10.1071/CH13324_AC

Australian Journal of Chemistry 2013, 66(12), 1570-1575

Supplementary Material

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

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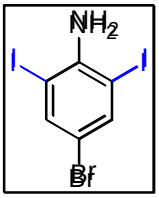
1. General Information

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 F_{254}. 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, mutiplet. 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

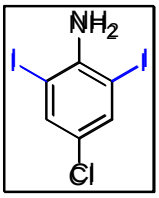
An appropriate aniline derivative (7.9 mmol, 1.0 equiv.), iodine (17.3 mmol, 2.2 equiv) and silver (I) sulfate (3.2 mmol, 1.1 equiv) were dissolved in ethanol (40 mL) and stirred for 24 h at room temperature. The mixture was filtered over Celite® 545 to remove AgI precipitate. Water (200 mL) was added 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)**



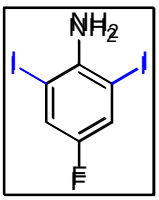
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **74%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Synthesis* **2006** *(20),* 3467-3477 *and Org. Biomol. Chem.* **2011***, V.9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

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



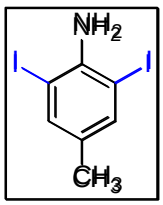
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **72%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *J. Org. Chem.* **2011***; 76,* 2123-2131.

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



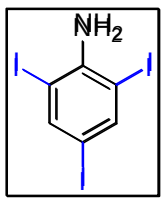
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **31%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.44 (d, 2H, *J* = 7.4 Hz), 4.46 (s, 2H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 154.8, 152.3, 142.7, 125.6, 125.3, 78.9, 78.8. **IR** (cast film, cm-1) 3377, 3258, 3037, 2957, 1616, 1584, 1419, 1004, 900. **Mp**: 67-69 °C. **HRMS** (CI) *m/z* for $C_6H_4FI_2N$ $[M-H]^-$: calcd. 362.8417; found, 362.8434.

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



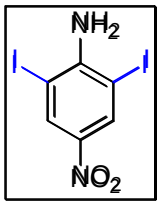
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **56%** yield as a brownish solid. The spectroscopic data for this compound are matched the previous report by *Int. J. ChemTech. Res.* **2009***;* 1 *(4):* 1005-1007.

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



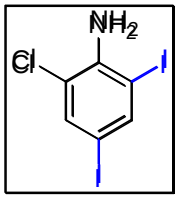
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **51%** yield as a brown solid. The spectroscopic data for this compound are matched the previous report by *Russ. Chem. Bull., Int. Ed.* **2004***; 53* (2), 471-473.

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



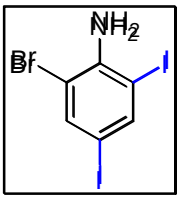
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **32%** yield as a yellow solid. The spectroscopic data for this compound are matched the previous report by *Tetrahedron*. **2002**; *58*: 3977-3983.

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



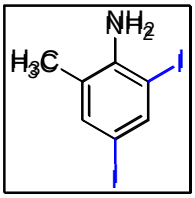
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **88%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.79 (s, 1H), 7.50 (s, 1H), 4.56 (s, 2H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 144.4, 143.2, 137.2, 118.2, 83.7, 77.4. **IR** (cast film, cm^{-1}) 3416, 3387, 3055, 1652, 1167, 1121, 950. **Mp**: 71-73 °C. **HRMS** (CI) *m/z* for $C_6H_4ClI_2N$ $[M-H]^-$: calcd. 377.8122; found, 377.8179.

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



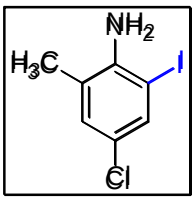
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **89%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.84 (s, 1H), 7.67 (s, 1H), 4.64 (s, 2H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 145.1, 143.9, 140.1, 107.5, 83.4, 77.9. **IR** (cast film, cm^{-1}) 3412, 3314, 2954, 1622, 1095, 930. **Mp**: 65-67 °C. **HRMS** (CI) *m/z* for $C_6H_4BrI_2N$ $[M+H]^-$: 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)



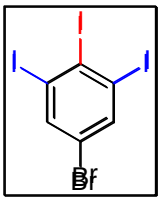
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **28%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.50 (s, 1H), 7.02 (s, 1H), 4.06 (s, 2H), 2.20 (s, 3H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 143.6, 135.5, 130.1, 123.2, 122.8, 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 C_7H_7ClIN $[M-H]^-$: 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.

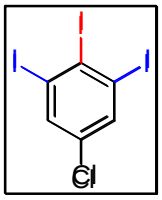
A solution of $NaNO_2$ (1.53 mmol, 1.1 equiv.) in water (1.0 mL) was added dropwise to a mixture of aniline salt in water (3.5 mL) and concentrated hydrochloric acid (1.5 mL) below 5 °C, and the mixture was stirred for 10 min. A solution of potassium iodide (2.1 mmol, 1.5 equiv.) in water (1.0 mL) was 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)



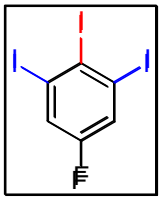
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **81%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.97 (s, 2H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 141.7, 140.2, 122.6, 106.7. **IR** (cast film, cm^{-1}) 2987, 1642, 1546, 1051, 860. **Mp**: 138-140 °C. **HRMS** (CI) *m/z* for $C_6H_2BrI_3$ [M^+]: calcd. 533.6474; found, 533.6199.

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



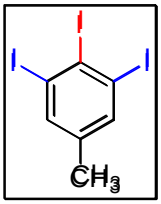
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **88%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.86 (s, 2H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 138.3, 135.3, 119.2, 106.8. **IR** (cast film, cm^{-1}) 3025, 1633, 1492, 894. **Mp**: 118-120 °C. **HRMS** (CI) *m/z* for $C_6H_2ClI_3$ [M^+]: calcd. 489.6979; found, 489.6939.

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



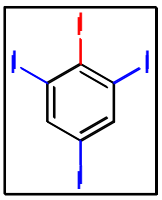
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **45%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.62 (d, 2H, *J* = 4.0 Hz). δ_C (100 MHz, *d*-$CDCl_3$) δ: 161.8, 159.3, 126.2, 125.9, 115.1, 105.5, 105.4. **IR** (cast film, cm^{-1}) 3065, 2941, 1596, 1435, 1187, 852. **Mp**: 127-129 °C. **HRMS** (CI) *m/z* for $C_6H_2FI_3$ [M^+]: calcd. 473.7275; found, 473.7164.

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



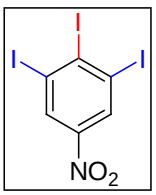
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **59%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.69 (s, 1H), 2.17 (s, 3H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 140.9, 139.1, 116.2, 106.2, 19.4. **IR** (cast film, cm^{-1}) 3149, 2985, 1614, 1587, 1215, 1146, 840. **Mp**: 113-115 °C. **HRMS** (CI) *m/z* for $C_7H_5I_3$ [M^+]: calcd. 469.8286; found, 469.9000.

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



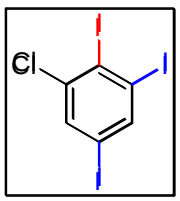
The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **46%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 8.13 (s, 2H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 146.1, 121.0, 108.1, 94.9. **IR** (cast film, cm^{-1}) 2987, 1534, 1145, 1009, 873. **Mp**: 133-135 °C. **HRMS** (CI) *m/z* for $C_6H_2I_4$ [M^+]: calcd. 581.6335; found, 581.6158.

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



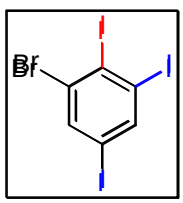
The title compound was prepared using the general procedure for halogenation of aniline derivatives and isolated in **21%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *J. Am. Chem. Soc.*, **2007**, *129(16), 4886-4887* and *J. Labelled Compd. Rad.*, **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*-$CDCl_3$) δ: 8.07 (s, 1H), 7.71 (s, 1H). δ_C (100 MHz, *d*-$CDCl_3$) 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 $C_6H_2ClI_3$ [M^+]: calcd. 489.6979; found, 489.6775.

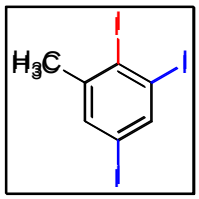
3.8 Synthesis of 1-bromo-2,3,5-triiodobenzene (23)



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*-$CDCl_3$) 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 $C_6H_2BrI_3$ [M^+]: calcd. 533.6474; found, 533.6423.

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

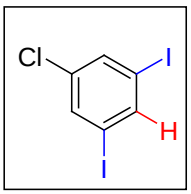


The title compound was prepared using the general procedure for diazotization aniline derivatives and isolated in **45%** yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 8.00 (s, 1H), 7.49 (s, 1H), 2.54 (s, 3H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 145.8, 143.9, 136.6, 113.7, 110.5, 93.8, 31.8. **IR** (cast film, cm^{-1}) 3125, 2945, 2913, 1642, 1574, 1351, 1121, 931. **Mp**: 114-116 °C. **HRMS** (CI) *m/z* for $C_7H_5I_3$ [M^+]: calcd. 469.8286; found, 469.8613.

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

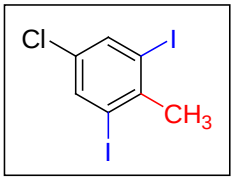
To a solution of triiodoarene (0.42 mmol) in 15 mL of a mixture of THF at −78 °C was added dropwise isopropyl magnesium chloride (2M in THF, 0.23 mL, 0.46 mmol). The mixture was stirred at that temperature for 2 h and then, electrophile was added. The solution was slowly warmed to room temperature and 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)



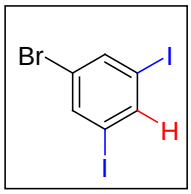
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 65% yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.94 (s, 1H), 7.68 (s, 2H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 143.0, 136.1, 135.1, 93.9. **IR** (cast film, cm^{-1}) 2945, 2916, 1623, 1586, 1414, 1125, 762. **Mp**: 131-133 °C. **HRMS** (CI) *m/z* for $C_6H_3ClI_2$ [M^+]: calcd. 363.8013; found, 363.7996.

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



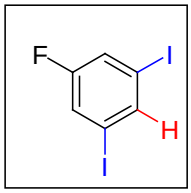
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 47% yield as a white solid. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.82 (s, 2H), 2.72 (s, 3H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 141.2, 138.3, 131.8, 97.8, 33.7. **IR** (cast film, cm^{-1}) 3148, 2975, 2846, 1634, 1573, 1243, 1147, 876. **Mp**: 145-147 °C. **HRMS** (CI) *m/z* for $C_7H_5ClI_2$ [M^+]: calcd. 377.8169; found, 377.8144.

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



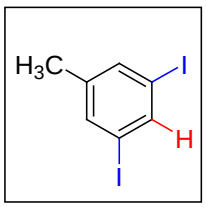
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 68% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Org. Biomol. Chem.,* **2011***, 9(12),* 4440-4443.

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



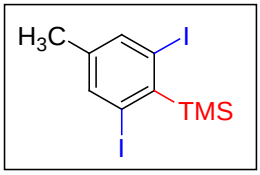
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 71% yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Revue Roumaine d Chimie,* **1989***, 34(3),* 807-810.

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.

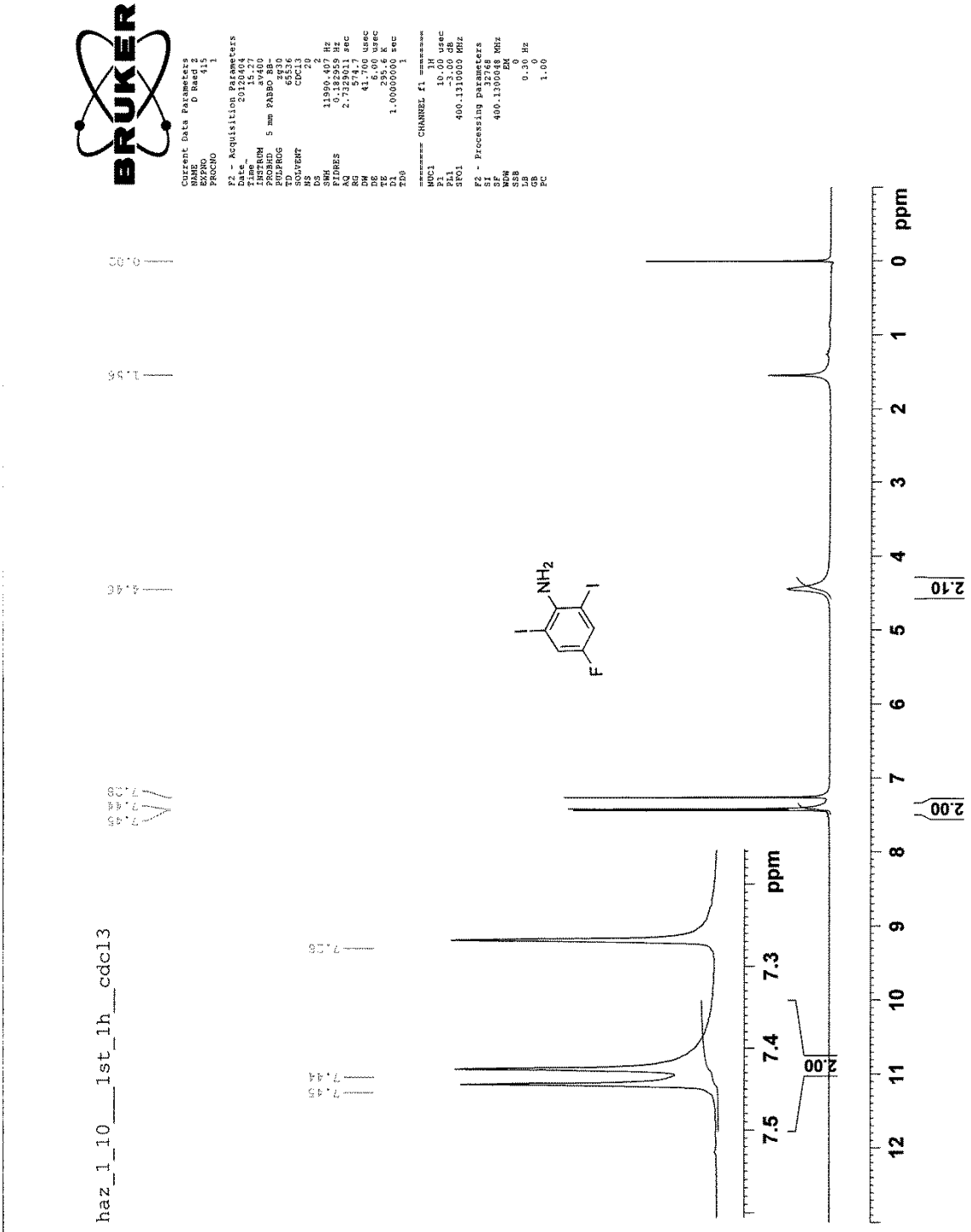
4.6 Synthesis of (2,6-diiodo-4-methylphenyl)trimethylsilane (30)



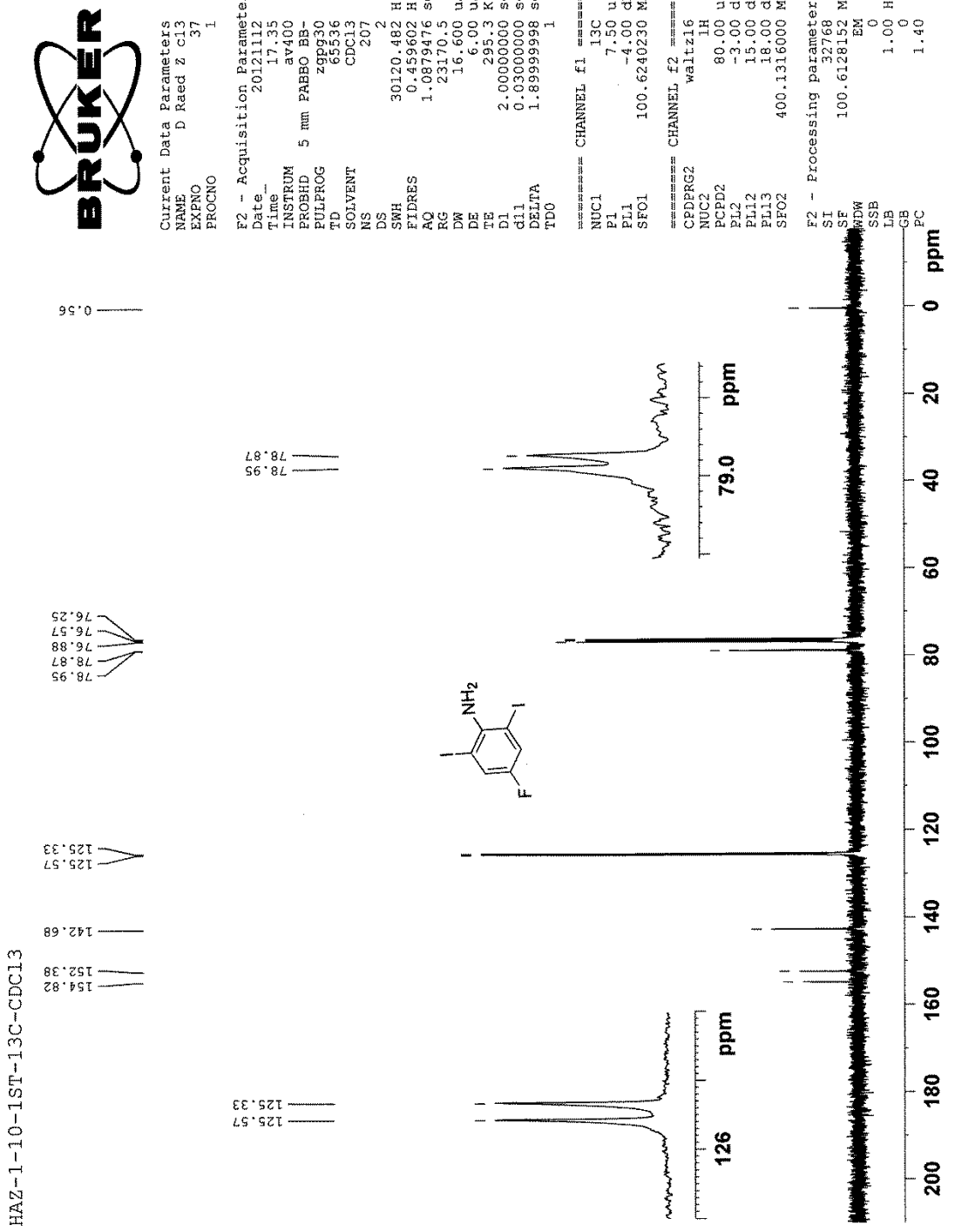
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 39% yield as colorless oil. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.81 (s, 2H), 2.18 (s, 3H), 0.64 (s, 9H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 141.8, 141.2, 136.9, 102.6, 19.1, 4.53. **IR** (cast film, cm^{-1}) 3201, 2918, 2894, 1687, 1548, 1423, 1250, 1146, 944. **HRMS** (CI) *m/z* for $C_{10}H_{14}I_2Si$ [M^+]: calcd. 415.8954; found, 415.8913.

5. NMR Spectra for New Compounds

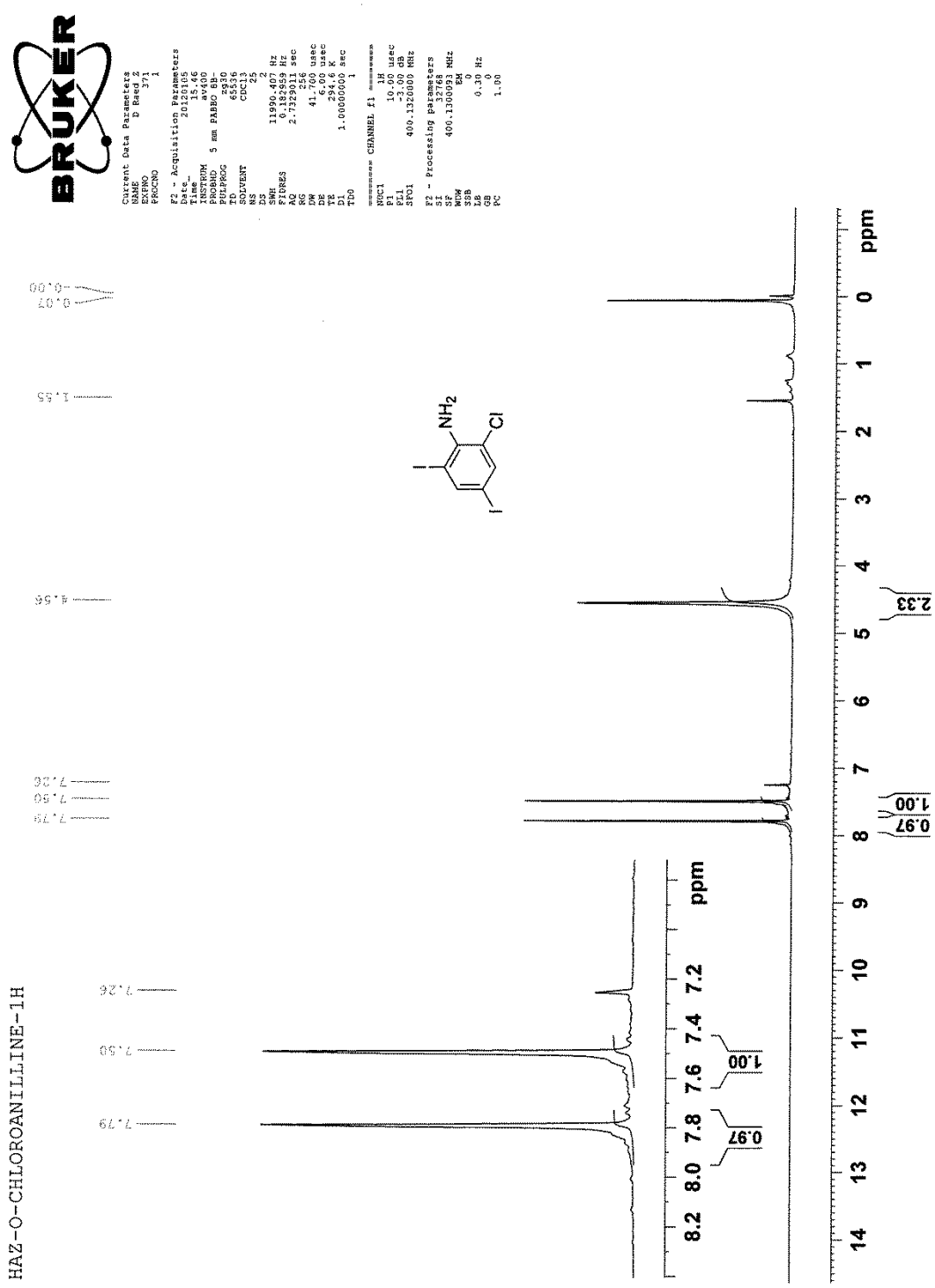
5.1 ^{1}H-NMR of 4-fluoro-2,6-diiodoaniline in (6) *d*-$CDCl_3$ at 25 ^{o}C.



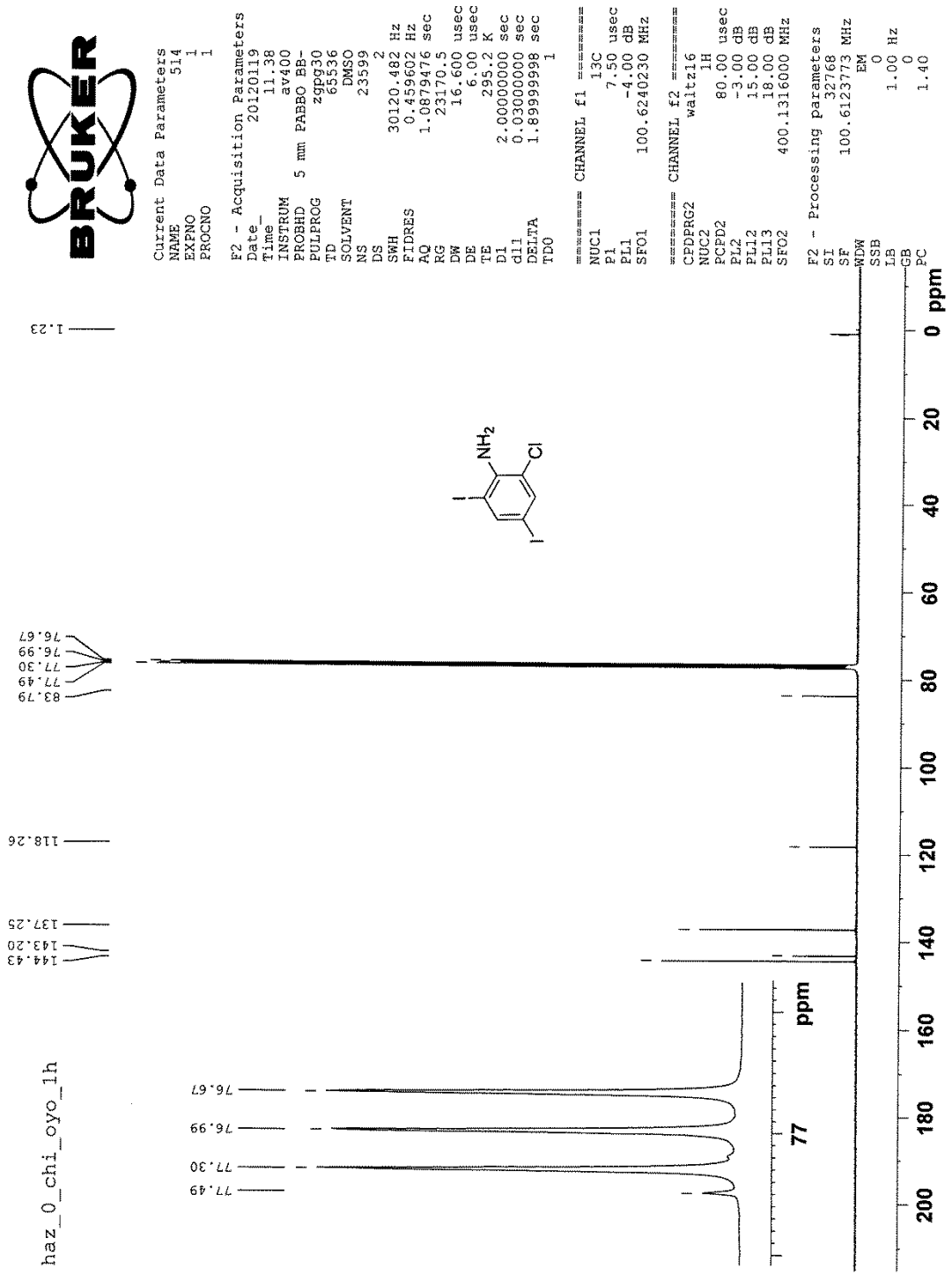
5.2 ^{13}C-NMR of 4-fluoro-2,6-diiodoaniline (6) in *d*-$CDCl_3$ at 25 ^{o}C.



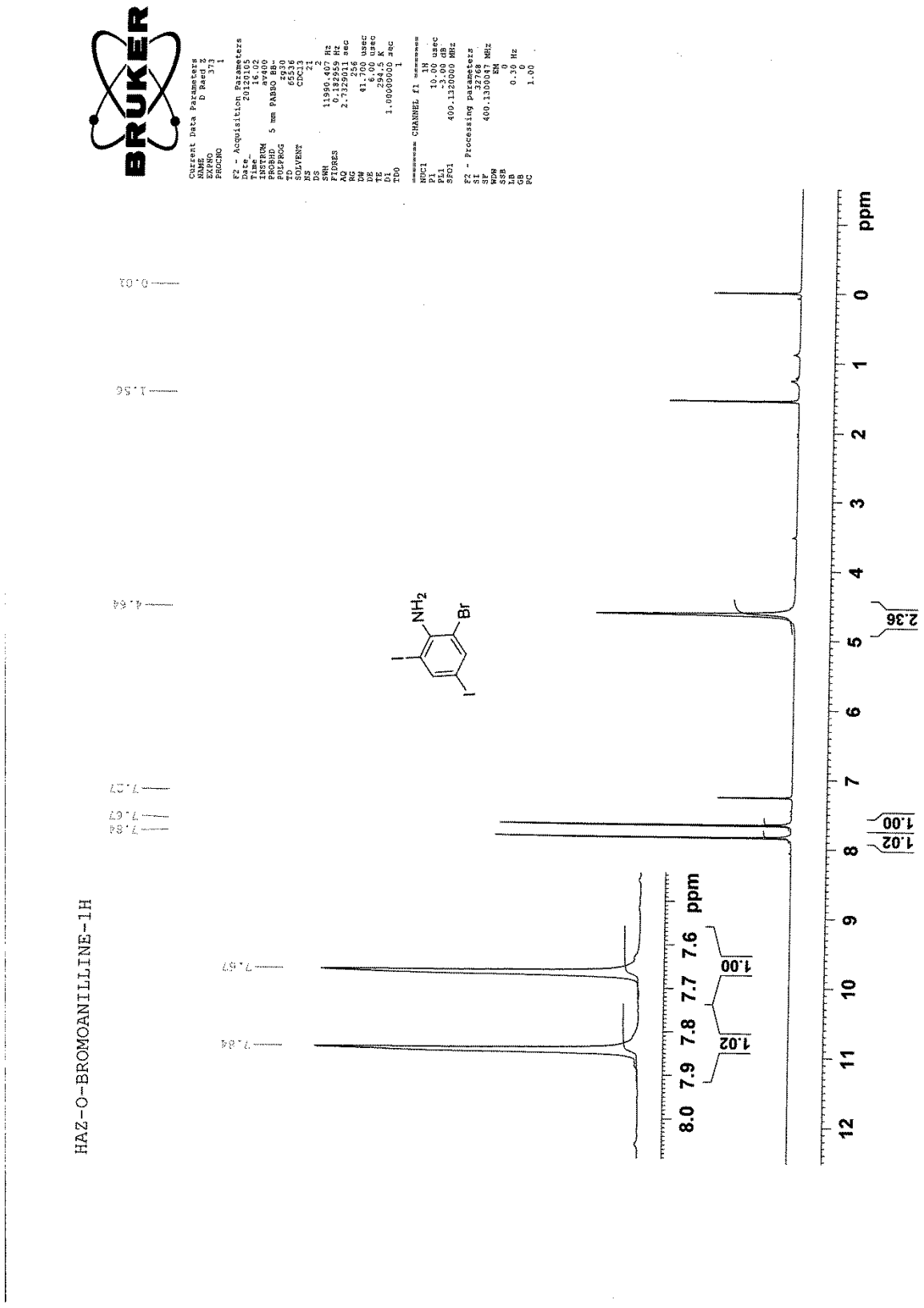
5.3 ^{1}H-NMR of 2-chloro-4,6-diiodoaniline (10) in *d*-$CDCl_3$ at 25 ^{o}C.



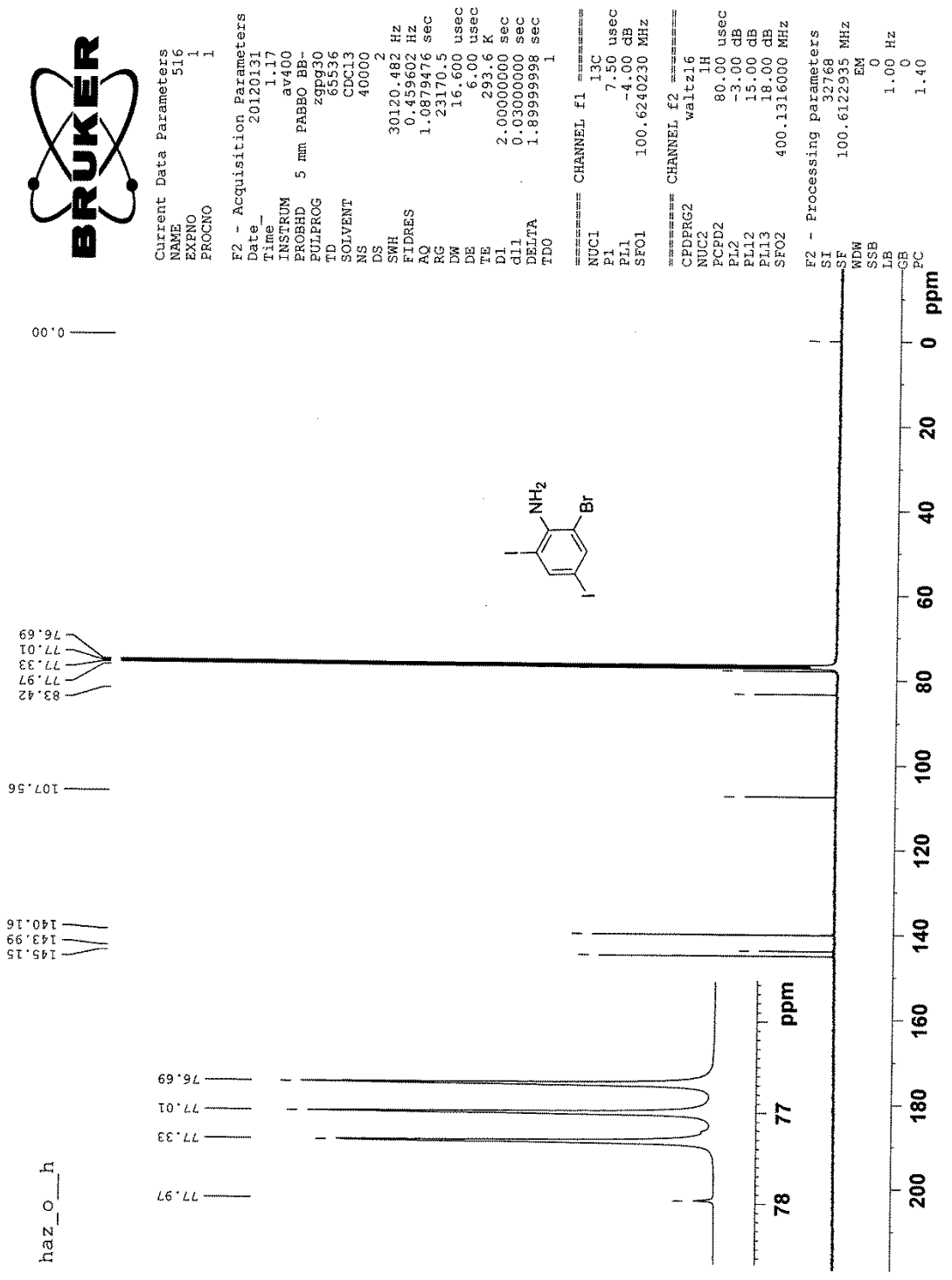
5.4 ^{13}C-NMR of 6-chloro-2,4-diiodoaniline (10) in *d*-$CDCl_3$ at 25 ^{o}C.



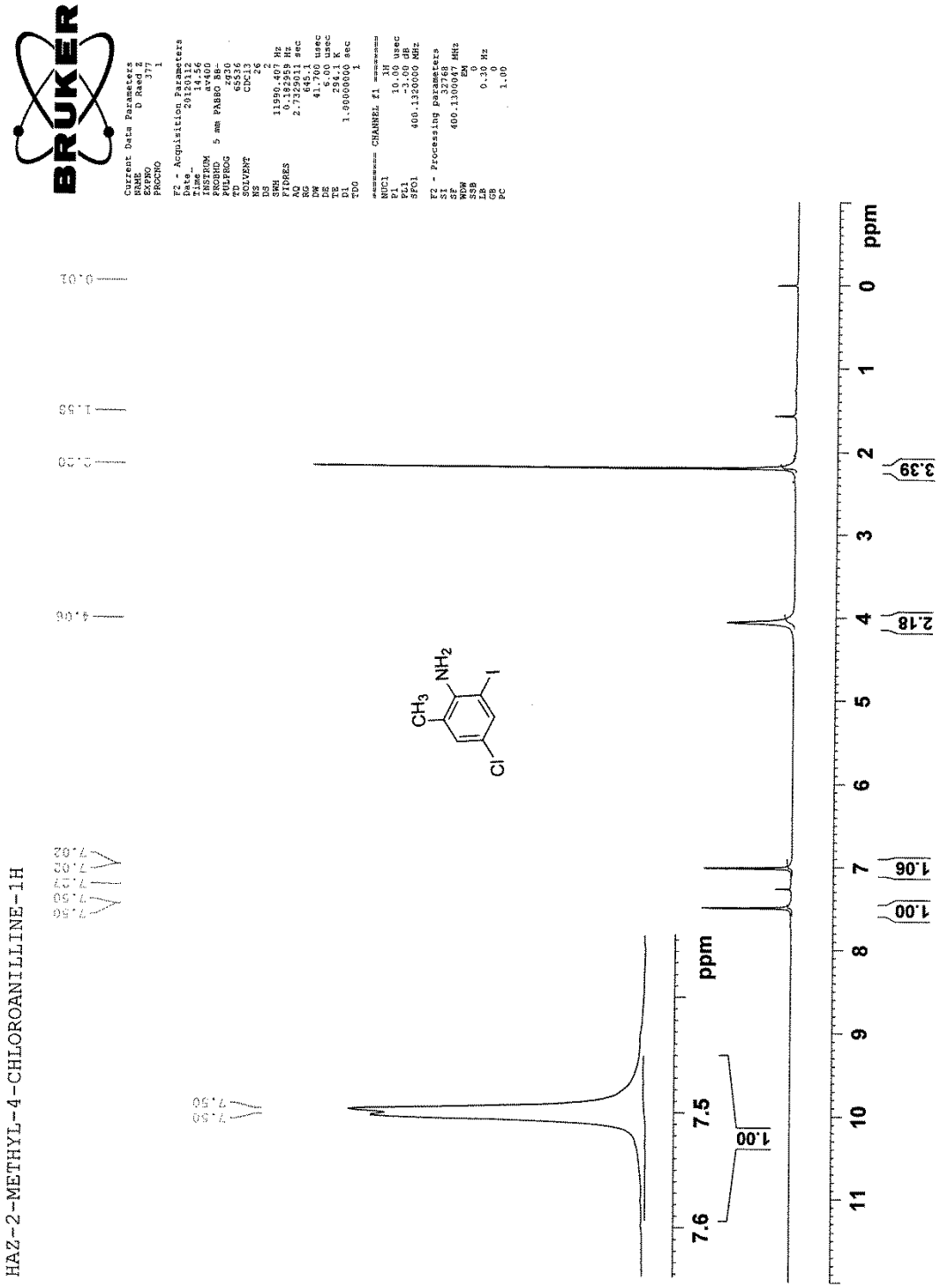
5.5 ^{1}H-NMR of 6-bromo-2,4-diiodoaniline (11) in *d*-$CDCl_3$ at 25 ^{o}C.



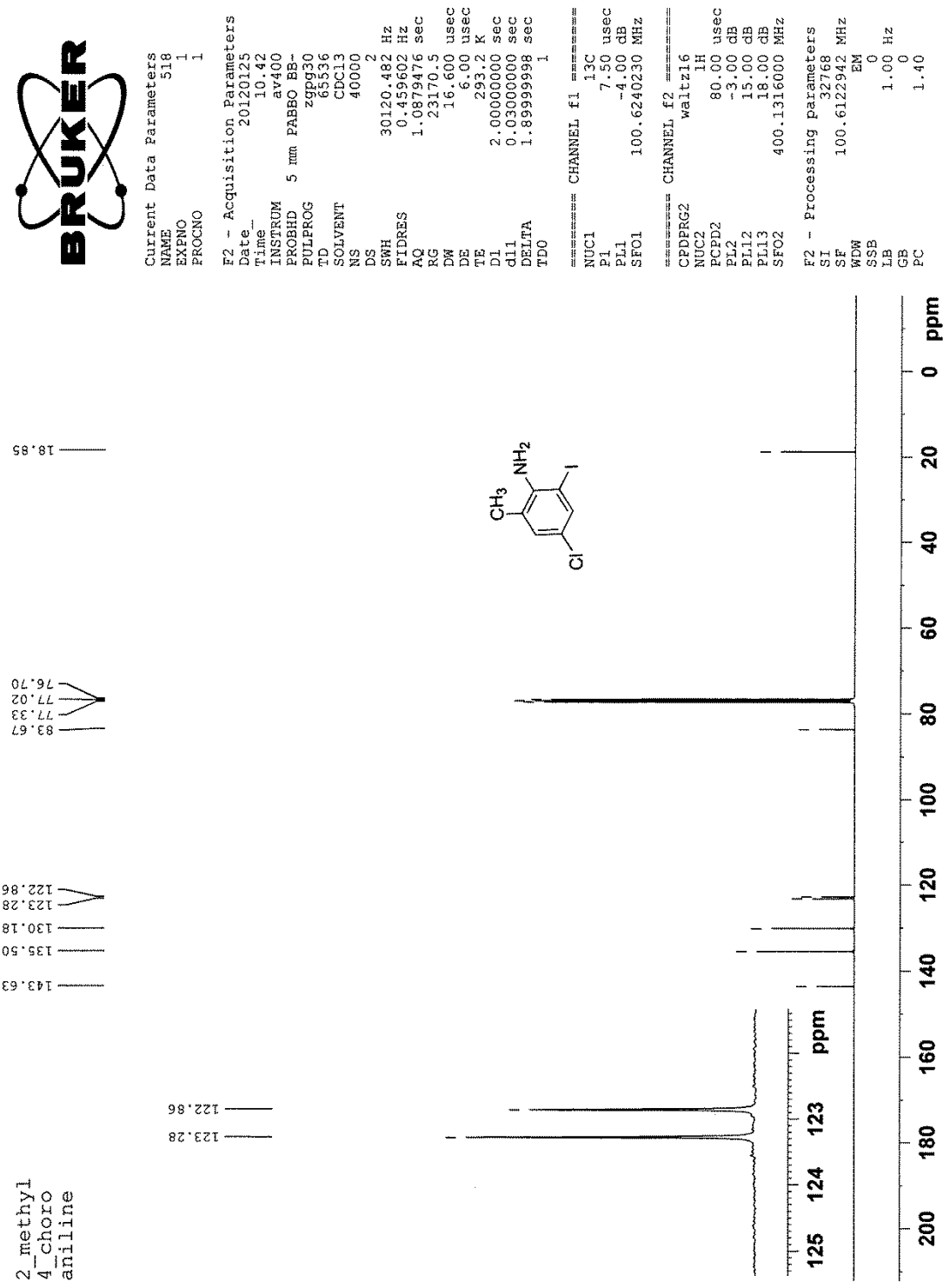
5.6 ^{13}C-NMR of 6-bromo-2,4-diiodoaniline (11) in *d*-$CDCl_3$ at 25 ^{o}C.



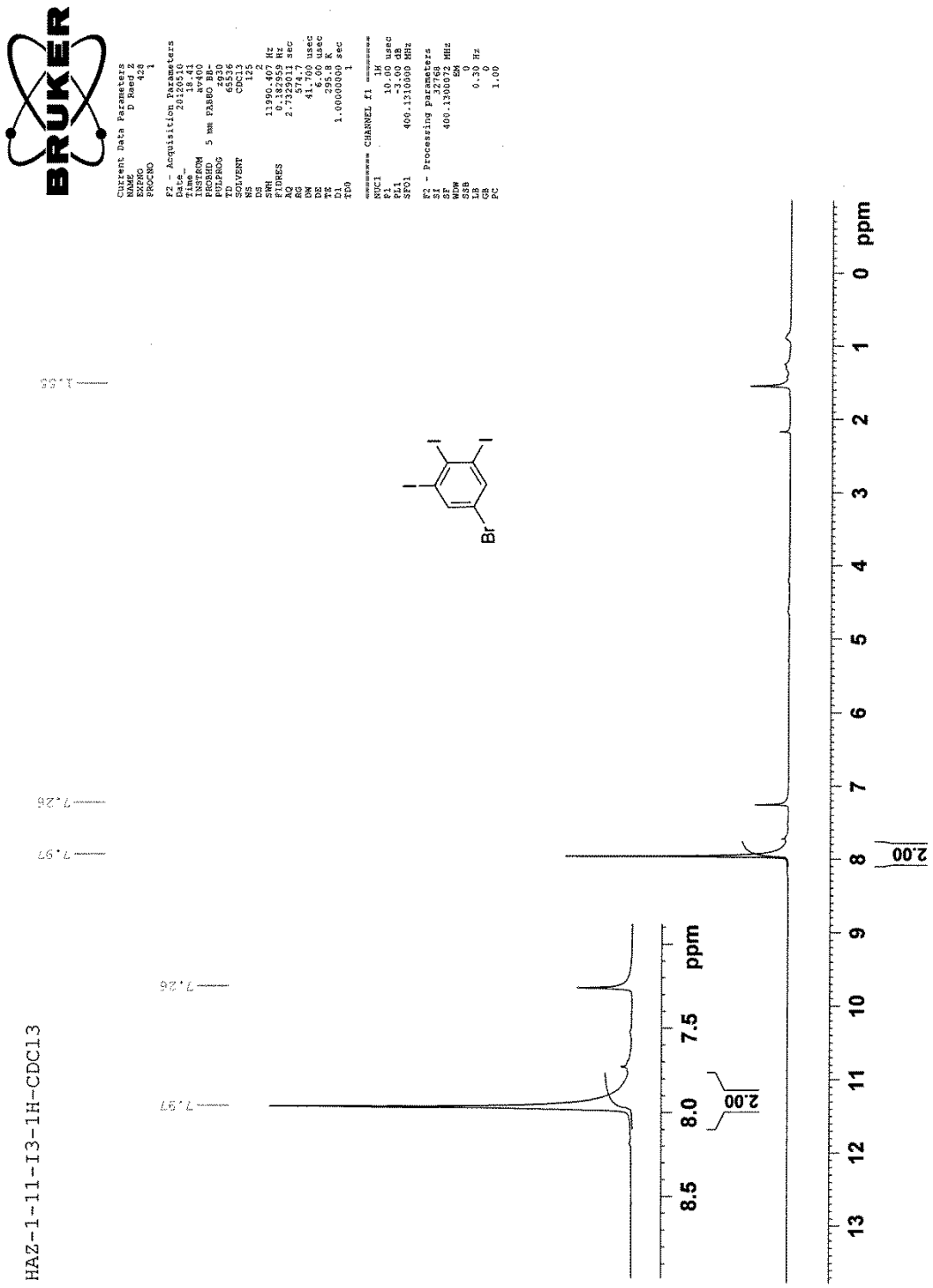
5.7 ^{1}H-NMR of 4-chloro-2-iodo-6-methylaniline (13) in *d*-$CDCl_3$ at 25 ^{o}C.



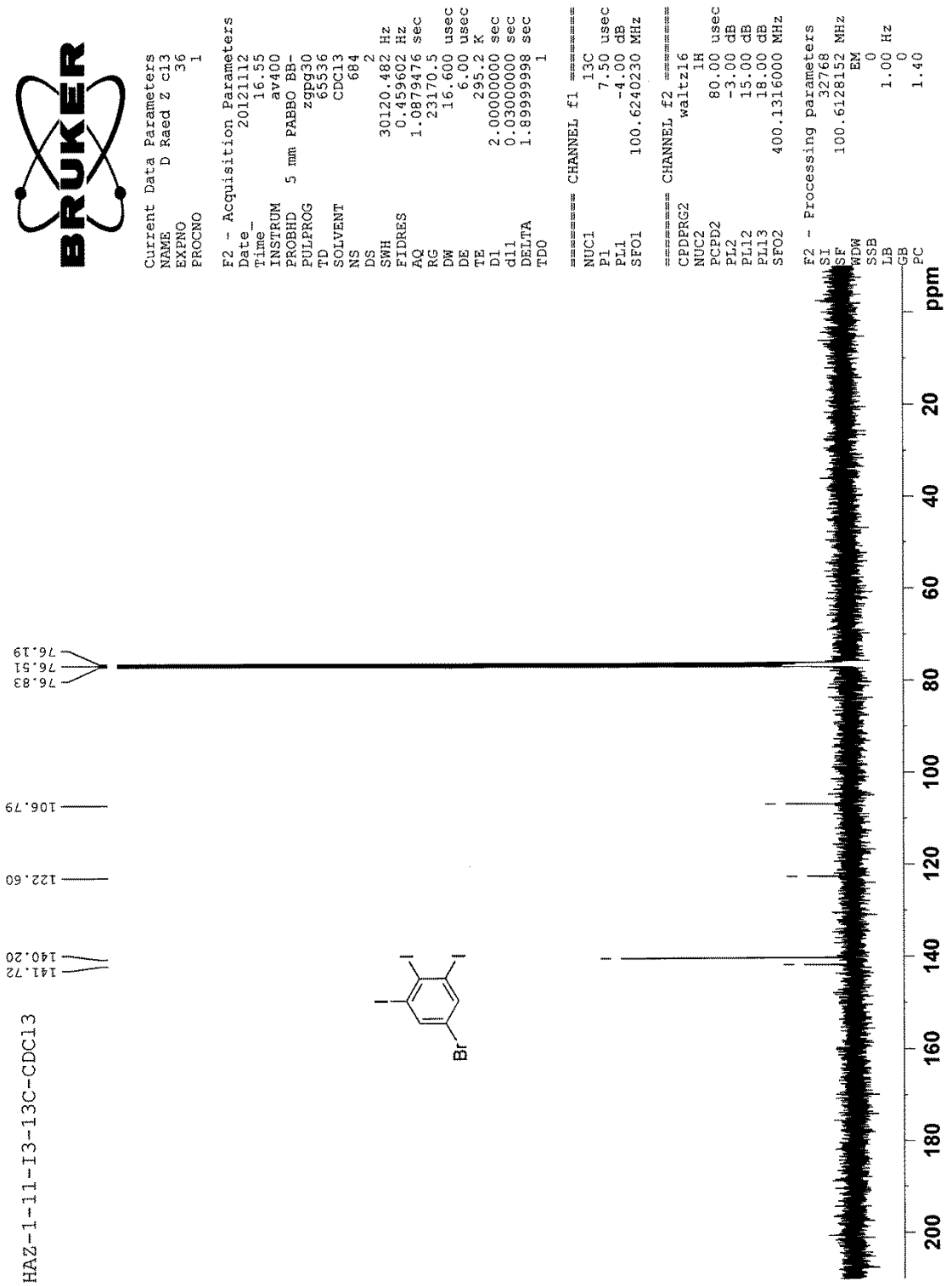
5.8 ^{13}C-NMR of 4-chloro-2-iodo-6-methylaniline (13) in *d*-$CDCl_3$ at 25 ^{o}C.



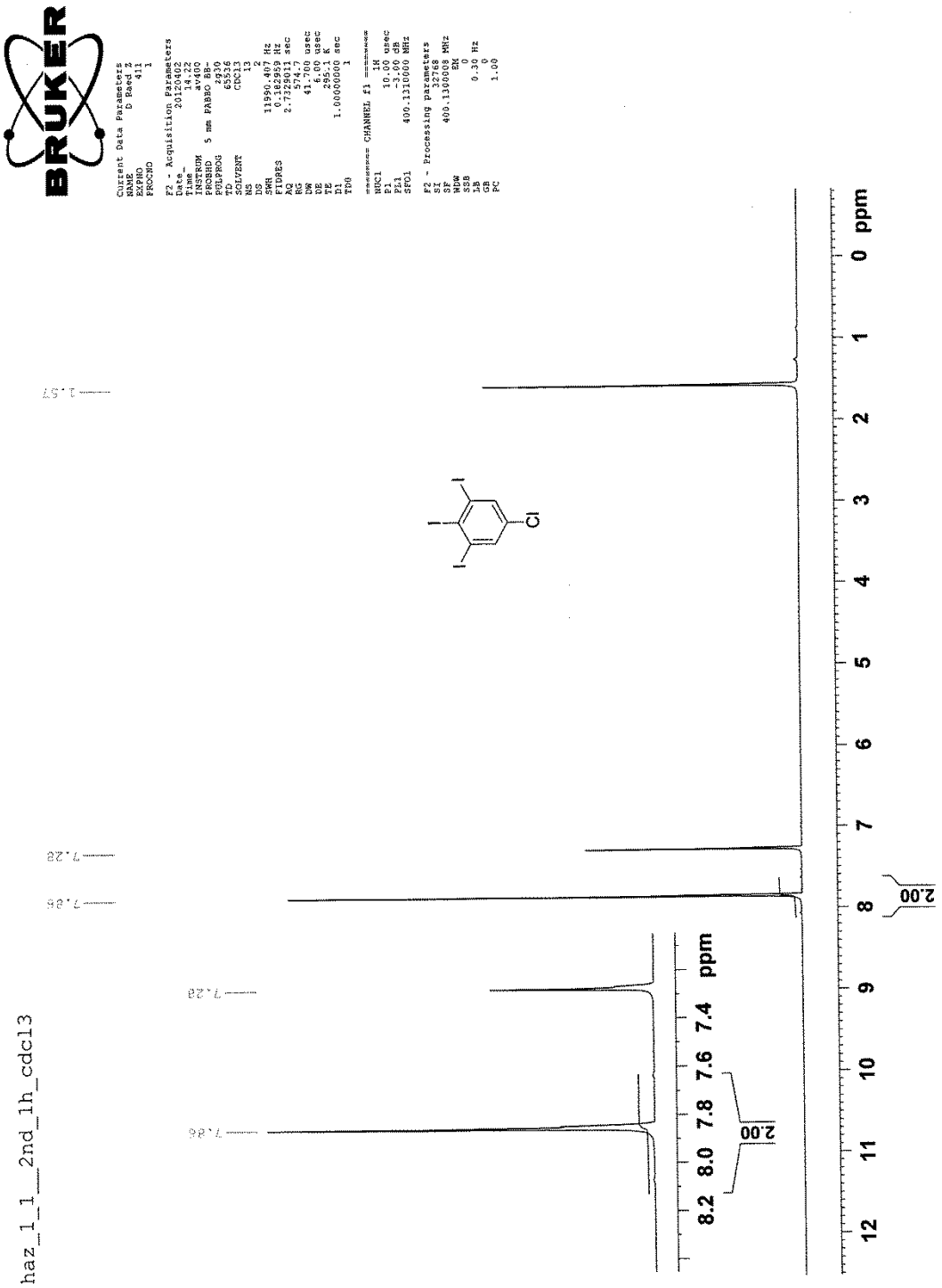
5.9 ^{1}H-NMR of 5-bromo-1,2,3-triiodobenzene (16) in *d*-$CDCl_3$ at 25 ^{o}C.



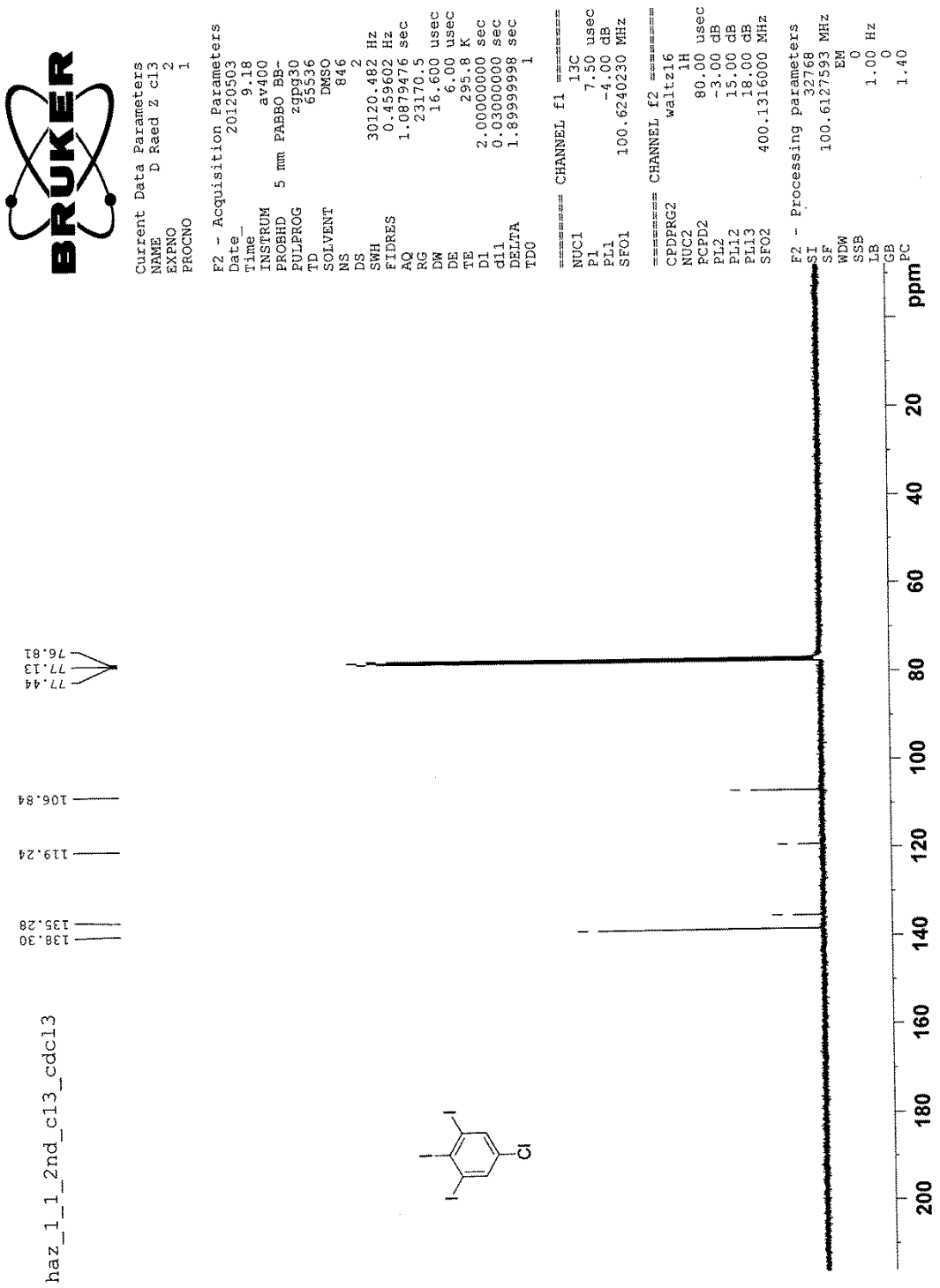
5.10 ^{13}C-NMR of 5-bromo-1,2,3-triiodobenzene (16) in *d*-$CDCl_3$ at 25 °C.



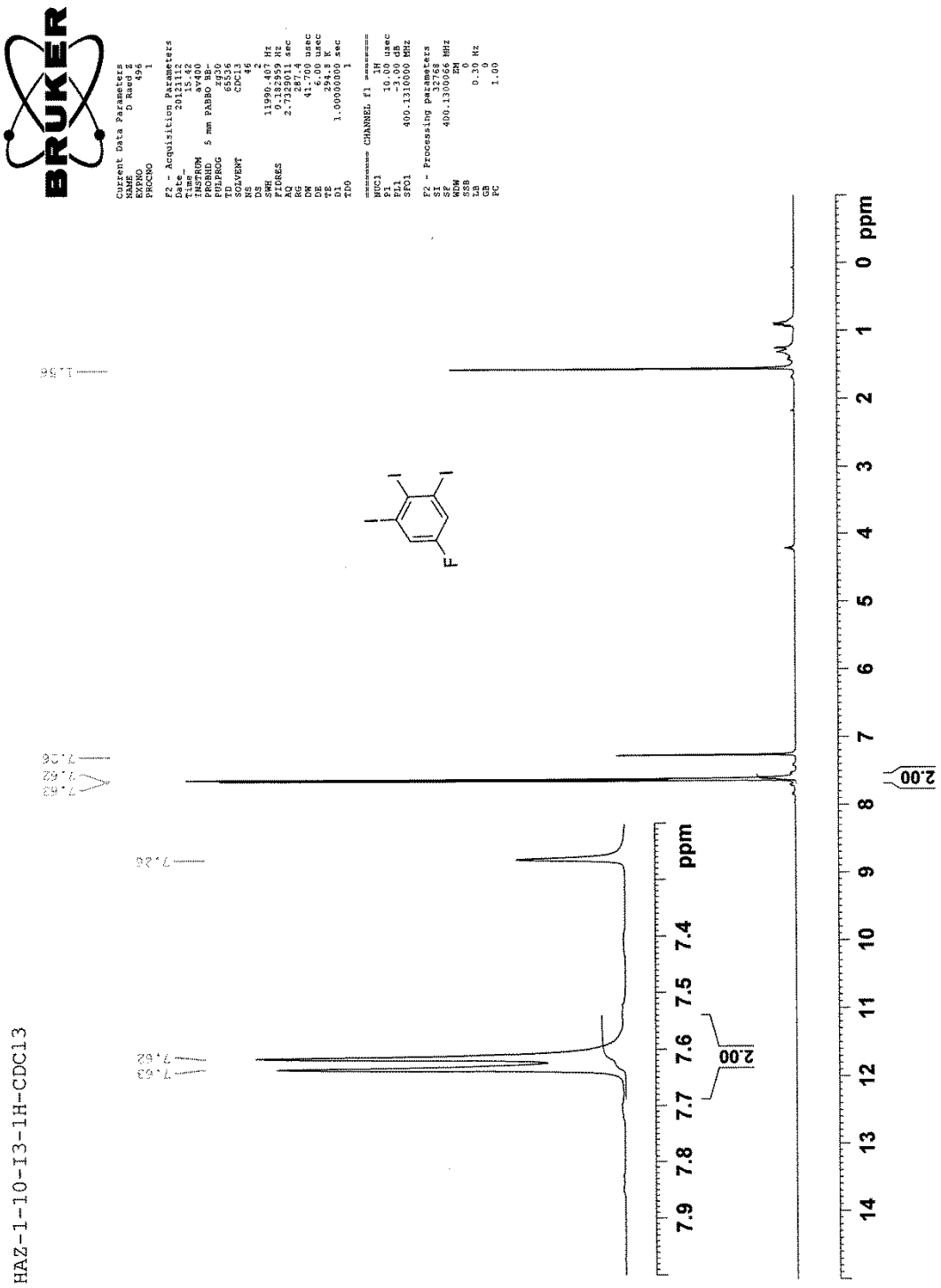
5.11 ^{1}H-NMR of 5-chloro-1,2,3-triiodobenzene (17) in *d*-$CDCl_3$ at 25 ^{o}C.



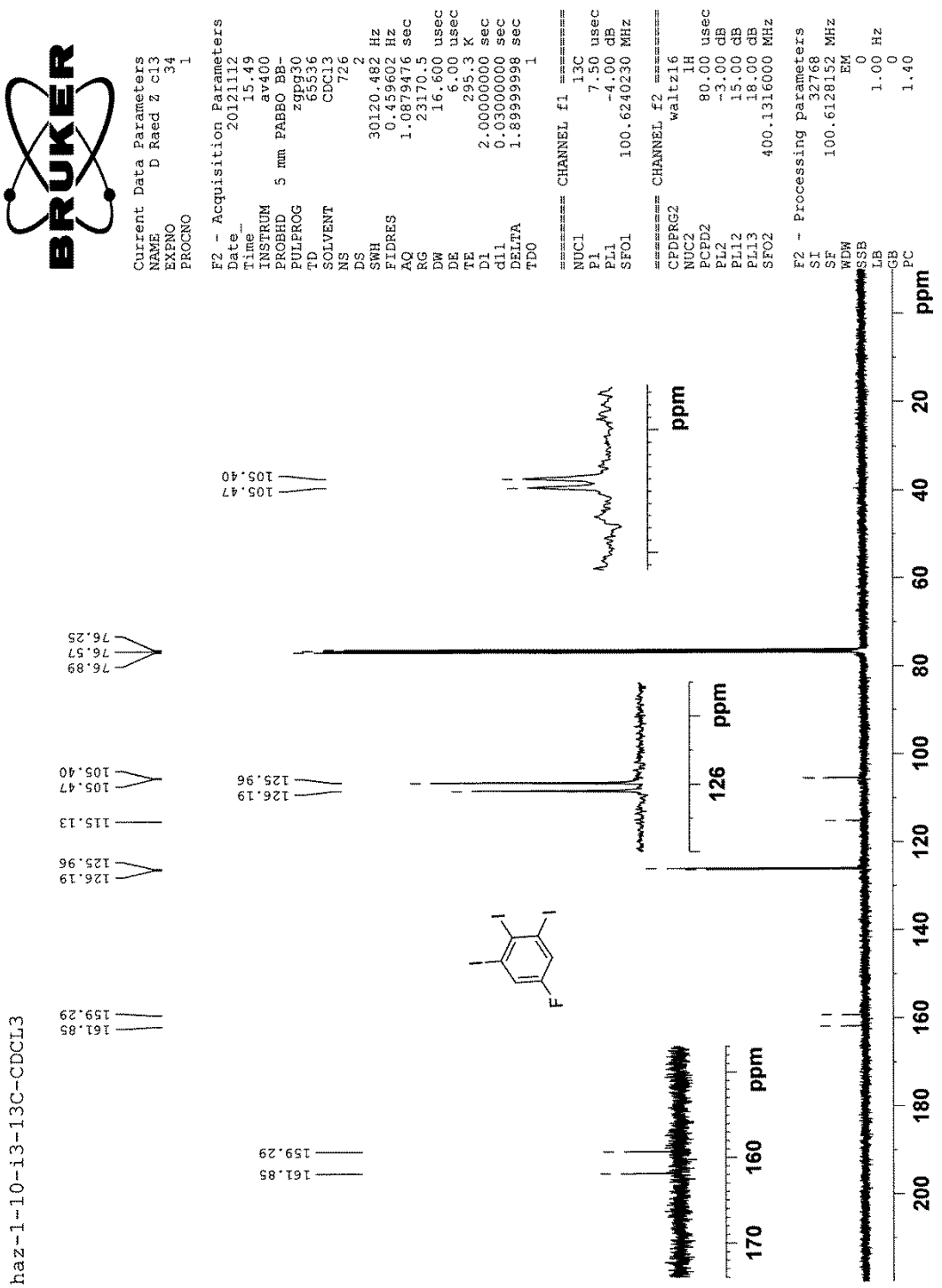
5.12 ^{13}C-NMR of 5-chloro-1,2,3-triiodobenzene (17) in *d*-$CDCl_3$ at 25 ^{o}C.



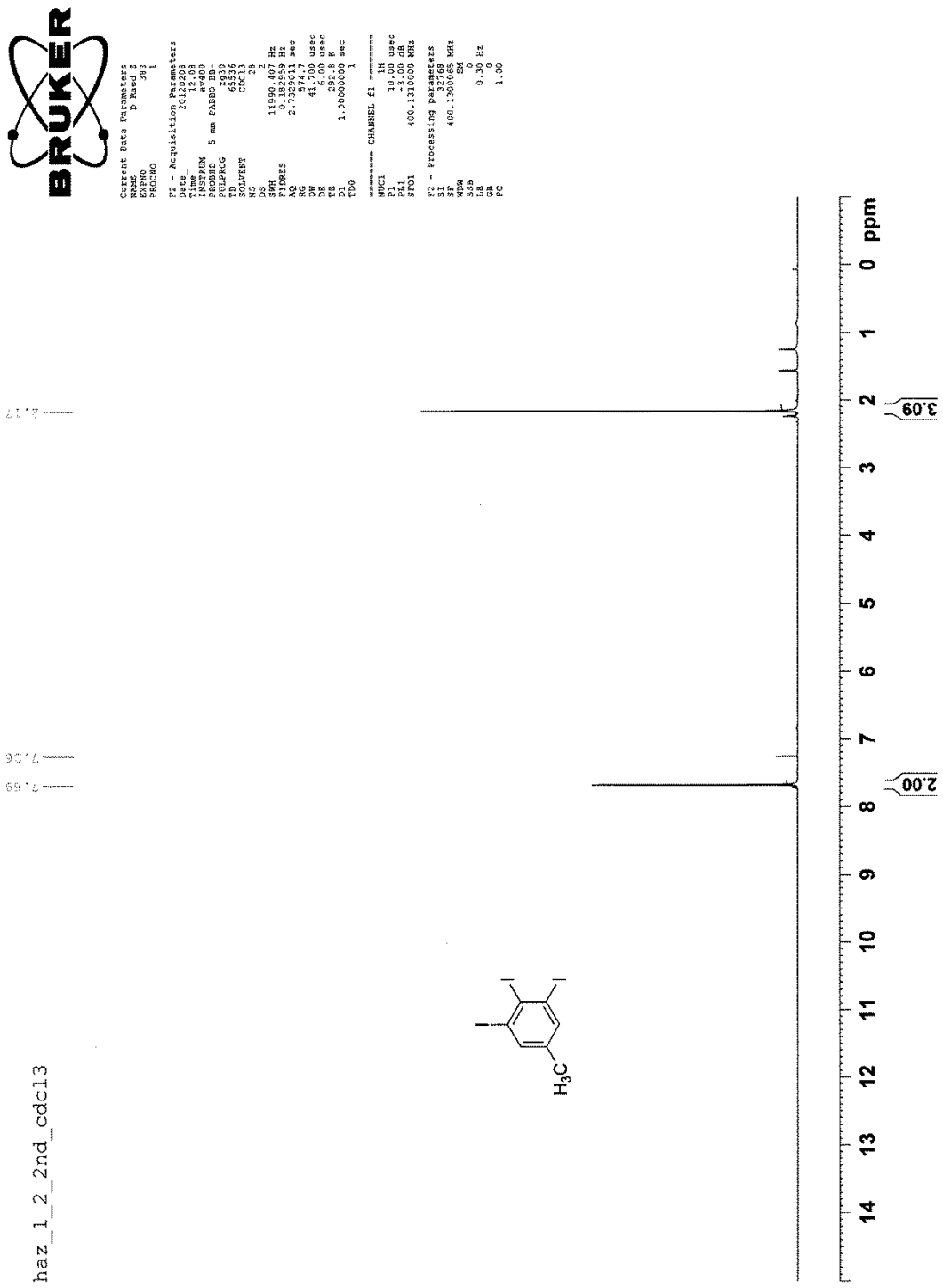
5.13 ^{1}H-NMR of 5-fluoro-1,2,3-triiodobenzene (18) in *d*-$CDCl_3$ at 25 ^{o}C.



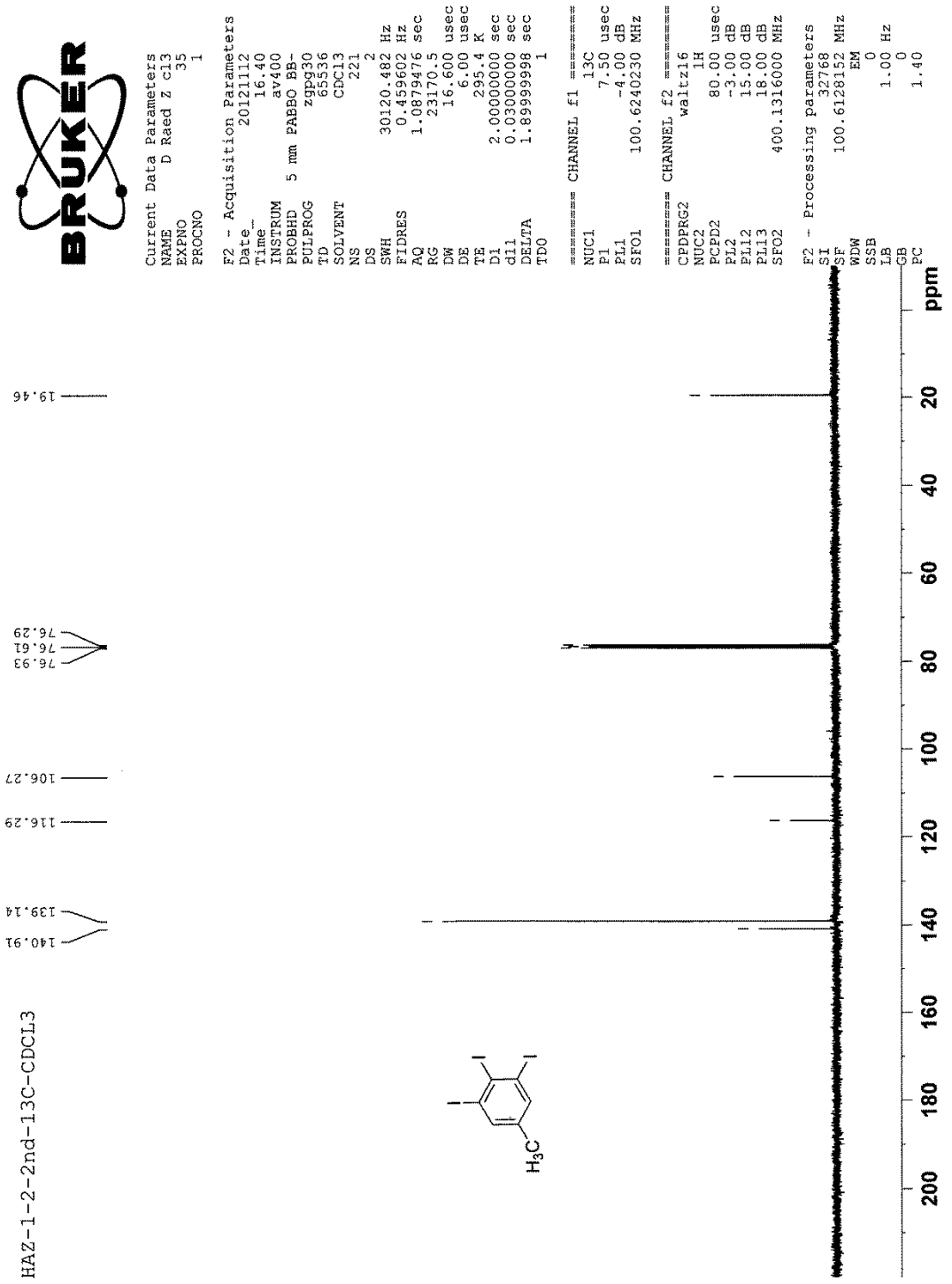
5.14 ^{13}C-NMR of 5-fluoro-1,2,3-triiodobenzene (18) in *d*-$CDCl_3$ at 25 ^{o}C.



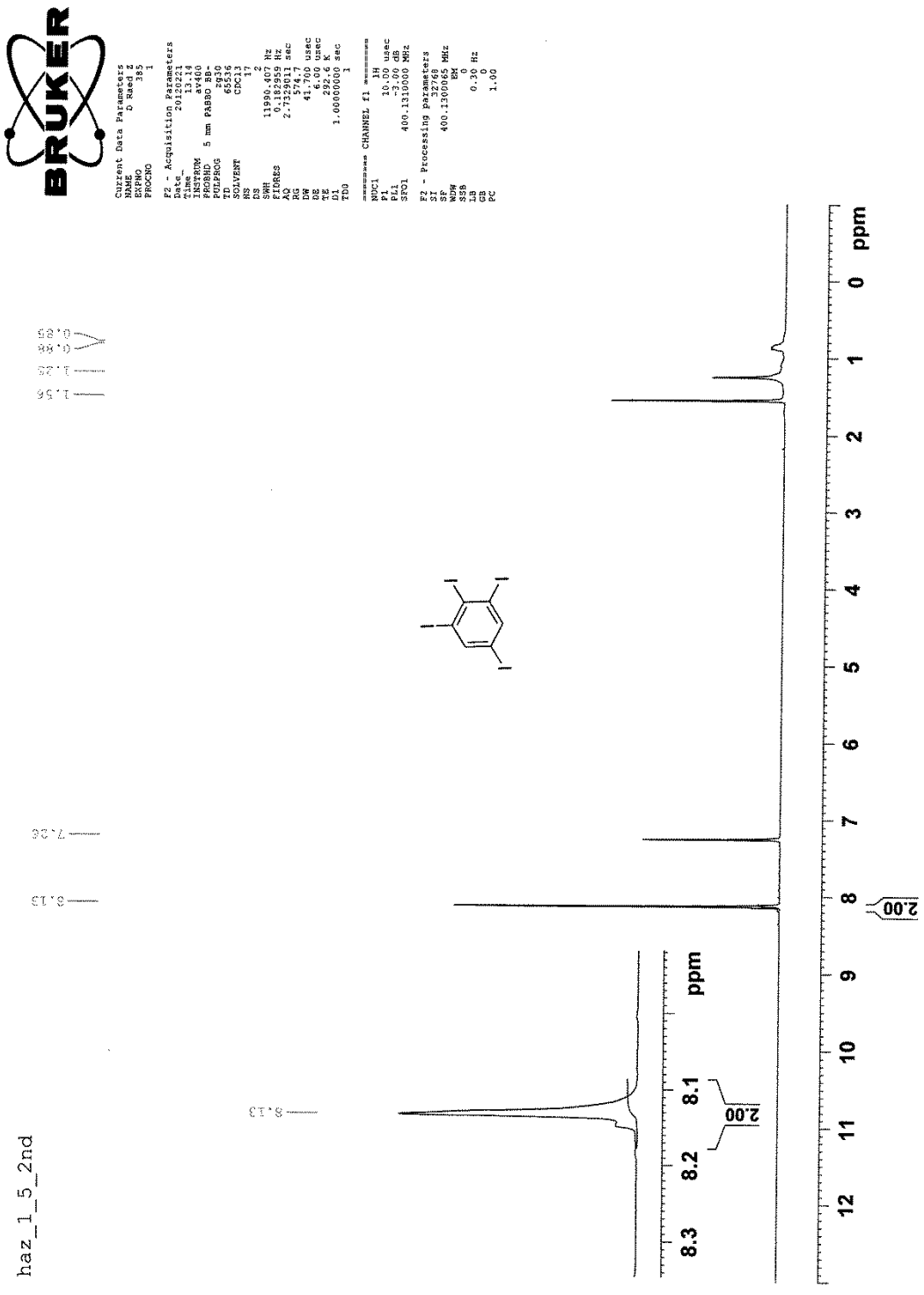
5.15 ^{1}H-NMR of 1,2,3-triiodo-5-methylbenzene (19) in *d*-$CDCl_3$ at 25 ^{o}C.



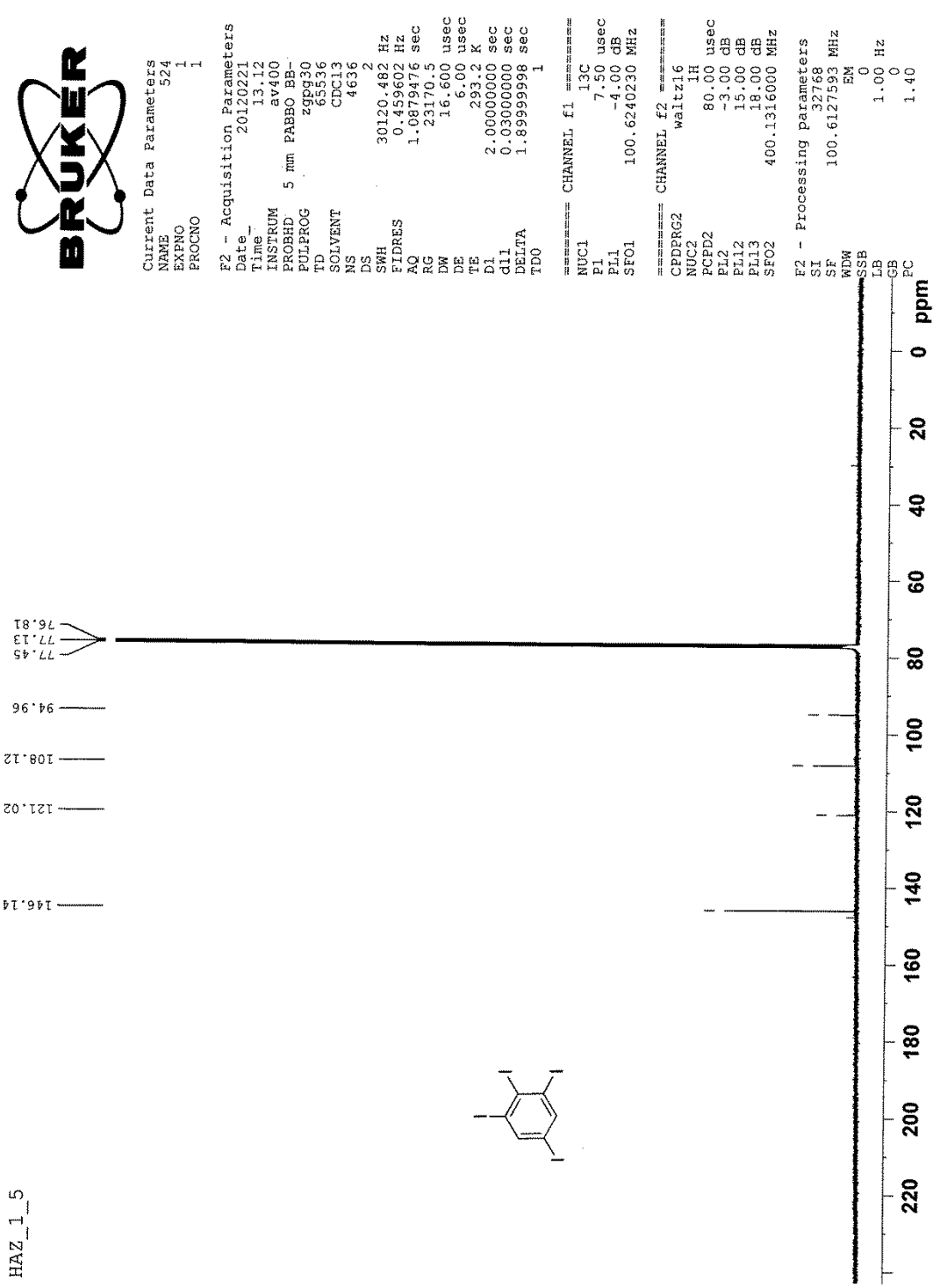
5.16 ^{13}C-NMR of 1,2,3-triiodo-5-methylbenzene (19) in *d*-$CDCl_3$ at 25 ^{o}C.



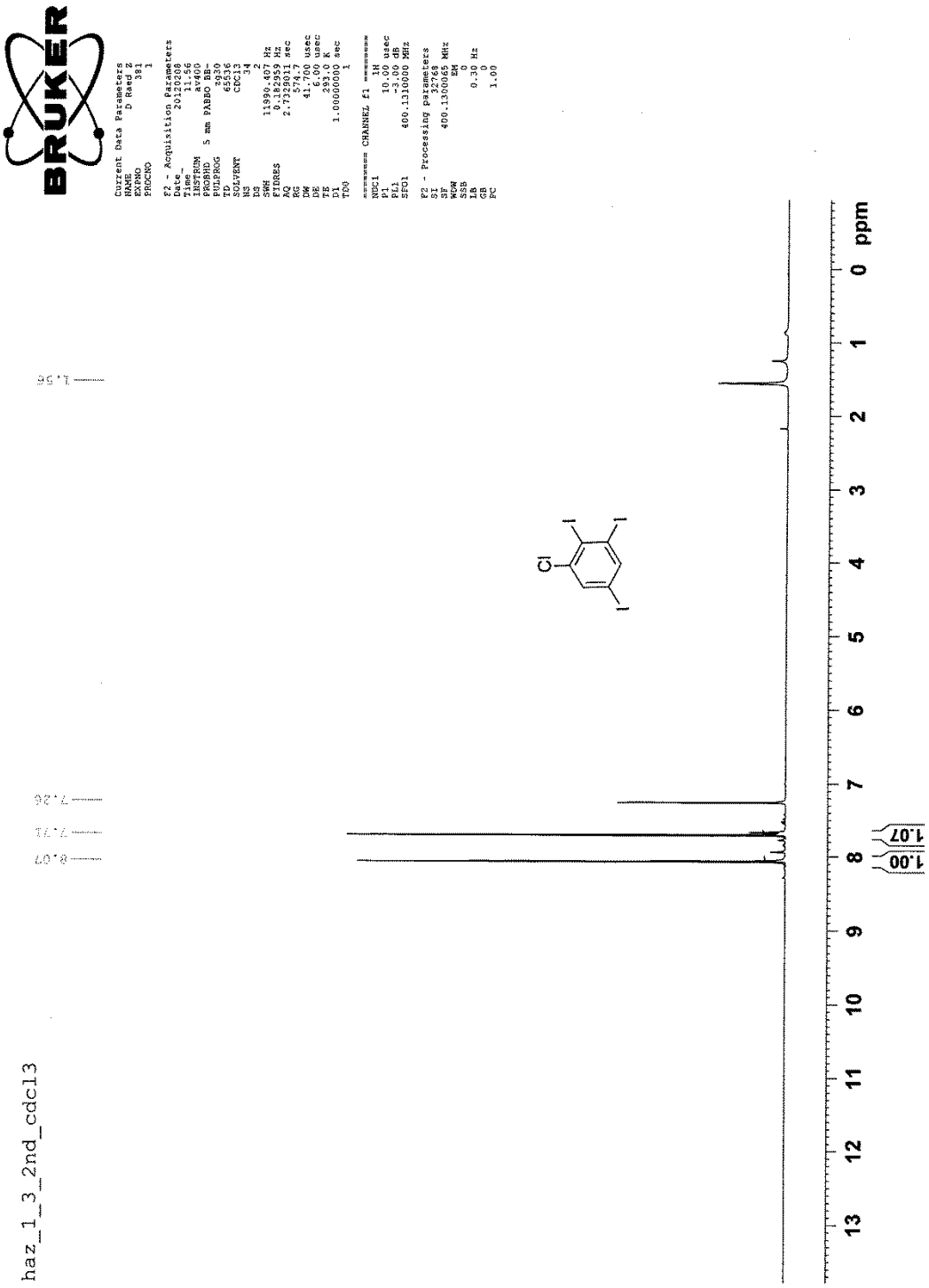
5.17 ^{1}H-NMR of 1,2,3,5-tetraiodobenzene (20) in *d*-$CDCl_3$ at 25 ^{o}C.



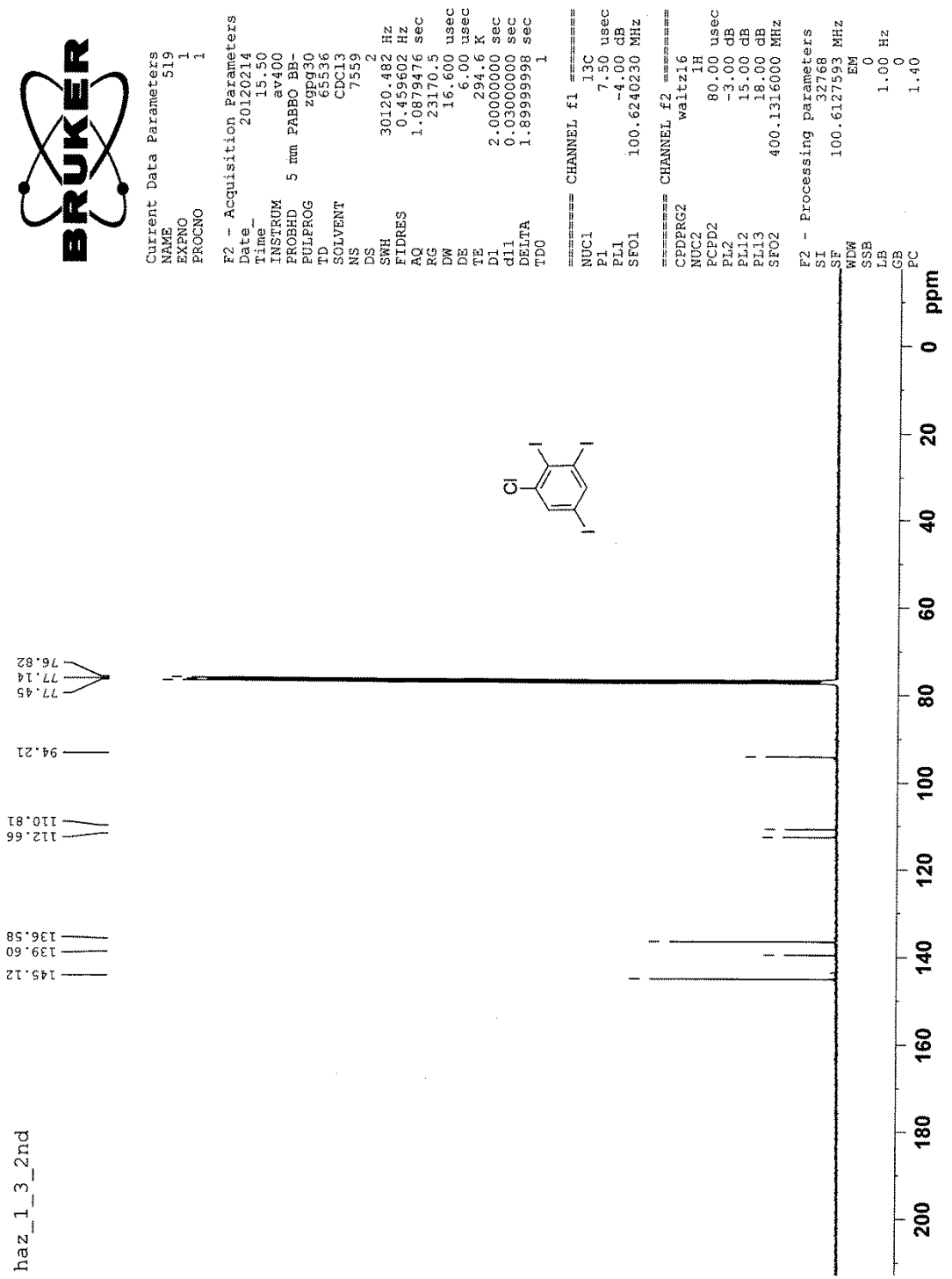
5.18 ^{13}C-NMR of 1,2,3,5-tetraiodobenzene (20) in *d*-$CDCl_3$ at 25 ^{o}C.



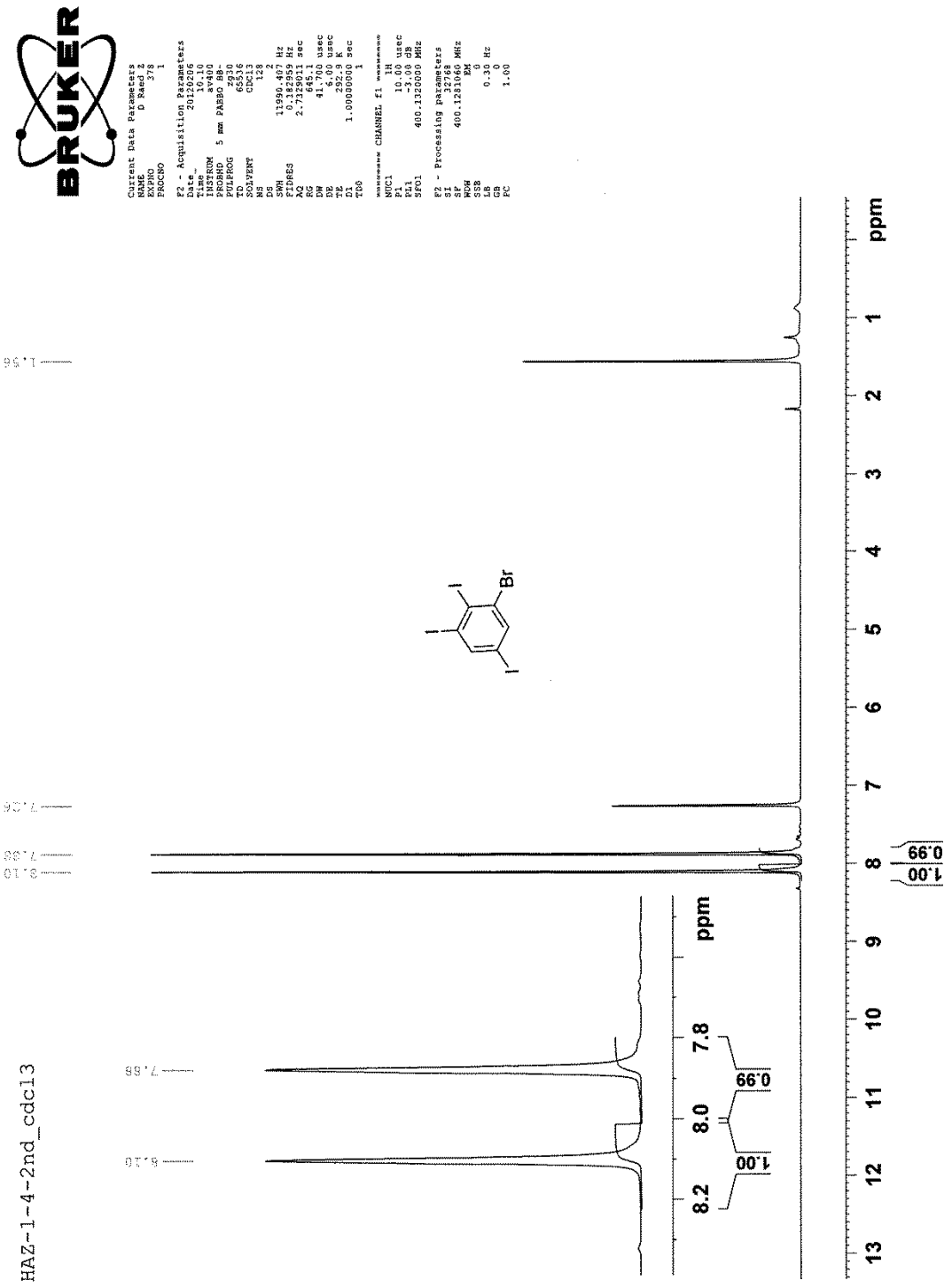
5.19 ^{1}H-NMR of 1-chloro-2,3,5-triiodobenzene (22) in *d*-$CDCl_3$ at 25 ^{o}C.



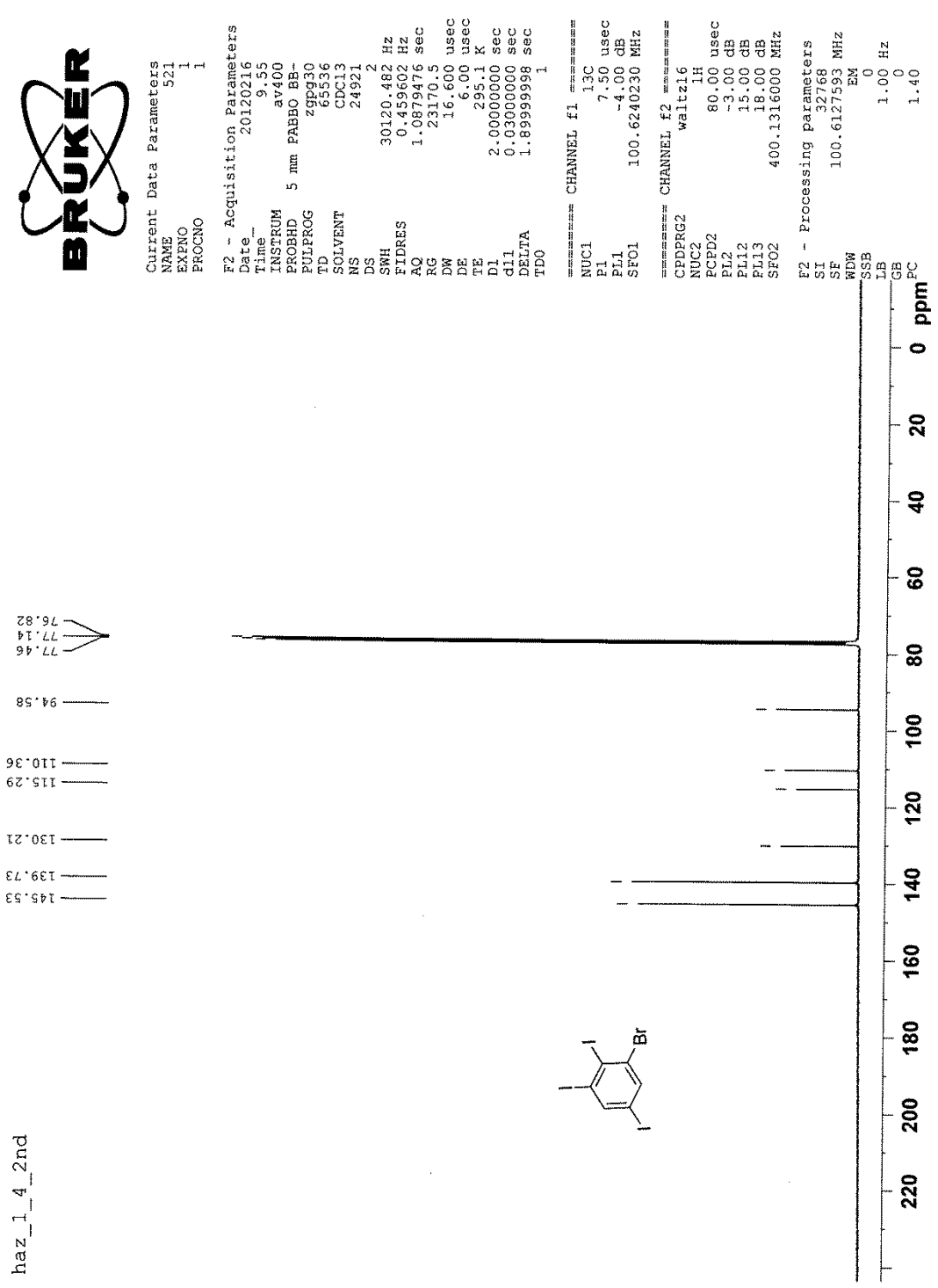
5.20 ^{13}C-NMR of 1-chloro-2,3,5-triiodobenzene (22) in *d*-$CDCl_3$ at 25 ^{o}C.



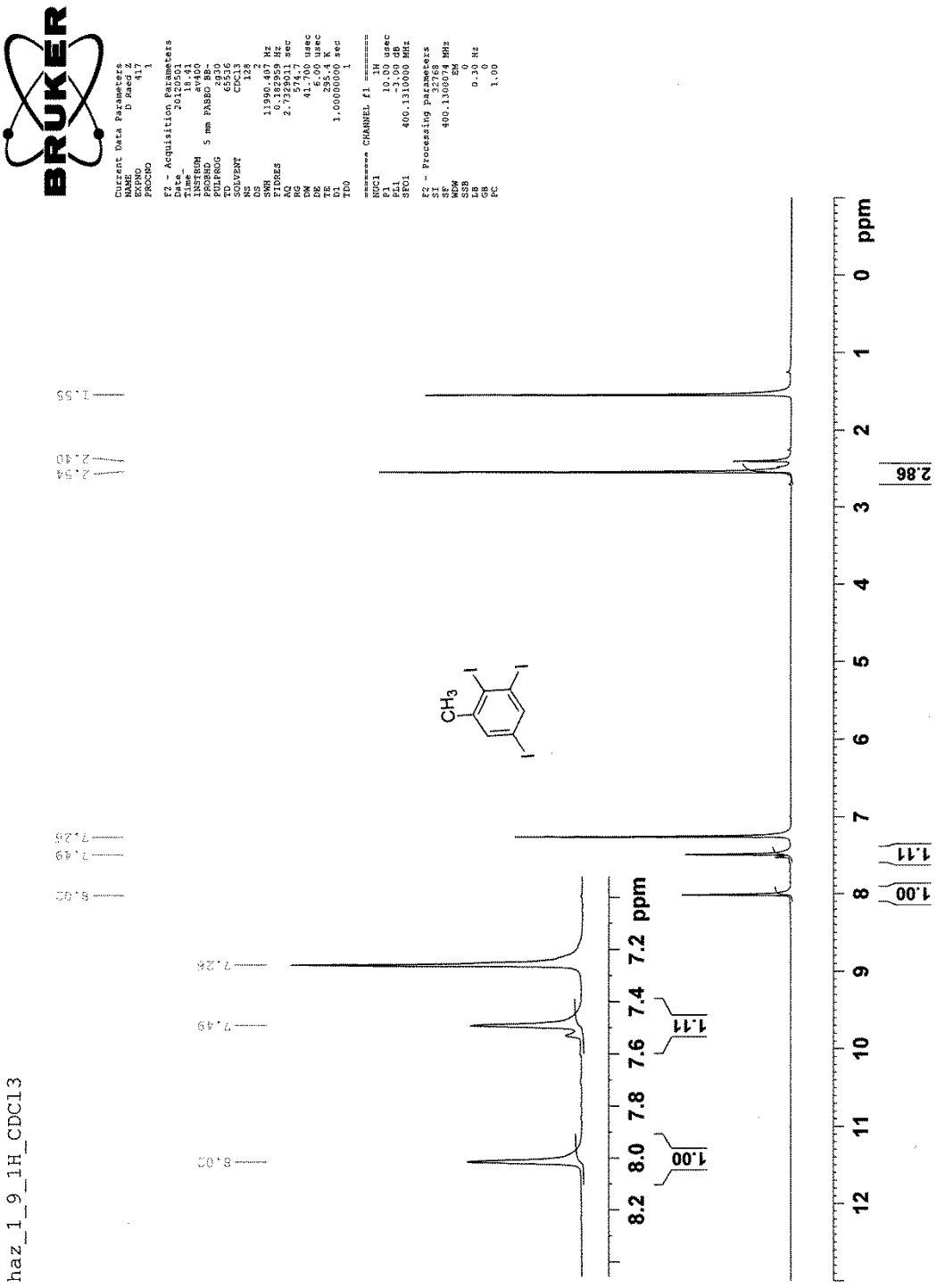
5.21 ^{1}H-NMR of 1-bromo-2,3,5-triiodobenzene (23) in *d*-$CDCl_3$ at 25 ^{o}C.



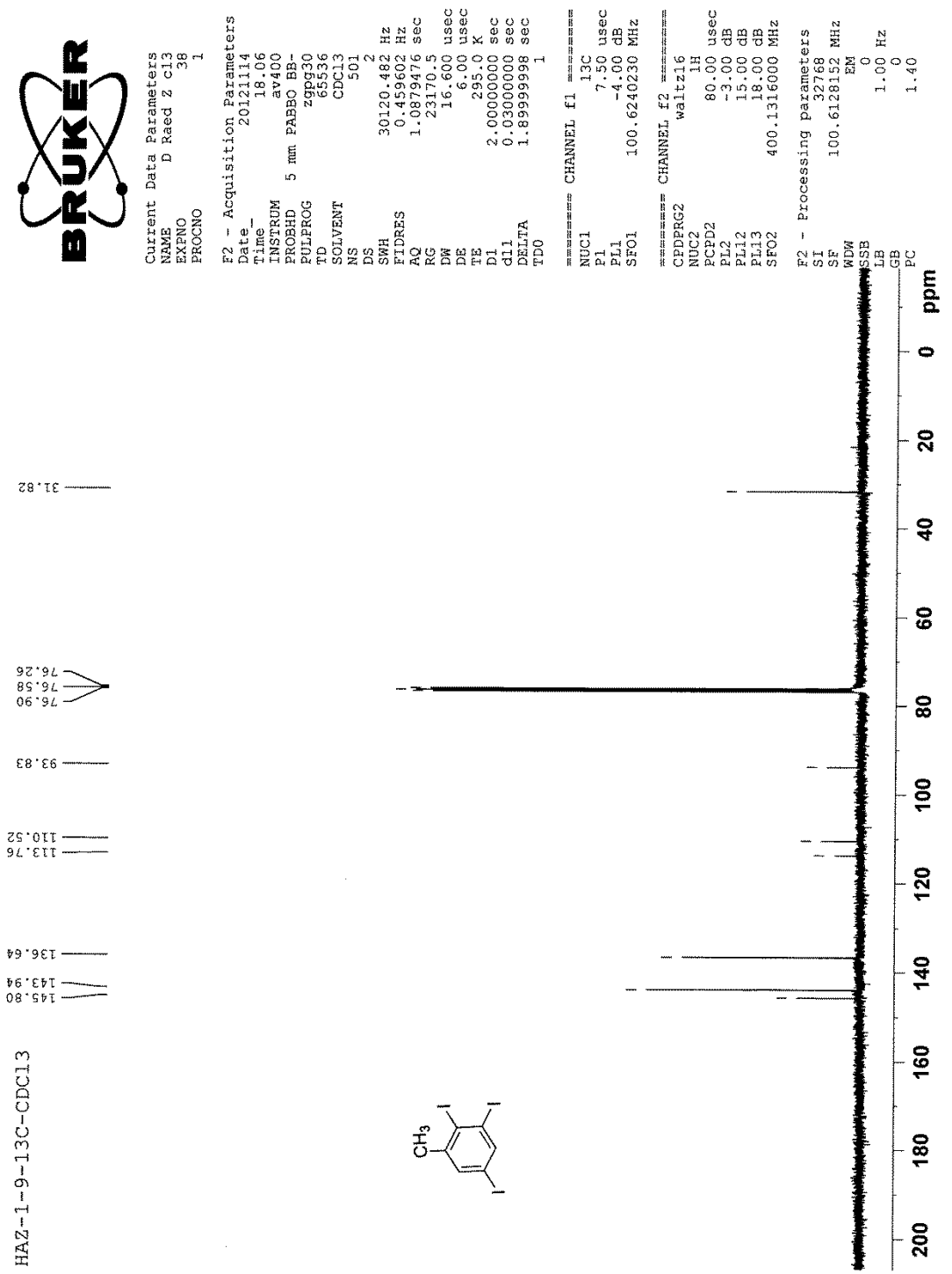
5.22 ^{13}C-NMR of 1-bromo-2,3,5-triiodobenzene (23) in *d*-$CDCl_3$ at 25 ^{o}C.



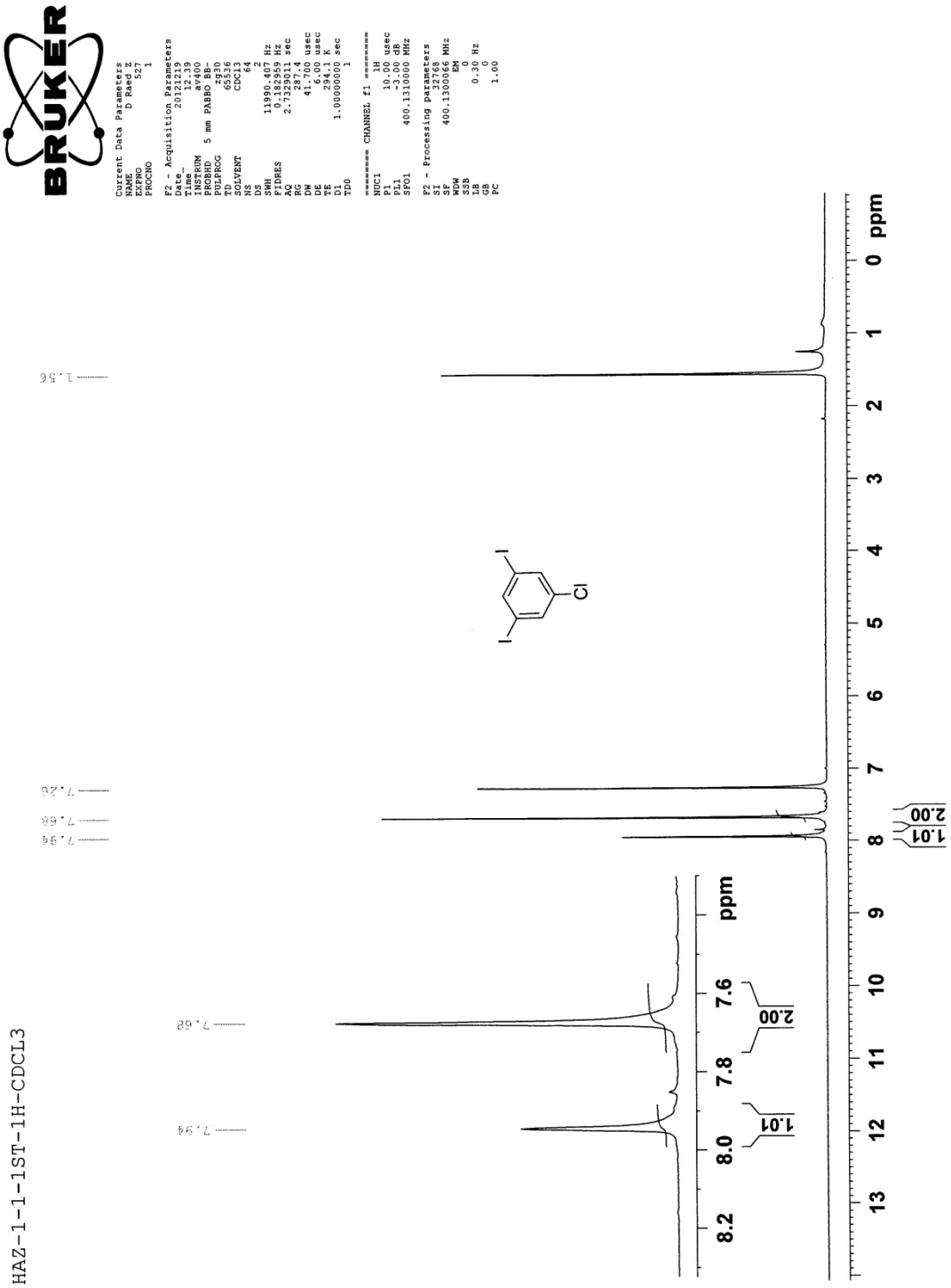
5.23 ^{1}H-NMR of 1,2,5-triiodo-3-methylbenzene (24) in *d*-$CDCl_3$ at 25 ^{o}C.



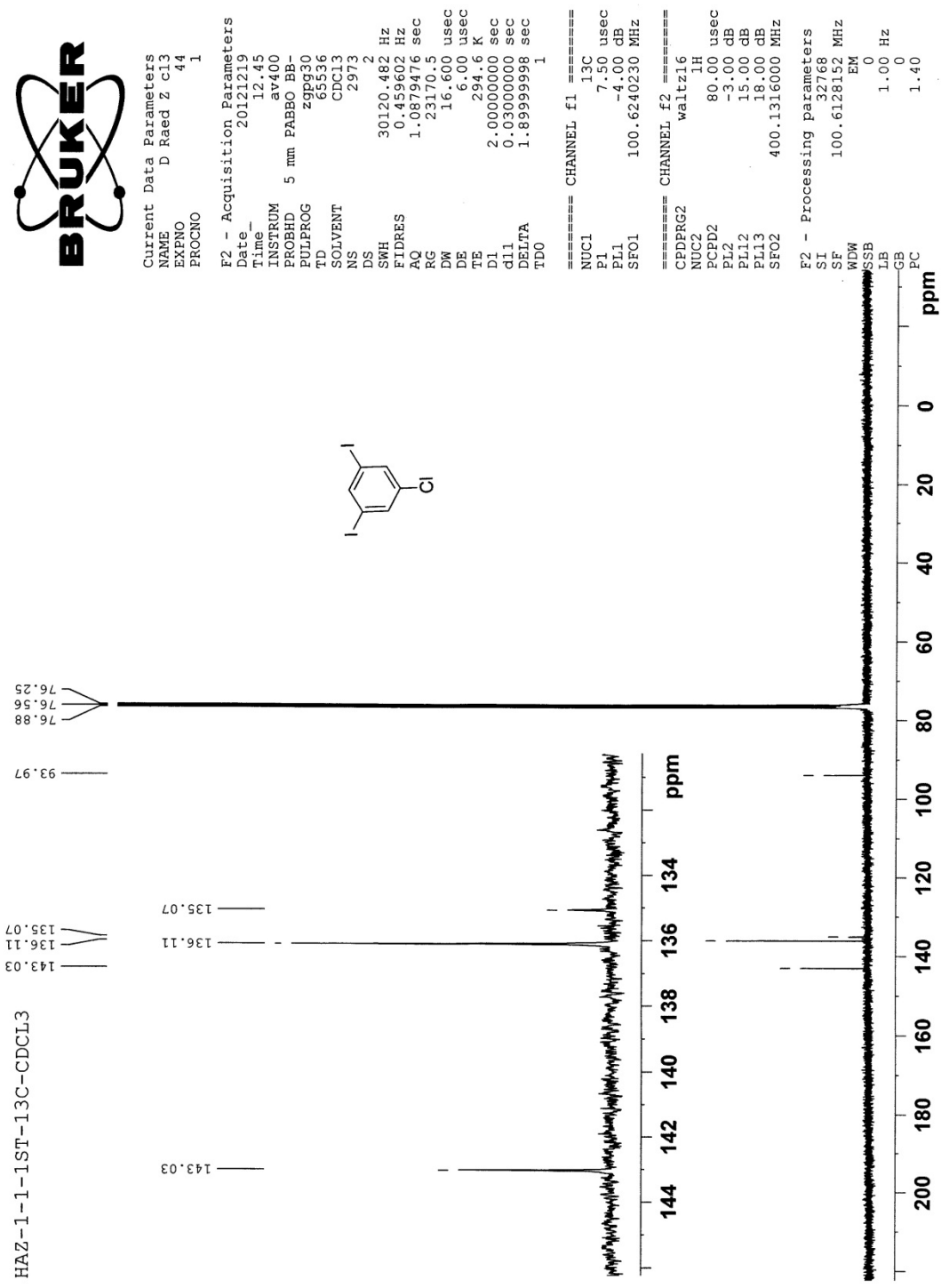
5.24 ^{13}C-NMR of 1,2,5-triiodo-3-methylbenzene (24) in *d*-$CDCl_3$ at 25 ^{o}C.



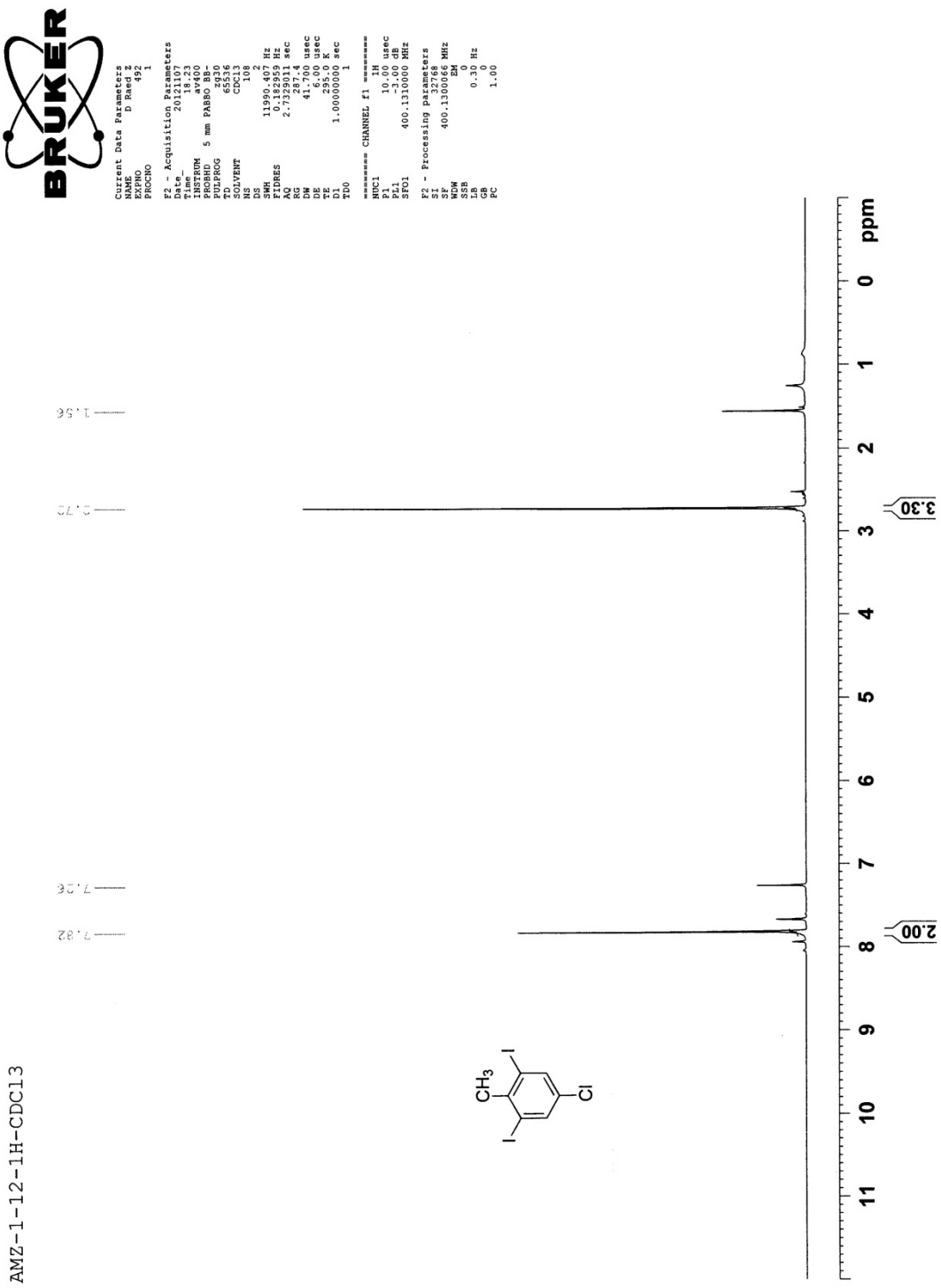
5.25 ^{1}H-NMR of 1-chloro-3,5-diiodobenzene (25) in *d*-$CDCl_3$ at 25 ^{o}C.



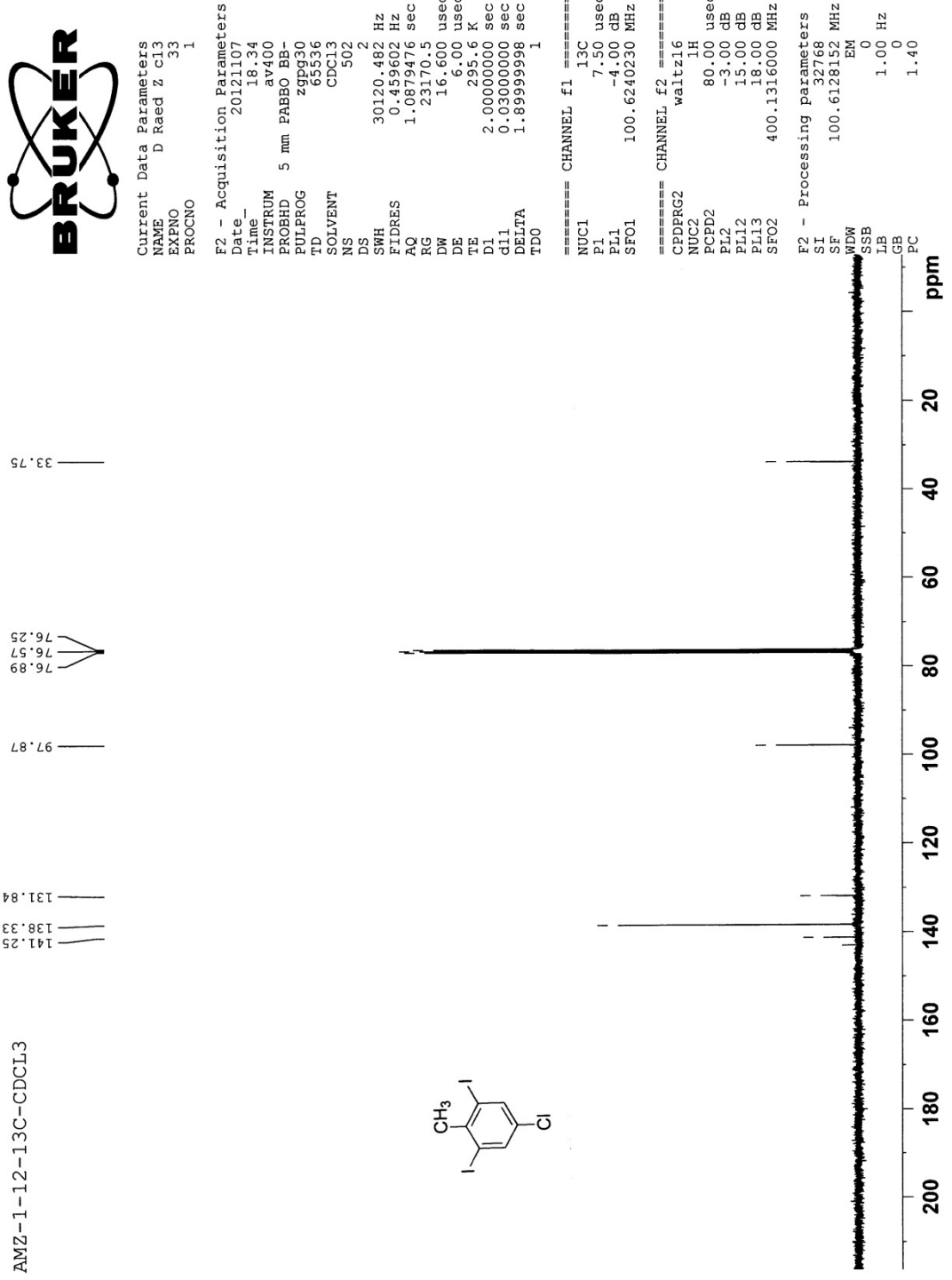
5.26 ^{13}C-NMR of 1-chloro-3,5-diiodobenzene (25) in *d*-$CDCl_3$ at 25 ^{o}C.



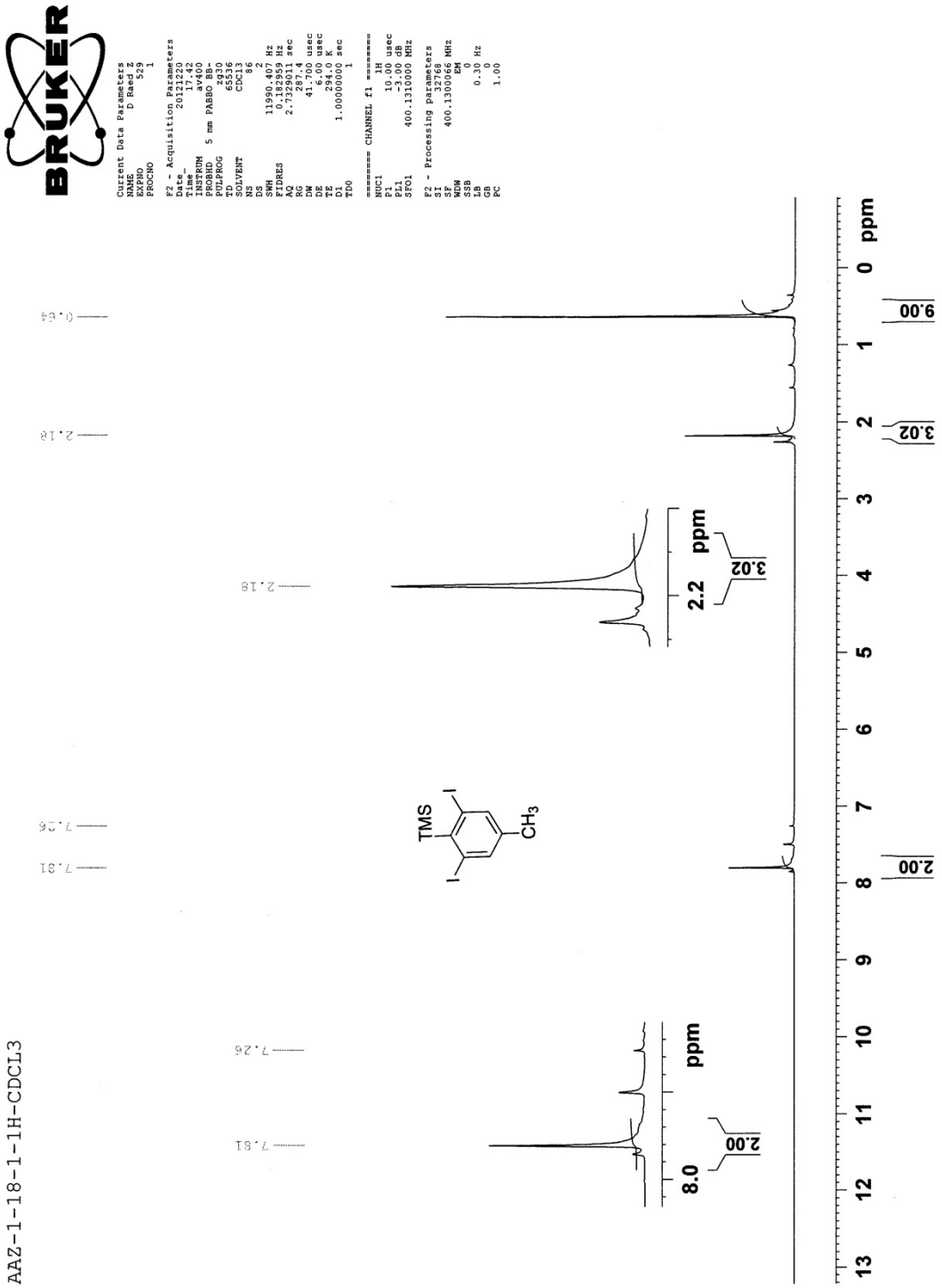
5.27 ^{1}H-NMR of 5-chloro-1,3-diiodo-2-methylbenzene (26) in *d*-$CDCl_3$ at 25 ^{o}C.



5.28 ^{13}C-NMR of 5-chloro-1,3-diiodo-2-methylbenzene (26) in *d*-$CDCl_3$ at 25 ^{o}C.



5.29 ^{1}H-NMR of (2,6-diiodo-4-methylphenyl)trimethylsilane (30) in *d*-$CDCl_3$ at 25 ^{o}C.



5.30 ^{13}C-NMR of (2,6-diiodo-4-methylphenyl)trimethylsilane (30) in *d*-$CDCl_3$ at 25 ^{o}C.