

Supplementary Material

A simple and efficient two-step synthesis of 1,2,3-triiodoarenes via consecutive *C-H* iodination/ *ipso*-iododecarboxylation strategy: A potential application towards *ortho*-diiodoarenes by regioselective metal-iodine exchange reaction

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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, chlorobenzene (C₆H₅Cl), chloroform, ethyl acetate, palladium (II) diacetate [Pd(OAc)₂], iodobenzene diacetate PhI(OAc)₂, *N*-iodosuccinimide (NIS), sodium sulfate anhyd., iso-propylmagnesium chloride (*i*-PrMgCl, 2M), iodine (I₂). Anhydrous solvents were distilled over appropriate drying agents prior to use were used as supplied. 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). Reaction scale is between 2-5 gm. NMR spectra were recorded on a Bruker-Avance 400 MHz spectrometer. The residual solvent protons (¹H) or the solvent carbon (¹³C) were used as internal standards. ¹H-NMR data are presented as follows: chemical shift in ppm (δ) downfield from trimethylsilane (TMS) (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 using Chemical Ionization (CI) and electrospray ionization (ESI) techniques.

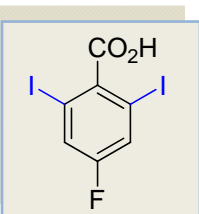
2. General Procedure for Iodination of Benzoic Acid Derivatives

(General procedure by Jin-Quan Yu, *Angew. Chem. Int. Ed.*, **2008**, *47*, 5215-5219)

In a flamed dry 100 mL RBF, substrate (16.4 mmol, 1.0 equiv.), palladium acetate (0.05 equiv.), iodobenzene diacetate (1.5 equiv.) and iodine (1.5 equiv.) were dissolved in anhydrous dimethylformamide (DMF) (40 mL) under atmospheric air. The flask was then sealed with a septa and the reaction mixture was stirred at 100 °C for 24 hr. The reaction mixture was cooled to room temperature, diluted with ethyl acetate and then washed with 0.5 N HCl (20 mL × 4). The organic phase was washed with brine, dried over Na₂SO₄ and concentrated in a rotary evaporator. The

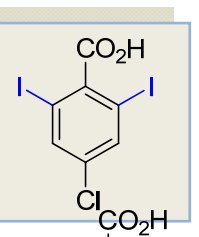
residue was purified by column chromatography on silica gel (3:1 hexane : EtOAc) to give the iodination product.

2.1 Synthesis of 4-fluoro-2,6-diiodobenzoic acid (6)

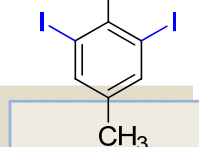


The title compound was prepared using the general procedure for Iodination of arene carboxylic acids and isolated in **85%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47 (28): 5215-9.

2.2 Synthesis of 4-chloro-2,6-diiodobenzoic acid (7)



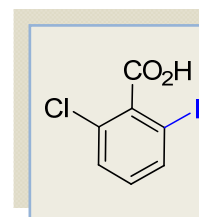
The title compound was prepared using the general procedure for Iodination of arene carboxylic acids and isolated in **87%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47 (28): 5215-9.



2.3 Synthesis of 2,6-diiodo-4-methylbenzoic acid (10)

The title compound was prepared using the general procedure for Iodination of arene carboxylic acids and isolated in **85%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47 (28): 5215-9.

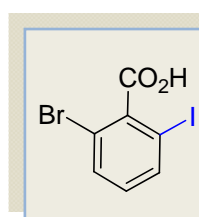
2.4 Synthesis of 2-chloro-6-iodobenzoic acid (12)



(28): 5215-9.

The title compound was prepared using the general procedure for Iodination of arene carboxylic acids and isolated in **91%** yield as a yellowish solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47

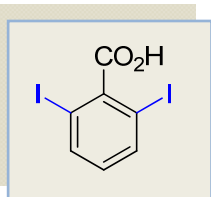
2.5 Synthesis of 2-bromo-6-iodobenzoic acid (13)



The title compound was prepared using the general procedure for Iodination of arene carboxylic acids and isolated in **86%** yield as a yellowish solid. The spectroscopic data for this compound are

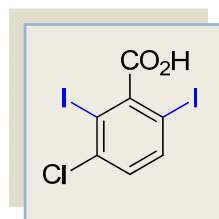
matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47 (28): 5215-9.

2.6 Synthesis of 2,6-diiodobenzoic acid (14)



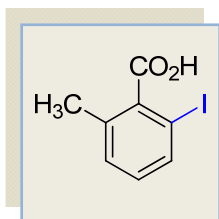
The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in **82%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47 (28): 5215-9.

2.7 Synthesis of 3-chloro-2,6-diiodobenzoic acid (15)



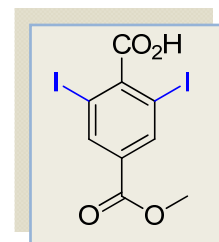
The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in **81%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃): 7.74 (d, 1H, *J* = 8.0 Hz), 7.20 (d, 1H, *J* = 8.0 Hz). δ_{C} (100 MHz, *d*-CDCl₃) δ : 172.2, 146.8, 142.6, 139.3, 130.3, 95.7, 86.9. **IR** (cast film, cm⁻¹) 1790, 1652, 1548, 1152, 965. **Mp**: 148-150 °C. **HRMS** (ESI) *m/z* for C₇H₂ClI₂O₂ [M-H]⁻: calcd. 406.7833; found, 406.7824.

2.8 Synthesis of 2-iodo-6-methylbenzoic acid (17)



The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in **69%** yield as a yellowish solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47 (28): 5215-9.

2.9 Synthesis of methyl 3,4,5-triiodobenzoate (20)

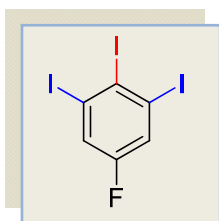


The title compound was prepared using the general procedure for iodination of arene carboxylic acids and isolated in **77%** yield as a pale-yellow solid. The spectroscopic data for this compound are matched the previous report by *Angew. Chem. Int. Ed.* **2008**; 47 (28): 5215-9.

3. General Procedure for *Ips*o-Iododecarboxylation of Iodinated Benzoic acid Derivatives

A mixture of *ortho*-diiodobenzoic acid derivative (1.0 mmol, 1.0 equiv.), *N*-iodosuccinamide (NIS, 2.0 equiv.) and 1,2-dichloroethane (DCE, 10 mL) were added to a flamed dry 100 mL RBF and the mixture was irradiated with 100W (tungsten lamp) under reflux conditions for 24 h. The reaction mixture was then cooled to room temperature. The mixture was then washed sat. NaHSO₃, sat. NaHCO₃ and with brine. The organic layers were then dried over anhydrous Na₂SO₄, filtered and concentrated in a rotary evaporator. The residue was then purified by flash chromatography on silica gel (100% hexane) to give the desired iodinated product.

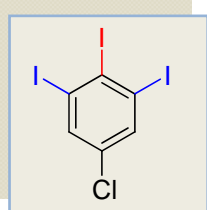
3.1 Synthesis of 5-fluoro-1,2,3-triiodobenzene (21)



The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **82%** yield as a white solid. The spectroscopic data for this compound are matched our previous report by *Aust. J. Chem.*

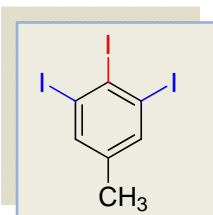
2013, 66, 1570–1575.

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



The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **84%** yield as a white solid. The spectroscopic data for this compound are matched our previous report by *Aust. J. Chem.* **2013**, 66, 1570–1575.

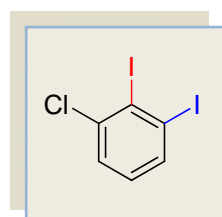
3.3 Synthesis of 1,2,3-triiodo-5-methylbenzene (23)



The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **61%** yield as a white solid. The spectroscopic data for this compound are matched our previous report by *Aust. J. Chem.*

2013, 66, 1570–1575.

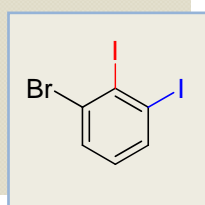
3.4 Synthesis of 1-chloro-2,3-diiodobenzene (24)



The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **83%** yield as a white solid. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.75 (d, 1H,

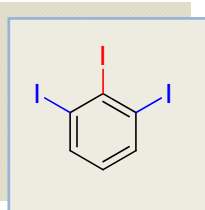
$J = 8.0$ Hz), 7.39 (d, 1H, $J = 8.1$ Hz), 6.98 (dd, 1H, $J = 8.0$, $J = 8.1$ Hz). δ_c (100 MHz, d -CDCl₃) δ : 138.5, 137.0, 130.0, 127.8, 112.3, 109.3. **IR** (cast film, cm⁻¹) 2978, 2948, 1642, 1581, 1100, 741. **Mp**: 83-84 °C. **HRMS** (CI) m/z for C₆H₃ClI₂ [M⁺]: calcd. 363.8013; found, 363.8008.

3.5 Synthesis of 1-bromo-2,3-diiodobenzene (25)



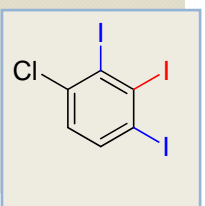
The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **80%** yield as a white solid. δ_H (400 MHz, d -CDCl₃) δ : 7.81 (d, 1H, $J = 8.0$ Hz), 7.58 (d, 1H, $J = 8.0$ Hz), 6.89 (dd, 1H, $J = 8.0$ Hz). δ_c (100 MHz, d -CDCl₃) δ : 137.5, 131.3, 130.2, 129.1, 114.9, 108.8. **IR** (cast film, cm⁻¹) 2987, 2962, 1601, 1542, 1118, 624. **Mp**: 92-93 °C. **HRMS** (CI) m/z for C₆H₃BrI₂ [M⁺]: calcd. 407.7507; found, 407.7497

3.6 Synthesis of 1,2,3-triiodobenzene (26)



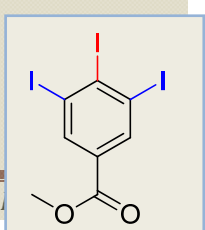
The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **81%** yield as a white solid. δ_H (400 MHz, d -CDCl₃) δ : 7.85 (d, 2H, $J = 8.0$ Hz), 6.68 (t, 1H, $J = 8$ Hz). δ_c (100 MHz, d -CDCl₃) δ : 138.2, 130.4, 120.7, 106.7. **IR** (cast film, cm⁻¹) 3121, 2968, 1624, 1510, 1015, 462. **Mp**: 111-112 °C. **HRMS** (CI) m/z for C₆H₃I₃ [M⁺]: calcd. 455.7369; found, 455.7362.

3.7 Synthesis of 1-chloro-2,3,4-triiodobenzene (27)



The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **65%** yield as a white solid. δ_H (400 MHz, d -CDCl₃) δ : 7.83 (d, 1H, $J = 8.0$ Hz), 7.13 (d, 1H, $J = 8$ Hz). δ_c (100 MHz, d -CDCl₃) δ : 138.9, 137.7, 128.9, 123.1, 112.6, 103.8. **IR** (cast film, cm⁻¹) 3088, 2941, 1605, 1544, 1142, 570. **Mp**: 85-86 °C. **HRMS** (CI) m/z for C₆H₂ClI₃ [M⁺]: calcd. 489.6979; found, 489.6970.

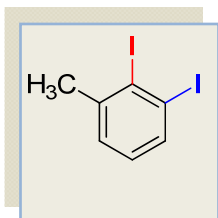
3.8 Synthesis of methyl 3,4,5-triiodobenzoate (28)



The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **75%** yield as a white solid. δ_H (400 MHz, d -CDCl₃) δ : 8.40 (s, 2H).

δ_c (100 MHz, *d*-CDCl₃) δ : 163.5, 139.0, 138.6, 129.3, 106.5, 52.3. **IR** (cast film, cm⁻¹) 3058, 2967, 1548, 1520, 1047, 654. **Mp**: 145-146 °C. **HRMS** (CI) *m/z* for C₈H₅I₃O₂ [M⁺]: calcd. 513.7424; found, 513.7415.

3.9 Synthesis of 1,2-diiodo-3-methylbenzene (29)

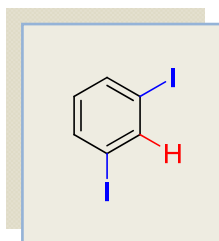


The title compound was prepared using the general procedure for *ipso*-iododecarboxylation of benzoic acid derivatives and isolated in **53%** yield as a white solid. The spectroscopic data for this compound are matched our previous report by *J. Org. Chem.*, **1991**, *56*, 6573-6579.

4. General Procedure for Metal-Halogen Exchange Reaction of 1,2,3-triiodoarenes

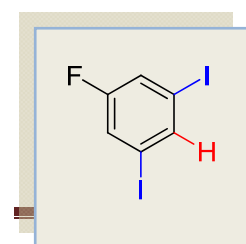
To a solution of triiodoarene (0.66 mmol, 1.0 equiv.) in 15 mL of a mixture of THF at -78 °C was added dropwise isopropyl magnesium chloride (2M in THF, 0.23 mL, 0.70 mmol). The mixture was stirred at that temperature for 2 h. The electrophile was then added and the solution was slowly warmed to room temperature and stirred overnight. Saturated NH₄Cl was added and the resulting mixture was stirred for 30 min. The aqueous layer was extracted with Et₂O (2 x 50 mL). The organic layer was washed with brine, dried with anhydrous Na₂SO₄, 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 1,3-diiodobenzene (30)



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 *Adv. Syn. Cat.*, **2011** 353(9), 1438-1442.

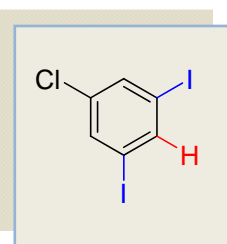
4.2 Synthesis of 1-fluoro-3,5-diiodobenzene (31)



The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **67%** yield as a white solid. The spectroscopic data for this compound are

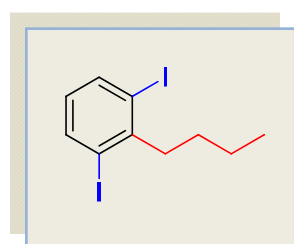
matched the previous report by *Revue Roumaine d Chimie*, **1989**, 34(3), 807-810.

4.3 Synthesis of 1-chloro-3,5-diiodobenzene (32)



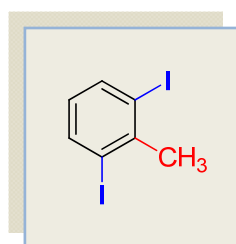
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **69%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Aust. J. Chem.* **2013**, 66, 1570–1575.

4.4 Synthesis of 2-butyl-1,3-diiodobenzene (33)



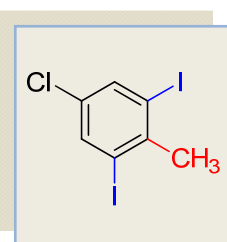
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **68%** yield as colorless oil. δ_{H} (300 MHz, *d*-CDCl₃) δ : 7.80 (d, 2H, *J* = 7.8 Hz), 6.47 (t, 1H, *J* = 7.8 Hz), 3.04 (t, 2H, *J* = 6.9 Hz), 1.52 (m, 4H), 1.01 (t, 3H, *J* = 6.9 Hz). δ_{C} (75 MHz, *d*-CDCl₃) δ : 146.5, 140.0, 129.1, 98.9, 46.6, 30.4, 22.8, 13.8. **IR** (cast film, cm⁻¹) 3050, 2971, 1612, 1594, 1217, 1078, 684. **HRMS** (CI) *m/z* for C₁₀H₁₂I₂ [M⁺]: calcd. 385.9028; found, 385.9016.

4.5 Synthesis of 1,3-diiodo-2-methylbenzene (34)



The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in 64% yield as colorless oil. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.82 (d, 2H, *J* = 8.0 Hz), 6.49 (t, 1H, *J* = 8.0 Hz), 2.75 (s, 3H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 142.5, 139.2, 128.6, 99.0, 34.6. **IR** (cast film, cm⁻¹) 3102, 2942, 2810, 1583, 1512, 1389, 1125, 1014, 894. **HRMS** (CI) *m/z* for C₇H₆I₂ [M⁺]: calcd. 343.8559; found, 343.8548.

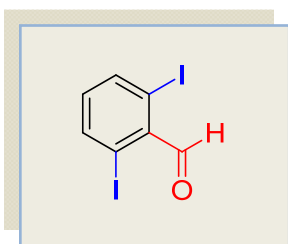
4.6 Synthesis of 5-chloro-1,3-diiodo-2-methylbenzene (35)



The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **54%** yield as a white solid. The spectroscopic data for this compound are matched the previous report by *Aust. J. Chem.* **2013**, 66, 1570–

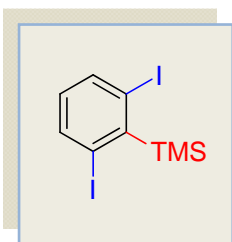
1575.

4.7 Synthesis of (2,6-diiodophenyl)(pyridin-2-yl)methanol (36)



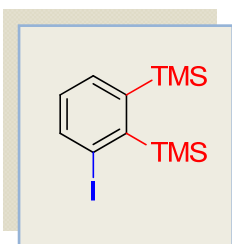
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **61%** yield as colorless oil. δ_{H} (400 MHz, *d*-CDCl₃) δ : 9.79 (s, 1H), 8.02 (d, 2H, *J* = 8.0 Hz), 6.68 (t, 1H, *J* = 8.0 Hz). δ_{C} (100 MHz, *d*-CDCl₃) δ : 195.7, 141.6, 135.6, 134.6, 97.5. **IR** (cast film, cm⁻¹) 3114, 2974, 2711, 1713, 1234, 1019, 787. **HRMS** (CI) *m/z* for C₇H₄I₂O [M⁺]: calcd. 357.8352; found, 357.8347.

4.8 Synthesis of (2,6-diiodophenyl)trimethylsilane (37)



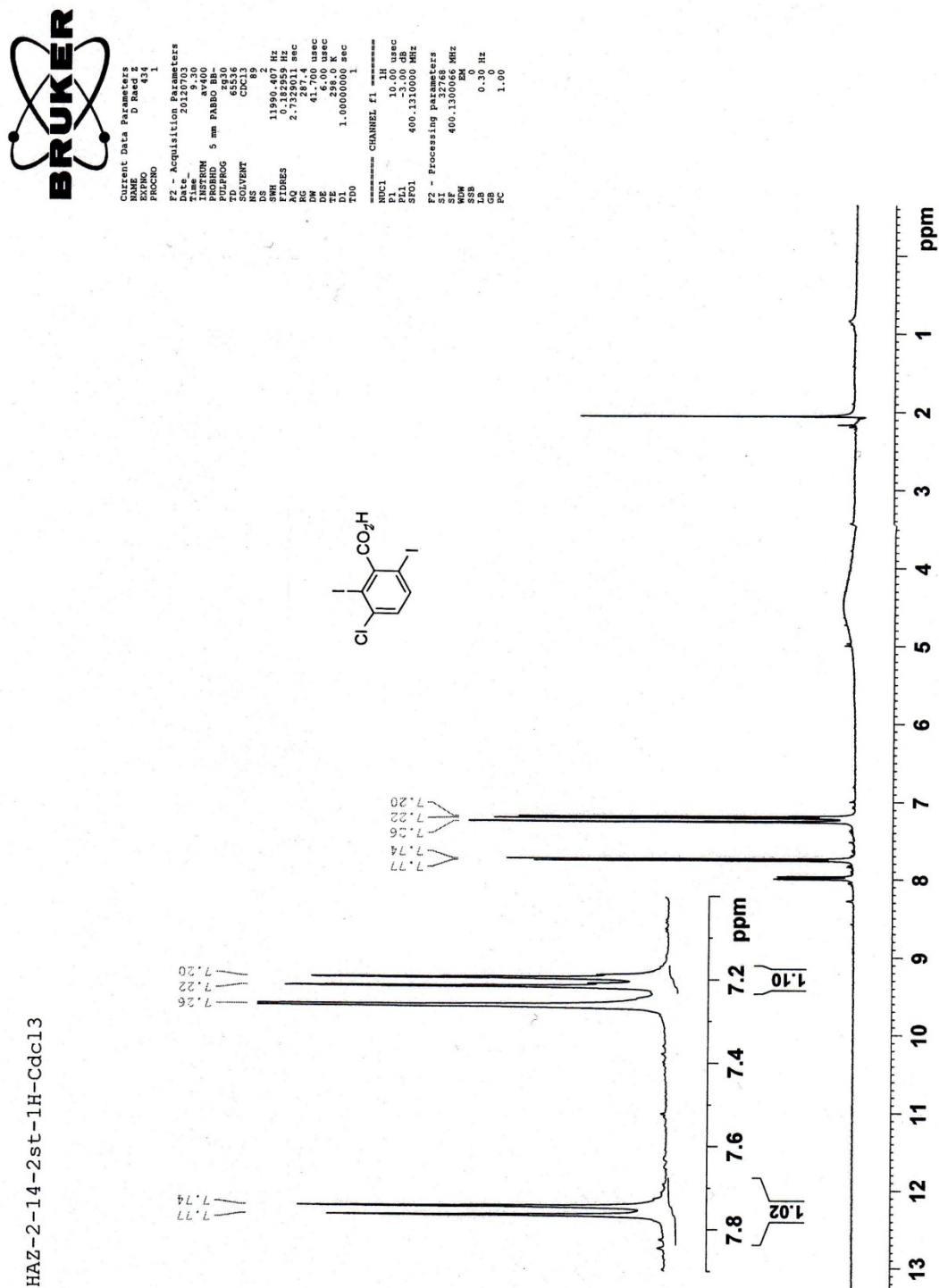
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **70%** yield as colorless oil. δ_{H} (500 MHz, *d*-CDCl₃) δ : 7.94 (d, 2H, *J* = 7.5 Hz), 6.53 (t, 1H, *J* = 7.5 Hz), 0.64 (s, 9H). δ_{C} (125 MHz, *d*-CDCl₃) δ : 141.4, 131.2, 103.3, 4.9. **IR** (cast film, cm⁻¹) 2986, 2915, 1589, 1468, 1269, 1146, 849. **HRMS** (CI) *m/z* for C₉H₁₂I₂Si [M⁺]: calcd. 401.8798; found, 401.8788.

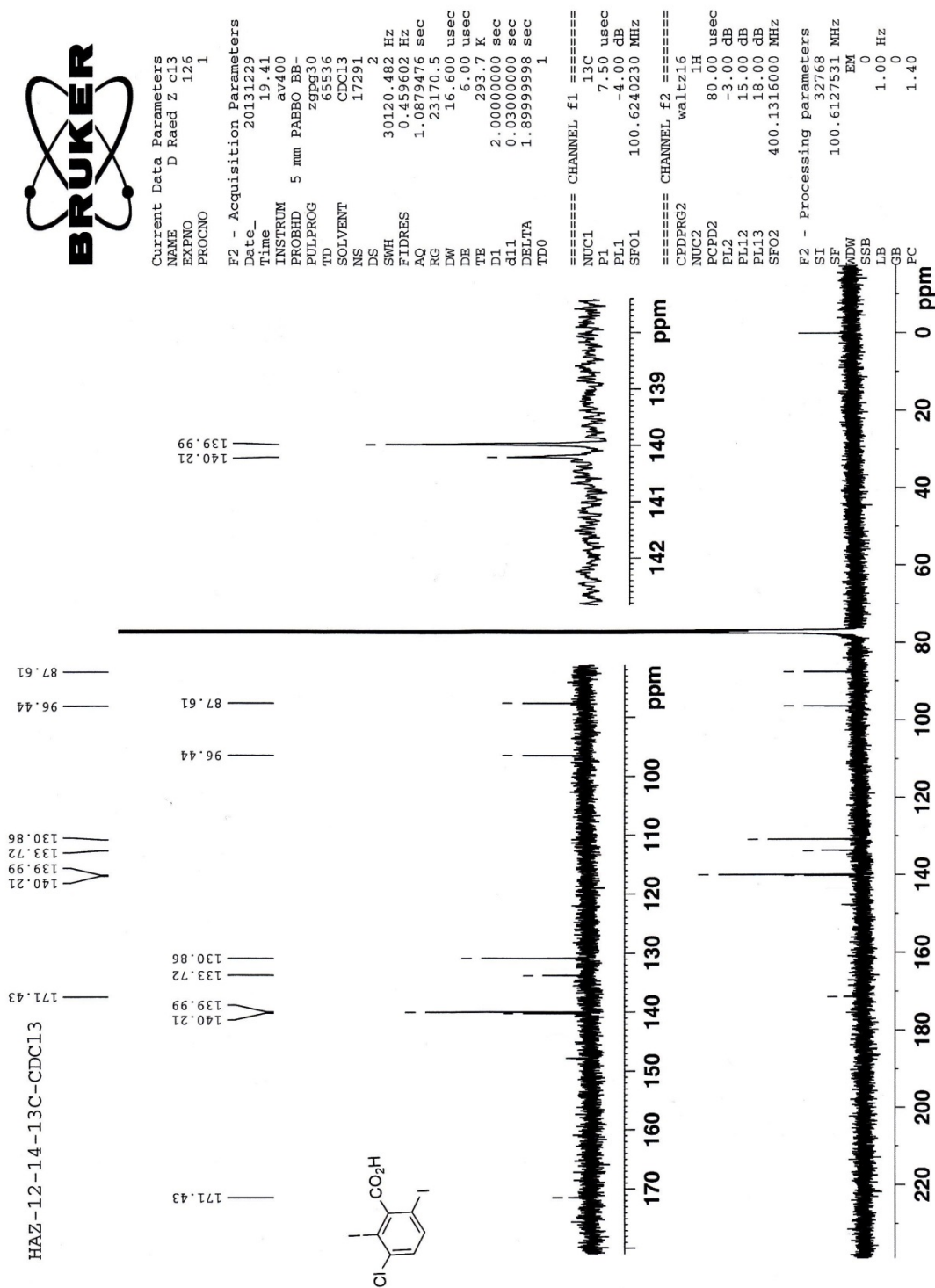
4.9 Synthesis of (3-iodo-1,2-phenylene)bis(trimethylsilane) (38)

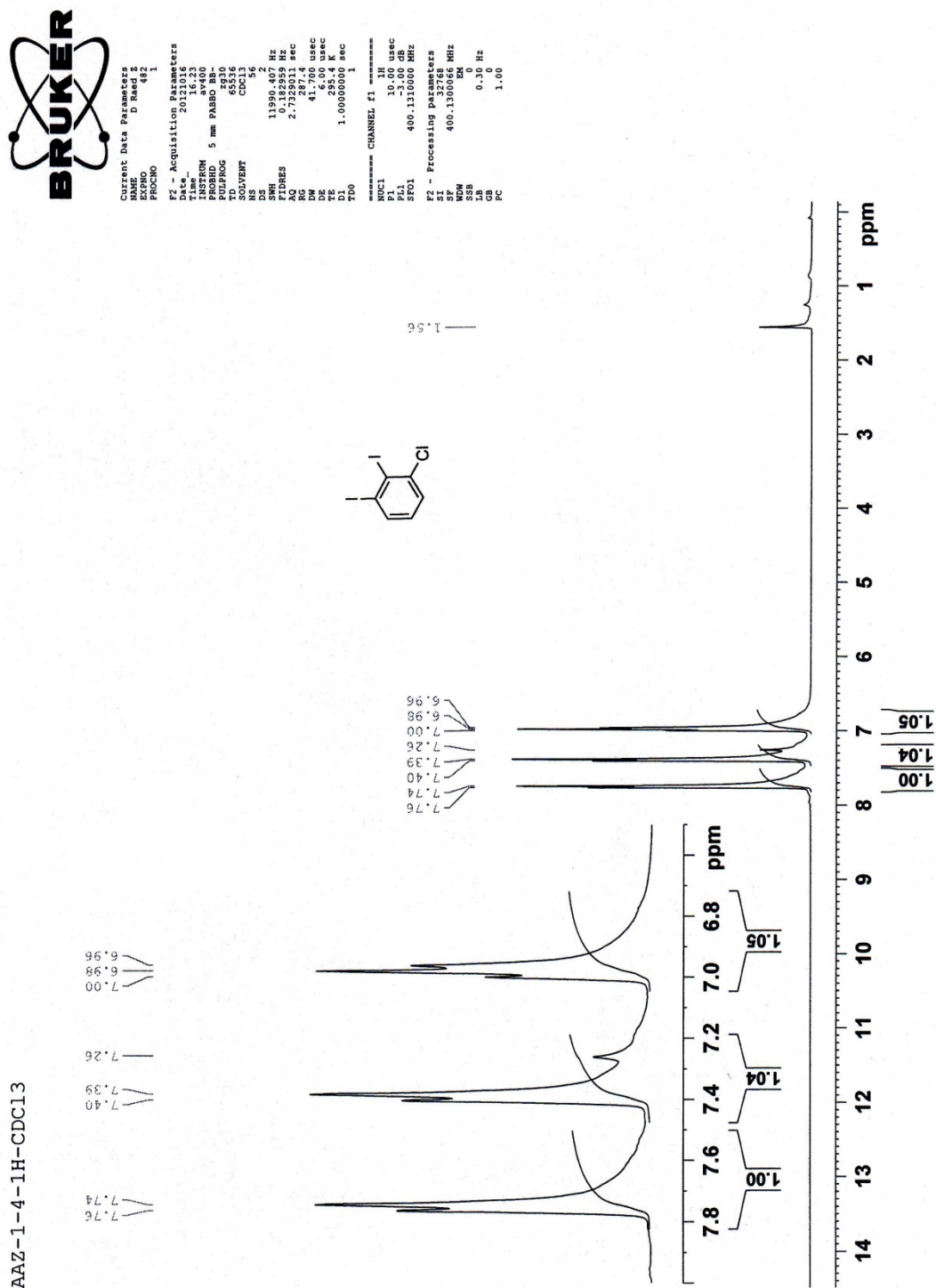


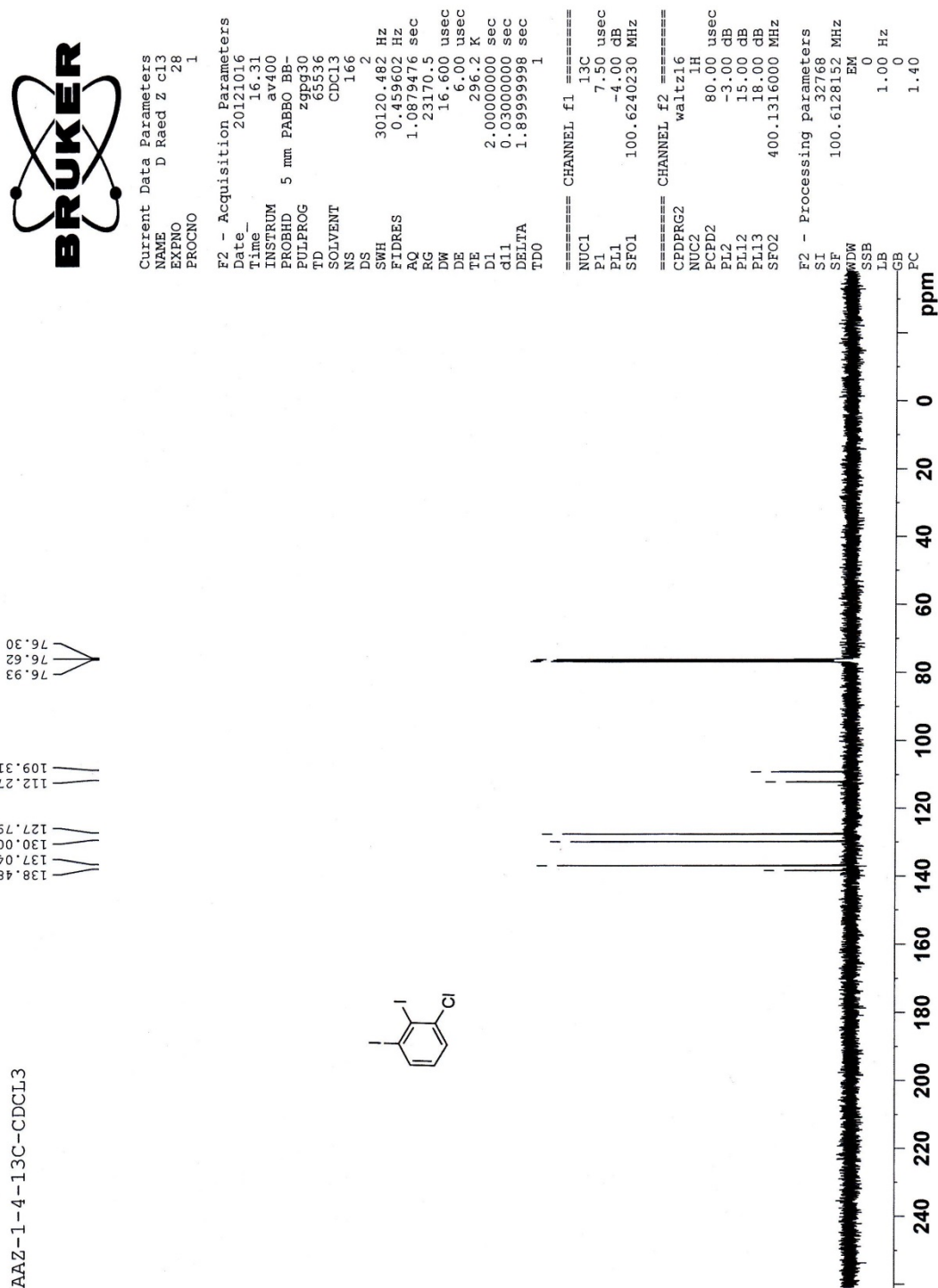
The title compound was prepared using the general procedure for Metal-Halogen exchange reaction and isolated in **34%** yield as colorless oil. δ_{H} (400 MHz, *d*-CDCl₃) δ : 7.88 (dd, 1H, *J* = 8.0, *J* = 2.0 Hz), 7.60 (dd, 1H, *J* = 8.0 Hz, *J* = 2.0 Hz), 6.88 (t, 1H, *J* = 8.0 Hz), 0.55 (s, 9H), 0.36 (s, 9H). δ_{C} (100 MHz, *d*-CDCl₃) δ : 151.0, 150.8, 140.5, 134.1, 127.9, 107.2, 3.43, 2.54. **IR** (cast film, cm⁻¹) 2984, 2946, 1612, 1578, 1272, 1269, 1146, 844, 789. **HRMS** (CI) *m/z* for C₁₂H₂₁ISi₂ [M⁺]: calcd. 348.0226; found, 348.0217.

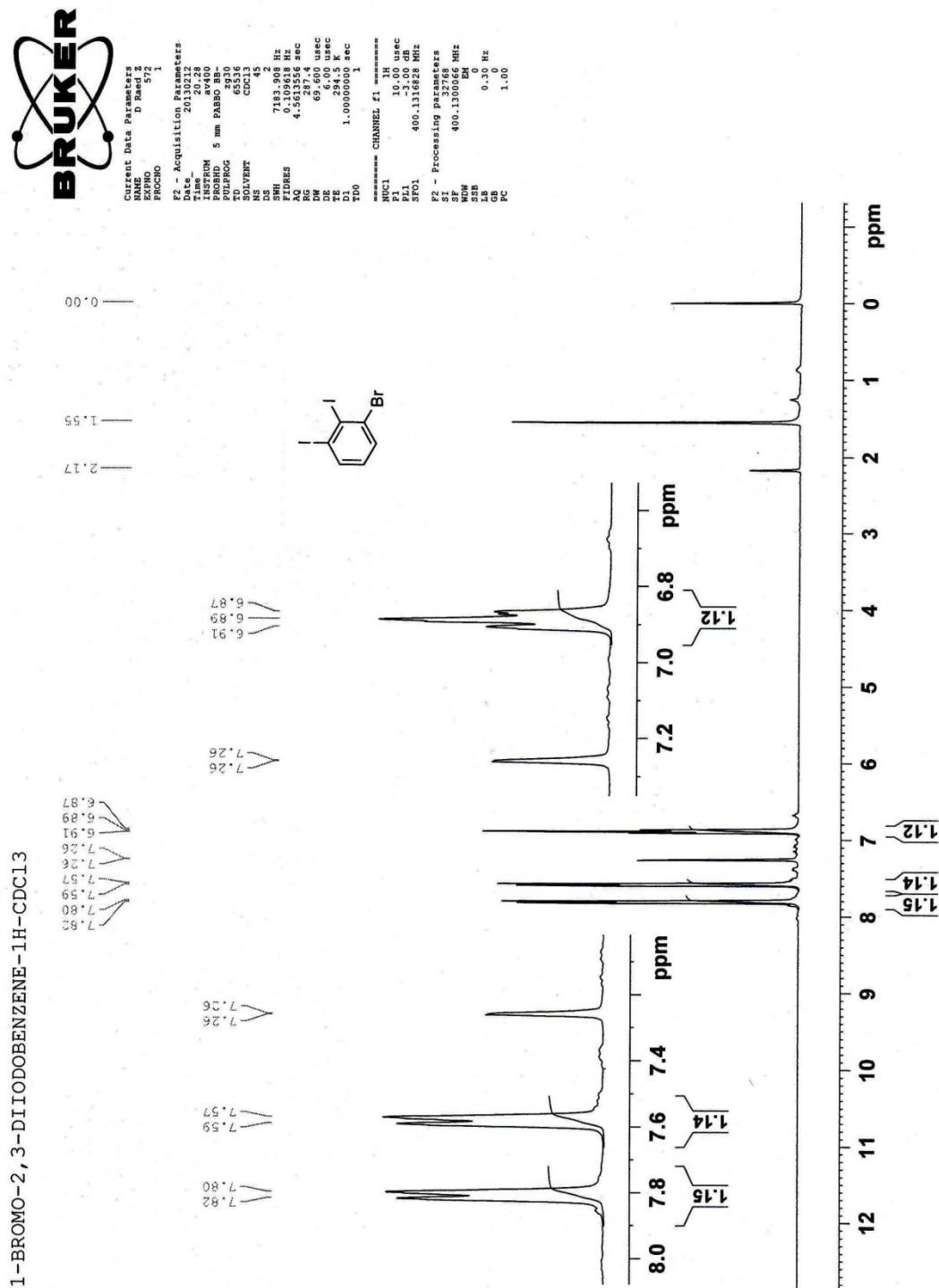
5. NMR Spectra for New Compounds

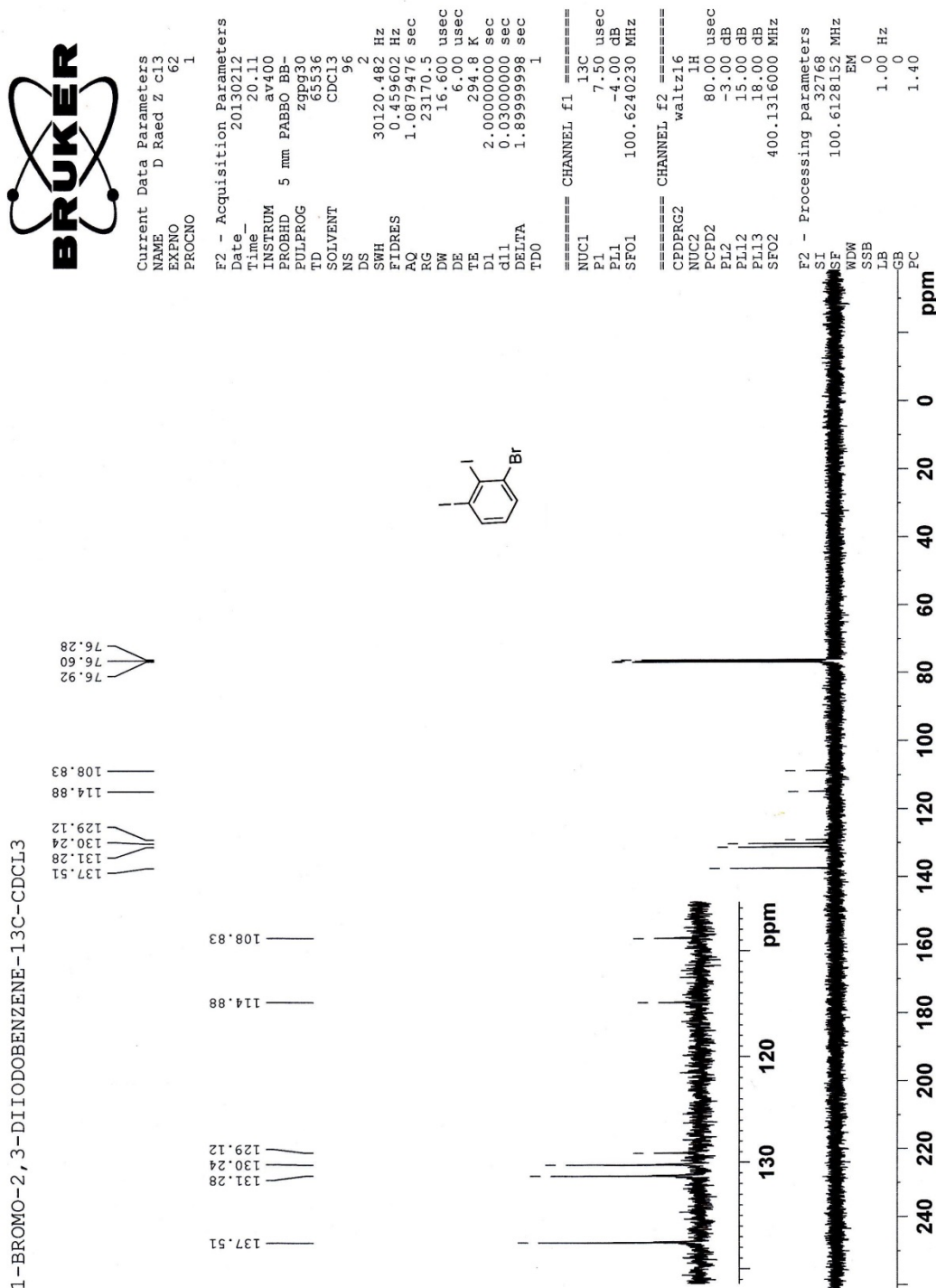
5.1 $^1\text{H-NMR}$ of 3-chloro-2,6-diiodobenzoic acid (15) in $d\text{-CDCl}_3$ at 25 °C.

5.2 ^{13}C -NMR of 3-chloro-2,6-diiodobenzoic acid (15) in $d\text{-CDCl}_3$ at 25 °C.

5.3 $^1\text{H-NMR}$ of 1-chloro-2,3-diiodobenzene (24) in $d\text{-CDCl}_3$ at 25 °C.

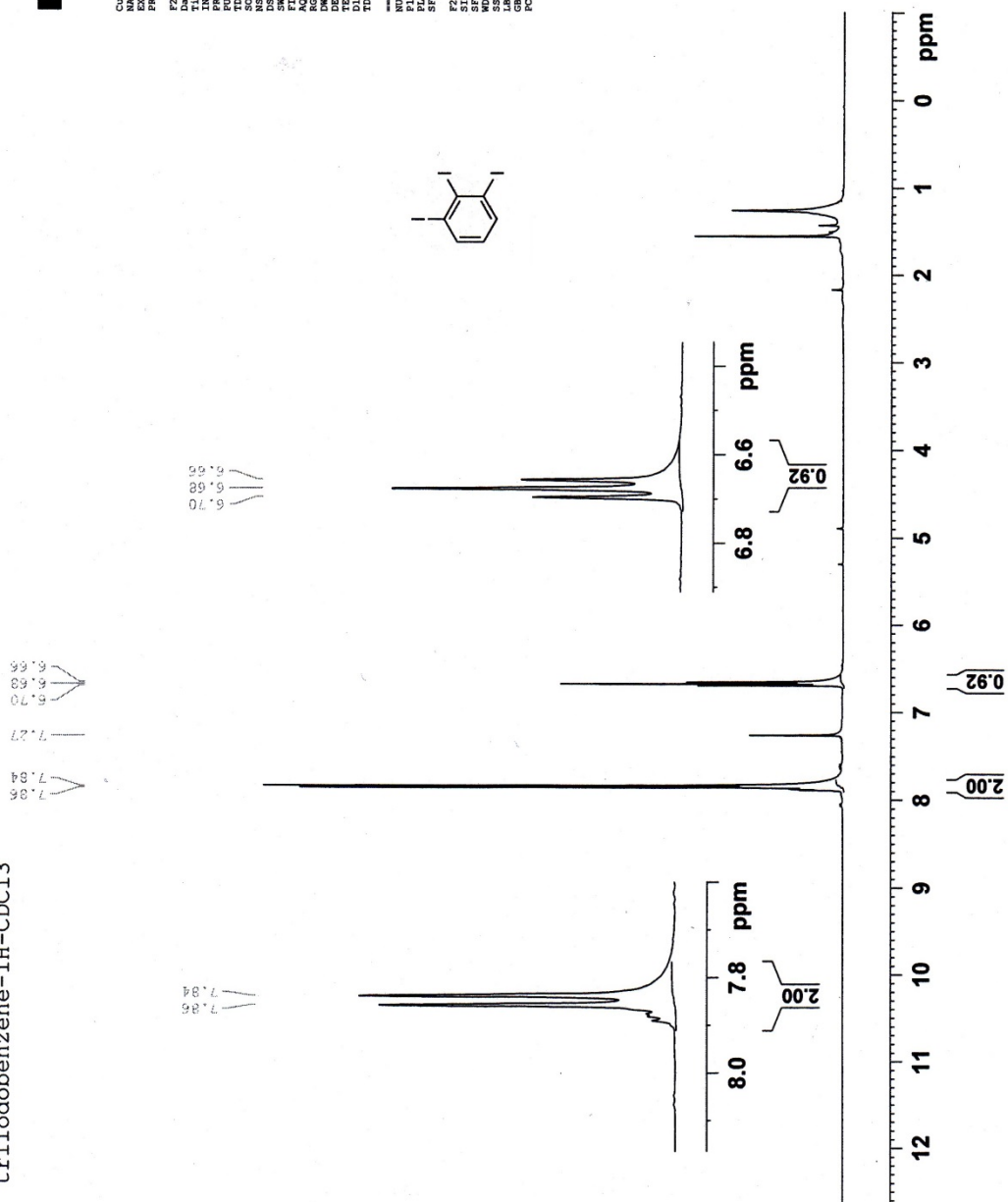
5.4 ^{13}C -NMR of 1-chloro-2,3-diiodobenzene (24) in $d\text{-CDCl}_3$ at 25 °C.

5.5 ¹H-NMR of 1-bromo-2,3-diiodobenzene (25) in *d*-CDCl₃ at 25 °C.

5.6 ^{13}C -NMR of 1-bromo-2,3-diiodobenzene (25) in $d\text{-CDCl}_3$ at 25 °C.

5.7 $^1\text{H-NMR}$ of 1,2,3-triiodobenzene (26) in $d\text{-CDCl}_3$ at 25 °C.

Current Data Parameters
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 SOLVENT CDCl3
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 SH 11990.407 Hz
 FIDRES 0.182959 Hz
 AQ 2.772911 sec
 RG 327
 DM 41.700 usec
 DE 6.00 usec
 D1 1.0000000 sec
 TDO 1
 CHANNEL f1
 NUC1 ^1H
 P1 10.00 usec
 PL1 -1.00 dB
 SFO1 400.1310000 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300086 MHz
 SD 0
 LB 0.30 Hz
 GB 0
 PC 1.00

triiodobenzene- $^1\text{H-CDCl}_3$ 

5.8 ^{13}C -NMR of 1,2,3-triiodobenzene (26) in $d\text{-CDCl}_3$ at 25 °C.

Current Data Parameters
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 EXPNO 18
 PROCNO 1

F2 - Acquisition Parameters

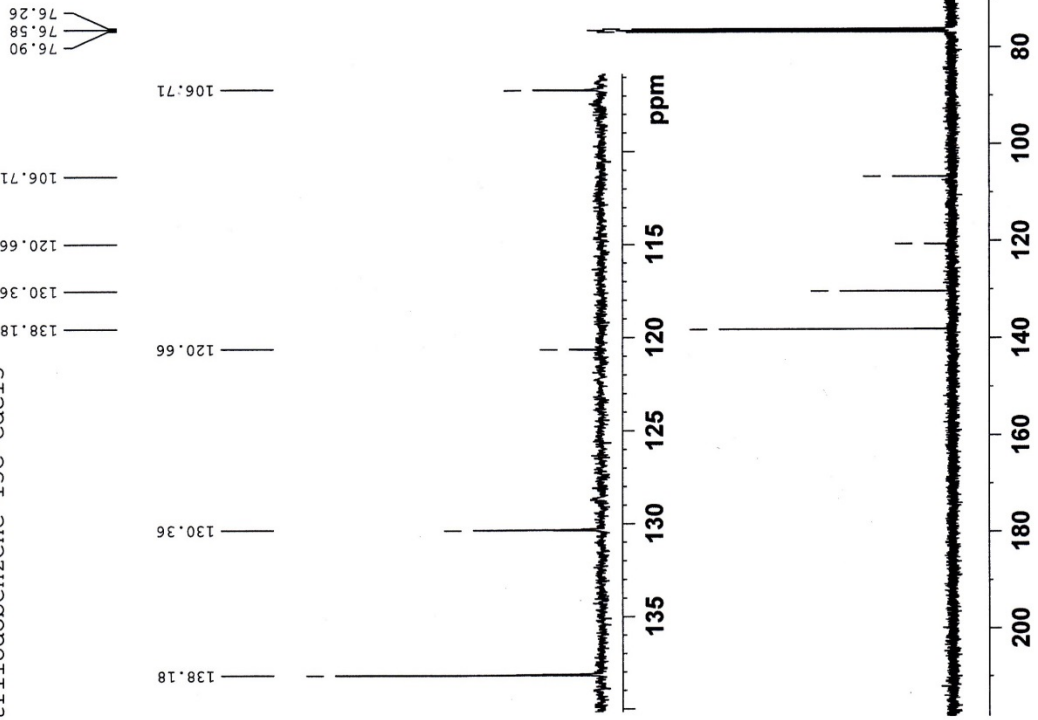
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 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

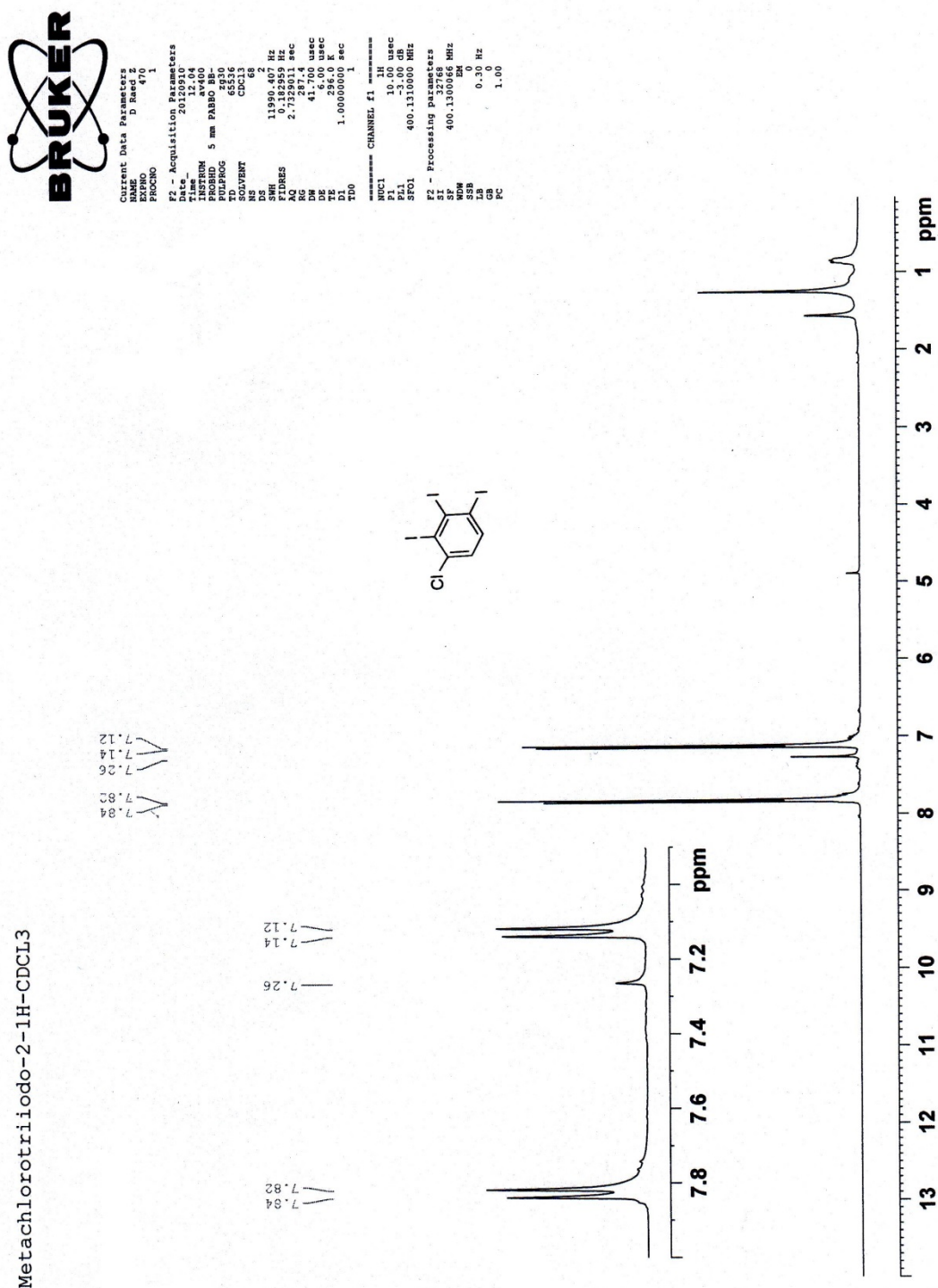
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 SF01 100.6240230 MHz

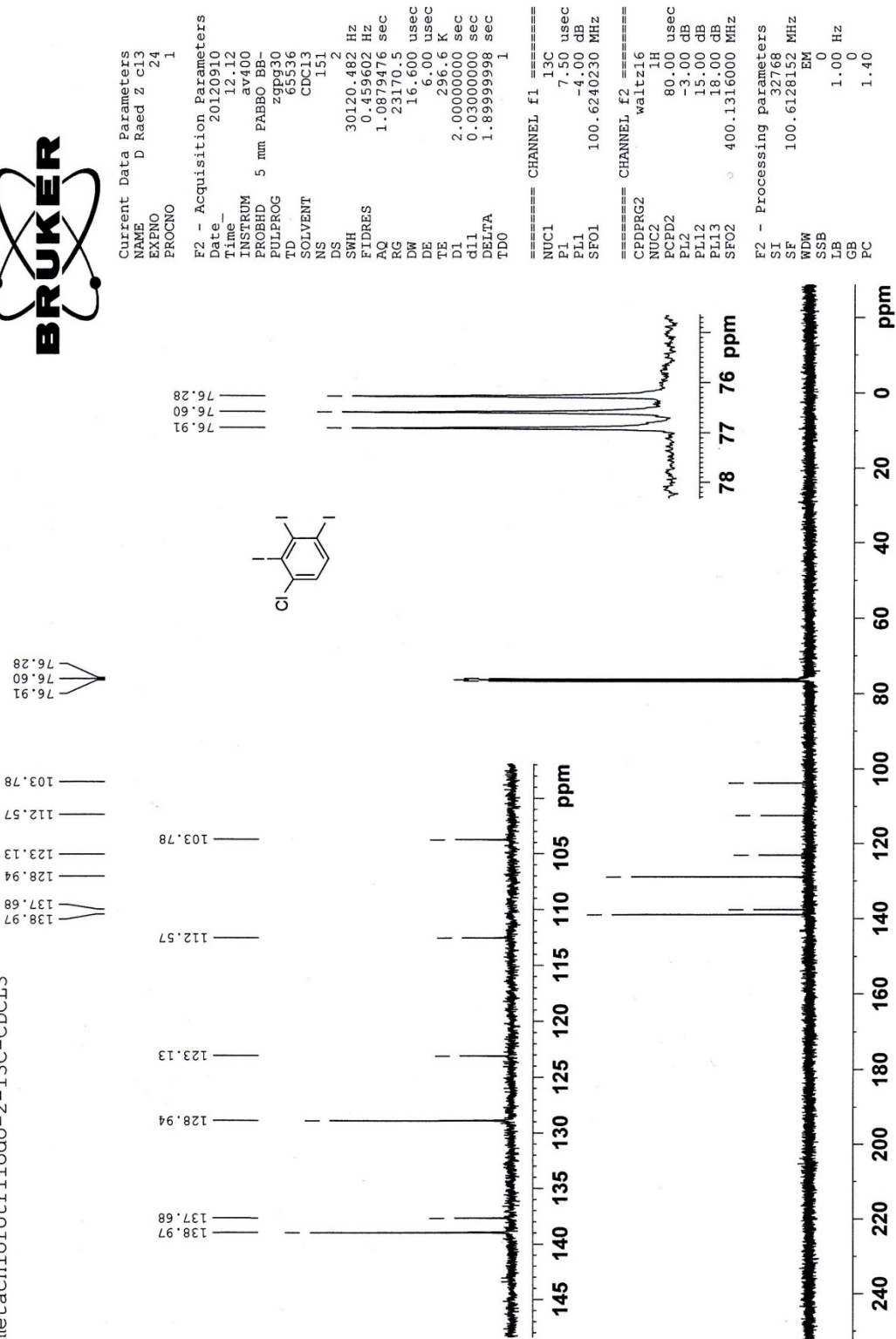
===== CHANNEL f2 =====
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 F2PRG2 80.00 usec
 PL2 -3.00 dB
 PL12 15.00 dB
 PL13 18.00 dB
 SFO2 400.1316000 MHz

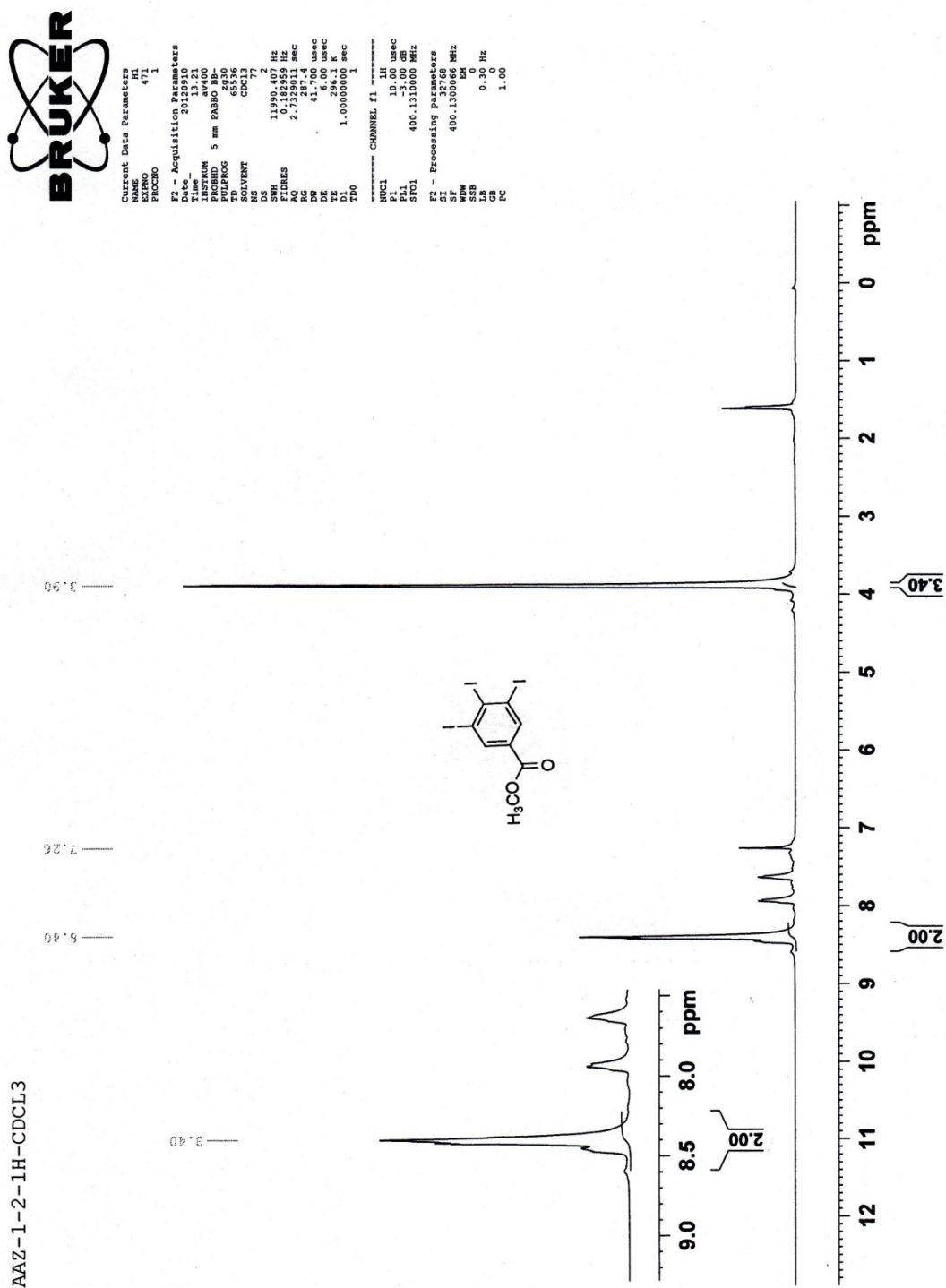
F2 - Processing parameters
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 GB 0
 PC 1.40

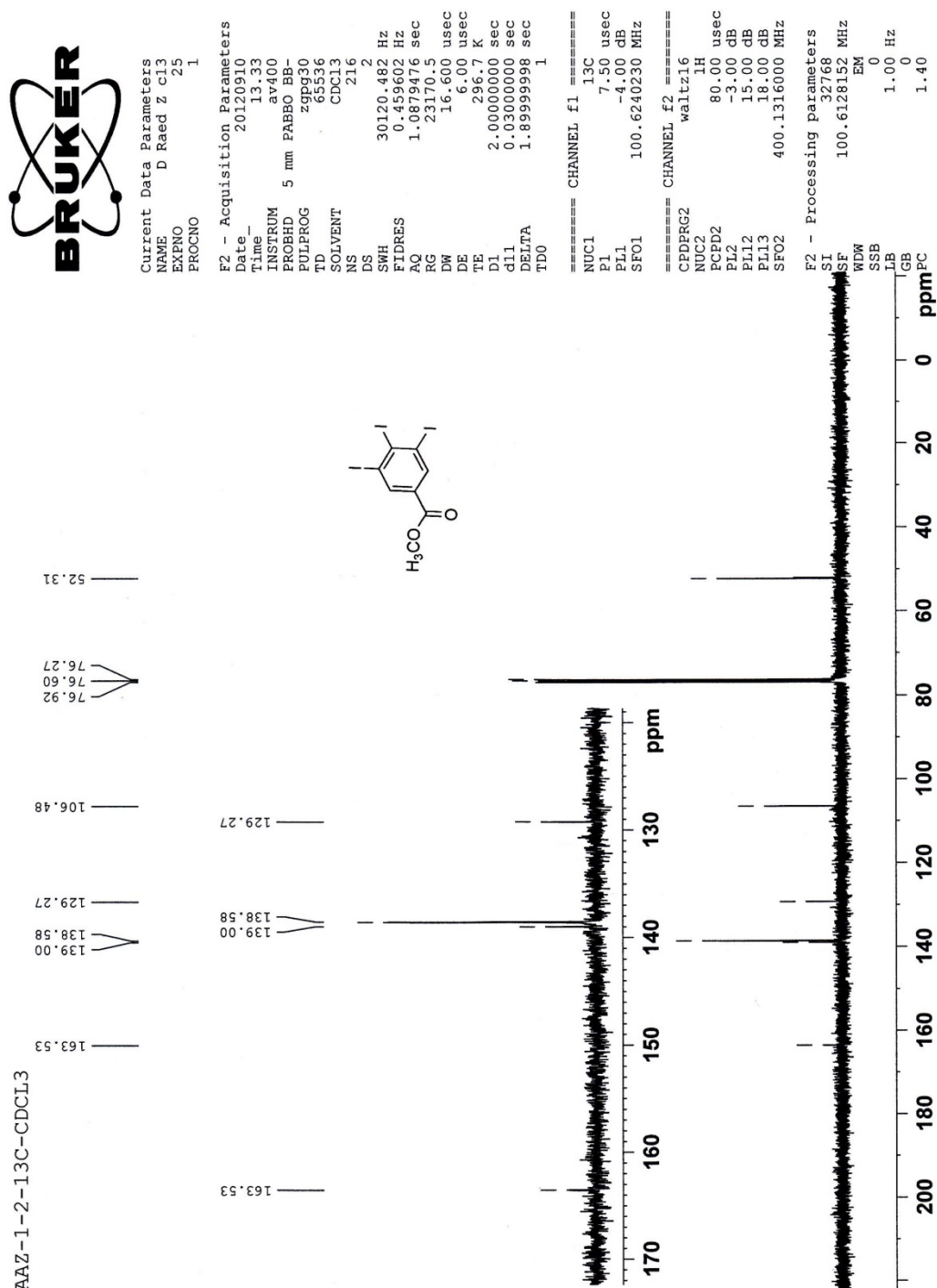
triiodobenzene- ^{13}C -cdcl3

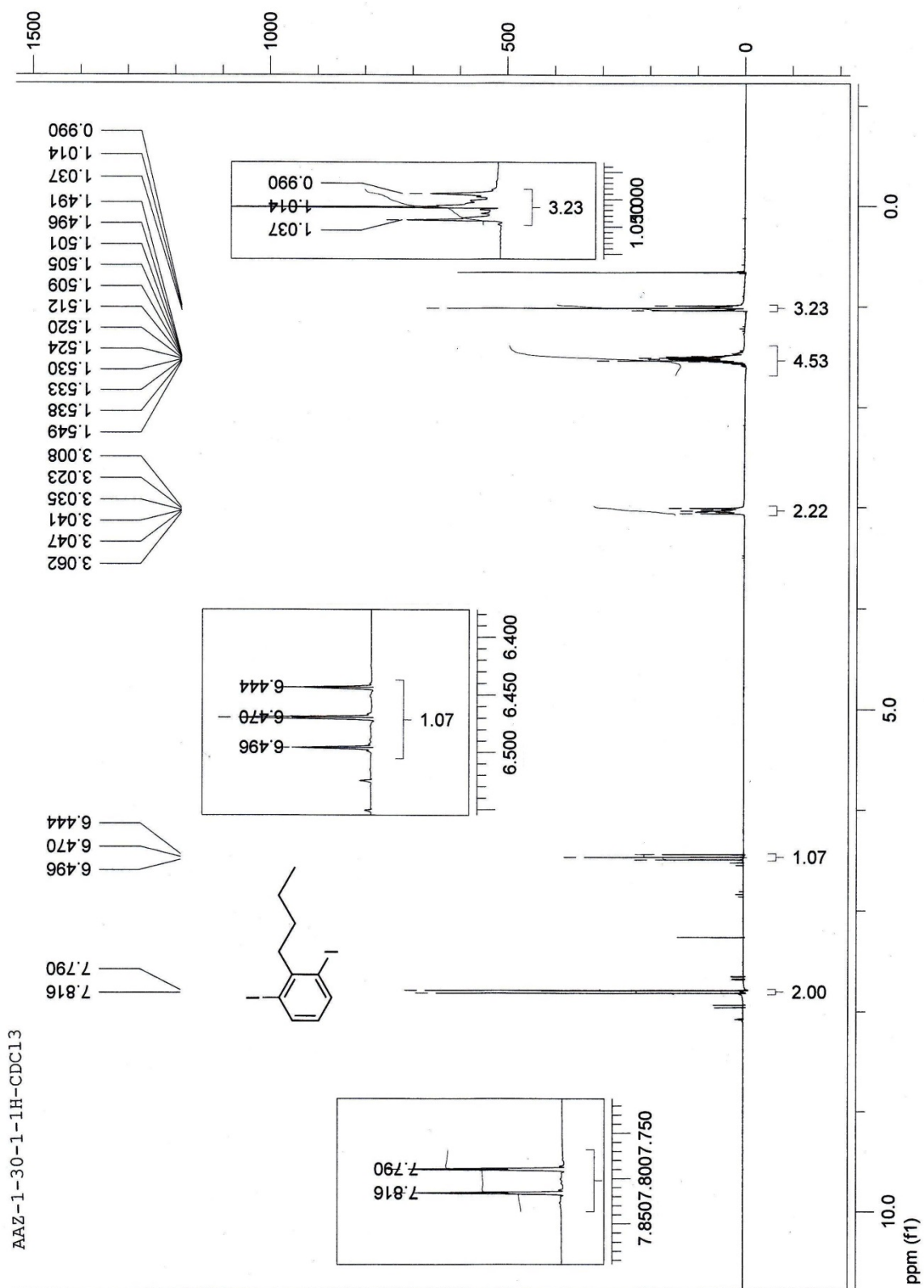


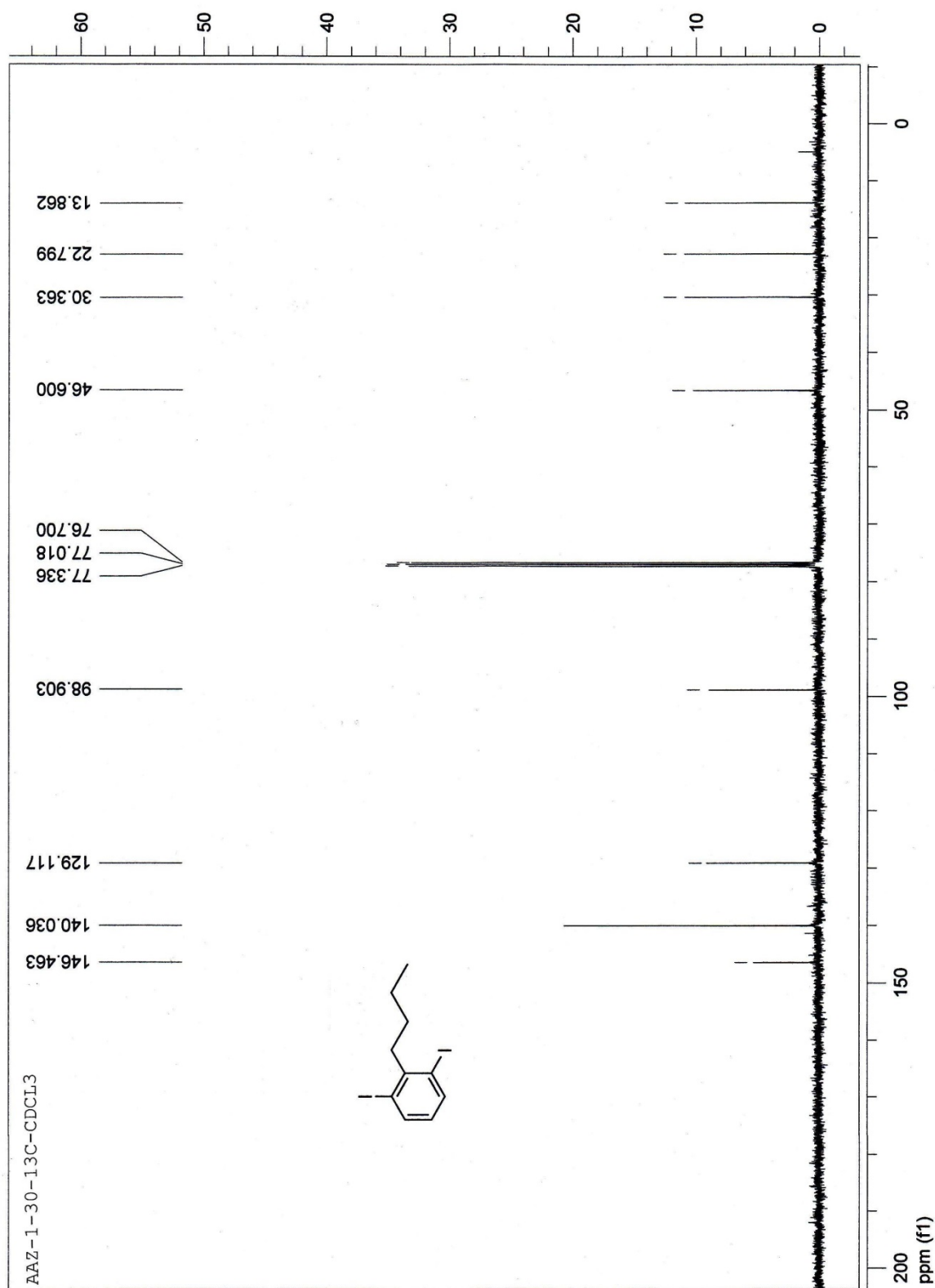
5.9 $^1\text{H-NMR}$ of 1-chloro-2,3,4-triiodobenzene (27) in $d\text{-CDCl}_3$ at 25 °C.

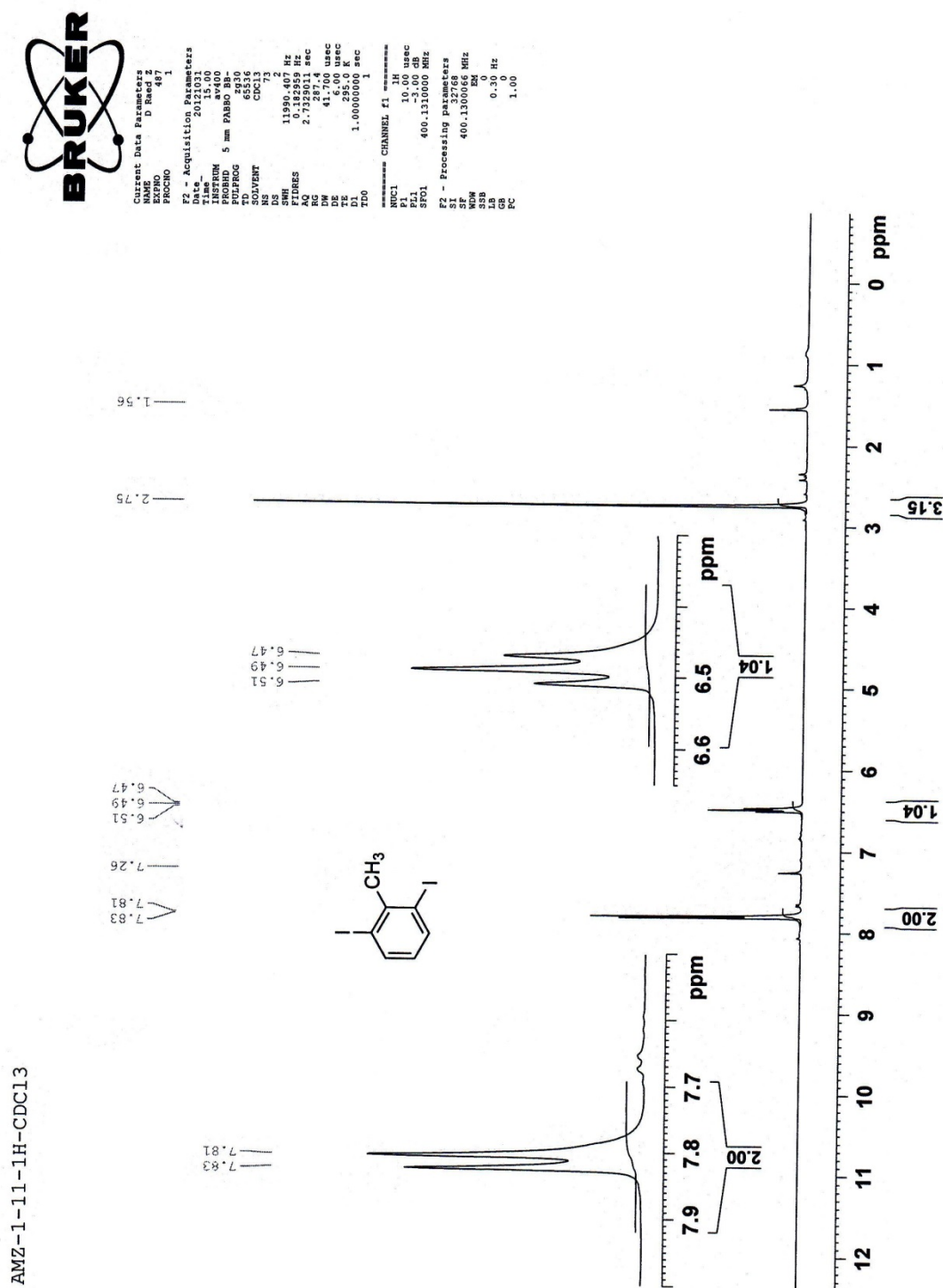
5.10 ^{13}C -NMR of 1-chloro-2,3,4-triiodobenzene (27) in $d\text{-CDCl}_3$ at 25°C .metachlorotriiodo-2- ^{13}C - CDCl_3 

5.11 $^1\text{H-NMR}$ of methyl 3,4,5-triiodobenzoate (28) in $d\text{-CDCl}_3$ at 25 °C.

5.12 ^{13}C -NMR of methyl 3,4,5-triiodobenzoate (28) in $d\text{-CDCl}_3$ at 25 °C.

5.13 $^1\text{H-NMR}$ of 2-butyl-1,3-diiodobenzene (33) in $d\text{-CDCl}_3$ at 25 °C.

5.14 ^{13}C -NMR of 2-butyl-1,3-diiodobenzene (33) in $d\text{-CDCl}_3$ at 25 °C.

5.15 $^1\text{H-NMR}$ of 1,3-diiodo-2-methylbenzene (34) in $d\text{-CDCl}_3$ at 25 °C.

5.16 ^{13}C -NMR of 1,3-diiodo-2-methylbenzene (34) in $d\text{-CDCl}_3$ at 25 °C.

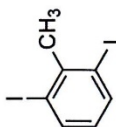
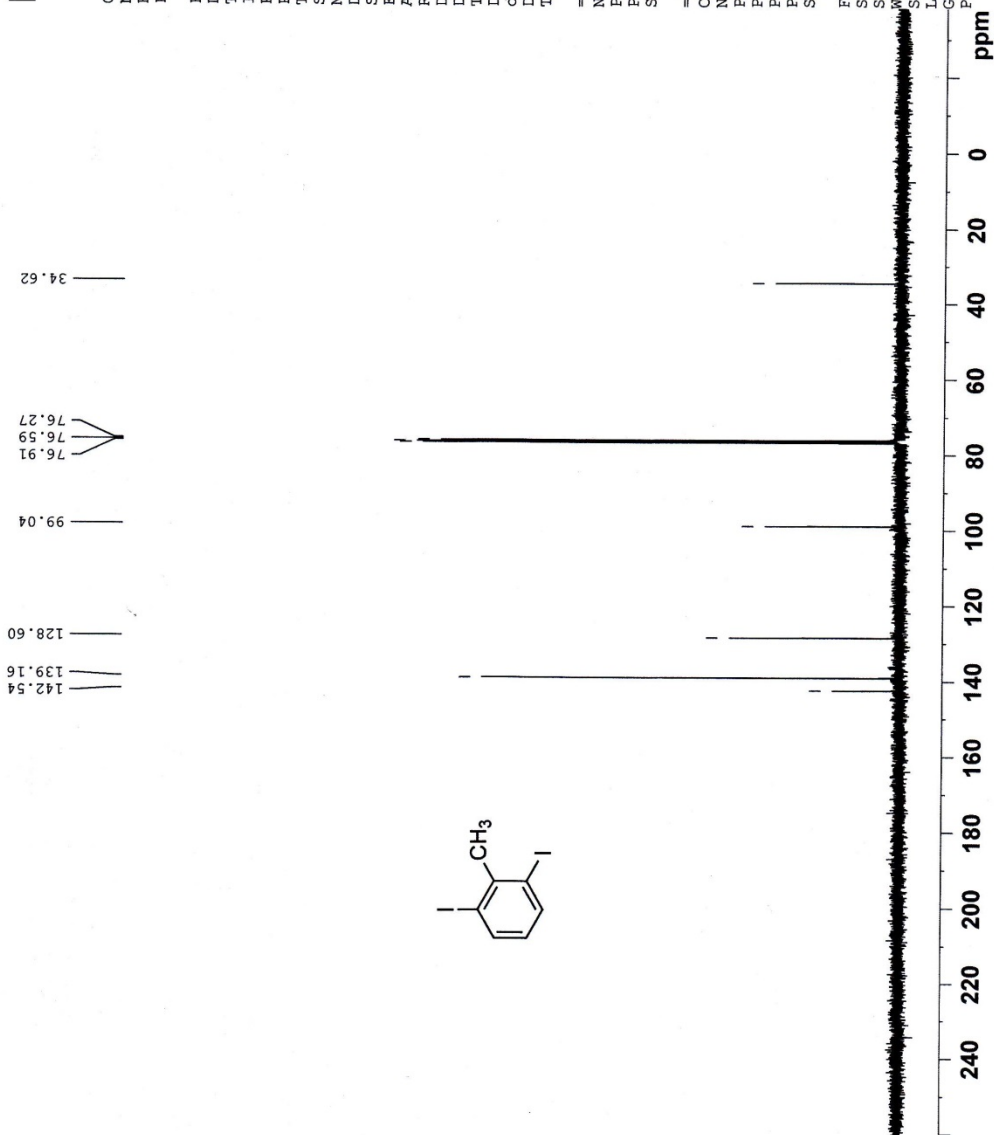
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 PROCNO 1

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 DS 2
 SWH 30120.482 Hz
 FIDRES 0.459602 Hz
 AQ 1.0879476 sec
 RG 23170.5
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 DE 6.00 usec
 TE 295.5 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TDO 1

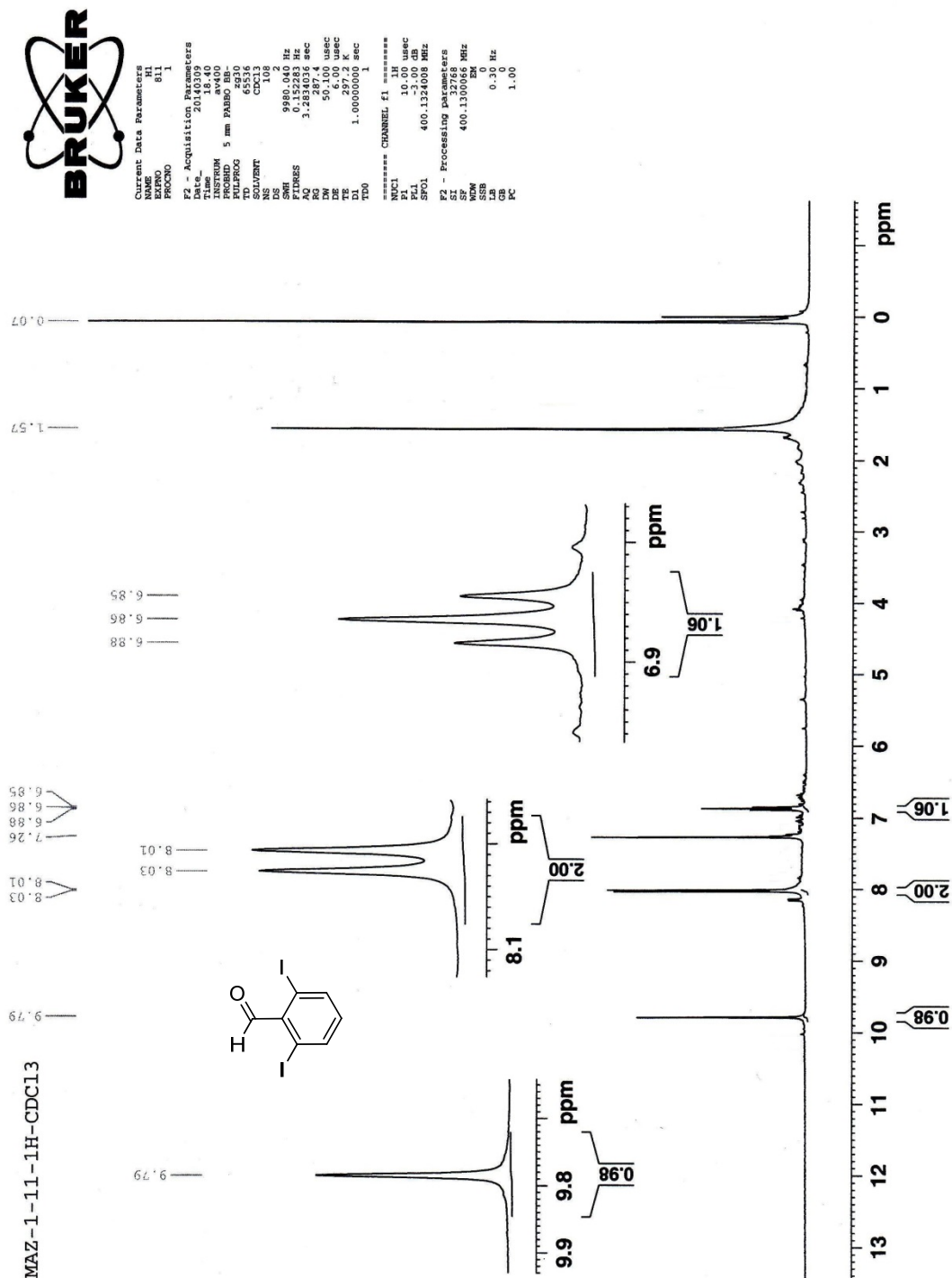
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 P1 7.50 usec
 PL1 -4.00 dB
 SF01 100.6240230 MHz

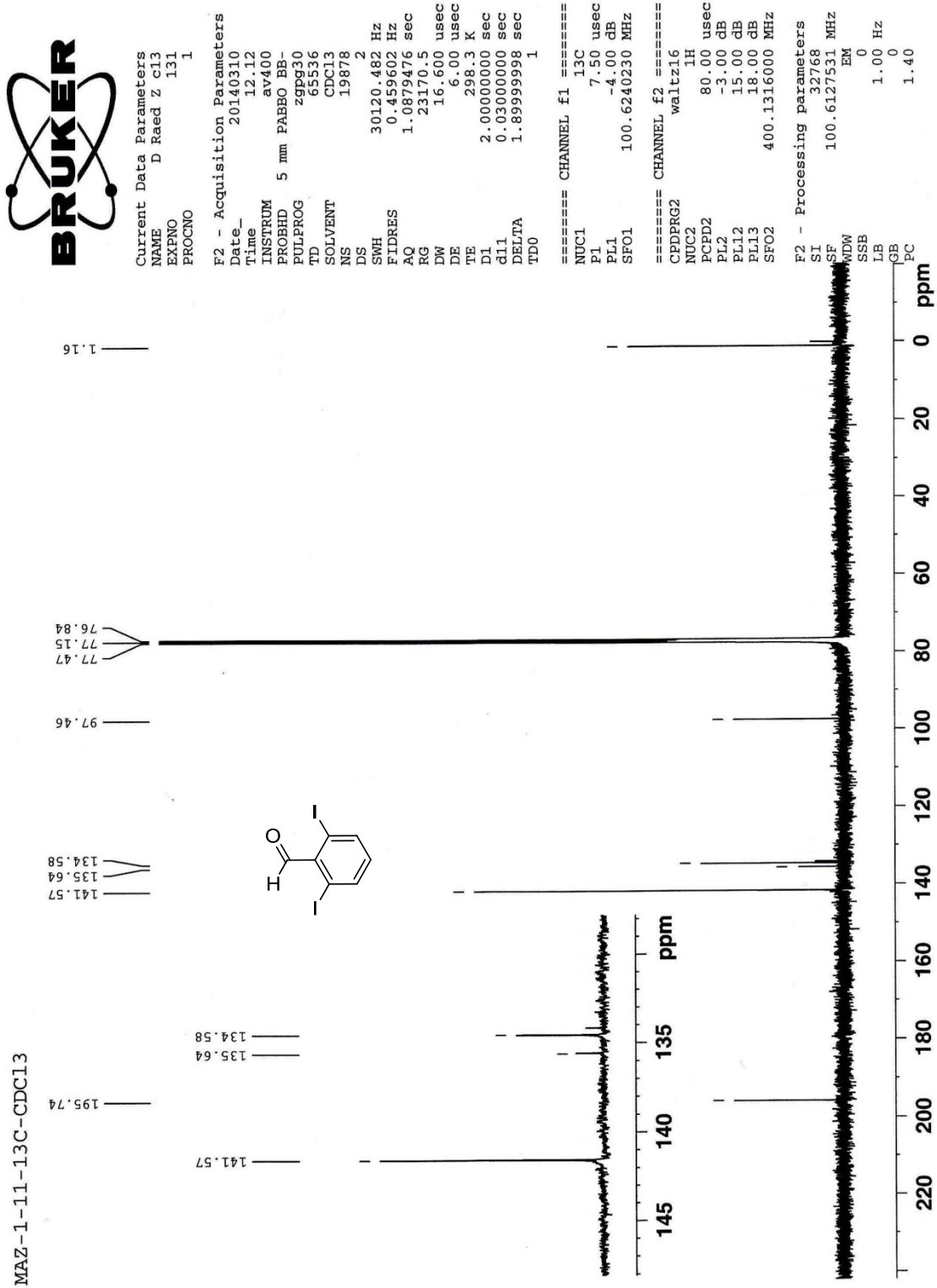
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ^1H
 FCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.00 dB
 PL13 18.00 dB
 SF02 400.1316000 MHz

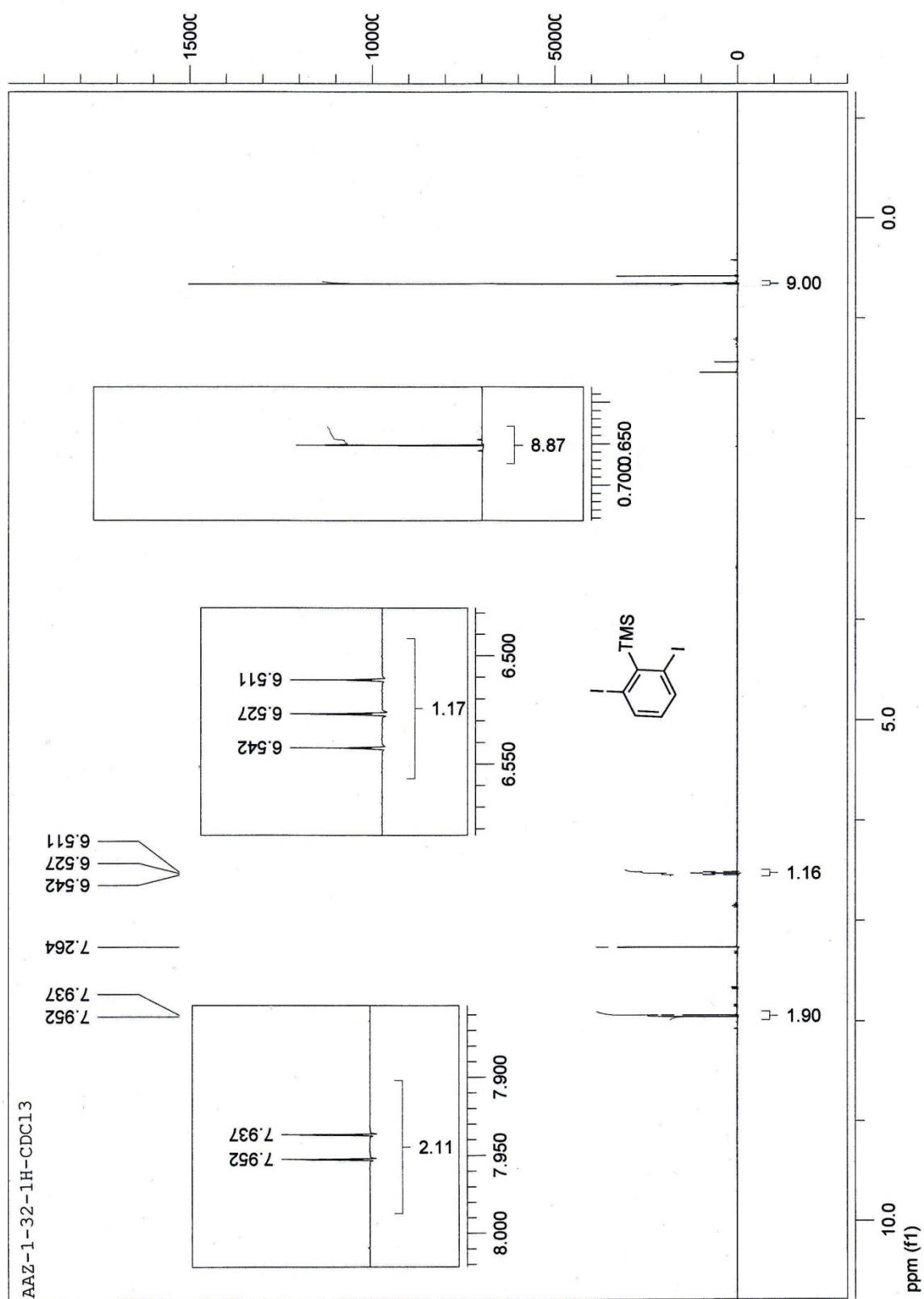
F2 - Processing parameters
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 SF 100.6128152 MHz
 EM 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

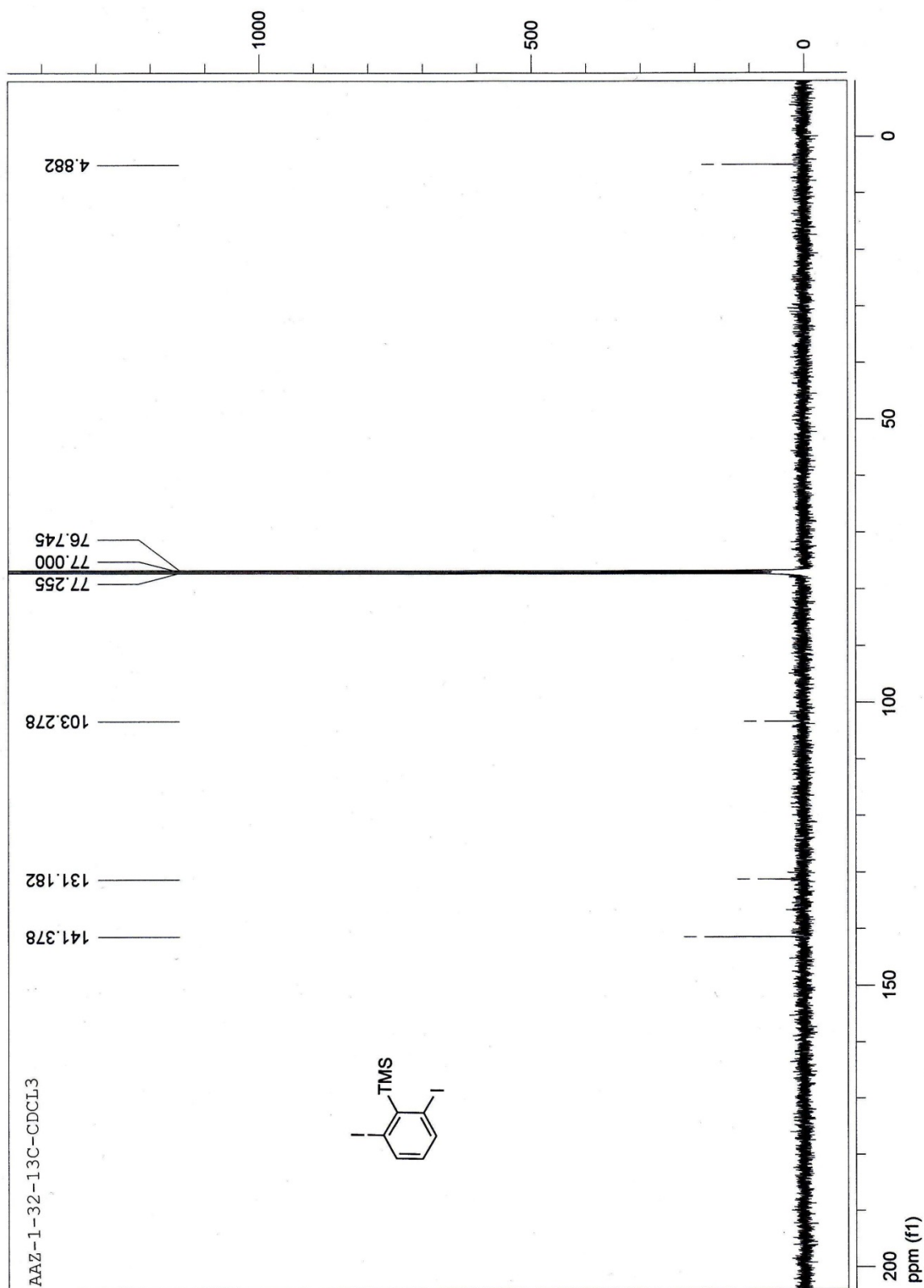


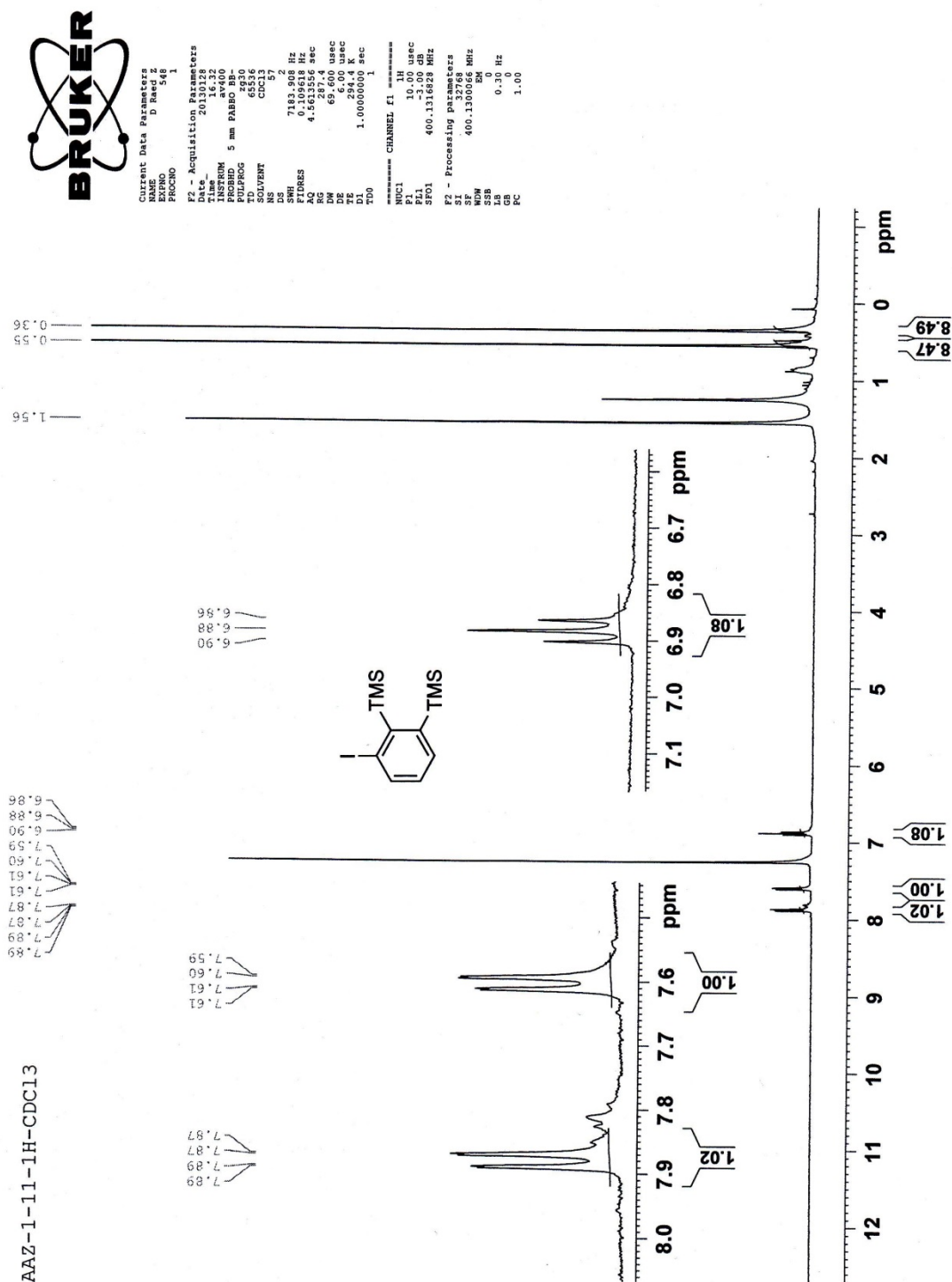
AMZ-1-11-13C-CDCL3

5.17 ¹H-NMR of 2,6-diiodobenzaldehyde (36) in *d*-CDCl₃ at 25 °C.

5.18 ^{13}C -NMR of 2,6-diiodobenzaldehyde (36) in *d*- CDCl_3 at 25 °C.

5.19 $^1\text{H-NMR}$ of (2,6-diiodophenyl)trimethylsilane (37) in $d\text{-CDCl}_3$ at 25 °C.

5.20 ^{13}C -NMR of (2,6-diiodophenyl)trimethylsilane (37) in $d\text{-CDCl}_3$ at 25 °C.

5.21 ¹H-NMR of (3-iodo-1,2-phenylene)bis(trimethylsilane) (38) in *d*-CDCl₃ at 25 °C.

5.22 ^{13}C -NMR of (3-iodo-1,2-phenylene)bis(trimethylsilane) (38) in CDCl_3 at 25 °C.