TOTAL SYNTHESIS OF MARINE DERIVED NATURAL PRODUCTS: KEALIIQUINONE AND AGELIFERIN

by

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ABSTRACT

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An intramolecular Diels-Alder reaction of vinylimidazoles has been developed and applied towards the total synthesis of benzimidazole and tetrahydrobenzimidazole natural products, namely kealiiquinone and ageliferin. A common feature of many imidazole-containing natural products including those of interest in this dissertation is the presence of a 2-amino or 2-imidazolone substituent.

A novel method for achieving C2 substitutions has been developed employing chloro-containing reagents. *N*-Chlorosuccinimide in conjunction with a base has been used to give 2-imidazolones directly from imidazolium salts. This method was then optimized through the use of sodium hypochlorite. This chemistry provides the desired 2-imidazolones in yields ranging from 46-88% in benzimidazole and 37-80% in simple imidazole systems. *N*-Chlorocarbamates have been used to give 2-iminoimidazoles in yields of 72-95% in benzimidazoles and 36-71% in simple imidazoles providing an intermediate that can be easily deprotected to unmask a 2-amino functionality. The major advantage of these procedures is the ease of functionalization

without the need for strictly anhydrous conditions and the use of strong bases (i.e. BuLi and LDA).

An approach to the total synthesis of kealiiquinone is described involving construction of a propynoate system containing a vinyl imidazole and an aryl substituted, which then participates in the intramolecular Diels-Alder chemistry. The resulting heterocycle was oxidized to give a benzimidazole intermediate, and utilizing the sodium hypochlorite oxidation protocol efficiently gave a 2-imidazolone intermediate in 74% from which the remaining quinone ring was constructed. Among the final transformations of this synthesis was a benzyl deprotection involving triflic acid which was effective at removing the benzyl group as well as an aryl methyl ether resulting in isolation of a phenol intermediate. Methylation protocols have been investigated; however, conditions for a chemoselective *o*-methylation have yet to be identified.

The Diels-Alder chemistry of propynoates has been extended to access the core structure of ageliferin. Optimization was necessary at points leading up to and after the Diels-Alder reaction. The construction of a key ester intermediate was prepared from a propargyl alcohol directly using a cyano-mediated, tandem oxidation/substitution protocol greatly reducing the steps needed for the preparation of the propiolate derivative. Following the Diels-Alder reaction, the stereochemistry of ageliferin was set through a $Pd(OAc)_2$ catalyzed hydrogenation of a double bond to give an all *cis* tetrahydrobenzimidazole intermediate. Methanolysis of a lactone functionality of this intermediate provided the desired *trans* relationship as a result of epimerization of the ring opened product, intersecting a previous route.

Therefore, we have provided examples of benzimidazole and tetrahydrobenzimidazole alkaloids that can be accessed through an intramolecular Diels-Alder reaction of easily prepared propynoate systems. Continued studies are underway to achieve the final transformations of these target molecules, as well as utilizing intermediates of these syntheses to access related natural products, particularly in the case of ageliferin.

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LIST OF ABBREVIATIONS

APCI	Atmospheric-pressure chemical ionization
ATR	Attenuated total reflectance
Boc	*tert*-Butoxycarbonyl
BOM	Benzyloxymethyl
Bn	Benzyl
CDI	Carbonyl diimidazole
DCC	Dicyclohexylcarbodiimide
DBU	1,8-Diazabicycloundec-7-ene
DIBAL	Diisobutylaluminum hydride
DMAP	4-Dimethylaminopyridine
DMAS	*N*,*N*-Dimethylaminosulfonyl
DMF	*N*,*N*-Dimethylformamide
DMP	Dess-Martin periodinane
EDCI	1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide
ESI	Electrospray ionization
FT	Fourier Transform
GI_{50}	Concentration that yields 50% growth
HRMS	High resolution mass spectroscopy
HSV	Herpes simplex virus
IBX	2-Iodoxybenzoic acid

IC_{50}	Half maximal inhibitory concentration
im	Imidazole
LC_{50}	Median lethal dose
LDA	Lithium diisopropylamide
LiHMDS	Lithium *bis*(trimethylsilyl)amide
LTMP	Lithium 2,2,6,6-tetramethylpiperidide
MIC	Minimum inhibitory concentration
MOM	Methoxymethyl
μW	Microwave
NBS	*N*-Bromosuccinimide
NCS	*N*-Chlorosuccinimide
n-BuLi	*n*-Butyl lithium
NMR	Nuclear magnetic resonance
NOE	Nuclear Overhauser effect
PCC	Pyridinium chlorochromate
PLE	Pig liver esterase
PMB	*p*-Methoxybenzyl
PPA	Polyphosphoric acid
SEM	2-Trimethylsilylethoxymethoxy
t-BuLi	*tert*-Butyl lithium
TBAF	Tetra-*n*-butylammonium fluoride
TBS	*tert*-Butyldimethylsilyl
TGI	Total growth inhibition
THF	Tetrahydrofuran

TfOH	Trifluoromethanesulfonic acid
TMS	Trimethylsilyl
Tris	2,4,6-Triisopropylbenzenesulfonyl
Ts	*p*-Toluenesulfonyl
p-TSA	*p*-toluenesulfonic acid
δ	Chemical shift (NMR)

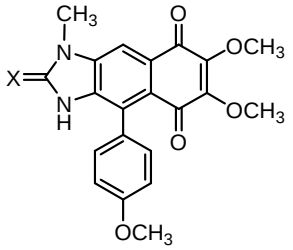
CHAPTER 1

INTRODUCTION

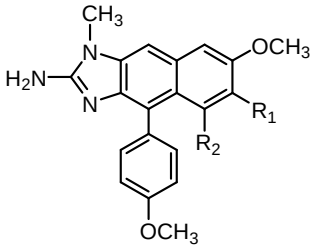
Benzimidazole-containing natural products have been isolated from marine sponges of the *Leucetta* genus, while those containing a tetrahydrobenzimidazole belong to the oroidin family of bromopyrrole alkaloids.[1] Interesting and potentially therapeutic biological activities are often associated with these alkaloids, as well as synthetically challenging structural motifs making them ideal synthetic targets.

1.1 Benzimidazole Natural Products

A number of imidazole-containing alkaloids including those having a benzimidazole framework have been isolated from the marine sponge *Leucetta chagosensis*. In addition, an amino or imidazolone substituent is typically located at C2 of the imidazole.[2] The kealiiquinones (**1-2**) and kealiinines (**3a-c**) are representative of this class of natural products that have been isolated from *Leucetta* sponges (Figure 1.1).



1: Kealiiquinone X = O
2: 2-Amino-2-deoxy-kealiiquinone X = NH



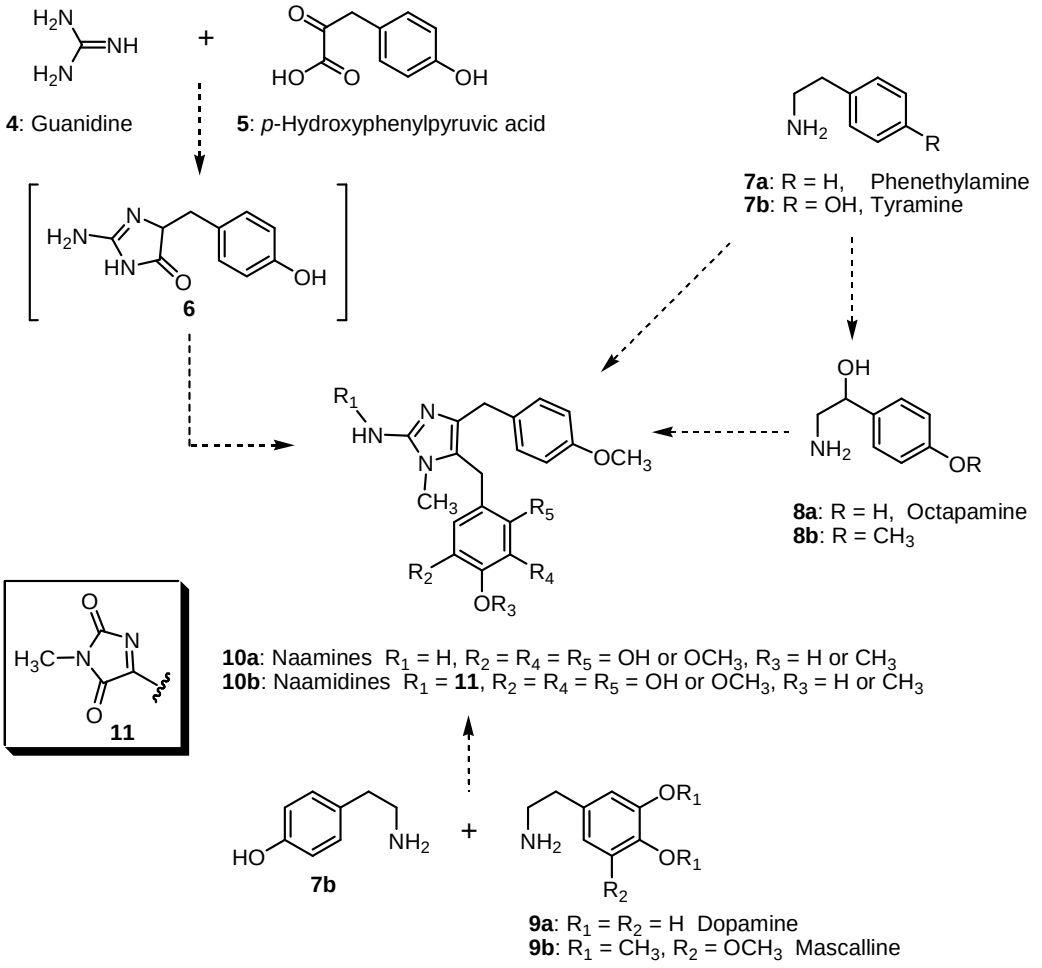
3a: Kealiinine A R_1 = OH, R_2 = H
3b: Kealiinine B R_1 = OCH_3, R_2 = H
3c: Kealiinine C R_1 = OCH_3, R_2 = OCH_3

Figure 1.1 Benzimidazole natural products from *Leucetta* sponges

Kealiiquinone (**1**) was isolated as long, red needles in 1990 by Scheuer and co-workers from marine sponges harvested off the coast of Saipan and Guam in Micronesia.[3] It was found

to be inactive against initial toxicity assays including KB cells and HSV II virus, but no broad scale evaluation was or subsequently been performed. 2-Deoxy-2-aminokealiiquinone (**2**) was isolated in 1997 by Schmitz and co-workers also from a sponge taken from Micronesia, but no biological activity has been reported for this alkaloid.[4]

Kealiinine A-C (**3a-c**) were isolated in 2004 by Proksch and co-workers from sponges collected in Indonesia.[5] Kealiinine A (**3a**) was obtained as a yellowish brown powder and was found to give a 50% mortality rate in brine shrimp assay, *Artemia salina*, at a concentration of 20 µg/mL. No biological activity data has been reported for the other kealiinines (**3b-c**). It

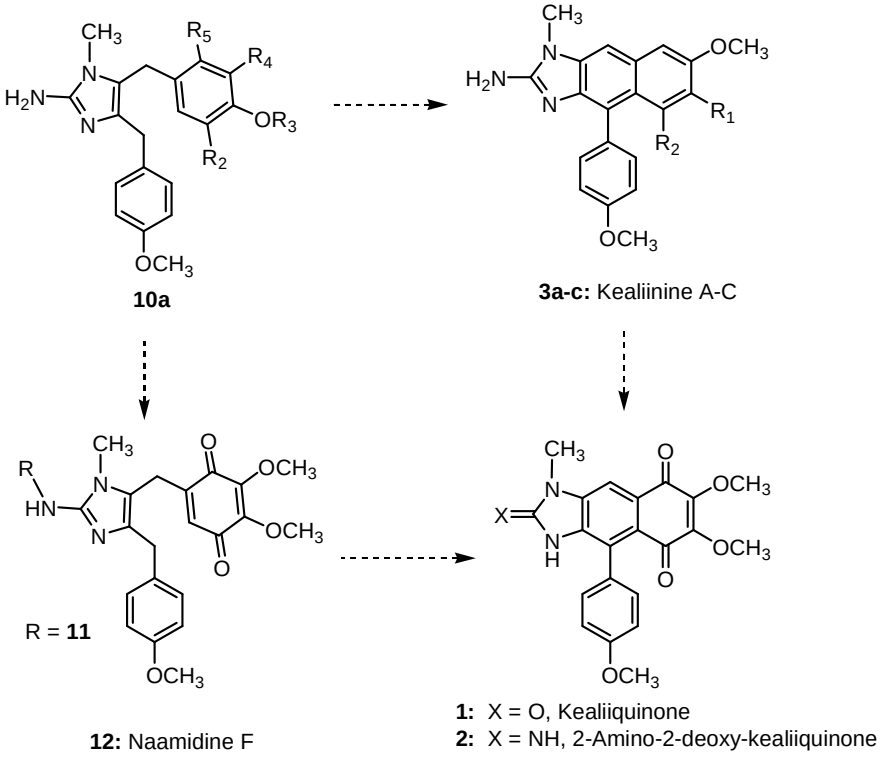


Scheme 1.1

should be noted that recent studies emerging from our lab towards their total syntheses are suggesting that their reported structures may in fact be incorrect.

Biosynthetically, the kealiiquinone (**1-2**) and kealiinines (**3a-c**) are proposed to arise through cyclization and oxidation events of *Leucetta* alkaloids having 4,5-disubstituted bis-benzyl frameworks characteristic of the naamines (**10a**) and naamidines (**10b**).[2] Several pathways for the biosynthesis of **10a-b** have been proposed; however, no experimental evidence has been presented to support these hypotheses (Scheme 1.1).

Condensation of guanidine (**4**) and *p*-hydroxyphenylpyruvic acid (**5**) leading to intermediate **6**, followed by further substitution is one possible biosynthetic pathway that results in the general naamine (**10a**) framework.[6, 7] Another possible pathway involves the coupling of



Scheme 1.2

two units of phenethylamine (**7a**) or tyramine (**7b**) to give **10a**.[8] An oxidation event to give octapamine (**8a**) or **8b** prior to coupling can also lead to **10a**. It has also been proposed that tyramine (**7b**) can couple with dopamine (**9a**) or mascalline (**9b**) to give the naamine framework.[9]

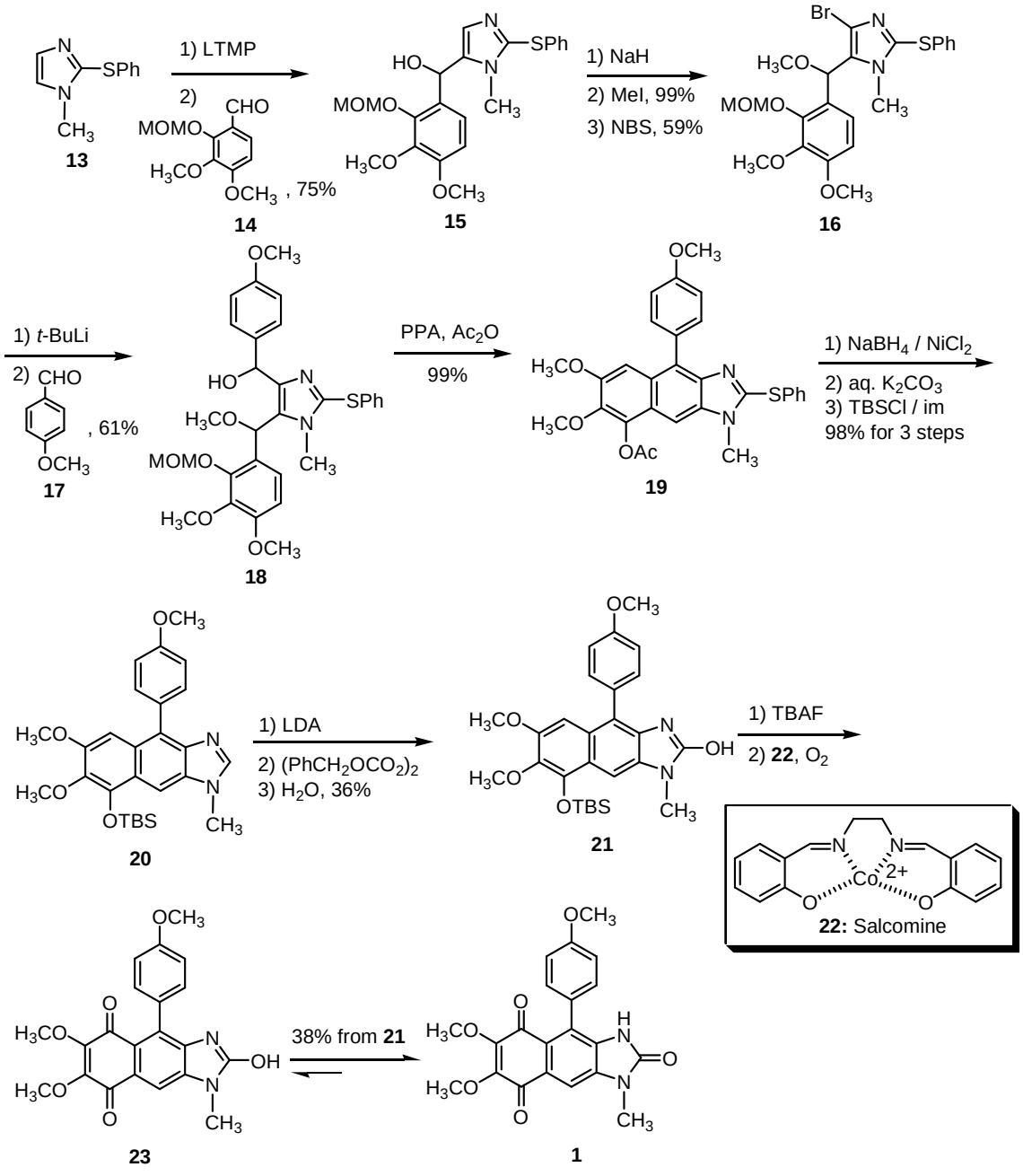
The biosynthesis of kealiinine A-C (**3a-c**) can then proceed via an oxidative cyclization of **10a**. The kealiiquinones (**1-2**) can be produced by oxidation of the kealiinines (**10a**→**3**→**1**-**2**) or by oxidation of **10a** to **12** prior to cyclization (**10a**→**12**→**1-2**) (Scheme 1.2).[10]

1.2 Total Synthesis of Kealiiquinone and a Regioisomer

A total synthesis of kealiiquinone was reported by Ohta and co-workers in 1995 starting from 2-thiophenylimidazole (**13**) (Scheme 1.3).[11, 12] Chemoselective C5 lithiation with lithium tetramethylpiperidide (LTMP) followed by addition of aldehyde **14** resulted in the formation of alcohol **15**. This alcohol was then converted to methyl ether **16** upon treatment with NaH, followed by MeI, and C4 bromination with NBS. Metal-halogen exchange with *t*-BuLi, then addition of *p*-anisaldehyde (**17**) resulted in C4 alkylation to give alcohol **18**. Acid-mediated cyclization of **18** to give naphthimidazole **19** was achieved with polyphosphoric acid (PPA); however, the acidic conditions also caused the MOM deprotection of the phenolic hydyroxyl group which was re-protected as the acetate. Desulfurization was accomplished with a $NaBH_4/NiCl_2$ complex, followed by deprotection of the acetate, then re-protection of the phenol with TBSCl to give **20**. C2 functionalization was achieved by lithiation with LDA, then quenching with dibenzyl peroxydicarbonate, and finally addition of water to give **21**. TBS deprotection and oxidation with salcomine (**22**) provided kealiiquinone (**1**) in 11 steps and 2.6% overall yield.

In his synthesis, Ohta has taken advantage of the well-documented C-H acidity trends of imidazoles allowing substitutions to occur following the order of C5, then C4 of C2 substituted systems.[13, 14] In addition, C2 functionalization was accomplished through a common, but poor

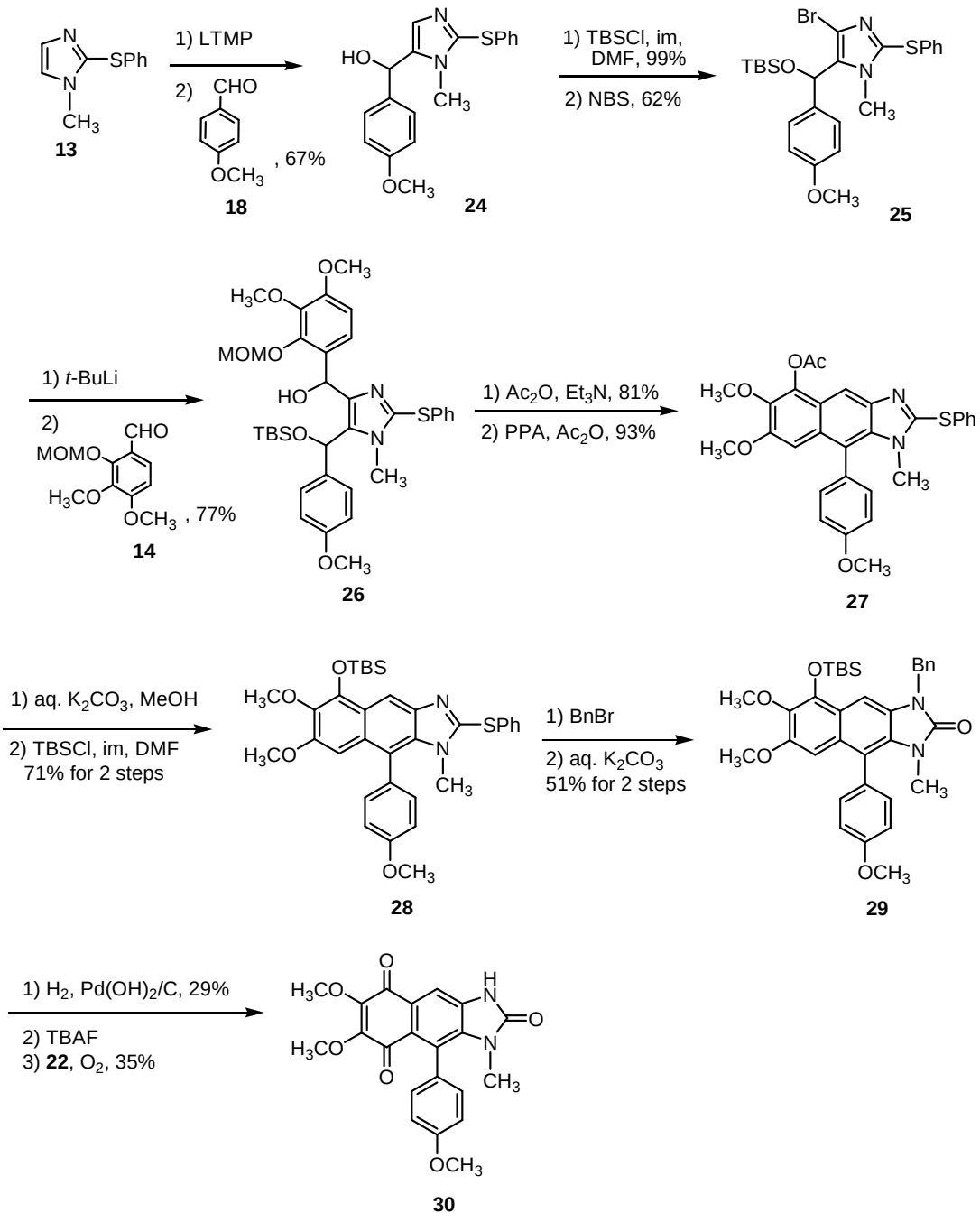
yielding metalation/electrophilic quench sequence involving a peroxide to ultimately lead to the desired imidazolone.



Scheme 1.3

The synthesis of a regioisomer of kealiiquinone was reported by Ohta and co-wokers in 2001.[15] In this synthesis, a new method of C2 functionalization utilizing imidazolium salts was developed and applied instead of the previously reported low yielding procedure. Following a similar approach to that applied to the synthesis of kealiiquinone, 2-thiophenylimidazole (**13**) was treated with LTMP, followed by *p*-anisaldehyde (**17**) to give the C5 alcohol **24** that was converted to the silyl ether, and brominated with NBS to give **25** (Scheme 1.4). Metal-halogen exchange and addition of aldehyde **14** gave alcohol **26** which was protected as the acetate prior to cyclization with PPA and Ac_2O to give naphthimidazole **27**. The acetate protecting group was exchanged for TBS providing intermediate **28**. Treatment of **28** with BnBr produced an imidazolium salt that when heated in aq. K_2CO_3 resulted in substitution of the thiophenyl C2 substituent to provide the 2-imidazolone **29**. Hydrogenation to remove the benzyl group, deprotection of the TBS ether, and then oxidation with salcomine (**22**) provided the regioisomer of kealiiquinone **30** in 12 steps and 0.62% overall yield.

Both synthetic kealiiquinone (**1**) and the regioisomer **30** were screened against 39 human cancer cell lines by the Japanese Foundation for Cancer Research.[15] Among the 39 cell lines screened, five were breast cancers (HBC-4, BSY-1, HBC-5, MCF-7 and MDA-MB-231), six were central nervous system cancers (U251, SF-268, SF-295, SF-539, SNB-75, and SNB-78), five were colon cancers (HCC2998, KM-12, HT-29, HCT-15, and HCT-116), seven were lungs cancers (NCI-H23, NCI-H226, NCI-H522, NCI-H460, A549, DMS273, and DMS114), five were ovarian cancers (OVCAR-3, OVCAR-4, OVCAR-5, OVCAR-8, and SK-OV-3), six were stomach cancers (St-4, MKN1, MKN7, MKN28, MKN45, and MKN74), two were renal cancers (RXF-631L and ACHN), two were prostate cancers (DU-145 and PC-3), and a melanoma (LOX-IMVI). The mean concentrations to achieve GI_{50}, TGI, and LC_{50} against the panel for kealiiquinone (**1**) and regioisomer **30** were 39.8, 79.4, and 97.7 μM, and 51.3, 91.2, and 100 μM. It was also noted by the Japanese Foundation for Cancer Research that both compounds appear to work by a unique mechanism of action presumably from the gathered reactivity profile.

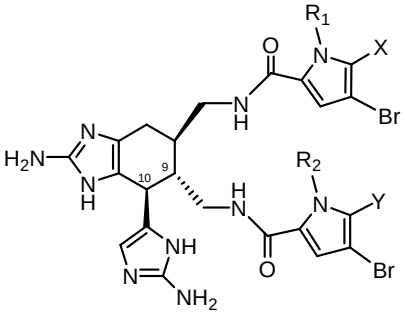


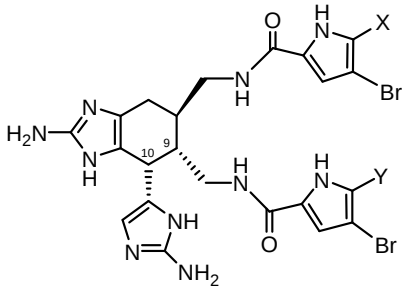
13 **18** **24** **25** **14** **26** **27** **28** **29** **30**

Scheme 1.4

1.3 Tetrahydrobenzimidazole Natural Products

A handful of tetrahydrobenzimidazole containing alkaloids belonging to the oriodin family of natural products have been isolated from a number of marine sponges including those of the *Agelas, Axinella* and *Astrosclera* genera.[1] Among them, the ageliferins (**31a-h**) and nagelamides E-G (**32a-c**) have received increasing attention in recent years. The two differ only in the stereochemistry at C10, and are characterized by the bromopyrrole side-chains and 2-aminoimidazoles.





31a: Ageliferin $R_1 = R_2 = X = Y = H$
31b: Bromoageliferin $R_1 = R_2 = X = H$, Y = Br
31c: Dibromoageliferin $R_1 = R_2 = H$, X = Y = Br
31d: *N*(1')-Methylageliferin R_1 = Me, $R_2 = X = Y = H$
31e: *N*(1),*N*(1')-Dimethylageliferin $R_1 = R_2$ = Me, X = Y = H
31f: *N*(1')-Methyl-2-bromoageliferin R_1 = Me, $R_2 = X = H$, Y = Br
31g: *N*(1')-Methyl-2'-bromoageliferin R_1 = Me, R_2 = H, Y = H, X = Br
31h: *N*(1')-methyl-2,2'-dibromoageliferin R_1 = Me, R_2 = H, X = Y = Br

32a: Nagelamide E X = Y = H
32b: Nagelamide F X = Br, Y = H
32c: Nagelamide G X = Y = Br

Figure 1.2 Oroidin-derived tetrahydrobenzimidazole natural products

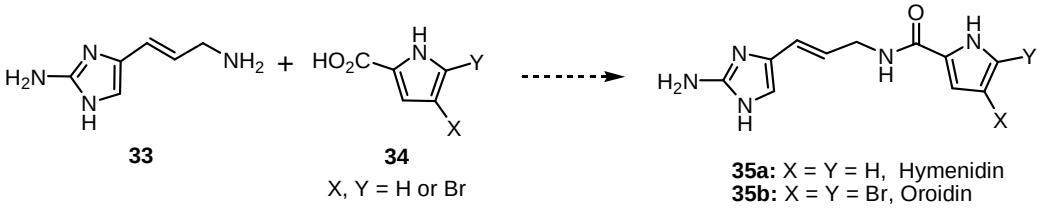
Ageliferin (**31a**), bromoageliferin (**31b**), and dibromoageliferin (**31c**) were isolated and their structures elucidated in 1990 by Kobayashi and co-workers from a sponge of the *Agelas* species harvested off the coast of Okinawa, Japan,[16] and Rinehart and co-workers from a sponge taken from the Caribbean in 1991.[17] These compounds were suspected of being actomyosin ATPase activators, and therefore, were tested for ATPase activity of myofibrils from rabbit skeletal muscle. It was found that compounds **31a-c** elevated activity to 150, 190, and 200% of the control at concentrations of 30, 1, and 1 μM.[16] The *N*-methyl derivatives (**31d-h**) were isolated in 1996 by Williams and Faulkner.[18]

The nagelamides E-G (**32a-c**) were isolated in 2004 also by Kobayashi and co-workers and also from an Okinawan sponge.[19] In this isolation report, nagelamides E-G (**32a-c**) and the ageliferins (**31a-c**) were found to be active against Gram-positive bacteria, *Micrococcus luteus* and *Bacillus subtillis*, and the Gram-negative bacterium *Escherichia coli* (Table 1.1). Nagelamide G (**32c**) also showed inhibitory activity against protein phosphatase type 2A with an IC_{50} = 13 μM.

Table 1.1 Antibacterial activity of nagelamides E-G and ageliferins (MIC, μg/mL)

Compd	*M. luteus*	*B. subtillis*	*E. coli*
32a	4.17	16.7	33.3
32b	4.17	16.7	33.3
32c	2.08	16.7	33.3
31a	16.7	33.3	>33.3
31b	>33.3	>33.3	>33.3
31c	4.17	8.33	33.3

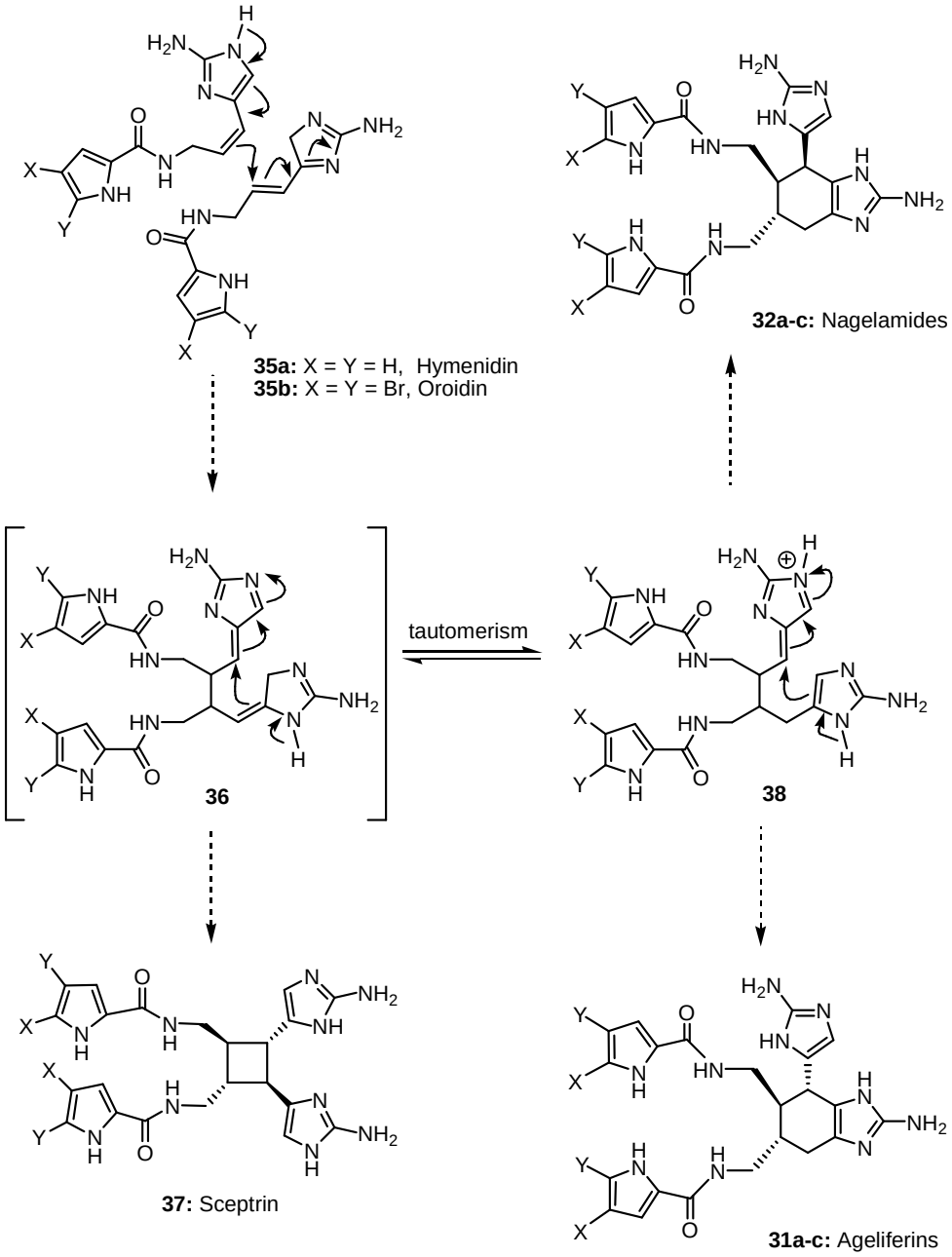
The biosynthesis of compounds **31a-h** and **32a-c** are proposed by Al-Mourabit to proceed through a head-to-head dimerization of two different tautomeric forms of hymenidin (**35a**) and/or oroidin (**35b**), two bromopyrrole imidazoles arising from the coupling of **33** and **34** (Scheme 1.5 and 1.6).[20] Cyclization of intermediate **36** can then lead to sceptrin (**37**), or intermediate **36** can tautomerize to intermediate **38**. This can then undergo a similar cyclization to give the ageliferins (**31a-c**).



33 **34**
X, Y = H or Br

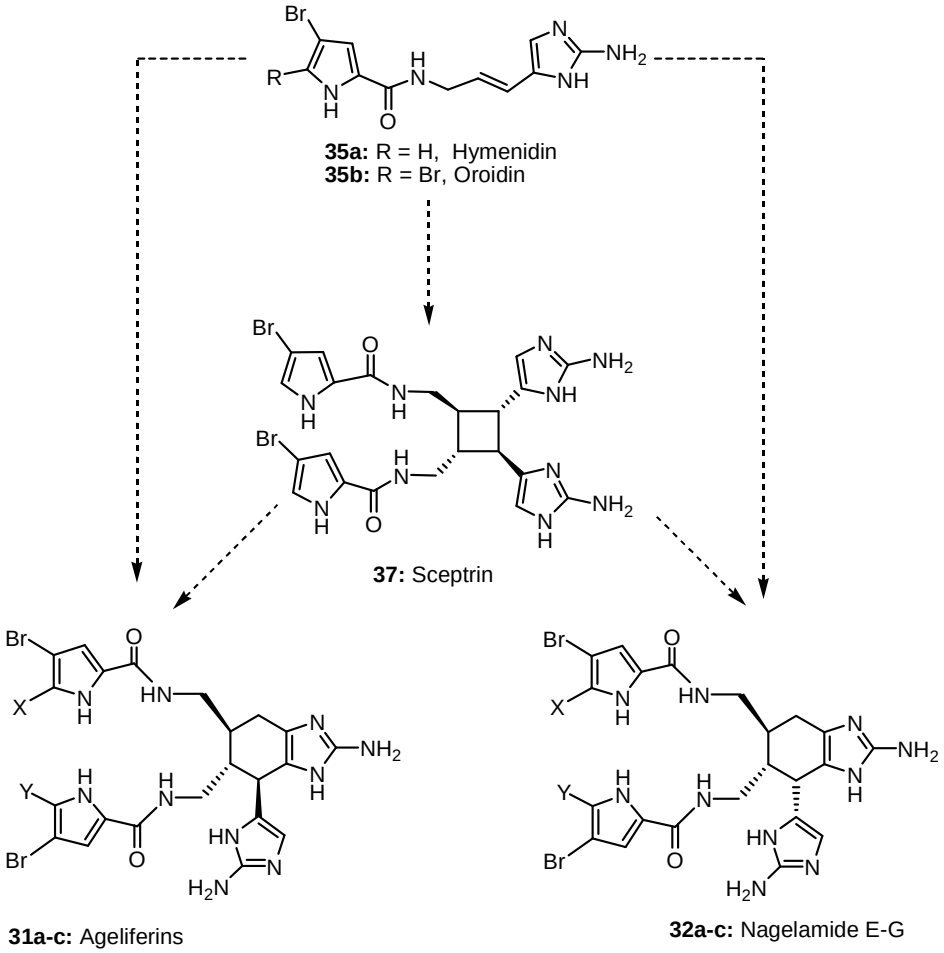
35a: X = Y = H, Hymenidin
35b: X = Y = Br, Oroidin

Scheme 1.5


35a: X = Y = H, Hymenidin
35b: X = Y = Br, Oroidin
32a-c: Nagelamides
tautomerism
36
38
37: Sceptrin
31a-c: Ageliferins

Scheme 1.6

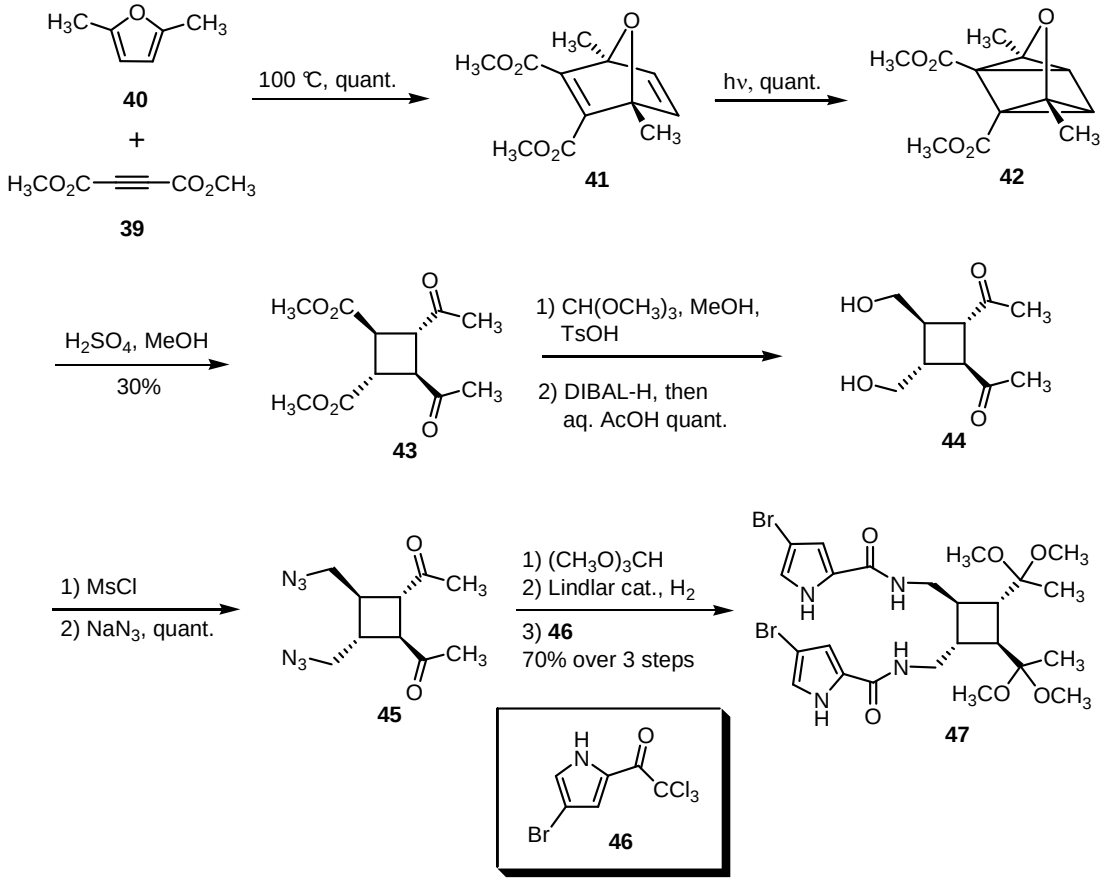
It has also been proposed that the alkaloids of interest can form via a direct, net [4+2] cycloaddition of some combination of hymenidin (**35a**) and/or oroidin (**35b**) (Scheme 1.7).[16, 19, 21] This has been attempted by Lindel, however, cyclooroidin was produced from these studies.[22] Baran has also proposed, based on the total synthesis of ageliferin (**31a**) and nagelamide E (**32a**), that the common precursor of these, and other dimeric alkaloids of this family, may actually be sceptrin (**37**) which undergoes a [1,3]-rearrangement in this case to provide **31a** and **32a**.[23] It should be pointed out that there is no evidence for any of their proposals from experimental studies.



Scheme 1.7

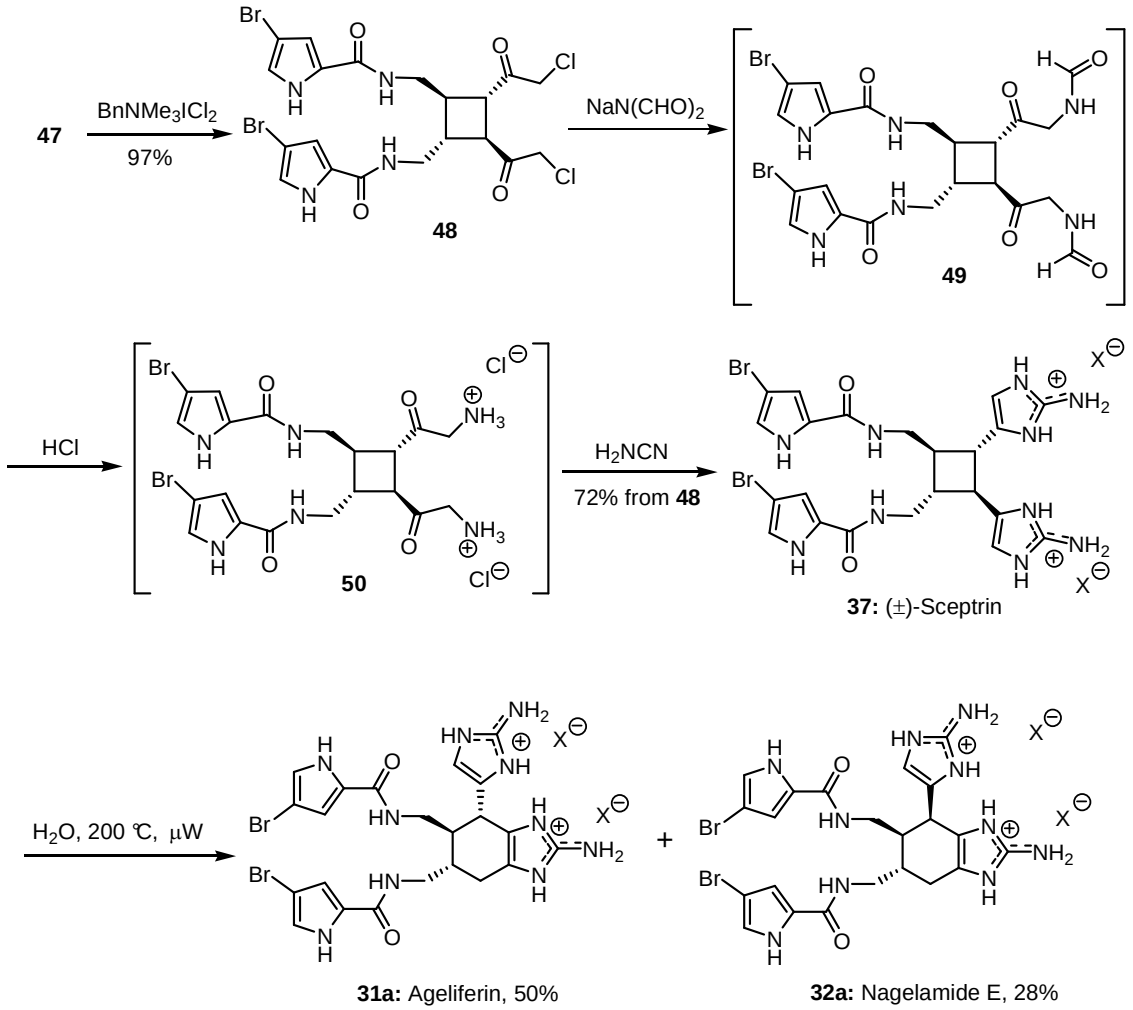
1.4 Baran's Total Synthesis of Ageliferin and Nagelamide E

The total syntheses of the oroidin alkaloids have been led in recent years by Baran and co-workers. Ageliferin (**31a**) and nagelamide E (**32a**) were reported in 2007 through a rearrangement and cyclization of sceptrin (**37**).[21, 23] The synthesis began with a Diels-Alder reaction of 2,5-dimethylfuran (**40**) and dimethyl acetylenedicarboxylate (DMAD) (**39**) to yield bicycle **41** (Scheme 1.8). A [2+2] cyclization of **41** led to intermediate **42** which when treated with H_2SO_4 caused a fragmentation that resulted in the all *trans*-cyclobutane **43**. Protection of the ketones, followed by reduction of the esters with DIBAL-H gave diol **44**. The diol was



Scheme 1.8

converted to the mesylate and then displaced with NaN_3 to give azide **45**. Protection of the ketones, reduction of the azides with Lindlar catalyst, and then acylation with bromopyrrole **46** gave cyclobutane **47**.

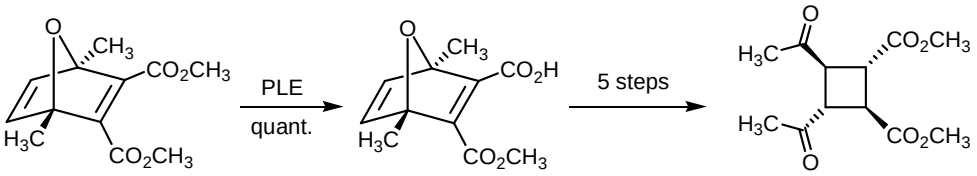


Scheme1.9

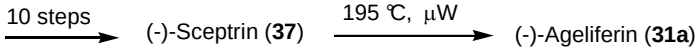
Sceptrin (**37**) was then completed by taking intermediate **47** and treating it with benzyl trimethylammonium dichloriodate to give the bis-chloroketone **48** (Scheme 1.9). The 2-aminoimidazoles were then constructed using sodium diformamide to give **49** which was then hydrolyzed with HCl giving **50**. Lastly, cyanamide was used to complete the ring construction, and the total synthesis of (±)-sceptrin (**37**). Microwave heating of the bis-acetate salt of sceptrin

(**37**) then gave a 50% yield of ageliferin (**31a**), 28% yield of nagelamide E (**32a**), and 12% recovered starting material.

In addition to the racemic total syntheses, an enantioselective synthesis of (-)-sceptrin (**37**) and (-)-ageliferin (**31a**) has been reported, also by Baran, based on the construction of an enantiopure cyclobutane intermediate **(+)-43** via enzymatic resolution of **41** that led to (-)-sceptrin (Scheme 1.10).[24] The microwave-assisted [1,3]-rearrangement and cyclization of (-)-sceptrin then leads to (-)-agleiferin which was found to give consistent circular dichroism (CD) data as the isolated material.



41 **(-)-42** (75% *ee*) **(+)-43** (>95% *ee*)



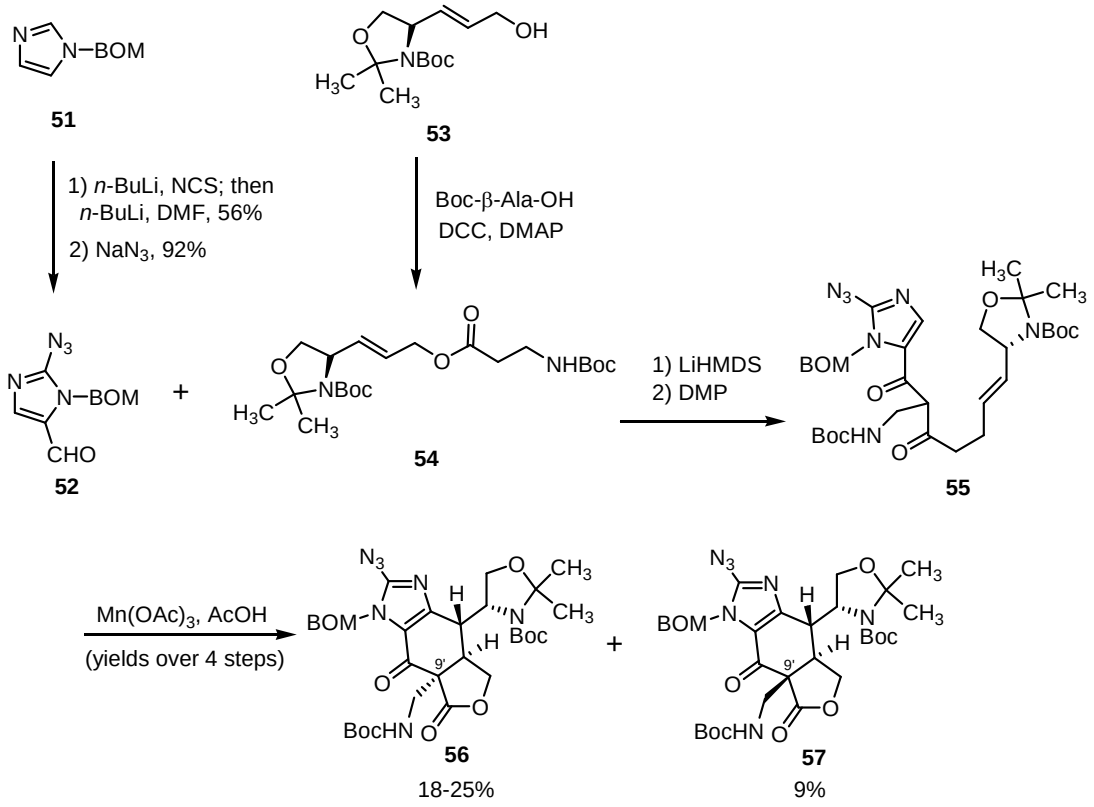
Scheme 1.10

1.5 Chen's Total Synthesis of Ageliferin

In September of 2011, Chou Chen and co-workers reported the asymmetric total synthesis of *ent*-ageliferin (**31a**) through a Mn(III)-mediated radical cyclization that allowed for the construction of the cyclic framework of ageliferin.[25] This synthesis supports the proposed biosynthetic net [4+2] cyclization theory through a single-electron transfer to affect the dimerization of two hymenidin (**35a**) molecules.

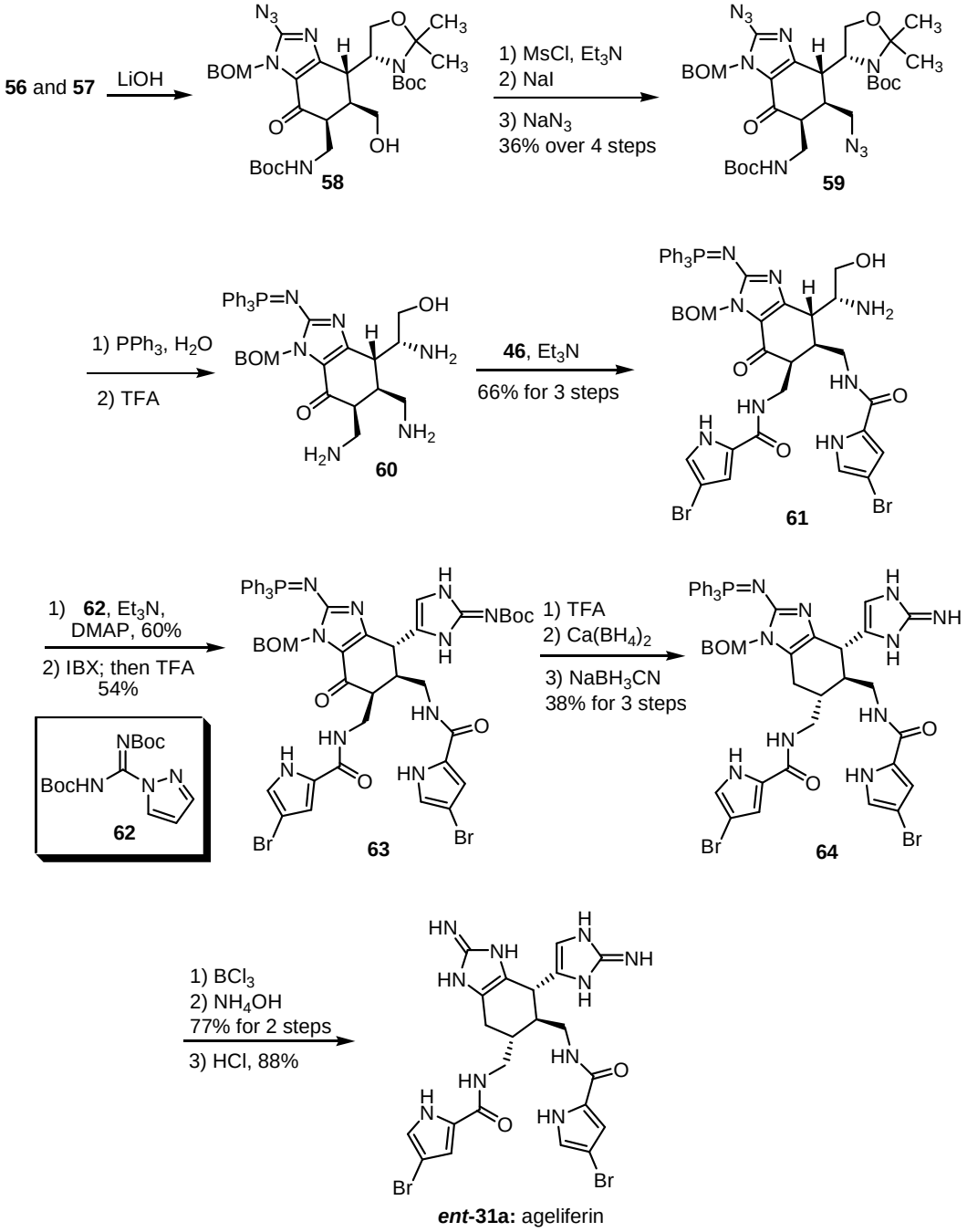
The synthesis began with the preparation of the 2-azidoimidazole **52** from BOM-protected imidazole **51** (Scheme 1.11). The C2 position of imidazole **51** was first chlorinated by treatment with BuLi followed by NCS. The 2-chloro intermediate was then treated with

additional BuLi followed by DMF to give the formylated product in a one-pot operation. Finally, substitution with NaN_3 provided azide **52**. Separately, allylic alcohol **53** was coupled with Boc-β-Ala-OH to give **54**. The Aldol condensation of **54** and **52** was achieved with LiHMDS and the resulting alcohol was oxidized with Dess-Martin periodinane (DMP) to give **55**.



Scheme 1.11

The construction of intermediate **55** was a critical step since it set up the $Mn(OAc)_3$ mediated cyclization developed to provide cycloadducts **56** and **57**. Both diastereomers (**56** and **57**) isolated from the cyclization reaction result in **58** after decarboxylation due to an epimerization at C9' (Scheme 1.12). The alcohol of **58** was converted to azide **59** by first converting the alcohol to the mesylate, followed by conversion to the iodide and lastly substitution with NaN_3.



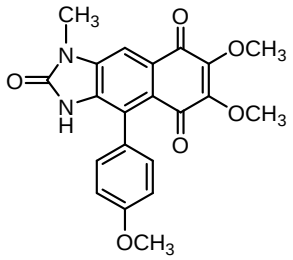
Scheme 1.12

The azido groups were then reduced by a Staudinger reaction, however, the C2 imide was more stable to hydrolysis and was left as a protecting group. Triamine **60** was obtained

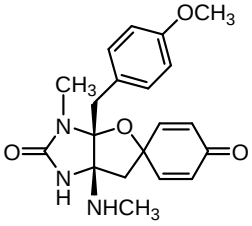
following treatment of **59** with TFA, and was then coupled with pyrrole **46** to give the side-chains and intermediate **61**. The remaining imidazole ring was constructed from condensation of **61** with guanidine **62** which cyclized to provide **63** after oxidation with IBX. Acid catalyzed epimerization and reduction of the ketone resulted in **64** of which the BOM group was removed in two steps; first with BCl_3, then basic hydrolysis with NH_4OH. The imide was then hydrolyzed with HCl to give ageliferin (**31a**) which had a CD spectrum consistent with *ent*-**31a**.

1.6 2-Imidazolone Natural Products

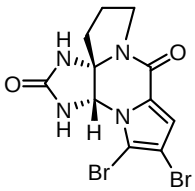
The 2-aminoimidazole moiety has been recognized as a common feature among imidazole containing marine alkaloids and has received substantial recognition within the synthetic community;[1] however, examples of 2-imidazolone containing natural products remain scarce. To date a handful of natural products having a 2-imidazolone group have been isolated including kealiiquinone (**1**), spiroleucettadine (**65**),[26, 27] dibromophakellstatin (**66**),[28] and agelastatin A (**67**)[29] (Figure 1.3).



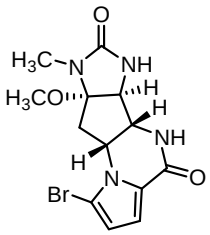
1: Kealiiquinone



65: Spiroleucettadine



66: Dibromophakellstatin



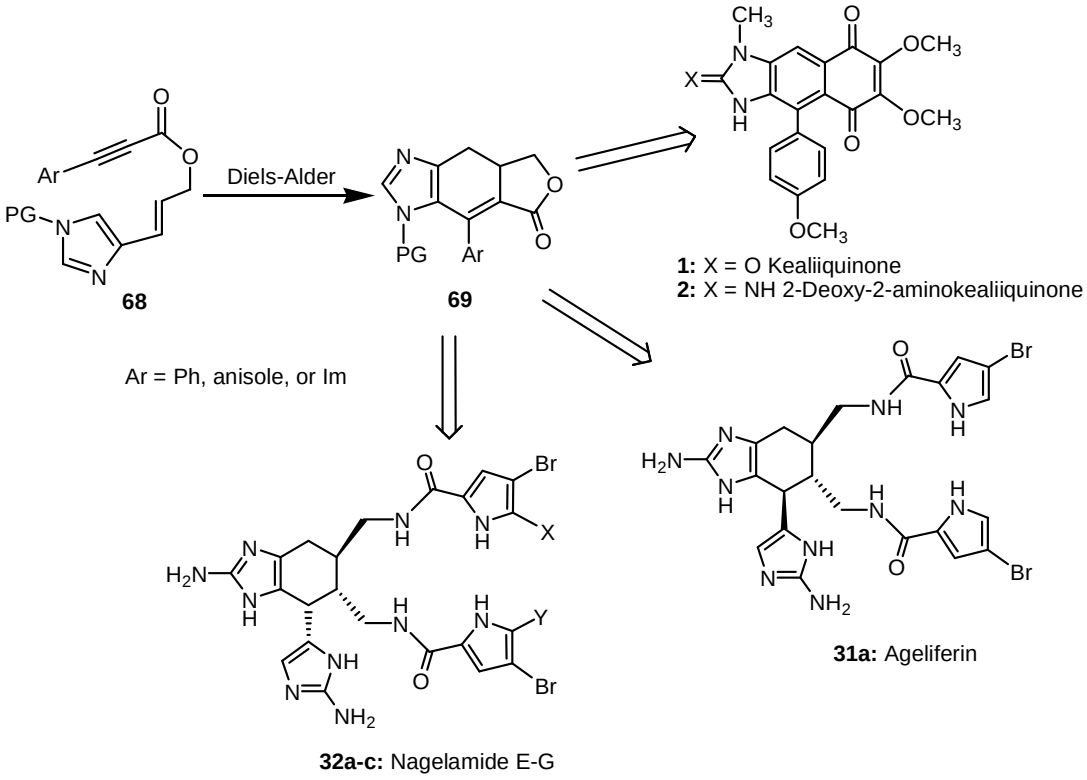
67: Agelastatin A

Figure 1.3 2-Imidazolone containing natural products

Synthetic routes to access 2-imidazolones have been developed and mostly involve condensation reactions to construct the functionalized ring rather than functionalization of a pre-existing imidazole (see chapter 2). Ohta's total syntheses of kealiiquinone (**1**)[11, 12] and regioisomer **30**[15] are excellent examples of the latter.

1.7 Our Approach to Kealiiquinone and Ageliferin

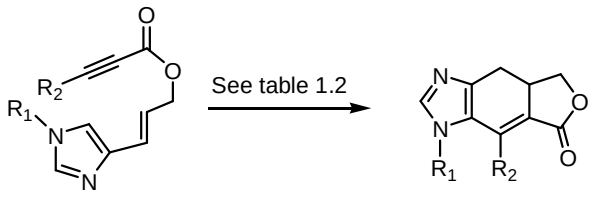
Our approach to the benzimidazole and tetrahydrobenzimidazole frameworks of the introduced *Leucetta* and oroidin alkaloids is centered on an intramolecular Diels-Alder (IMDA) reaction of a propynoate system such as **68** to access heterocycles such as **69** which can be further elaborated to achieve the targeted natural products (Scheme 1.13).



Scheme 1.13

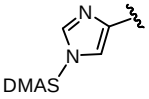
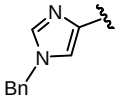
The use of a vinylic imidazole as a diene in Diels-Alder reactions has been demonstrated by us in a number of examples.[30] In addition, several examples of IMDA reactions involving propynoates have been described by us.[31-34] For example, propynoates **68a-c** were shown to successfully undergo cyclization to afford **69a-c** in yields ranging from 30-85% (Table 1.2). Bis-imidazole substrates have also been shown to participate in the Diels-Alder chemistry as seen in the cyclization of **68d-e** to give **69d-e**.[33] These examples demonstrate that the Diels-Alder reactions can directly lead to the general frameworks needed

for the target molecules, the kealiiquinones (**1-2**) and the ageliferin family. In addition, the lactone provides functionality that can be manipulated to construct desired ring systems or for incorporation of the pyrrole-carboxamide side-chains.



68a-e **69a-e**

Table 1.2 Examples of Intramolecular Diels-Alder reactions of propynoates

68	R_1	R_2	Conditions (℃/h) [a]	**69** (%)
a	DMAS	H	180/48	30
b	DMAS	Ph	130/48	73
c	Bn	Ph	130/24	85
d	Bn		120/12	82
e	Bn		130/12	70

a) Benzene was the solvent used in examples **a-c**. Dichloromethane was the solvent used in examples **d-e**.

CHAPTER 2

C2-SUBSTITUTIONS OF IMIDAZOLES

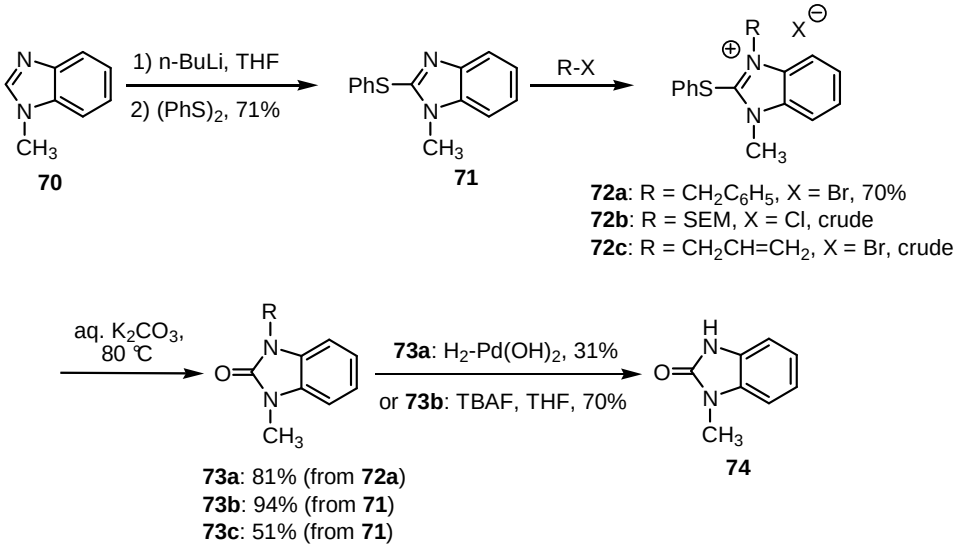
Methods for C2 functionalization of imidazoles have been evolving for some twenty years; one method involving metalation followed by quenching with an electrophile based on chemistry described by Katritzsky[35] and Lipshutz[36] has been popular in our group. However, issues associated with the use of strong and potentially nucleophilic bases, as well as functional group compatibility, limit this method's effectiveness in certain cases. More recently, substitution employing transition metal catalyzed processes based on the premise of C-H activation have been developed.[37-41] However, these employ amines of limited use for further elaboration and generally require harsh reaction conditions (>100 ℃) which limit functional group compatibility. This chapter will focus on the development of a new strategy for C2-substitutions in imidazoles leading to 2-imidazolones and 2-aminoimidazoles using an approach developed to side-step the need for metalation in these transformations.

2.1 Synthesis of 2-Imidazolones

A number of natural products containing a 2-imidazolone moiety have been identified including kealiiquinone (**1**), spiroleucettadine (**65**), dibromophakellstatin (**66**), and agelastatin A (**67**) (see Fig. 1.3). Methods for preparing 2-imidazolones from imidazoles are limited and often inefficient. One such method involves lithiation of the imidazole followed by oxidation of the organometallic with a peroxide such as $(TMSO)_2$[36] or $(PhCO_2)_2$.[11] In addition, benzimidazoles have been shown to undergo C2 oxidation when treated with Ac_2O and H_2O_2 at reflux.[42] However, both procedures suffer from low yields and relatively harsh reaction conditions. The more popular approach is to construct the functionalized ring via condensation reactions. 2-Benzimidazolones have been successfully prepared from benzene-1,2-diamines with

phosgene,[43-47] triphosgene,[48, 49] or carbonyl diimidazole (CDI).[50, 51] Examples involving transition metal catalyzed methods have also been developed,[39-41] as well as base-induced cyclizations of aryl ureas.[52]

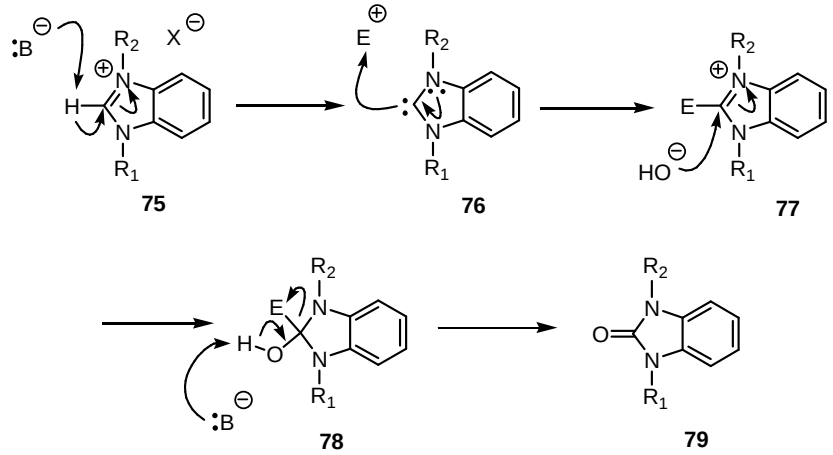
Ohta has provided the most promising example of 2-imidazolone construction from benzimidazoles during the development of his synthesis of a regioisomer of kealiiquinone.[15] Benzimidazolium salts of 2-thiobenzimidazoles, **72a-c**, were hydrolyzed to give 2-imidazolones **73a-c** in moderate to good yields depending on the protecting groups employed (Scheme 2.1). While this approach proved to be effective, it required the pre-functionalization of the C2 position via a lithiation of **70**, a step we wished to avoid if at all possible, as well as heating at elevated temperatures in the presence of a relatively strong base to affect hydrolysis of **72**.



Scheme 2.1

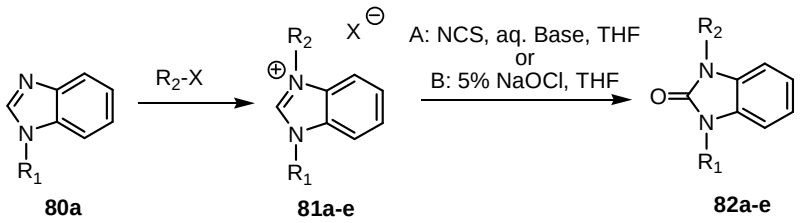
What is demonstrated nicely in this chemistry is that nucleophilic substitution of imidazolium salts can occur at C2 if a suitable leaving group is in that position and that deprotection of at least one *N*-substituent can be accomplished leading to synthetically useful 2-imidazolones such as **74**. Taking this into consideration, we turned our attention to the benzimidazolium salts themselves with the anticipation of directly functionalizing the C2 position

by taking advantage of the increased acidity of the proton in the C2 position.[53-55] We hypothesized that by treating benzimidazolium salt **75** with a base we could generate a stabilized carbene **76** that could then be trapped with a suitable electrophile leading to **77**. Nucleophilic attack with hydroxide providing **78**, and then elimination of the electrophile would give imidazolone **79**, thereby, inducing the transformation in one pot and without the need for pre-functionalization (Scheme 2.2).



Scheme 2.2

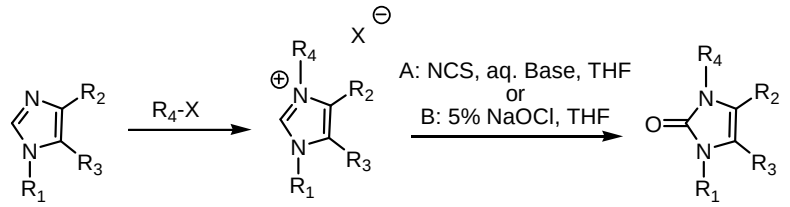
To test this hypothesis, benzimidazolium salts **81a-b** were prepared and treated with a variety of bases including NaH, Na_2CO_3, K_2CO_3, and NaOH in the presence of *N*-chlorosuccinimide (NCS), an electrophilic source of chlorine, in THF at room temperature, and found that the expected 2-imidazolones **82a-b** were isolated under these mild reaction conditions in yields ranging from 53-84% (Table 2.1). From this initial screen of bases, it was decided to use 2M NaOH as the standard base in the development of this methodology since it provided the best yields of **82a-b**. Analogs **81c-e** with varying protecting groups, including Bn, MOM, and SEM were prepared and found to be tolerant of the reaction conditions producing imidazolones **82c-e** in moderate yields; however, the base employed in these examples was 1M Na_2CO_3. It was found that the use of this base minimized the suspected hydrolysis and decarboxylation of the urea, and other unidentified by-products observed using NaOH.



80a **81a-e** **82a-e**

Table 2.1 Results of benzimidazole C2 oxidations

	R_1	R_2	X	**81** (%)	**82** (%)	
					A	B
a	Bn	Me	I	92	84	85
b	PMB	Me	I	82	83	81
c	Bn	Bn	Cl	54	50	88
d	Me	MOM	Cl	91	46	81
e	Me	SEM	Cl	96	49	79



80b **81f-j** **82f-j**

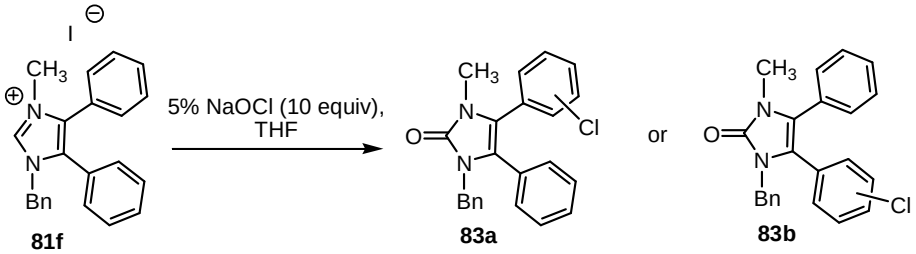
Table 2.2 Results of simple imidazole C2 oxidations

	R_1	R_2	R_3	R_4	X	**81** (%)	**82** (%)	
							A	B
f	Bn	Ph	Ph	Me	I	81	86	79
g	Me	Ph	Ph	Me	I	92	72	71
h	Me	H	H	Bn	Cl	84	37	39
i	Me	H	H	SEM	Cl	91	48	36
j	Bn	Ph	H	Me	I	94	52	67

Note: 2M NaOH was the base used except in the preparation of **82c-e** in which case 1M Na_2CO_3 was used

As reaction conditions were being optimized, alternative electrophilic sources of chlorine were also being investigated, and we found that household bleach (~5% aq. NaOCl) was an excellent reagent to affect this transformation. Treating the salts **81a-e** in THF with an excess of bleach (~10 equiv) resulted in oxidation to the imidazolones **82a-e** in increased yields and enhanced purities. The chemistry was then extended to successfully include simple imidazoles, **81f-j**, of varying substitution on the 4- and 5- positions (Table 2.2).

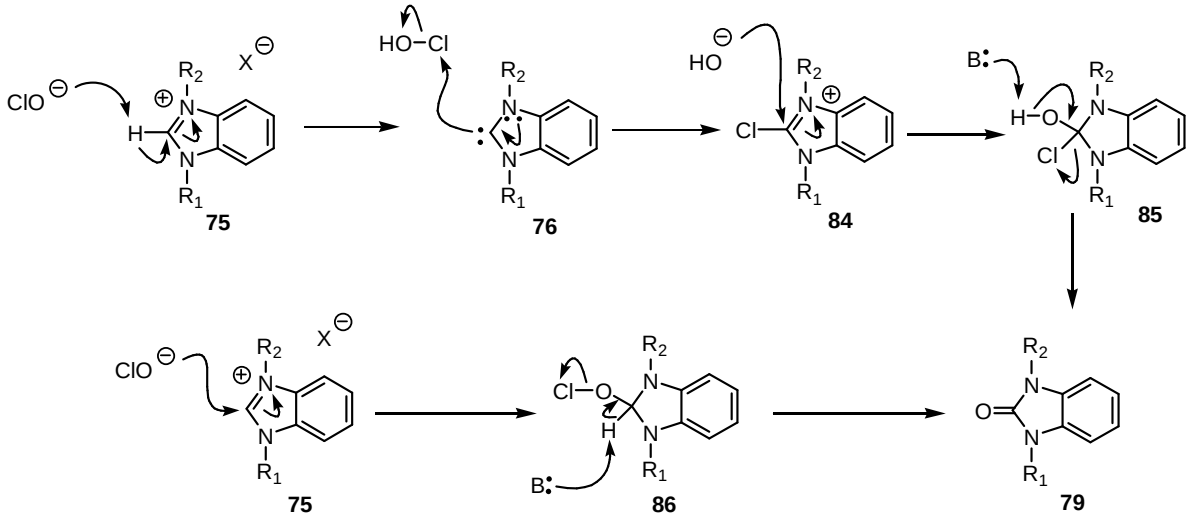
During the course of preparing derivatives **82f-j**, careful attention to the equivalents of NaOCl had to be taken. We found that treating **81f** with excess bleach resulted in the chlorination of one of the phenyl rings along with C2 oxidation leading to **83a** or **83b** as evidenced by NMR spectroscopy and mass spectrometry data (Scheme 2.3). Given the large numbers of aromatic protons and carbons we have been unable to unequivocally assign a structure. Inhibiting the chlorination event was achieved by treating **81f-g** with ~1 equiv of bleach in the presence of excess base (NaOH) to obtain 79% and 71% yield for the desired imidazolones **82f-g**. The same protocol was used in the preparation of **82h-j** as well.



Scheme 2.3

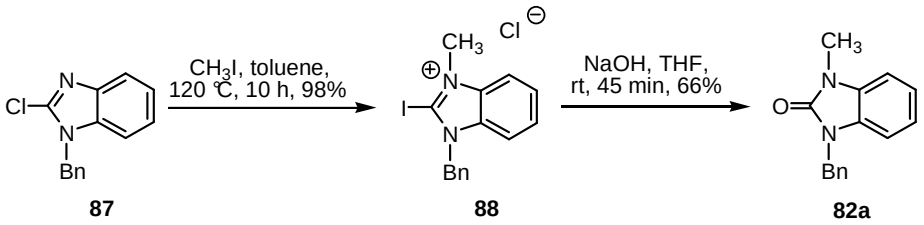
Mechanistically, the oxidation involving NaOCl can occur through at least two possible pathways (Scheme 2.5). Hypochlorite can act as a nucleophile and attack the electron deficient C2 position of **75** resulting in intermediate **86** which upon deprotonation and loss of chloride results in imidazolone **79**. The nucleophilic nature of hypochlorite has been extensively studied and is believed to be enhanced even through the alpha effect.[56, 57] Alternatively, hypochlorite can act as a base which deprotonates the C2 position leading to **76** which can trap the

electrophilic chlorine of the hypochlorite ion leading to **84**; the proposed intermediate in the NCS method.[58-60] Deprotonation and loss of chloride then gives **79**. Whether the two different oxidation methods share a common intermediate is debatable, as is what that intermediate might be. In order to narrow down the likely intermediates of this transformation, a few experiments were conducted.



Scheme 2.5

Initially, 2-chlorobenzimidazole **87** was converted to its benzimidazolium salt **88** (Scheme 2.6). However, it appears the salt obtained from the methylation was actually the 2-iodo derivative as evidenced by NMR spectroscopy and particularly mass spectrometry data. Treating **88** with aqueous sodium hydroxide in THF at room temperature resulted in formation of **82a** in good yield. Therefore, a 2-halobenzimidazolium salt can undergo facile substitution in the presence of a nucleophile, in this case OH^-, to afford C2 substituted imidazoles. Other examples of 2-haloimidazolium salts undergoing substitution reactions have also been reported in literature.[58-60] It is unclear as to why the 2-iodo salt was obtained in the methylation reaction. It is possible that it is a consequence of the elevated temperature needed to effect methylation. Further detailed studies will be required to elucidate the exact mechanism of this transformation.

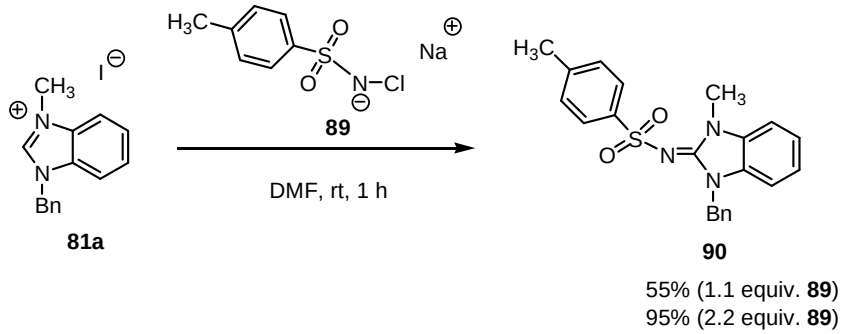


Scheme 2.6

2.2 Synthesis of 2-Iminoimidazoles

2-Aminoimidazole-containing natural products such as those highlighted in Chapter 1 pose an equally challenging synthetic issue as the imidazolone construction. A method for direct nitrogen substitution involves lithiation followed by trapping of TsN_3 or $TrisN_3$.[61, 62] The resulting azide intermediate can then be reduced to the amine via hydrogenation with Pd/C or Lindlar catalyst, with $NaBH_4$, or through a Staudinger reaction.[25] Following this protocol has allowed for the completion of a number of total synthesis efforts in our lab.[63-69]

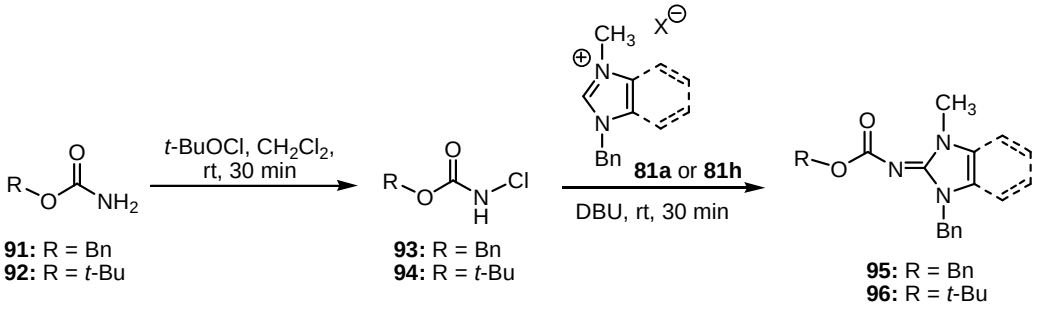
During the screening of electrophilic chloro-containing reagents for application toward the imidazolone chemistry, chloramine-T (**89**) was employed with the intent that the approach could be extended to include *N*-chloro-containing reagents. Addition of 1 equivalent of



Scheme 2.7

chloramine-T to a solution of **81a** in DMF resulted in formation of the tosyl derivative **90** in 55% yield along with sulfonamide; however, addition of 2 equivalents of **89** provided **90** in 95% yield. This shows that in the absence of an external base, the reaction will proceed to give the substitution product, however, does not definitively prove the nucleophilic attack mechanism presented in Scheme 2.5.

The successful application of chloramine-T in the substitution chemistry prompted more investigation of *N*-chloro-containing reagents. The preparation of *N*-chlorocarbamates **93** or **94** by reaction of carbamate **91** or **92** with *t*-BuOCl is known[70] and a one-pot procedure for their

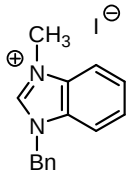
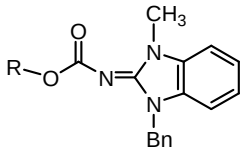
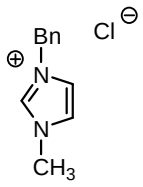
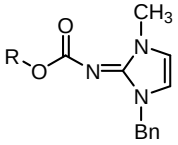


91: R = Bn
92: R = *t*-Bu

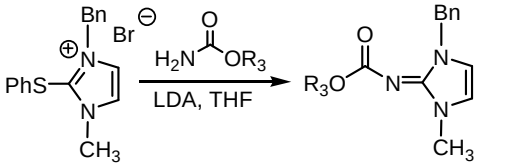
93: R = Bn
94: R = *t*-Bu

95: R = Bn
96: R = *t*-Bu

Table 2.3 Results of 2-Iminoimidazole synthesis

81	2-Iminoimidazoles
 **81a**	 **95a:** R = Bn (86%) **96a:** R = *t*-Bu (72%)
 **81h**	 **95b:** R = Bn (36%) **96b:** R = *t*-Bu (71%)

preparation and reaction with imidazolium salts was developed. The expected 2-imino derivatives **95a-b** were isolated in good yield when either *N*-chloro-*t*-butyl- (**93**) or benzylcarbamate (**94**) were reacted with **81a** in the presence of DBU (Table 2.3). Simple imidazolium salts also participate in the chemistry as illustrated by the preparation of **96a-b** from **81h** in 71% and 36% yields.

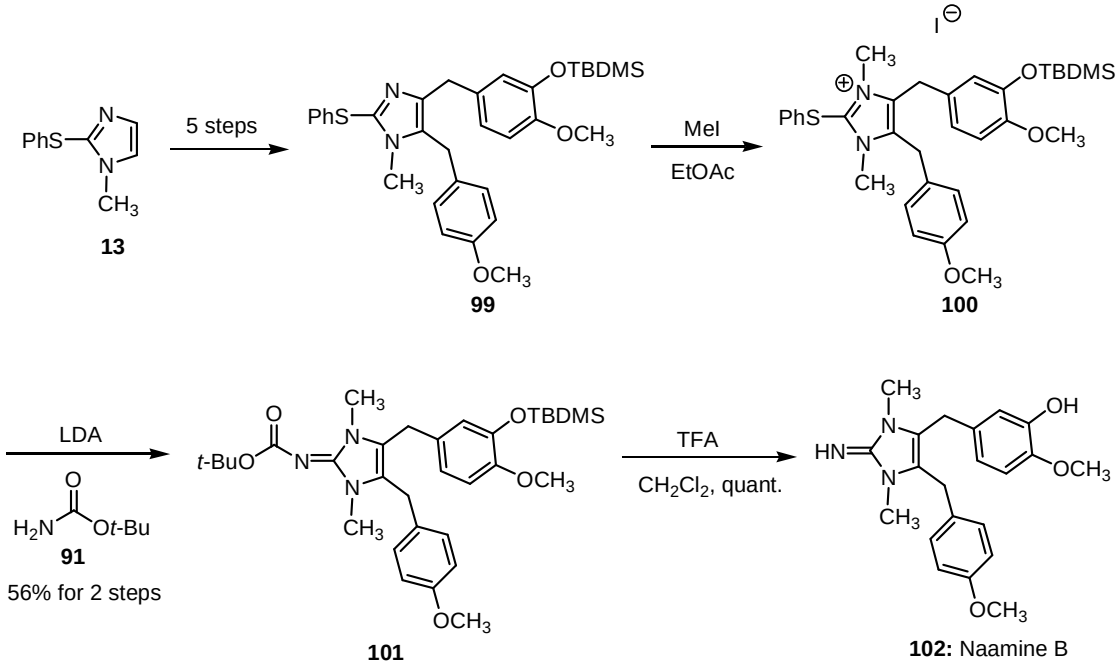


97a

98a: R_3 = Me, 86%
98b: R_3 = Ph, 49%
98c: R_3 = *t*-Bu, 71%

Scheme 2.8

Ohta has demonstrated the effectiveness of using 2-iminoimidazoles as intermediates en route to 2-aminoimidazoles.[71] Treating carbamates with LDA, followed by addition of 2-



13

99

100

91

56% for 2 steps

101

102: Naamine B

Scheme 2.9

thioimidazolium salt **97a** resulted in formation of the desired 2-iminoimidazole derivatives **98a-c** in a similar manner as seen for the synthesis of 2-imidazolones (Scheme 2.8).

This method was applied to the total synthesis of naamine B (**102**) in which the anion of *t*-butyl carbamate (**91**) formed from deprotonation with LDA was reacted with imidazolium salt **100** to give 2-iminoimidazole **101** (Scheme 2.9).[71] Treating **101** with TFA resulted in deprotection of the *N*-Boc substitutent as well as the silyl protecting group to give naamine B (**102**). Our strategy would appear to offer an advantage of this method as it circumvents the need for the introduction of the carbamate through the use of LDA and the installation of a C2-activating substituent.

2.3 Conclusions

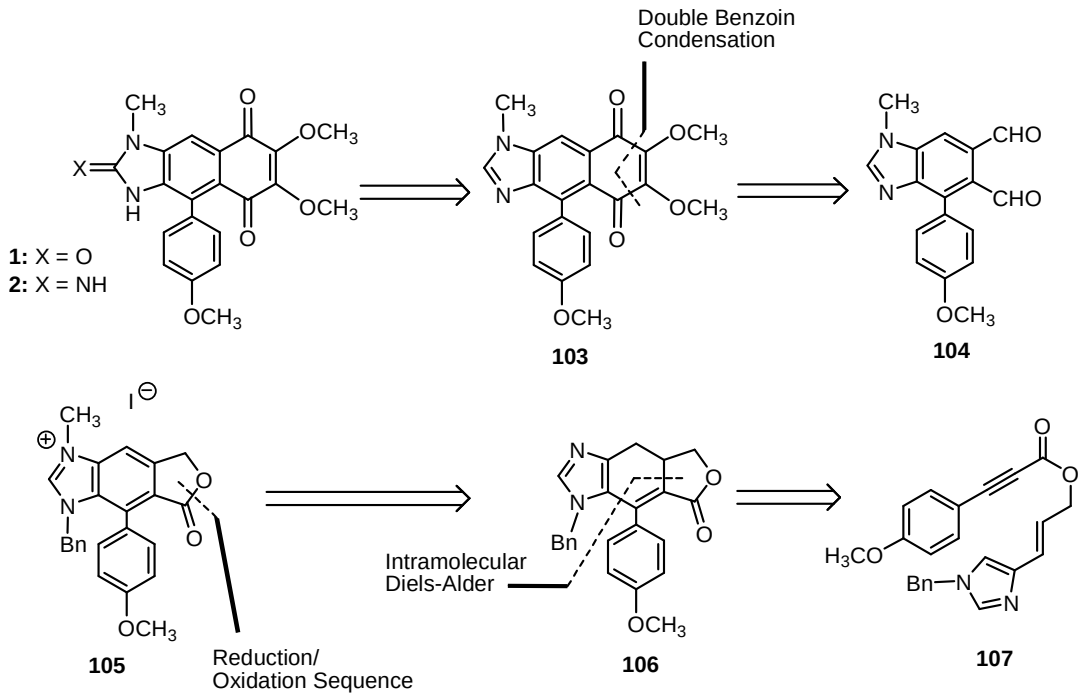
C2 functionalization of imidazoles involving chloro-containing reagents has been developed and successfully applied to a number of simple imidazole systems. NCS, NaOCl, chloramine-T, and *N*-chlorocarbamates all participate in the chemistry to provide 2-imidazolones or 2-iminoimidazoles from imidazolium salts. While the exact mechanism of this transformation is still to be determined, it is likely that a 2-haloimidazolium intermediate is involved based on literature examples and our own experiments. The application of this method toward the synthesis of kealiiquinone is described in the following chapter.

CHAPTER 3

TOTAL SYNTHESIS OF KEALIIQUINONE

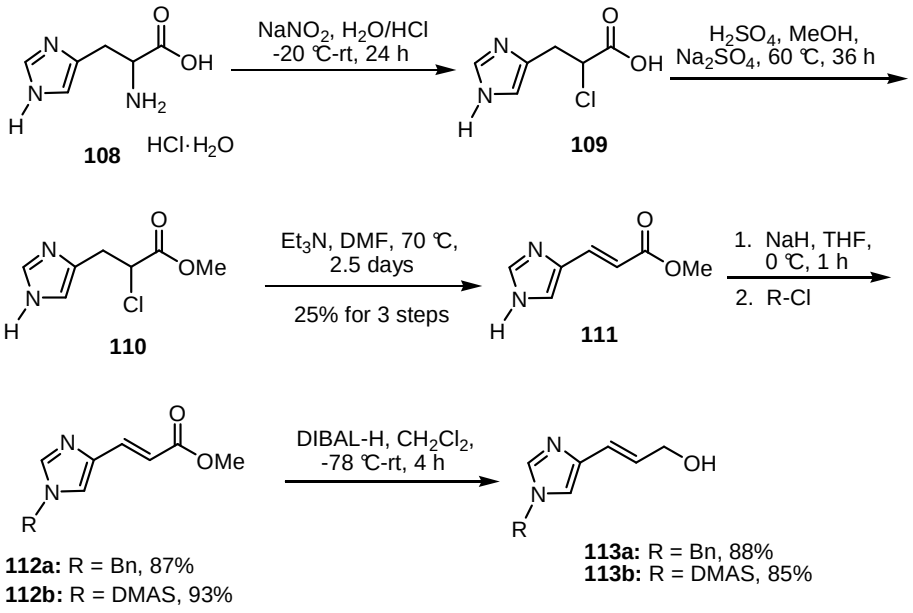
3.1 First Generation Approach

The total synthesis of kealiiquinone (**1**) was initially envisioned to occur through a late-stage C2-functionalization of dimethoxyquinone intermediate **103** with the intention of being able to access both kealiiquinone (**1**) and 2-deoxy-2-aminokealiiquinone (**2**) from the same common intermediate (Scheme 3.1). The quinone ring of intermediate **103** would be constructed via a proposed double benzoin condensation of dialdehyde **104** which would result from debenzylation and a reduction/oxidation sequence of imidazolium salt **105**. The ring



Scheme 3.1

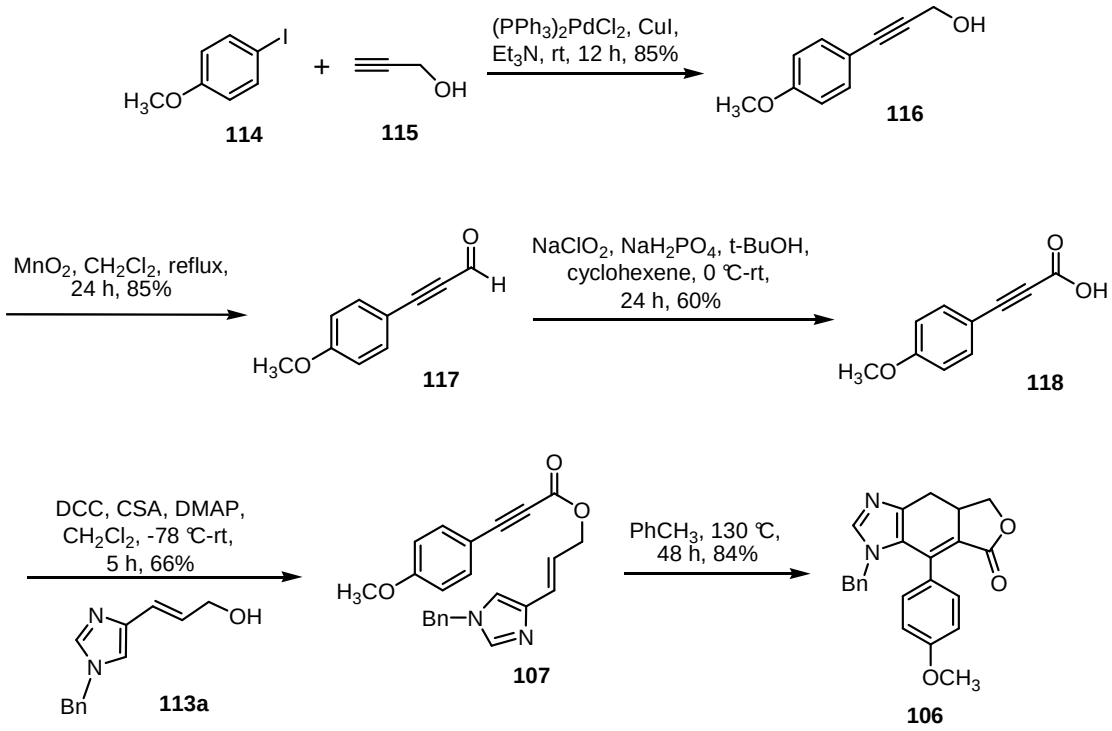
system of **105** would ultimately arise from intermediate **106**; the IMDA product from the cyclization of propynoate **107**.



Scheme 3.2

The synthesis of propynoate **107** required the preparation of two essential coupling partners, allylic alcohol **113a** and propiolic acid **118**. Alcohol **113a** is a common building block in our lab and a method to prepare it in five steps starting from histidine (**108**) has been developed (Scheme 3.2).[33, 72]

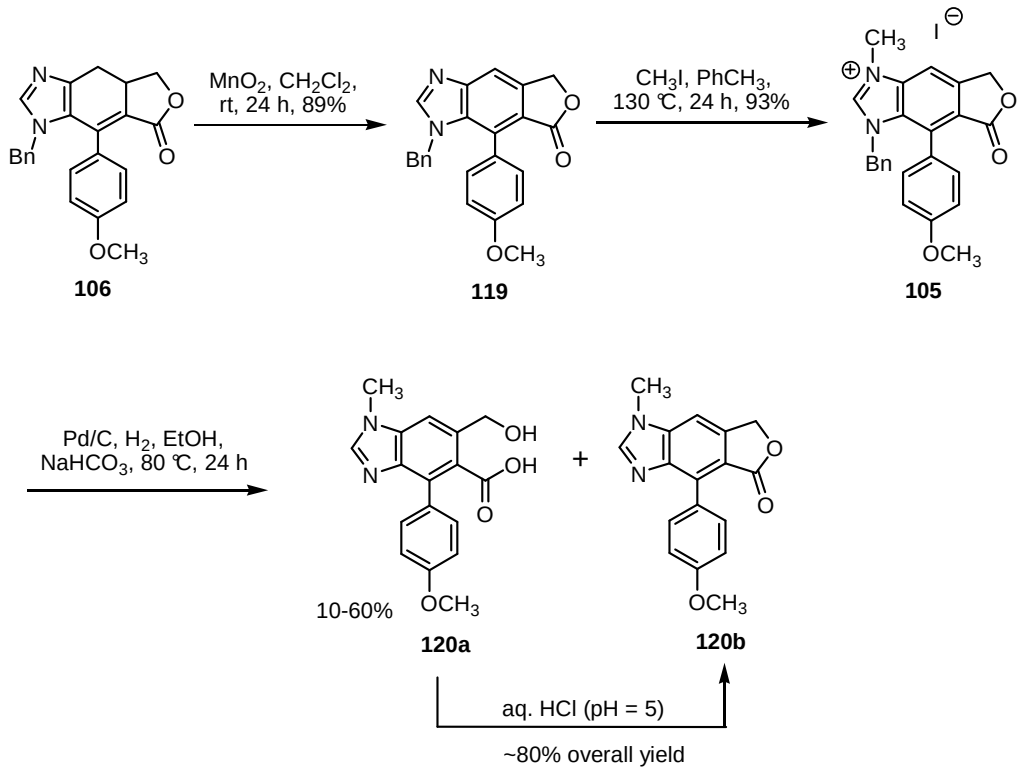
Propiolic acid **118** was prepared following literature procedures from 4-iodoanisole (**114**) and propargyl alcohol (**115**); coupled through a Sonogashira reaction (Scheme 3.3).[73] The resulting alcohol **116** was oxidized with MnO_2 to the aldehyde **117**,[74] and then to the acid by a Pinnick oxidation to give the desired partner **118**. Unfortunately, attempts to oxidize **116** directly to acid **118** were unsuccessful.[75, 76] Once the two partners were prepared, they were coupled together using DCC to give propynoate **107**. Heating **107** in toluene in a re-sealable pressure vessel for 48 h provided the Diels-Alder cycloadduct **106**.



Scheme 3.3

Treating **106** with MnO_2 resulted in aromatization by a dehydrogenation event to give **119** (Scheme 3.4).[77] The resulting benzimidazole intermediate **119** was then heated in the presence of iodomethane to provide the imidazolium salt **105**. Debenzylation of **105** was achieved through hydrogenation with Pd/C in the presence of $NaHCO_3$.[78]

The debenzylation of **105** was an interesting transformation. When attempted without base, no reaction was observed. The base is presumably acting to quench any hydroiodic acid that may be forming and thereby preventing poisoning of the catalyst. It was also observed that some reactions were low yielding, and that upon washing the catalyst with water, a white solid could be isolated from the aqueous layer. ^{1}H NMR spectra of the solid was strikingly similar to the expected product, and with the solubility and chromatography characteristics (the compound stays at the baseline of TLC plates), we hypothesized that the aqueous $NaHCO_3$ hydrolyzed the lactone leading to hydroxy acid intermediate **120a**. As further confirmation, we found that after acidifying the aqueous layer after filtration to pH 5 with dilute HCl, the desired lactone was



Scheme 3.4

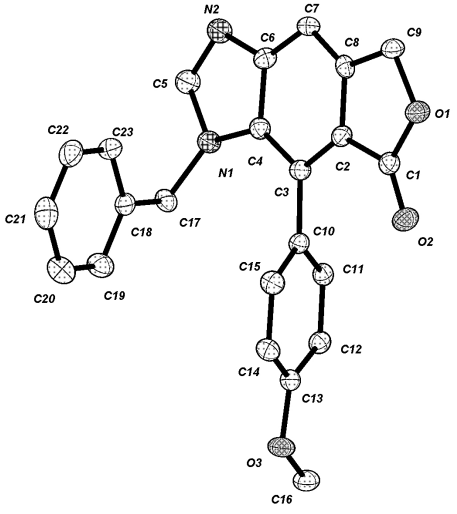
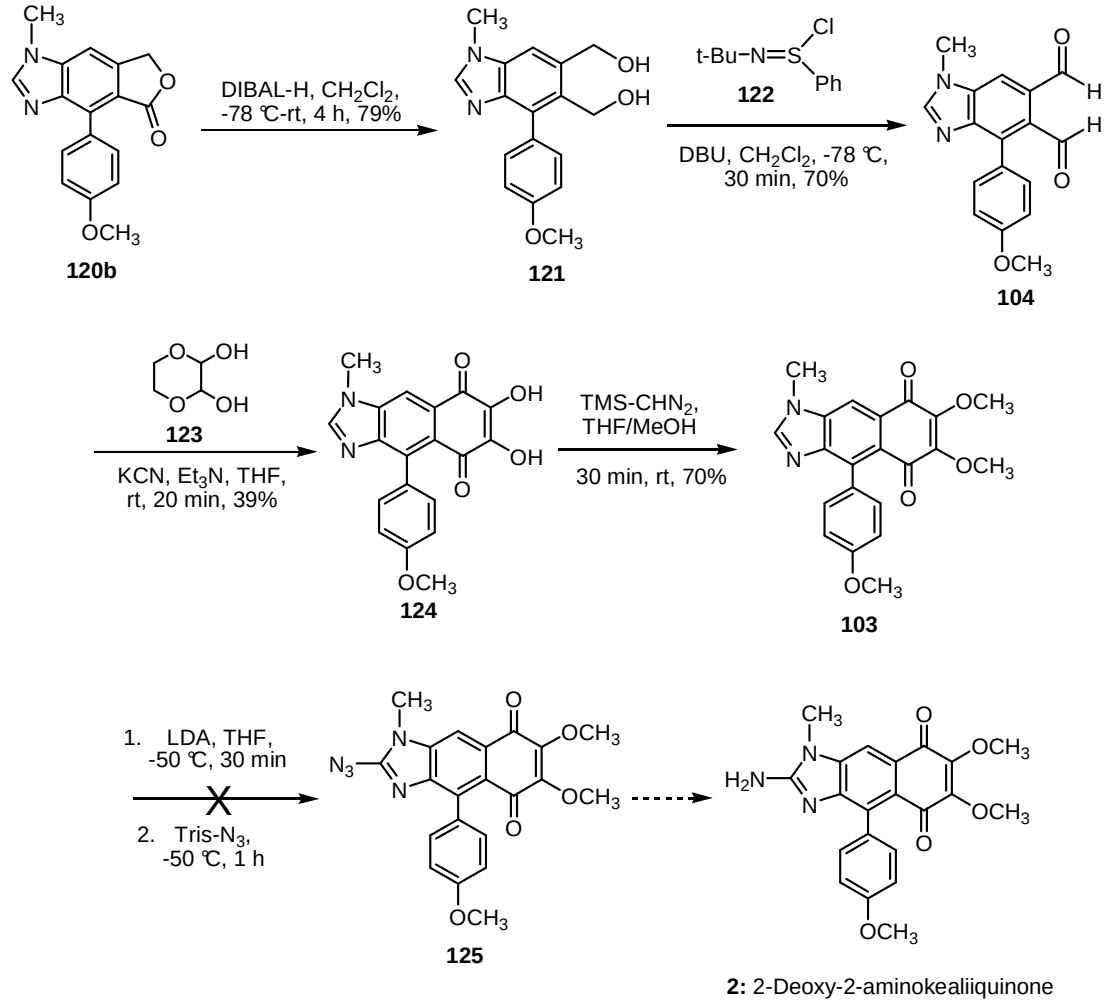


Figure 3.1 X-ray structure of benzimidazole **119**

isolated by extraction. Therefore, the by-product **120a** obtained from the deprotection of **105** was converted to the desired product **120b** during work-up of the reaction to afford an overall yield of 80%.

Lactone **120b** was then reduced with DIBAL-H to afford diol **121** (Scheme 3.5). The oxidation of diol **121** to provide the dialdehyde **104** was especially problematic and required extensive screening of reagents. It was found that protocols involving more traditional and well-known oxidation methods and reagents such as MnO_2, IBX, DMP, and PCC resulted in the



Scheme 3.5

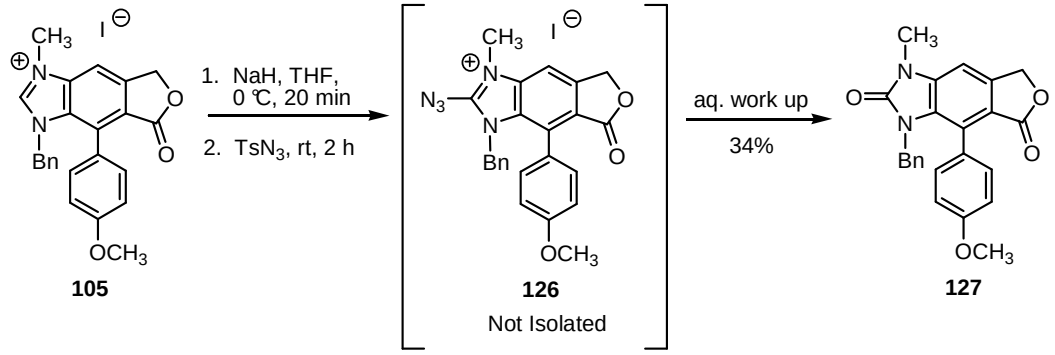
almost exclusive isolation of the lactone **120b**. Swern oxidation did provide the desired dialdehyde **104** in moderate yield on one occasion, but proved difficult to reproduce. Finally, a method involving *N*-*t*-butylbenzenesulfinimidoyl chloride (**122**)[79] was found to effectively and consistently oxidize diol **121** to the dialdehyde **104** in 70% yield and on sufficient scale to advance the project.

Dialdehyde **104** was such a critical intermediate because it allowed for the construction of the dihydroxyquinone **124** through a presumed double benzoin-like condensation with 2,3-dihydroxy-1,4-dioxane (**123**).[80] The hydroxyl groups of **125** were then methylated with TMS-diazomethane to give dimethoxyquinone **103**.[81] Quinone **103** was treated with LDA, followed by $TrisN_3$ in attempts to functionalize the C2 position with an azide which could then be reduced to give 2-deoxy-2-aminokealiiquinone (**2**) in an approach that has been successfully applied in a number of situations (see Chapter 2). However, the desired azide **125** was never isolated from the complex, crude reaction mixtures.

Multiple attempts to functionalize **103** in this manner were unsuccessful. Finally, an attempted deuterium labeling experiment was performed in which **103** was treated with LDA just as before and quenched with D_2O after approximately 30 minutes. Upon the addition of D_2O to the reaction, a bright red, insoluble precipitate was observed, and no unreacted starting material or the expected deuterium labeled product was identified from ^1H NMR spectroscopy. From these investigations, we concluded that C2 functionalization was not going to be successful at this stage and with this approach. Lithiation and azidation was attempted on lactone **120b**; however, the poor solubility of **120b** in most organic solvents, even in the presence of LDA, resulted in the isolation of unreacted starting material.

Azidation was then attempted on imidazolium salt **105** with the expectation that deprotonation could be achieved with a much weaker base than BuLi or LDA (Scheme 3.6). By treating **105** with NaH at 0 ℃, followed by TsN_3 we anticipated obtaining azide **126**. However,

the product isolated from the reaction mixture was the imidazolone **127** presumably from nucleophilic addition of water to **126** during work up and loss of the azide.

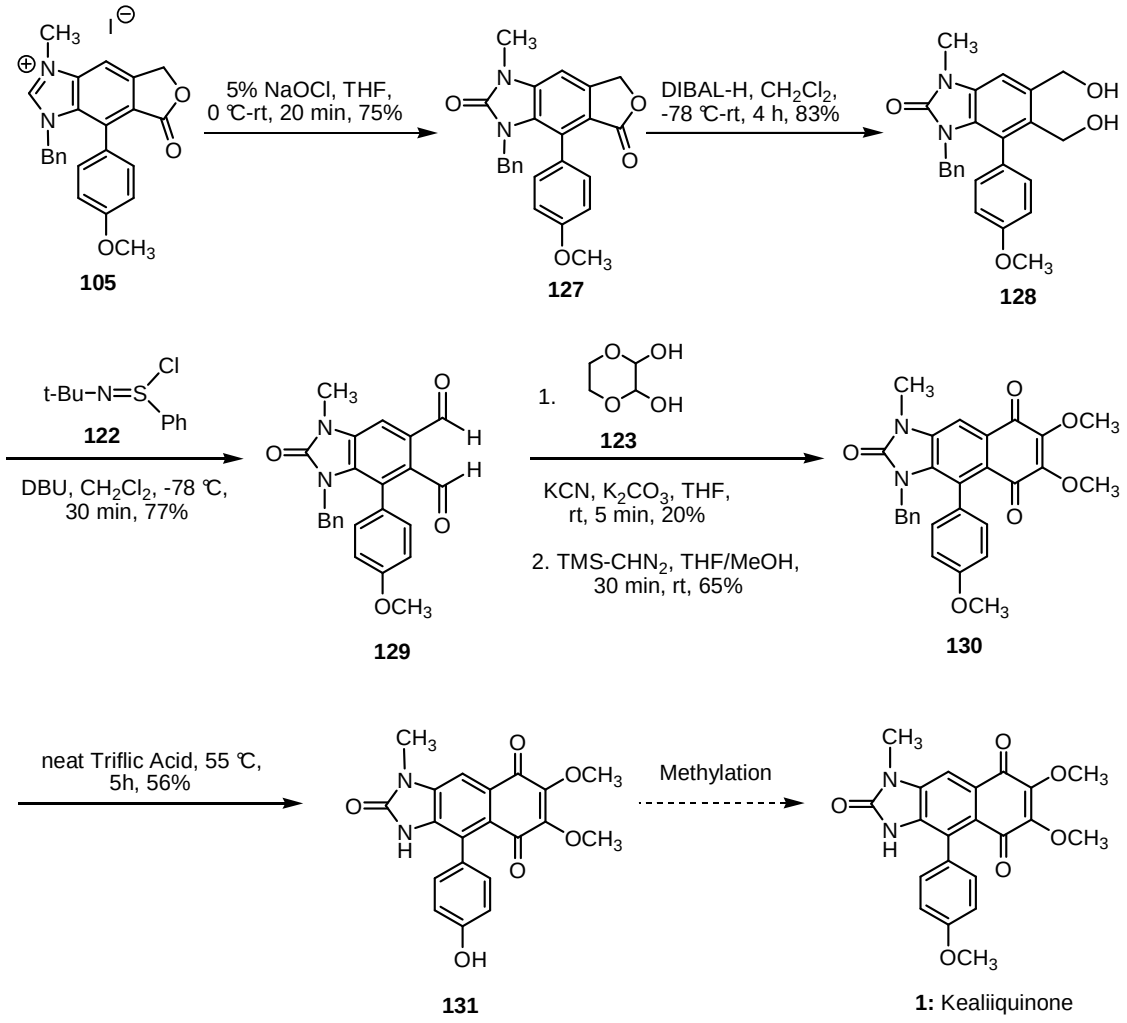


Scheme 3.6

The difficulties encountered up to this point and the unexpected formation of **127** from the imidazolium salt prompted an investigation into the functionalization of imidazolium salts and ultimately resulted in the 2-imidazolone and 2-iminoimidazole methodology described in Chapter 2. Application of this methodology resulted in the development of the second generation synthetic approach to kealiiquinone.

3.2 Second Generation Approach

The second generation approach to kealiiquinone was centered on construction of the 2-imidazolone from the developed methodology. Treating imidazolium salt **105** with sodium hypochlorite resulted in the desired oxidation and preparation of imidazolone **127** (Scheme 3.7). The lactone of **127** was functionalized in a similar manner as seen in the first generation synthesis to provide dialdehyde **129**. The dihydroxyquinone was constructed and methylated to



Scheme 3.7

give dimethoxyquinone **130** through reactions largely identical to those previously described (Scheme 3.4).

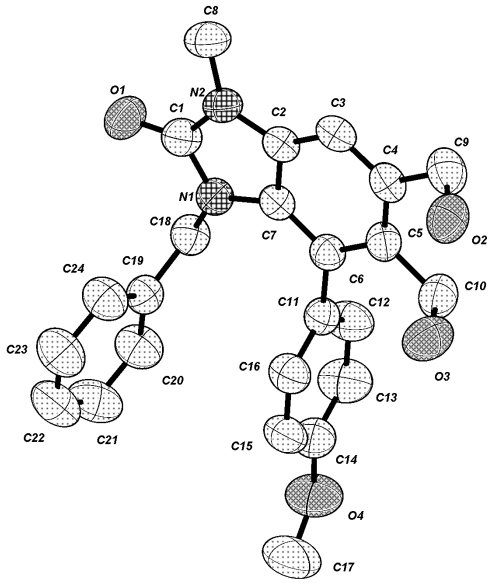
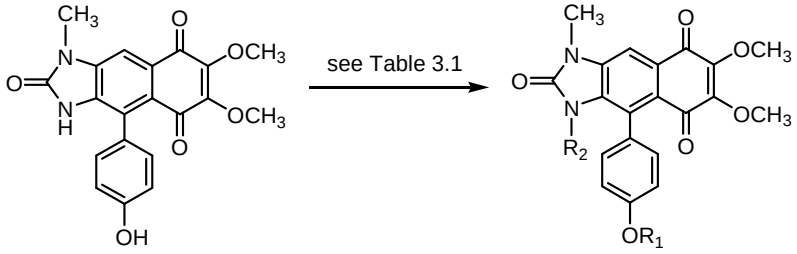


Figure 3.2 X-ray structure of 2-imidazolone **128**

In contrast to amines, few general methods exist for the debenzylation of amides, and the somewhat sensitive quinone ring served to complicate and narrow the selection of potential processes. Accordingly, the debenzylation of intermediate **130** was identified as potentially problematic early on, but given we were already committed to the route we continued. Imidazolones **82a** and **127** were used as models to test some of these methods including hydrogenation with Pd/C,[15] oxidation with potassium *t*-butoxide/O_2,[82] radical bromination with NBS,[83, 84] and acidic hydrolysis using *p*-TSA[85] all without success.

Triflic acid has been used to successfully debenzylate a variety of amides including some relatively elaborate examples.[86, 87] Treatment of **130** with neat triflic acid at 55 ℃ resulted in the desired debenzylation, and an undesired demethylation of the 4-methoxy group of the phenyl substituent resulting in phenol **131**.[86] Attempts to affect chemoselective *O*-methylation were unsuccessful and resulted in either both *N*- and *O*-methylation when Me_2SO_4 was used

with K_2CO_3 or selective *N*-methylation with 0.5 equivalents of Me_2SO_4 and LiOH[88] or TMS-CHN_2 with DIPEA (Table 3.1).[89]



131

132a: $R_1 = R_2 = Me$
132b: $R_1 = H$, $R_2 = Me$

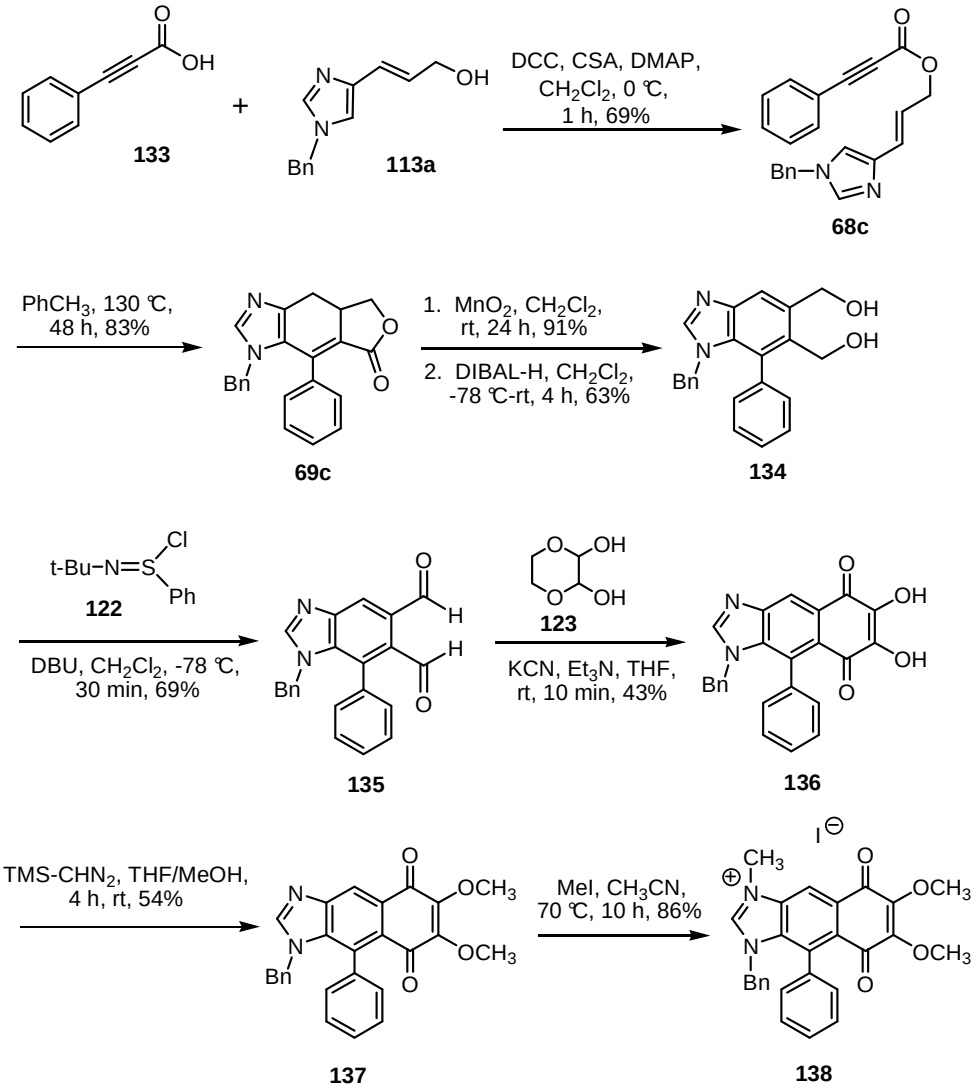
Table 3.1 Conditions screened for *O*-methylation

Conditions	R_1	R_2
1) Me_2SO_4, K_2CO_3, acetone, rt	Me	Me
2) Me_2SO_4 (0.5 equiv.), LiOH, THF, rt	H	Me
3) TMS-CHN_2, DIPEA, CH_3CN, rt	H	Me

Chemoselectivity becomes an increasing issue in systems containing reactive heteroatoms of similar pK_a resulting in mixtures of methylated products.[90] In addition, the rate of *N*-methylation of heterocycles has been shown to be faster than *O*-methylation of phenols under certain conditions most likely due to the greater nucleophilicity of nitrogen.[91] These two factors can be used to explain what has been observed in our methylation attempts. The pK_a of the phenolic –OH must not be sufficiently different from that of the imidazol-2-one -NH to impart any selectivity, and the rate of *N*-methylation must be significantly faster than that of *O*-methylation. Further studies are being conducted to screen alternative reaction conditions to achieve the desired methylation.

3.3 Synthesis of Analogs

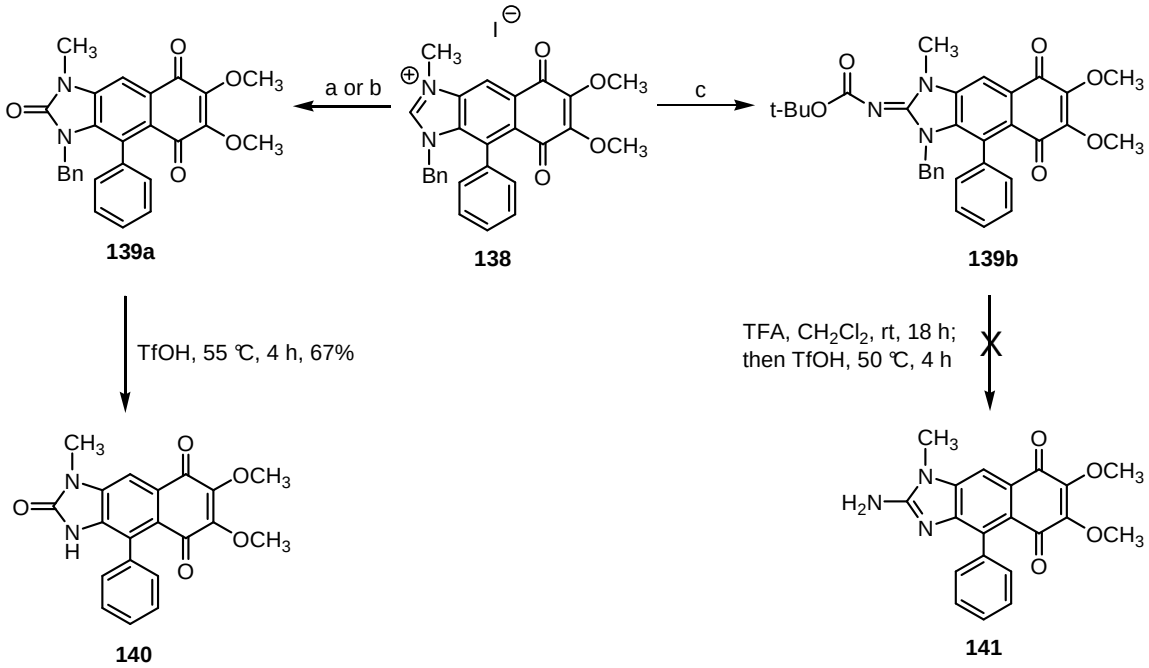
Several analogs of kealiiquinone were also prepared based on the routes designed to access the late stage intermediates in the first and second generation approaches. Compound **68c** prepared from phenyl propiolic acid (**133**) and alcohol **113a** was studied early on as a model system to develop the Diels-Alder reaction to give **69c** (Scheme 3.8). Applying the same



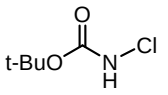
Scheme 3.8

transformations as previously described in sections 3.1 and 3.2 permitted the preparation of dimethoxyquinone **137**. The sequence in this route was changed somewhat in that methylation to form the imidazolium salt **138** was delayed until after the construction of the quinone ring.

The motivation for delaying formation of the imidazolium salt **138** until the end of the synthesis was to have an intermediate that could be functionalized at the C2 position leading to both a 2-imidazolone and 2-amino substituent much like the first generation approach. Treating **138** with sodium hypochlorite (NaOCl) at 0 ℃ or NCS and 1M K_2CO_3 resulted in oxidation to the 2-imidazolone **139a** (Scheme 3.9). Additionally, treating **138** with *N*-chloro *t*-butylcarbamate (**83**) and DBU, the 2-iminoimidazole intermediate **139b** was prepared.



a) 5% NaOCl, THF, 0 ℃, 10 min, 26% b) NCS, 1M K_2CO_3, THF, rt, 30 min, 60%

c)  , DBU, CH_2Cl_2, 30 min, 30%

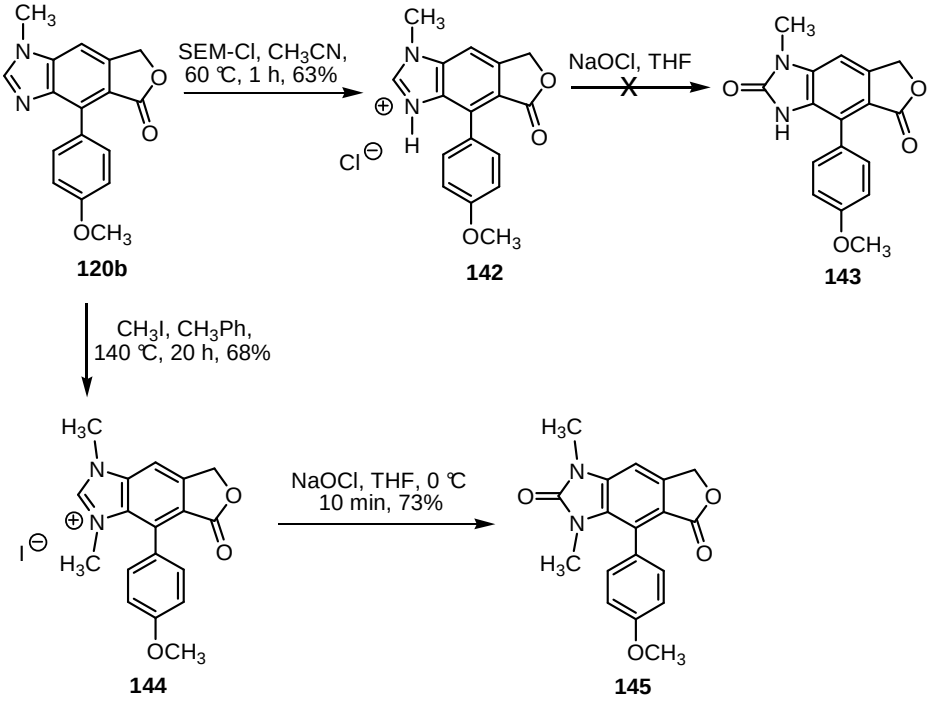
83

Scheme 3.9

Deprotection of **139a** was accomplished with TfOH to give **140**, while an attempt to deprotect **139b** stepwise with TFA then TfOH to give **141** was unsuccessful. Therefore, a desmethoxy analog **140** of kealiiquinone (**1**) was constructed through a late stage C2 functionalization of **138**.

In addition to the synthesis of analog **140** which is interesting in itself from the standpoint of biological activity, we have demonstrated that the quinone ring is tolerant to both *N*-alkylation leading to imidazolium salts and C2-substitution chemistry employing *N*-chloro-containing reagents. This has led to functionalization attempts on other late stage intermediates in the kealiiquinone sequences.

3.4 Alternative Functionalization Attempts



Scheme 3.10

As the chemistry towards kealiiquinone developed, some of the late stage intermediates from the first generation approach were re-examined as potential intermediates to access the

natural products. For example, lactone **120b** was treated with SEM-Cl in an attempt to produce the SEM imidazolium salt (Scheme 3.10). However, the compound isolated from that reaction was what we propose from spectroscopic data was the HCl salt **142**; a consequence perhaps of HCl produced from the decomposition of SEM-Cl or the initially formed imidazolium intermediate. Salt **142** failed to provide the imidazolone **143** when treated with NaOCl. An X-ray crystal structure of **142** was obtained (Figure 3.3).

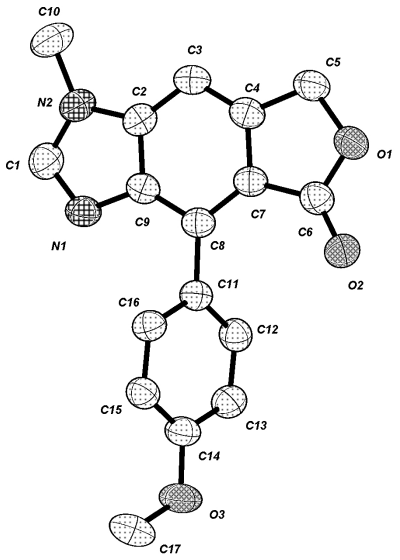
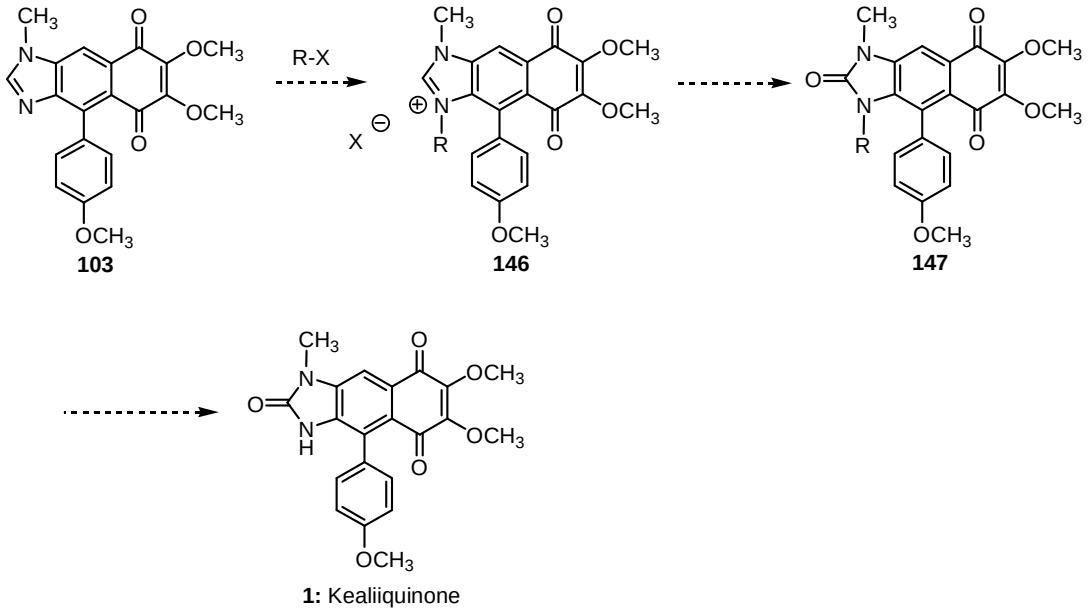


Figure 3.3 X-ray structure of lactone **142**

N3 is in a relatively hindered environment being in such close proximity to the phenyl ring; therefore, a smaller methyl group was used to test whether substitution could occur in that position. When **120b** was heated with iodomethane, the dimethyl salt **144** was formed and this was oxidized to imidazolone **145**. Lactone **120b** has very limited solubility in a number of organic solvents as previously mentioned. As a result, elevated temperatures are needed in order to dissolve **120b** to get it to react, and this can be problematic with certain reagents. Dimethoxyquinone **103** is readily soluble in organic solvents and based on the synthesis of the

kealiiquinone analogs **140** and **141**, C2 functionalization is possible if an imidazolium salt can be formed with a suitable substituent (Scheme 3.11).



Scheme 3.11

3.5 Conclusions

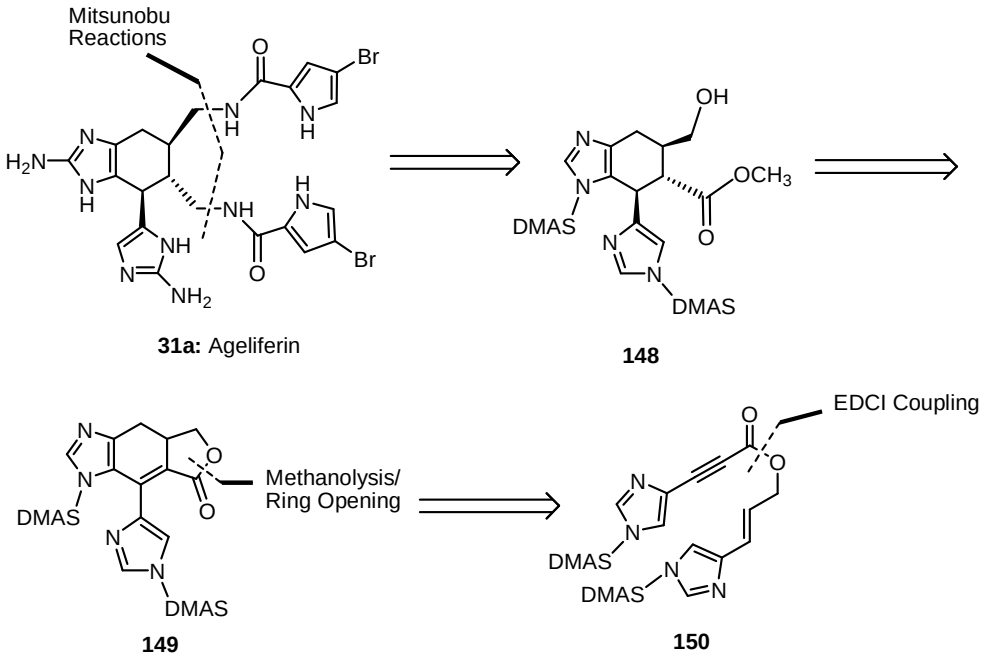
Considerable progress towards kealiiquinone has been made utilizing an intramolecular Diels-Alder reaction to prepare polysubstituted benzimidazole intermediates. Successful functionalization to construct the quinone ring as well as C2 functionalization utilizing a method developed to specifically address issues with this transformation in these systems has allowed for preparation of both 7'-desmethyl (**131**) and 4'-desmethoxy (**140**) derivatives of kealiiquinone (**1**). Ultimately, if the problems associated with benzyl deprotection of imidazolone **130** can be avoided with the use of another protecting group, then this will provide a concise and flexible strategy for the assembly of this family of natural products. This is currently being investigated for future approaches.

CHAPTER 4

STUDIES TOWARD THE TOTAL SYNTHESIS OF AGELIFERIN

4.1 Current Approach to Ageliferin

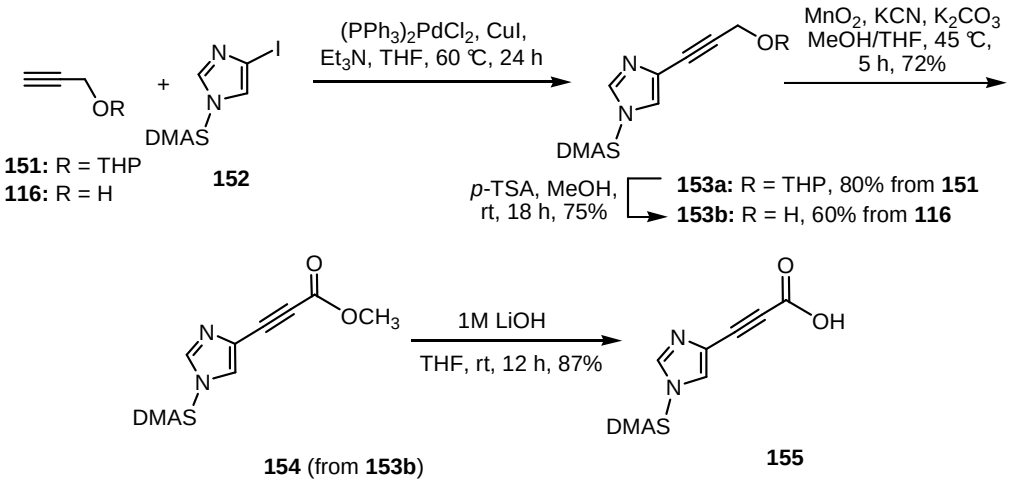
The general approach to ageliferin (**31a**) adopted from previous synthetic attempts and strategies toward other oroidin alkaloids has involved construction of the amide bonds containing the pyrrole rings through a series of Mitsonubu reactions, C2 amination and deprotection (not necessarily in that order) of a hydroxy ester intermediate **148** (Scheme 4.1). Intermediate **148** would be obtained from the cycloadduct **149** through reduction of the double bond and methanolysis to open the lactone ring. Propynoate **150** would serve as the precursor in the Diels-Alder reaction leading to **149**.



Scheme 4.1

Just as in the construction of other propynoates, the coupling partners necessary for intermediate **150** are allylic alcohol **113b** and propiolic acid **155**. The preparation of the acid has undergone a series of optimizations since the original lengthy procedure involving an ortho ester intermediate.[33] Conditions for a Sonogashira reaction of THP-protected propargyl alcohol **151** and 4-iodoimidazole **152** were developed allowing access to **153a** (Scheme 4.2). Deprotection of the THP ether was accomplished with *p*-TSA giving the propargyl alcohol **153b**. This route was eventually streamlined to omit the THP-protection/deprotection steps by developing conditions to achieve the Sonogashira coupling of propargyl alcohol (**116**) and 4-iodoimidazole **152** directly.[92]

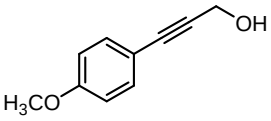
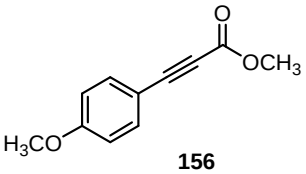
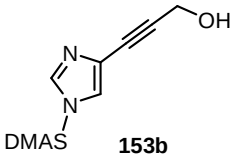
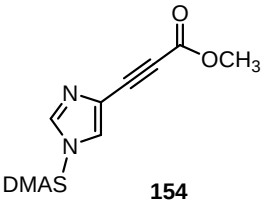
The oxidation of alcohol **153b** was found to be more difficult than initially expected. Attempts to oxidize **153b** to the aldehyde with MnO_2 similar to that of the anisole system were unsuccessful, as were attempts to oxidize directly to the acid. Ultimately, a method involving the tandem oxidation and cyano-mediated substitution of propargyl alcohols to methyl esters was utilized to give ester **154**.[93] This reaction required some optimization to make it applicable to the imidazole-containing system, in particular, reduction of the amount of cyanide ion used. Hydrolysis of ester **154** to the desired acid **155** was achieved with aqueous LiOH in THF.



Scheme 4.2

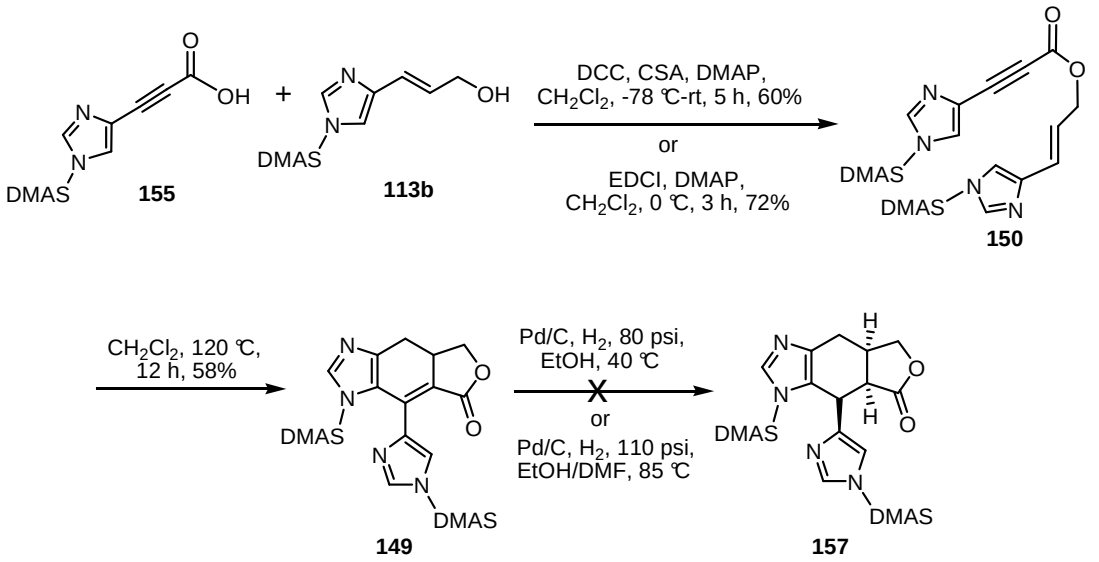
The oxidation conditions were initially screened on alcohol **118** having the anisole ring. A promising 63% yield of the expected methyl ester **156** was isolated from the reaction mixture with no further purification necessary (Table 4.1). However, when alcohol **153b** was subjected to the reaction conditions, the reaction proved to be low to moderate yielding (35-51%), and the resulting ester was highly colored and difficult to purify. The stoichiometric amount of NaCN and the possible evolution of HCN during the course of the reaction were concerning from the standpoint of scale-up and possible reaction with the acid-labile DMAS protecting group. In an attempt to neutralize the evolution of HCN and use only a catalytic amount of cyanide, 1 equivalent of NaCN was replaced with 0.2 equivalents of KCN and an excess of K_2CO_3. Gratifyingly, this modification provided **154** in increased yield and purity.

Table 4.1 Oxidation of propargyl alcohols to methyl esters

Propargyl alcohol (**118** or **153b**)	Methyl ester (**156** or **154**)
 **118**	 **156** 63% from Method A
 **153b**	 **154** 35-51% from Method A 72% from Method B

Method A: NaCN (1.0 equiv), MnO_2, THF/MeOH, 45 ℃

Method B: KCN (0.20 equiv), K_2CO_3 (2.0 equiv), MnO_2, THF/MeOH, 45 ℃



Scheme 4.3

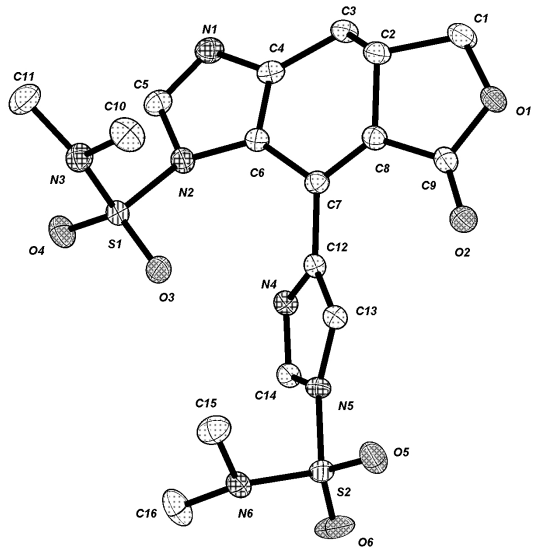
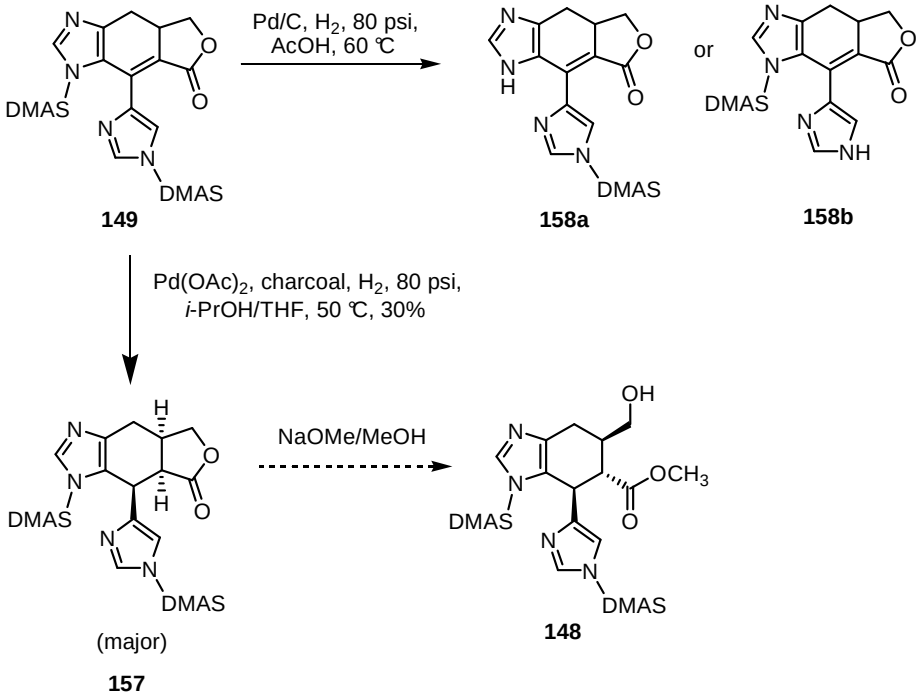


Figure 4.1 X-ray structure of cycloadduct **149**

Acid **155** and alcohol **113b** were initially coupled together using DCC to give propynoate **150** as a sticky, tan solid (Scheme 4.3). When **150** from the DCC coupling was heated in CH_2Cl_2, the reaction was observed to turn dark brown over the course of several hours and a polar precipitate was observed to form on the walls of the flask. Previous

experiences in our lab indicated that this was a poor omen for an effective cycloaddition. The desired Diels-Alder product **149** was never obtained from these attempts. When the coupling was repeated with EDCI, **150** was obtained as a white, crystalline solid, and the Diels-Alder reaction provided cycloadduct **149** in almost 60% yield. We concluded that an impurity associated with the DCC coupling must have been accelerating decomposition of **150** during the Diels-Alder reaction.

Reduction of the double bond resulting from the Diels-Alder reaction of propynoate systems has been previously accomplished by hydrogenation with Pd/C to give an all *cis*-product such as **157** (Scheme 4.3).[33] However, attempts to reduce **149** in this manner were unsuccessful. We found intermediate **149** to be completely insoluble in warm EtOH resulting in the isolation of starting material when the hydrogenation conditions in Scheme 4.3 were applied. Using a mixture of EtOH and DMF as solvents dissolved **149**, but the desired reduction was not achieved with 110 psi of hydrogen at 85 ℃; unreact ed **149** was recovered. When the solvent



Scheme 4.4

was changed from EtOH to AcOH, the Diels-Alder product **149** was soluble, however, instead of reduction, deprotection of a DMAS group occurred to give **158a** or **158b** (Scheme 4.4). The desired reduction was ultimately achieved using $Pd(OAc)_2$ and charcoal as the catalyst in a mixture of *i*-PrOH and THF for the hydrogenation to give **157**.[94] While the expected all *cis* product **157** was isolated, it became apparent that the acetate present in the reaction was catalyzing epimerization events as evidenced by what is believed to be a mixture of diastereomers that was also recovered. However, these diastereomers may be useful intermediates toward the synthesis of other oroidin-derived natural products such as the nagelamides (**32a-c**).

The lactone of **157** can be treated with NaOMe to open the ring and provide **148** (Scheme 4.4). It has been demonstrated that similar systems undergo an epimerization to relieve steric strain upon opening of the lactone resulting in the desired *trans* stereochemistry of **148**.[33] At this point, the desired stereochemistry would be set, and this route intercepts intermediates prepared from other approaches to ageliferin (**31a**).

4.2 Conclusions

An intramolecular Diels-Alder reaction of propynoate **150** was used in a similar manner as the approach to kealiiquinone to obtain an intermediate which can be functionalized to access ageliferin. Tetrahydrobenzimidazole **148** provides an advanced intermediate with the appropriate stereochemistry as well as the hydroxyl and methyl ester handles that can be further functionalized to ultimately install the bromopyrrole side chains. This approach offers a substantially simplified route to intermediate **148** which has been constructed through other routes developed to access the oroidin alkaloids.

CHAPTER 5

EXPERIMENTAL SECTION

5.1 General methods

All reagents were purchased from commercial suppliers and used without purification unless otherwise specified. All reactions involving moisture sensitive reagents were conducted in oven-dried glassware under a dry nitrogen atmosphere. All solvents used in moisture sensitive reactions were purified by Innovative Technology's Pure-Solve solvent purification system.

NMR spectra were recorded on JEOL ECX 300 MHz and Eclipse+ 500 MHz spectrometers. ^{1}H NMR spectra were recorded in $CDCl_3$ (unless otherwise indicated) at a spectrometer frequency of 300.53 MHz or 500.13 MHz using residual $CHCl_3$ (δ = 7.26 ppm) as an internal reference. For spectra recorded in other solvents, residual MeOH (δ = 3.31 ppm) or DMSO (δ = 2.50 ppm) were used as internal references. ^{13}C NMR spectra were recorded in $CDCl_3$ (unless otherwise indicated) at a spectrometer frequency of 75.57 MHz or 125.76 MHz using residual $CHCl_3$ (δ = 77.2 ppm) as an internal reference. For spectra recorded in other solvents, residual MeOH (δ = 39.5 ppm) or DMSO (δ = 49.0 ppm) were used as internal references.

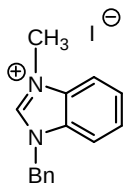
Melting points were recorded on a Laboratory Devices Inc. Mel Temp apparatus and are uncorrected. Infrared (IR) spectra were obtained on a Bruker ALPHA FT-IR Spectrometer using neat samples (ATR spectroscopy), or a Bruker Vector 22 spectrometer using either KBr pellets for solids or neat films on NaCl plates for liquids; all absorptions are reported in cm^{-1}. High resolution mass spectra (HRMS) were recorded by the Department of Chemistry and Biochemistry, University of Florida, Gainsville by electrospray ionization (ESI) or atmospheric-

pressure chemical ionization (APCI) unless otherwise indicated. All mass spectral data are reported as *m/z* (relative intensity).

Analytical thin layer chromatography (TLC) was performed on Sorbent Technologies Silica G TLC aluminum backed plates (200 μm thickness). Liquid chromatography was performed using Sorbent Technologies Standard Grade Silica Gel (230 x 400 mesh).

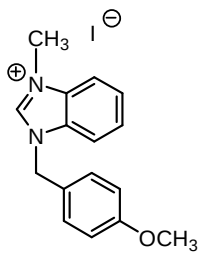
5.2 Synthesis

1-Benzyl-3-methyl-1*H*-benzimidazol-3-ium iodide (81a):



To 1-Benzyl-1*H*-benzimidazole (1.00 g, 4.80 mmol) in toluene (30 mL) was added MeI (1.50 mL, 24.0 mmol) and heated at 110 ℃ for 1 8 h at which time additional aliquots of MeI (0.5 equiv) were added until starting material was no longer detected by TLC. The resulting precipitated solids were filtered and washed with EtOAc to give **81a** as a pale, yellow solid (1.55 g, 92%). mp: 157-159 ℃ (lit. 138 ℃); [95] ^{1}H NMR: δ = 11.01 (s, 1H), 7.73 (d, *J* = 8.6 Hz, 1H), 7.62-7.58 (m, 2H), 7.54-7.51 (m, 3H), 7.35-7.30 (m, 3H), 5.81 (s, 2H), 4.24 (s, 3H); ^{13}C NMR: δ = 142.3, 132.5, 132.2, 131.1, 129.5, 129.4, 128.6, 127.5, 113.9, 113.2, 51.6, 34.4; IR (cm^{-1}): 3005, 1561, 1462, 1432, 1344, 1191, 749, 702; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{15}H_{15}N_2$ is 223.1230 found 223.1236.

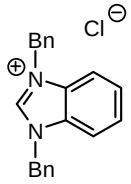
1-(4-methoxybenzyl)-3-methyl-1*H*-benzimidazol-3-ium iodide (81b):



To 1-(4-Methoxybenzyl)-1*H*-benzimidazole (1.00 g, 4.20 mmol) in toluene (60 mL) was added MeI (0.50 mL, 8.4 mmol) and heated at 110 ℃ for 18 h at which time additional aliquots of MeI (0.50 equiv) were added until starting material was no longer detected by TLC. The precipitated solids were filtered and washed with EtOAc and then Et_2O to give **81b** as a beige

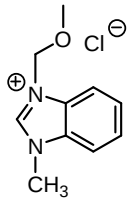
solid (1.30 g, 82%). mp: 197-200 ℃; ^{1}H NMR (CD_3OD): δ = 9.45 (s, 1H), 7.95-7.87 (m, 2H), 7.73-7.62 (m, 2H), 7.44 (d, *J* = 8.9 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 5.64 (s, 2H), 4.13 (s, 3H), 3.78 (s, 3H); ^{13}C NMR (CD_3OD): δ = 160.6, 142.0, 132.6, 131.3, 130.0, 126.9, 126.8, 124.9, 114.4, 113.5, 113.1, 54.5, 50.1, 32.7; IR (cm^{-1}): 2974, 1610, 1567, 1516, 1451, 1248, 1185, 1020, 783, 761; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{16}H_{17}N_2O$ is 253.1335 found 253.1329.

1,3-Dibenzyl-1*H*-benzimidazol-3-ium chloride (81c):



To 1-Benzyl-1*H*-benzimidazole (0.75 g, 3.6 mmol) in toluene (30 mL) was added benzyl chloride (0.50 mL, 4.3 mmol) and the mixture was heated at reflux for 18 h. The precipitated solids were filtered and washed with Et_2O to give **81c** as an off-white solid (0.65 g, 54%). mp: 211-212 ℃ (lit. 21 0-211 ℃);[96] ^{1}H NMR (CD_3OD): δ = 9.73 (s, 1H), 7.85 (dd, *J* = 6.2 Hz, *J* = 3.1 Hz, 2H), 7.62 (dd, *J* = 6.2 Hz, *J* = 3.1 Hz, 2H), 7.48-7.38 (m, 10H), 4.87 (s, 4H); ^{13}C NMR (CD_3OD): δ = 142.0, 133.3, 131.7, 129.1, 129.0, 128.1, 127.1, 113.7, 50.7; IR (cm^{-1}): 3419, 3355, 3036, 2954, 1557, 1460, 1426, 1374, 1189, 1016, 756, 740, 701.

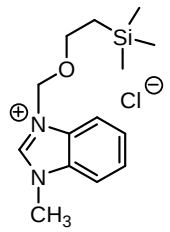
3-Methoxymethyl-1-methyl-1*H*-benzimidazol-3-ium chloride (81d):



To 1-Methyl-1*H*-benzimidazole (0.50 g, 3.8 mmol) in THF was added MOM-Cl (0.34 mL, 4.5 mmol) and stirred at rt for 18 h. The solvent was removed by rotary evaporation and the resulting solids were suspended in Et_2O, filtered and washed with Et_2O to give **81d** as a white, hygroscopic solid (0.73 g, 91%) that was stored at rt under N_2. mp: 115-118 ℃ (lit. 88-90 ℃);[55] ^{1}H NMR (CD_3OD): δ = 9.67 (s, 1H), 8.01-7.97 (m, 2H), 7.76-7.71 (m, 2H), 5.87 (s, 2H), 4.18 (s, 3H), 3.44 (s, 3H); ^{13}C NMR (CD_3OD): δ =

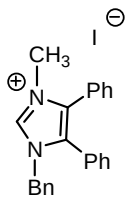
142.7, 132.7, 131.0, 127.3, 127.2, 113.6, 113.2, 78.5, 56.4, 32.7; IR (cm^{-1}): 3351, 3048, 1563, 1467, 1099, 1060, 1005, 765.

1-Methyl-3-trimethylsilylethoxymethyl-1*H*-benzimidazol-3-ium chloride (81e):



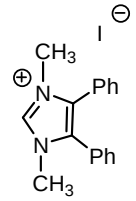
To 1-Methyl-1*H*-benzimidazole (0.50 g, 3.8 mmol) in THF (15 mL) was added SEM-Cl (0.80 mL, 4.5 mmol) and stirred at rt for 18 h. The solvent was removed by rotary evaporation and the resulting white solid was suspended in Et_2O, filtered and washed with Et_2O to give **81e** as a white solid (1.09 g, 96%). mp: 114-115 ℃; ^{1}H NMR: δ = 11.47 (s, 1H), 7.87-7.84 (m, 1H), 7.74-7.71 (m, 1H), 7.67-7.62 (m, 2H), 6.04 (s, 2H), 4.29 (s, 3H), 3.69 (t, *J* = 8.6 Hz, 2H), 0.90 (t, *J* = 8.3 Hz, 2H), -0.08 (s, 9H); ^{13}C NMR: δ = 143.8, 132.4, 130.8, 127.5, 114.3, 112.9, 68.3, 34.0, 17.9, -1.3; IR (cm^{-1}): 3442, 3367, 3027, 2954, 1569, 1420, 1250, 1113, 834, 763; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{14}H_{23}N_2OSi$ is 263.1574 found 263.1573.

1-Benzyl-3-methyl-4,5-diphenyl-1*H*-imidazol-3-ium iodide (81f):



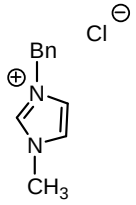
To 1-Benzyl-4,5-diphenyl-1*H*-imidazole (0.15 g, 0.48 mmol) in THF (10 mL) was added MeI (0.10 mL, 1.4 mmol) and stirred at 70 ℃ for 18 h. An additional aliquot of MeI (3 equiv) was added and heating resumed for a further 3 h. The solids were filtered and washed with EtOAc to give **81f** as a white solid (0.18 g, 81%). mp: 223-225 ℃; ^{1}H NMR: δ = 10.36 (s, 1H), 7.45-7.34 (m, 6H), 7.31-7.25 (m, 5H), 7.22-7.19 (m, 4H), 5.40 (s, 2H), 3.91 (s, 3H); ^{13}C NMR: δ = 136.7, 133.0, 132.8, 132.0, 131.1, 130.6, 130.5, 129.3, 128.9, 124.9, 124.5, 51.5, 35.5; IR (cm^{-1}): 3033, 1561, 1497, 1438, 1076, 766, 728, 702; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{23}H_{21}N_2$ is 325.1749 found 325.1695.

1,3-Dimethyl-4,5-diphenyl-1*H*-imidazol-3-ium iodide (81g):



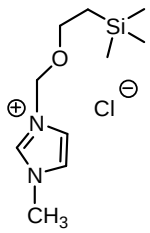
To 1-Methyl-4,5-diphenyl-1*H*-imidazole (0.15 g, 0.64 mmol) in toluene (5 mL) was added MeI (0.20 mL, 3.2 mmol) and stirred at 125 ℃ for 3 h. The solution was cooled to rt and the solids were filtered and washed with EtOAc to give **81g** as a pale yellow solid (0.22 g, 92%). mp: 214-216 ℃; ^{1}H NMR: δ = 10.37 (s, 1H), 7.44-7.37 (m, 6H), 7.29-7.27 (m, 4H), 3.91 (s, 6H); ^{13}C NMR: δ = 137.3, 132.4, 130.7, 130.5, 129.3, 124.7, 35.3; IR (cm^{-1}): 3067, 1577, 1468, 1218, 849, 766, 706; HR-APCIMS (*m/z*): Calcd. for $[M]^{+}$ $C_{17}H_{17}N_2$ is 249.1386 found 249.1392.

3-Benzyl-1-methyl-1*H*-imidazol-3-ium chloride (81h):



To 1-Methylimidazole (1.00 mL, 12.5 mmol) in THF (50 mL) was added BnCl (7.20 mL, 62.7 mmol) and heated at reflux overnight. The solvent was decanted and the resulting oil was triturated with Et_2O and dried under vacuum to give **81h** as an off-white semi-solid (2.20 g, 84%).[97] ^{1}H NMR: δ = 10.65 (s, 1H), 7.45-7.42 (m, 3H), 7.34-7.31 (m, 4H), 5.54 (s, 2H), 4.02 (s, 3H); ^{13}C NMR: δ = 138.0, 133.3, 129.5, 129.5, 129.0, 123.6, 121.8, 53.4, 36.7.

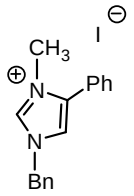
1-Methyl-3-trimethylsilylethoxymethyl-1*H*-imidazol-3-ium chloride (81i):



To 1-Methylimidazole (0.25 mL, 3.1 mmol) in THF (5 mL) was added SEM-Cl (0.66 mL, 3.7 mmol) and stirred at rt. Within minutes a second layer was observed, and after 30 minutes the layers were allowed to separate. The top layer was drawn off with a pipet, and the resulting oil was dried under vacuum to give **81i** as a clear, colorless oil (0.71 g, 91%). ^{1}H NMR: δ = 10.81 (s, 1H), 7.46 (s, 1H), 7.42 (s, 1H), 5.73 (s, 2H), 4.12 (s,3H), 3.65 (t, *J* = 8.0 Hz, 2H), 0.93 (t, *J* = 8.0 Hz), -0.02 (s, 9H); ^{13}C

NMR: δ = 138.5, 123.7, 120.7, 78.9, 68.6, 36.9, 18.0, -1.3; IR (cm^{-1}): 2953, 1575, 1557, 1248, 1157, 1096, 857, 833, 739; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{10}H_{21}N_2OSi$ is 231.1418 found 213.1424.

1-Benzyl-3-methyl-4-phenyl-1*H*-imidazol-3-ium iodide (81j):



To 1-Benzyl-4-phenyl-imidazole (0.20 g, 0.85 mmol) in THF (10 mL) was added MeI (0.16 mL, 2.6 mmol) and heated at reflux overnight. The solvent was removed and the resulting residue was triturated with EtOAc, then Et_2O to give **81j** as a yellow semi-solid (0.30 g, 94%). ^{1}H NMR: δ = 10.31 (s, 1H), 7.59-7.56 (m, 2H), 7.47-7.45 (m, 3H), 7.39-7.36 (m, 5H), 7.30-7.29 (m, 1H), 5.62 (s, 2H), 3.90 (s, 3H); ^{13}C NMR: δ = 137.3, 136.0, 132.8, 130.8, 129.7, 129.6, 129.5, 129.5, 124.6, 119.1, 53.6, 35.4; IR (cm^{-1}): 3027, 1556, 1495, 1443, 1146, 762, 741, 697; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{17}H_{17}N_2$ is 249.1386 found 249.1395.

General procedure for oxidation of 1-Benzyl-3-methyl-1*H*-benzimidazol-3-ium iodide (81a):

Method A: A benzimidazolium salt (0.29 mmol) in THF (10 mL) was cooled to 0 ℃ and NaH (60% dispersion in mineral oil, 0.017 g, 0.43 mmol) was added and stirred under N_2 at 0 ℃ for 10 min. NCS (0.050 g, 0.37 mmol) was added and stirred for 20 min. 2M NaOH (5 mL) was added and the reaction mixture was allowed to come to room temp and stir for 10 min. The reaction was diluted with water (10 mL) and the aqueous layer extracted with EtOAc (2x15 mL). The combined organic extracts were washed with water (10 mL), followed by brine (10 mL), dried (anhyd. Na_2SO_4), and concentrated.

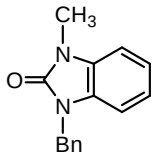
Method B: To a benzimidazolium salt (0.29 mmol) in THF (10 mL) was added 1M K_2CO_3/Na_2CO_3 or 2M NaOH (1.1 mmol) followed by NCS (0.050 g, 0.37 mmol) and stirred at room temp for 1 h (except in the case of Na_2CO_3 which required stirring for 18 h). The reaction was diluted with water (10 mL) and the aqueous layer extracted with EtOAc (2x15 mL). The combined organic extracts were washed with water (10 mL), followed by brine (10 mL), dried (anhyd. Na_2SO_4), and concentrated.

Method C: Benzimidazolium salt (0.34 mmol) in THF (10 mL) was cooled to 0 ℃ and 5% NaOCl (5 mL) was added. The reaction was allowed to come to room temp and stir for 10 min. The reaction was diluted with water (10 mL) and the aqueous layer extracted with EtOAc (2 x 15 mL). The combined organic extracts were washed with water (10 mL), followed by brine (10 mL), dried (anhyd. Na_2SO_4), and concentrated.

General procedure for the oxidation of 1-Benzyl-3-methyl-4,5-diphenyl-1*H*-imidazol-3-ium iodide (81f):

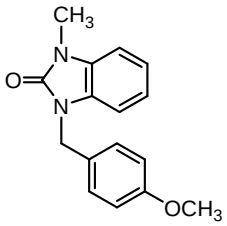
Method D: To an imidazolium salt (0.10 g, 0.22 mmol) in THF (5 mL) was added 1M NaOH (1.0 mL, 0.88 mmol) followed by slow, dropwise addition of 5% NaOCl (0.40 mL) over about 10 minutes at room temp. The reaction was diluted with water and extracted with EtOAc (2 x 10 mL). The combined organic extracts were washed with water (10 mL), followed by brine (10 mL), dried (anhyd. Na_2SO_4), and concentrated.

1-Benzyl-3-methyl-1,3-dihydro-2*H*-benzimidazol-2-one (82a):



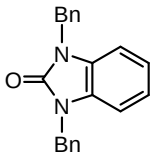
From **81a** (0.12 g, 0.34 mmol) following Method C and purification by column chromatography (4:6 EtOAc/Hexane), **82a** was obtained as a white solid (0.070 g, 85%). mp: 87-89 ℃ (lit. 87-88 ℃); [15] ^{1}H NMR: δ = 7.34-7.29 (m, 4H), 7.27-7.25 (m, 1H), 7.09-7.06 (m, 1H), 7.02-6.97 (m, 2H), 6.87 (d, *J* = 7.5 Hz, 1H), 5.08 (s, 2H), 3.46 (s, 3H); ^{13}C NMR: δ = 154.7, 136.5, 130.2, 129.3, 128.8, 127.8, 127.6, 121.4, 121.3, 108.3, 107.5, 45.0, 27.3; IR (cm^{-1}): 3043, 2946, 1705, 1494, 1435, 1398, 1246, 1121, 1003, 731.

1-(4-methoxybenzyl)-3-methyl-1,3-dihydro-2*H*-benzimidazol-2-one (82b):



From **81b** (0.12 g, 0.31 mmol) following Method C and purification by column chromatography (6:4 EtOAc/Hexane), **82b** was obtained as a white solid (0.069 g, 81%). mp: 102-104 ℃; ^{1}H NMR: δ = 7.27 (d, *J* = 8.6 Hz, 2H), 7.06 (td, *J* = 7.5 Hz, *J* = 8.0 Hz, 1H), 7.00 (td, *J* = 8.0 Hz, *J* = 8.6 Hz, 1H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.89 (d, *J* = 7.5 Hz, 1H), 6.83 (d, *J* = 8.6 Hz, 2H), 5.01 (s, 2H), 3.76 (s, 3H), 3.45 (s, 3H); ^{13}C NMR: δ = 159.2, 154.6, 130.2, 129.2, 129.0, 128.6, 121.3, 114.2, 108.3, 107.5, 55.3, 44.5, 27.3; IR (cm^{-1}): 2972, 1699, 1609, 1513, 1499, 1245, 1170, 1023, 740, 724; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{16}H_{16}N_2O_2Na$ is 291.1104 found 291.1115, Calcd. for $[2M+Na]^+$ is 559.2316 found 559.2346.

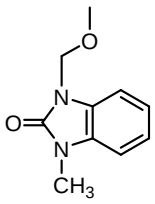
1,3-Dibenzyl-1,3-dihydro-2*H*-benzimidazol-2-one (82c):



From **81c** (0.15 g, 0.45 mmol) following Method C and purification by column chromatography (3:7 EtOAc/Hexane), **82c** was obtained as an off-white solid (0.12 g, 88%). mp: 110-112 ℃ (lit. 110 ℃); [98] ^{1}H NMR: δ = 7.35-7.26 (m, 10H),

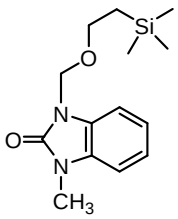
6.97 (dd, *J* = 5.5 Hz, *J* = 3.4 Hz, 2H), 6.88 (dd, *J* = 5.5 Hz, *J* = 3.4 Hz, 2H), 5.12 (s, 4H); ^{13}C NMR: δ = 154.7, 136.4, 129.4, 128.9, 127.8, 127.6, 121.5, 108.4, 45.1; IR (cm^{-1}): 3025, 2926, 1691, 1489, 1436, 1407, 1356, 1164, 750, 696, 659.

3-Methoxymethyl-1-methyl-1,3-dihydro-2*H*-benzimidazol-2-one (82d):



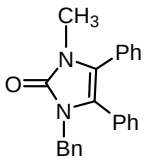
From **81d** (0.10 g, 0.47 mmol) following Method C and purification by column chromatography (1:1 EtOAc/Hexane), **82d** was obtained as a white solid (0.073 g, 81%). mp: 68-70 ℃; ^{1}H NMR: δ = 7.18-7.10 (m, 3H), 7.01-6.98 (m, 1H), 5.30 (s, 2H), 3.43 (s, 3H), 3.36 (s, 3H); ^{13}C NMR: δ = 154.6, 130.2, 128.7, 122.1, 121.7, 108.7, 107.7, 72.6, 56.4, 27.2; IR (cm^{-1}): 2937, 1689, 1622, 1501, 1380, 1031, 907, 750, 721, 636; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{10}H_{12}N_2O_2Na$ is 215.0791 found 215.0782; Calcd. for $[2M+Na]^+$ $C_{20}H_{24}N_4O_4Na$ is 407.1690 found 407.1708.

1-Methyl-3-trimethylsilylethoxymethyl-1,3-dihydro-2*H*-benzimidazol-2-one (82e):



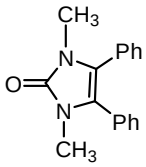
From **81e** (0.036 g, 0.12 mmol) following Method C and purification by column chromatography (2:8 EtOAc/Hexane), **82e** was obtained as a colorless oil[15] (0.026 g, 79%). ^{1}H NMR: δ = 7.18-7.15 (m,1H), 7.13-7.09 (m, 2H), 6.99-6.96 (m, 1H), 5.31 (s, 2H), 3.60 (t, *J* = 8.3 Hz, 2H), 3.41 (s, 3H), 0.91 (t, *J* = 8.3 Hz, 2H), -0.04 (s, 9H); ^{13}C NMR: δ = 154.5, 130.2, 128.8, 122.0, 121.6, 108.8, 107.5, 70.8, 66.2, 27.2, 17.9, -1.3; IR (cm^{-1}): 2951, 1694, 1449, 1374, 1247, 1072, 832, 695.

1-Benzyl-3-methyl-4,5-diphenyl-1,3-dihydro-2*H*-imidazol-2-one (82f):



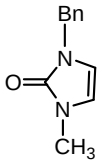
From **81f** (0.10 g, 0.22 mmol) following Method D and purification by column chromatography (4:6 EtOAc/Hexane), **82f** was obtained as a white solid (0.059 g, 79%). mp: 122-124 ℃; ^{1}H NMR: δ = 7.25-7.24 (m, 4H), 7.23-7.21 (m, 2H), 7.20-7.18 (m, 1H), 7.16-7.14 (m, 2H), 7.07-7.06 (m, 2H), 7.03-7.01 (m, 2H), 4.85 (s, 2H), 3.29 (s, 3H); ^{13}C NMR: δ = 154.0, 138.0, 130.7, 130.1, 129.1, 129.1, 128.5, 128.2, 128.0, 127.6, 127.3, 121.6, 121.1, 45.4, 29.3; IR (cm^{-1}): 3060, 2948, 1674, 1600, 1498, 1450, 1387, 748, 703; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{23}H_{21}N_2O$ is 341.1648 found 341.1633; Calcd. for $[M+Na]^+$ $C_{23}H_{20}N_2ONa$ is 363.1468 found 363.1472; Calcd. for $[2M+Na]^+$ $C_{46}H_{40}N_4O_2Na$ is 703.3043 found 703.3056.

1,3-Dimethyl-4,5-diphenyl-1,3-dihydro-2*H*-imidazol-2-one (82g):



From **81g** (0.060 g, 0.16 mmol) following Method D and purification by column chromatography (silica gel was neutralized with Et_3N prior to purification) (8:2 EtOAc/Hexane to EtOAc), **82g** was obtained as a white, crystalline solid (0.030 g, 71%). mp: 186-189 ℃ (lit. 203 ℃; [99] 183-184 ℃); [100] ^{1}H NMR: δ = 7.29-7.26 (m, 6H), 7.16-7.14 (m, 4H), 3.24 (2, 6H); ^{13}C NMR: δ = 154.0, 130.2, 129.2, 128.6, 128.0, 121.3, 29.1; IR (cm^{-1}): 2873, 1680, 1458, 1418, 1379, 1021, 778, 767, 746, 709; HR-APCIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{17}H_{16}N_2O$ is 265.1335 found 265.1331.

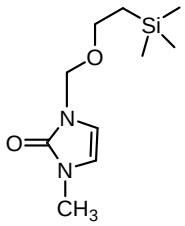
3-Benzyl-1-methyl-1,3-dihydro-2*H*-imidazol-2-one (82h):



From **81h** (0.20 g, 0.96 mmol) following Method D and purification by column chromatography (EtOAc), **82h** was obtained as a white solid (0.070 g, 39%). mp: 60-62 ℃ (lit. 63-64 ℃); [15] ^{1}H NMR: δ = 7.34-7.26 (m, 3H), 7.25-7.23 (m, 2H), 6.15 (d, *J* =

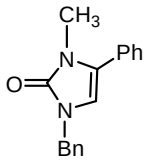
3.4 Hz, 1H), 6.09 (d, *J* = 3.4 Hz, 1H), 4.76 (s, 2H), 3.26 (s, 3H); ^{13}C NMR: δ = 153.3, 137.0, 128.8, 127.9, 127.8, 111.8, 110.0, 47.3, 30.6; IR (cm^{-1}): 3118, 2944, 1659, 1643, 1477, 1450, 1408, 1373, 1241, 810, 739.

1-Methyl-3-trimethylsilylethoxymethyl-1,3-dihydro-2*H*-imidazol-2-one (82i):



From **81i** (0.15 g, 0.60 mmol) following Method D and purification by column chromatography (1:1 EtOAc/Hexane), **82i** was obtained as a colorless oil[15] (0.050 g, 36%). ^{1}H NMR: δ = 6.31 (d, *J* = 2.9 Hz, 1H), 6.17 (d, *J* = 2.9 Hz, 1H), 4.98 (s, 2H), 3.55 (t, *J* = 8.0 Hz, 2H), 3.24 (s, 3H), 0.89 (t, *J* =8.0 Hz, 2H), -0.02 (s, 9H); ^{13}C NMR: δ = 153.3, 112.5, 109.7, 72.7, 66.1, 30.4, 17.9, -1.3; IR (cm^{-1}): 2951, 1675, 1467, 1412, 1246, 1080, 917, 857, 833, 761.

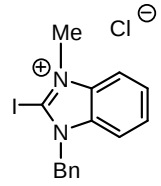
1-Benzyl-3-methyl-4-phenyl-1,3-dihydro-2*H*-imidazol-2-one (82j):



From **81j** (0.30 g, 0.80 mmol) following Method D and purification by column chromatography (6:4 EtOAc/Hexane), **82j** was obtained as a yellow oil (0.14 g, 67%). ^{1}H NMR: δ = 7.40-7.30 (m, 10H), 6.16 (s, 1H), 4.85 (s, 2H), 3.33 (s, 3H); ^{13}C NMR: δ = 154.0, 137.0, 128.9, 128.1, 128.0, 127.9, 127.7, 125.2, 107.7, 107.5, 47.3, 29.4; IR (cm^{-1}): 3031, 2929, 1668, 1453, 1403, 1174, 724, 695; HR-ESIMS (*m/z*): Calcd for $[M+Na]^+$ is $C_{17}H_{16}N_2ONa$ is 287.1155 found 287.1168.

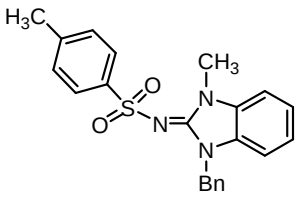
1-Benzyl-2-iodo-3-methyl-1*H*-benzimidazol-3-ium chloride (88):

To 1-Benzyl-2-chloro-1*H*-benzimidazole (**87**)[101] (0.50 g, 2.1 mmol) in toluene (10 mL) was added MeI (0.64 mL, 10 mmol) and heated at 125 ℃ for 18 h. The solids were filtered and



washed with EtOAc to give **88** as a pale yellow solid (0.78 g, 98%). mp: 222-223 ℃; ^{1}H NMR (DMSO-d_6): δ = 8.05 (d, *J* = 8.6 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.59 (t, *J* = 7.5 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.37-7.28 (m, 5H), 5.81 (s, 2H), 4.08 (s, 3H); ^{13}C NMR (DMSO-d_6): δ = 134.6, 134.2, 133.1, 129.4, 128.9, 127.7, 126.9, 126.8, 121.7, 114.2, 113.8, 52.2, 39.7; IR (cm^{-1}): 2906, 1455, 1385, 1309, 1171, 1029, 744, 700; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{15}H_{14}IN_2$ is 349.0196 found 349.0205.

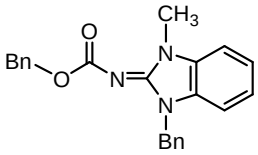
_N_-[(2_E_)-1-benzyl-3-methyl-1,3-dihydro-2_H_-benzimidazol-2-ylidene]-4-methylbenzene sulfonamide (90):



To **81a** (0.075 g, 0.21 mmol) in DMF (3 mL) was added chloramine-T trihydrate (**89**) (0.13 g, 0.47 mmol) and stirred at rt for 1 h. The reaction was diluted with water (5 mL) and extracted with EtOAc (2 x 5 mL). The combined organic extracts were washed with 2M NaOH (5 mL), water (5 mL), and then brine (5 mL). The organics were dried (anhyd. Na_2SO_4) and concentrated. The resulting solids were recrystallized from CH_2Cl_2/hexane to provide **90** as pale yellow crystals (0.080 g, 95%). mp: 165-167 °C; ^{1}H NMR: δ = 7.82 (d, *J* = 8.6 Hz, 2H), 7.26-7.24 (m, 5H), 7.20-7.16 (m, 5H), 7.06 (d, *J* = 8.0 Hz, 1H), 5.18 (s, 2H), 3.94 (s, 3H), 2.37 (s, 3H); ^{13}C NMR: δ = 148.6, 142.7, 141.5, 135.1, 131.3, 129.7, 129.2, 128.9, 128.0, 127.5, 125.9, 123.7, 123.4, 110.3, 109.9, 47.0, 32.4, 21.5; IR (cm^{-1}): 1579, 1567, 1445, 1245, 1132, 1088; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{22}H_{22}N_3O_2S$ is 392.1427 found 392.1420.

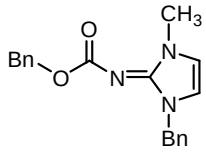
1-Benzyl-2-benzyloxycarbonylimino-3-methyl-1,3-dihydro-2_H_-benzimidazole (95a):

To benzyl carbamate (**92**) (0.095 g, 0.63 mmol) in CH_2Cl_2 (10 mL) was added *t*-BuOCl (0.080 mL, 0.71 mmol) and stirred at rt for 30 min. The reaction was cooled to 0 ℃ and DBU (0.13



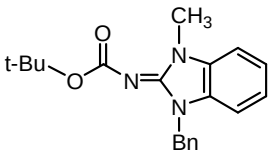
mL, 0.86 mmol) was added followed by **81a** (0.10 g, 0.29 mmol) and stirred at rt for 30 min. The reaction was quenched with water (10 mL), the layers were separated and the aqueous layer extracted with CH_2Cl_2 (5 mL). The combined organic extracts were washed with 2M NaOH (5 mL), water (5 mL) and brine (5 mL). The organic layer was dried (anhyd. Na_2SO_4), concentrated, and purified by column chromatograpy (7:3 EtOAc/Hexane) to give **95a** as a white solid (0.090 g, 85%). mp: 76-77 ℃; ^{1}H NMR: δ = 7.44 (d, *J* = 7.5 Hz, 2H), 7.32 (t, *J* = 6.9 Hz, 2H), 7.29-7.26 (m, 3H), 7.25-7.18 (m, 5H), 7.14 (td, *J* = 2.3 Hz, *J* = 7.5 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 5.29 (s, 2H), 5.20 (s, 2H), 3.57 (s, 3H); ^{13}C NMR: δ = 159.0, 153.4, 137.9, 135.3, 131.5, 130.2, 128.9, 128.4, 128.1, 128.0, 127.6, 127.5, 123.1, 123.0, 110.1, 109.3, 67.2, 47.0, 31.0; IR (cm^{-1}): 3031, 2947, 1651, 1575, 1493, 1427, 1376, 1308, 1261, 1222, 1174; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{23}H_{22}N_3O_2$ is 372.1707 found 372.1709.

1-Benzyl-2-benzyloxycarbonylimino-3-methyl-2,3-dihydro-1*H*-imidazole (95b):



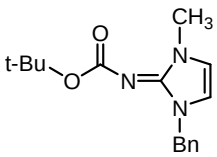
Prepared following the same procedure as **95a** from **81h** (0.15 g 0.72 mmol) and purified by column chromatography (8% MeOH in EtOAc) to give **95b** as a white solid (0.082 g, 36%). mp: 93-95 ℃ (li t. 93-95 ℃); [71] ^{1}H NMR: δ = 7.43 (d, *J* = 7.5 Hz, 2H), 7.32-7.29 (m, 5H), 7.24-7.21 (m, 13H), 6.49 (d, *J* = 2.9 Hz, 1H), 6.37 (d, *J* = 2.9 Hz, 1H), 5.16 (s, 2H), 4.98 (s, 2H), 3.46 (s, 3H); ^{13}C NMR: δ = 159.4, 150.8, 138.4, 135.2, 129.0, 128.5, 128.4, 128.3, 127.9, 127.4, 116.5, 114.1, 66.8, 50.0, 34.0; IR (cm^{-1}): 3031, 2944, 1631, 1549, 1486, 1454, 1381, 1313, 1233, 1165, 1066.

1-Benzyl-2-*tert*-butylcarbonylimino-3-methyl-1,3-dihydro-2*H*-benzimidazole (96a):



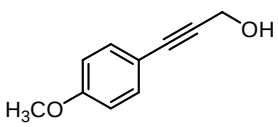
Prepared following the same procedure as **95a** from **81a** (0.074 g, 0.63 mmol) and purified by column chromatography (1:1 EtOAc/Hexane) to give **96a** as a white solid (0.069 g, 72%). mp: 141-142 ℃; ^{1}H NMR: δ = 7.29-7.26 (m, 3H), 7.25-7.23 (m, 2H), 7.18-7.16 (m, 2H), 7.11-7.07 (m, 1H), 6.97 (d, *J* = 8.6 Hz, 1H), 5.30 (s, 2H), 3.58 (s, 3H), 1.53 (s, 9H); ^{13}C NMR: δ = 159.0, 153.2, 135.5, 131.6, 130.3, 128.8, 127.8, 127.5, 122.8, 122.7, 110.0, 108.9, 78.2, 46.9, 31.1, 28.5; IR (cm^{-1}): 3061, 2972, 1647, 1581, 1495, 1457, 1361, 1320, 1151, 1056; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{20}H_{24}N_3O_2$ is 338.1863 found 338.1858; Calcd. for $[M+Na]^+$ $C_{20}H_{23}N_3O_2Na$ is 360.1682 found 360.1684.

1-Benzyl-2-*tert*-butylcarbonylimino-3-methyl-2,3-dihydro-1*H*-imidazole (96b):



Prepared following the same procedure as **95a** from **81h** (0.15 g, 0.72 mmol) and purified by column chromatography (8% MeOH in EtOAc) to give **96b** as a clear, colorless oil (0.15 g, 71%). (lit. mp: 88-91 ℃);[71] ^{1}H NMR: δ = 7.33-7.29 (m, 3H), 7.24-7.22 (m, 2H); 6.46 (d, *J* = 2.3 Hz, 1H), 6.30 (d, *J* = 2.3 Hz, 1H), 4.98 (s, 2H), 3.46 (s, 3H), 1.50 (s, 9H); ^{13}C NMR: δ = 159.5, 150.9, 135.5, 129.0, 128.5, 128.3, 116.2, 114.0, 77.0, 49.9, 34.2, 28.7; IR (cm^{-1}): 2973, 1626, 1548, 1483, 1453, 1361, 1318, 1240, 1153, 1066.

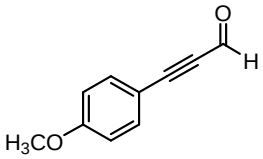
3-(4-Methoxyphenyl)prop-2-yn-1-ol (116):



To 4-Iodoanisole (**114**) (20.0 g, 0.0854 mol) in THF (400 mL) was added propargyl alcohol (**115**) (7.5 mL, 0.13 mol) and Et_3N (24.0 mL, 0.172 mol). $(PPh_3)_2PdCl_2$ (0.60 g, 0.85 mmol) and CuI (0.81 g, 4.2

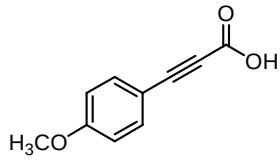
mmol) were added and the reaction mixture was stirred at rt under N_2 for 20 h. Water (10 mL) was added and the solvent reduced. EtOAc (100 mL) was added and the solids filtered through Celite and washed with EtOAc. The organic layer was washed with water (2x), followed by brine, dried and concentrated. The crude residue was purified by column chromatography (3:7 EtOAc/Hexane) to give **116** as an orange solid (11.7 g, 85%). mp: 65-68 ℃ (lit. 52 ℃);[73] ^{1}H NMR: δ = 7.37 (d, *J* = 8.9 Hz, 2H), 6.83 (d, *J* = 8.9 Hz, 2H), 4.48 (s, 2H), 3.81 (s, 3H); ^{13}C NMR: δ = 159.8, 133.3, 114.7, 114.0, 85.9, 85.7, 55.4, 51.8.

3-(4-methoxyphenyl)prop-2-ynal (117):



To **116** (1.0 g, 6.2 mmol) in CH_2Cl_2 (30 mL) was added MnO_2 (~85% act.) (5.0 g) and stirred at 45 ℃ for 18-48 h until the reaction was deemed complete by TLC. The solids were filtered through Celite and washed with CH_2Cl_2. The filtrate was reduced to give **117** as a light brown solid (0.83 g, 85%). mp: 48-50 ℃ (lit. 47-48.5 ℃);[102] ^{1}H NMR: δ = 9.39 (s, 1H), 7.55 (d, *J* = 8.9 Hz, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 3.85 (s, 3H); ^{13}C NMR: δ = 176.8, 162.2, 135.5, 114.6, 111.2, 96.7, 88.8, 55.5.

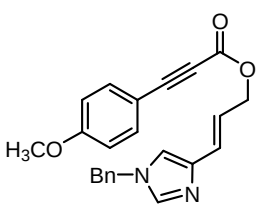
3-(4-Methoxyphenyl)prop-2-ynoic acid (118):



To **117** (4.4 g, 0.028 mol) in *t*-BuOH (100 mL) was added cylcohexene (8.9 mL, 0.084 mol) and cooled to 0 ℃. A solution of $NaOCl_2$ (6.2 g, 0.069 mol) and $NaH_2PO_4 \bullet H_2O$ (16 g, 0.12 mol) in H_2O (100 mL) was added slowly in several portions. The reaction was allowed to come to rt and stirred for 10 h. The aqueous layer was made basic (pH~9) with the addition of solid Na_2CO_3 and extracted with EtOAc (3x). These organic extracts were later discarded. The aqueous layer was then acidified (pH~5) with the addition of solid citric acid and extracted with EtOAc

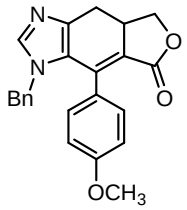
(3x). The combined organic extracts were dried and concentrated to give **118** as a yellow solid (2.9 g, 60%). mp: 130-132 ℃ (lit. 144-145 ℃); [103] ^{1}H NMR (CD_3OD): δ = 7.50 (d, *J* = 8.9 Hz, 2H), 6.40 (d, *J* = 8.9 Hz, 2H), 3.81 (s, 3H); ^{13}C NMR (CD_3OD): δ = 161.9, 155.7, 134.5, 114.2, 111.2, 86.3, 79.9, 54.6.

(2*E*)-3-(1-Benzyl-1*H*-imidazol-4-yl)prop-2-enyl 3-(4-methoxyphenyl)propynoate (107):



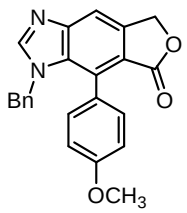
118 (2.00 g, 11.4 mmol), alcohol **133a** (2.94 g, 13.7 mmol), DMAP (0.14 g, 1.2 mmol) and CSA (0.16 g, 0.70 mmol) were dissolved in CH_2Cl_2 (50 mL) and cooled to -78 ℃ under N_2. DCC (3.50 g, 17.1 mmol) dissolved in CH_2Cl_2 (20 mL) was added dropwise. The mixture was allowed to come to rt and stir for 2 h. The solids were filtered through Celite and washed with CH_2Cl_2. The filtrate was reduced and the resulting brown residue was purified by column chromatography (8:2 EtOAc/Hexane) to afford **107** as an off-white solid (2.53 g, 56%). mp: 65-67 ℃; ^{1}H NMR: δ = 7.52 (s, 1H), 7.49 (d, *J* = 2.1 Hz, 2H), 7.36-7.31 (m, 3H), 7.15-7.13 (m, 2H), 6.86 (m, 3H), 6.58 (d, *J* = 15.6 Hz, 1H), 6.47-6.38 (dt, *J* = 15.5 Hz, *J* = 6.2 Hz, 1H), 5.06 (s, 2H), 4.82 (d, *J* = 6.6 Hz, 2H), 3.81 (s, 3H); ^{13}C NMR: δ = 161.6, 154.2, 139.8, 137.9, 135.9, 135.0, 129.1, 128.5, 127.4, 127.0, 120.6, 117.9, 114.4, 111.5, 87.3, 80.1, 66.5, 55.5, 51.0; IR (KBr, cm^{-1}): 3105, 2195, 1899, 1697, 1602, 1567, 1541; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{23}H_{21}N_2O_3$ is 373.1547 found 373.1564; Cald. for $[M+Na]^+$ $C_{23}H_{20}N_2O_3Na$ is 395.1366 found 395.1368; Calcd. for $[2M+H]^+$ $C_{46}H_{42}N_4O_6$ is 745.3021 found 745.3024; Calcd. for $[2M+Na]^+$ $C_{46}H_{40}N_4O_6Na$ is 767.2840 found 767.2844.

3-Benzyl-4-(4-methoxyphenyl)-3,7,7a,8-tetrahydro-5*H*-furo[3,4-*f*]benzimidazol-5-one (106):



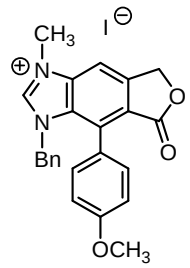
107 (2.54 g, 6.84 mmol) was dissolved in toluene (200 mL) and purged with N_2 for 15 minutes. The solution was then heated at 130 ℃ in a sealed tube for 48 h. The solvent was removed and the resulting solids washed with diethyl ether to afford **106** as an off-white solid (2.14 g, 84%). mp: 199-201 ℃; ^{1}H NMR: δ = 7.44 (s, 1H), 7.24-7.10 (m, 5H), 6.85 (dd, *J* = 5.7 Hz, *J* = 1.8 Hz, 2H), 6.64 (dd, *J* = 5.1, *J* = 1.5 Hz, 2H), 4.68 (dd, *J* = 16.5, *J* = 7.5 Hz, 2H), 4.43 (d, *J* = 15.6 Hz, 1H), 4.01 (t, *J* = 8.7 Hz, 1H), 3.83 (s, 3H), 3.67-3.58 (m, 1H), 3.06 (dd, *J* = 15.0, *J* = 8.7 Hz, 1H), 2.73 (dd, *J* = 17.1, *J* = 15.9 Hz, 1H); ^{13}C NMR: δ = 168.1, 160.3, 144.4, 141.2, 139.4, 135.8, 128.7, 128.4, 128.1, 126.6, 123.9, 117.2, 70.6, 55.4, 50.3, 38.1, 28.0; IR (KBr, cm^{-1}): 3465, 2946, 1737, 1621, 1512, 1252; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{23}H_{21}N_2O_3$ is 373.1547 found 373.1571; Calcd. for $[2M+H]^+$ $C_{46}H_{42}N_4O_6$ is 745.3021 found 745.3045.

3-Benzyl-4-(4-methoxyphenyl)-3,7-dihydro-5*H*-furo[3,4-*f*]benzimidazole-5-one (119):



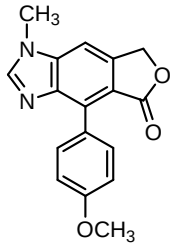
106 (7.0 g, 19 mmol) was dissolved in CH_2Cl_2 (500 mL) and MnO_2 (18.0 g, 188 mmol) was added. The mixture stirred at rt for 24 h. The solids were filtered and washed with CH_2Cl_2 followed by MeOH. The filtrate was concentrated to give **119** as an off-white solid (6.24 g, 89%). mp: 176-178 ℃; ^{1}H NMR: δ = 8.01 (s, 1H), 7.82 (s, 1H), 7.21-7.16 (m, 3H), 7.06 (d, *J* = 8.9 Hz, 2H), 6.83 (d, *J* = 8.9 Hz, 2H), 6.59 (d, *J* = 7.5 Hz, 2H), 5.35 (s, 2H), 4.97 (s, 2H), 3.84 (s, 3H); ^{13}C NMR: δ = 170.0, 159.8, 149.4, 148.9, 140.5, 135.8, 132.7, 131.0, 128.7, 128.0, 127.6, 126.2, 124.2, 118.5, 113.3, 112.4, 68.0, 55.3, 50.3; IR (KBr, cm^{-1}): 2947, 1739, 1614, 1500, 1347, 1253; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{23}H_{18}N_2O_3Na$ is 393.1210 found 393.1210; Calcd. for $[2M+Na]^+$ $C_{46}H_{36}N_4O_6Na$ is 763.2527 found 763.2521.

3-Benzyl-4-(4-methoxyphenyl)-1-methyl-5-oxo-5,7-dihydro-3*H*-furo[3,4-f]benzimidazol-1-ium iodide (105):



119 (0.37 g, 1.0 mmol) was dissolved in toluene (20 mL) and MeI (0.20 mL, 3.2 mmol) was added. The mixture was heated at 120 ℃ for 24 h during which additional aliquots of methyl iodide (0.5 equiv) were added until no starting material could be detected by TLC. The solids were filtered and washed with EtOAc to afford **105** as a pale yellow solid (0.48 g, 93%). mp: 198-200 ℃; ^{1}H NMR: δ = 10.50 (s, 1H), 8.03 (s, 1H), 7.25-7.12 (m, 5H), 6.94-6.87 (m, 4H), 5.43 (s, 2H), 5.27 (s, 2H), 4.32 (s, 3H), 3.86 (s, 3H); ^{13}C NMR: δ = 167.7, 160.8, 146.4, 144.8, 136.8, 132.2, 131.4, 131.1, 130.1, 129.2, 127.7, 122.8, 121.0, 113.9, 106.7, 68.0, 55.5, 53.2, 35.4; IR (cm^{-1}): 2939, 1775, 1612, 1575, 1501, 1451, 1378, 1337; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{24}H_{21}N_2O_3$ is 385.1547 found 385.1548.

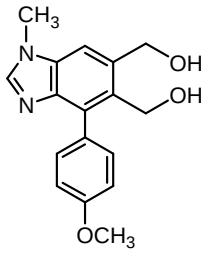
4-(4-Methoxyphenyl)-1-methyl-1,7-dihydro-furo[3,4-f]benzimidazol-5-one (120b):



To **105** (0.44 g, 0.86 mmol) was added EtOH (50 mL) and sat. $NaHCO_3$ (5 mL), followed by 10% Pd/C (0.10 g). The reaction mixture was heated at 80 ℃ under H_2 (1 atm) for 24 h. The solids were filtered and washed with CH_2Cl_2/MeOH followed by water. The aqueous layer was then acidified to pH = 5 with 3M HCl and stirred for 2 h. The aqueous layer was extracted multiple times with CH_2Cl_2. The combined organic extracts were dried (Na_2SO_4) and concentrated. The resulting solid was recrystallized from MeOH to afford **120b** as a white solid (0.19 g, 76%). mp: >250 ℃; ^{1}H NMR (DMSO-d_6): δ = 8.32 (s, 1H), 7.69 (s, 1H), 7.55 (d, *J* = 9.2 Hz, 2H), 6.98 (d, *J* = 9.2 Hz, 2H), 5.39 (s, 2H), 3.89 (s, 3H), 3.80 (s, 3H); ^{13}C NMR (DMSO-d_6): δ = 170.5, 159.7, 147.9, 143.1, 142.6, 139.8, 133.4, 133.2, 125.4, 114.6, 113.2, 102.9, 68.4, 55.7, 31.6; IR (KBr, cm^{-1}): 3044, 2957, 2835, 1739, 1603, 1501, 1453; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$

$C_{17}H_{15}N_2O_3$ is 295.1077 found 295.1092; Calcd. for $[M+Na]^+$ $C_{17}H_{14}N_2O_3Na$ is 317.0897 found 317.0910.

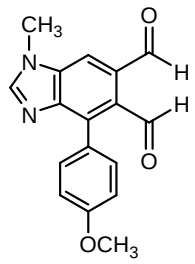
[4-(4-Methoxyphenyl)-1-methyl-1*H*-benzimidazole-5,6-diyl]dimethanol (121):



120b (0.19 g, 0.65 mmol) was dissolved in CH_2Cl_2 (5 mL) and cooled to -78 ℃ under N_2. DIBAL-H (1M in Hexane) (1.5 mL, 1.5 mmol) was added dropwise and the reaction mixture was allowed to warm to rt and stirred for 3 h. The reaction mixture was cooled to 0 ℃ and water was added slowly followed by MeOH. The solids were filtered and washed with CH_2Cl_2 and MeOH. The organic layer was separated and the aqueous layer was extracted multiple times with CH_2Cl_2. The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. The resulting white solid was triturated with Et_2O to afford **121** as a white solid (0.15 g, 79%). mp: 245-247 ℃; ^{1}H NMR (DMSO-d_6): δ = 8.02 (s, 1H), 7.58 (s, 1H), 7.43 (d, *J* = 8.6 Hz, 2H), 6.98 (d, *J* = 8.6 Hz, 2H), 5.25 (t, *J* = 5.5 Hz, 1H), 4.83 (d, *J* = 5.2 Hz, 2H), 4.79 (t, *J* = 4.8 Hz, 1H), 4.43 (d, *J* = 4.5 Hz, 2H), 3.81 (s, 3H), 3.79 (s, 3H); ^{13}C NMR (DMSO-d_6): δ = 158.8, 144.7, 141.6, 137.6, 134.2, 133.3, 132.5, 129.9, 129.8, 113.5, 108.4, 62.4, 58.5, 55.6, 31.2; IR (cm^{-1}): 3074, 2889, 1608, 1574, 1340, 1237, 1174, 1015; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{17}H_{19}N_2O_3$ is 299.1390 found 299.1411; Calcd. for $[M+H-H_2O]^+$ $C_{17}H_{21}N_2O_4$ is 281.1285 found 281.1291.

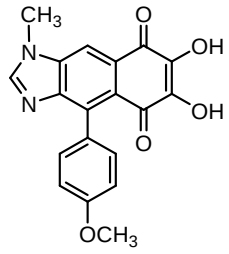
4-(4-Methoxyphenyl)-1-methyl-1*H*-benzimidazole-5,6-dicarbaldehyde (104):

121 (0.10 g, 0.34 mmol) was dissolved in CH_2Cl_2 and DBU (0.2 mL, 1.34 mmol) was added. The solution was cooled to -78 ℃ under N_2 and *N-tert*-butylbenzenesulfinimidoyl chloride (**122**) (0.22 g, 1.02 mmol) dissolved in CH_2Cl_2 (5 mL) was added dropwise. The mixture was stirred at



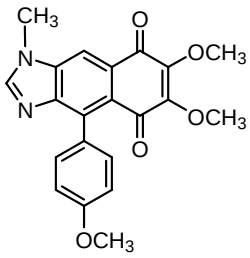
-78 ℃ for 30 min. The reaction was quenched with sat. $NaHCO_3$ and the organic layer separated. The aqueous layer was extracted with CH_2Cl_2 (2x). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. The resulting residue was purified by column chromatography (the silica gel was neutralized with 5% Et_3N prior to purification) (EtOAc to 2% MeOH in EtOAc) to afford **104** as an off-white solid (0.069 g, 70%). ^{1}H NMR: δ = 10.61 (s, 1H), 10.16 (s, 1H), 8.09 (s, 1H), 8.05 (s, 1H), 7.49 (d, 2H, *J* = 8.4 Hz), 7.08 (d, 2H, *J* = 8.4 Hz), 3.97 (s, 3H), 3.89 (s, 3H); ^{13}C NMR: δ = 193.5, 192.9, 160.2, 147.7, 145.3, 139.8, 136.7, 133.1, 132.7, 129.5, 125.1, 114.1, 109.7, 55.5, 31.7; IR (KBr, cm^{-1}): 3105, 2928, 2881, 1777, 1751, 1682, 1663, 1566; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{17}H_{15}N_2O_3$ is 295.1077 found 295.1110; Calcd. for $[M+Na]^+$ $C_{17}H_{14}N_2O_3Na$ is 317.0897 found 317.0938.

6,7-Dihydroxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]imidazole-5,8-dione (124):



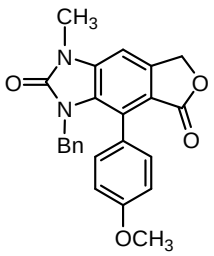
To **104** (0.074 g, 0.27 mmol) in EtOH (1.5 mL) was added **123** (0.050 g, 0.81 mmol) followed by the simultaneous addition of KCN (0.021 g, 0.33 mmol) in H_2O (0.5 mL) and Et_3N (0.040 mL, 0.27 mmol). The reaction was stirred at rt for 15 min and then quenched with 10% HCl until a pH=5 was reached. The aqueous layer was extracted with CH_2Cl_2 (3x) and discarded. The aqueous layer was reduced and the resulting solids filtered and washed with acetone to give **124** as a red-orange solid (0.035 g, 39%). ^{1}H NMR (DMSO-d_6): δ = 9.73 (brs, 2H), 8.34 (s, 1H), 8.18 (s, 1H), 7.16 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.6 Hz, 2H), 3.93 (s, 3H), 3.78 (s, 3H); ^{13}C NMR (DMSO-d_6): δ = 181.8, 181.3, 158.9, 148.8, 146.8, 141.9, 139.4, 137.1, 135.8, 131.1, 129.9, 127.5, 121.5, 113.4, 109.6, 55.6, 31.9; IR (cm^{-1}): 2981, 1642, 1627, 1608, 1337, 1243, 1211, 1021; HR-ESIMS (negative mode) (*m/z*): Calcd. for $[M-H]^-$ $C_{19}H_{13}N_2O_5$ is 349.0830 found 349.0832.

6,7-Dimethoxyoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]imidazole-5,8-dione (103):



To **124** (0.023 g, 0.066 mmol) in THF (3 mL) was added MeOH (1 mL) and TMS-CHN_2 (2.0M in Et_2O) (0.080 mL, 0.16 mmol) and stirred at rt for 1 h. The reaction was diluted with CH_2Cl_2 and washed with sat. $NaHCO_3$ (1x), dried (anhyd. Na_2SO_4) and concentrated. The resulting residue was purified by column chromatography (2:8 acetone/Hexane to 1:1 acetone/Hexane) to give **103** as an orange residue (0.016 g, 64%). ^{1}H NMR: δ = 8.24 (s, 1H), 8.00 (s, 1H), 7.28 (d, *J* = 8.6 Hz, 2H), 7.02 (d, *J* = 8.6 Hz, 2H), 4.10 (s, 3H), 4.01 (s, 3H), 3.95 (s, 3H), 3.86 (s, 3H); ^{13}C NMR: δ = 182.3, 182.0, 159.1, 148.9, 147.4, 146.5, 136.8, 130.6, 130.2, 128.8, 128.3, 122.8, 114.5, 113.8, 108.8, 61.4, 61.3, 55.3, 31.7; IR (cm^{-1}): 2924, 2852, 1658, 1618, 1515, 1456, 1341, 1307, 1245, 1215, 1054; HR-APCIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{21}H_{18}N_2O_5$ is 379.1288 found 379.1296.

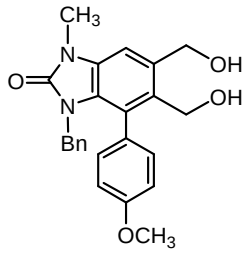
3-Benzyl-4-(4-methoxyphenyl)-1-methyl-3,7-dihydro-1*H*-furo[3,4-*f*]benzimidazole-2,5-dione (127):



105 (1.0 g, 1.9 mmol) in THF (40 mL) was cooled to 0 °C and 5% NaOCl (30 mL) was added dropwise. The reaction was allowed to come to rt and stir for 30 min. The reaction was diluted with EtOAc and the aqueous layer extracted with EtOAc (2x). The combined organic extracts were washed with H_20, then brine, dried (anhyd. Na_2SO_4) and concentrated. The resulting residue was purified by column chromatography (7:3 EtOAc/Hexane) to give **127** as an off-white solid (0.58 g, 75%). mp: 185-187 ℃; ^{1}H NMR: δ = 7.12-7.09 (m, 3H), 7.03 (s, 1H), 6.94 (d, *J* = 8.6 Hz, 2H), 6.77 (d, *J* = 8.9, 2H), 6.58-6.56 (m, 2H), 5.23 (s, 2H), 4.79 (s, 2H), 3.83 (s, 3H), 3.57 (s, 3H); ^{13}C NMR: δ = 169.8, 159.7, 155.6, 141.8, 136.4, 136.2, 131.3, 128.2,

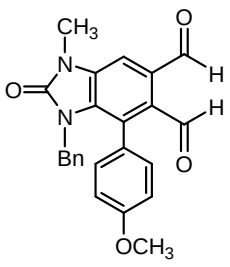
127.8, 127.2, 126.0, 124.0, 123.6, 117.1, 113.1, 99.5, 67.7, 55.3, 45.8, 27.8; IR (cm^{-1}): 1748, 1705, 1609, 1517, 1496, 1352, 1244, 1028; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{24}H_{20}N_2O_4Na$ is 423.1315 found 423.1316.

[3-Benzyl-4-(4-methoxyphenyl)-1-methyl-1*H*-benzimidazole-5,6-diyl]dimethanol-2-one (128):



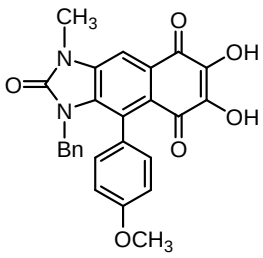
127 (1.4 g, 3.5 mmol) was dissolved in CH_2Cl_2 (25 mL) and cooled to -78 ℃ under N_2. DIBAL-H (1M in hexanes) (9.8 mL, 9.8 mmol) was added dropwise, then allowed to come to rt and stirred for 4 h. The reaction mixture was cooled to 0 ℃ and water was added slowly followed by MeOH. The solids were filtered through Celite and washed with CH_2Cl_2. Water was added to the filtrate and the organic layer separated. The aqueous layer was extracted with CH_2Cl_2 (2x). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. The resulting solids were washed with hexane, then Et_2O to give **128** as an off-white solid (1.17g, 83%). mp: 205-206 °C; ^{1}H NMR: δ = 7.11-7.09 (m, 3H), 7.03 (s, 1H), 6.87 (d, *J* = 8.6 Hz, 2H), 6.70 (d, *J* = 8.6 Hz, 2H), 6.59-6.57 (m, 2H), 4.81 (s, 2H), 4.64 (s, 2H), 4.38 (s, 2H), 3.82 (s, 3H), 3.46 (s, 3H); ^{13}C NMR: δ = 159.4, 155.5, 137.1, 134.0, 132.5, 131.5, 130.1, 128.1, 127.2, 126.9, 125.8, 125.6, 113.4, 108.5, 65.1, 59.5, 55.4, 45.4, 27.4; IR (cm^{-1}): 3313, 2930, 1669, 1611, 1515, 1398, 1244, 1181; HR-APCIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{24}H_{25}N_2O_4$ is 405.1809 found 405.1826; Calcd. for $[M+Na]^+$ $C_{24}H_{24}N_2O_4Na$ is 427.1628 found 427.1643.

3-Benzyl-4-(4-methoxyphenyl)-1-methyl-1*H*-benzimidazole-5,6-dicarbaldehyde-2-one (129):



128 (0.22 g, 0.54 mmol) was dissolved in CH_2Cl_2 (10 mL) and cooled to -78 ℃ under N_2. DBU (0.32 mL, 2.2 mmol) was added followed by **122** (0.38 g, 1.7 mmol) in CH_2Cl_2 (1 mL) and the reaction was stirred at -78 ℃ for 30 min. Sat. $NaHCO_3$ was added and the solution was allowed to come to rt. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (2x). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. The resulting residue was purified by column chromatography (the silica gel was neutralized with Et_3N prior to purification) (4:6 EtOAc/Hexane) to give **129** as an off-white solid (0.17 g, 77%). mp: 152-155 ℃ ; ^{1}H NMR: δ = 10.40 (s, 1H), 9.67 (s, 1H), 7.68 (s, 1H), 7.14-7.08 (m, 3H), 6.92 (d, *J* = 8.6 Hz, 2H), 6.74 (d, *J* = 8.6 Hz, 2H), 6.54 (d, *J* = 6.8 Hz, 2H), 4.74 (s, 2H), 3.84 (s, 3H), 3.59 (s, 3H); ^{13}C NMR: δ = 192.6, 192.0, 160.1, 155.6, 136.1, 133.7, 132.2, 131.9, 131.8, 130.2, 128.3, 128.0, 127.3, 125.6, 123.6, 113.7, 106.5, 55.5, 45.8, 27.9; IR (cm^{-1}): 2934, 1701, 1675, 1606, 1513, 1357, 1243, 1076; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{24}H_{20}N_2O_4Na$ is 423.1315 found 423.1309.

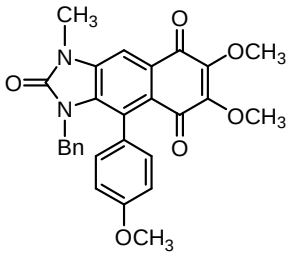
3-Benzyl-6,7-dihydroxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione:



To **129** (0.10 g, 0.25 mmol) in THF (3 mL) was added **123** (0.042 g, 0.75 mmol) followed by addition of KCN (0.029 g, 0.44 mmol) and K_2CO_3 (0.17 g, 1.3 mmol) in H_2O (1 mL). The reaction was stirred at rt for 5 min., then quenched with 6M HCl and diluted with H_2O (5 mL). The aqueous layer was extracted with EtOAc (2x). The combined organic extracts were washed wth H_2O, then brine, dried (anhyd. Na_2SO_4) and concentrated.

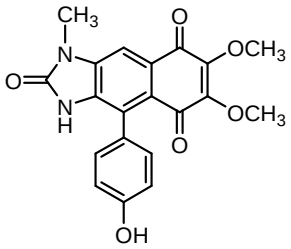
An orange-red residue was recovered and cold MeOH was added. The resulting solids were filtered to give the dihydroxyquinone as an orange solid (0.022 g, 20%). mp: 204-206 ℃; ^{1}H NMR (DMSO-d_6): δ = 9.65 (s, 1H), 9.60 (s,1H), 7.80 (s, 1H), 7.13-7.11 (m, 3H), 6.79 (d, *J* = 8.6 Hz, 2H), 6.63 (d, *J* = 8.6 Hz, 2H), 6.56-6.54 (m, 2H), 4.44 (s, 2H), 3.71 (s, 3H), 3.50 (s, 3H); ^{13}C NMR (DMSO-d_6): δ = 181.6, 181.1, 159.1, 155.1, 141.0, 138.4, 137.3, 134.0, 131.2, 130.5, 128.5, 127.7, 127.3, 127.0, 125.7, 125.0, 122.6, 113.5, 106.0, 55.6, 45.5, 28.1; IR (cm^{-1}): 3240, 1697, 1654, 1597, 1514, 1400, 1283, 1243

3-Benzyl-6,7-dimethoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (130):



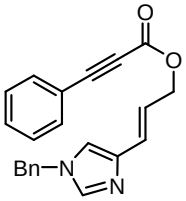
The dihydroxyquinone prepared above (0.022 g, 0.048 mmol) was dissolved in THF (3 mL) and MeOH (0.5 mL) was added followed by TMS-CHN_2 (2.0M in Et_2O) (0.07 mL, 0.15 mmol) and stirred at rt for 30 min. The reaction was diluted with EtOAc and washed with 2M Na_2CO_3 (2x), then brine. The organic extracts were dried (anhyd. Na_2SO_4), and concentrated. The resulting residue was purified by column chromatography (1:1 EtOAc/Hexane) to give **130** as an orange solid (0.015 g, 65%). mp: 176-178 ℃; ^{1}H NMR: δ = 7.82 (s, 1H), 7.13-7.11 (m, 3H), 6.79 (d, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 8.9 Hz, 2H), 6.58-6.56 (m, 2H), 4.60 (s, 2H), 4.03 (s, 3H), 3.92 (s, 3H), 3.81 (s, 3H), 3.60 (s, 3H); ^{13}C NMR: δ = 182.0, 181.5, 159.2, 155.4, 148.1, 145.2, 136.6, 134.0, 130.1, 128.2, 127.1, 127.0, 125.5, 124.0, 113.5, 105.4, 61.4, 61.3, 55.3, 45.8, 27.9; IR (cm^{-1}): 2950, 1716, 1651, 1619, 1601, 1455, 1241, 1211; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{28}H_{24}N_2O_6Na$ is 507.1527 found 507.1533.

4-(4-Hydroxyphenyl)-6,7-dimethoxy-1-methyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (131):



To **130** (0.030 g, 0.062 mmol) was added TfOH (2.5 mL) and heated at 55 ℃ in a vial for 4 h. The reaction was allowed to cool to rt and poured into EtOAc. Water was added and the solution neutralized with sat. $NaHCO_3$. The aqueous layer was extracted with EtOAc (2x). The combined organic extracts were washed with brine, dried (anhyd. Na_2SO_4) and concentrated. The resulting residue was purified by column chromatography (8:2 EtOAc/Hexane) to give **131** as an orange solid (0.010 g, 56%). mp: >290 ℃; ^{1}H NMR (DMSO-d_6): δ = 10.95 (s, 1H), 9.41 (s, 1H), 7.63 (s, 1H), 6.96 (d, *J* = 8.6 Hz, 2H), 6.75 (d, *J* = 8.6 Hz, 2H), 3.90 (s, 3H), 3.82 (s, 3H), 3.35 (s, 3H); ^{13}C NMR (DMSO-d_6): δ = 181.9, 181.7, 157.2, 155.3, 148.4, 145.6, 134.4, 133.1, 130.3, 127.0, 126.4, 124.5, 123.2, 115.8, 104.9, 61.2 (2C), 27.3; IR (cm^{-1})= 3200, 2921, 2851, 1702, 1656, 1609, 1515, 1105, 1059, 1033; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{20}H_{16}N_2O_6Na$ is 403.0901 found 403.0911; Calcd. for $[2M+Na]^+$ $C_{56}H_{48}N_4O_{12}Na$ is 783.1909 found 783.1914.

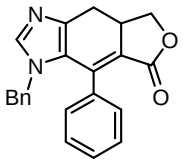
(2*E*)-3-(1-Benzyl-1*H*-imidazol-4-yl)prop-2-enyl 3-phenylpropynoate (68c):



To alcohol **113a** (6.0 g, 28 mmol) in CH_2Cl_2 (200 mL) was added acid **133** (4.9 g, 33 mmol), DMAP (0.34 g, 2.8 mmol), and CSA (0.39 g, 1.7 mmol). The solution was then cooled to 0 ℃ under N_2 and DCC (8.7 g, 42 mmol) in CH_2Cl_2 (20 mL) was added dropwise and the reaction was stirred at 0 ℃ for 2 h. The solids were filtered through Celite and washed with CH_2Cl_2. The filtrate was reduced and the resulting residue was purified by column chromatography (6:4 EtOAc/Hexane) to give **68c** as a pale yellow solid (6.6 g, 69%). mp: 57-61 ℃; ^{1}H NMR: δ = 7.11-7.53 (m, 11H), 6.34 (s, 1H), 6.36-6.59 (m, 2H), 5.02 (s, 2H), 4.81 (d, *J* = 6.3 Hz, 2H); ^{13}C NMR: δ = 153.9, 139.8,

138.0, 136.0, 133.1, 130.7, 129.1, 128.7, 128.4, 127.4, 127.3, 120.3, 119.7, 118.0, 86.4, 80.7, 66.7, 50.9; IR (NaCl disk, cm^{-1}): 3068, 2936, 2219, 1705, 1491, 1444, 1285, 1186, 1170, 966; HR-ESIMS (*m/z*): Calcd for $C_{22}H_{19}N_2O_2$ $[M+H]^+$ is 343.1441 found 343.1453; Calcd for $C_{44}H_{35}N_4O_4Na$ $[2M+Na]^+$ is 707.2629 found 707.2639.

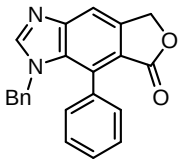
3-Benzyl-3,7,7a,8-tetrahydro-4-phenyl-5*H*-furo[3,4-*f*]benzimidazol-5-one (69c):



68c (6.6 g, 19 mmol) in toluene (600 mL) was degassed with N_2 for 20 min. The solution was then heated to 130 ℃ in a re-seal able pressure vessel for 48 h. The reaction was allowed to cool to rt at which point the product precipitated from the toluene. The solids were filtered and washed with Et_2O. The filtrate was reduced and the resulting solids washed with Et_2O and filtered to recover a second crop to give **69c** as an off-white solid (5.5 g, 83%). mp: 228-231 ℃; ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ = 7.80 (s, 1H), 7.14-7.34 (m, 8H), 6.50-6.52 (m, 2H), 4.88 (d, *J* = 16.2 Hz, 1H), 4.58 (t, *J* = 9.0 Hz, 1H), 4.16 (d, *J* = 15.9 Hz, 1H), 3.97 (t, *J* = 8.7 Hz, 1H), 3.52-3.61 (m, 1H), 2.91, (dd, *J* = 15.9, 15.6, Hz, 1H), 2.66 (dd, *J* = 17.4, 15.9 Hz, 1H); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ = 167.9, 144.7, 142.6, 139.1, 137.3, 132.6, 129.2, 128.9, 128.8, 128.3, 128.0, 127.6, 126.4, 117.6, 70.7, 49.4, 37.9, 27.5,; IR (KBr, cm^{-1}): 3048, 2936, 1734, 1609, 1525, 1350, 1255, 1214, 1188, 1098, 1074, 1018; HR-ESIMS (*m/z*): Calcd for $C_{22}H_{19}N_2O_2$ $[M+H]^+$ is 343.1441 found 343.1439; Calcd for $C_{22}H_{18}N_2O_2Na$ $[M+Na]^+$ is 365.1260 found 365.1265; Calcd for $C_{44}H_{36}N_4O_4Na$ $[2M+Na]^+$ is 707.2629 found 707.2627.

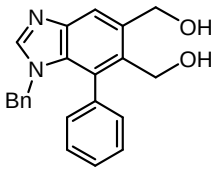
3-benzyl-4-phenyl-3,7-dihydro-5*H*-furo[3,4-*f*]benzimidazol-5-one:

To **69c** (5.5 g, 16 mmol) in CH_2Cl_2 (250 mL) was added MnO_2 (~85% act.) (15 g) and heated at 40 ℃ for 48 h. The solids were filtered through C elite and washed with CH_2Cl_2. The filtrate



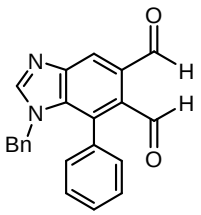
was reduced to give the product as a white solid (5.0 g, 91%). mp: 185-186 ℃; ^{1}H NMR: δ = 8.02 (s, 1H), 7.85 (s, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.21 (d, *J* = 7.5 Hz, 1H), 7.18-7.13 (m, 4H), 6.54 (d, *J* = 7.5 Hz, 2H), 5.37 (s, 2H), 4.93 (s, 2H); ^{13}C NMR: δ = 169.8, 148.9, 140.5, 135.7, 132.3, 129.8, 128.8, 128.6, 128.1, 127.9, 127.6, 126.2, 118.4, 112.6, 68.1, 50.3; IR (cm^{-1}): 3005, 2923, 1752, 1497, 1363, 1251, 1130, 1018; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{22}H_{16}N_2O_2Na$ is 363.1104 found 363.1114; Calcd. for $[2M+Na]^+$ $C_{44}H_{32}N_4O_4Na$ is 703.2316 found 703.2327.

[3-Benzyl-4-phenyl-1*H*-benzimidazole-5,6-diyl]dimethanol (134):



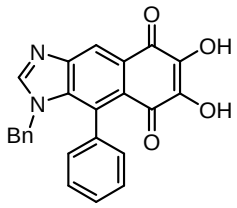
The benzimidazole prepared above (5.0 g, 15 mmol) was dissolved in CH_2Cl_2 (100 mL) and cooled to -78 ℃ under N_2. DIBAL-H (1M in hexanes) (41 mL, 41 mmol) was added dropwise, then allowed to come to rt and stir overnight. The reaction mixture was cooled to 0 ℃ and water was added slowly followed by MeOH. The solids were filtered through Celite and washed with CH_2Cl_2. Water was added to the filtrate and the organic layer separated. The aqueous layer was extracted with CH_2Cl_2 (2x). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. The resulting solids were washed with Et_2O to give **134** as a white solid (3.2 g, 63%). mp: 191-193 ℃; ^{1}H NMR: δ = 7.81 (s, 1H), 7.79 (s, 1H), 7.35 (t, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 7.4 Hz, 2H), 7.18-7.12 (m, 3H), 7.08 (d, *J* = 6.9 Hz, 2H), 6.50 (d, *J* = 6.9 Hz, 2H), 4.91 (s, 2H), 4.74 (s, 2H), 4.49 (s, 2H); ^{13}C NMR: δ = 146.0, 143.4, 136.5, 135.9, 135.1, 134.1, 131.5, 130.4, 128.6, 128.1, 128.0, 127.7, 127.7, 126.0, 121.2, 65.4, 59.6, 49.6; IR (cm^{-1}): 3056, 2856, 1504, 1441, 1311, 1175, 1018, 1000; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{22}H_{21}N_2O_2$ is 345.1598 found 345.1604; Calcd. for $[M+Na]^+$ $C_{28}H_{24}N_2O_6Na$ is 507.1527 found 507.1533.

3-Benzyl-4-phenyl-1*H*-benzimidazole-5,6-dicarbaldehyde (135):



134 (3.0 g, 8.7 mmol) was dissolved in CH_2Cl_2 (75 mL) and cooled to -78 ℃ under N_2. DBU (4.8 mL, 35 mmol) was added followed by **122** (4.7 g, 22 mmol) in CH_2Cl_2 (3 mL) and the reaction was stirred at -78 ℃ for 30 min. Sat. $NaHCO_3$ was added and the solution was allowed to come to rt. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (2x). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. The resulting residue was purified by column chromatography (the silica gel was neutralized with Et_3N prior to purification) (6:4 EtOAc/Hexane) to give **135** as a white solid (2.0 g, 68%). mp: 167-169 ℃; ^{1}H NMR: δ = 10.49 (s, 1H), 9.88 (s, 1H), 8.43 (s,1H), 8.06 (s, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 7.5 Hz, 1H), 7.21-7.14 (m, 4H), 6.51 (d, *J* = 7.5 Hz, 2H), 4.86 (s, 2H); ^{13}C NMR: δ = 192.8, 192.4, 149.7, 146.8, 135.4, 134.0, 132.9, 132.4, 132.0, 131.4, 130.5, 129.2, 128.9, 128.4, 128.2, 125.9, 121.8, 50.2; IR (cm^{-1}): 3070, 2871, 1756, 1678, 1595, 1488, 1455, 1303, 1213; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{22}H_{16}N_2O_2Na$ is 363.1104 found 363.1108.

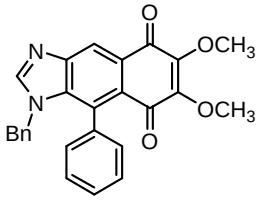
3-Benzyl-6,7-dihydroxy-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-5,8-dione (136):



To **135** (0.15 g, 0.44 mmol) in THF was added **123** (0.037 g, 0.66 mmol) followed by the simultaneous addition of KCN (0.029 g, 0.44 mmol) in H_2O (1 mL) and Et_3N (0.060 mL, 0.44 mmol). The reaction was stirred at rt for 15 min., then quenched with H_2O (5 mL). 1M HCl was added until pH = 5 and the aqueous layer was extracted with EtOAc (2x). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. An orange-red residue was recovered and recrystallized from MeOH to give **136** as an orange-red solid (0.074 g, 43%). mp: 280 ℃ (decomp.); ^{1}H NMR (DMSO-d_6): δ = 9.71 (brs, 2H), 8.46 (s, 1H), 8.30 (s, 1H), 7.32-7.29 (m,

1H), 7.19-7.16 (m, 5H), 7.01-7.00 (m, 2H), 6.45 (d, *J* = 4.6 Hz, 2H), 4.75 (s, 2H); ^{13}C NMR (DMSO-d_6): δ = 181.6, 181.1, 151.2, 146.6, 141.8, 139.6, 137.6, 136.5, 135.5, 129.3, 129.1, 128.8, 128.2, 127.8, 127.8, 127.1, 125.9, 122.9, 118.7, 49.3; IR (cm^{-1}): 2831, 1662, 1615, 1595, 1569, 1343, 1315, 1206, 1189, 905; HR-APCIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{24}H_{17}N_2O_4$ is 397.1183 found 397.1196.

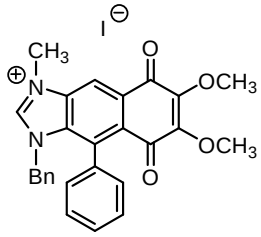
3-Benzyl-6,7-dimethoxy-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-5,8-dione (137):



136 (0.084 g, 0.21 mmol) was suspended in THF (5 mL) and MeOH (2.0 mL) was added followed by TMS-CHN_2 (2.0M in Et_2O) (0.37 mL, 0.74 mmol) and stirred at rt for 4 hr. The reaction was diluted with EtOAc and washed with sat. $NaHCO_3$ (2x), dried (anhyd. Na_2SO_4), and concentrated. The resulting residue was purified by column chromatography (1:1 EtOAc/Hexane) to give**137** as a yellow solid (0.045 g, 54%). mp: 189-191 ℃; ^{1}H NMR: δ = 8.65 (s, 1H), 7.92 (s, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.23-7.18 (m, 3H), 7.04 (d, *J* = 7.5 Hz, 2H), 6.57 (d, *J* = 8.0 Hz, 2H), 4.65 (s, 2H), 4.07 (s, 3H), 3.94 (s, 3H); ^{13}C NMR: δ = 182.1, 181.9, 149.5, 148.7, 147.0, 146.4, 136.1, 135.9, 135.8, 129.2, 128.8, 128.5, 128.3, 128.1, 127.9, 127.8, 126.1, 124.0, 120.2, 61.4, 61.3, 50.0; IR (cm^{-1}): 3009, 2940, 1649, 1596, 1441, 1348, 1291, 1027; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{26}H_{21}N_2O_4$ is 425.1496 found 425.1490; Calcd. for $[M+Na]^+$ $C_{26}H_{20}N_2O_4Na$ is 447.1315 found 447.1325.

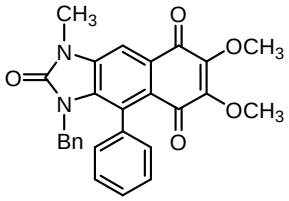
3-Benzyl-6,7-dimethoxy-1-methyl-4-phenyl-1*H*-naphtho[2,3-*d*]imidazolium-5,8-dione iodide (138):

To **137** (0.045 g, 0.11 mmol) in CH_3CN (8 mL) was added MeI (0.20 mL) and heated at 70 ℃ for 15 h. The reaction mixture was concentrated and the resulting residue was triturated with



Et_2O to give **138** as a red-orange solid (0.052 g, 86%). mp: 184-188 ℃; ^{1}H NMR: δ = 10.33 (s, 1H), 8.49 (s, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.29-7.27 (m, 3H), 7.18 (d, *J* = 7.5 Hz, 2H), 6.96 (d, *J* = 7.5 Hz, 2H), 4.93 (s, 2H), 4.26 (s,3H), 4.10 (s, 3H), 3.98 (s, 3H); ^{13}C NMR: δ = 180.2, 180.0, 149.3, 147.7, 146.4, 134.9, 132.9, 132.9, 132.3, 132.2, 131.1, 129.4, 129.3, 128.9, 128.6, 128.0, 127.0, 111.8, 61.6 (2C), 53.0, 34.9; IR (cm^{-1}): 3031, 2945, 1718, 1660, 1609, 1452, 1314, 1209, 1052, 916; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{27}H_{23}N_2O_4$ is 439.1652 found 439.1634.

3-Benzyl-6,7-dimethoxy-1-methyl-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (139a):

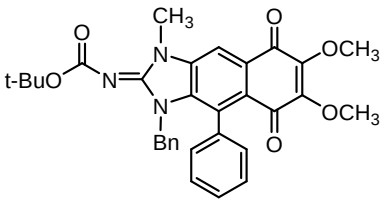


Condition A: **138** (0.052 g, 0.092 mmol) was suspended in THF (3 mL) and cooled to 0 ℃. 5% NaOCl (0.55 mL) was added dropwise and stirred at 0 ℃ for 10 min. The reaction mixture was diluted with water (5 mL) and extracted with EtOAc (2x). The combined organic extracts were washed with sat. NaCl, dried (anhyd. Na_2SO_4) and concentrated. The resulting yellow residue was purified by column chromatography (1:1 EtOAc/Hexane) to give **139a** as a yellow solid (0.011 g, 26%).

Condition B: To **138** (0.031 g, 0.055 mmol) in THF (1 mL) was added 1M K_2CO_3 (0.27 mL) and NCS (0.008 g, 0.060 mmol) and stirred at rt for 30 min. The reaction was diluted with water (4 mL) and extracted with EtOAc (2 x 5 mL). The combined organic extracts were washed with brine, dried and concentrated. The resulting residue was purified by column chromatography (1:1 EtOAc/Hexane) to give **139a** as a yellow solid (0.015 g, 60%). mp: 160-162 ℃; ^{1}H NMR: δ = 7.84 (s, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.17 (t, *J* = 7.5 Hz, 2H), 7.13-7.09 (m, 3H), 6.89 (d, *J* =

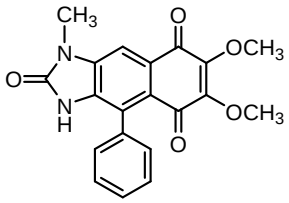
6.9 Hz, 2H), 6.53 (d, *J* = 6.3 Hz, 2H), 4.52 (s, 2H), 4.03 (s, 3H), 3.91 (s, 3H), 3.60 (s, 3H); ^{13}C NMR: δ = 181.9, 181.4, 155.4, 148.1, 145.3, 136.5, 135.2, 134.0, 131.4, 129.0, 128.2, 128.1, 127.9, 127.5, 127.1, 125.6, 125.5, 123.7, 105.5, 61.4, 61.3, 45.7, 27.9; IR (cm^{-1}): 2927, 1709, 1651, 1620, 1601, 1452, 1344, 1209, 1059; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{27}H_{22}N_2O_5Na$ is 477.1421 found 447.1438; Calcd. for $[2M+Na]^+$ $C_{54}H_{44}N_4O_{10}Na$ is 931.2950 found 931.2965.

3-Benzyl-2-*tert*-butylcarbonylimino-6,7-dimethoxy-1-methyl-4-phenyl-1*H*-naphtho [2,3-*d*]imidazole-2,5,8-trione (139b):



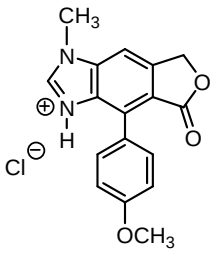
To **92** (0.019 g, 0.16 mmol) in CH_2Cl_2 (2 mL) was added *t*-BuOCl (0.020 mL, 0.17 mmol) and stirred at rt for 20 min. The reaction was cooled to 0 ℃ and DBU (0.033 mL, 0.22 mmol) was added followed by **138** in CH_2Cl_2 (3 mL). The reaction was stirred at rt for 30 min, then diluted with water. The aqueous layer was extracted with CH_2Cl_2 (2x) and the combined organic extracts were washed with brine, dried (anhyd. Na_2SO_4) and concentrated. The crude residue was purified by column chromatography (1:1 EtOAc/Hexane) to give **139b** as a yellow-orange residue (0.010 g, 30%). ^{1}H NMR: δ = 7.98 (s, 1H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.18 (t, *J* = 7.5 Hz, 2H), 7.12-7.07 (m, 3H), 6.84 (d, *J* = 6.9 Hz, 2H), 6.44 (d, *J* = 6.9 Hz, 2H), 4.70 (s, 2H), 4.03 (s, 3H), 3.90 (s,3H), 3.68 (s,3H), 1.46 (s,9H); ^{13}C NMR: δ = 181.6, 181.1, 158.3, 153.9, 148.3, 145.5, 135.8, 135.4, 134.6, 132.3, 128.9, 128.2, 128.2, 128.1, 128.0, 127.1, 127.1, 125.4, 124.5, 106.6, 78.9, 61.4, 61.3, 47.3, 31.8, 28.3; IR (cm^{-1}): 2929, 1655, 1581, 1443, 1308, 1207, 1147, 1047, 1026; HR-ESIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{32}H_{32}N_3O_6$ is 554.2286 found 554.2290; Calcd. for $[M+Na]^+$ $C_{32}H_{31}N_3O_6Na$ is 576.2105 found 576.2116.

6,7-Dimethoxy-1-methyl-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (140):



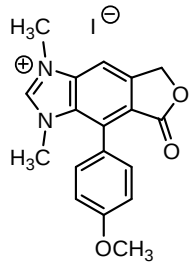
To **139a** (0.025 g, 0.055 mmol) was added TfOH (2 mL) and heated at 55 ℃ for 4 h. The reaction was quenched with water and neutralized with sat. $NaHCO_3$. The aqueous layer was extracted with EtOAc (2x) and the combined organic extracts were washed with brine, dried and concentrated. The resulting residue was purified by column chromatography (6:4 EtOAc/Hexane) to give **140** as a yellow waxy solid (0.010 g, 67%). ^{1}H NMR: δ = 8.11 (brs, 1H), 7.74 (s, 1H), 7.49-7.43 (m, 3H), 7.22-7.21 (m, 2H), 4.07 (s, 3H), 3.97 (s, 3H), 3.47 (s, 3H); ^{13}C NMR: δ = 181.9, 181.5, 154.2, 147.6, 146.0, 135.3, 134.1, 131.6, 129.1, 128.3, 128.1, 127.8, 124.3, 123.0, 105.5, 61.5, 61.4, 27.4; IR (cm^{-1}): 2919, 1708, 1648, 1623, 1443, 1332, 1245, 1194, 1056, 1026; HR-APCIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{20}H_{17}N_2O_5$ is 365.1132 found 365.1144.

4-(4-Methoxyphenyl)-1-methyl-5-oxo-5,7-dihydro-1*H*-furo[3,4-*f*][3,1]benzimidazol-3-ium chloride (142)



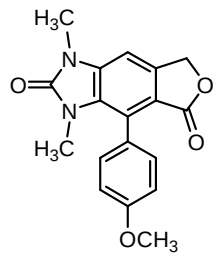
To **120b** (0.072 g, 0.24 mmol) in acetonitrile (10 mL) was added SEM-Cl and stirred at 60 ℃ for 1 h. The reaction was cooled to 0 ℃ and the precipitated solids were filtered and washed with Et_2O to give **142** as a white solid (0.051 g, 63%). mp: 255-258 ℃; ^{1}H NMR (CD_3OD): δ = 9.56 (d, *J* = 4.6 Hz, 1H), 8.06 (s, 1H), 7.51 (d, *J* = 8.6 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 2H), 5.52 (s, 2H), 4.19 (s, 3H), 3.89 (s, 3H); ^{13}C NMR (CD_3OD): δ = 169.3, 161.1, 145.7, 145.1, 136.4, 131.3, 130.5, 122.0, 119.9, 113.8, 105.2, 68.5, 54.6, 32.7; IR (cm^{-1}): 3155, 2942, 1728, 1623, 1506, 1392, 1243, 1142, 1110, 1012; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{18}H_{17}N_2O_3$ is 317.0897 found 317.0903.

4-(4-Methoxyphenyl)-1,3-dimethyl-5-oxo-5,7-dihydro-3*H*-furo[3,4-f]benzimidazol-1-ium iodide (144):



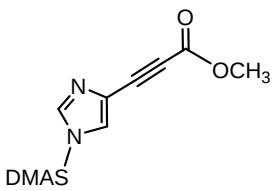
To **120b** (0.025 g, 0.085 mmol) in toluene (10 mL) was added MeI (0.030 mL, 0.42 mmol) and heated to 140 ℃ for 20 h. The solvent was reduced to give **144** (0.025 g, 68%) as an off-white solid. mp: 253-256 ℃; ^{1}H NMR (CD_3OD): δ = 8.13 (s, 1H), 7.37 (d, *J* = 8.6 Hz, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 5.51 (s, 2H), 4.18 (s, 3H), 3.88 (s, 3H), 3.54 (s, 3H); ^{13}C NMR (CD_3OD): δ = 168.9, 160.9, 145.2, 136.7, 131.1, 130.8, 130.5, 121.8, 113.3, 106.3, 68.4, 54.6, 36.2, 32.9; IR (cm^{-1}): 2960, 1772, 1612, 1577, 1378, 1241, 1065, 1018, 823; HR-ESIMS (*m/z*): Calcd. for $[M]^+$ $C_{18}H_{17}N_2O_3$ is 309.1234 found 309.1229.

4-(4-Methoxyphenyl)-1,3-dimethyl-3,7-dihydro-1*H*-furo[3,4-*f*]benzimidazole-2,5-dione (145):



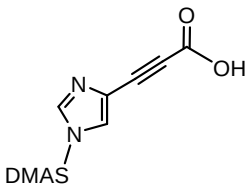
144 (0.010 g, 0.023 mmol) in THF was cooled to 0 ℃ and 5% NaOCl was added and stirred for 10 min. The reaction was diluted with water and extracted with EtOAc (2x). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated. The resulting solids were purified by PTLC (EtOAc) to give **145** as a white solid (0.006 g, 73%). mp: 239-240 ℃; ^{1}H NMR: δ = 7.26 (d, *J* = 8.6 Hz, 2H), 6.98 (d, *J* = 8.6 Hz, 2H), 6.98 (s, 1H), 5.25 (s, 2H), 3.87 (s, 3H), 3.49 (s, 3H), 2.92 (s, 3H); ^{13}C NMR: δ = 170.0, 159.9, 155.4, 141.5, 135.8, 131.3, 128.9, 124.1, 123.4, 116.8, 113.3, 99.3, 67.9, 55.3, 30.3, 27.7; IR (cm^{-1}): 2930, 2838, 1761, 1700, 1609, 1520, 1482, 1357, 1287, 1248, 1196, 1179, 1139, 1078; HR-APCIMS (*m/z*): Calcd. for $[M+H]^+$ $C_{18}H_{17}N_2O_4$ is 325.1183 found 325.1191.

Methyl 3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]prop-2-ynoate (154):



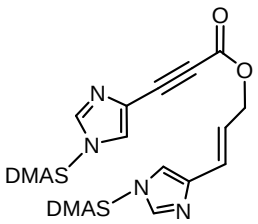
To **153b** (0.50 g, 2.18 mmol) in THF (9 mL) was added MeOH (1 mL), MnO_2 (3.0 g, 33 mmol), KCN (0.030 g, 0.44 mmol), and K_2CO_3 (0.36 g, 2.6 mmol) and heated at 45 ℃ for 5 h. The solids were filtered through Celite and washed with EtOAc. The filtrate was washed with H_2O, then brine, dried (anhyd. Na_2SO_4) and concentrated to give **154** as a tan solid (0.40 g, 71%). mp: 124-127 ℃; ^{1}H NMR: δ = 7.87 (d, *J* = 1.4 Hz, 1H), 7.61 (d, *J* = 1.4 Hz), 3.83 (s, 3H), 2.90 (s, 6 H); ^{13}C NMR: δ = 154.0, 137.1, 124.7, 123.3, 82.1, 78.6, 53.0, 38.3; IR (cm^{-1}): 3138, 3123, 2222, 1700, 1386, 1333, 1291, 1172, 1080, 1013; HR-APCIMS (*m/z*): Calcd. for $[M+H]^+$ $C_9H_{12}N_3O_4S$ is 258.0543 found 258.0543.

3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]prop-2-ynoic acid (155):



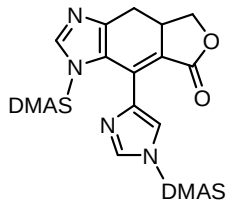
To **154** (6.5 g, 0.025 mol) in THF (150 mL) was added LiOH•H_2O (5.3 g, 0.13 mol) in water (130 mL) and stirred at rt for 18 h. The reaction was diluted with water and acidified with 6M HCl until pH = 4. The precipitated solids were filtered and washed with cold water. The filtrate was extracted with EtOAc (2x). The combined organic extracts were concentrated and the resulting solids were washed with cold water. The two crops were combined to give **155** as a white solid (5.7 g, 92%). mp: 150 ℃ (decomp) (lit. 138-140 ℃);[33] ^{1}H NMR (DMSO-d_6): δ = 8.34 (d, *J* = 1.2 Hz, 1H), 8.29 (d, *J* = 1.2 Hz, 1H), 2.82 (s, 6H); ^{13}C NMR (DMSO-d_6): δ = 154.6, 138.7, 126.7, 122.3, 83.4, 78.6, 39.7.

(2*E*)-3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]prop-2-en-1-yl 3-[1-(*N*,*N*-dimethyl sulfamoyl)-1*H*-imidazol-4-yl]prop-2-ynoate (150):



155 (2.61 g, 10.7 mmol), **113b** (1.77 g, 7.66 mmol), and DMAP (0.23 g, 1.91 mmol) in CH_2Cl_2 (50 mL) was cooled to 0 ℃ and EDCI (1.80 g, 9.19 mmol) was added and stirred at 0 ℃ for 2 h. The organic layer was washed with H_2O (2x), followed by brine, dried (anhyd. Na_2SO_4), and concentrated. The resulting residue was purified by column chromatography (8:2 EtOAc/Hexane) to give **150** as a white solid (2.52 g, 72%). mp: 119-121 ℃; ^{1}H NMR: δ = 7.86 (d, *J* = 1.1 Hz, 1H), 7.84 (d, *J* = 1.1 Hz, 1H), 7.61 (d, *J* = 1.1 Hz, 1H), 7.16 (d, *J* = 1.1 Hz, 1H), 6.59-4.87 (m, 2H), 4.87 (d, *J* = 4.6 Hz, 2H), 2.89 (s, 6H), 2.86 (s, 6H); ^{13}C NMR: δ = 153.3, 140.5, 137.2, 137.1, 124.7, 124.7, 123.8, 123.4, 115.1, 82.2, 78.8, 66.0, 38.3, 38.3; IR (cm^{-1}): 3141, 3100, 2221, 1684, 1392, 1292, 1172, 1067, 963; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{16}H_{20}N_6O_6S_2Na$ is 479.0778 found 479.0795; Calcd. for $[2M+Na]^+$ $C_{32}H_{40}N_{12}O_{12}S_4Na$ is 935.1664 found 935.1687.

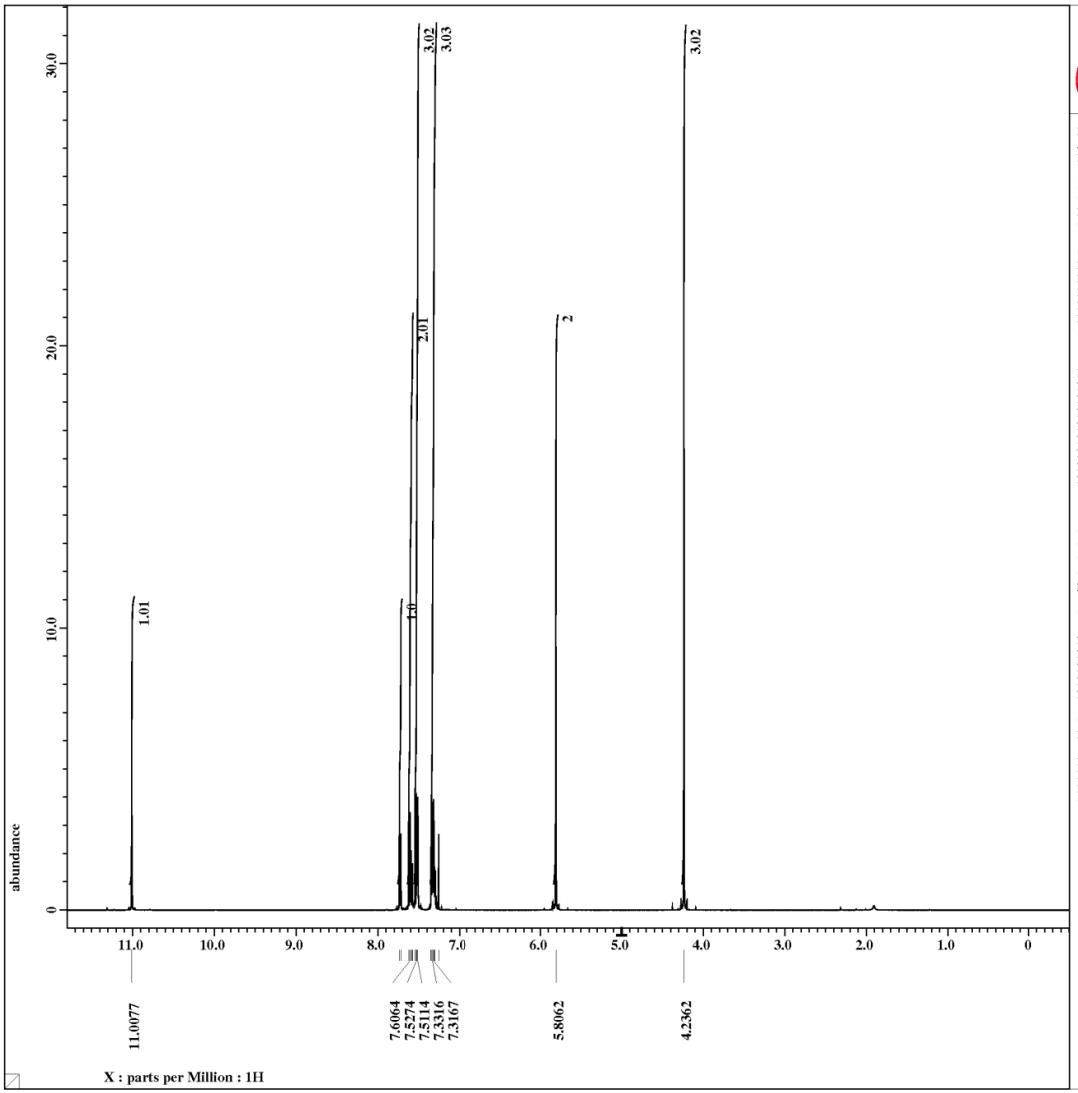
3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]-3,7,7a,8-tetrahydro-4-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]-5*H*-furo[3,4-*f*]benzimidazole-5-one (149):



150 (1.0 g, 2.2 mmol) was dissolved in dichloromethane (150 mL) and purged with N_2 for 10 minutes. The solution was then heated to 130 ℃ in a sealed tube for 20 h. The solvent was removed and the resulting solids washed with ethanol to afford **149** as a pale yellow solid (0.58 g, 58%). mp: 169-172 ℃; ^{1}H NMR: δ = 7.98 (s, 1H), 7.91 (d, *J* = 1.7 Hz, 1H), 7.78 (d, *J* = 1.1 Hz, 1H), 4.73 (t, *J* = 9.2 Hz, 1H), 4.06 (t, *J* = 8.6 Hz, 1H), 3.63-3.58 (m, 1H), 3.07 (dd, *J* = 8.6 Hz, *J* = 16.6 Hz, 1H), 2.89 (s, 6H), 2.76-2.73 (m, 1H), 2.72 (s, 6H); ^{13}C NMR: δ = 167.5, 146.1, 142.1, 135.8, 133.7, 131.3, 128.5, 121.9, 120.4, 70.7, 38.3, 38.1, 37.5, 27.9; IR (cm^{-1}): 1732, 1386,

1172, 1081, 963, 723; HR-ESIMS (*m/z*): Calcd. for $[M+Na]^+$ $C_{16}H_{20}N_6O_6S_2Na$ is 479.0778 found 479.0764.

APPENDIX 1
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-3-methyl-1*H*-benzimidazol-3-ium iodide (**81a**)



JEOL

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Author            = delta
Experiment        = single_pulse.ex2
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Revision_time     = 18-OCT-2011 11:27:25
Current_time      = 18-OCT-2011 11:28:06

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Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
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Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 21.2[dC]
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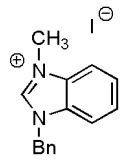

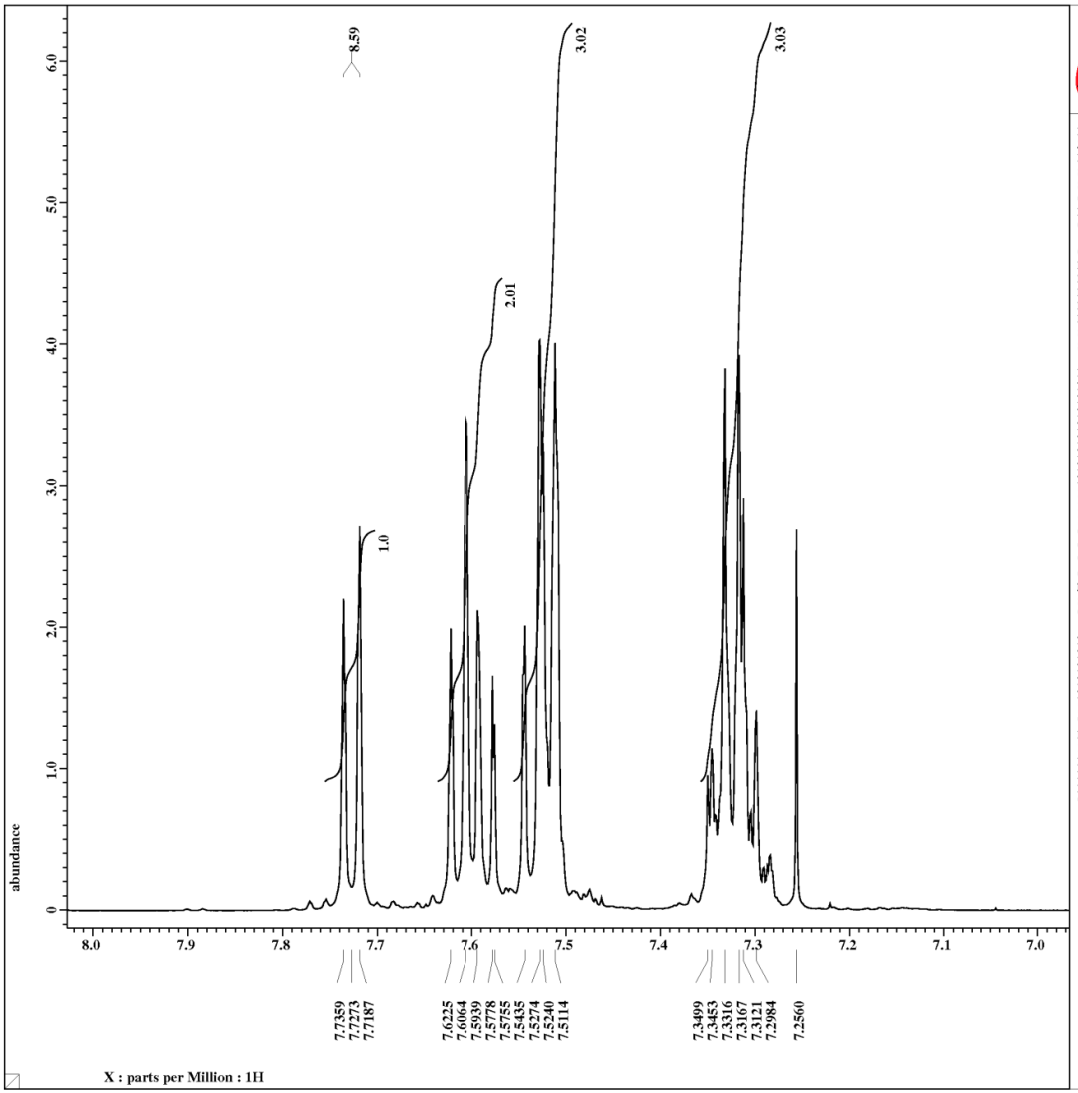

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Experiment = single_pulse.ex2
Sample_id = S#419087
Solvent = CHLOROFORM-D
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Revision_time = 18-OCT-2011 11:28:26
Current_time = 18-OCT-2011 11:29:04

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16

X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
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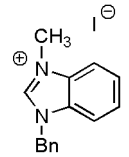
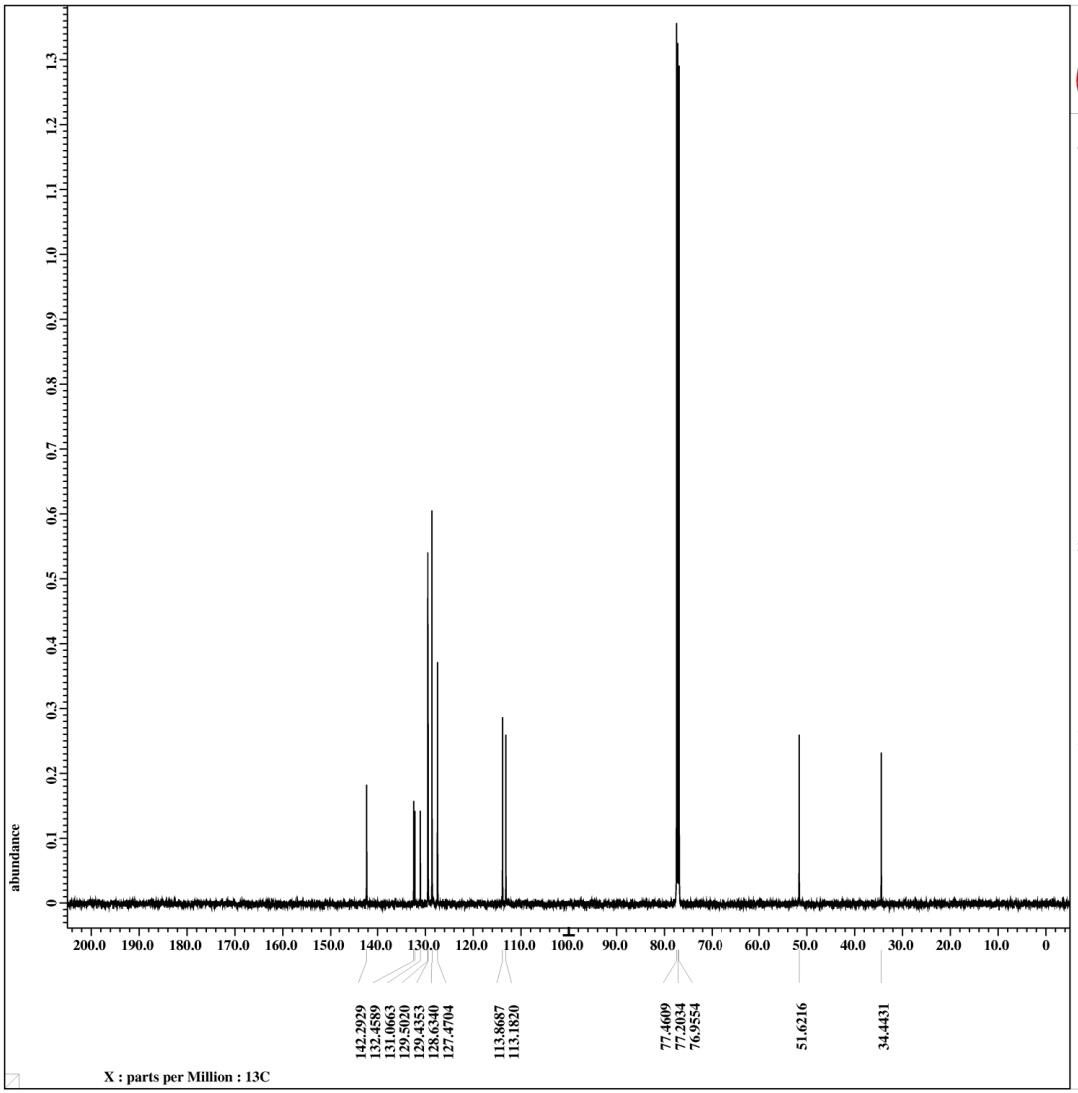
Temp_get = 21.2[dC]





JEOL

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Revision_time     = 11-JAN-2011 12:29:11
Current_time      = 11-JAN-2011 12:30:08

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Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
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X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 300
Total_scans       = 300

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 21.7[dC]
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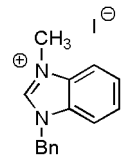

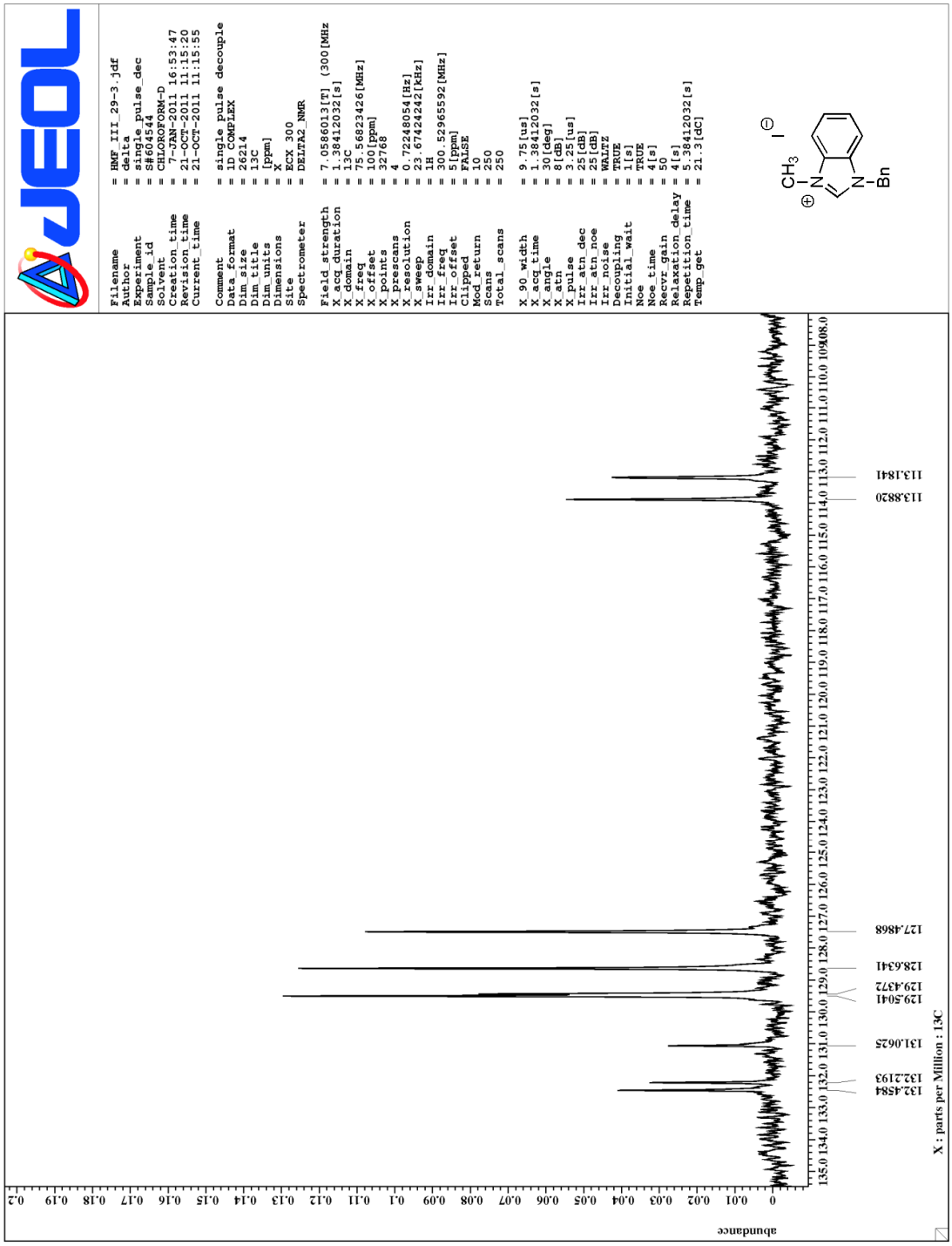
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Creation_time = 7-JAN-2011 16:53:47
Revision_time = 21-OCT-2011 11:15:20
Current_time = 21-OCT-2011 11:15:55
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Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
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X_domain = 13C
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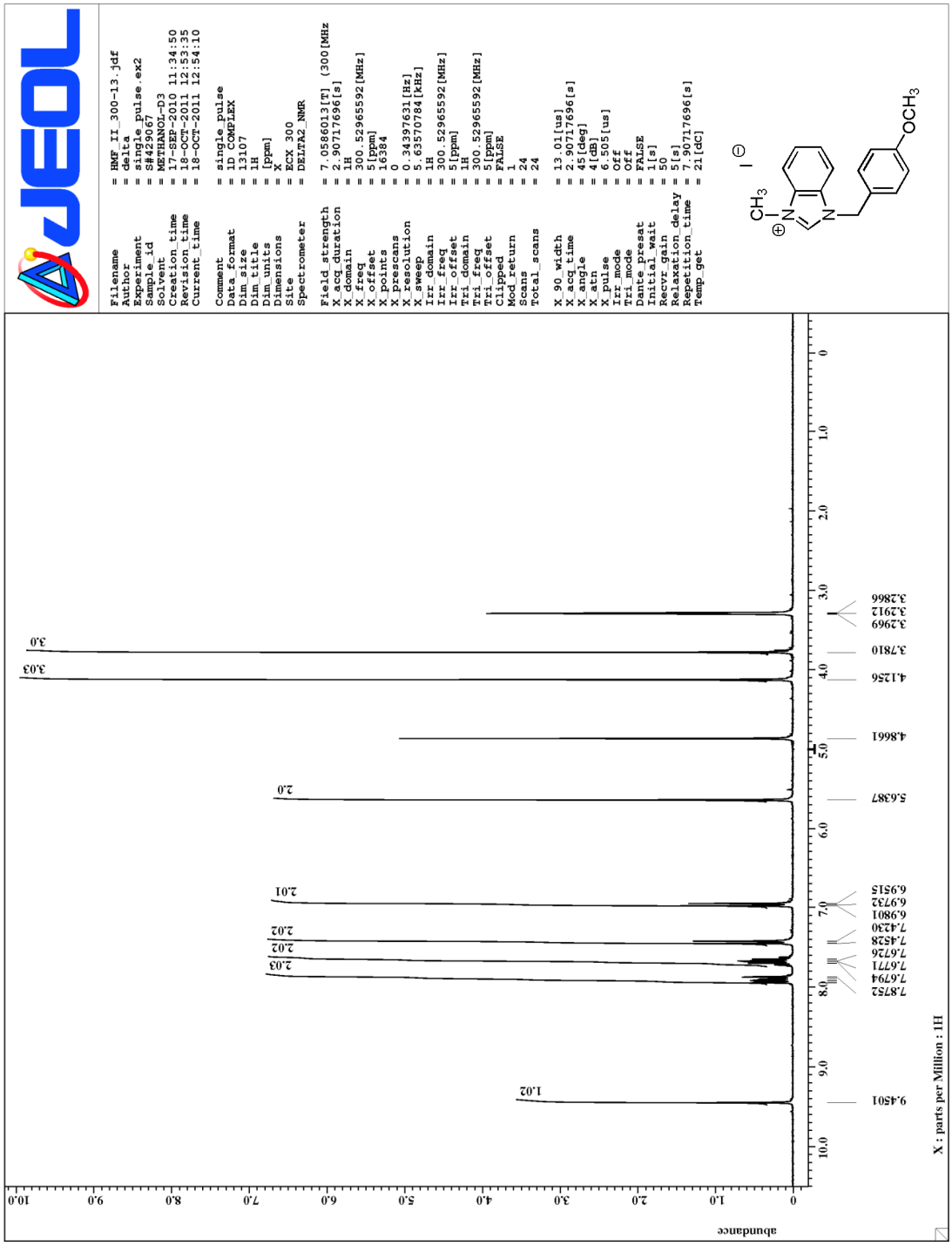
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 250
Total_scans = 250
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 5.38412032[s]
Temp_get = 21.3[dC]
JEOL
abundance
X : parts per Million : 13C
113.1841
113.8820
127.4868
128.6341
129.4372
129.5041
131.0625
132.2193
132.4584

APPENDIX 2
^{1}H and ^{13}C NMR Spectra of
1-(4-methoxybenzyl)-3-methyl-1*H*-benzimidazol-3-ium iodide (**81b**)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Creation_time = 17-SEP-2010 11:34:50
Revision_time = 18-OCT-2011 12:53:35
Current_time = 18-OCT-2011 12:54:10
Comment = single_pulse
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Dim_units = [ppm]
Dimensions = X
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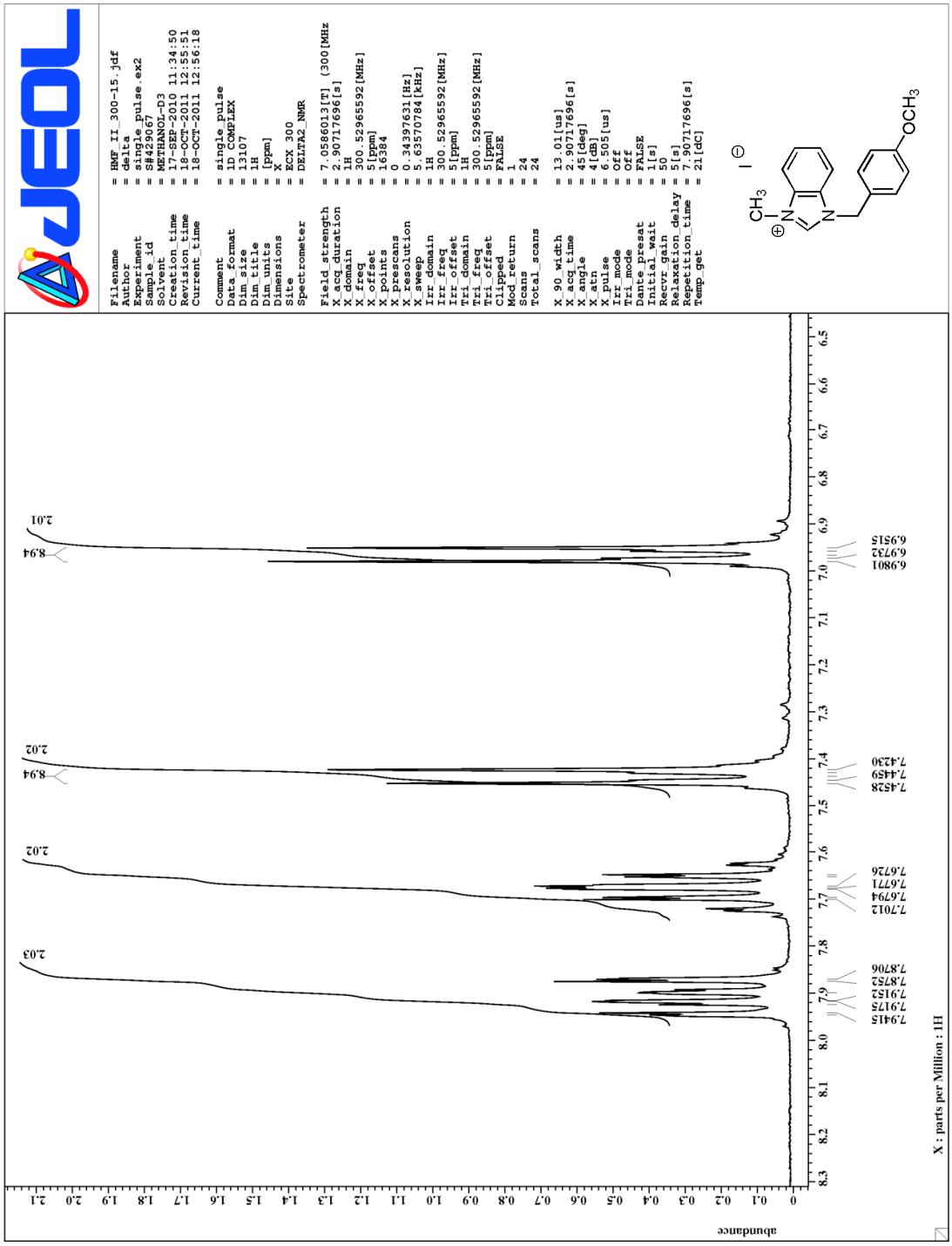
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21[dC]
I⊖
CH3
N
N
OCH3
abundance
X : parts per Million : 1H
9.4501
7.8752
7.8752
7.6794
7.6771
7.6726
7.4528
7.4230
6.9801
6.9732
6.9515
5.6387
4.8661
4.1256
3.7810
3.2969
3.2912
3.2866
1.02
2.03
2.02
2.02
2.01
2.0
3.03
3.0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Revision_time = 18-OCT-2011 12:55:51
Current_time = 18-OCT-2011 12:56:18
Comment = single_pulse
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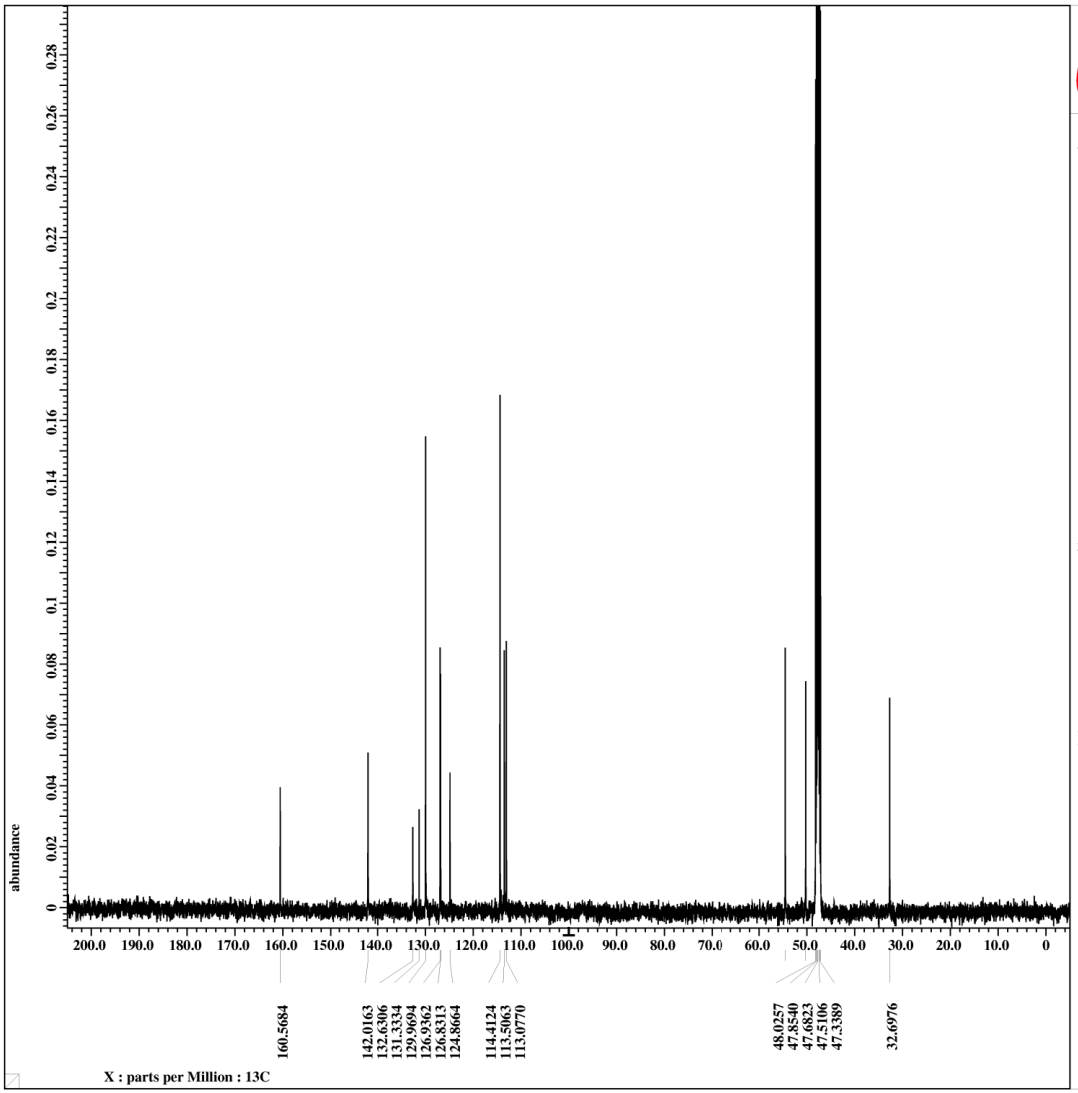
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21[dC]
CH3
OCH3
abundance
X : parts per Million : 1H
2.03
2.02
2.02
2.01
8.94
8.94
7.9415
7.9175
7.9152
7.8752
7.8706
7.7012
7.6794
7.6771
7.6726
7.4528
7.4459
7.4230
6.9801
6.9732
6.9515



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Solvent = METHANOL-D3
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Revision_time = 17-JAN-2011 16:17:18
Current_time = 17-JAN-2011 16:18:20

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Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 870.0
Total_scans = 870.0

X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
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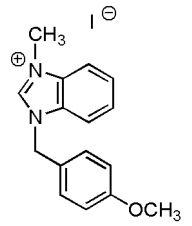
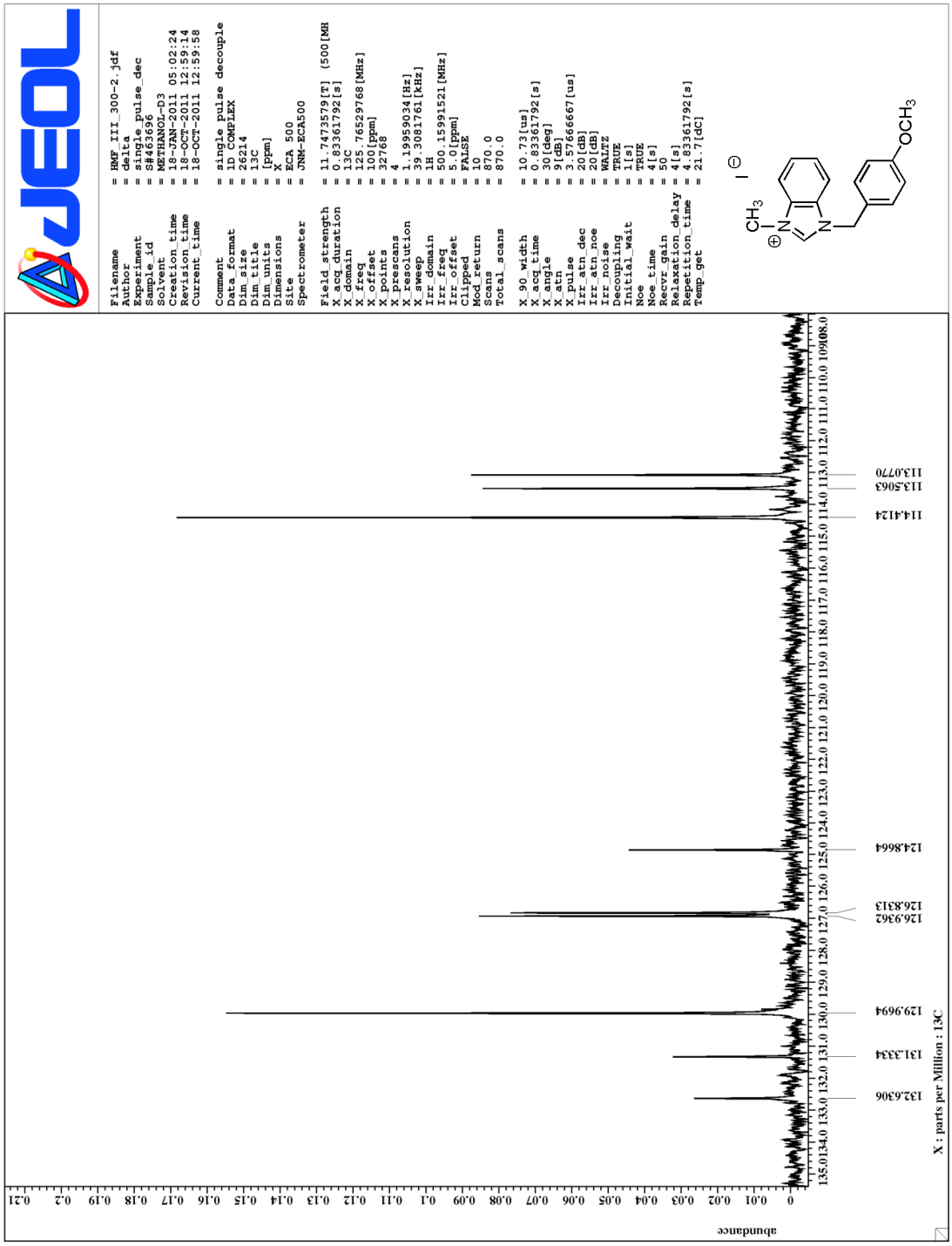
Temp_get = 21.7[dC]

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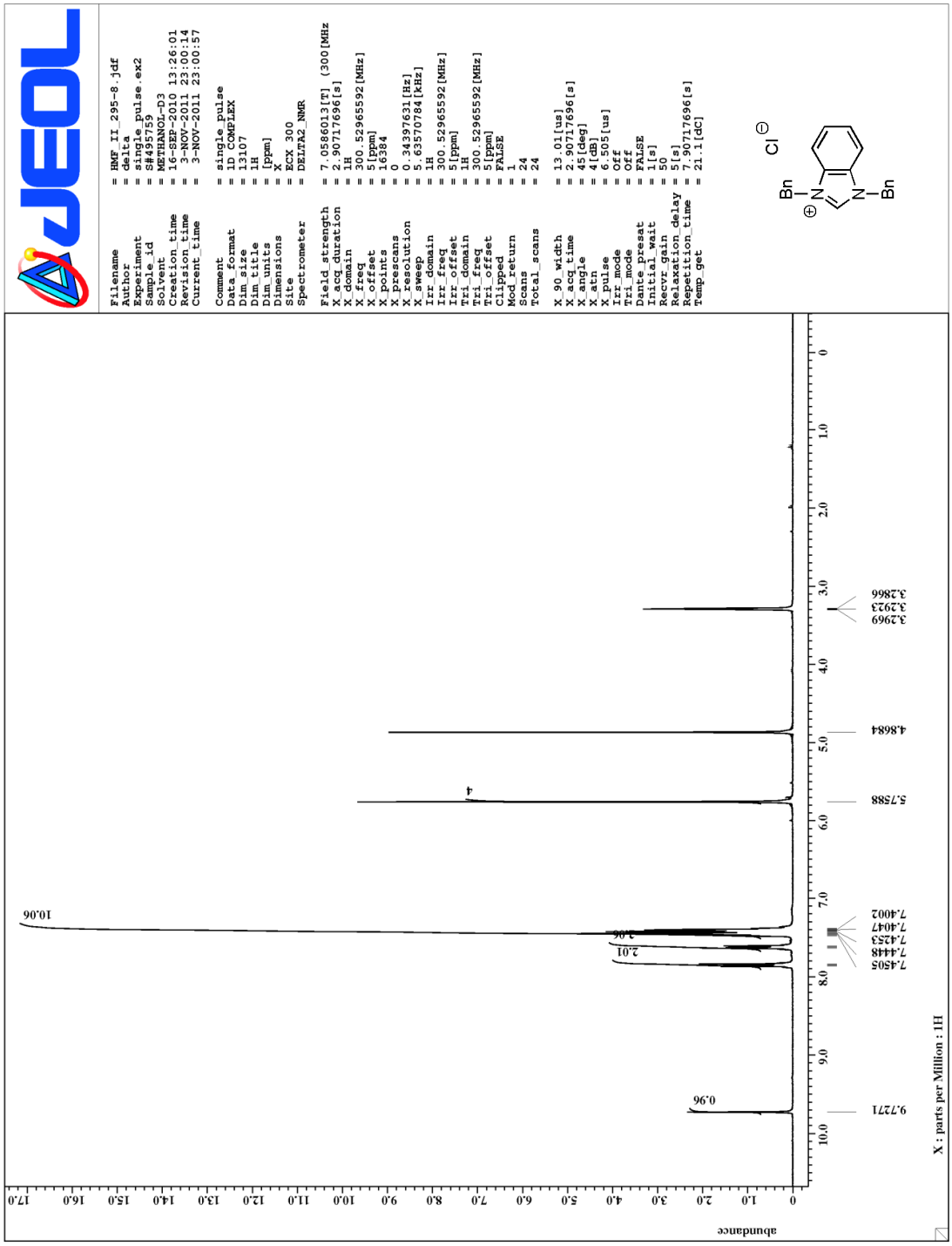
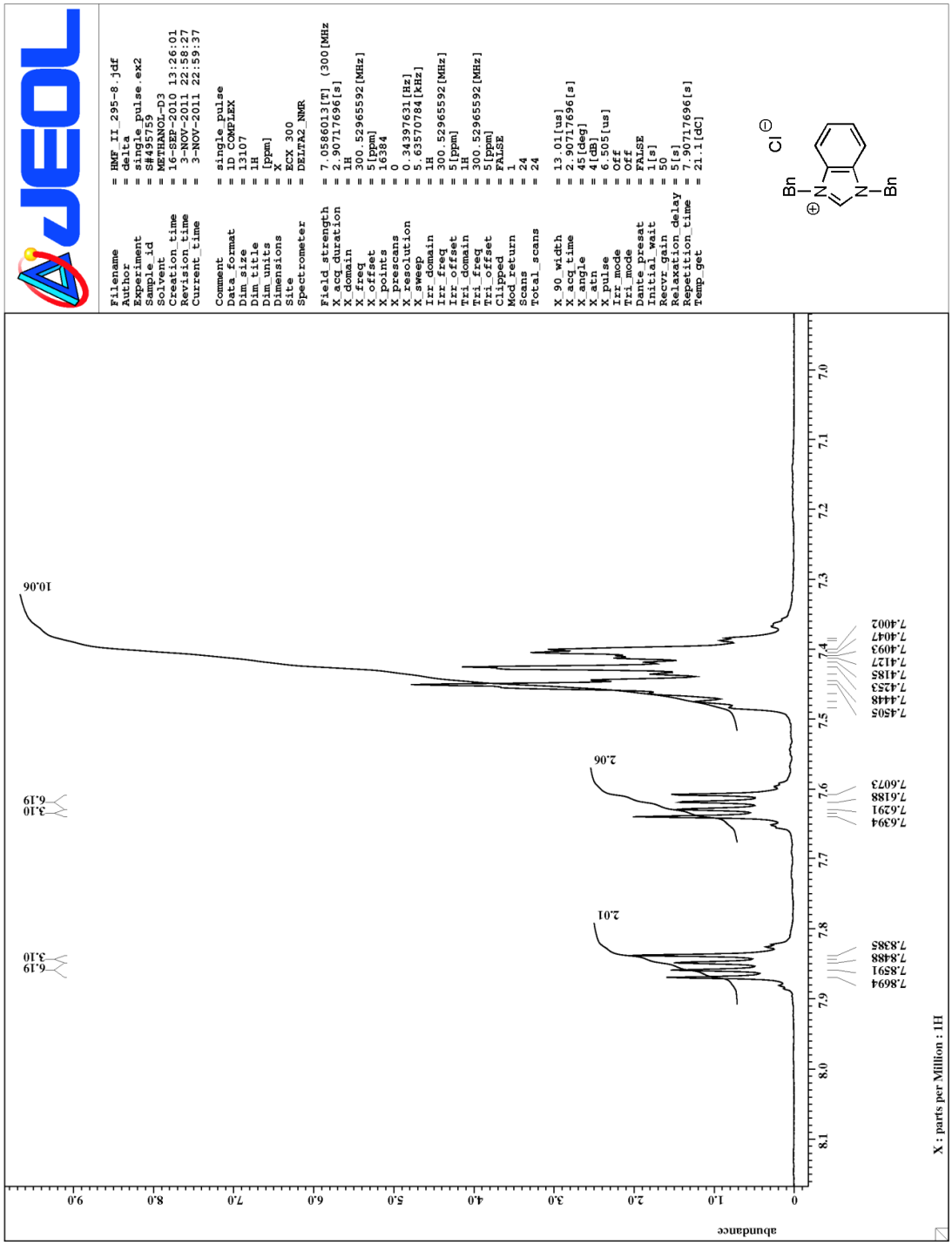
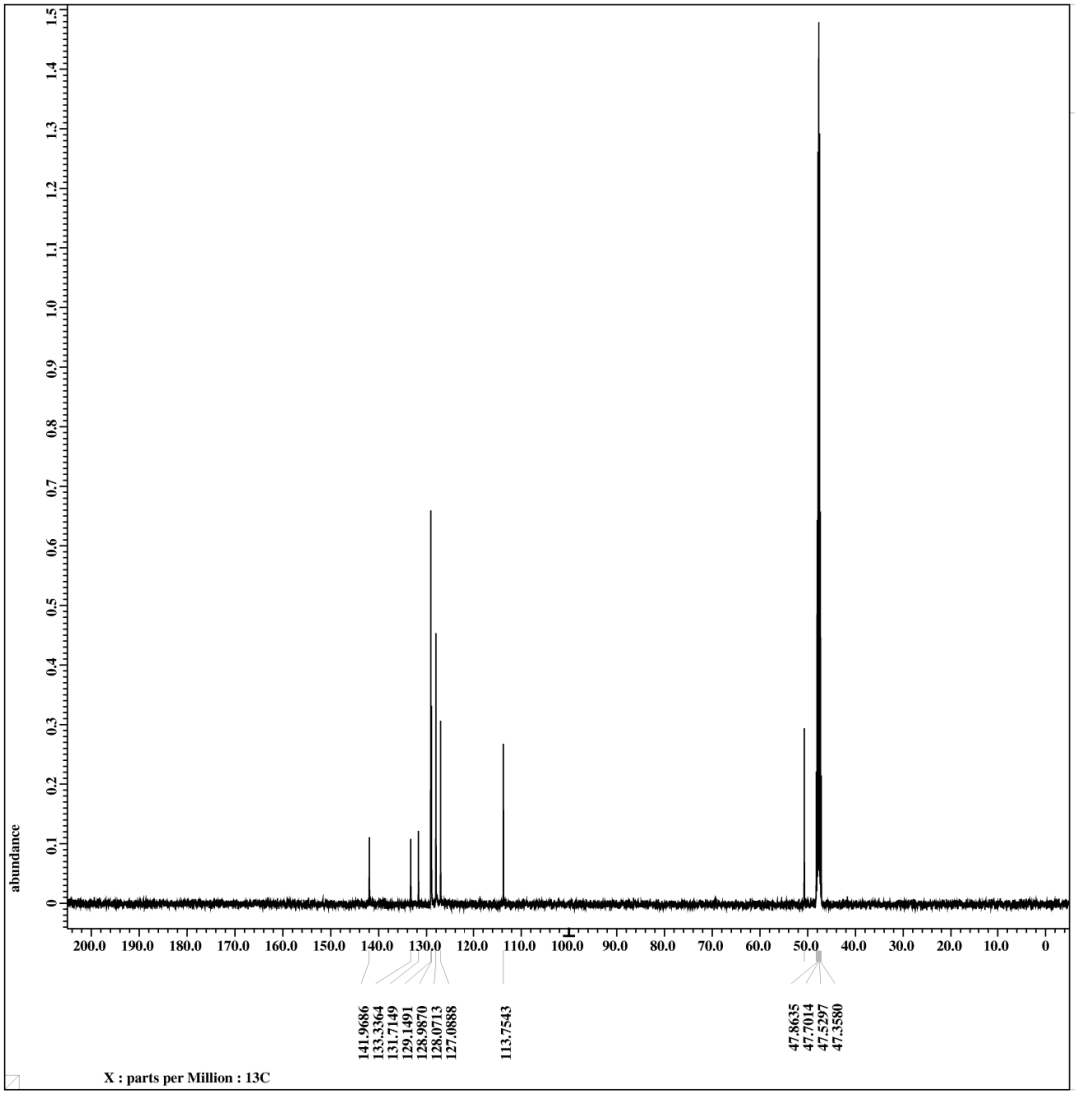
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 870.0
Total_scans = 870.0
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.7[dC]
CH3
OCH3
abundance
X : parts per Million : 13C
132.6306
131.3334
129.9694
126.9362
126.8313
124.8664
114.4124
113.5063
113.0770

APPENDIX 3
^{1}H and ^{13}C NMR Spectra of
1,3-Dibenzyl-1*H*-benzimidazol-3-ium chloride (**81c**)


Filename = HMF_II_295-8.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#495759
Solvent = METHANOL-D3
Creation_time = 16-SEP-2010 13:26:01
Revision_time = 3-NOV-2011 23:00:14
Current_time = 3-NOV-2011 23:00:57
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21.1[dC]
Cl
Bn
Bn
10.06
2.06
2.01
4
0.96
9.7271
7.4505
7.4448
7.4253
7.4047
7.4002
5.7588
4.8684
3.2969
3.2923
3.2866
abundance
X : parts per Million : 1H


JEOL
Filename = HMF_II_295-8.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#495759
Solvent = METHANOL-D3
Creation_time = 16-SEP-2010 13:26:01
Revision_time = 3-NOV-2011 22:58:27
Current_time = 3-NOV-2011 22:59:37
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21.1[dC]
Cl
Bn
Bn
abundance
X : parts per Million : 1H
10.06
2.06
2.01
6.19
3.10
6.19
3.10
7.8694
7.8591
7.8488
7.8385
7.6394
7.6291
7.6188
7.6073
7.4505
7.4448
7.4253
7.4185
7.4127
7.4093
7.4047
7.4002



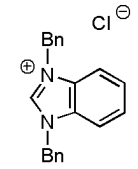
JEOL

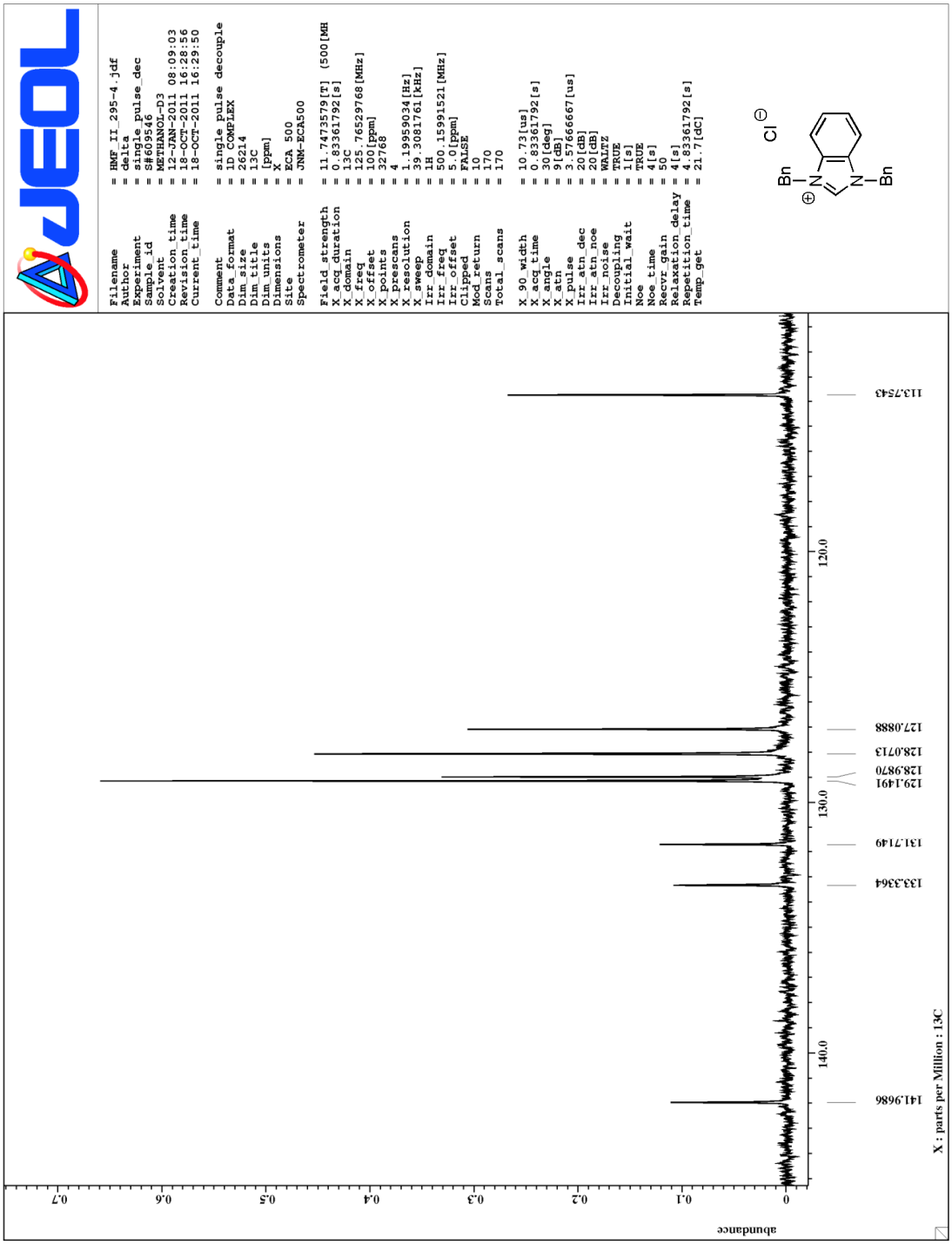
```
Filename          = HMF_II_295-2.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#609546
Solvent           = METHANOL-D3
Creation_time     = 12-JAN-2011 08:09:03
Revision_time     = 12-JAN-2011 10:18:48
Current_time      = 12-JAN-2011 10:19:30

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

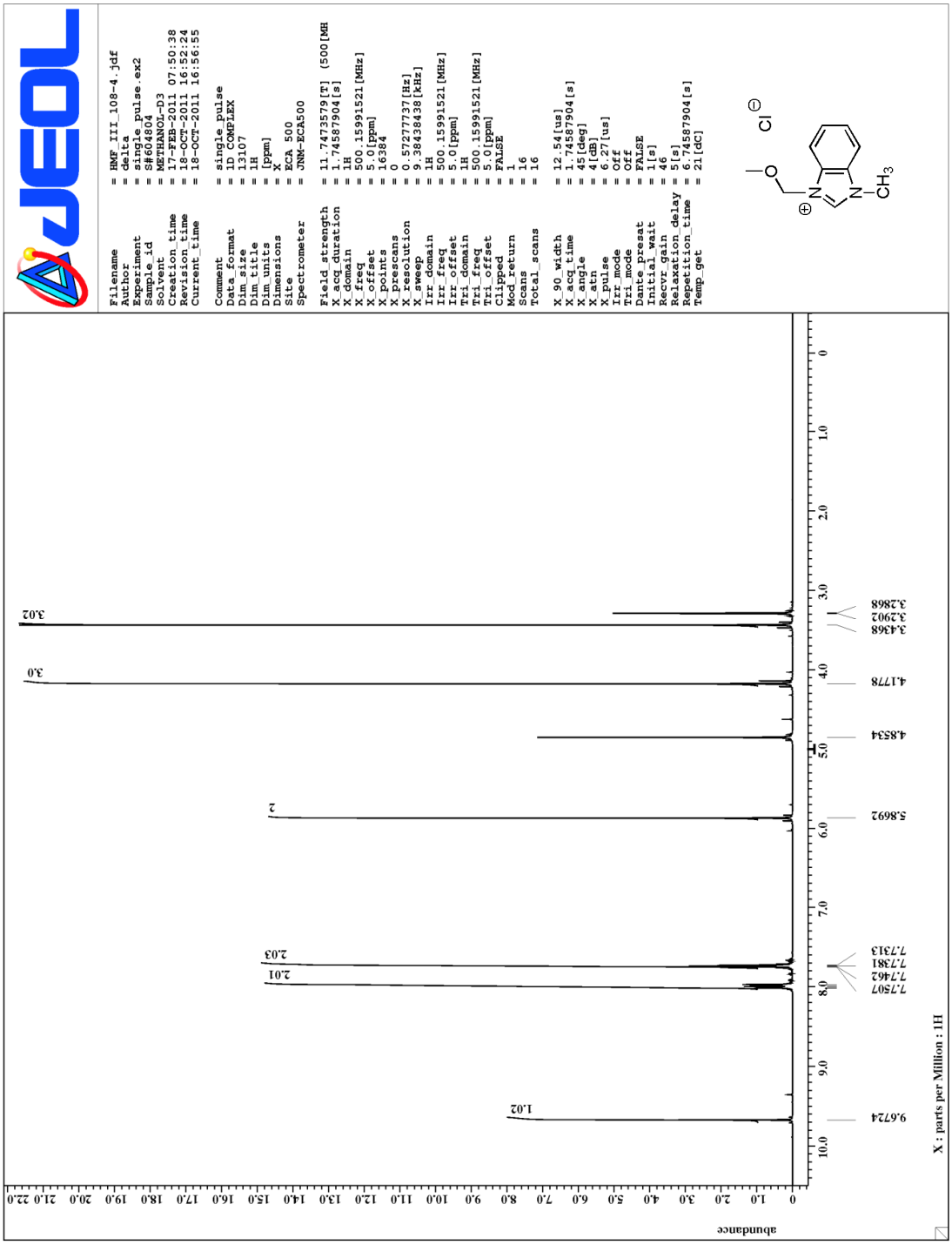
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 170
Total_scans       = 170

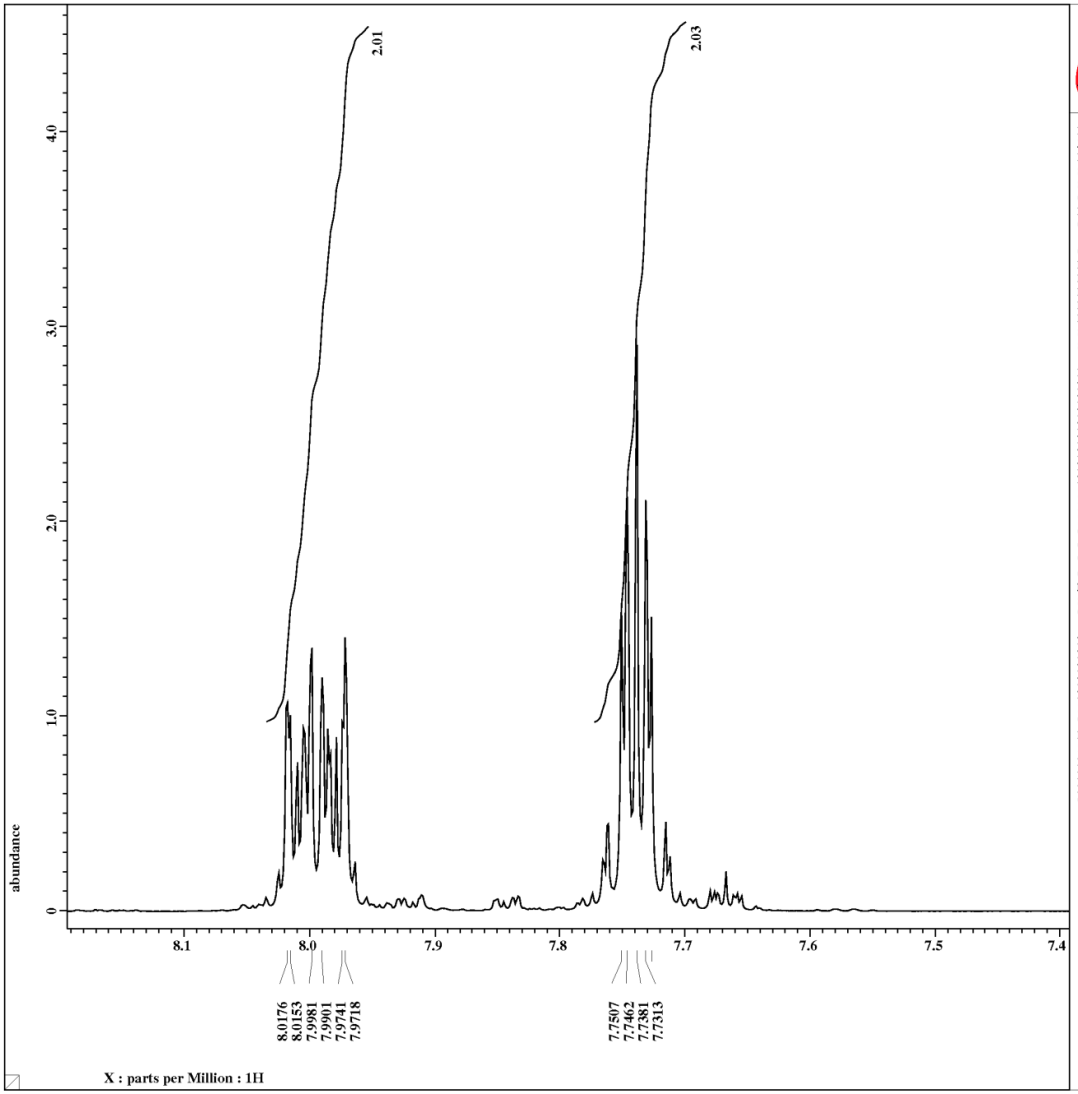
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 21.7[dC]
```




JEOL
Filename = RMF_II_295-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#609546
Solvent = METHANOL-D3
Creation_time = 12-JAN-2011 08:09:03
Revision_time = 18-OCT-2011 16:28:56
Current_time = 18-OCT-2011 16:29:50
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 170
Total_scans = 170
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.7[dC]
Cl
Bn
Bn
abundance
X : parts per Million : 13C
141.9686
133.3364
131.7149
129.1491
128.9870
128.0713
127.0888
113.7543

APPENDIX 4
^{1}H and ^{13}C NMR Spectra of
3-Methoxymethyl-1-methyl-1*H*-benzimidazol-3-ium chloride (**81d**)


JEOL
Filename = RMF III_108-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#604804
Solvent = METHANOL-D3
Creation_time = 17-FEB-2011 07:50:38
Revision_time = 18-OCT-2011 16:52:24
Current_time = 18-OCT-2011 16:56:55
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21[dC]
Cl
N
N
CH3
abundance
X : parts per Million : 1H
9.6724
7.7507
7.7462
7.7381
7.7313
5.8692
4.8534
4.1778
3.4368
3.2902
3.2868
1.02
2.03
2.01
2
3.0
3.02



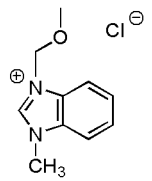
JEOL

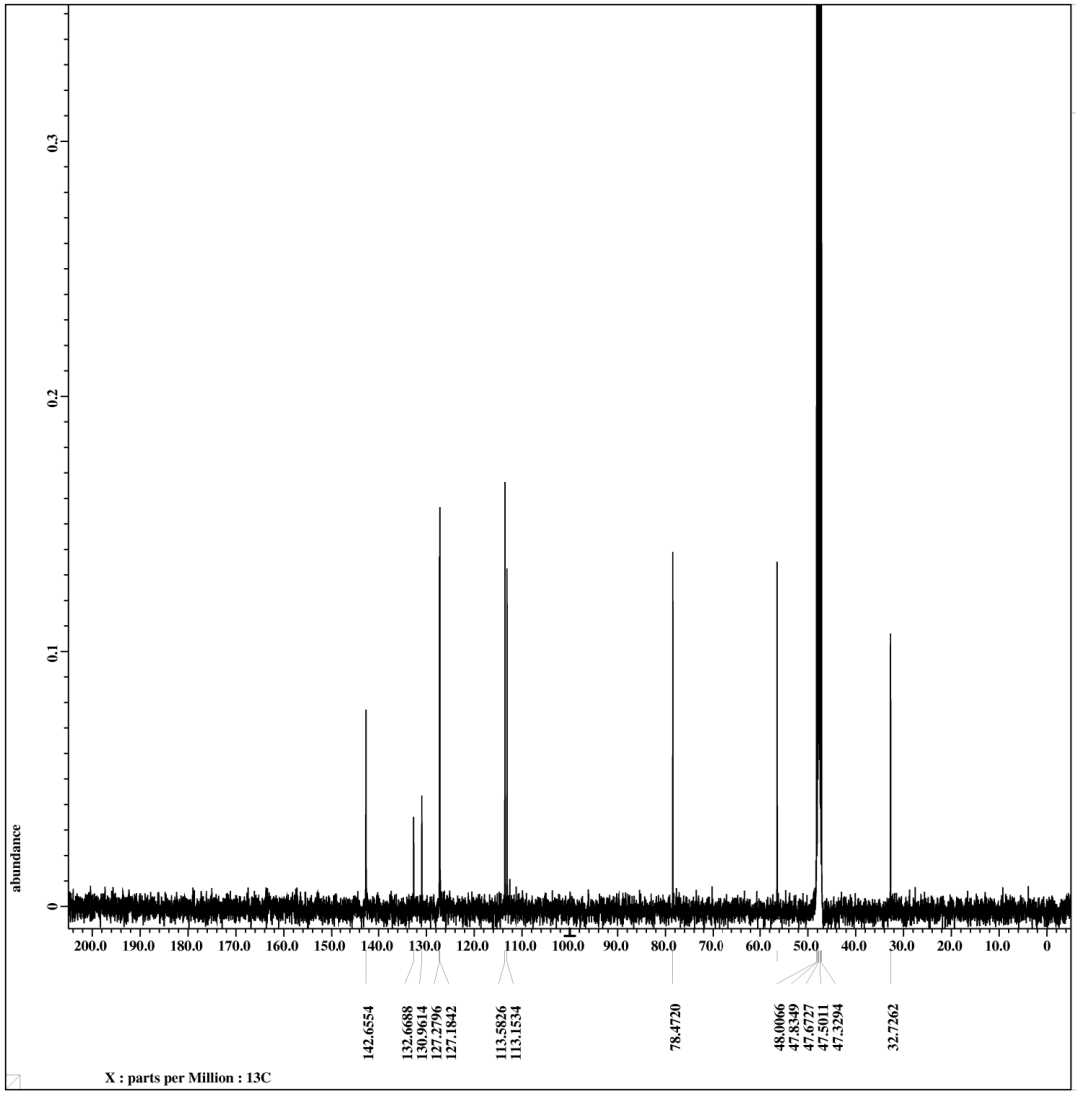
```
Filename          = HMF_III_108-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#604804
Solvent           = METHANOL-D3
Creation_time     = 17-FEB-2011 07:50:38
Revision_time     = 18-OCT-2011 16:52:24
Current_time      = 18-OCT-2011 16:53:29

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

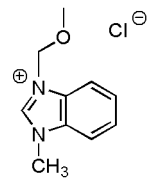
X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 21[dC]
```

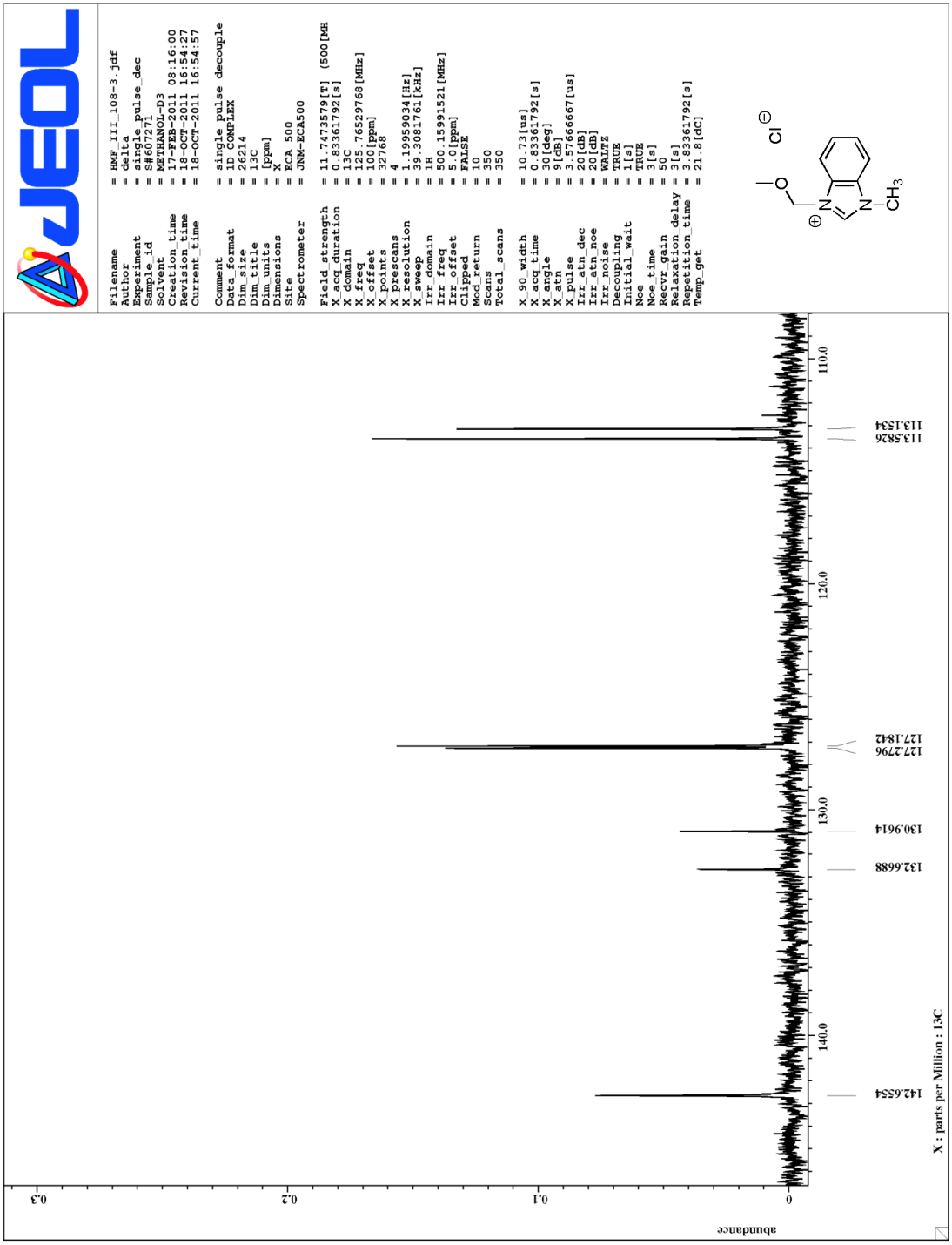




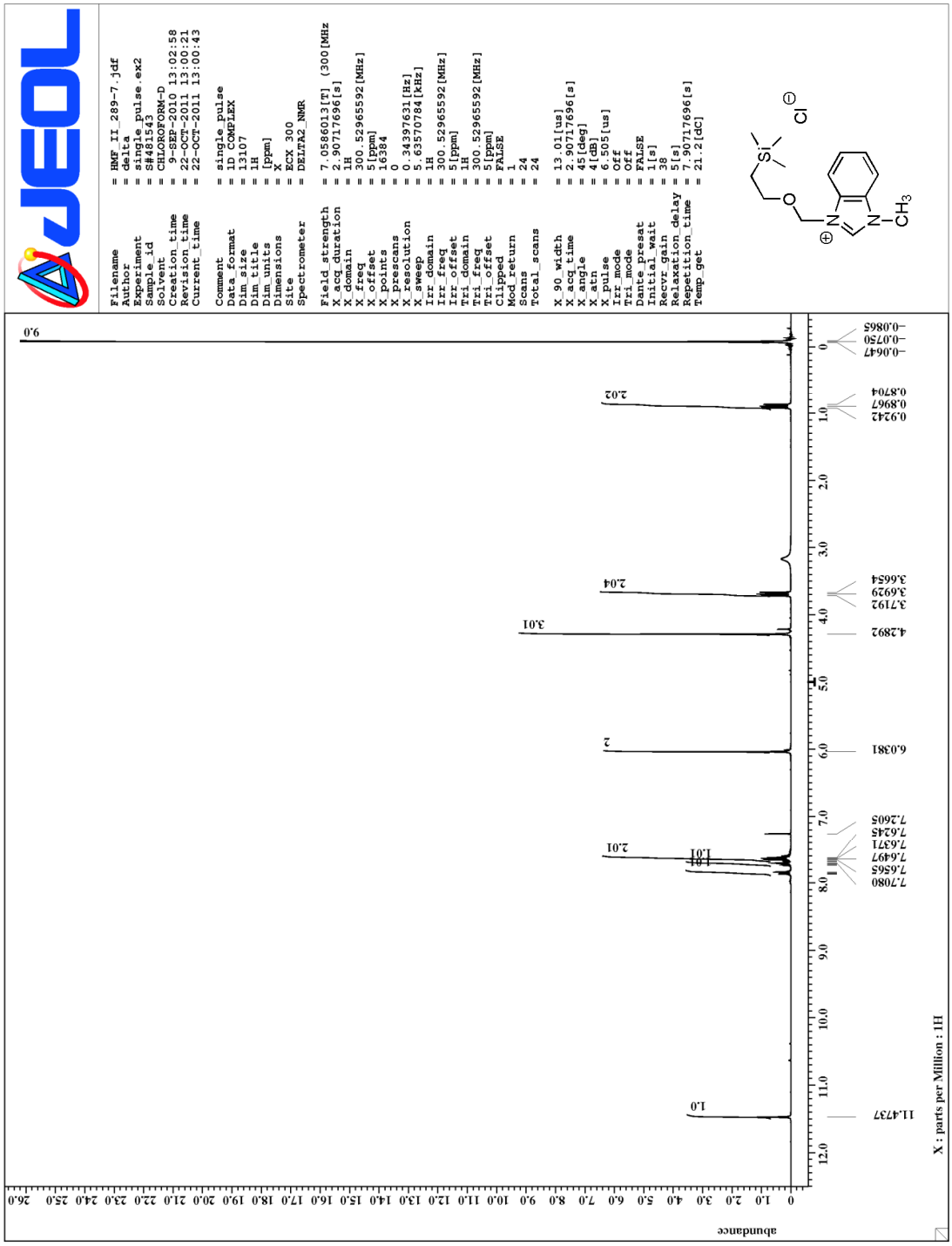
JEOL

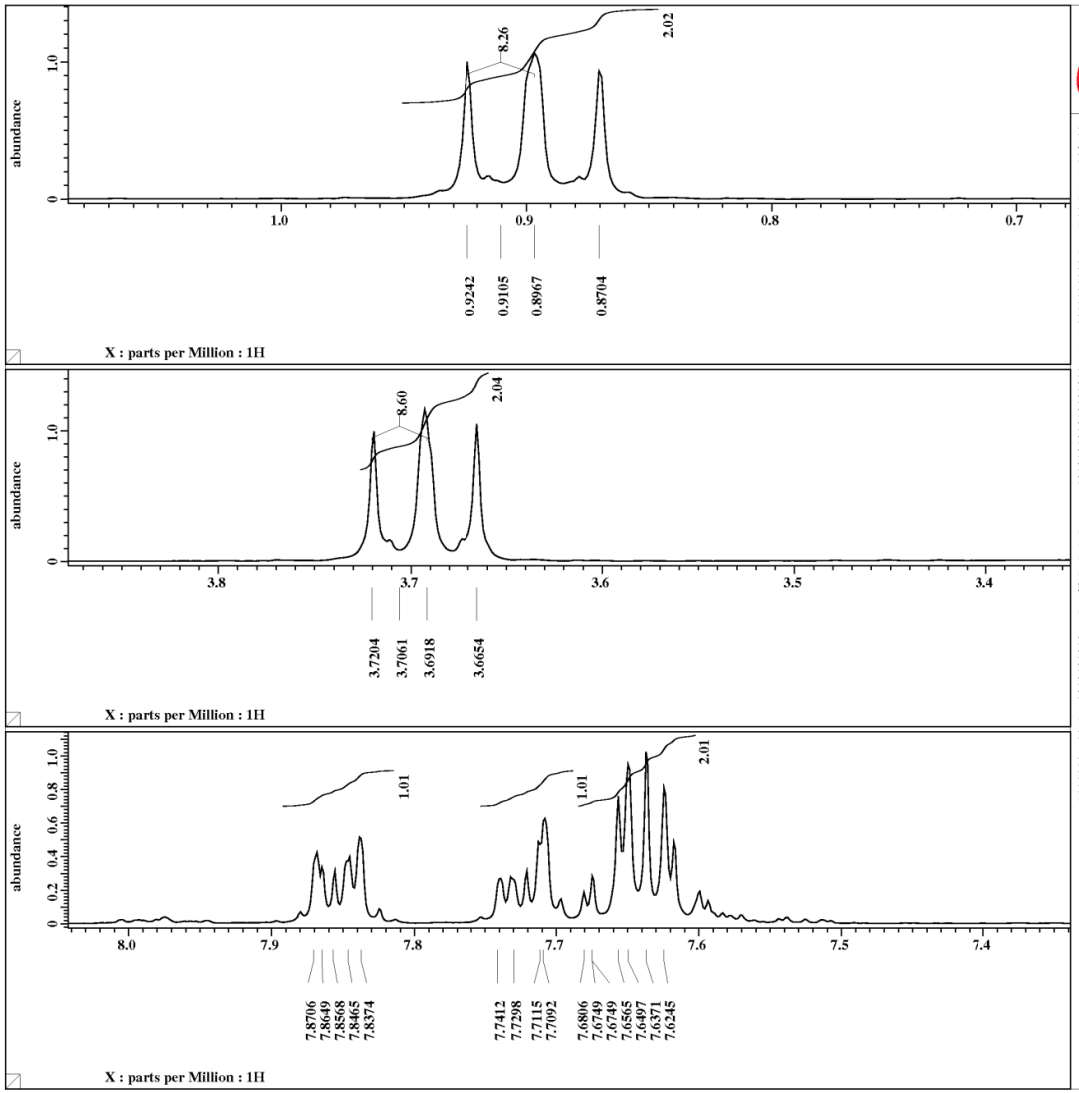
Filename	= HMF_III_108-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#607271
Solvent	= METHANOL-D3
Creation_time	= 17-FEB-2011 08:16:00
Revision_time	= 20-FEB-2011 17:49:29
Current_time	= 20-FEB-2011 17:50:27
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 10
Scans	= 350
Total_scans	= 350
X_90_width	= 10.73[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 9[dB]
X_pulse	= 3.57666667[us]
Irr_atn_dec	= 20[dB]
Irr_atn_noe	= 20[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 3[s]
Recvr_gain	= 50
Relaxation_delay	= 3[s]
Repetition_time	= 3.83361792[s]
Temp_get	= 21.8[dC]




JEOL
Filename = HMF_III_108-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#607271
Solvent = METHANOL-D3
Creation_time = 17-FEB-2011 08:16:00
Revision_time = 18-OCT-2011 16:54:27
Current_time = 18-OCT-2011 16:54:57
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 350
Total_scans = 350
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe_time = TRUE
Recyc_time = 3[s]
Relaxation_delay = 50
Repetition_time = 3[s]
Temp_get = 3.83361792[s]
= 21.8[dC]
Cl
N
N
CH3
abundance
0
0.1
0.2
0.3
110.0
120.0
130.0
140.0
113.5826
113.1534
127.2796
127.1842
130.9614
132.6688
142.6554
X : parts per Million : 13C

APPENDIX 5
^{1}H and ^{13}C NMR Spectra of
1-Methyl-3-trimethylsilylethoxymethyl-1*H*-benzimidazol-3-ium chloride (**81e**)


JEOL
Filename = HMF_II_289-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#481543
Solvent = CHLOROFORM-D
Creation_time = 9-SEP-2010 13:02:58
Revision_time = 22-OCT-2011 13:00:21
Current_time = 22-OCT-2011 13:00:43
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIMA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21.2[dC]
Cl
Si
O
N
N
CH3
abundance
X : parts per Million : 1H
9.0
2.02
2.04
3.01
2
2.01
1.0
1.01
0.9242
0.8967
0.8704
-0.0647
-0.0750
-0.0865
3.7192
3.6929
3.6654
4.2892
6.0381
7.7080
7.6565
7.6497
7.6371
7.6245
7.2605
11.4737

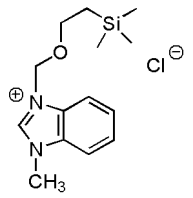


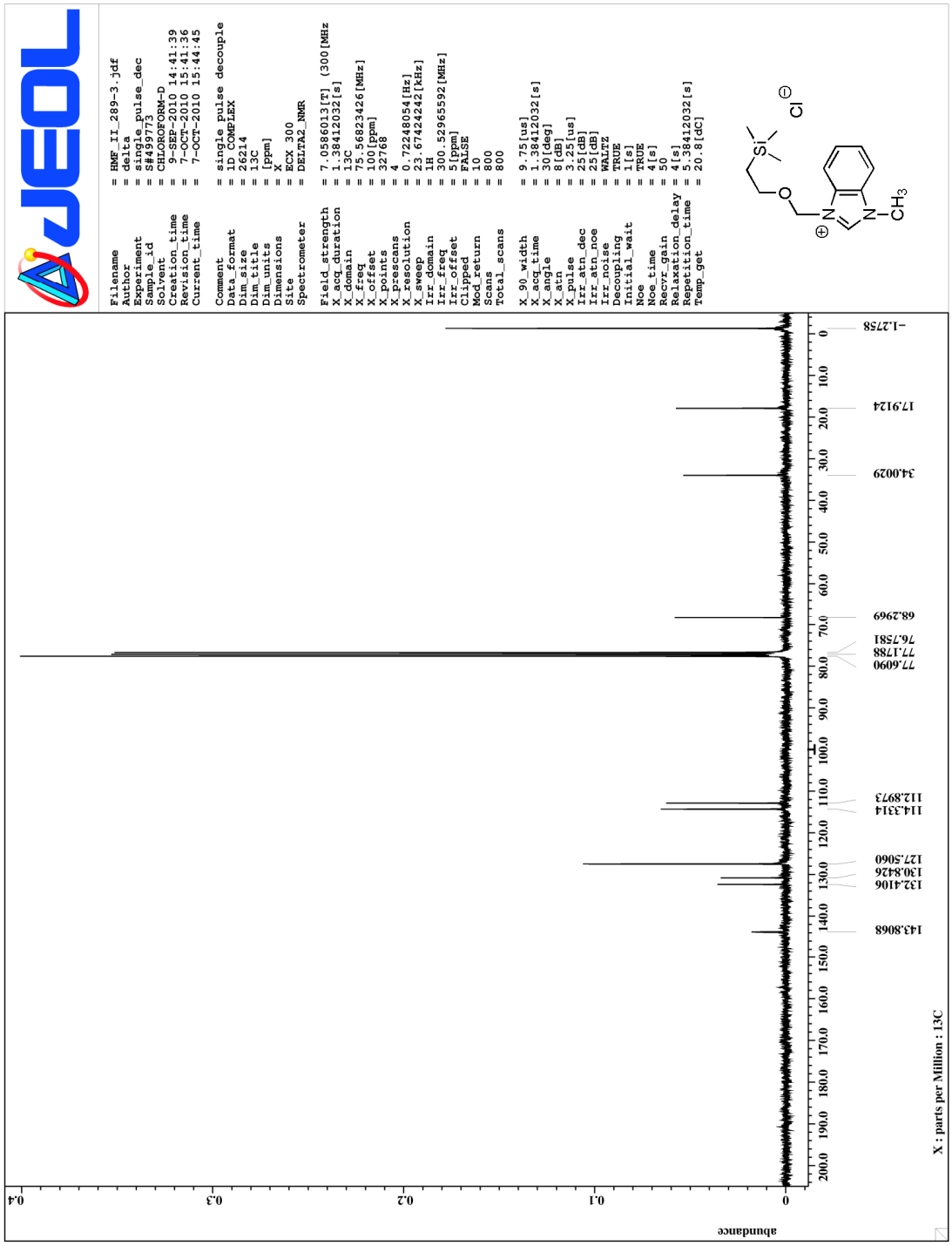

```
Filename          = HMF_II_289-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#481543
Solvent           = CHLOROFORM-D
Creation_time     =  9-SEP-2010 13:02:58
Revision_time     = 22-OCT-2011 13:01:29
Current_time      = 22-OCT-2011 13:02:09

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 300
Spectrometer      = DELTA2_NMR

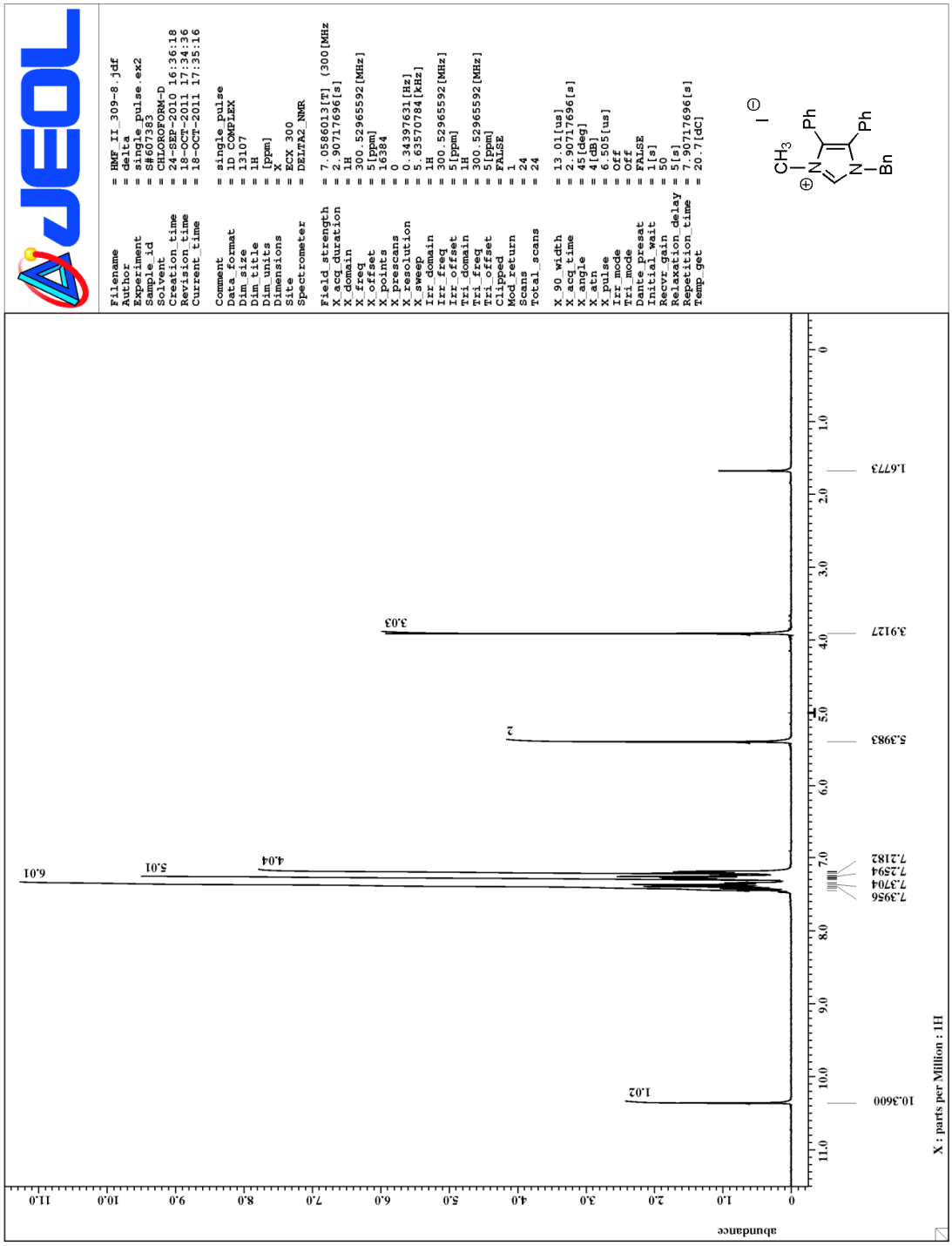
Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 2.90717696[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.34397631[Hz]
X_sweep           = 5.63570784[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 300.52965592[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 24
Total_scans       = 24

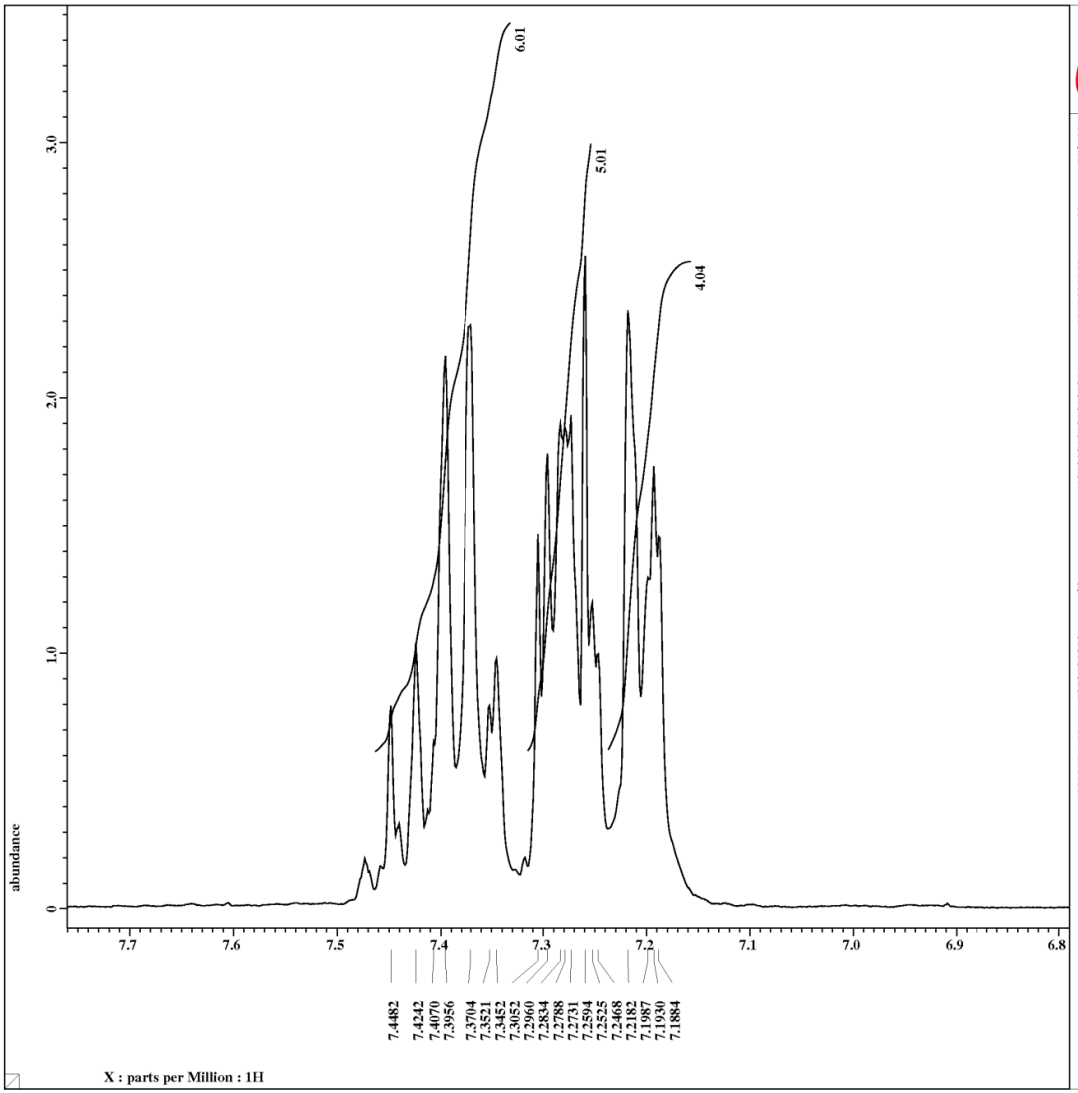
X_90_width        = 13.01[us]
X_acq_time        = 2.90717696[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.505[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 7.90717696[s]
Temp_get          = 21.2[dC]
```




JEOL
Filename = RMF_II_289-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#499773
Solvent = CHLOROFORM-D
Creation_time = 9-SEP-2010 14:41:39
Revision_time = 7-OCT-2010 15:41:36
Current_time = 7-OCT-2010 15:44:45
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 800
Total_scans = 800
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 5.38412032[s]
Temp_get = 20.8[dC]
Cl
Si
O
N
N
CH3
abundance
0
0.1
0.2
0.3
0.4
-1.2758
17.9124
34.0029
68.2969
76.7581
77.1788
77.6090
112.8973
114.3314
127.5060
130.8426
132.4106
143.8068
X : parts per Million : 13C

APPENDIX 6
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-3-methyl-4,5-diphenyl-1*H*-imidazol-3-ium iodide (**81f**)


JEOL
Filename = HMF_II_309-8.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#607383
Solvent = CHLOROFORM-D
Creation_time = 24-SEP-2010 16:36:18
Revision_time = 18-OCT-2011 17:34:36
Current_time = 18-OCT-2011 17:35:16
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.7[dC]
CH3
Ph
Ph
Bn
abundance
X : parts per Million : 1H
10.3600
7.3956
7.3704
7.2594
7.2182
5.3983
3.9127
1.6773
6.01
5.01
4.04
3.03
2
1.02

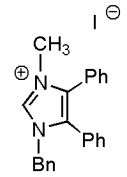


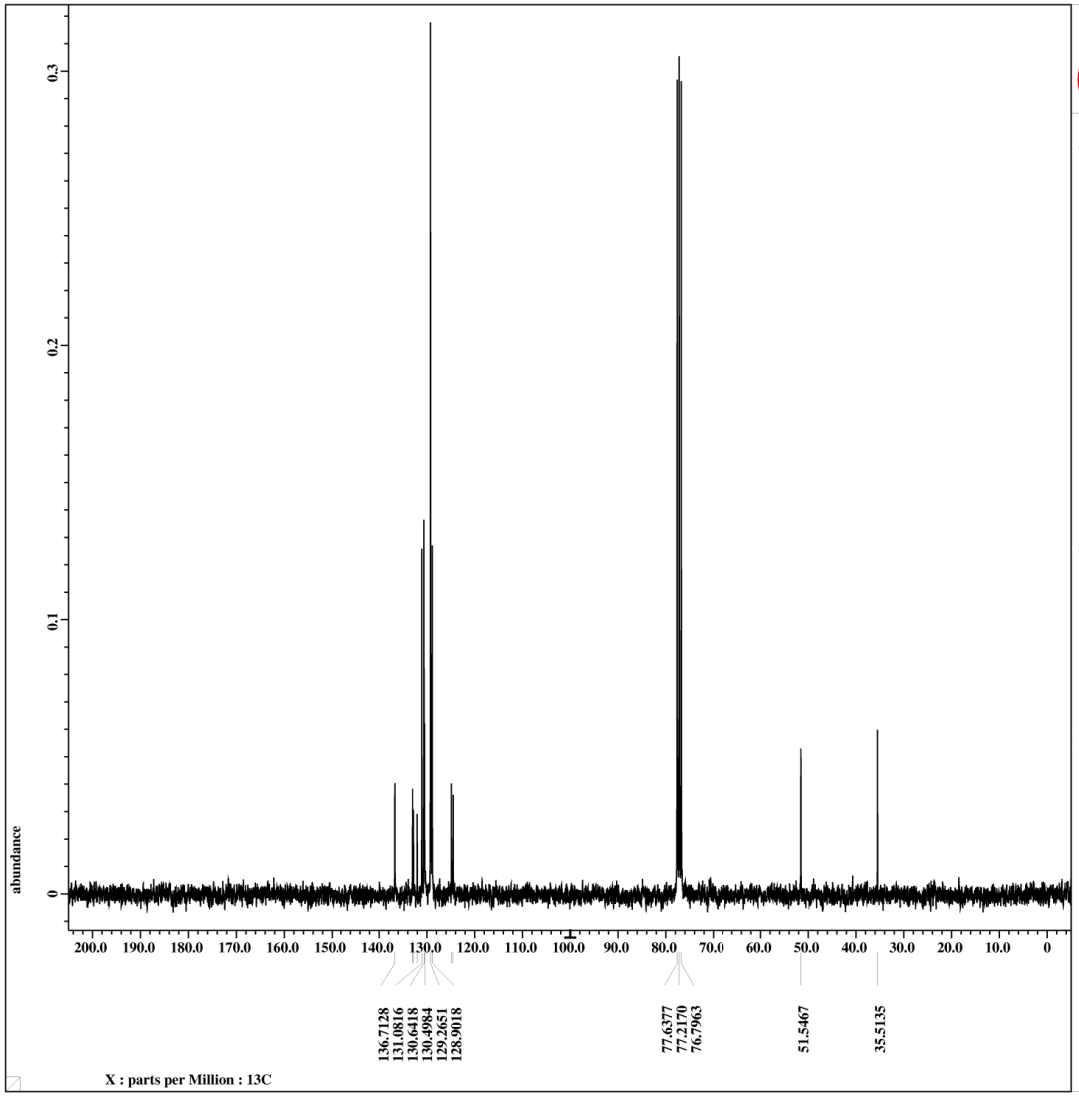
```
Filename          = HMF_II_309-8.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#607383
Solvent           = CHLOROFORM-D
Creation_time     = 24-SEP-2010 16:36:18
Revision_time     = 18-OCT-2011 17:34:36
Current_time      = 18-OCT-2011 17:35:39

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 300
Spectrometer      = DELTA2_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 2.90717696[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.34397631[Hz]
X_sweep           = 5.63570784[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 300.52965592[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 24
Total_scans       = 24

X_90_width        = 13.01[us]
X_acq_time        = 2.90717696[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.505[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 7.90717696[s]
Temp_get          = 20.7[dC]
```





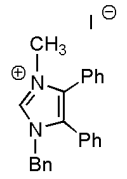
JEOL

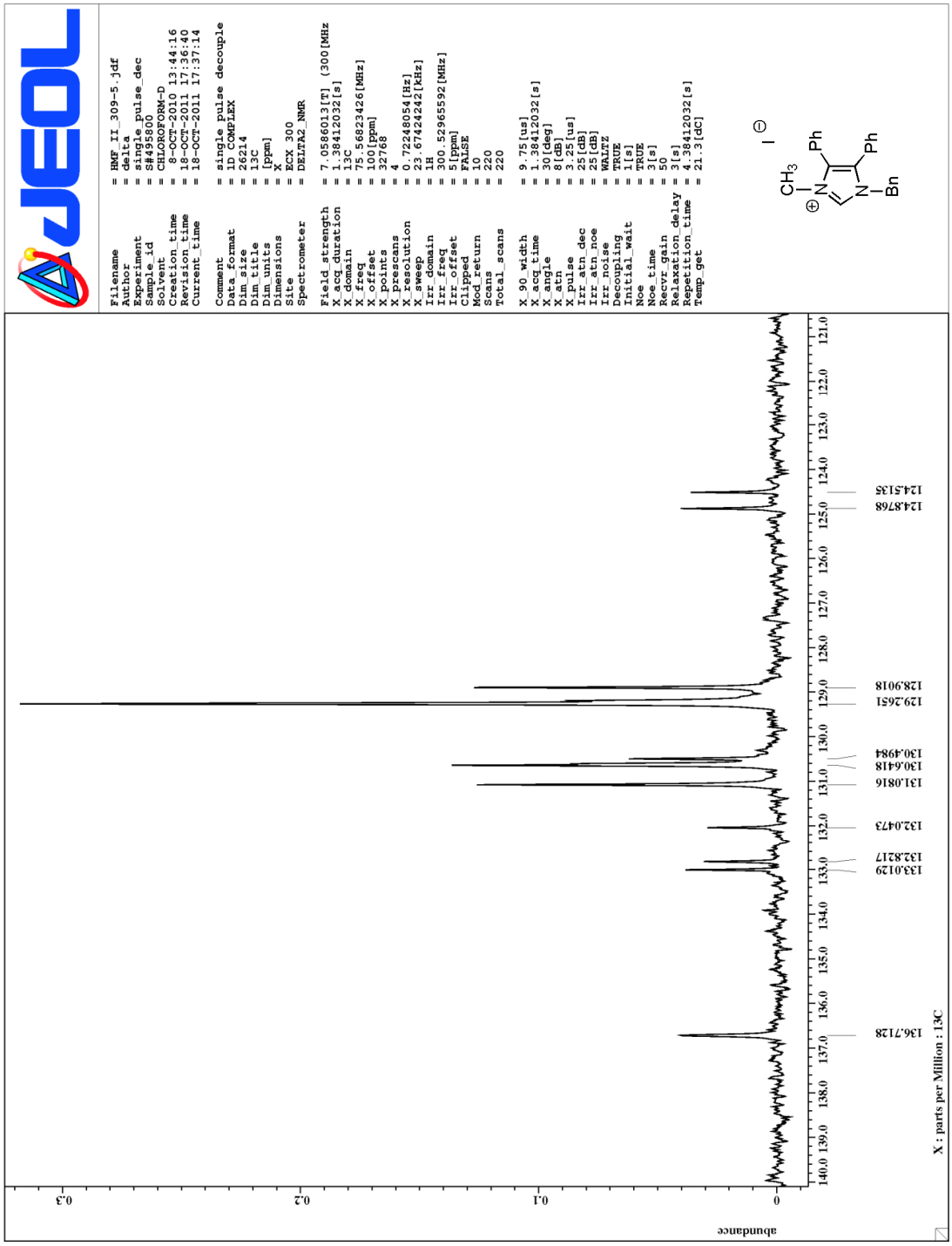
```
Filename          = HMF_II_309-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#495800
Solvent           = CHLOROFORM-D
Creation_time     =  8-OCT-2010 13:44:16
Revision_time     = 19-NOV-2010 23:45:05
Current_time      = 19-NOV-2010 23:46:58

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 300
Spectrometer      = DELTA2_NMR

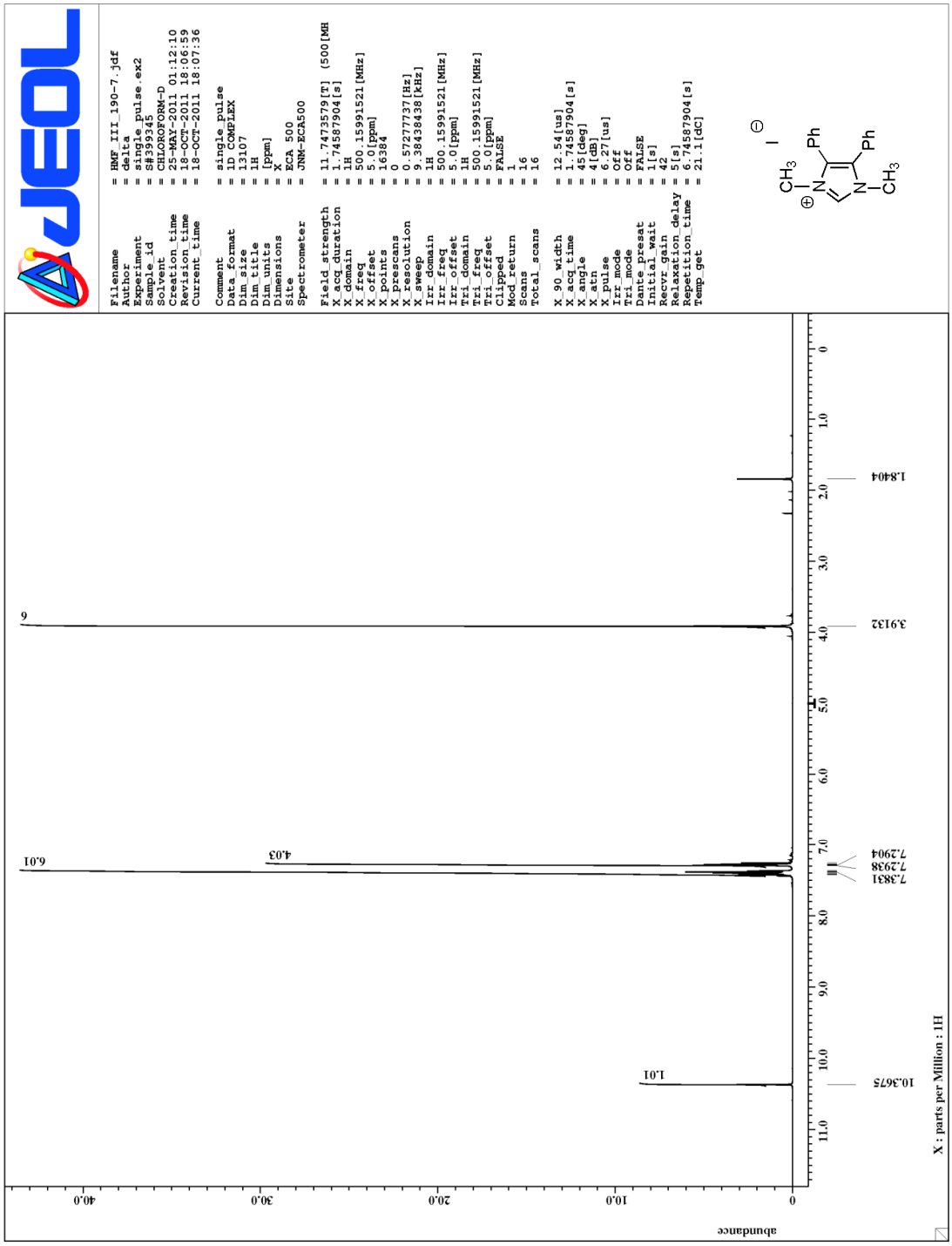
Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.38412032[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.72248054[Hz]
X_sweep           = 23.67424242[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 220
Total_scans       = 220

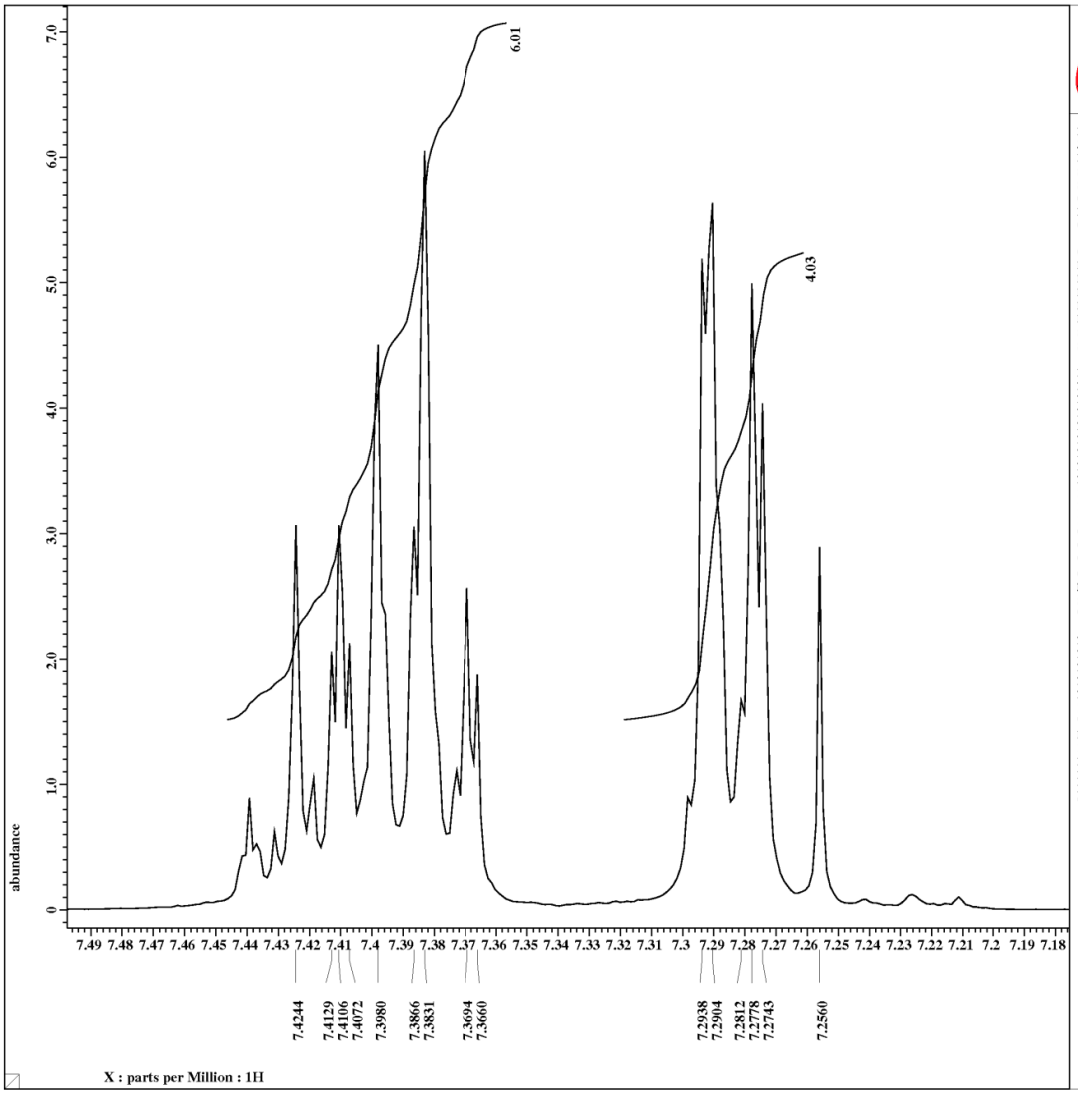
X_90_width        = 9.75[us]
X_acq_time        = 1.38412032[s]
X_angle           = 30[deg]
X_atn             = 8[dB]
X_pulse           = 3.25[us]
Irr_atn_dec       = 25[dB]
Irr_atn_noe       = 25[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 3[s]
Recvr_gain        = 50
Relaxation_delay  = 3[s]
Repetition_time   = 4.38412032[s]
Temp_get          = 21.3[dC]
```

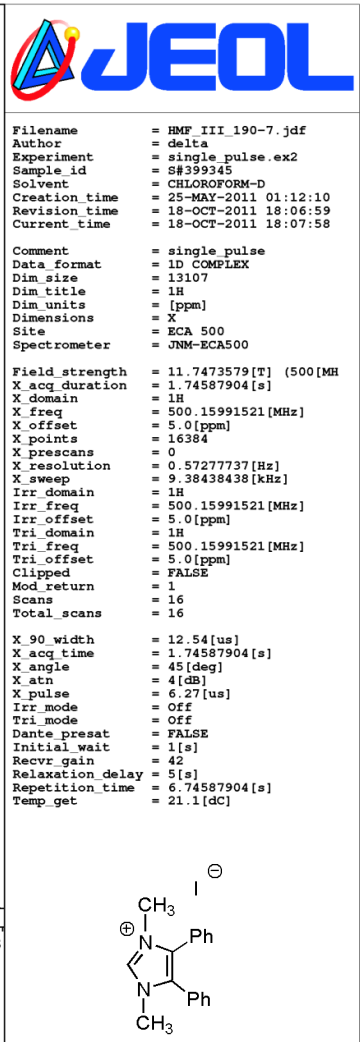


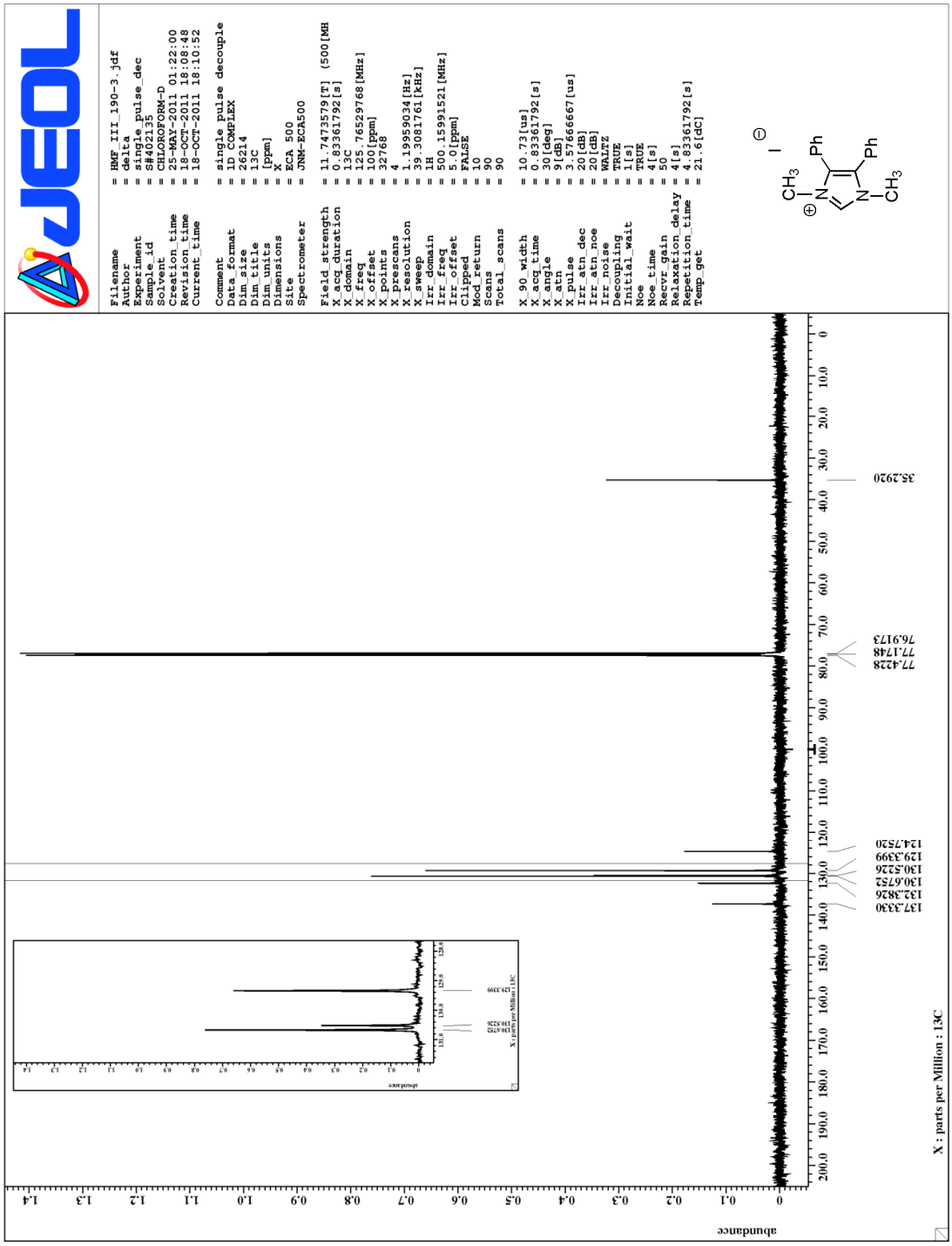

JEOL
Filename = BMF_II_309-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#495800
Solvent = CHLOROFORM-D
Creation_time = 8-OCT-2010 13:44:16
Revision_time = 18-OCT-2011 17:36:40
Current_time = 18-OCT-2011 17:37:14
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DEIFRA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 220
Total_scans = 220
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.38412032[s]
Temp_get = 21.3[dC]
CH3
N
Ph
Ph
N
Bn
I
abundance
0
0.1
0.2
0.3
140.0 139.0 138.0 137.0 136.0 135.0 134.0 133.0 132.0 131.0 130.0 129.0 128.0 127.0 126.0 125.0 124.0 123.0 122.0 121.0
136.7128
133.0129
132.8217
132.0473
131.0816
130.6418
130.4984
129.2651
128.9018
124.8768
124.5135
X : parts per Million : 13C

APPENDIX 7
^{1}H and ^{13}C NMR Spectra of
1,3-Dimethyl-4,5-diphenyl-1*H*-imidazol-3-ium iodide (**81g**)

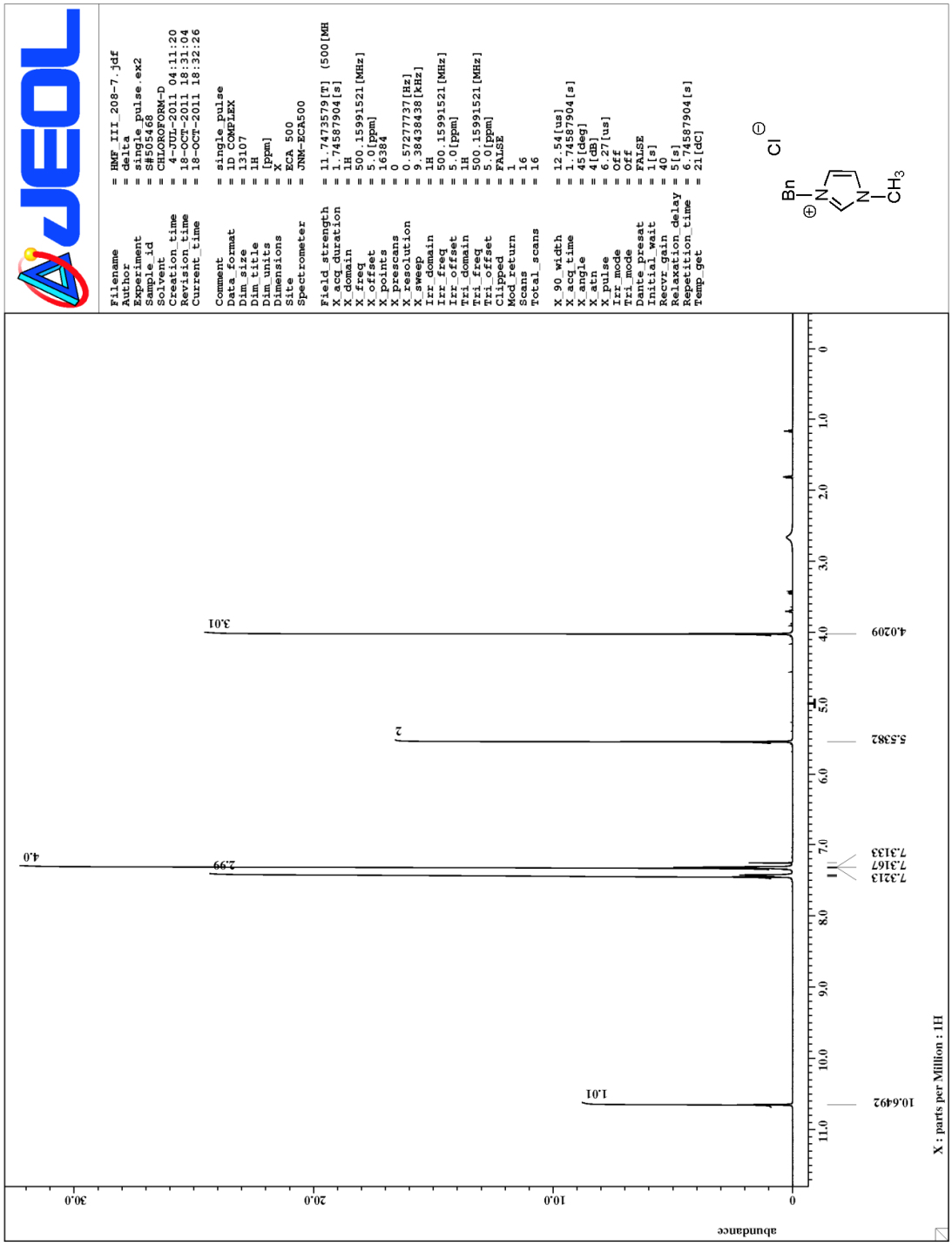

JEOL
Filename = HMF III 190-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#399345
Solvent = CHLOROFORM-D
Creation_time = 25-MAY-2011 01:12:10
Revision_time = 18-OCT-2011 18:06:59
Current_time = 18-OCT-2011 18:07:36
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.1[dC]
CH3
N
Ph
Ph
N
CH3
I
6
6.01
4.03
1.01
1.8404
3.9132
7.3831
7.2938
7.2904
10.3675
abundance
X : parts per Million : 1H

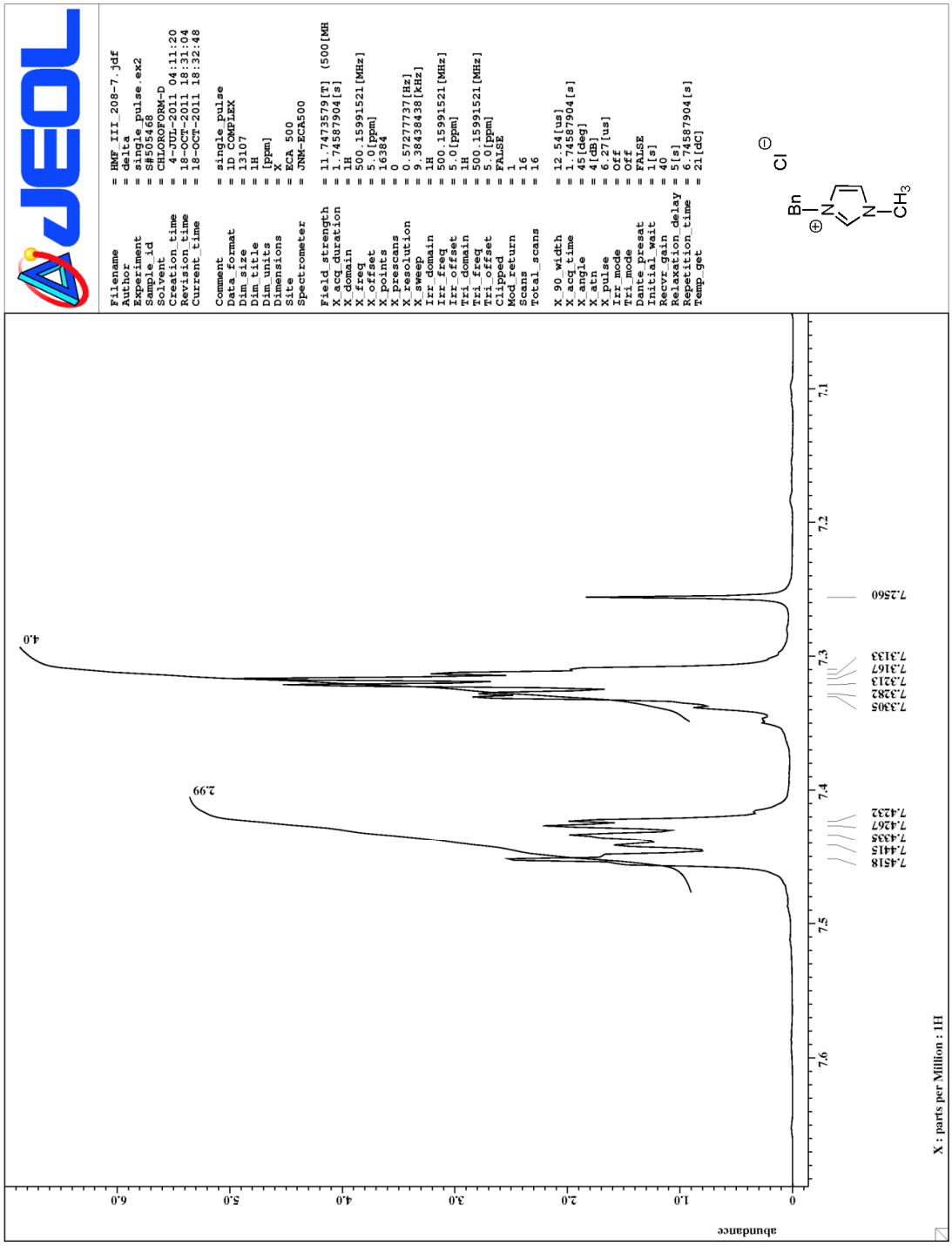

abundance
6.01
4.03
7.49 7.48 7.47 7.46 7.45 7.44 7.43 7.42 7.41 7.4 7.39 7.38 7.37 7.36 7.35 7.34 7.33 7.32 7.31 7.3 7.29 7.28 7.27 7.26 7.25 7.24 7.23 7.22 7.21 7.2 7.19 7.18
7.4244
7.4129
7.4106
7.4072
7.3980
7.3866
7.3831
7.3694
7.3660
7.2938
7.2904
7.2812
7.2778
7.2743
7.2560
X : parts per Million : 1H

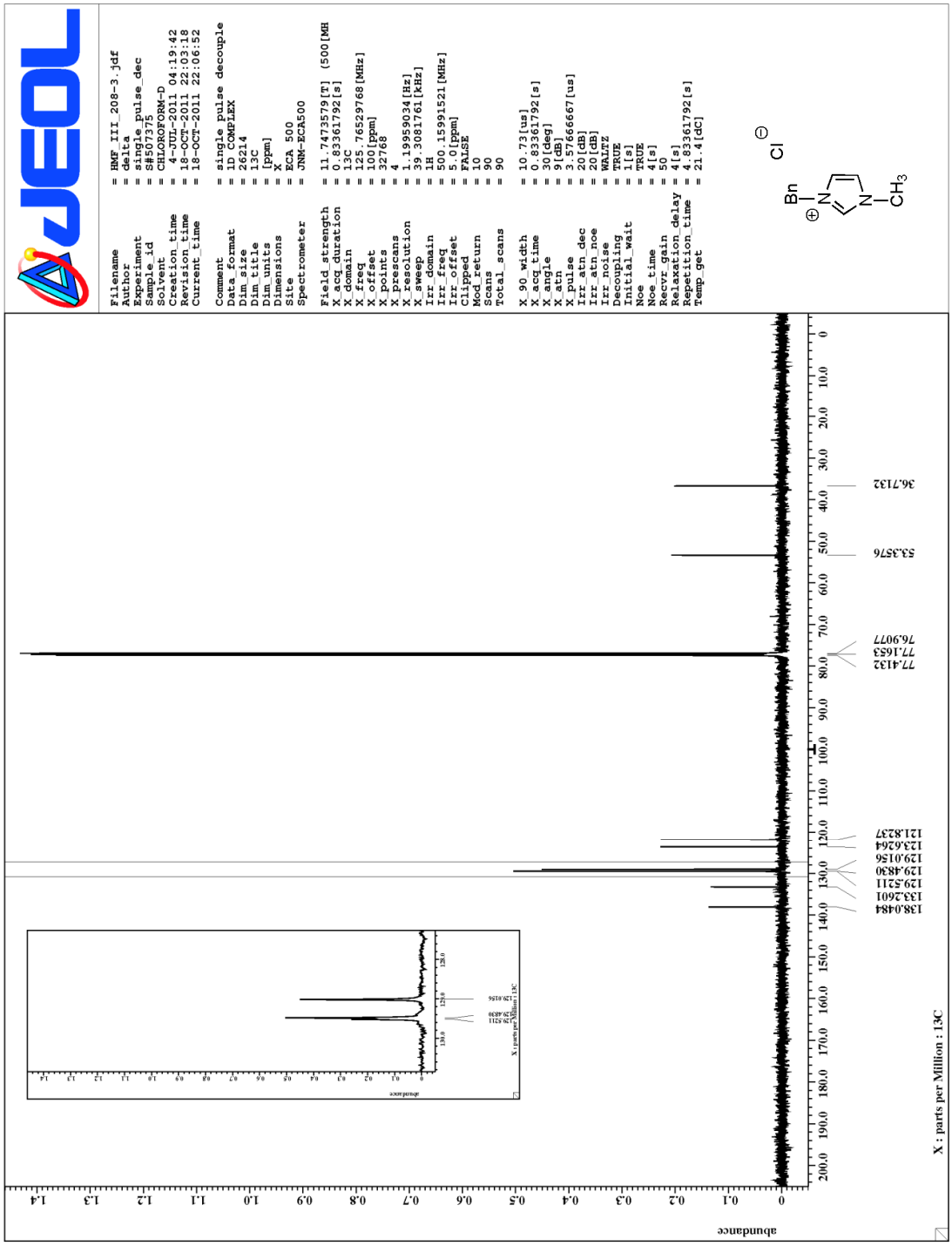

Filename = HMF_III_190-7.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#399345
Solvent = CHLOROFORM-D
Creation_time = 25-MAY-2011 01:12:10
Revision_time = 18-OCT-2011 18:06:59
Current_time = 18-OCT-2011 18:07:58
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.1[dC]
CH3
N
Ph
Ph
N
CH3
I


Filename = HMF III_190-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#4021135
Solvent = CHLOROFORM-D
Creation_time = 25-MAY-2011 01:22:00
Revision_time = 18-OCT-2011 18:08:48
Current_time = 18-OCT-2011 18:10:52
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 90
Total_scans = 90
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.6[dC]
35.2920
76.9173
77.1748
77.4228
124.7520
129.3399
130.5226
130.6752
132.3826
137.3330
abundance
X : parts per Million : 13C

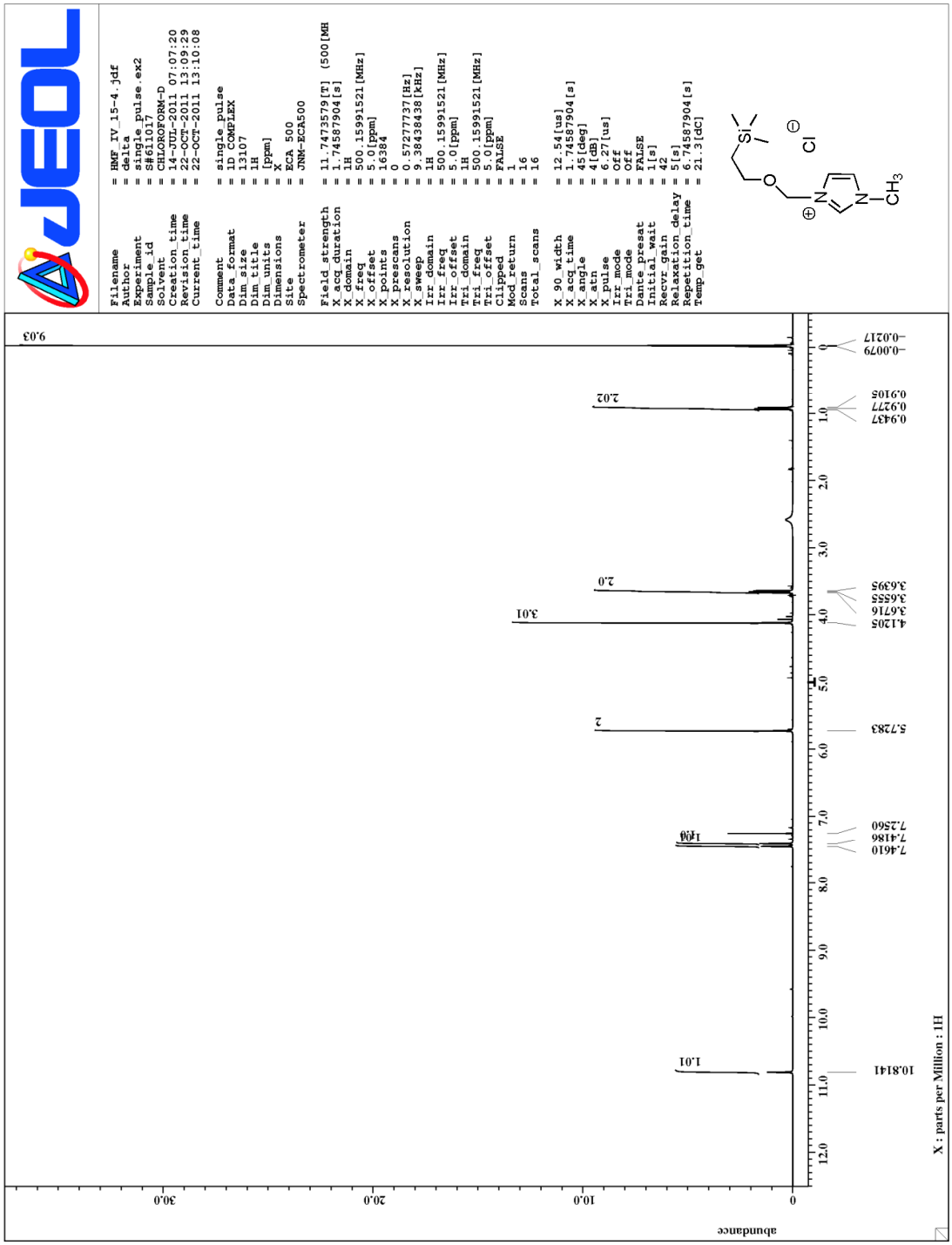
APPENDIX 8
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-1-methyl-1*H*-imidazol-3-ium chloride (**81h**)

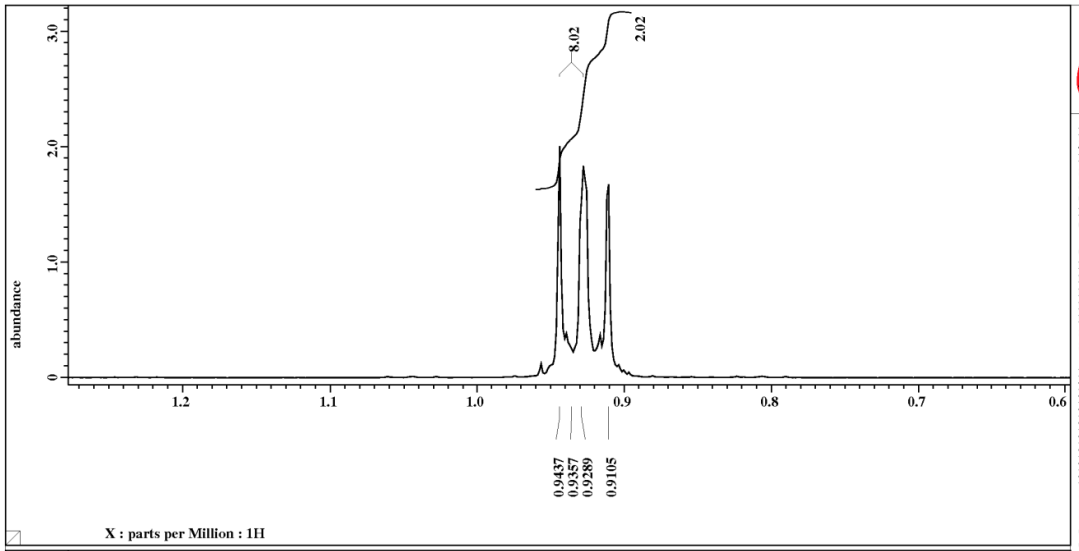

Filename = RMF_III_208-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#505468
Solvent = CHLOROFORM-D
Creation_time = 4-JUL-2011 04:11:20
Revision_time = 18-OCT-2011 18:31:04
Current_time = 18-OCT-2011 18:32:26
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21[dC]
Cl
Bn
CH3
abundance
X : parts per Million : 1H
4.0
2.99
3.01
2
1.01
10.6492
7.3213
7.3167
7.3133
5.5382
4.0209

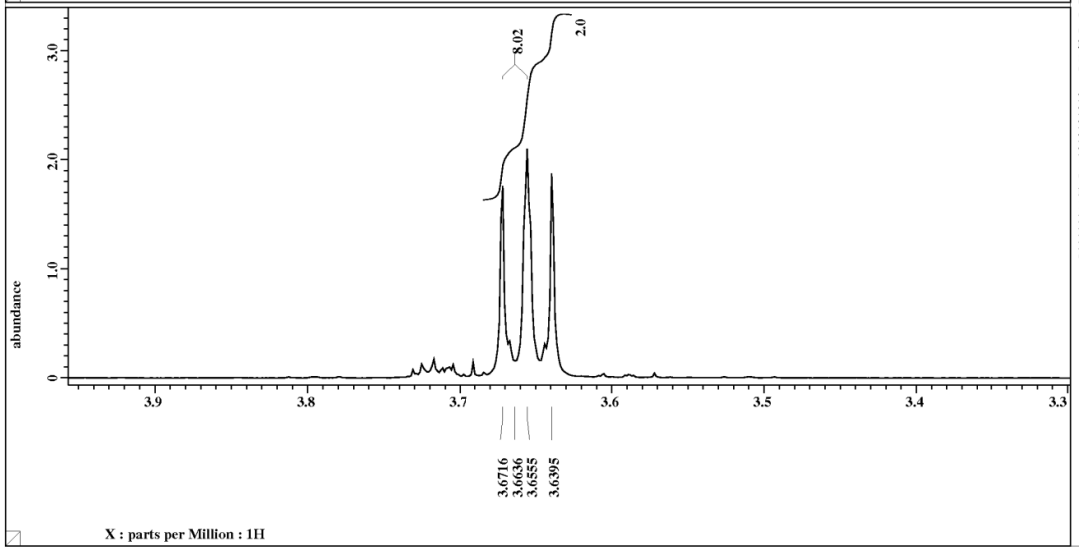

JEOL
Filename = HMF III_208-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#505468
Solvent = CHLOROFORM-D
Creation_time = 4-JUL-2011 04:11:20
Revision_time = 18-OCT-2011 18:31:04
Current_time = 18-OCT-2011 18:32:48
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21[dC]
Cl⊖
Bn
CH3
abundance
X : parts per Million : 1H
4.0
2.99
7.2560
7.3305
7.3382
7.3513
7.3167
7.3133
7.4518
7.4415
7.4335
7.4267
7.4232


JEOL
Filename = HMF III_208-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#507375
Solvent = CHLOROFORM-D
Creation_time = 4-JUL-2011 04:19:42
Revision_time = 18-OCT-2011 22:03:18
Current_time = 18-OCT-2011 22:06:52
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 90
Total_scans = 90
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.4[dC]
Cl
Bn
N
N
CH3
abundance
X : parts per Million : 13C
138.0484
133.2601
129.5211
129.4830
129.0156
123.6264
121.8237
77.4132
77.1653
76.9077
53.3576
36.7132

APPENDIX 9
^{1}H and ^{13}C NMR Spectra of
1-Methyl-3-trimethylsilylethoxymethyl-1*H*-imidazol-3-ium chloride (**81i**)


JEOL
Filename = HMF_IV_15-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#611017
Solvent = CHLOROFORM-D
Creation_time = 14-JUL-2011 07:07:20
Revision_time = 22-OCT-2011 13:09:29
Current_time = 22-OCT-2011 13:10:08
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.3[dC]
Cl
abundance
X : parts per Million : 1H
9.03
2.02
3.01
2.0
2
1.01
10.8141
7.4610
7.4186
7.2560
5.7283
4.1205
3.6716
3.6555
3.6395
0.9437
0.9277
0.9105
0.0079
0.0217





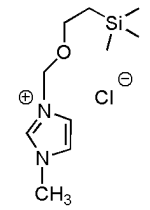
JEOL

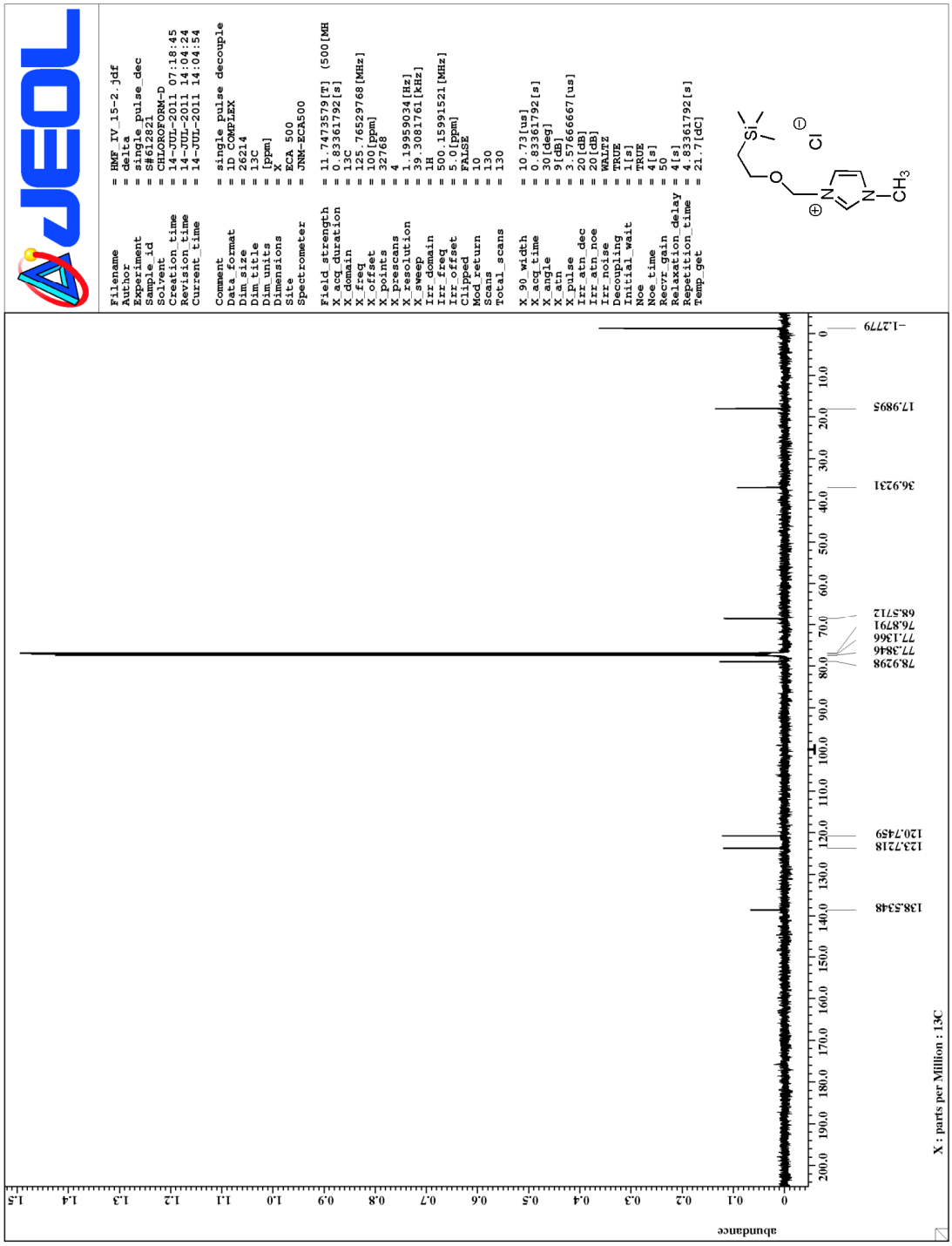
```
Filename          = HMF_IV_15-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#611017
Solvent           = CHLOROFORM-D
Creation_time     = 14-JUL-2011 07:07:20
Revision_time     = 22-OCT-2011 13:10:52
Current_time      = 22-OCT-2011 13:13:20

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

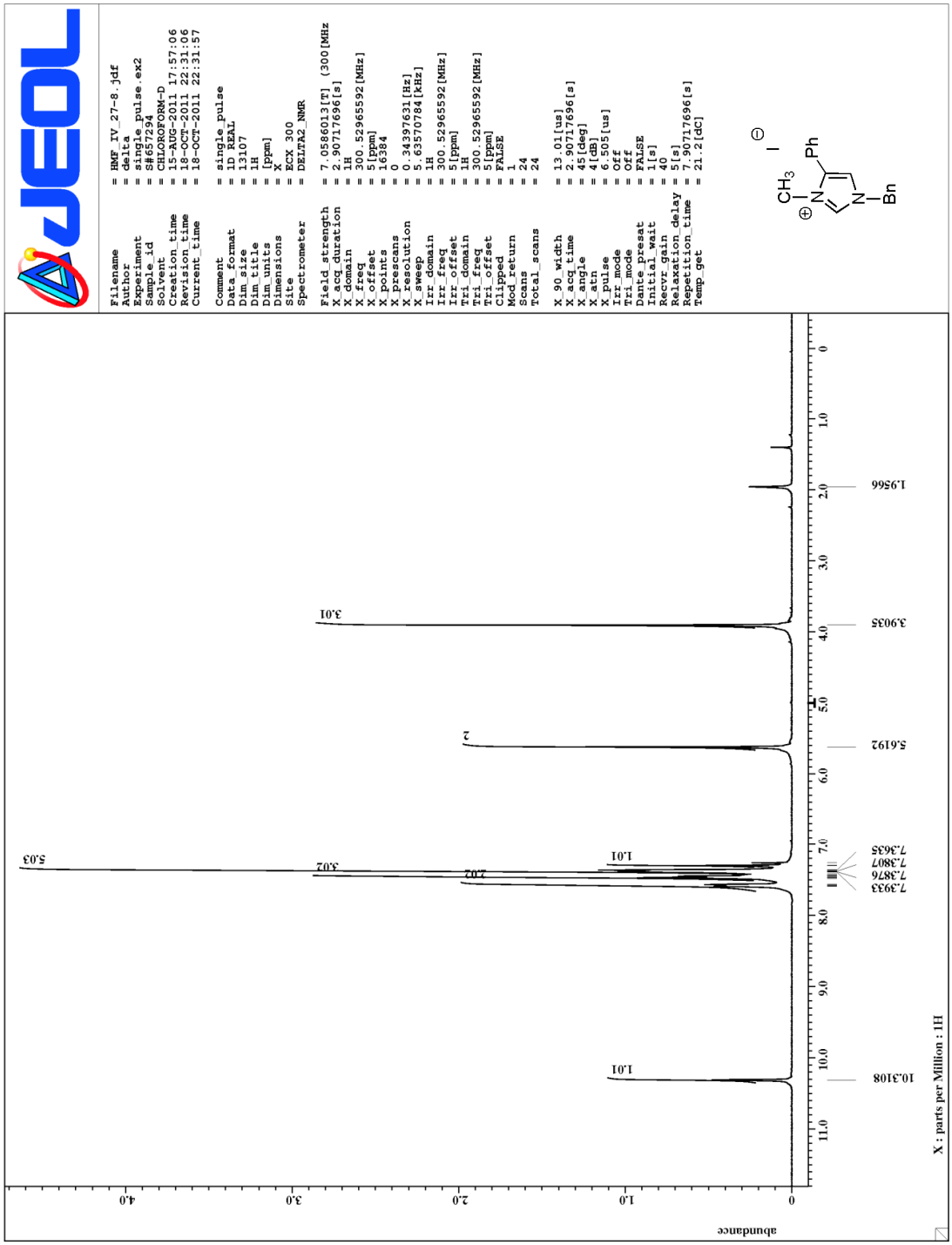
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

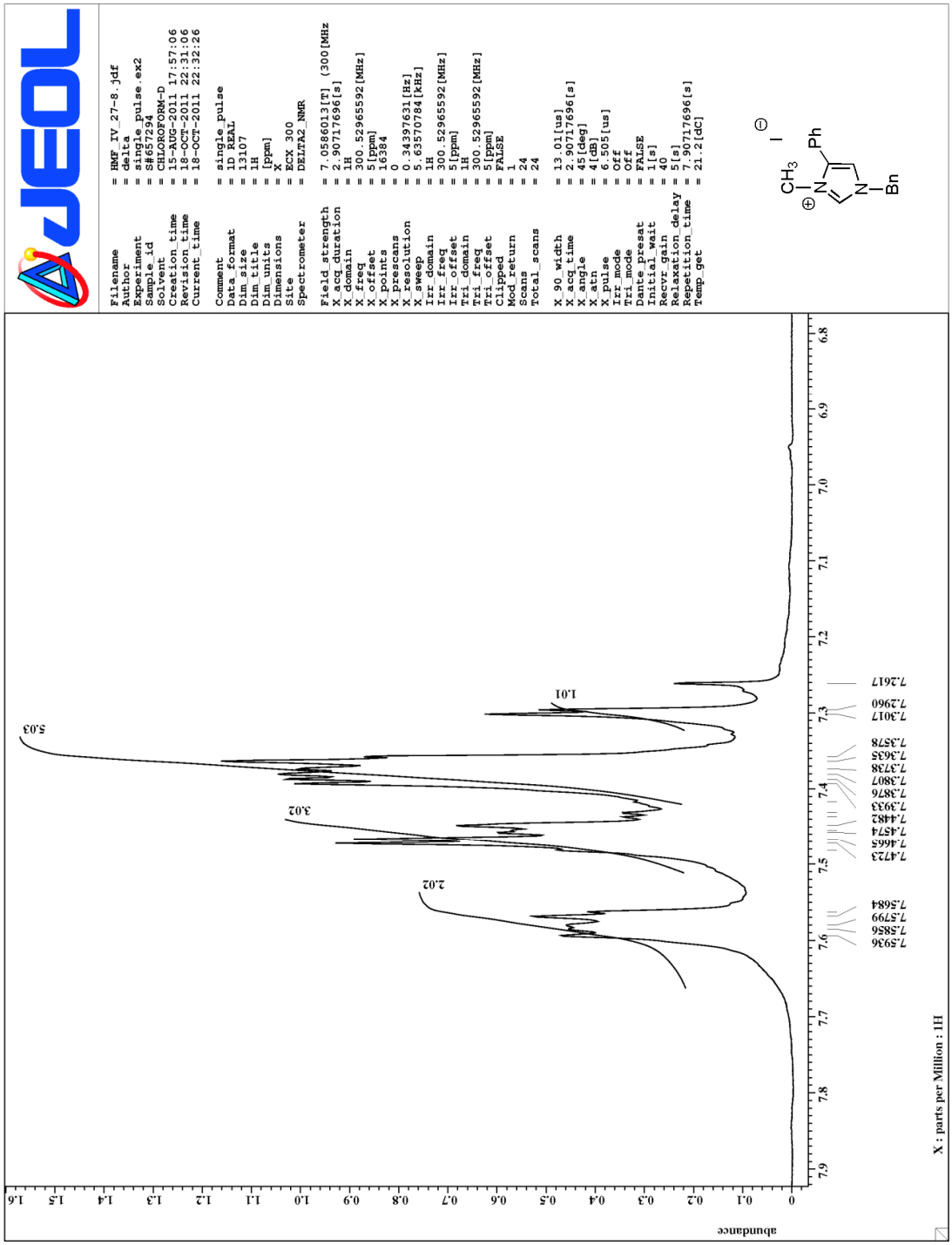
X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 21.3[dC]
```

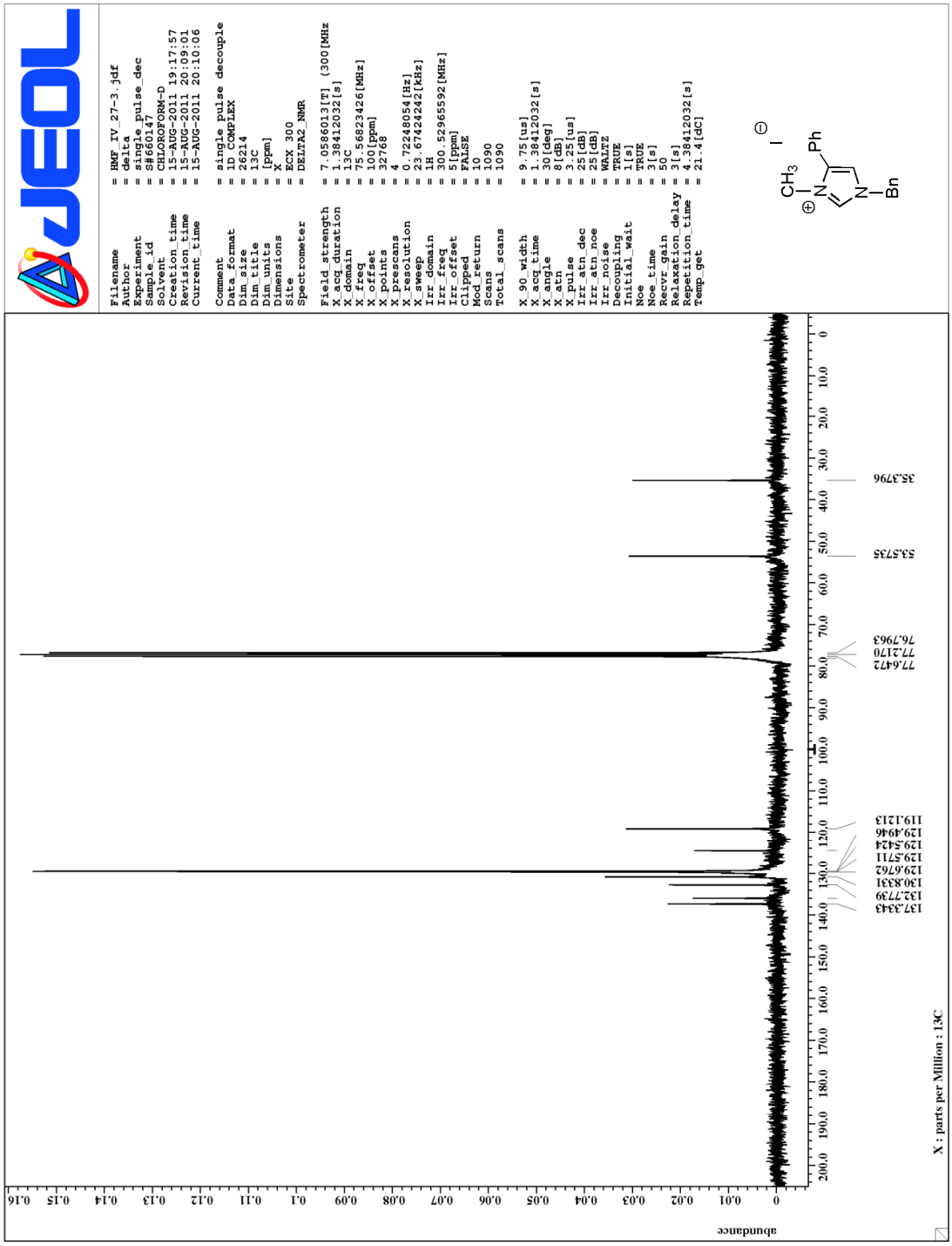


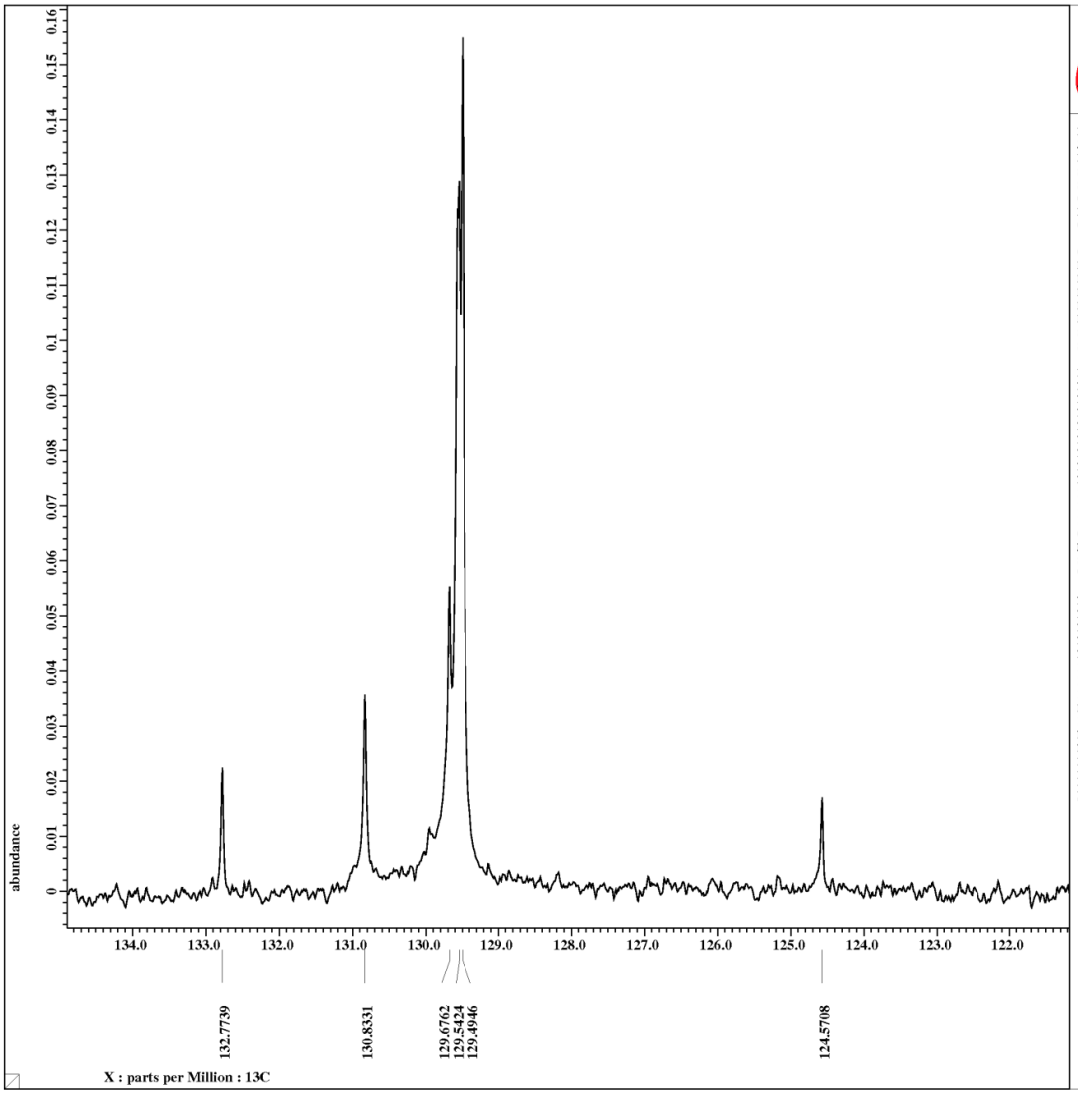

JEOL
Filename = HMF_IV_15-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#612821
Solvent = CHLOROFORM-D
Creation_time = 14-JUL-2011 07:18:45
Revision_time = 14-JUL-2011 14:04:24
Current_time = 14-JUL-2011 14:04:54
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 130
Total_scans = 130
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 2.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe_time = TRUE
Recyr_gain = 4[s]
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.7[dC]
Si
Cl
N
N
CH3
-1.2779
17.9895
36.9231
68.5712
76.8791
77.1366
77.3846
78.9298
120.7459
123.7218
138.5348
abundance
X : parts per Million : 13C

APPENDIX 10
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-3-methyl-4-phenyl-1*H*-imidazol-3-ium iodide (**81j**)


JEOL
Filename = HMF_IV_27-8.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#657294
Solvent = CHLOROFORM-D
Creation_time = 15-AUG-2011 17:57:06
Revision_time = 18-OCT-2011 22:31:06
Current_time = 18-OCT-2011 22:31:57
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIFAZ_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21.2[dC]
CH3
Ph
Bn
5.03
3.02
2.02
1.01
1.01
3.01
2
1.01
10.3108
7.3933
7.3876
7.3807
7.3635
5.6192
3.9035
1.9566
abundance
X : parts per Million : 1H


JEOL
Filename = HMF_IV_27-8.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#657294
Solvent = CHLOROFORM-D
Creation_time = 15-AUG-2011 17:57:06
Revision_time = 18-OCT-2011 22:31:06
Current_time = 18-OCT-2011 22:32:26
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21.2[dC]
CH3
Ph
Bn
5.03
3.02
2.02
1.01
7.2617
7.3017
7.2960
7.3578
7.3635
7.3738
7.3807
7.3876
7.3933
7.4482
7.4574
7.4665
7.4723
7.5684
7.5799
7.5856
7.5936
abundance
X : parts per Million : 1H


Filename = RMF_IV_27-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#660147
Solvent = CHLOROFORM-D
Creation_time = 15-AUG-2011 19:17:57
Revision_time = 15-AUG-2011 20:09:01
Current_time = 15-AUG-2011 20:10:06
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1090
Total_scans = 1090
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.38412032[s]
Temp_get = 21.4[dC]
abundance
X : parts per Million : 13C
137.3343
132.7739
130.8331
129.6762
129.5711
129.5424
129.4946
119.1213
77.6472
77.2170
76.7963
53.5735
35.3796



JEOL

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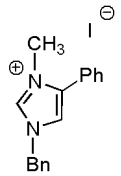
Filename          = HMF_IV_27-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#660147
Solvent           = CHLOROFORM-D
Creation_time     = 15-AUG-2011 19:17:57
Revision_time     = 18-OCT-2011 23:40:02
Current_time      = 18-OCT-2011 23:43:33

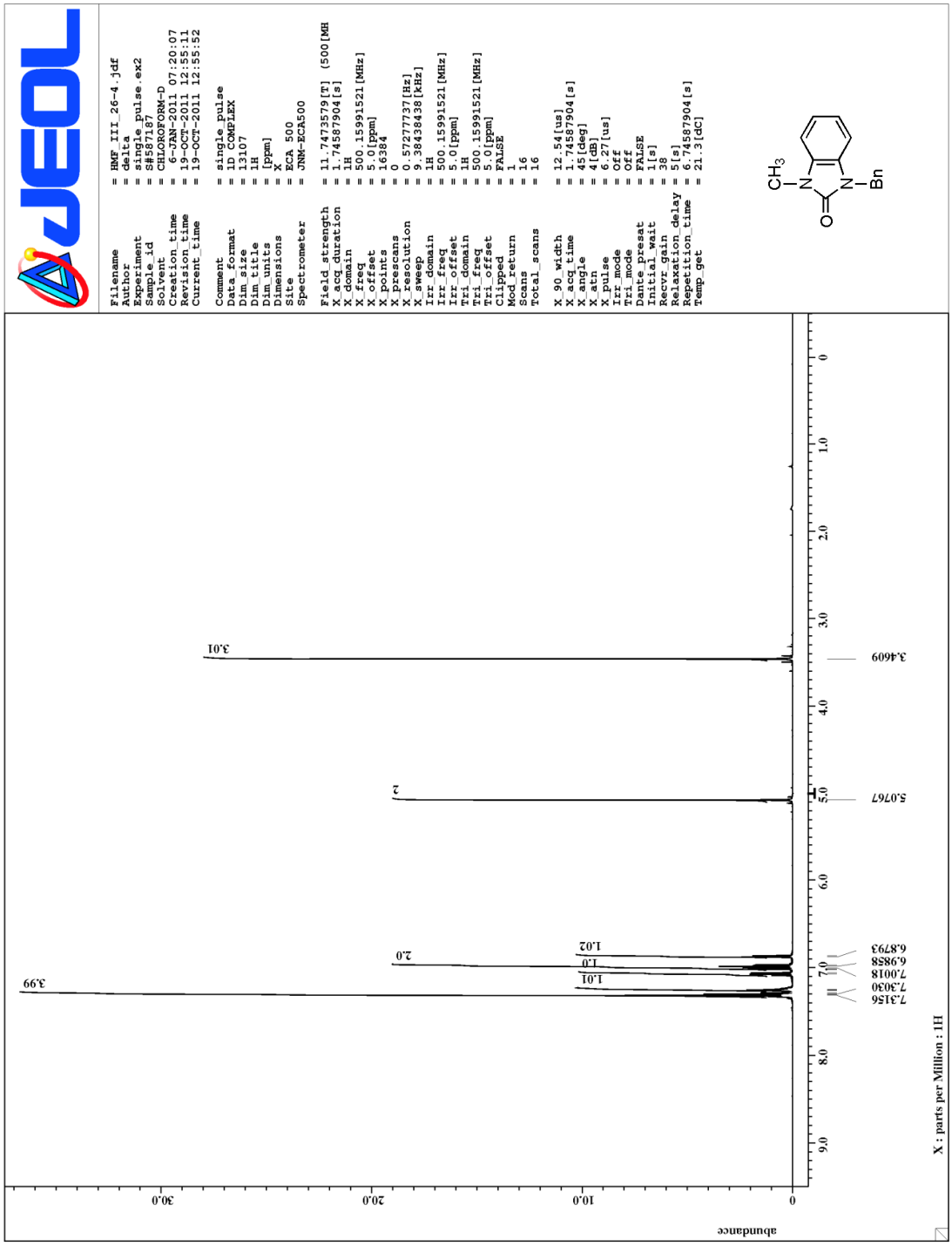
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 300
Spectrometer      = DELTA2_NMR

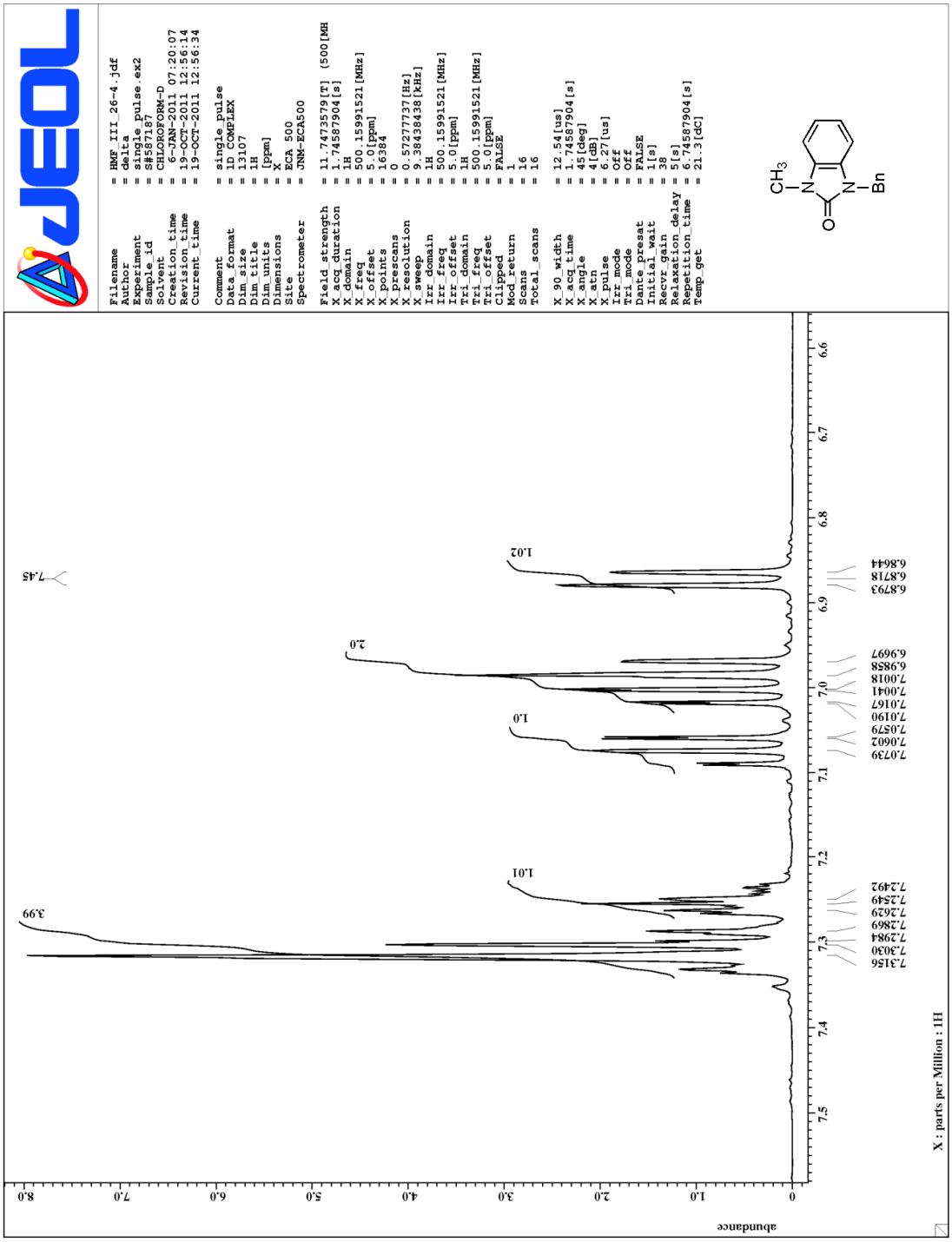
Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.38412032[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.72248054[Hz]
X_sweep           = 23.67424242[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 1090
Total_scans       = 1090

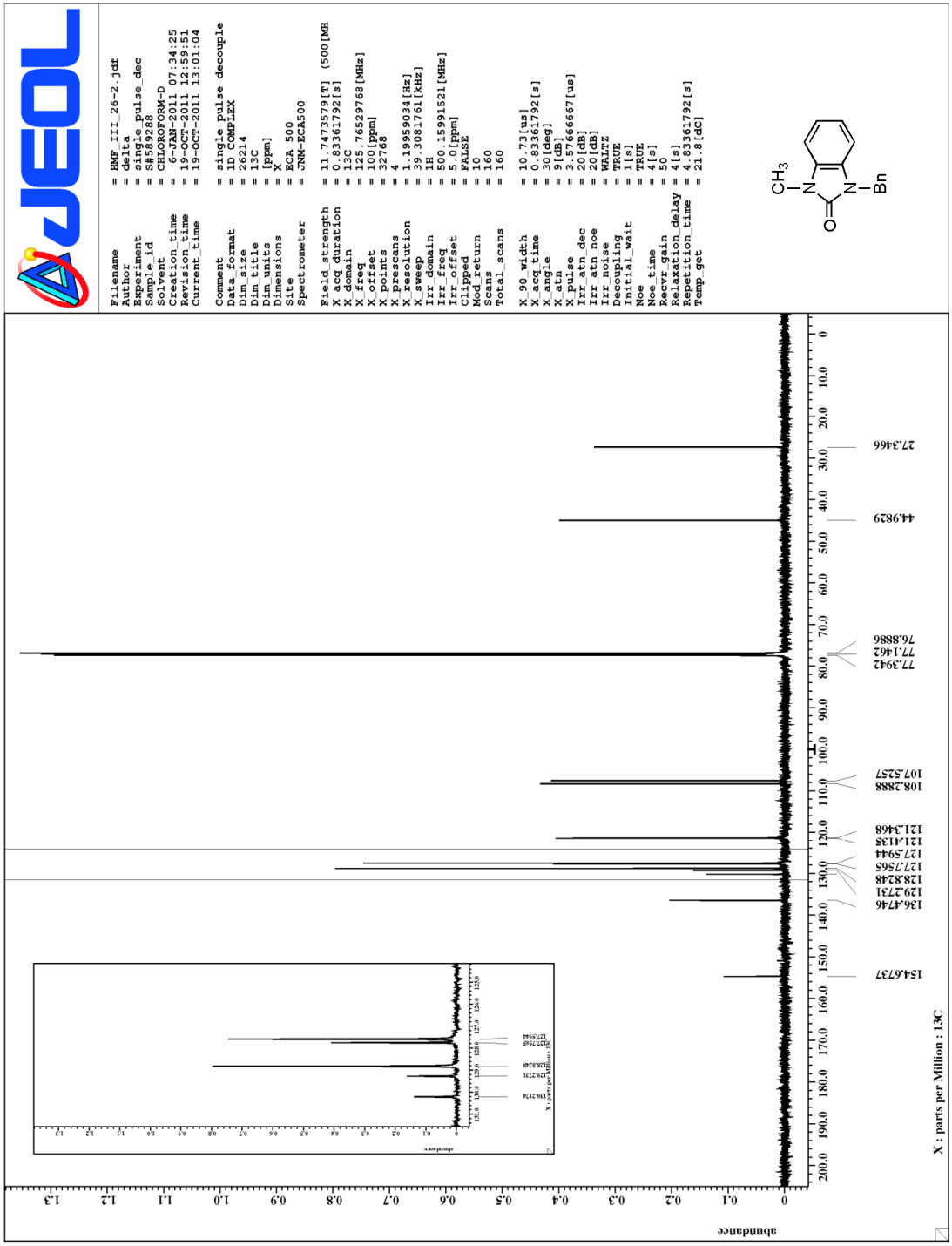
X_90_width        = 9.75[us]
X_acq_time        = 1.38412032[s]
X_angle           = 30[deg]
X_atn             = 8[dB]
X_pulse           = 3.25[us]
Irr_atn_dec       = 25[dB]
Irr_atn_noe       = 25[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 3[s]
Recvr_gain        = 50
Relaxation_delay  = 3[s]
Repetition_time   = 4.38412032[s]
Temp_get          = 21.4[dC]
```



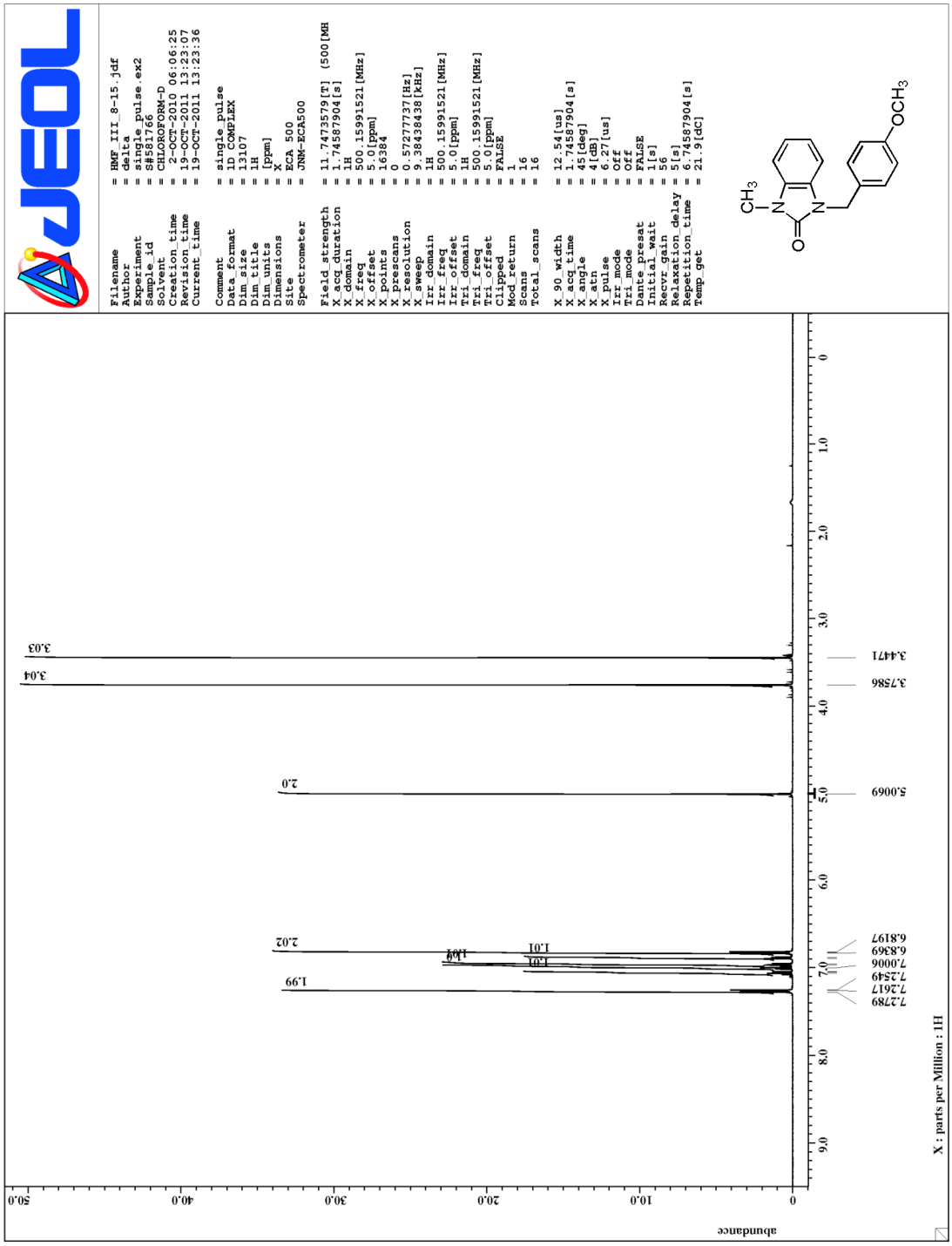
APPENDIX 11
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-3-methyl-1,3-dihydro-2*H*-benzimidazol-2-one (**82a**)

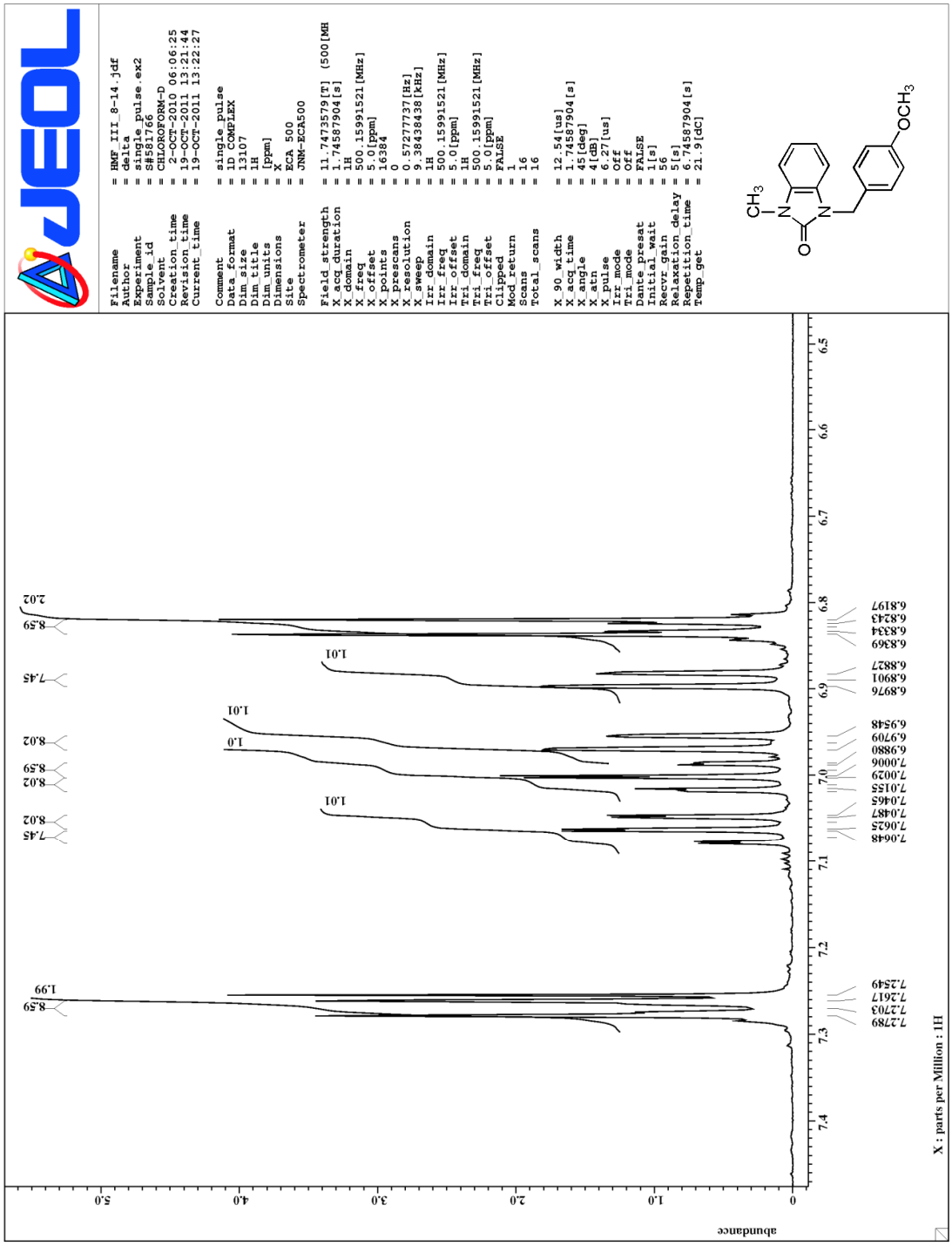

JEOL
Filename = RMF_III_26-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#587187
Solvent = CHLOROFORM-D
Creation_time = 6-JAN-2011 07:20:07
Revision_time = 19-OCT-2011 12:55:11
Current_time = 19-OCT-2011 12:55:52
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.3[dC]
CH3
N
N
Bn
O
abundance
3.99
3.01
2.0
1.02
1.0
1.01
1.0
7
3.4609
5.0767
7.3156
7.3030
7.0018
6.9858
6.8793
X : parts per Million : 1H

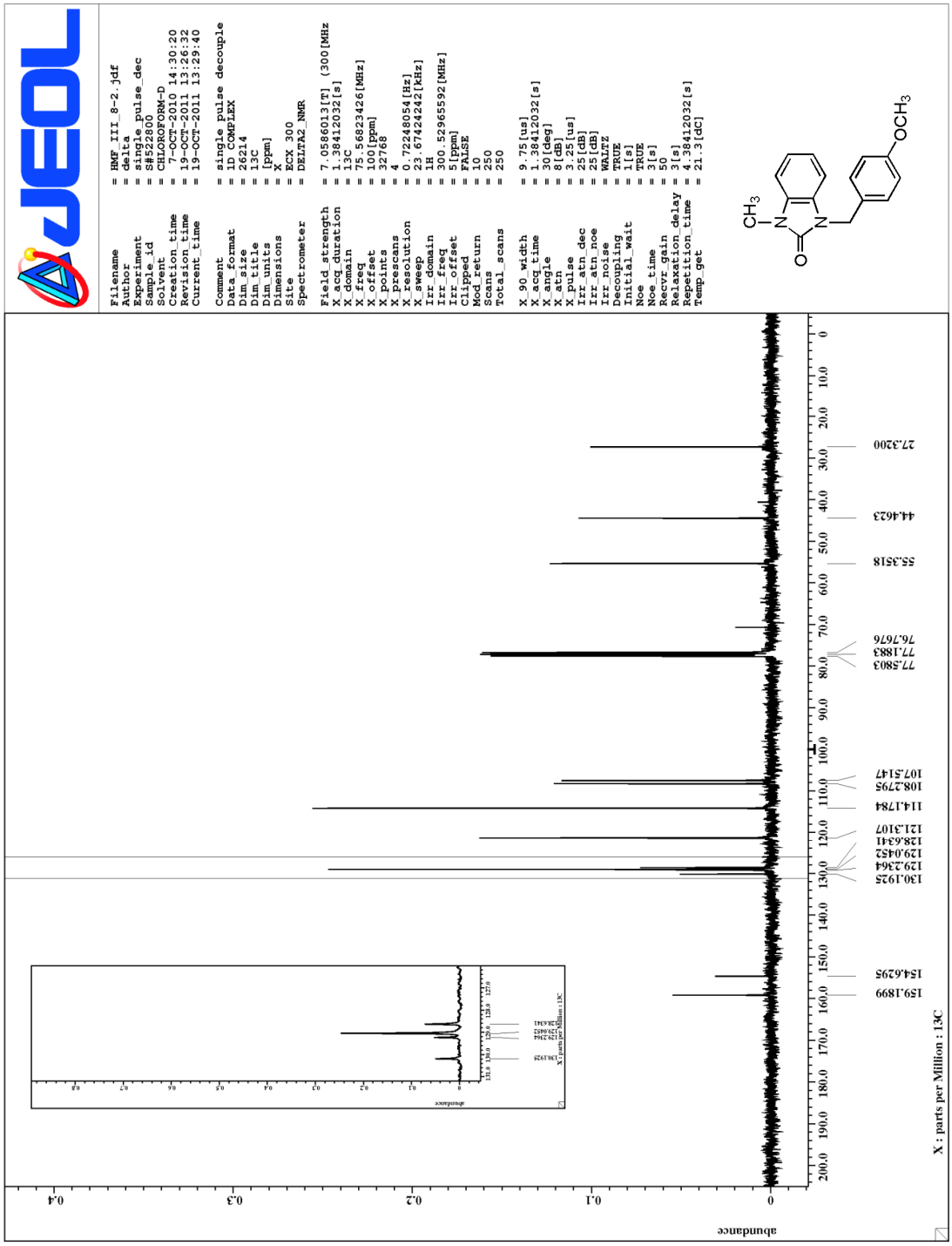

JEOL
Filename = RMF_III_26-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#587187
Solvent = CHLOROFORM-D
Creation_time = 6-JAN-2011 07:20:07
Revision_time = 19-OCT-2011 12:56:14
Current_time = 19-OCT-2011 12:56:34
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45.0[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.3[dC]
CH3
N
N
Bn
O
abundance
X : parts per Million : 1H
3.99
1.01
1.0
2.0
1.02
7.45
7.3156
7.3030
7.2984
7.2869
7.2629
7.2549
7.2492
7.0739
7.0602
7.0579
7.0190
7.0167
7.0041
7.0018
6.9858
6.9697
6.8793
6.8718
6.8644


Filename = BMF III_26-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#589288
Solvent = CHLOROFORM-D
Creation_time = 6-JAN-2011 07:34:25
Revision_time = 19-OCT-2011 12:59:51
Current_time = 19-OCT-2011 13:01:04
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817614[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 160
Total_scans = 160
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.8[dC]
CH3
N
N
Bn
O
27.3466
44.9829
76.8886
77.1462
77.3942
107.5257
108.2888
121.3468
121.4135
127.5944
127.7565
128.8248
129.2731
136.4746
154.6737
abundance
X : parts per Million : 13C

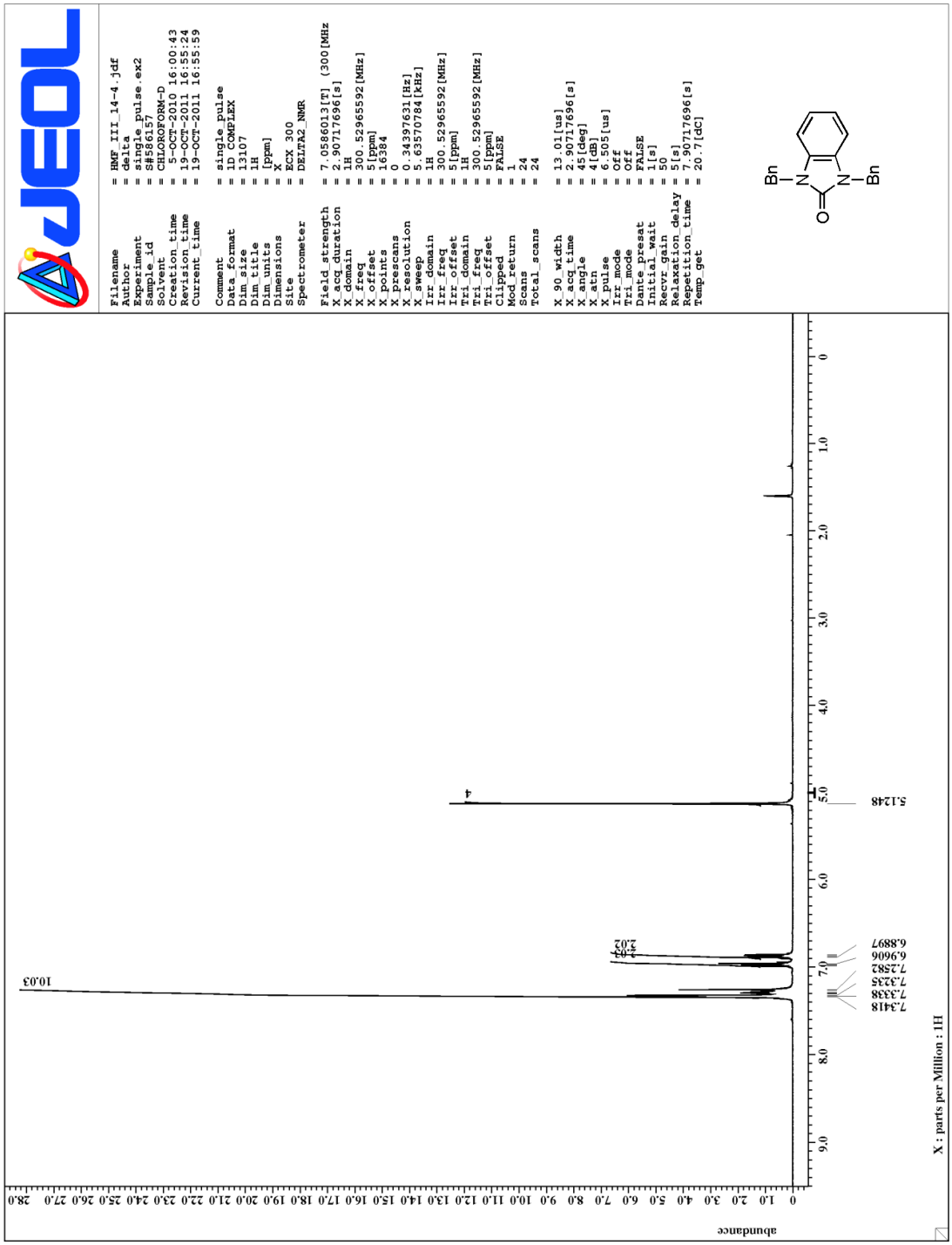
APPENDIX 12
^{1}H and ^{13}C NMR Spectra of
1-(4-methoxybenzyl)-3-methyl-1,3-dihydro-2*H*-benzimidazol-2-one (**82b**)

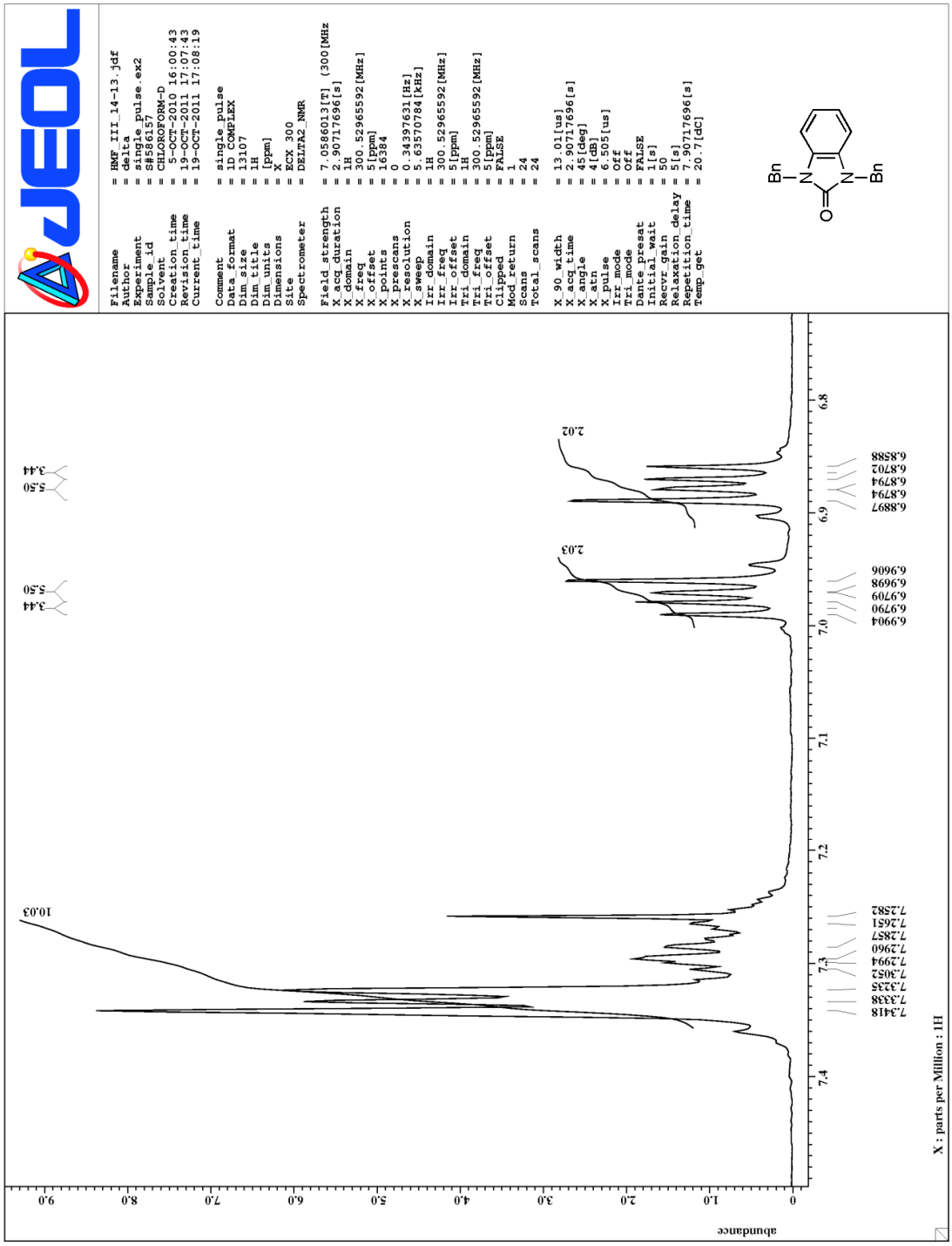

JEOL
Filename = HMF III_8-15.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#581766
Solvent = CHLOROFORM-D
Creation_time = 2-OCT-2010 06:06:25
Revision_time = 19-OCT-2011 13:23:07
Current_time = 19-OCT-2011 13:23:36
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 56
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
CH3
N
N
O
OCH3
abundance
X : parts per Million : 1H
3.03
3.04
2.0
2.02
1.99
1.01
1.01
1.09
3.4471
3.7586
5.0069
7.2789
7.2617
7.2549
7.0006
6.8369
6.8197

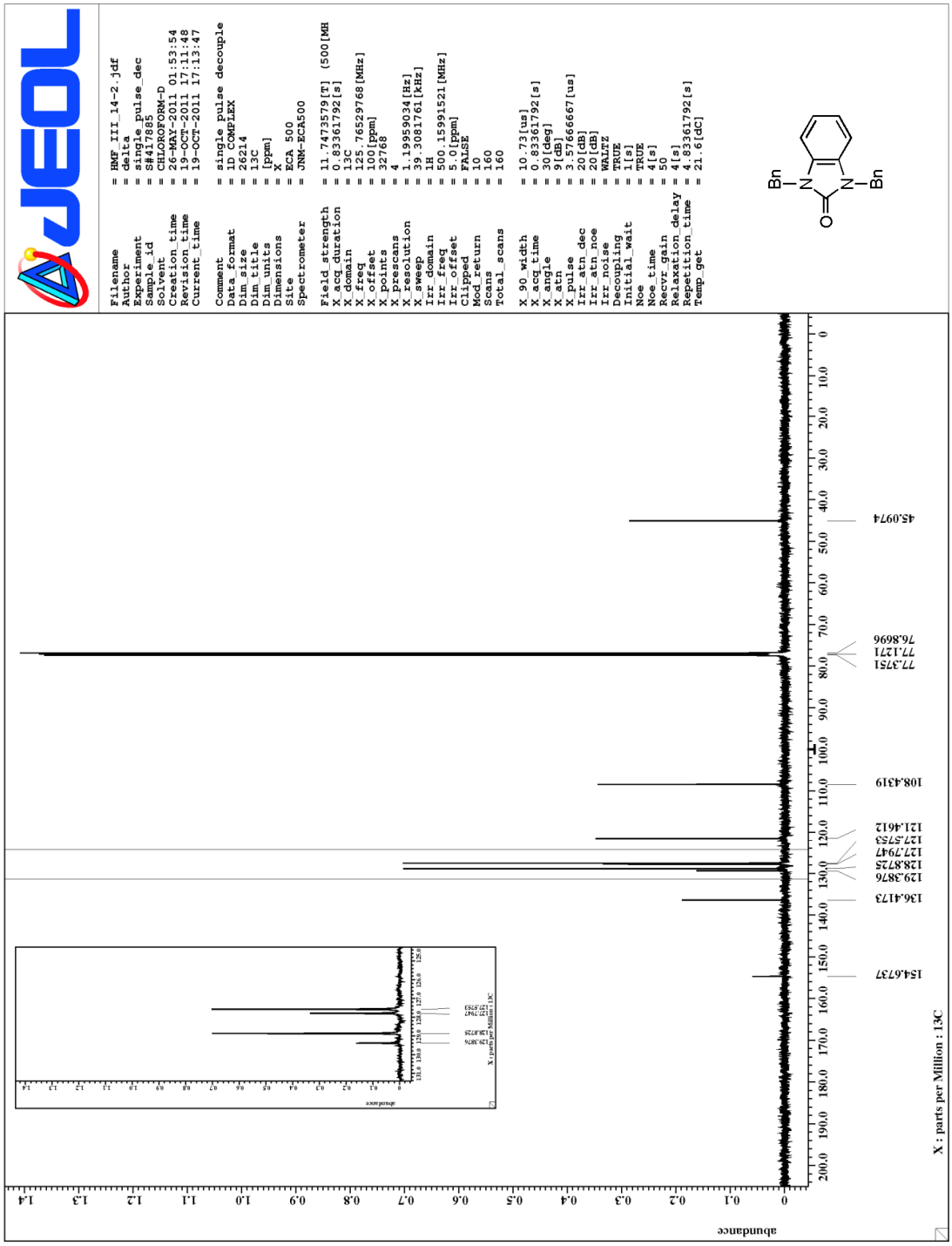

JEOL
Filename = HMF III_8-14.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#581766
Solvent = CHLOROFORM-D
Creation_time = 2-OCT-2010 06:06:25
Revision_time = 19-OCT-2011 13:21:44
Current_time = 19-OCT-2011 13:22:27
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 56
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
CH3
OCH3
abundance
X : parts per Million : 1H
8.59
1.99
7.45
8.02
8.02
8.59
8.02
1.0
1.01
1.01
8.59
2.02
1.01
7.2789
7.2703
7.2617
7.2549
7.0648
7.0625
7.0487
7.0465
7.0155
7.0029
7.0006
6.9880
6.9709
6.9548
6.8976
6.8901
6.8827
6.8369
6.8334
6.8243
6.8197


JEOL
Filename = HMF III_8-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#522800
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2010 14:30:20
Revision_time = 19-OCT-2011 13:26:32
Current_time = 19-OCT-2011 13:29:40
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 250
Total_scans = 250
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.38412032[s]
Temp_get = 21.3[dC]
CH3
OCH3
abundance
X : parts per Million : 13C
159.1899
154.6295
130.1925
129.2364
129.0452
128.6341
121.3107
114.1784
108.2795
107.5147
77.5803
77.1883
76.7676
55.3518
44.4623
27.3200

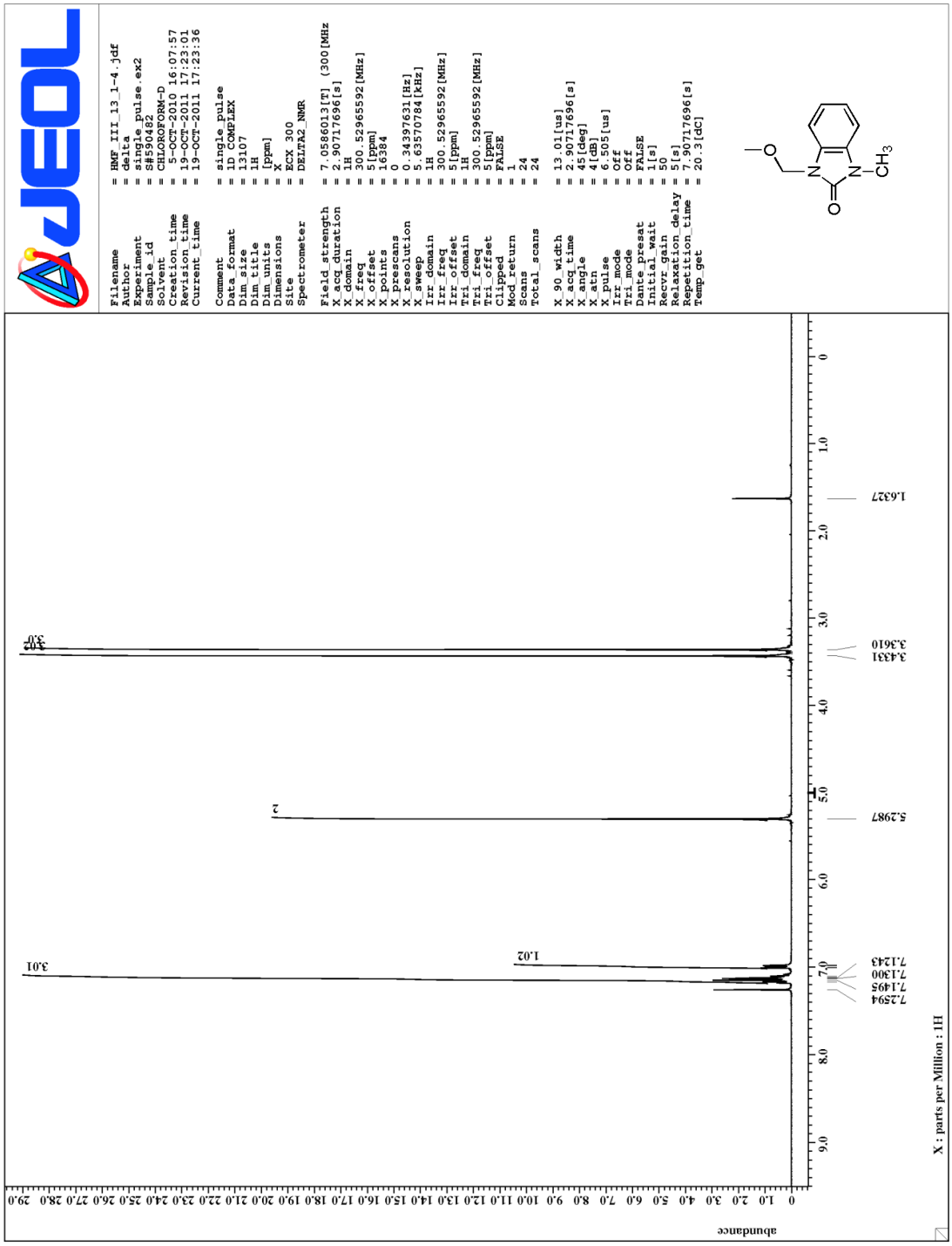
APPENDIX 13
^{1}H and ^{13}C NMR Spectra of
1,3-Dibenzyl-1,3-dihydro-2*H*-benzimidazol-2-one (**82c**)


Filename = RMF_III_14-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#586157
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2010 16:00:43
Revision_time = 19-OCT-2011 16:55:24
Current_time = 19-OCT-2011 16:55:59
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.7[dC]
Bn
N
N
Bn
O
abundance
10.03
2.02
2.07
4
5.1248
7.3418
7.3338
7.3335
7.2582
6.9606
6.8897
X : parts per Million : 1H


JEOL
Filename = HMF III 14-13.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#586157
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2010 16:00:43
Revision_time = 19-OCT-2011 17:07:43
Current_time = 19-OCT-2011 17:08:19
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.7[dC]
Bn
N
N
Bn
O
abundance
X : parts per Million : 1H
10.03
2.02
2.03
3.44
5.50
3.44
5.50
7.3418
7.3338
7.3235
7.3052
7.2994
7.2960
7.2857
7.2651
7.2582
6.9904
6.9790
6.9709
6.9698
6.9606
6.8897
6.8794
6.8794
6.8702
6.8588


Filename = BMF_III_14-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#417885
Solvent = CHLOROFORM-D
Creation_time = 26-MAY-2011 01:53:54
Revision_time = 19-OCT-2011 17:11:48
Current_time = 19-OCT-2011 17:13:47
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 160
Total_scans = 160
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.6[dC]
Bn
N
N
Bn
O
154.6737
136.4173
129.3876
128.8725
127.7947
127.5753
121.4612
108.4319
77.3751
77.1271
76.8696
45.0974
abundance
X : parts per Million : 13C

APPENDIX 14
^{1}H and ^{13}C NMR Spectra of
3-Methoxymethyl-1-methyl-1,3-dihydro-2*H*-benzimidazol-2-one (**82d**)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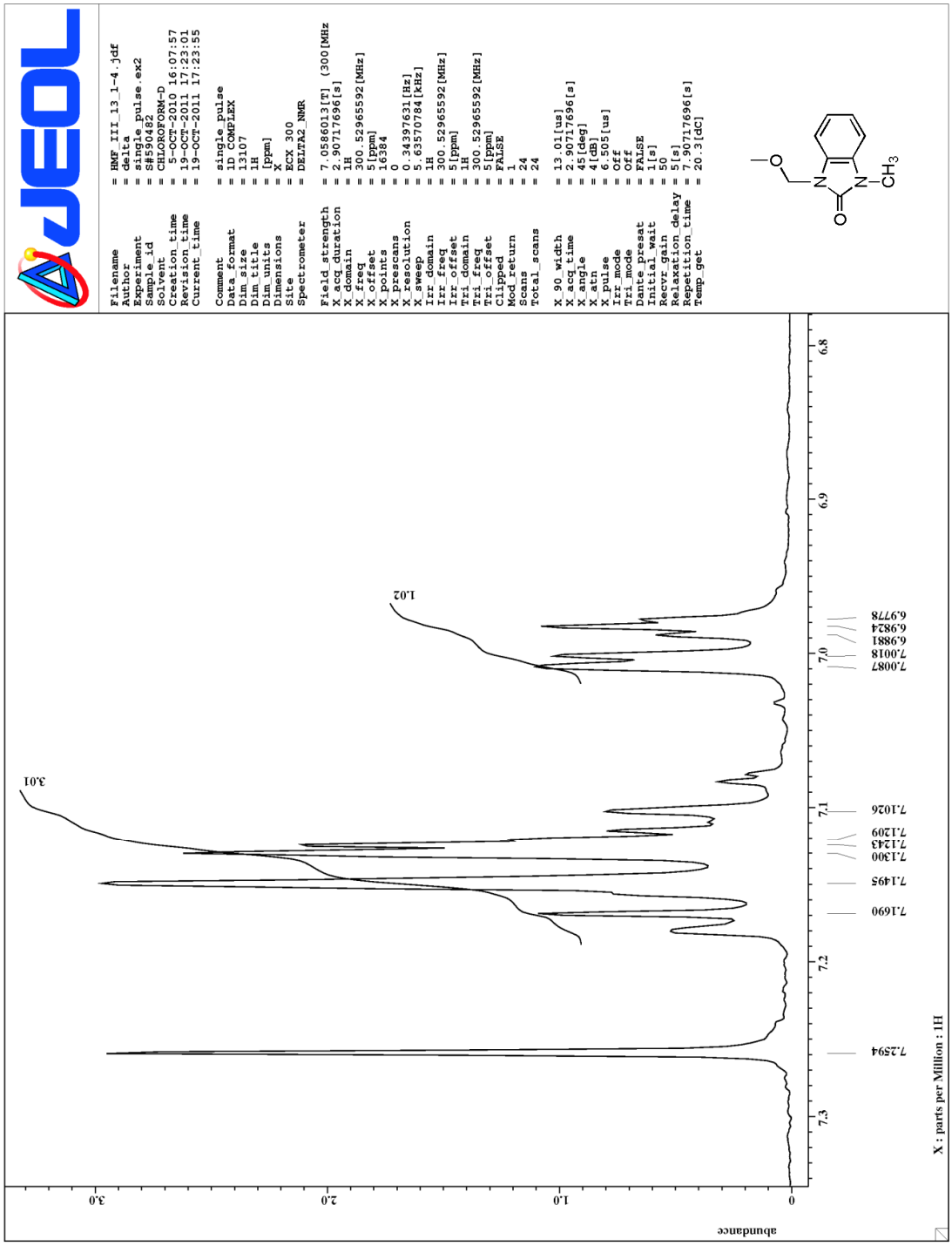
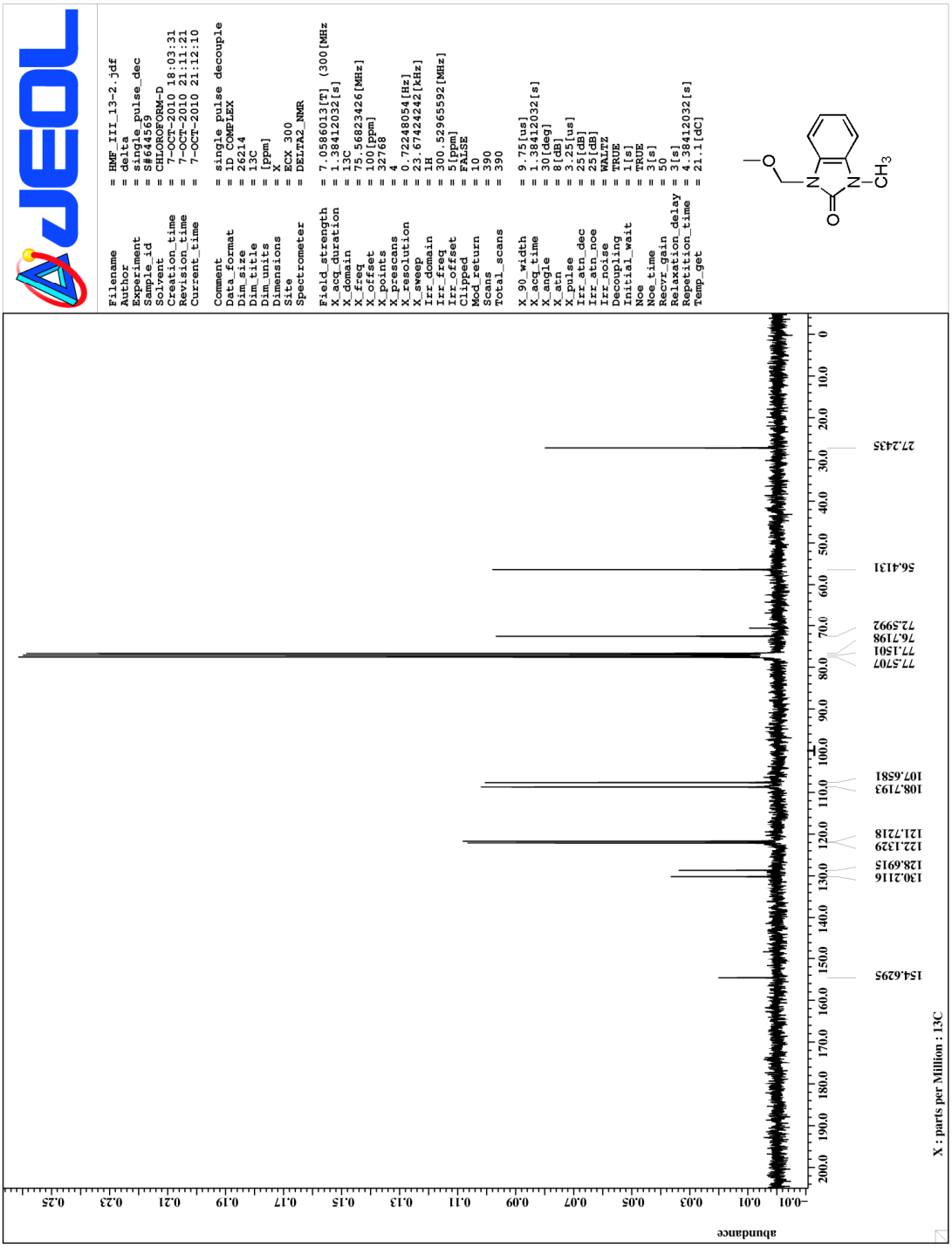
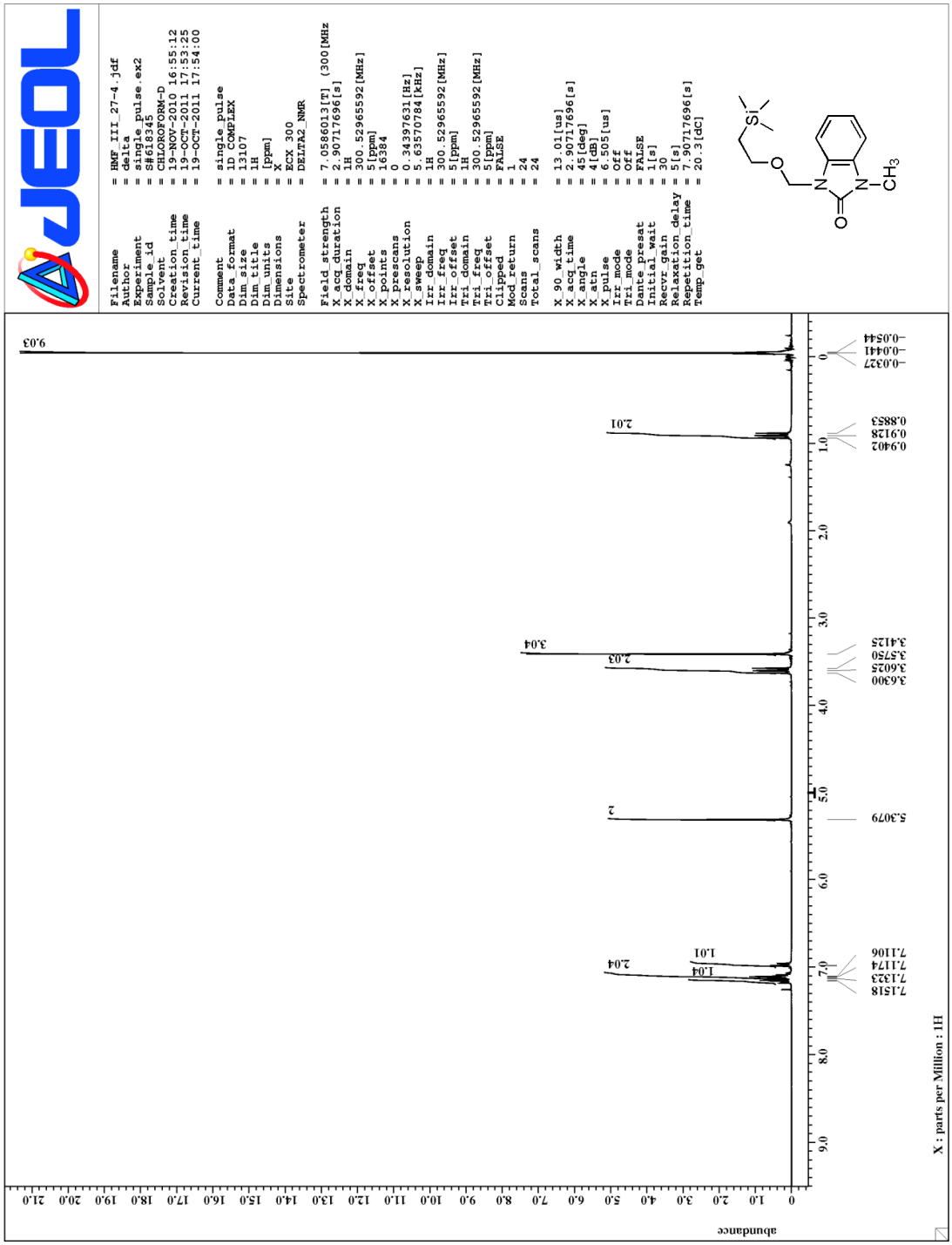
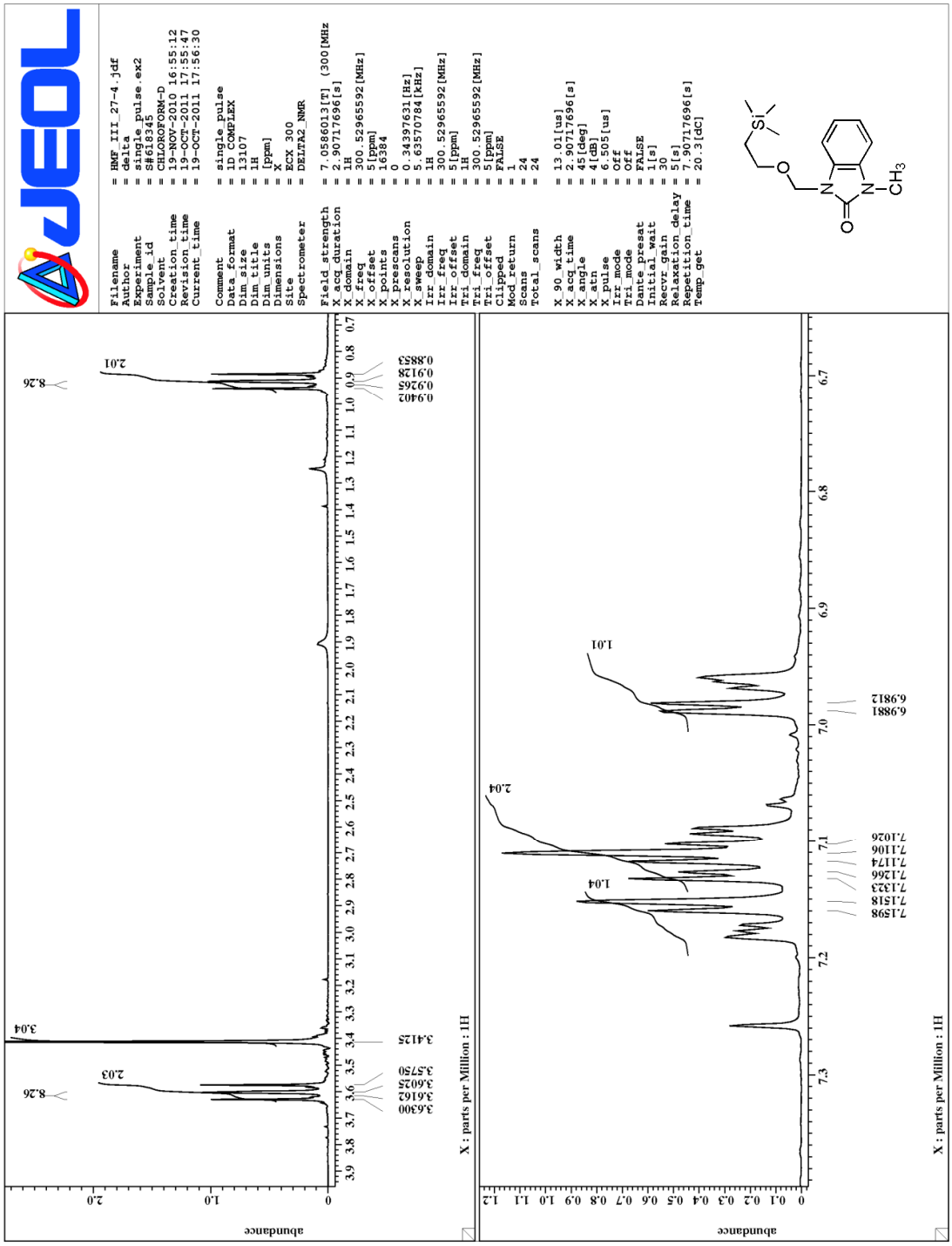
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#590482
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2010 16:07:57
Revision_time = 19-OCT-2011 17:23:01
Current_time = 19-OCT-2011 17:23:36
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DEIMPA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.3[dC]
3.01
1.02
2
3.02
1.6327
3.4331
3.3610
5.2987
7.2594
7.1495
7.1300
7.1243
abundance
X : parts per Million : 1H

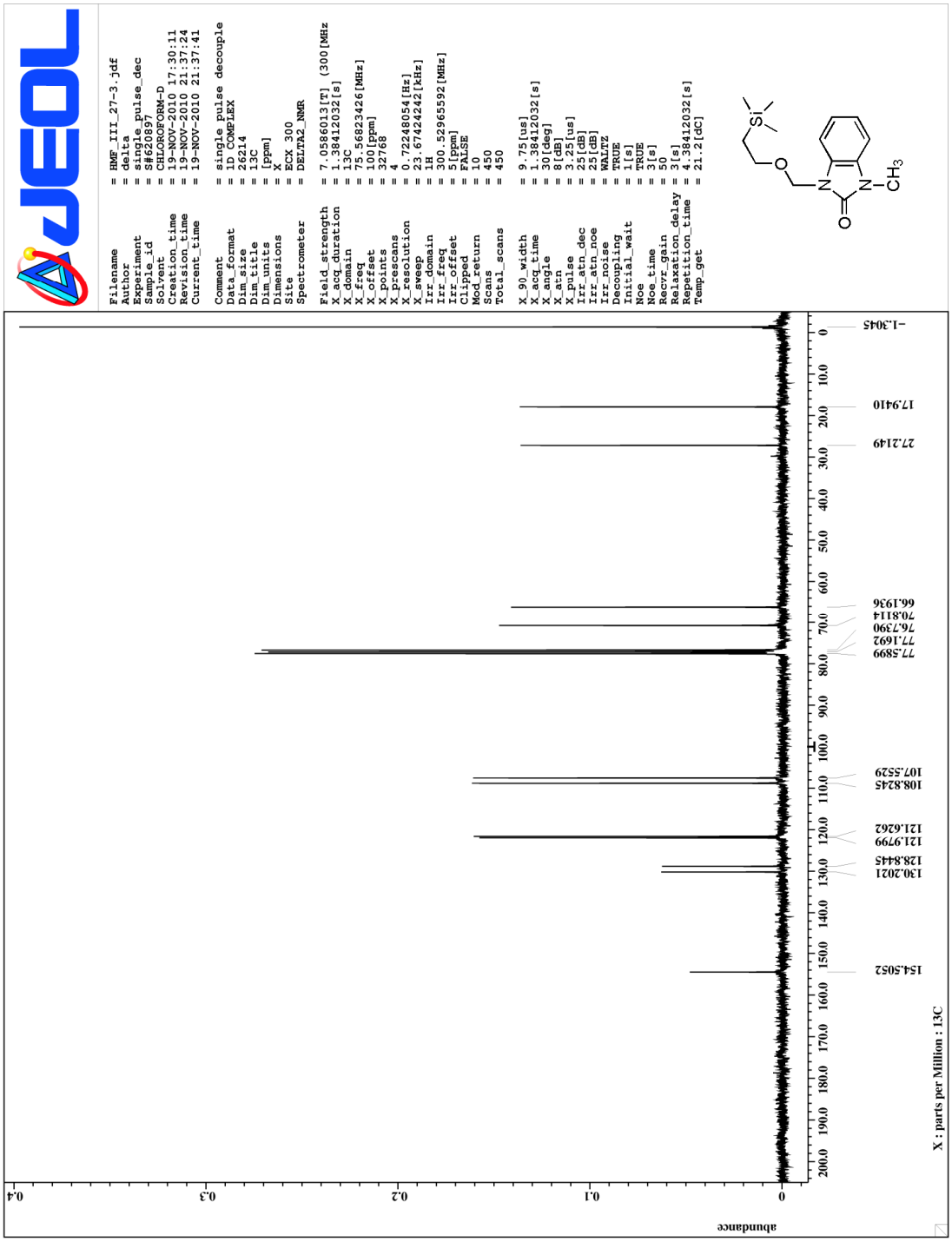

JEOL
Filename = HMF III 13 1-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#590482
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2010 16:07:57
Revision_time = 19-OCT-2011 17:23:01
Current_time = 19-OCT-2011 17:23:55
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.3[dC]
3.01
1.02
7.2594
7.1690
7.1495
7.1300
7.1243
7.1209
7.1026
7.0087
7.0018
6.9881
6.9824
6.9778
abundance
X : parts per Million : 1H


JEOL
Filename = HMF_III_13-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#644569
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2010 18:03:31
Revision_time = 7-OCT-2010 21:11:21
Current_time = 7-OCT-2010 21:12:10
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEFAULT_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 390
Total_scans = 390
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Nce = TRUE
Nce_time = 3[s]
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.38412032[s]
Temp_get = 21.1[dC]
O
N
N
CH3
abundance
X : parts per Million : 13C
27.2435
56.4131
72.5992
76.7198
77.1501
77.5707
107.6581
108.7193
121.7218
122.1329
128.6915
130.2116
154.6295

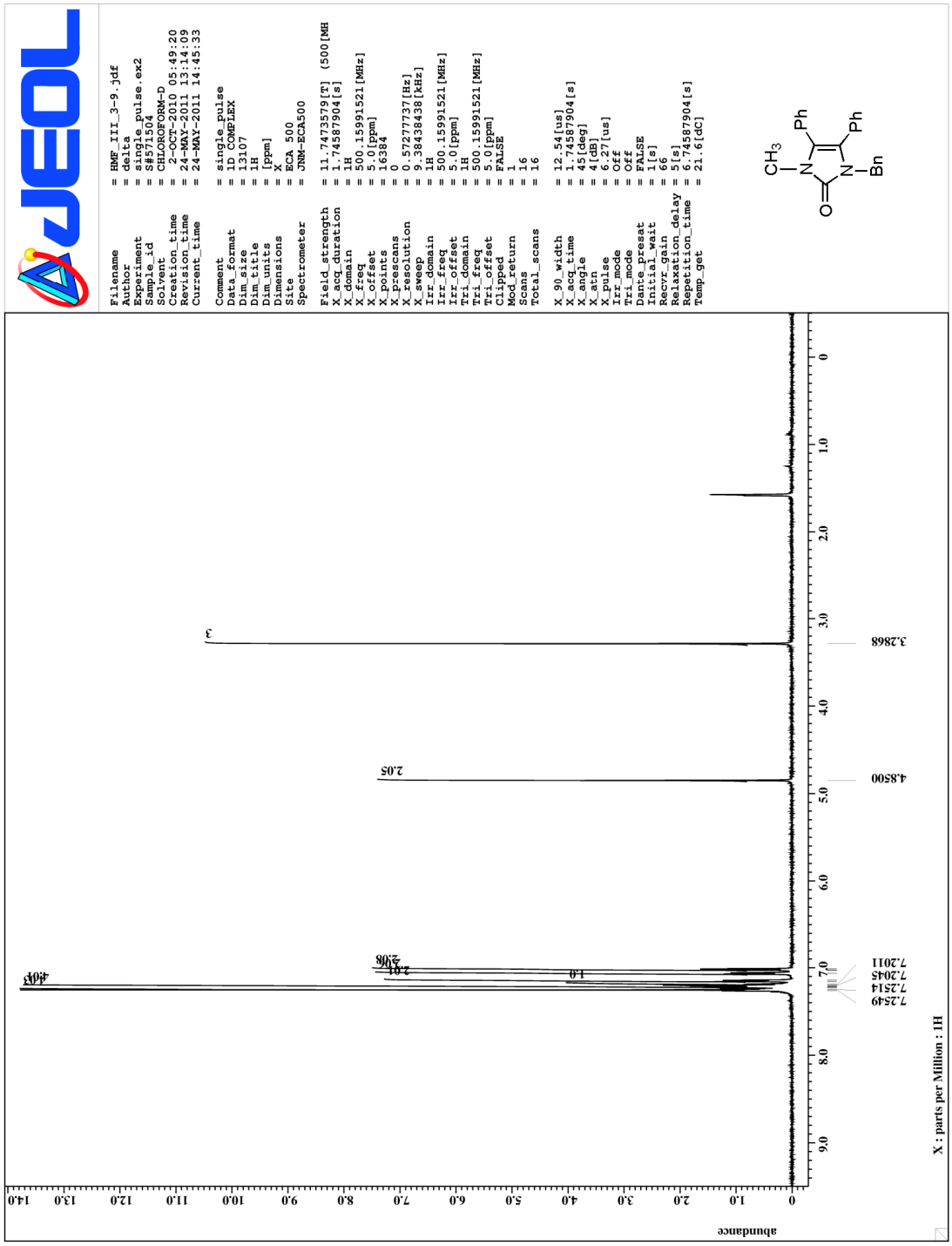
APPENDIX 15
^{1}H and ^{13}C NMR Spectra of
1-Methyl-3-trimethylsilylethoxymethyl-1,3-dihydro-2*H*-benzimidazol-2-one (**82e**)


JEOL
Filename = HMF III_27-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#618345
Solvent = CHLOROFORM-D
Creation_time = 19-NOV-2010 16:55:12
Revision_time = 19-OCT-2011 17:53:25
Current_time = 19-OCT-2011 17:54:00
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DEUTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 30
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.3[dC]
CH3
abundance
X : parts per Million : 1H
9.03
2.01
3.04
2.03
2
2.04
1.04
1.01
0.9402
0.9128
0.8853
3.6300
3.6025
3.5750
3.4125
5.3079
7.1518
7.1323
7.1174
7.1106
-0.0327
-0.0441
-0.0544


JEOL
Filename = RMF III_27-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#618345
Solvent = CHLOROFORM-D
Creation_time = 19-NOV-2010 16:55:12
Revision_time = 19-OCT-2011 17:55:47
Current_time = 19-OCT-2011 17:56:30
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 30
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.3[dC]
abundance
X : parts per Million : 1H
8.26
2.01
0.9402
0.9265
0.9128
0.8853
3.6300
3.6162
3.6025
3.5750
3.4125
2.03
8.26
3.04
abundance
X : parts per Million : 1H
6.9881
6.9812
7.1598
7.1518
7.1323
7.1266
7.1174
7.1106
7.1026
1.01
2.04
1.04
2.04
Si
O
N
N
CH3
O


JEOL
Filename = RMF_III_27-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#620897
Solvent = CHLOROFORM-D
Creation_time = 19-NOV-2010 17:30:11
Revision_time = 19-NOV-2010 21:37:24
Current_time = 19-NOV-2010 21:37:41
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 450
Total_scans = 450
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Nce_time = TRUE
Recyr_time = 3[s]
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.38412032[s]
Temp_get = 21.2[dC]
Si
O
N
N
CH3
O
abundance
0
0.1
0.2
0.3
0.4
X : parts per Million : 13C
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-1.3045
17.9410
27.2149
66.1936
70.8114
76.7390
77.1692
77.5899
107.5529
108.8245
121.6262
121.9799
128.8445
130.2021
154.5052

APPENDIX 16
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-3-methyl-4,5-diphenyl-1,3-dihydro-2*H*-imidazol-2-one (**82f**)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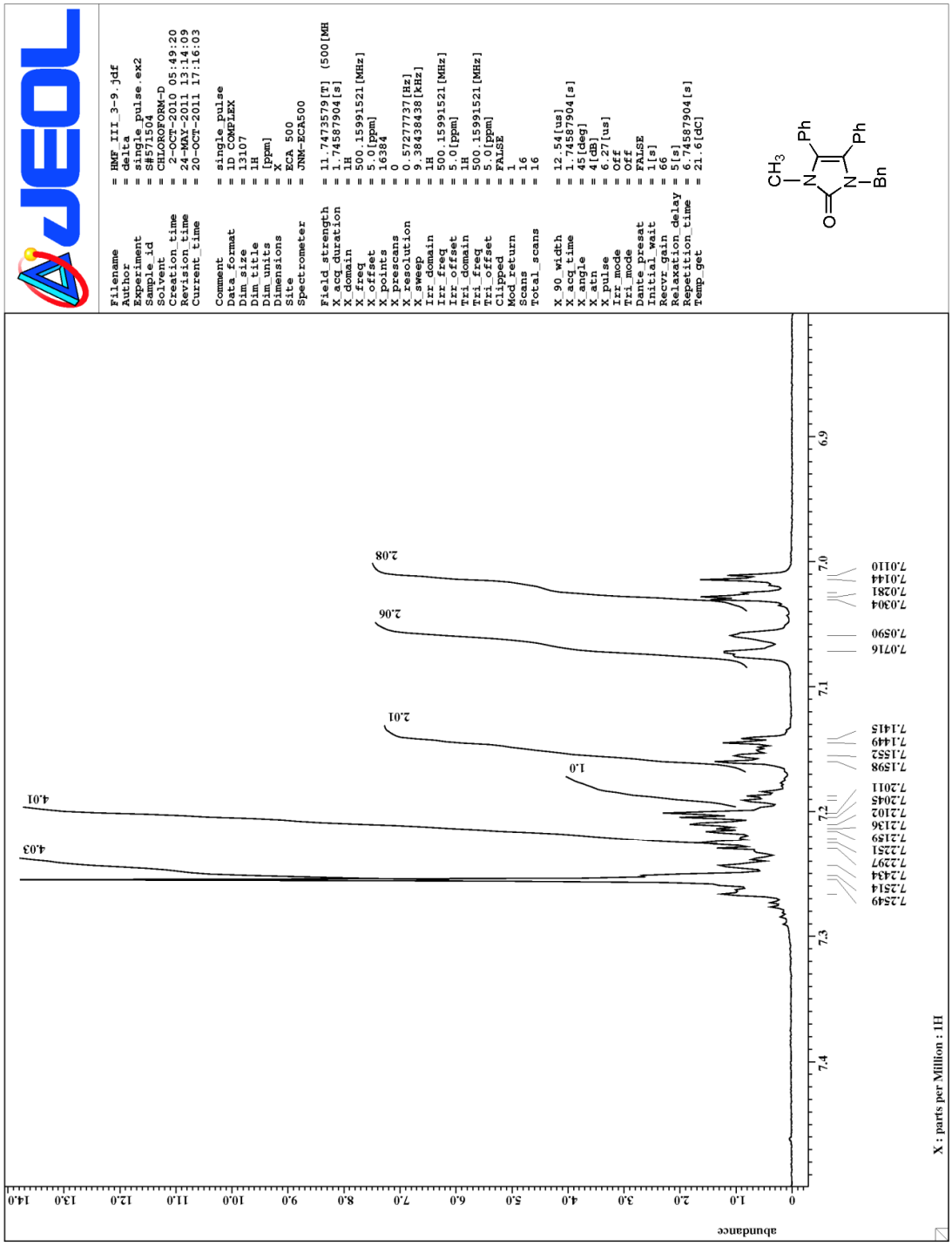
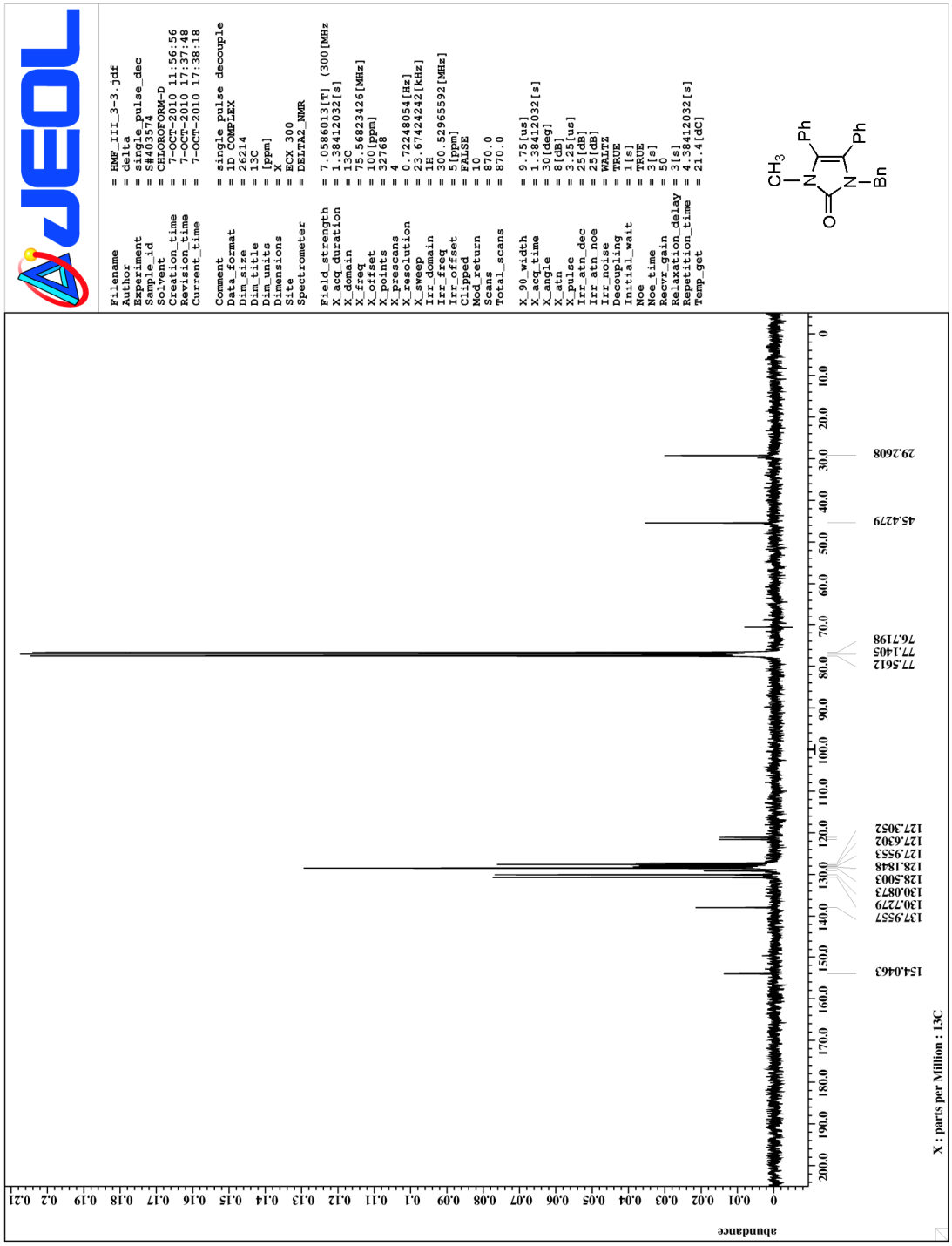
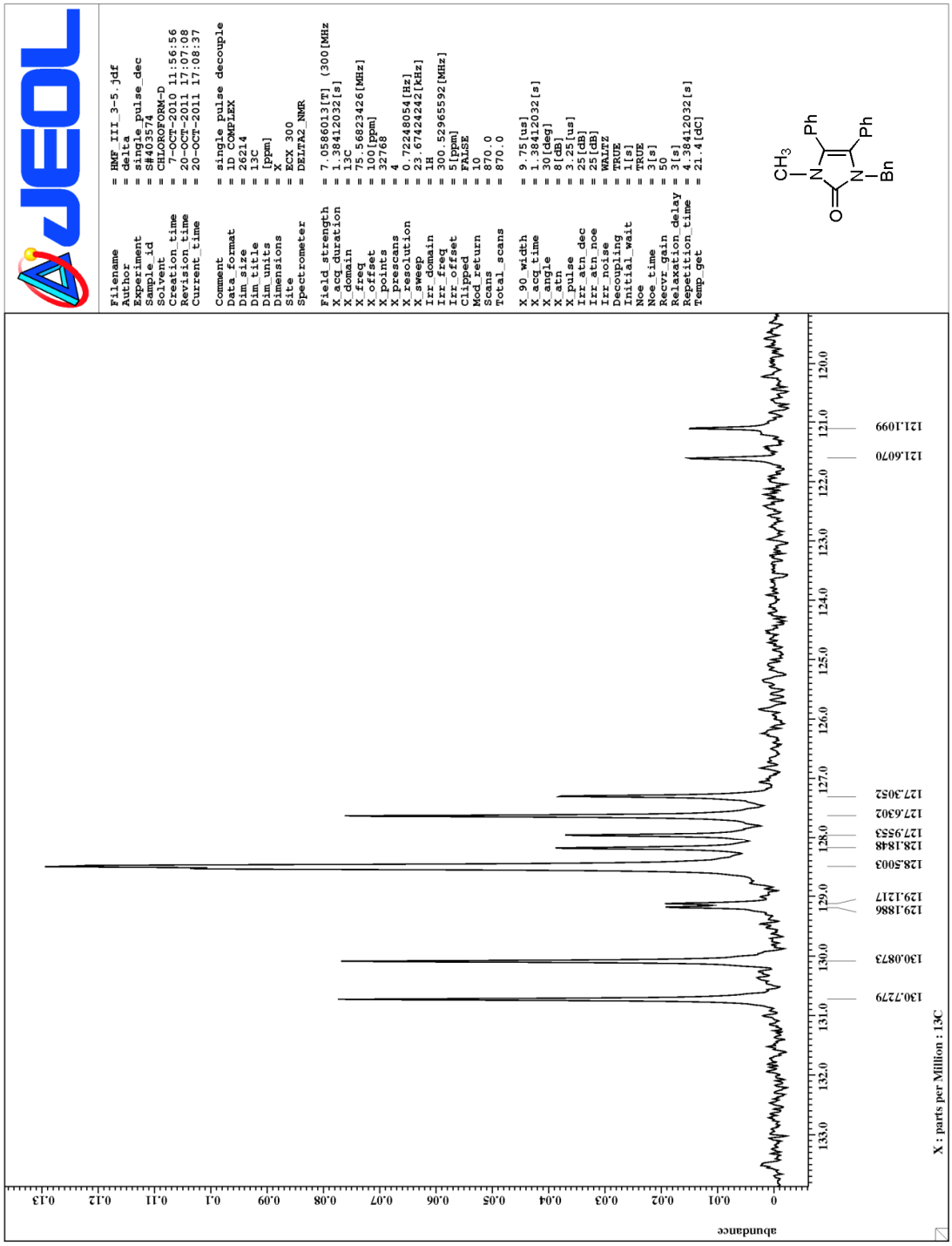
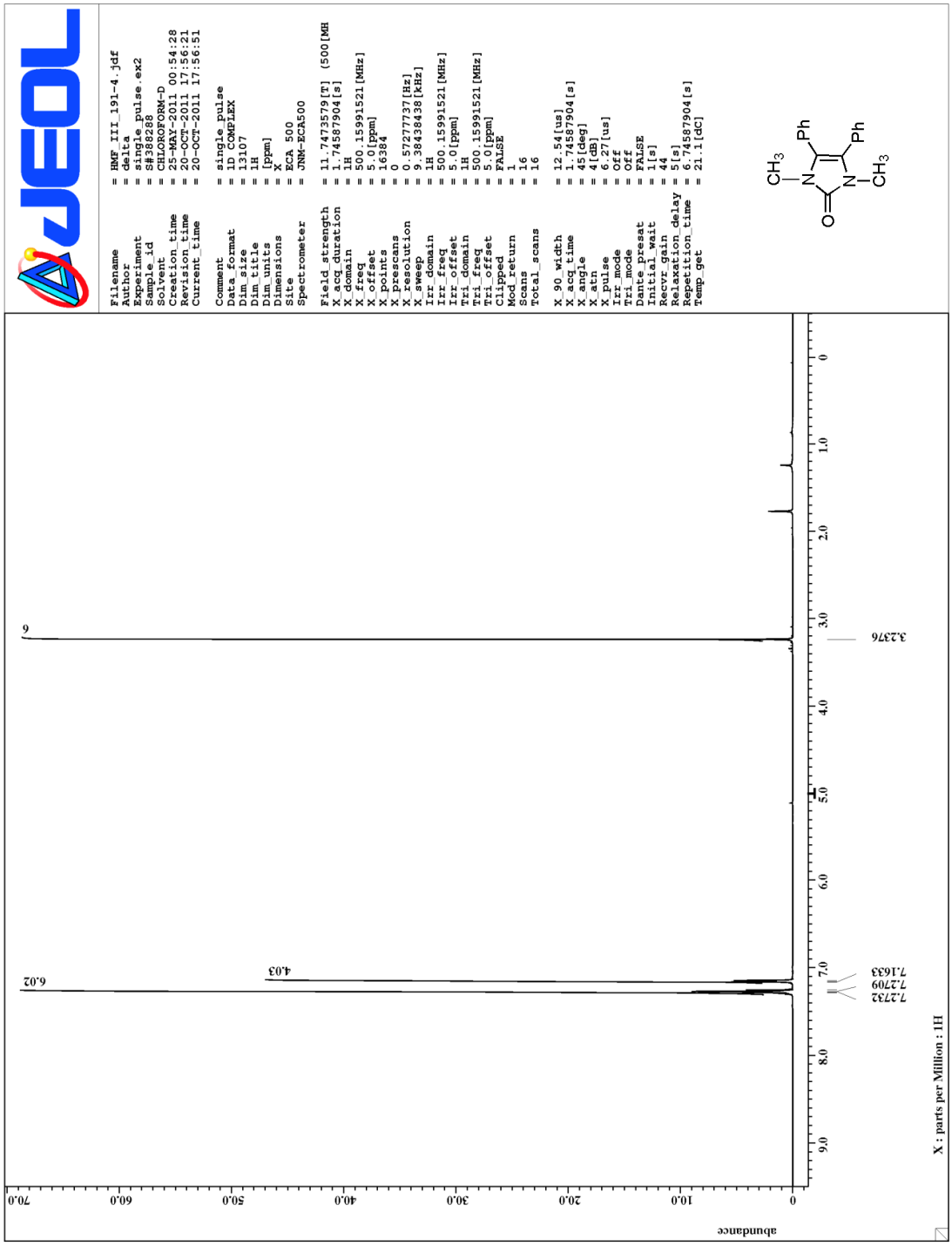
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#571504
Solvent = CHLOROFORM-D
Creation_time = 2-OCT-2010 05:49:20
Revision_time = 24-MAY-2011 13:14:09
Current_time = 24-MAY-2011 14:45:33
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recyr_gain = 66
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.6[dC]
CH3
Ph
Ph
Bn
N
N
O
abundance
X : parts per Million : 1H
3.2868
4.8500
7.2549
7.2514
7.2045
7.2011
3
2.05
1.0
2.08
4.07

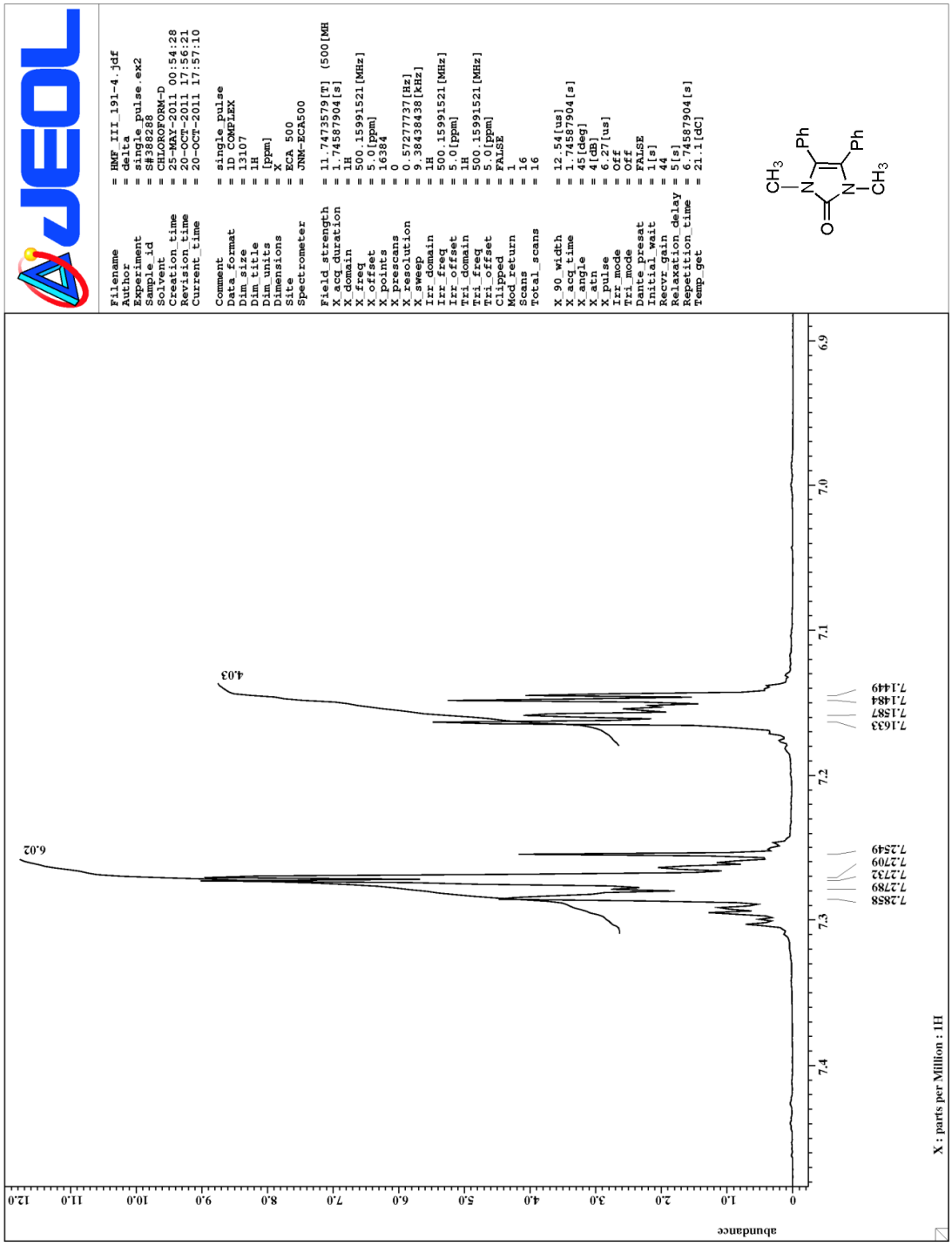

JEOL
Filename = RMF III_3-9.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#571504
Solvent = CHLOROFORM-D
Creation_time = 2-OCT-2010 05:49:20
Revision_time = 24-MAY-2011 13:14:09
Current_time = 20-OCT-2011 17:16:03
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 66
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.6[dC]
CH3
Ph
Ph
Bn
N
N
O
4.03
4.01
2.08
2.06
2.01
1.0
abundance
X : parts per Million : 1H
7.2549
7.2514
7.2434
7.2297
7.2251
7.2159
7.2136
7.2102
7.2045
7.2011
7.1598
7.1552
7.1449
7.1415
7.0716
7.0590
7.0304
7.0281
7.0144
7.0110

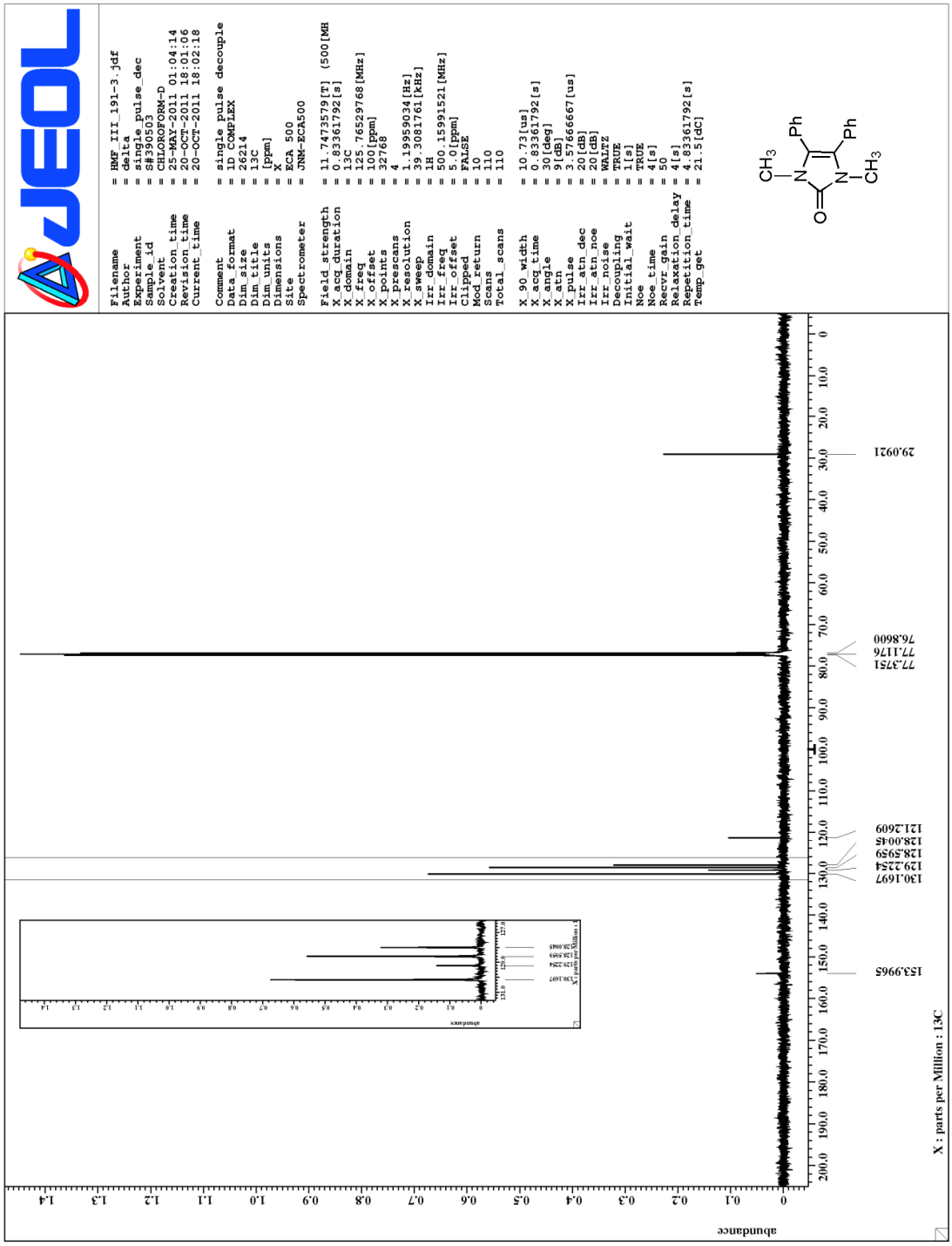

JEOL
Filename = RMF_III_3-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#403574
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2010 11:56:56
Revision_time = 7-OCT-2010 17:37:48
Current_time = 7-OCT-2010 17:38:18
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
X_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 870.0
Total_scans = 870.0
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 2.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Nce = TRUE
Nce_time = 3[s]
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.38412032[s]
Temp_get = 21.4[dC]
CH3
N
Ph
Ph
N
Bn
O
abundance
X : parts per Million : 13C
154.0463
137.9557
130.7279
130.0873
128.5003
128.1848
127.9553
127.6302
127.3052
77.5612
77.1405
76.7198
45.4279
29.2608


JEOL
Filename = BMF_III_3-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#403574
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2010 11:56:56
Revision_time = 20-OCT-2011 17:07:08
Current_time = 20-OCT-2011 17:08:37
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 870.0
Total_scans = 870.0
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 4.38412032[s]
Temp_get = 21.4[dC]
CH3
N
Ph
Ph
N
Bn
O
abundance
X : parts per Million : 13C
121.1099
121.6070
127.3052
127.6302
127.9553
128.1848
128.5003
129.1217
129.1886
130.0873
130.7279

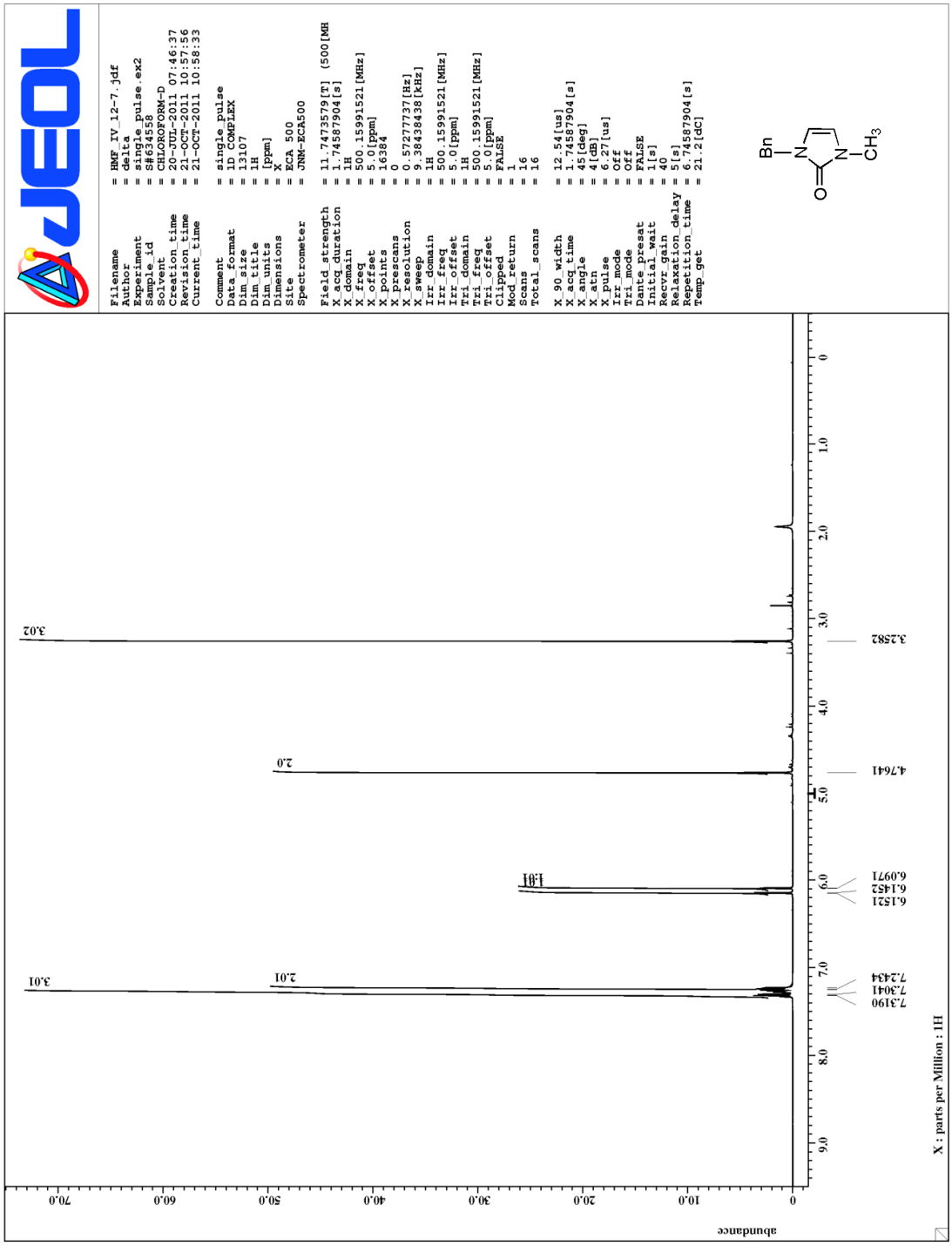
APPENDIX 17
^{1}H and ^{13}C NMR Spectra of
1,3-Dimethyl-4,5-diphenyl-1,3-dihydro-2*H*-imidazol-2-one (**82g**)

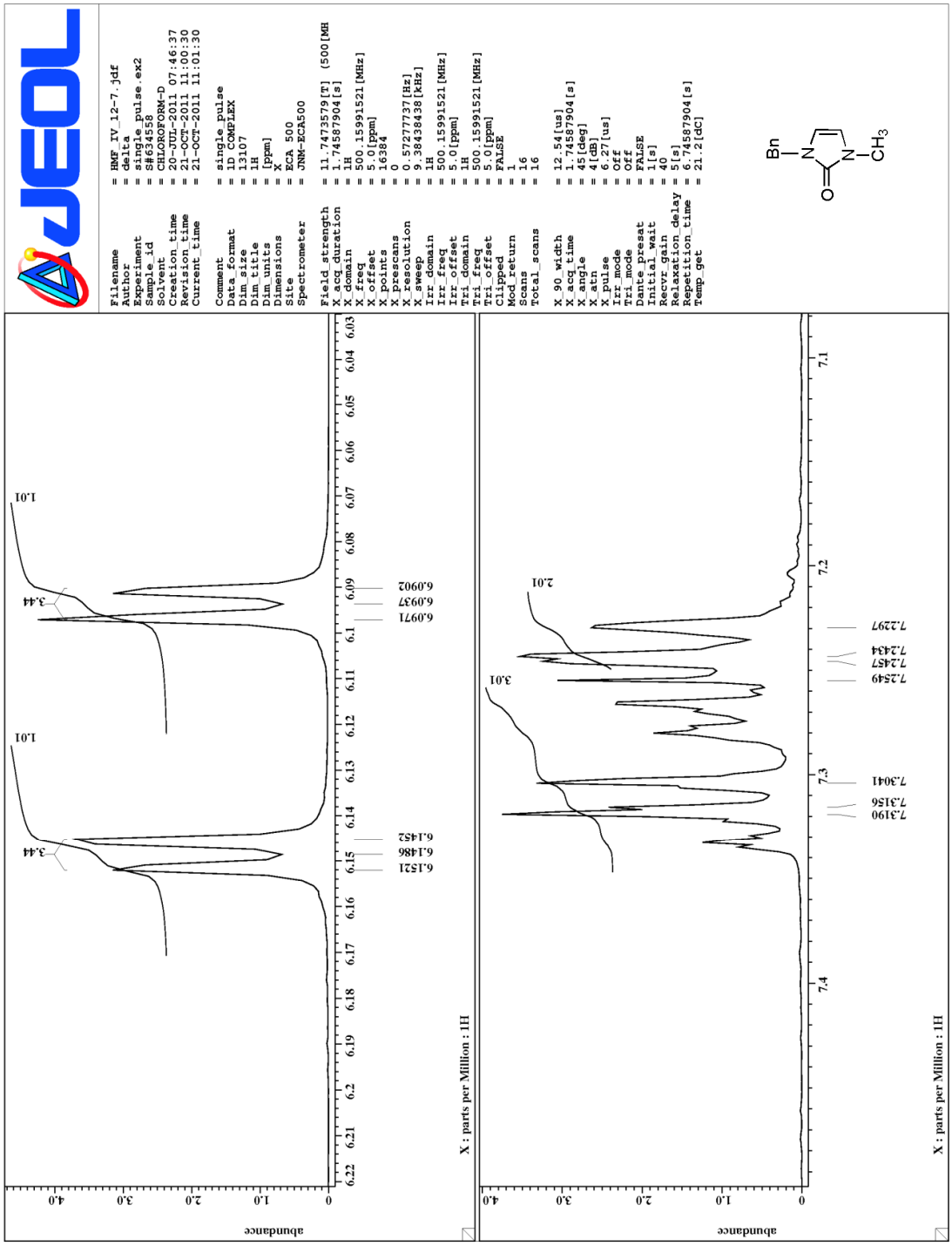

JEOL
Filename = HMF_III_191-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#388288
Solvent = CHLOROFORM-D
Creation_time = 25-MAY-2011 00:54:28
Revision_time = 20-OCT-2011 17:56:21
Current_time = 20-OCT-2011 17:56:51
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.1[dC]
CH3
Ph
Ph
CH3
N
N
O
6
6.02
4.03
3.2376
7.2732
7.2709
7.1633
abundance
X : parts per Million : 1H

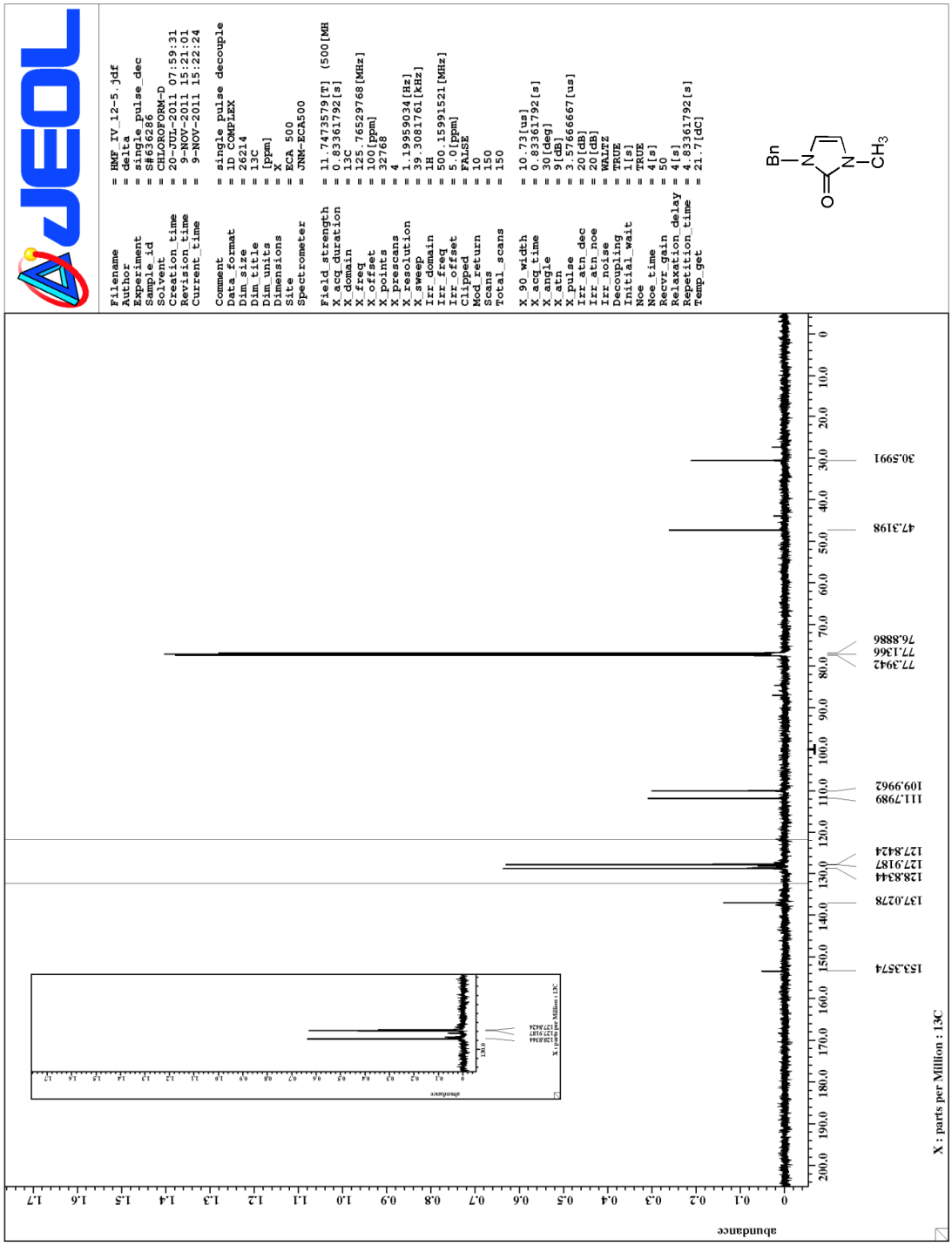

JEOL
Filename = RMF_III_191-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#388288
Solvent = CHLOROFORM-D
Creation_time = 25-MAY-2011 00:54:28
Revision_time = 20-OCT-2011 17:56:21
Current_time = 20-OCT-2011 17:57:10
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.1[dC]
Ph
Ph
CH3
CH3
N
N
O
6.02
4.03
7.2858
7.2789
7.2732
7.2709
7.2549
7.1633
7.1587
7.1484
7.1449
abundance
X : parts per Million : 1H


Filename = HMF_III_191-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#390503
Solvent = CHLOROFORM-D
Creation_time = 25-MAY-2011 01:04:14
Revision_time = 20-OCT-2011 18:01:06
Current_time = 20-OCT-2011 18:02:18
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 110
Total_scans = 110
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.5[dC]
CH3
N
Ph
O
abundance
X : parts per Million : 13C
153.9965
130.1697
129.2254
128.5959
128.0045
121.2609
77.3751
77.1176
76.8600
29.0921

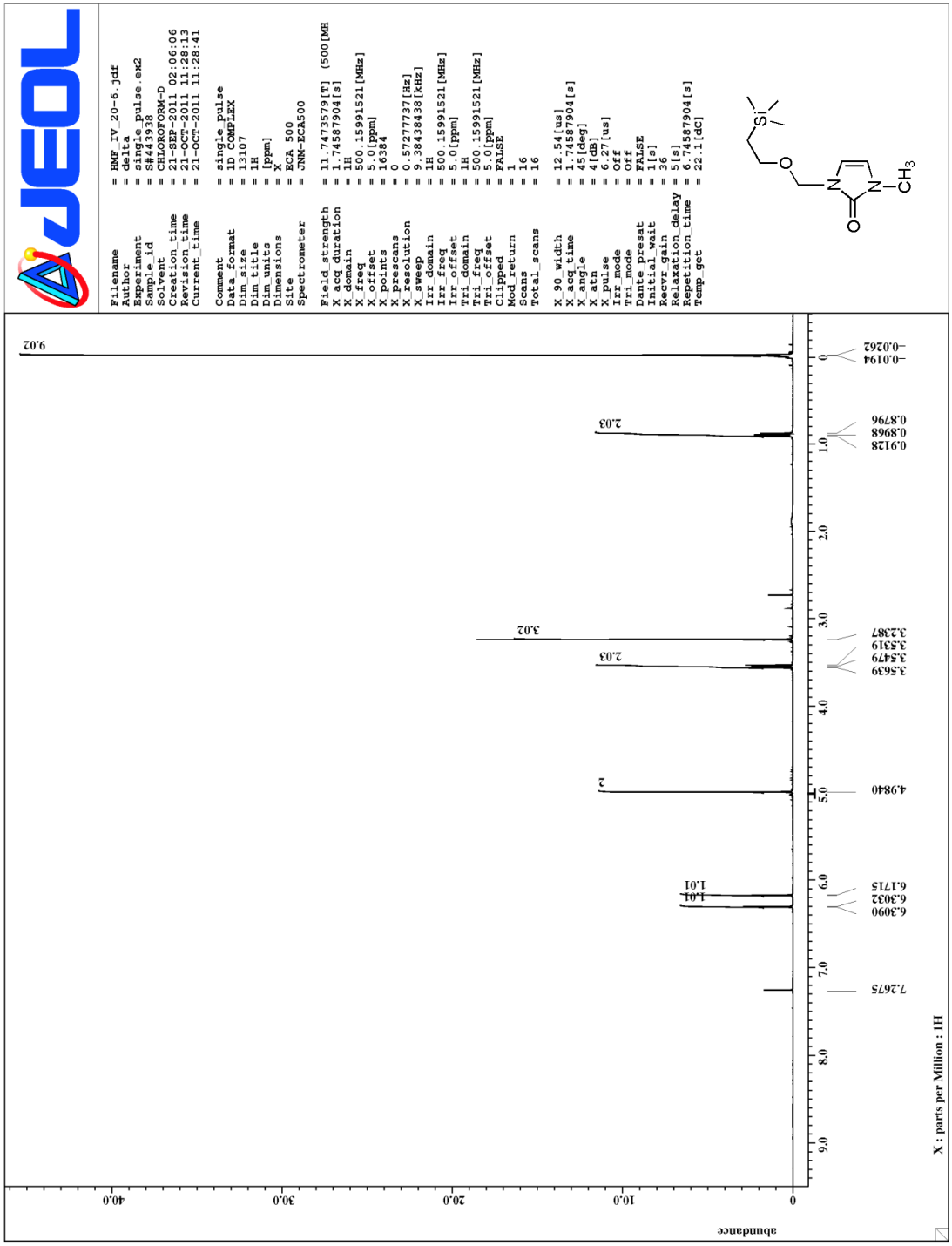
APPENDIX 18
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-1-methyl-1,3-dihydro-2*H*-imidazol-2-one (**82h**)

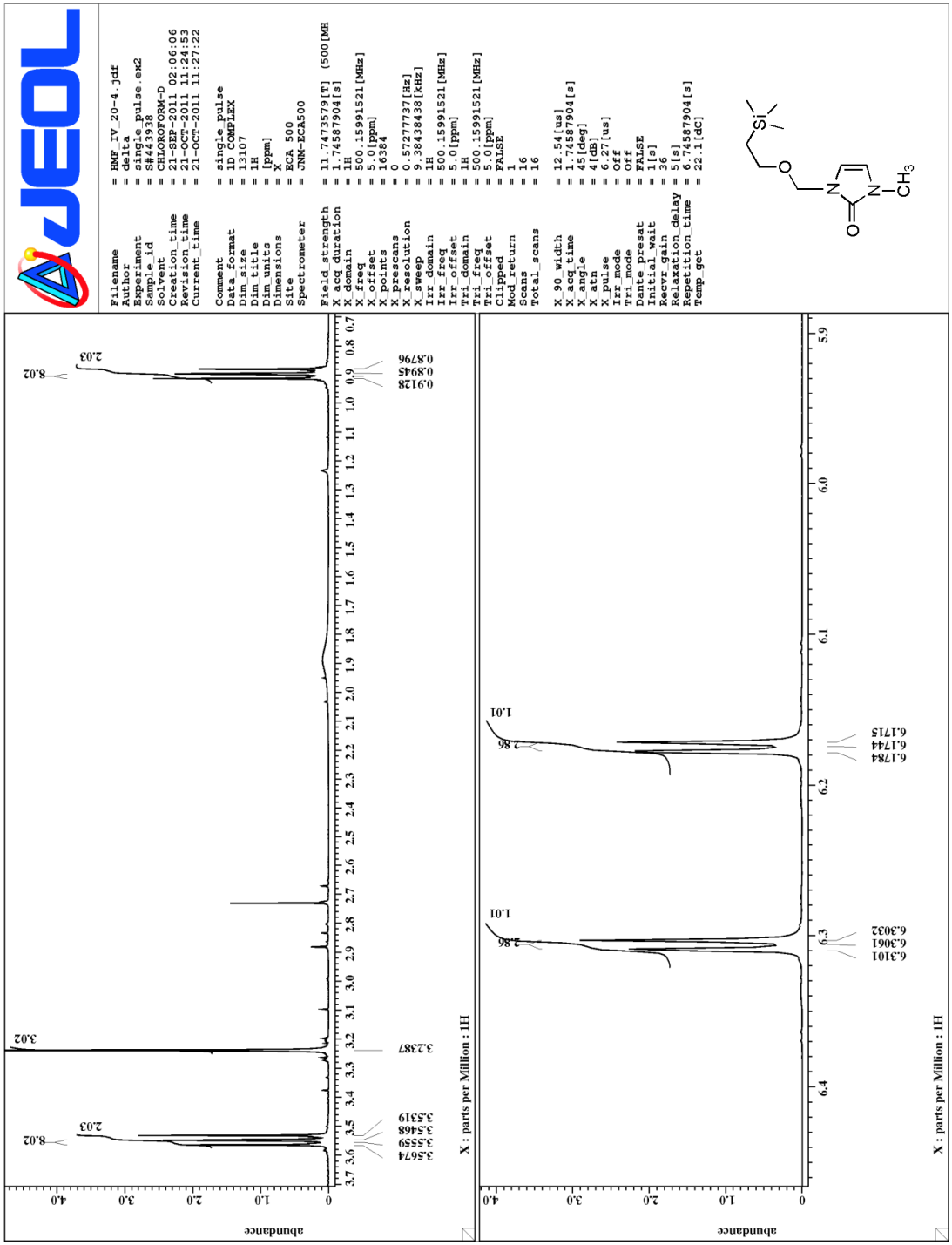

Filename = HMF_IV_12-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#634558
Solvent = CHLOROFORM-D
Creation_time = 20-JUL-2011 07:46:37
Revision_time = 21-OCT-2011 10:57:56
Current_time = 21-OCT-2011 10:58:33
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
Bn
N
N
CH3
O
abundance
X : parts per Million : 1H
3.02
2.0
1.01
2.01
3.01
3.2582
4.7641
6.1521
6.1452
6.0971
7.3190
7.3041
7.2434

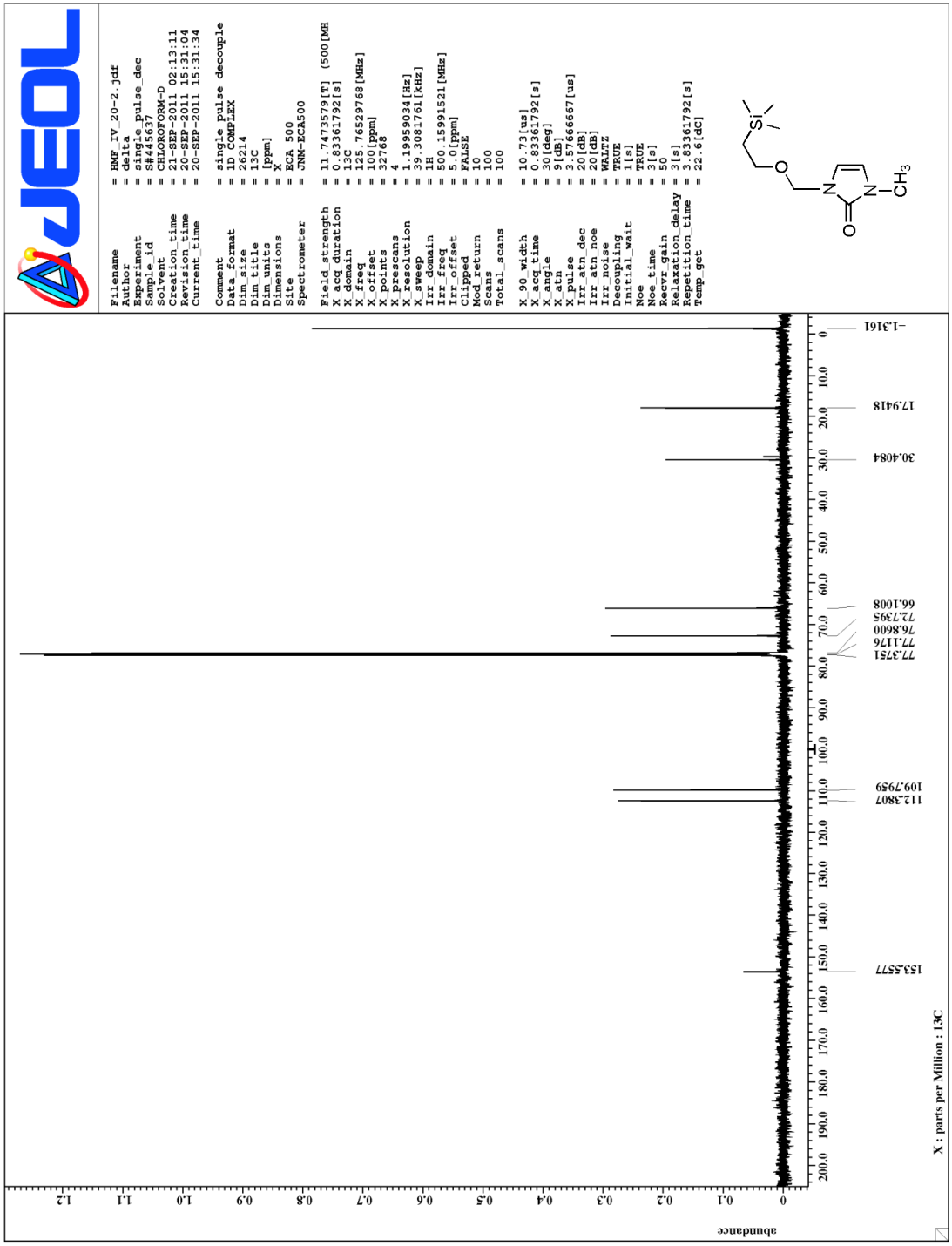

JEOL
Filename = HMF_IV_12-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#634558
Solvent = CHLOROFORM-D
Creation_time = 20-JUL-2011 07:46:37
Revision_time = 21-OCT-2011 11:00:30
Current_time = 21-OCT-2011 11:01:30
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384381[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
Bn
N
N
O
CH3
abundance
X : parts per Million : 1H
1.01
3.44
6.0971
6.0937
6.0902
6.1521
6.1486
6.1452
2.01
3.01
7.2297
7.2434
7.2457
7.2549
7.3041
7.3156
7.3190


JEOL
Filename = HMF_IV_12-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#636286
Solvent = CHLOROFORM-D
Creation_time = 20-JUL-2011 07:59:31
Revision_time = 9-NOV-2011 15:21:01
Current_time = 9-NOV-2011 15:22:24
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817614[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 150
Total_scans = 150
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.7[dC]
Bn
N
N
CH3
O
153.3574
137.0278
128.8344
127.9187
127.8424
111.7989
109.9962
77.3942
77.1366
76.8886
47.3198
30.5991
abundance
X : parts per Million : 13C

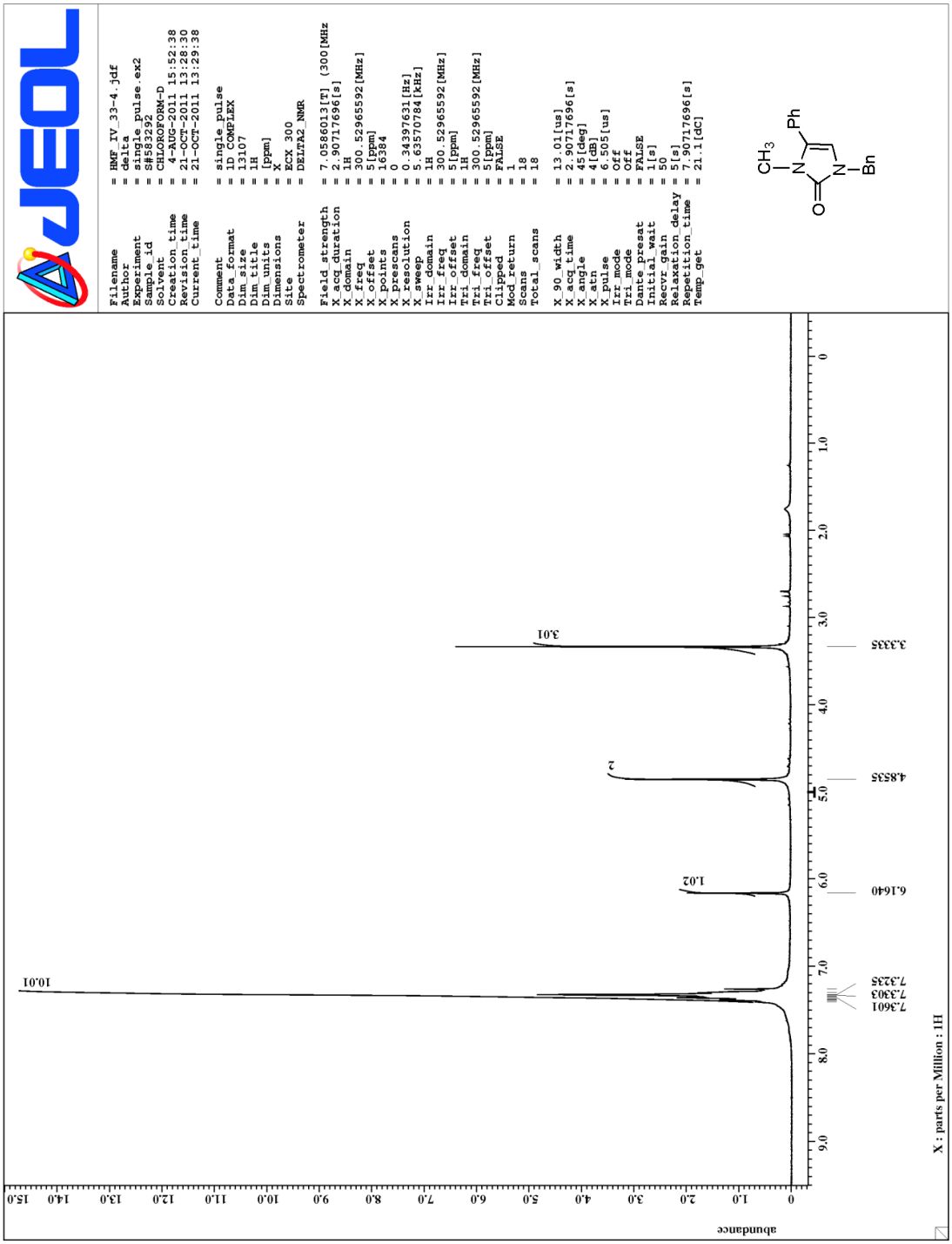
APPENDIX 19
^{1}H and ^{13}C NMR Spectra of
1-Methyl-3-trimethylsilylethoxymethyl-1,3-dihydro-2*H*-imidazol-2-one (**82i**)

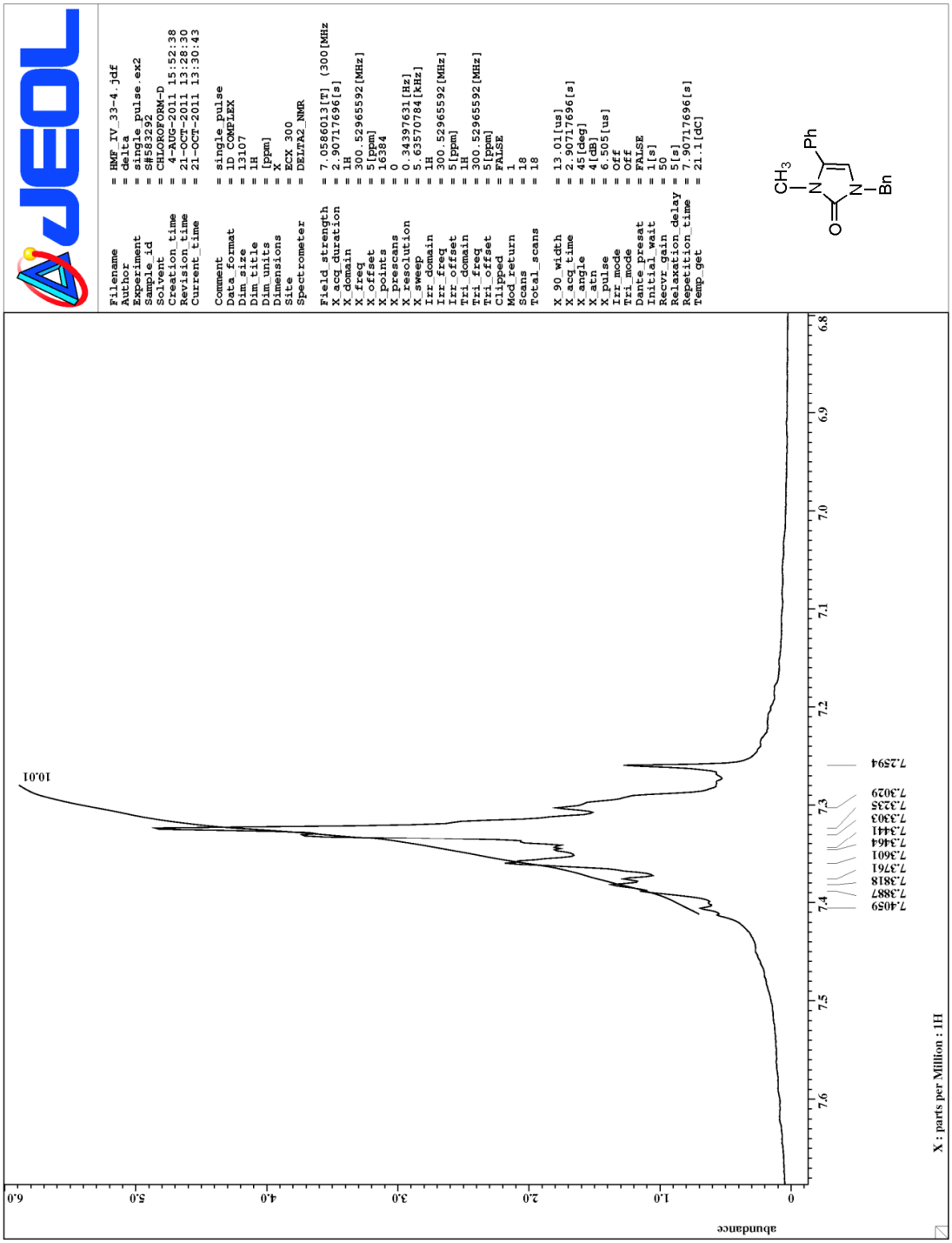

Filename = RMF_IV_20-6.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#443938
Solvent = CHLOROFORM-D
Creation_time = 21-SEP-2011 02:06:06
Revision_time = 21-OCT-2011 11:28:13
Current_time = 21-OCT-2011 11:28:41
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.1[dC]
JEOL
abundance
X : parts per Million : 1H
9.02
3.02
2.03
2.03
2
1.01
1.01
7.2675
6.3090
6.3032
6.1715
4.9840
3.5639
3.5479
3.5319
3.2387
0.9128
0.8968
0.8796
-0.0194
-0.0262

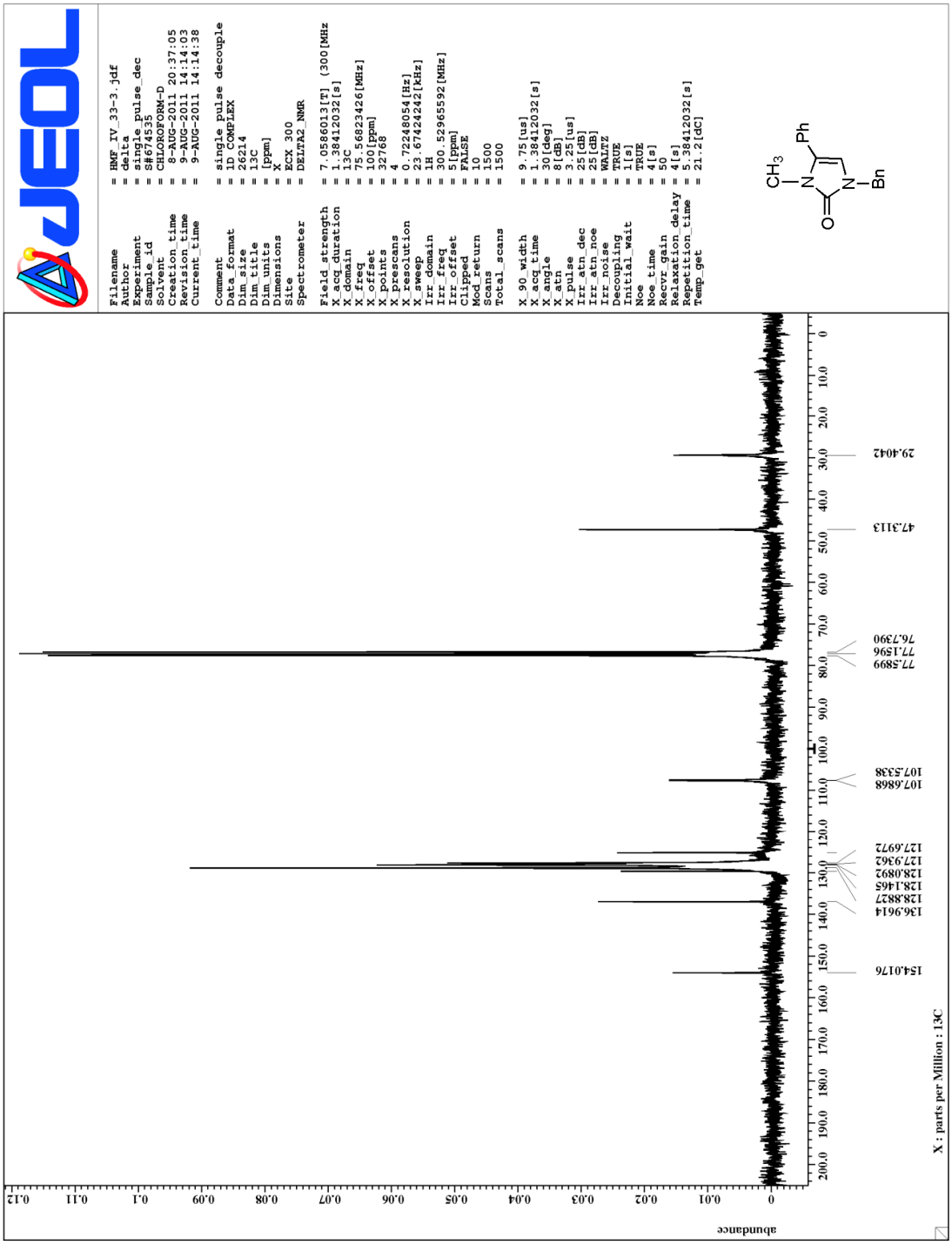

JEOL
Filename = HMF_IV_20-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#443938
Solvent = CHLOROFORM-D
Creation_time = 21-SEP-2011 02:06:06
Revision_time = 21-OCT-2011 11:24:53
Current_time = 21-OCT-2011 11:27:22
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.1[dC]
8.02
2.03
3.02
3.2387
3.5574
3.5559
3.5468
3.5319
0.9128
0.8945
0.8796
abundance
X : parts per Million : 1H
6.3101
6.3061
6.3032
6.1784
6.1744
6.1715
2.86
1.01

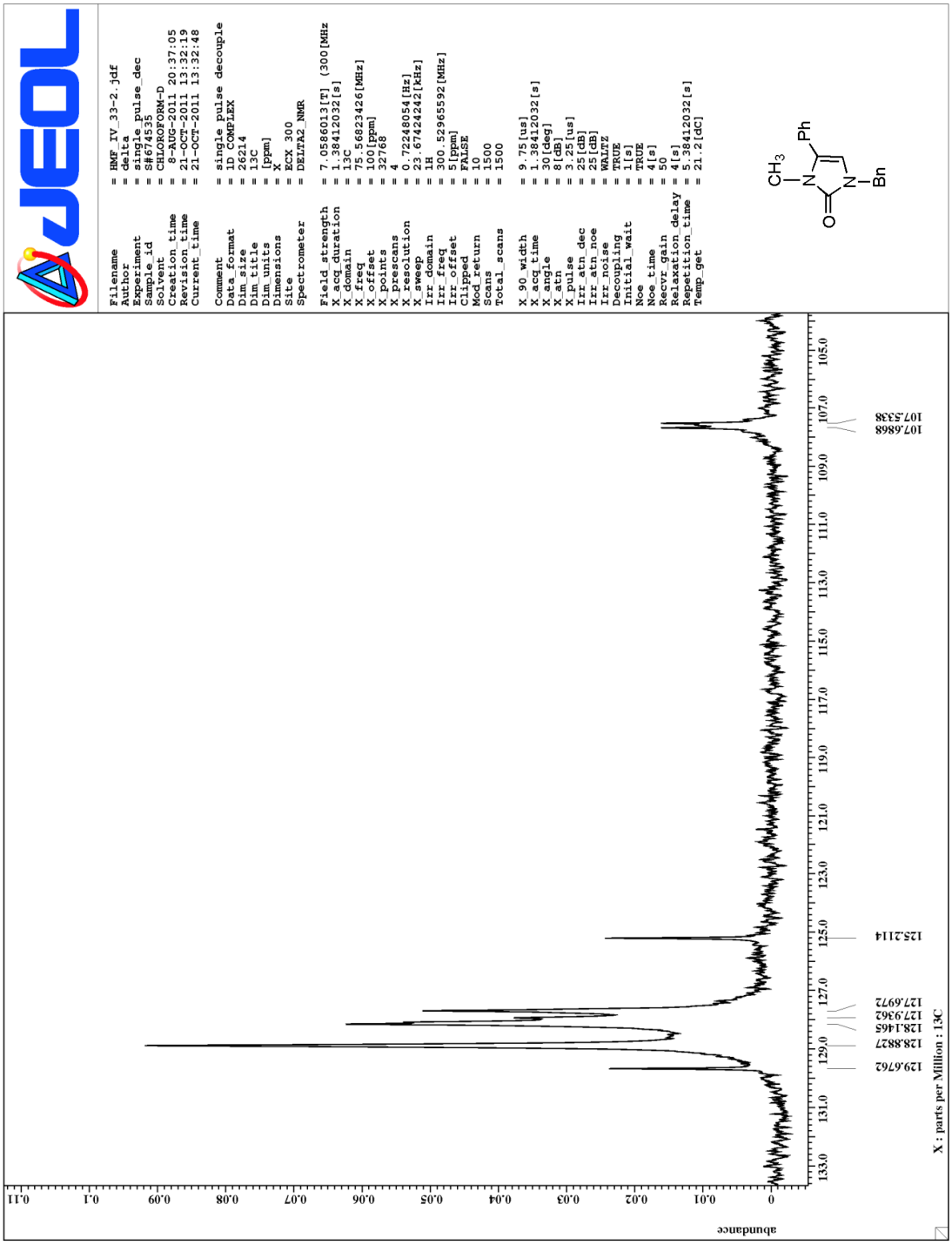

Filename = HMF_IV_20-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#445637
Solvent = CHLOROFORM-D
Creation_time = 21-SEP-2011 02:13:11
Revision_time = 20-SEP-2011 15:31:04
Current_time = 20-SEP-2011 15:31:34
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 100
Total_scans = 100
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.6[dC]
-1.3161
17.9418
30.4084
66.1008
72.7395
76.8600
77.1176
77.3751
109.7959
112.3807
153.5577
abundance
X : parts per Million : 13C

APPENDIX 20
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-3-methyl-4-phenyl-1,3-dihydro-2*H*-imidazol-2-one (**82j**)

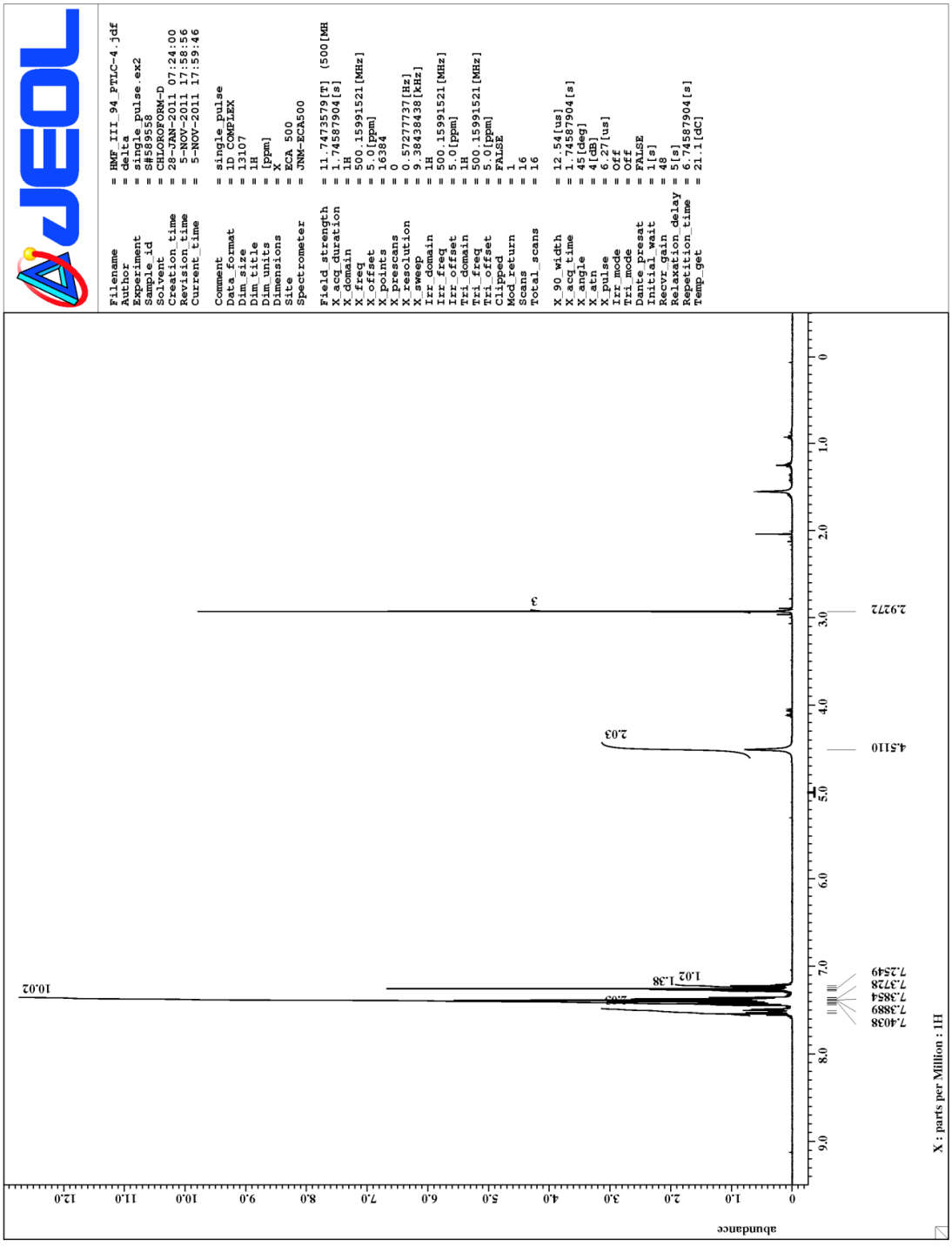

Filename = RMF_IV_33-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#583292
Solvent = CHLOROFORM-D
Creation_time = 4-AUG-2011 15:52:38
Revision_time = 21-OCT-2011 13:28:30
Current_time = 21-OCT-2011 13:29:38
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 18
Total_scans = 18
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21.1[dC]
CH3
Ph
Bn
10.01
1.02
2
3.01
3.3335
4.8535
6.1640
7.3601
7.3303
7.3235
abundance
X : parts per Million : 1H

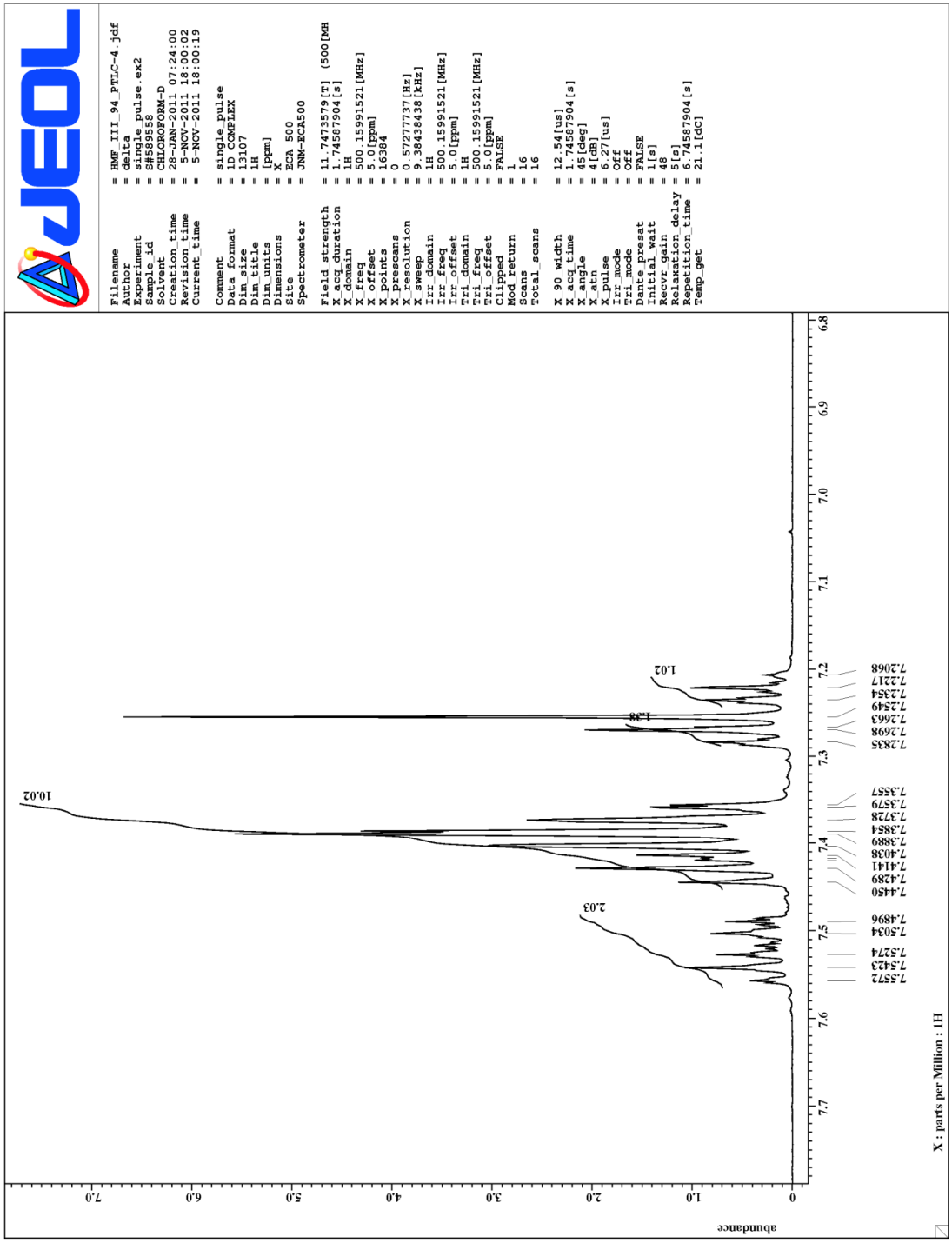

JEOL
Filename = HMF_IV_33-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#583292
Solvent = CHLOROFORM-D
Creation_time = 4-AUG-2011 15:52:38
Revision_time = 21-OCT-2011 13:28:30
Current_time = 21-OCT-2011 13:30:43
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 18
Total_scans = 18
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 21.1[dC]
CH3
Ph
Bn
10.01
abundance
X : parts per Million : 1H
7.4059
7.3887
7.3818
7.3761
7.3601
7.3464
7.3441
7.3303
7.3335
7.3029
7.2594

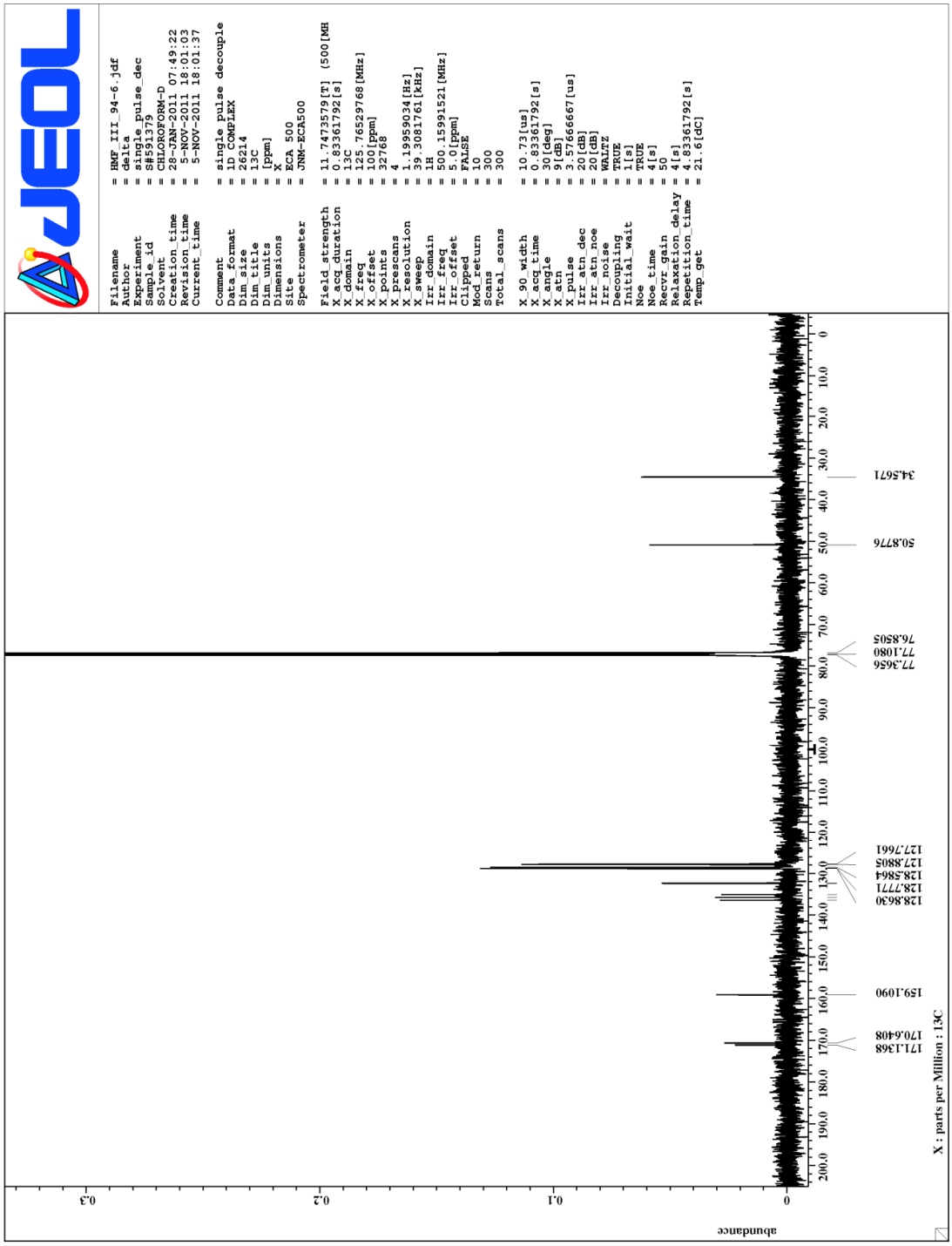

Filename = RMF_IV_33-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#674535
Solvent = CHLOROFORM-D
Creation_time = 8-AUG-2011 20:37:05
Revision_time = 9-AUG-2011 14:14:03
Current_time = 9-AUG-2011 14:14:38
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1500
Total_scans = 1500
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe_time = TRUE
Recyr_gain = 4[s]
Relaxation_delay = 50
Repetition_time = 4[s]
Temp_get = 5.38412032[s]
= 21.2[dC]
CH3
Ph
Bn
O
29.4042
47.3113
76.7390
77.1596
77.5899
107.5338
107.6868
127.6972
127.9362
128.0892
128.1465
128.8827
136.9614
154.0176
abundance
X : parts per Million : 13C

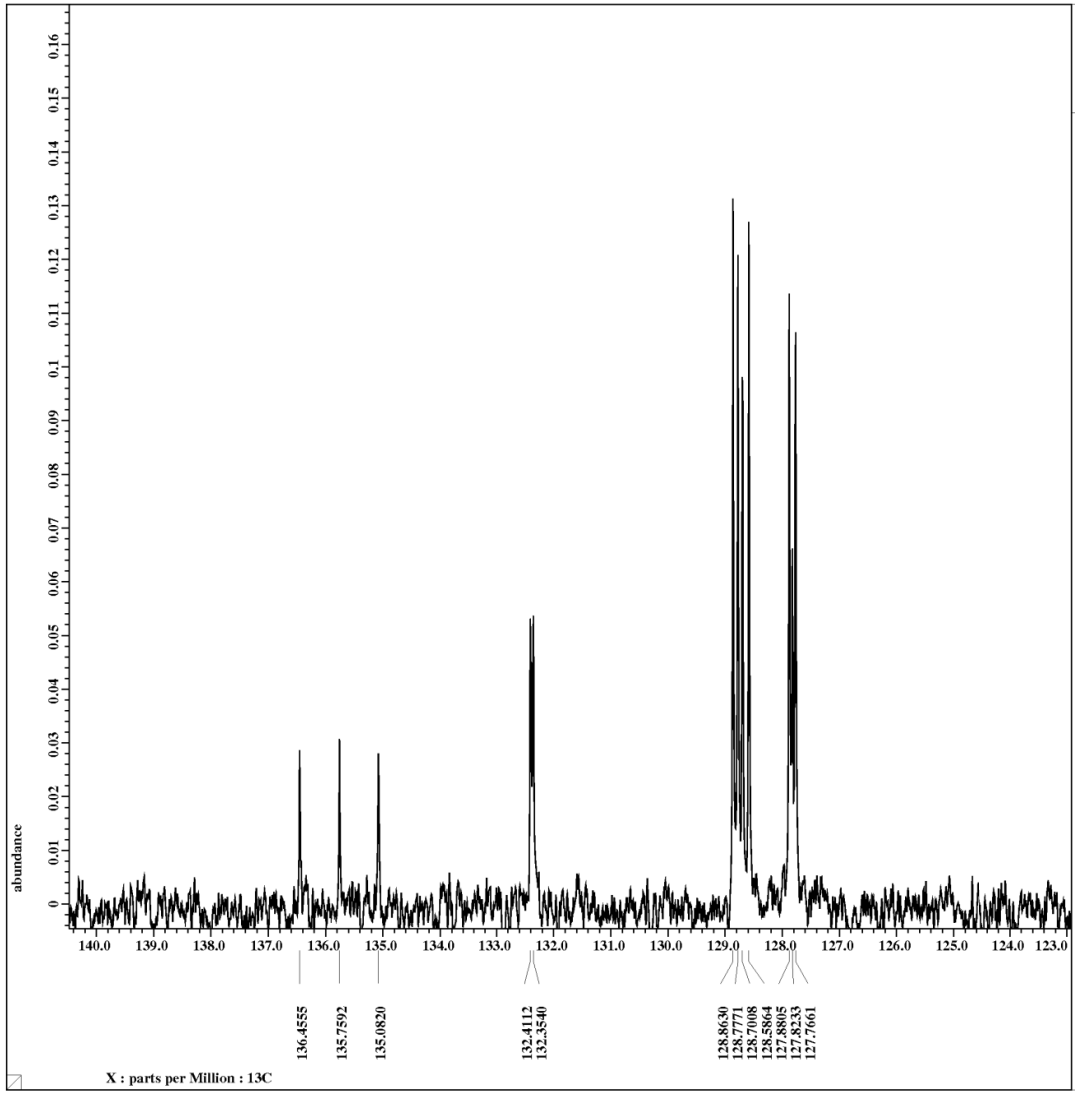

JEOL
Filename = HMF_IV_33-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#674535
Solvent = CHLOROFORM-D
Creation_time = 8-AUG-2011 20:37:05
Revision_time = 21-OCT-2011 13:32:19
Current_time = 21-OCT-2011 13:32:48
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1500
Total_scans = 1500
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 5.38412032[s]
Temp_get = 21.2[dC]
CH3
Ph
N
N
Bn
O
abundance
X : parts per Million : 13C
129.6762
128.8827
128.1465
127.9362
127.6972
125.2114
107.6868
107.5338

APPENDIX 21
^{1}H and ^{13}C NMR Spectra of unidentified oxidation product (**83**)


JEOL
Filename = RMF_III_94_PTLC-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#589558
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2011 07:24:00
Revision_time = 5-NOV-2011 17:58:56
Current_time = 5-NOV-2011 17:59:46
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.1[dC]
abundance
X : parts per Million : 1H
10.02
1.02
1.38
2.03
3
2.9272
4.5110
7.4038
7.3889
7.3854
7.3728
7.2549


Filename = RMF_III_94_PTLC-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#589558
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2011 07:24:00
Revision_time = 5-NOV-2011 18:00:02
Current_time = 5-NOV-2011 18:00:19
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.1[dC]
abundance
10.02
2.03
1.38
1.02
7.5572
7.5423
7.5374
7.5034
7.4896
7.4450
7.4289
7.4141
7.4038
7.3889
7.3854
7.3728
7.3579
7.3557
7.2835
7.2698
7.2663
7.2549
7.2354
7.2217
7.2068
X : parts per Million : 1H


Filename = HMF_III_94-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#591379
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2011 07:49:22
Revision_time = 5-NOV-2011 18:01:03
Current_time = 5-NOV-2011 18:01:37
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 300
Total_scans = 300
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.6[dC]
abundance
X : parts per Million : 13C
34.5671
50.8776
76.8505
77.1080
77.3656
128.8630
128.7771
128.5864
127.8805
127.7661
159.1090
171.1368
170.6408



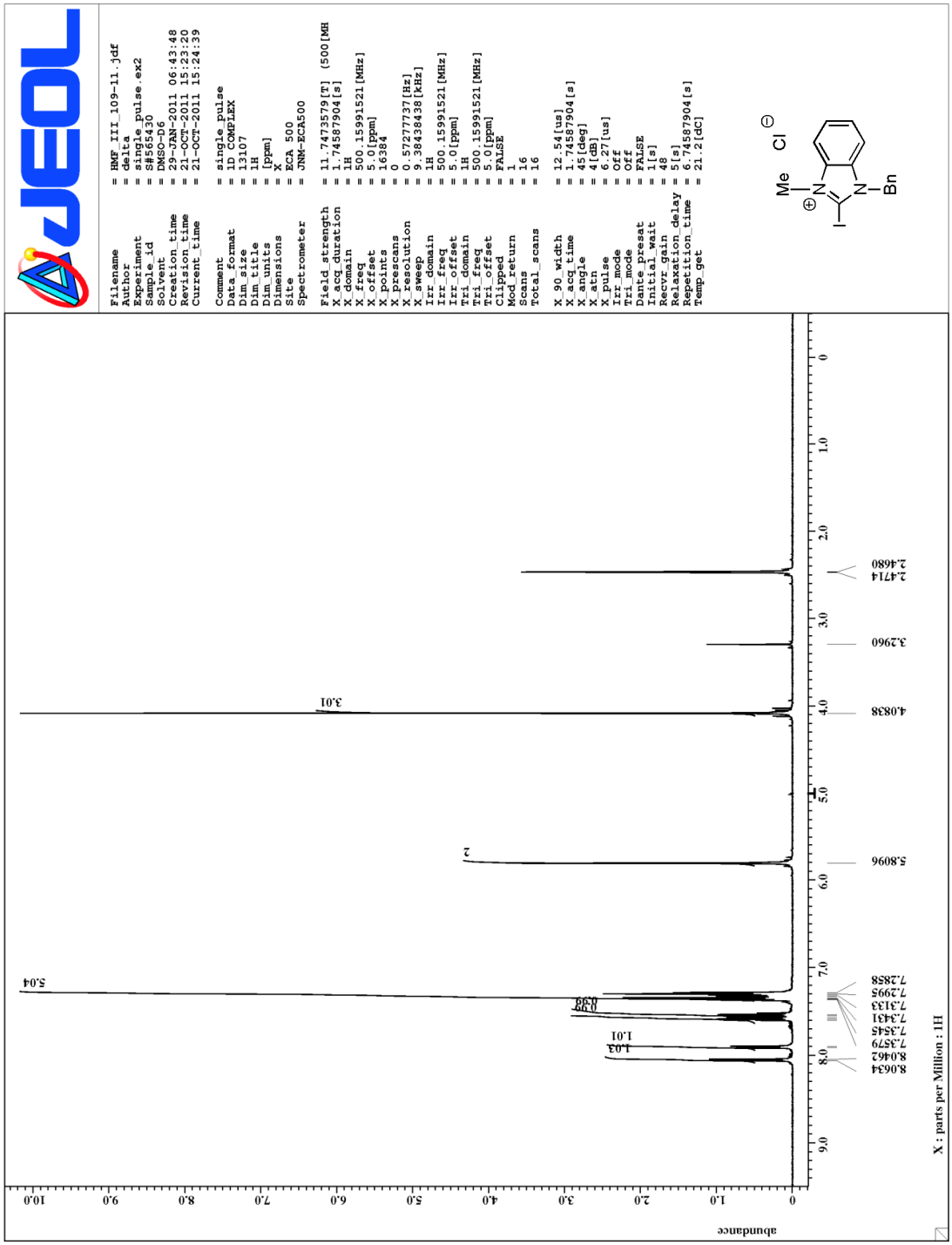
```
Filename          = HMF_III_94-6.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#591379
Solvent           = CHLOROFORM-D
Creation_time     = 28-JAN-2011 07:49:22
Revision_time     =  5-NOV-2011 18:01:03
Current_time      =  5-NOV-2011 18:02:04

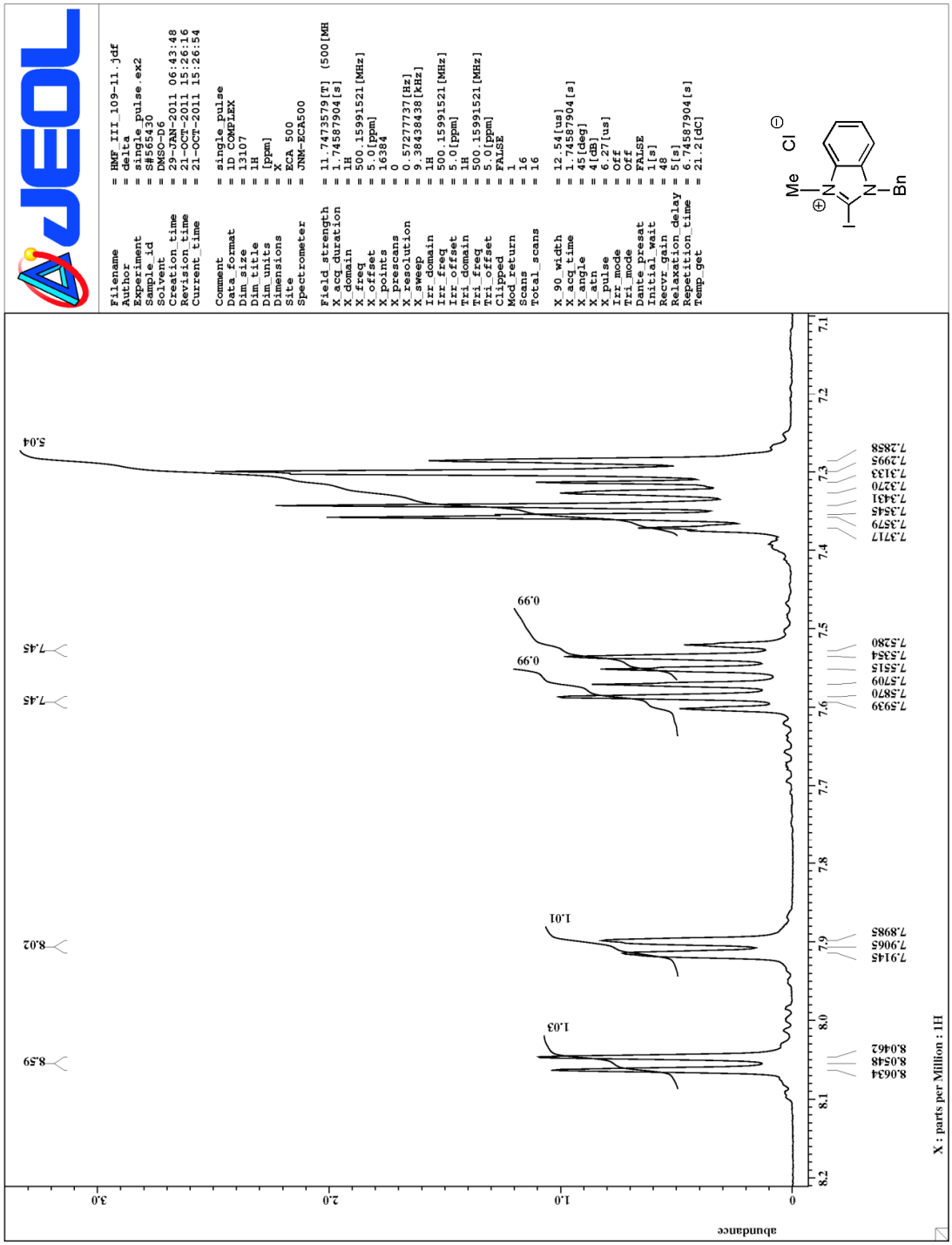
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

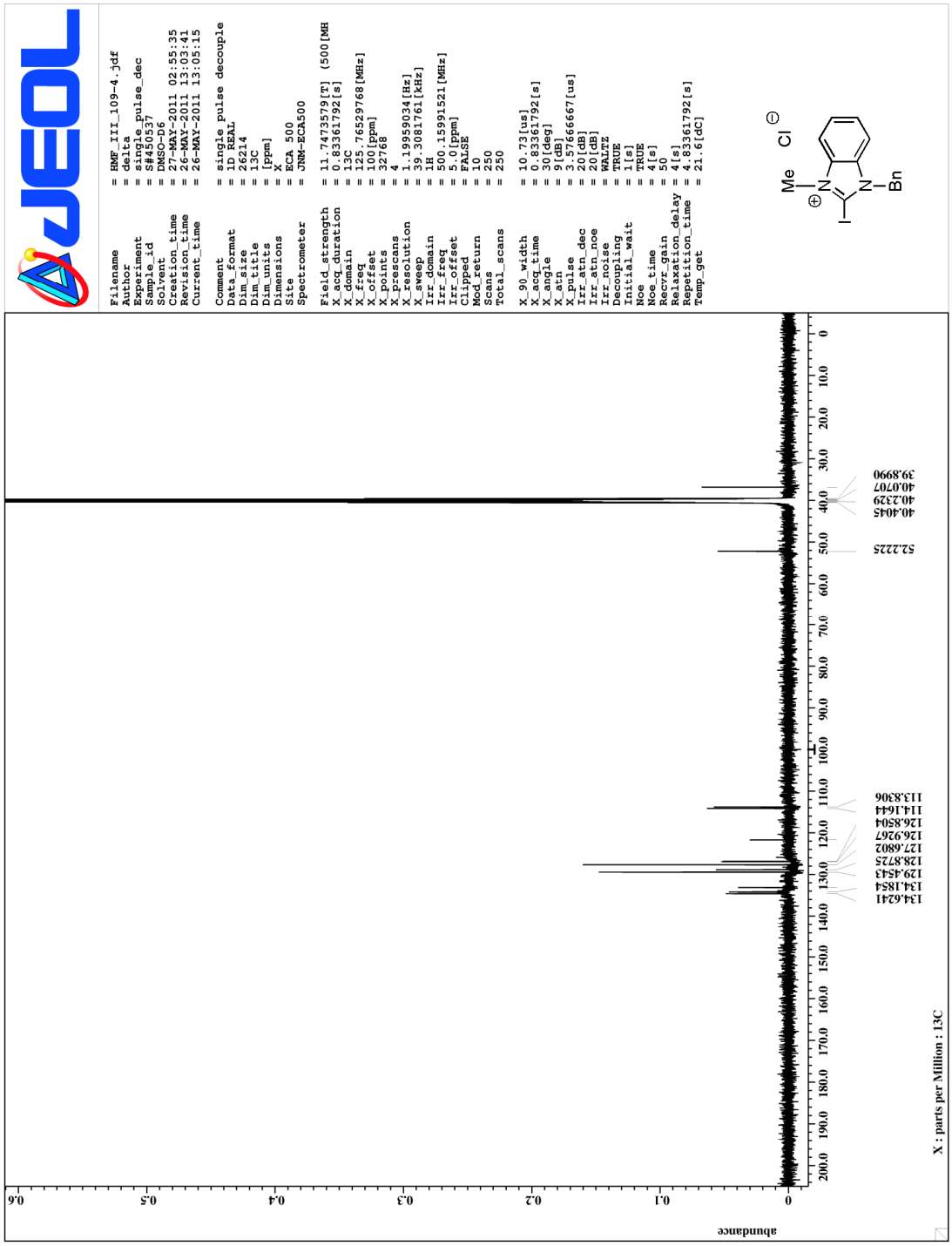
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 300
Total_scans       = 300

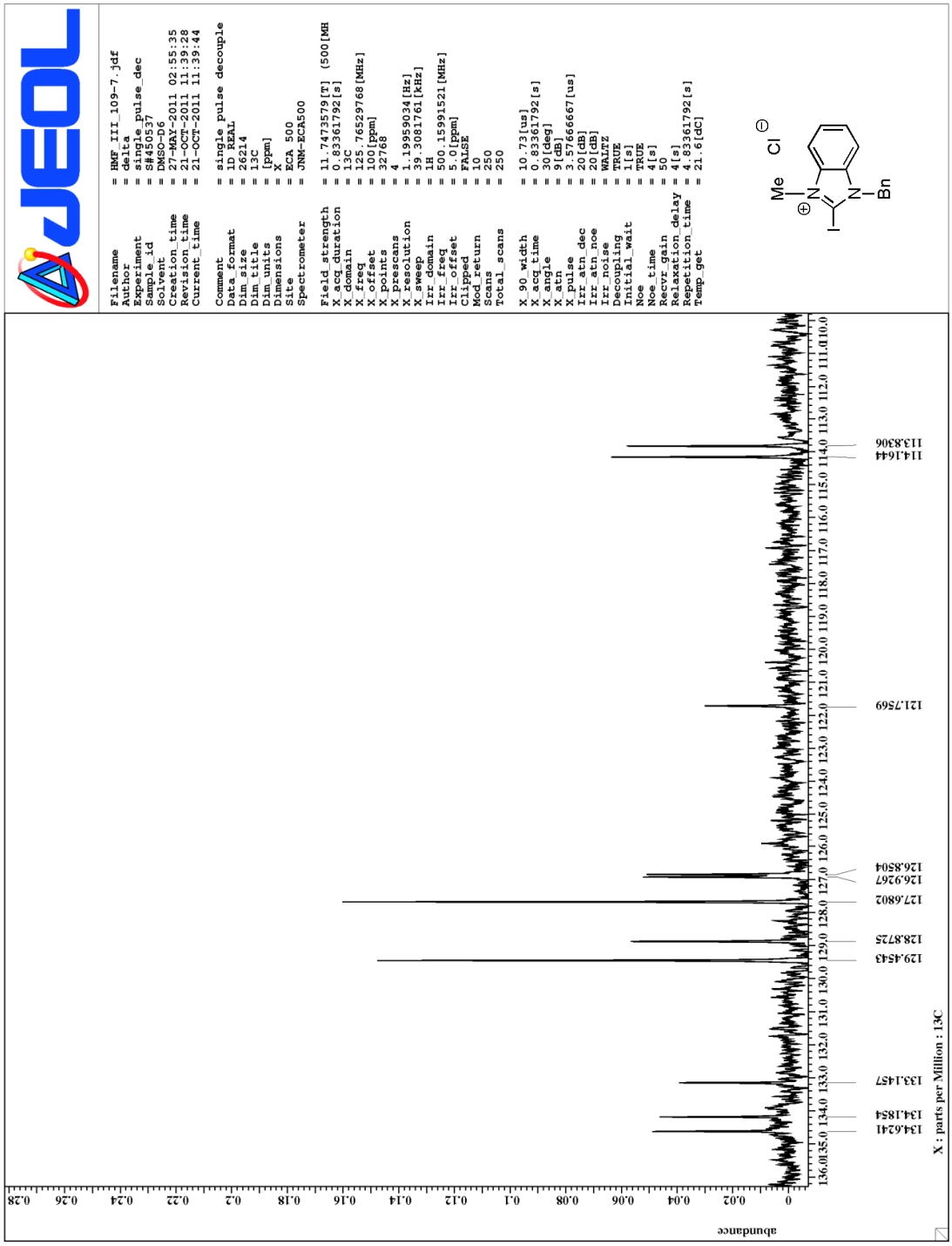
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 21.6[dC]
```

APPENDIX 22
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-2-iodo-3-methyl-1*H*-benzimidazol-3-ium chloride (**88**)

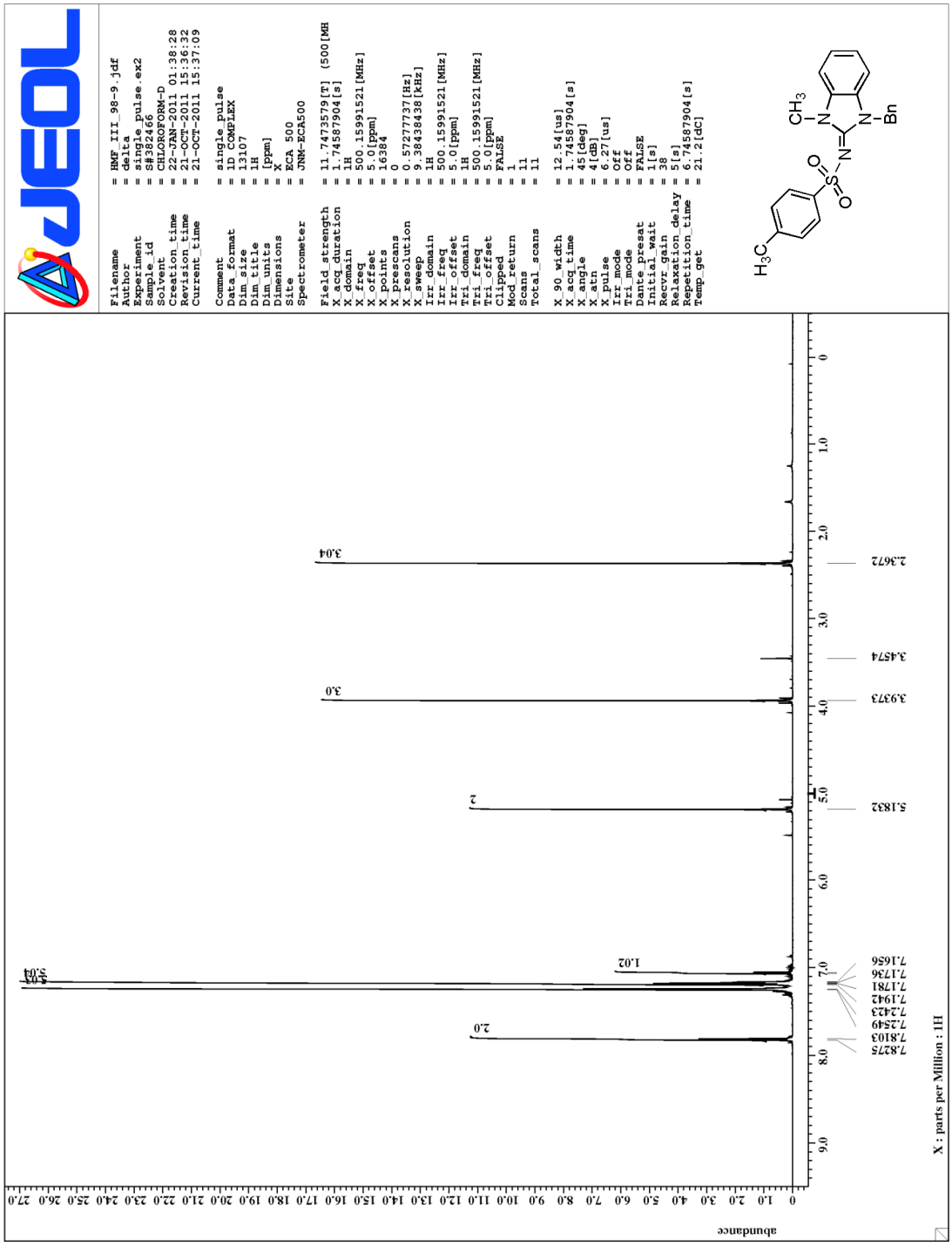

JEOL
Filename = RMF_III_109-11.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#565430
Solvent = DMSO-D6
Creation_time = 29-JAN-2011 06:43:48
Revision_time = 21-OCT-2011 15:23:20
Current_time = 21-OCT-2011 15:24:39
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
Me
Bn
Cl
abundance
X : parts per Million : 1H
5.04
3.01
2
1.03
1.01
0.99
0.99
8.0634
8.0462
7.3579
7.3545
7.3431
7.3133
7.2995
7.2858
5.8096
4.0838
3.2960
2.4714
2.4680

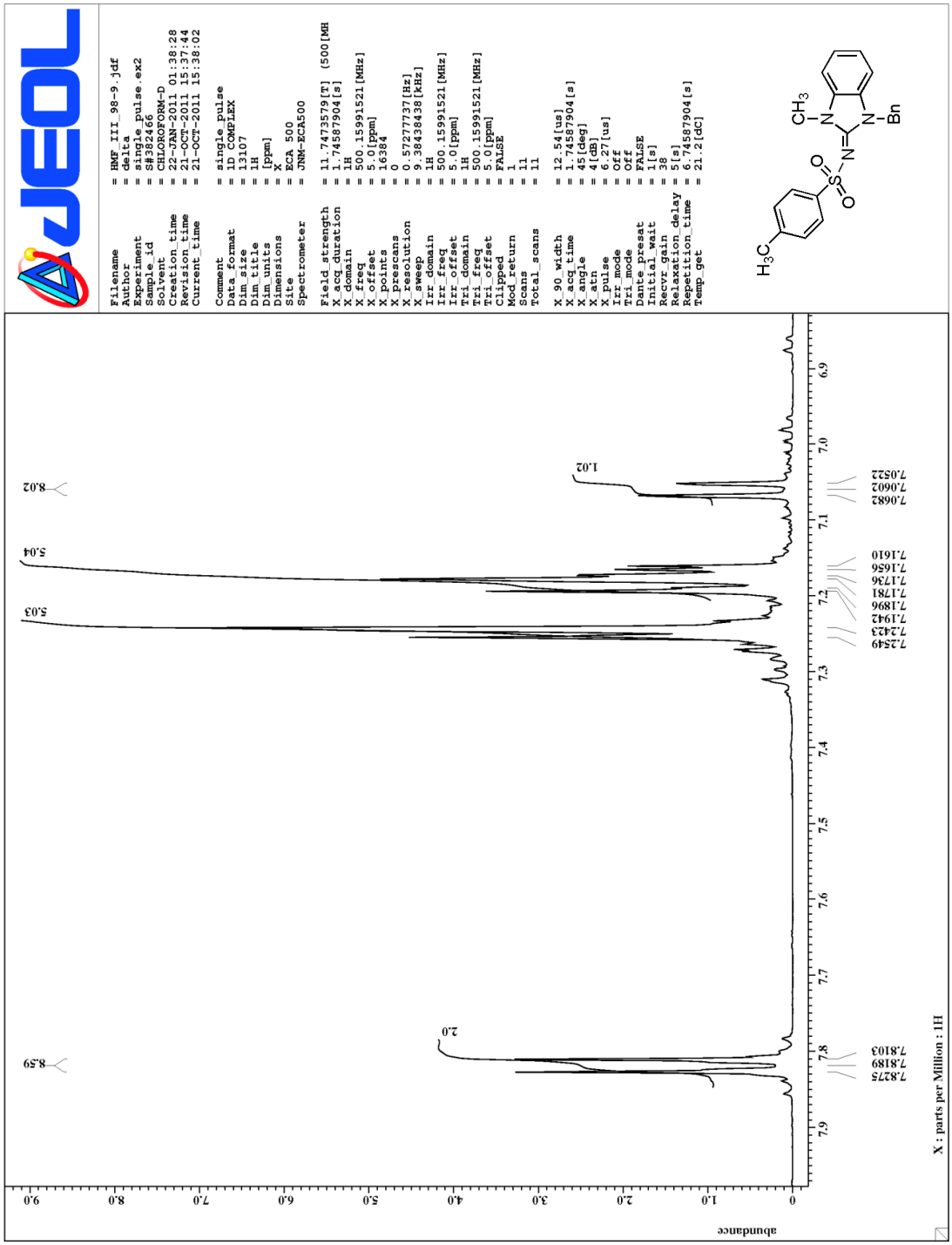

Filename = BMF_III_109-11.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#565430
Solvent = DMSO-D6
Creation_time = 29-JAN-2011 06:43:48
Revision_time = 21-OCT-2011 15:26:16
Current_time = 21-OCT-2011 15:26:54
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
Me
Cl
Bn
abundance
X : parts per Million : 1H
8.59
8.02
7.45
7.45
5.04
1.03
1.01
0.99
0.99
8.0634
8.0548
8.0462
7.9145
7.9065
7.8985
7.5939
7.5870
7.5709
7.5515
7.5354
7.5280
7.3717
7.3579
7.3545
7.3431
7.3170
7.3133
7.2995
7.2858

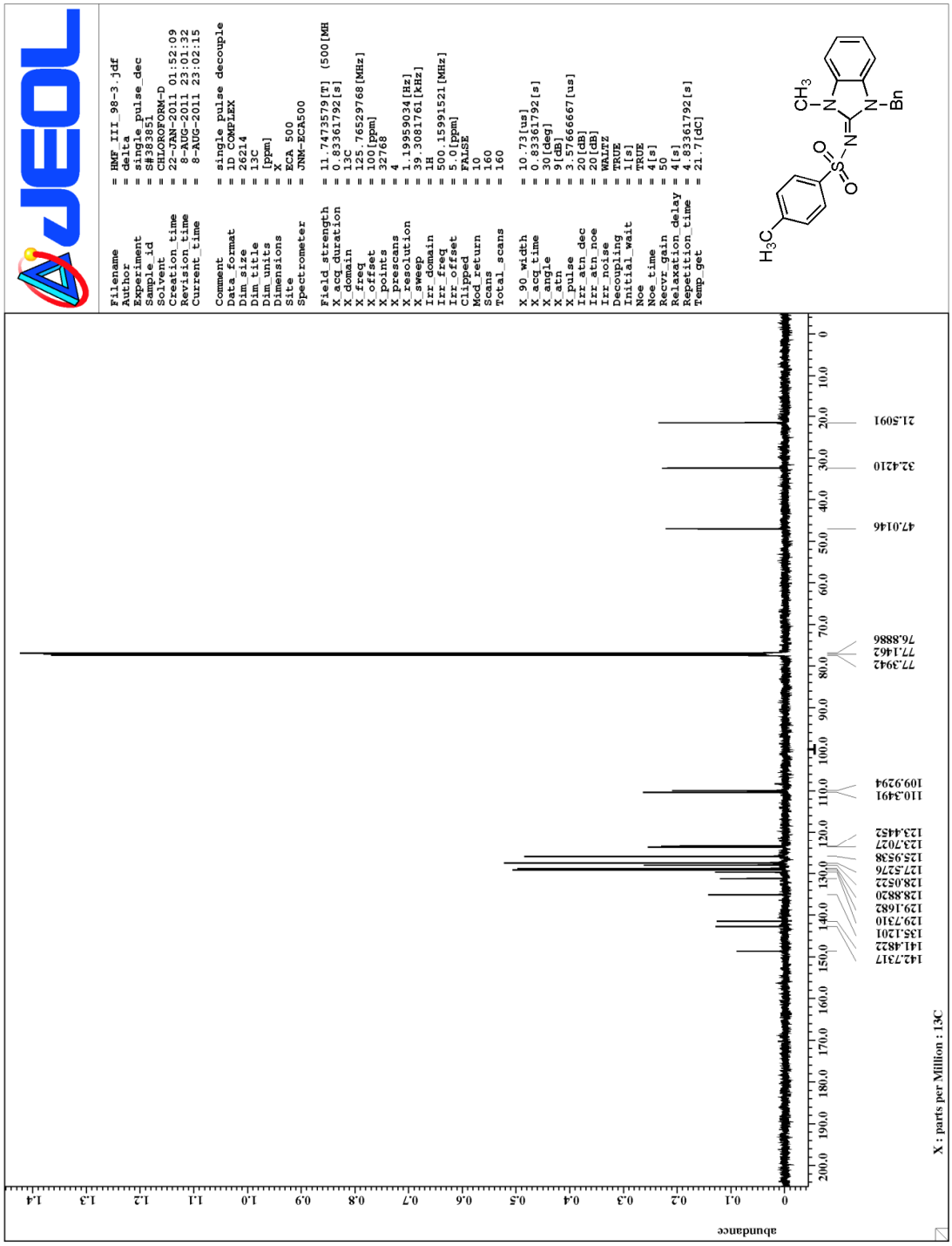

Filename = RMF_III_109-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#450537
Solvent = DMSO-D6
Creation_time = 27-MAY-2011 02:55:35
Revision_time = 26-MAY-2011 13:03:41
Current_time = 26-MAY-2011 13:05:15
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 250
Total_scans = 250
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Nce = TRUE
Nce_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.6[dC]
Me
Cl
Bn
abundance
X : parts per Million : 13C
134.6241
134.1854
129.4543
128.8725
127.6802
126.9267
126.8504
114.1644
113.8306
52.2225
40.4045
40.2329
40.0707
39.8990

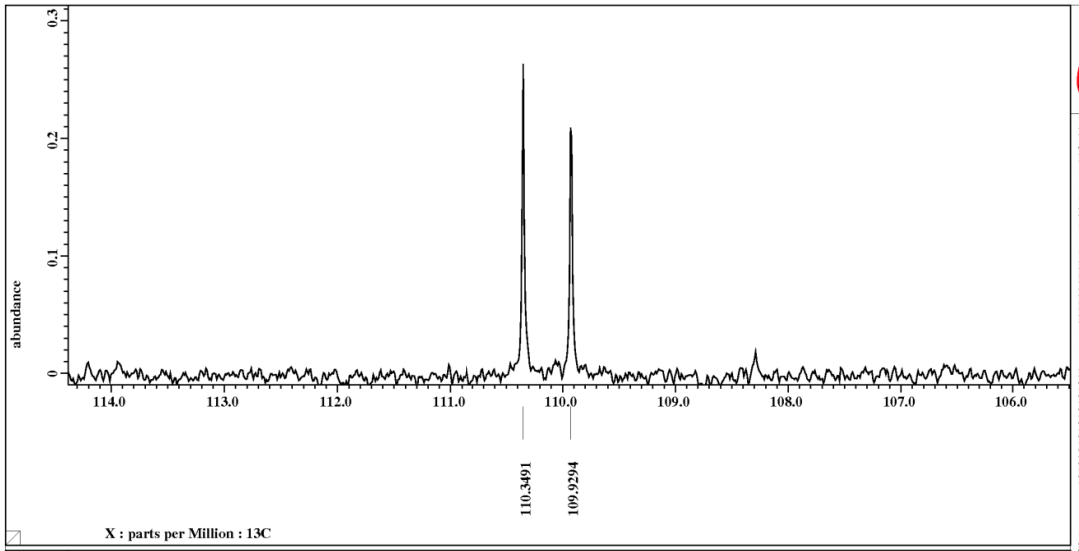

Filename = RMF_III_109-7.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#450537
Solvent = DMSO-D6
Creation_time = 27-MAY-2011 02:55:35
Revision_time = 21-OCT-2011 11:39:28
Current_time = 21-OCT-2011 11:39:44
Comment = single_pulse_decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 250
Total_scans = 250
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.6[dC]
Me
Cl
Bn
I
abundance
X : parts per Million : 13C
134.6241
134.1854
133.1457
129.4543
128.8725
127.6802
126.9267
126.8504
121.7569
114.1644
113.8306

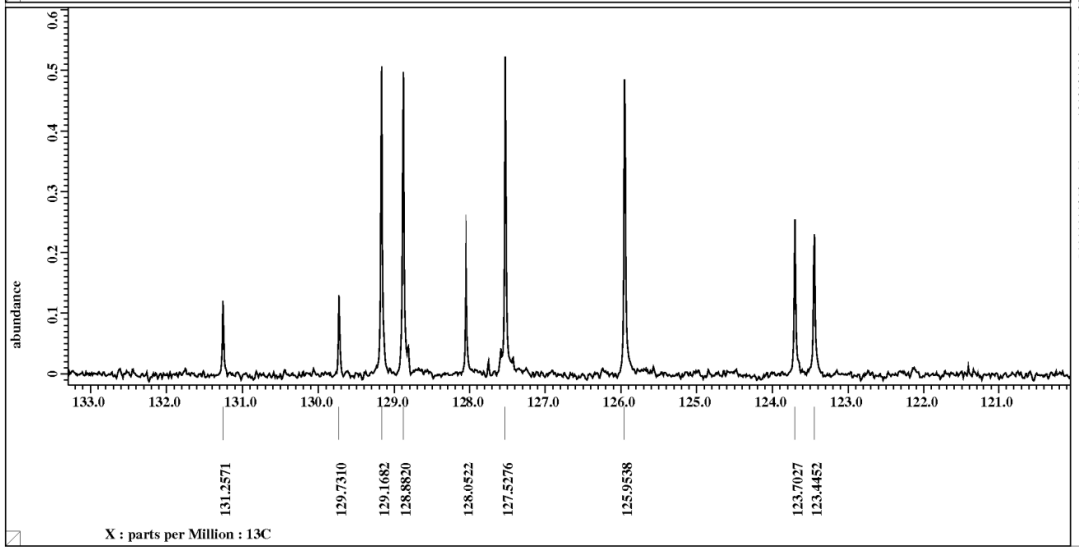
APPENDIX 23
^{1}H and ^{13}C NMR Spectra of
N-[(2*E*)-1-benzyl-3-methyl-1,3-dihydro-2*H*-benzimidazol-2-ylidene]-4-methylbenzenesulfonamide (**90**)


Filename = RMF_III_98-9.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#382466
Solvent = CHLOROFORM-D
Creation_time = 22-JAN-2011 01:38:28
Revision_time = 21-OCT-2011 15:36:32
Current_time = 21-OCT-2011 15:37:09
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 11
Total_scans = 11
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
CH3
Bn
H3C
abundance
X : parts per Million : 1H
3.04
3.0
2.0
1.02
2.3672
3.4574
3.9373
5.1832
7.8275
7.8103
7.2549
7.2423
7.1942
7.1781
7.1736
7.1656

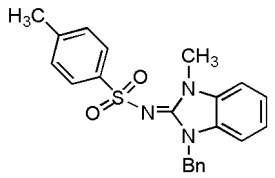

JEOL
Filename = RMF_III_98-9.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#382466
Solvent = CHLOROFORM-D
Creation_time = 22-JAN-2011 01:38:28
Revision_time = 21-OCT-2011 15:37:44
Current_time = 21-OCT-2011 15:38:02
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 11
Total_scans = 11
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
CH3
Bn
H3C
abundance
X : parts per Million : 1H
8.59
8.02
5.04
5.03
2.0
1.02
7.8375
7.8189
7.8103
7.2549
7.2423
7.1942
7.1896
7.1781
7.1736
7.1656
7.1610
7.0682
7.0602
7.0522


Filename = RMF_III_98-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#383851
Solvent = CHLOROFORM-D
Creation_time = 22-JAN-2011 01:52:09
Revision_time = 8-AUG-2011 23:01:32
Current_time = 8-AUG-2011 23:02:15
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 160
Total_scans = 160
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.7[dC]
21.5091
32.4210
47.0146
76.8886
77.1462
77.3942
109.9294
110.3491
123.4452
123.7027
125.9538
127.5276
128.0522
128.8820
129.1682
129.7310
135.1201
141.4822
142.7317
abundance
X : parts per Million : 13C

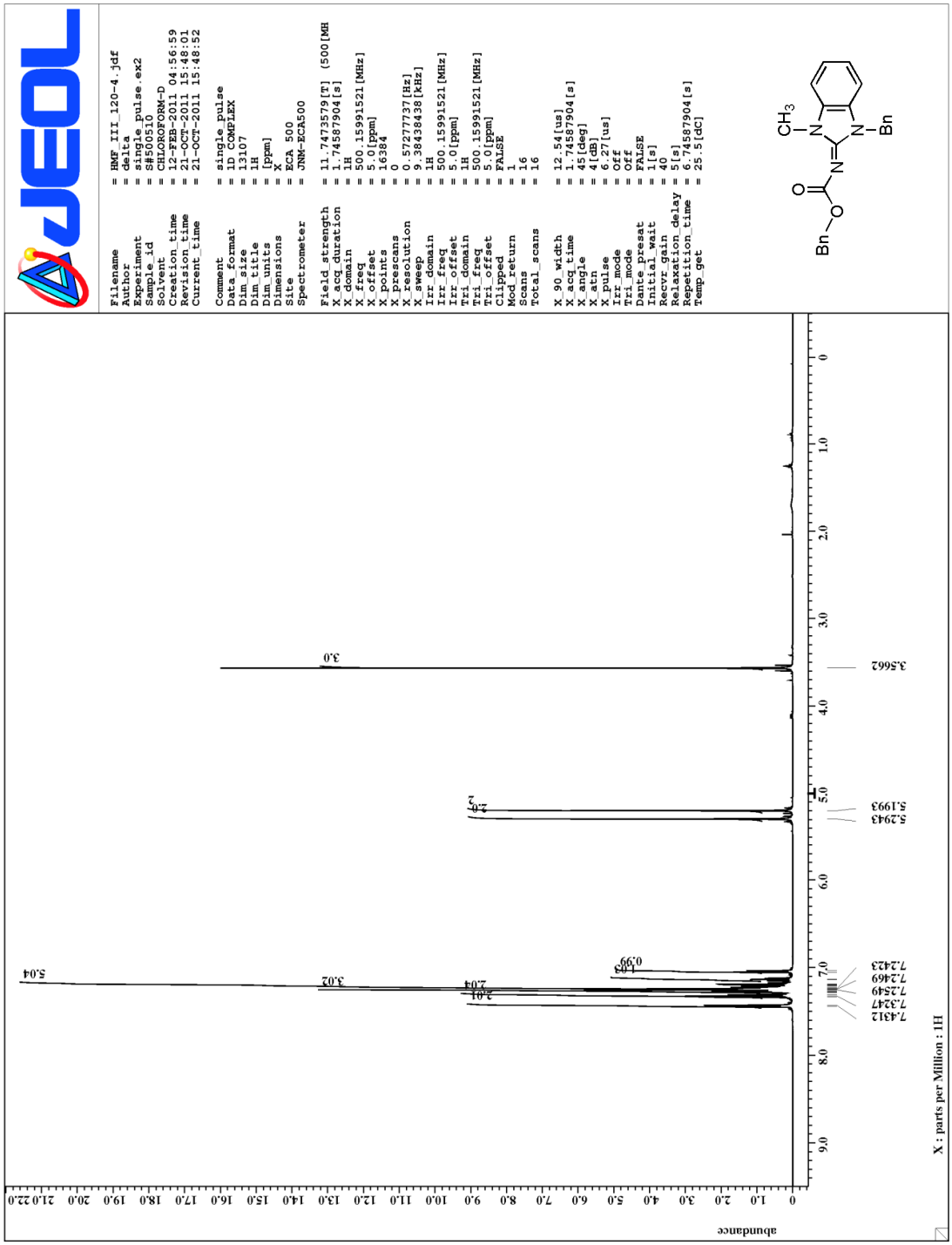


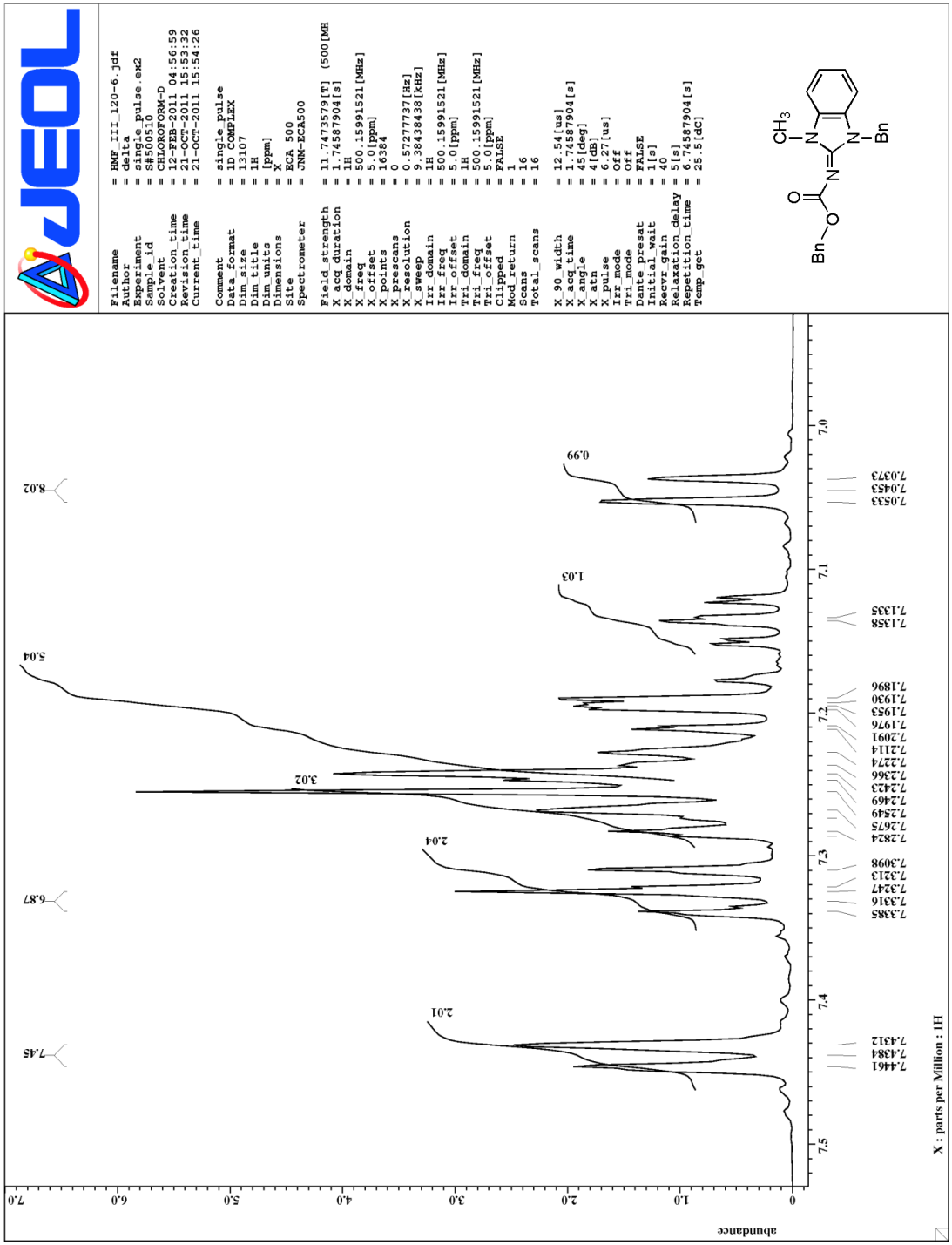


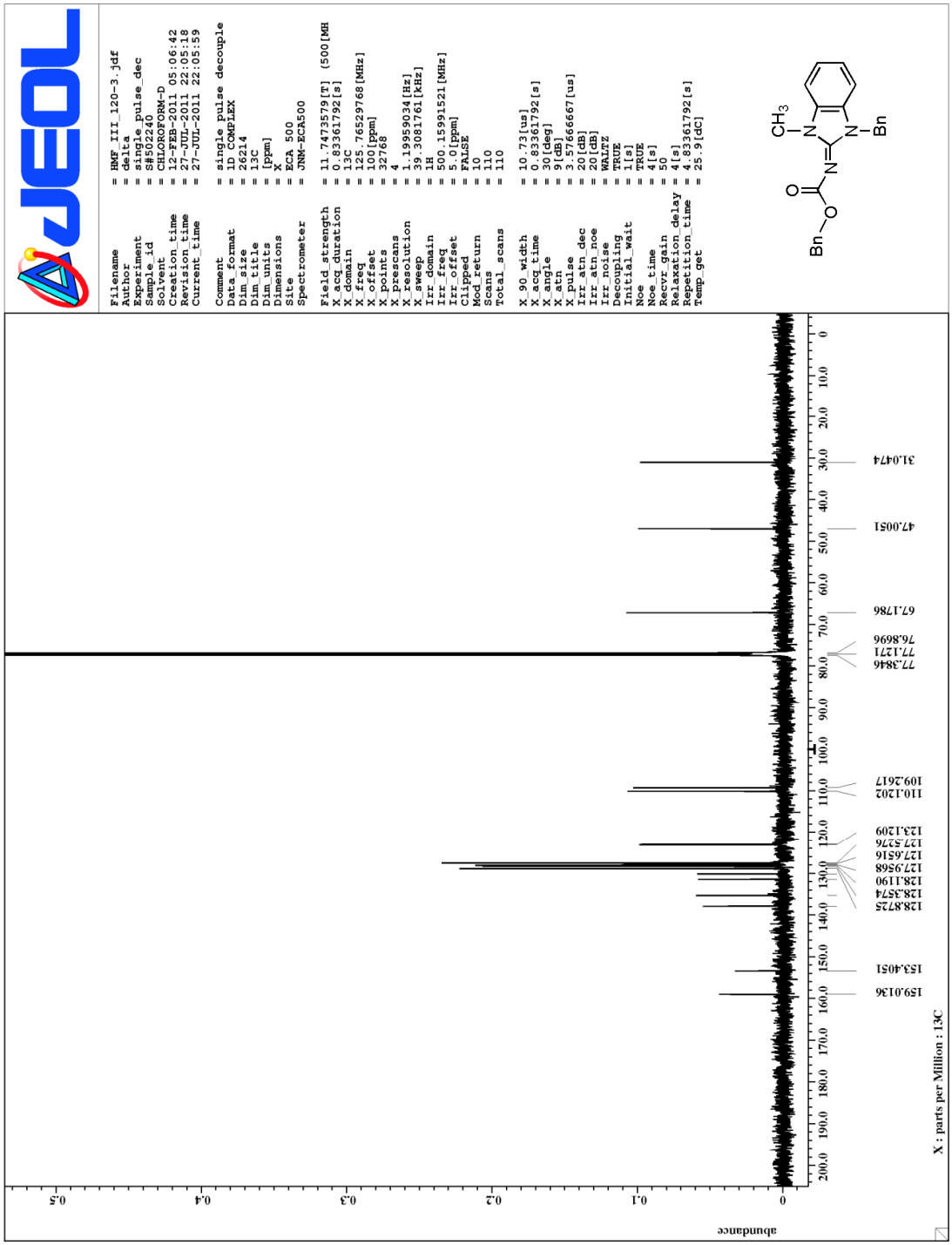
Filename	= HMF_III_98-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#383851
Solvent	= CHLOROFORM-D
Creation_time	= 22-JAN-2011 01:52:09
Revision_time	= 18-OCT-2011 11:14:22
Current_time	= 18-OCT-2011 11:15:34
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 10
Scans	= 160
Total_scans	= 160
X_90_width	= 10.73[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 9[dB]
X_pulse	= 3.57666667[us]
Irr_atn_dec	= 20[dB]
Irr_atn_noe	= 20[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 4[s]
Recvr_gain	= 50
Relaxation_delay	= 4[s]
Repetition_time	= 4.83361792[s]
Temp_get	= 21.7[dC]

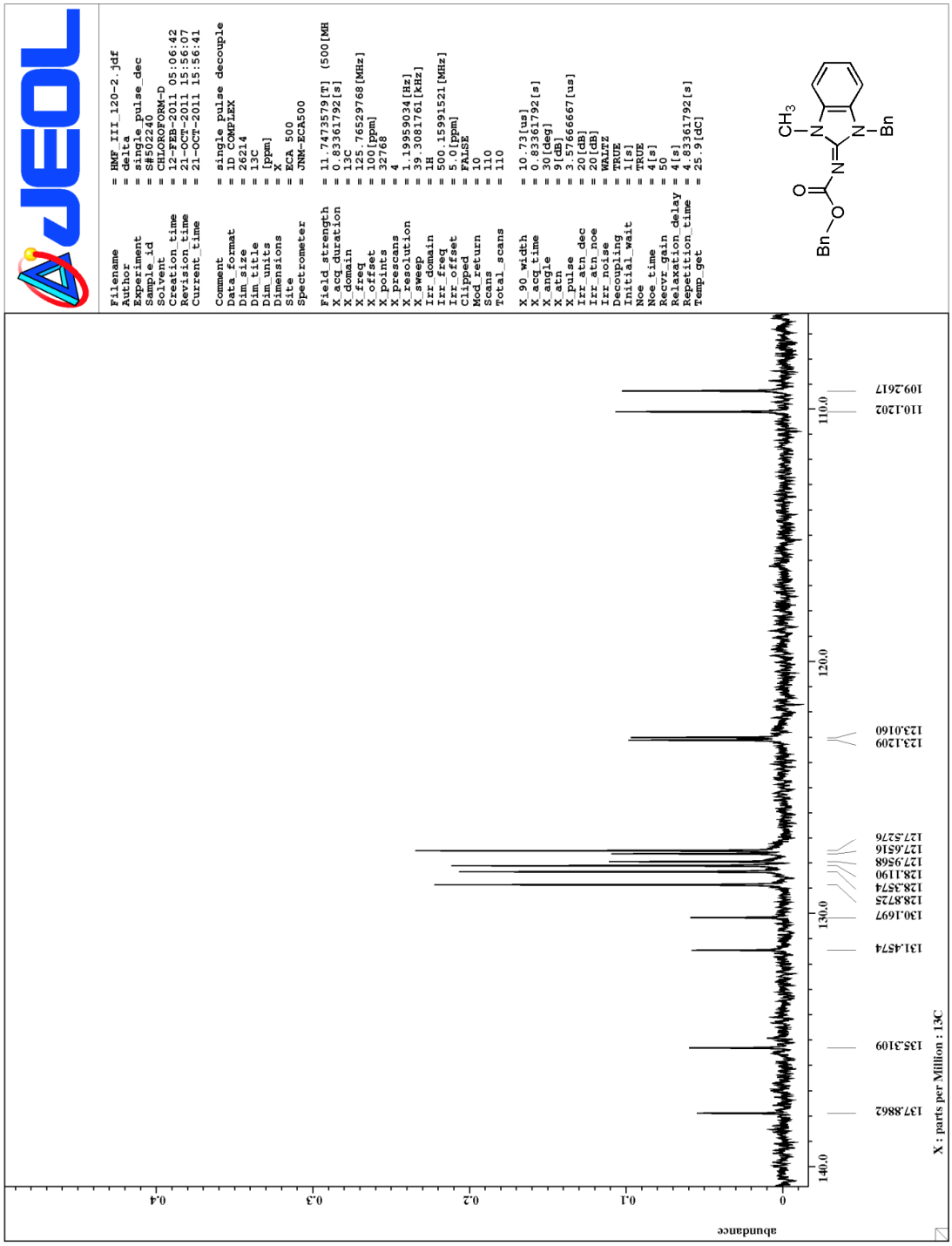


APPENDIX 24
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-2-benzyloxycarbonylimino-3-methyl-1,3-dihydro-2*H*-benzimidazole (**95a**)

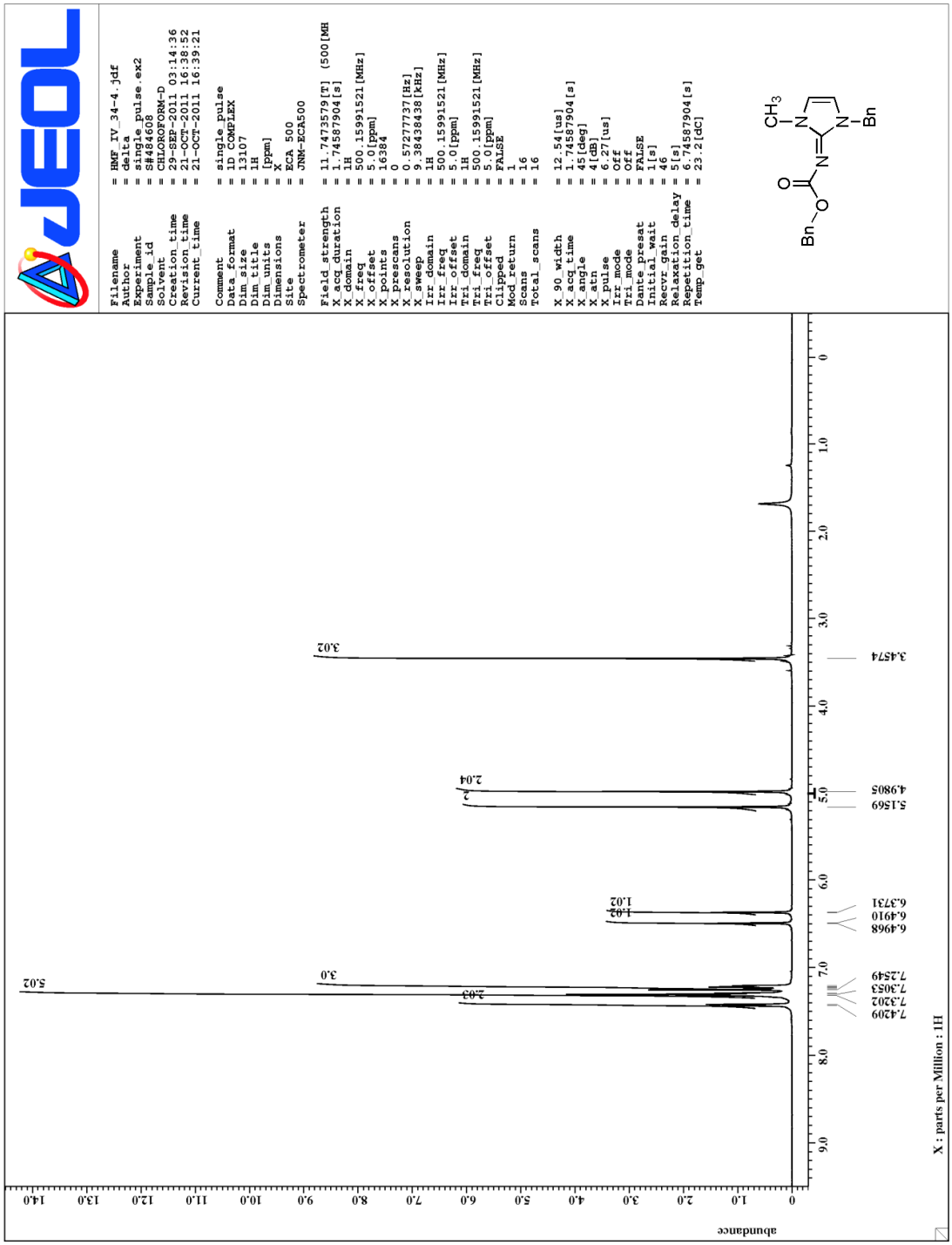

JEOL
Filename = HMF III_120-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#500510
Solvent = CHLOROFORM-D
Creation_time = 12-FEB-2011 04:56:59
Revision_time = 21-OCT-2011 15:48:01
Current_time = 21-OCT-2011 15:48:52
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 25.5[dC]
CH3
Bn
Bn
Bn
3.5662
5.2943
5.1993
7.4312
7.3247
7.2549
7.2469
7.2423
5.04
3.02
2.04
2.01
1.03
0.99
3.0
2.0
abundance
X : parts per Million : 1H

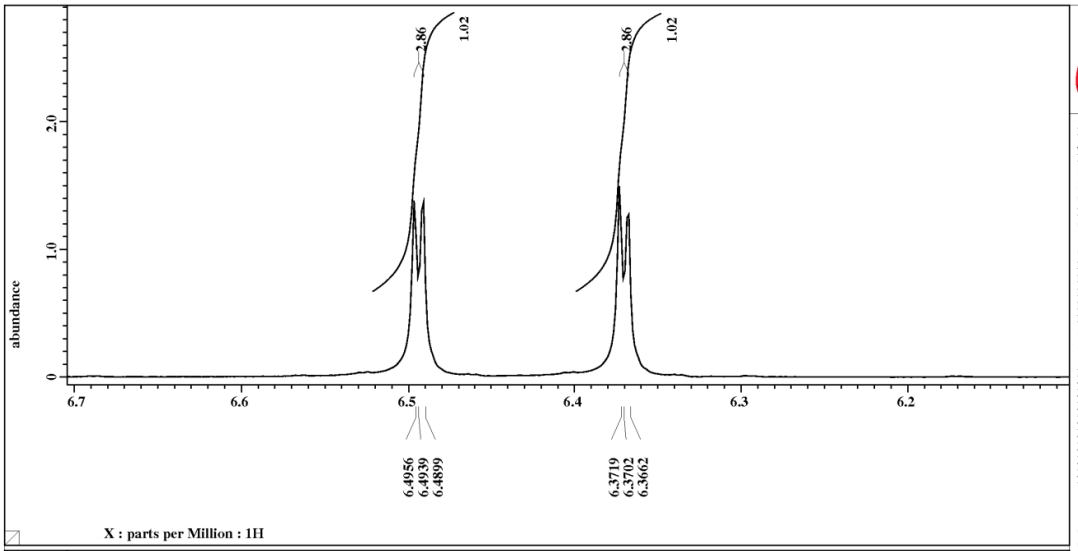

JEOL
Filename = HMF III_120-6.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#500510
Solvent = CHLOROFORM-D
Creation_time = 12-FEB-2011 04:56:59
Revision_time = 21-OCT-2011 15:53:32
Current_time = 21-OCT-2011 15:54:26
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 25.5[dC]
CH3
N
N
Bn
O
N
O
O
Bn
abundance
X : parts per Million : 1H
8.02
7.45
6.87
5.04
3.02
2.04
2.01
1.03
0.99
7.4461
7.4384
7.4312
7.3385
7.3316
7.3247
7.3213
7.3098
7.2824
7.2675
7.2549
7.2469
7.2423
7.2366
7.2274
7.2114
7.2091
7.1976
7.1953
7.1930
7.1896
7.1358
7.1335
7.0533
7.0453
7.0373

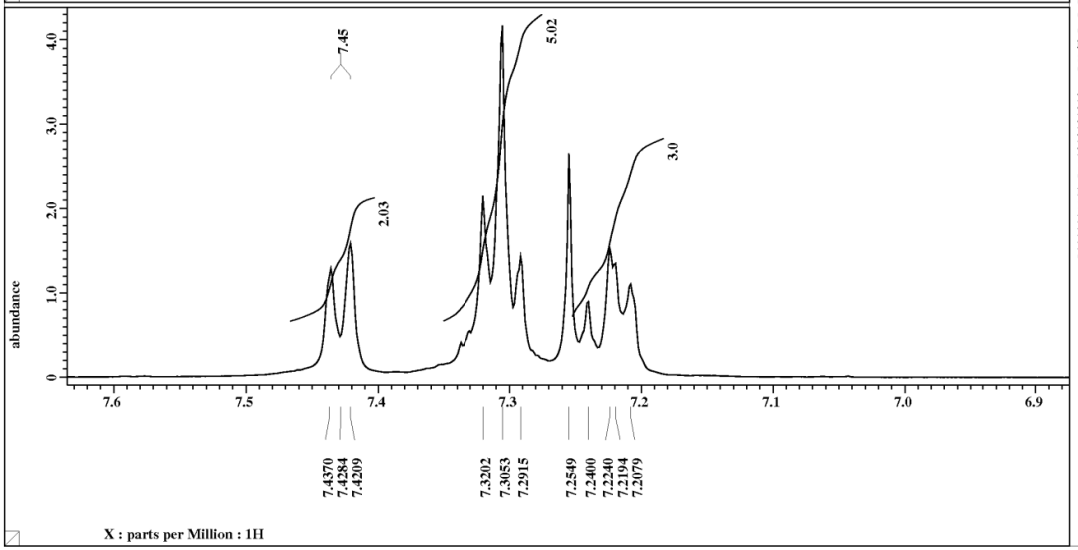

Filename = HMF III_120-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#502240
Solvent = CHLOROFORM-D
Creation_time = 12-FEB-2011 05:06:42
Revision_time = 27-JUL-2011 22:05:18
Current_time = 27-JUL-2011 22:05:59
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 110
Total_scans = 110
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 25.9[dC]
CH3
N
N
Bn
O
N
O
Bn
abundance
X : parts per Million : 13C
31.0474
47.0051
67.1786
76.8696
77.1271
77.3846
109.2617
110.1202
123.1209
127.5276
127.6516
127.9568
128.1190
128.3574
128.8725
153.4051
159.0136


Filename = HMF_III_120-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#502240
Solvent = CHLOROFORM-D
Creation_time = 12-FEB-2011 05:06:42
Revision_time = 21-OCT-2011 15:56:07
Current_time = 21-OCT-2011 15:56:41
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 110
Total_scans = 110
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 25.9[dC]
CH3
N
N
Bn
O
N
O
O
Bn
abundance
0
0.1
0.2
0.3
0.4
140.0
130.0
120.0
110.0
137.8862
135.3109
131.4574
130.1697
128.8725
128.3574
128.1190
127.9568
127.6516
127.5276
123.1209
123.0160
110.1202
109.2617
X : parts per Million : 13C

APPENDIX 25
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-2-benzyloxycarbonylimino-3-methyl-2,3-dihydro-1*H*-imidazole (**95b**)


Filename = HMF_IV_34-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#484608
Solvent = CHLOROFORM-D
Creation_time = 29-SEP-2011 03:14:36
Revision_time = 21-OCT-2011 16:38:52
Current_time = 21-OCT-2011 16:39:21
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.2[dC]
CH3
N
N
Bn
N
O
O
Bn
abundance
X : parts per Million : 1H
5.02
3.0
2.03
1.02
3.02
2.04
3.4574
5.1569
4.9805
6.4968
6.4910
6.3731
7.4209
7.3202
7.3053
7.2549



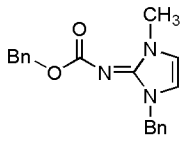


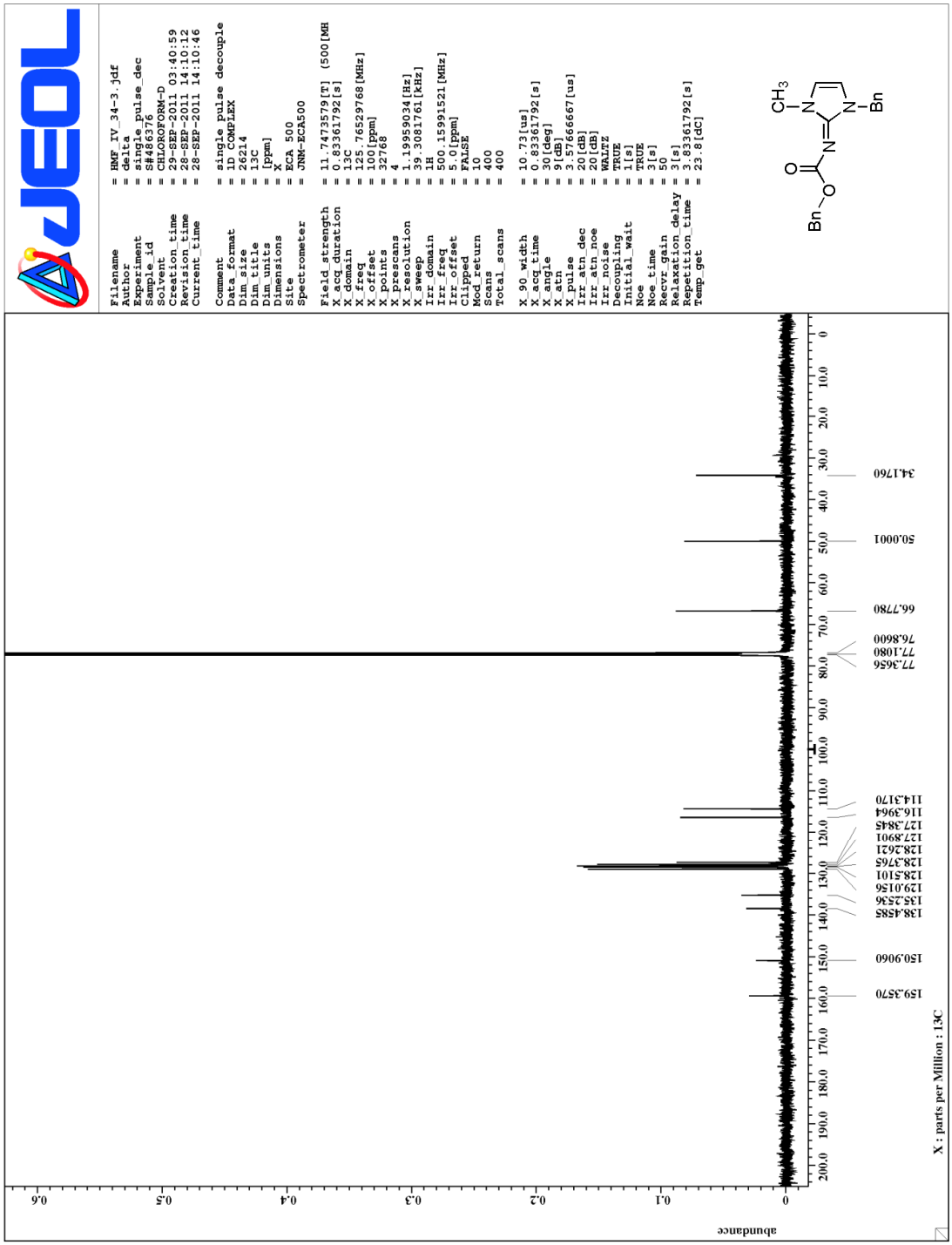

```
Filename          = HMF_IV_34-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#484608
Solvent           = CHLOROFORM-D
Creation_time     = 29-SEP-2011 03:14:36
Revision_time     = 21-OCT-2011 16:40:05
Current_time      = 21-OCT-2011 16:40:40

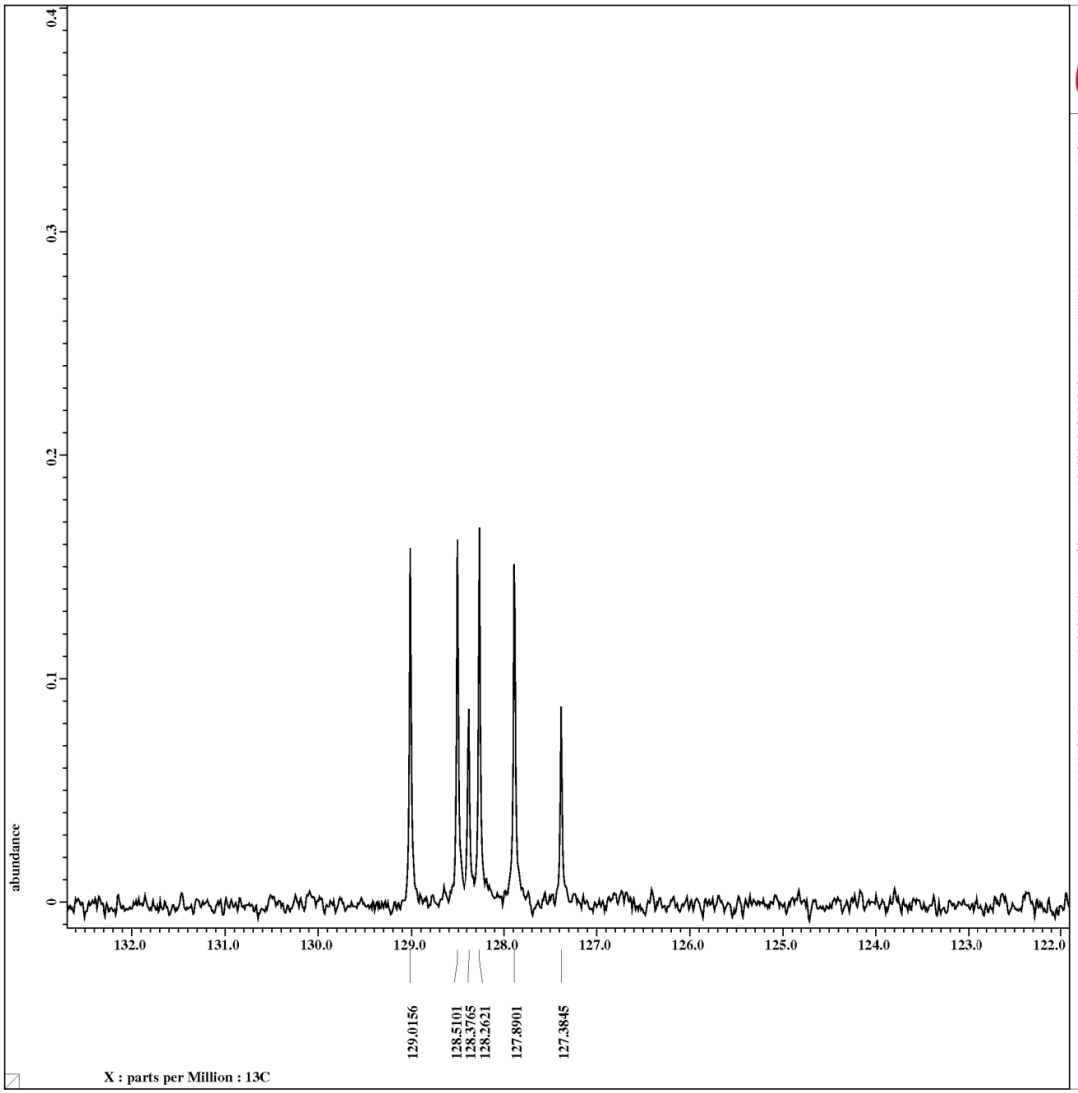
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 23.2[dC]
```




JEOL
Filename = HMF_IV_34-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#486376
Solvent = CHLOROFORM-D
Creation_time = 29-SEP-2011 03:40:59
Revision_time = 28-SEP-2011 14:10:12
Current_time = 28-SEP-2011 14:10:46
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 400
Total_scans = 400
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 23.8[dC]
CH3
N
N
Bn
O
O
Bn
abundance
X : parts per Million : 13C
34.1760
50.0001
66.7780
76.8600
77.1080
77.3656
114.3170
116.3964
127.3845
127.8901
128.2621
128.3765
128.5101
129.0156
135.2536
138.4585
150.9060
159.3570



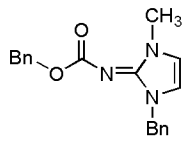
JEOL

```
Filename          = HMF_IV_34-2.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#486376
Solvent           = CHLOROFORM-D
Creation_time     = 29-SEP-2011 03:40:59
Revision_time     = 21-OCT-2011 16:35:38
Current_time      = 21-OCT-2011 16:36:05

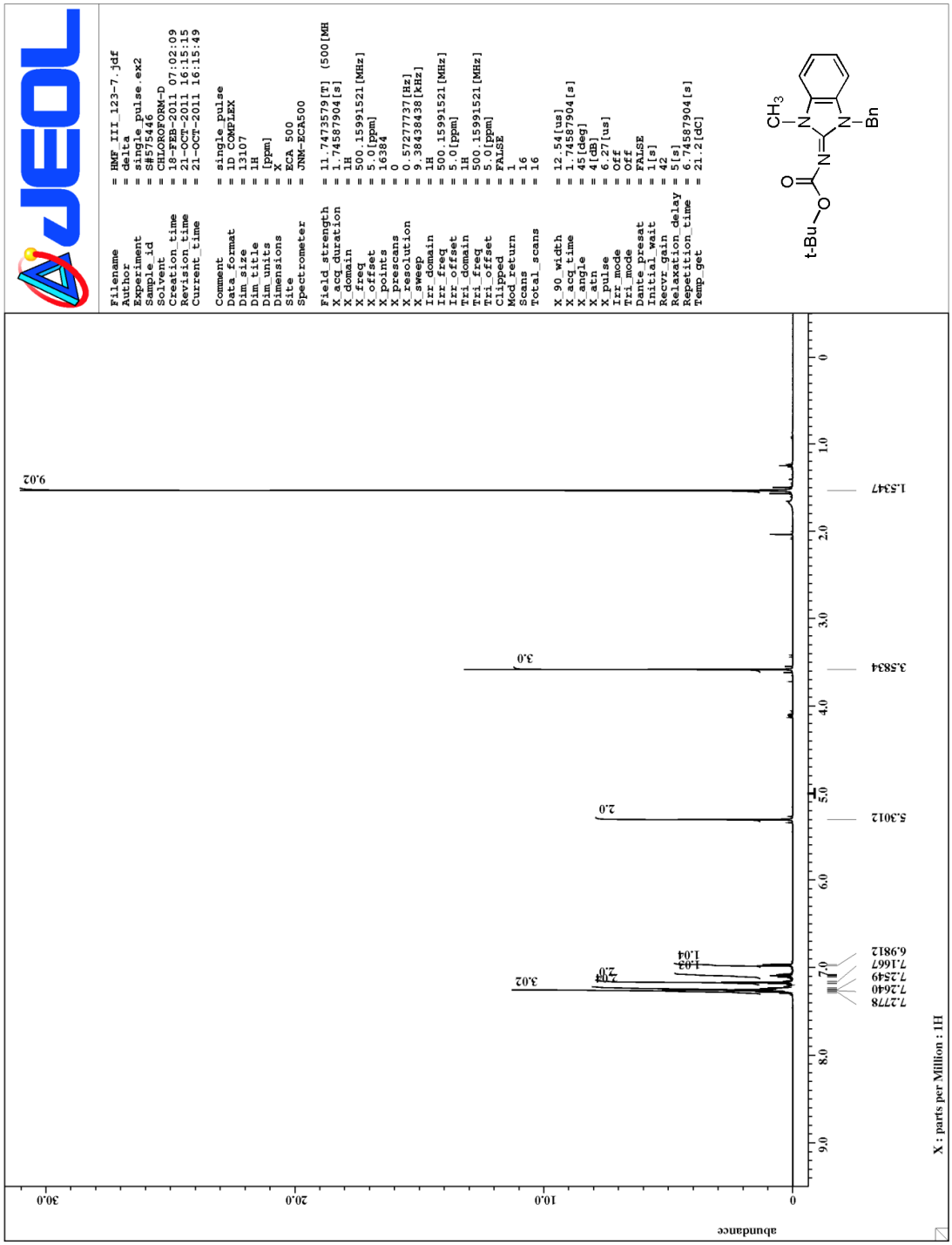
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

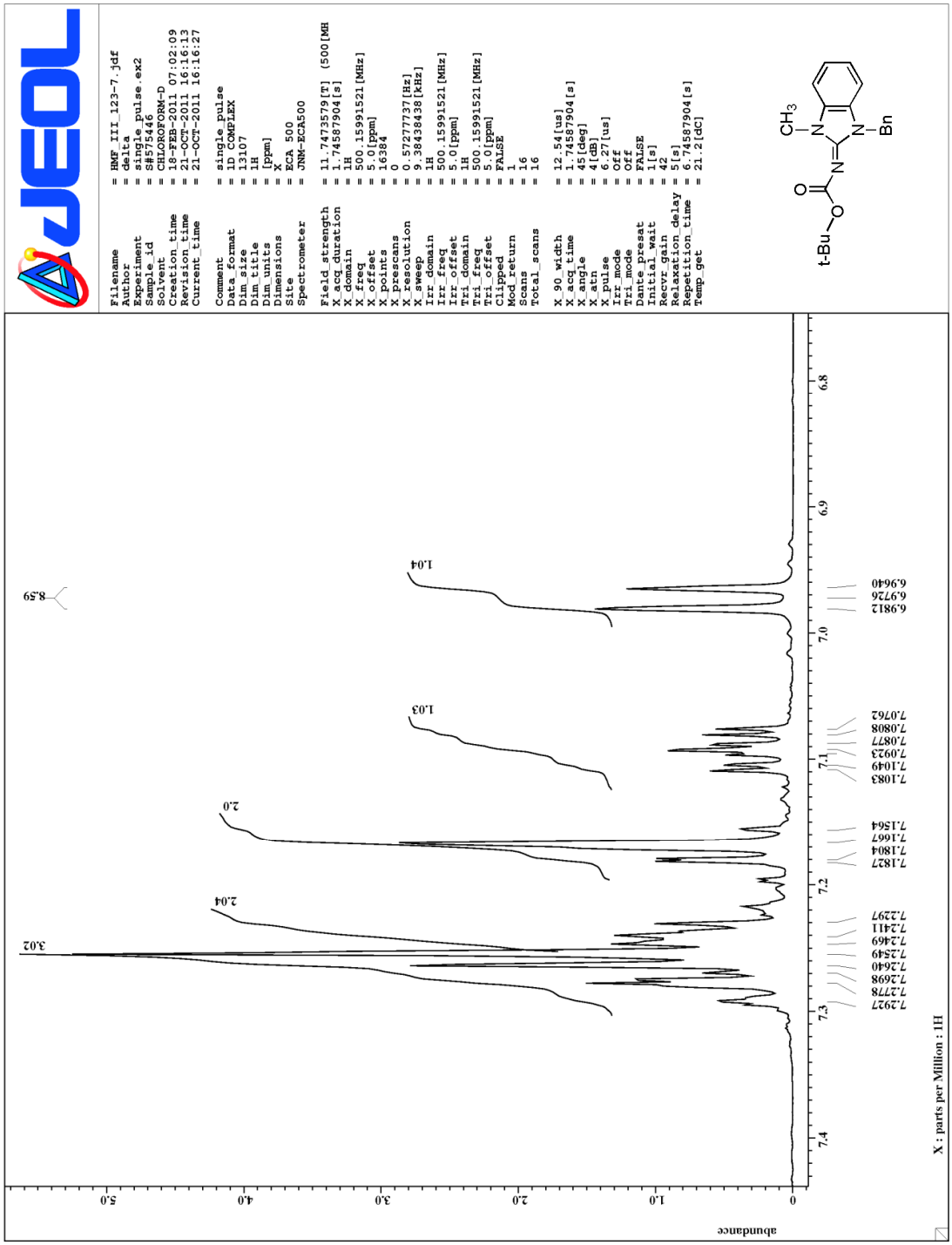
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 400
Total_scans       = 400

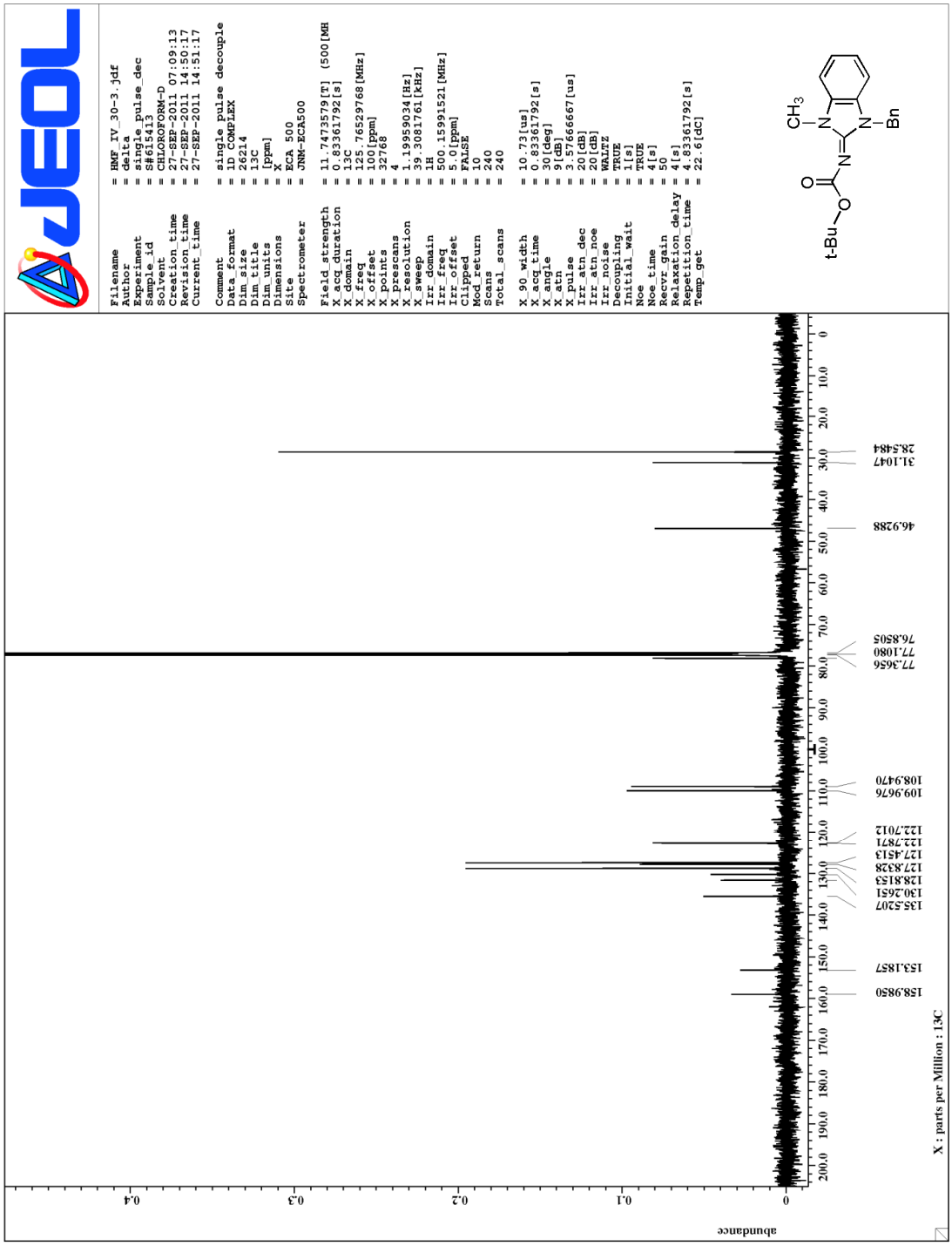
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 3[s]
Recvr_gain        = 50
Relaxation_delay  = 3[s]
Repetition_time   = 3.83361792[s]
Temp_get          = 23.8[dC]
```

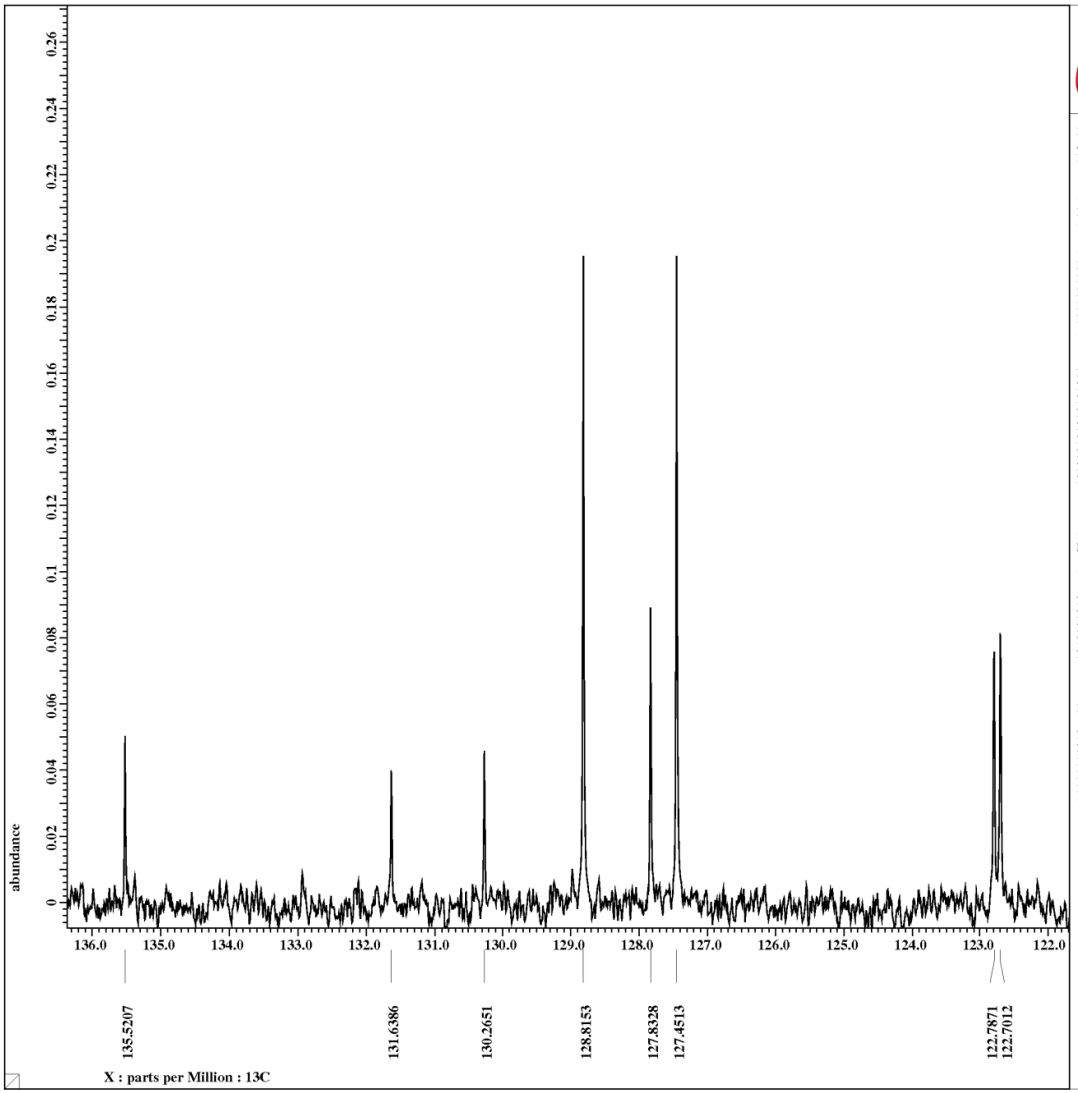


APPENDIX 26
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-2-*tert*-butylcarbonylimino-3-methyl-1,3-dihydro-2*H*-benzimidazole (**96a**)


Filename = RMF_III_123-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#575446
Solvent = CHLOROFORM-D
Creation_time = 18-FEB-2011 07:02:09
Revision_time = 21-OCT-2011 16:15:15
Current_time = 21-OCT-2011 16:15:49
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
CH3
N
N
Bn
O
N
O
t-Bu
abundance
X : parts per Million : 1H
9.02
3.0
2.0
2.0
1.04
3.02
1.5347
3.5834
5.3012
6.9812
7.1667
7.2549
7.2640
7.2778


JEOL
Filename = HMF III_123-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#575446
Solvent = CHLOROFORM-D
Creation_time = 18-FEB-2011 07:02:09
Revision_time = 21-OCT-2011 16:16:13
Current_time = 21-OCT-2011 16:16:27
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.159991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384438[kHz]
Irr_domain = 1H
Irr_freq = 500.159991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.159991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.2[dC]
CH3
N
N
Bn
O
N
O
t-Bu
abundance
X : parts per Million : 1H
8.59
3.02
2.04
2.04
2.0
1.04
1.03
7.2927
7.2778
7.2698
7.2640
7.2549
7.2469
7.2411
7.2297
7.1827
7.1804
7.1667
7.1564
7.1083
7.1049
7.0923
7.0877
7.0808
7.0762
6.9812
6.9726
6.9640


Filename = HMF_IV_30-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#615413
Solvent = CHLOROFORM-D
Creation_time = 27-SEP-2011 07:09:13
Revision_time = 27-SEP-2011 14:50:17
Current_time = 27-SEP-2011 14:51:17
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 240
Total_scans = 240
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.6[dC]
CH3
N
N
Bn
O
N
O
t-Bu
abundance
X : parts per Million : 13C
158.9850
153.1857
135.5207
130.2651
128.8153
127.8328
127.4513
122.7871
122.7012
109.9676
108.9470
77.3656
77.1080
76.8505
46.9288
31.1047
28.5484

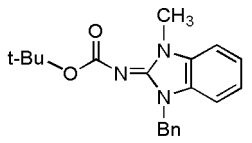



```
Filename          = HMF_IV_30-2.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#615413
Solvent           = CHLOROFORM-D
Creation_time     = 27-SEP-2011 07:09:13
Revision_time     = 21-OCT-2011 16:23:27
Current_time      = 21-OCT-2011 16:24:58

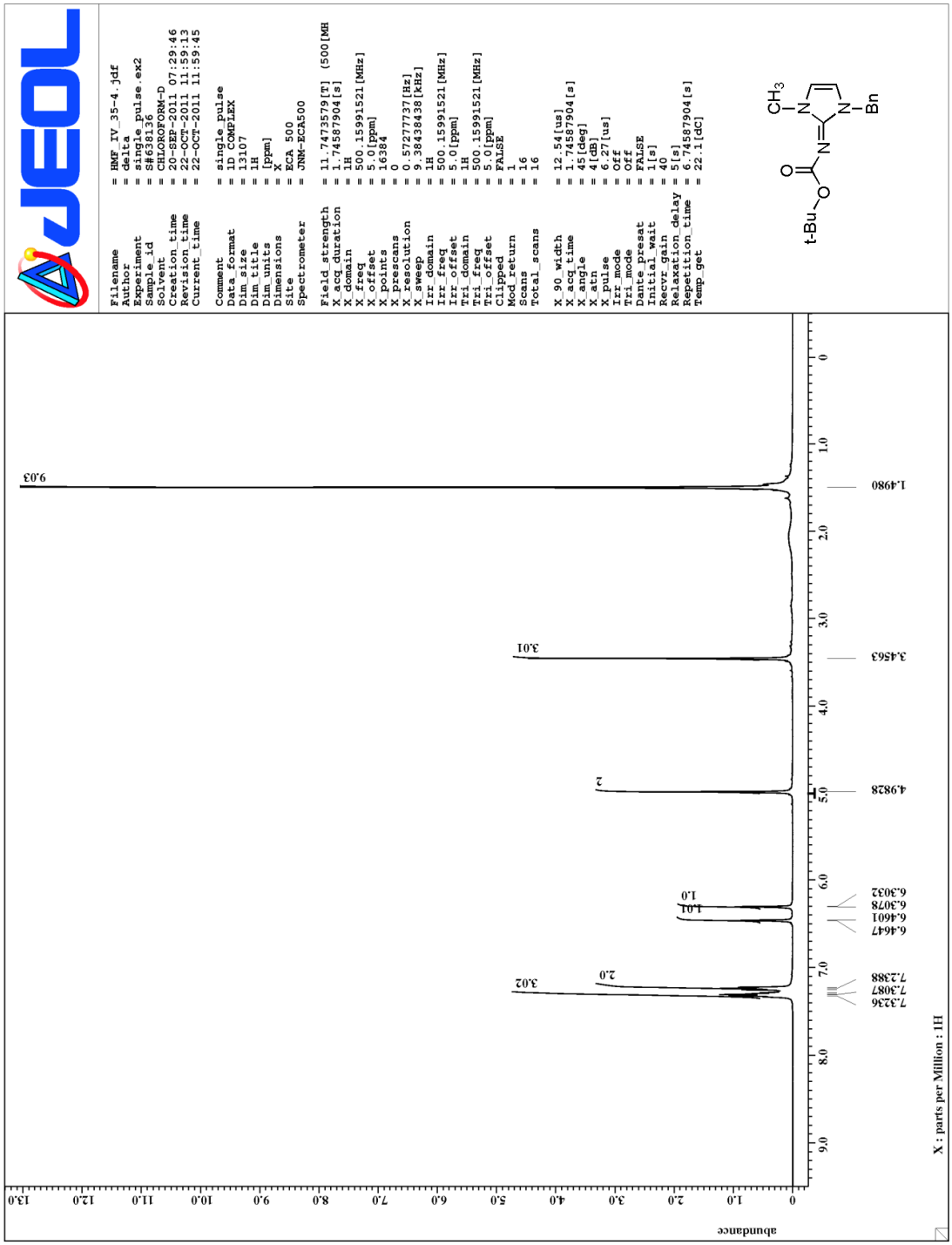
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

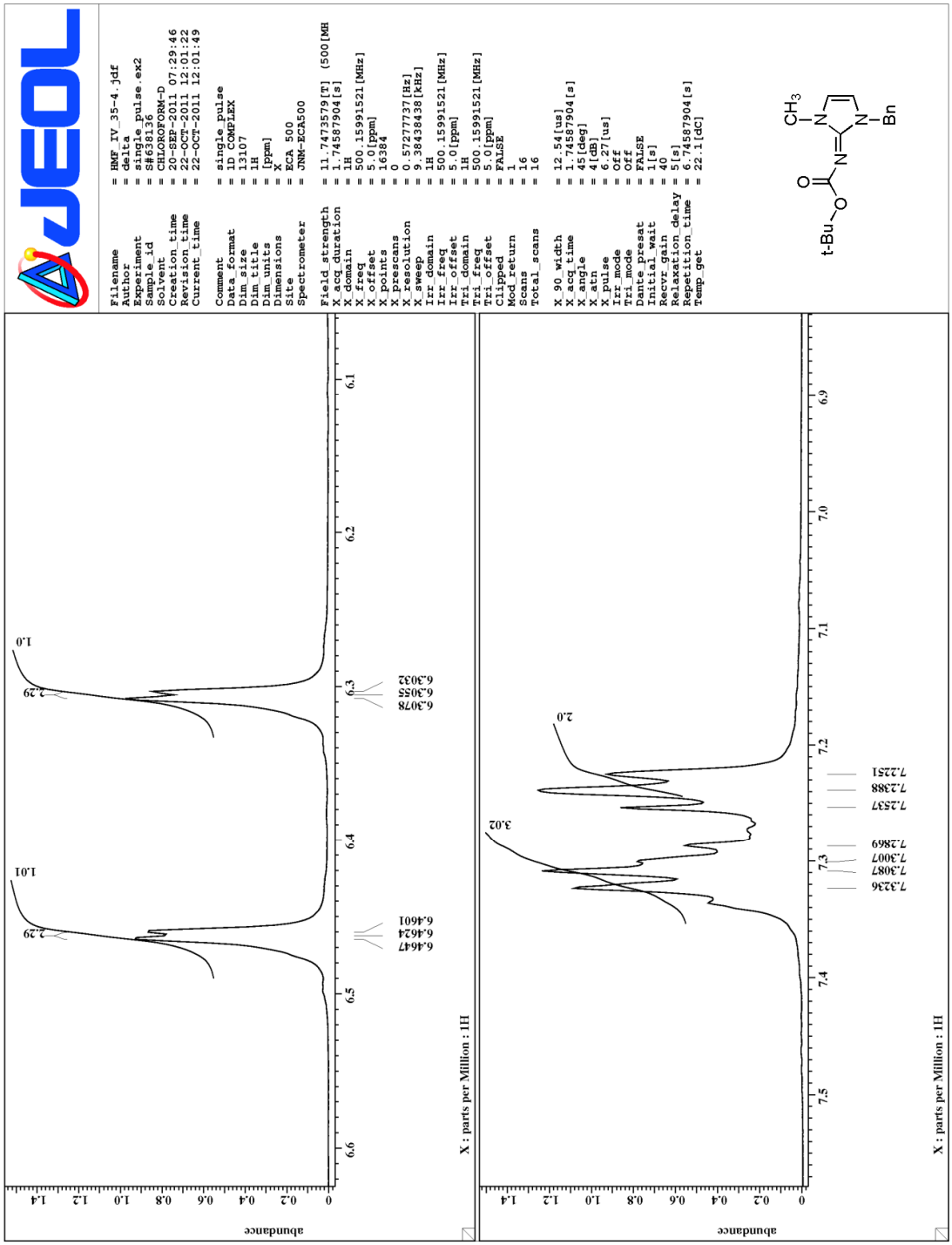
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 240
Total_scans       = 240

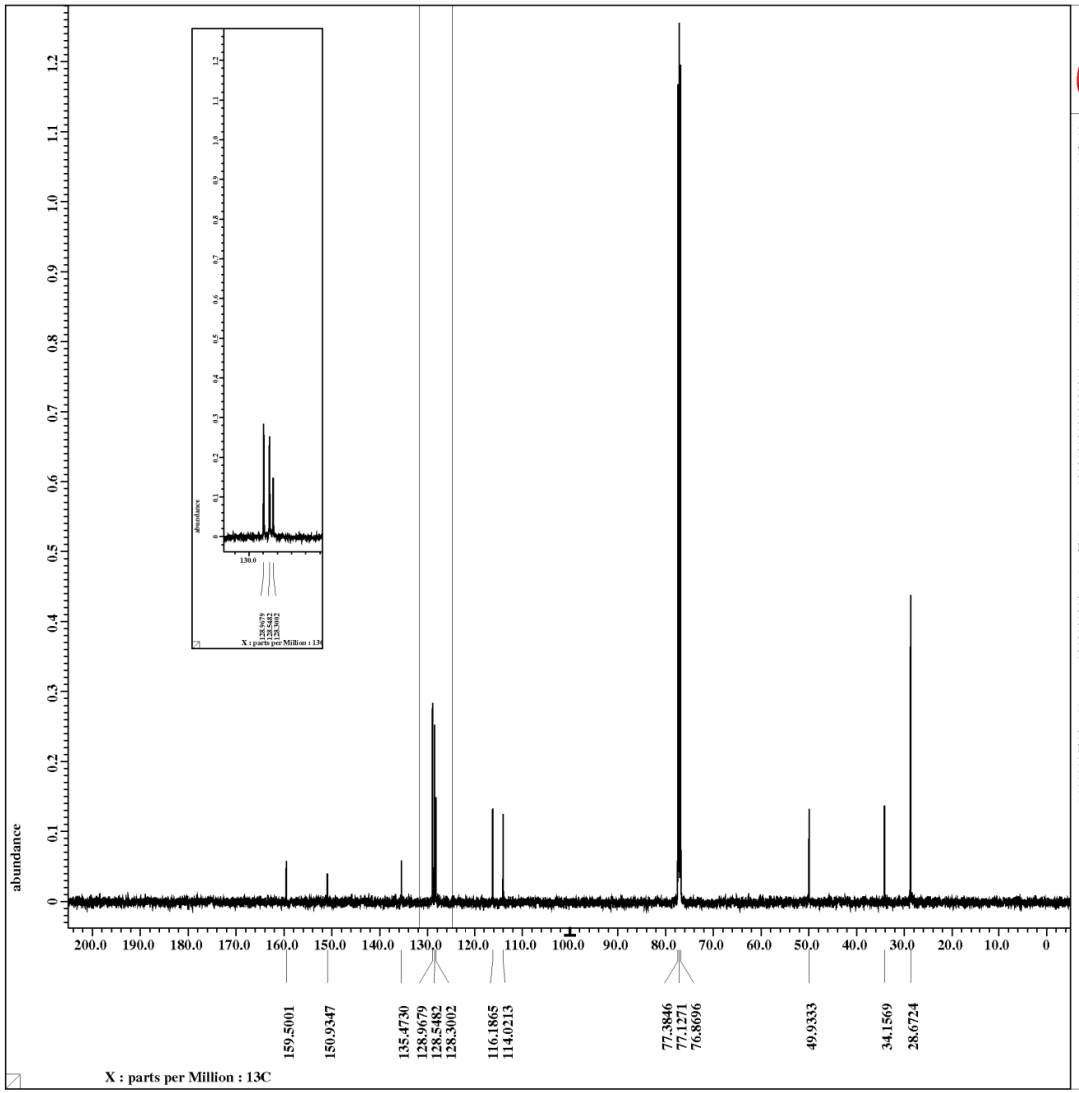
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 22.6[dC]
```



APPENDIX 27
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-2-*tert*-butylcarbonylimino-3-methyl-2,3-dihydro-1*H*-imidazole (**96b**)


Filename = HMF_IV_35-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#638136
Solvent = CHLOROFORM-D
Creation_time = 20-SEP-2011 07:29:46
Revision_time = 22-OCT-2011 11:59:13
Current_time = 22-OCT-2011 11:59:45
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.1[dC]
CH3
N
N
Bn
O
N
O
t-Bu
abundance
9.03
3.01
2
3.02
2.0
1.01
1.0
1.4980
3.4563
4.9828
6.4647
6.4601
6.3078
6.3032
7.3236
7.3087
7.2388
X : parts per Million : 1H


JEOL
Filename = HMF_IV_35-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#638136
Solvent = CHLOROFORM-D
Creation_time = 20-SEP-2011 07:29:46
Revision_time = 22-OCT-2011 12:01:22
Current_time = 22-OCT-2011 12:01:49
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384381[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.1[dC]
CH3
N
N
Bn
O
O
t-Bu
abundance
X : parts per Million : 1H
1.01
2.29
6.4647
6.4624
6.4601
6.3078
6.3055
6.3032
3.02
2.0
7.3236
7.3087
7.3007
7.2869
7.2537
7.2388
7.2251



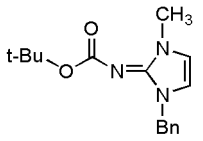
JEOL

```
Filename          = HMF_IV_35-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#639878
Solvent           = CHLOROFORM-D
Creation_time     = 20-SEP-2011 07:49:07
Revision_time     = 22-OCT-2011 11:52:51
Current_time      = 22-OCT-2011 11:55:16

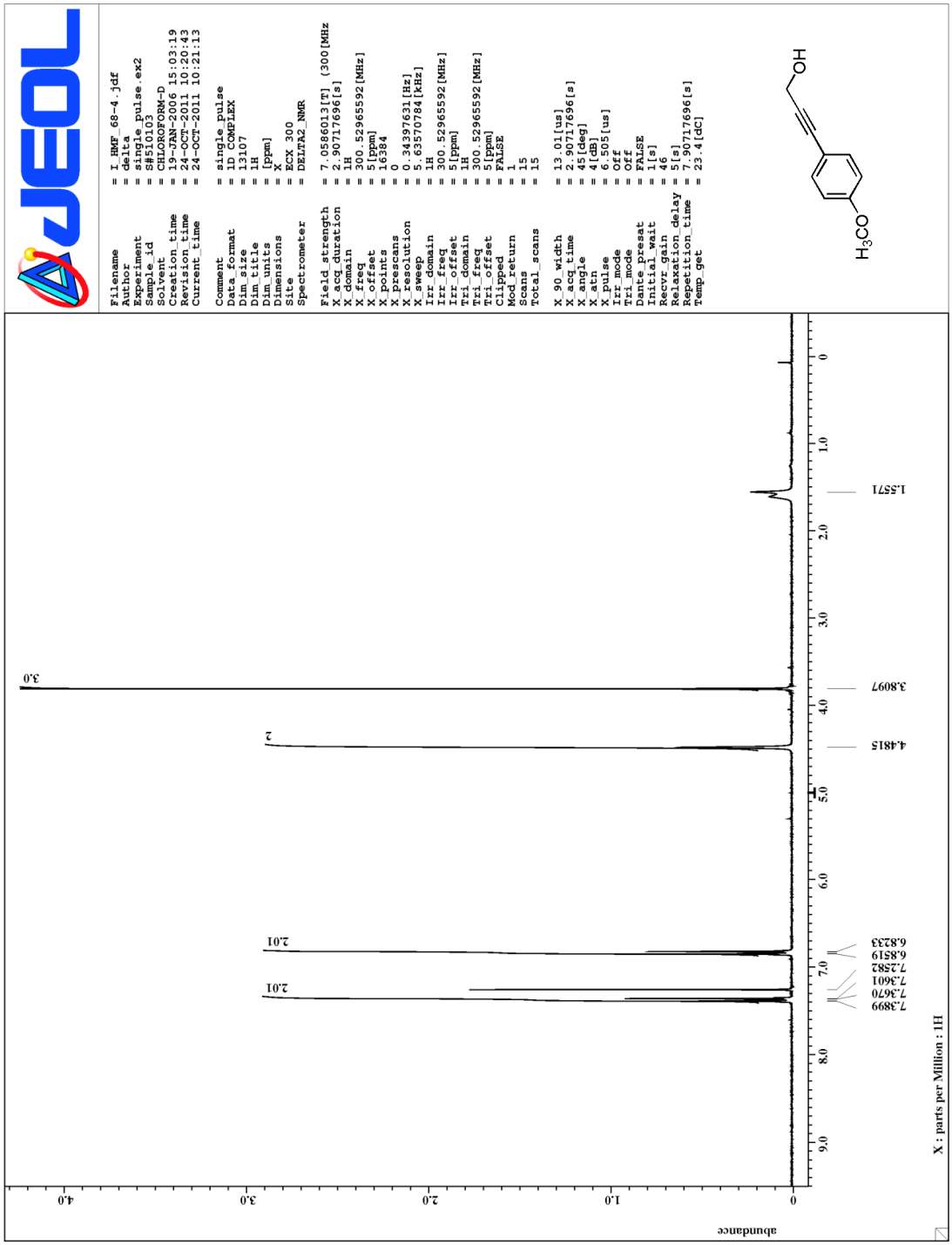
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

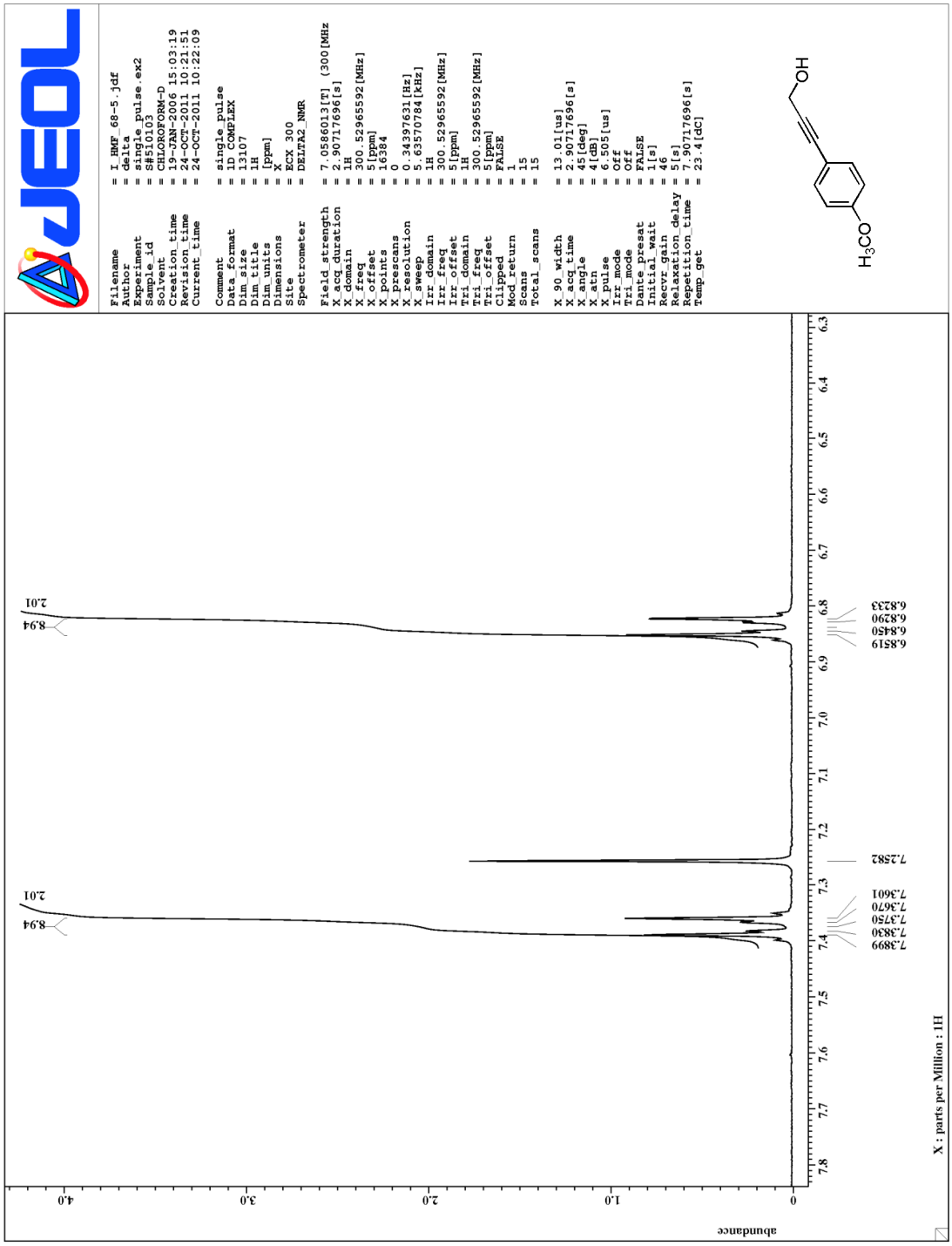
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 230.0
Total_scans       = 230.0

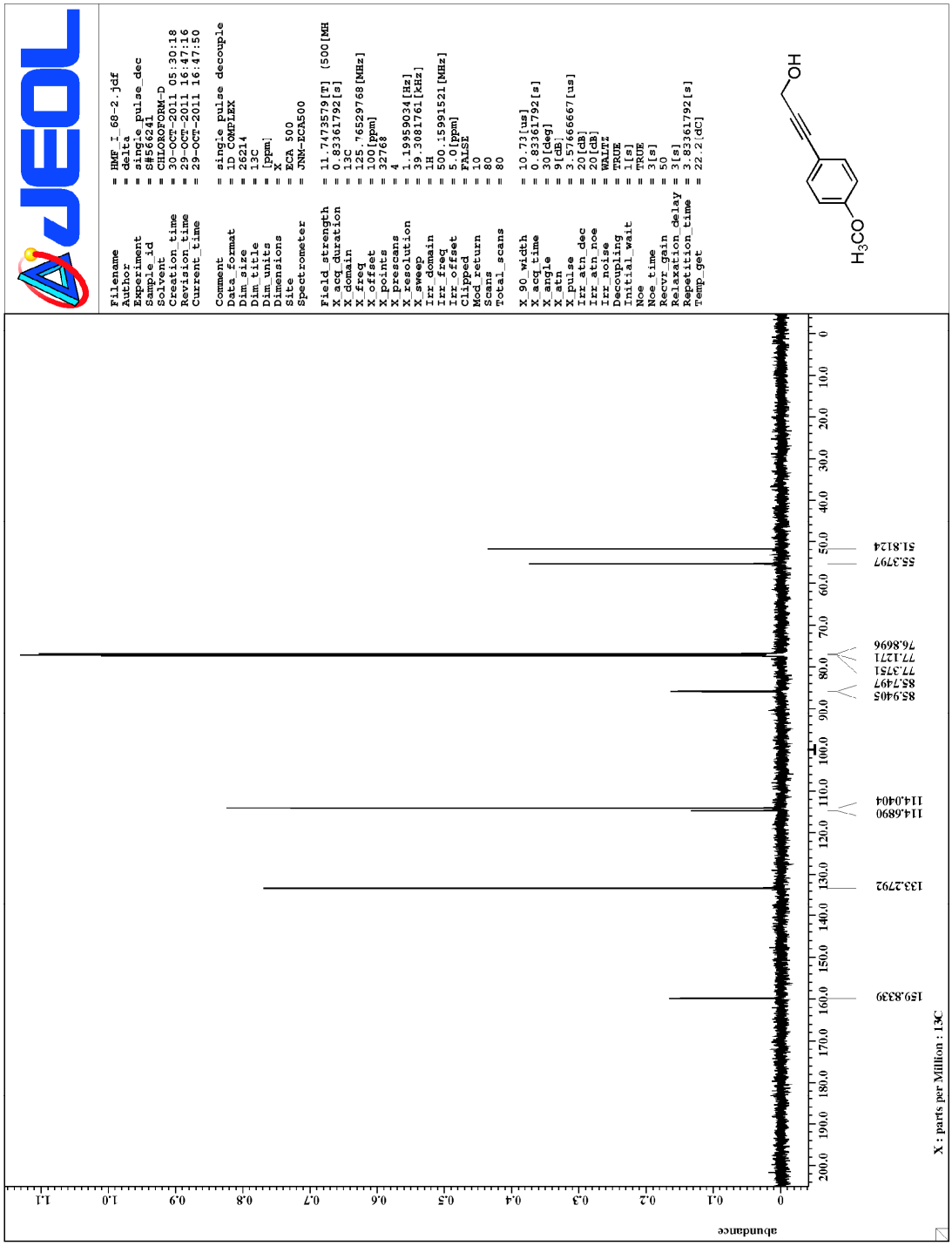
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 22.7[dC]
```



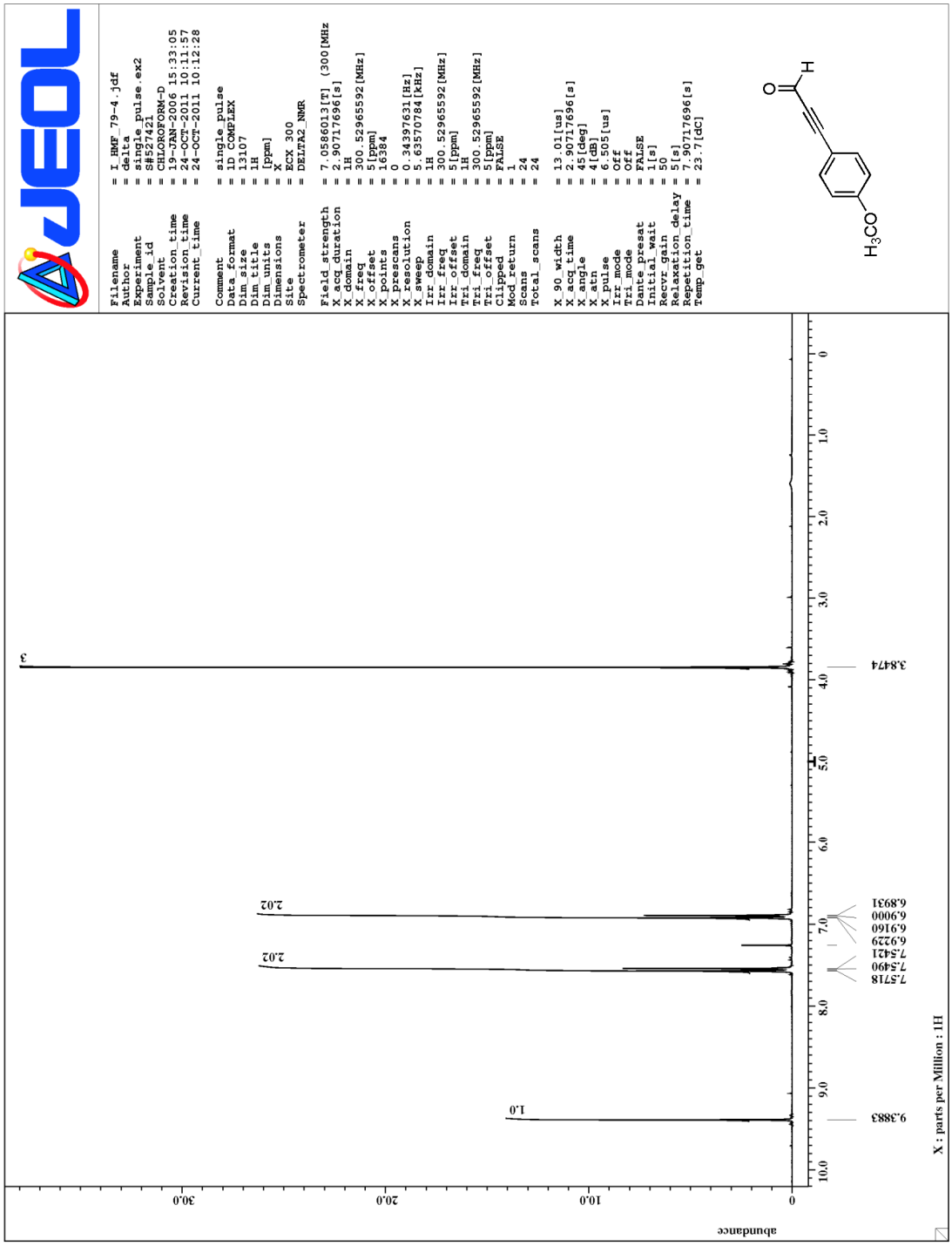
APPENDIX 28
^{1}H and ^{13}C NMR Spectra of 3-(4-methoxyphenyl)prop-2-yn-1-ol (**116**)

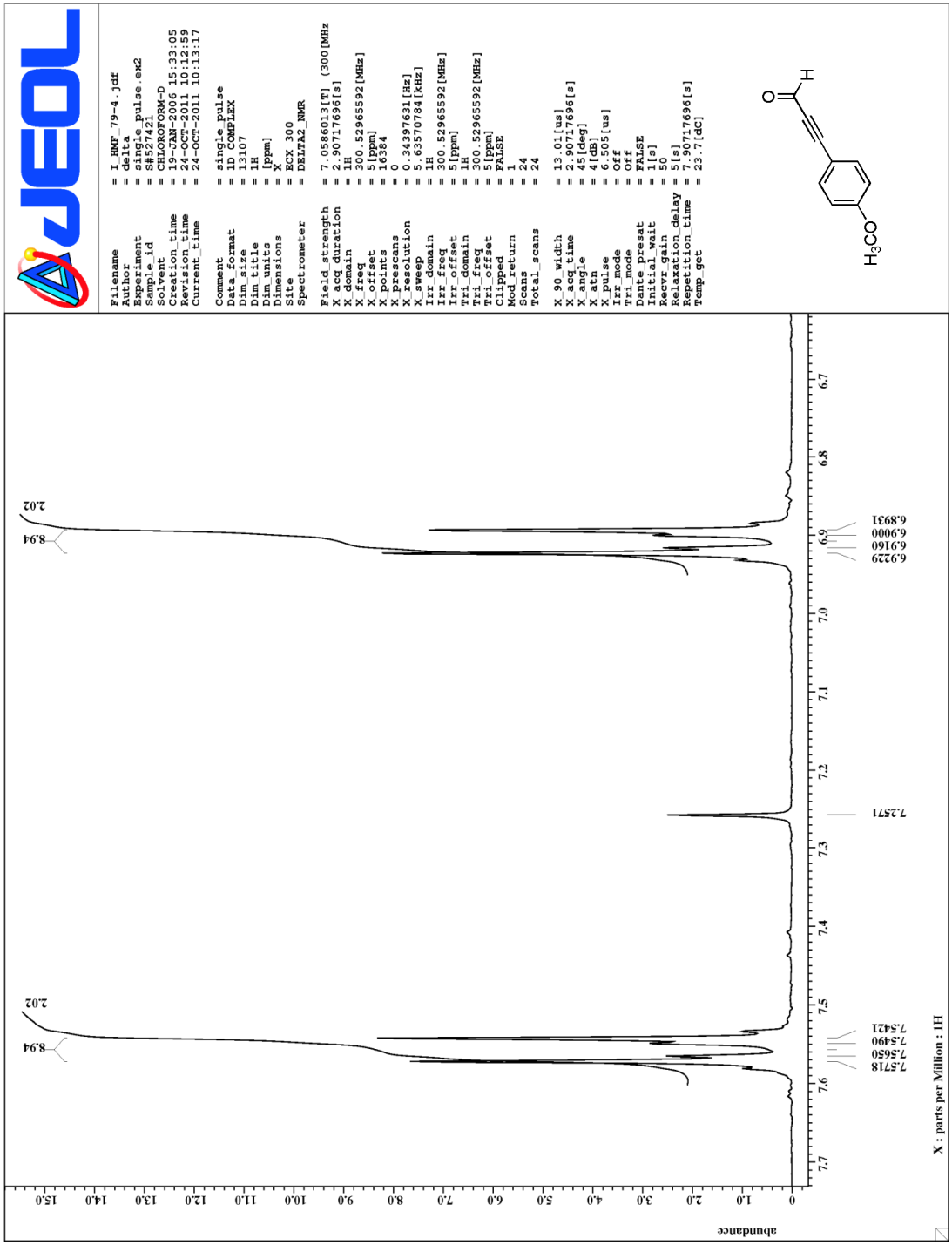

Filename = I_HMF_68-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#5101103
Solvent = CHLOROFORM-D
Creation_time = 19-JAN-2006 15:03:19
Revision_time = 24-OCT-2011 10:20:43
Current_time = 24-OCT-2011 10:21:13
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 15
Total_scans = 15
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.4[dC]
OH
H3CO
abundance
X : parts per Million : 1H
3.0
2
2.01
2.01
1.5571
3.8097
4.4815
7.3899
7.3670
7.3601
7.2582
6.8519
6.8233

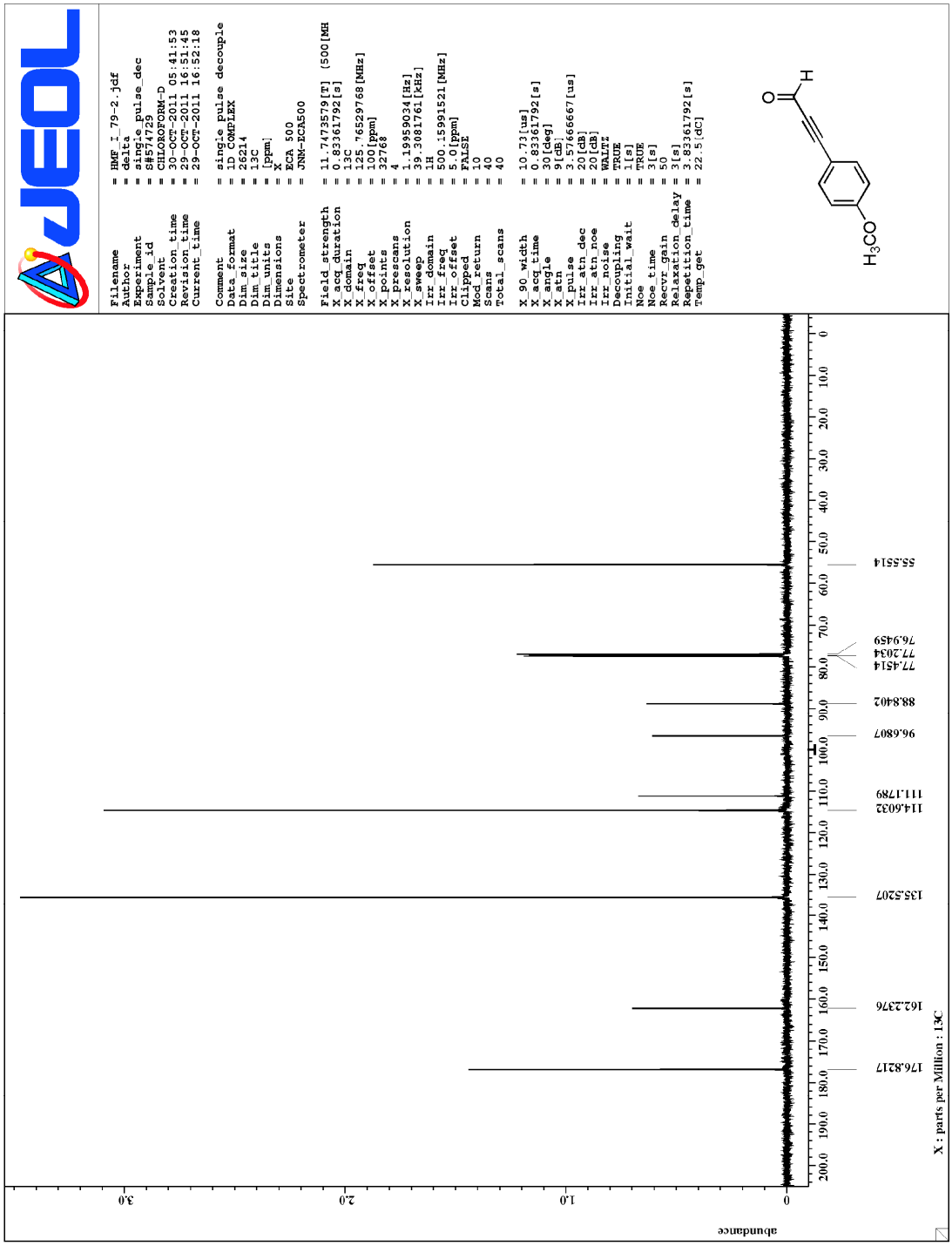

JEOL
Filename = I_HMF_68-5.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#5101103
Solvent = CHLOROFORM-D
Creation_time = 19-JAN-2006 15:03:19
Revision_time = 24-OCT-2011 10:21:51
Current_time = 24-OCT-2011 10:22:09
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 15
Total_scans = 15
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.4[dC]
OH
H3CO
abundance
X : parts per Million : 1H
8.94
2.01
8.94
2.01
7.3899
7.3830
7.3750
7.3679
7.3601
7.2582
6.8519
6.8450
6.8390
6.8333


JEOL
Filename = HMF_I_68-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#566241
Solvent = CHLOROFORM-D
Creation_time = 30-OCT-2011 05:30:18
Revision_time = 29-OCT-2011 16:47:16
Current_time = 29-OCT-2011 16:47:50
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 80
Total_scans = 80
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.5716666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.2[dC]
OH
H3CO
abundance
X : parts per Million : 13C
159.8339
133.2792
114.6890
114.0404
85.9405
85.7497
77.3751
77.1271
76.8696
55.3797
51.8124

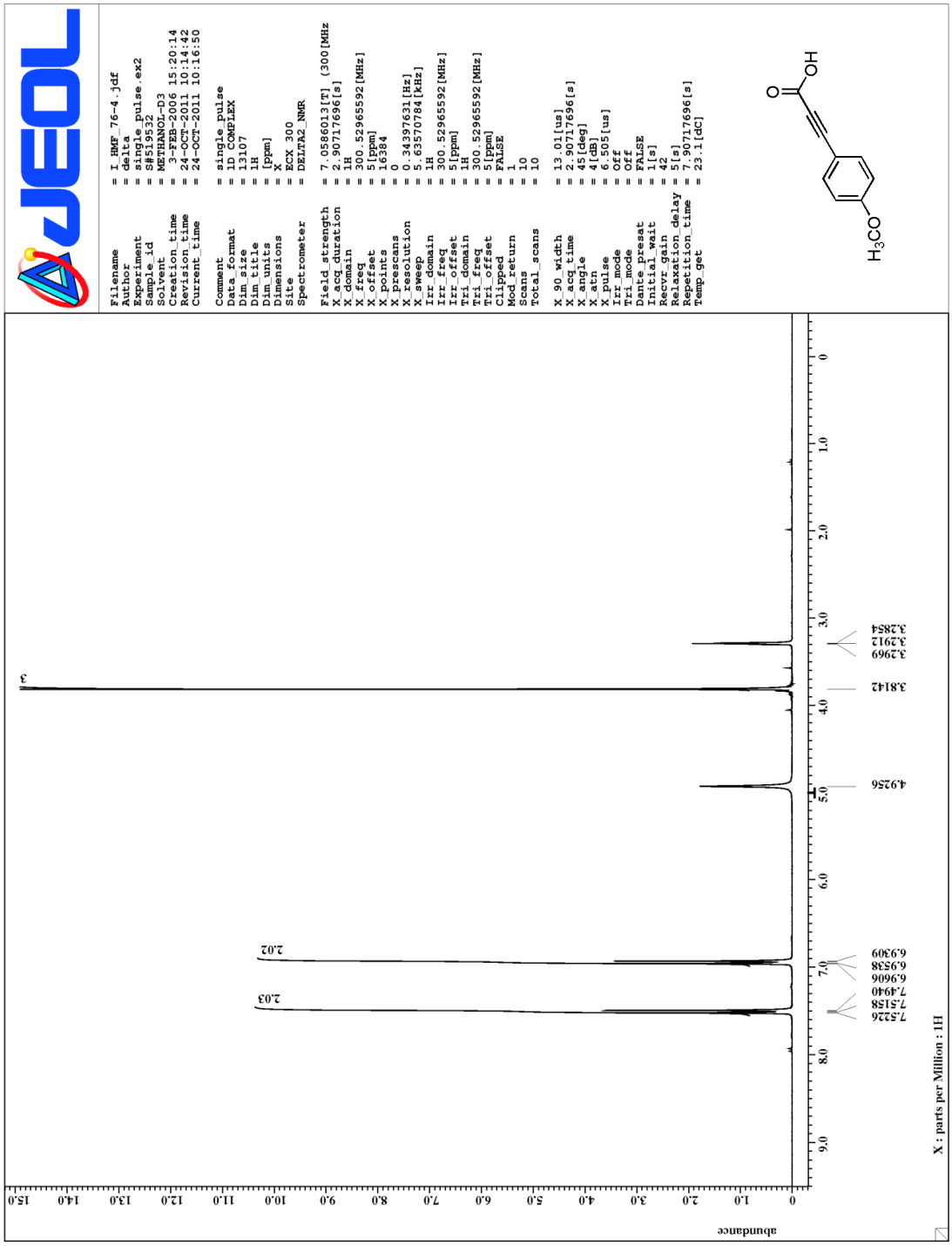
APPENDIX 29
^{1}H and ^{13}C NMR Spectra of 3-(4-methoxyphenyl)prop-2-ynal (**117**)

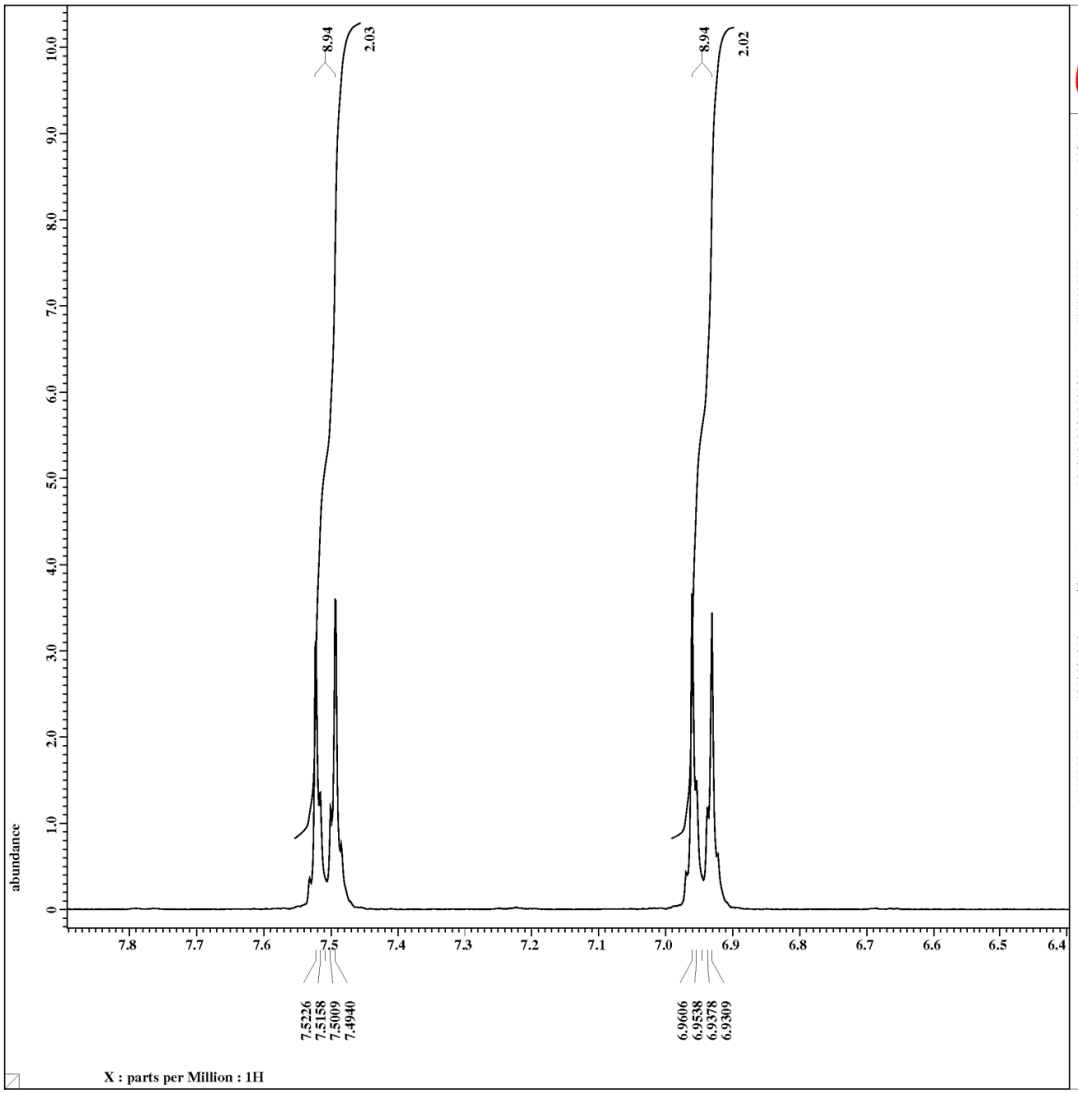

JEOL
Filename = I_HMF_79-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#527421
Solvent = CHLOROFORM-D
Creation_time = 19-JAN-2006 15:33:05
Revision_time = 24-OCT-2011 10:11:57
Current_time = 24-OCT-2011 10:12:28
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIMPA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.7[dC]
H
O
H3CO
abundance
X : parts per Million : 1H
3
2.02
2.02
1.0
9.3883
7.5718
7.5490
7.5421
6.9229
6.9160
6.9000
6.8931
3.8474


JEOL
Filename = I_HMF_79-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#527421
Solvent = CHLOROFORM-D
Creation_time = 19-JAN-2006 15:33:05
Revision_time = 24-OCT-2011 10:12:59
Current_time = 24-OCT-2011 10:13:17
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIFAZ_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.7[dC]
O
H
H3CO
abundance
X : parts per Million : 1H
8.94
2.02
8.94
2.02
7.5718
7.5650
7.5490
7.5421
7.2571
6.9229
6.9160
6.9000
6.8931


JEOL
Filename = HMF_I_79-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#574729
Solvent = CHLOROFORM-D
Creation_time = 30-OCT-2011 05:41:53
Revision_time = 29-OCT-2011 16:51:45
Current_time = 29-OCT-2011 16:52:18
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = EQA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 40
Total_scans = 40
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 91[dB]
X_pulse = 3.5716666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.5[dC]
H
O
H3CO
abundance
3.0
2.0
1.0
0
X : parts per Million : 13C
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
176.8217
162.2376
135.5207
114.6032
111.1789
96.6807
88.8402
77.4514
77.2034
76.9459
55.5514

APPENDIX 30
^{1}H and ^{13}C NMR Spectra of 3-(4-methoxyphenyl)prop-2-ynoic acid (**118**)


Filename = I_HMF_76-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#519532
Solvent = METHANOL-D3
Creation_time = 3-FEB-2006 15:20:14
Revision_time = 24-OCT-2011 10:14:42
Current_time = 24-OCT-2011 10:16:50
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 10
Total_scans = 10
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.1[dC]
H3CO
OH
abundance
X : parts per Million : 1H
3
2.02
2.03
7.5226
7.5158
7.4940
6.9606
6.9538
6.9309
4.9256
3.8142
3.2969
3.2912
3.2854



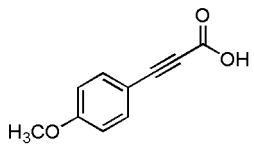
JEOL

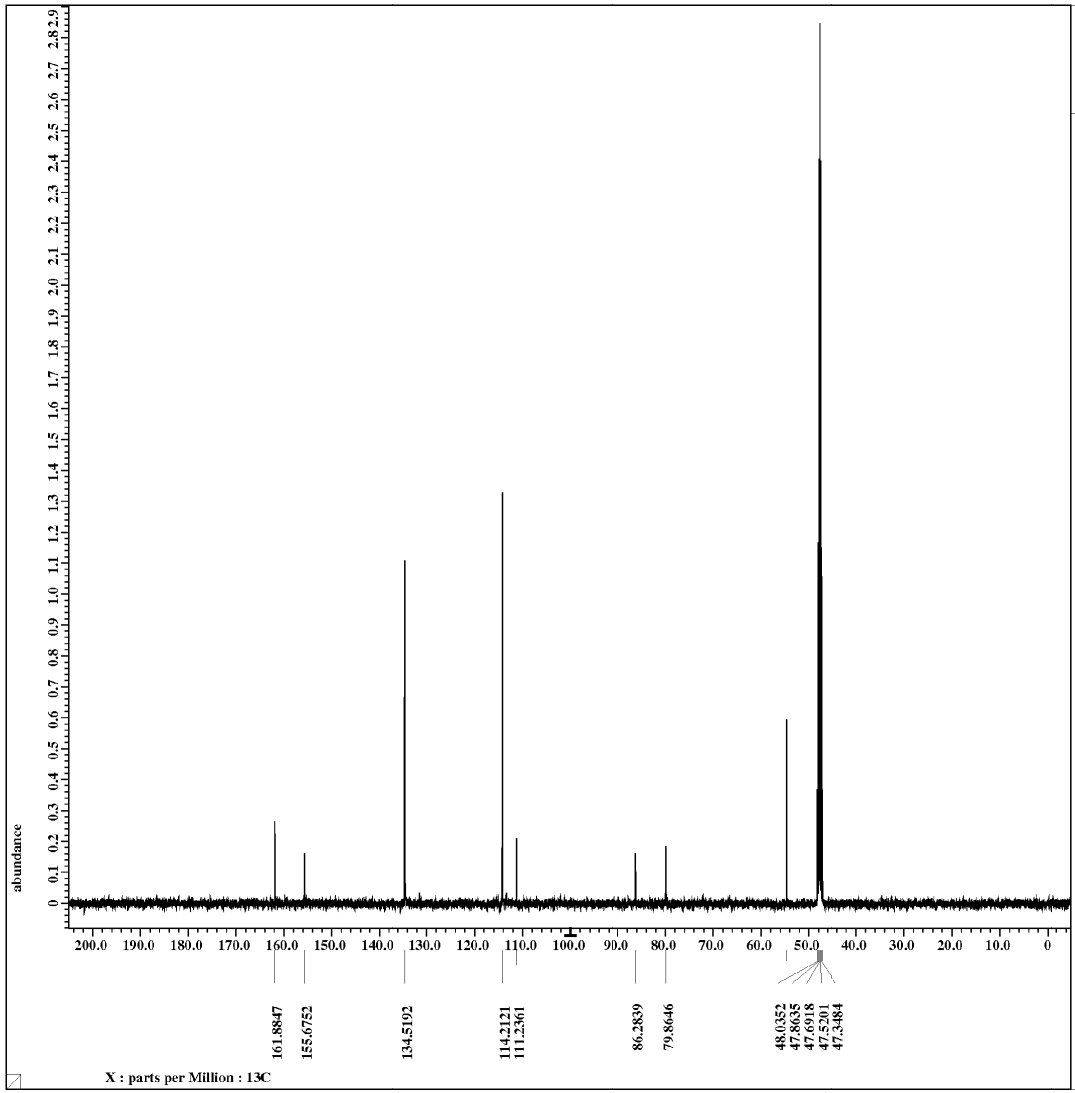
```
Filename          = I_HMF_76-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#519532
Solvent           = METHANOL-D3
Creation_time     =  3-FEB-2006 15:20:14
Revision_time     = 24-OCT-2011 10:17:54
Current_time      = 24-OCT-2011 10:18:23

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 300
Spectrometer      = DELTA2_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 2.90717696[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.34397631[Hz]
X_sweep           = 5.63570784[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 300.52965592[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 10
Total_scans       = 10

X_90_width        = 13.01[us]
X_acq_time        = 2.90717696[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.505[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 5[s]
Repetition_time   = 7.90717696[s]
Temp_get          = 23.1[dC]
```



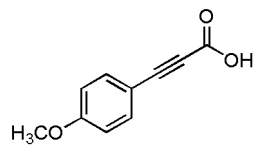



```
Filename          = HMF_I_76-2.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#581857
Solvent           = METHANOL-D3
Creation_time     = 30-OCT-2011 05:55:41
Revision_time     = 29-OCT-2011 16:54:39
Current_time      = 29-OCT-2011 16:55:37

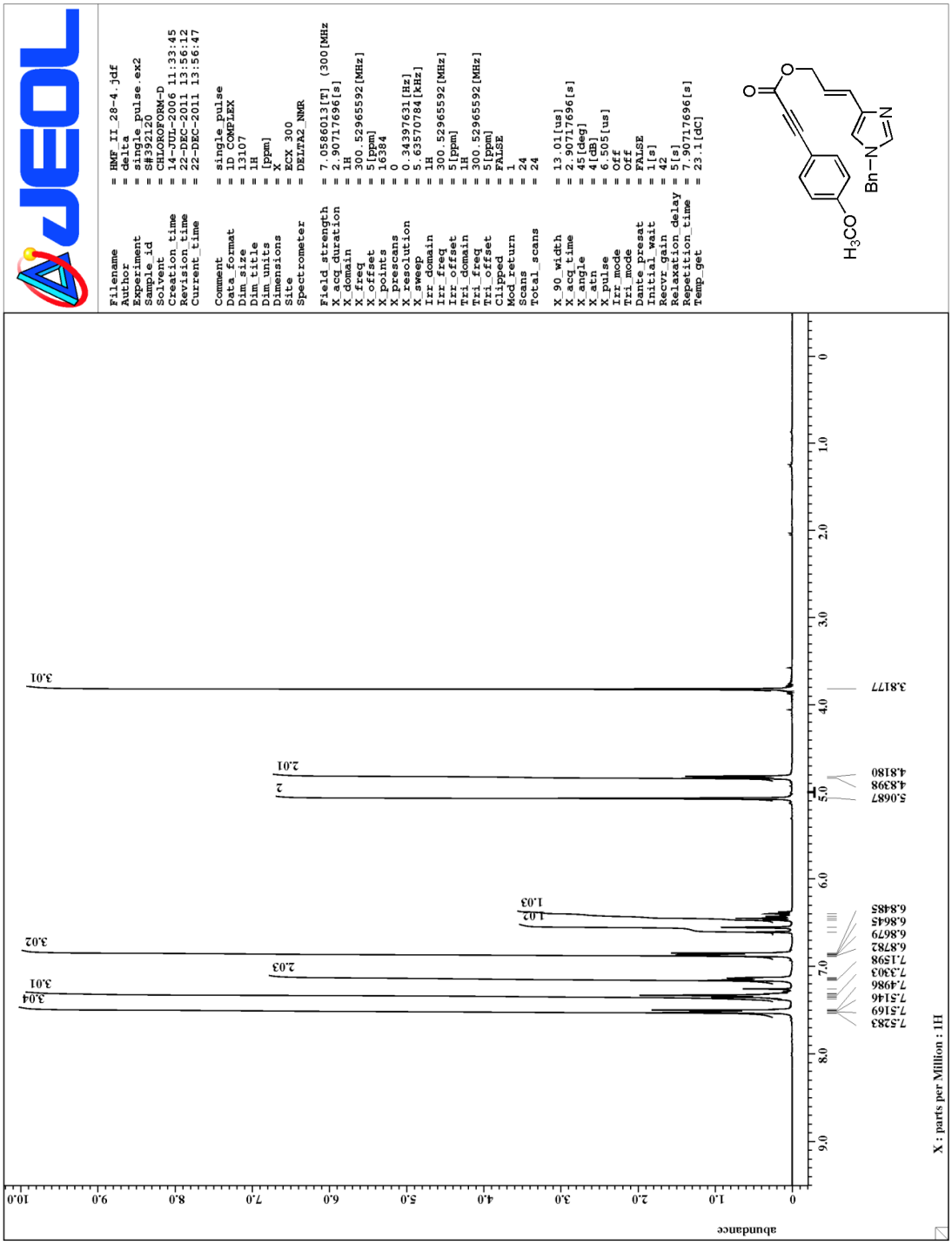
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

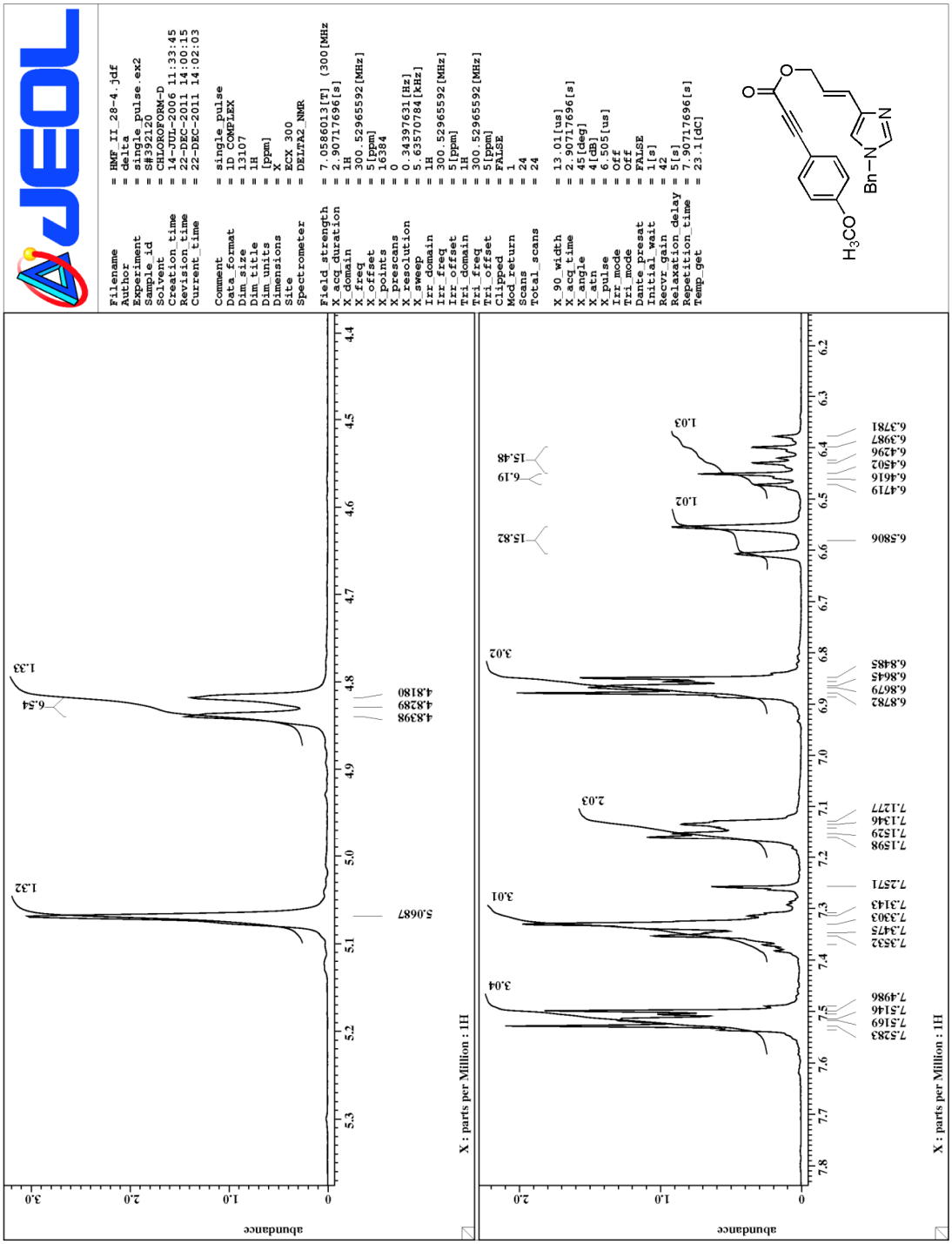
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 70
Total_scans       = 70

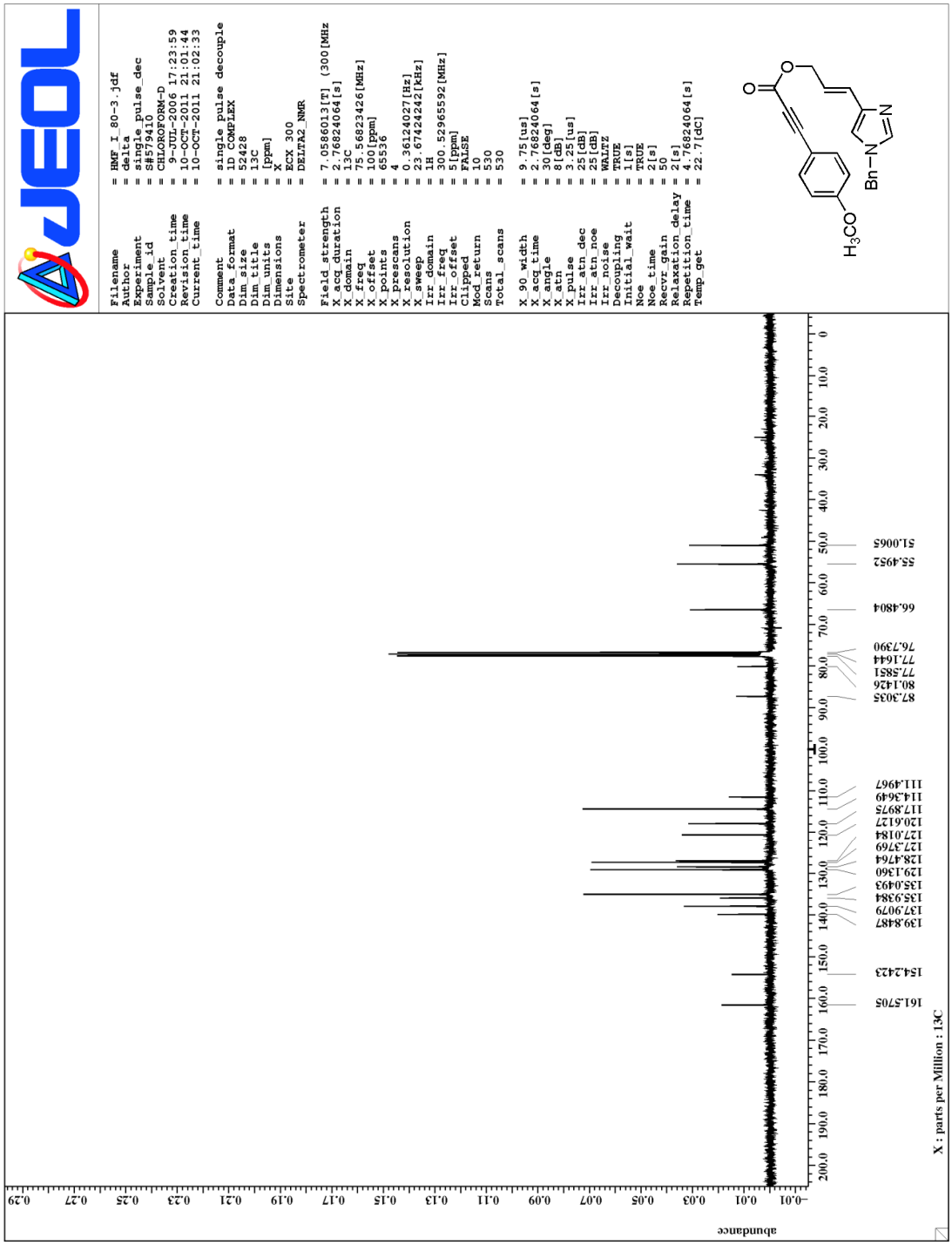
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 3[s]
Recvr_gain        = 50
Relaxation_delay  = 3[s]
Repetition_time   = 3.83361792[s]
Temp_get          = 22.9[dC]
```

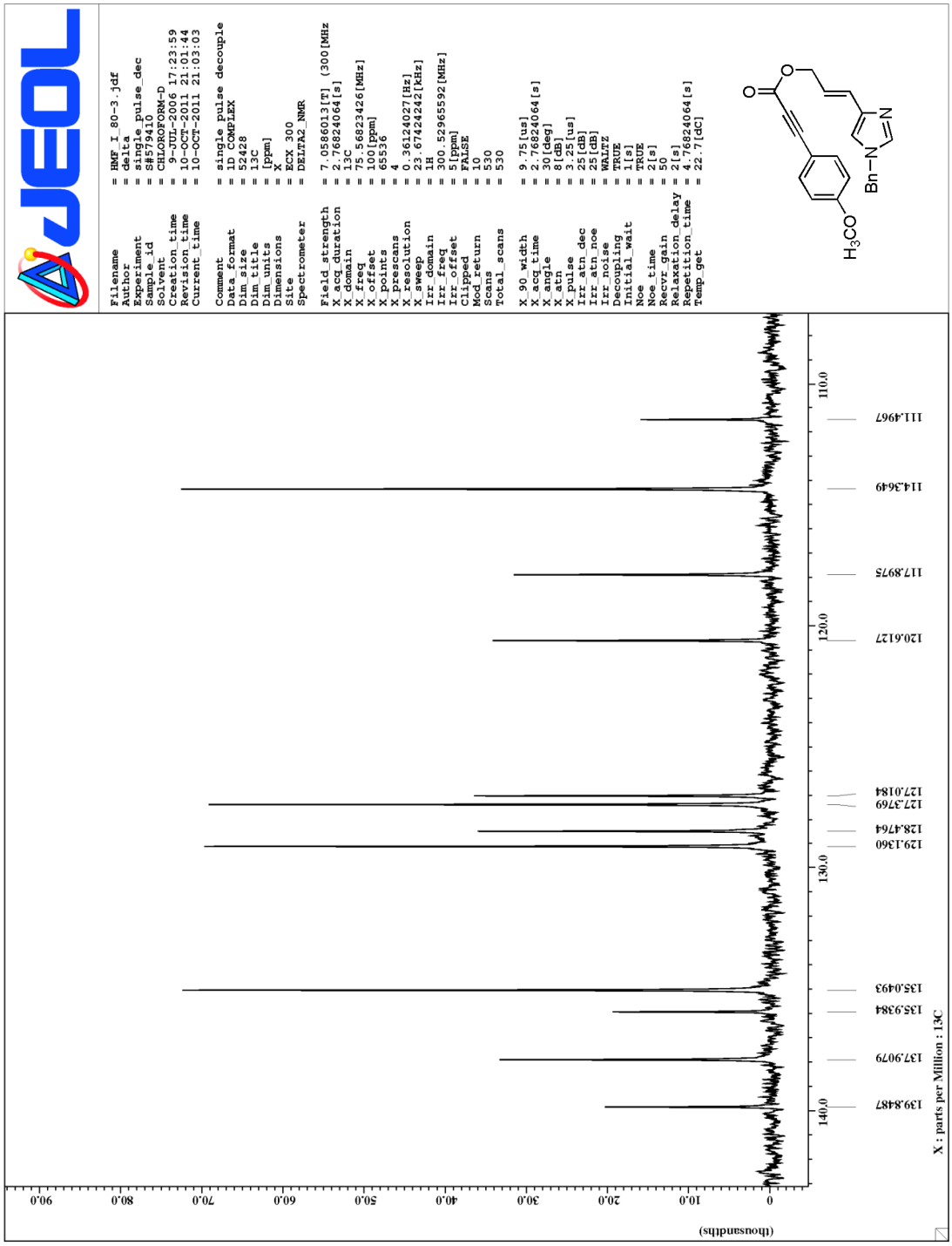


APPENDIX 31
^{1}H and ^{13}C NMR Spectra of
(2*E*)-3-(1-Benzyl-1*H*-imidazol-4-yl)prop-2-enyl 3-(4-methoxyphenyl)propynoate (**107**)

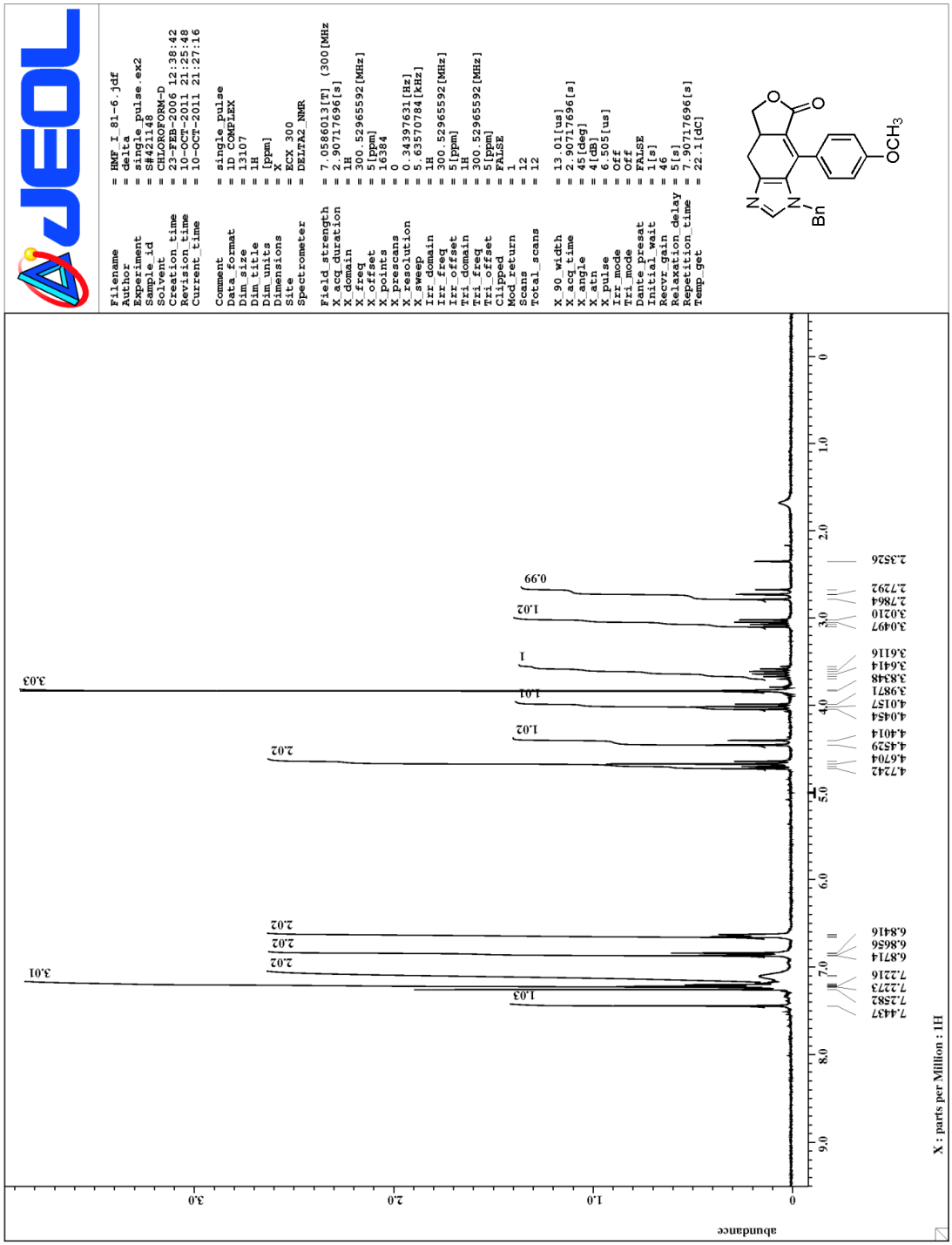

JEOL
Filename = HMF II_28-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#392120
Solvent = CHLOROFORM-D
Creation_time = 14-JUL-2006 11:33:45
Revision_time = 22-DEC-2011 13:56:12
Current_time = 22-DEC-2011 13:56:47
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.1[dC]
H3CO
Bn–N
N
abundance
X : parts per Million : 1H
3.01
2.01
2
3.02
2.03
3.04
3.01
1.02
1.03
3.8177
5.0687
4.8398
4.8180
7.5283
7.5169
7.5146
7.4986
7.3303
7.1598
6.8782
6.8679
6.8645
6.8485

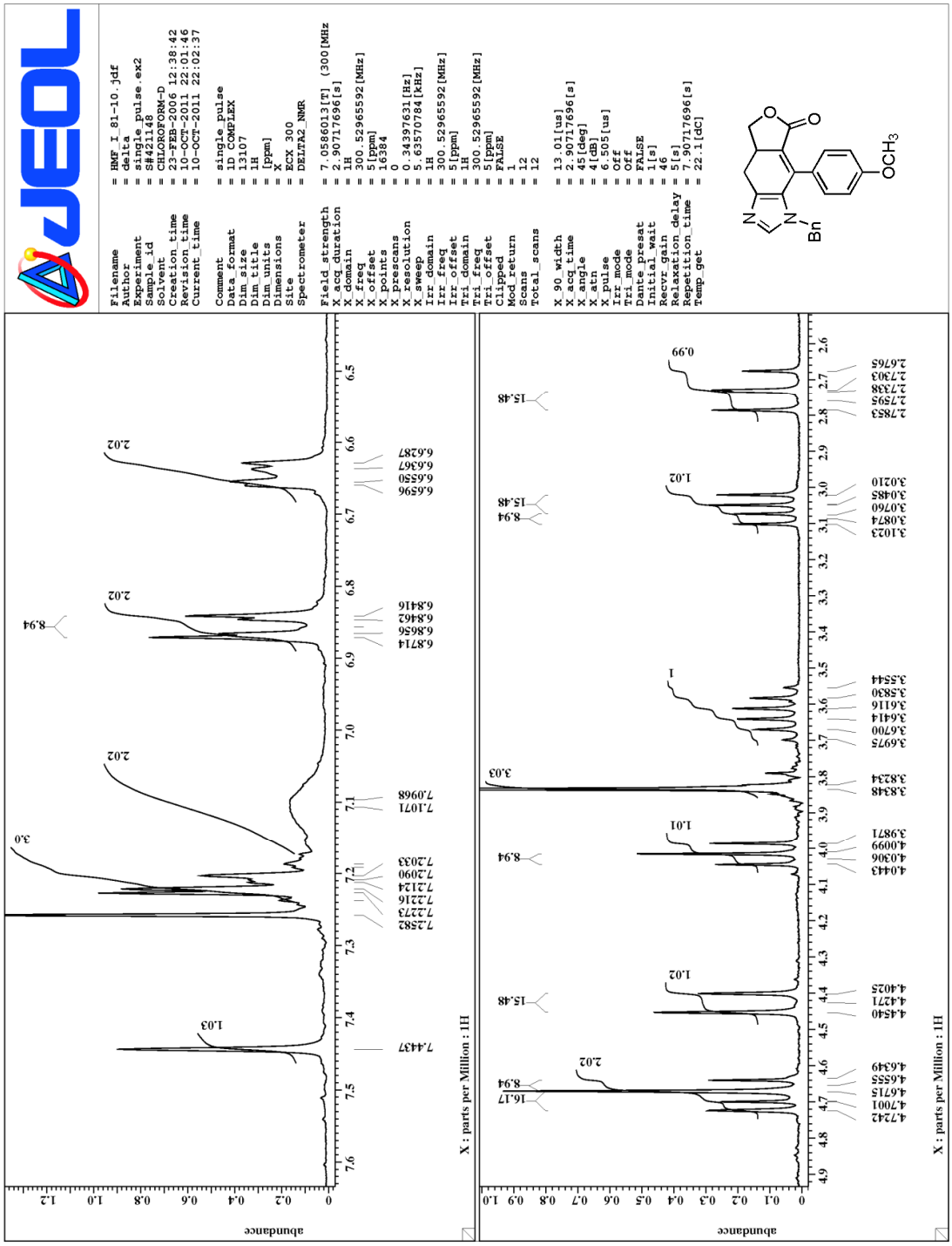

JEOL
Filename = HMF II_28-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#392120
Solvent = CHLOROFORM-D
Creation_time = 14-JUL-2006 11:33:45
Revision_time = 22-DEC-2011 14:00:15
Current_time = 22-DEC-2011 14:02:03
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.1[dC]
H3CO
Bn–N
N
abundance
X : parts per Million : 1H
1.32
6.54
1.33
5.0687
4.8398
4.8289
4.8180
3.04
2.03
3.01
3.02
15.82
6.19
15.48
1.02
1.03
7.5383
7.5169
7.5146
7.4986
7.3532
7.3475
7.3303
7.3143
7.2571
7.1598
7.1529
7.1346
7.1277
6.8782
6.8679
6.8645
6.8485
6.5806
6.4719
6.4616
6.4502
6.4296
6.3987
6.3781

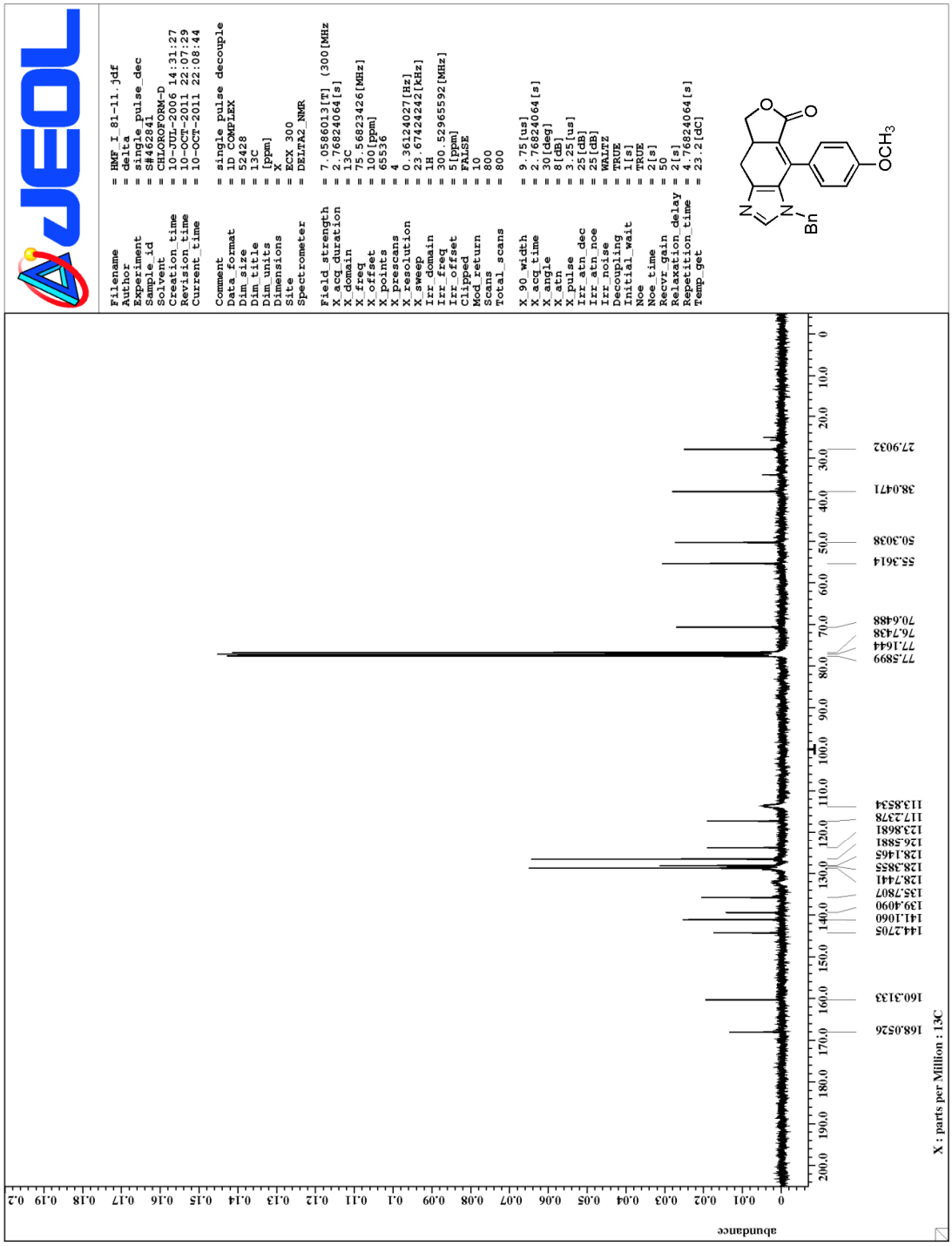

JEOL
Filename = BMF_I_80-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#579410
Solvent = CHLOROFORM-D
Creation_time = 9-JUL-2006 17:23:59
Revision_time = 10-OCT-2011 21:01:44
Current_time = 10-OCT-2011 21:02:33
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIFAZ_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 530
Total_scans = 530
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recyr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 22.7[dC]
H3CO
Bn–N
N
abundance
X : parts per Million : 13C
161.5705
154.2423
139.8487
137.9079
135.9384
135.0493
129.1360
128.4764
127.3769
127.0184
120.6127
117.8975
114.3649
111.4967
87.3035
80.1426
77.5851
77.1644
76.7390
66.4804
55.4952
51.0065

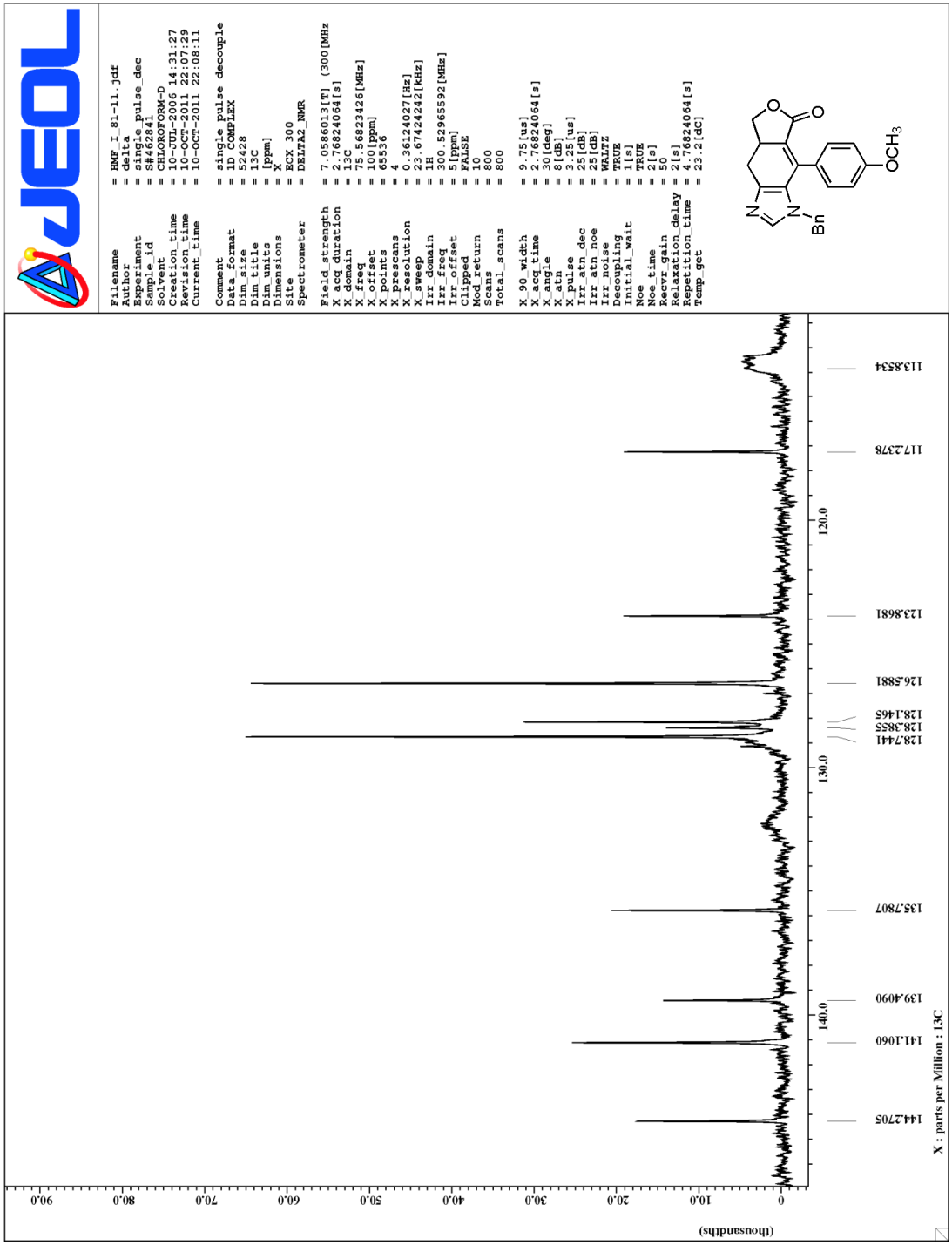

JEOL
Filename = HMF_I_80-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#579410
Solvent = CHLOROFORM-D
Creation_time = 9-JUL-2006 17:23:59
Revision_time = 10-OCT-2011 21:01:44
Current_time = 10-OCT-2011 21:03:03
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIFA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 530
Total_scans = 530
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 22.7[dC]
O
O
Bn–N
N
H3CO
(thousandths)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
110.0
120.0
130.0
140.0
111.4967
114.3649
117.8975
120.6127
127.0184
127.3769
128.4764
129.1360
135.0493
135.9384
137.9079
139.8487
X : parts per Million : 13C

APPENDIX 32
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-4-(4-methoxyphenyl)-3,7,7a,8-tetrahydro-5*H*-furo[3,4-*f*]benzimidazol-5-one (**106**)

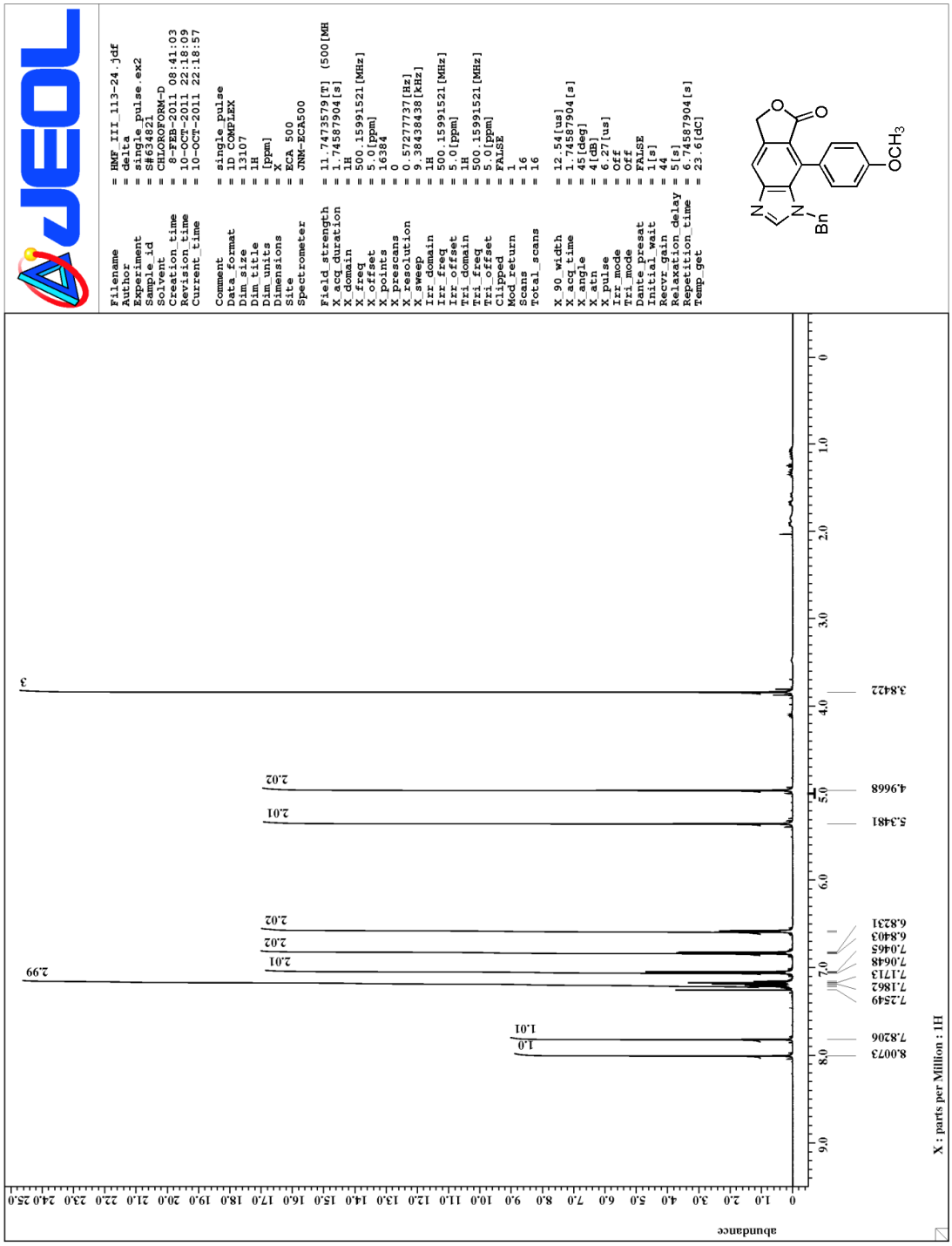

JEOL
Filename = RMF_I_81-6.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#421148
Solvent = CHLOROFORM-D
Creation_time = 23-FEB-2006 12:38:42
Revision_time = 10-OCT-2011 21:25:48
Current_time = 10-OCT-2011 21:27:16
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 22.1[dC]
N
N
Bn
O
O
OCH3
abundance
X : parts per Million : 1H
3.01
2.02
2.02
2.02
1.03
3.03
2.02
1.02
1.01
1.02
I
1.02
0.99
7.4437
7.2582
7.2273
7.2216
6.8714
6.8656
6.8416
4.7242
4.6704
4.4529
4.4014
4.0454
4.0157
3.9871
3.8348
3.6414
3.6116
3.0497
3.0210
2.7864
2.7292
2.3526


JEOL
Filename = HMF_I_81-10.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#421148
Solvent = CHLOROFORM-D
Creation_time = 23-FEB-2006 12:38:42
Revision_time = 10-OCT-2011 22:01:46
Current_time = 10-OCT-2011 22:02:37
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 22.1[dC]
OCH3
Bn
N
O
abundance
X : parts per Million : 1H
3.0
1.03
2.02
2.02
2.02
8.94
7.4437
7.3582
7.3273
7.2216
7.2124
7.2090
7.2033
7.1071
7.0968
6.8714
6.8656
6.8462
6.8416
6.6596
6.6550
6.6367
6.6287
3.03
1.01
1.02
15.48
8.94
2.02
16.17
8.94
15.48
1.02
8.94
15.48
0.99
4.7242
4.7001
4.6715
4.6555
4.6349
4.5540
4.4271
4.4025
4.0443
4.0306
4.0099
3.9871
3.8348
3.8234
3.6975
3.6700
3.6414
3.6116
3.5830
3.5544
3.1023
3.0874
3.0760
3.0485
3.0210
2.7853
2.7595
2.7338
2.7303
2.6765


Filename = HMF_I_81-11.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#462841
Solvent = CHLOROFORM-D
Creation_time = 10-JUL-2006 14:31:27
Revision_time = 10-OCT-2011 22:07:29
Current_time = 10-OCT-2011 22:08:44
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIFA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 800
Total_scans = 800
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recyr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 23.2[dC]
OCH3
Bn
27.9032
38.0471
50.3038
55.3614
70.6488
76.7438
77.1644
77.5899
113.8534
117.2378
123.8681
126.5881
128.1465
128.3855
128.7441
135.7807
139.4090
141.1060
144.2705
160.3133
168.0526
X : parts per Million : 13C
abundance


JEOL
Filename = RMF_I_81-11.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#462841
Solvent = CHLOROFORM-D
Creation_time = 10-JUL-2006 14:31:27
Revision_time = 10-OCT-2011 22:07:29
Current_time = 10-OCT-2011 22:08:11
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DEFPA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 800
Total_scans = 800
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recyr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 23.2[dC]
OCH3
N
N
Bn
(thousandths)
X : parts per Million : 13C
144.2705
141.1060
139.4090
135.7807
128.7441
128.3855
128.1465
126.5881
123.8681
117.2378
113.8534

APPENDIX 33
^{1}H and ^{13}C NMR Spectra of
1-Benzyl-4-(4-methoxyphenyl)-3,7-dihydro-5*H*-furo[3,4-*f*]benzimidazol-5-one (**119**)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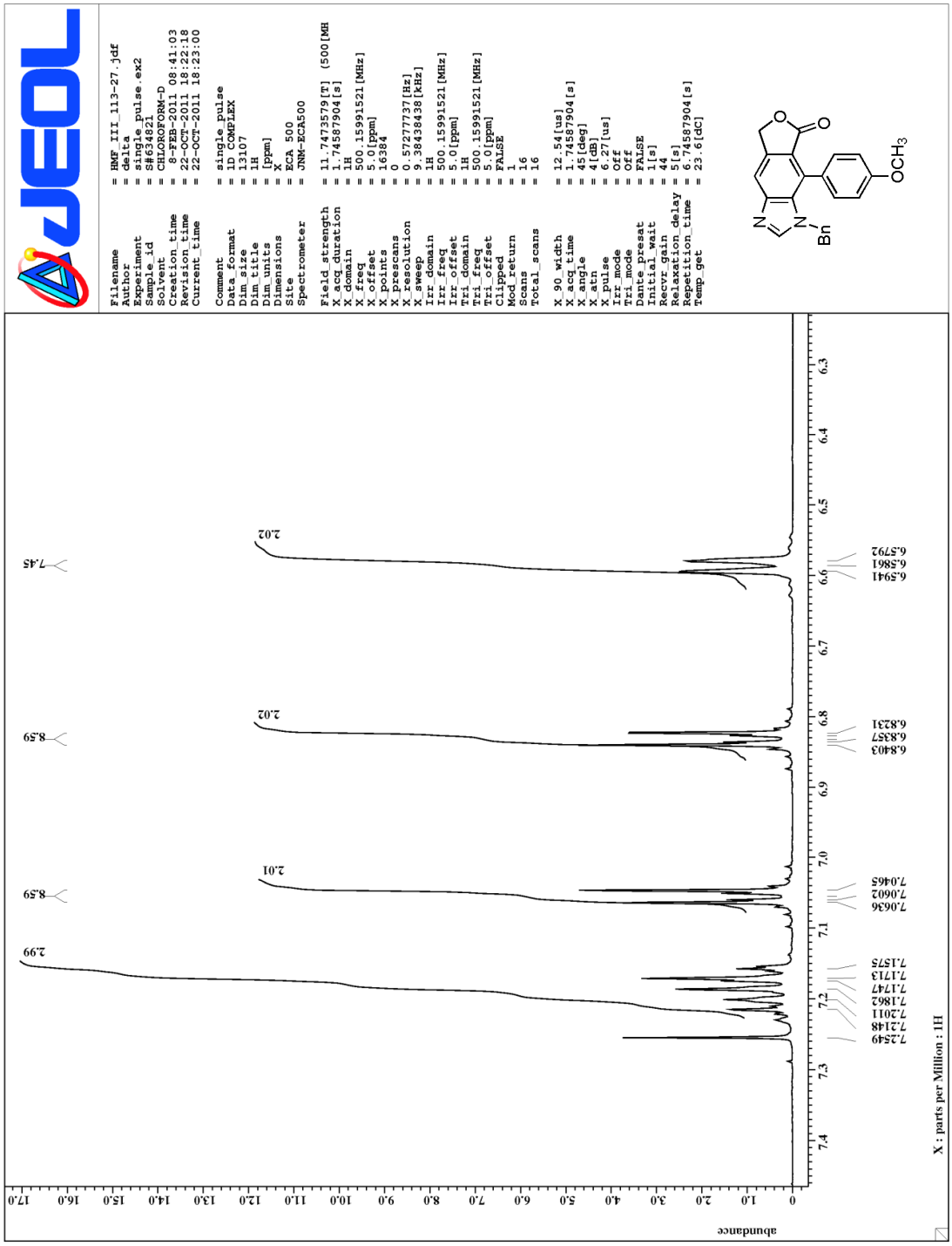
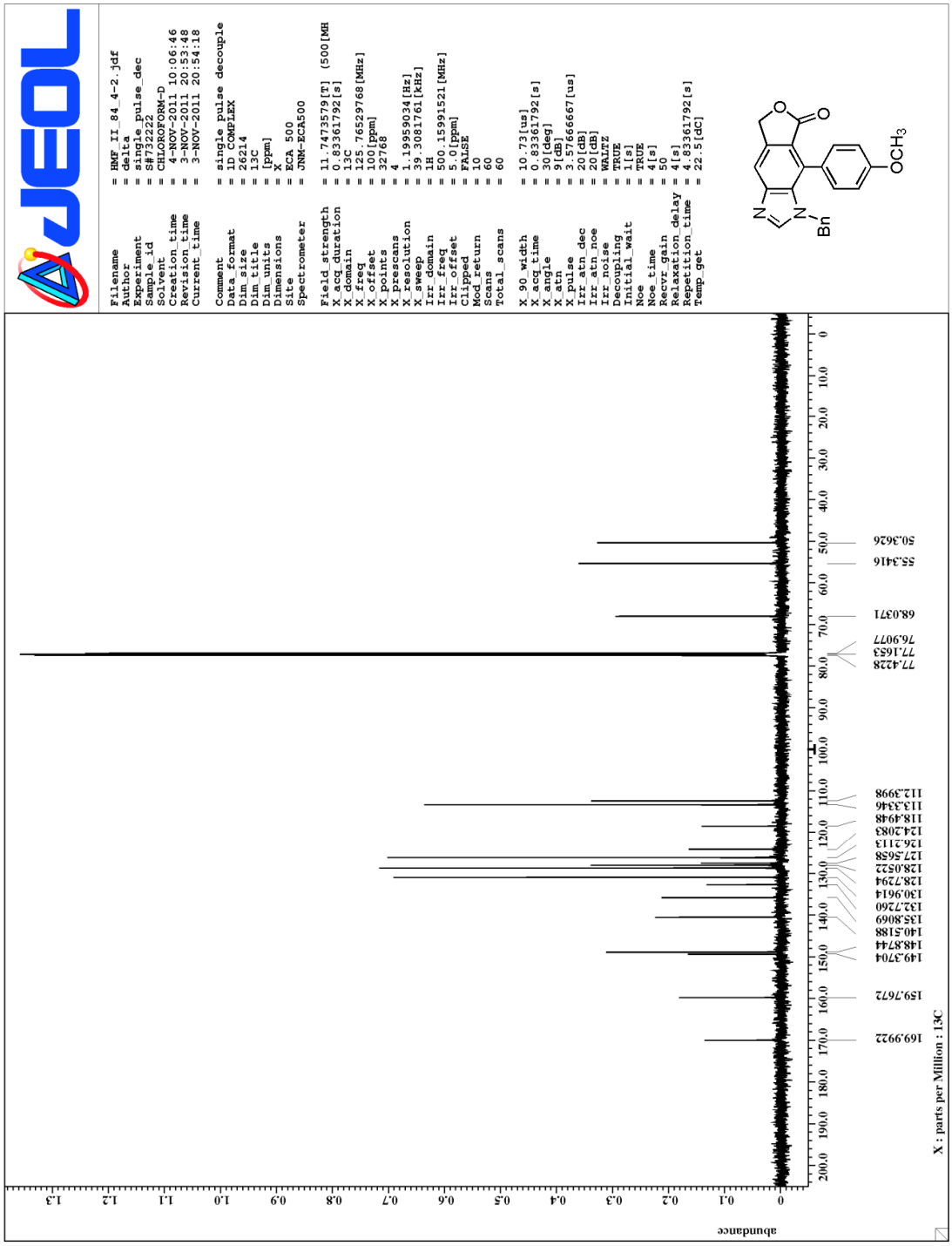
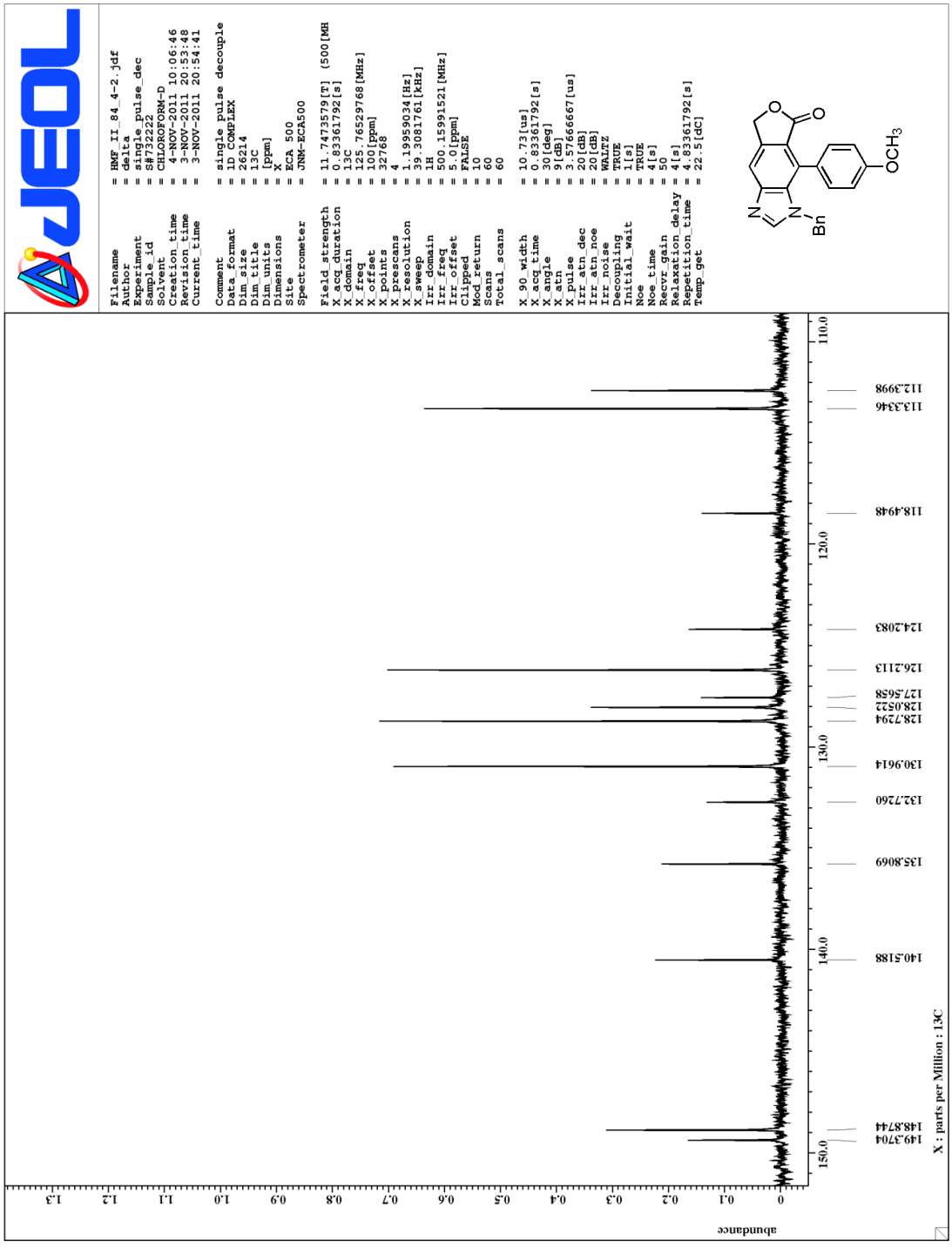
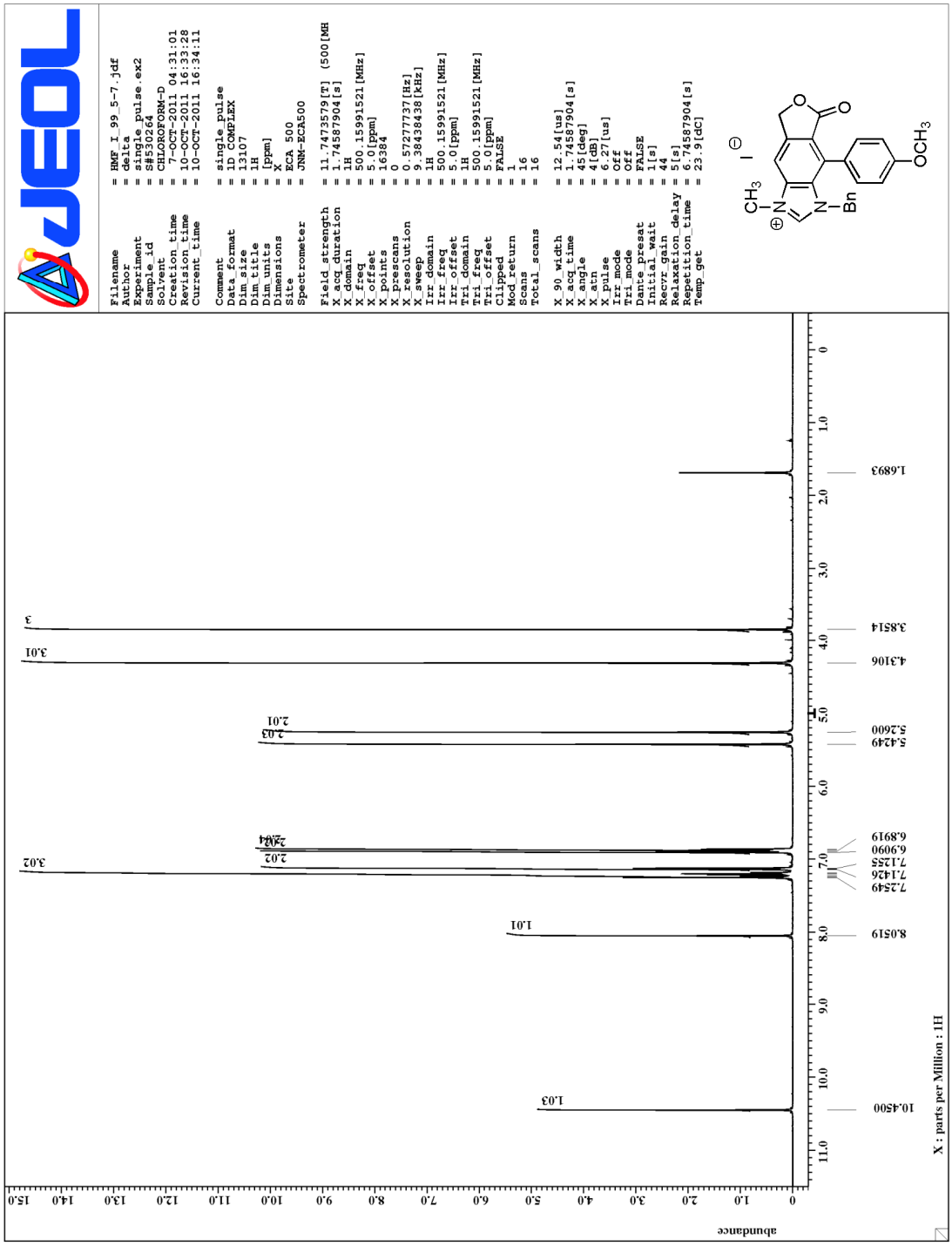
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#634821
Solvent = CHLOROFORM-D
Creation_time = 8-FEB-2011 08:41:03
Revision_time = 10-OCT-2011 22:18:09
Current_time = 10-OCT-2011 22:18:57
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.6[dC]
OCH3
Bn
abundance
X : parts per Million : 1H
3
2.99
2.02
2.01
2.02
2.01
2.02
1.01
1.0
3.8422
4.9668
5.3481
6.8231
6.8403
7.0465
7.0648
7.1713
7.1862
7.2549
7.8206
8.0073


JEOL
Filename = RMF_III_113-27.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#634821
Solvent = CHLOROFORM-D
Creation_time = 8-FEB-2011 08:41:03
Revision_time = 22-OCT-2011 18:22:18
Current_time = 22-OCT-2011 18:23:00
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.6[dC]
Bn
OCH3
abundance
X : parts per Million : 1H
7.45
8.59
8.59
2.99
2.01
2.02
2.02
7.2549
7.2148
7.2011
7.1862
7.1747
7.1713
7.1575
7.0636
7.0602
7.0465
6.8403
6.8357
6.8231
6.5941
6.5861
6.5792


JEOL
Filename = HMF_II_84_4-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#732222
Solvent = CHLOROFORM-D
Creation_time = 4-NOV-2011 10:06:46
Revision_time = 3-NOV-2011 20:53:48
Current_time = 3-NOV-2011 20:54:18
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 60
Total_scans = 60
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.5[dC]
OCH3
N
N
Bn
abundance
X : parts per Million : 13C
169.9922
159.7672
149.3704
148.8744
140.5188
135.8069
132.7260
130.9614
128.7294
128.0522
127.5658
126.3113
124.2083
118.4948
113.3346
112.3998
77.4228
77.1653
76.9077
68.0371
55.3416
50.3626


Filename = HMF_II_84_4-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#732222
Solvent = CHLOROFORM-D
Creation_time = 4-NOV-2011 10:06:46
Revision_time = 3-NOV-2011 20:53:48
Current_time = 3-NOV-2011 20:54:41
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 60
Total_scans = 60
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.5[dC]
OCH3
Bn
abundance
X : parts per Million : 13C
149.3704
148.8744
140.5188
135.8069
132.7260
130.9614
128.7294
128.0522
127.5658
126.2113
124.2083
118.4948
113.3346
112.3998

APPENDIX 34
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-4-(4-methoxyphenyl)-1-methyl-5-oxo-5,7-dihydro-3*H*-furo[3,4-*f*]benzimidazol-1-ium iodide (**105**)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Experiment = single_pulse_ex2
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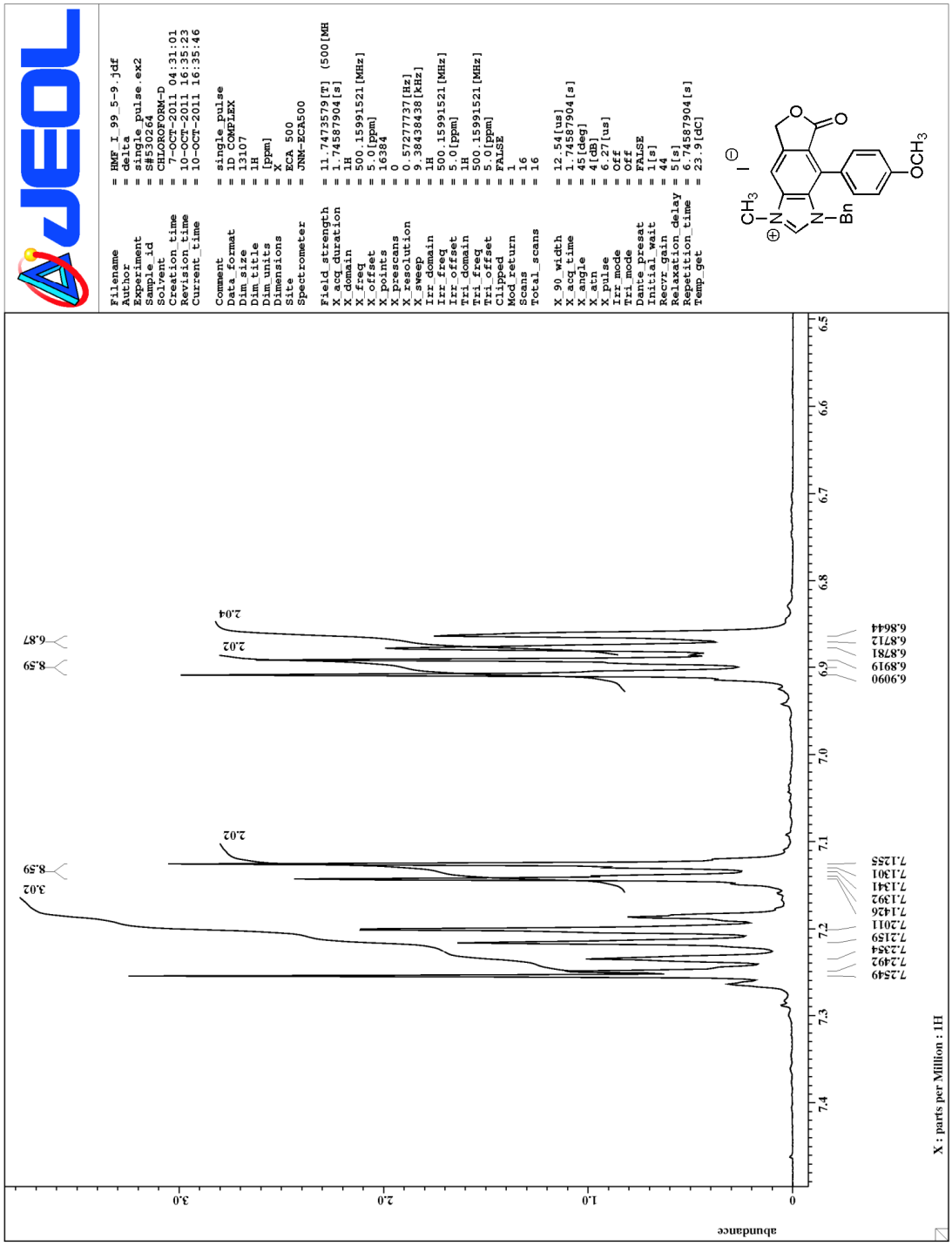
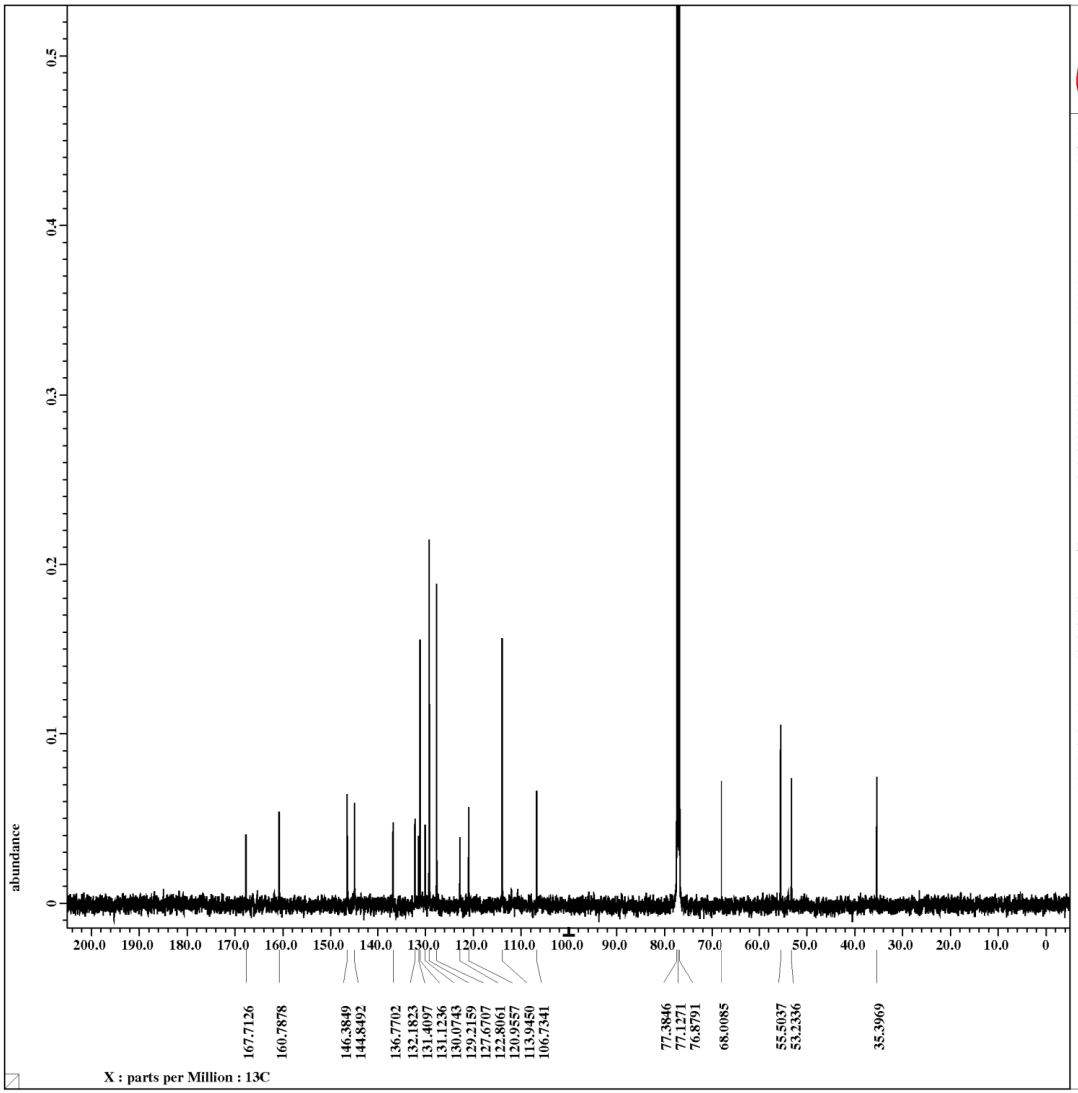
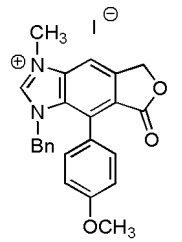
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2011 04:31:01
Revision_time = 10-OCT-2011 16:33:28
Current_time = 10-OCT-2011 16:34:11
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384381[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 41[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.9[dC]
CH3
Bn
OCH3
abundance
X : parts per Million : 1H
10.4500
8.0519
7.2549
7.1426
7.1255
6.9090
6.8919
5.4249
5.2600
4.3106
3.8514
1.6893
1.03
1.01
3.02
2.02
2.03
2.01
3.01
3

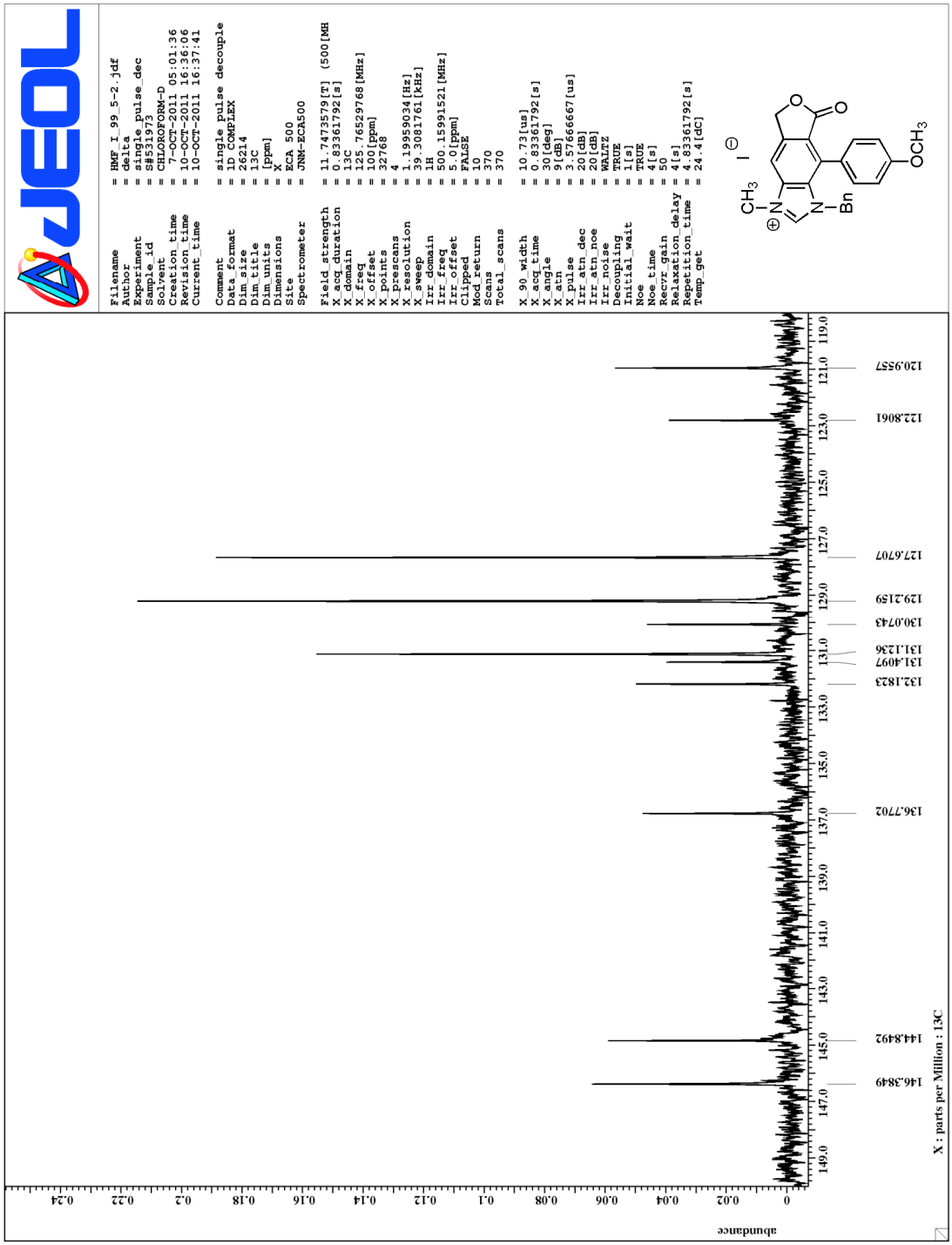

Filename = HMF_I_99_5-9.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#530264
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2011 04:31:01
Revision_time = 10-OCT-2011 16:35:23
Current_time = 10-OCT-2011 16:35:46
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.9[dC]
abundance
X : parts per Million : 1H
3.02
8.59
2.02
2.04
2.02
8.59
6.87
7.2549
7.2492
7.2354
7.2159
7.2011
7.1426
7.1392
7.1341
7.1301
7.1255
6.9090
6.8919
6.8781
6.8712
6.8644
OCH3
Bn
CH3
I



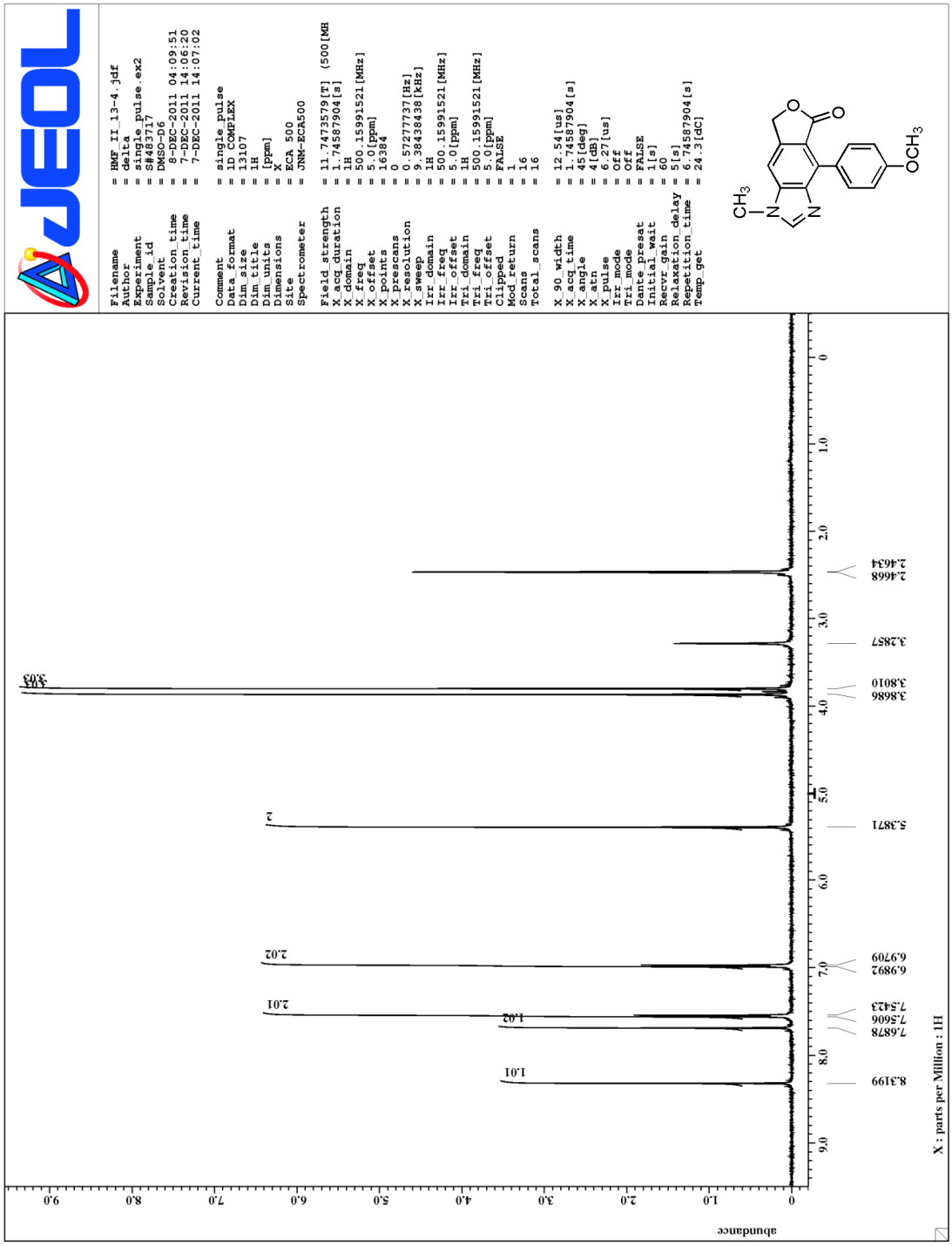
JEOL

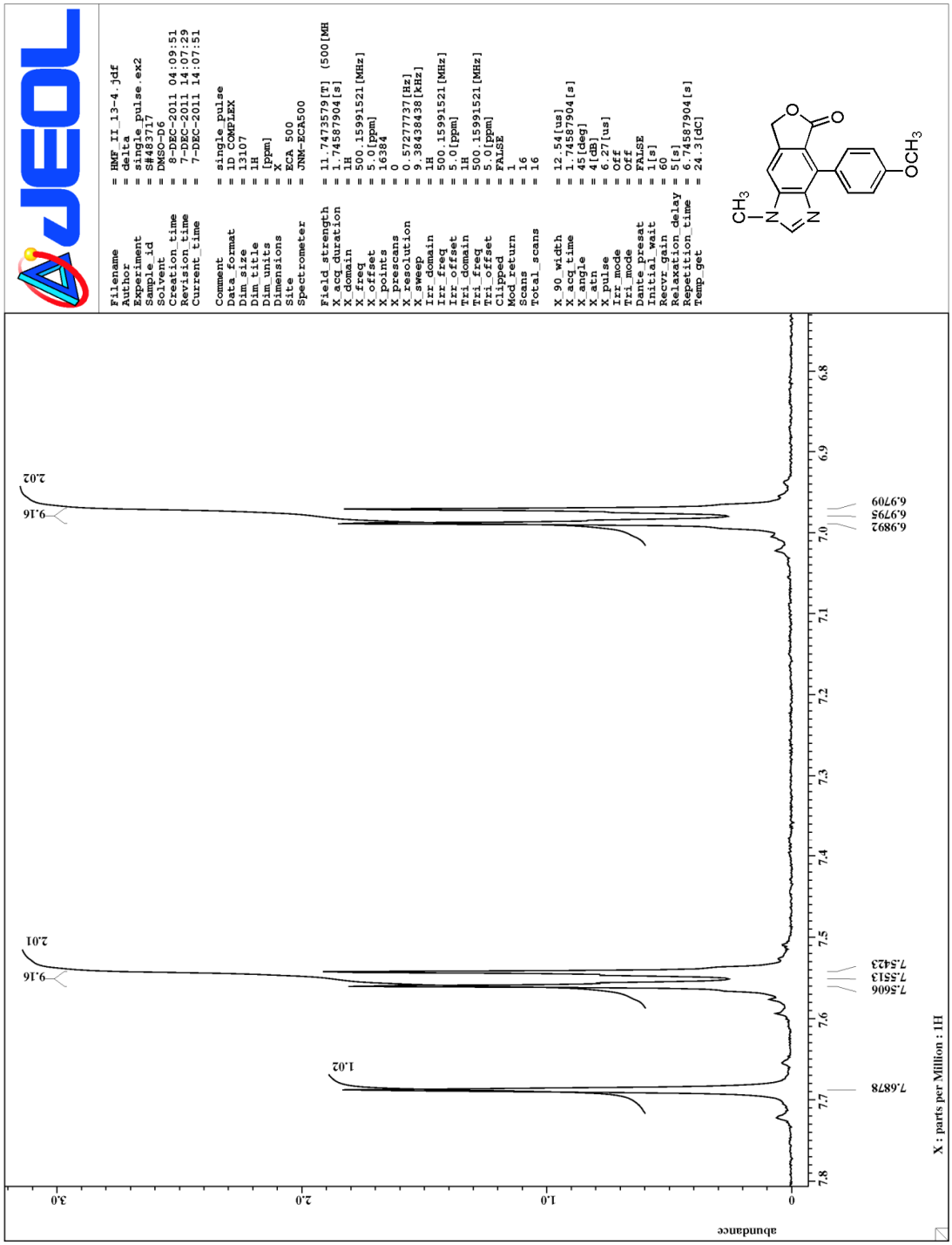
Filename	= HMF_I_99_5-2.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#531973
Solvent	= CHLOROFORM-D
Creation_time	= 7-OCT-2011 05:01:36
Revision_time	= 10-OCT-2011 16:36:06
Current_time	= 10-OCT-2011 16:36:51
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 10
Scans	= 370
Total_scans	= 370
X_90_width	= 10.73[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 9[dB]
X_pulse	= 3.57666667[us]
Irr_atn_dec	= 20[dB]
Irr_atn_noe	= 20[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 4[s]
Recvr_gain	= 50
Relaxation_delay	= 4[s]
Repetition_time	= 4.83361792[s]
Temp_get	= 24.4[dC]

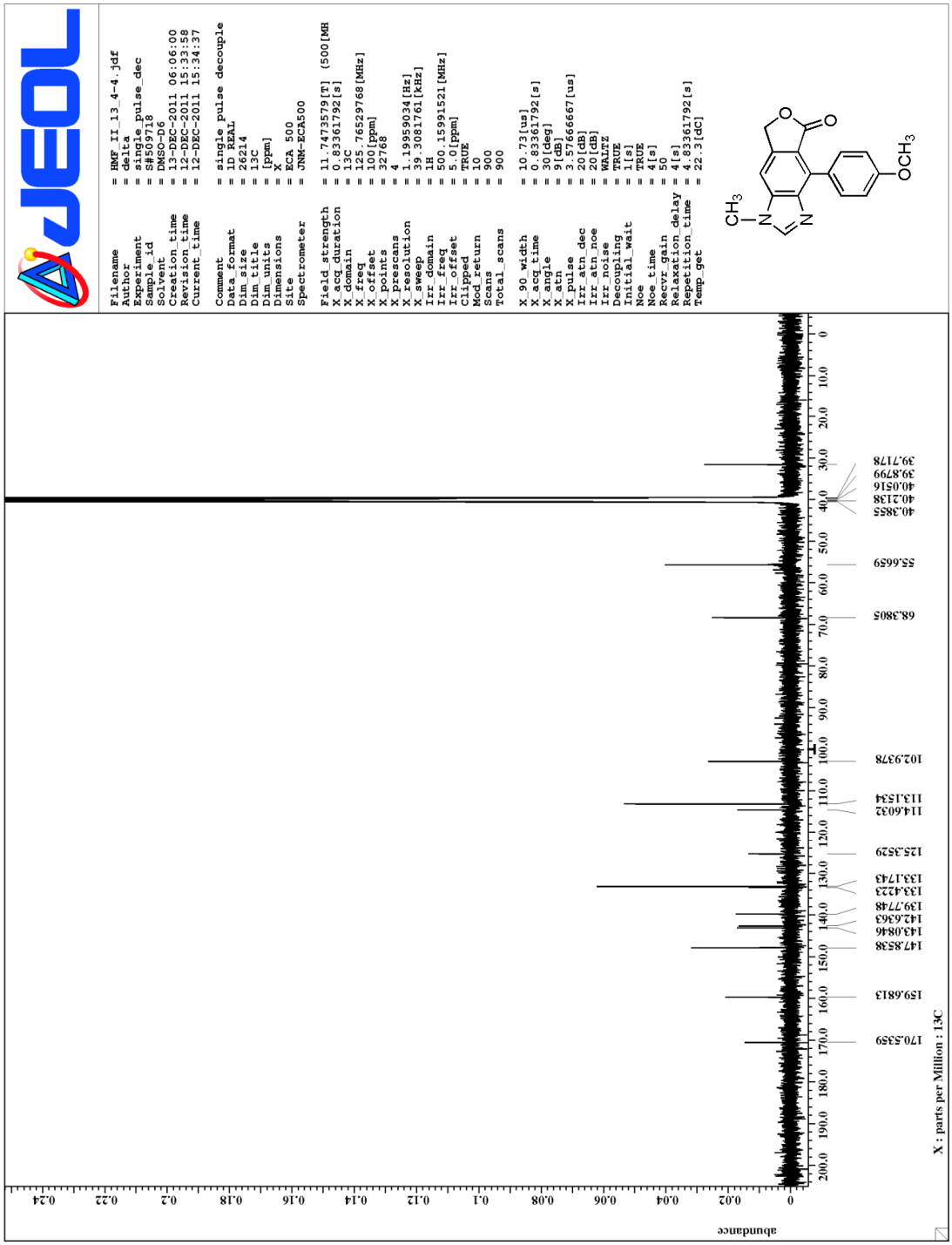



Filename = BMF_I_99_5-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#531973
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2011 05:01:36
Revision_time = 10-OCT-2011 16:36:06
Current_time = 10-OCT-2011 16:37:41
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 370
Total_scans = 370
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 24.4[dC]
120.9557
122.8061
127.6707
129.2159
130.0743
131.1236
131.4097
132.1823
136.7702
144.8492
146.3849
X : parts per Million : 13C
abundance

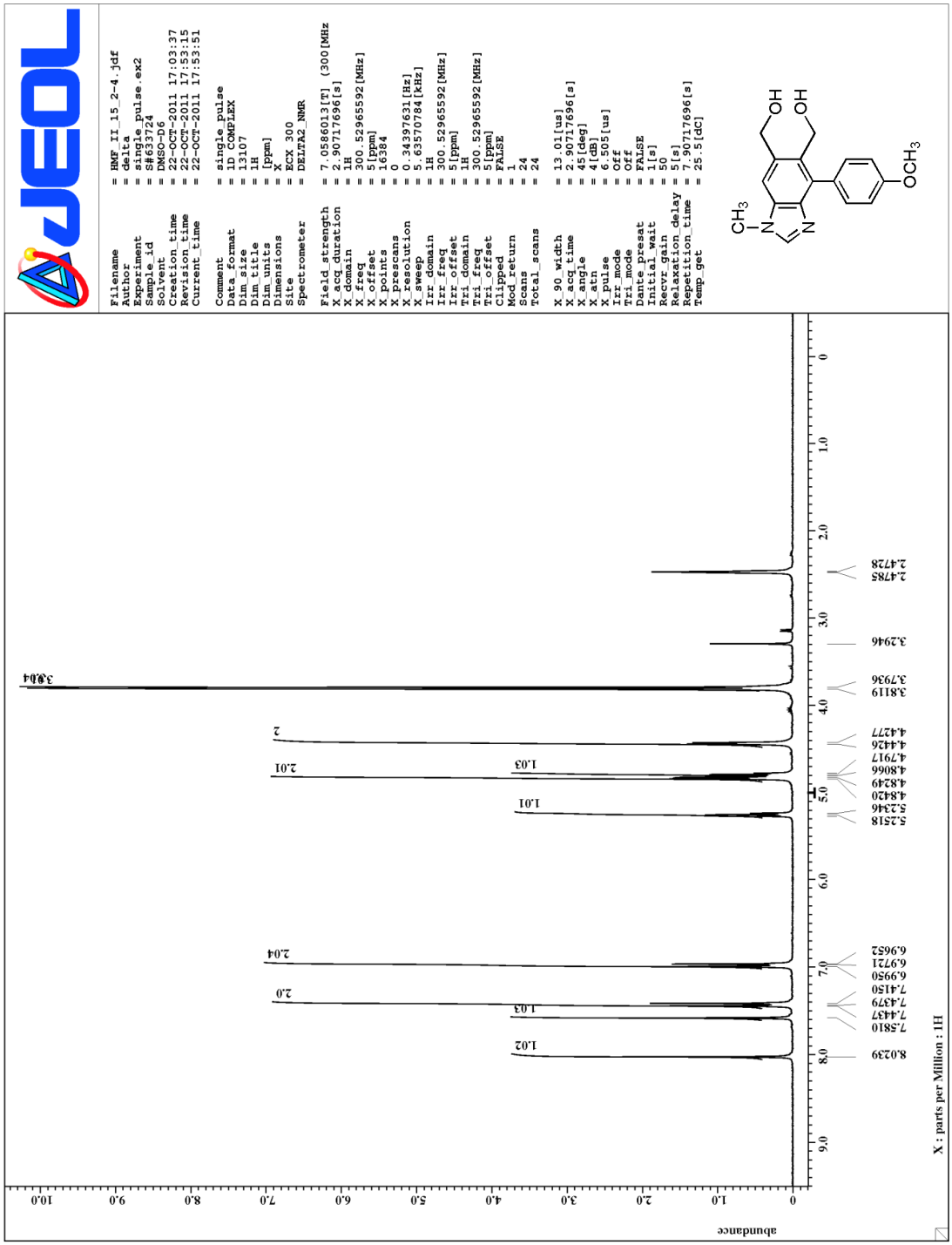
APPENDIX 35
^{1}H and ^{13}C NMR Spectra of
4-(4-Methoxyphenyl)-1-methyl-1,7-dihydro-furo[3,4-f]benzimidazol-5-one (**120b**)

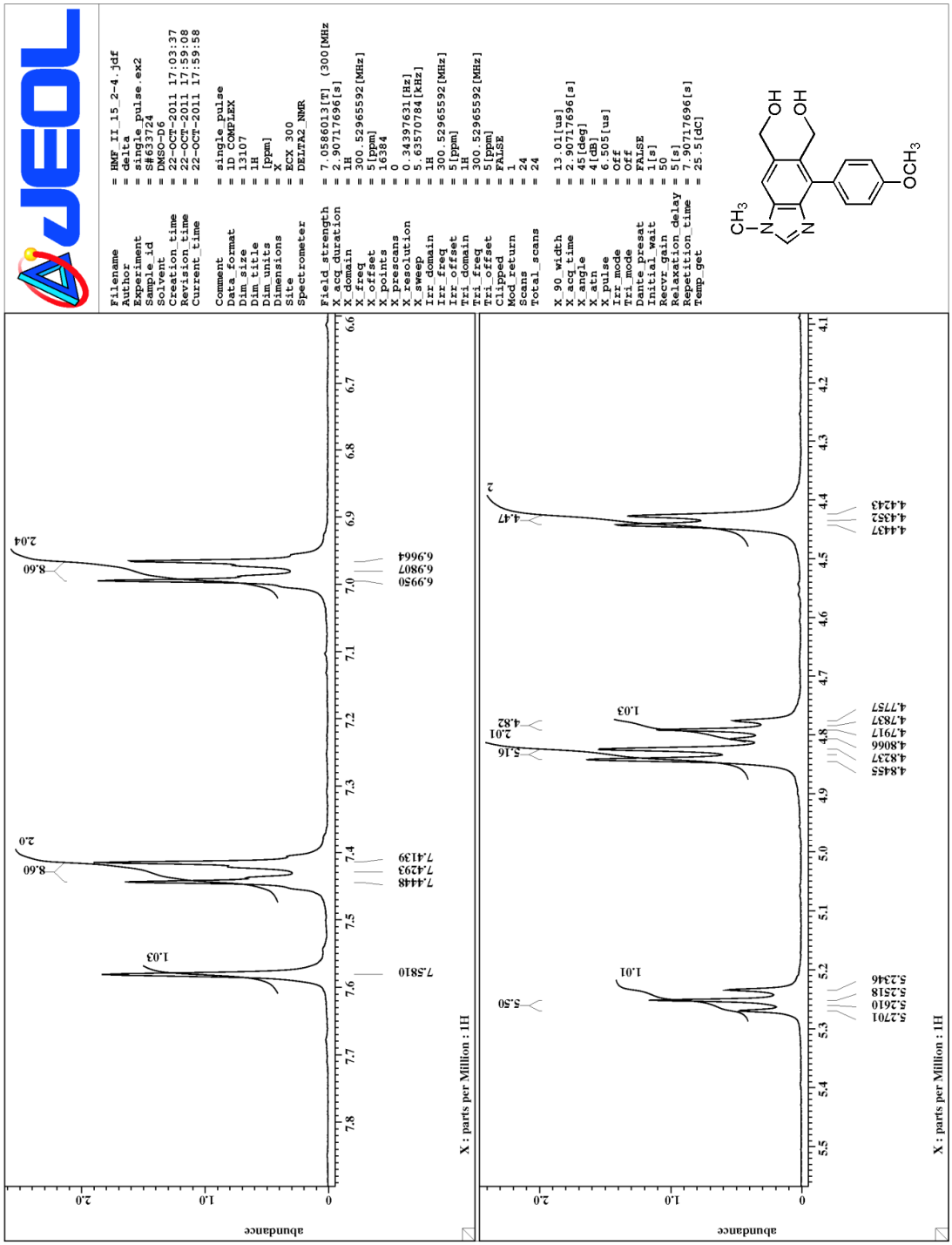

JEOL
Filename = HMF_II_13-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#483717
Solvent = DMSO-D6
Creation_time = 8-DEC-2011 04:09:51
Revision_time = 7-DEC-2011 14:06:20
Current_time = 7-DEC-2011 14:07:02
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 60
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 24.3[dC]
CH3
OCH3
abundance
X : parts per Million : 1H
3.03
2
2.02
2.01
1.02
1.01
8.3199
7.6878
7.5606
7.5423
6.9892
6.9709
5.3871
3.8686
3.8010
3.2857
2.4668
2.4634

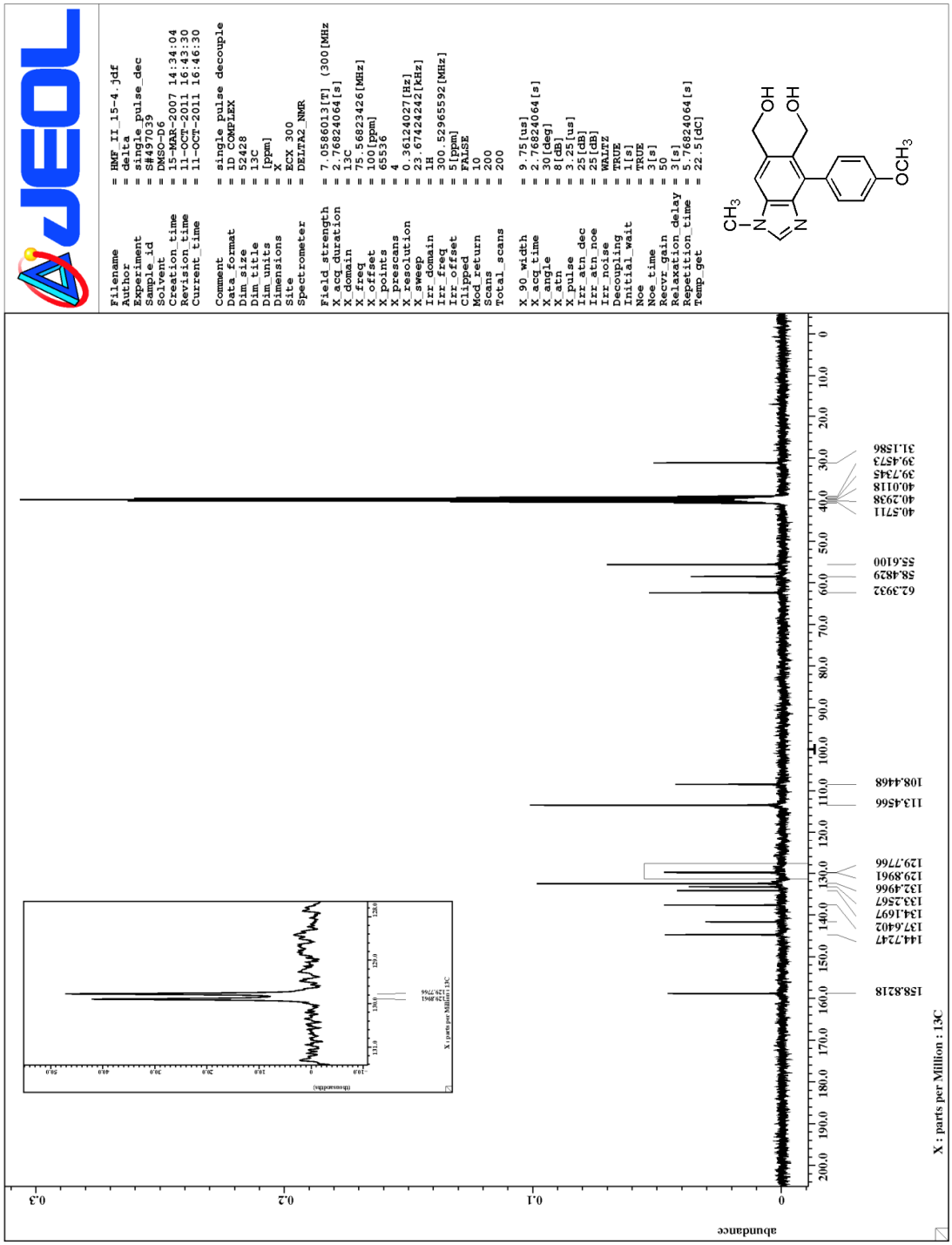

JEOL
Filename = RMF_II_13-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#483717
Solvent = DMSO-D6
Creation_time = 8-DEC-2011 04:09:51
Revision_time = 7-DEC-2011 14:07:29
Current_time = 7-DEC-2011 14:07:51
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 60
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 24.3[dC]
CH3
N
N
O
O
OCH3
abundance
3.0
2.0
1.0
0
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
2.02
9.16
2.01
9.16
1.02
7.6878
7.5606
7.5513
7.5423
6.9892
6.9795
6.9709
X : parts per Million : 1H

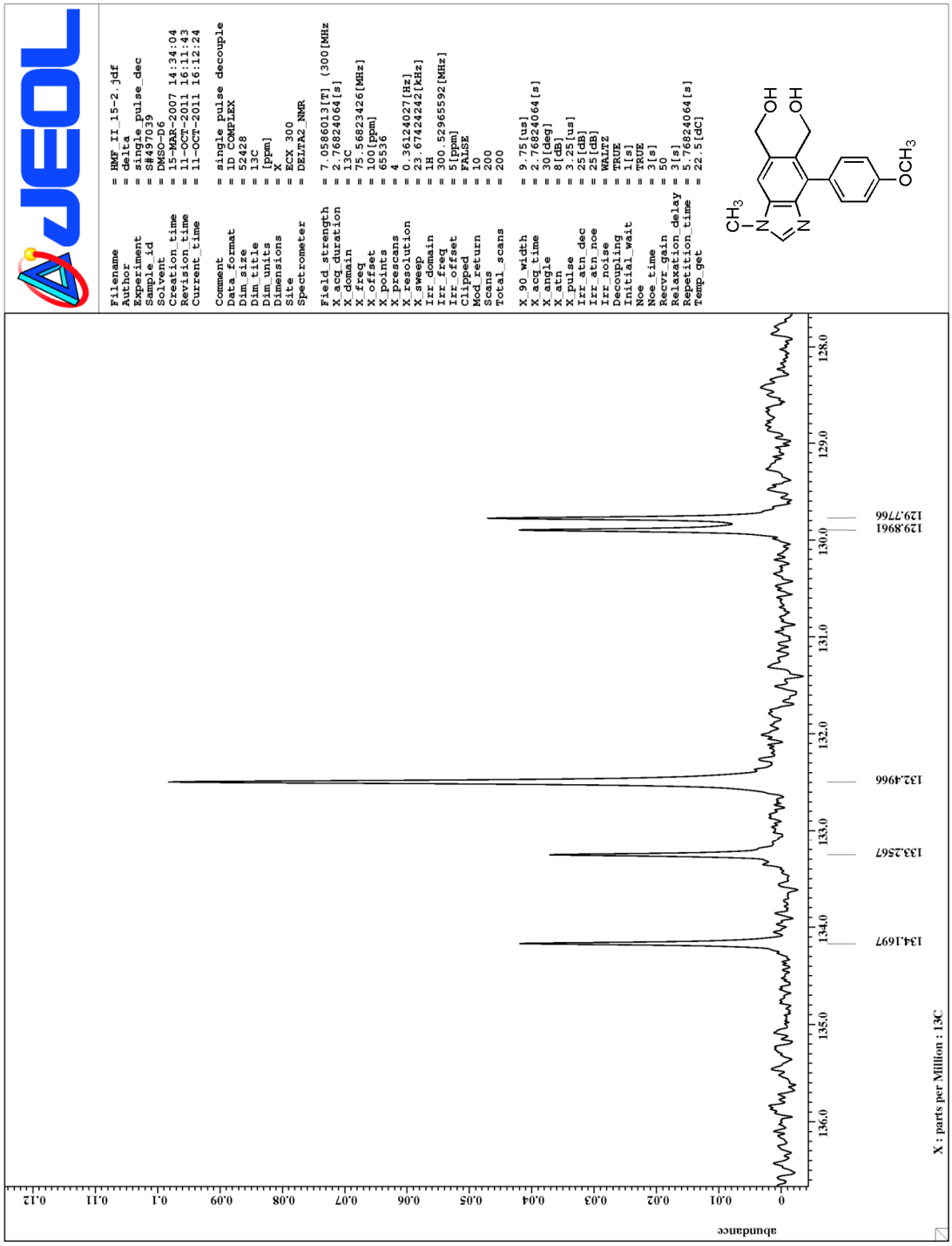

JEOL
Filename = HMF_II_13_4-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#509718
Solvent = DMSO-D6
Creation_time = 13-DEC-2011 06:06:00
Revision_time = 12-DEC-2011 15:33:58
Current_time = 12-DEC-2011 15:34:37
Comment = single_pulse_decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = TRUE
Mod_return = 10
Scans = 900
Total_scans = 900
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.3[dC]
CH3
N
N
O
O
OCH3
abundance
X : parts per Million : 13C
170.5359
159.6813
147.8538
143.0846
142.6363
139.7748
133.4223
133.1743
125.3529
114.6032
113.1534
102.9378
68.3805
55.6659
40.3855
40.2138
40.0516
39.8799
39.7178

APPENDIX 36
^{1}H and ^{13}C NMR Spectra of
[4-(4-Methoxyphenyl)-1-methyl-1*H*-benzimidazole-5,6-diyl]dimethanol (**121**)

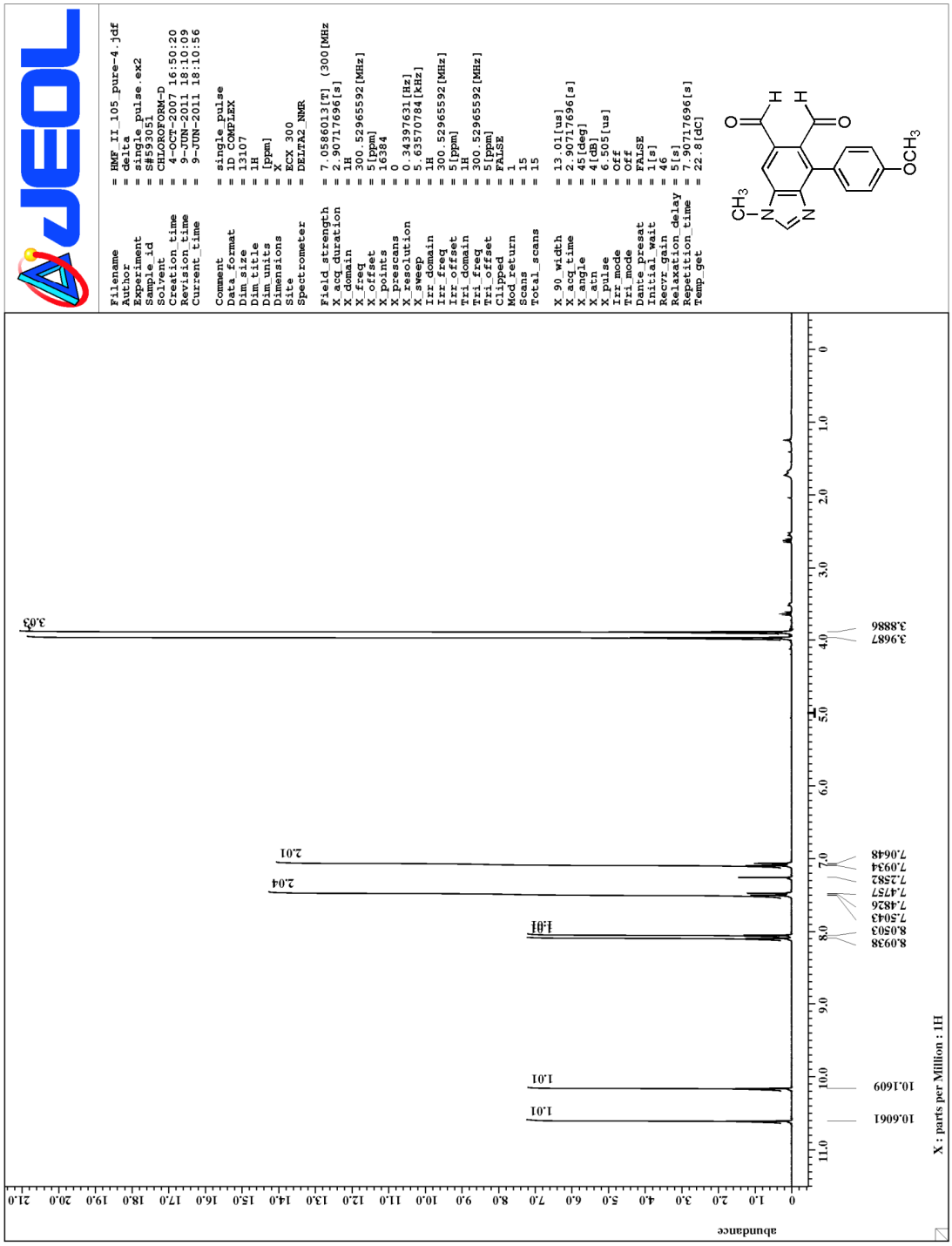

JEOL
Filename = HMF_II_15_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#633724
Solvent = DMSO-D6
Creation_time = 22-OCT-2011 17:03:37
Revision_time = 22-OCT-2011 17:53:15
Current_time = 22-OCT-2011 17:53:51
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 25.5[dC]
OH
OH
CH3
N
N
OCH3
abundance
X : parts per Million : 1H
8.0239
7.5810
7.4437
7.4379
7.4150
6.9950
6.9721
6.9652
5.2518
5.2346
4.8420
4.8249
4.8066
4.7917
4.4426
4.4277
3.8119
3.7936
3.2946
2.4785
2.4728
3.00
2.01
1.03
1.01
2.04
2.00
1.03
1.02
2

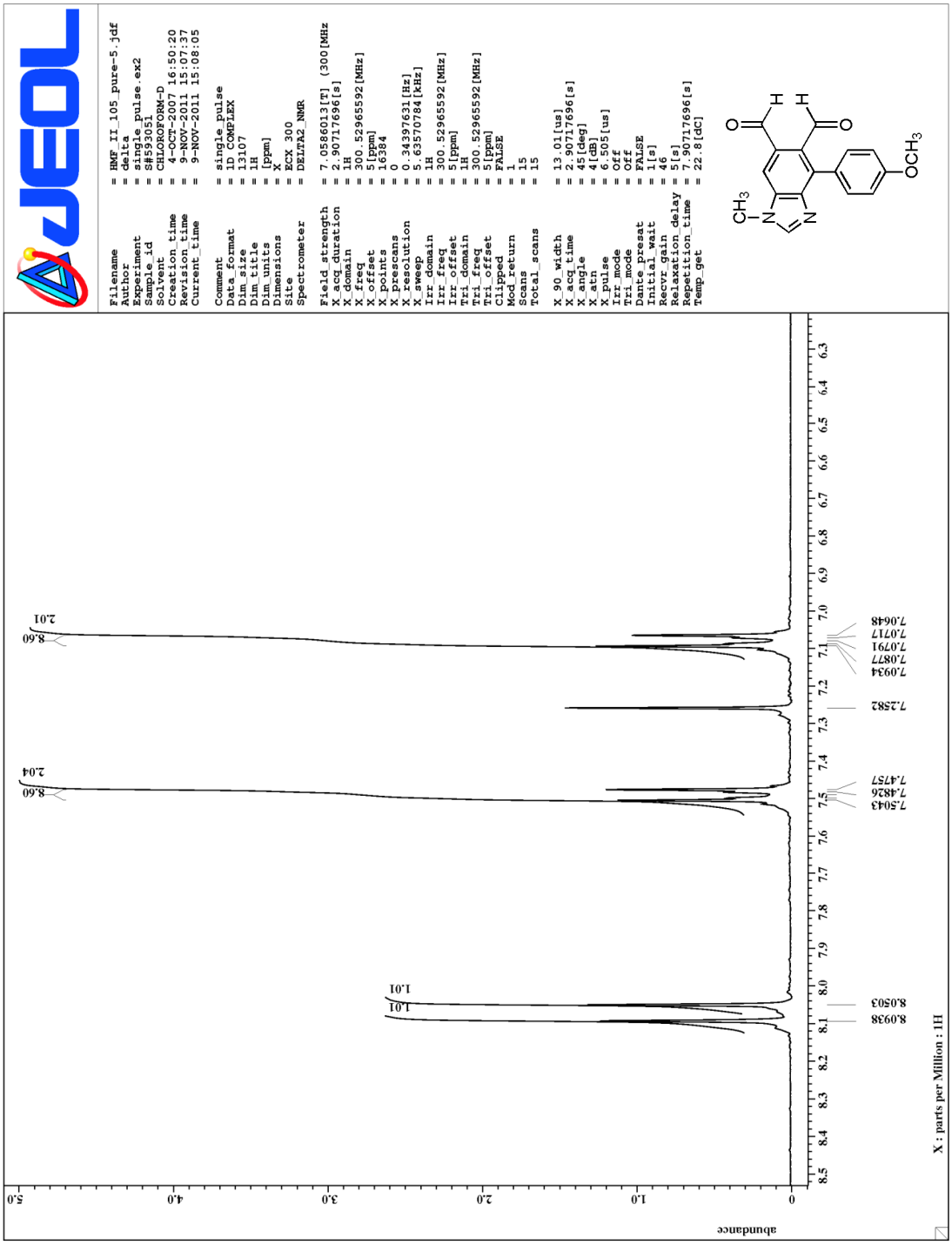

JEOL
Filename = HMF_II_15_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#633724
Solvent = DMSO-D6
Creation_time = 22-OCT-2011 17:03:37
Revision_time = 22-OCT-2011 17:59:08
Current_time = 22-OCT-2011 17:59:58
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEUTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 25.5[dC]
abundance
X : parts per Million : 1H
8.60
8.60
2.04
2.0
1.03
7.5810
7.4448
7.4293
7.4139
6.9950
6.9807
6.9664
5.50
5.2701
5.2610
5.2518
5.2346
5.16
4.8455
4.8237
4.8066
4.7917
4.7837
4.7757
4.82
2.01
4.47
4.4437
4.4352
4.4243
1.03
1.01
2
OH
OH
CH3
N
N
OCH3

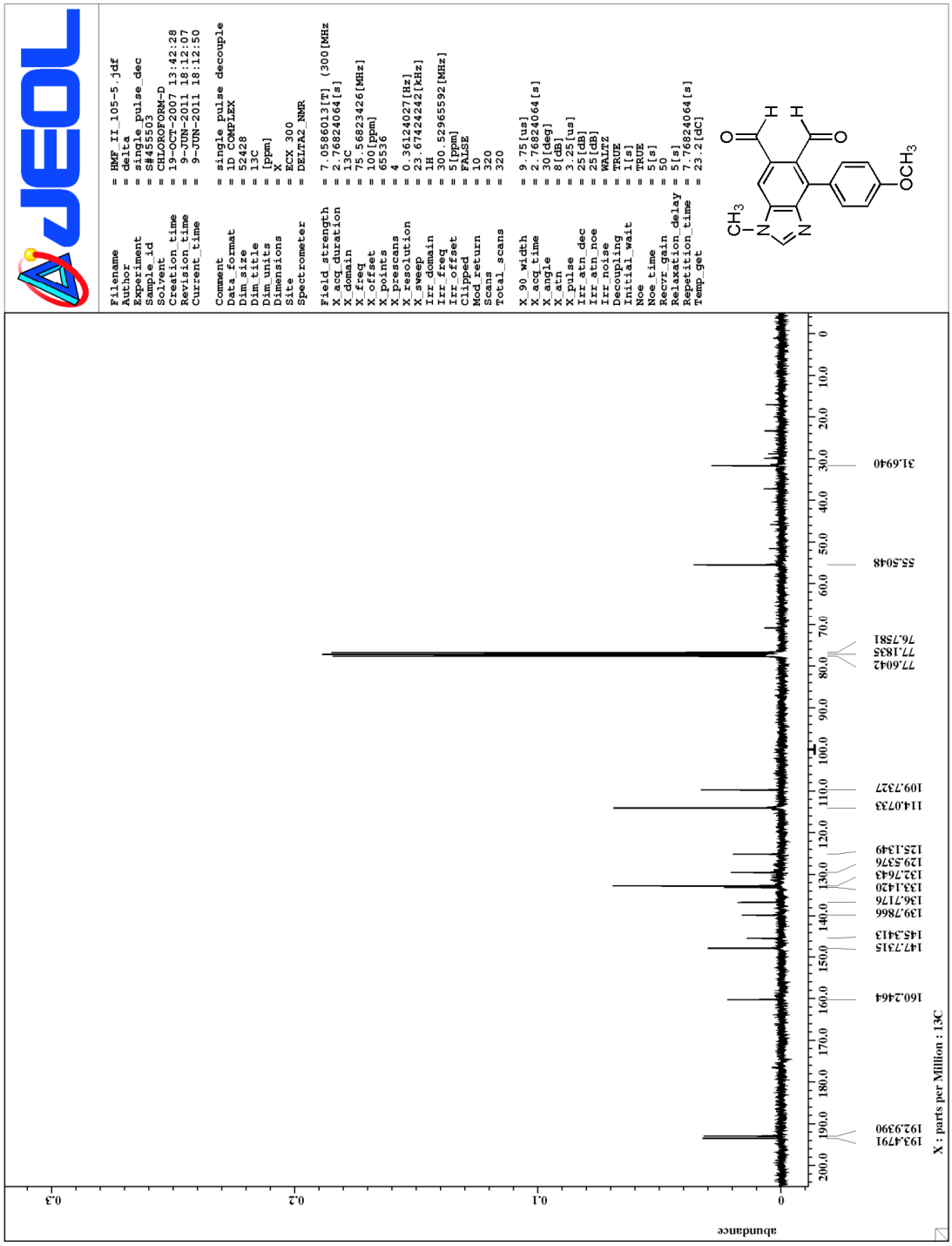

JEOL
Filename = HMF II 15-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#497039
Solvent = DMSO-D6
Creation_time = 15-MAR-2007 14:34:04
Revision_time = 11-OCT-2011 16:43:30
Current_time = 11-OCT-2011 16:46:30
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DEIFAZ_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 200
Total_scans = 200
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 5.76824064[s]
Temp_get = 22.5[dC]
OH
OH
CH3
N
N
OCH3
abundance
X : parts per Million : 13C
158.8218
144.7247
137.6402
134.1697
133.2567
132.4966
129.8961
129.7766
113.4566
108.4468
62.3932
58.4829
55.6100
40.5711
40.2938
40.0118
39.7345
39.4573
31.1586


Filename = HMF_II_15-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#497039
Solvent = DMSO-D6
Creation_time = 15-MAR-2007 14:34:04
Revision_time = 11-OCT-2011 16:11:43
Current_time = 11-OCT-2011 16:12:24
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 200
Total_scans = 200
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 5.76824064[s]
Temp_get = 22.5[dC]
OH
OH
CH3
N
N
OCH3
abundance
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
0.12
136.0
135.0
134.0
133.0
132.0
131.0
130.0
129.0
128.0
134.1697
133.2567
132.4966
129.8961
129.7766
X : parts per Million : 13C

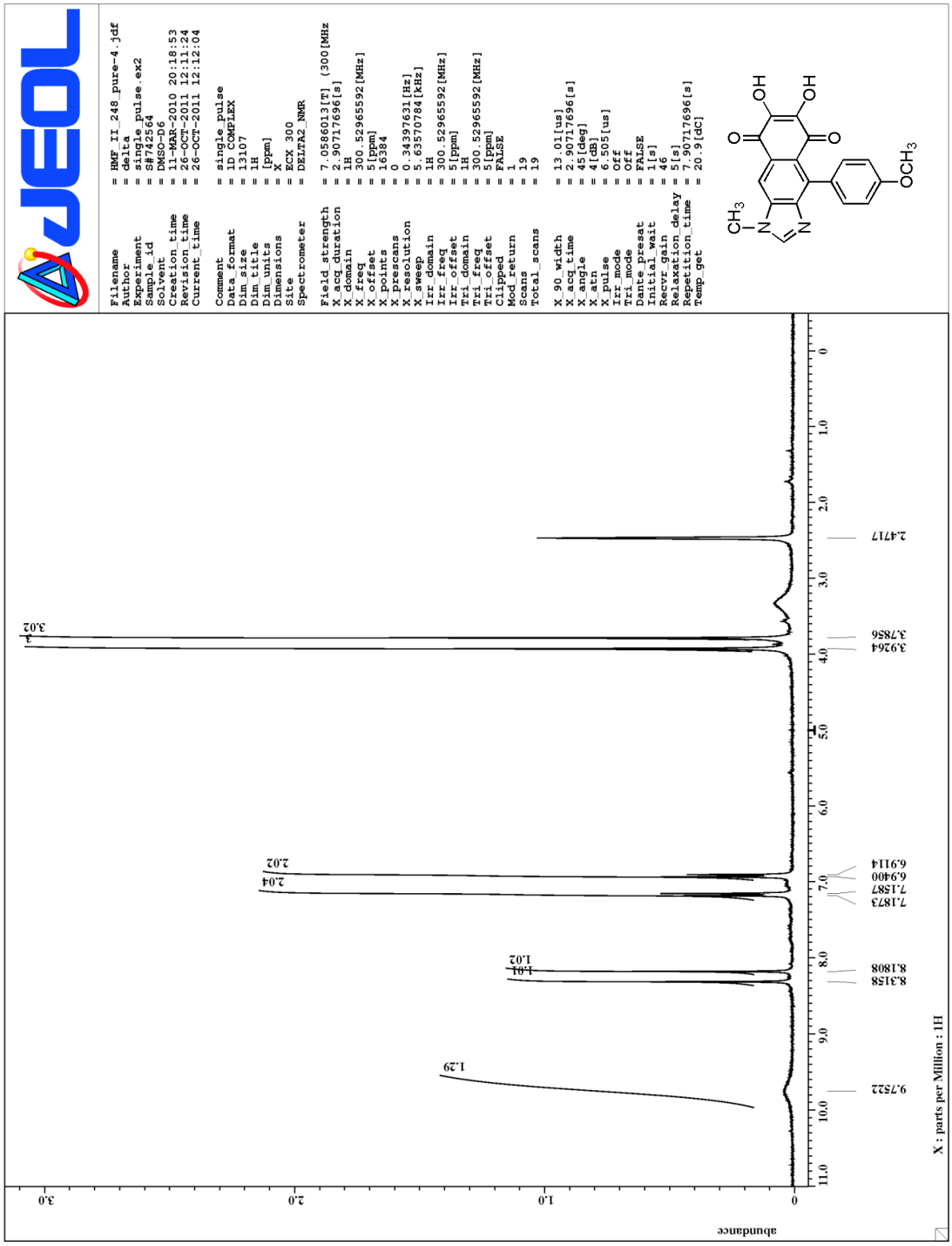
APPENDIX 37
^{1}H and ^{13}C NMR Spectra of
4-(4-Methoxyphenyl)-1-methyl-1*H*-benzimidazole-5,6-dicarbaldehyde (**104**)

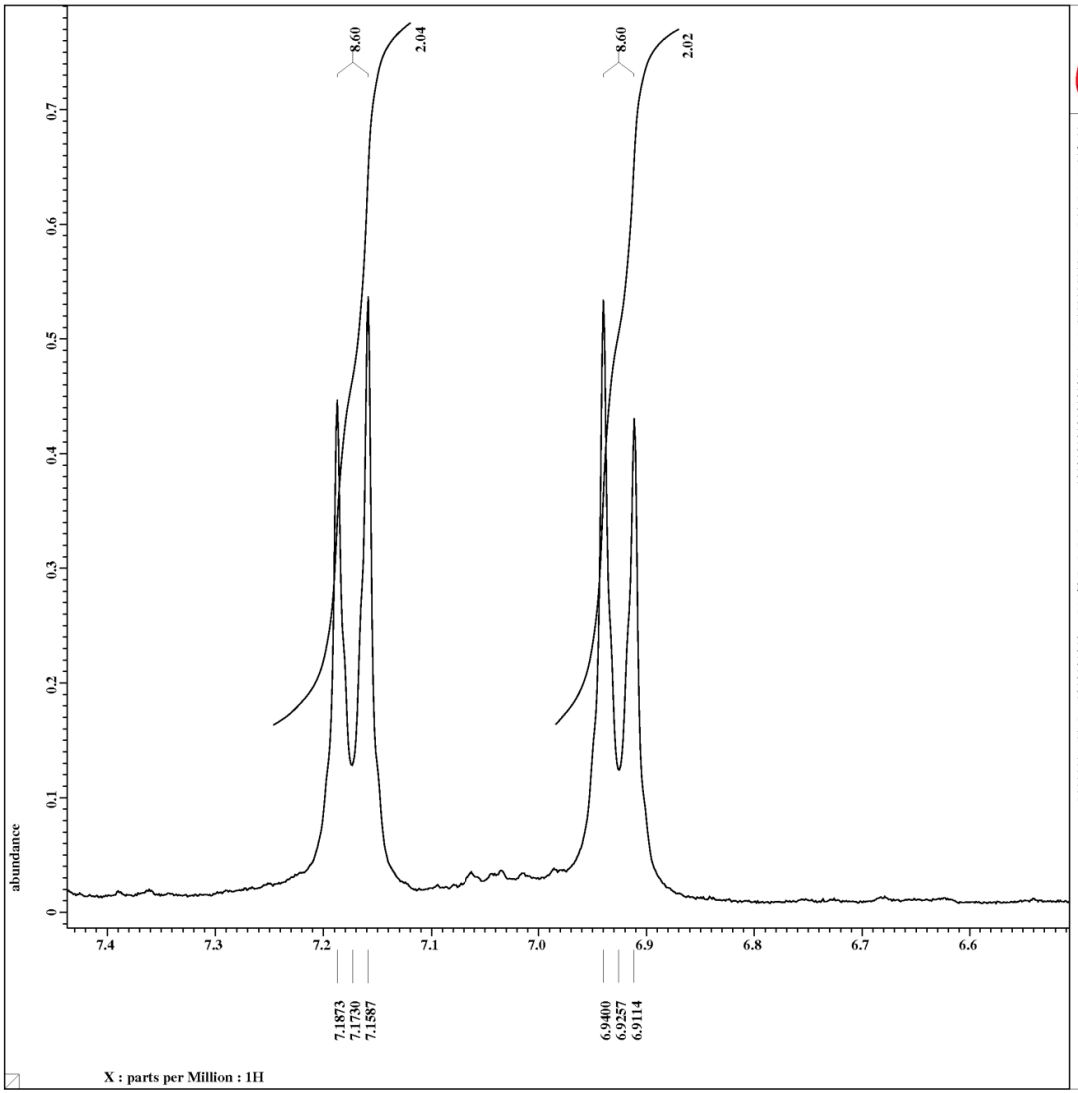

JEOL
Filename = HMF_II_105_pure-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#593051
Solvent = CHLOROFORM-D
Creation_time = 4-OCT-2007 16:50:20
Revision_time = 9-JUN-2011 18:10:09
Current_time = 9-JUN-2011 18:10:56
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DEIFRAZ_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 15
Total_scans = 15
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recyr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 22.8[dC]
CH3
N
N
O
H
H
O
OCH3
3.03
2.01
2.04
1.01
1.01
1.01
10.1609
10.6061
8.0938
8.0503
7.5043
7.4826
7.4757
7.2582
7.0934
7.0648
3.9687
3.8886
abundance
X : parts per Million : 1H


JEOL
Filename = RMF_II_105_pure-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#593051
Solvent = CHLOROFORM-D
Creation_time = 4-OCT-2007 16:50:20
Revision_time = 9-NOV-2011 15:07:37
Current_time = 9-NOV-2011 15:08:05
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 15
Total_scans = 15
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 22.8[dC]
O
H
H
O
CH3
N
N
OCH3
abundance
X : parts per Million : 1H
8.60
2.01
8.60
2.04
1.01
1.01
8.0938
8.0503
7.5043
7.4826
7.4757
7.2582
7.0934
7.0877
7.0791
7.0717
7.0648


Filename = HMF II_105-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#455503
Solvent = CHLOROFORM-D
Creation_time = 19-OCT-2007 13:42:28
Revision_time = 9-JUN-2011 18:12:07
Current_time = 9-JUN-2011 18:12:50
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 320
Total_scans = 320
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 5[s]
Recyr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 7.76824064[s]
Temp_get = 23.2[dC]
abundance
X : parts per Million : 13C
31.6940
55.5048
76.7581
77.1835
77.6042
109.7327
114.0733
125.1349
129.5376
132.7643
133.1420
136.7176
139.7866
145.3413
147.7315
160.2464
192.9390
193.4791

APPENDIX 38
^{1}H and ^{13}C NMR Spectra of
6,7-Dihydroxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]
imidazole-5,8-dione (**124**)


JEOL
Filename = HMF_II_248_pure-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#742564
Solvent = DMSO-D6
Creation_time = 11-MAR-2010 20:18:53
Revision_time = 26-OCT-2011 12:11:24
Current_time = 26-OCT-2011 12:12:04
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIMA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 19
Total_scans = 19
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.9[dC]
OH
OH
O
O
OCH3
CH3
N
N
abundance
X : parts per Million : 1H
9.7522
8.3158
8.1808
7.1873
7.1587
6.9400
6.9114
3.9264
3.7856
2.4717
1.29
1.02
1.01
2.04
2.02
3.02
3



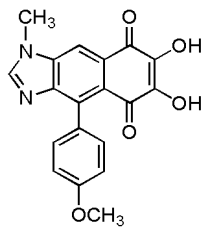
JEOL

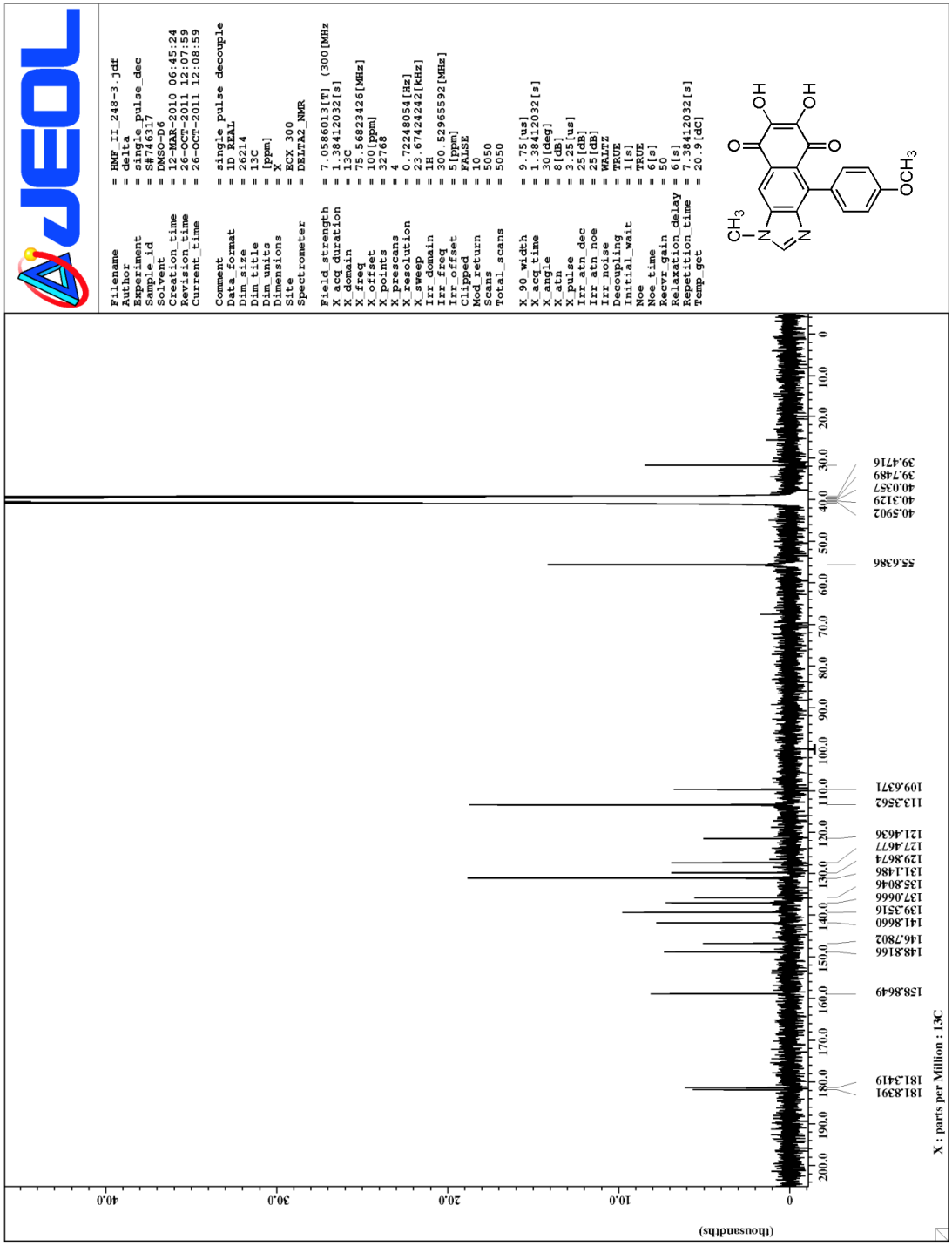
```
Filename          = HMF_II_248_pure-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#742564
Solvent           = DMSO-D6
Creation_time     = 11-MAR-2010 20:18:53
Revision_time     = 26-OCT-2011 12:12:27
Current_time      = 26-OCT-2011 12:12:41

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 300
Spectrometer      = DELTA2_NMR

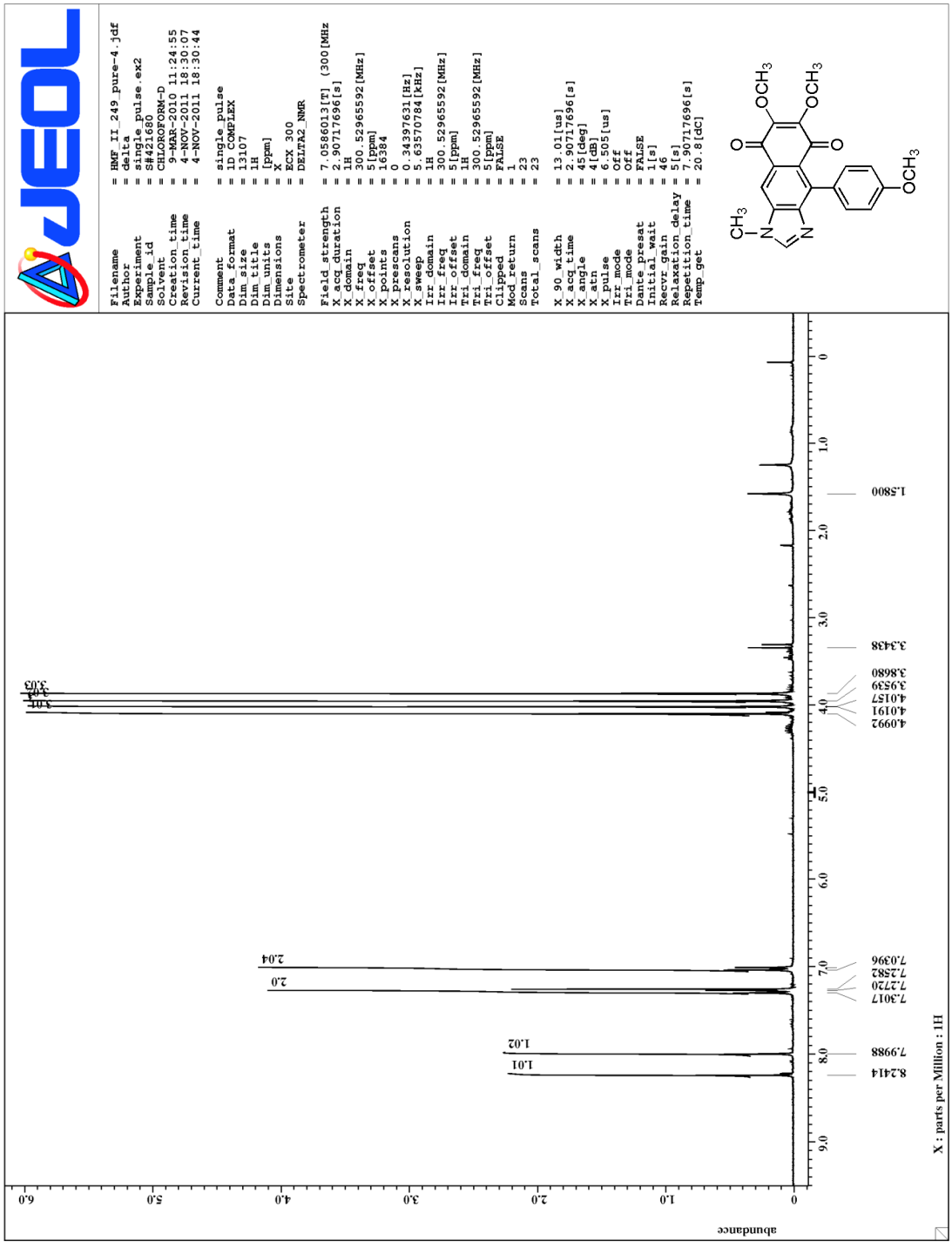
Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 2.90717696[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.34397631[Hz]
X_sweep           = 5.63570784[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 300.52965592[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 19
Total_scans       = 19

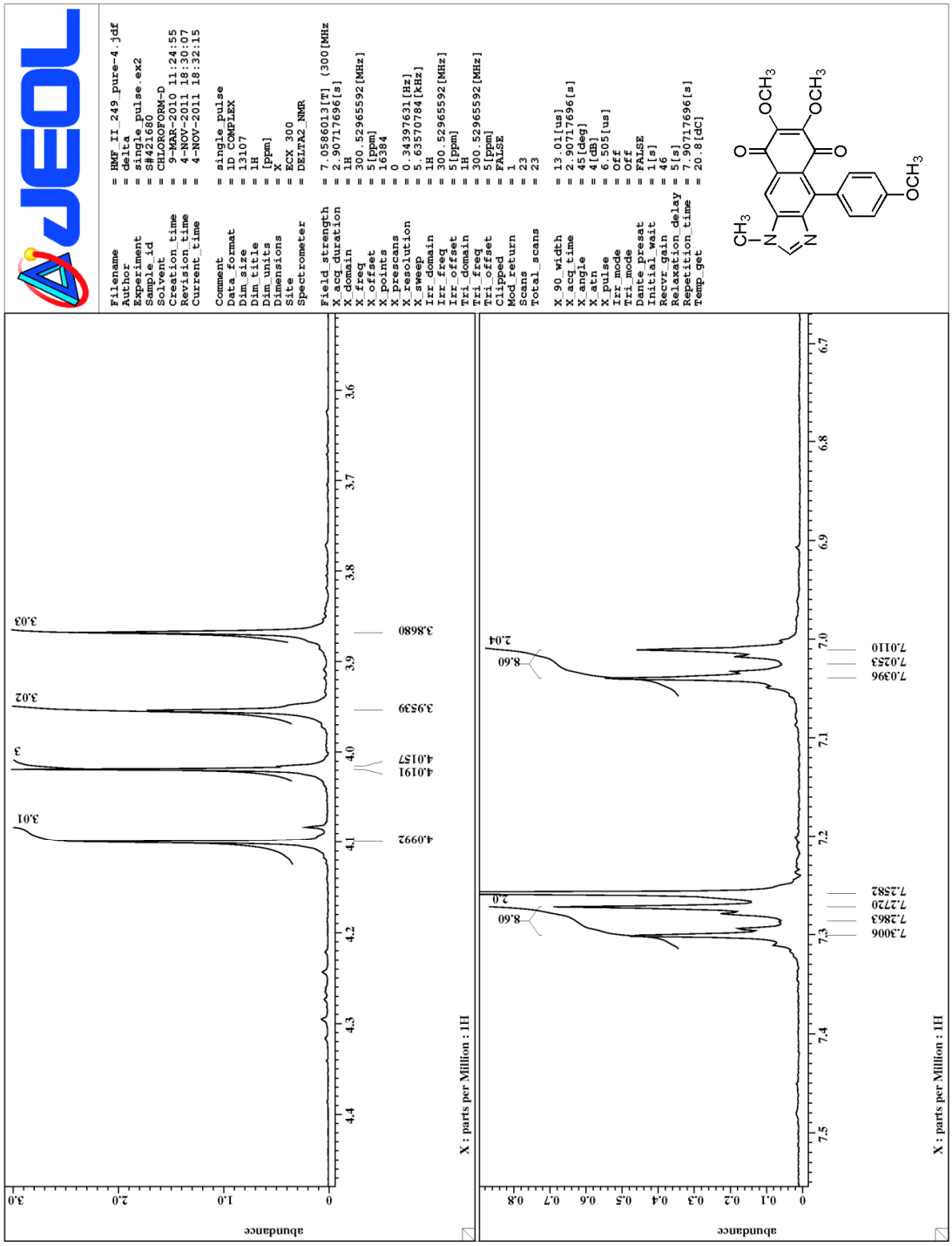
X_90_width        = 13.01[us]
X_acq_time        = 2.90717696[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.505[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 7.90717696[s]
Temp_get          = 20.9[dC]
```

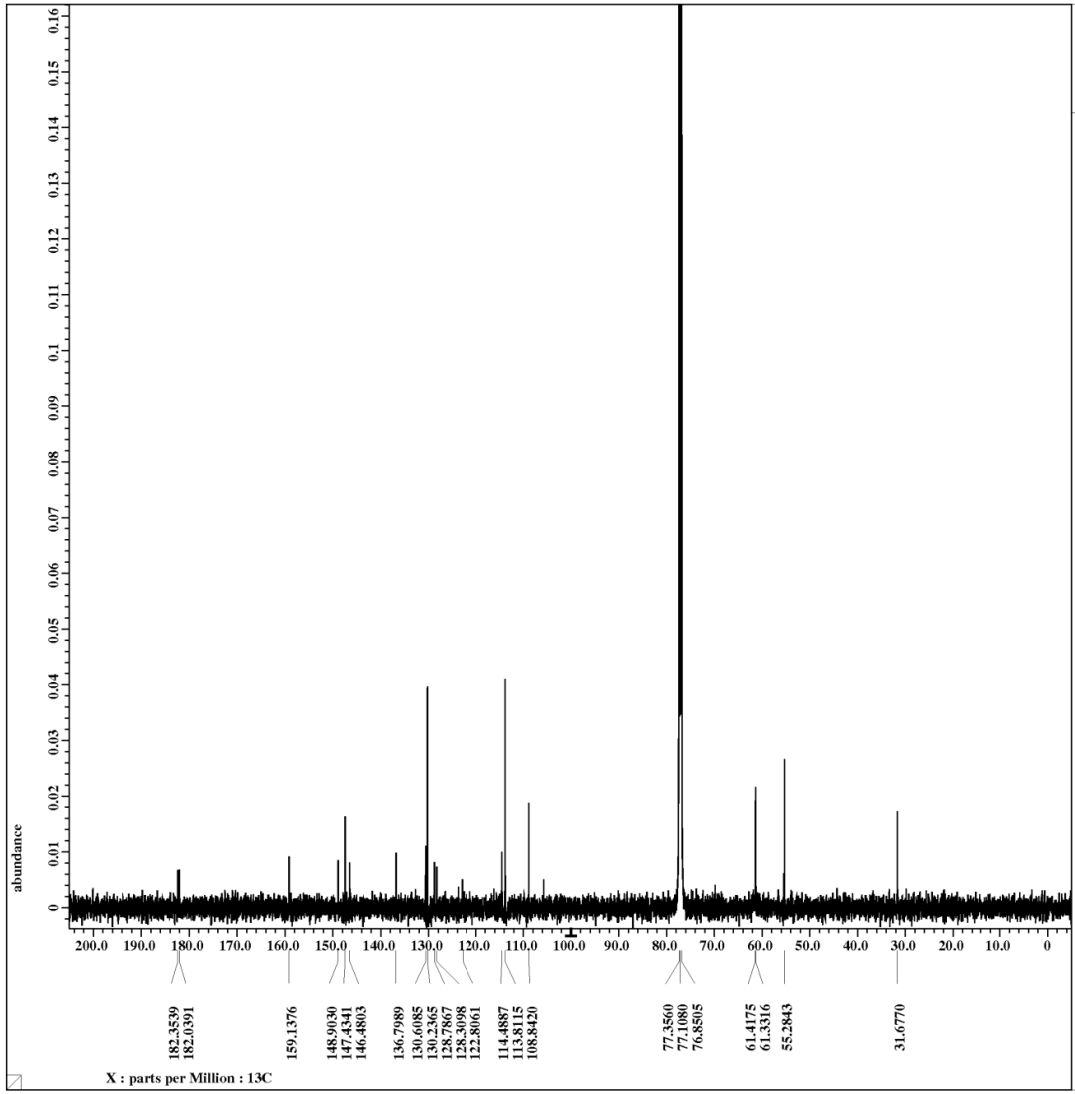



Filename = HMF_II_248-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#746317
Solvent = DMSO-D6
Creation_time = 12-MAR-2010 06:45:24
Revision_time = 26-OCT-2011 12:07:59
Current_time = 26-OCT-2011 12:08:59
Comment = single_pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DEIFPA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.38412032[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.72248054[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 5050
Total_scans = 5050
X_90_width = 9.75[us]
X_acq_time = 1.38412032[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 6[s]
Recvr_gain = 50
Relaxation_delay = 6[s]
Repetition_time = 7.38412032[s]
Temp_get = 20.9[dC]
OH
OH
O
O
CH3
N
N
OCH3
(thousandths)
X : parts per Million : 13C
181.8391
181.3419
158.8649
148.8166
146.7802
141.8660
139.3516
137.0666
135.8046
131.1486
129.8674
127.4677
121.4636
113.3562
109.6371
55.6386
40.5902
40.3129
40.0357
39.7489
39.4716

APPENDIX 39
^{1}H and ^{13}C NMR Spectra of
6,7-Dimethoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]
imidazole-5,8-dione (**103**)


Filename = HMF_II_249_pure-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#421680
Solvent = CHLOROFORM-D
Creation_time = 9-MAR-2010 11:24:55
Revision_time = 4-NOV-2011 18:30:07
Current_time = 4-NOV-2011 18:30:44
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.05860131[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 23
Total_scans = 23
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.8[dC]
OCH3
OCH3
OCH3
CH3
N
N
O
O
abundance
X : parts per Million : 1H
8.2414
7.9988
7.3017
7.2720
7.2582
7.0396
4.0992
4.0191
4.0157
3.9539
3.8680
3.3438
1.5800
1.01
1.02
2.0
2.04
3.01
3.04
3.03


Filename = HMF_II_249_pure-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#421680
Solvent = CHLOROFORM-D
Creation_time = 9-MAR-2010 11:24:55
Revision_time = 4-NOV-2011 18:30:07
Current_time = 4-NOV-2011 18:32:15
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.05860131[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 23
Total_scans = 23
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 20.8[dC]
OCH3
OCH3
OCH3
CH3
N
N
O
O
3.01
3
3.02
3.03
4.0992
4.0191
4.0157
3.9539
3.8680
8.60
2.04
8.60
2.0
7.3006
7.2863
7.2720
7.2582
7.0396
7.0253
7.0110
abundance
X : parts per Million : 1H



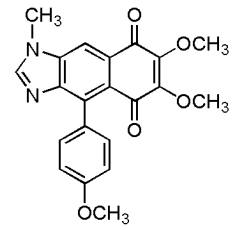
JEOL

```
Filename          = HMF_II_80_5-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#467662
Solvent           = CHLOROFORM-D
Creation_time     = 23-DEC-2011 06:19:46
Revision_time     = 22-DEC-2011 15:50:59
Current_time      = 22-DEC-2011 15:53:23

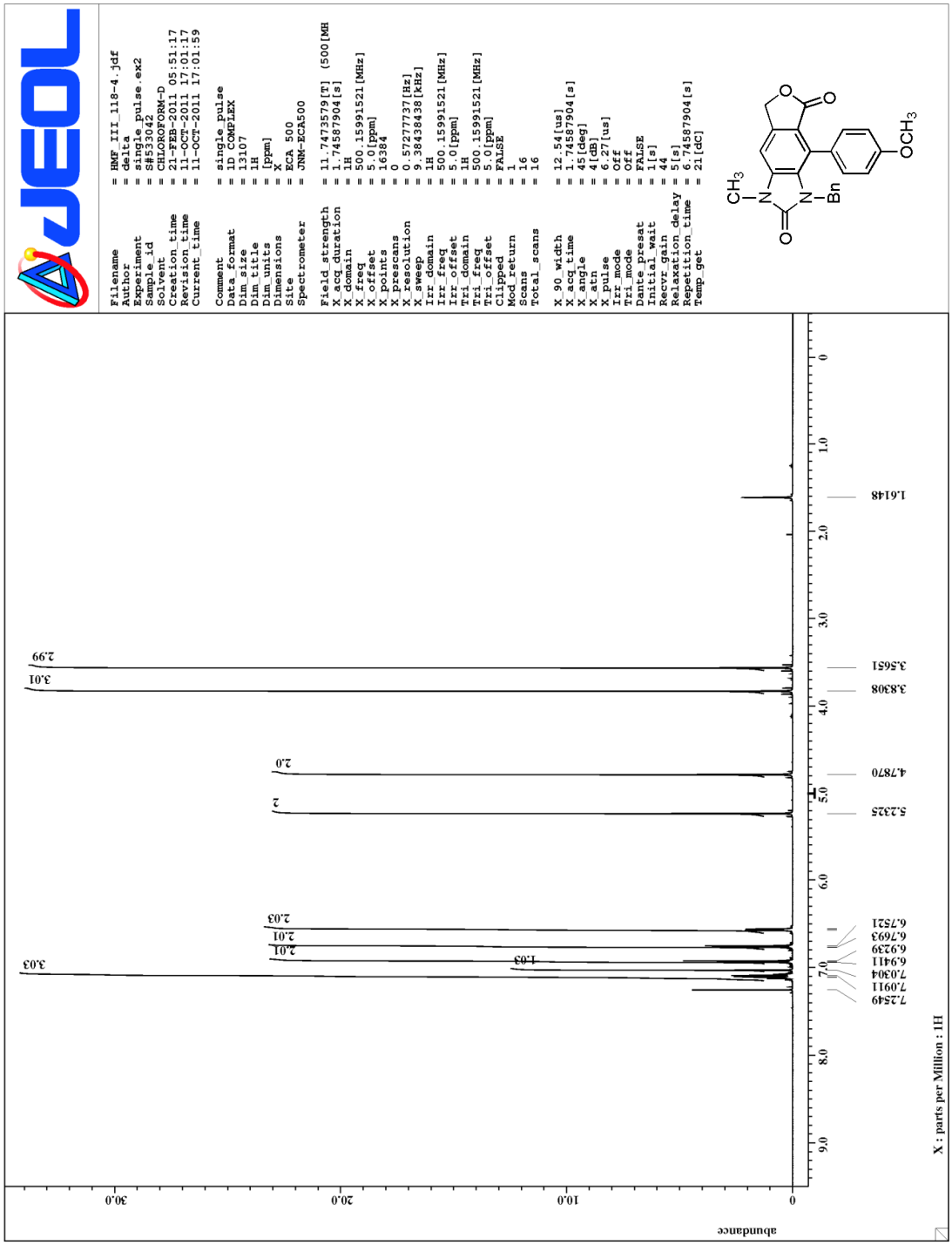
Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

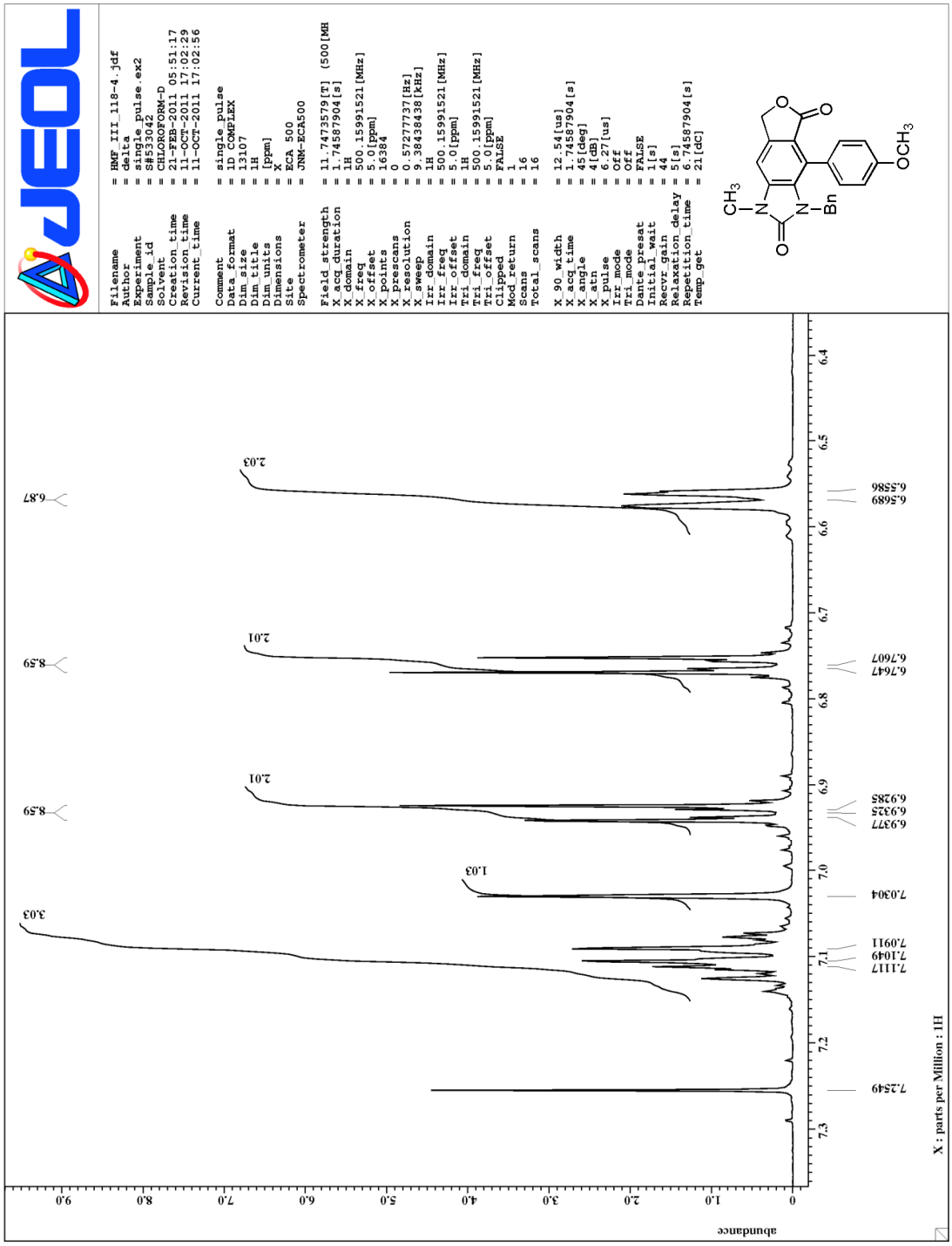
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 1940
Total_scans       = 1940

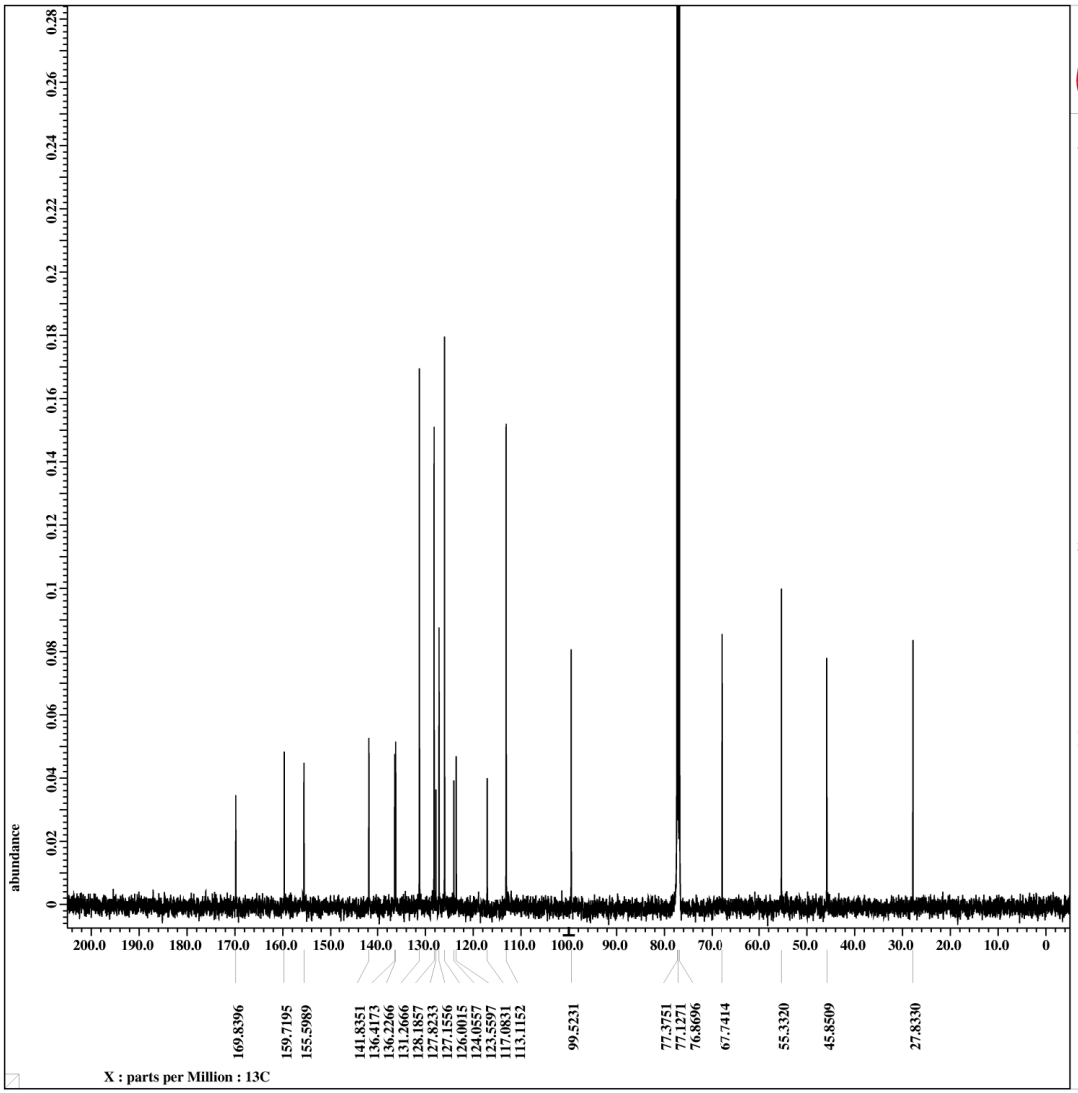
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 22.2[dC]
```



APPENDIX 40
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-4-(4-methoxyphenyl)-1-methyl-3,7-dihydro-1*H*-furo[3,4-*f*]benzimidazole-2,5-dione (**127**)


JEOL
Filename = RMF_III_118-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#533042
Solvent = CHLOROFORM-D
Creation_time = 21-FEB-2011 05:51:17
Revision_time = 11-OCT-2011 17:01:17
Current_time = 11-OCT-2011 17:01:59
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21[dC]
CH3
N
N
Bn
O
O
O
O
OCH3
abundance
X : parts per Million : 1H
3.03
2.99
3.01
2.0
2
2.03
2.01
2.01
1.03
1.6148
3.5651
3.8308
4.7870
5.2325
7.2549
7.0911
7.0304
6.9411
6.9239
6.7693
6.7521


JEOL
Filename = HMF_III_118-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#533042
Solvent = CHLOROFORM-D
Creation_time = 21-FEB-2011 05:51:17
Revision_time = 11-OCT-2011 17:02:29
Current_time = 11-OCT-2011 17:02:56
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21[dC]
CH3
N
N
Bn
O
O
O
OCH3
abundance
X : parts per Million : 1H
7.2549
7.1117
7.1049
7.0911
7.0304
6.9377
6.9325
6.9285
6.7647
6.7607
6.5689
6.5586
3.03
1.03
2.01
2.01
2.03
8.59
8.59
6.87



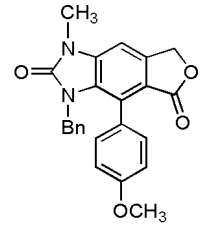
JEOL

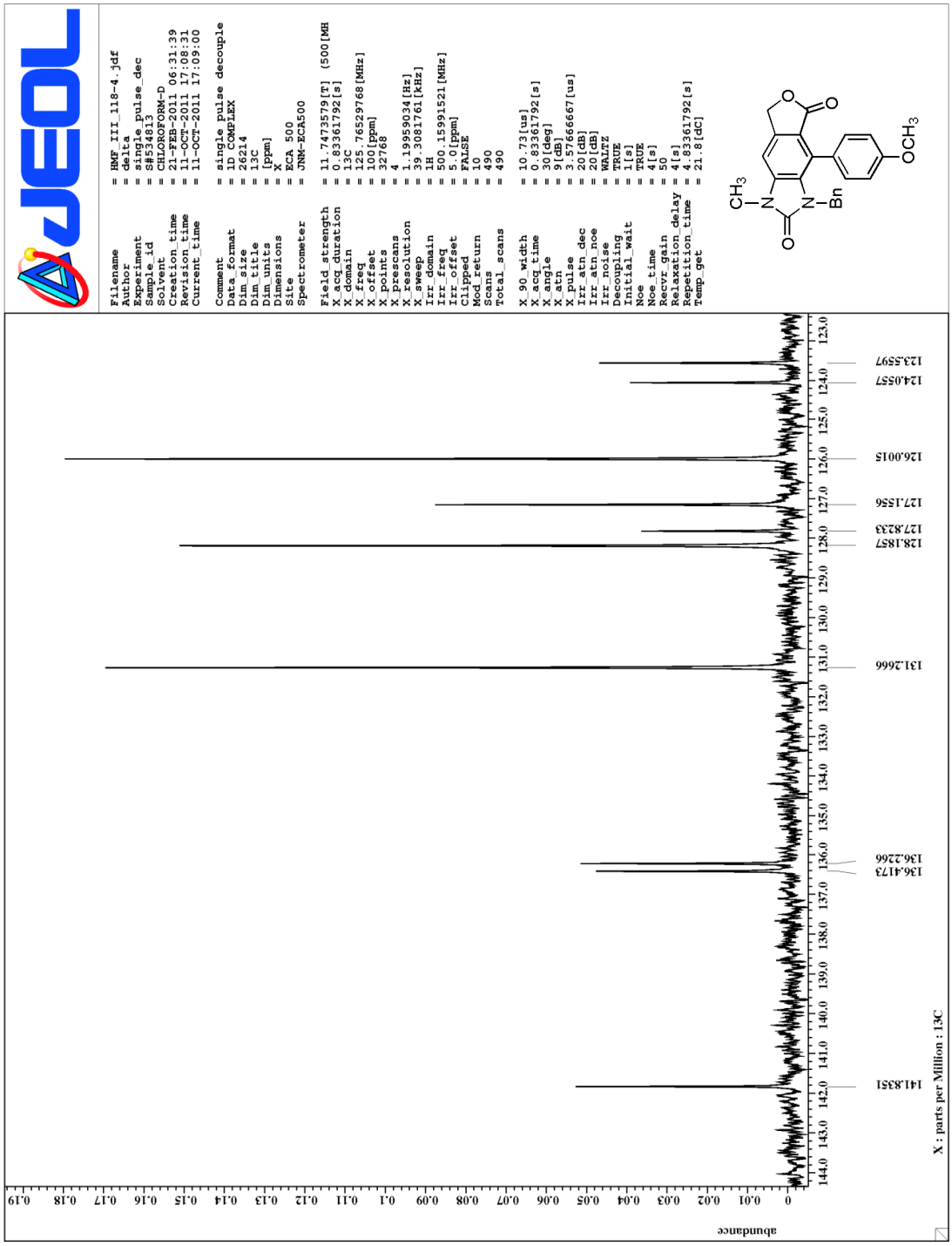
```
Filename          = HMF_III_118-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#534813
Solvent           = CHLOROFORM-D
Creation_time     = 21-FEB-2011 06:31:39
Revision_time     = 20-FEB-2011 17:59:50
Current_time      = 20-FEB-2011 18:00:26

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

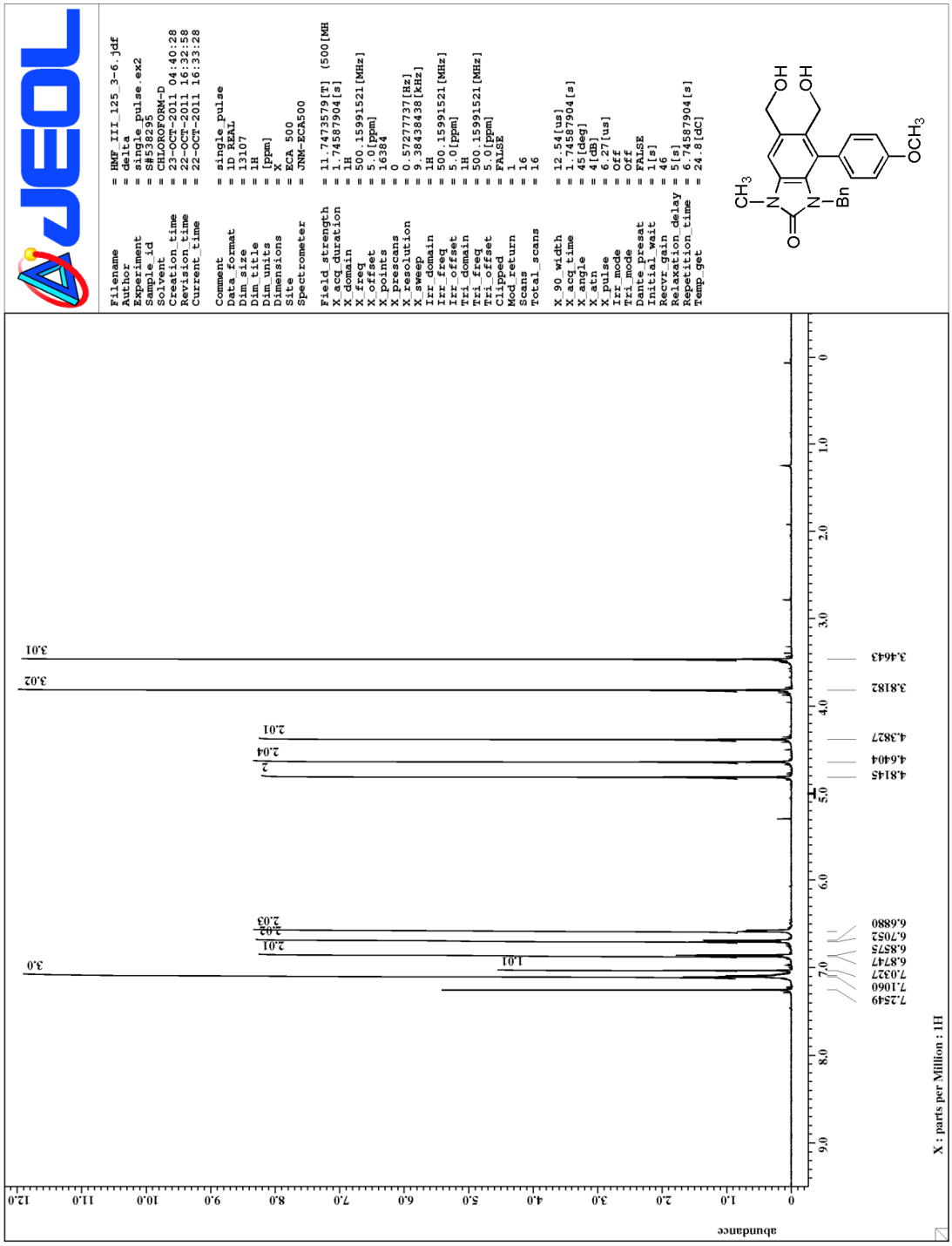
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 490
Total_scans       = 490

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 21.8[dC]
```




JEOL
Filename = HMF_III_118-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#534813
Solvent = CHLOROFORM-D
Creation_time = 21-FEB-2011 06:31:39
Revision_time = 11-OCT-2011 17:08:31
Current_time = 11-OCT-2011 17:09:00
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 490
Total_scans = 490
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.8[dC]
CH3
N
Bn
O
OCH3
123.5597
124.0557
126.0015
127.1556
127.8233
128.1857
131.2666
136.4173
136.2266
141.8351
abundance
X : parts per Million : 13C

APPENDIX 41
^{1}H and ^{13}C NMR Spectra of
[3-Benzyl-4-(4-methoxyphenyl)-1-methyl-1*H*-benzimidazole-5,6-diyl]dimethanol-2-dione (**128**)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Author = delta
Experiment = single_pulse_ex2
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Solvent = CHLOROFORM-D
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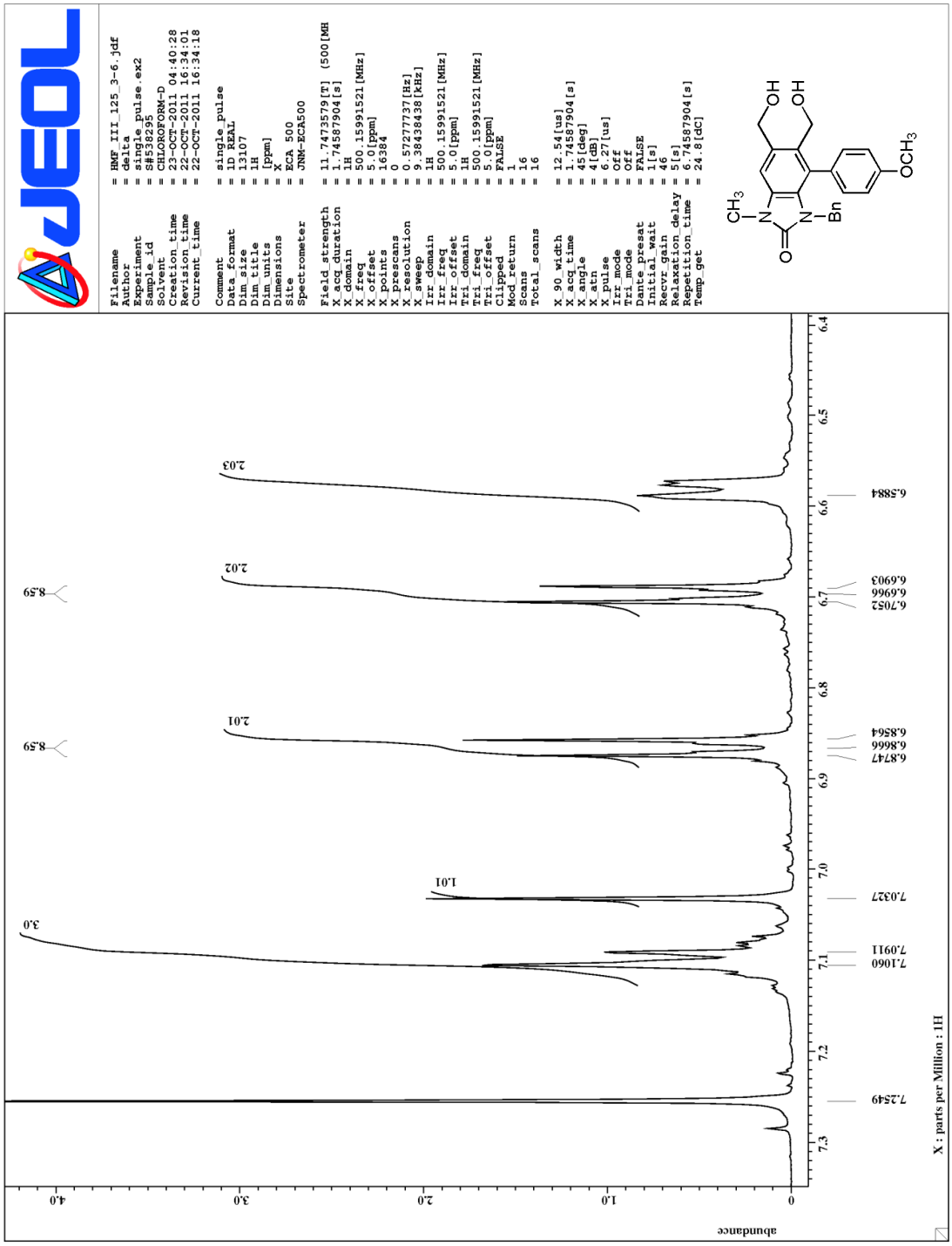
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 24.8[dC]
OH
OH
CH3
N
N
Bn
O
OCH3
abundance
X : parts per Million : 1H
3.0
1.01
2.01
2.03
2.03
2.01
2.04
2.01
3.02
3.01
7.2549
7.1060
7.0327
6.8747
6.8575
6.7052
6.6880
4.8145
4.6404
4.3827
3.8182
3.4643


JEOL
Filename = HMF III_125_3-6.jdf
Author = delta
Experiment = single_pulse_ex2
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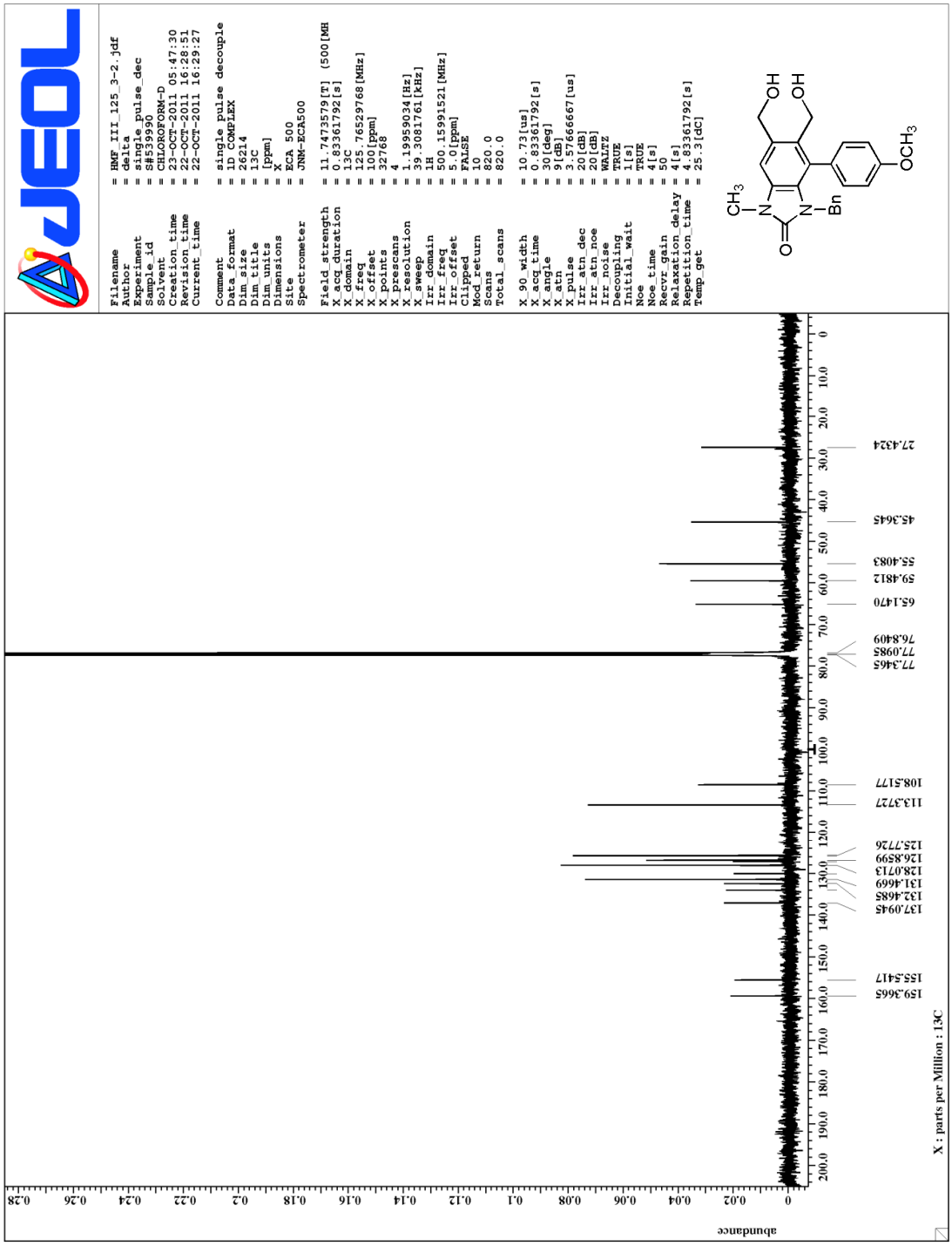
Solvent = CHLOROFORM-D
Creation_time = 23-OCT-2011 04:40:28
Revision_time = 22-OCT-2011 16:34:01
Current_time = 22-OCT-2011 16:34:18
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 24.8[dC]
CH3
N
N
Bn
O
OH
OH
OCH3
abundance
X : parts per Million : 1H
7.2549
7.1060
7.0911
7.0327
6.8747
6.8666
6.8564
6.7052
6.6966
6.6903
6.5884
3.0
1.01
2.01
2.02
2.03
8.59
8.59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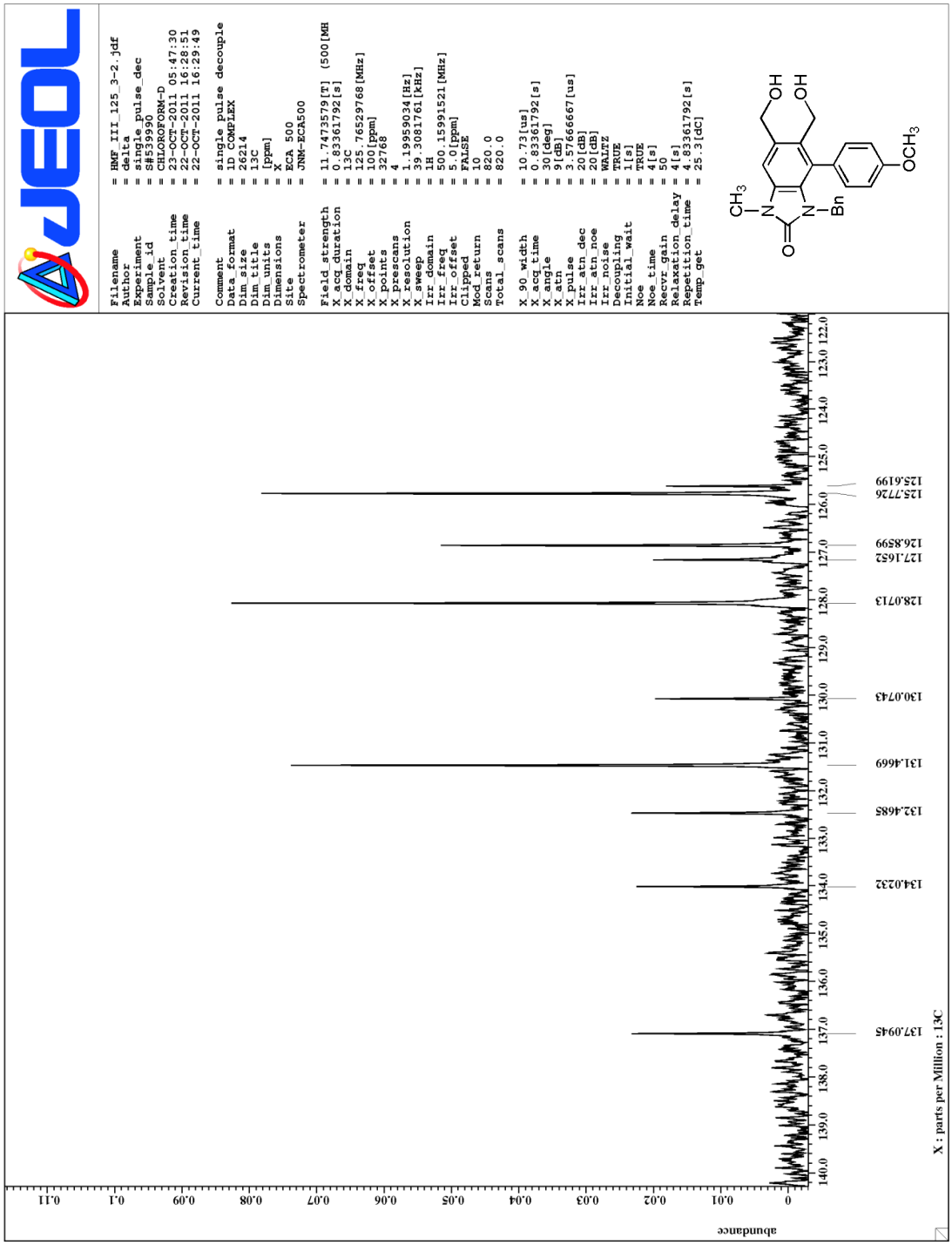
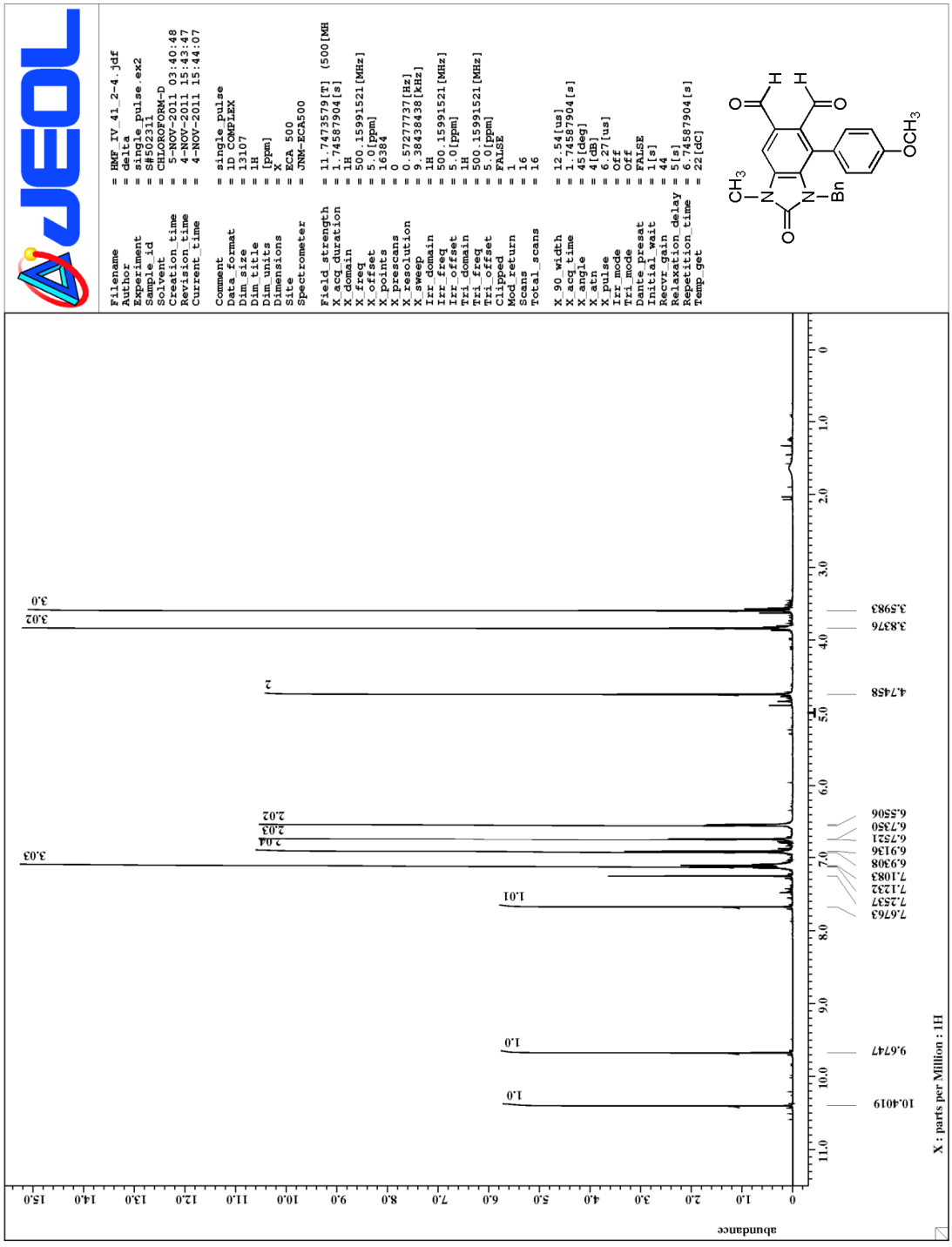
Author = delta
Experiment = single_pulse_dec
Sample_id = S#539990
Solvent = CHLOROFORM-D
Creation_time = 23-OCT-2011 05:47:30
Revision_time = 22-OCT-2011 16:28:51
Current_time = 22-OCT-2011 16:29:27
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 820.0
Total_scans = 820.0
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 25.3[dC]
CH3
N
O
N
Bn
OH
OH
OCH3
abundance
X : parts per Million : 13C
27.4324
45.3645
55.4083
59.4812
65.1470
76.8409
77.0985
77.3465
108.5177
113.3727
125.7726
126.8599
128.0713
131.4669
132.4685
137.0945
155.5417
159.3665


Filename = HMF_III_125_3-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#539990
Solvent = CHLOROFORM-D
Creation_time = 23-OCT-2011 05:47:30
Revision_time = 22-OCT-2011 16:28:51
Current_time = 22-OCT-2011 16:29:49
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 820.0
Total_scans = 820.0
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 25.3[dC]
OH
OH
CH3
N
N
Bn
O
OCH3
abundance
X : parts per Million : 13C
137.0945
134.0232
132.4685
131.4669
130.0743
128.0713
127.1652
126.8599
125.7726
125.6199

APPENDIX 42
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-4-(4-methoxyphenyl)-1-methyl-1*H*-benzimidazole-
5,6-dicarbaldehyde-2-one (**129**)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Solvent = CHLOROFORM-D
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Revision_time = 4-NOV-2011 15:43:47
Current_time = 4-NOV-2011 15:44:07
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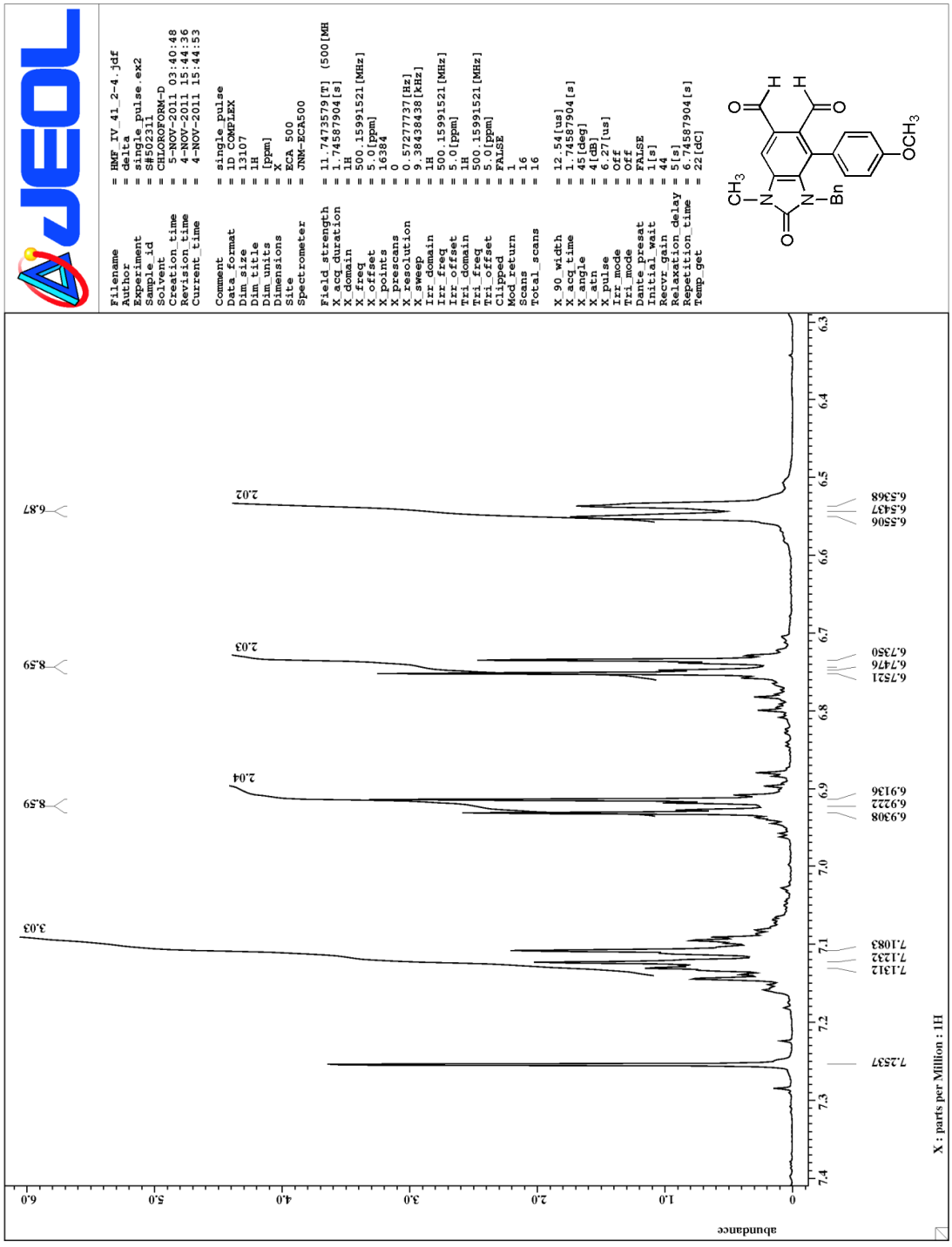
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22[dC]
CH3
N
N
Bn
O
O
H
H
O
OCH3
abundance
X : parts per Million : 1H
3.0
3.02
2
3.03
2.04
2.03
2.02
1.01
1.0
1.0
3.5983
3.8376
4.7458
6.5506
6.7521
6.7730
6.9136
6.9308
7.1083
7.1232
7.2537
7.6763
9.6747
10.4019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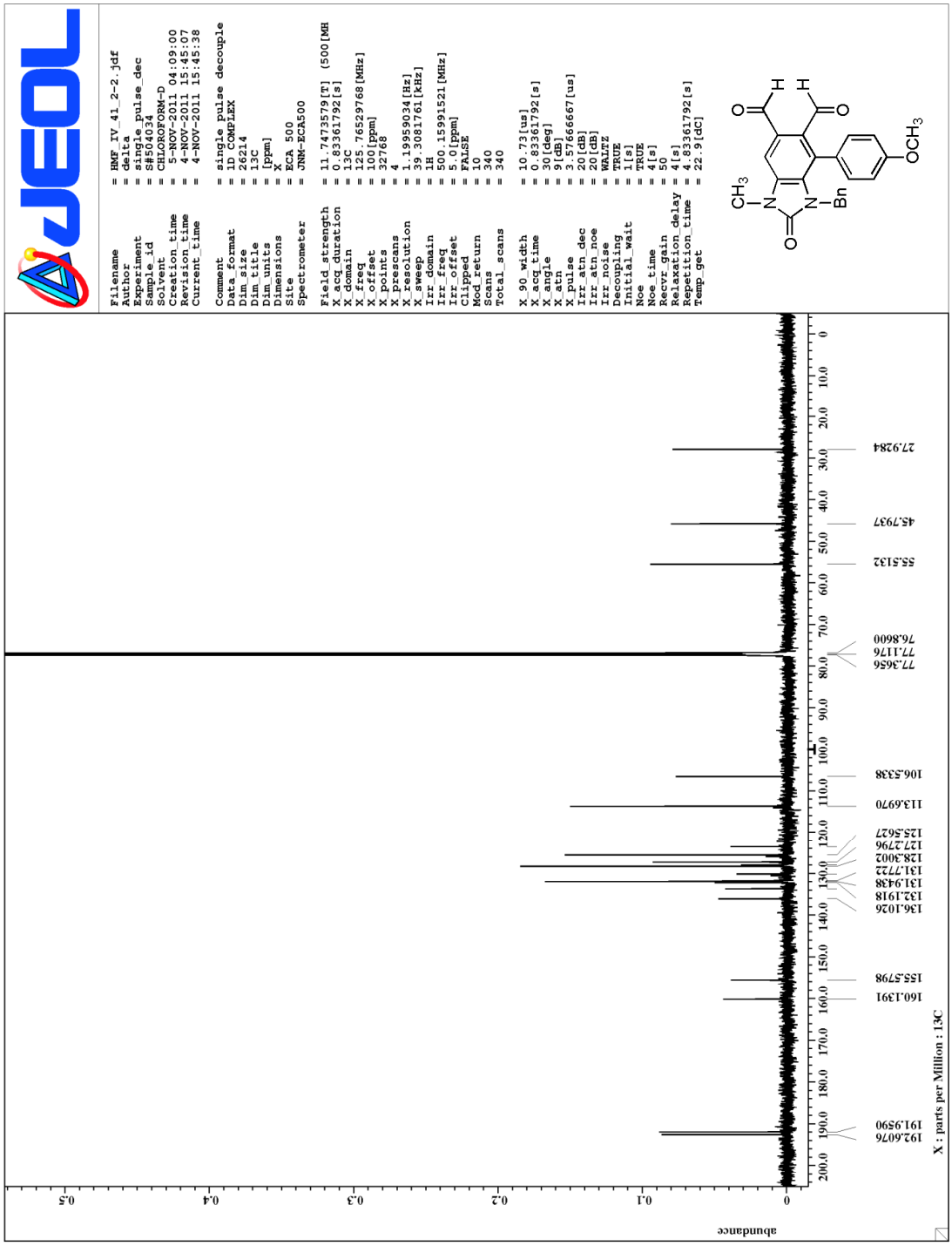
Filename = HMF_IV_41_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#502311
Solvent = CHLOROFORM-D
Creation_time = 5-NOV-2011 03:40:48
Revision_time = 4-NOV-2011 15:44:36
Current_time = 4-NOV-2011 15:44:53
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22[dC]
CH3
N
N
Bn
O
O
H
H
O
OCH3
abundance
X : parts per Million : 1H
3.03
8.59
8.59
6.87
2.04
2.03
2.02
7.2537
7.1312
7.1232
7.1083
6.9308
6.9222
6.9136
6.7521
6.7476
6.7350
6.5506
6.5437
6.5368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Revision_time = 4-NOV-2011 15:45:07
Current_time = 4-NOV-2011 15:45:38
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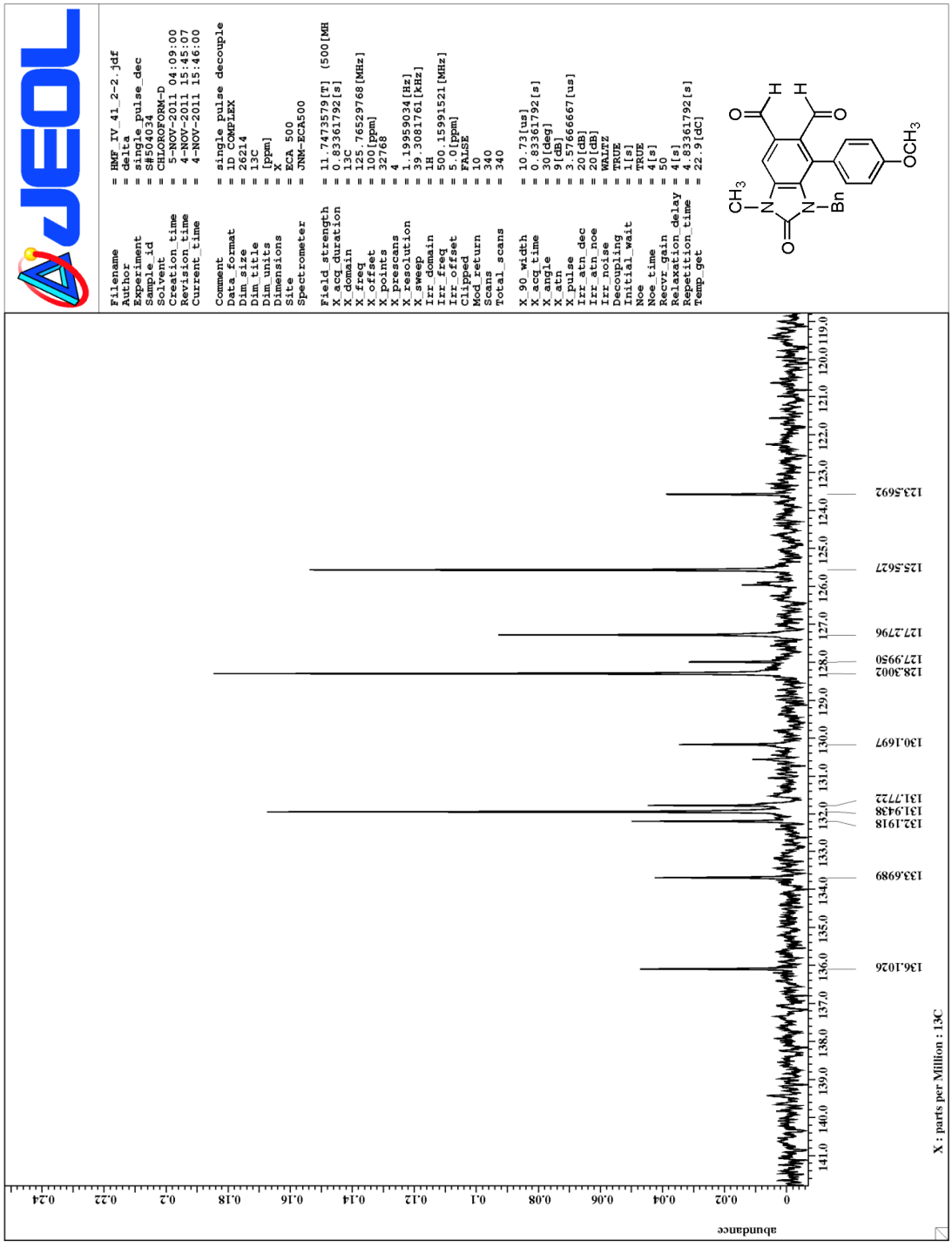
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817611kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 340
Total_scans = 340
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.9[dC]
CH3
N
N
Bn
O
H
H
O
O
CH3
abundance
X : parts per Million : 13C
192.6076
191.9590
160.1391
155.5798
136.1026
132.1918
131.9438
131.7722
128.3002
127.2796
125.5627
113.6970
106.5338
77.3656
77.1176
76.8600
55.5132
45.7937
27.9284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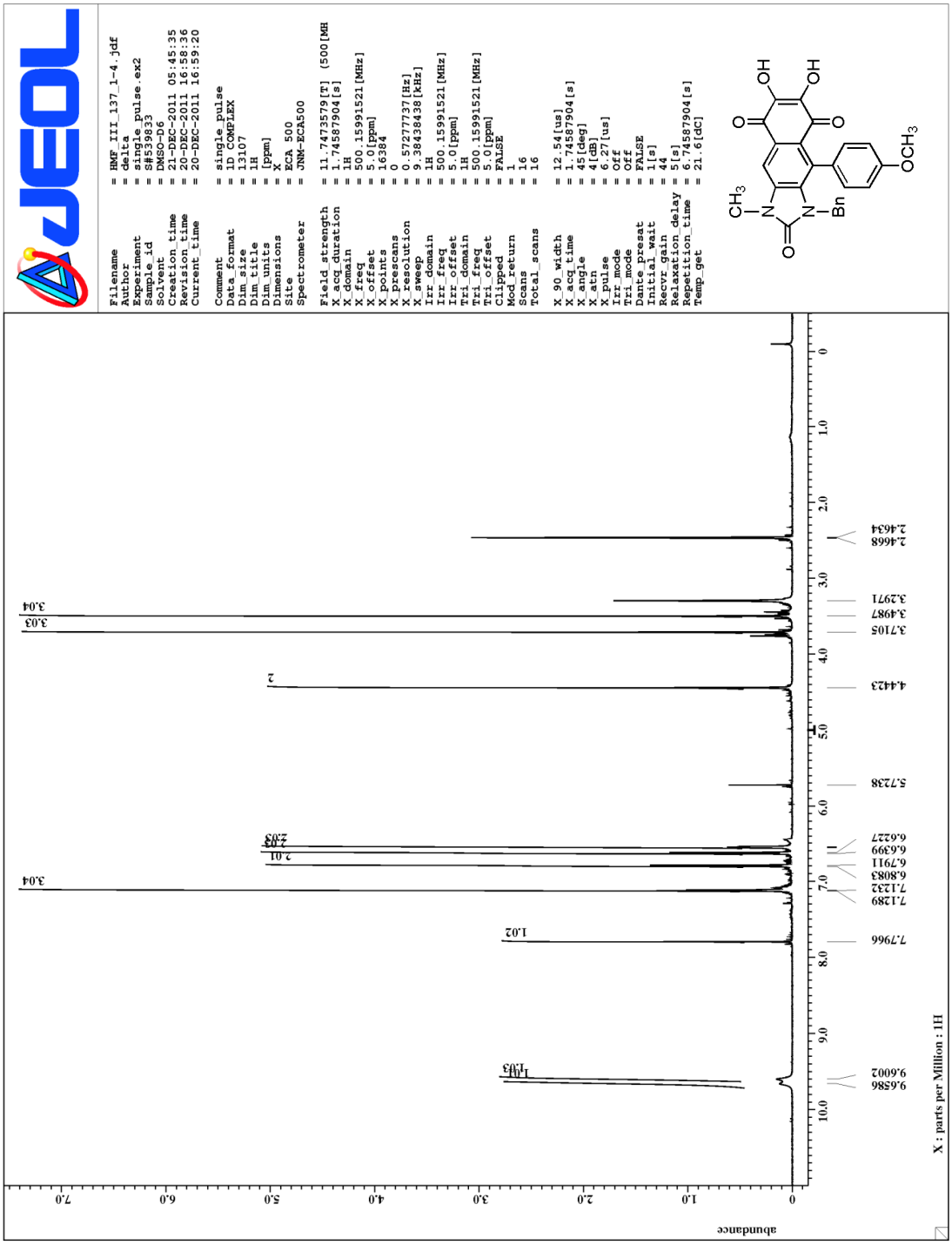
Author = delta
Experiment = single_pulse_dec
Sample_id = S#5040034
Solvent = CHLOROFORM-D
Creation_time = 5-NOV-2011 04:09:00
Revision_time = 4-NOV-2011 15:45:07
Current_time = 4-NOV-2011 15:46:00
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 340
Total_scans = 340
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.9[dC]
abundance
X : parts per Million : 13C
123.5692
125.5627
127.2796
127.9950
128.3002
130.1697
131.7722
131.9438
132.1918
133.6989
136.1026
CH3
N
N
Bn
O
O
H
H
OCH3

APPENDIX 43
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-6,7-dihydroxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho
[2,3-*d*]imidazole-2,5,8-dione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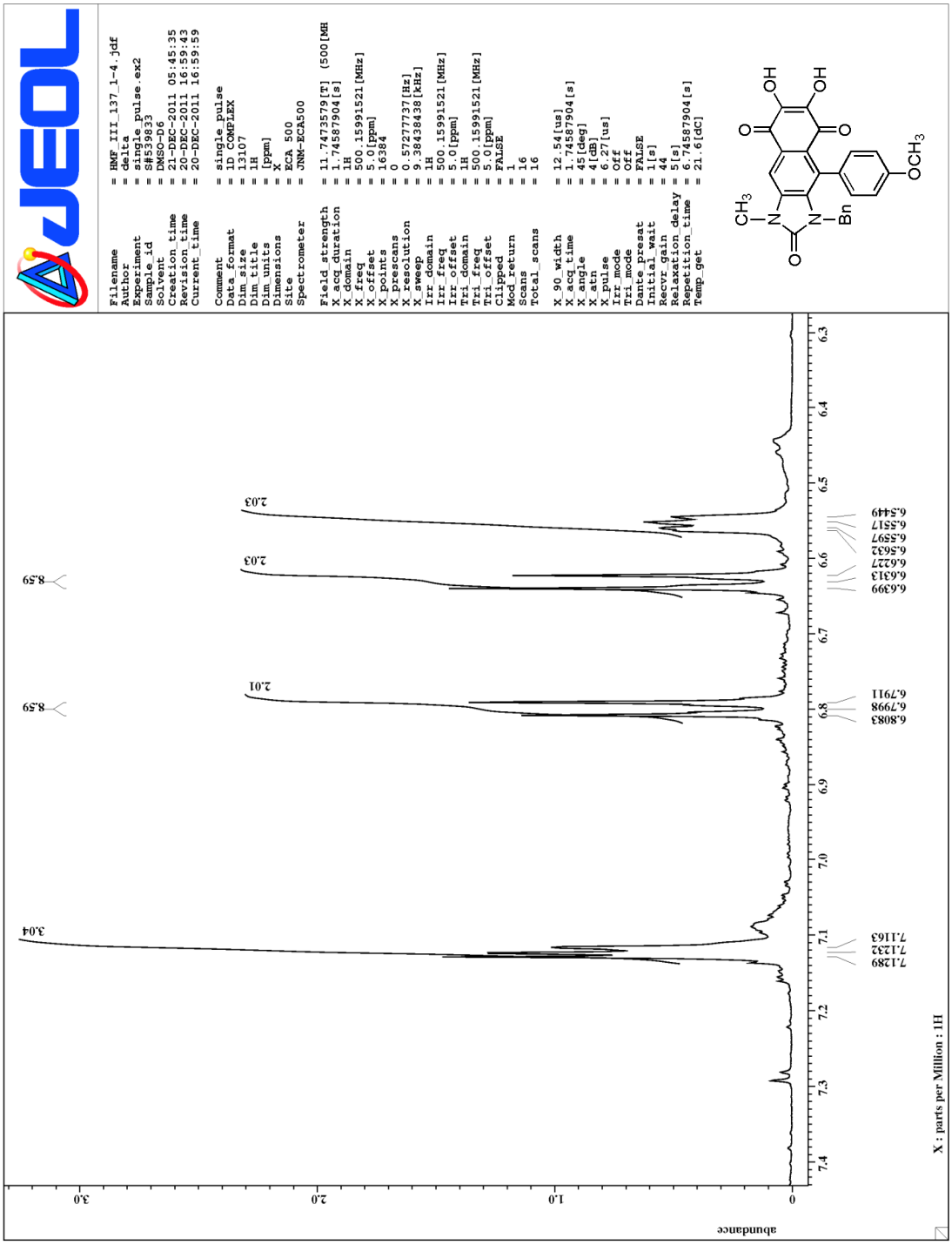
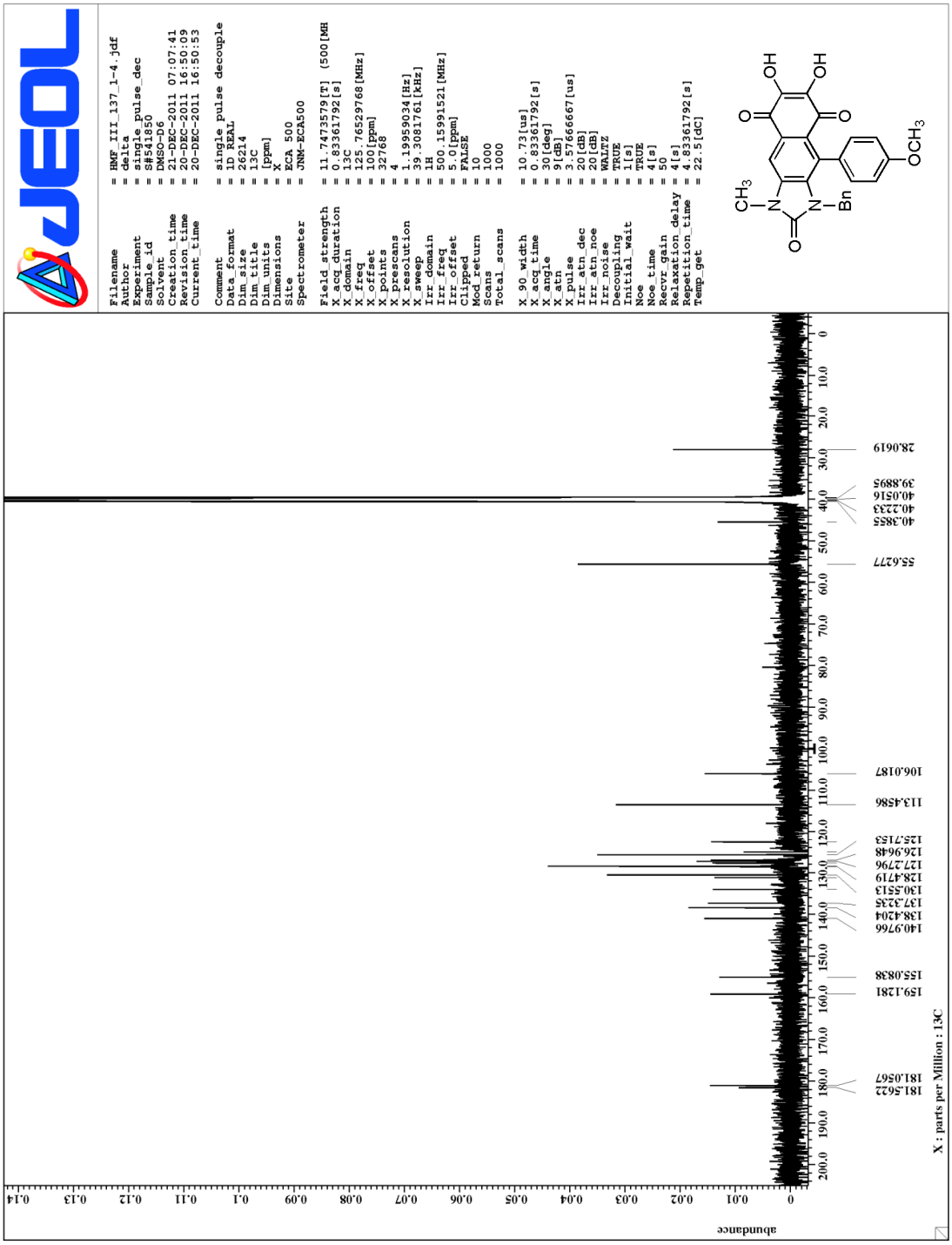
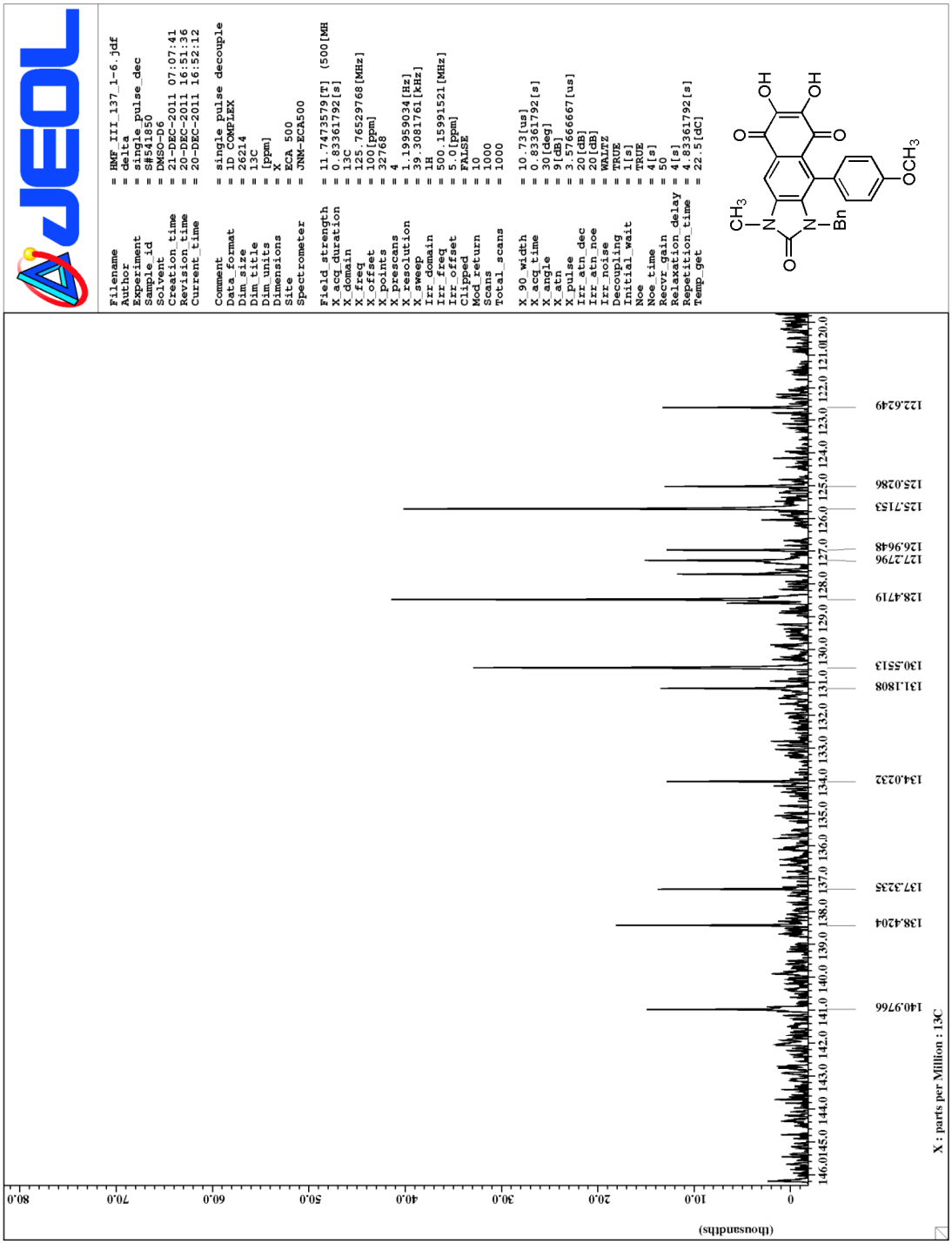
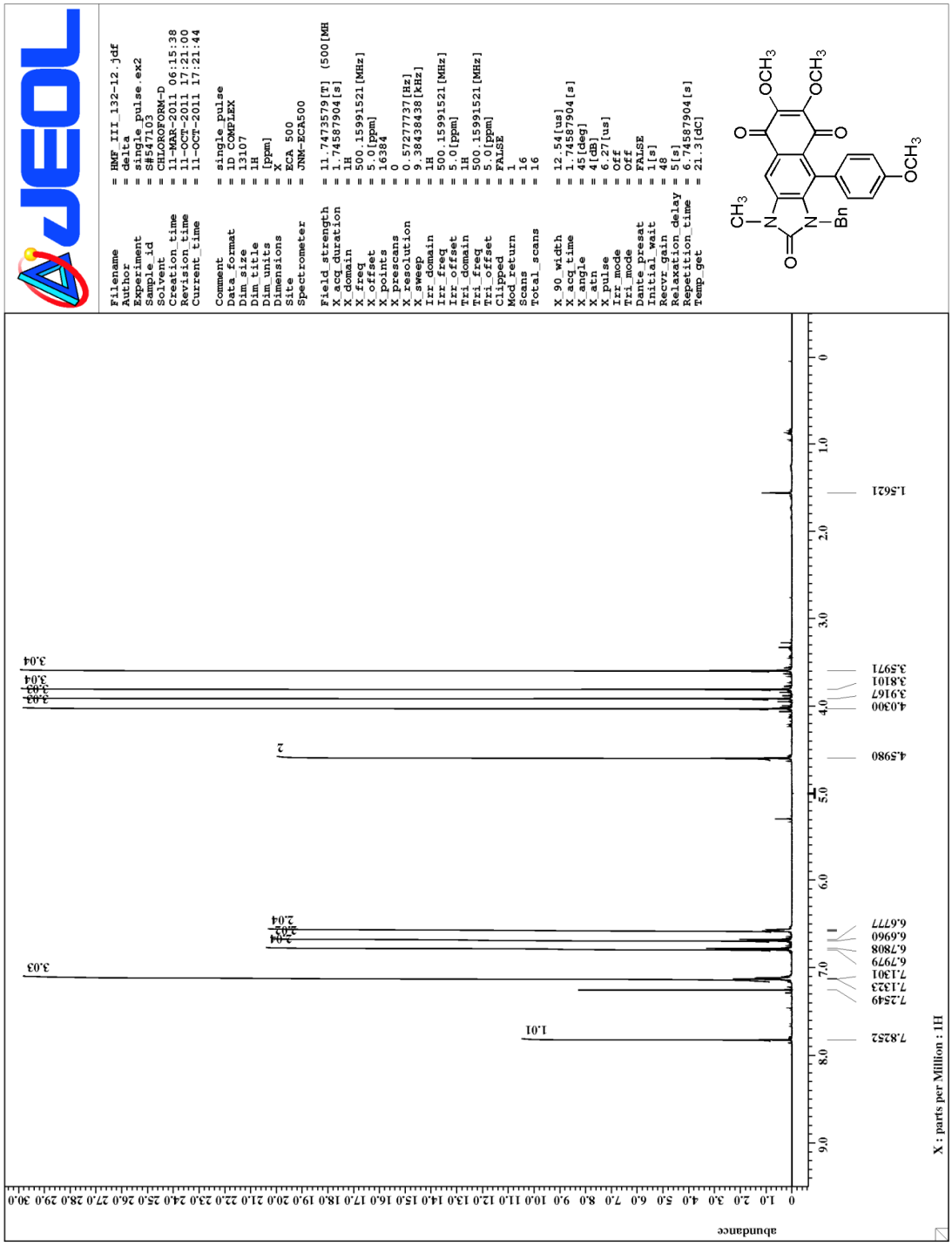
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#539833
Solvent = DMSO-D6
Creation_time = 21-DEC-2011 05:45:35
Revision_time = 20-DEC-2011 16:58:36
Current_time = 20-DEC-2011 16:59:20
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.6[dC]
OH
OH
O
O
O
N
N
CH3
Bn
OCH3
abundance
X : parts per Million : 1H
9.6586
9.6002
7.7966
7.1289
7.1232
6.8083
6.7911
6.6399
6.6227
5.7238
4.4423
3.7105
3.4987
3.2971
2.4668
2.4634
3.04
3.03
2.01
2.03
1.02
1.01
1.03

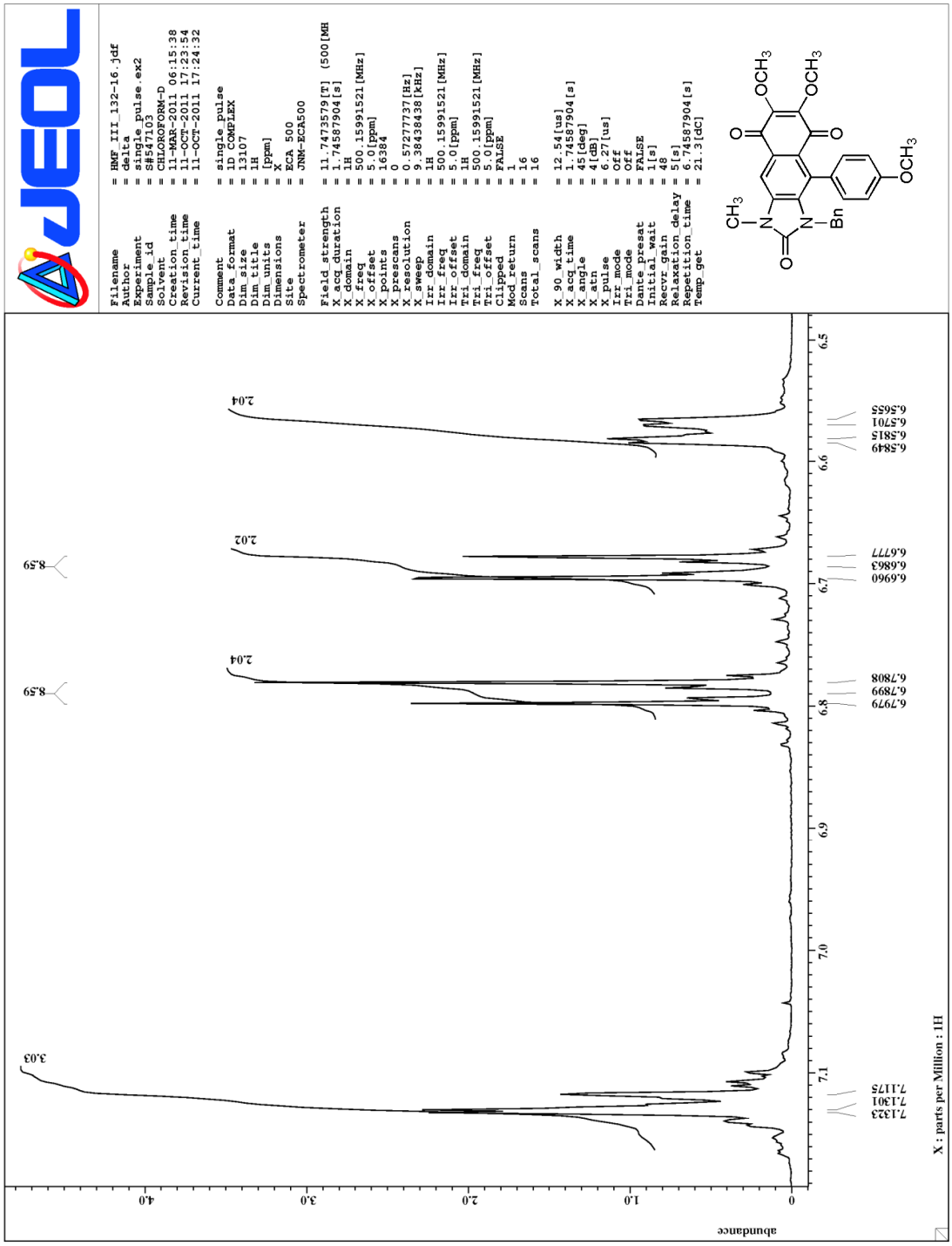

JEOL
Filename = RMF_III_137_1-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#539833
Solvent = DMSO-D6
Creation_time = 21-DEC-2011 05:45:35
Revision_time = 20-DEC-2011 16:59:43
Current_time = 20-DEC-2011 16:59:59
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.6[dC]
OH
OH
O
O
O
O
CH3
N
N
Bn
OCH3
abundance
X : parts per Million : 1H
3.04
8.59
8.59
2.01
2.03
2.03
7.1289
7.1332
7.1163
6.8083
6.7998
6.7911
6.6399
6.6313
6.6227
6.5632
6.5597
6.5517
6.5449

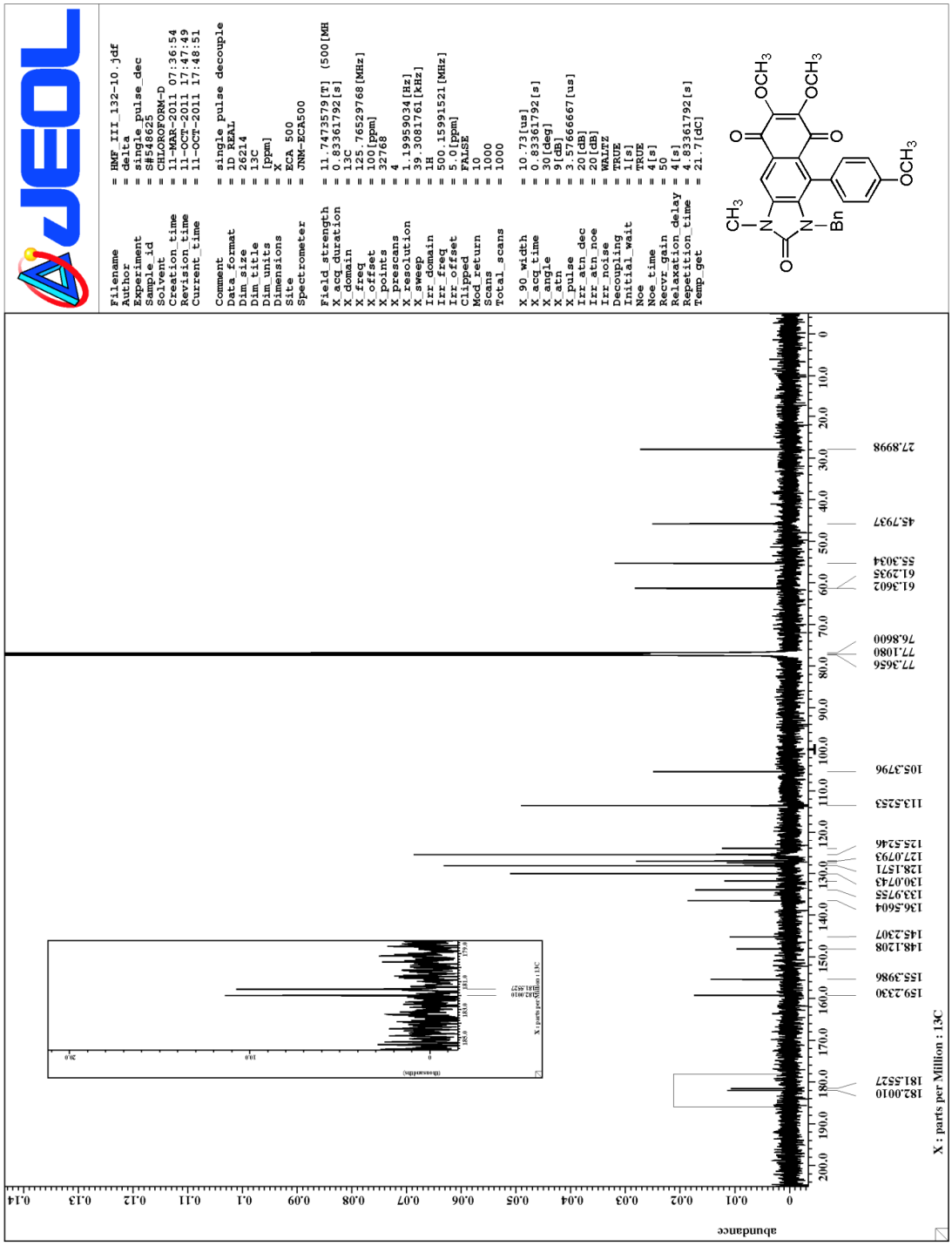

Filename = HMF III 137_1-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#541850
Solvent = DMSO-D6
Creation_time = 21-DEC-2011 07:07:41
Revision_time = 20-DEC-2011 16:50:09
Current_time = 20-DEC-2011 16:50:53
Comment = single_pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1000
Total_scans = 1000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe_time = TRUE
Recyr_gain = 4[s]
Relaxation_delay = 50
Repetition_time = 4[s]
Temp_get = 4.83361792[s]
22.5[dC]
OH
OH
O
O
CH3
N
N
Bn
O
OCH3
abundance
X : parts per Million : 13C
28.0619
39.8895
40.0516
40.2233
40.3855
55.6277
106.0187
113.4586
125.7153
126.9648
127.2796
128.4719
130.5513
137.3235
138.4204
140.9766
155.0838
159.1281
181.0567
181.5622

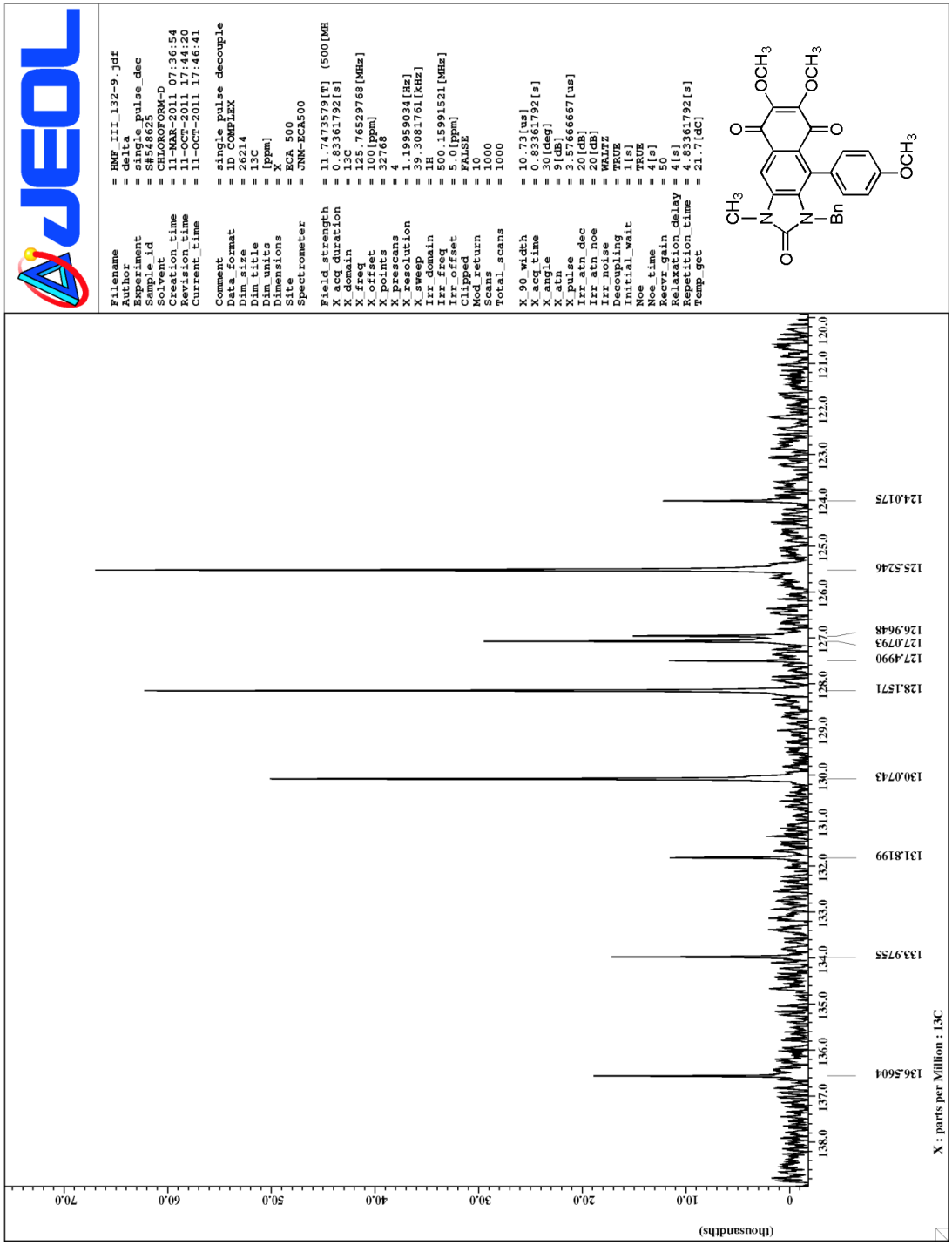

Filename = HMF III 137_1-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#541850
Solvent = DMSO-D6
Creation_time = 21-DEC-2011 07:07:41
Revision_time = 20-DEC-2011 16:51:36
Current_time = 20-DEC-2011 16:52:12
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1000
Total_scans = 1000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.5[dC]
OH
OH
O
O
O
N
N
CH3
Bn
OCH3
122.6249
125.0286
125.7153
126.9648
127.2796
128.4719
130.5513
131.1808
134.0232
137.3235
138.4204
140.9766
(thousandths)
X : parts per Million : 13C

APPENDIX 44
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-6,7-dimethoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]
imidazole-2,5,8-trione (**130**)

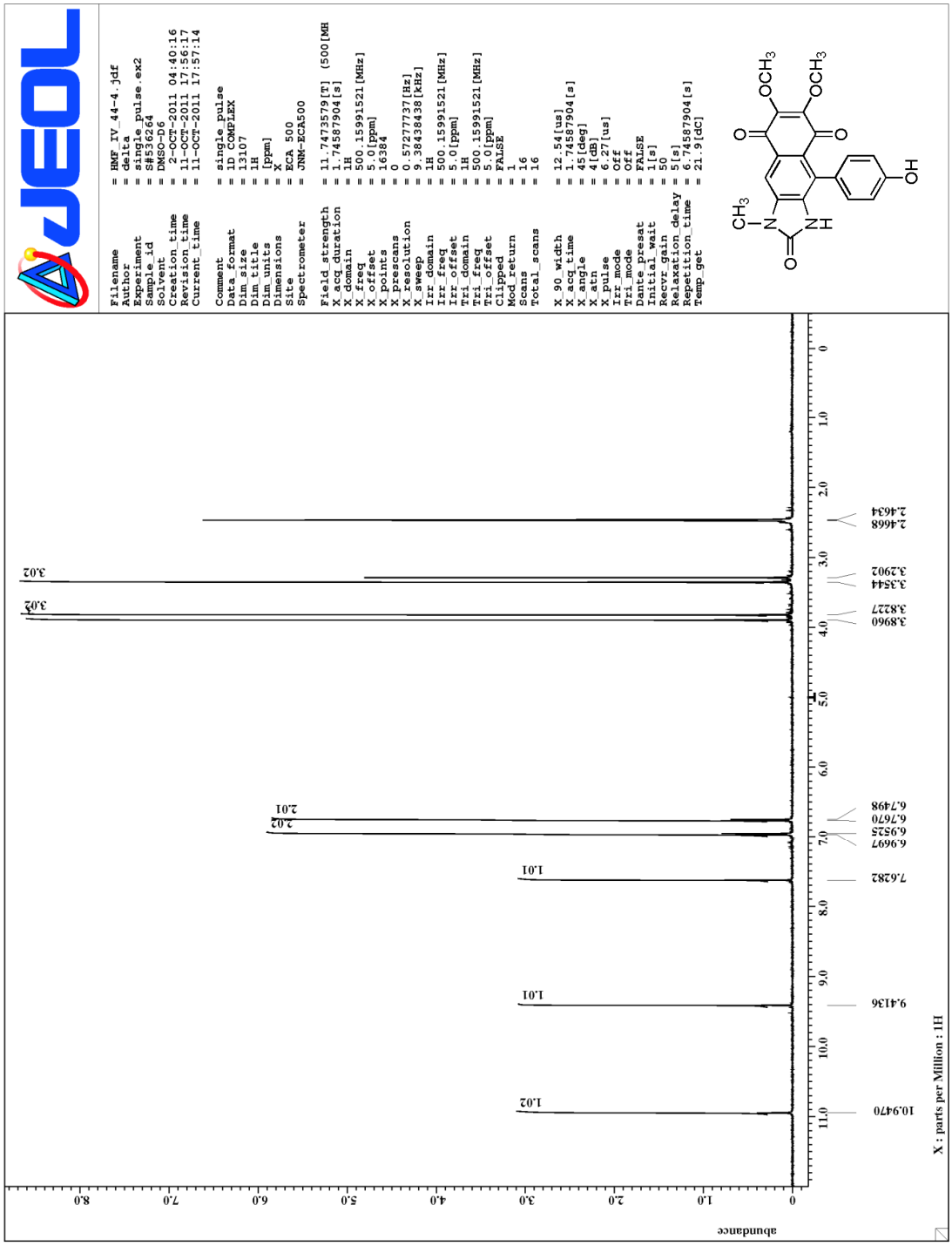

Filename = RMF_III_132-12.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#547103
Solvent = CHLOROFORM-D
Creation_time = 11-MAR-2011 06:15:38
Revision_time = 11-OCT-2011 17:21:00
Current_time = 11-OCT-2011 17:21:44
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.3[dC]
OCH3
OCH3
OCH3
CH3
N
N
Bn
O
O
O
abundance
X : parts per Million : 1H
1.5621
3.5971
3.8101
3.9167
4.0300
4.5980
7.8252
7.2549
7.1323
7.1301
6.7979
6.7808
6.6960
6.6777
3.04
3.03
2.04
1.01
2

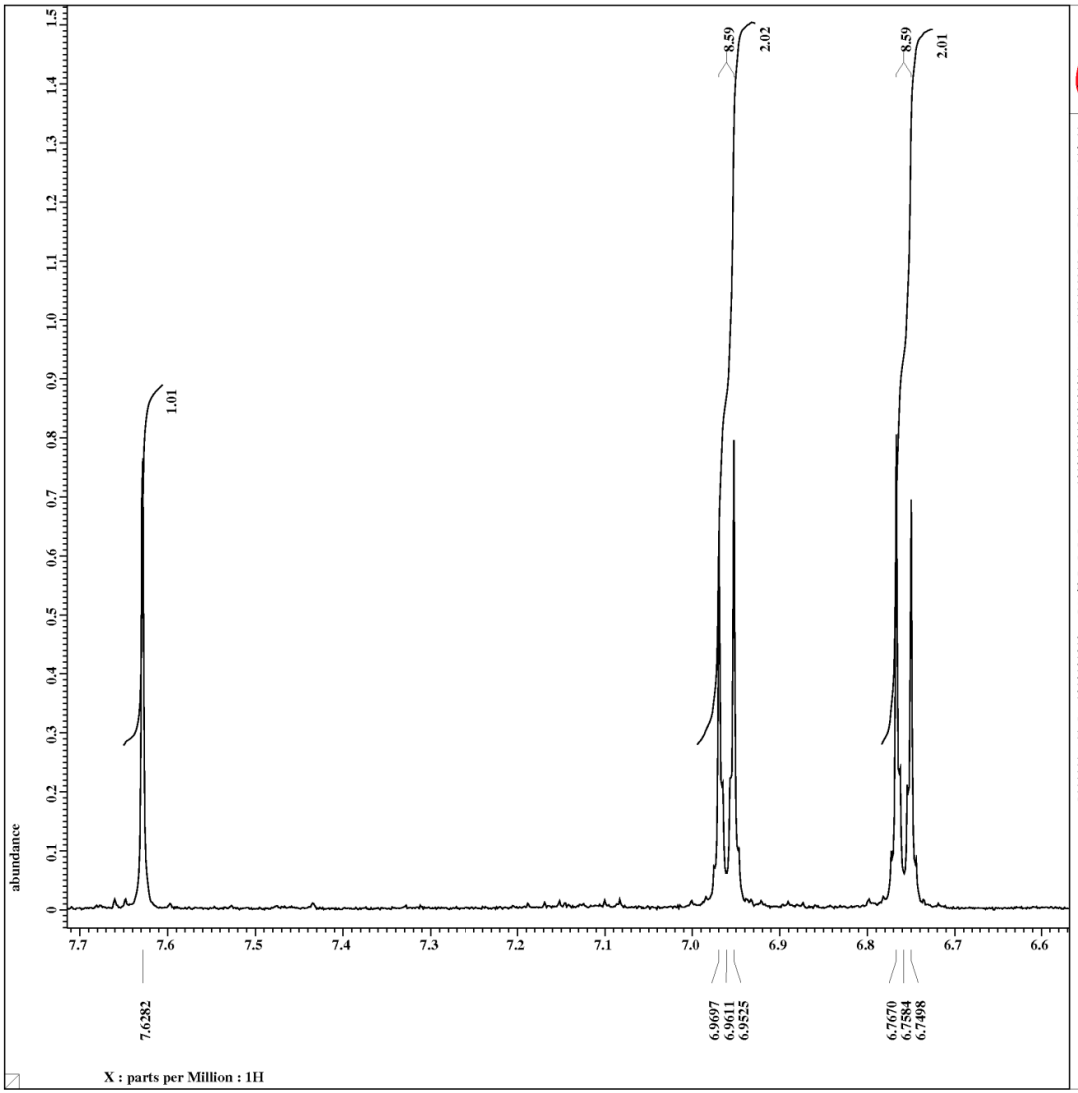

JEOL
Filename = RMF_III_132-16.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#547103
Solvent = CHLOROFORM-D
Creation_time = 11-MAR-2011 06:15:38
Revision_time = 11-OCT-2011 17:23:54
Current_time = 11-OCT-2011 17:24:32
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.3[dC]
OCH3
OCH3
OCH3
CH3
N
N
Bn
O
O
O
abundance
X : parts per Million : 1H
3.03
8.59
8.59
8.59
2.04
2.02
2.04
2.04
7.1323
7.1301
7.1175
6.7979
6.7899
6.7808
6.6960
6.6863
6.6777
6.5849
6.5815
6.5701
6.5655


JEOL
Filename = HMF_III_132-10.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#548625
Solvent = CHLOROFORM-D
Creation_time = 11-MAR-2011 07:36:54
Revision_time = 11-OCT-2011 17:47:49
Current_time = 11-OCT-2011 17:48:51
Comment = single_pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1000
Total_scans = 1000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.7[dC]
OCH3
OCH3
OCH3
CH3
N
N
Bn
O
O
O
abundance
X : parts per Million : 13C
182.0010
181.5527
159.2330
155.3986
148.1208
145.2307
136.5604
133.9755
130.0743
128.1571
127.0793
125.5246
113.5253
105.3796
77.3656
77.1080
76.8600
61.3602
61.2935
55.3034
45.7937
27.8998


Filename = HMF_III_132-9.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#548625
Solvent = CHLOROFORM-D
Creation_time = 11-MAR-2011 07:36:54
Revision_time = 11-OCT-2011 17:44:20
Current_time = 11-OCT-2011 17:46:41
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1000
Total_scans = 1000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recyr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 21.7[dC]
OCH3
OCH3
OCH3
CH3
N
N
Bn
O
O
O
O
(thousandths)
X : parts per Million : 13C
124.0175
125.5246
126.9648
127.0793
127.4990
128.1571
130.0743
131.8199
133.9755
136.5604

APPENDIX 45
^{1}H and ^{13}C NMR Spectra of
4-(4-hydroxyphenyl)-6,7-dimethoxy-1-methyl-1*H*-naphtho[2,3-*d*]
imidazole-2,5,8-trione (**131**)


JEOL
Filename = HMF_IV_44-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#536264
Solvent = DMSO-D6
Creation_time = 2-OCT-2011 04:40:16
Revision_time = 11-OCT-2011 17:56:17
Current_time = 11-OCT-2011 17:57:14
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
OCH3
OCH3
CH3
N
NH
O
OH
abundance
X : parts per Million : 1H
10.9470
9.4136
7.6282
6.9697
6.9525
6.7670
6.7498
3.8960
3.8227
3.3544
3.2902
2.4668
2.4634
1.02
1.01
1.01
1.02
2.02
2.01
3.02
3.02

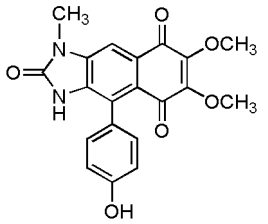


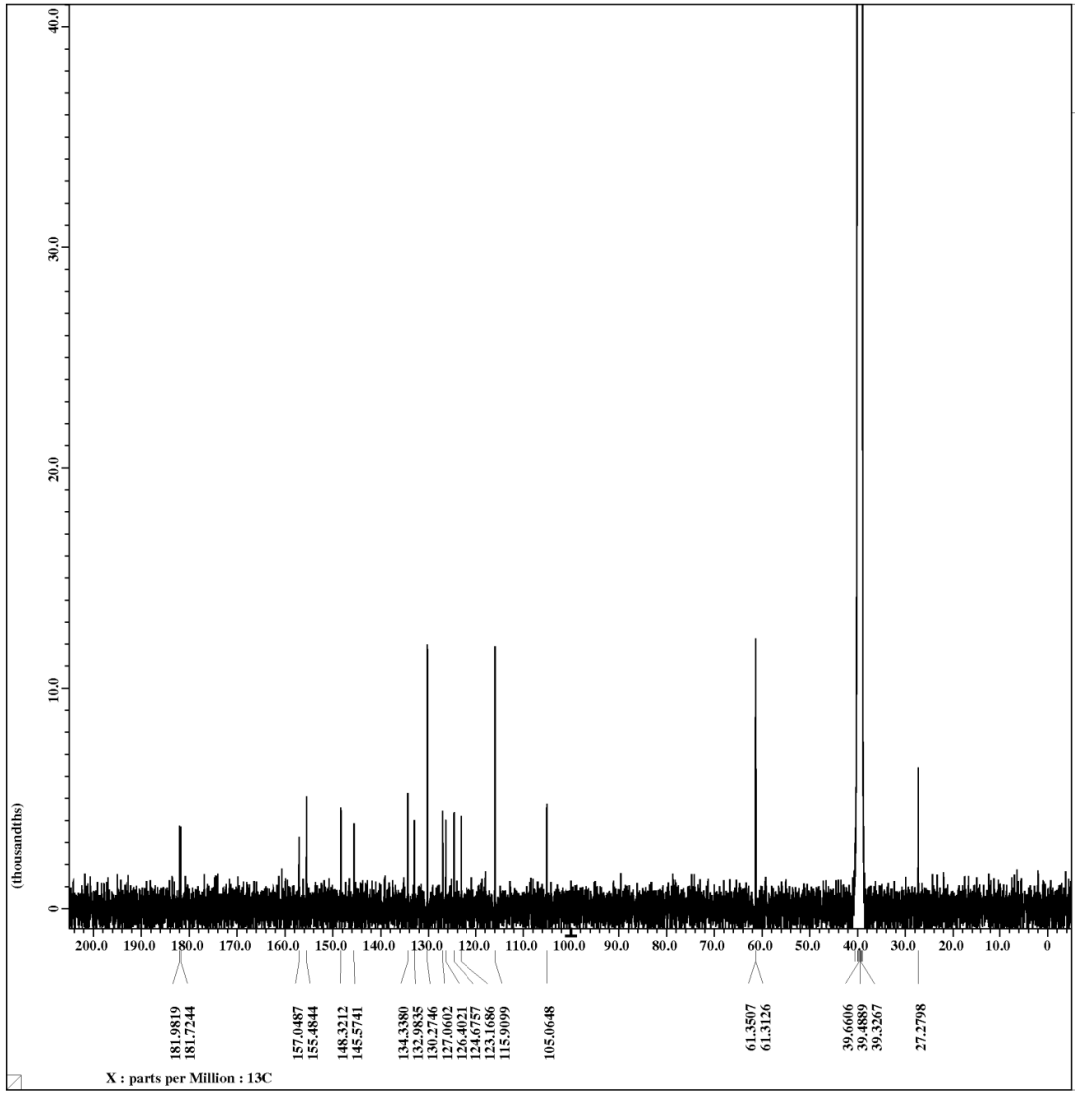

```
Filename          = HMF_IV_44-7.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#536264
Solvent           = DMSO-D6
Creation_time     =  2-OCT-2011 04:40:16
Revision_time     = 11-OCT-2011 17:58:44
Current_time      = 11-OCT-2011 17:59:14

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 21.9[dC]
```



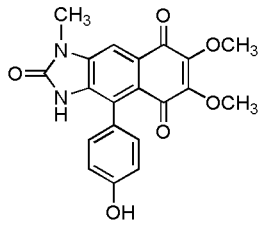


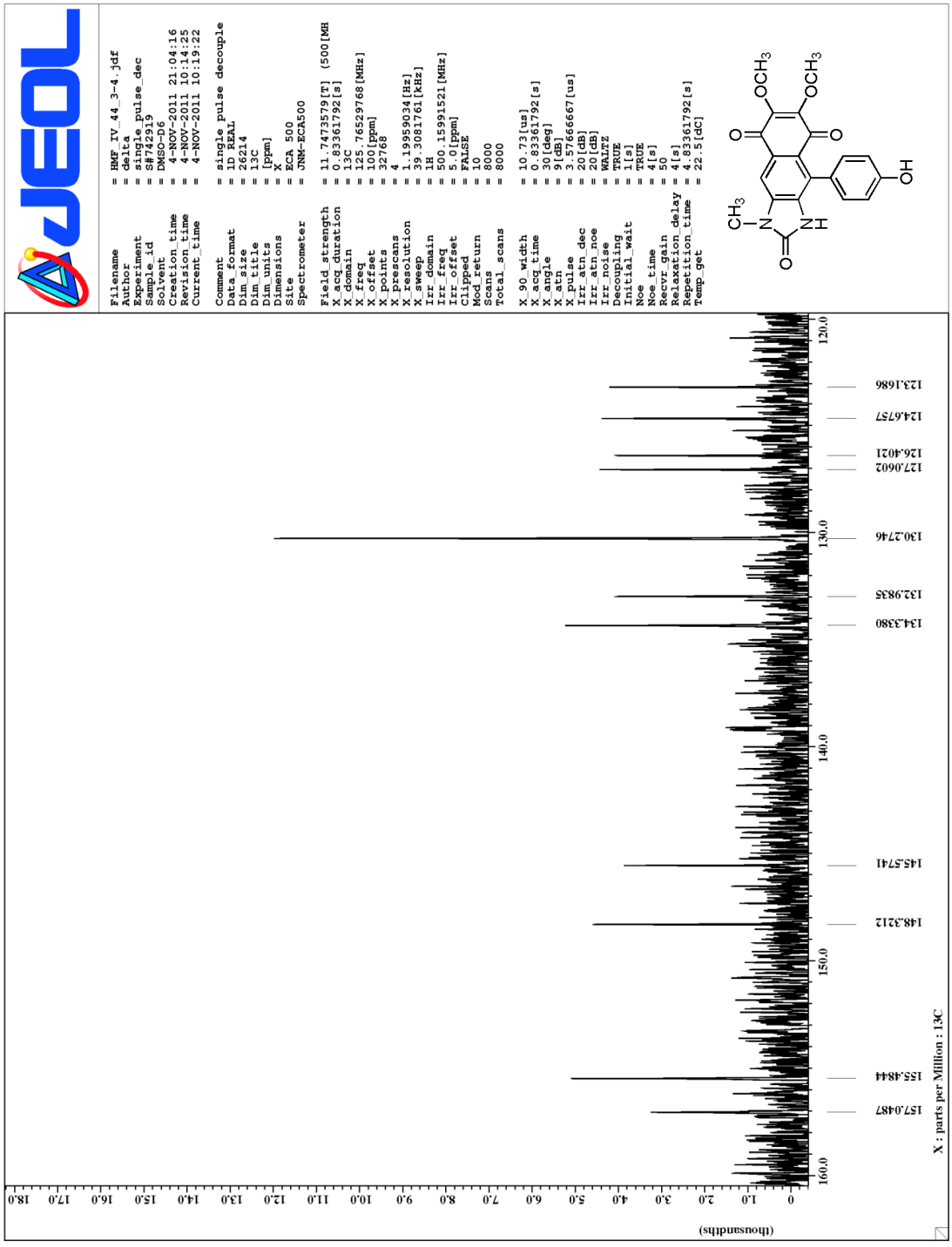
Filename = HMF_IV_44_3-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#742919
Solvent = DMSO-D6
Creation_time = 4-NOV-2011 21:04:16
Revision_time = 4-NOV-2011 10:14:25
Current_time = 4-NOV-2011 10:16:47

Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500

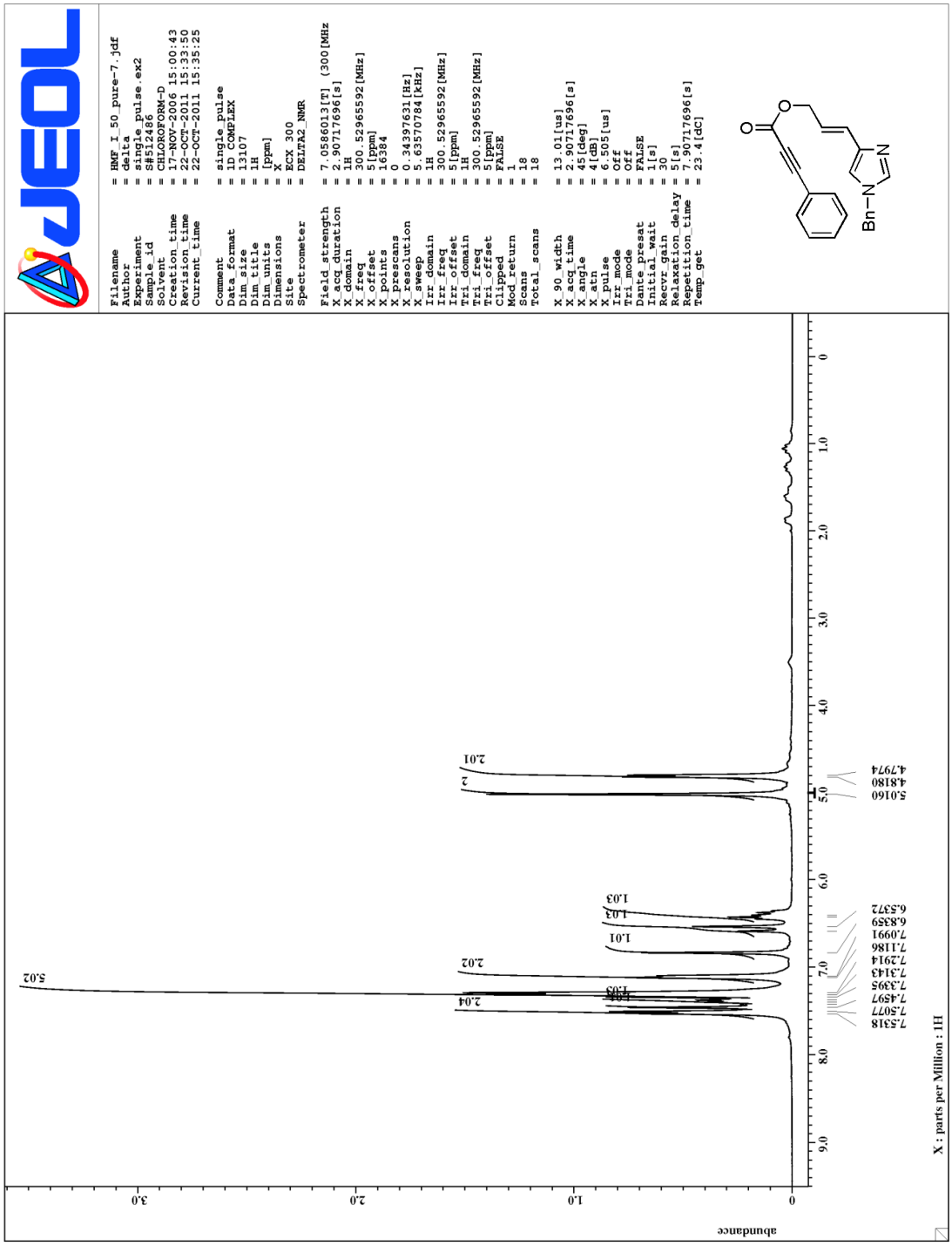
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 8000
Total_scans = 8000

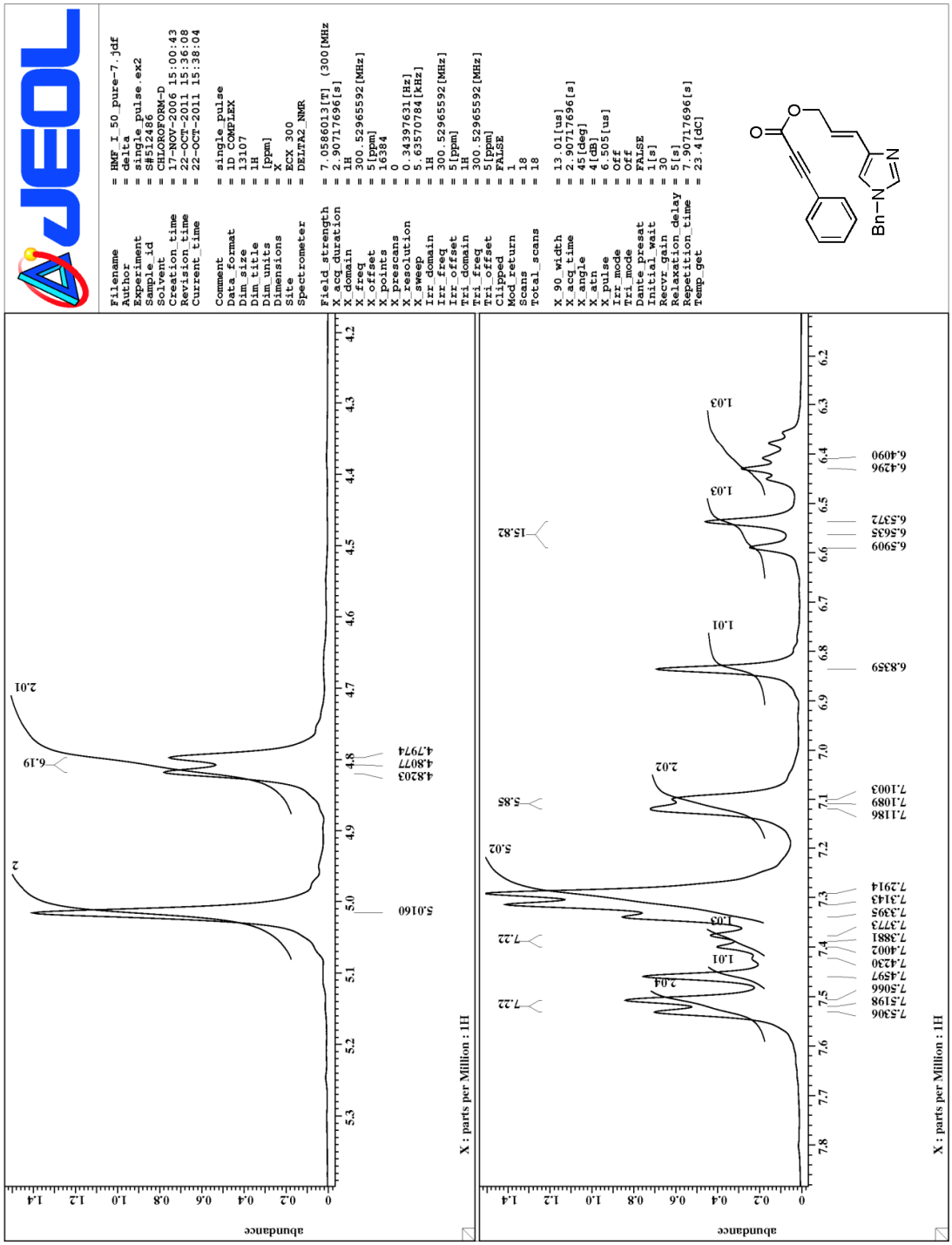
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.5[dC]

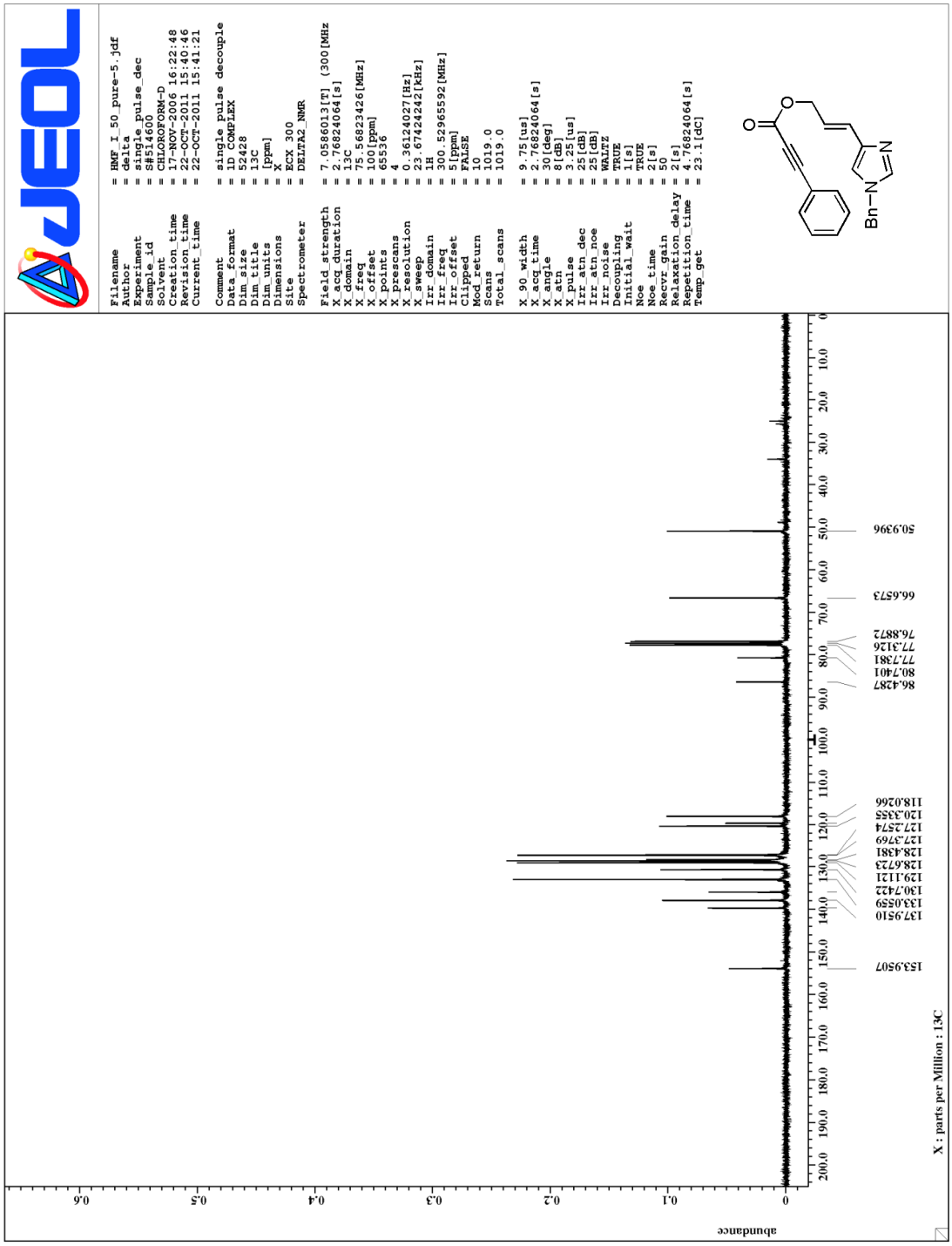


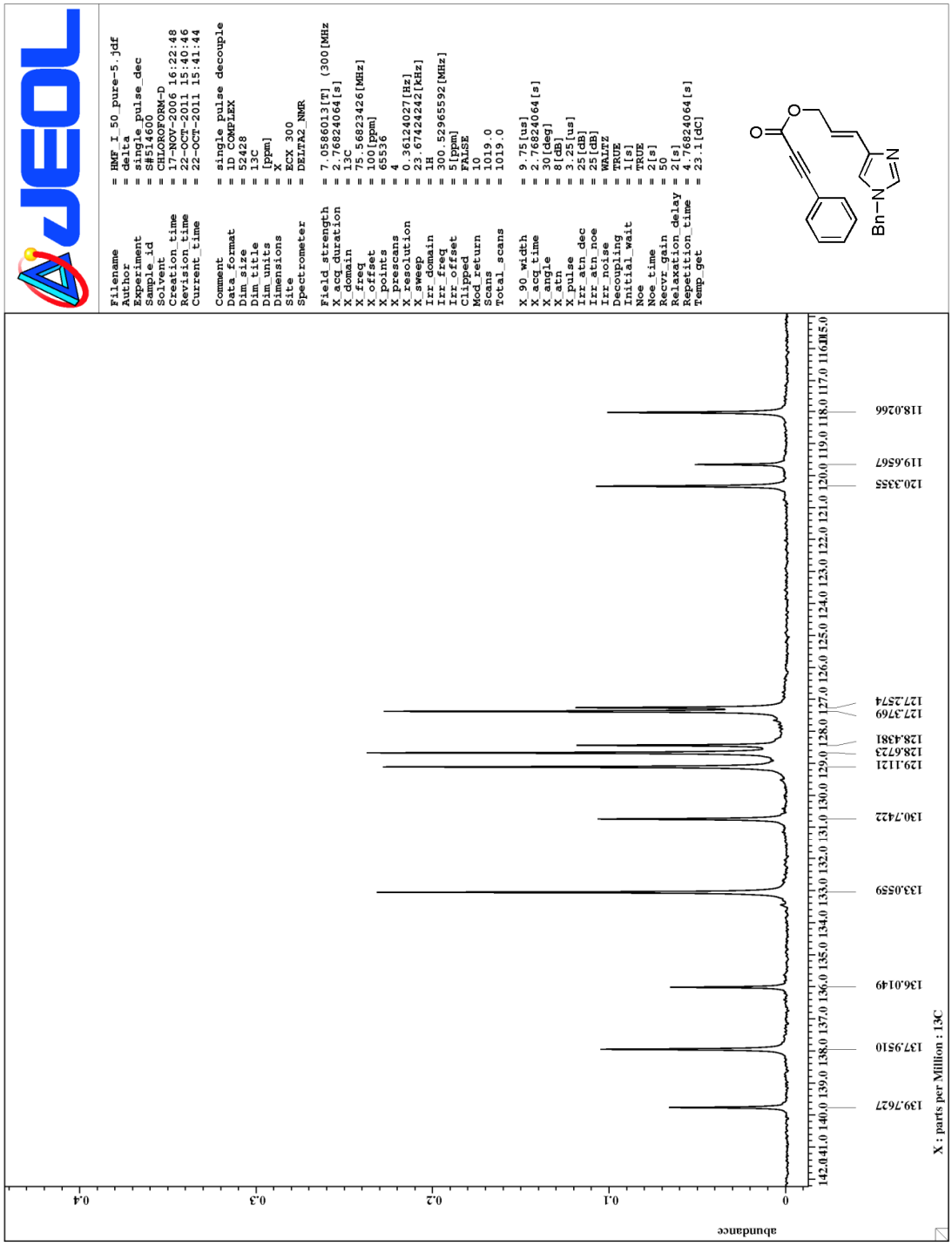

JEOL
Filename = HMF_IV_44_3-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#742919
Solvent = DMSO-D6
Creation_time = 4-NOV-2011 21:04:16
Revision_time = 4-NOV-2011 10:14:25
Current_time = 4-NOV-2011 10:19:22
Comment = single_pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159915211MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 8000
Total_scans = 8000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.5[dC]
OCH3
OCH3
CH3
N
H
N
O
O
O
OH
(thousandths)
X : parts per Million : 13C
157.0487
155.4844
148.3212
145.5741
134.3380
132.9835
130.2746
127.0602
126.4021
124.6757
123.1686

APPENDIX 46
^{1}H and ^{13}C NMR Spectra of
(2*E*)-3-(1-Benzyl-1*H*-imidazol-4-yl)prop-2-enyl 3-phenylpropynoate (**68c**)

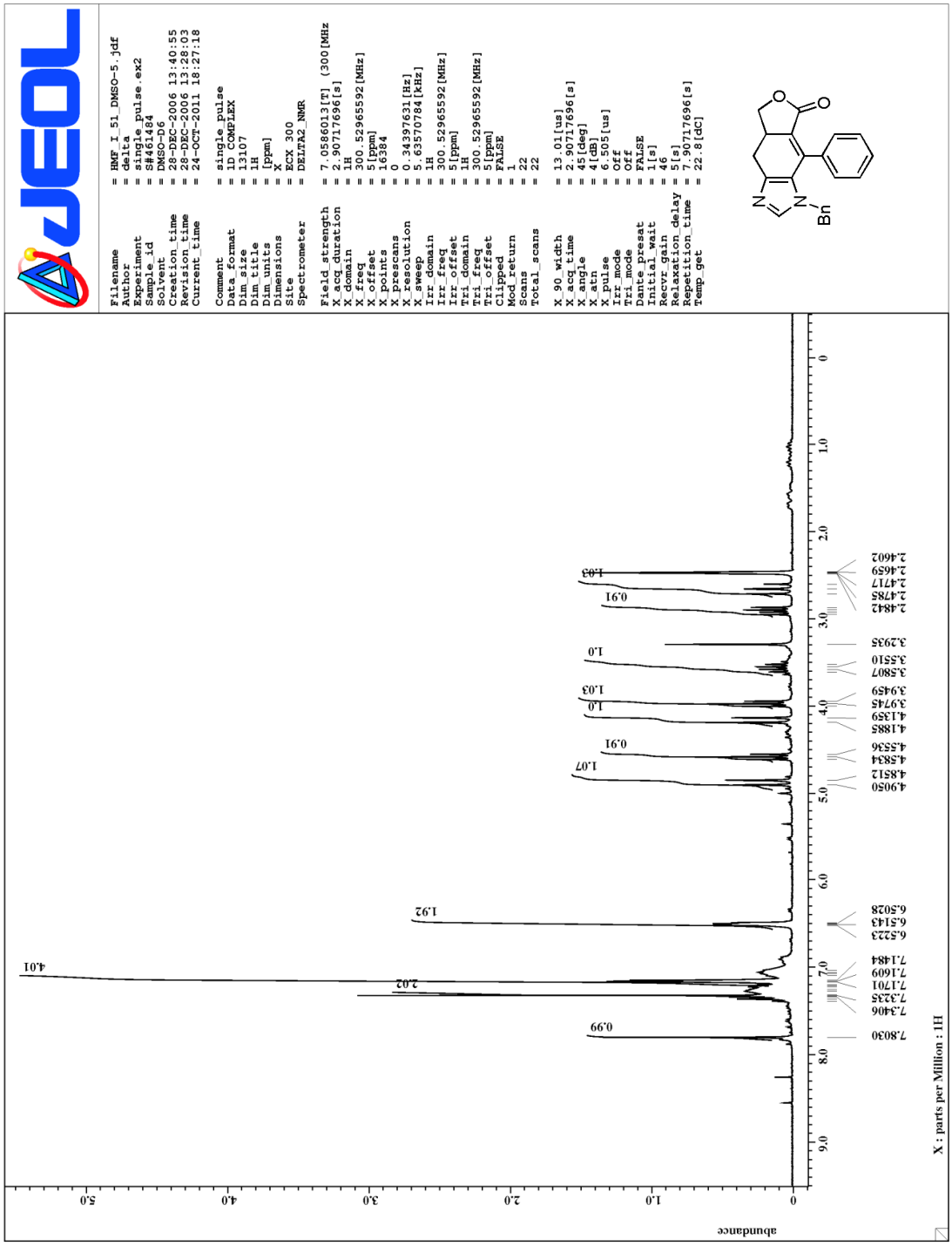

JEOL
Filename = HMF_I_50_pure-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#512486
Solvent = CHLOROFORM-D
Creation_time = 17-NOV-2006 15:00:43
Revision_time = 22-OCT-2011 15:33:50
Current_time = 22-OCT-2011 15:35:25
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEIMPA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 18
Total_scans = 18
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 30
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.4[dC]
Bn—N
N
O
O
abundance
X : parts per Million : 1H
5.02
2.04
2.02
2.01
1.03
1.02
1.01
2
5.0160
4.8180
4.7974
7.5318
7.5077
7.4897
7.3395
7.3143
7.2914
7.1186
7.0991
6.8359
6.5372

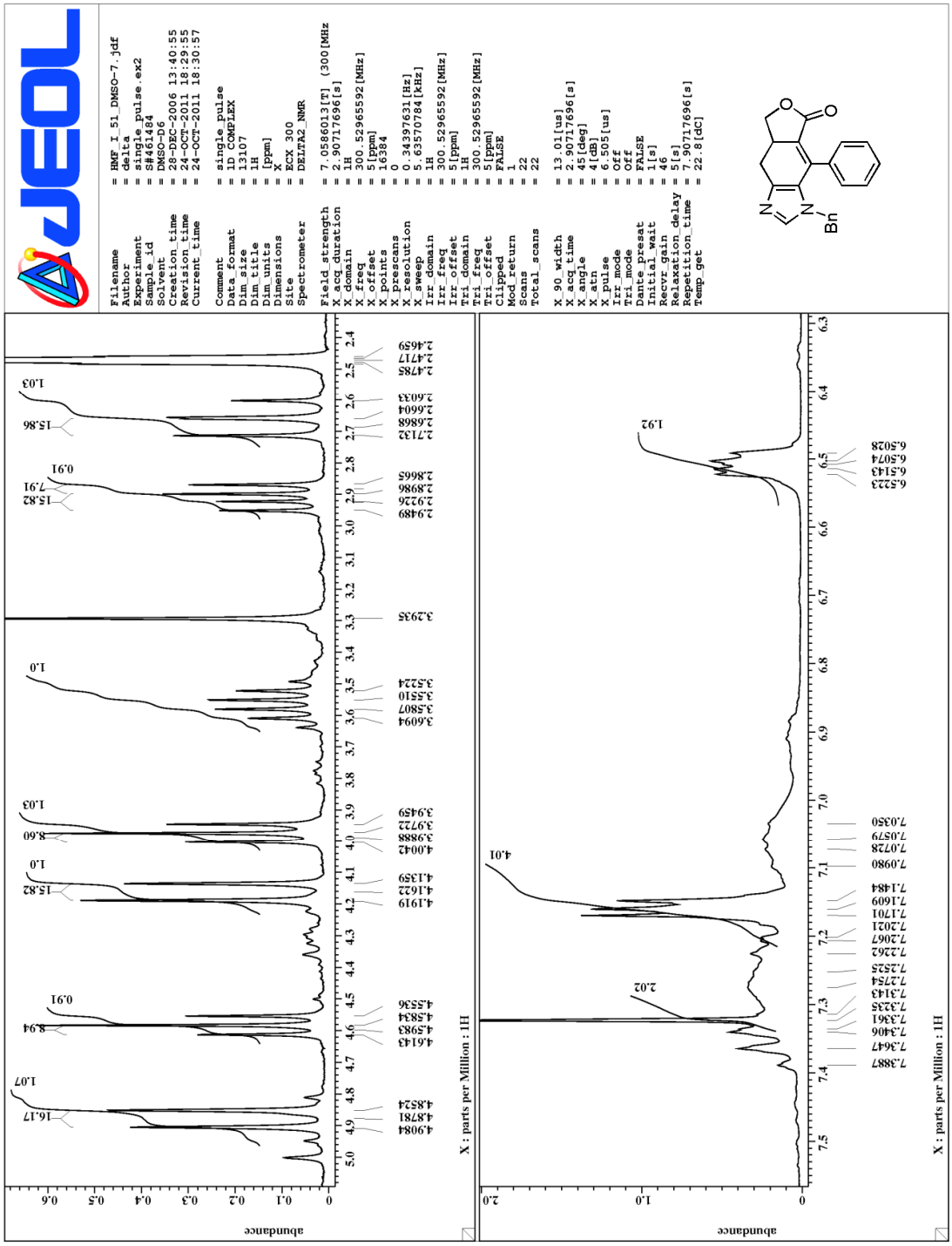

JEOL
Filename = HMF_I_50_pure-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#512486
Solvent = CHLOROFORM-D
Creation_time = 17-NOV-2006 15:00:43
Revision_time = 22-OCT-2011 15:36:08
Current_time = 22-OCT-2011 15:38:04
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 18
Total_scans = 18
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 30
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.4[dC]
Bn—N
abundance
X : parts per Million : 1H
2.01
6.19
2
5.0160
4.8203
4.8077
4.7974
15.82
5.85
5.02
7.22
2.04
1.01
1.03
2.02
6.8359
6.5909
6.5635
6.5372
6.4296
6.4090
7.5306
7.5198
7.5066
7.4597
7.4230
7.4002
7.3881
7.3773
7.3395
7.3143
7.2914
7.1186
7.1089
7.1003

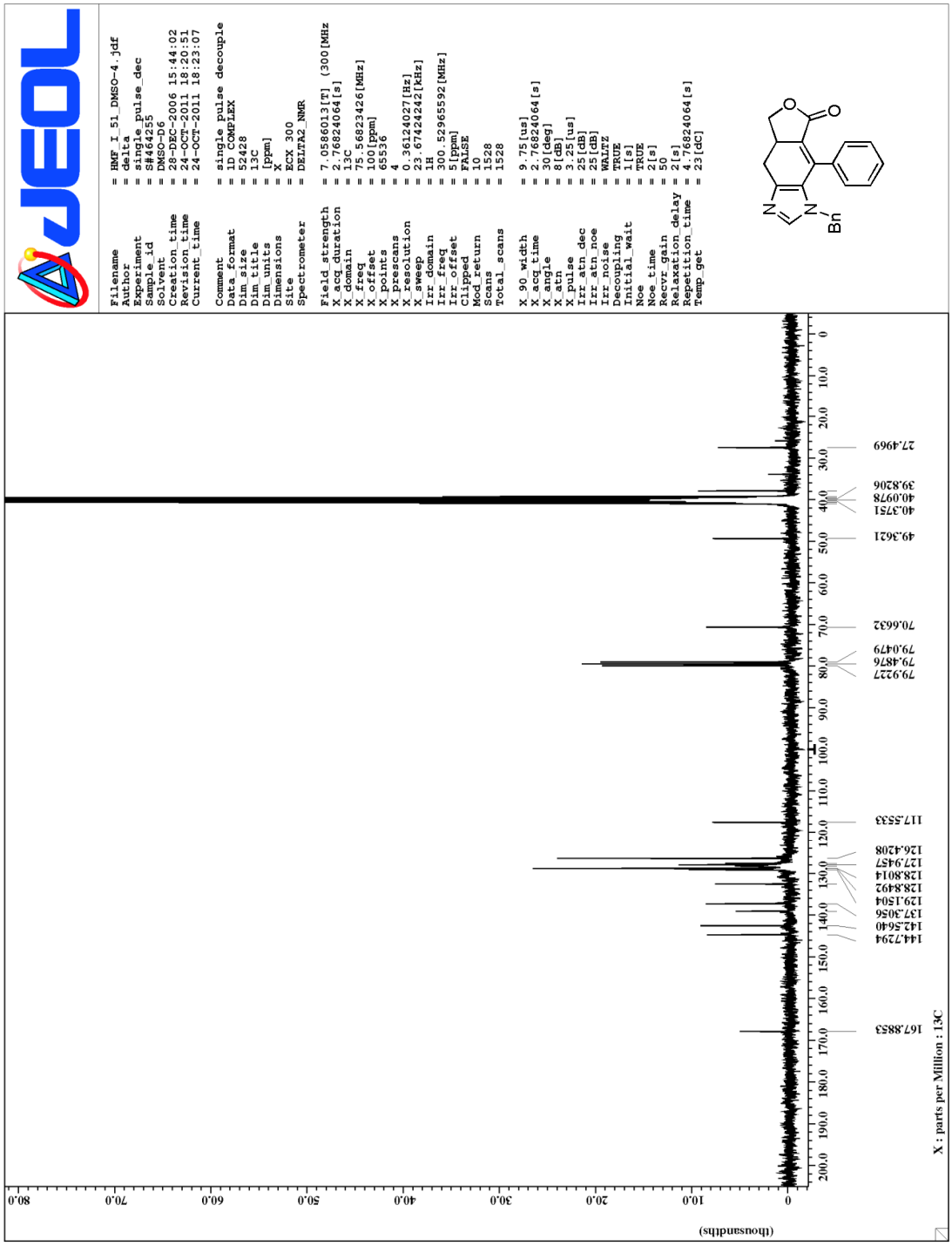

JEOL
Filename = HMF_I_50_pure-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#514600
Solvent = CHLOROFORM-D
Creation_time = 17-NOV-2006 16:22:48
Revision_time = 22-OCT-2011 15:40:46
Current_time = 22-OCT-2011 15:41:21
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1019.0
Total_scans = 1019.0
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 23.1[dC]
Bn−N
N
O
O
abundance
X : parts per Million : 13C
153.9507
137.9510
133.0559
130.7422
129.1121
128.6723
128.4381
127.3769
127.2574
120.3355
118.0266
86.4287
80.7401
77.7381
77.3126
76.8872
66.6573
50.9396

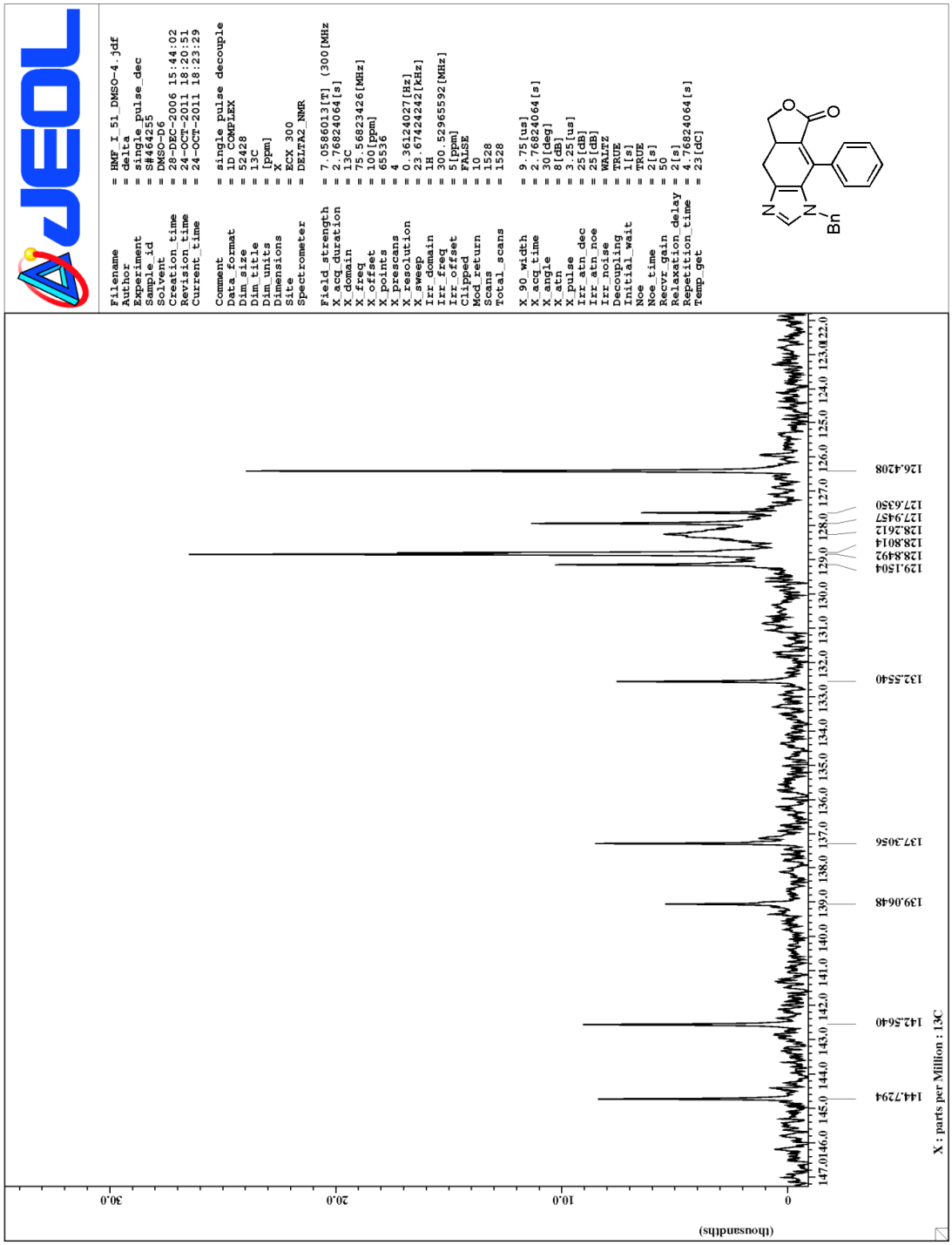

Filename = HMF_I_50_pure-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#514600
Solvent = CHLOROFORM-D
Creation_time = 17-NOV-2006 16:22:48
Revision_time = 22-OCT-2011 15:40:46
Current_time = 22-OCT-2011 15:41:44
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEUTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1019.0
Total_scans = 1019.0
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 23.1[dC]
Bn—N
abundance
X : parts per Million : 13C
139.7627
137.9510
136.0149
133.0559
130.7422
129.1121
128.6723
128.4381
127.3769
127.2574
120.3355
119.6567
118.0266

APPENDIX 47
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-3,7,7a,8-tetrahydro-4-phenyl-5*H*-furo[3,4-*f*]benzimidazol-5-one (**69c**)

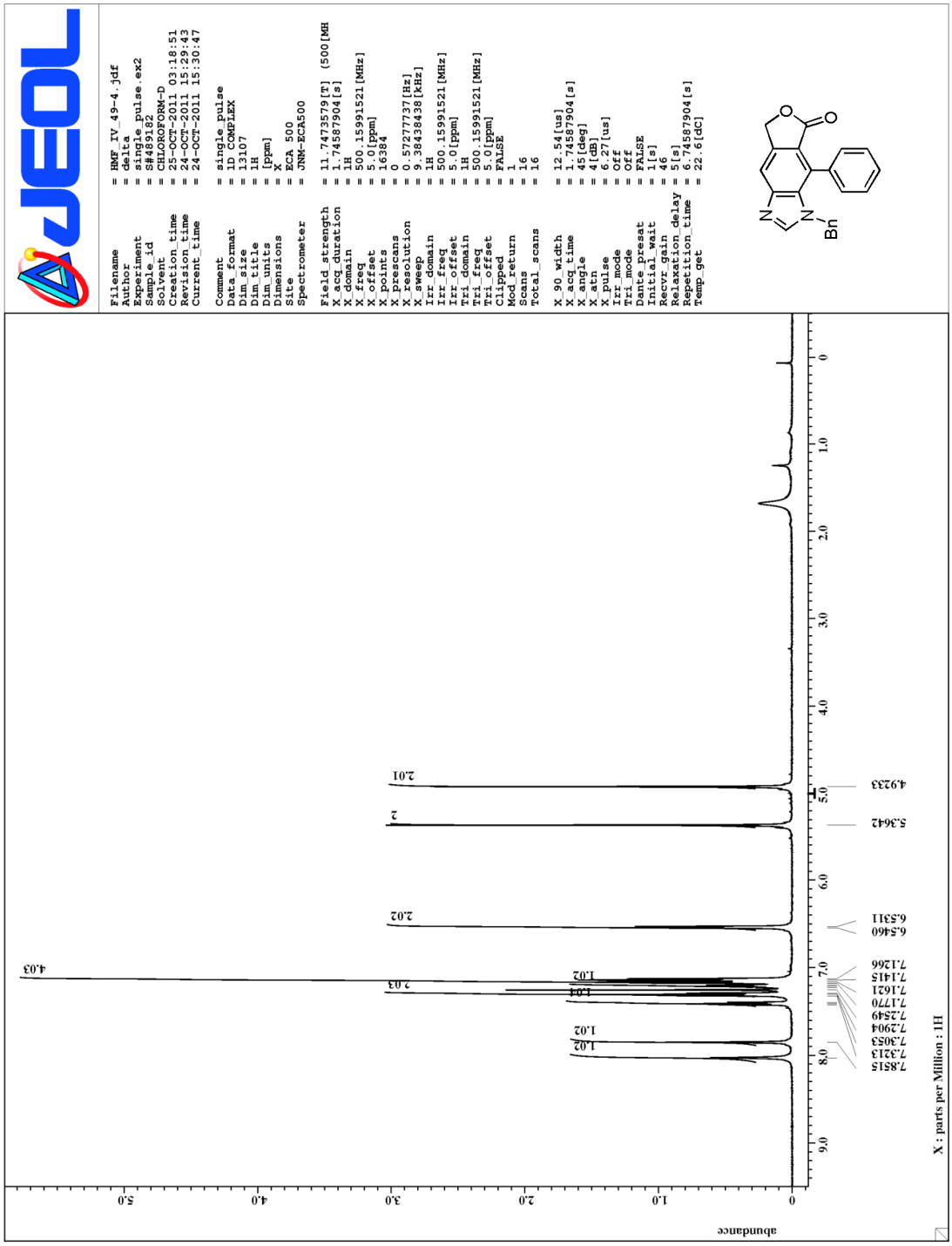

Filename = HMF_I_51_DMSO-5.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#461484
Solvent = DMSO-D6
Creation_time = 28-DEC-2006 13:40:55
Revision_time = 28-DEC-2006 13:28:03
Current_time = 24-OCT-2011 18:27:18
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 22
Total_scans = 22
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 22.8[dC]
Bn
abundance
X : parts per Million : 1H
4.01
2.02
1.92
0.99
1.07
0.91
1.03
1.0
1.03
1.0
0.91
1.03
7.8030
7.3406
7.3235
7.1701
7.1609
7.1484
6.5223
6.5143
6.5028
4.9050
4.8512
4.5834
4.5536
4.1885
4.1359
3.9745
3.9459
3.5807
3.5510
3.2935
2.4842
2.4785
2.4717
2.4659
2.4602

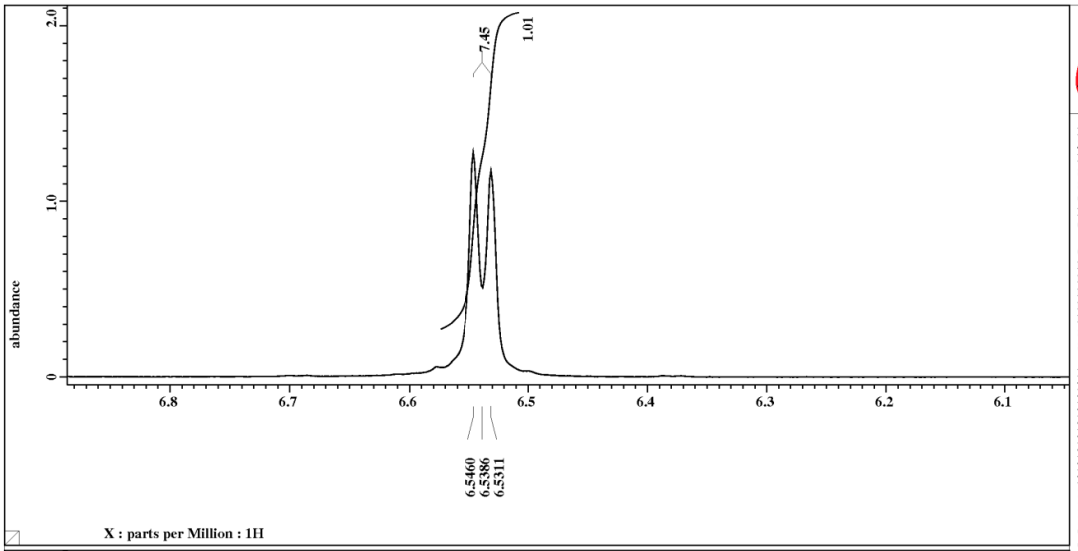

JEOL
Filename = HMF_I_51_DMSO-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#461484
Solvent = DMSO-D6
Creation_time = 28-DEC-2006 13:40:55
Revision_time = 24-OCT-2011 18:29:55
Current_time = 24-OCT-2011 18:30:57
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DEUTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 22
Total_scans = 22
X_90_width = 13.01[us]
X_acq_time = 2.90717696[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 22.8[dC]
Bn
abundance
X : parts per Million : 1H
16.17
1.07
8.94
0.91
15.82
1.0
8.60
1.03
3.29
1.0
15.82
7.91
0.91
15.86
1.03
4.01
2.02
1.92

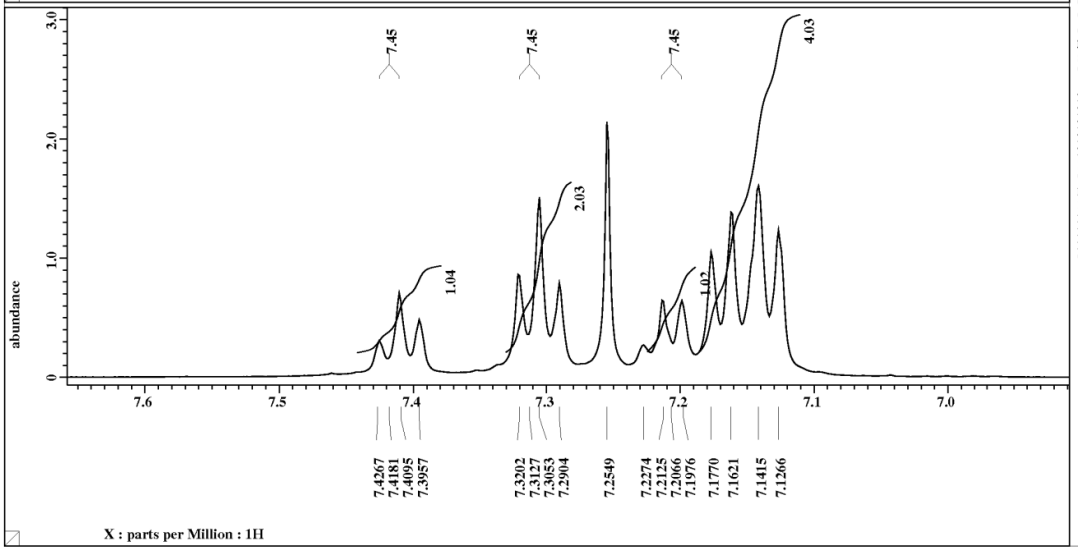

JEOL
Filename = HMF_I_51_DMSO-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#464255
Solvent = DMSO-D6
Creation_time = 28-DEC-2006 15:44:02
Revision_time = 24-OCT-2011 18:20:51
Current_time = 24-OCT-2011 18:23:07
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1528
Total_scans = 1528
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 23[dC]
N
N
Bn
(thousandths)
X : parts per Million : 13C
167.8853
144.7294
142.5640
137.3056
129.1504
128.8492
128.8014
127.9457
126.4208
117.5533
79.9227
79.4876
79.0479
70.6632
49.3621
40.3751
40.0978
39.8206
27.4969


Filename = HMF_I_51_DMSO-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#464255
Solvent = DMSO-D6
Creation_time = 28-DEC-2006 15:44:02
Revision_time = 24-OCT-2011 18:20:51
Current_time = 24-OCT-2011 18:23:29
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX_300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1528
Total_scans = 1528
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 23[dC]
Bn
126.4208
127.6350
127.9457
128.2612
128.8014
128.8492
129.1504
132.5540
137.3056
139.0648
142.5640
144.7294
X : parts per Million : 13C
(thousandths)

APPENDIX 48
^{1}H and ^{13}C NMR Spectra of
3-benzyl-4-phenyl-3,7-dihydro-5*H*-furo[3,4-*f*]benzimidazol-5-one


JEOL
Filename = HMF_IV_49-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#489182
Solvent = CHLOROFORM-D
Creation_time = 25-OCT-2011 03:18:51
Revision_time = 24-OCT-2011 15:29:43
Current_time = 24-OCT-2011 15:30:47
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.6[dC]
Bn
abundance
X : parts per Million : 1H
4.03
2.01
2
2.02
2.03
1.04
1.02
1.02
1.02
4.9233
5.3642
6.5460
6.5311
7.1266
7.1415
7.1621
7.1770
7.2549
7.2904
7.3053
7.3213
7.8515





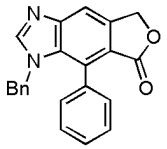
JEOL

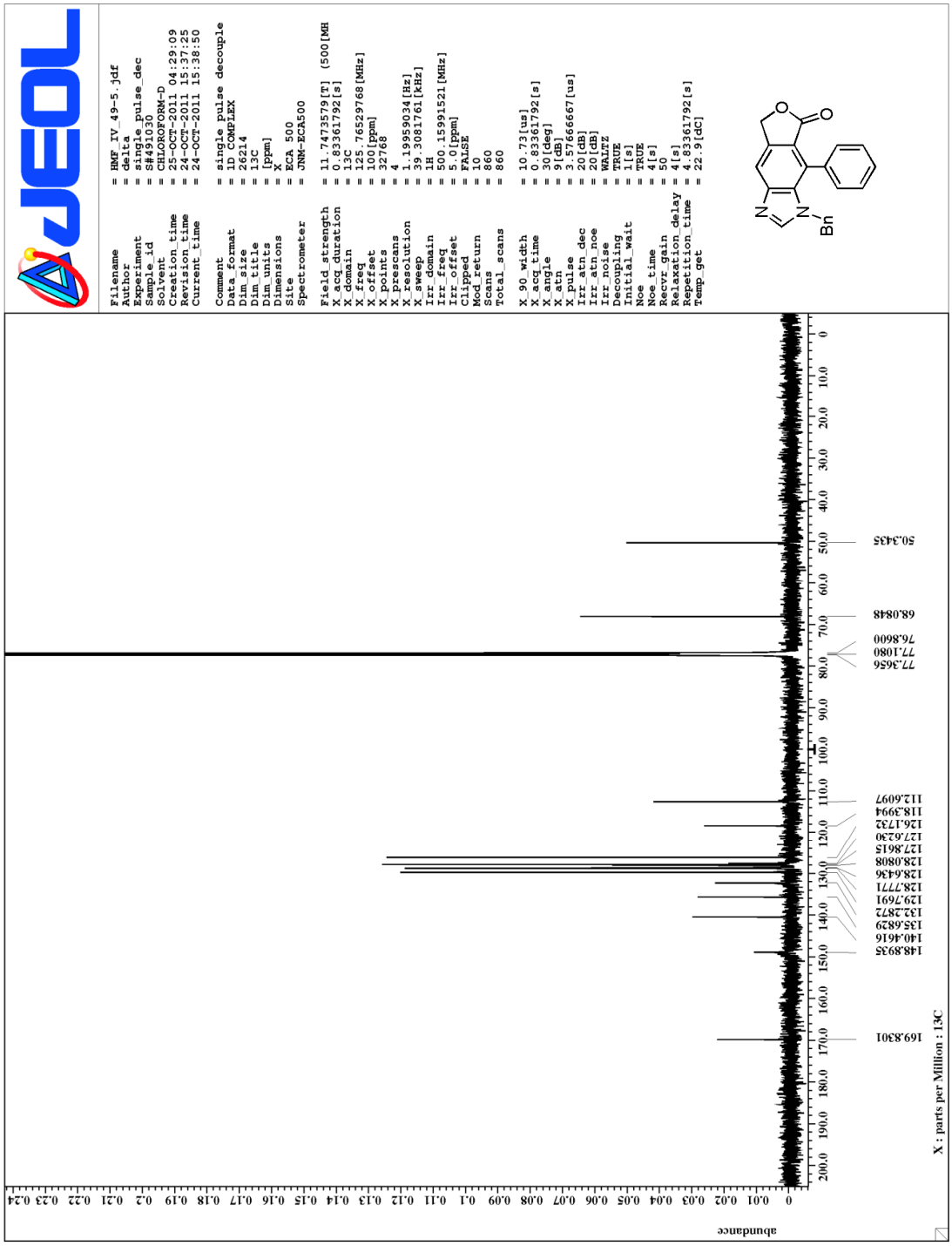
```
Filename          = HMF_IV_49-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#489182
Solvent           = CHLOROFORM-D
Creation_time     = 25-OCT-2011 03:18:51
Revision_time     = 24-OCT-2011 15:34:48
Current_time      = 24-OCT-2011 15:41:21

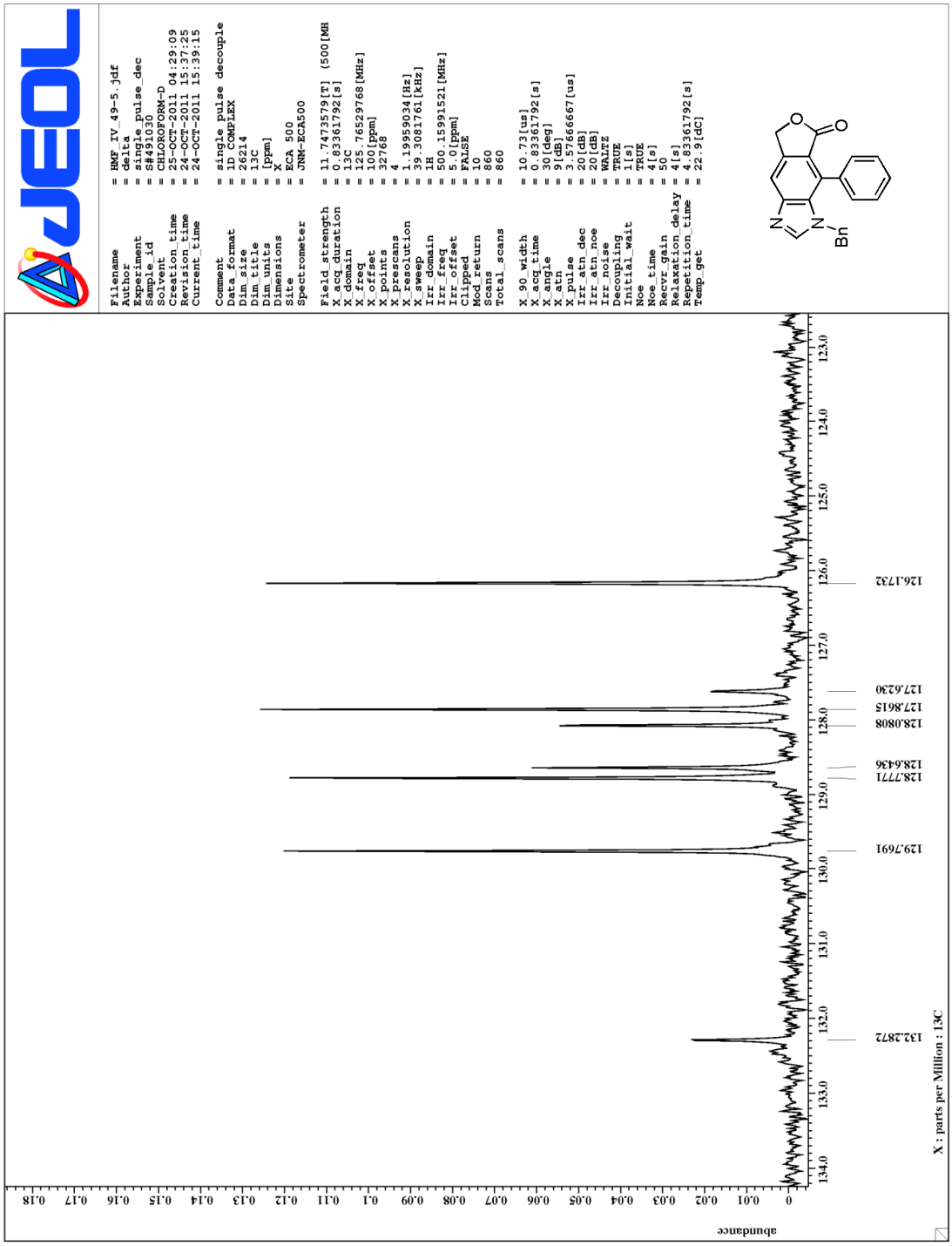
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

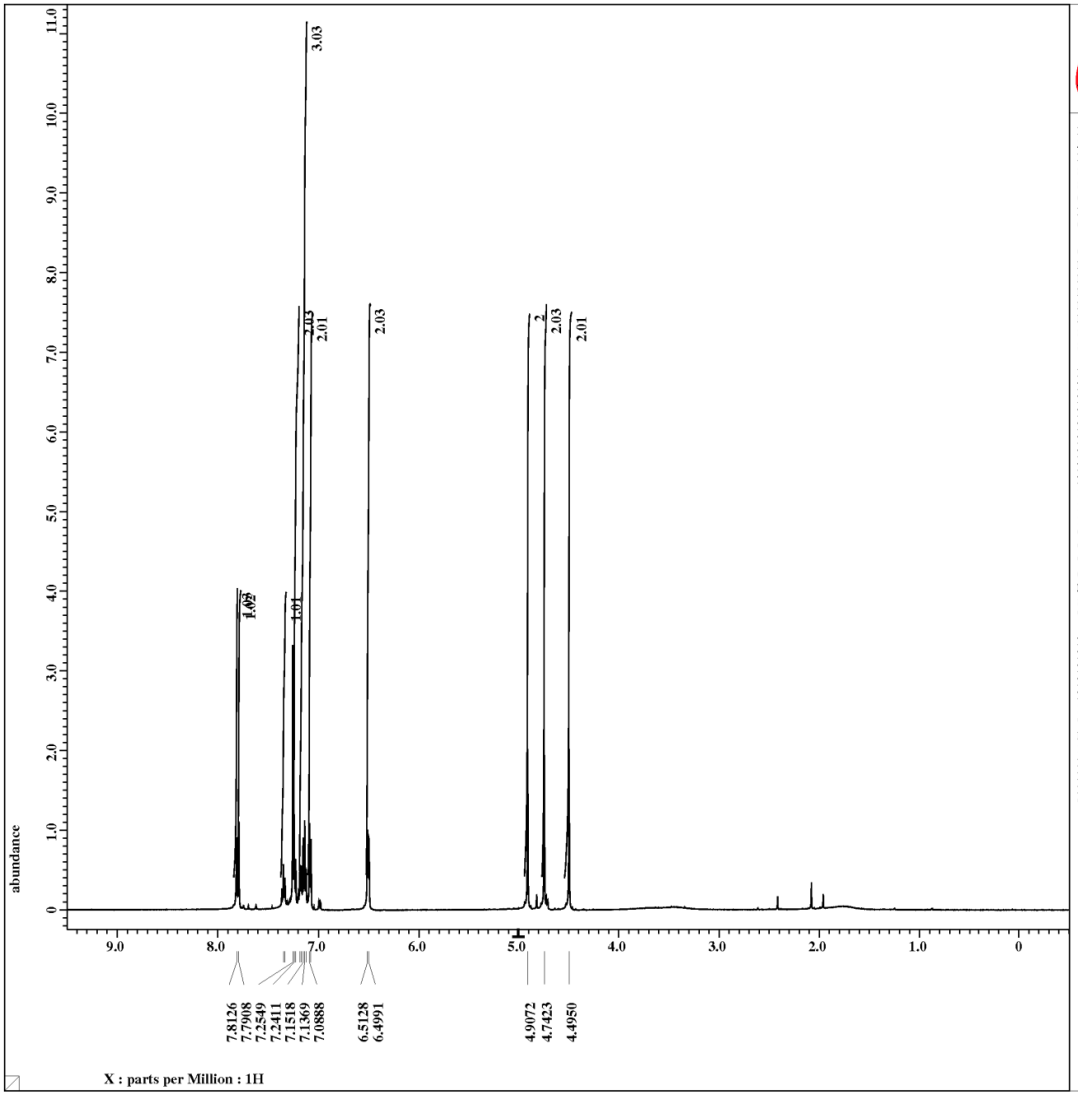
X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.6[dC]
```




JEOL
Filename = BMF_IV_49-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#491030
Solvent = CHLOROFORM-D
Creation_time = 25-OCT-2011 04:29:09
Revision_time = 24-OCT-2011 15:37:25
Current_time = 24-OCT-2011 15:38:50
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 860
Total_scans = 860
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.9[dC]
N
N
Bn
50.3435
68.0848
76.8600
77.1080
77.3656
112.6097
118.3994
126.1732
127.6230
127.8615
128.0808
128.6436
128.7771
129.7691
132.2872
135.6829
140.4616
148.8935
169.8301
abundance
X : parts per Million : 13C


Filename = HMF_IV_49-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#491030
Solvent = CHLOROFORM-D
Creation_time = 25-OCT-2011 04:29:09
Revision_time = 24-OCT-2011 15:37:25
Current_time = 24-OCT-2011 15:39:15
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 860
Total_scans = 860
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.9[dC]
O
O
N
N
Bn
126.1732
127.6230
127.8615
128.0808
128.6436
128.7771
129.7691
132.2872
abundance
X : parts per Million : 13C

APPENDIX 49
^{1}H and ^{13}C NMR Spectra of
[3-Benzyl-4-phenyl-1*H*-benzimidazole-5,6-diyl]dimethanol (**134**)

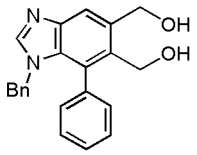


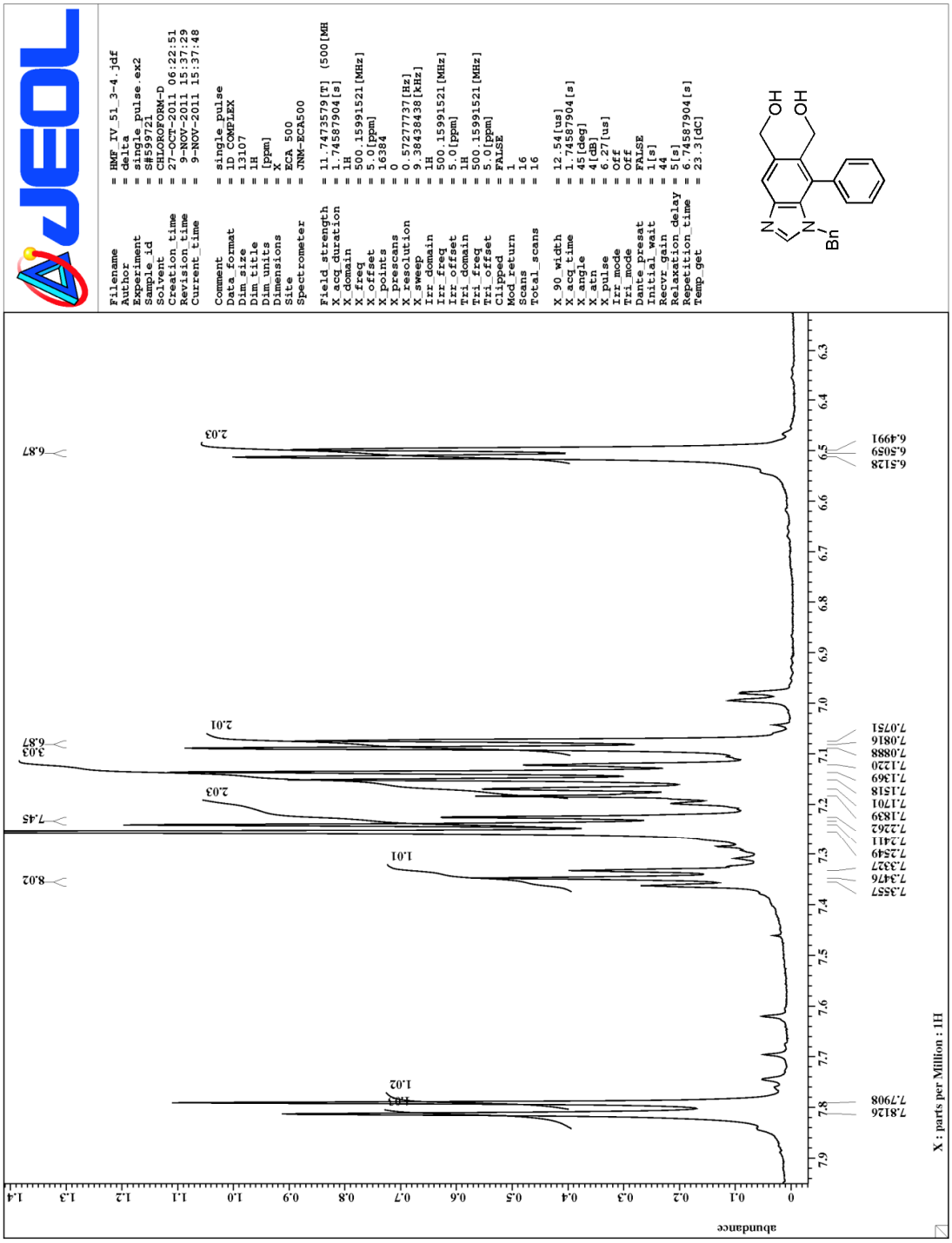

```
Filename          = HMF_IV_51_3-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#599721
Solvent           = CHLOROFORM-D
Creation_time     = 27-OCT-2011 06:22:51
Revision_time     =  9-NOV-2011 15:42:53
Current_time      =  9-NOV-2011 15:43:18

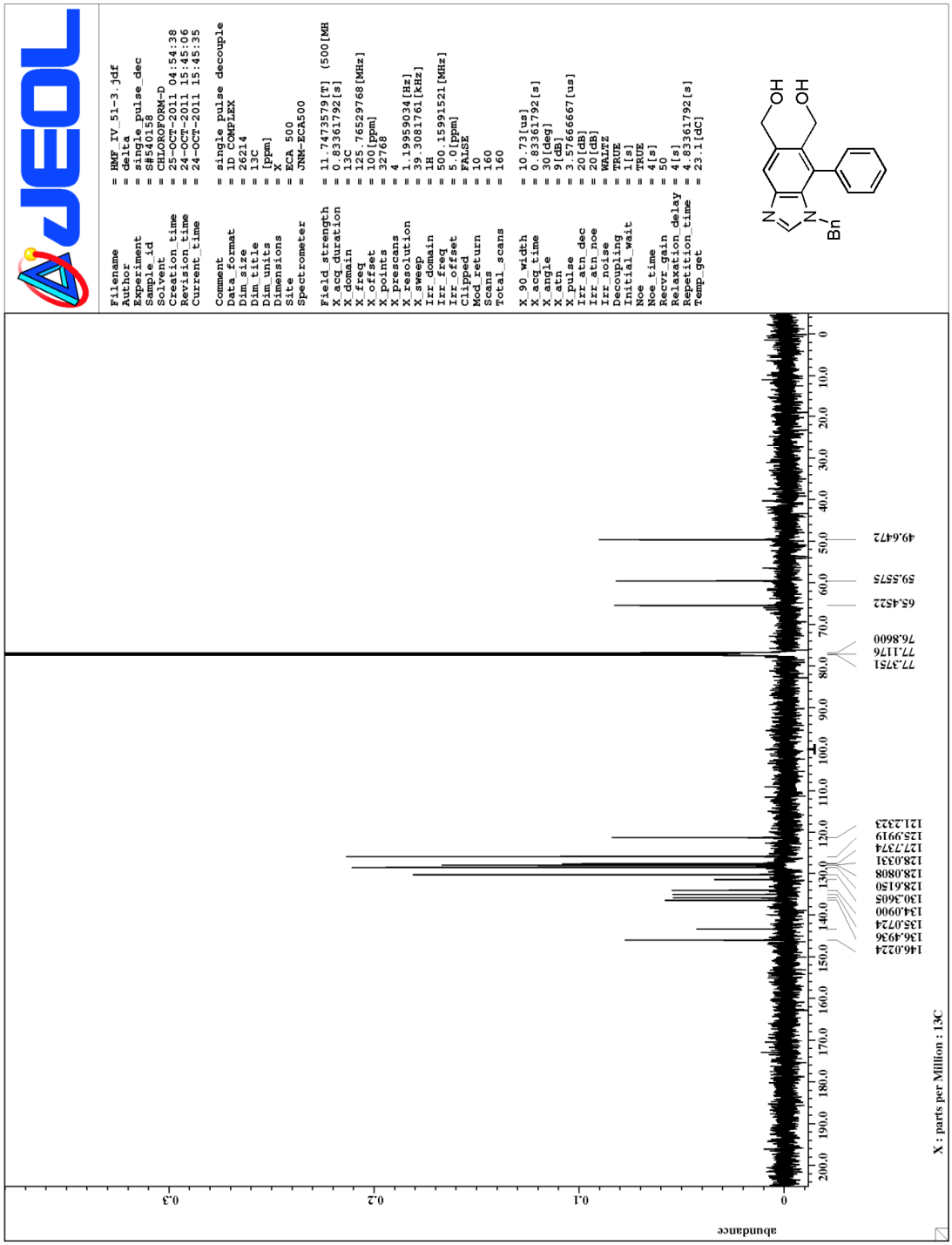
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

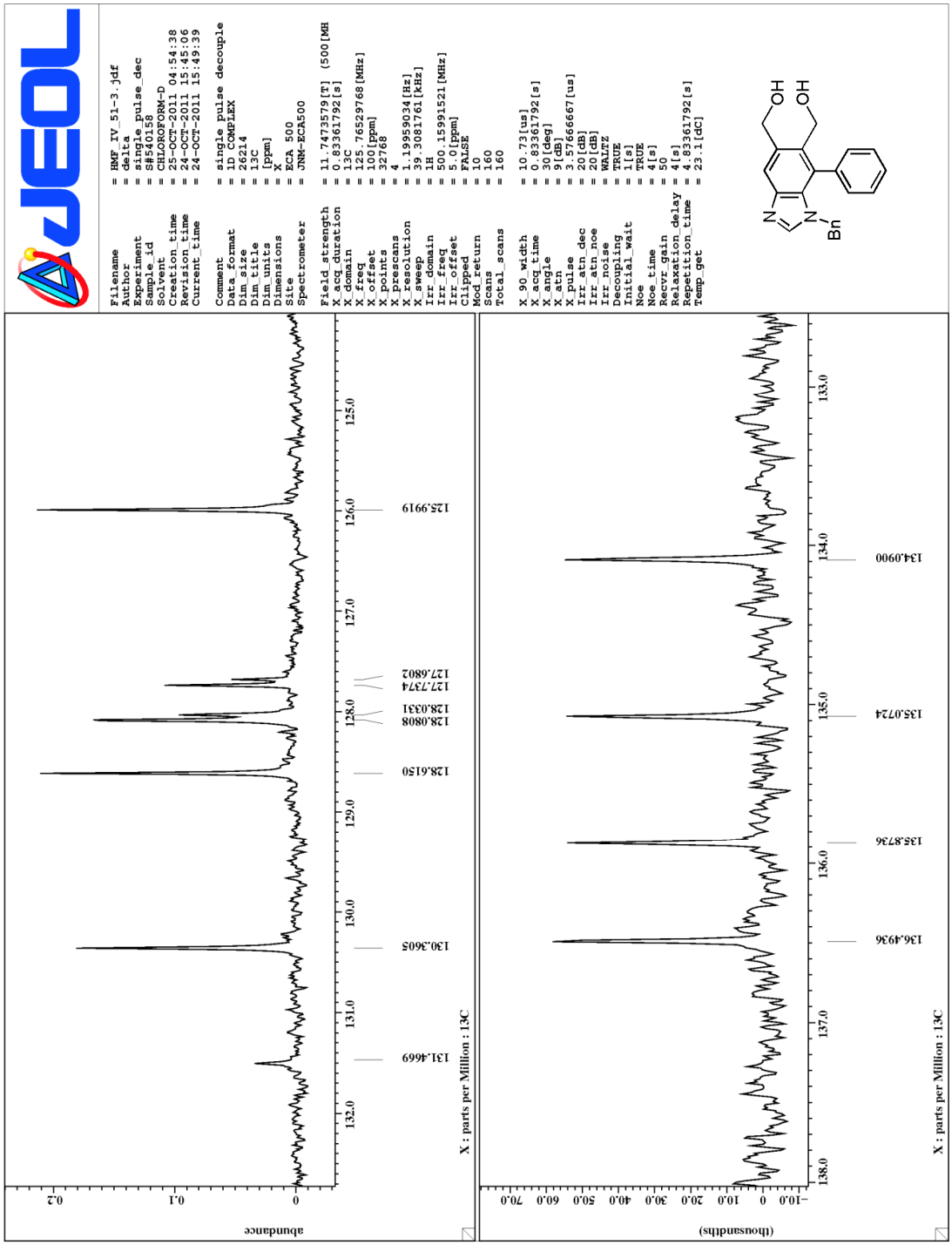
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 23.3[dC]
```

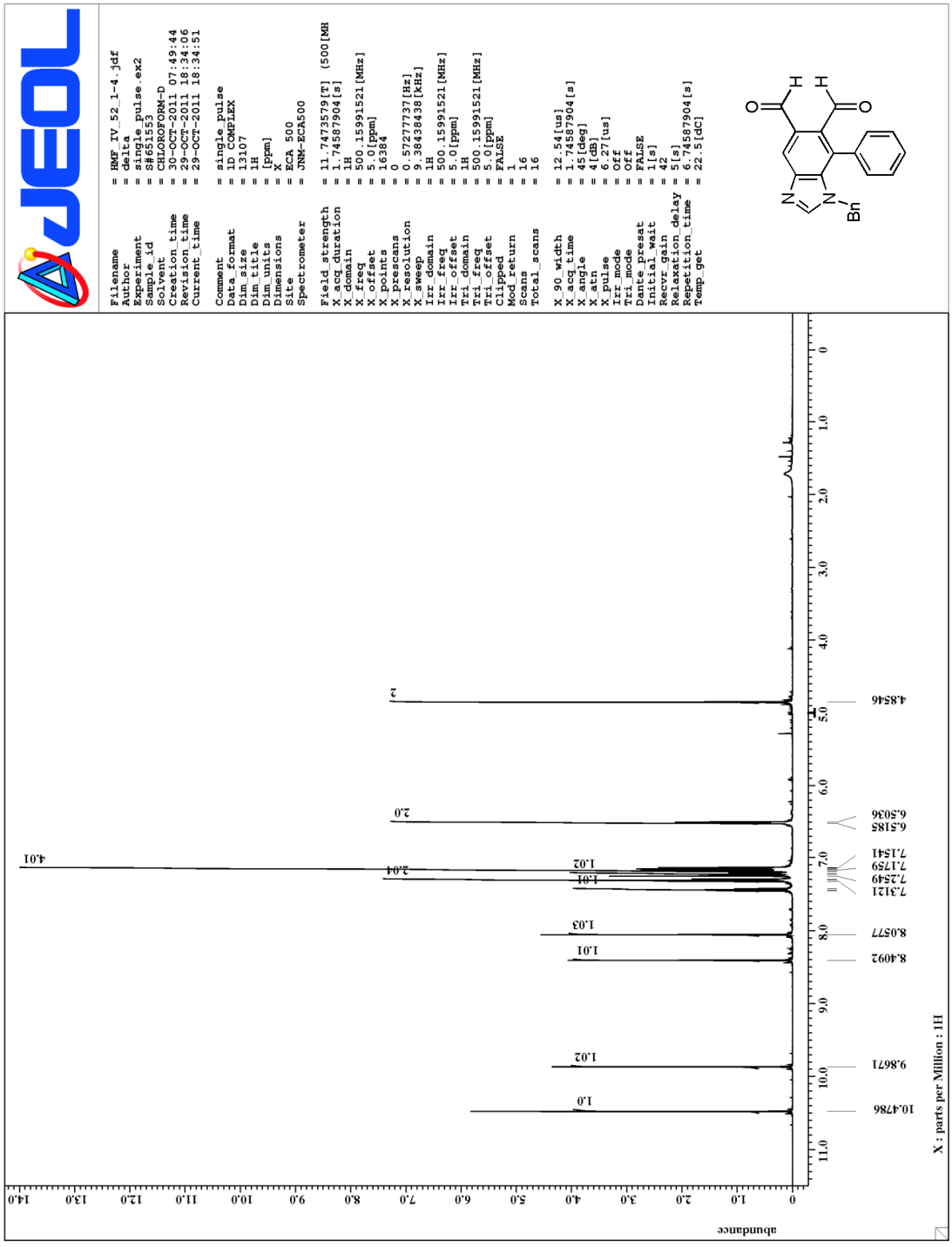


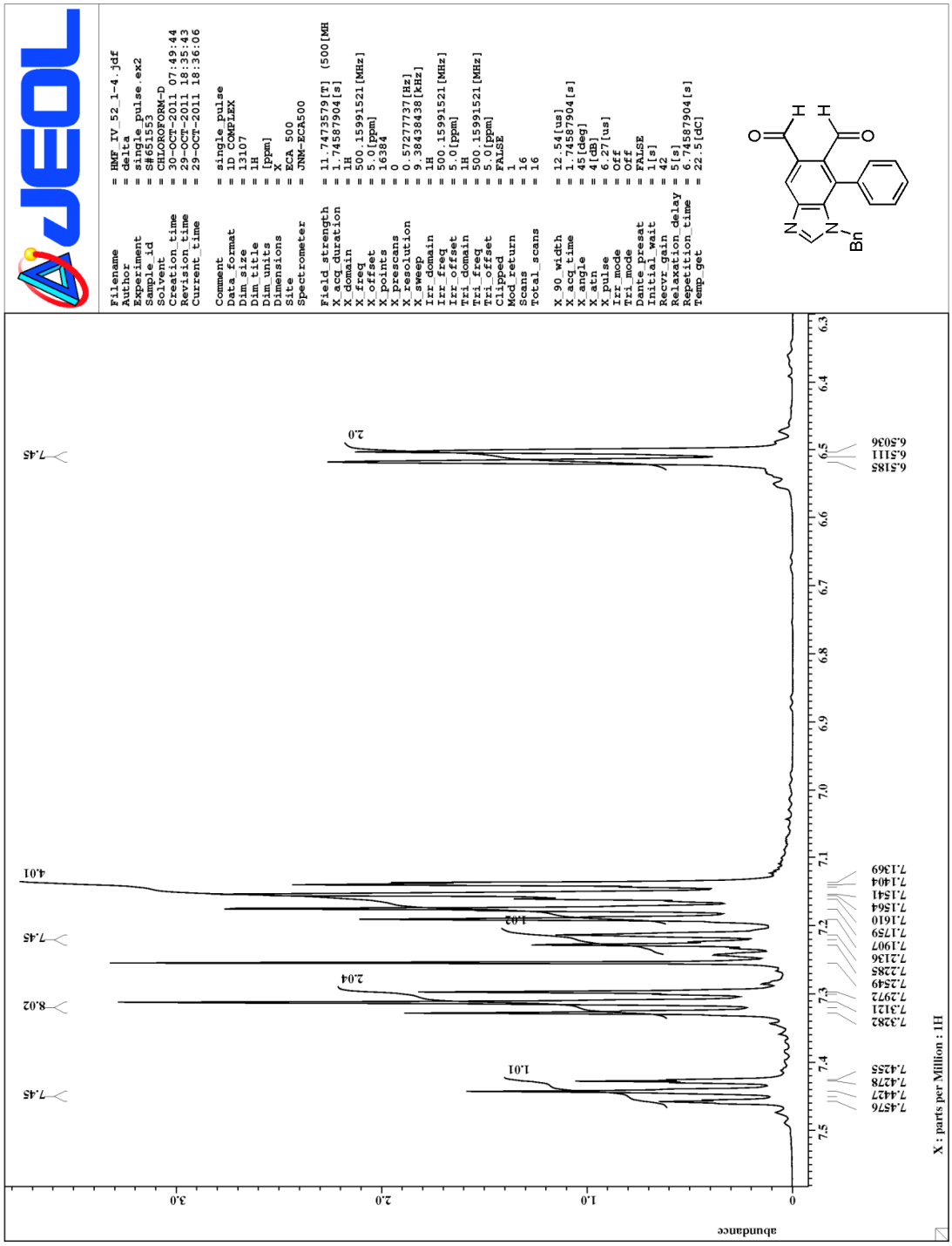

JEOL
Filename = RMF_IV_51_3-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#599721
Solvent = CHLOROFORM-D
Creation_time = 27-OCT-2011 06:22:51
Revision_time = 9-NOV-2011 15:37:29
Current_time = 9-NOV-2011 15:37:48
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.3[dC]
OH
OH
N
N
Bn
abundance
X : parts per Million : 1H
6.4991
6.5059
6.5128
7.0751
7.0816
7.0888
7.1220
7.1369
7.1518
7.1701
7.1839
7.2262
7.2411
7.2549
7.3327
7.3476
7.7908
7.8126
6.87
2.03
2.01
3.03
6.87
2.03
7.45
1.01
8.02
1.02
1.03

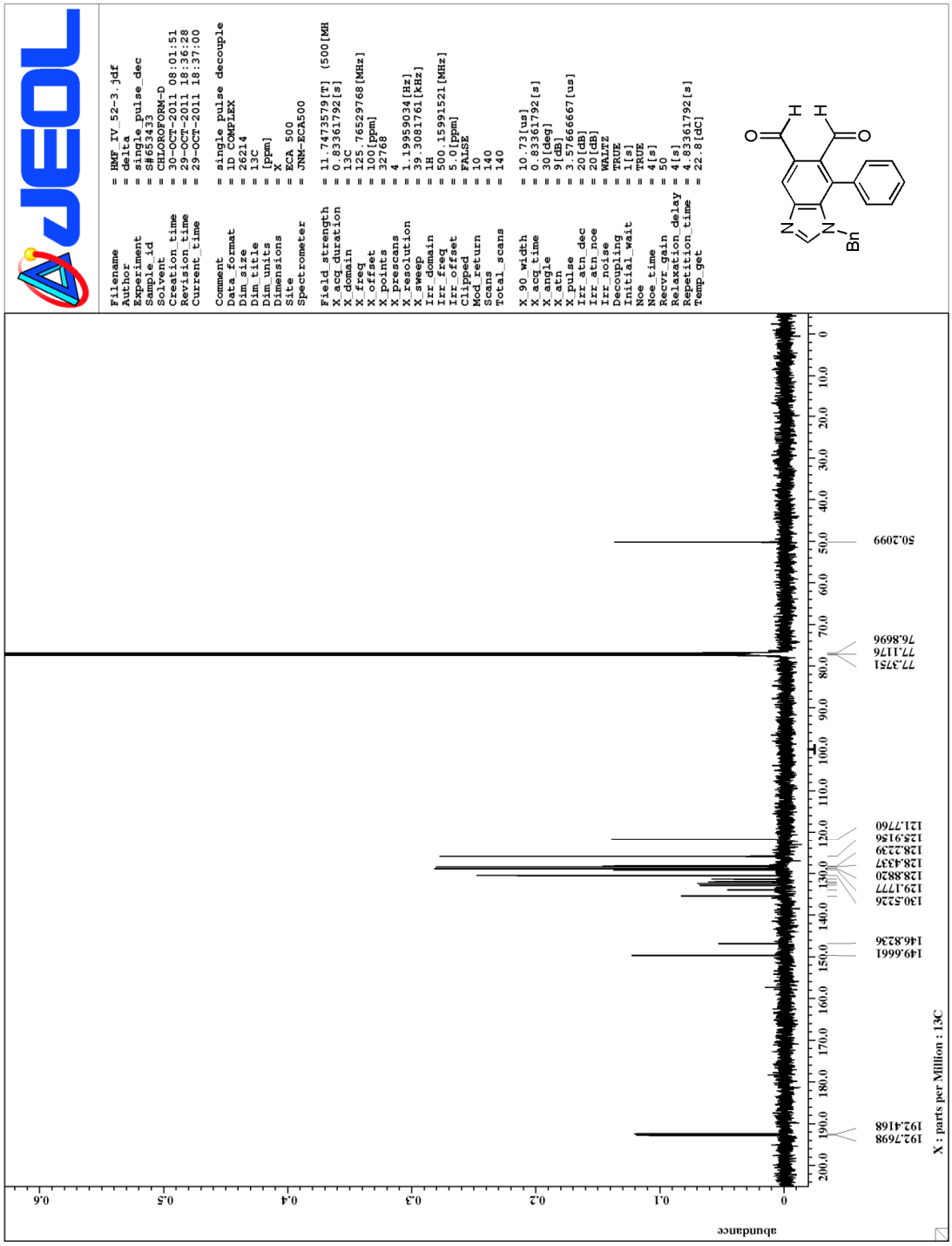

JEOL
Filename = HMF_IV_51-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#5401S8
Solvent = CHLOROFORM-D
Creation_time = 25-OCT-2011 04:54:38
Revision_time = 24-OCT-2011 15:45:06
Current_time = 24-OCT-2011 15:45:35
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 160
Total_scans = 160
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 23.1[dC]
OH
OH
N
N
Bn
abundance
0
0.1
0.2
0.3
49.6472
59.5575
65.4522
76.8600
77.1176
77.3751
121.2323
125.9919
127.7374
128.0331
128.0808
128.6150
130.3605
134.0900
135.0724
136.4936
146.0224
X : parts per Million : 13C

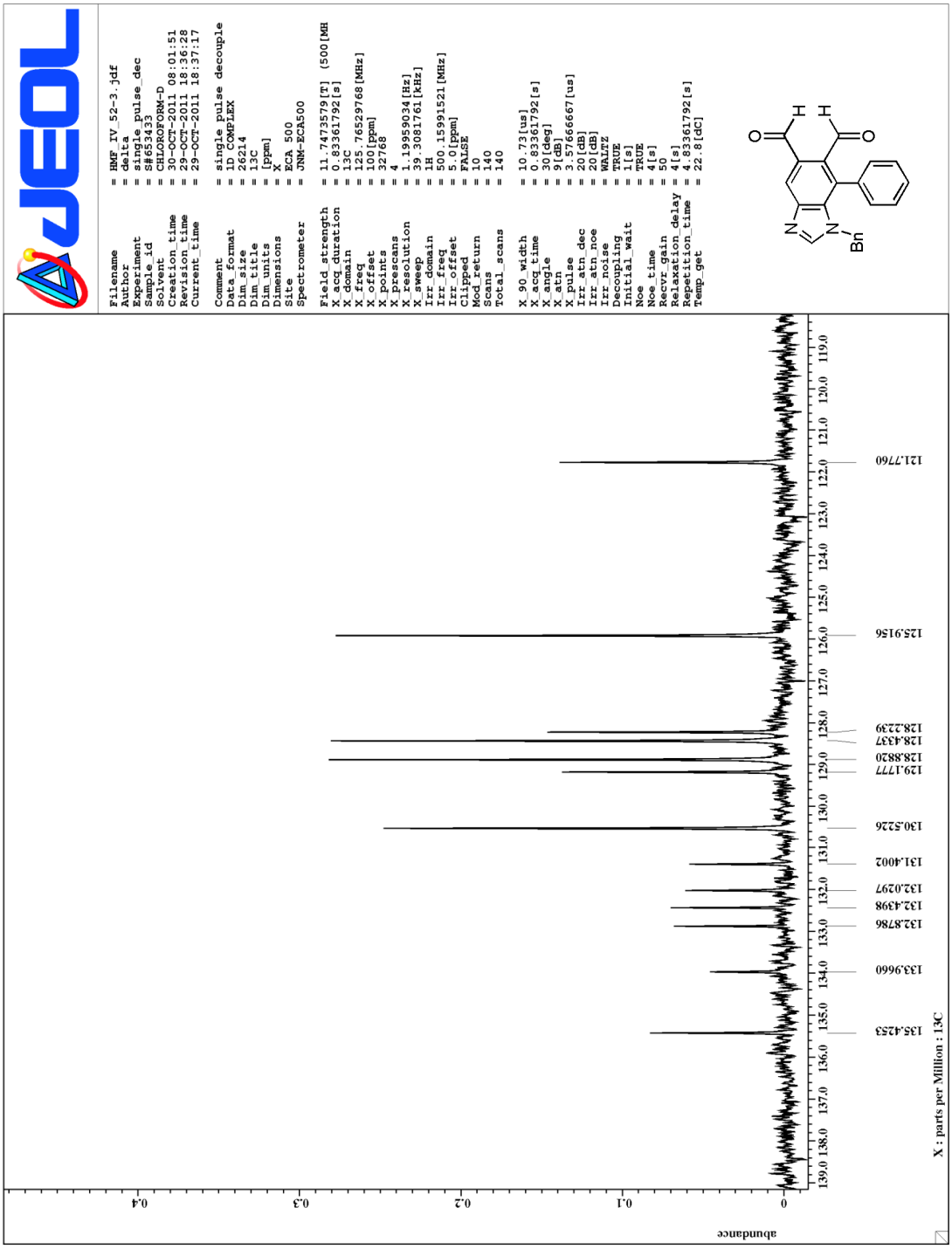

JEOL
Filename = HMF_IV_51-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#5401S8
Solvent = CHLOROFORM-D
Creation_time = 25-OCT-2011 04:54:38
Revision_time = 24-OCT-2011 15:45:06
Current_time = 24-OCT-2011 15:49:39
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 160
Total_scans = 160
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 23.1[dC]
OH
OH
N
N
Bn
abundance
X : parts per Million : 13C
125.9919
127.7374
127.6802
128.0808
128.0331
128.6150
130.3605
131.4669
(thousandths)
134.0900
135.0724
135.8736
136.4936

APPENDIX 50
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-4-phenyl-1*H*-benzimidazole-5,6-dicarbaldehyde (**135**)

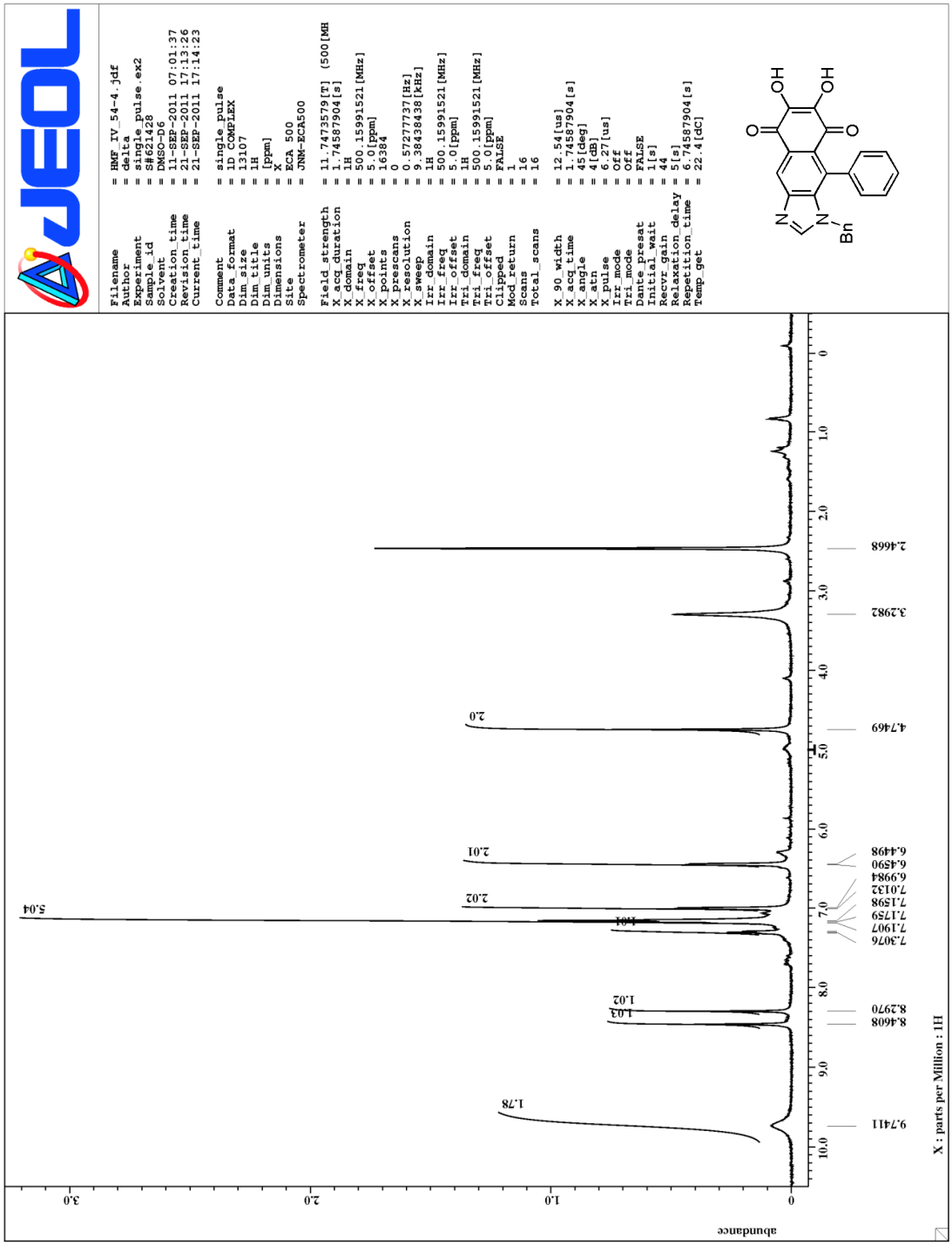

Filename = HMF_IV_52_1-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#651553
Solvent = CHLOROFORM-D
Creation_time = 30-OCT-2011 07:49:44
Revision_time = 29-OCT-2011 18:34:06
Current_time = 29-OCT-2011 18:34:51
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.745879044[s]
Temp_get = 22.5[dC]
Bn
abundance
X : parts per Million : 1H
10.4786
9.8671
8.4092
8.0577
7.3121
7.2549
7.1759
7.1541
6.5185
6.5036
4.8546
4.01
2.01
2.0
1.02
1.01
1.03
1.0

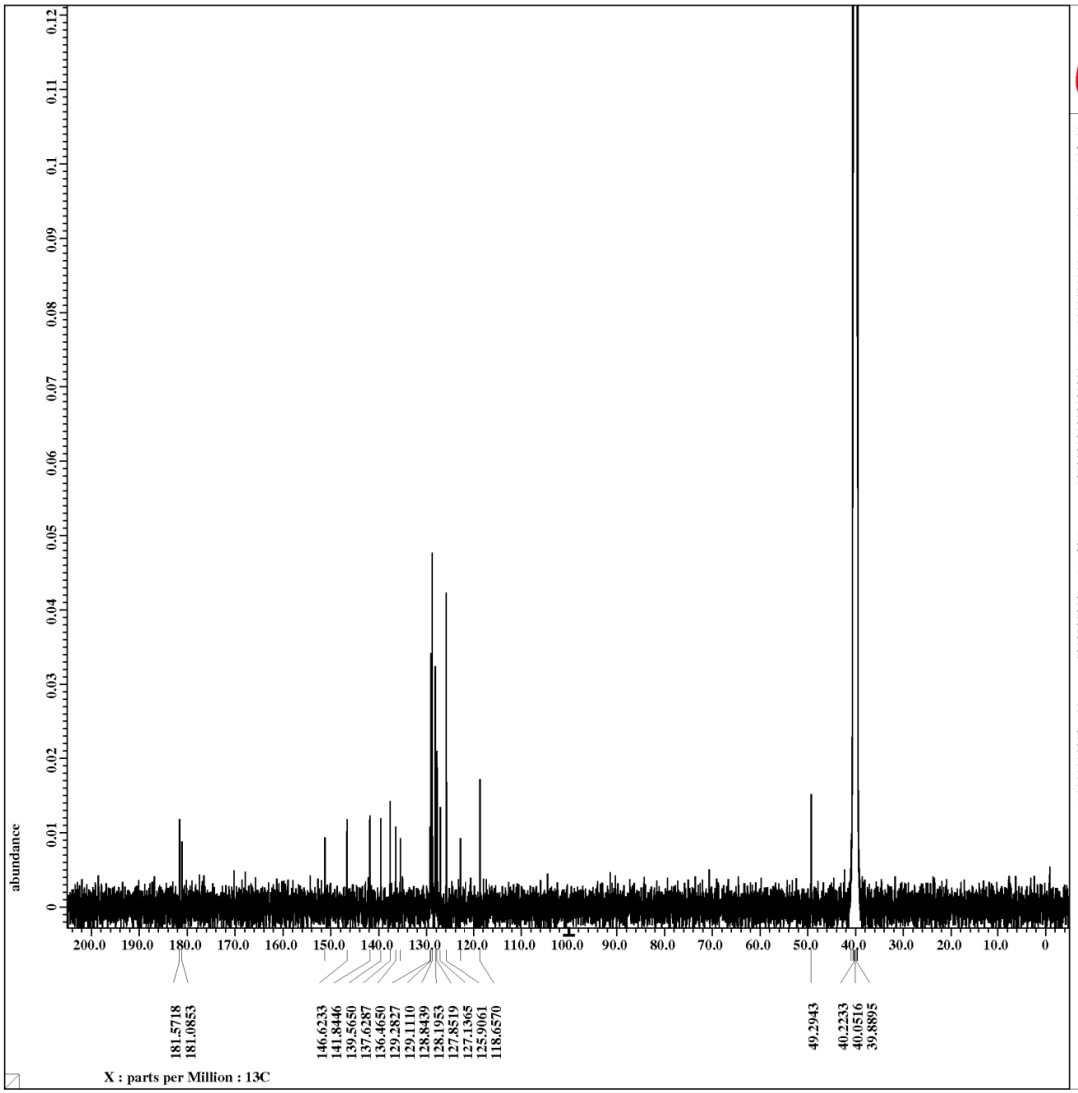

Filename = HMF_IV_52_1-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#651553
Solvent = CHLOROFORM-D
Creation_time = 30-OCT-2011 07:49:44
Revision_time = 29-OCT-2011 18:35:43
Current_time = 29-OCT-2011 18:36:06
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.5[dC]
Bn
abundance
X : parts per Million : 1H
7.45
8.02
4.01
2.04
1.01
2.0
1.02
6.5036
6.5111
6.5185
7.1369
7.1404
7.1541
7.1564
7.1610
7.1759
7.1907
7.2136
7.2285
7.2549
7.2972
7.3121
7.3282
7.4255
7.4278
7.4427
7.4576


JEOL
Filename = HMF_IV_52-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#653433
Solvent = CHLOROFORM-D
Creation_time = 30-OCT-2011 08:01:51
Revision_time = 29-OCT-2011 18:36:28
Current_time = 29-OCT-2011 18:37:00
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159915211[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 140
Total_scans = 140
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.8[dC]
H
H
O
O
N
N
Bn
abundance
X : parts per Million : 13C
192.7698
192.4168
149.6661
146.8236
130.5226
129.1777
128.8820
128.4337
128.2239
125.9136
121.7760
77.3751
77.1176
76.8696
50.2099


Filename = HMF_IV_52-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#653433
Solvent = CHLOROFORM-D
Creation_time = 30-OCT-2011 08:01:51
Revision_time = 29-OCT-2011 18:36:28
Current_time = 29-OCT-2011 18:37:17
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 140
Total_scans = 140
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.8[dC]
H
H
O
O
N
N
Bn
abundance
0.4
0.3
0.2
0.1
0
139.0 138.0 137.0 136.0 135.0 134.0 133.0 132.0 131.0 130.0 129.0 128.0 127.0 126.0 125.0 124.0 123.0 122.0 121.0 120.0 119.0
121.7760
125.9156
128.2239
128.4337
128.8820
129.1777
130.5226
131.4002
132.0297
132.4398
132.8786
133.9660
135.4253
X : parts per Million : 13C

APPENDIX 51
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-6,7-dihydroxy-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-5,8-dione (**136**)


JEOL
Filename = HMF_IV_54-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#621428
Solvent = DMSO-D6
Creation_time = 11-SEP-2011 07:01:37
Revision_time = 21-SEP-2011 17:13:26
Current_time = 21-SEP-2011 17:14:23
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.4[dC]
OH
OH
O
O
N
N
Bn
abundance
X : parts per Million : 1H
9.7411
8.4608
8.2970
7.3076
7.1907
7.1759
7.1598
7.0132
6.9984
6.4590
6.4498
4.7469
3.2982
2.4668
5.04
1.78
1.01
1.03
1.02
2.02
2.01
2.0

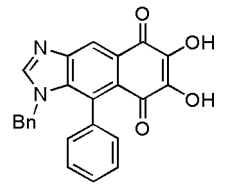


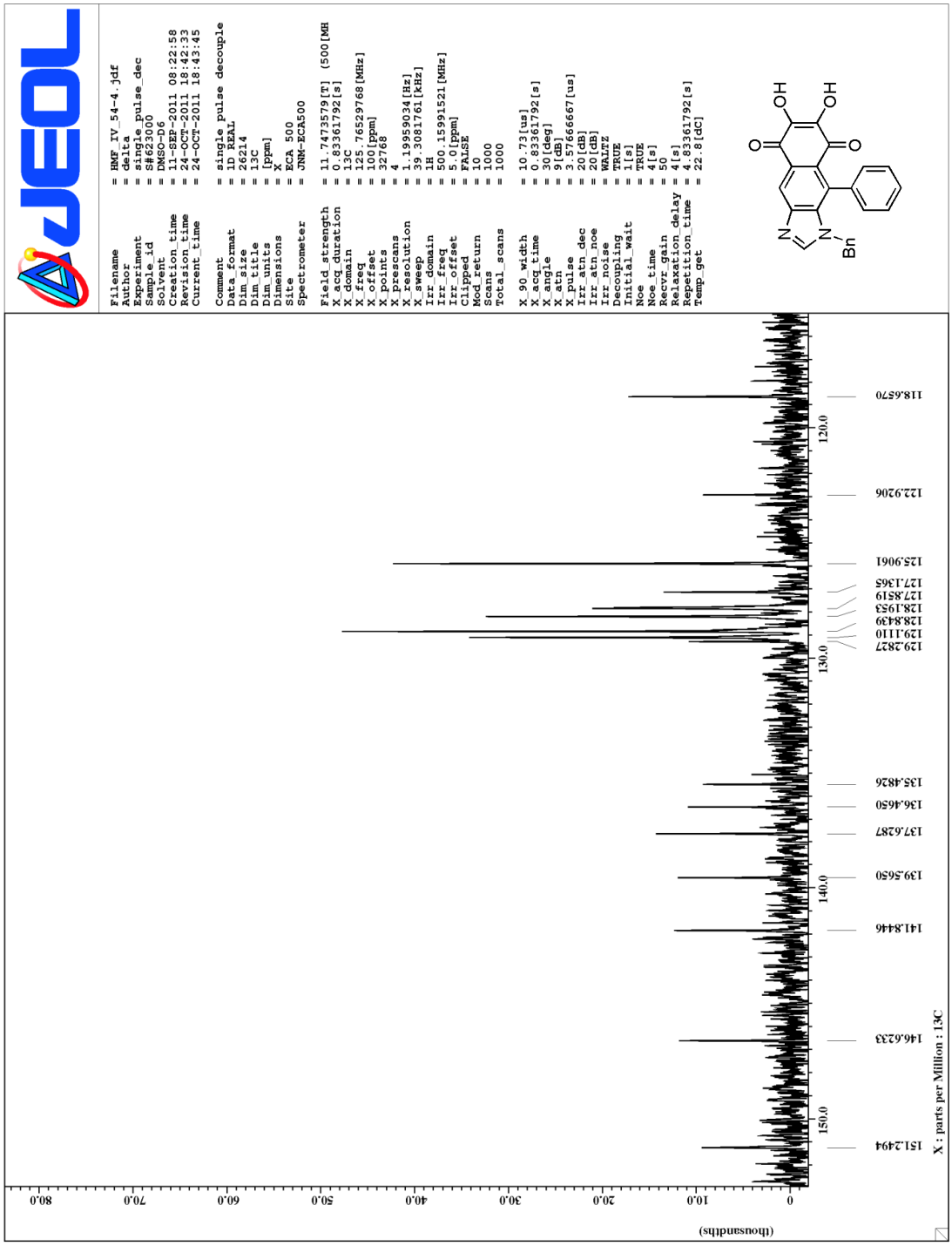
```
Filename          = HMF_IV_54-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#623000
Solvent           = DMSO-D6
Creation_time     = 11-SEP-2011 08:22:58
Revision_time     = 24-OCT-2011 18:42:33
Current_time      = 24-OCT-2011 18:43:16

Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

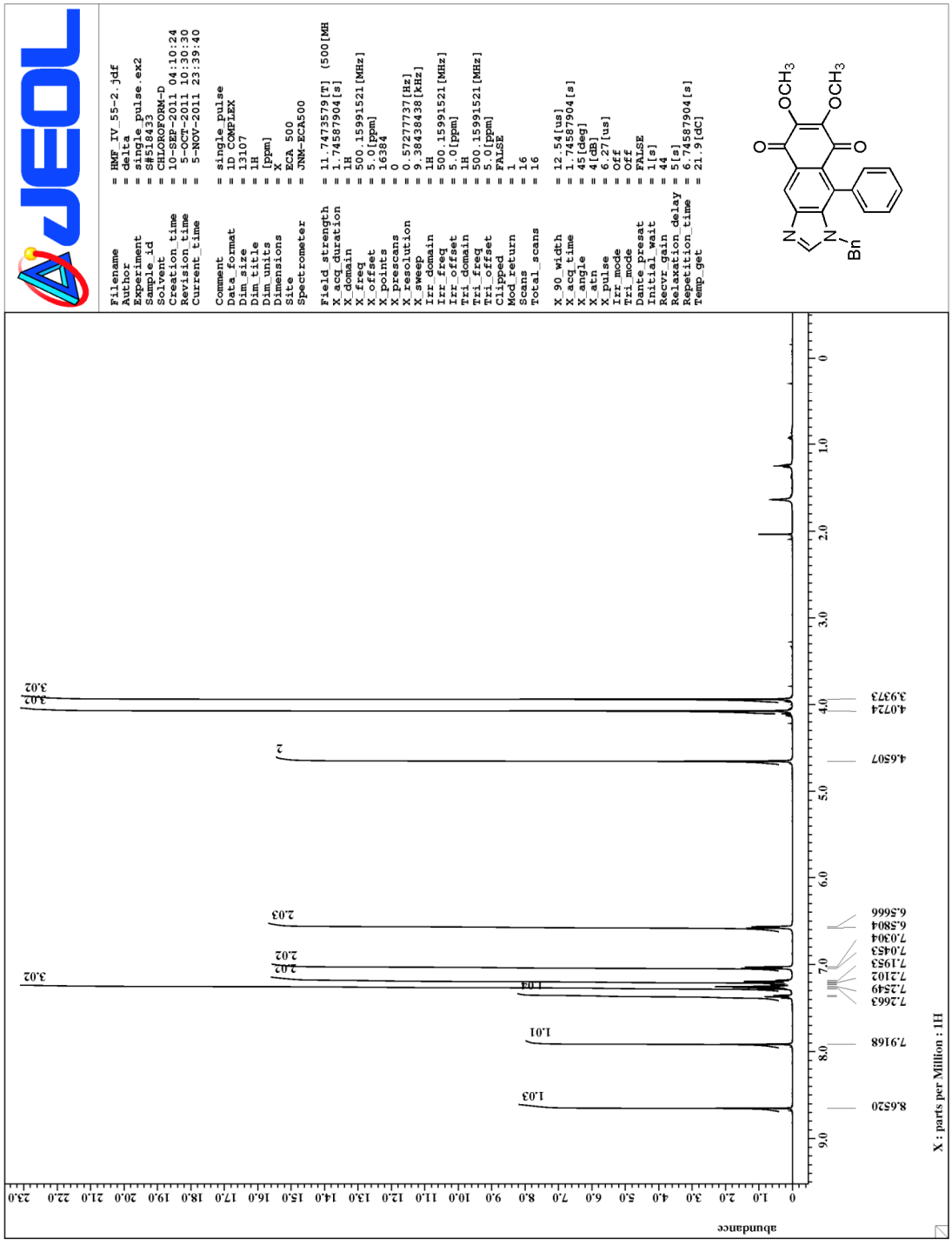
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 1000
Total_scans       = 1000

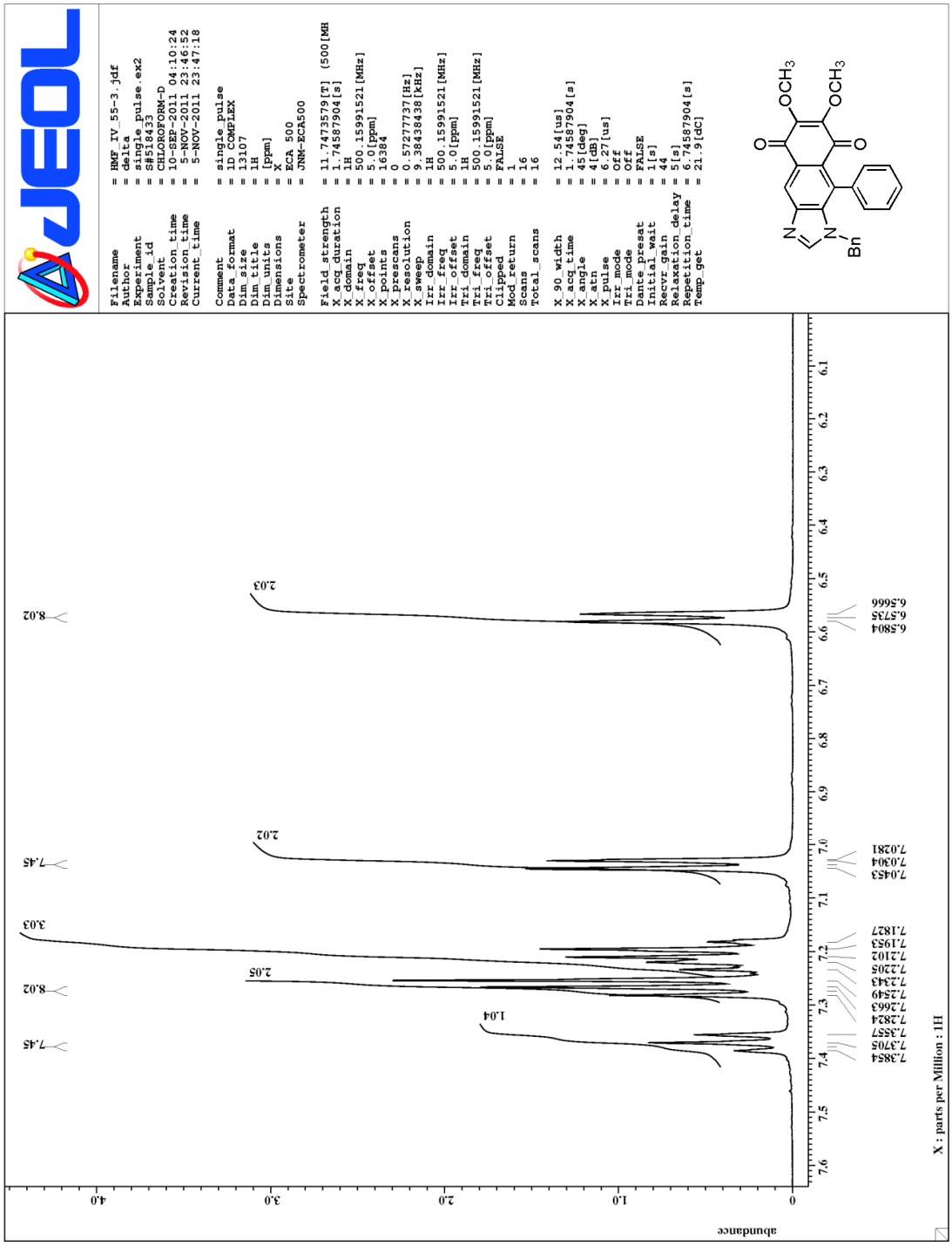
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 22.8[dC]
```

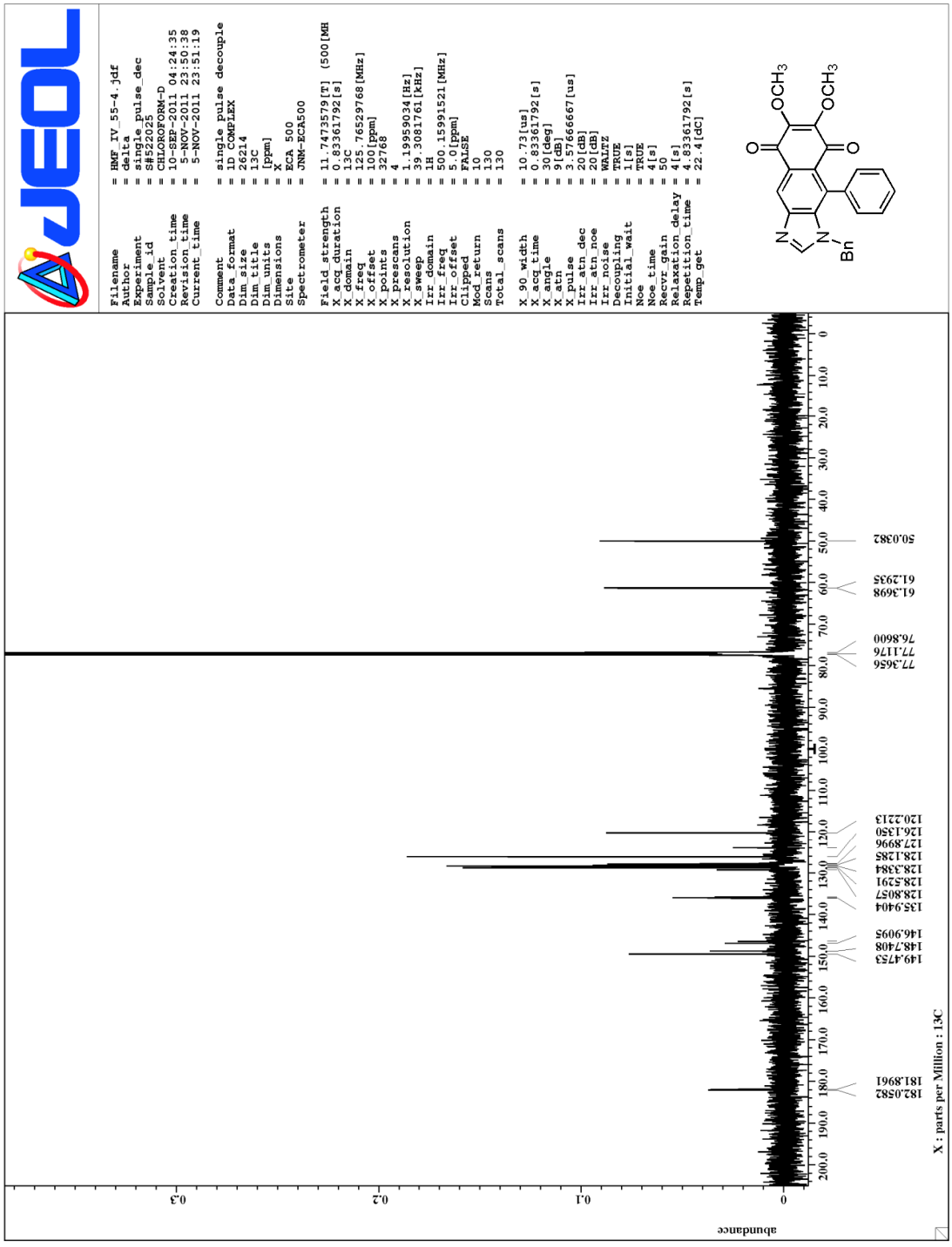


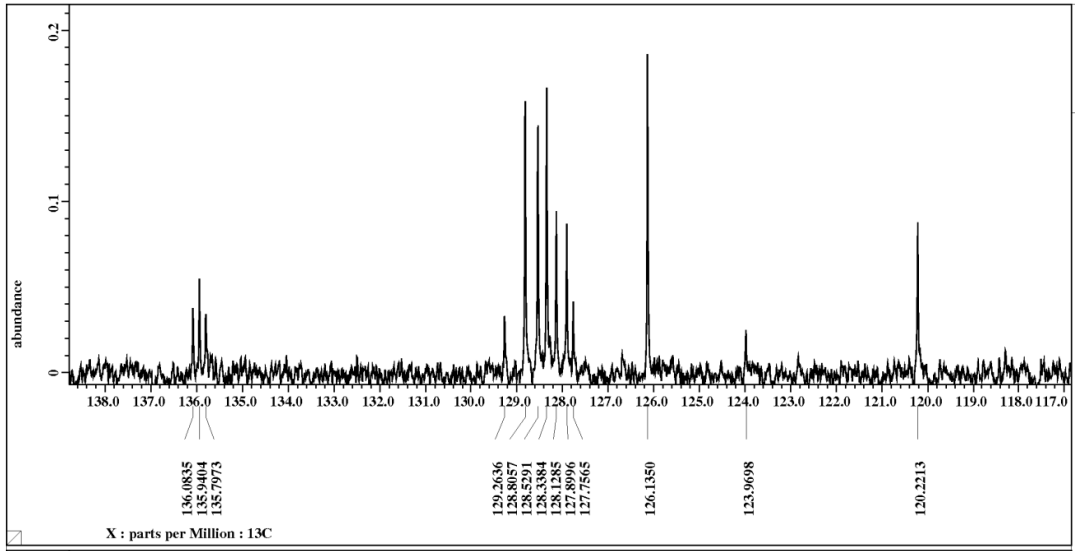

JEOL
Filename = HMF_IV_54-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#623000
Solvent = DMSO-D6
Creation_time = 11-SEP-2011 08:22:58
Revision_time = 24-OCT-2011 18:42:33
Current_time = 24-OCT-2011 18:43:45
Comment = single_pulse_decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1000
Total_scans = 1000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.8[dC]
OH
OH
O
O
N
N
Bn
(thousandths)
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
150.0
140.0
130.0
120.0
151.2494
146.6233
141.8446
139.5650
137.6287
136.4650
135.4826
129.2827
129.1110
128.8439
128.1953
127.8519
127.1365
125.9061
122.9206
118.6570
X : parts per Million : 13C

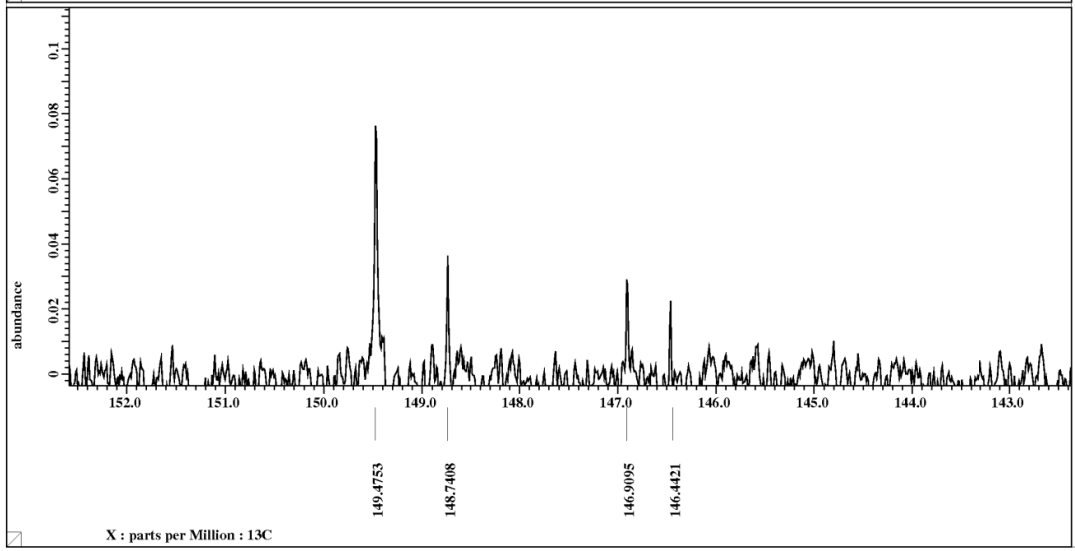
APPENDIX 52
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-6,7-dimethoxy-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (**137**)


JEOL
Filename = HMF_IV_55-2.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#518433
Solvent = CHLOROFORM-D
Creation_time = 10-SEP-2011 04:10:24
Revision_time = 5-OCT-2011 10:30:30
Current_time = 5-NOV-2011 23:39:40
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6.27[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
OCH3
OCH3
N
N
Bn
abundance
X : parts per Million : 1H
3.02
3.02
2
2.03
2.02
2.02
3.02
1.03
1.01
1.03
8.6520
7.9168
7.2663
7.2549
7.2102
7.1953
7.0453
7.0304
6.5804
6.5666
4.6507
4.0724
3.9373


Filename = HMF_IV_55-3.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#5184433
Solvent = CHLOROFORM-D
Creation_time = 10-SEP-2011 04:10:24
Revision_time = 5-NOV-2011 23:46:52
Current_time = 5-NOV-2011 23:47:18
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
OCH3
OCH3
N
N
Bn
abundance
X : parts per Million : 1H
8.02
7.45
3.03
8.02
7.45
2.03
2.02
2.05
1.04
6.5666
6.5735
6.5804
7.0281
7.0304
7.0453
7.1827
7.1953
7.2102
7.2205
7.2343
7.2549
7.2663
7.2824
7.3557
7.3705
7.3854


Filename = HMF_IV_55-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#522025
Solvent = CHLOROFORM-D
Creation_time = 10-SEP-2011 04:24:35
Revision_time = 5-NOV-2011 23:50:38
Current_time = 5-NOV-2011 23:51:19
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 130
Total_scans = 130
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.4[dC]
OCH3
OCH3
N
N
Bn
abundance
0
0.1
0.2
0.3
182.0582
181.8961
149.4753
148.7408
146.9095
135.9404
128.8057
128.5291
128.3384
128.1285
127.8996
126.1350
120.2213
77.3656
77.1176
76.8600
61.3698
61.2935
50.0382
X : parts per Million : 13C



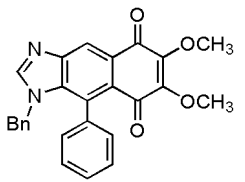



```
Filename          = HMF_IV_55-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#522025
Solvent           = CHLOROFORM-D
Creation_time     = 10-SEP-2011 04:24:35
Revision_time     =  5-NOV-2011 23:50:38
Current_time      =  5-NOV-2011 23:52:15

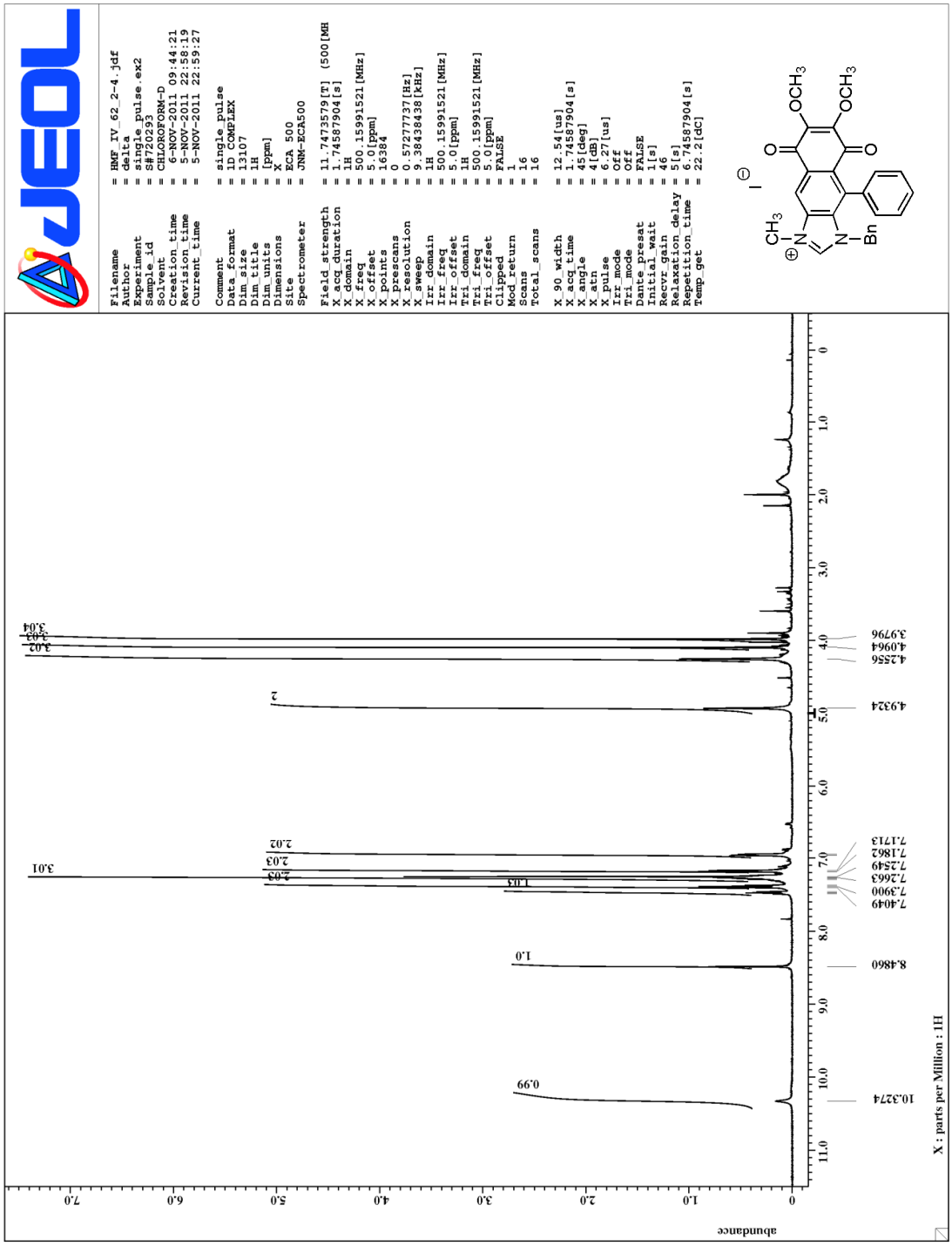
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

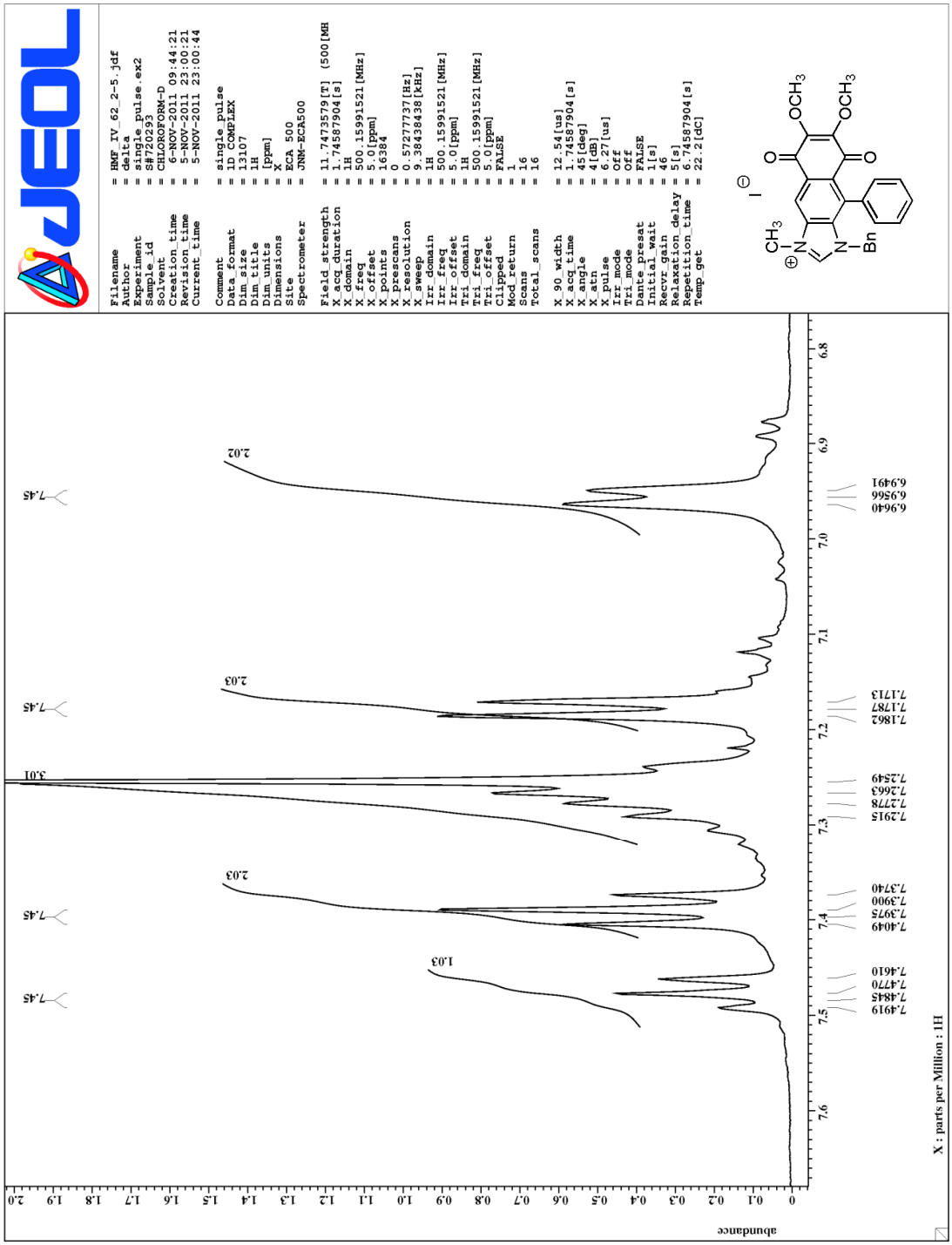
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 130
Total_scans       = 130

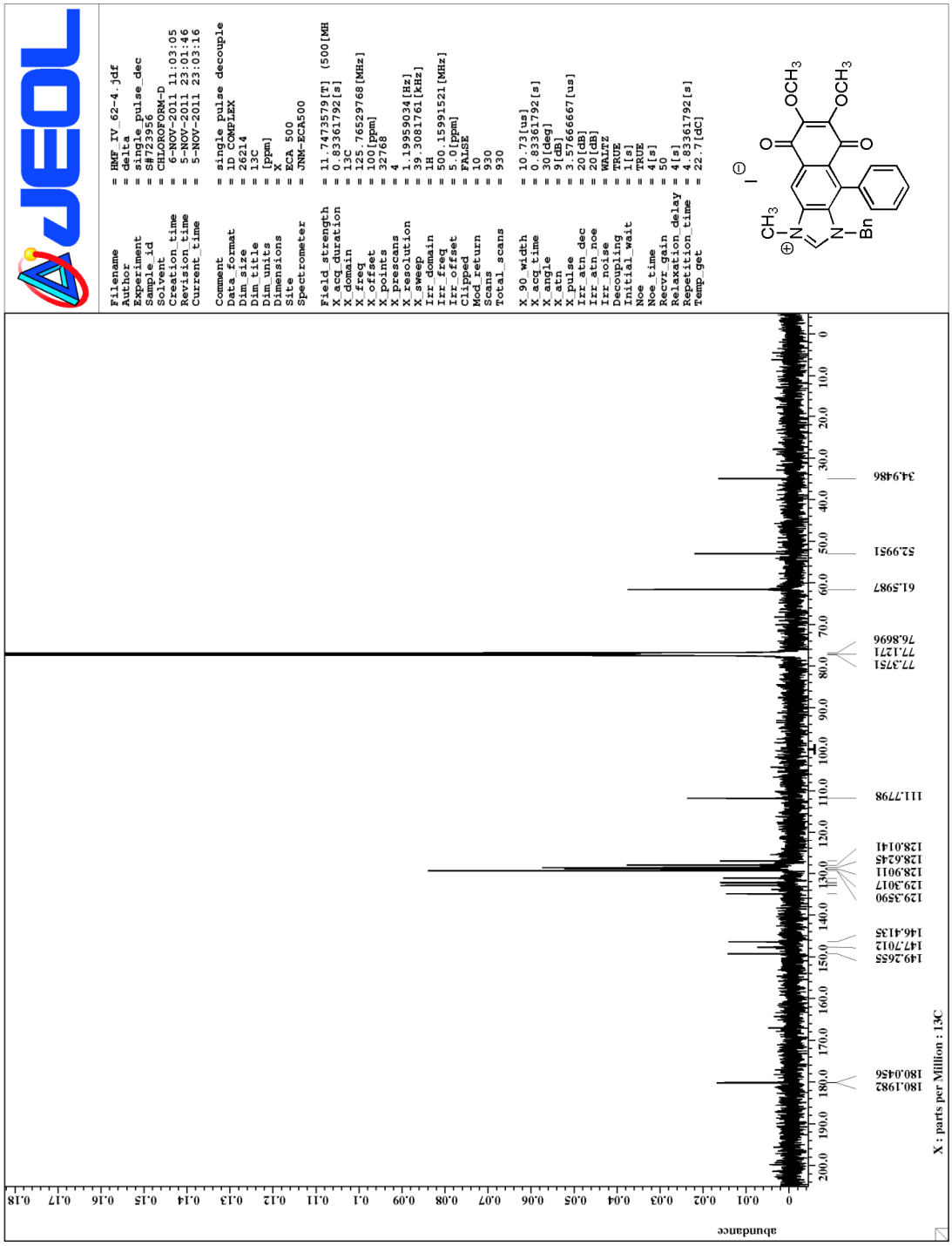
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 22.4[dC]
```

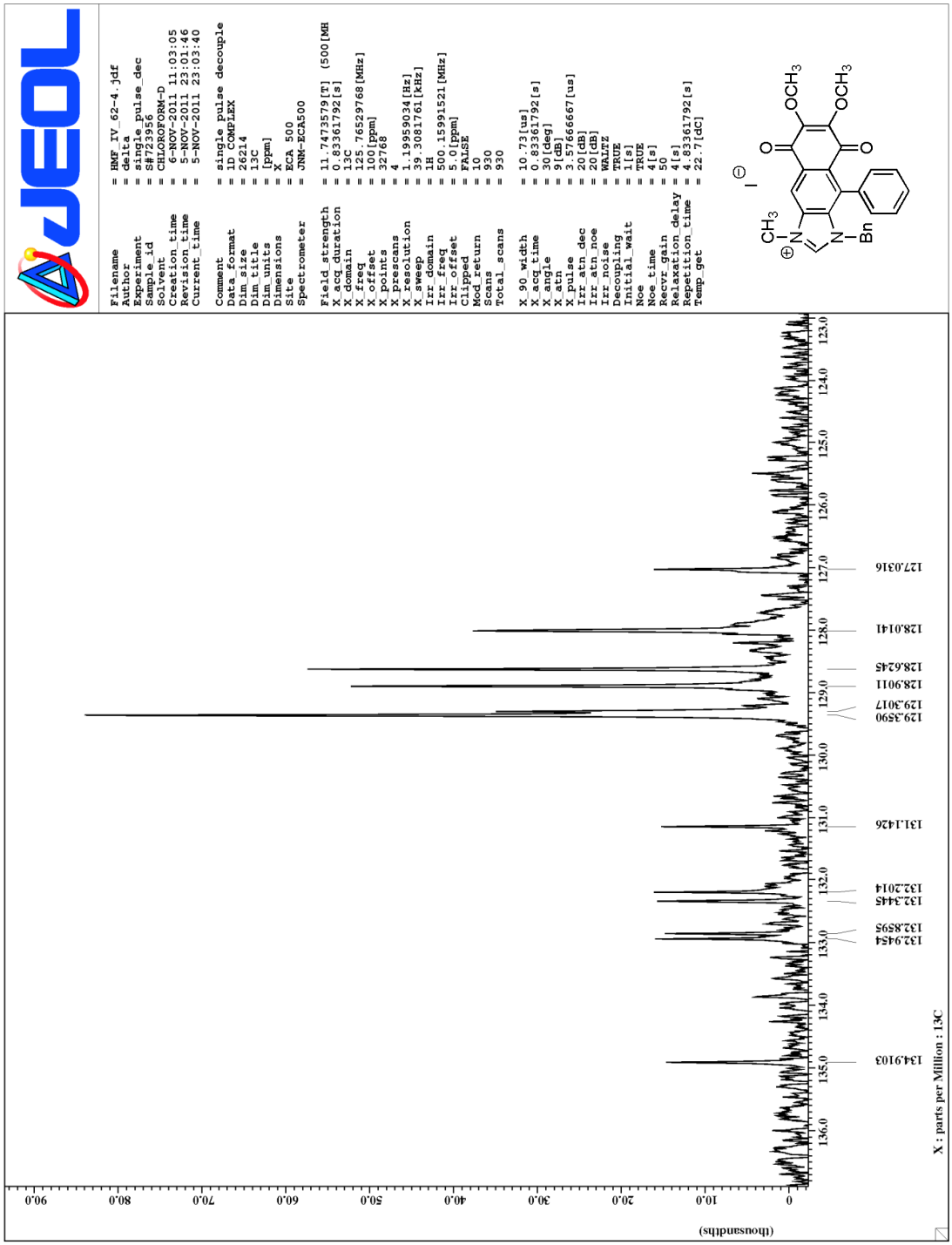


APPENDIX 53
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-6,7-dimethoxy-1-methyl-4-phenyl-1*H*-naphtho[2,3-*d*]imidazolium-5,8-dione iodide (**138**)

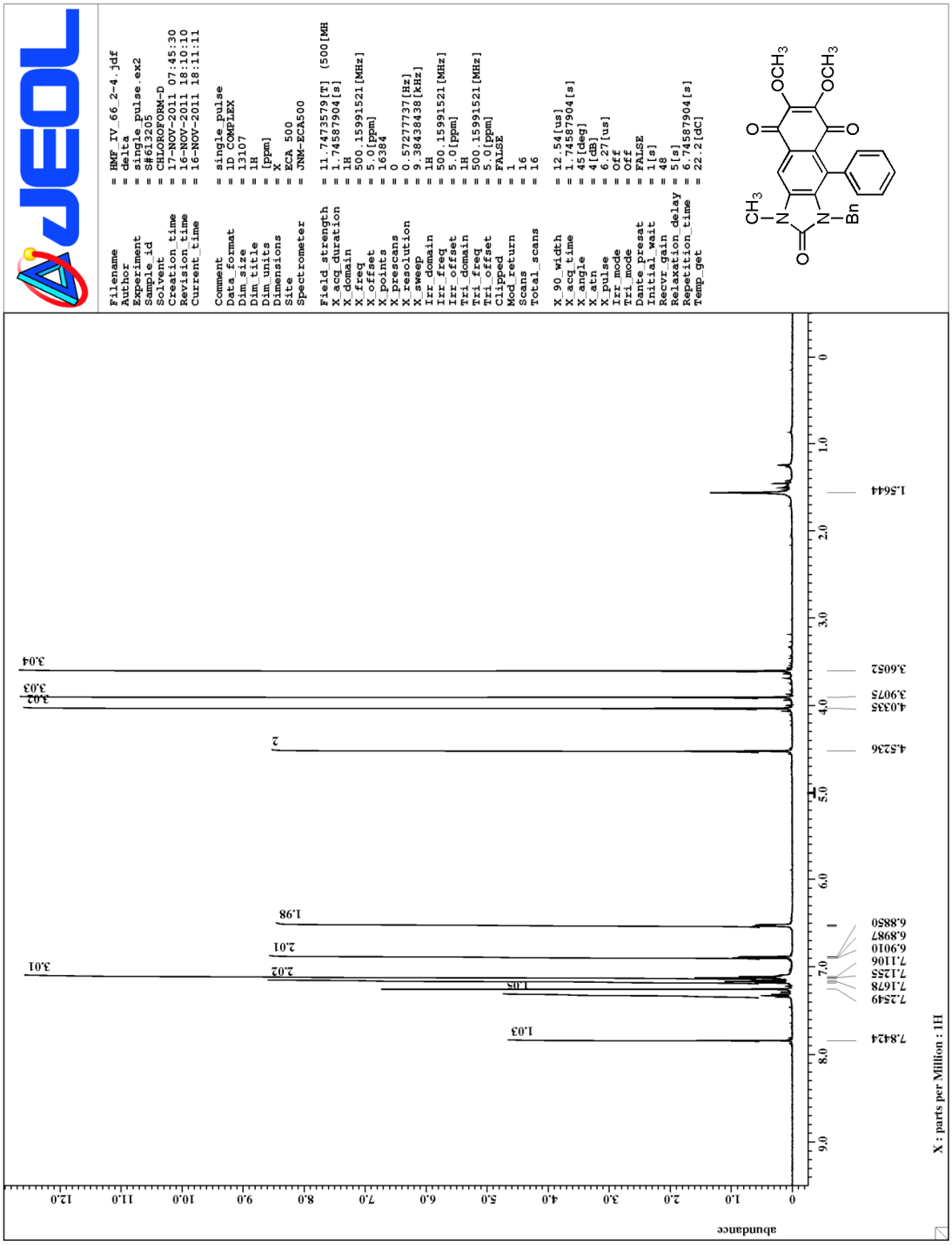

JEOL
Filename = RMF_IV_62_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#720293
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 09:44:21
Revision_time = 5-NOV-2011 22:58:19
Current_time = 5-NOV-2011 22:59:27
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.2[dC]
OCH3
OCH3
CH3
Bn
abundance
X : parts per Million : 1H
10.3274
8.4860
7.4049
7.3900
7.2663
7.2549
7.1862
7.1713
4.9324
4.2556
4.0964
3.9796
3.02
3.03
3.04
3.01
2
2.02
2.03
2.03
1.03
1.0
0.99

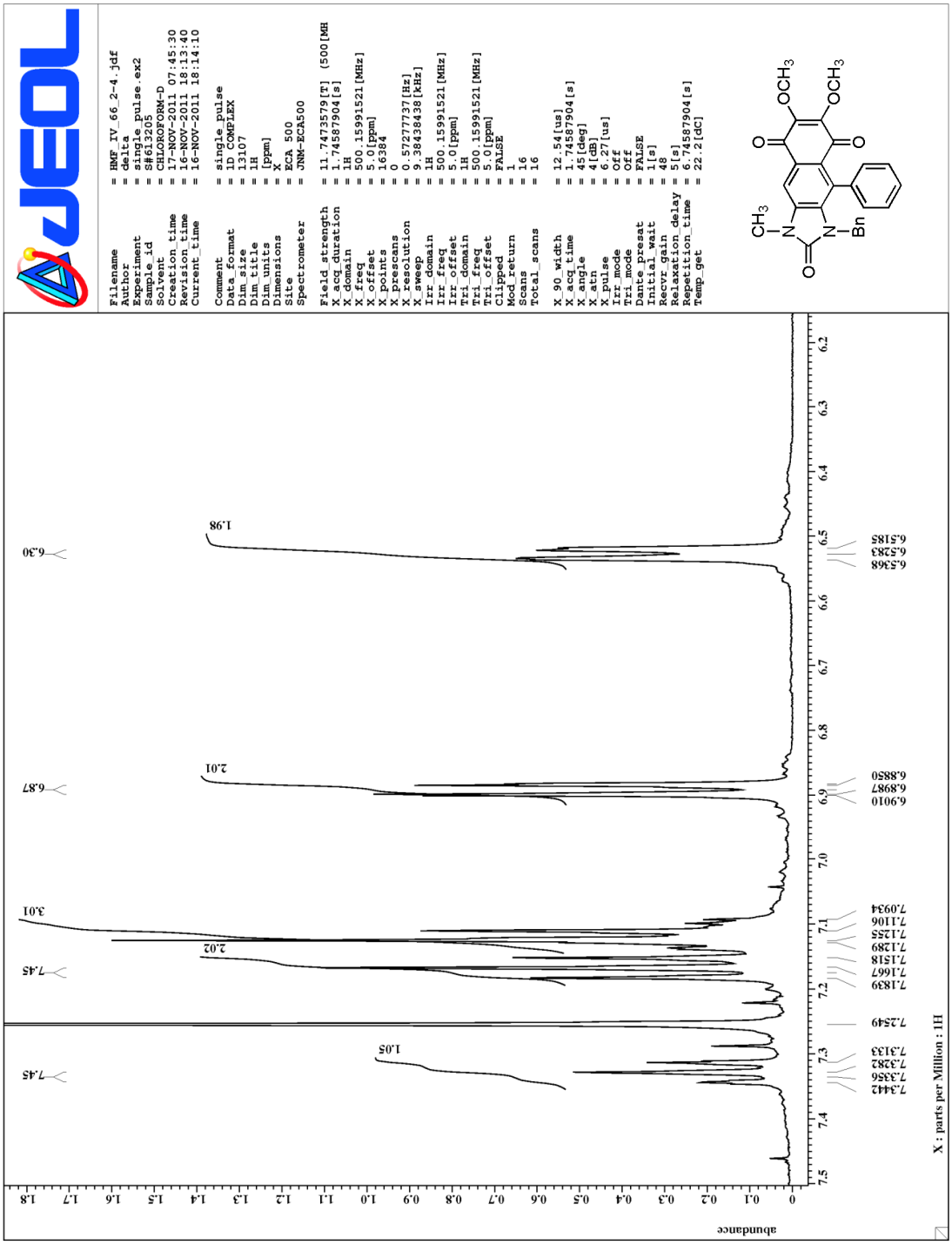

Filename = RMF_IV_62_2-5.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#720293
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 09:44:21
Revision_time = 5-NOV-2011 23:00:21
Current_time = 5-NOV-2011 23:00:44
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.159911521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384381[kHz]
Irr_domain = 1H
Irr_freq = 500.159911521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.159911521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.2[dC]
abundance
X : parts per Million : 1H
7.4919
7.4845
7.4770
7.4610
7.4049
7.3975
7.3900
7.3740
7.2915
7.2778
7.2663
7.2549
7.1862
7.1787
7.1713
6.9640
6.9566
6.9491
1.03
2.03
2.03
3.01
2.03
2.02
7.45
7.45
7.45
7.45

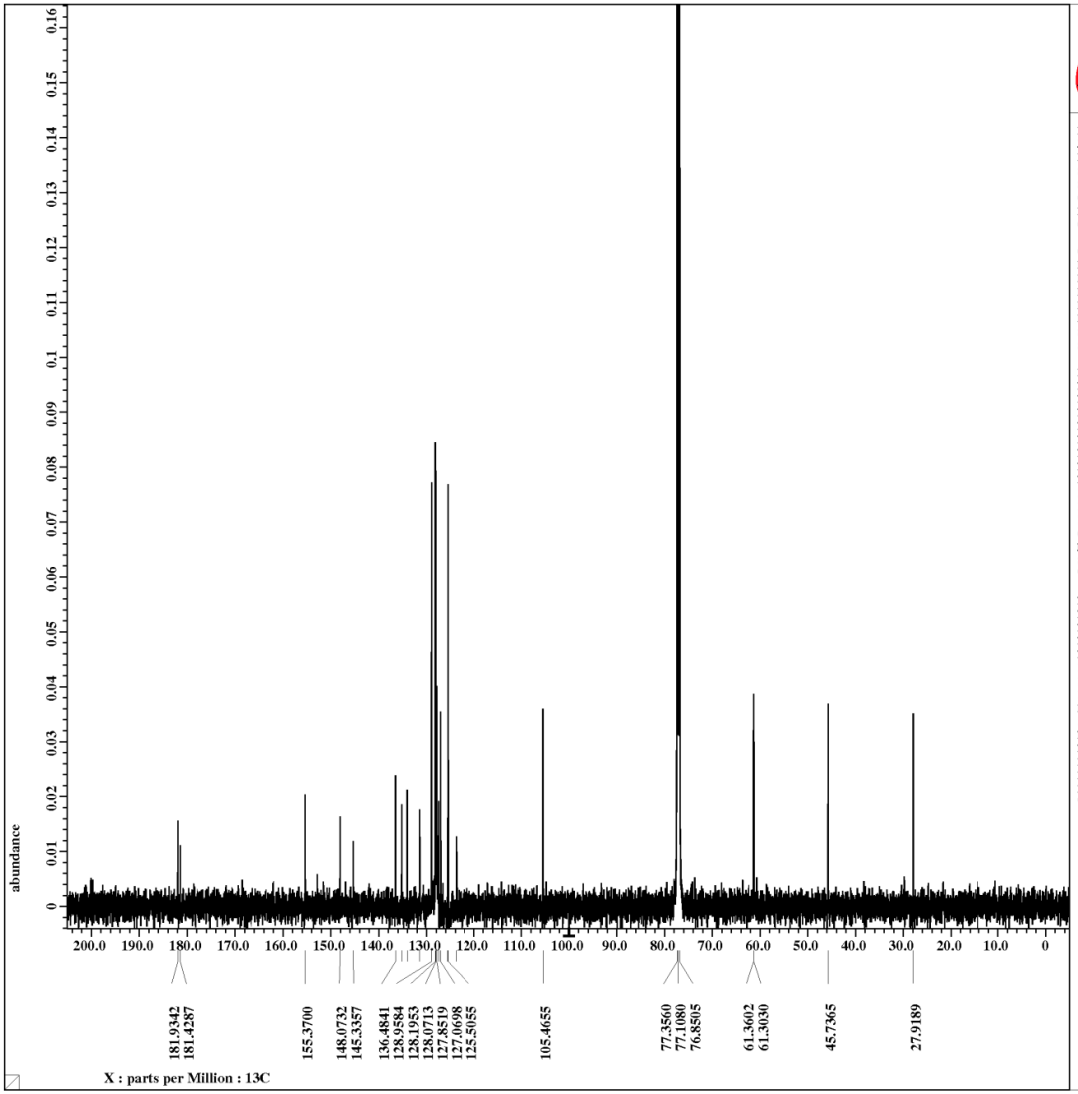

Filename = RMF_IV_62-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#723956
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 11:03:05
Revision_time = 5-NOV-2011 23:01:46
Current_time = 5-NOV-2011 23:03:16
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 930
Total_scans = 930
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.7[dC]
OCH3
OCH3
CH3
Bn
34.9486
52.9951
61.5987
76.8696
77.1271
77.3751
111.7798
128.0141
128.6245
128.9011
129.3017
129.3590
146.4135
147.7012
149.2655
180.0456
180.1982
X : parts per Million : 13C
abundance


Filename = HMF_IV_62-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#723956
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 11:03:05
Revision_time = 5-NOV-2011 23:01:46
Current_time = 5-NOV-2011 23:03:40
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 930
Total_scans = 930
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.7[dC]
127.0316
128.0141
128.6245
128.9011
129.3017
129.3590
131.1426
132.2014
132.3445
132.8595
132.9454
134.9103
X : parts per Million : 13C
(thousandths)

APPENDIX 54
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-6,7-dimethoxy-1-methyl-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (**139a**)


Filename = HMF_IV_66_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#613205
Solvent = CHLOROFORM-D
Creation_time = 17-NOV-2011 07:45:30
Revision_time = 16-NOV-2011 18:10:10
Current_time = 16-NOV-2011 18:11:11
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45.0[deg]
X_atn = 6.27[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.2[dC]
OCH3
OCH3
CH3
N
N
Bn
O
O
O
O
abundance
X : parts per Million : 1H
1.5644
3.6052
3.9075
4.0335
4.5236
7.8424
7.2549
7.1678
7.1255
7.1106
6.9010
6.8987
6.8850
3.04
3.03
3.02
3.01
2.02
2.01
1.98
1.05
1.03
2


JEOL
Filename = RMF_IV_66_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#613205
Solvent = CHLOROFORM-D
Creation_time = 17-NOV-2011 07:45:30
Revision_time = 16-NOV-2011 18:13:40
Current_time = 16-NOV-2011 18:14:10
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.2[dC]
OCH3
OCH3
CH3
N
N
Bn
O
O
O
abundance
X : parts per Million : 1H
6.30
6.87
7.45
7.45
3.01
2.02
2.01
1.98
1.05
6.5368
6.5383
6.5185
6.9010
6.8987
6.8850
7.1839
7.1667
7.1518
7.1289
7.1255
7.1106
7.0934
7.2549
7.3442
7.3356
7.3382
7.3133

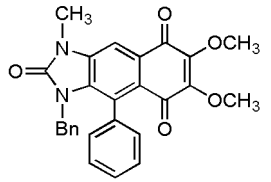


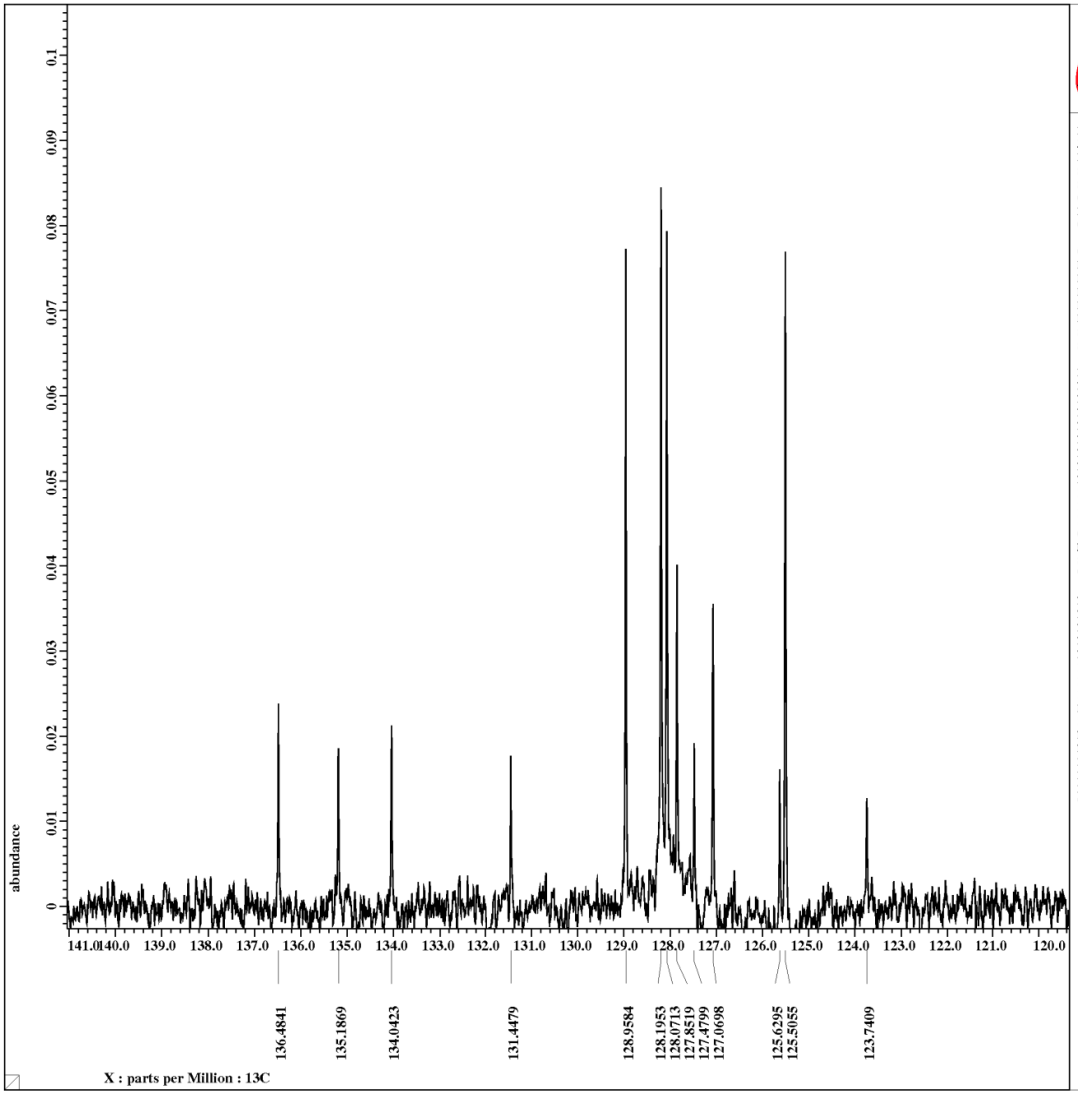
```
Filename          = HMF_IV_58-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#570634
Solvent           = CHLOROFORM-D
Creation_time     = 21-SEP-2011 06:52:15
Revision_time     = 25-OCT-2011 17:02:12
Current_time      = 25-OCT-2011 17:02:53

Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 950
Total_scans       = 950

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 22.8[dC]
```





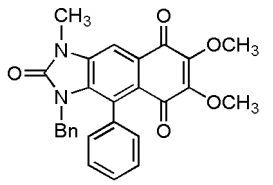
JEOL

```
Filename          = HMF_IV_58-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#570634
Solvent           = CHLOROFORM-D
Creation_time     = 21-SEP-2011 06:52:15
Revision_time     = 25-OCT-2011 17:02:12
Current_time      = 25-OCT-2011 17:03:17

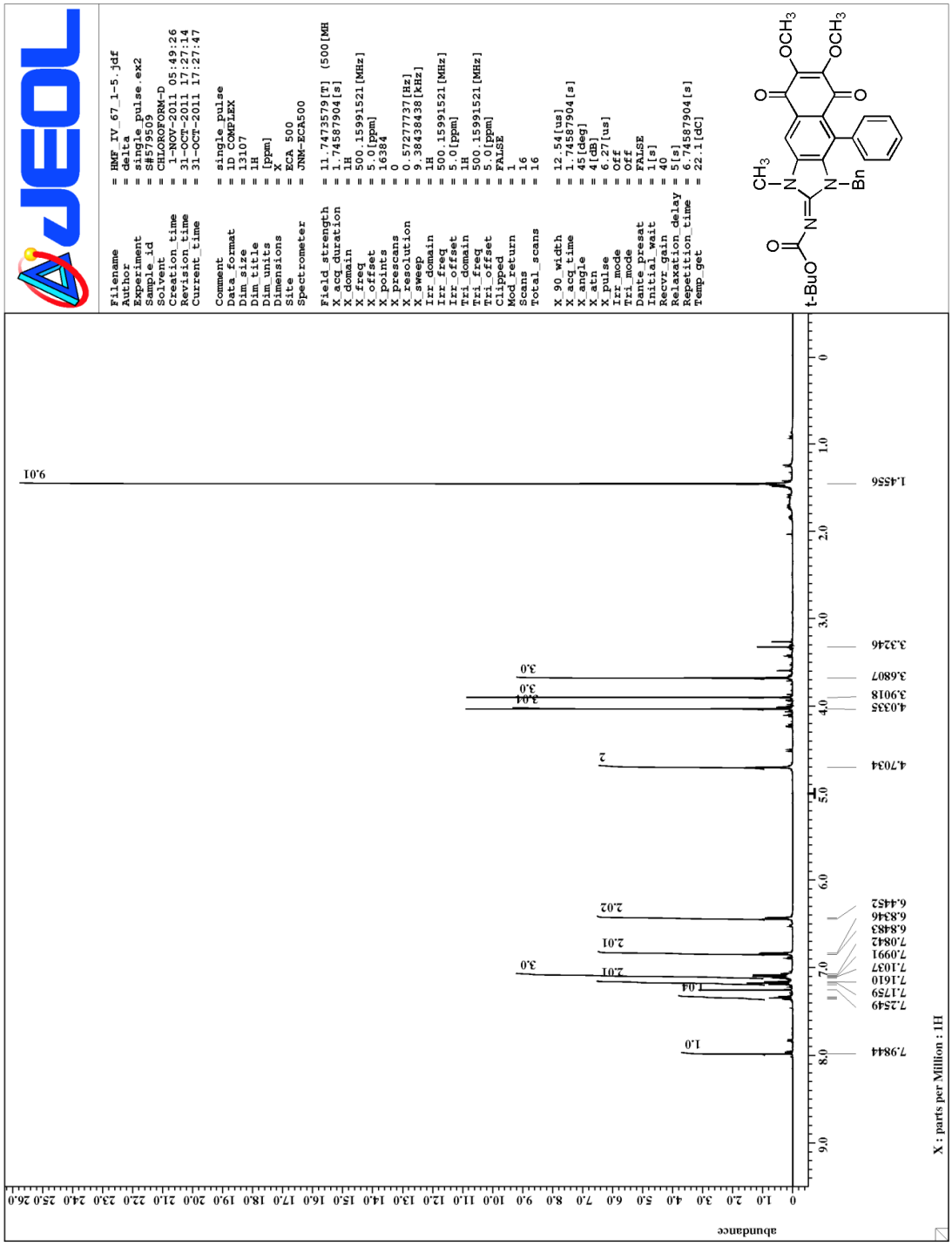
Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

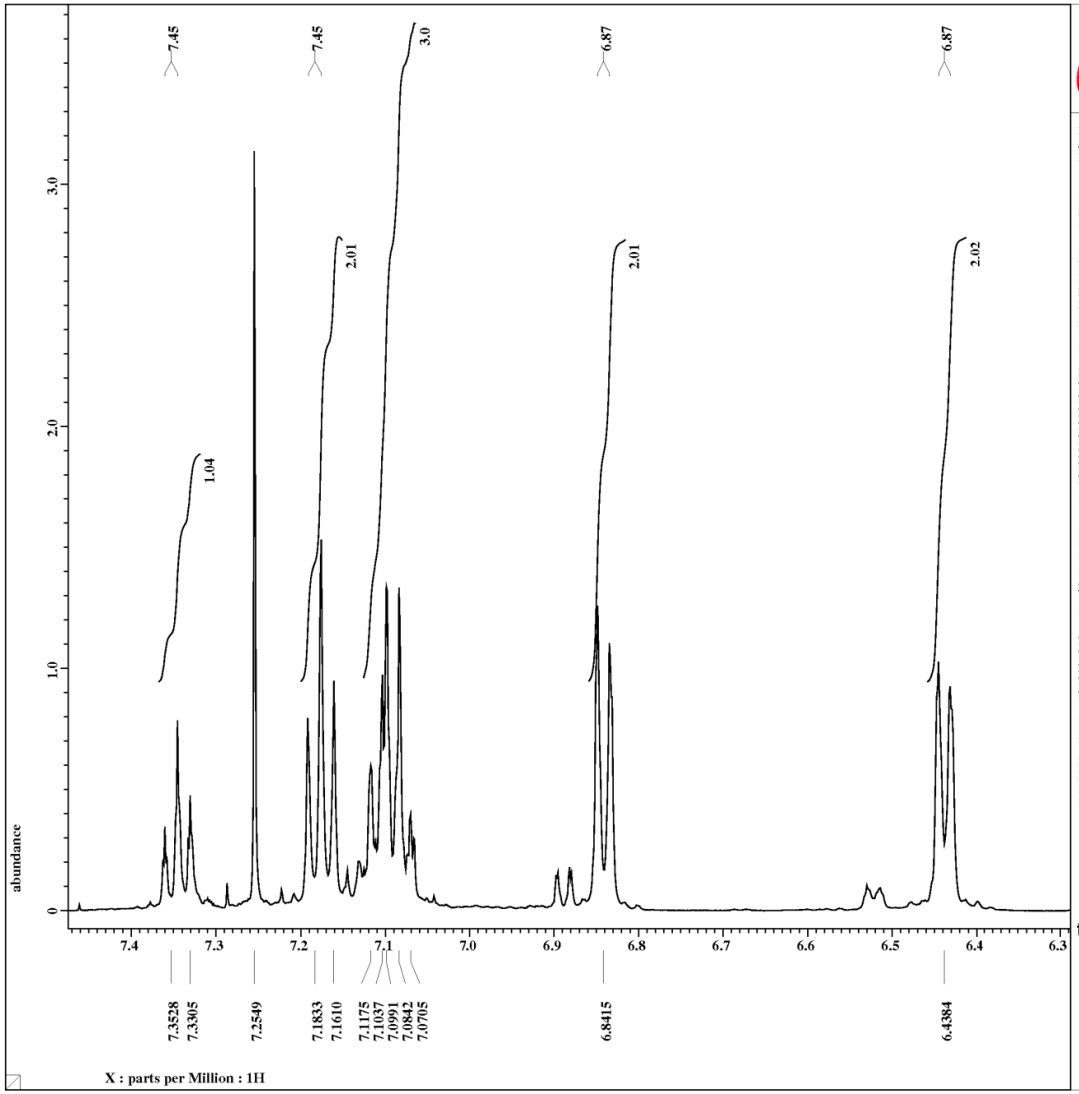
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 950
Total_scans       = 950

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 4[s]
Recvr_gain        = 50
Relaxation_delay  = 4[s]
Repetition_time   = 4.83361792[s]
Temp_get          = 22.8[dC]
```



APPENDIX 55
^{1}H and ^{13}C NMR Spectra of
3-Benzyl-2-*tert*-butylcarbonylimino-6,7-dimethoxy-1-methyl-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (**139b**)


JEOL
Filename = RMF_IV_67_1-5.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#579509
Solvent = CHLOROFORM-D
Creation_time = 1-NOV-2011 05:49:26
Revision_time = 31-OCT-2011 17:27:14
Current_time = 31-OCT-2011 17:27:47
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.1[dC]
OCH3
OCH3
CH3
Bn
t-BuO
abundance
X : parts per Million : 1H
9.01
3.0
3.0
3.01
3.0
2
2.02
2.01
2.01
3.0
1.04
1.0
1.4556
3.3246
3.6807
3.9018
4.0335
4.7034
6.4452
6.8346
6.8483
7.0842
7.0991
7.1037
7.1610
7.1759
7.2549
7.9844

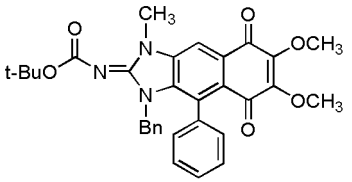


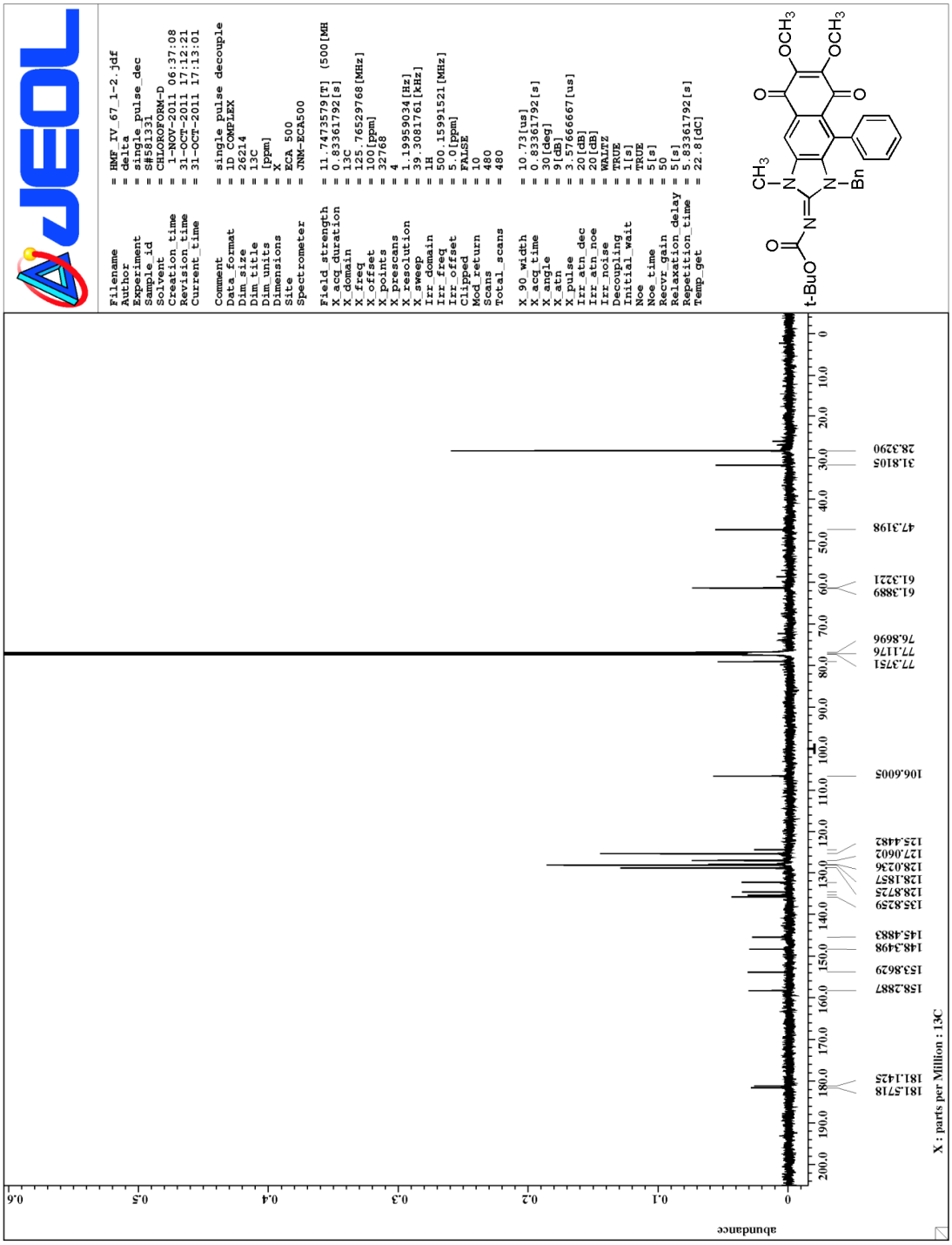

```
Filename          = HMF_IV_67_1-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#579509
Solvent           = CHLOROFORM-D
Creation_time     =  1-NOV-2011 05:49:26
Revision_time     = 31-OCT-2011 17:28:24
Current_time      = 31-OCT-2011 17:28:41

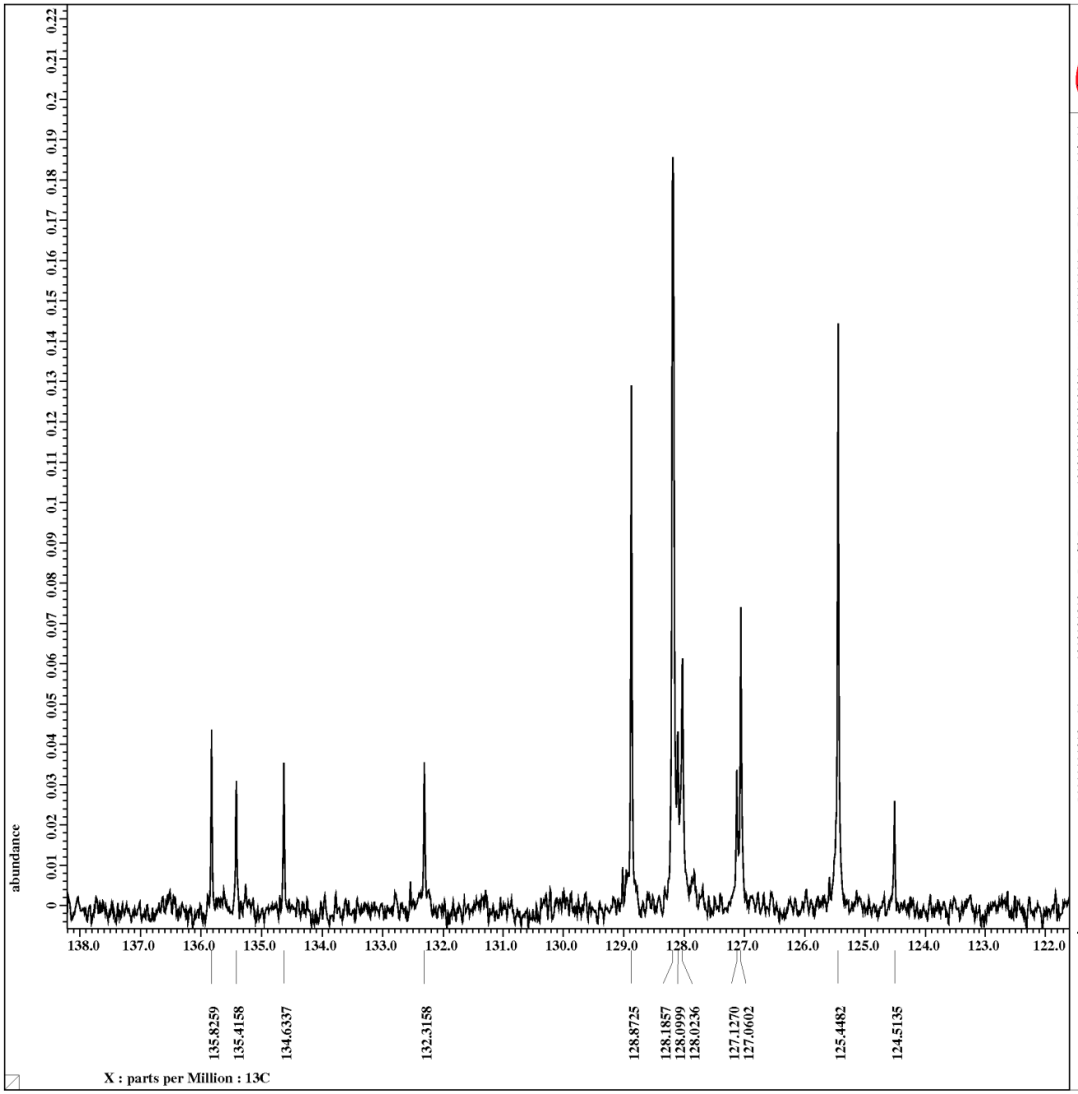
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.1[dC]
```




JEOL
Filename = HMF_IV_67_1-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#581331
Solvent = CHLOROFORM-D
Creation_time = 1-NOV-2011 06:37:08
Revision_time = 31-OCT-2011 17:12:21
Current_time = 31-OCT-2011 17:13:01
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 480
Total_scans = 480
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_seqfil = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe_time = TRUE
Recyr_gain = 5[s]
Relaxation_delay = 50
Repetition_time = 5[s]
Temp_get = 5.83361792[s]
= 22.8[dC]
OCH3
OCH3
CH3
Bn
t-BuO
abundance
X : parts per Million : 13C
181.5718
181.1425
158.2887
153.8629
148.3498
145.4883
135.8259
128.8725
128.1857
128.0236
127.0602
125.4482
106.6005
77.3751
77.1176
76.8696
61.3889
61.3221
47.3198
31.8105
28.3290

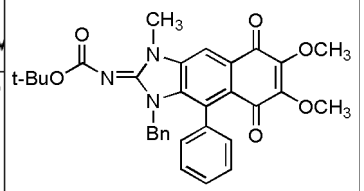


```
Filename          = HMF_IV_67_1-2.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#581331
Solvent           = CHLOROFORM-D
Creation_time     =  1-NOV-2011 06:37:08
Revision_time     = 31-OCT-2011 17:12:21
Current_time      = 31-OCT-2011 17:13:22

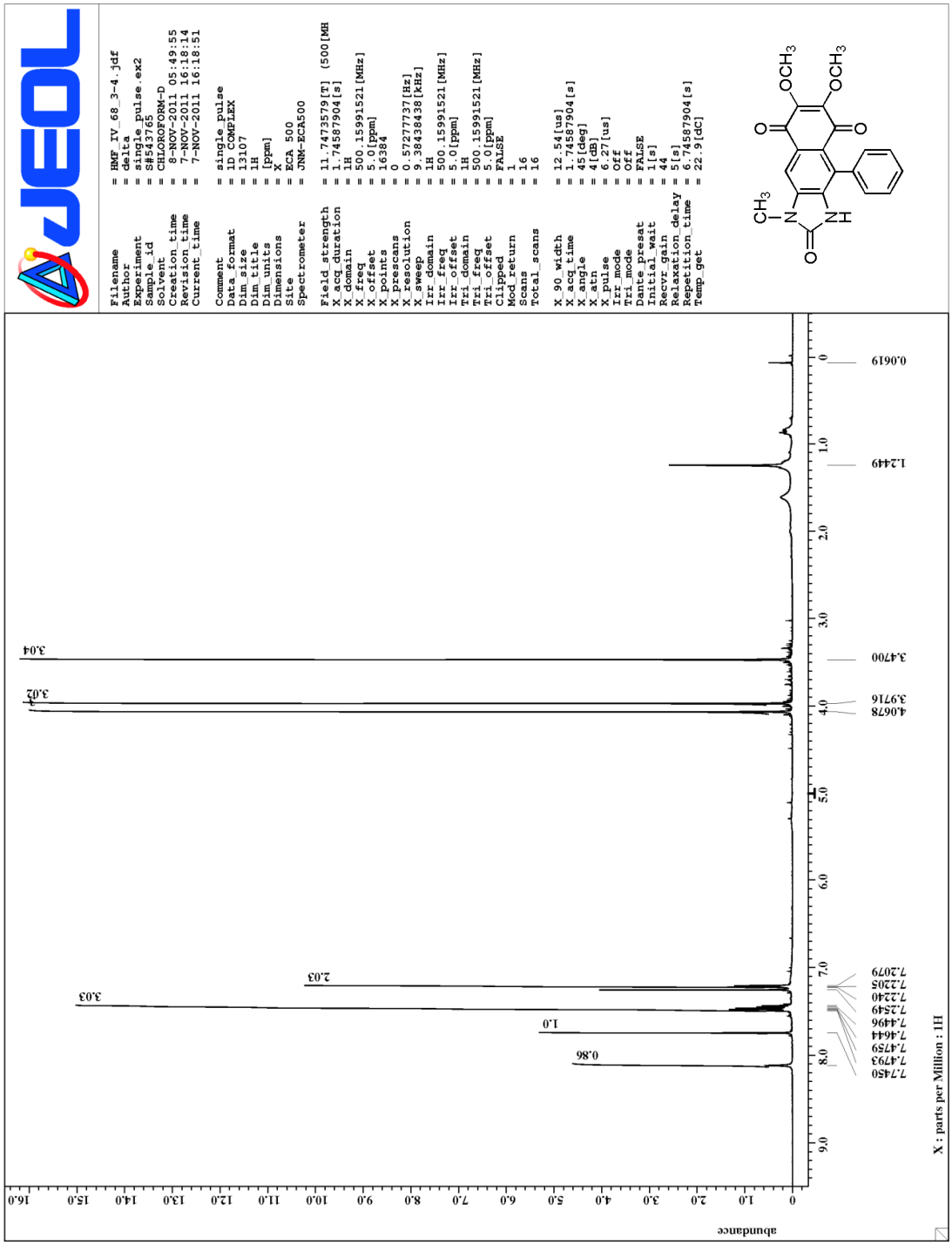
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

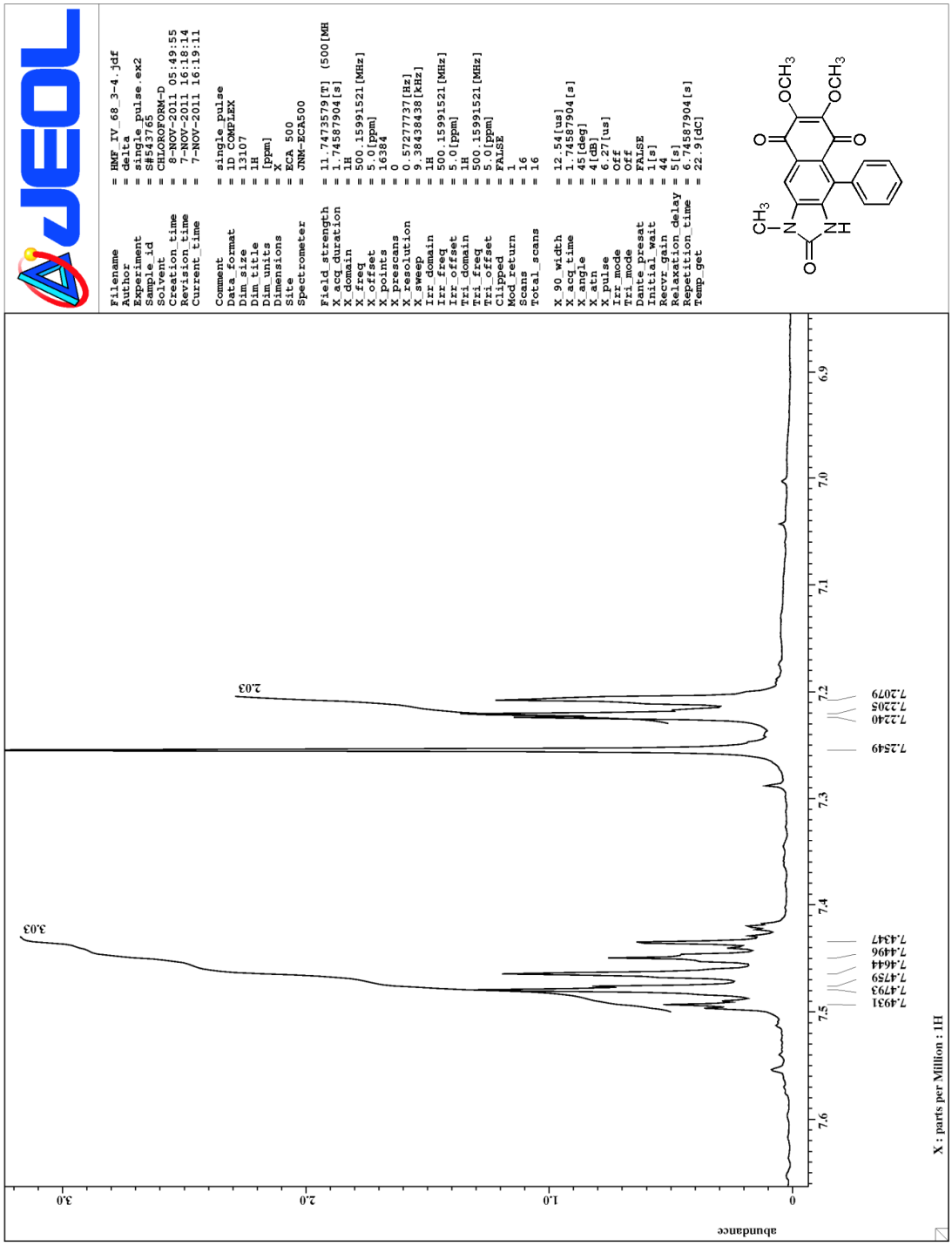
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 480
Total_scans       = 480

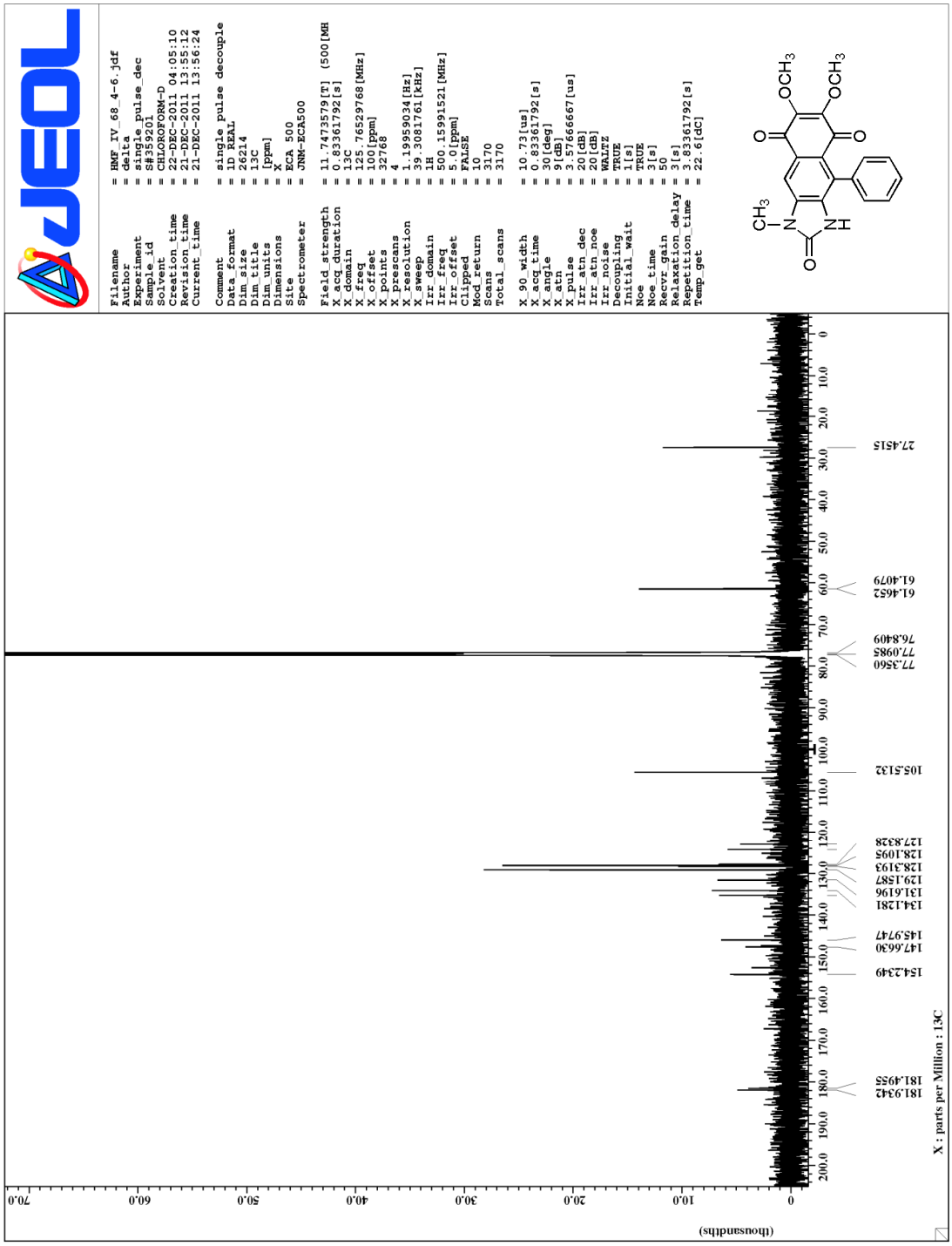
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.83361792[s]
Temp_get          = 22.8[dC]
```

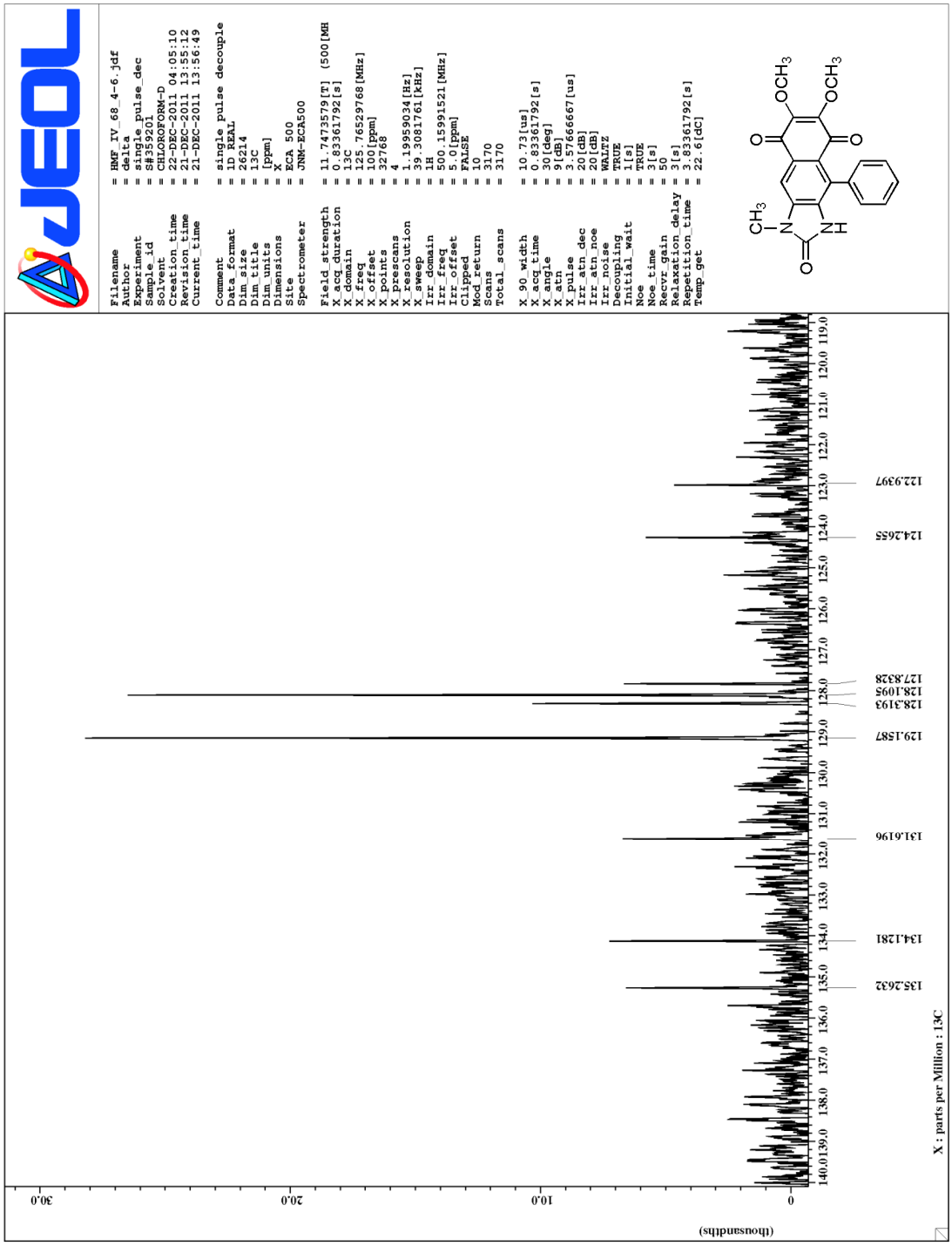


APPENDIX 56
^{1}H and ^{13}C NMR Spectra of
6,7-dimethoxy-1-methyl-4-phenyl-1*H*-naphtho[2,3-*d*]imidazole-2,5,8-trione (**140**)

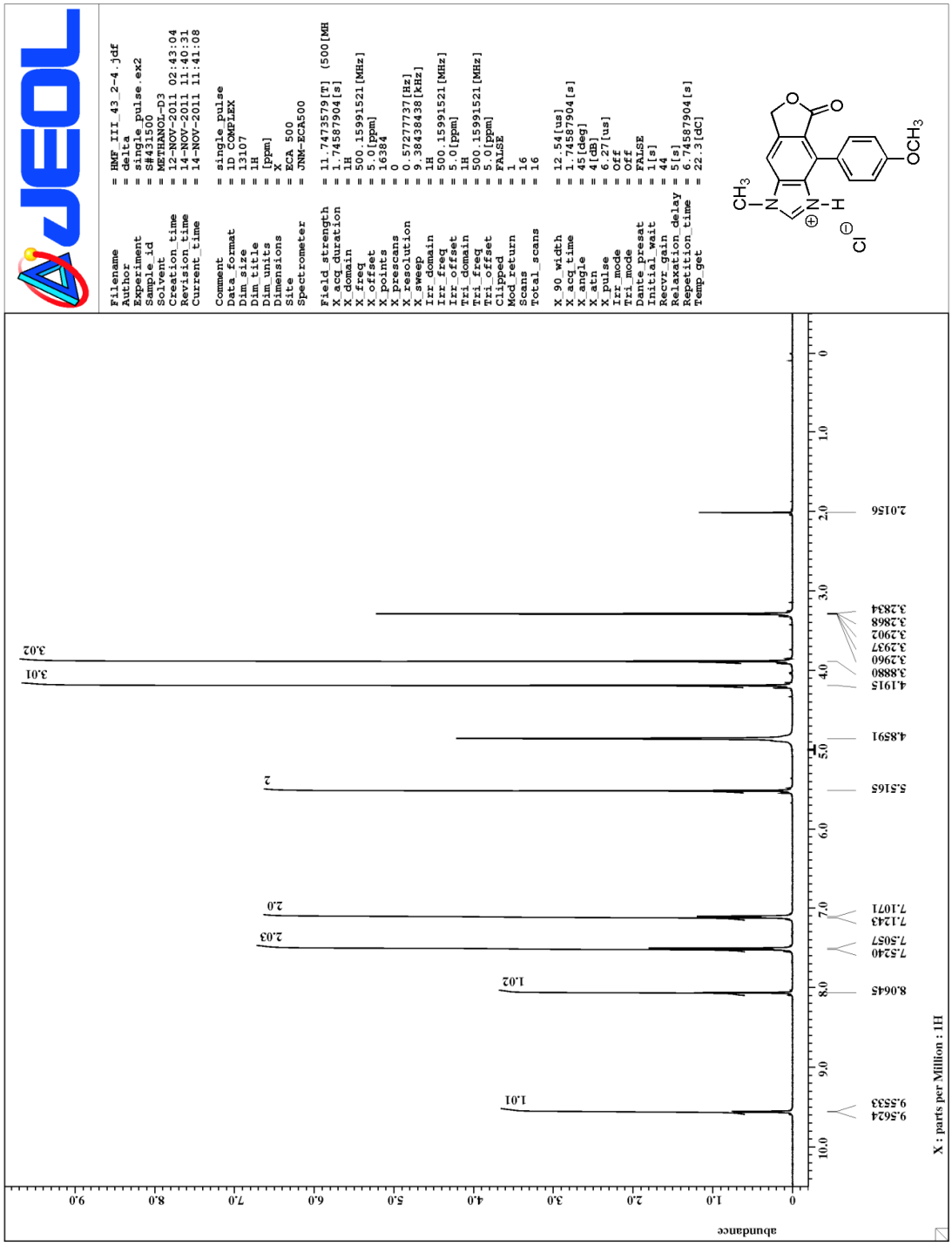

Filename = HMF_IV_68_3-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#543765
Solvent = CHLOROFORM-D
Creation_time = 8-NOV-2011 05:49:55
Revision_time = 7-NOV-2011 16:18:14
Current_time = 7-NOV-2011 16:18:51
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.9[dC]
OCH3
OCH3
CH3
N
NH
O
O
O
O
abundance
X : parts per Million : 1H
3.04
3.02
3.03
2.03
1.0
0.86
0.0619
1.2449
3.4700
3.9716
4.0678
7.2079
7.2205
7.2240
7.2549
7.4496
7.4644
7.4759
7.4793
7.7450

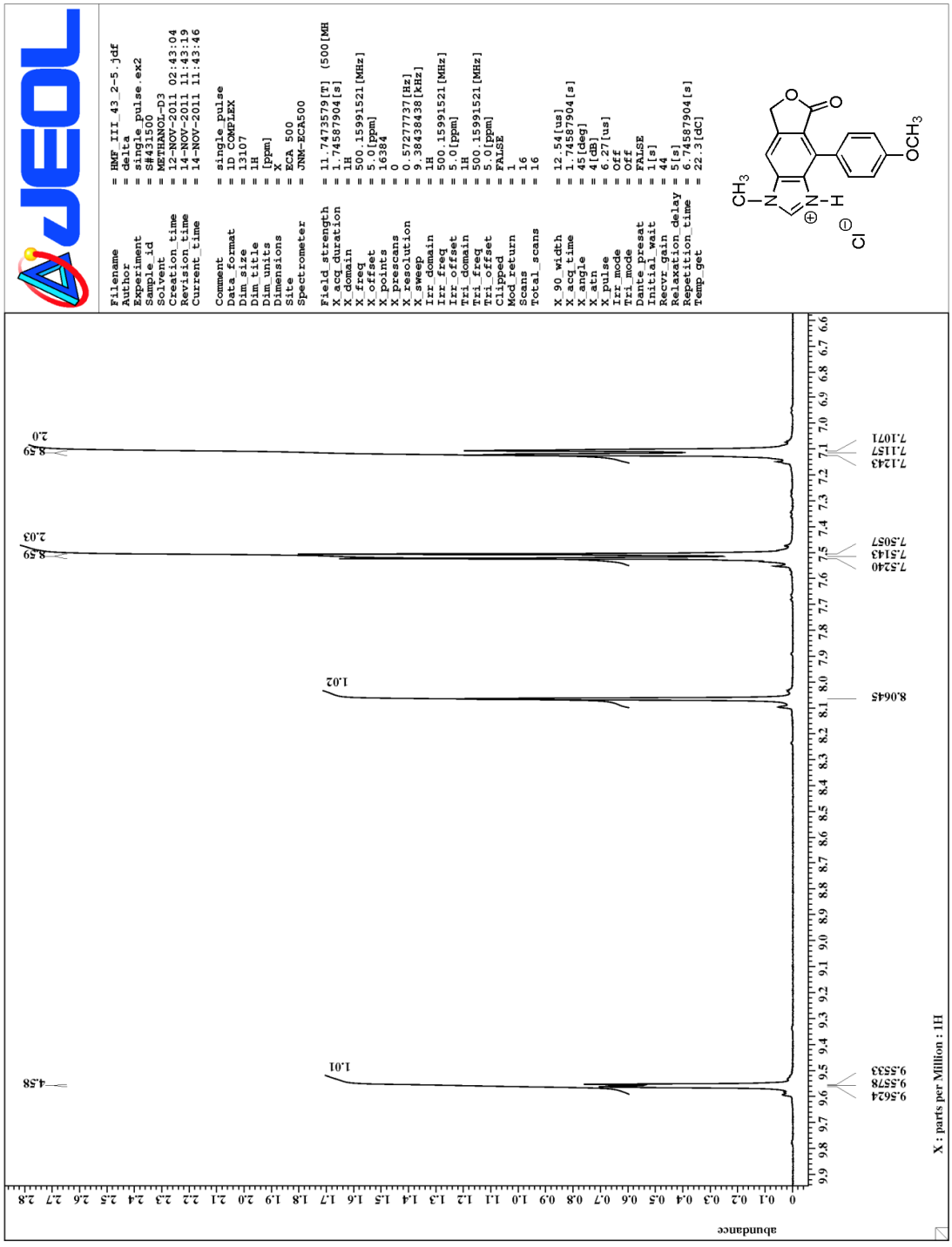

Filename = HMF_IV_68_3-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#543765
Solvent = CHLOROFORM-D
Creation_time = 8-NOV-2011 05:49:55
Revision_time = 7-NOV-2011 16:18:14
Current_time = 7-NOV-2011 16:19:11
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.9[dC]
OCH3
OCH3
CH3
N
H
N
O
O
O
abundance
3.03
2.03
7.4931
7.4793
7.4759
7.4644
7.4496
7.4347
7.2549
7.2240
7.2205
7.2079
X : parts per Million : 1H

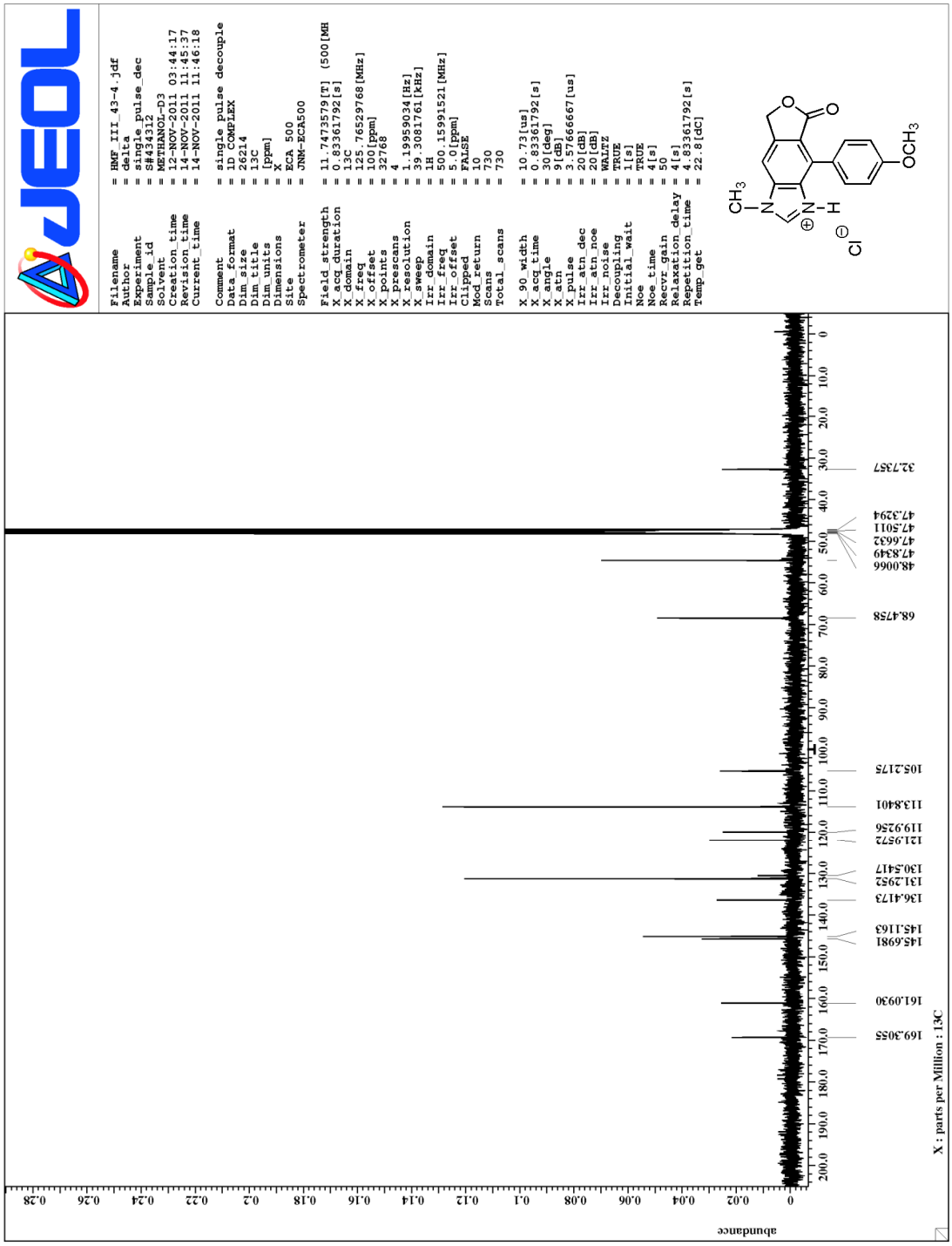

Filename = HMF_IV_68_4-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#359201
Solvent = CHLOROFORM-D
Creation_time = 22-DEC-2011 04:05:10
Revision_time = 21-DEC-2011 13:55:12
Current_time = 21-DEC-2011 13:56:24
Comment = single_pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 3170
Total_scans = 3170
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.6[dC]
OCH3
OCH3
CH3
N
H
O
O
O
(thousandths)
X : parts per Million : 13C
181.9342
181.4955
154.2349
147.6630
145.9747
134.1281
131.6196
129.1587
128.3193
128.1095
127.8328
105.5132
77.3560
77.0985
76.8409
61.4652
61.4079
27.4515


JEOL
Filename = RMF_IV_68_4-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#359201
Solvent = CHLOROFORM-D
Creation_time = 22-DEC-2011 04:05:10
Revision_time = 21-DEC-2011 13:55:12
Current_time = 21-DEC-2011 13:56:49
Comment = single_pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 3170
Total_scans = 3170
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.6[dC]
OCH3
OCH3
CH3
N
NH
O
O
O
(thousandths)
30.0
20.0
10.0
0
140.0 139.0 138.0 137.0 136.0 135.0 134.0 133.0 132.0 131.0 130.0 129.0 128.0 127.0 126.0 125.0 124.0 123.0 122.0 121.0 120.0 119.0
122.9397
124.6555
127.8328
128.1095
128.3193
129.1587
131.6196
134.1281
135.2632
X : parts per Million : 13C

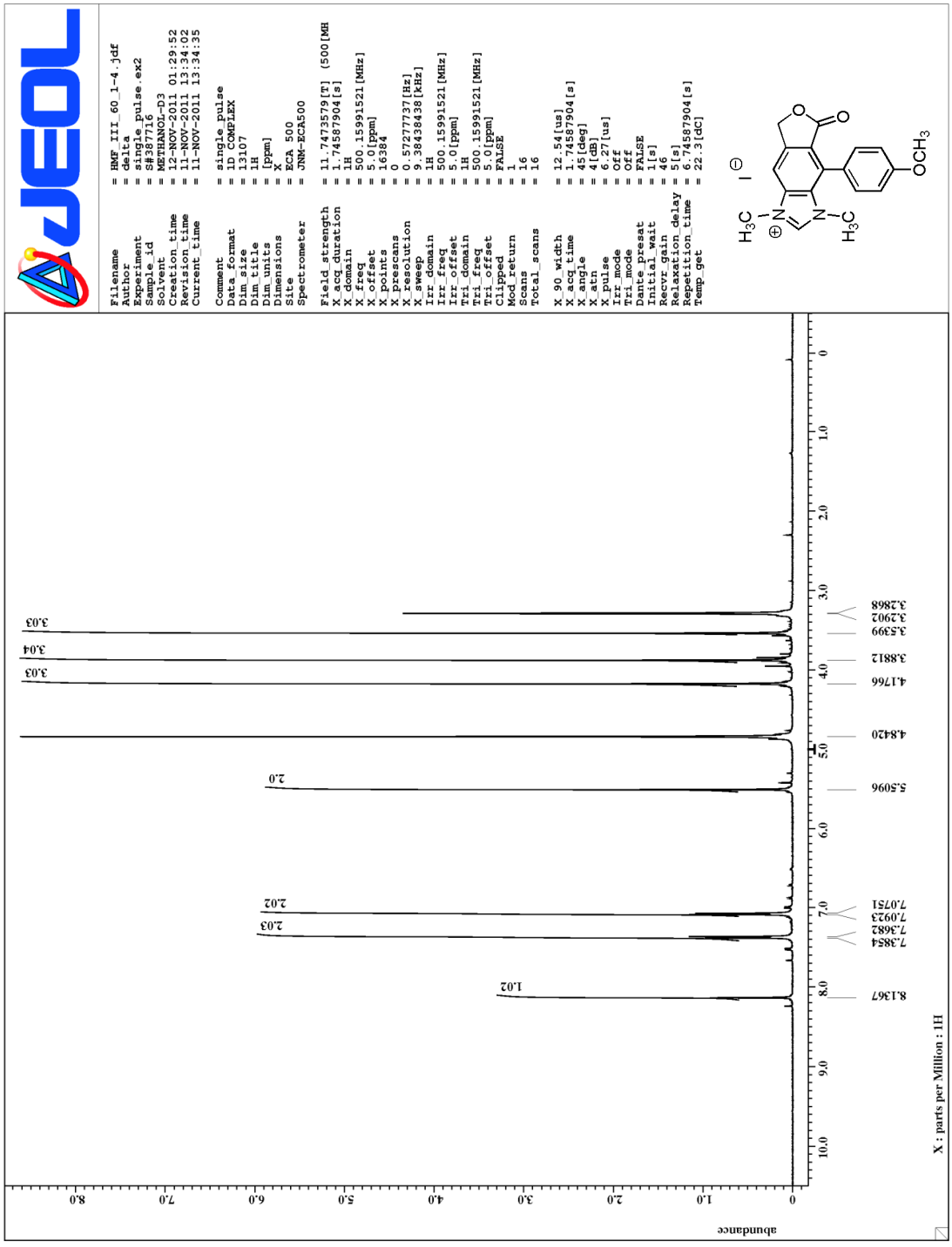
APPENDIX 57
^{1}H and ^{13}C NMR Spectra of
4-(4-methoxyphenyl)-1-methyl-5-oxo-5,7-dihydro-1*H*-furo[3,4-*f*]
[3,1]benzimidazol-3-ium chloride (**142**)

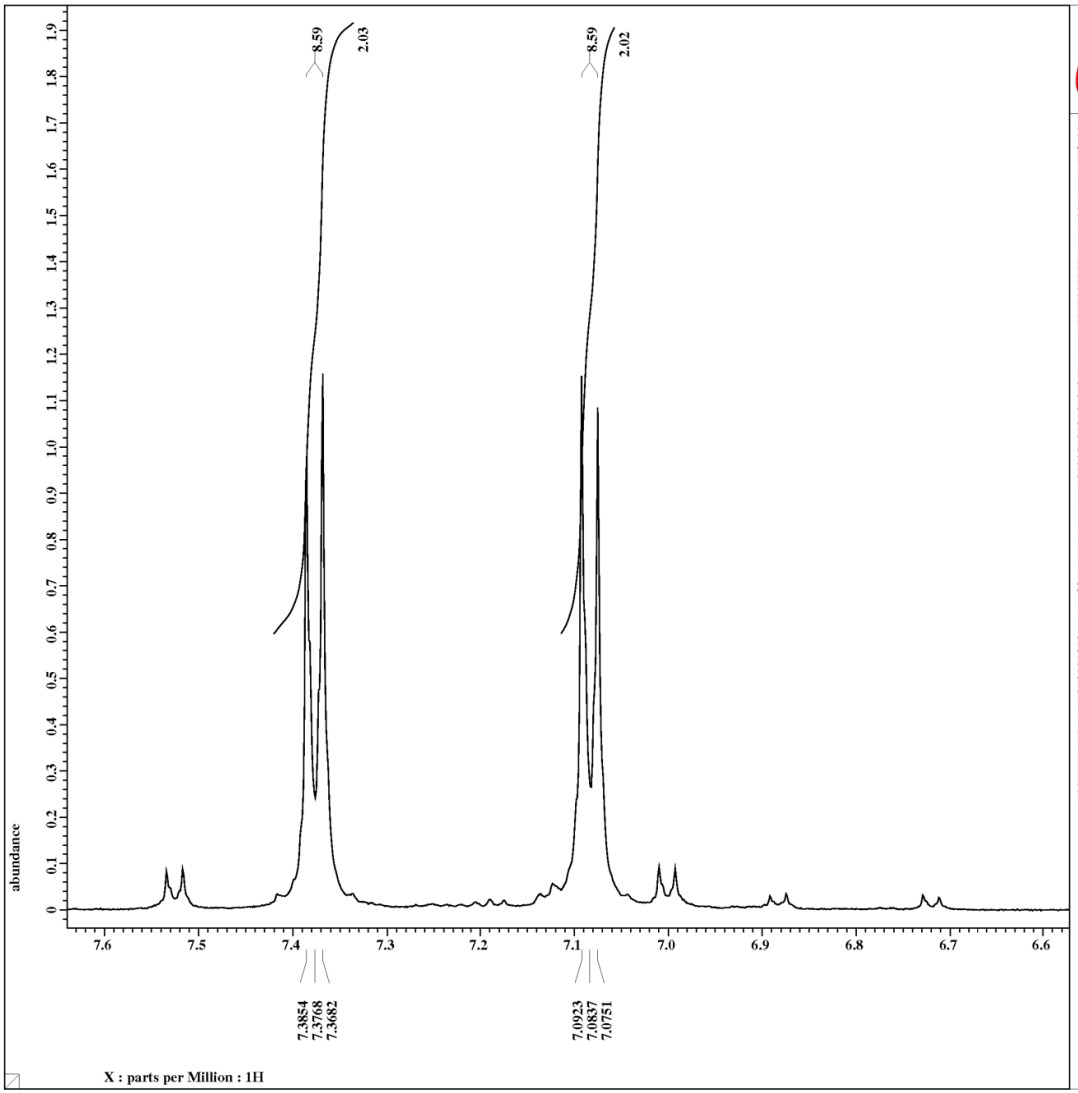

Filename = HMF_III_43_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#431500
Solvent = METHANOL-D3
Creation_time = 12-NOV-2011 02:43:04
Revision_time = 14-NOV-2011 11:40:31
Current_time = 14-NOV-2011 11:41:08
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.3[dC]
CH3
OCH3
Cl
abundance
X : parts per Million : 1H
9.5624
9.5533
8.0645
7.5240
7.5057
7.1243
7.1071
5.5165
4.8591
4.1915
3.8880
3.2960
3.2937
3.2902
3.2868
3.2834
2.0156
1.01
1.02
2.03
2.0
2
3.01
3.02


Filename = HMF_III_43_2-5.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#431500
Solvent = METHANOL-D3
Creation_time = 12-NOV-2011 02:43:04
Revision_time = 14-NOV-2011 11:43:19
Current_time = 14-NOV-2011 11:43:46
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.3[dC]
CH3
OCH3
Cl
abundance
X : parts per Million : 1H
4.58
1.01
1.02
2.03
2.00
8.59
8.59
9.5624
9.5578
9.5533
8.0645
7.5240
7.5143
7.5057
7.1243
7.1157
7.1071


Filename = HMF_III_43-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#434312
Solvent = METHANOL-D3
Creation_time = 12-NOV-2011 03:44:17
Revision_time = 14-NOV-2011 11:45:37
Current_time = 14-NOV-2011 11:46:18
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.30817611kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 730
Total_scans = 730
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.8[dC]
32.7357
47.3294
47.5011
47.6632
47.8349
48.0066
68.4758
105.2175
113.8401
119.9256
121.9572
130.5417
131.2952
136.4173
145.1163
145.6981
161.0930
169.3055
abundance
X : parts per Million : 13C

APPENDIX 58
^{1}H and ^{13}C NMR Spectra of
4-(4-methoxyphenyl)-1,3-dimethyl-5-oxo-5,7-dihydro-3*H*-furo[3,4-f]
benzimidazol-1-ium iodide (**144**)


Filename = HMF_III_60_1-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#387716
Solvent = METHANOL-D3
Creation_time = 12-NOV-2011 01:29:52
Revision_time = 11-NOV-2011 13:34:02
Current_time = 11-NOV-2011 13:34:35
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.3[dC]
abundance
X : parts per Million : 1H
3.03
3.04
3.03
2.0
2.03
2.02
1.02
8.1367
7.3854
7.3682
7.0923
7.0751
5.5096
4.8420
4.1766
3.8812
3.5399
3.2902
3.2868



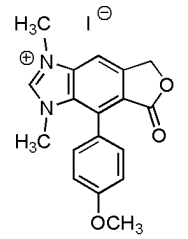
JEOL

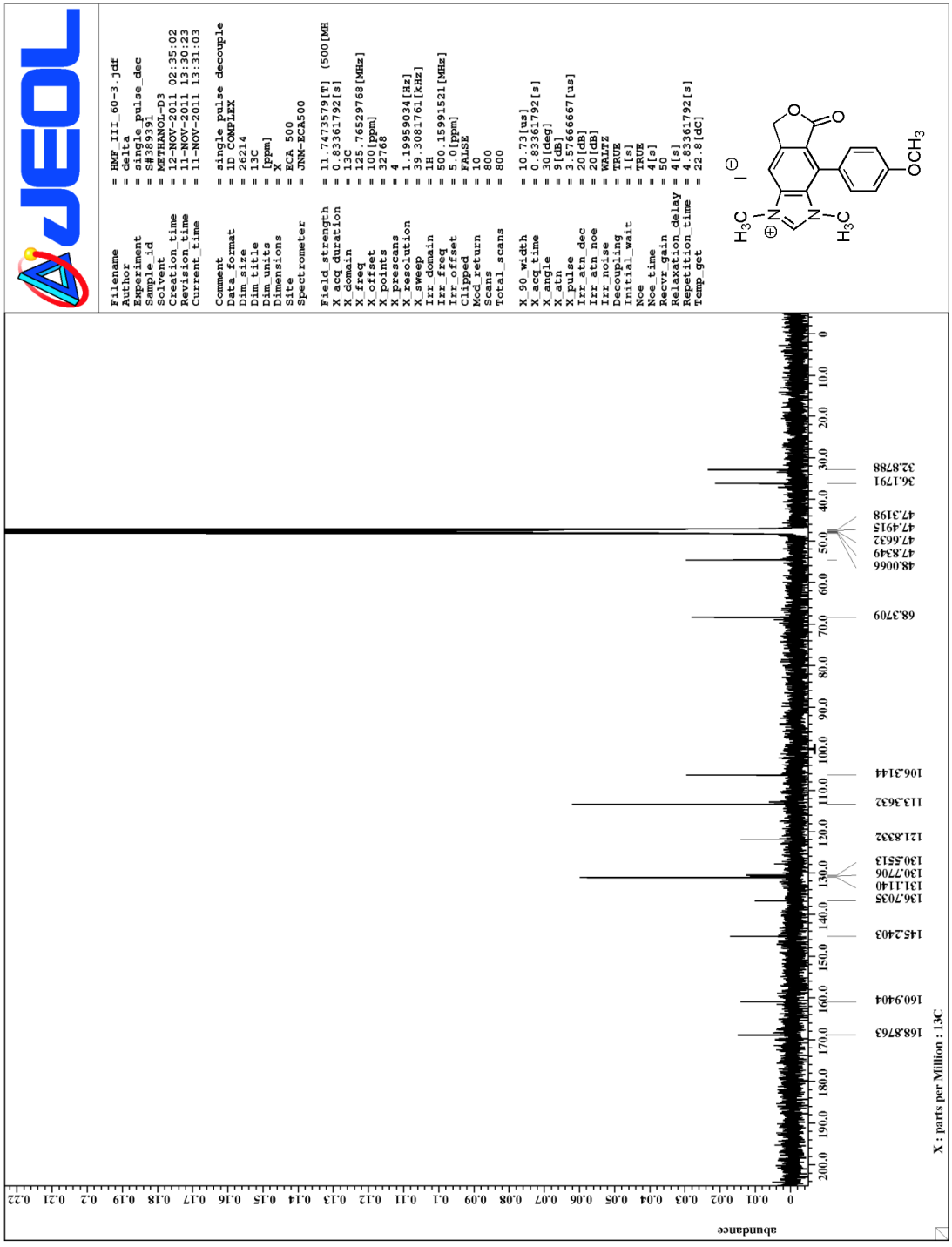
```
Filename          = HMF_III_60_1-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#387716
Solvent           = METHANOL-D3
Creation_time     = 12-NOV-2011 01:29:52
Revision_time     = 11-NOV-2011 13:35:00
Current_time      = 11-NOV-2011 13:35:14

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

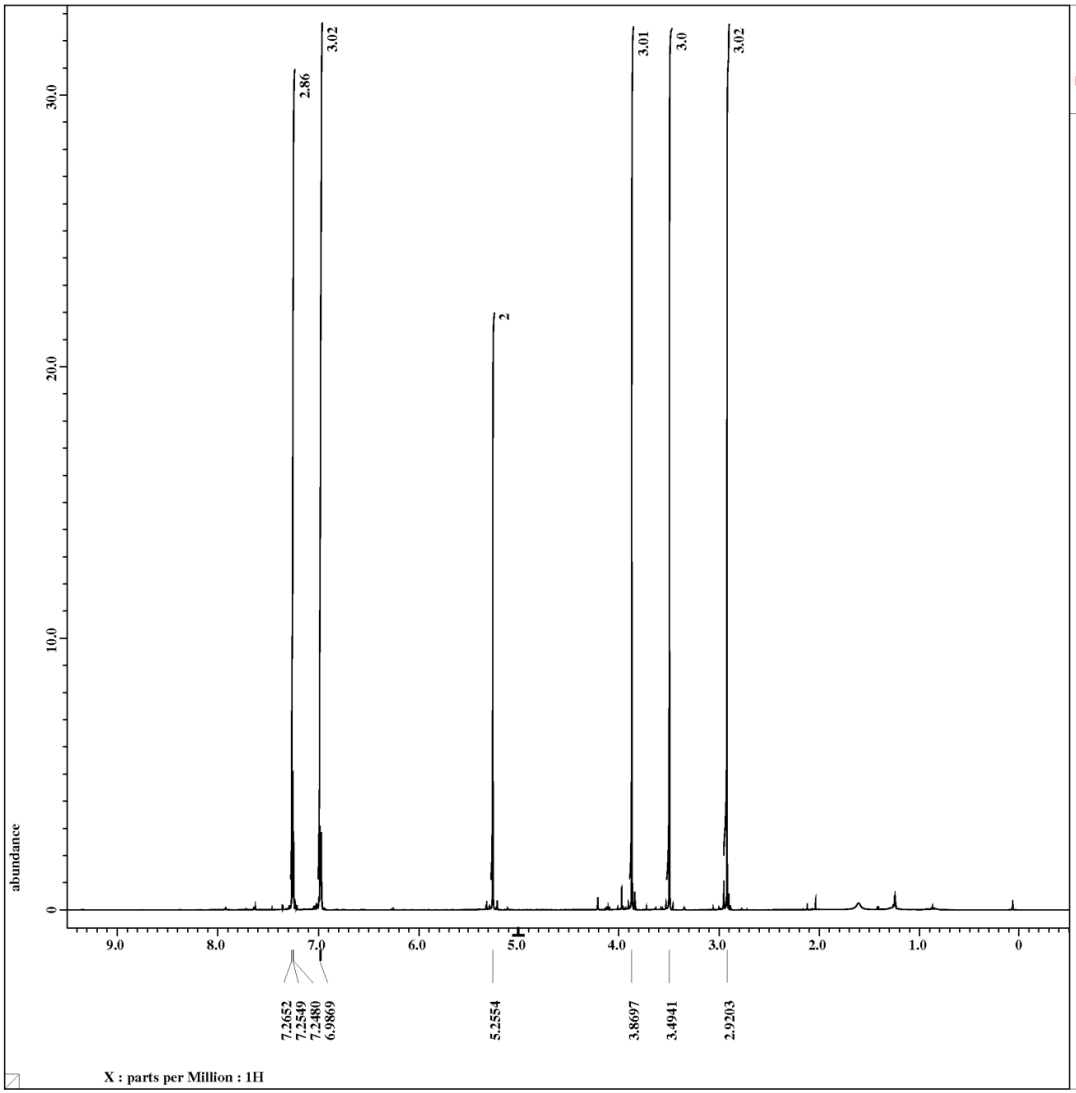
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.3[dC]
```




Filename = HMF_III_60-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#389391
Solvent = METHANOL-D3
Creation_time = 12-NOV-2011 02:35:02
Revision_time = 11-NOV-2011 13:30:23
Current_time = 11-NOV-2011 13:31:03
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 800
Total_scans = 800
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.8[dC]
abundance
X : parts per Million : 13C
32.8788
36.1791
47.3198
47.4915
47.6632
47.8349
48.0066
68.3709
106.3144
113.3632
121.8332
130.5513
130.7706
131.1140
136.7035
145.2403
160.9404
168.8763

APPENDIX 59
^{1}H and ^{13}C NMR Spectra of
4-(4-Methoxyphenyl)-1,3-dimethyl-3,7-dihydro-1*H*-furo[3,4-*f*]benzimidazole-2,5-dione (**145**)



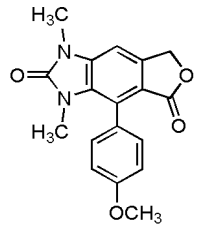
JEOL

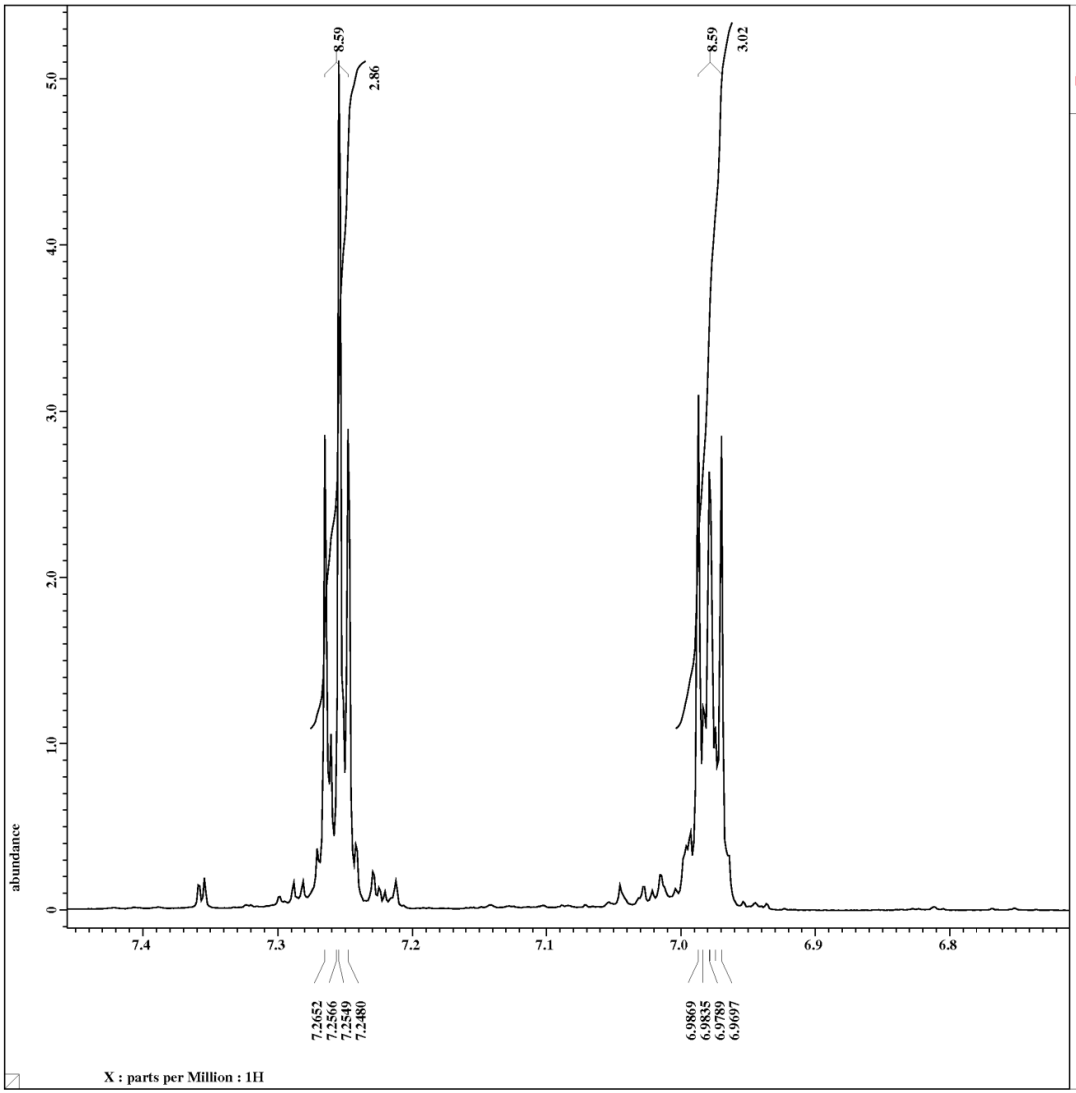
```
Filename          = HMF_IV_64_3-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#730304
Solvent           = CHLOROFORM-D
Creation_time     = 18-NOV-2011 11:01:07
Revision_time     = 17-NOV-2011 21:17:23
Current_time      = 17-NOV-2011 21:17:56

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.3[dC]
```





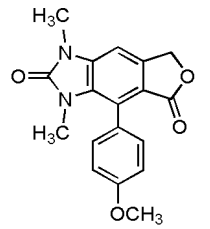
JEOL

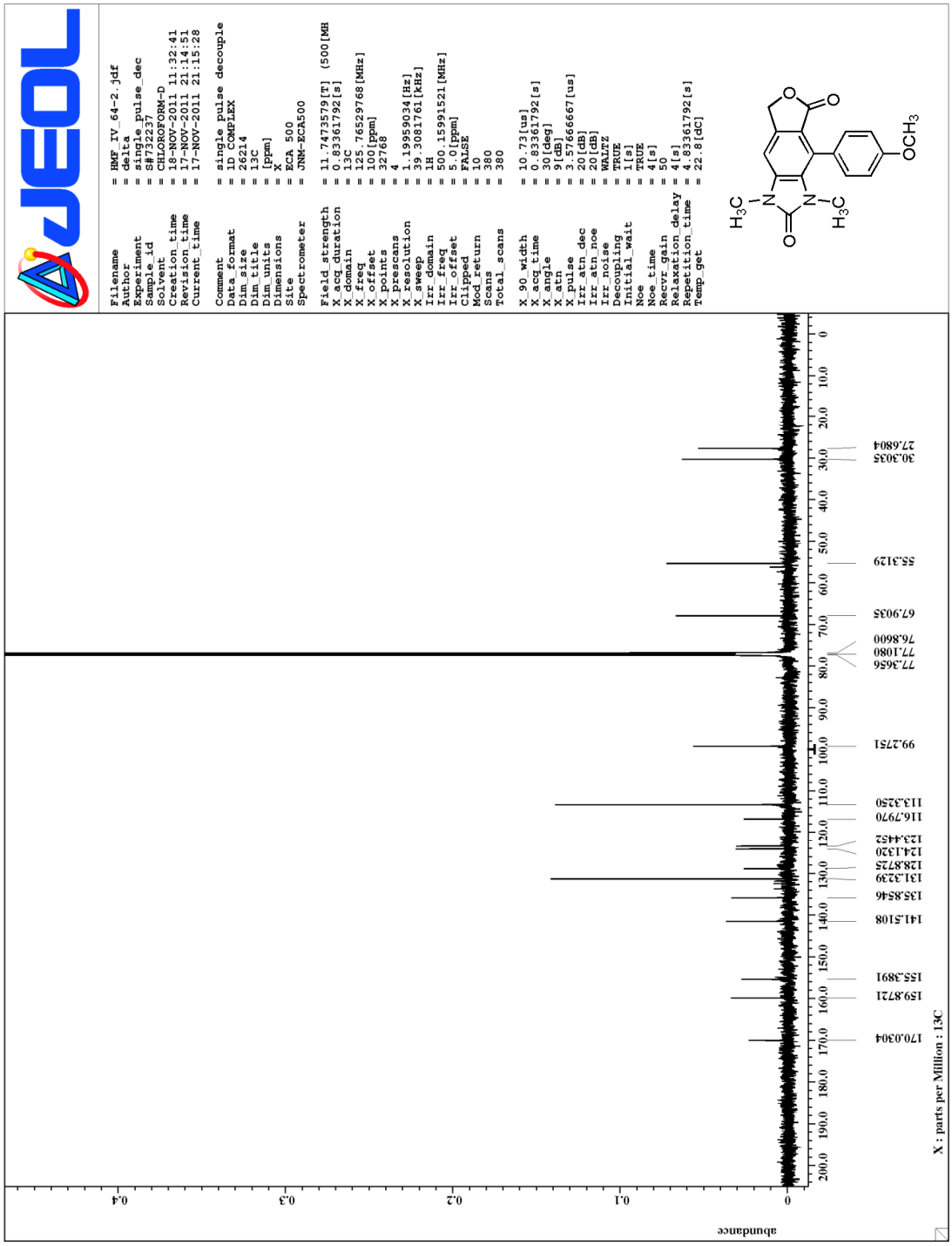
```
Filename          = HMF_IV_64_3-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#730304
Solvent           = CHLOROFORM-D
Creation_time     = 18-NOV-2011 11:01:07
Revision_time     = 17-NOV-2011 21:18:17
Current_time      = 17-NOV-2011 21:18:36

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

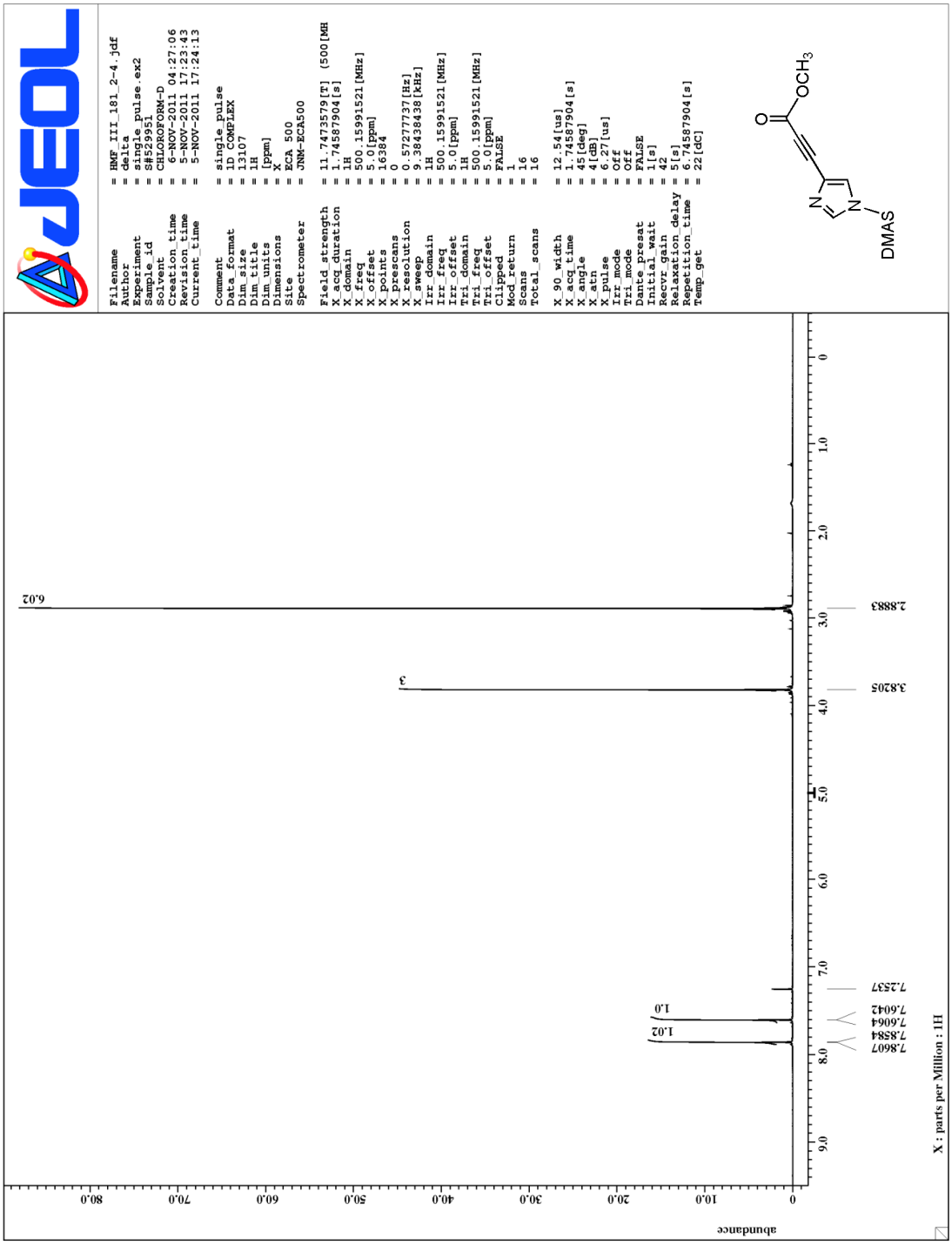
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

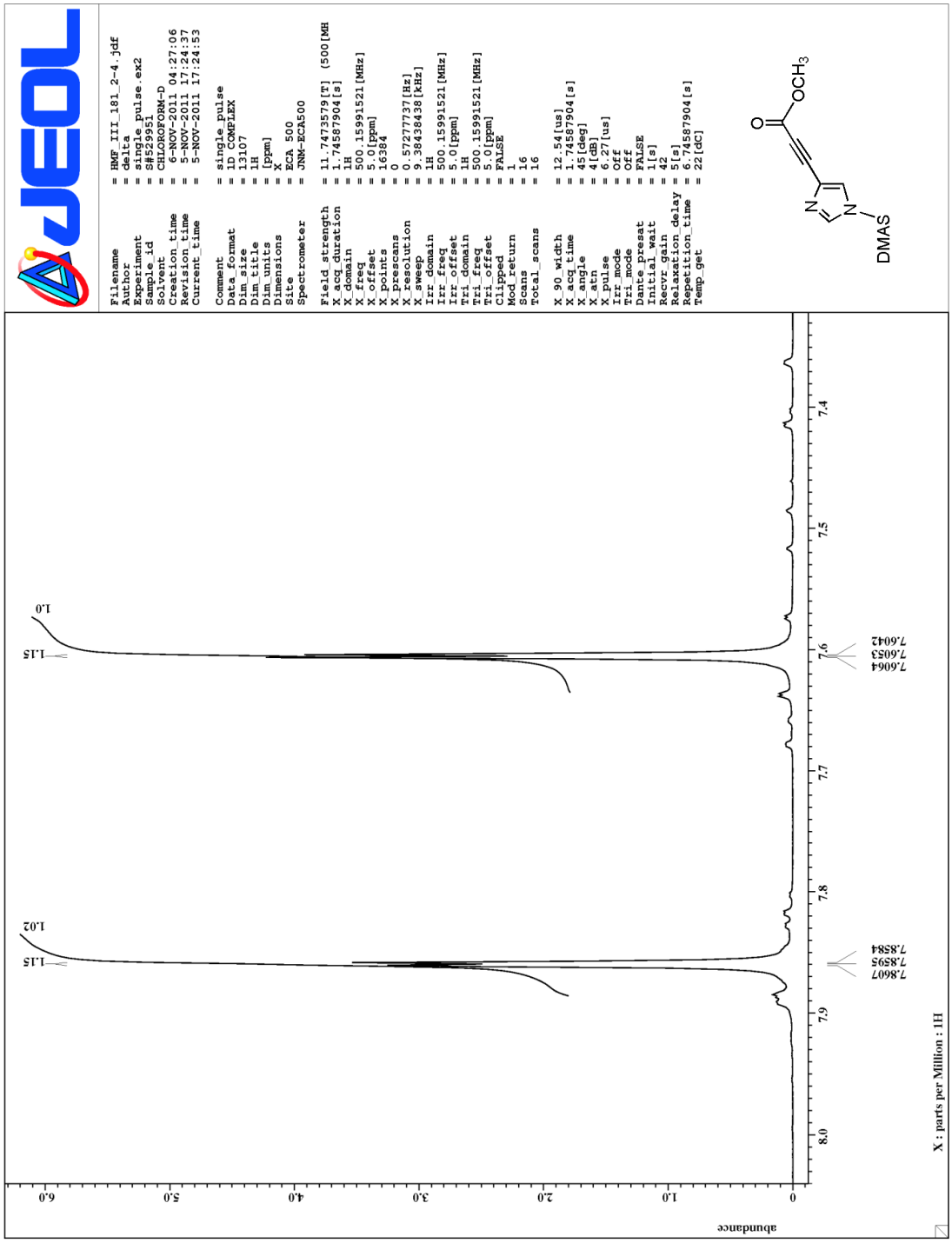
X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.3[dC]
```

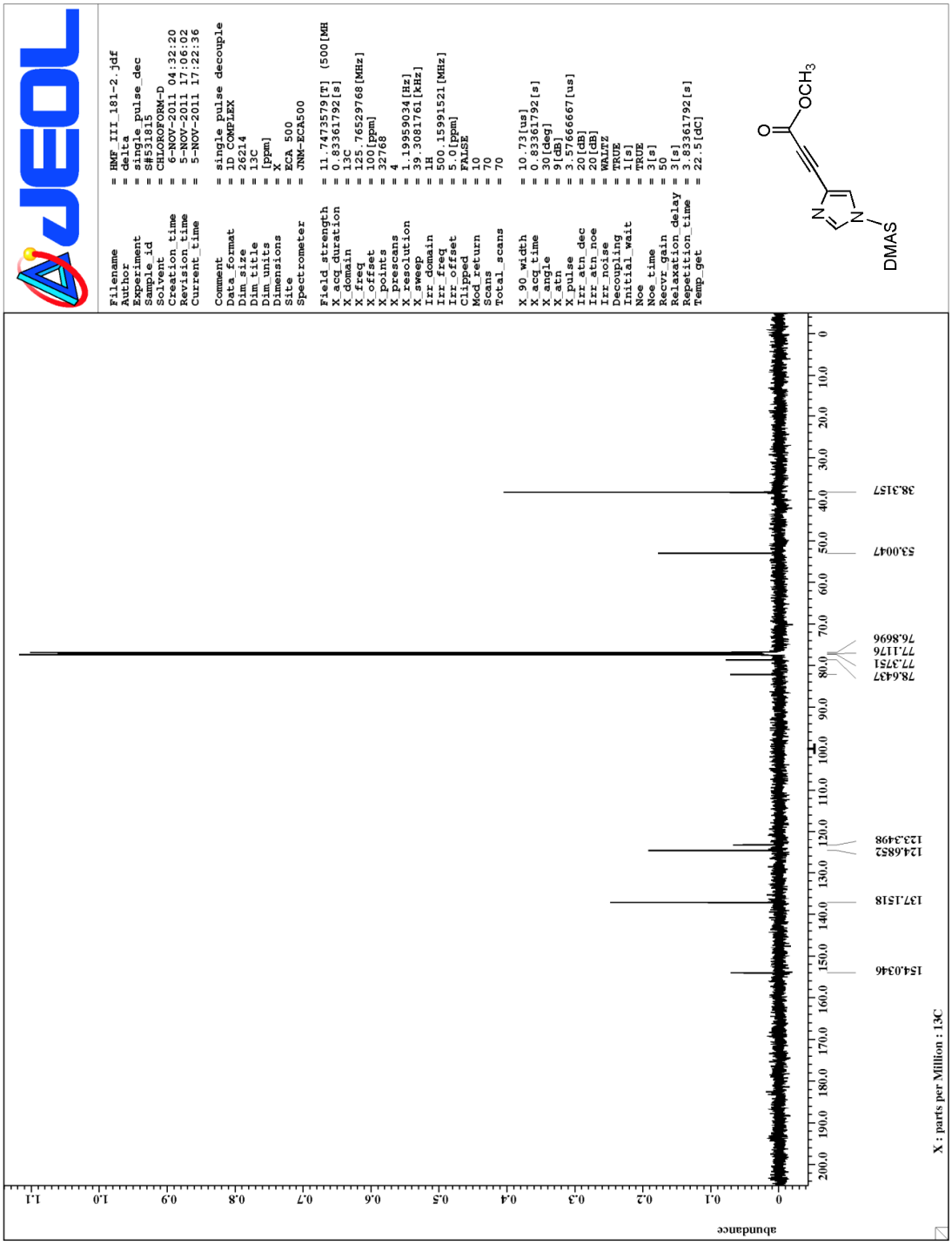



Filename = HMF_IV_64-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#7322237
Solvent = CHLOROFORM-D
Creation_time = 18-NOV-2011 11:32:41
Revision_time = 17-NOV-2011 21:14:51
Current_time = 17-NOV-2011 21:15:28
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159915211MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 380
Total_scans = 380
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 4[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 4.83361792[s]
Temp_get = 22.8[dC]
abundance
X : parts per Million : 13C
170.0304
159.8721
155.3891
141.5108
135.8546
131.3239
128.8725
124.1320
123.4452
116.7970
113.3250
99.2751
77.3656
77.1080
76.8600
67.9035
55.3129
30.3035
27.6804

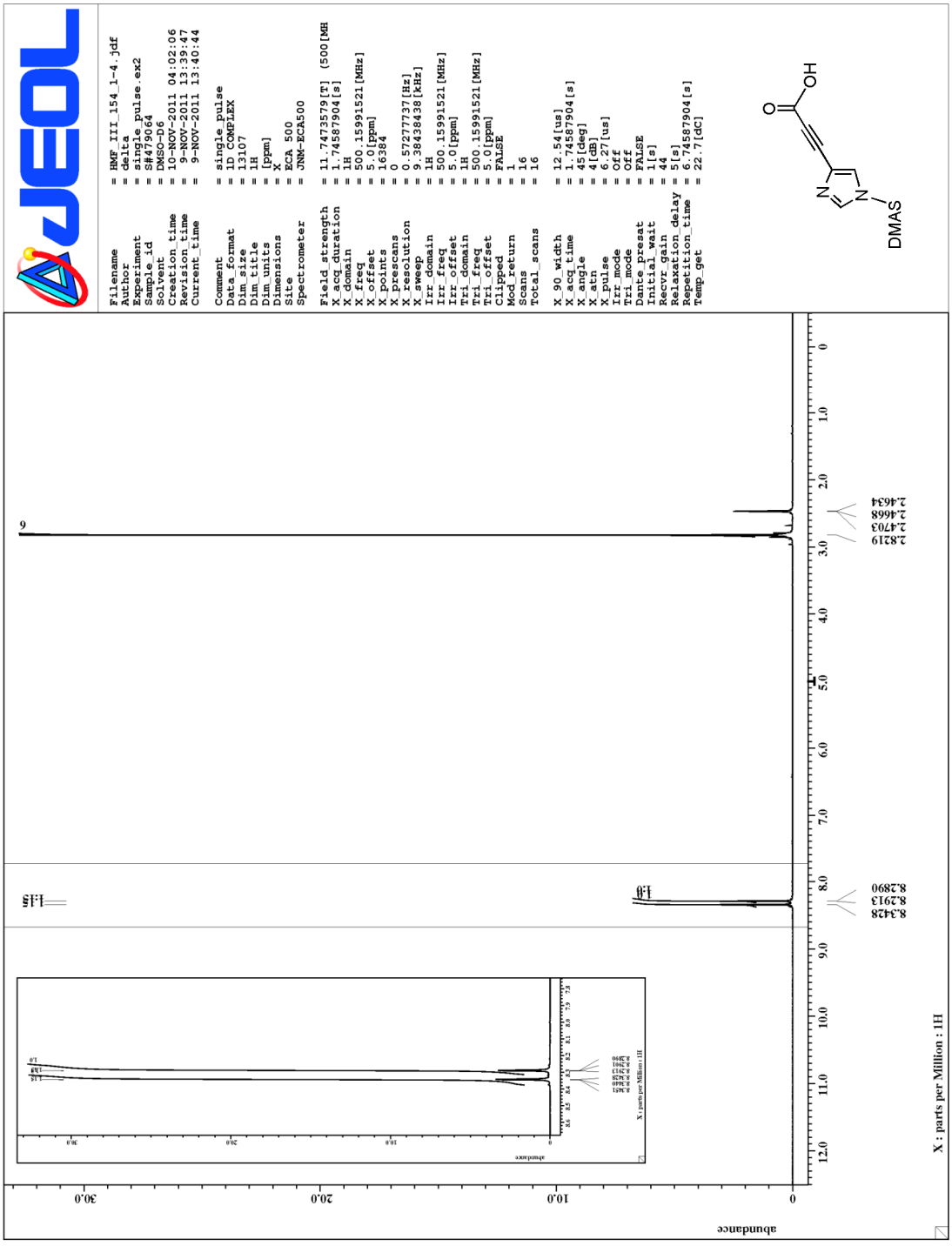
APPENDIX 60
^{1}H and ^{13}C NMR Spectra of
Methyl 3-[1-(*N*,*N*-dimethylsulfamoyl)1H-imidazol-4-yl]prop-2-ynoate (**154**)

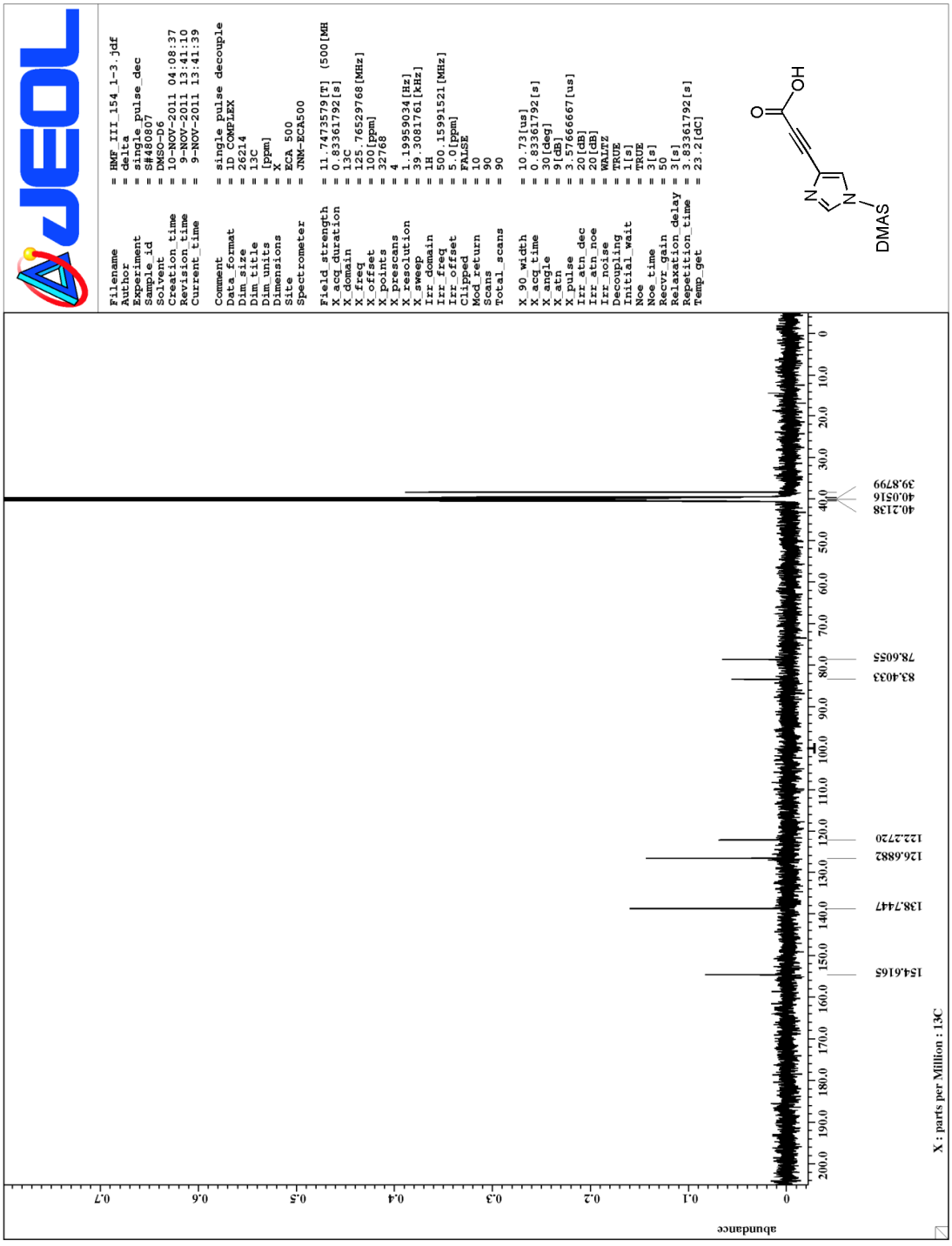

Filename = RMF_III_181_2-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#529951
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 04:27:06
Revision_time = 5-NOV-2011 17:23:43
Current_time = 5-NOV-2011 17:24:13
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22[dC]
OCH3
DMAS
6.02
3
1.02
1.0
2.8883
3.8205
7.2537
7.6042
7.6064
7.8584
7.8607
abundance
X : parts per Million : 1H


JEOL
Filename = RMF_III_181_2-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#529951
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 04:27:06
Revision_time = 5-NOV-2011 17:24:37
Current_time = 5-NOV-2011 17:24:53
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.745879041[s]
Temp_get = 22[dC]
OCH3
DMAS
abundance
X : parts per Million : 1H
1.0
1.15
1.02
1.15
1.0
7.8607
7.8595
7.8584
7.6064
7.6053
7.6042

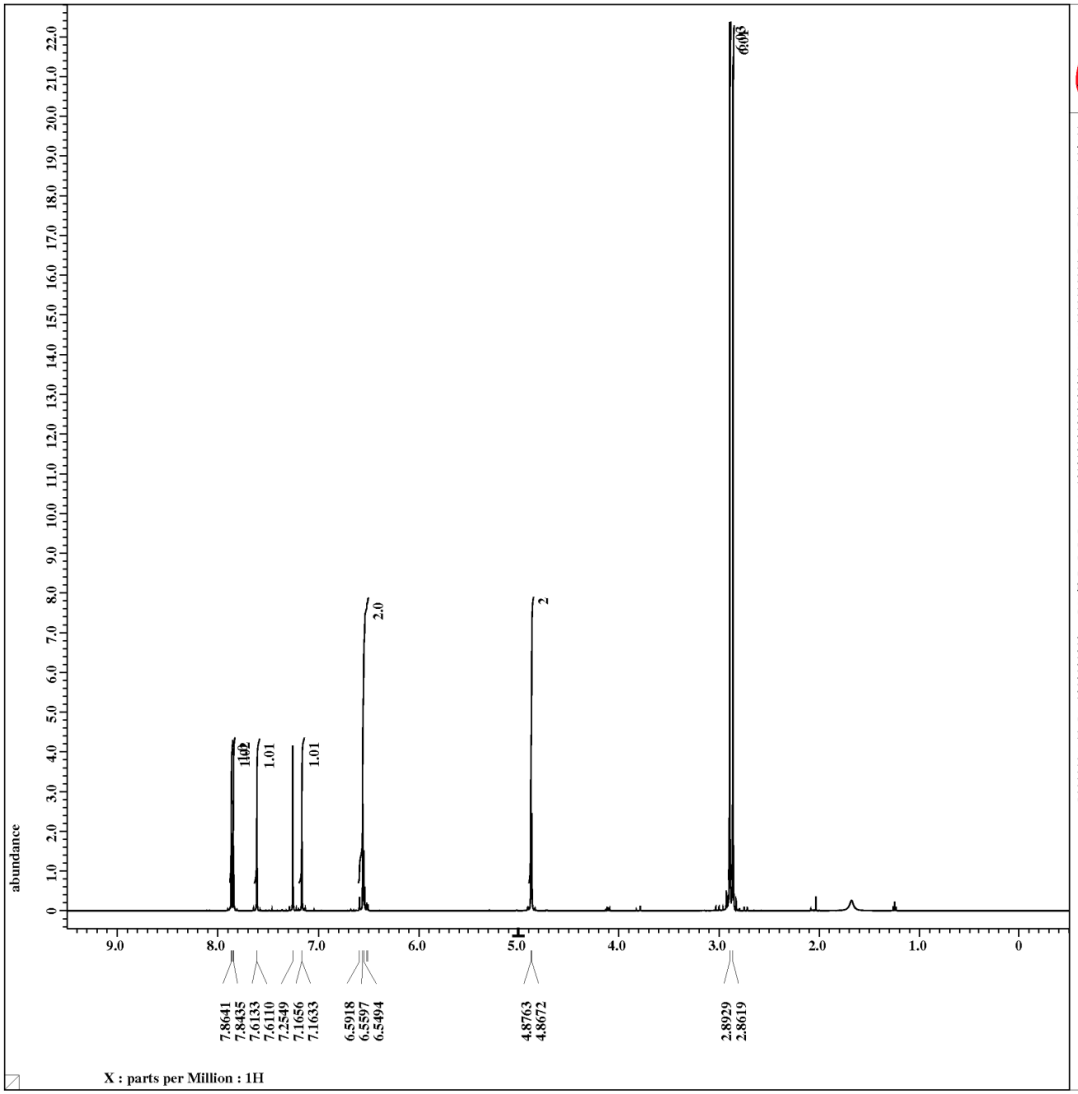

Filename = HMF III 181-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#531815
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 04:32:20
Revision_time = 5-NOV-2011 17:06:02
Current_time = 5-NOV-2011 17:22:36
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 70
Total_scans = 70
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_seqfile = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe_time = TRUE
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.5[dC]
OCH3
N
N
DMAS
38.3157
53.0047
76.8696
77.1176
77.3751
78.6437
123.3498
124.6852
137.1518
154.0346
abundance
X : parts per Million : 13C

APPENDIX 61
^{1}H and ^{13}C NMR Spectra of
3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]prop-2-ynoic acid (**155**)


Filename = RMF_III_154_1-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#479064
Solvent = DMSO-D6
Creation_time = 10-NOV-2011 04:02:06
Revision_time = 9-NOV-2011 13:39:47
Current_time = 9-NOV-2011 13:40:44
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.384384438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.745879044[s]
Temp_get = 22.7[dC]
OH
DMAS
6
1.15
1.0
8.3428
8.2913
8.2890
2.8219
2.4703
2.4668
2.4634
abundance
X : parts per Million : 1H


Filename = RMF_III_154_1-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#480807
Solvent = DMSO-D6
Creation_time = 10-NOV-2011 04:08:37
Revision_time = 9-NOV-2011 13:41:10
Current_time = 9-NOV-2011 13:41:39
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = ONM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.765297681MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 90
Total_scans = 90
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 23.2[dC]
OH
N
N
DMAS
abundance
X : parts per Million : 13C
154.6165
138.7447
126.6882
122.2720
83.4033
78.6055
40.2138
40.0516
39.8799

APPENDIX 62
^{1}H and ^{13}C NMR Spectra of
(2*E*)-3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]prop-2-en-1-yl 3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]prop-2-ynoate (**150**)



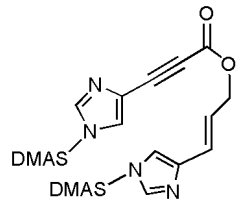
JEOL

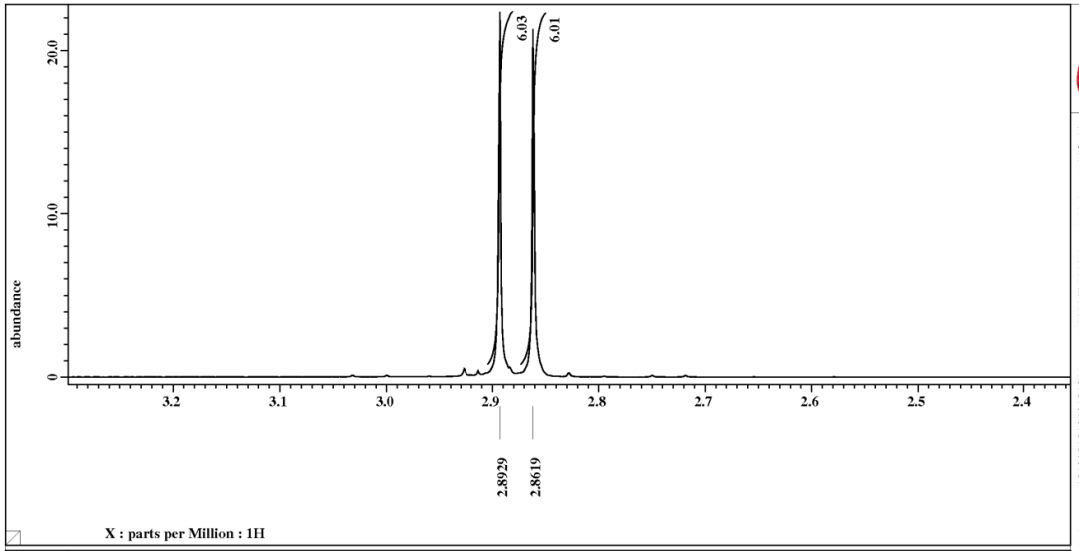
```
Filename          = HMF_III_155_2-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#537655
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2011 04:39:30
Revision_time     =  5-NOV-2011 16:21:06
Current_time      =  5-NOV-2011 16:21:37

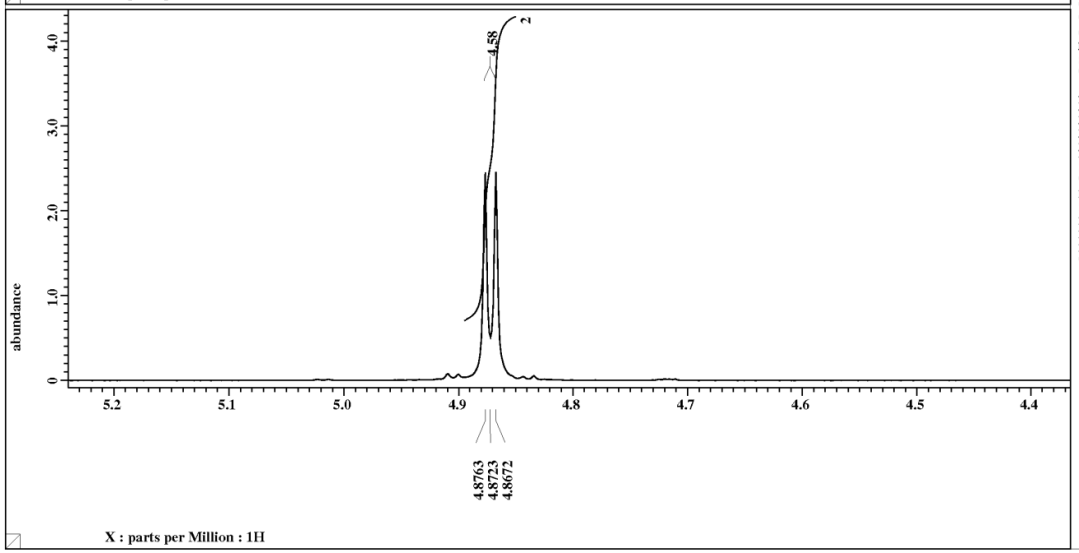
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.2[dC]
```





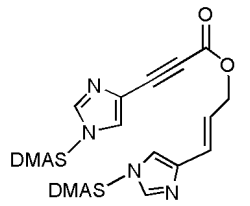


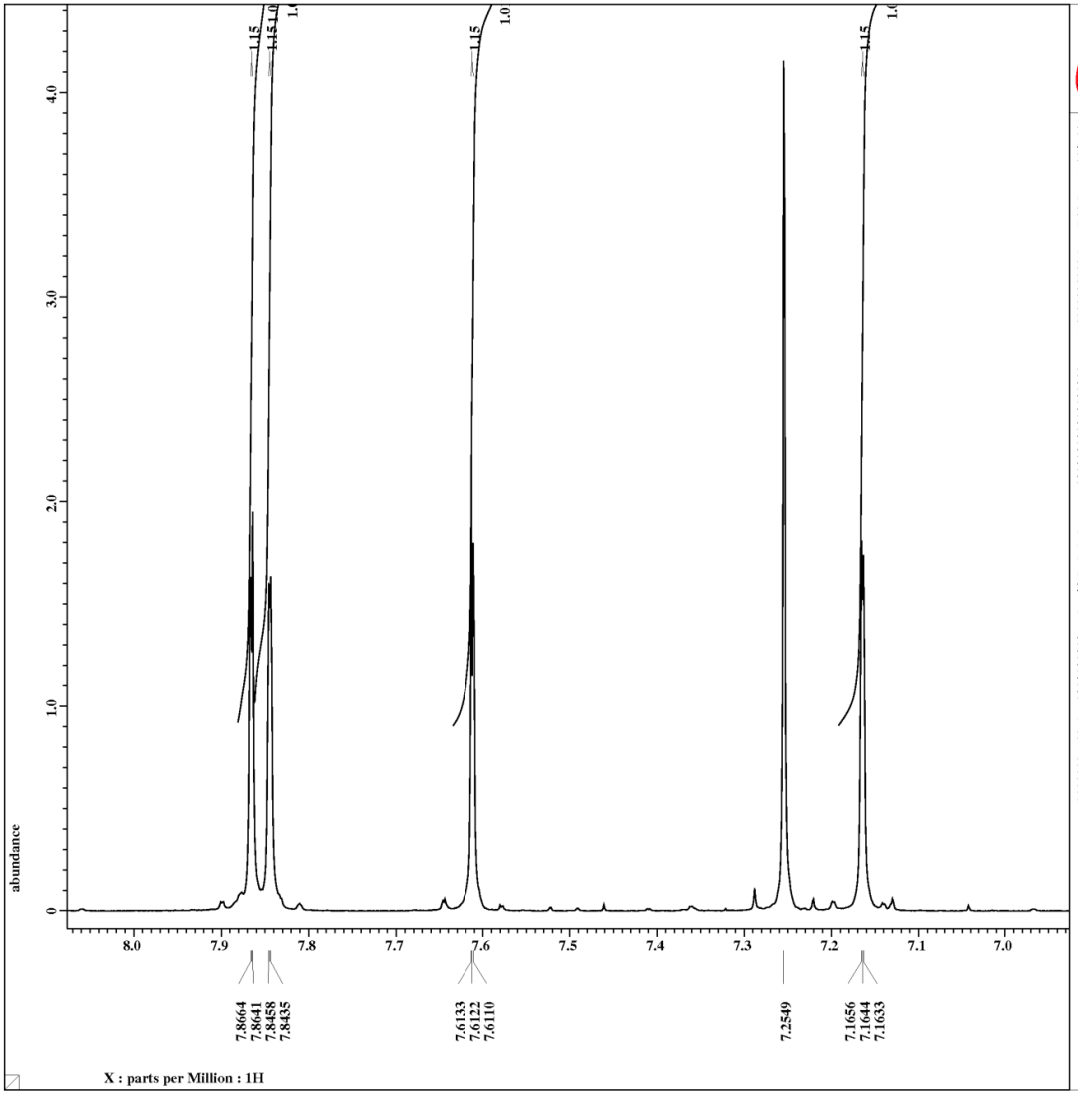

```
Filename          = HMF_III_155_2-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#537655
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2011 04:39:30
Revision_time     =  5-NOV-2011 16:21:06
Current_time      =  5-NOV-2011 16:23:14

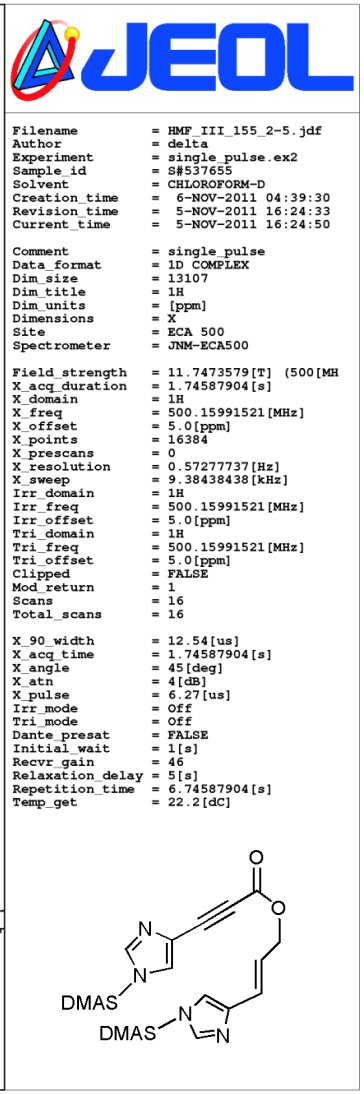
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

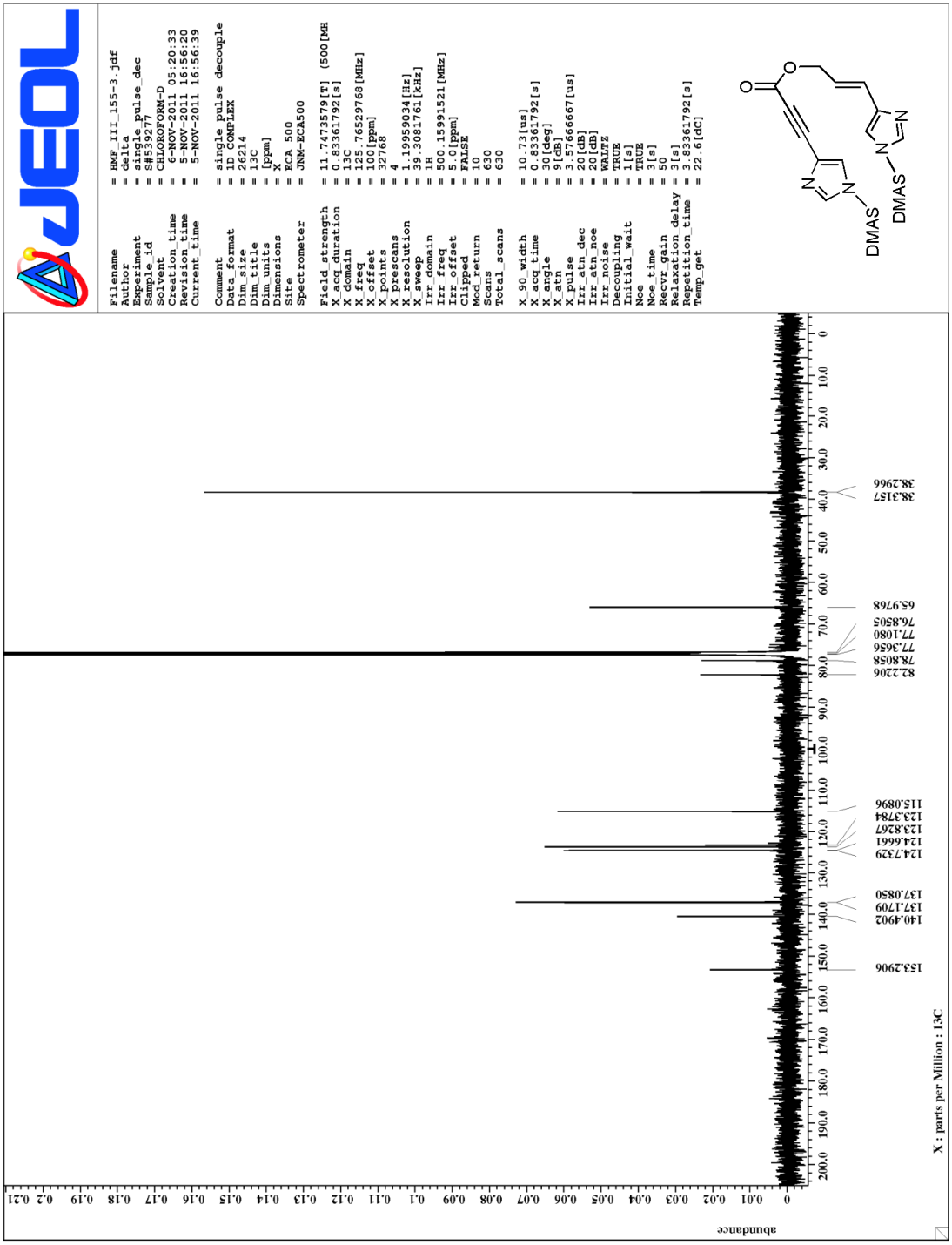
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

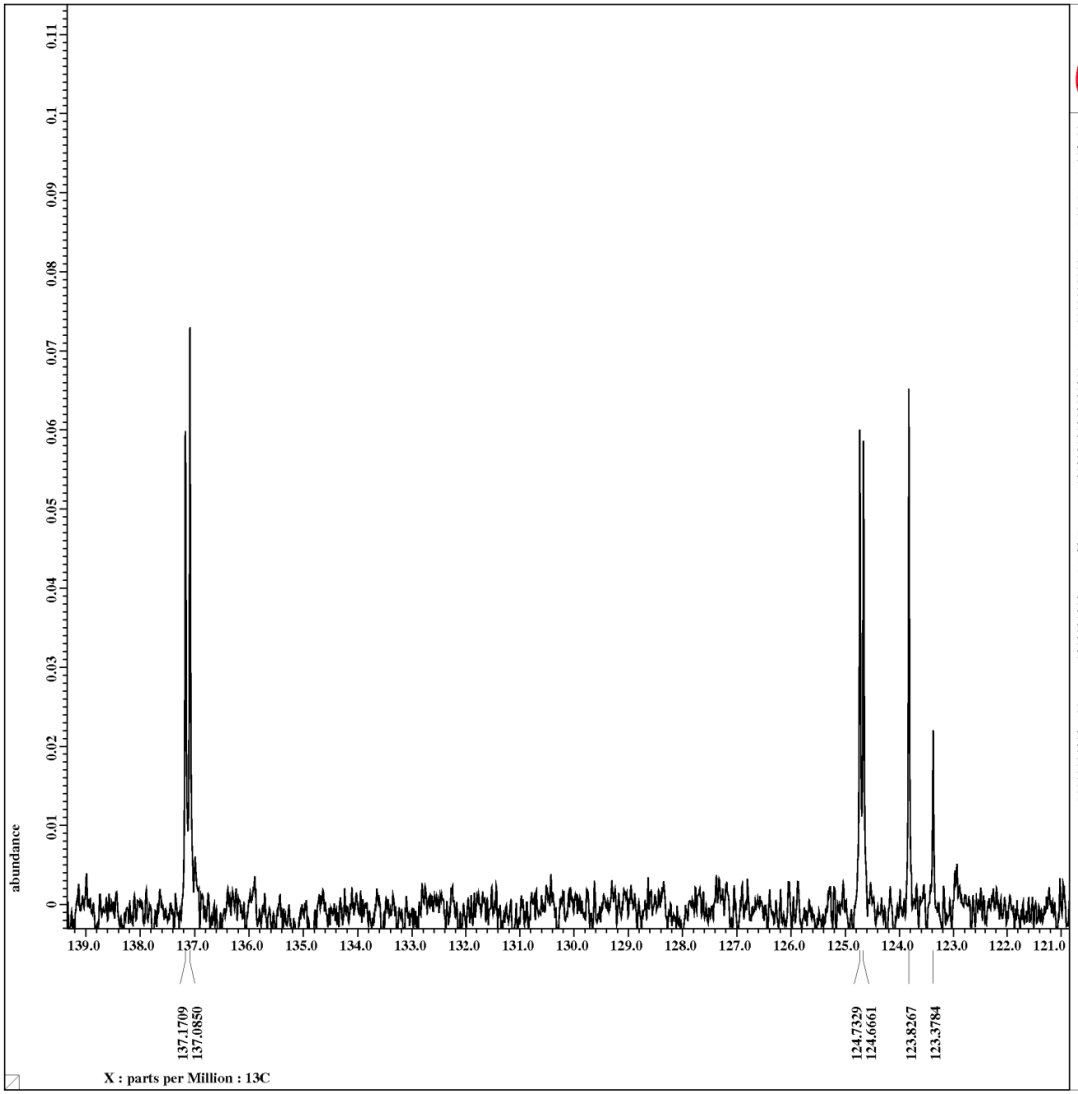
X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.2[dC]
```




abundance
4.0
3.0
2.0
1.0
0
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
7.8664
7.8641
7.8458
7.8435
7.6133
7.6122
7.6110
7.2549
7.1656
7.1644
7.1633
X : parts per Million : 1H


Filename = HMF_III_155_2-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#537655
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 04:39:30
Revision_time = 5-NOV-2011 16:24:33
Current_time = 5-NOV-2011 16:24:50
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.2[dC]
O
O
N
N
DMAS
N
N
DMAS


JEOL
Filename = RMF_III_155-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#539277
Solvent = CHLOROFORM-D
Creation_time = 6-NOV-2011 05:20:33
Revision_time = 5-NOV-2011 16:56:20
Current_time = 5-NOV-2011 16:56:39
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 630
Total_scans = 630
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57166667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recyr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.6[dC]
DMAS
DMAS
abundance
X : parts per Million : 13C
153.2906
140.4902
137.1709
137.0850
124.7329
124.4661
123.8267
123.3784
115.0896
82.2206
78.8058
77.3656
77.1080
76.8505
65.9768
38.3157
38.2966



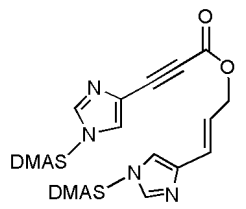
JEOL

```
Filename          = HMF_III_155-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#539277
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2011 05:20:33
Revision_time     =  5-NOV-2011 16:56:20
Current_time      =  5-NOV-2011 16:57:13

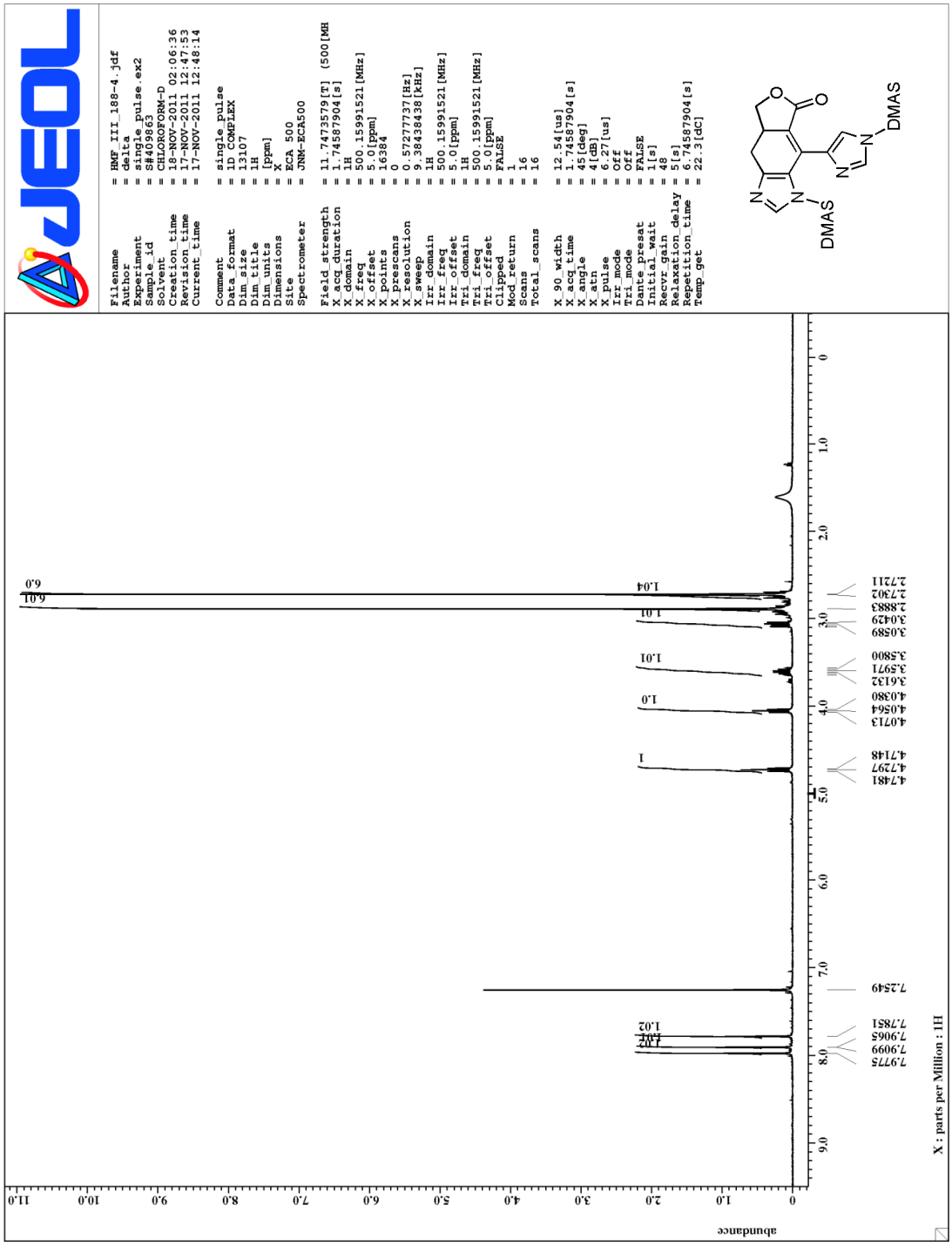
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

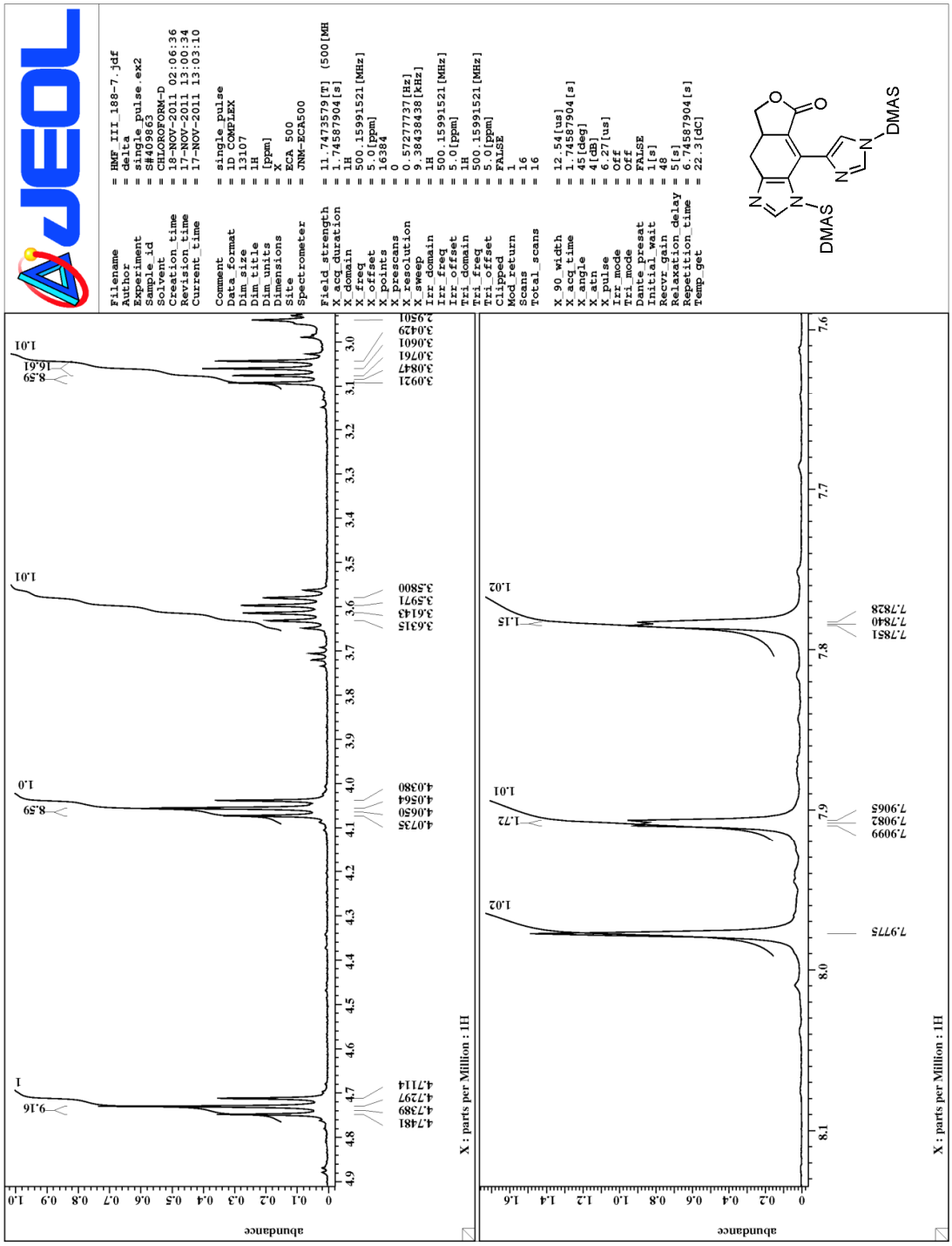
Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 630
Total_scans       = 630

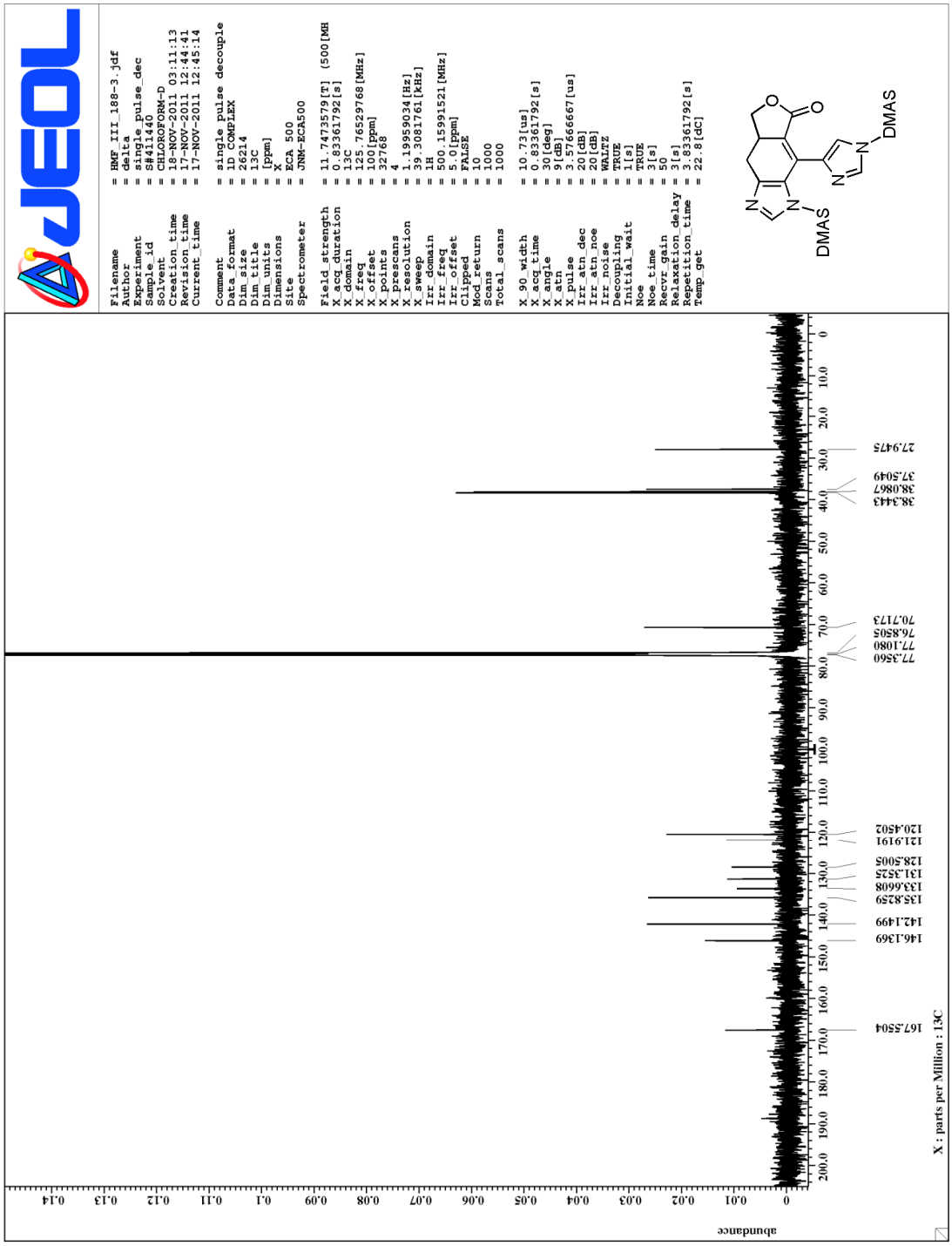
X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 3[s]
Recvr_gain        = 50
Relaxation_delay  = 3[s]
Repetition_time   = 3.83361792[s]
Temp_get          = 22.6[dC]
```



APPENDIX 63
^{1}H and ^{13}C NMR Spectra of
3-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]-3,7,7a,8-tetrahydro-4-[1-(*N*,*N*-dimethylsulfamoyl)-1*H*-imidazol-4-yl]-5*H*-furo[3,4-*f*]benzimidazole-5-one (**149**)


JEOL
Filename = RMF_III_188-4.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#409863
Solvent = CHLOROFORM-D
Creation_time = 18-NOV-2011 02:06:36
Revision_time = 17-NOV-2011 12:47:53
Current_time = 17-NOV-2011 12:48:14
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 6[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.3[dC]
DMAS
DMAS
abundance
X : parts per Million : 1H
6.01
1.04
1.01
1.01
1.0
1
1.02
7.2549
4.7481
4.7297
4.7148
4.0713
4.0564
4.0380
3.6132
3.5971
3.5800
3.0589
3.0429
2.8883
2.7302
2.7211
7.9775
7.9099
7.9065
7.7851


JEOL
Filename = RMF_III_188-7.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#409863
Solvent = CHLOROFORM-D
Creation_time = 18-NOV-2011 02:06:36
Revision_time = 17-NOV-2011 13:00:34
Current_time = 17-NOV-2011 13:03:10
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.3[dC]
DMAS
DMAS
abundance
X : parts per Million : 1H
9.16
8.59
8.59
16.61
1.0
1.01
1.01
4.7481
4.7389
4.7297
4.7114
4.0735
4.0650
4.0564
4.0380
3.6315
3.6143
3.5971
3.5800
3.0921
3.0847
3.0761
3.0601
3.0429
2.9501
1.02
1.72
1.01
1.15
1.02
7.9773
7.9099
7.9082
7.9065
7.7851
7.7840
7.7828


Filename = HMF III 188-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#411440
Solvent = CHLOROFORM-D
Creation_time = 18-NOV-2011 03:11:13
Revision_time = 17-NOV-2011 12:44:41
Current_time = 17-NOV-2011 12:45:14
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = UNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1000
Total_scans = 1000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.5716666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 3[s]
Recvr_gain = 50
Relaxation_delay = 3[s]
Repetition_time = 3.83361792[s]
Temp_get = 22.8[dC]
DMAS
DMAS
27.9475
37.5049
38.0867
38.3443
70.7173
76.8505
77.1080
77.3560
120.4502
121.9191
128.5005
131.3525
133.6608
135.8259
142.1499
146.1369
167.5504
abundance
X : parts per Million : 13C

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BIOGRAPHICAL INFORMATION

Heather Lima was born in Houston, TX and was raised in El Paso, TX. She obtained a B.S. in biochemistry from The University of Texas at Arlington in 2005. During her undergraduate studies, she began working with Dr. Carl Lovely in the field of organic chemistry and total synthesis. She continued her work with Dr. Lovely as a graduate student at which time the work she had done as an undergraduate was applied toward the total synthesis of natural products including kealiiquinone and ageliferin. She obtained her Ph.D. in chemistry in 2011, and subsequently joined the synthesis group of Cerilliant Corporation, a company located in Round Rock, TX that produces and markets high quality certified reference standards.