

Supplementary Material

A Mild and Convenient Synthesis of 1,2,3-Triiodoarenes via Consecutive Iodination/Diazotization/Iodination Strategy

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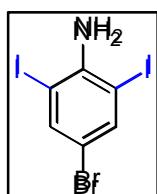
1. General Information

All commercial reagents and chromatography solvents were used as obtained unless otherwise stated. Ethanol, diethyl ether, hydrochloric acid, silver (I) sulfate (Ag_2SO_4 , BDH, analytical reagent), silver nitrate (AgNO_3 , EM Science, 99%), potassium iodide (KI, BDH), and iodine (I_2). Anhydrous solvents were distilled over appropriate drying agents prior to use. Analytical thin layer chromatography (TLC) was performed on Merck silica gel 60 F₂₅₄. Merck Silica gel 60 (0.063 – 0.2 mm) was used for column chromatography. Visualization of TLC was accomplished with UV light (254 nm). NMR spectra were recorded on a Bruker-Avance 400 MHz spectrometer. The residual solvent protons (^1H) or the solvent carbon (^{13}C) were used as internal standards. ^1H -NMR data are presented as follows: chemical shift in ppm (δ) downfield from trimethylsilane (multiplicity, integration, coupling constant). The following abbreviations are used in reporting NMR data: s, singlet; bs, broad singlet; d, doublet; t, triplet; q, quartet; dq, doublet of quartets; dd, doublet of doublets; m, multiplet. High resolution mass spectra were recorded by the Jordan University of Science & Technology using Chemical Ionization (CI) technique.

2. General Procedure for Halogenations of Aniline Derivatives

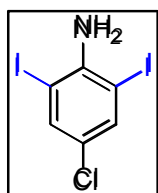
An appropriate aniline derivative (7.9 mmol, 1.0 equiv.), iodine (17.3 mmol, 2.2 equiv) and silver (I) sulfate (3.2 mmol, 1.1 equiv) were dissolved in ethanol (40 mL) and stirred for 24 h at room temperature. The mixture was filtered over Celite® 545 to remove AgI precipitate. Water (200 mL) was added to the filtrate and the mixture was then extracted with ethyl acetate (3 X 50 mL). The combined organic layers were washed with aqueous sodium sulfite to remove excess iodine, washed with brine, dried over Na_2SO_4 , filtered and concentrated. The residue was chromatographed on silica gel (hexane/ethyl acetate 7:1) to yield the pure desired product.

2.1 Synthesis of 4-bromo-2,6-diiodoaniline (4)



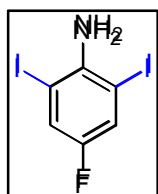
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in 74% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Synthesis* **2006** (20), 3467-3477 and *Org. Biomol. Chem.* **2011**, V.9 (12), 4440-4443.

2.2 Synthesis of 4-chloro-2,6-diiodoaniline (5)



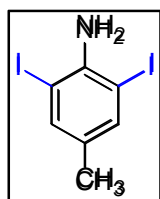
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **72%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *J. Org. Chem.* **2011**; 76, 2123-2131.

2.3 Synthesis of 4-fluoro-2,6-diiodoaniline (6)



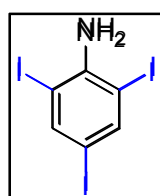
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **31%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.44 (d, 2H, *J* = 7.4 Hz), 4.46 (s, 2H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 154.8, 152.3, 142.7, 125.6, 125.3, 78.9, 78.8. **IR** (cast film, cm⁻¹) 3377, 3258, 3037, 2957, 1616, 1584, 1419, 1004, 900. **Mp**: 67-69 °C. **HRMS** (CI) *m/z* for C₆H₄FI₂N [M-H]⁻: calcd. 362.8417; found, 362.8434.

2.4 Synthesis of 2,6-diiodo-4-methylaniline (7)



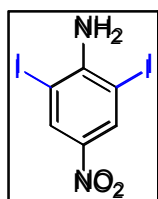
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **56%** yield as a brownish solid. The spectroscopic data for this compound are matched the previous report by *Int. J. ChemTech. Res.* **2009**; 1 (4): 1005-1007.

2.5 Synthesis of 2,4,6-triiodoaniline (8)



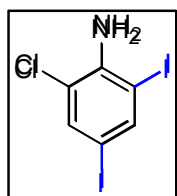
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **51%** yield as a brown solid. The spectroscopic data for this compound are matched the previous report by *Russ. Chem. Bull., Int. Ed.* **2004**; 53 (2), 471-473.

2.6 Synthesis of 2,6-diiodo-4-nitroaniline (9)



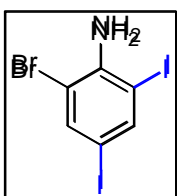
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **32%** yield as a yellow solid. The spectroscopic data for this compound are matched the previous report by *Tetrahedron.* **2002**; 58: 3977-3983.

2.7 Synthesis of 2-chloro-4,6-diiodoaniline (10)



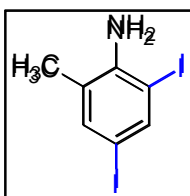
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **88%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.79 (s, 1H), 7.50 (s, 1H), 4.56 (s, 2H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 144.4, 143.2, 137.2, 118.2, 83.7, 77.4. **IR** (cast film, cm⁻¹) 3416, 3387, 3055, 1652, 1167, 1121, 950. **Mp**: 71-73 °C. **HRMS** (CI) *m/z* for C₆H₄ClI₂N [M-H]⁻: calcd. 377.8122; found, 377.8179.

2.8 Synthesis of 2-bromo-4,6-diiodoaniline (11)



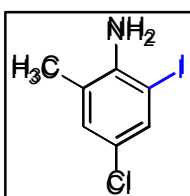
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **89%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.84 (s, 1H), 7.67 (s, 1H), 4.64 (s, 2H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 145.1, 143.9, 140.1, 107.5, 83.4, 77.9. **IR** (cast film, cm⁻¹) 3412, 3314, 2954, 1622, 1095, 930. **Mp**: 65-67 °C. **HRMS** (CI) *m/z* for C₆H₄BrI₂N [M+H]⁺: calcd. 423.7616; found, 423.7640.

2.9 Synthesis of 2,4-diiodo-6-methylaniline (12)



The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **25%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Bull. Chem. Soc. Jpn.* **1988**; 61, 600-602 and *Monatshefte Fur chemie.* **2004**; 135, 1009-1014.

2.10 Synthesis of 4-chloro-2-iodo-6-methylaniline (13)



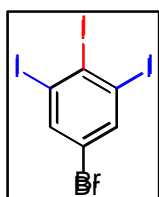
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **28%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.50 (s, 1H), 7.02 (s, 1H), 4.06 (s, 2H), 2.20 (s, 3H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 143.6, 135.5, 130.1, 123.2, 122.8, 83.6, 18.8. **IR** (cast film, cm⁻¹) 3511, 3395, 3120, 2915, 1682, 1154, 1321, 892. **Mp**: 58-60 °C. **HRMS** (CI) *m/z* for C₇H₇ClIN [M-H]⁻: calcd. 265.9312; found, 265.9178.

3. General Procedure for Diazotization of Aniline Derivatives

A concentrated HCl was added dropwise to a solution of iodinated aniline derivative (1.4 mmol, 1.0 equiv.) in 15 mL diethyl ether at 0 °C until no precipitate is formed. The aniline salt was then filtered, washed with cold diethyl ether and collected.

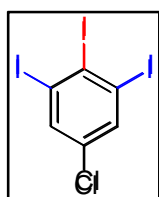
A solution of NaNO_2 (1.53 mmol, 1.1 equiv.) in water (1.0 mL) was added dropwise to a mixture of aniline salt in water (3.5 mL) and concentrated hydrochloric acid (1.5 mL) below 5 °C, and the mixture was stirred for 10 min. A solution of potassium iodide (2.1 mmol, 1.5 equiv.) in water (1.0 mL) was then added dropwise to the reaction mixture. The mixture was stirred for 15 min without cooling, at 50 °C for 30 min and then followed by 45 min at 80 °C. The mixture was cooled to 0 °C, and a solution of 5 % aqueous sodium sulfite (30 mL) was added. The organic layer was separated, and the aqueous layer was extracted with diethyl ether (30 mL, 3 times). The combined organic layers were washed with brine, dried over Na_2SO_4 , filtered and concentrated. The residue was chromatographed on silica gel (hexane) to yield the pure desired product.

3.1 Synthesis of 5-bromo-1,2,3-triiodobenzene (16)



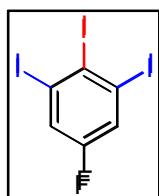
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **81%** yield as a white solid. δ_{H} (400 MHz, *d*- CDCl_3) δ : 7.97 (s, 2H). δ_{C} (100 MHz, *d*- CDCl_3) δ : 141.7, 140.2, 122.6, 106.7. **IR** (cast film, cm^{-1}) 2987, 1642, 1546, 1051, 860. **Mp**: 138-140 °C. **HRMS** (CI) *m/z* for $\text{C}_6\text{H}_2\text{BrI}_3$ [M^+]: calcd. 533.6474; found, 533.6199.

3.2 Synthesis of 5-chloro-1,2,3-triiodobenzene (17)



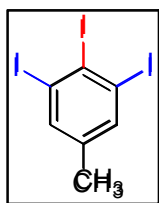
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **88%** yield as a white solid. δ_{H} (400 MHz, *d*- CDCl_3) δ : 7.86 (s, 2H). δ_{C} (100 MHz, *d*- CDCl_3) δ : 138.3, 135.3, 119.2, 106.8. **IR** (cast film, cm^{-1}) 3025, 1633, 1492, 894. **Mp**: 118-120 °C. **HRMS** (CI) *m/z* for $\text{C}_6\text{H}_2\text{ClI}_3$ [M^+]: calcd. 489.6979; found, 489.6939.

3.3 Synthesis of 5-fluoro-1,2,3-triiodobenzene (18)



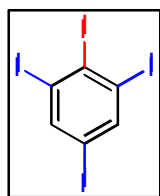
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **45%** yield as a white solid. δ_{H} (400 MHz, *d*- CDCl_3) δ : 7.62 (d, 2H, $J = 4.0$ Hz). δ_{C} (100 MHz, *d*- CDCl_3) δ : 161.8, 159.3, 126.2, 125.9, 115.1, 105.5, 105.4. **IR** (cast film, cm^{-1}) 3065, 2941, 1596, 1435, 1187, 852. **Mp**: 127-129 °C. **HRMS** (CI) *m/z* for $\text{C}_6\text{H}_2\text{FI}_3$ [M^+]: calcd. 473.7275; found, 473.7164.

3.4 Synthesis of 1,2,3-triiodo-5-methylbenzene (19)



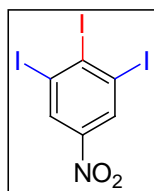
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **59%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.69 (s, 1H), 2.17 (s, 3H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 140.9, 139.1, 116.2, 106.2, 19.4. **IR** (cast film, cm⁻¹) 3149, 2985, 1614, 1587, 1215, 1146, 840. **Mp**: 113-115 °C. **HRMS** (CI) *m/z* for C₇H₅I₃ [M⁺]: calcd. 469.8286; found, 469.9000.

3.5 Synthesis of 1,2,3,5-tetraiodobenzene (20)



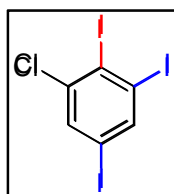
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **46%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 8.13 (s, 2H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 146.1, 121.0, 108.1, 94.9. **IR** (cast film, cm⁻¹) 2987, 1534, 1145, 1009, 873. **Mp**: 133-135 °C. **HRMS** (CI) *m/z* for C₆H₂I₄ [M⁺]: calcd. 581.6335; found, 581.6158.

3.6 Synthesis of 1-chloro-2,3,5-triiodobenzene (21)



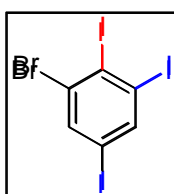
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **21%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *J. Am. Chem. Soc.*, **2007**, *129*(16), 4886-4887 and *J. Labelled Compd. Rad.*, **1987**, *24*(8), 949-55.

3.7 Synthesis of 1-chloro-2,3,5-triiodobenzene (22)



The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **66%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 8.07 (s, 1H), 7.71 (s, 1H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 145.1, 139.6, 136.5, 112.6, 110.8, 94.2. **IR** (cast film, cm⁻¹) 3012, 2983, 1642, 1548, 1124, 863. **Mp**: 112-114 °C. **HRMS** (CI) *m/z* for C₆H₂ClI₃ [M⁺]: calcd. 489.6979; found, 489.6775.

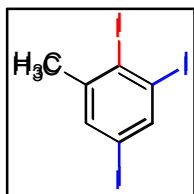
3.8 Synthesis of 1-bromo-2,3,5-triiodobenzene (23)



The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **67%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 8.10 (s, 1H), 7.88 (s, 1H). δ_{C} (100 MHz, *d*-

CDCl_3) δ : 145.5, 139.7, 130.2, 115.2, 110.3, 94.5. **IR** (cast film, cm^{-1}) 3042, 2968, 1604, 1523, 1154, 760. **Mp**: 113-115 °C. **HRMS** (CI) m/z for $\text{C}_6\text{H}_2\text{BrI}_3$ [M^+]: calcd. 533.6474; found, 533.6423.

3.9 Synthesis of 1,2,5-triiodo-3-methylbenzene (24)

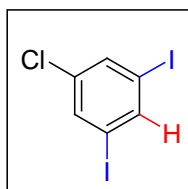


The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **45%** yield as a white solid. δ_{H} (400 MHz, $d\text{-CDCl}_3$) δ : 8.00 (s, 1H), 7.49 (s, 1H), 2.54 (s, 3H). δ_{C} (100 MHz, $d\text{-CDCl}_3$) δ : 145.8, 143.9, 136.6, 113.7, 110.5, 93.8, 31.8. **IR** (cast film, cm^{-1}) 3125, 2945, 2913, 1642, 1574, 1351, 1121, 931. **Mp**: 114-116 °C. **HRMS** (CI) m/z for $\text{C}_7\text{H}_5\text{I}_3$ [M^+]: calcd. 469.8286; found, 469.8613.

4. General procedure for Metal-Halogen exchange reactions of 1,2,3-triiodoarenes

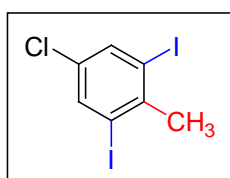
To a solution of triiodoarene (0.42 mmol) in 15 mL of a mixture of THF at -78 °C was added dropwise isopropyl magnesium chloride (2M in THF, 0.23 mL, 0.46 mmol). The mixture was stirred at that temperature for 2 h and then, electrophile was added. The solution was slowly warmed to room temperature and stirred overnight. Saturated NH_4Cl was added and the resulting mixture was stirred 30 min. at room temperature. The aqueous layer was extracted with Et_2O (2 x 50 mL). Drying of the organic phase with Na_2SO_4 , filtered and then the solvent was evaporated under reduced pressure. The crude product was purified by flash chromatography (100% hexane) to yield the pure desired product.

4.1 Synthesis of 1-chloro-3,5-diiodobenzene (25)



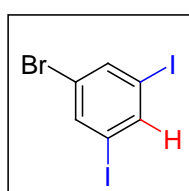
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **65%** yield as a white solid. δ_{H} (400 MHz, $d\text{-CDCl}_3$) δ : 7.94 (s, 1H), 7.68 (s, 2H). δ_{C} (100 MHz, $d\text{-CDCl}_3$) δ : 143.0, 136.1, 135.1, 93.9. **IR** (cast film, cm^{-1}) 2945, 2916, 1623, 1586, 1414, 1125, 762. **Mp**: 131-133 °C. **HRMS** (CI) m/z for $\text{C}_6\text{H}_3\text{ClI}_2$ [M^+]: calcd. 363.8013; found, 363.7996.

4.2 Synthesis of 5-chloro-1,3-diiodo-2-methylbenzene (26)



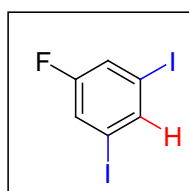
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 47% yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.82 (s, 2H), 2.72 (s, 3H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 141.2, 138.3, 131.8, 97.8, 33.7. **IR** (cast film, cm⁻¹) 3148, 2975, 2846, 1634, 1573, 1243, 1147, 876. **Mp**: 145-147 °C. **HRMS** (CI) *m/z* for C₇H₅ClI₂ [M⁺]: calcd. 377.8169; found, 377.8144.

4.3 Synthesis of 1-bromo-3,5-diiodobenzene (27)



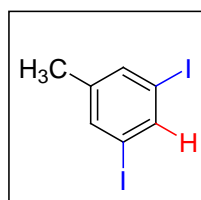
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 68% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Org. Biomol. Chem.*, **2011**, 9(12), 4440-4443.

4.4 Synthesis of 1-fluoro-3,5-diiodobenzene (28)



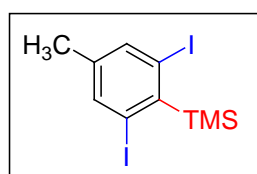
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 71% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Revue Roumaine d Chimie*, **1989**, 34(3), 807-810.

4.5 Synthesis of 1,3-diiodo-5-methylbenzene (29)



The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 73% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.*, **2008**, 47(33), 6208-6211.

4.6 Synthesis of (2,6-diiodo-4-methylphenyl)trimethylsilane (30)



The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 39% yield as colorless oil. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.81 (s, 2H), 2.18 (s, 3H), 0.64 (s, 9H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 141.8, 141.2, 136.9, 102.6, 19.1, 4.53. **IR** (cast film, cm⁻¹) 3201, 2918, 2894, 1687, 1548, 1423, 1250, 1146, 944. **HRMS** (CI) *m/z* for C₁₀H₁₄I₂Si [M⁺]: calcd. 415.8954; found, 415.8913.

5. NMR Spectra for New Compounds

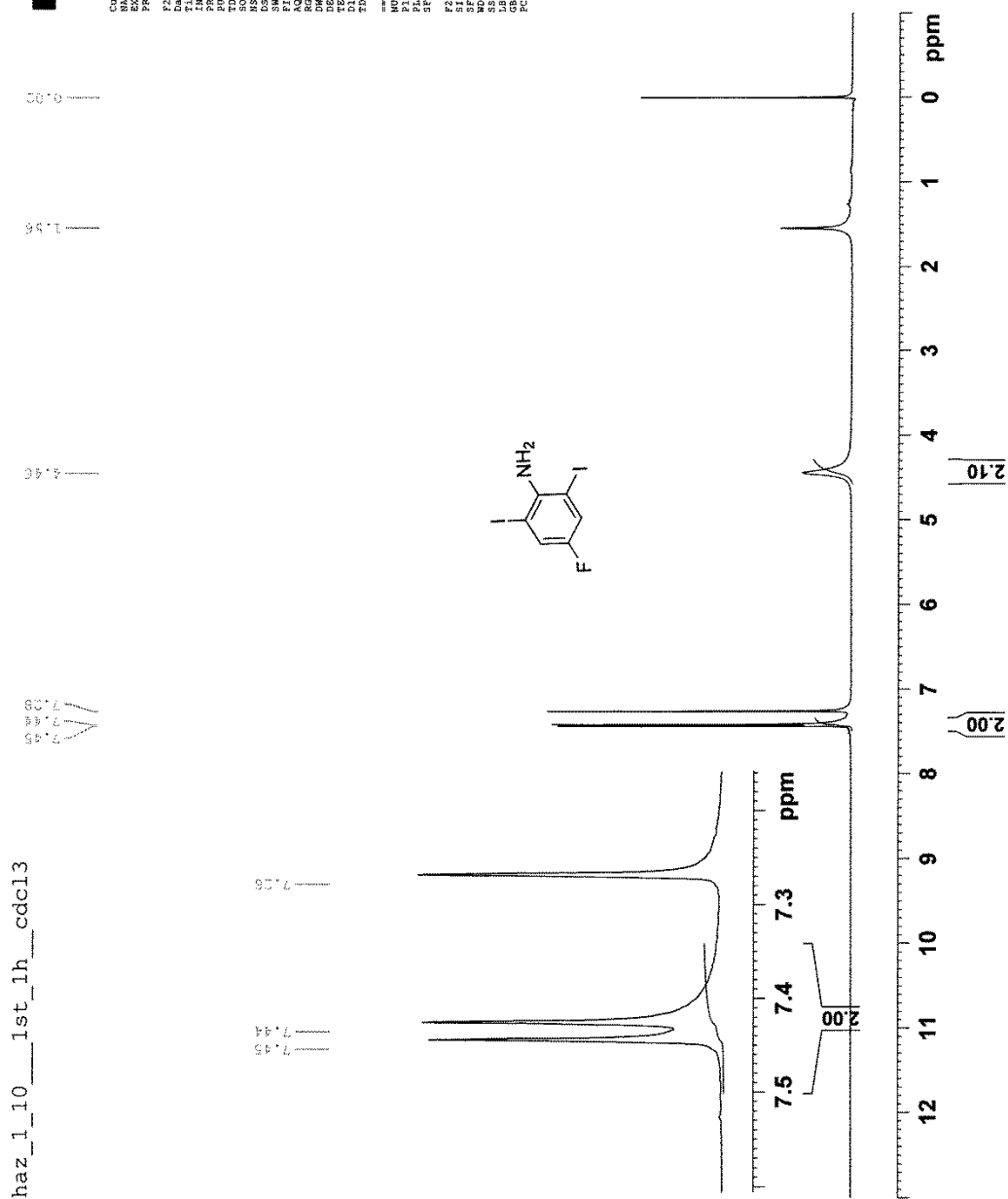
5.1 $^1\text{H-NMR}$ of 4-fluoro-2,6-diiodoaniline in (6) $d\text{-CDCl}_3$ at 25 °C.

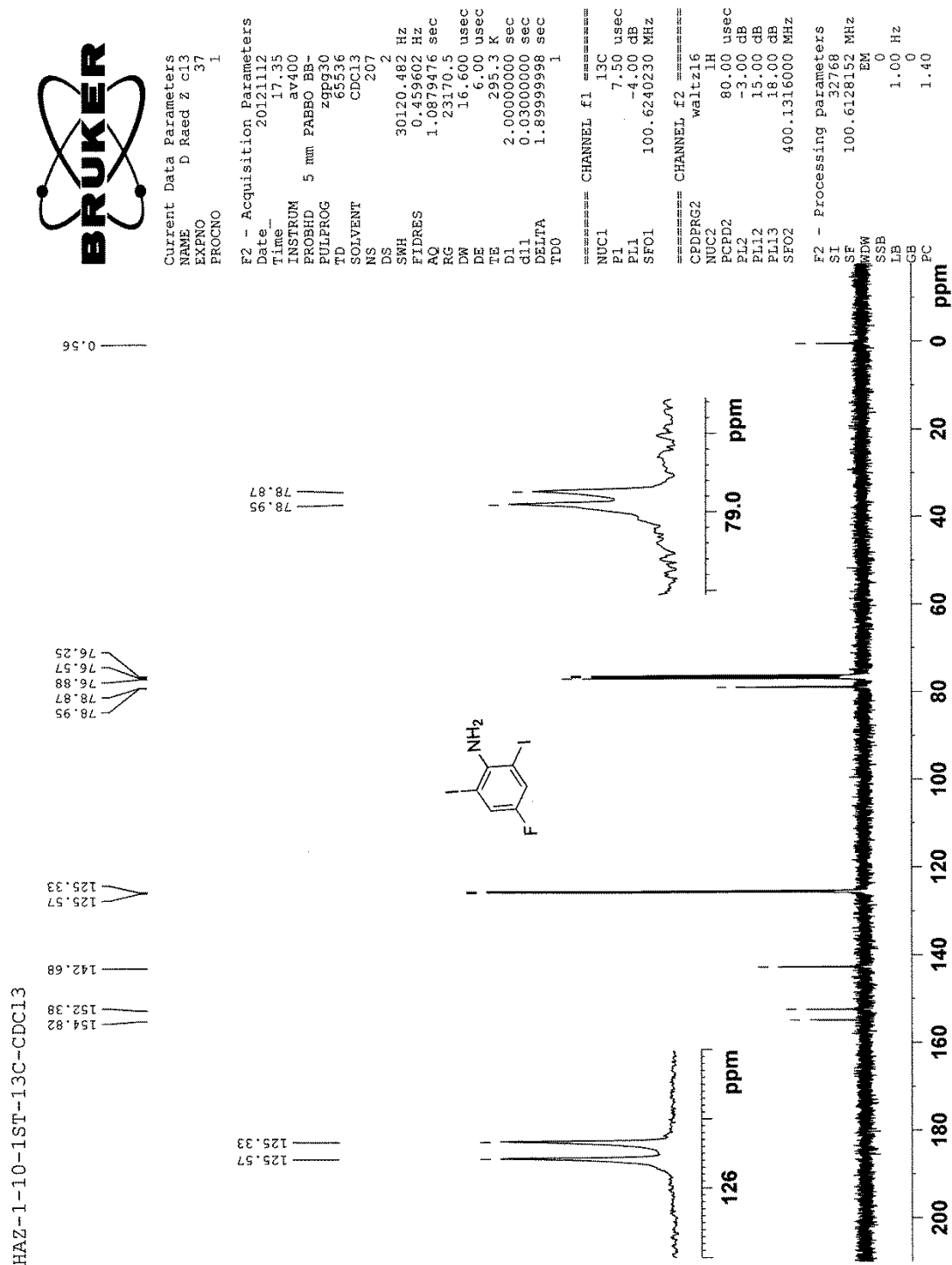
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 PRNUC10 41.1

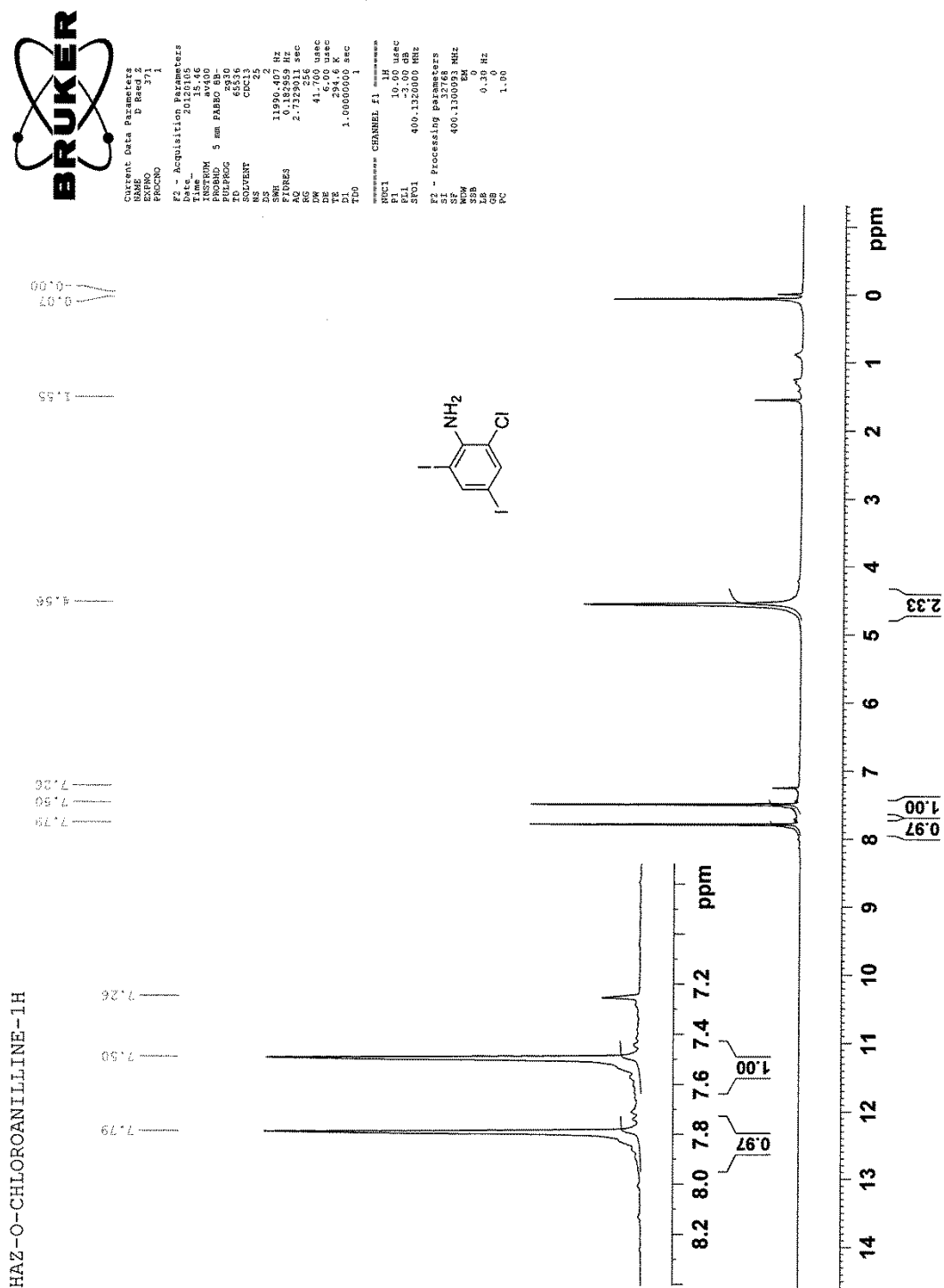
F2 - Acquisition Parameters
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 Time_ 15.27
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 PULPROG zgpg30
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 NS 20
 DS 2
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 SF 400.130000 MHz
 FIDRES 2.7132011 sec
 AQ 37.70 usec
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 DE 5.00 usec
 TE 295.6 K
 ZD 1.0000000 sec

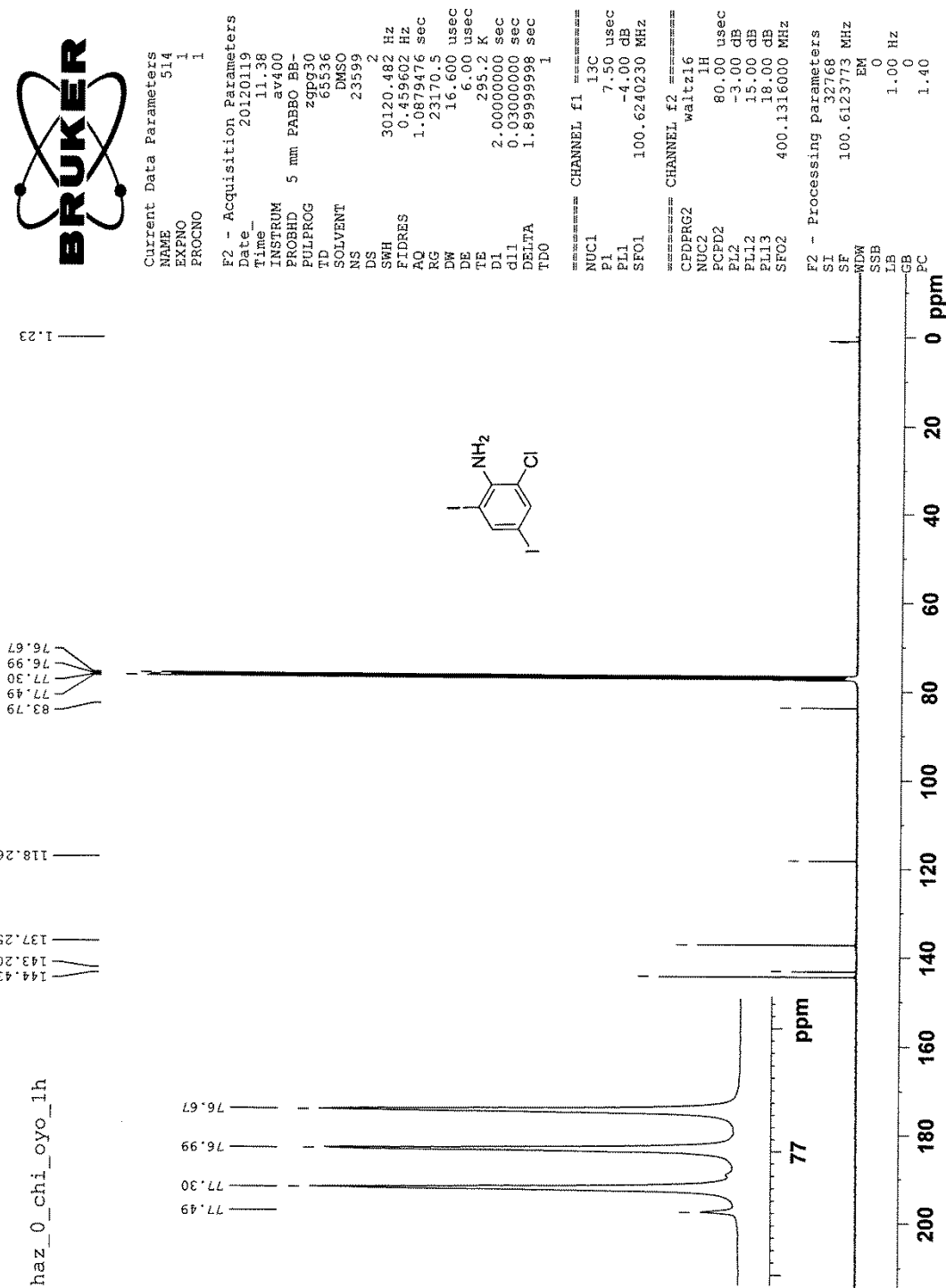
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 PL 0.00 dB
 SFO1 400.130000 MHz

F2 - Processing Parameters
 SF 400.130048 MHz
 DS 2
 GB 0.30 Hz
 PC 1.00

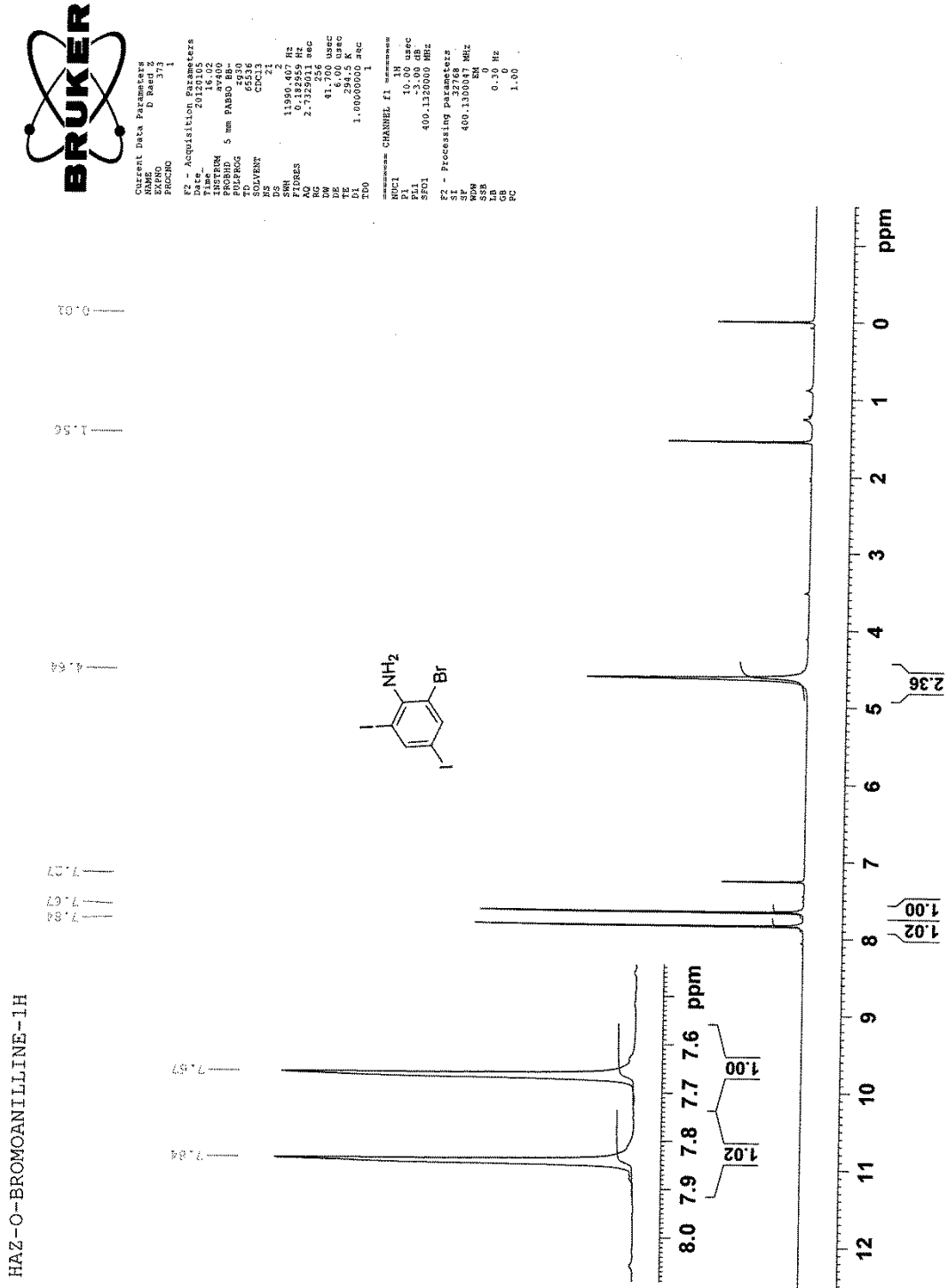


5.2 ^{13}C -NMR of 4-fluoro-2,6-diiodoaniline (6) in $d\text{-CDCl}_3$ at 25 °C.

5.3 ¹H-NMR of 2-chloro-4,6-diiodoaniline (10) in d-CDCl₃ at 25 °C.

5.4 ¹³C-NMR of 6-chloro-2,4-diiodoaniline (10) in *d*-CDCl₃ at 25 °C.

5.5 ¹H-NMR of 6-bromo-2,4-diiodoaniline (11) in *d*-CDCl₃ at 25 °C.



5.6 ^{13}C -NMR of 6-bromo-2,4-diiodoaniline (**11**) in $d\text{-CDCl}_3$ at 25 °C.

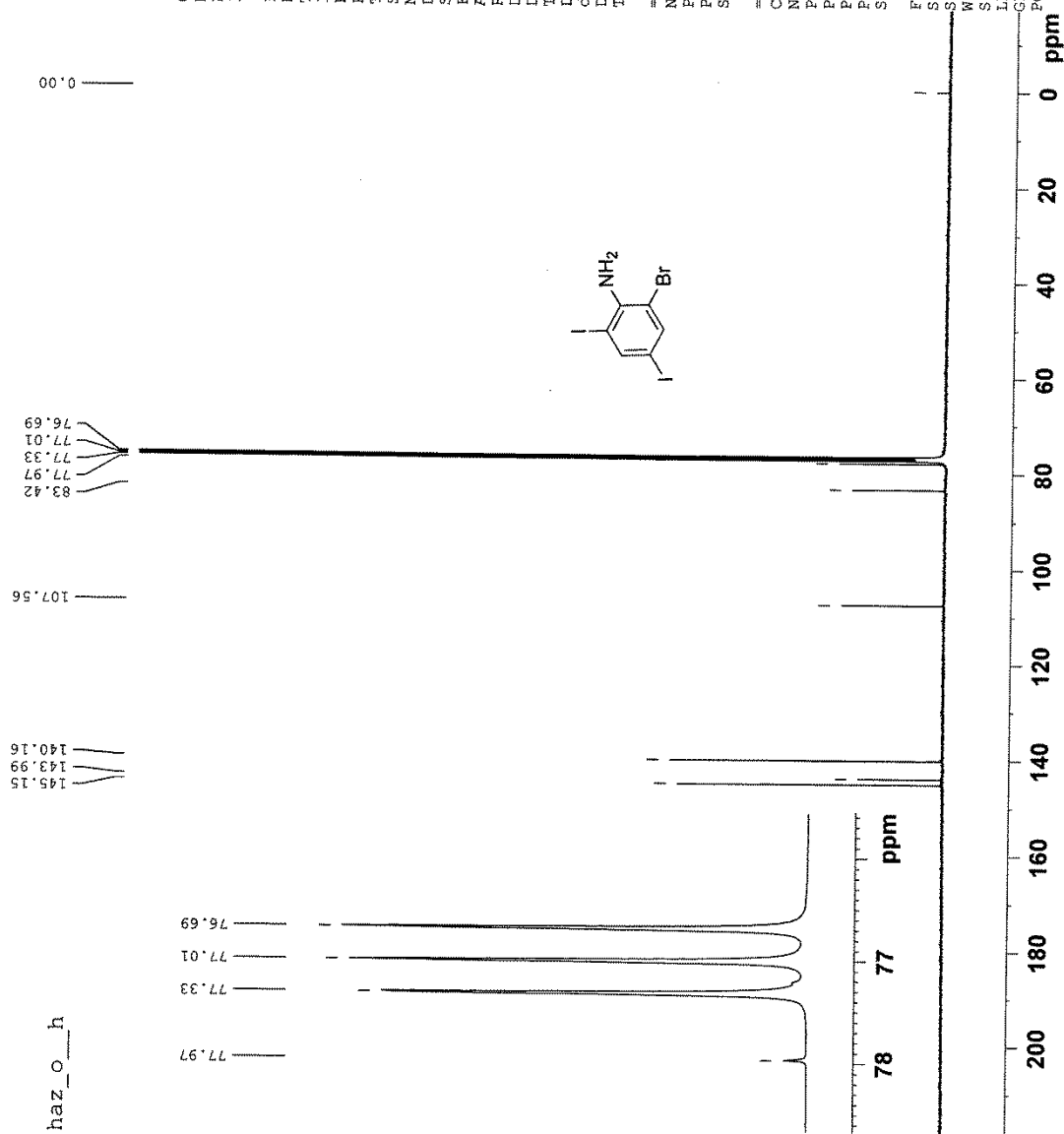
Current Data Parameters
 NAME 516
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 INSTRUM av400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 85536
 SOLVENT CDCl3
 NS 40000
 DS 2
 SWH 30120.482 Hz
 FIDRES 0.459602 Hz
 AQ 1.0879476 sec
 RG 23170.5
 DW 16.600 usec
 DE 6.00 usec
 TE 293.6 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TDO 1

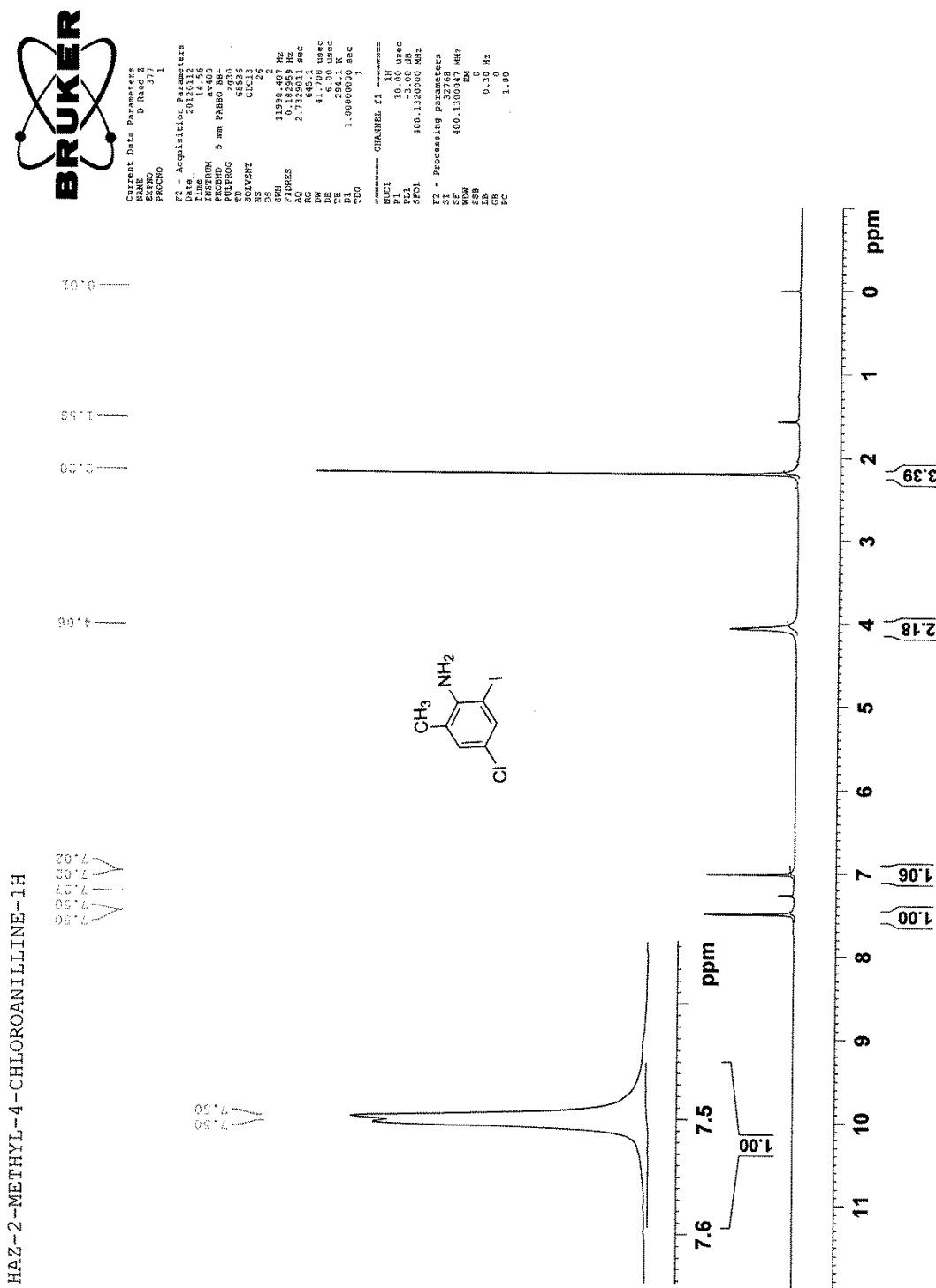
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 NUC1 ^{13}C
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 PL1 -4.00 dB
 SFO1 100.6240230 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 ^1H
 FCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.00 dB
 PL13 18.00 dB
 SFO2 400.1316000 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6122935 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



5.7 ¹H-NMR of 4-chloro-2-iodo-6-methylaniline (13) in d-CDCl₃ at 25 °C.



5.8 ¹³C-NMR of 4-chloro-2-iodo-6-methylaniline (13) in *d*-CDCl₃ at 25 °C.

```

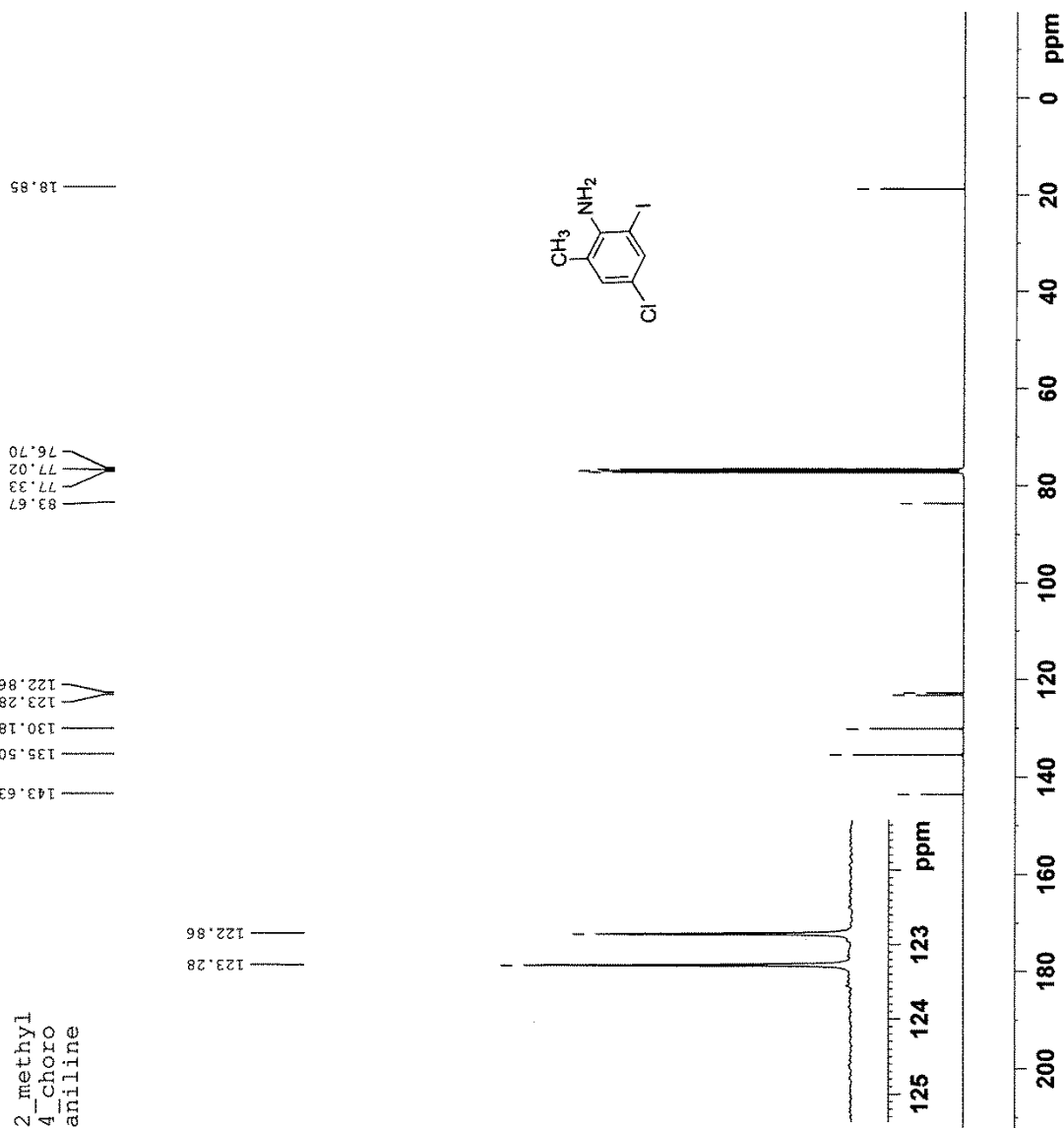
Current Data Parameters
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EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
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Time_         10.42
INSTRUM       av400
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            40000
DS            2
SWH           30120.482 Hz
FIDRES        0.459602 Hz
AQ            1.0879476 sec
RG            23170.5
DE            16.600 usec
TE            293.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA        1.89999998 sec
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1            7.50 usec
PL1           -4.00 dB
SFO1         100.6240230 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -3.00 dB
PL12          15.00 dB
PL13          18.00 dB
SFO2         400.1316000 MHz

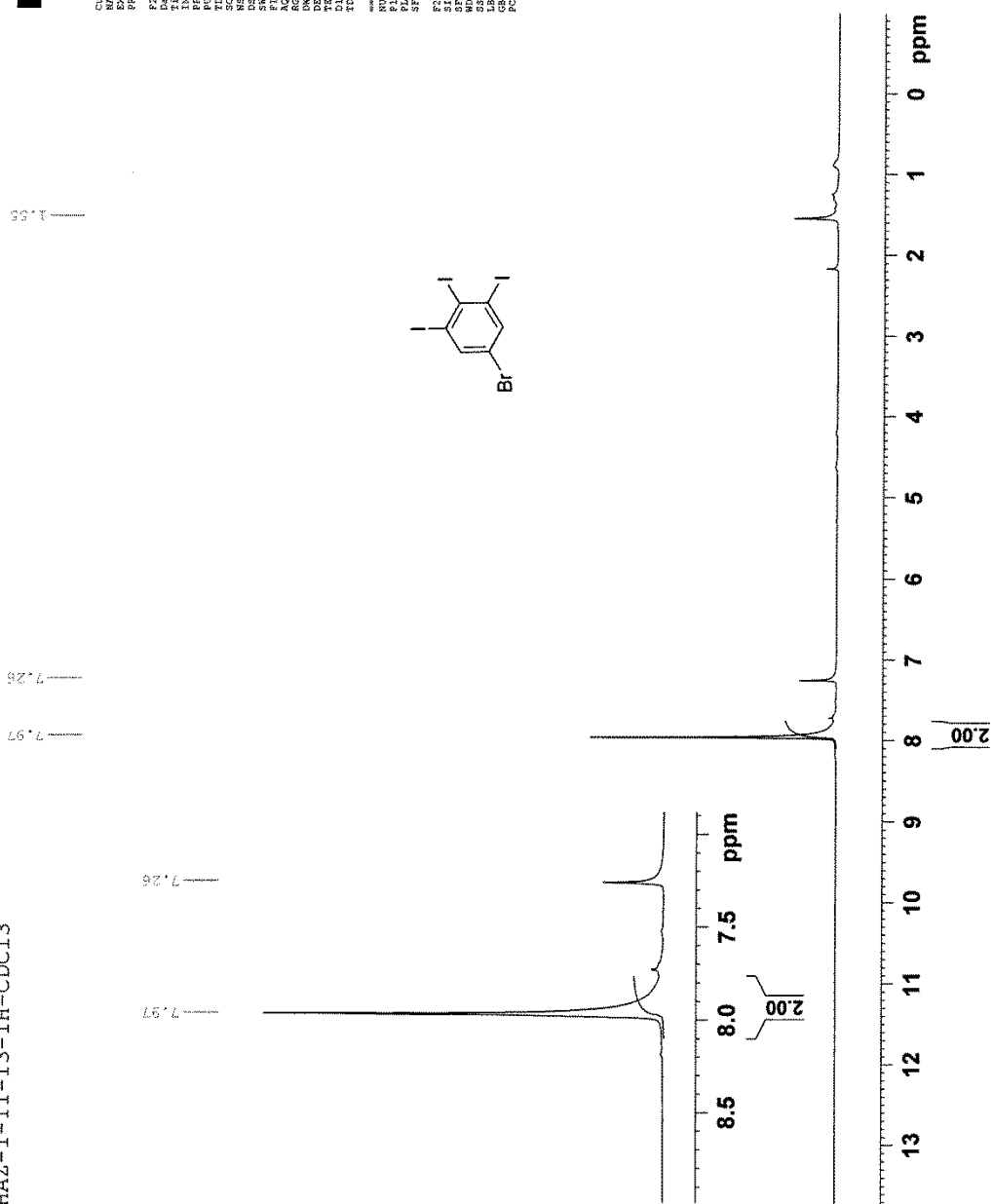
F2 - Processing parameters
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SF           100.6122942 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
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5.9 $^1\text{H-NMR}$ of 5-bromo-1,2,3-triiodobenzene (16) in $d\text{-CDCl}_3$ at 25 $^\circ\text{C}$.

Current Data Parameters
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20120510
 Time 13:03
 INSTRUM spect
 PULPROG zgpg30
 SOLVENT CDCl3
 NS 125
 DS 4
 SWH 13990.407 Hz
 FIDRES 0.182559 Hz
 AQ 2.7329011 sec
 RG 327.5
 DW 41.700 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.0000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL 0.00 dB
 SFO1 400.1310000 MHz
 F2 - Processing Parameters
 SI 327.5
 SF 400.1340072 MHz
 SD 327.5
 LB 0.30 Hz
 GB 0
 PC 1.00

HAZ-1-11-13-1H-CDCl3



5.10 ^{13}C -NMR of 5-bromo-1,2,3-triodobenzene (16) in $d\text{-CDCl}_3$ at 25 °C.

Current Data Parameters
 NAME D Raed Z cl3
 EXPNO 36
 PROCNO 1

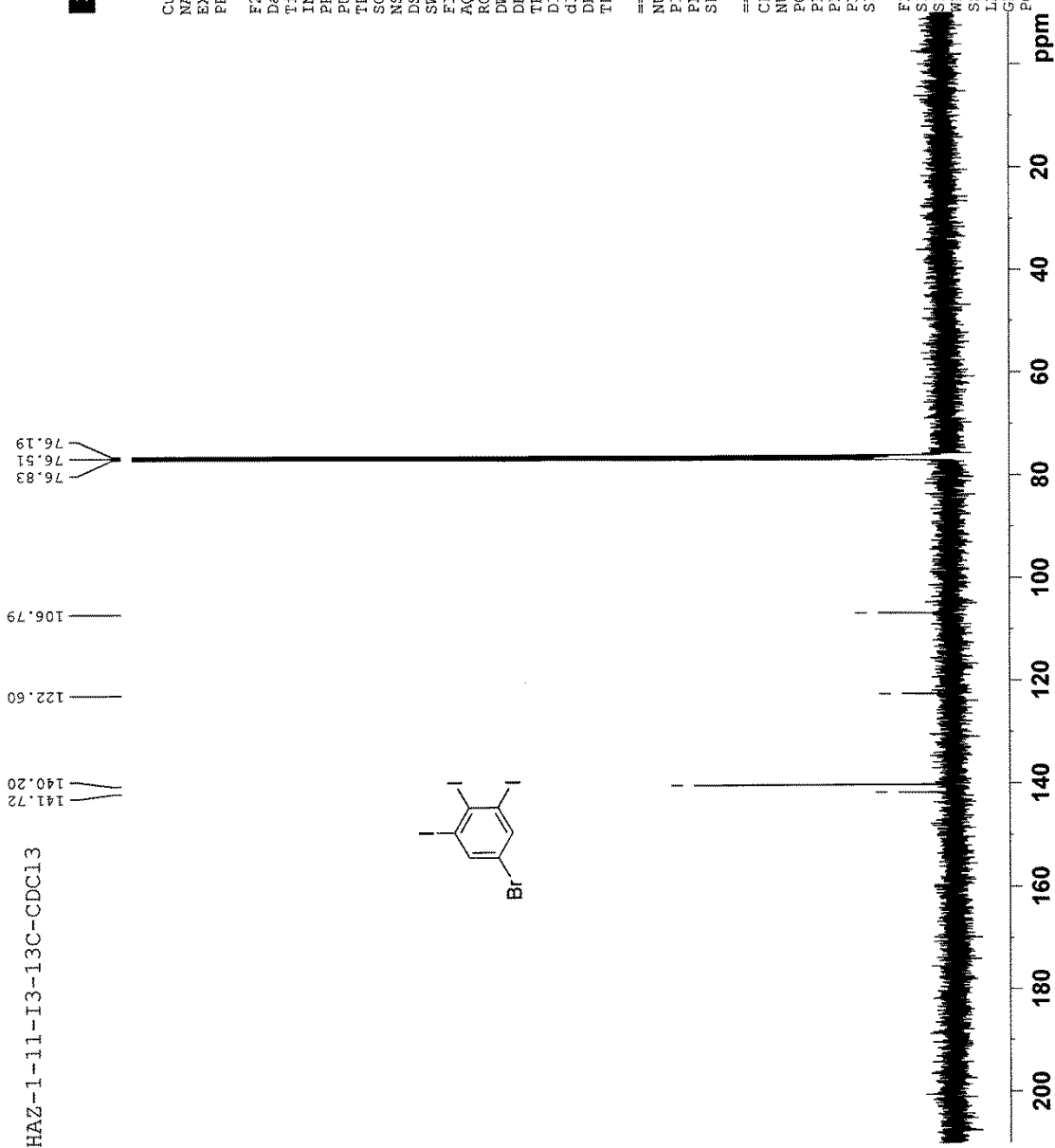
F2 - Acquisition Parameters

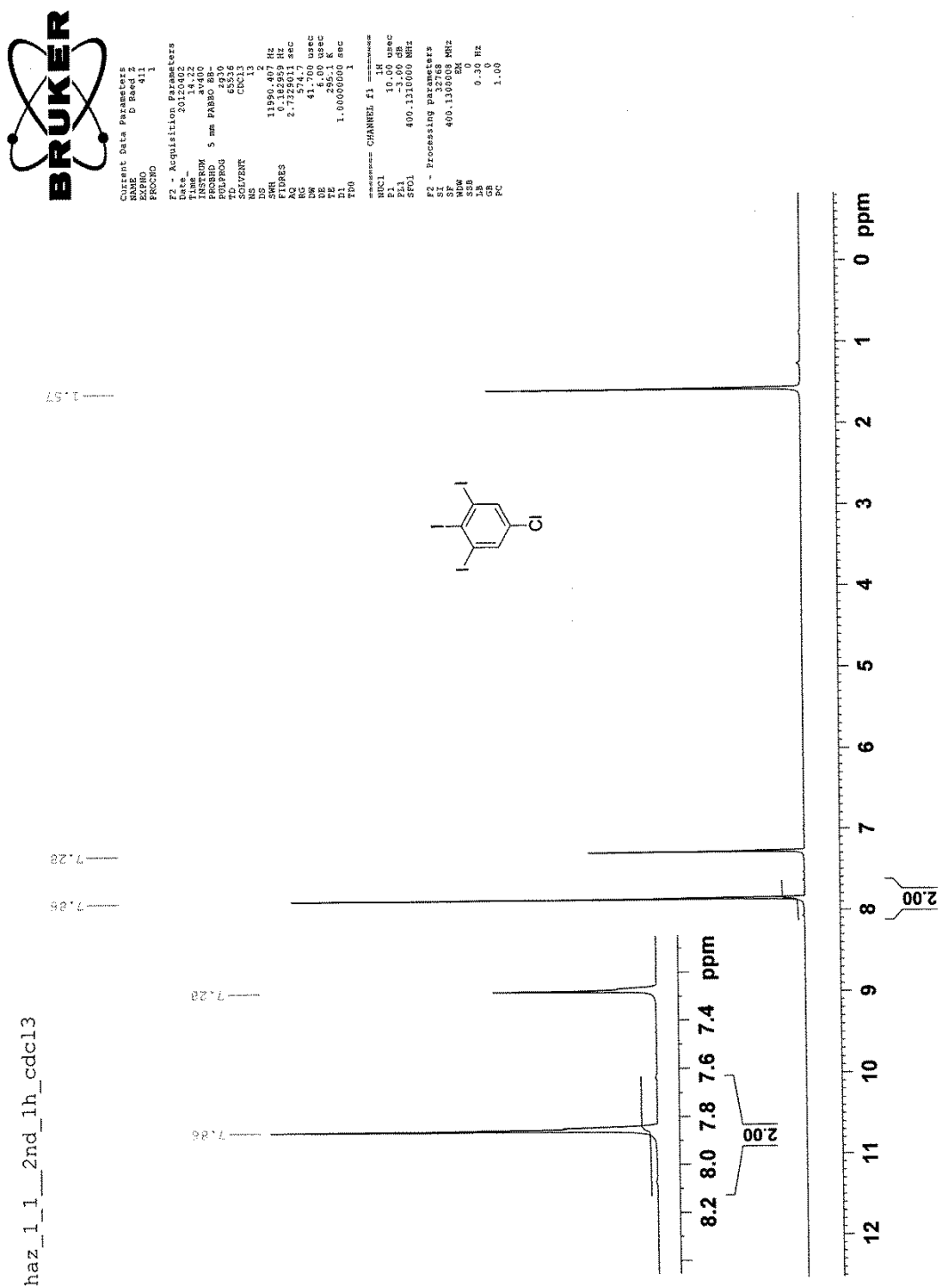
Date_ 20121112
 Time_ 16.55
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 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 684
 DS 2
 SWH 30120.482 Hz
 FIDRES 0.459602 Hz
 AQ 1.0879476 sec
 RG 23170.5
 DW 16.600 usec
 DE 6.00 usec
 TE 295.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 ^{13}C
 P1 7.50 usec
 PL1 -4.00 dB
 SFO1 100.6240230 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ^1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PLI2 15.00 dB
 PLI3 18.00 dB
 SFO2 400.1316000 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6128152 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



5.11 $^1\text{H-NMR}$ of 5-chloro-1,2,3-triiodobenzene (**17**) in $d\text{-CDCl}_3$ at 25°C .

5.12 ¹³C-NMR of 5-chloro-1,2,3-triodobenzene (17) in *d*-CDCl₃ at 25 °C.

Current Data Parameters
 NAME D Raed Z c13
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120503
 Time_ 9.18
 INSTRUM av400
 PROBHD 5 mm FAPBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 846
 DS 2
 SWH 30120.482 Hz
 FIDRES 0.459602 Hz
 AQ 1.0879476 sec
 RG 23170.5
 DW 16.600 usec
 DE 6.00 usec
 TE 295.8 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89899998 sec
 TDO 1

CHANNEL f1
 NUC1 13C
 P1 7.50 usec
 PL1 -4.00 dB
 SFO1 100.6240230 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PLI2 15.00 dB
 PLI3 18.00 dB
 SFO2 400.1316000 MHz

F2 - Processing parameters
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 SF 100.6127593 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

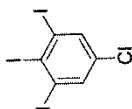
76.81
 77.13
 77.44

106.84

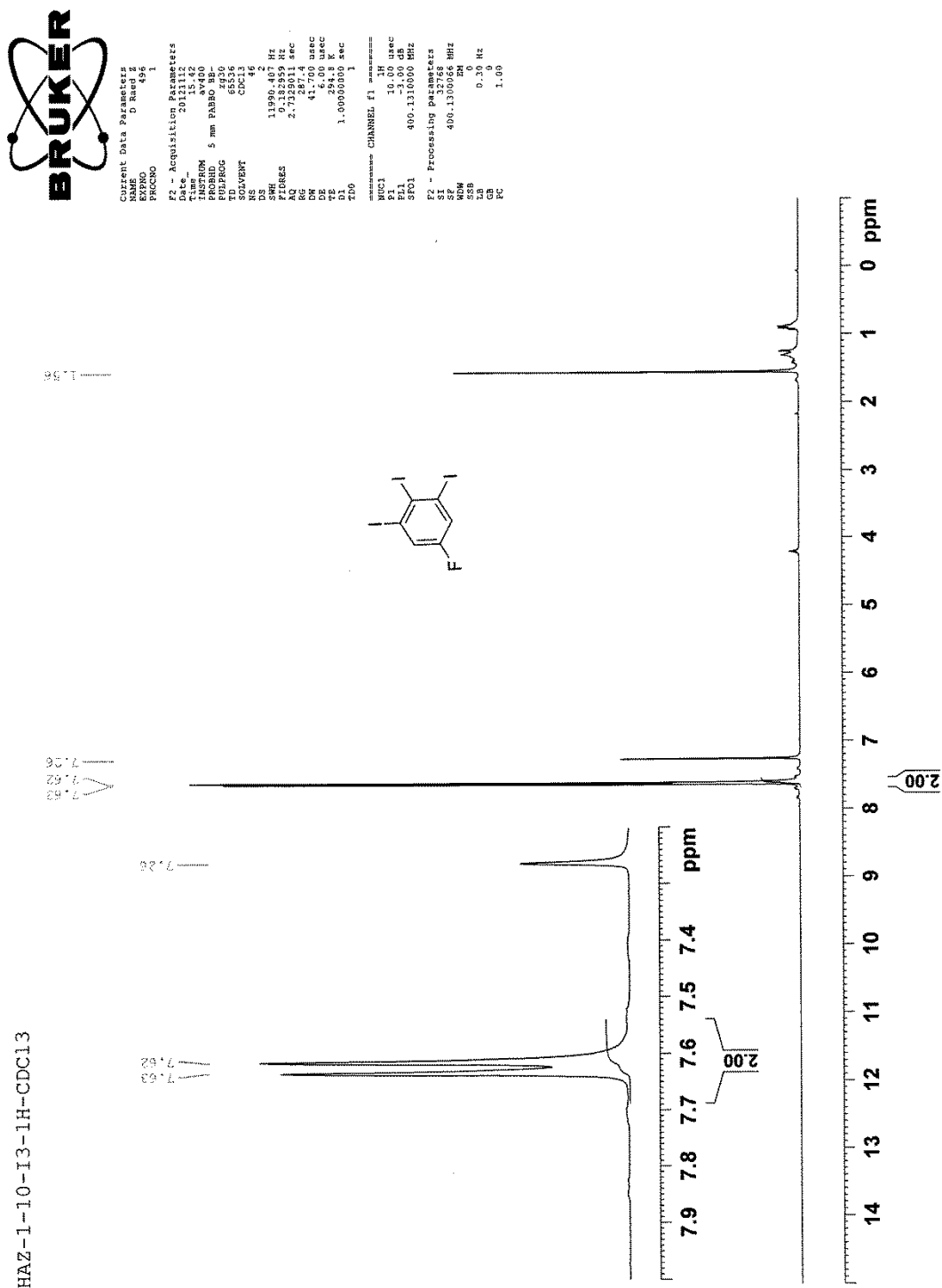
119.24

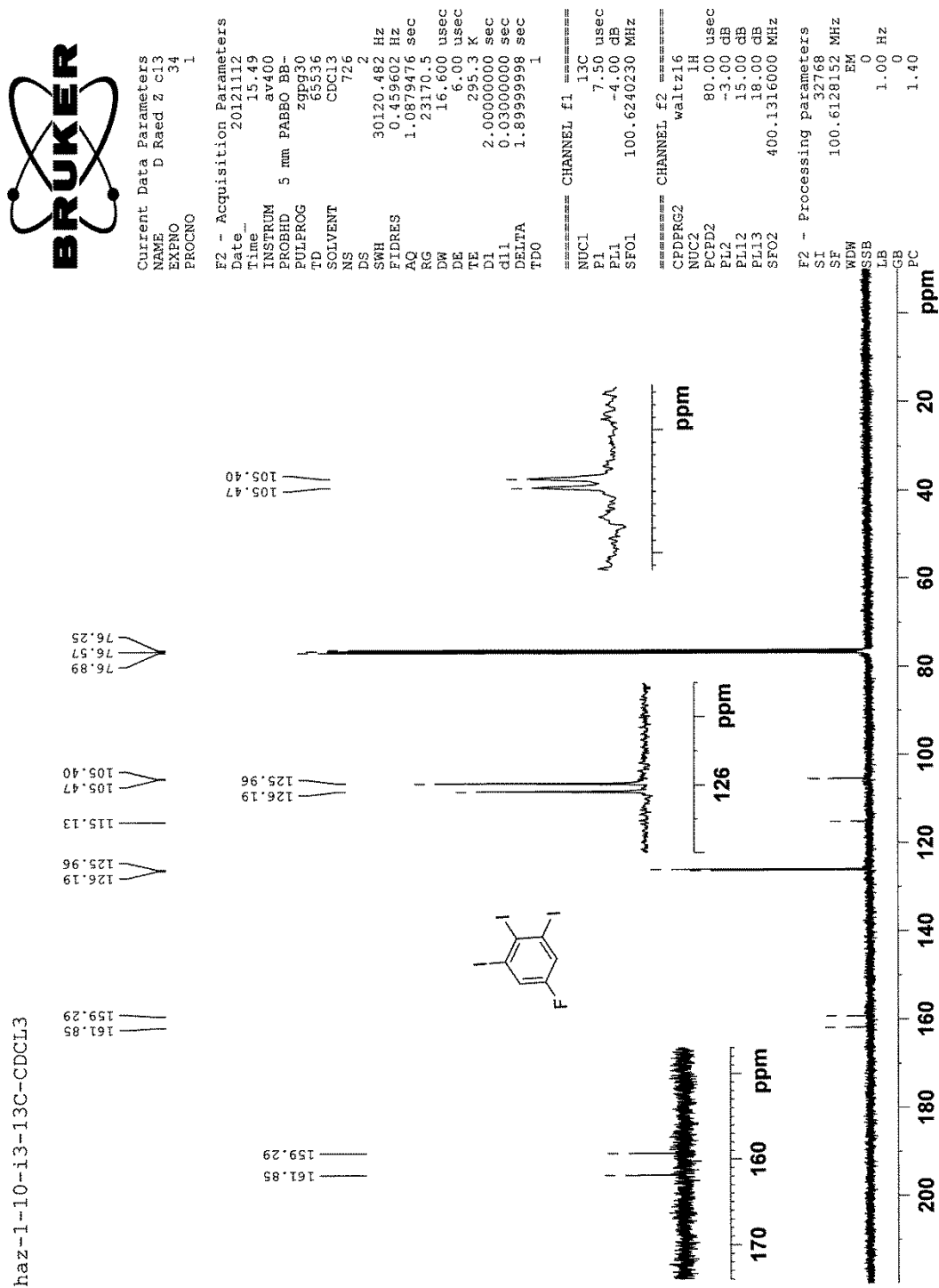
138.30
 139.28

haz_1_1_2nd_c13_cdcl3



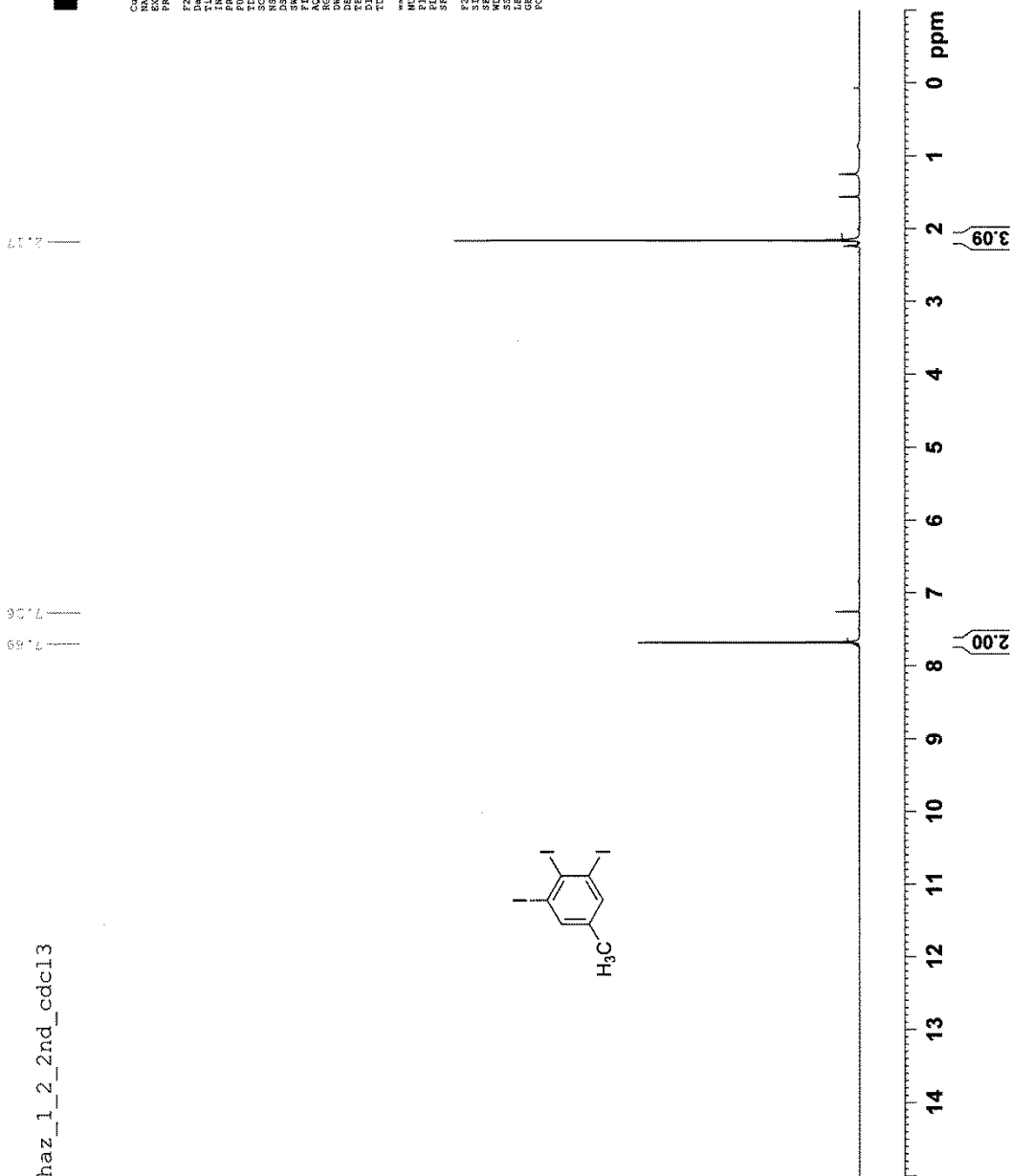
ppm

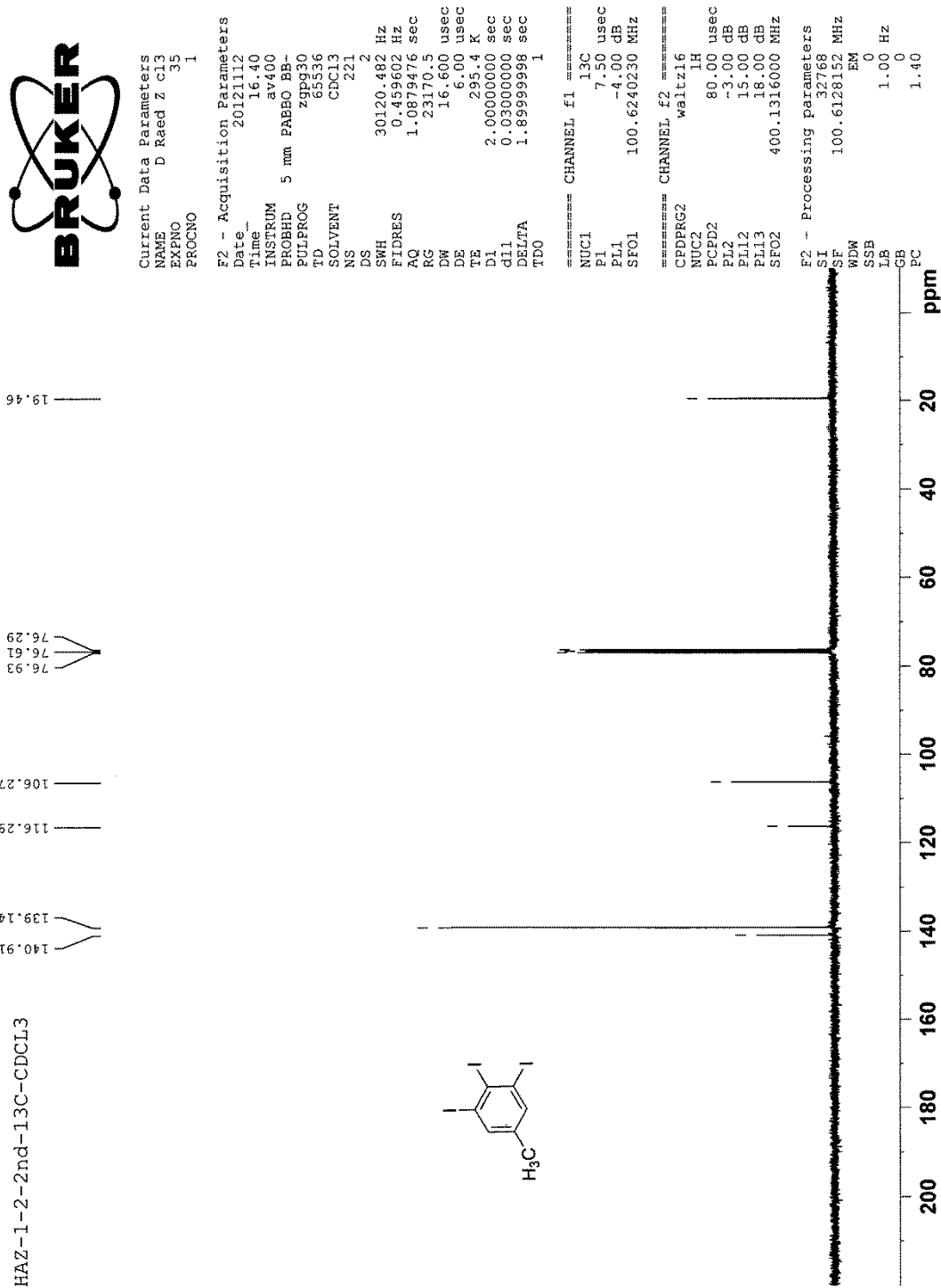
5.13 $^1\text{H-NMR}$ of 5-fluoro-1,2,3-triiodobenzene (18) in $d\text{-CDCl}_3$ at 25 $^\circ\text{C}$.

5.14 ^{13}C -NMR of 5-fluoro-1,2,3-triiodobenzene (18) in $d\text{-CDCl}_3$ at 25 °C.

5.15 $^1\text{H-NMR}$ of 1,2,3-triiodo-5-methylbenzene (19) in $d\text{-CDCl}_3$ at 25 °C.

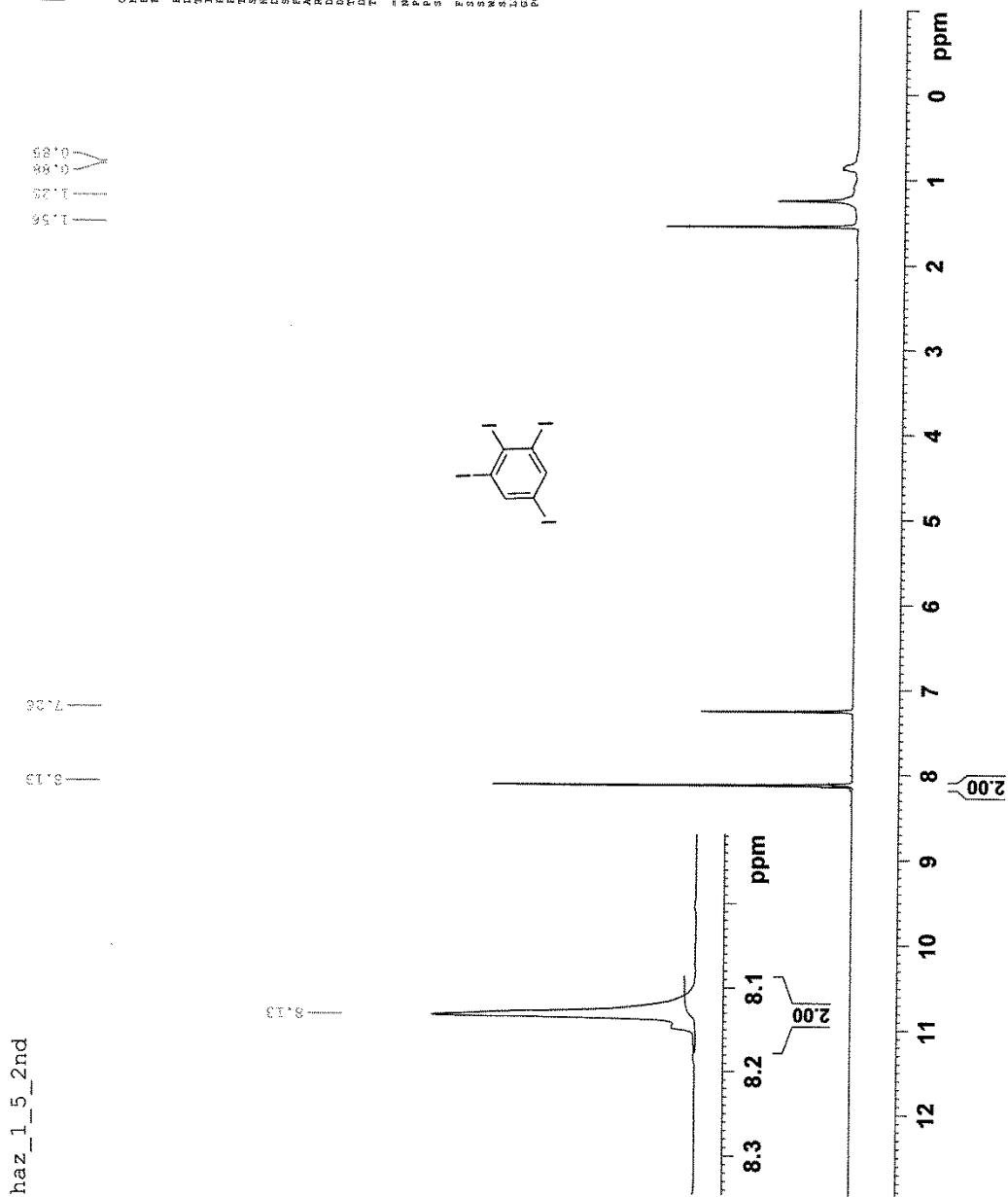
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 Time 12:40:08
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 PULPROG zgpg30
 SOLVENT CDCl3
 NS 28
 DS 4
 SWH 11990.407 Hz
 FIDRES 0.1822959 Hz
 AQ 2.7225011 sec
 RG 41.700 usec
 DW 6.00 usec
 DE 6.00 usec
 DI 1.0000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 SFO1 400.1310000 MHz
 F2 - Processing Parameters
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 SF 400.1300065 MHz
 SW 500
 LB 0
 GB 0
 PC 1.00

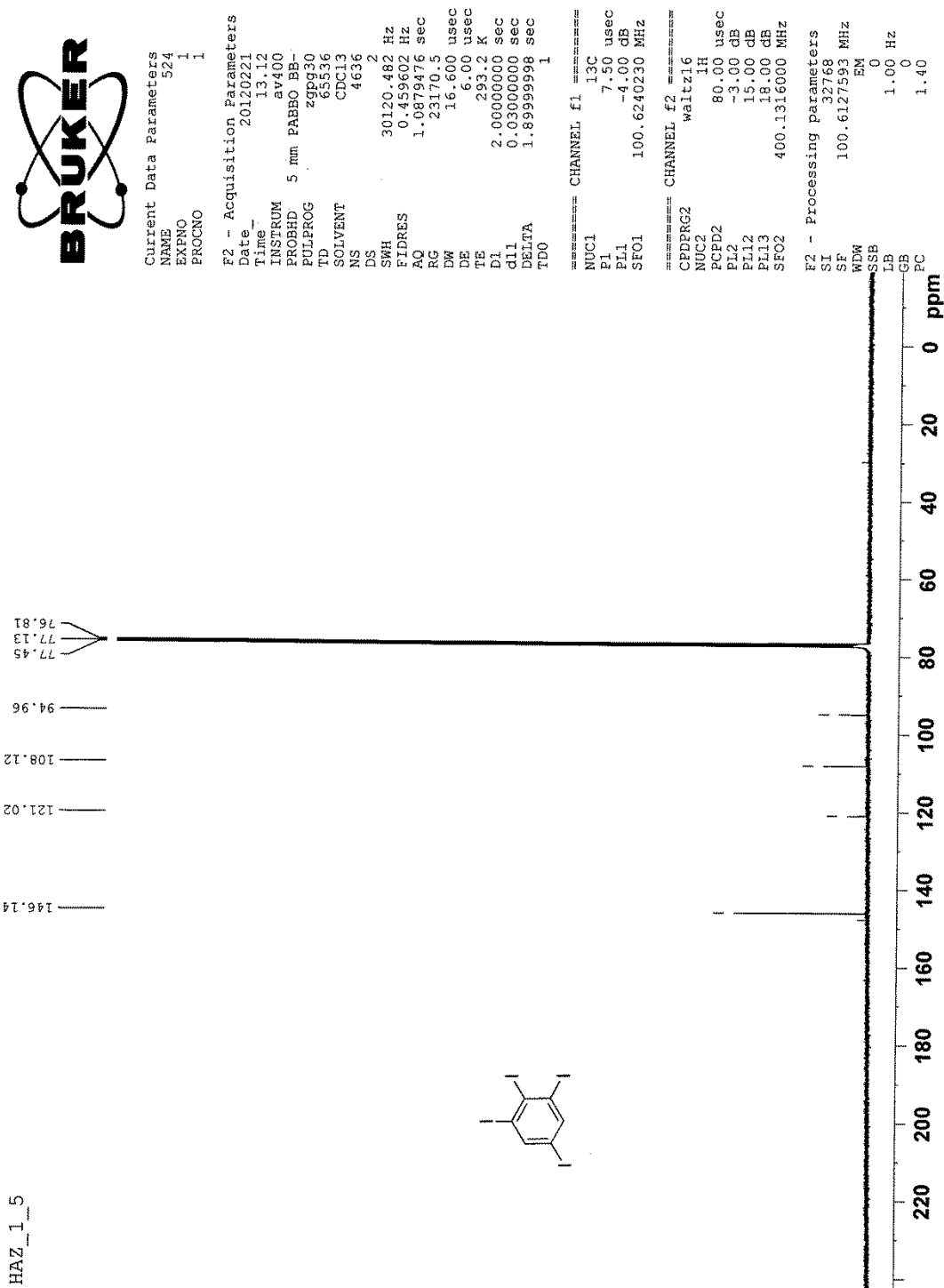


5.16 ^{13}C -NMR of 1,2,3-triiodo-5-methylbenzene (19) in $d\text{-CDCl}_3$ at 25 °C.

5.17 $^1\text{H-NMR}$ of 1,2,3,5-tetraiodobenzene (20) in $d\text{-CDCl}_3$ at 25 °C.

Current Data Parameters
 NAME D_384932
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 PROCNO 1
 F2 - Acquisition Parameters
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 Time_ 11:52:11
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 PROBHD 5 mm PABBO BB-
 P1PRG2 zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 17
 DS 1
 SWH 11990.407 Hz
 FIDRES 0.18295 Hz
 AQ 2.775241 sec
 RG 574.7
 DM 41.700 usec
 DE 6.00 usec
 TE 29.99
 D1 1.0000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 ^1H
 P 10.00 usec
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 SFO1 400.1310000 MHz
 F2 - Processing Parameters
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 SI 32768
 SSF 0
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 GC 1.00

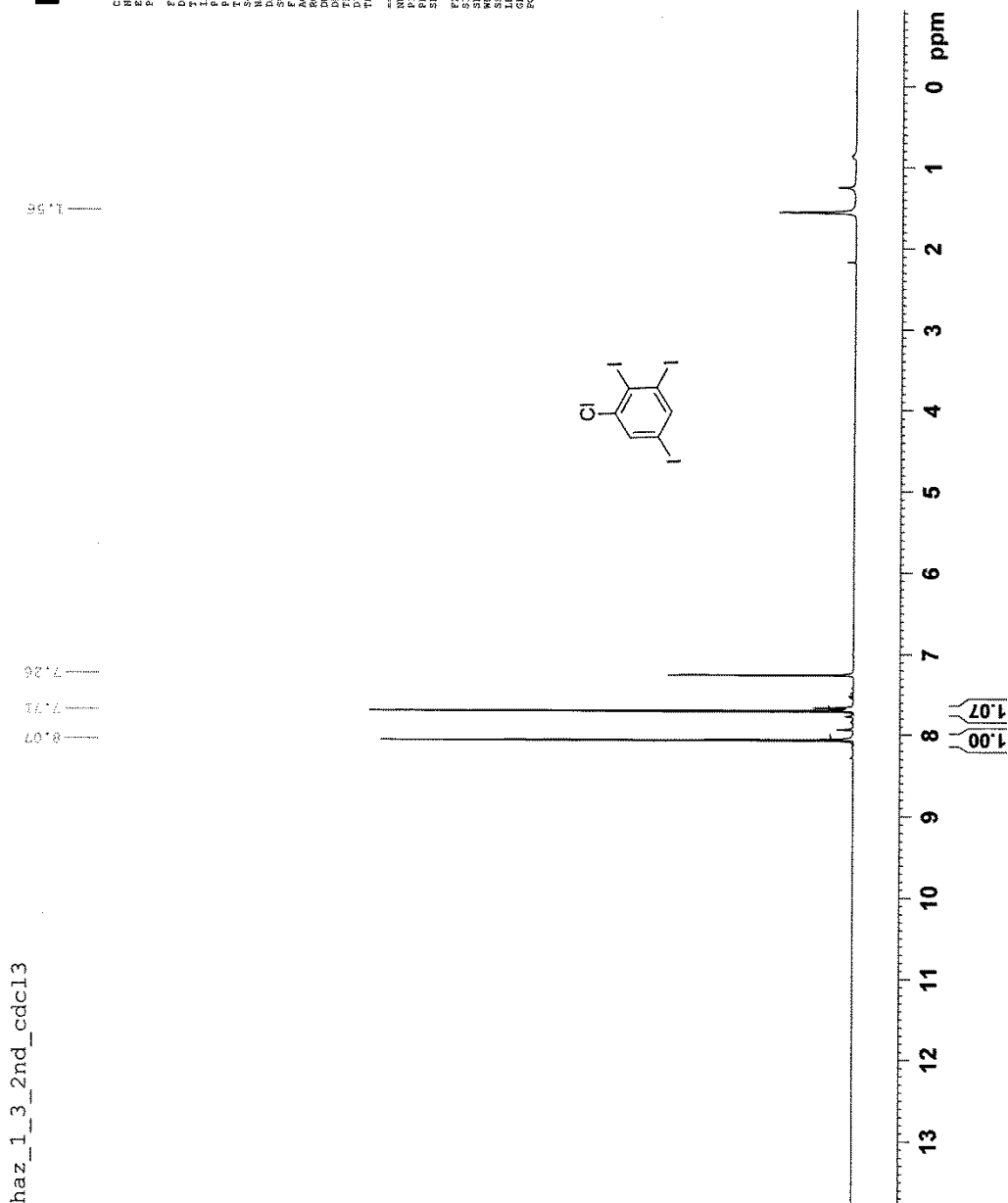


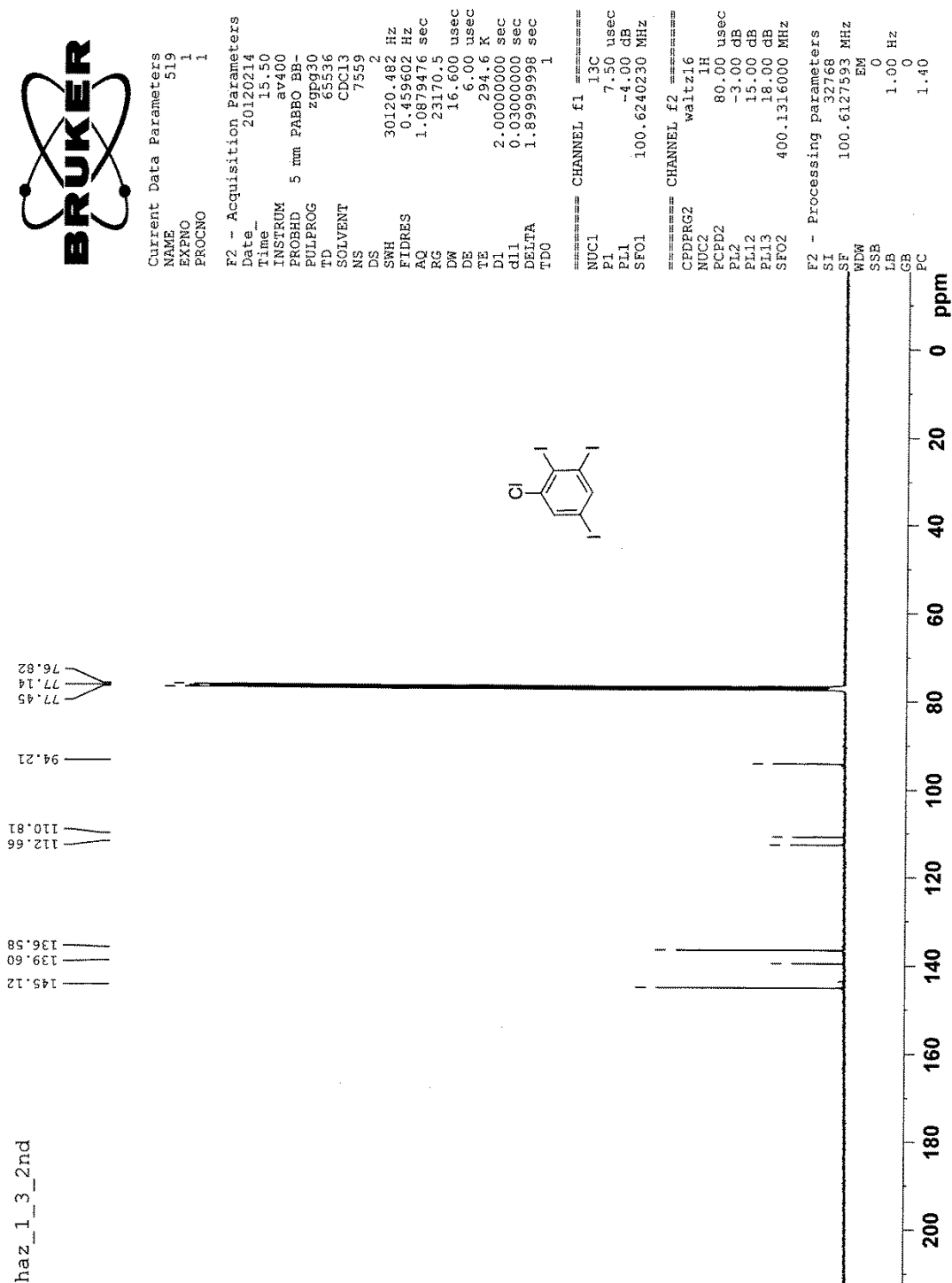
5.18 ¹³C-NMR of 1,2,3,5-tetraiodobenzene (20) in *d*-CDCl₃ at 25 °C.

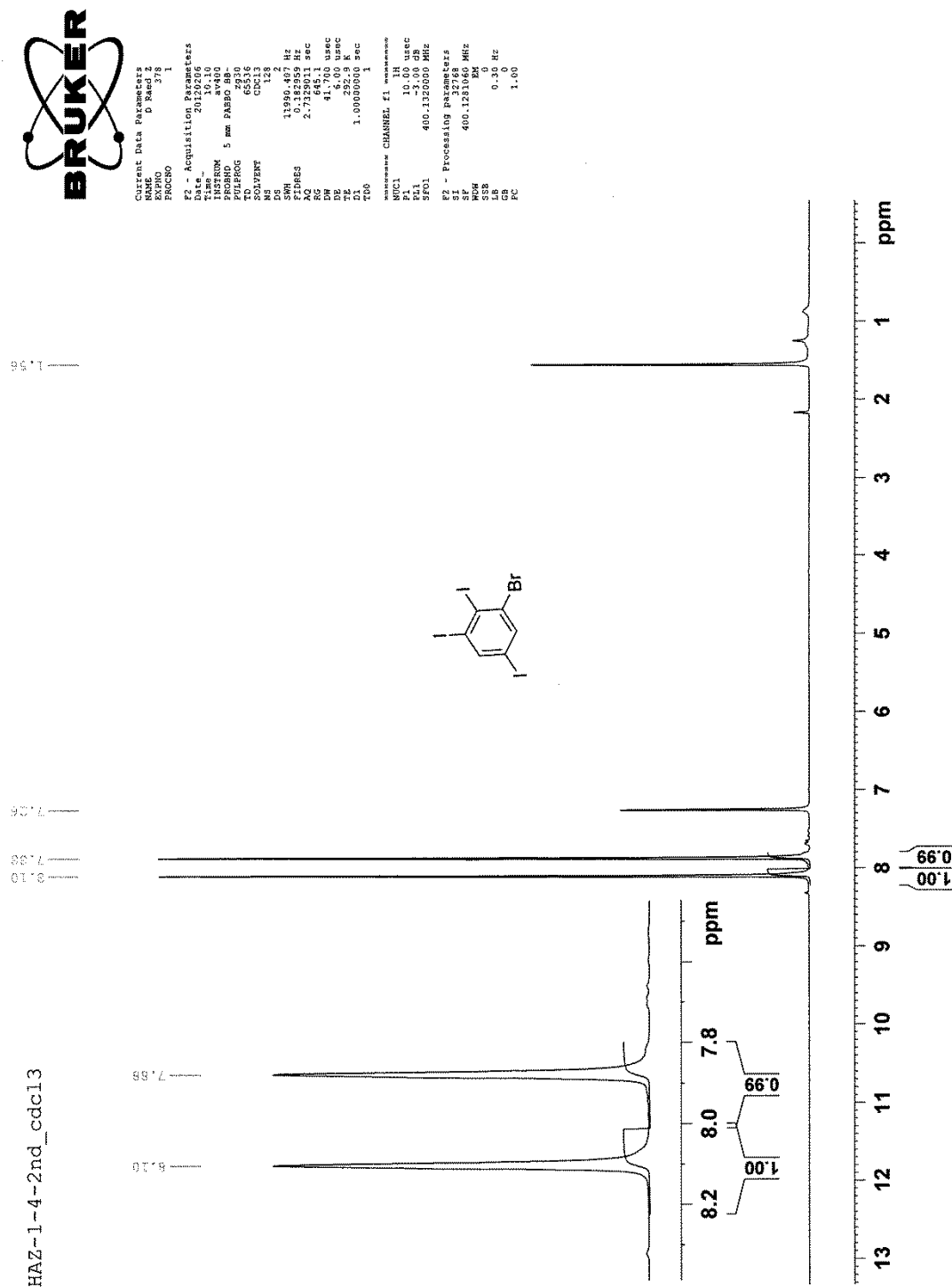
5.19 $^1\text{H-NMR}$ of 1-chloro-2,3,5-triodobenzene (22) in $d\text{-CDCl}_3$ at 25 °C.

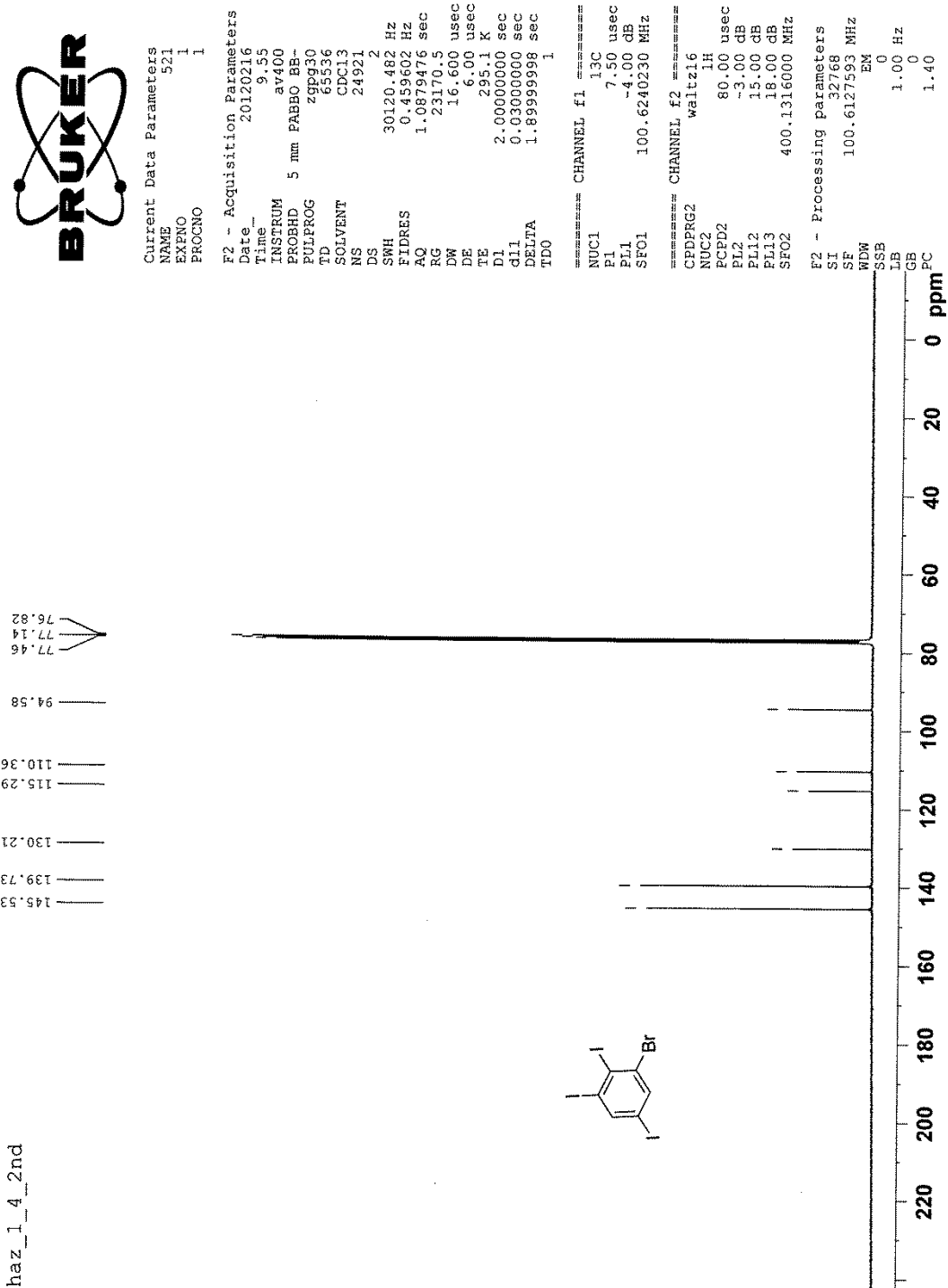
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 P1PRG0 3533
 TD 65536
 SOLVENT CDCl3
 NS 34
 DS 4
 SWH 11990.407 Hz
 FIDRES 0.182959 Hz
 AQ 2.772171 sec
 RG 574.1
 IQM 41.700 usec
 DE 8.00 usec
 TE 300.2 K
 D1 1.00600000 sec
 TDO 1

===== CHANNEL f1 =====
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 P1 15.00 usec
 PL1 -1.00 dB
 SFO1 400.130000 MHz
 F2 - Processing parameters
 SF 400.130000 MHz
 DS 4
 SSF 0
 LB 0.30 Hz
 GB 0
 EC 1.00



5.20 ^{13}C -NMR of 1-chloro-2,3,5-triiodobenzene (22) in *d*- CDCl_3 at 25 °C.

5.21 $^1\text{H-NMR}$ of 1-bromo-2,3,5-triiodobenzene (23) in $d\text{-CDCl}_3$ at 25 °C.

5.22 ¹³C-NMR of 1-bromo-2,3,5-triiodobenzene (23) in *d*-CDCl₃ at 25 °C.

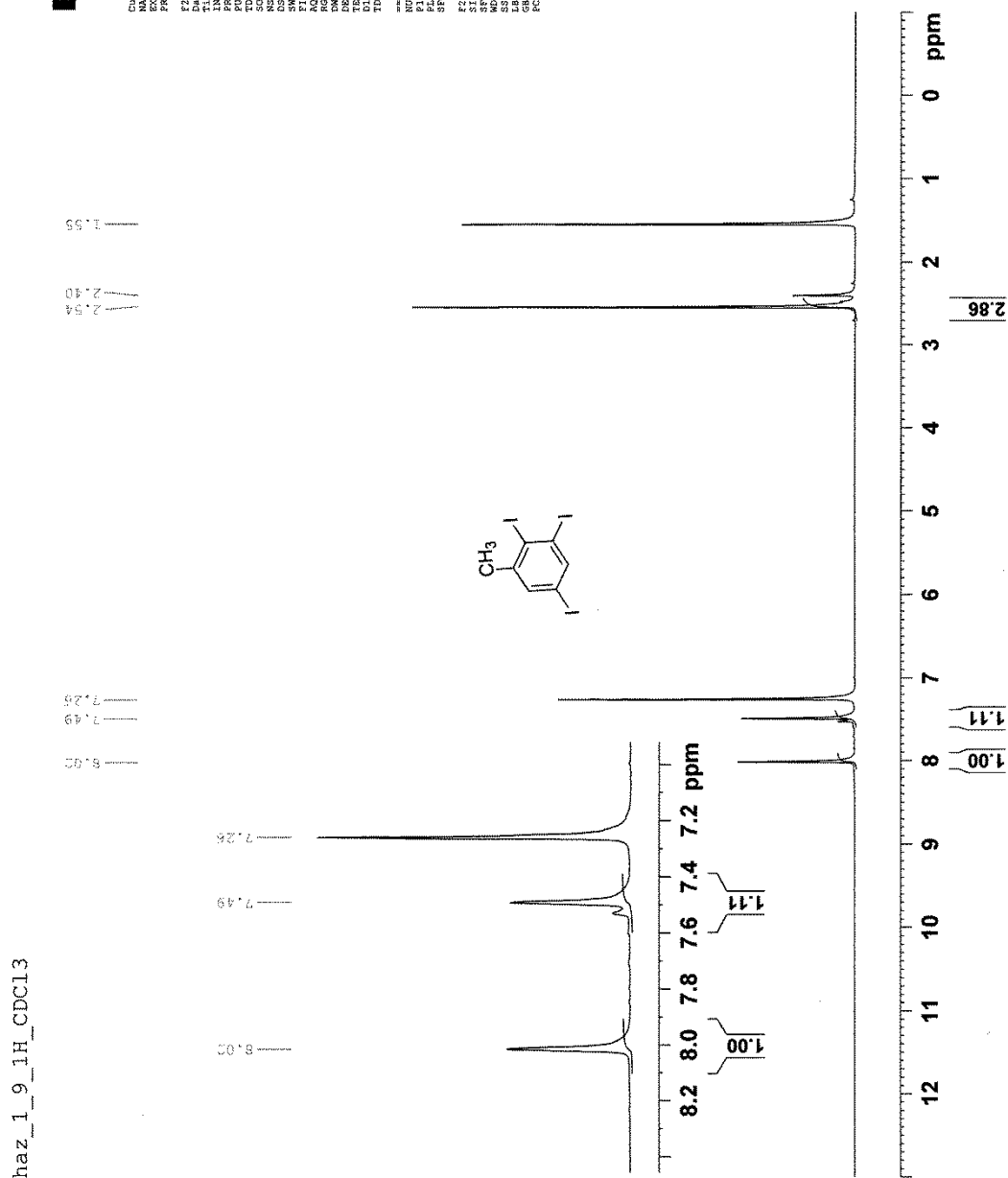
5.23 $^1\text{H-NMR}$ of 1,2,5-triiodo-3-methylbenzene (24) in $d\text{-CDCl}_3$ at 25 $^\circ\text{C}$.

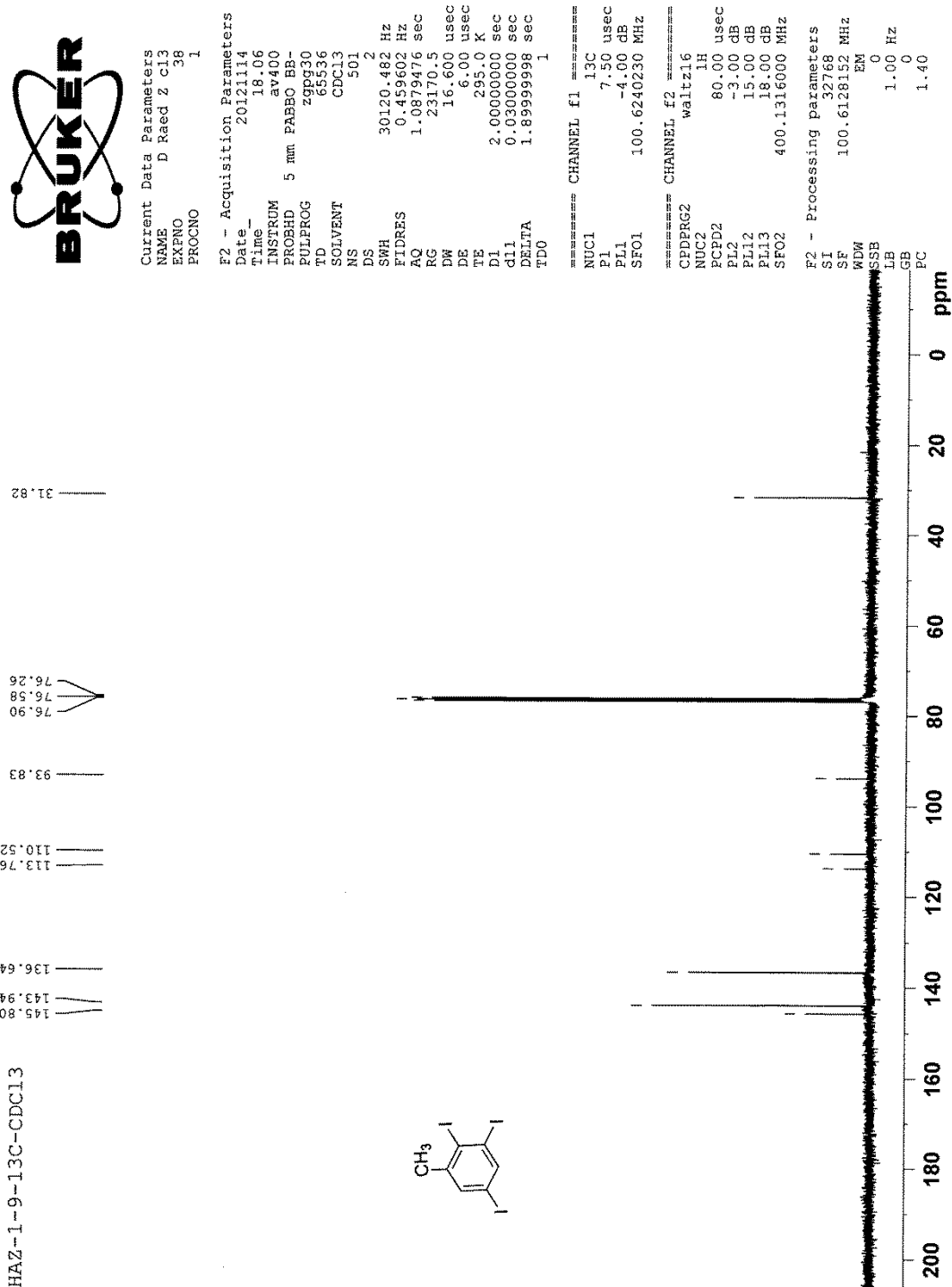
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 NAME D Base 2
 EXPNO 417
 PROCNO 1

F2 - Acquisition Parameters
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 ID 65316
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 AQ 2.7229011 sec
 RG 574.7
 DS 4
 SFO1 400.1300000 MHz
 DQ 6.00 usec
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 TD 1.00000000 sec

===== CHANNEL f1 =====
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 SFO1 400.1310000 MHz

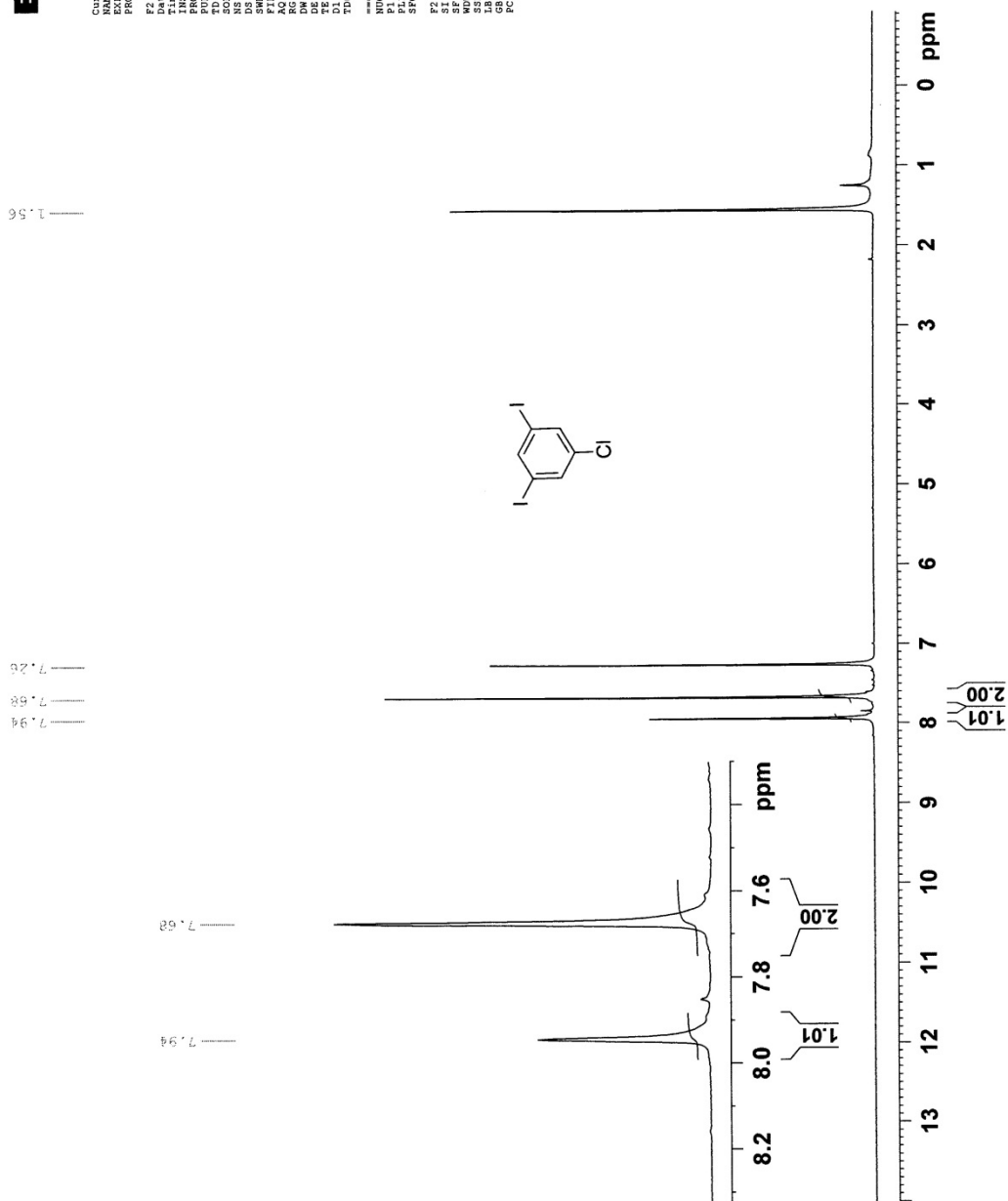
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 CC 0
 SC 1.00

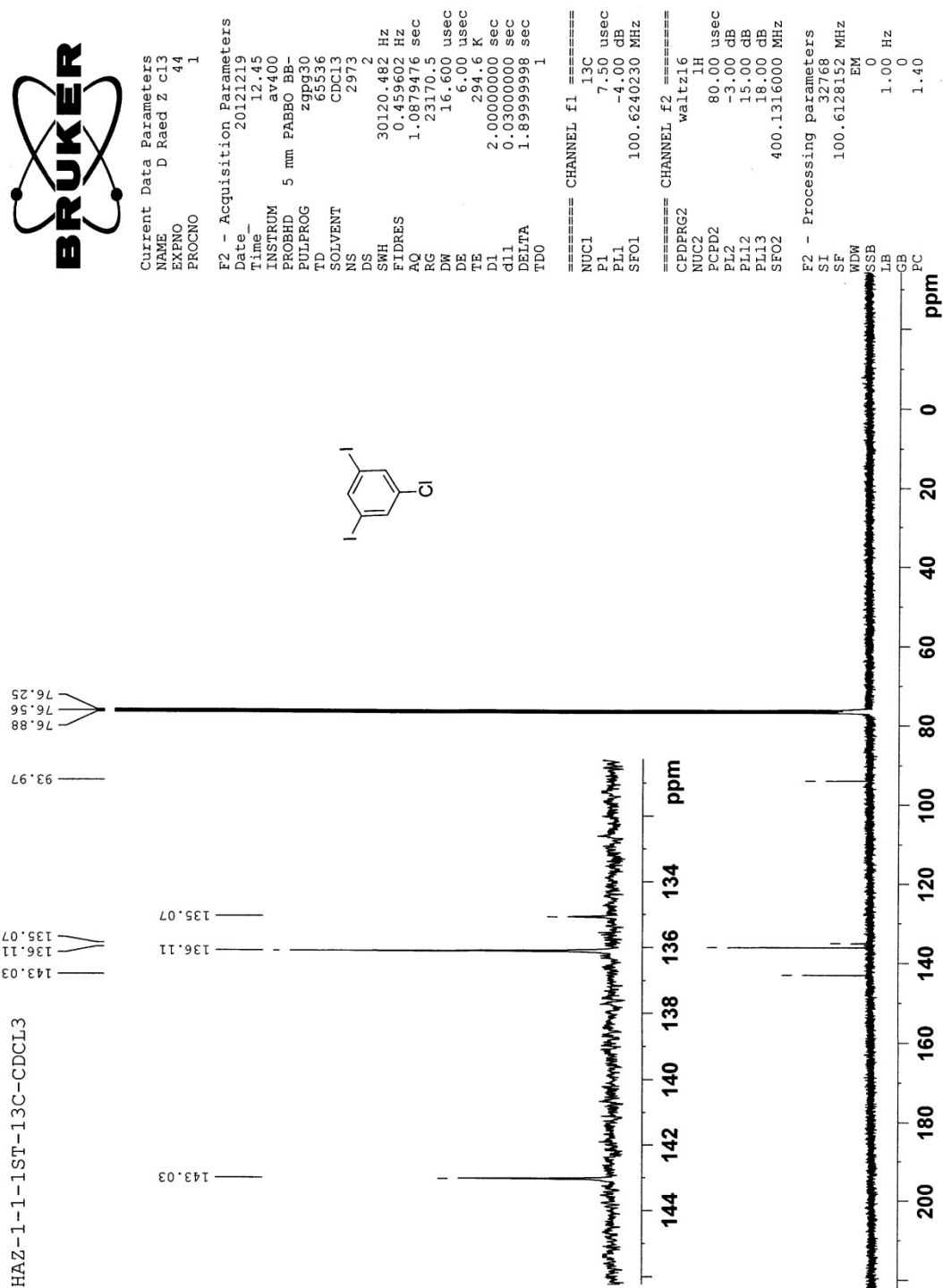


5.24 ^{13}C -NMR of 1,2,5-triiodo-3-methylbenzene (**24**) in *d*- CDCl_3 at 25 °C.

5.25 $^1\text{H-NMR}$ of 1-chloro-3,5-diiodobenzene (25) in $d\text{-CDCl}_3$ at 25 °C.

Current Data Parameters
 NAME D Read %
 EXPNO 527
 PROCNO 1
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 Date_ 201309
 Time_ 12:13
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6
 DS 2
 SWH 11990.407 Hz
 FIDRES 0.182955 Hz
 AQ 0.0200000 sec
 RG 279.74
 DM 41.700 usec
 DE 19.41 usec
 TE 294.1 K
 D1 1.00000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 ^1H
 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 400.1310000 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.130068 MHz
 WF 0
 SS 0
 LB 0.30 Hz
 GB 0
 PC 1.00

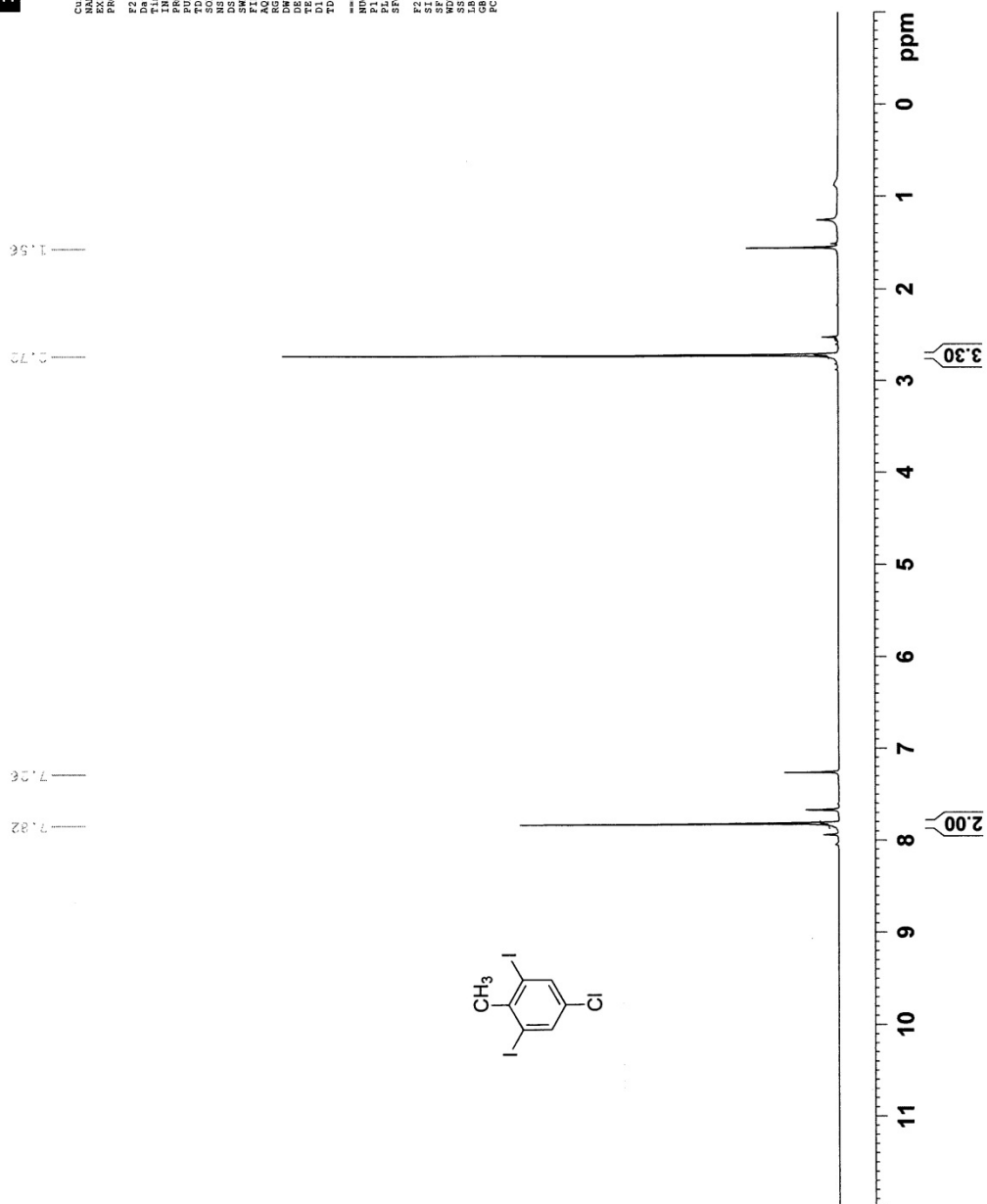
HAZ-1-1-1ST- $^1\text{H-CDCl}_3$ 

5.26 ¹³C-NMR of 1-chloro-3,5-diiodobenzene (25) in *d*-CDCl₃ at 25 °C.

5.27 $^1\text{H-NMR}$ of 5-chloro-1,3-diiodo-2-methylbenzene (26) in $d\text{-CDCl}_3$ at 25 °C.

Current Data Parameters
 NAME D_Head_2
 EXPNO 492
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20181223
 Time 18:23
 INSTRUM 5 mm PABEAV400
 PULPROG zgpg30
 FIDRES 0.000000
 TD 65536
 SOLVENT CDCl3
 DS 192
 SWH 11990.407 Hz
 FWHZ 655.360 Hz
 AQ 2.7329013 sec
 RG 287.4
 DW 41.700 usec
 DE 2.000 usec
 TE 296.0 K sec
 D1 1.0000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUCL 13C
 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 400.1310000 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1310000 MHz
 WDW EM
 SSB 0
 GB 0
 PC 1.00

AMZ-1-12-1H-CDCl3



5.28 ^{13}C -NMR of 5-chloro-1,3-diiodo-2-methylbenzene (26) in $d\text{-CDCl}_3$ at 25 °C.

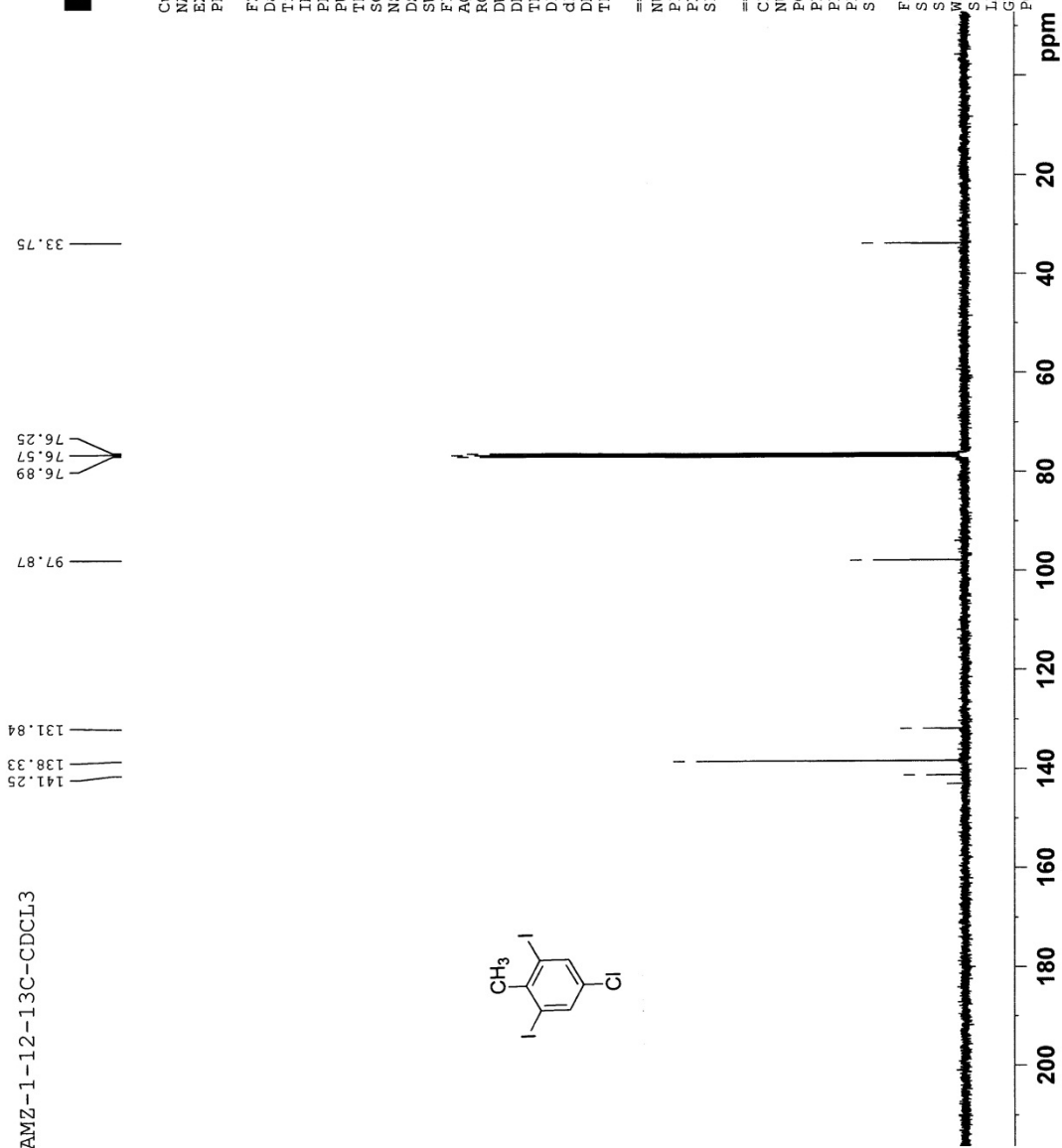
Current Data Parameters
 NAME D Raed 2 c13
 EXPNO 33
 PROCNO 1

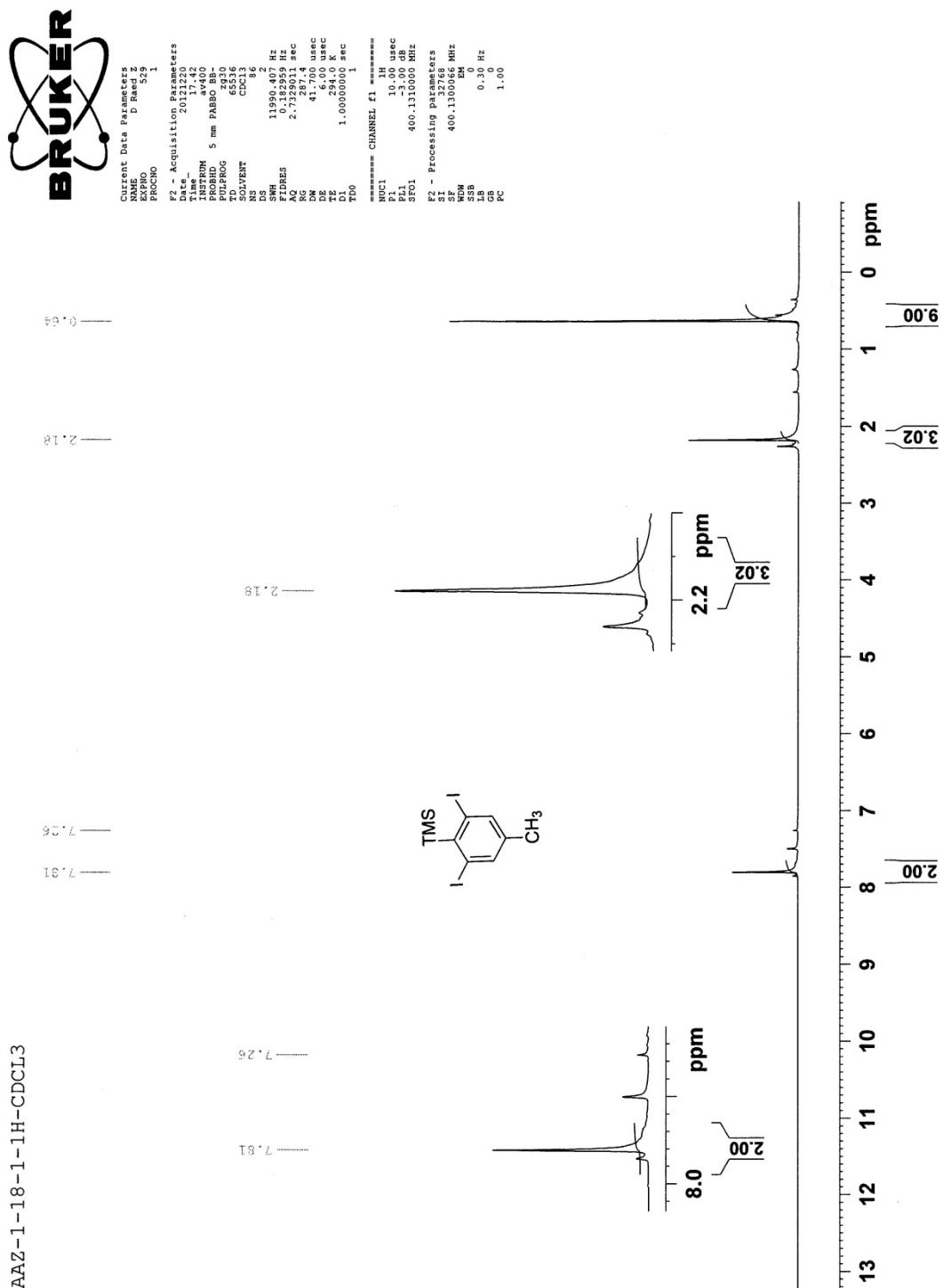
F2 - Acquisition Parameters
 Date_ 20121107
 Time 18.34
 INSTRUM av400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 502
 DS 2
 SWH 30120.482 Hz
 FIDRES 0.459602 Hz
 AQ 1.0879476 sec
 RG 23170.5
 DW 16.600 usec
 DE 6.00 usec
 TE 295.6 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.50 usec
 PL1 -4.00 dB
 SFO1 100.6240230 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.00 dB
 PL13 18.00 dB
 SFO2 400.1316000 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6128152 MHz
 EM 0
 WDW 0
 LB 1.00 Hz
 GB 0
 PC 1.40



5.29 $^1\text{H-NMR}$ of (2,6-diiodo-4-methylphenyl)trimethylsilane (30) in $d\text{-CDCl}_3$ at 25 $^\circ\text{C}$.

5.30 ^{13}C -NMR of (2,6-diiodo-4-methylphenyl)trimethylsilane (30) in $d\text{-CDCl}_3$ at 25 °C.