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REGIOSELECTIVE SYNTHESIS OF ORTHO-IODOBIPHENYLBORONIC ACID DERIVATIVES: A SUPERIOR CATALYST FOR CARBOXYLIC ACID ACTIVATION

Raed M. Al-Zoubi, Walid K. Al-Jammal and Robert McDonald*

Supporting Information

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1. Experimental Details and Compound Data.

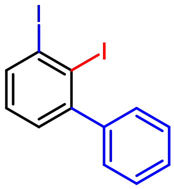
1.1 General Information

All commercial reagents and chromatography solvents were used as obtained unless otherwise stated. Ethanol, toluene, ethyl acetate, hexanes, anhydrous sodium sulfate (Na_2SO_4, BDH). 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 using Chemical Ionization (CI) and Electrospray ionization (ESI) techniques.

1.2 General procedure for the synthesis of 5-substituted-2,3-diiodobiphenyls

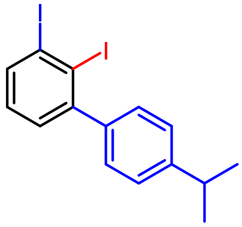
A flame dried round bottom flask was charged with arylboronic acid (0.72 mmol, 1.1 equiv.), 5-substituted-1,2,3-triiodobenzene (0.66 mmol, 1 equiv.), tetrakis-(triphenylphosphine) palladium (12% mol), 4 mL toluene, 2M solution of potassium carbonate (1.4 ml) and ethanol (0.4 ml) under argon. The mixture was heated to 100 ºC for 12 h. The mixture was cooled down to room temperature. The aqueous layer was extracted with ethyl acetate (2 x 50 mL). The organic layers were combined and washed with brine, dried with Na_2SO_4, filtered and then evaporated under reduced pressure. The crude product was purified by flash chromatography to yield the pure desired product.

1.2.1 Synthesis of 2,3-diiodo-1,1'-biphenyl (5)



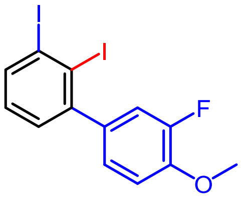
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**93**% yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.2 Synthesis of 2,3-diiodo-4'-isopropyl-1,1'-biphenyl (6)



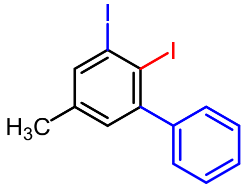
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**83%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.3 Synthesis of 3'-fluoro-2,3-diiodo-4'-methoxy-1,1'-biphenyl (7)



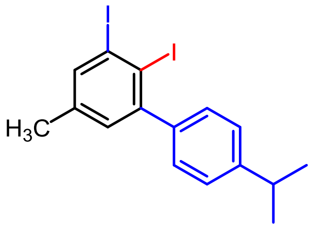
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**82%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.4 Synthesis of 3,4-Diiodo-5-phenyltoluene (8)



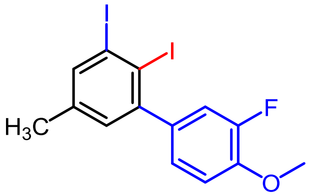
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as white solid (**80%** yields). The spectroscopic data for this compound are matched the previous report by F. Yang, K.-G. Ji, H.-T. Zhu, A. Shaukat, X.-Y. Liu, Y.-M. Liang, *Chem. - Eur. J.* **2011**, *17*, 4986-4990.

1.2.5 Synthesis of 2,3-diiodo-4'-isopropyl-5-methyl-1,1'-biphenyl (9)



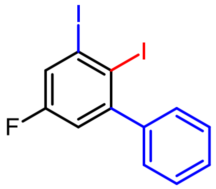
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**82%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.6 Synthesis of 3'-fluoro-2,3-diiodo-4'-methoxy-5-methyl-1,1'-biphenyl (10)



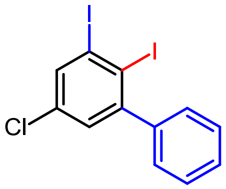
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as white solid (**91%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.7 Synthesis of 5-fluoro-2,3-diiodo-1,1'-biphenyl (11)



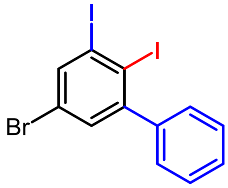
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**52%** yields). **IR** (cast film, cm^{-1}) 3012, 3008, 2975, 1607, 1589, 1358, 1272, 958, 745. **δ_H** (400MHz, *d*-$CDCl_3$) δ: 7.63 (dd, 1H, *J* = 2.8 Hz, *J* = 7.8 Hz), 7.35-7.45 (m, 3H), 7.20-7.24 (m, 2H), 6.99 (dd, 1H, *J* = 2.8 Hz, *J* = 8.6 Hz). **δ_C** (100 MHz, *d*-$CDCl_3$) δ: 163.1, 160.6, 150.7, 150.6, 145.9, 128.8, 128.4, 128.2, 125.9, 125.7, 116.6, 116.4, 109.9, 109.9, 106.9, 106.8. **HRMS** (ESI) m/z for $C_{12}H_8FI_2$ $[M+H]^+$: calcd. 424.8699; found, 424.8692.

1.2.8 Synthesis of 5-chloro-2,3-diiodo-1,1'-biphenyl (12)



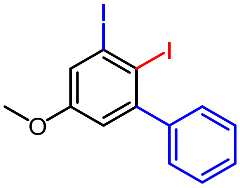
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**81%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.9 Synthesis of 5-bromo-2,3-diiodo-1,1'-biphenyl (13)



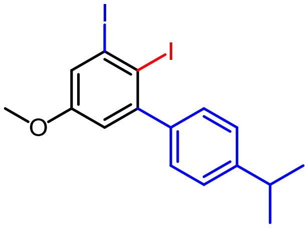
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**91%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.10 Synthesis of 2,3-diiodo-5-methoxy-1,1'-biphenyl (14)



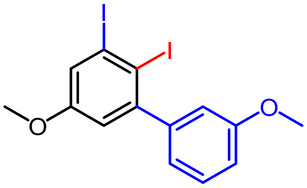
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**78%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem*. **2015**, 3374.

1.2.11 Synthesis of 2,3-diiodo-4'-isopropyl-1,1'-biphenyl (15)



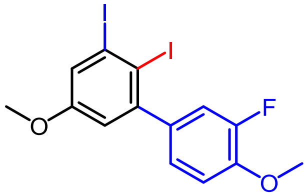
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**62%** yields). **IR** (cast film, cm^{-1}) 3042, 3016, 2975, 1598, 1587, 1281, 1113, 832. **δ$_H$** (400 MHz, *d*-CDCl$_3$) δ: 7.45 (d, 1H, *J* = 2.9 Hz) , 7.25 (d, 2H, *J* = 8.0 Hz), 7.17 (d, 2H, *J* = 8.2 Hz), 6.80 (d, 1H, *J* = 2.9 Hz), 2.98 (sept, 1H, *J* = 6.9 Hz), 1.30 (d, 6H, *J* = 6.9 Hz . **δ$_C$** (100 MHz, *d*-CDCl$_3$) δ: 159.6, 149.9, 148.7, 144.2, 128.8, 126.1, 124.5, 115.6, 110.1, 101.9, 55.8, 33.9, 24.1. **HRMS** (ESI) m/z for $C_{16}H_{17}I_2O$ $[M+H]^+$: calcd. Exact 478.9369; found, 478.9358.

1.2.12 Synthesis of 2,3-diiodo-3',5-dimethoxy-1,1'-biphenyl (16)



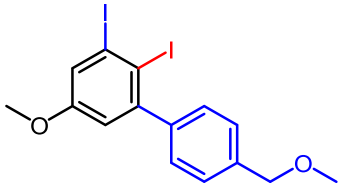
The title ompound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**52%** yields). **IR** (cast film, cm^{-1}) 3088, 3057, 2995, 1625, 1607, 1588, 1345, 1240, 1285, 1045, 768. **δ$_H$** (400 MHz, *d*-CDCl$_3$) δ: 7.46 (d, 1H, *J* = 2.9 Hz) , 7.31 (dd, 1H, *J* = 7.8 Hz, *J* = 8.0 Hz), 6.91 (dd, 1H, *J* = 2.4 Hz, *J* = 8.2 Hz), 6.75-6.88 (m, 3H), 3.84 (s, 3H), 3.78 (s, 3H). **δ$_C$** (100 MHz, *d*-CDCl$_3$) δ: 159.6, 159.1, 149.7, 147.9, 129.2, 124.6, 121.3, 115.4, 114.6, 113.6, 110.2, 101.4, 55.8, 55.5. **HRMS** (ESI) m/z for $C_{14}H_{13}I_2O_2$ $[M+H]^+$: calcd. 466.9005; found, 466.8994.

1.2.13 Synthesis of 3'-fluoro-2,3-diiodo-4',5-dimethoxy-1,1'-biphenyl (17)



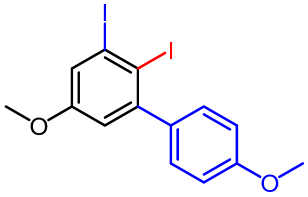
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as a white solid (**32%** yield). **IR** (cast film, cm^{-1}) 3079, 3045, 2994, 1621, 1612, 1582, 1317, 1243, 1120, 1086, 995, 817. **δ$_H$** (400 MHz, *d*-$CDCl_3$) δ:7.45 (d, 1H, *J* = 2.9 Hz), 6.90-7.10 (m, 3H), 6.77 (d, 1H, *J* = 2.9 Hz), 3.94 (s, 3H), 3.78 (s, 3H). **δ$_C$** (100 MHz, *d*-$CDCl_3$) δ: 159.6, 152.8, 150.4, 148.3, 147.6, 147.5, 139.6, 139.6, 124.9, 124.9, 124.6, 117.2, 116.9, 115.8, 112.8, 112.8, 110.2, 101.9, 56.4, 55.8. **M.p**: 114-116 °C. **HRMS** (ESI) m/z for $C_{14}H_{12}FI_2O_2$ $[M+H]^+$: calcd. 484.8911; found, 484.8901.

1.2.14 Synthesis of 2,3-diiodo-5-methoxy-4'-(methoxymethyl)-1,1'-biphenyl (18)



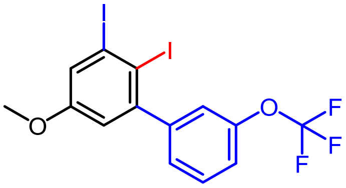
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as a white solid (**57%** yields). **IR** (cast film, cm^{-1}) 3011, 3009, 2987, 1608, 1542, 1448, 1266, 1210, 958, 752. **δ$_H$** (400 MHz, *d*-$CDCl_3$) δ: 7.45 (d, 1H, *J* = 2.9Hz), 7.38 (d, 2H, *J* = 8.0Hz), 7.23 (d, 2H, *J* = 8.1Hz), 6.78 (d, 1H, *J* = 2.9Hz), 4.52 (s, 2H), 3.77 (s, 3H), 3.44 (s, 3H). **δ$_C$** (100 MHz, *d*-$CDCl_3$) δ: 159.6, 149.6, 146.0, 138.1, 128.9, 127.4, 124.6, 115.6, 110.2, 101.6, 74.6, 58.5, 55.8. **M.p**: 71-75 °C. **HRMS** (ESI) m/z for $C_{15}H_{15}I_2O_2$ $[M+H]^+$: calcd. 480.9161; found, 480.9155.

1.2.15 Synthesis of 2,3-diiodo-4',5-dimethoxy-1,1'-biphenyl (19)



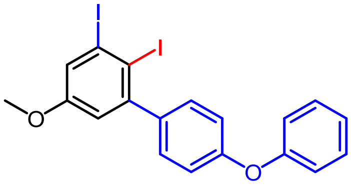
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as a white solid (**52%** yields). **IR** (cast film, cm^{-1}) 3014, 3004, 2985, 2893, 1607, 1594, 1542, 1278, 1240, 1146, 976, 714. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.42 (d, 1H, *J* = 2.9 Hz), 7.16 (d, 2H, *J* = 8.6 Hz), 7.91 (d, 2H, *J* = 8.7 Hz), 6.77 (d, 1H, *J* = 2.8 Hz), 3.83 (s, 3H), 3.74 (s, 3H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 159.5, 159.3, 149.5, 139.3, 130.1, 124.3, 115.7, 113.3, 110.0, 102.3, 55.7, 55.4. **M.p**: 106-108°C. **HRMS** (ESI) m/z for $C_{14}H_{13}I_2O_2$ $[M+H]^+$: calcd. Exact 466.9005; found, 466.8998.

1.2.16 Synthesis of 2,3-diiodo-5-methoxy-3'-(trifluoromethoxy)-1,1'-biphenyl (20)



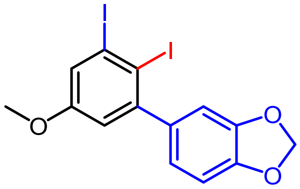
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**50%** yields). **IR** (cast film, cm^{-1}) 3077, 3012, 2998, 1622, 1601, 1473, 1314, 1245, 1276, 945, 844. δ_H (400 MHz, *d*-$CDCl_3$) δ: 7.48 (d, 1H, *J* = 2.9Hz),7.43 (dd, 1H, *J* = 8.0 Hz, *J* = 7.9 Hz), 7.23 (d, 1H, *J* = 8.2 Hz), 7.17 (d, 1H, *J* = 7.7 Hz), 7.13 (s, 1H), 6.79 (d, 1H, *J* = 2.9Hz), 3.79 (s, 3H). δ_C (100 MHz, *d*-$CDCl_3$) δ: 159.6, 148.8, 148.2, 148.1, 129.6, 127.4, 124.9, 1245, 121.9, 121.8, 120.5, 119.3, 116.8, 115.6, 110.4, 101.2, 55.8. **HRMS** (ESI) m/z for $C_{14}H_{10}F_3I_2O_2$ $[M+H]^+$: calcd. 520.8722; found, 520.8719.

1.2.17 Synthesis of 2,3-diiodo-5-methoxy-4'-phenoxy-1,1'-biphenyl (21)



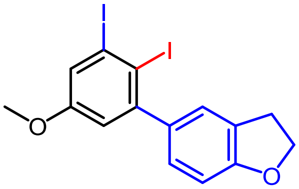
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as a white solid (**47%** yields). **IR** (cast film, cm^{-1}) 3124, 3055, 2996, 1644, 1646, 1541, 1347, 1247, 1223, 1104, 847. **δ_H** (400 MHz, *d*-$CDCl_3$) δ: 7.46 (d, 1H, *J* = 2.9 Hz), 7.38 (dd, 2H, *J* = 8.3 Hz, *J* = 7.5 Hz), 7.21 (d, 2H, *J* = 8.6 Hz), 7.14 (dd, 1H, *J* = 7.3 Hz , *J* = 7.4 Hz), 7.09 (d, 2H, *J* = 8.2 Hz), 7.02 (d, 2H, *J* = 8.6 Hz), 6.82 (d, 1H, *J* = 2.9 Hz), 3.79 (s, 3H). **δ_C** (100 MHz, *d*-$CDCl_3$) δ: 159.6, 157.3, 156.9, 149.3, 141.6, 130.4, 129.9, 124.5, 123.8, 119.5, 117.9, 115.7, 110.1, 102.1, 55.8. **M.p**: 94-96 °C. **HRMS** (ESI) m/z for $C_{19}H_{15}I_2O_2$ $[M+H]^+$: calcd. 528.9161; found, 528.9157.

1.2.18 Synthesis of 5-(2,3-diiodo-5-methoxyphenyl)benzo[d][1,3]dioxole (22)



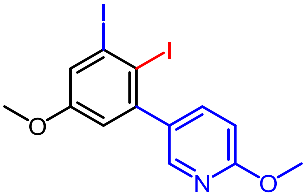
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as a white solid (**40%** yields). **IR** (cast film, cm^{-1}) 3012, 2978, 1604, 1547, 1533, 1301, 1244, 1239, 1147, 1007, 897, 748, 624. **δ_H** (400 MHz, *d*-$CDCl_3$) δ: 7.44 (d, 1H, *J* = 2.9 Hz), 6.83 (d, 1H, *J* = 8.0 Hz), 6.78 (d, 1H, *J* = 2.9 Hz), 6.72 (s, 1H), 6.68 (dd, 1H, *J* = 1.6 Hz, *J* = 7.9 Hz), 6.02 (s, 2H), 3.77 (s, 3H). **δ_C** (100 MHz, *d*-$CDCl_3$) δ: 159.6, 149.4, 147.4, 147.2, 140.8, 124.5, 122.5, 115.8, 110.0, 109.8, 107.9, 102.2, 101.4, 55.8. **M.p**: 123-125 °C. **HRMS** (ESI) m/z for $C_{14}H_{11}I_2O_3$ $[M+H]^+$: calcd. 480.8798; found, 480.8793.

1.2.19 Synthesis of 5-(2,3-diiodo-5-methoxyphenyl)-2,3-dihydrobenzofuran (23)



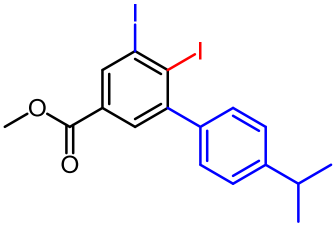
The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as a white solid (**30%** yields). **IR** (cast film, cm^{-1}) 3095, 3041, 3012, 2995, 1611, 1599, 1370, 1207, 1176, 1132, 892, 773. **δ_H** (400 MHz, *d*-$CDCl_3$) δ: 7.43 (d, 1H, *J* = 2.9Hz), 7.07 (s, 1H), 6.98 (d, 1H, *J* = 8.4Hz), 6.77-6.85 (m, 2H), 4.63 (t, 2H, *J* = 8.7Hz), 3.77 (s, 3H), 3.25 (t, 2H, *J* = 8.7Hz). **δ_C** (100 MHz, *d*-$CDCl_3$) δ: 159.9, 159.6, 149.9, 139.4, 128.9, 126.7, 125.6, 124.3, 115.7, 109.9, 108.7, 102.5, 71.6, 55.7, 29.8. **M.p**: 123-126 °C. **HRMS** (ESI) m/z for $C_{15}H_{13}I_2O_2$ $[M+H]^+$: calcd. 478.9005; found, 478.8995.

1.2.20 Synthesis of 5-(2,3-diiodo-5-methoxyphenyl)-2-methoxypyridine (24)



The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as colorless oil (**52%** yields). **IR** (cast film, cm^{-1}) 3112, 3075, 3012, 1625, 1593, 1432, 1328, 1195, 1109, 943, 814. **δ_H** (400 MHz, *d*-$CDCl_3$) δ: 8.05 (d, 1H, *J* = 2.2Hz), 7.49 (dd, 1H, *J* = 2.4 Hz, *J* = 8.5 Hz), 7.45 (d, 1H, *J* = 2.8Hz), 6.78 (m, 2H), 3.98 (s, 3H), 3.77 (s, 3H). **δ_C** (100 MHz, *d*-$CDCl_3$) δ: 163.7, 159.7, 146.4, 146.2, 139.5, 135.7, 124.8, 116.0, 110.3, 110.0, 102.4, 55.8, 53.7. **HRMS** (ESI) m/z for $C_{13}H_{12}I_2NO_2$ $[M+H]^+$: calcd. 467.8957; found, 467.8944.

1.2.21 Synthesis of methyl 5,6-diiodo-4'-isopropyl-[1,1'-biphenyl]-3-carboxylate (25)

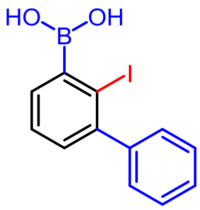


The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as white solid (**77%** yields). The spectroscopic data for this compound are matched our previous report by Al-Zoubi, R. M.; Al-Jammal, W. K.; El-Khateeb, M. Y.; McDonald, R. *Eur. J. Org. Chem.* **2015**, 3374.

1.3 General procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives and trimethylborate

To a solution of 2,3-diiodo-5-substituted biphenyl compound (1.0 mmol) in 20.0 mL anhyd. THF at -78 °C was added dropwize *iso*propyl magnesium chloride (2M in THF, 1.10 mmol, 1.10 equiv.). The mixture was stirred at that temperature for 2 h and then, trimethylborate (3.0 equiv.) was added. The solution was slowly warmed up 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). Organic layers were combined, filtered and then concentrated under reduced pressure using a rotary evaporator. A 20 mL diethylether was added and then extracted twice with 20 mL of aqueous NaOH (0.25M). Aqueous basic layers were combined; acidified using HCl solution (0.25M) and the resulting precipitate was then exctracted with 30 mL diethylether. Organic layers were combined, dried with anhyd. Sodium sulfate filtered and concentrated under reduced pressure using a rotary evaporator yield the pure desired product.

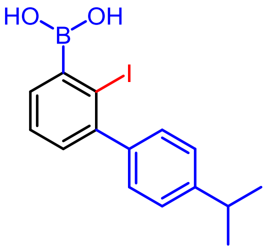
1.3.1 Synthesis of (2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (5_A)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**60%** yields). **IR** (cast film, cm^{-1}) 3421, 3095, 3025, 2955, 1601, 1598, 1106, 954, 782. **δ_H** (400MHz, d^6-

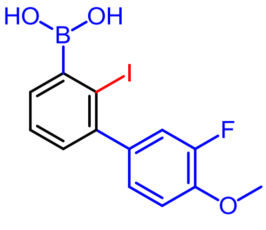
DMSO) δ: 8.31 (s, 2H), 7.37-7.46 (m, 4H), 7.24-7.28 (m, 2H), 7.21 (dd, 1H, *J* = 1.5 Hz, *J* = 7.4 Hz), 7.12 (dd, 1H, *J* = 1.5 Hz, *J* = 7.2 Hz). **δ_C** (100 MHz, d^6-DMSO) δ: 149.2, 145.5, 144.7, 131.4, 129.2, 129.1, 128.0, 127.5, 127.3, 102.6 . **δ_B** (128 MHz, *d*-acetone) δ: 29.2. **M.p**: 91-94 °C. **HRMS** (ESI) m/z for $C_{12}H_{11}BIO_2$ $[M+H]^+$: calcd. 324.9897; found, 324.9894.

1.3.2 Synthesis of (2-iodo-4'-isopropyl-[1,1'-biphenyl]-3-yl)boronic acid (6_A)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**54%** yields). **IR** (cast film, cm^{-1}) 3346, 3045, 2998, 1612, 1597, 1179, 1078, 967, 862. **δ_H** (400 MHz, d^6-DMSO) δ: 8.27 (s, 2H), 7.33 (dd, 1H, *J* = 7.4 Hz, *J* = 7.3 Hz) , 7.25 (d, 2H, *J* = 8.0 Hz), 7.12-7.15 (m, 3H), 7.05 (d, 1H, *J* = 7.0 Hz), 2.45 (sept, 1H, *J* = 6.4 Hz), 1.19 (d, 6H, *J* = 6.7 Hz) . **δ_C** (100 MHz, d^6-DMSO) δ: 149.5, 147.5, 145.5, 142.2, 131.2, 129.4, 129.1, 127.4, 125.9, 102.8, 33.2, 23.9. **δ_B** (128MHz, d^6-acetone) δ: 29.5. **M.p**: 114-116 °C. **HRMS** (ESI) m/z for $C_{15}H_{17}BIO_2$ $[M+H]^+$: calcd. Exact 367.0366; found, 367.0358.

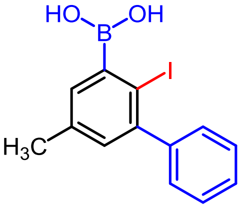
1.3.3 Synthesis of (3'-fluoro-2-iodo-4'-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (7_A)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**63%** yield). **IR** (cast film, cm^{-1}) 3396, 3097, 3017, 2986, 2934, 1619, 1597, 1547, 1352, 1247, 1101, 937, 782. **δ_H** (400 MHz, d^6-DMSO) δ: 8.30 (s, 2H), 7.38 (t, 1H, *J* = 7.4 Hz), 7.20-7.26 (m, 2H), 7.13 (dd, 1H, *J* = 1.6 Hz, *J* = 7.2 Hz), 7.08 (dd, 1H, *J* = 2.0 Hz, *J* =

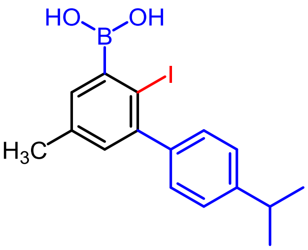
12.2 Hz), 7.01(d, 1H, *J* = 8.3 Hz), 3.39 (s, 3H). **δ_C** (100 MHz, d^6- DMSO) δ: 151.9, 149.5, 146.5, 146.5, 144.1, 137.5, 137.5, 131.5, 129.3, 127.4, 125.6, 125.6, 116.9, 116.7, 113.4, 113.4, 102.7, 56.1. **δ_B** (128 MHz, d^6-acetone) δ: 29.7. **M.p**: 119-121 °C. **HRMS** (ESI) m/z for $C_{13}H_{12}BFIO_3$ $[M+H]^+$: calcd. 372.9908; found, 372.9897.

1.3.4 Synthesis of (2-iodo-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (8_A)



The title compound was prepared using the general procedure for regioselective Suzuki-Miyaura cross-coupling reaction and isolated as a white solid (**47%** yield). **IR** (cast film, cm^{-1}) 3304, 3086, 3055, 2986, 2942, 1614, 1597, 1157, 1024, 935, 824. **δ_H** (400 MHz, d^6-DMSO) δ: 8.12 (s, 2H), 7.06 (s, 1H), 7.33-7.46 (m, 5H), 7.13 (s, 1H), 2.29 (s, 3H). **δ_C** (100 MHz, d^6-DMSO) δ: 145.8, 142.4, 139.1, 138.5, 128.7, 128.3, 128.2, 127.1, 99.9, 20.2. **δ_B** (128 MHz, d^6-acetone) δ: 29.3. **M.p**: 126-128 °C. **HRMS** (ESI) m/z for $C_{13}H_{13}BIO_2$ $[M+H]^+$: calcd. 339.0053; found, 339.0046.

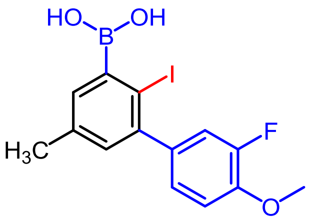
1.3.5 Synthesis of (2-iodo-4'-isopropyl-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (9_A)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**40%** yields). **IR** (cast film, cm^{-1}) 3318, 3104, 3095, 2984, 2812, 1601, 1575, 886, 776. **δ_H** (400 MHz, d^6-DMSO) δ: 8.24 (s, 2H), 7.29 (d, 2H, *J* = 8.0 Hz), 7.16 (d, 2H, *J* = 7.8 Hz), 7.02 (s, 1H), 6.93 (s, 1H), 2.62 (sept, 1H, *J* = 6.8 Hz), 2.25 (s, 3H), 1.23 (d, 6H, *J* = 6.8 Hz). **δ_C** (100 MHz, d^6-DMSO) δ: 147.4, 145.3, 142.2, 136.6, 132.1, 130.3, 129.1, 125.9, 98.8, 33.2, 23.9, 20.3. **δ_B** (128 MHz, d^6-acetone) δ: 29.5.

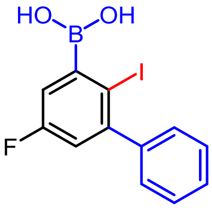
M.p: 117-119 °C. **HRMS** (ESI) m/z for $C_{16}H_{19}BIO_2$ $[M+H]^+$: calcd. 381.0523; found, 381.0520.

1.3.6 Synthesis of (3'-fluoro-2-iodo-4'-methoxy-5-methyl-[1,1'-biphenyl]-3-yl) boronic acid (10_A)



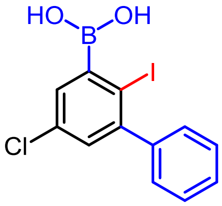
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**60%** yields). **IR** (cast film, cm^{-1}) 3406, 3120, 3089, 3009, 2979, 1596, 1442, 1243, 1124, 1106, 912, 795. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.25 (s, 2H), 7.21 (dd, 1H, *J* = 8.8 Hz , *J* = 8.7 Hz), 7.00-7.09 (m, 3H), 6.9 (d, 1H, *J* = 1.7 Hz), 3.88 (s, 3H), 2.25 (s, 3H). **δ_C** (100 MHz, *d*^{6}-DMSO) δ:151.9, 149.4, 149.1, 146.5, 146.4, 143.9, 137.5, 137.5, 136.9, 132.4, 130.2, 125.6, 125.6, 116.8, 116.7, 113.4, 98.9, 56.1, 20.3. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 29.3. **M.p**: 132-134 °C. **HRMS** (ESI) m/z for $C_{14}H_{14}BFIO_3$ $[M+H]^+$: calcd. 387.0065; found, 387.0059.

1.3.7 Synthesis of (5-fluoro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (11_A)



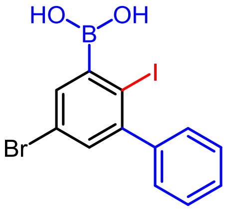
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**38%** yields). **IR** (cast film, cm^{-1}) 3298, 3084, 3068, 2994, 1631, 1599, 1425, 1348, 1275, 1186, 1015, 784. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.44 (s, 2H), 7.35-7.50 (m, 3H) , 7.27 (d, 2H, *J* = 6.9 Hz), 7.11 (dd, 1H, *J* = 2.9 Hz, *J* = 9.4 Hz), 7.01 (dd, 1H, *J* = 2.9 Hz, *J* = 8.6 Hz) . **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 162.9, 160.5, 147.8, 147.7, 143.6, 129.0, 128.3, 128.2, 128.1, 127.9, 118.0, 117.8, 116.2, 116.0, 96.2, 96.1. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 29.4. **M.p**: 135-137 °C. **HRMS** (ESI) m/z for $C_{12}H_{10}BFIO_2$ $[M+H]^+$: calcd. 342.9803; found, 342.9799.

1.3.8 Synthesis of (5-chloro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (12_A)



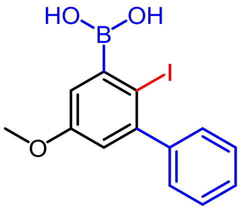
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**53%** yields). **IR** (cast film, cm^{-1}) 3314, 3109, 3058, 3017, 1621, 1597, 1414, 1283, 1138, 1017, 973, 712. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.50 (s, 2H), 7.40-7.48 (m, 4H), 7.25-7.29 (m, 2H), 7. 29 (d, 1H, *J* = 2.3 Hz), 7. 17 (d, 1H, *J* = 7.8 Hz). **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 151.4, 147.6, 143.4, 132.7, 130.7, 128.9, 128.4, 128.1, 127.9, 100.5. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 28.9. **M.p**: 138-140 °C. **HRMS** (ESI) m/z for $C_{12}H_{10}BClIO_2$ $[M+H]^+$: calcd. 358.9507; found, 358.9505.

1.3.9 Synthesis of (5-bromo-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (13_A)



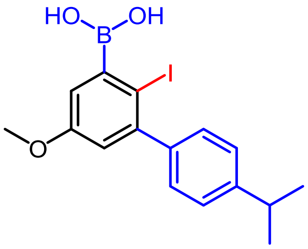
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**65%** yields). **IR** (cast film, cm^{-1}) 3274, 3108, 3061, 2941, 1612, 1599, 1296, 1101, 862, 697. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.47 (s, 2H), 7.40-7.48 (m, 3H), 7. 36 (d, 1H, *J* = 2.3 Hz), 7. 29 (d, 1H, *J* = 2.3 Hz), 7. 26 (d, 2H, *J* = 6.7 Hz). **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 152.1, 147.9, 143.3, 133.5, 131.2, 128.9, 128.2, 127.9, 121.4, 101.3. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 29.3. **M.p**: 140-144 °C. **HRMS** (ESI) m/z for $C_{12}H_{10}BBrIO_2$ $[M+H]^+$: calcd. 402.9002; found, 402.8997.

1.3.10 Synthesis of (2-iodo-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (14_A)



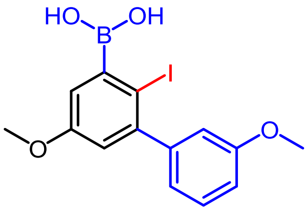
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**83%** yields). **IR** (cast film, cm^{-1}) 3288, 3087, 3025, 2984, 1601, 1587, 1289, 1148, 1012, 937, 812. **δ$_H$** (400 MHz, *d*^{6}-DMSO) δ: 8.32 (s, 2H), 7.38-7.45 (m, 3H), 7.27 (d, 2H, *J* = 6.8 Hz), 6.78 (d, 1H, *J* = 2.9 Hz), 6.75 (d, 1H, *J* = 2.9 Hz), 3.75 (s, 3H). **δ$_C$** (100 MHz, *d*^{6}-DMSO) δ: 158.6, 150.1, 146.7, 144.6, 129.1, 128.0, 127.6, 117.3, 115.2, 91.2, 55.3. **δ$_B$** (128 MHz, *d*^{6}-acetone) δ: 29.4. **M.p**: 119-121 °C. **HRMS** (ESI) m/z for $C_{13}H_{13}BIO_3$ $[M+H]^+$: calcd. 355.0002; found, 354.9997.

1.3.11 Synthesis of (2-iodo-4'-isopropyl-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (15_A)



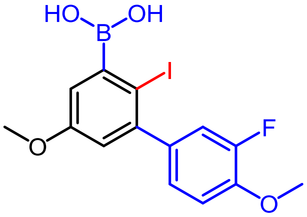
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**86%** yields). **IR** (cast film, cm^{-1}) 3369, 3107, 3081, 2932, 1621, 1598, 1272, 1133, 1098, 986, 862. **δ$_H$** (400 MHz, *d*^{6}-DMSO) δ: 8.30 (s, 2H), 7.30 (d, 2H, *J* = 8.0 Hz), 7.19 (d, 2H, *J* = 8.0 Hz), 6.77 (d, 1H, *J* = 2.9 Hz), 6.71 (d, 1H, *J* = 2.9 Hz), 3.75 (s, 3H), 2.95 (sep, 1H, *J* = 6.9Hz), 1.24 (d, 6H, *J* = 6.9Hz). **δ$_C$** (100 MHz, *d*^{6}-DMSO) δ:158.6, 150.0, 147.5, 146.6, 141.9, 128.9, 125.9, 117.2, 115.2, 91.3, 55.3, 33.1, 23.8. **δ$_B$** (128 MHz, *d*^{6}-acetone) δ: 28.1. **M.p**: 91-93 °C. **HRMS** (ESI) m/z for $C_{16}H_{19}BIO_3$ $[M+H]^+$: calcd. 397.0472; found, 397.0464.

1.3.12 Synthesis of (2-iodo-3',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (16_A)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**63%** yields). **IR** (cast film, cm^{-1}) 3312, 3087, 3014, 2998, 2888, 1642, 1609, 1299, 1283, 1114, 1107, 983. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.30 (s, 2H), 7.34 (dd, 1H, *J* = 7.9 Hz, *J* = 7.8 Hz), 6.95 (dd, 1H, *J* = 2.1 Hz, *J* = 8.3 Hz), 6.81 (d, 1H, *J* = 7.6 Hz), 6.73-6.78 (m, 2H), 6.73 (d, 1H, *J* = 2.9 Hz), 3.78 (s, 3H), 3.75 (s, 3H). **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 158.7, 158.6, 146.5, 145.8, 129.2, 121.5, 117.3, 115.2, 114.9, 112.9, 91.1, 55.3, 55.1. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 29.2. **M.p**: 129-131 °C. **HRMS** (ESI) m/z for $C_{14}H_{15}BIO_4$ $[M+H]^+$: calcd. 385.0108; found, 385.0105.

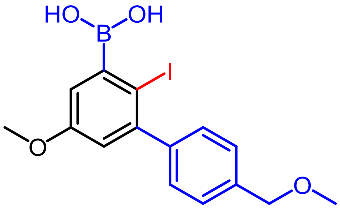
1.3.13 Synthesis of (3'-fluoro-2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl) boronic acid (17_A)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**77%** yields). **IR** (cast film, cm^{-1}) 3297, 3086, 3078, 3027, 2996, 1617, 1589, 1588, 1418, 1207, 1167, 1117, 862. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.31 (s, 2H), 7.23 (dd, 1H, *J* = 8.6 Hz, *J* = 8.7 Hz), 7.10 (dd, 1H, *J* = 1.7 Hz, *J* = 12.2 Hz), 7.04 (d, *J* = 8.4 Hz, 2H), 6.80 (d, 1H, *J* = 2.9 Hz), 6.75 (d, 1H, *J* = 2.9 Hz), 3.89 (s, 3H), 3.76 (s, 3H) . **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 158.6, 151.8, 150.1, 149.4, 146.6, 146.5, 145.2, 137.3, 137.2, 125.6, 125.6, 117.4, 116.8, 116.7, 115.3, 113.3, 91.4, 56.0, 55.3. **δ_B** (128 MHz, *d*^{6}-

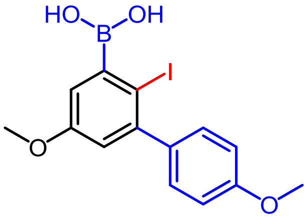
acetone) δ: 29.2. **M.p**: 134-136 °C. **HRMS** (ESI) m/z for $C_{14}H_{14}BFIO_4$ $[M+H]^+$: calcd. 403.0014; found, 403.0005.

1.3.14 Synthesis of (2-iodo-5-methoxy-4'-(methoxymethyl)-[1,1'-biphenyl]-3-yl) boronic acid (18_A)



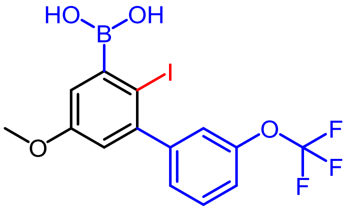
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**80%** yields). **IR** (cast film, cm^{-1}) 3213, 3099, 3085, 2988, 1621, 1608, 1598, 1249, 1120, 1108, 1096, 877, 648. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.31 (s, 2H), 7.37 (d, 2H, *J* = 7.8 Hz), 7.24 (d, 2H, *J* = 7.8 Hz), 6.78 (d, 1H, *J* = 2.8 Hz), 6.74 (d, 1H, *J* = 2.7 Hz), 4.46 (s, 2H), 3.75 (s, 3H), 3.32 (s, 3H). **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 158.6, 150.1, 146.5, 143.6, 137.6, 128.9, 127.1, 117.3, 115.2, 91.2, 73.4, 57.7, 55.3. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 30.0. **M.p**: 142-144 °C. **HRMS** (ESI) m/z for $C_{15}H_{17}BIO_4$ $[M+H]^+$: calcd. 399.0265; found, 399.0258.

1.3.15 Synthesis of (2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (19_A)



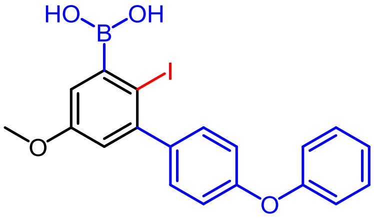
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**85%** yields). **IR** (cast film, cm^{-1}) 3295, 3076, 3028, 2981, 1623, 1601, 1573, 1253, 1208, 1147, 1129, 837, 728. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.31 (s, 2H), 7.18 (d, 2H, *J* = 8.2 Hz), 6.98 (d, 2H, *J* = 8.3 Hz), 6.75 (d, 1H, *J* = 2.0 Hz), 6.70 (d, 1H, *J* = 2.0 Hz), 3.79 (s, 3H), 3.74 (s, 3H) . **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 158.7, 158.6, 150.2, 146.4, 136.9, 130.3, 117.0, 115.4, 113.4, 91.9, 55.3, 55.2. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 29.4. **M.p**: 124-126 °C. **HRMS** (ESI) m/z for $C_{14}H_{15}BIO_4$ $[M+H]^+$: calcd. 385.0108; found, 385.0101.

1.3.16 Synthesis of (2-iodo-5-methoxy-3'-(trifluoromethoxy)-[1,1'-biphenyl]-3-yl)boronic acid (20$_A$)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**67%** yields). **IR** (cast film, cm^{-1}) 3312, 3097, 3058, 2942, 2828, 1589, 1546, 1427, 1343, 1242, 1168, 1127, 852, 742. **δ_H** (400 MHz, *d*^{6}-DMSO) δ: 8.32 (s, 2H), 7.59 (dd, 1H, *J* = 8.0 Hz , *J* = 7.9 Hz), 7.40 (d, 1H, *J* = 7.6 Hz), 7.30 (d, 1H, *J* = 7.6 Hz), 7.20 (s, 1H), 6.84 (d, 1H, *J* = 2.9 Hz), 6.77 (d, 1H, *J* = 2.9 Hz), 3.77 (s, 3H). **δ_C** (100 MHz, *d*^{6}-DMSO) δ: 158.6, 150.2, 147.8, 146.5, 144.9, 130.2, 128.3, 123.9, 121.7, 121.4, 120.1, 118.9, 117.9, 116.3, 115.2, 90.7, 55.4. **δ_B** (128 MHz, *d*^{6}-acetone) δ: 29.6. **M.p**: 126-128 °C. **HRMS** (ESI) m/z for $C_{14}H_{12}BF_3IO_4$ $[M+H]^+$: calcd. 438.9825; found, 438.9814.

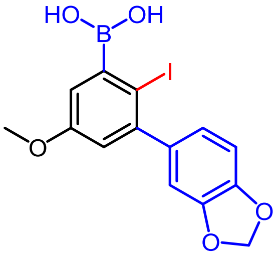
1.3.17 Synthesis of (2-iodo-5-methoxy-4'-phenoxy-[1,1'-biphenyl]-3-yl)boronic acid (21$_A$)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**59%** yields). **IR** (cast film, cm^{-1}) 3348, 3124, 3109, 3058, 2956, 1597, 1586, 1579, 1282, 1214, 1175, 1127, 812. **δ_H** (400 MHz, *d*^{6}-DMSO) δ:

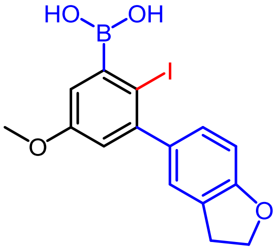
8.36 (s, 2H), 7.43 (dd, 2H, *J* = 8.0 Hz, *J* = 7.8 Hz), 7.26 (d, 2H, *J* = 8.5 Hz), 7.18 (dd, 1H, *J* = 7.4 Hz, *J* = 7.3 Hz), 7.07 (d, 2H, *J* = 7.8 Hz), 7.03 (d, 2H, *J* = 8.5 Hz), 6.80 (d, 1H, *J* = 3.0 Hz), 6.72 (d, 1H, *J* = 3.0 Hz), 3.75 (s, 3H). $\boldsymbol{\delta_C}$ (100 MHz, d^6-DMSO) δ: 158.6, 156.4, 156.3, 146.1, 139.6, 160.9. 130.2, 123.9, 119.1, 117.7, 117.4, 115.3, 91.6, 55.4. $\boldsymbol{\delta_B}$ (128 MHz, d^6-acetone) δ: 29.4. **M.p**: 129-131 °C. **HRMS** (ESI) m/z for $C_{19}H_{17}BIO_4$ $[M+H]^+$: calcd. 447.0265; found, 447.0262.

1.3.18 Synthesis of (3-(benzo[d][1,3]dioxol-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (22$_A$)



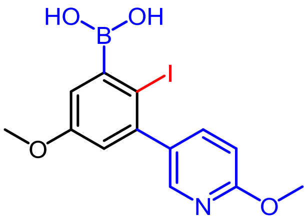
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**66%** yields). **IR** (cast film, cm^{-1}) 3298, 3108, 3068, 3028, 2994, 1619, 1578, 1348, 1286, 1274. 1208, 1142, 1115, 921, 689. $\boldsymbol{\delta_H}$ (400 MHz, d^6-DMSO) δ: 8.23 (s, 2H), 6.96 (d, 1H, *J* = 7.9 Hz), 6.69-6.78 (m, 3H), 6.53 (s, 1H), 6.06 (s, 2H), 3.75 (s, 3H). $\boldsymbol{\delta_C}$ (100 MHz, d^6-DMSO) δ: 158.5, 150.0, 146.7, 146.6, 146.3, 138.5, 122.7, 117.2, 115.3, 109.7, 107.9, 101.1, 91.7, 55.3. $\boldsymbol{\delta_B}$ (128 MHz, d^6-acetone) δ: 29.3. **M.p**: 108-110 °C. **HRMS** (ESI) m/z for $C_{14}H_{13}BIO_5$ $[M+H]^+$: calcd. 398.9901; found, 398.9893.

1.3.19 Synthesis of (3-(2,3-dihydrobenzofuran-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (23$_A$)



The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**53%** yields). **IR** (cast film, cm^{-1}) 3368, 3108, 3088, 3042, 2995, 1632, 1614, 1586, 1312, 1277, 1148, 986, 834. **δ$_H$** (400 MHz, *d*^{6}-DMSO) δ: 8.27 (s, 2H), 7.11 (s, 1H), 6.97 (dd, 1H, *J* = 1.6 Hz, *J* = 8.2 Hz), 6.80 (d, 1H, *J* = 8.2 Hz), 6.76 (d, 1H, *J* = 3.1 Hz), 6.71 (d, 1H, *J* = 3.0 Hz), 4.56 (t, 2H, *J* = 8.7 Hz), 3.75 (s, 3H), 4.56 (t, 2H, *J* = 8.6 Hz). **δ$_C$** (100 MHz, *d*^{6}-DMSO) δ: 159.1, 158.6, 150.1, 146.8, 136.9, 128.8, 126.9, 125.8, 116.9, 115.3, 108.2, 92.1, 71.1, 55.2, 29.1. **δ$_B$** (128 MHz, *d*^{6}-acetone) δ: 29.8. **M.p**: 137-139 °C. **HRMS** (ESI) m/z for $C_{15}H_{15}BIO_4$ $[M+H]^+$: calcd. 397.0108; found, 397.0104.

1.3.20 Synthesis of (2-iodo-5-methoxy-3-(6-methoxypyridin-3-yl)phenyl) boronic acid (24$_A$)



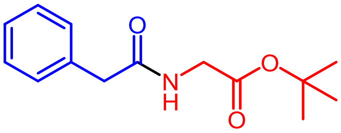
The title compound was prepared using the general procedure for MIE reaction of 2,3-diiodo-5-substituted biphenyl derivatives with borate and isolated as a white solid (**65%** yields). **IR** (cast film, cm^{-1}) 3307, 3109, 3086, 3062, 2997, 1614, 1598, 1388, 1286, 1214, 1178, 1137, 899. **δ$_H$** (400 MHz, *d*^{6}-DMSO) δ: 8.33 (s, 2H), 8.08 (d, 1H, *J* = 1.8 Hz), 7.61 (dd, 1H, *J* = 2Hz, *J* = 8.4 Hz), 6.89 (d, 1H, *J* = 8.5 Hz), 6.89 (d, 1H, *J* = 2.8 Hz), 6.76 (d, 1H, *J* = 2.8 Hz) 3.90 (s, 3H), 3.77 (s, 3H). **δ$_C$** (100 MHz, *d*^{6}-DMSO) δ: 162.8, 158.7, 150.1, 146.5, 143.1, 140.1, 133.8, 117.6, 115.6, 109.6, 92.1, 55.4, 53.3. **δ$_B$** (60 MHz, *d*^{6}-acetone) δ: 29.3. **M.p**: 139-141 °C. **HRMS** (ESI) m/z for $C_{13}H_{14}BINO_4$ $[M+H]^+$: calcd. 386.0061; found, 386.0058.

1.4 General Procedure for Organocatalytic Amidations

Into a 25 mL round bottom flask equipped with a stir bar were added carboxylic acid (0.55 mmol, 0.1M, 1.1 equiv), *ortho*-iodobiphenylboronic acid derivative (0.05 mmol, 10 mol%) and 1 g of activated 4Å molecular sieves (preactivation overnight in an oven at

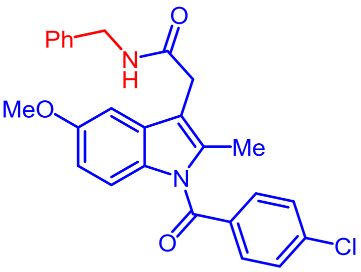
~250 °C or by Kugelrohr for 2h at 250 °C under high vacuum). Dichloromethane (7 mL) was added and the mixture was stirred for 10 min. Then, amine (0.5 mmol, 1 equiv) was added (in order to get reproducible results, it was necessary to use a gastight 100 µL syringe). The resulting mixture was stirred for 6 h at room temperature (24-25 °C). The reaction mixture was filtered through a pad of Celite® 545, the filtrate was washed with aqueous hydrochloric solution (pH = 4), aqueous sodium hydroxide solution (pH = 10-11) and brine, respectively. The organic layer was collected, dried over anhydrous Na_2SO_4, filtered and evaporated to yield the title amide product.

1.4.1 Synthesis of tert-butyl (2-phenylacetyl)glycinate (26)



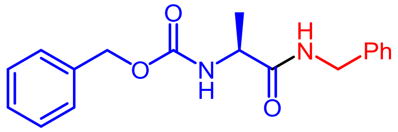
The title compound was prepared using the general procedure for organocatalytic amidation reaction and isolated as pale yellow oil (**74%** yields). **IR** (cast film, cm^{-1}) 3285, 3024, 3012, 1764, 1652, 1614, 1520, 864, 662. $\boldsymbol{\delta_H}$ (400 MHz, d^6-DMSO) δ: 7.26-7.35 (m, 5H), 6.16 (bs, 1H), 3.86 (d, 2H, *J* = 5.0 Hz), 3.58 (s, 2H), 1.42 (s, 9H). $\boldsymbol{\delta_C}$ (100 MHz, d^6-DMSO) δ: 171.1, 168.8, 134.7, 129.5, 128.9, 127.3, 82.2, 43.4, 42.2, 28.0. **HRMS** (ESI) m/z for $C_{14}H_{19}NO_3Na$ $[M+Na]^+$: calcd. 272.1263; found, 272.1269.

1.4.2 Synthesis of N-benzyl-2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetamide (27)



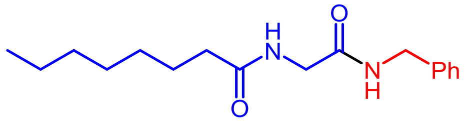
The title compound was prepared using the general procedure for organocatalytic amidation reaction and isolated as a white solid (**94%** yields). The spectroscopic data for this compound are matched the previous report by R. M. Al-Zoubi, O. Marion, D. G. Hall, *Angew. Chem. Int. Ed*. **2008**, *47*, 2876-2879.

1.4.3 Synthesis of benzyl (S)-(1-(benzylamino)-1-oxopropan-2-yl)carbamate (28)



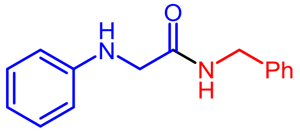
The title compound was prepared using the general procedure for organocatalytic amidation reaction and isolated as off-white solid (**95%** yields). **IR** (cast film, cm^{-1}) 3304, 3295, 3095, 3021, 2997, 1712, 1648, 1623, 1594, 824, 725. **δ_H** (400 MHz, *d*-$CDCl_3$) δ: 7.20-7.50 (m, 10H), 6.71 (d, 1H, *J* = 5.4 Hz), 5.00 (s, 2H), 5.74 (s, 1H), 3.94 (s, 2H), 3.72 (t, 1H, *J* = 6.6 Hz), 1.20 (d, 3H, *J* = 6.6 Hz). **δ_C** (100 MHz, *d*-$CDCl_3$) δ: 175.2, 155.2, 137.4, 136.7, 128.5, 128.4, 127.8, 127.8, 127.7, 65.1, 54.9, 51.1, 42.8, 18.9. **M.p**: 129-131 °C. **HRMS** (ESI) m/z for $C_{18}H_{20}N_2O_3Na$ $[M+Na]^+$: calcd. 335.1372; found, 335.1383.

1.4.4 Synthesis of N-(2-(benzylamino)-2-oxoethyl)octanamide (29)



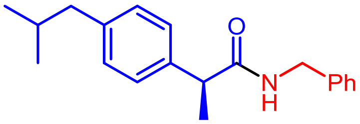
The title compound was prepared using the general procedure for organocatalytic amidation reaction and isolated as colorless oil (**40%** yields). **IR** (cast film, cm^{-1}) 3314, 3294, 3015, 3007, 2997, 1654, 1642, 1610, 1607, 1594, 945, 687. **δ_H** (400 MHz, *d*-$CDCl_3$) δ: 7.23-7.32 (m, 5H), 7.07 (s, 1H), 6.61 (s, 1H), 4.40 (d, 2H, *J* = 5.7 Hz), 3.94 (d, 2H, *J* = 5.1 Hz), 2.18 (t, 2H, *J* = 7.5 Hz), 1.56 (s, 2H), 1.10-1030 (m, 8H), 0.87 (t, 3H, *J* = 6.3 Hz). **δ_C** (100 MHz, *d*-$CDCl_3$) δ: 174.1, 169.2, 138.0, 128.8, 127.8, 127.6, 43.7, 43.5, 36.5, 31.8, 29.4, 29.1, 25.7, 22.7, 14.2, . **HRMS** (ESI) m/z for $C_{17}H_{26}N_2NaO_2$ $[M+Na]^+$: calcd. 313.1892; found, 313.1907.

1.4.5 Synthesis of N-benzyl-2-(phenylamino)acetamide (30)



The title compound was prepared using the general procedure for organocatalytic amidation reaction and isolated as a white solid (**50%** yields). The spectroscopic data for this compound are matched the previous report by A. J. A. Watson, R. J. Wakeham, A. C. Maxwell and J. M. J. Williams, *Tetrahedron* **2014**, 70, 3683-3690.

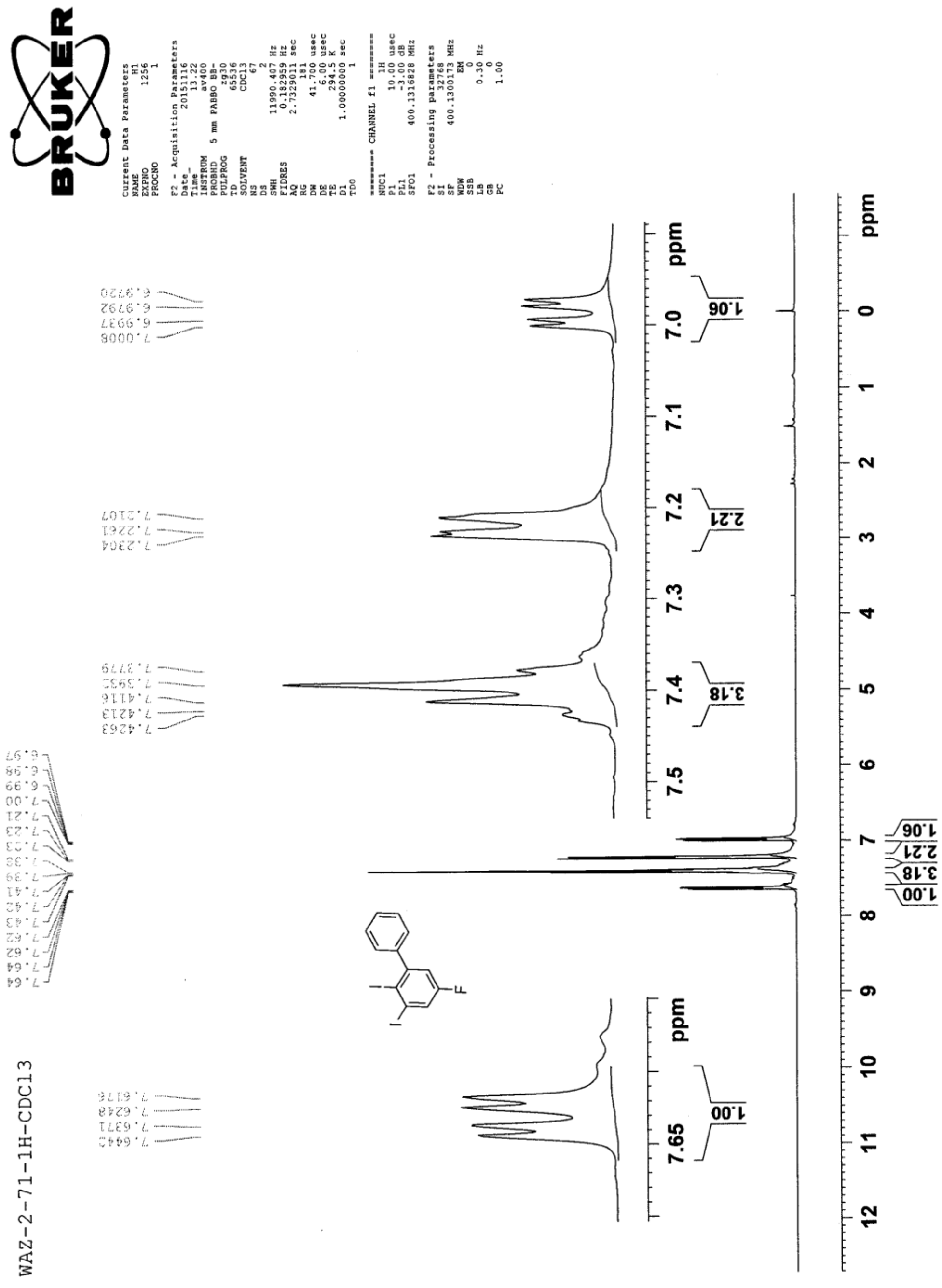
1.4.6 Synthesis of (S)-N-benzyl-2-(4-isobutylphenyl)propanamide (31)



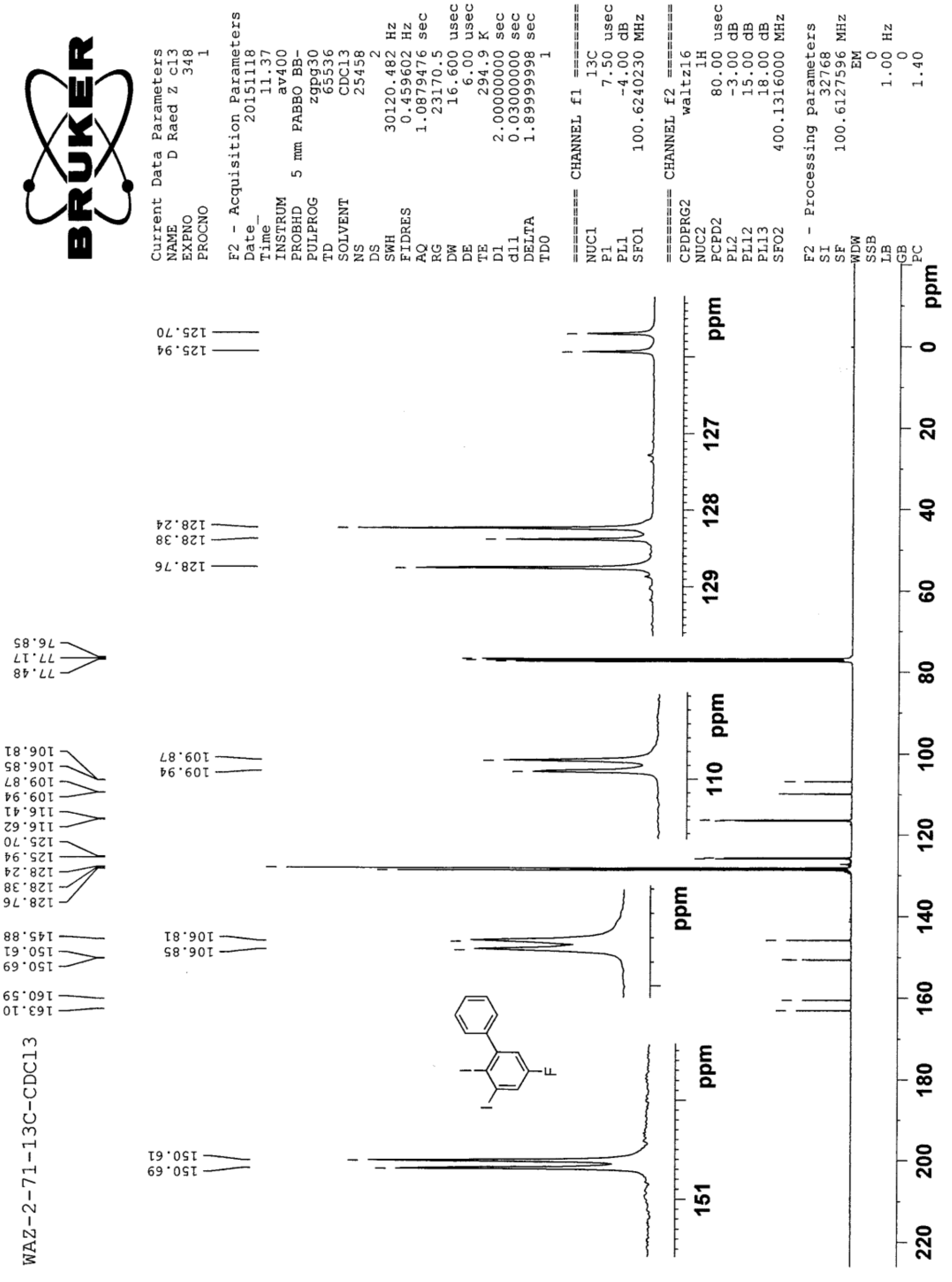
The title compound was prepared using the general procedure for organocatalytic amidation reaction and isolated as a white solid (**71%** yields). The spectroscopic data for this compound are matched the previous report by Sudrik, Surendra G.; Chavan, Sambhaji P.; Chandrakumar, K. R. S.; Pal, Sourav; Date, Sadgopal K.; Chavan, Subhash P.; Sonawane, Harikisan R. *J. Org. Chem.* **2002**, *67*, 1574-1579.

1.5 NMR Spectra for New Compound.

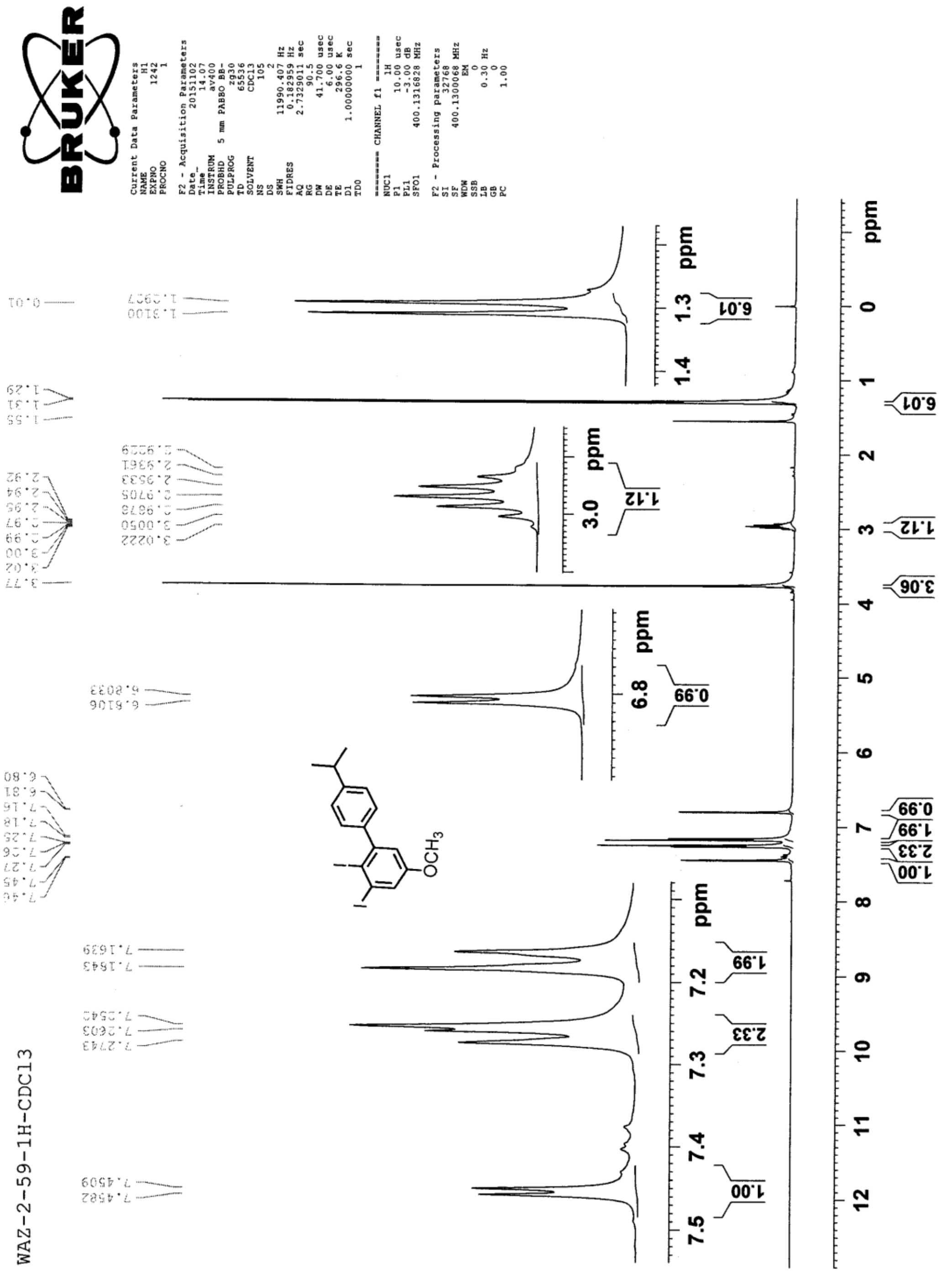
1.5.1 ^{1}H-NMR of 5-fluoro-2,3-diiodo-1,1'-biphenyl (11) in $CDCl_3$ at 25 °C.



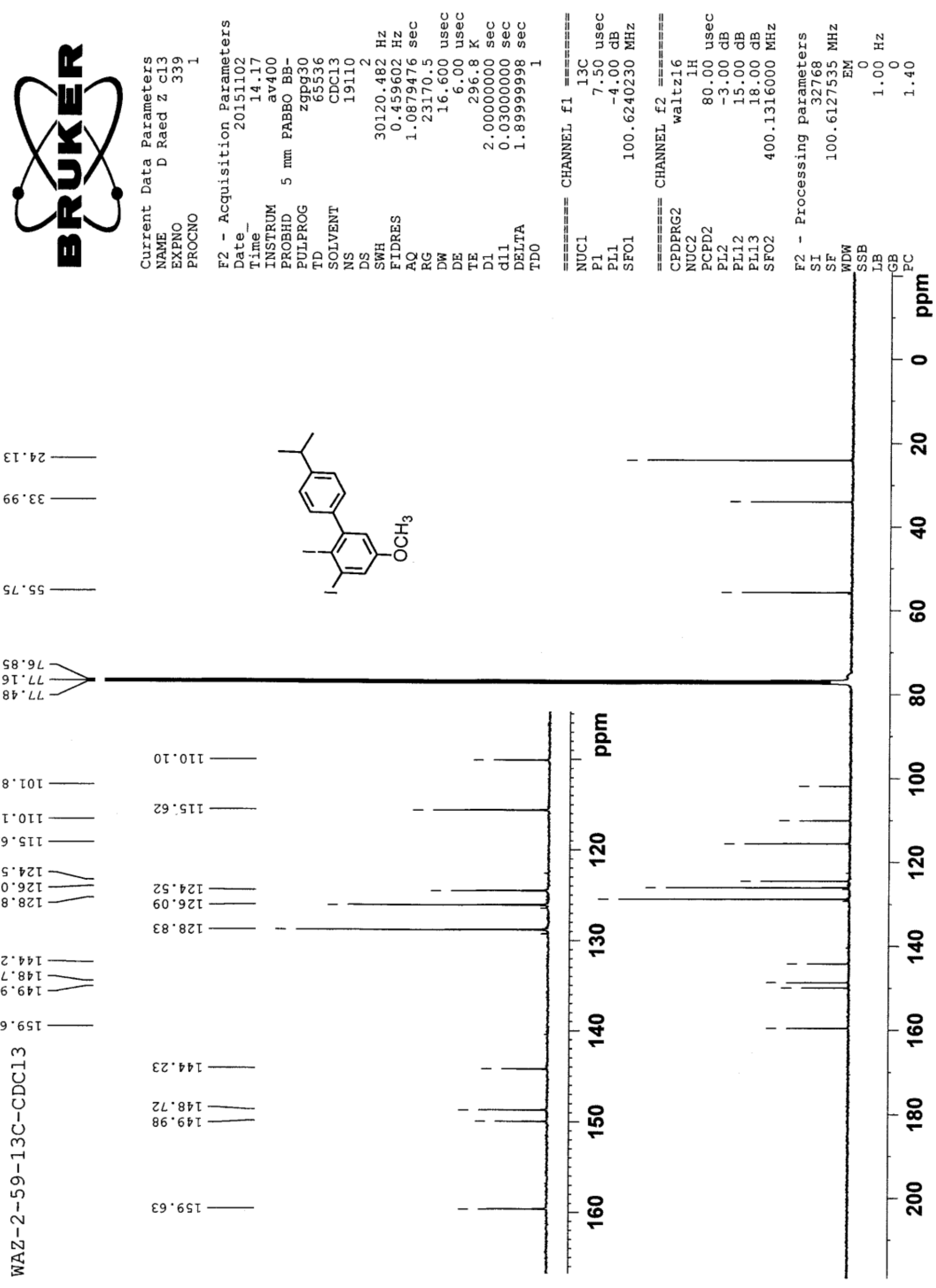
1.5.2 ^{13}C-NMR of 5-fluoro-2,3-diiodo-1,1'-biphenyl (11) in CDCl$_3$ at 25 ^{o}C.



1.5.3 ^{1}H-NMR of 2,3-diiodo-4'-isopropyl-1,1'-biphenyl (15) in $CDCl_3$ at 25 °C.

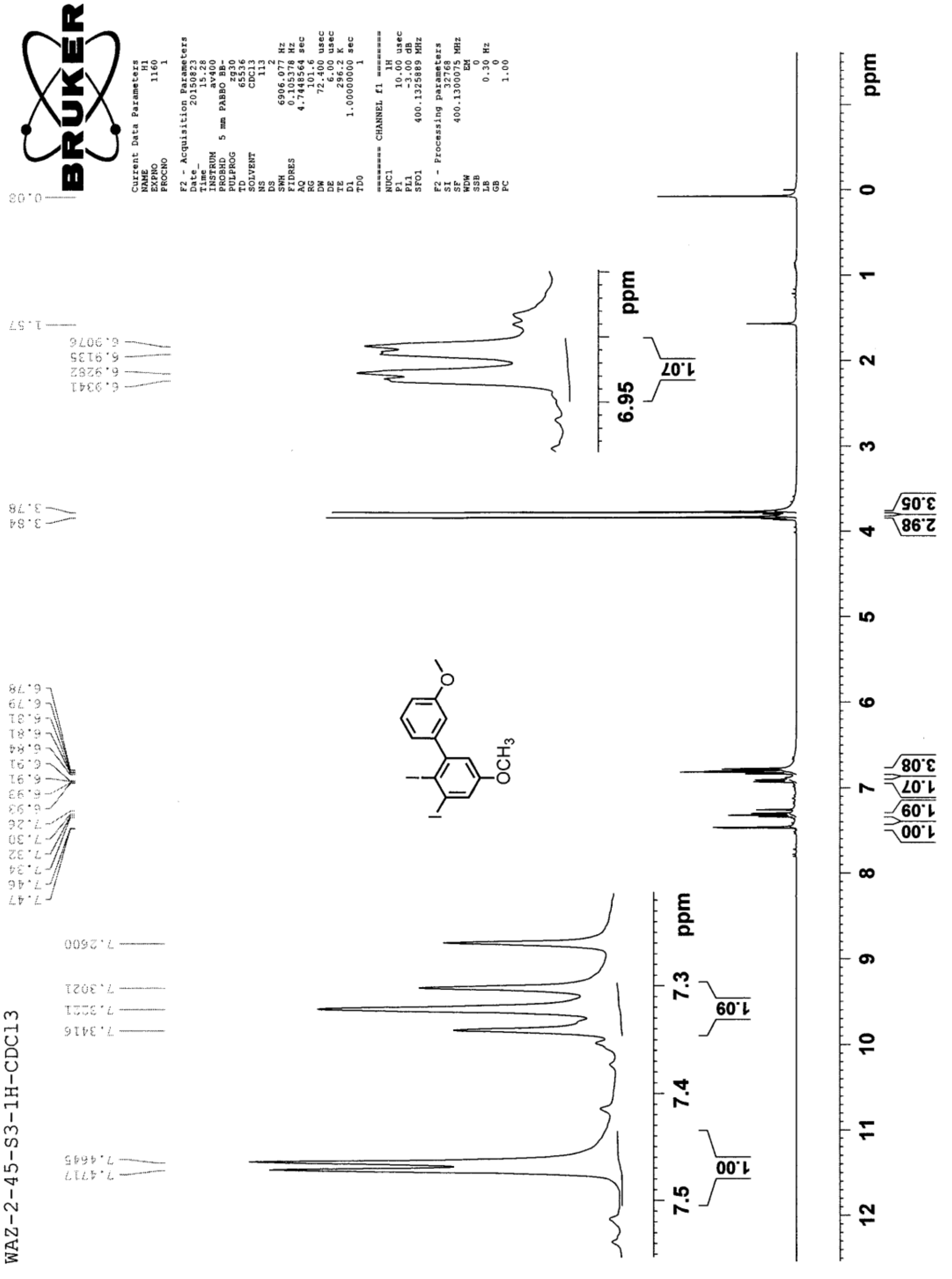


1.5.4 ^{13}C-NMR of 2,3-diiodo-4'-isopropyl-1,1'-biphenyl (15) in $CDCl_3$ at 25 ^{o}C.

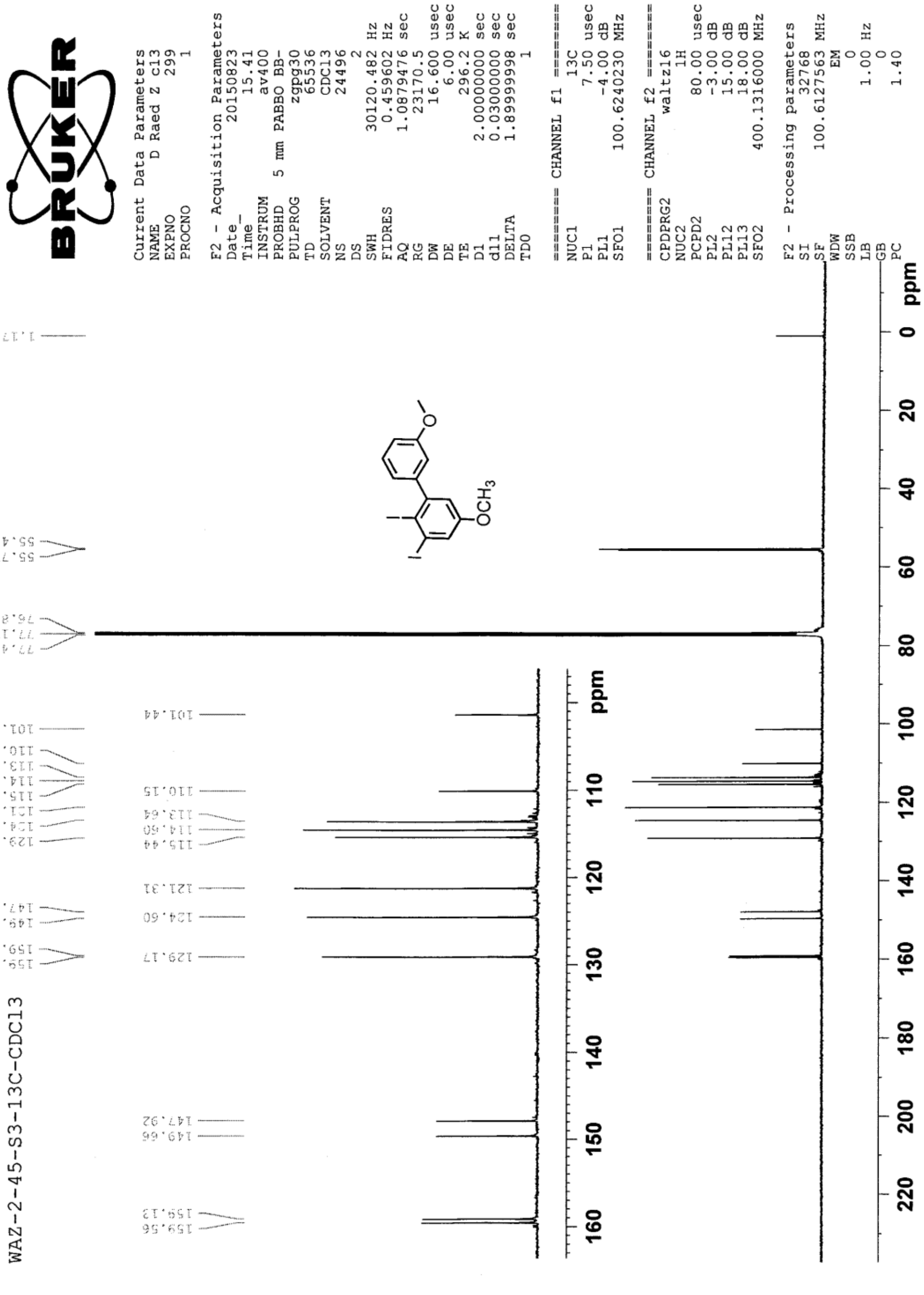


1.5.5

1.5.6 ^{1}H-NMR of 2,3-diiodo-3',5-dimethoxy-1,1'-biphenyl (16) in $CDCl_3$ at 25 ^oC.



1.5.7 ^{13}C-NMR of 2,3-diiodo-3',5-dimethoxy-1,1'-biphenyl (16) in $CDCl_3$ at 25 °C.



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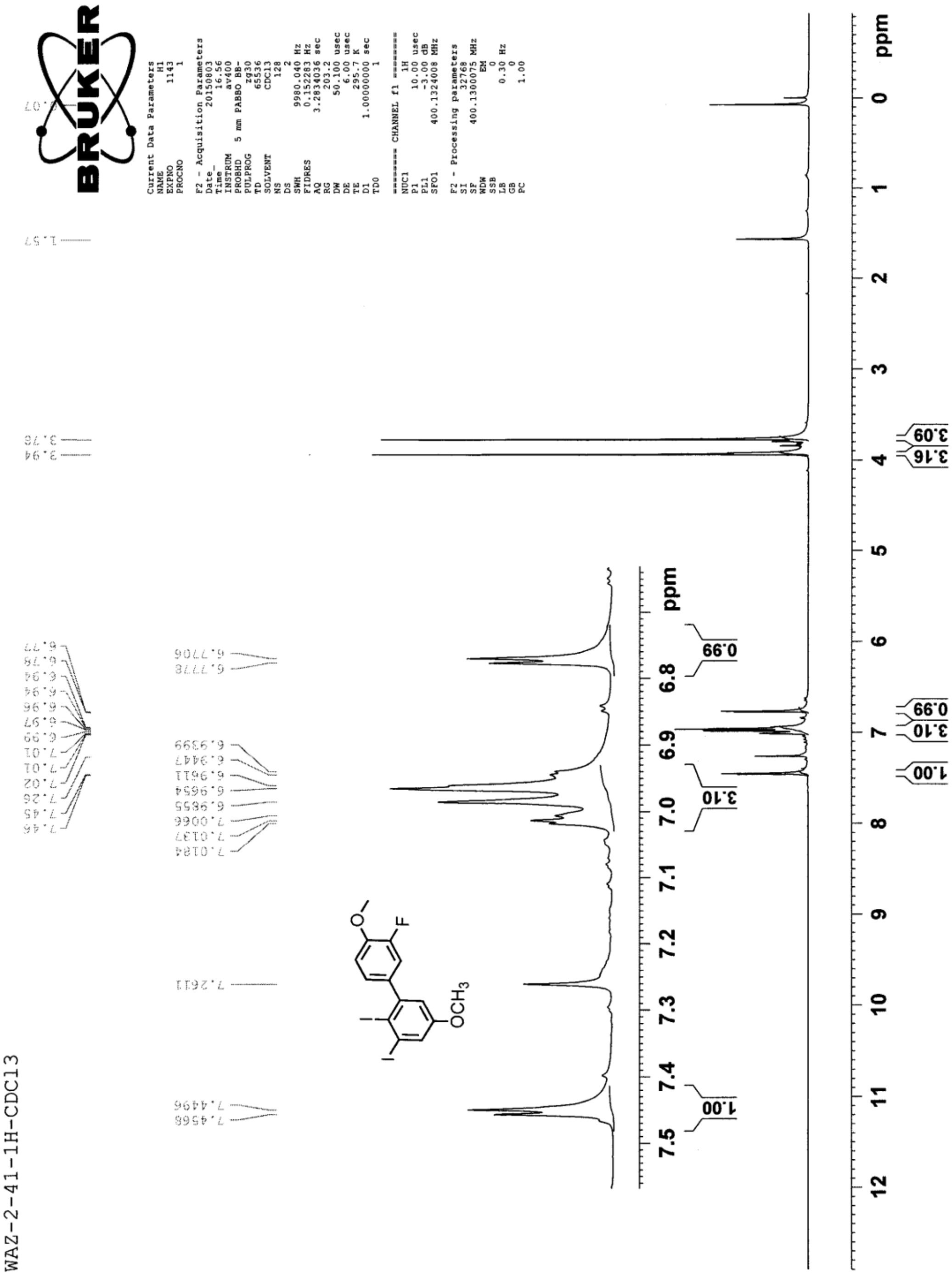
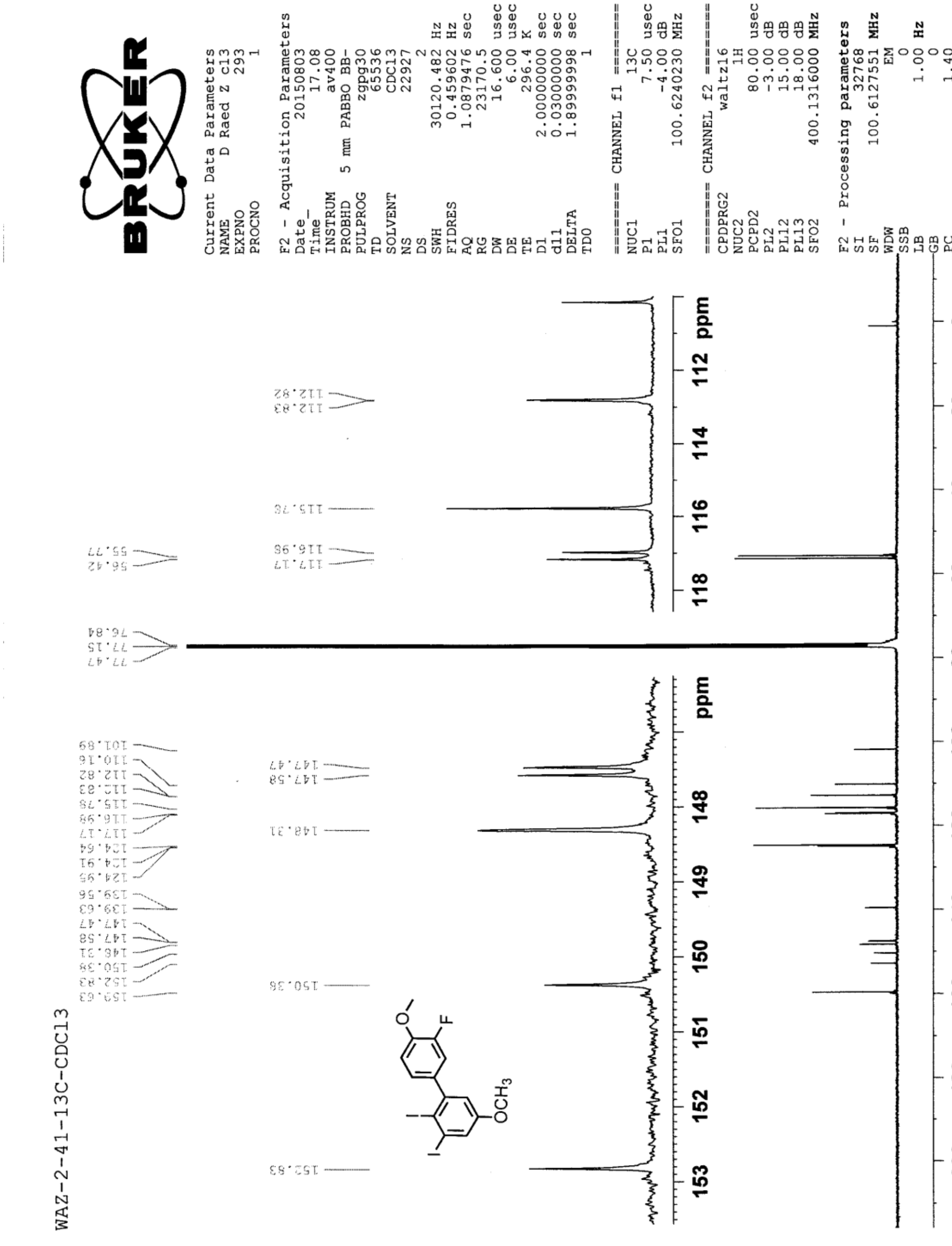
1.5.8

1.5.9 ^{1}H-NMR of 3'-fluoro-2,3-diiodo-4',5-dimethoxy-1,1'-biphenyl (17) in $CDCl_3$ at 25 °C.



1.5.10 ^{13}C-NMR of 3'-fluoro-2,3-diiodo-4',5-dimethoxy-1,1'-biphenyl (17) in $CDCl_3$ at 25 ^{o}C.



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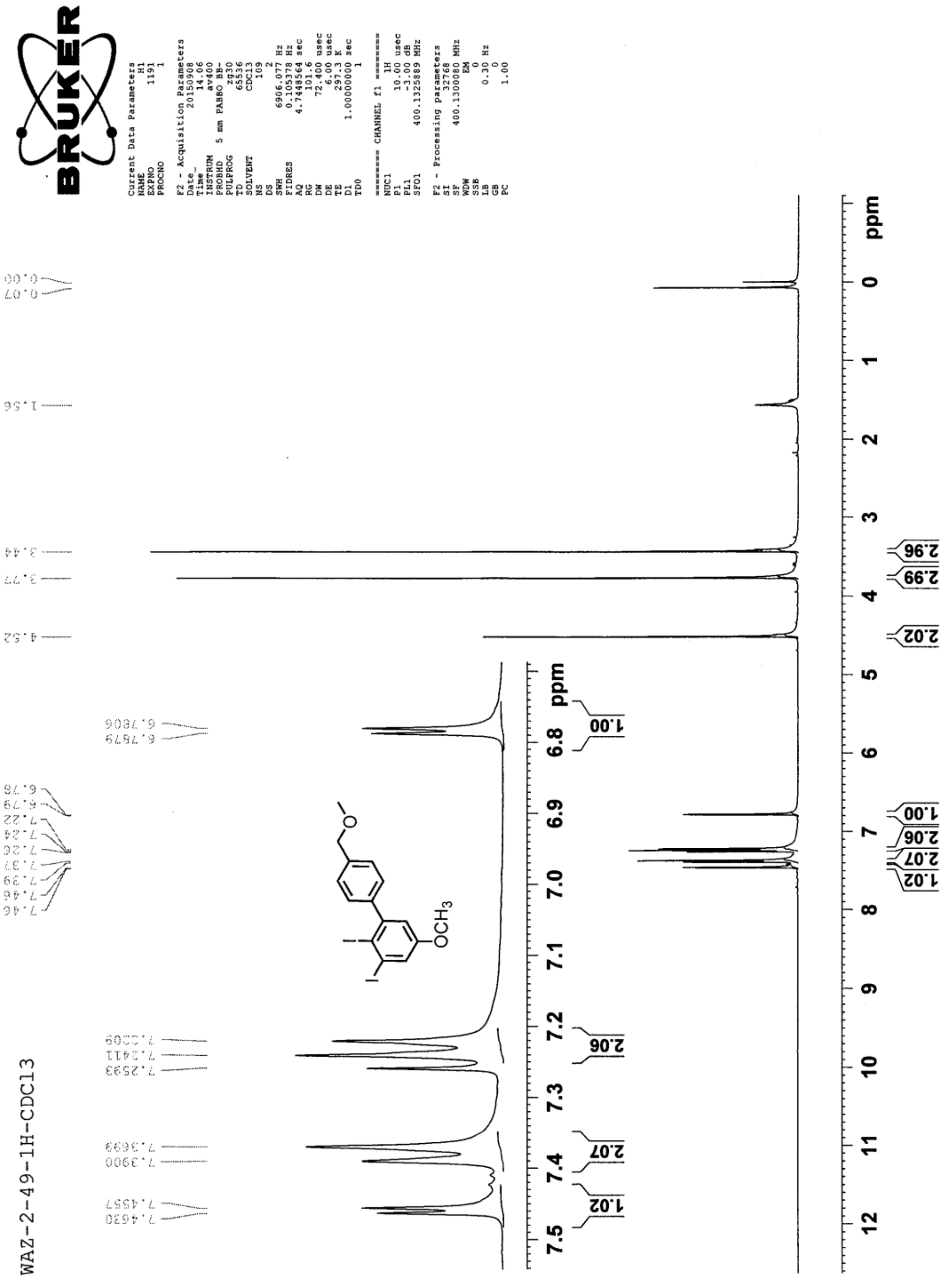
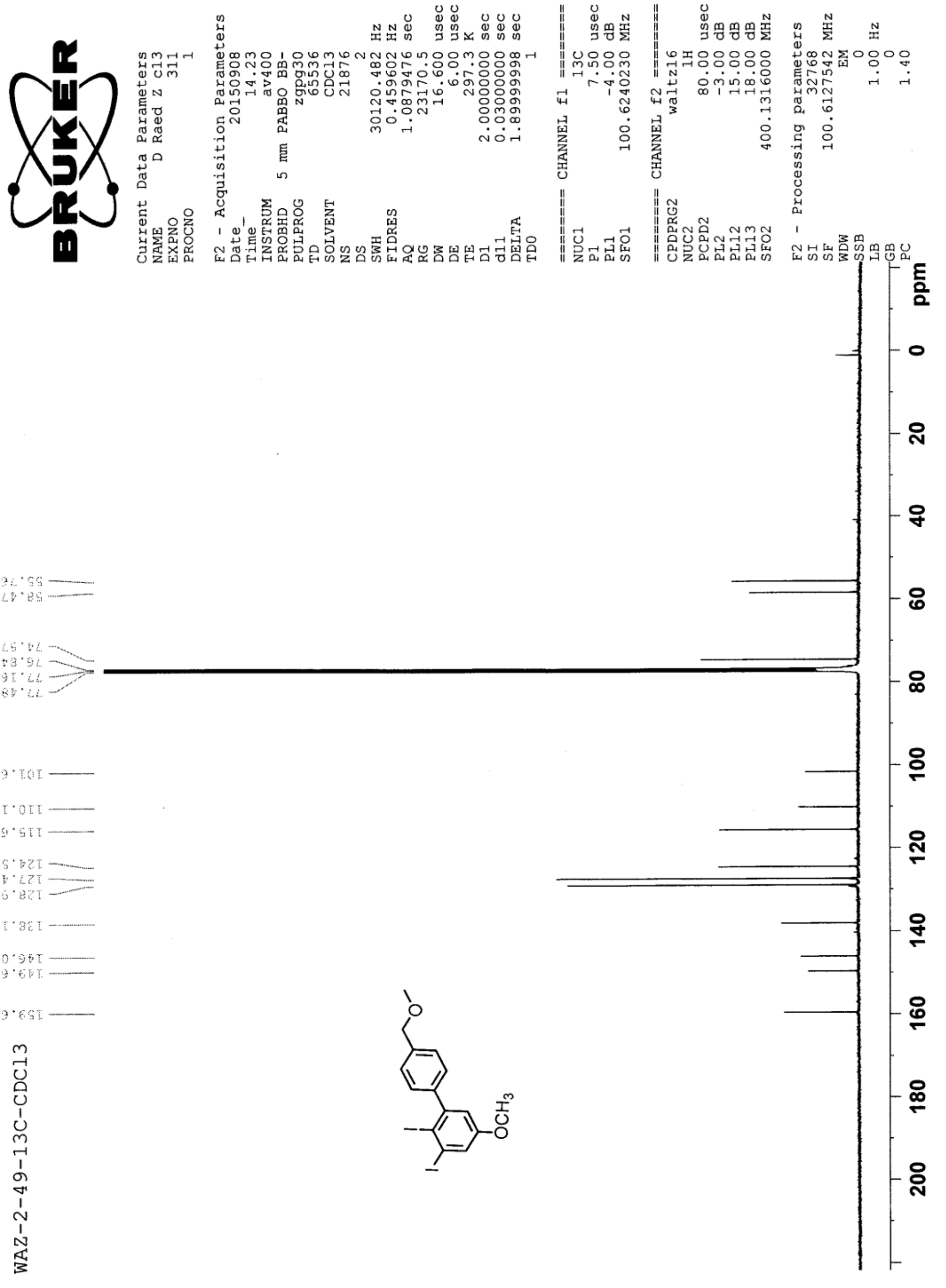
1.5.11

1.5.12 ^{1}H-NMR of 2,3-diiodo-5-methoxy-4'-(methoxymethyl)-1,1'-biphenyl (18) in $CDCl_3$ at 25 °C.



1.5.13 ^{13}C–NMR of 2,3-diiodo-5-methoxy-4'-(methoxymethyl)-1,1'-biphenyl (18) in $CDCl_3$ at 25 °C.



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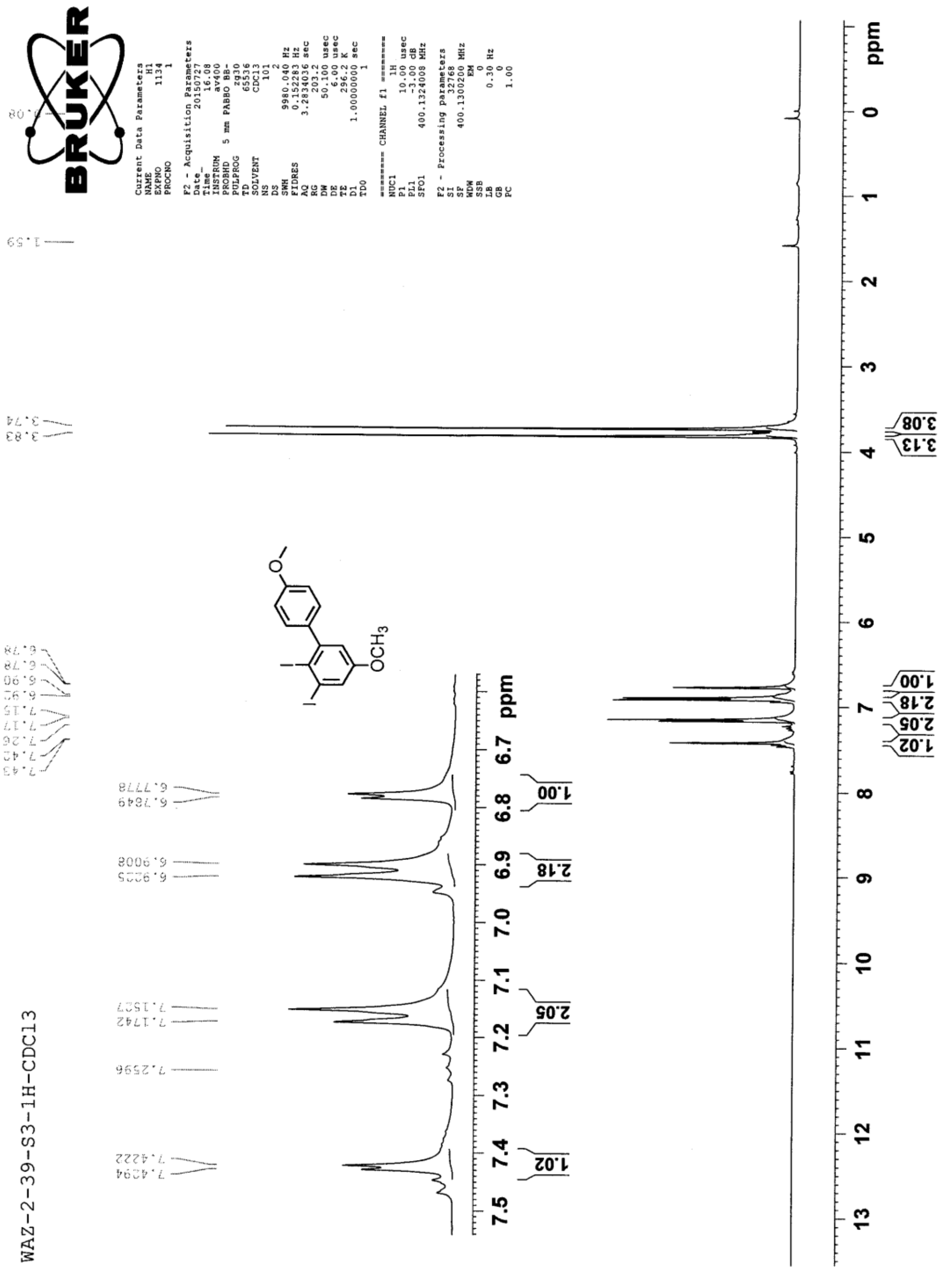
DELTA 1.89999998 sec
TD0 1

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

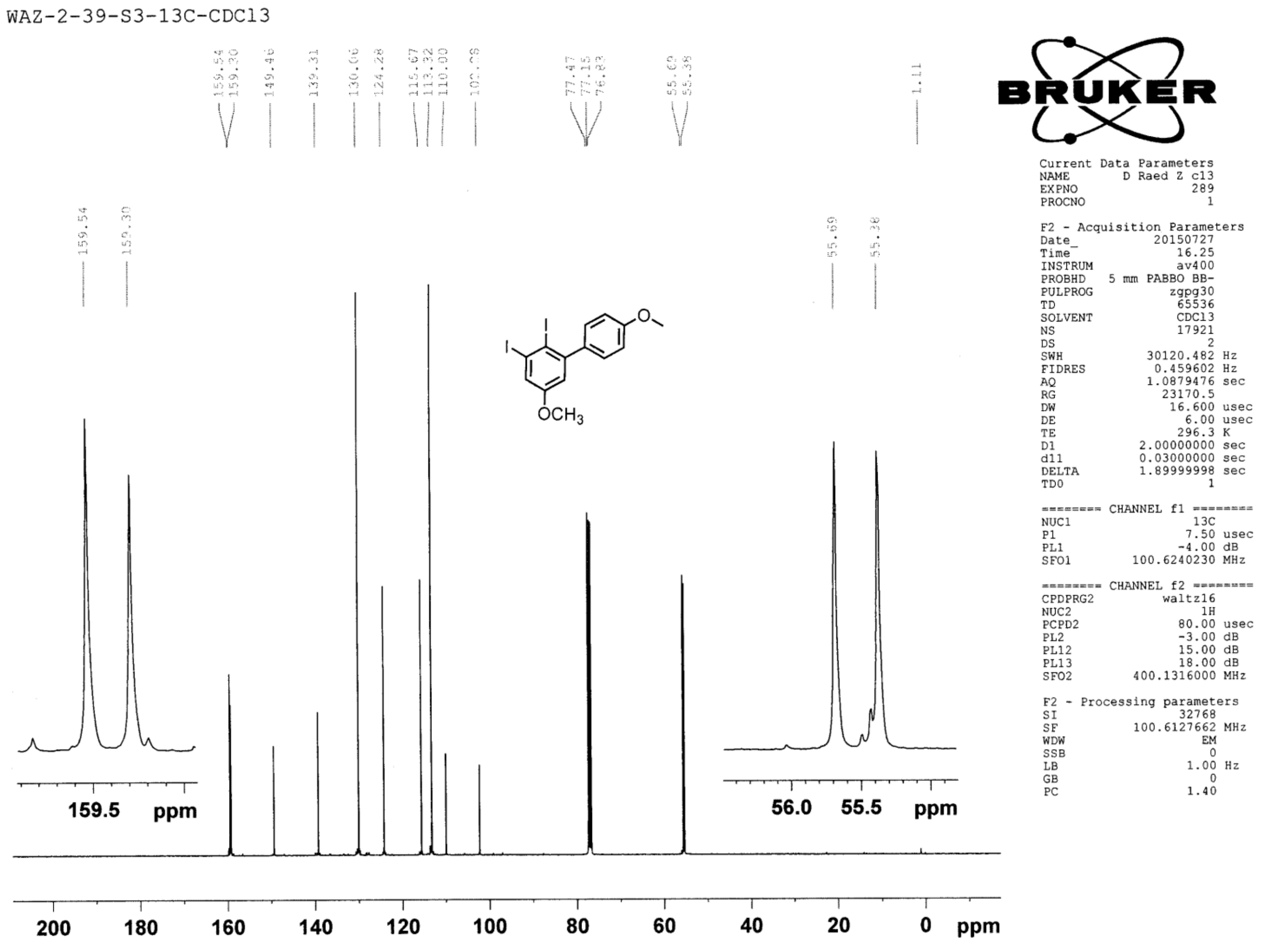
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 400.1316000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1.5.14 ^{1}H-NMR of 2,3-diiodo-4',5-dimethoxy-1,1'-biphenyl (19) in $CDCl_3$ at 25 °C.



1.5.15 ^{13}C-NMR of 2,3-diiodo-4',5-dimethoxy-1,1'-biphenyl (19) in $CDCl_3$ at 25 °C.



```
Current Data Parameters
NAME        D Raed Z c13
EXPNO                289
PROCNO                 1

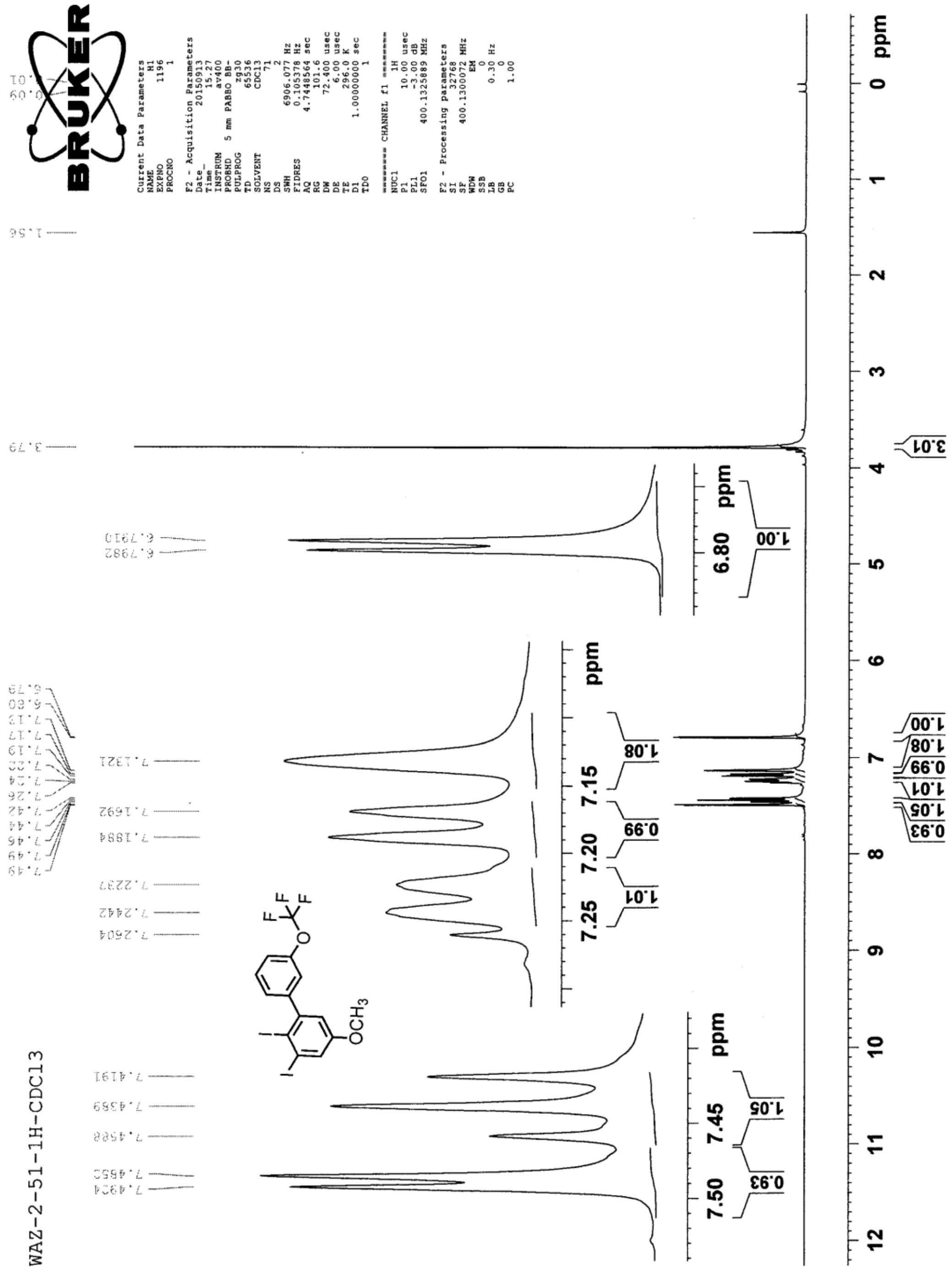
F2 - Acquisition Parameters
Date_           20150727
Time               16.25
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 17921
DS                     2
SWH            30120.482 Hz
FIDRES          0.459602 Hz
AQ             1.0879476 sec
RG               23170.5
DW                16.600 usec
DE                  6.00 usec
TE                 296.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

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

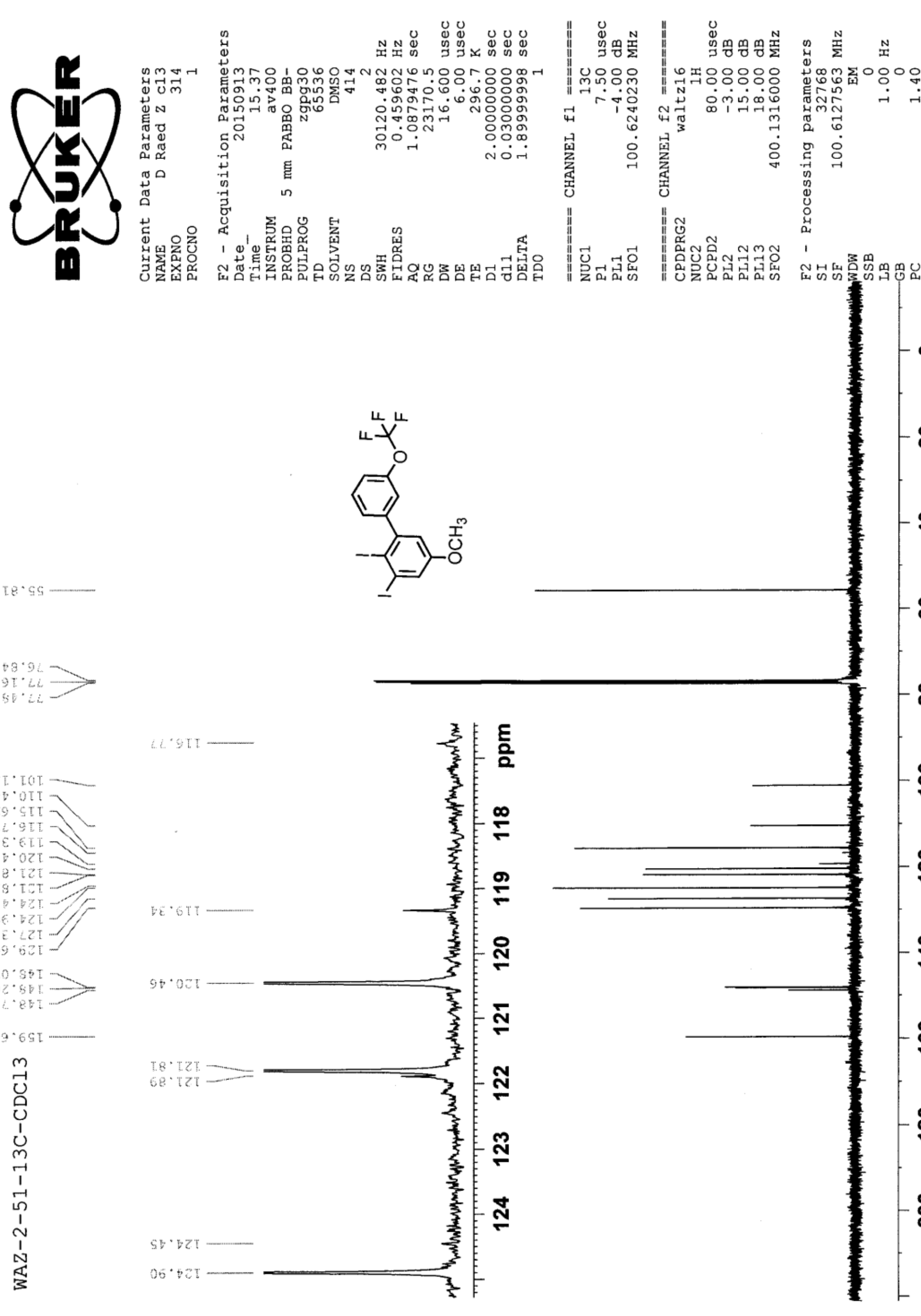
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               15.00 dB
PL13               18.00 dB
SFO2         400.1316000 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127662 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

1.5.16 ^{1}H-NMR of 2,3-diiodo-5-methoxy-3'-(trifluoromethoxy)-1,1'-biphenyl (20) in $CDCl_3$ at 25 ^oC.



1.5.17 ^{13}C-NMR of 2,3-diiodo-5-methoxy-3'-(trifluoromethoxy)-1,1'-biphenyl (20) in $CDCl_3$ at 25 °C.



```
Current Data Parameters
NAME        D Raed Z c13
EXPNO                314
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150913
Time               15.37
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                  414
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                296.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

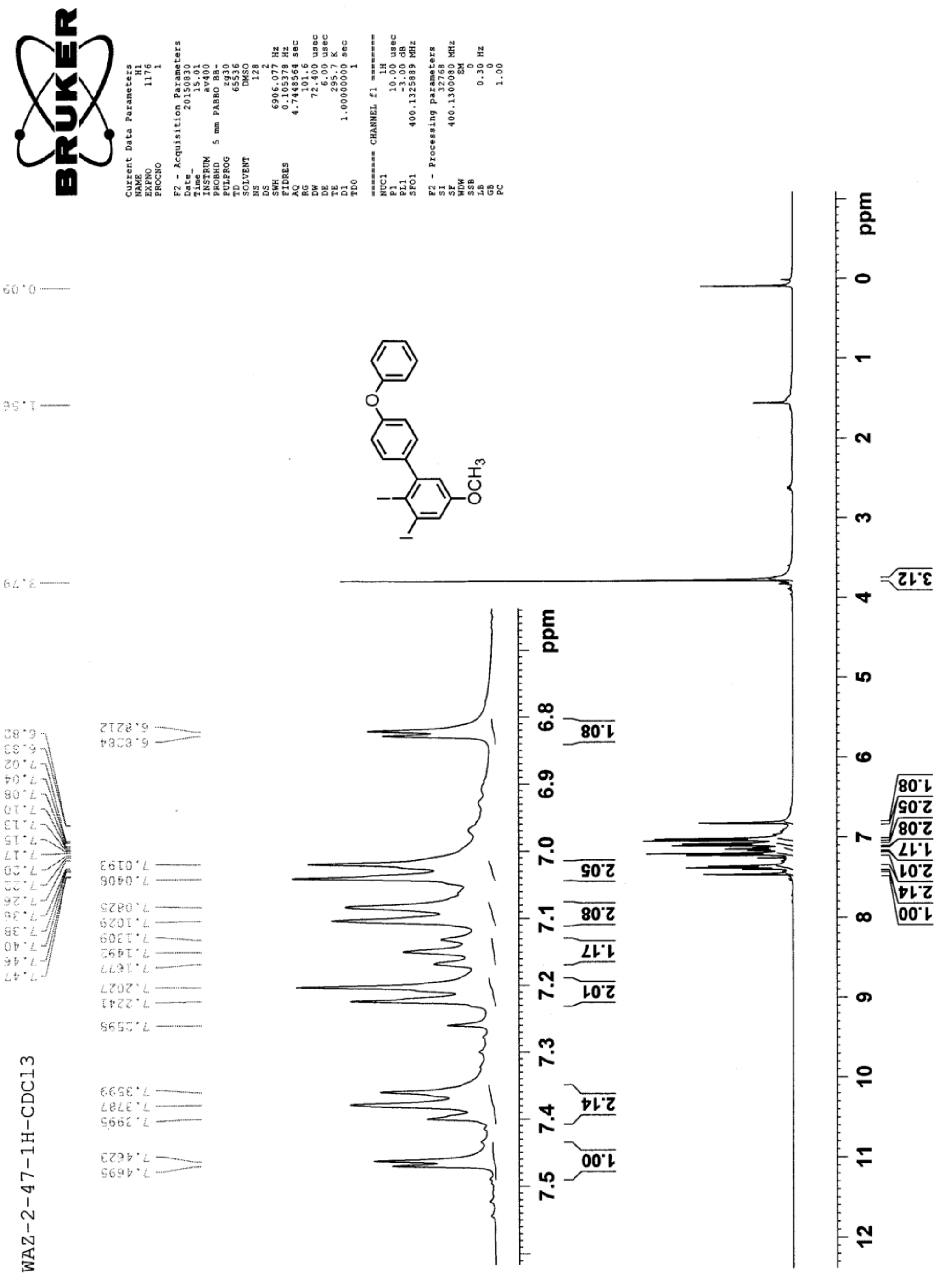
======== CHANNEL f1 ========
NUC1                13C
P1                 7.50 usec
PL1               -4.00 dB
SFO1        100.6240230 MHz

======== 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

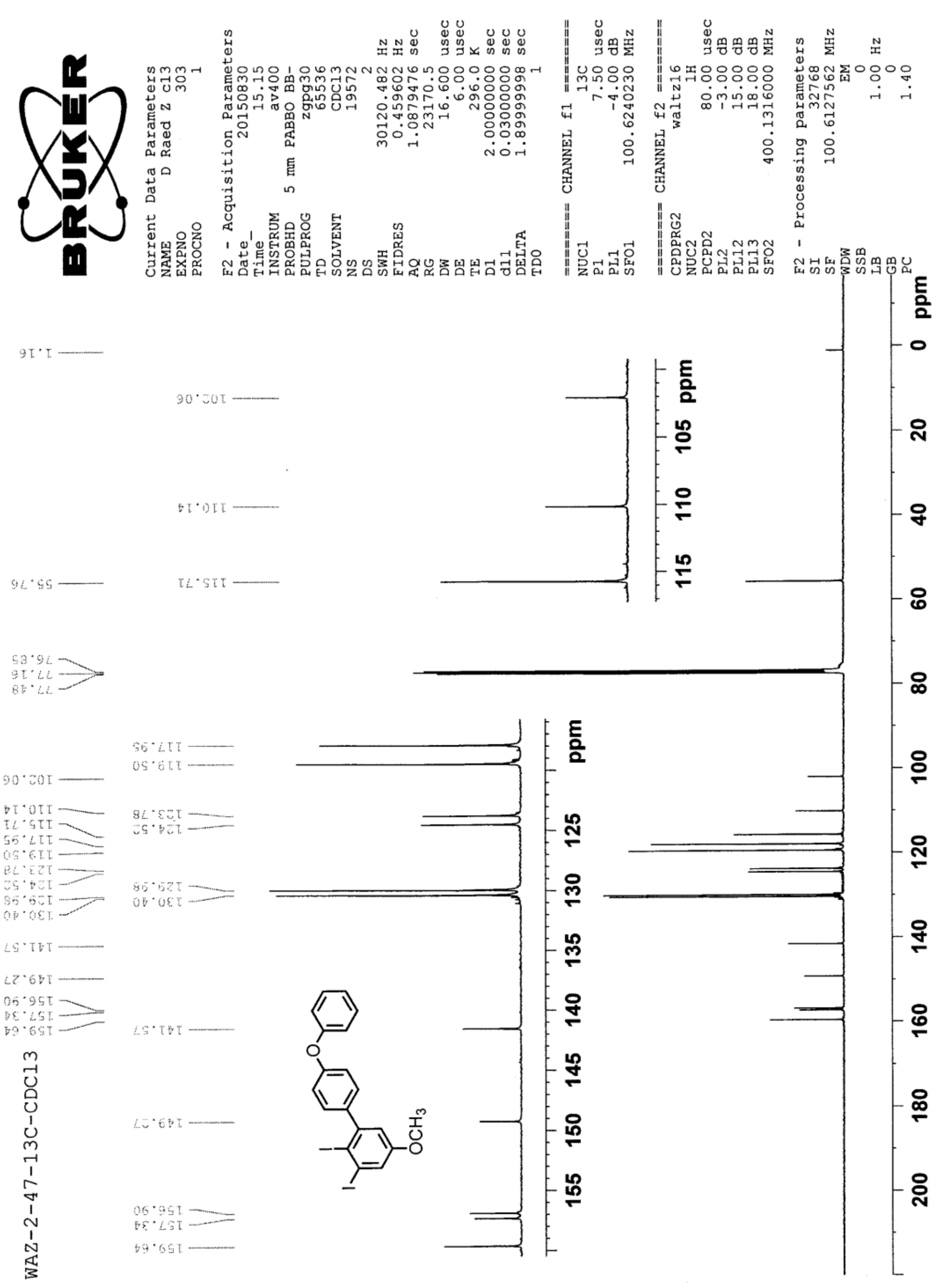
F2 - Processing parameters
SI                32768
SF          100.6127563 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

1.5.18 .

1.5.19 ^{1}H-NMR of 2,3-diiodo-5-methoxy-4'-phenoxy-1,1'-biphenyl (21) in $CDCl_3$ at 25 ^oC.



1.5.20 ^{13}C-NMR of 2,3-diiodo-5-methoxy-4'-phenoxy-1,1'-biphenyl (21) in $CDCl_3$ at 25 °C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  303
PROCNO                   1

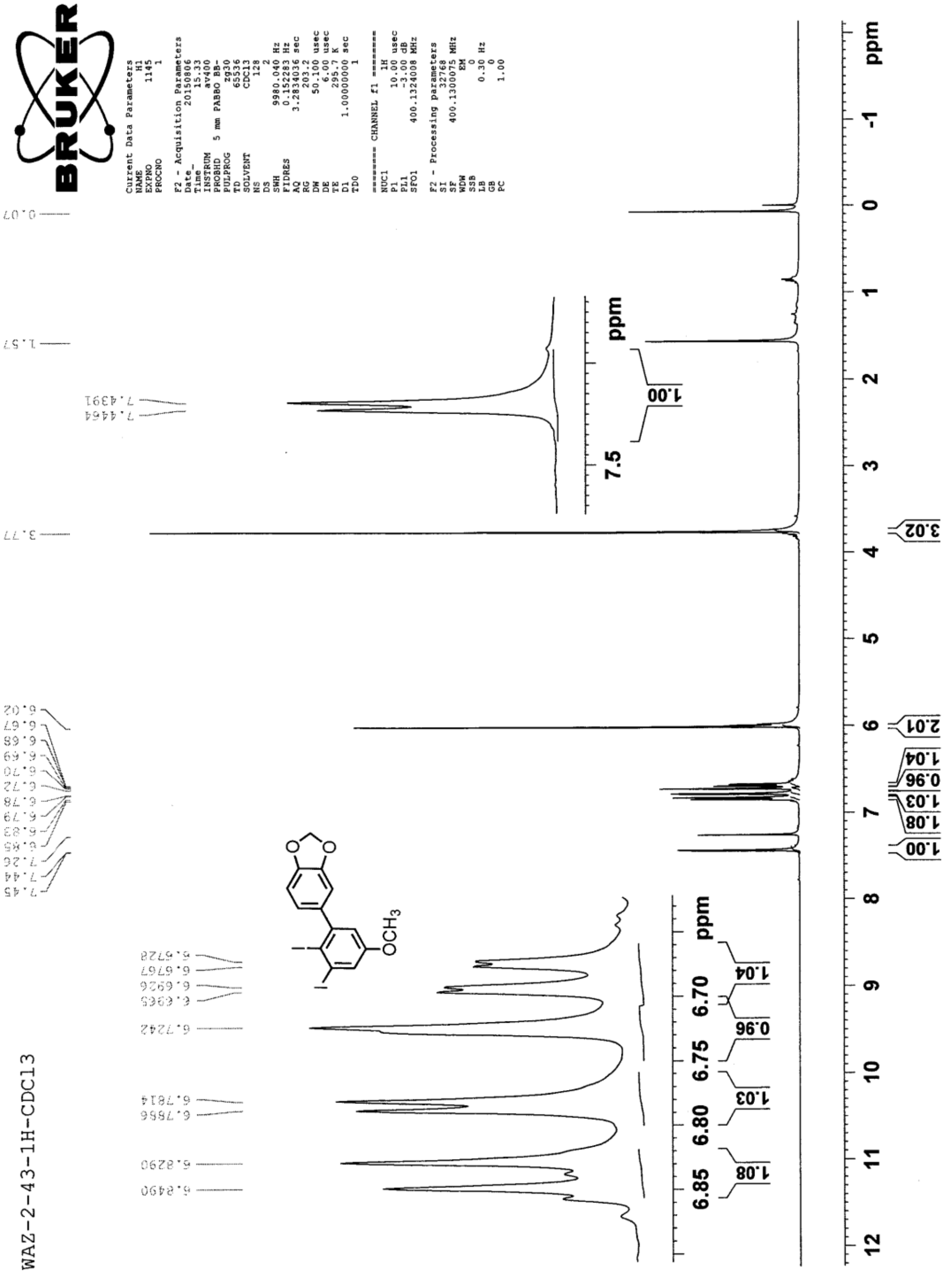
F2 - Acquisition Parameters
Date_             20150830
Time_                15.15
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT              CDC13
NS                   19572
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   296.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

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

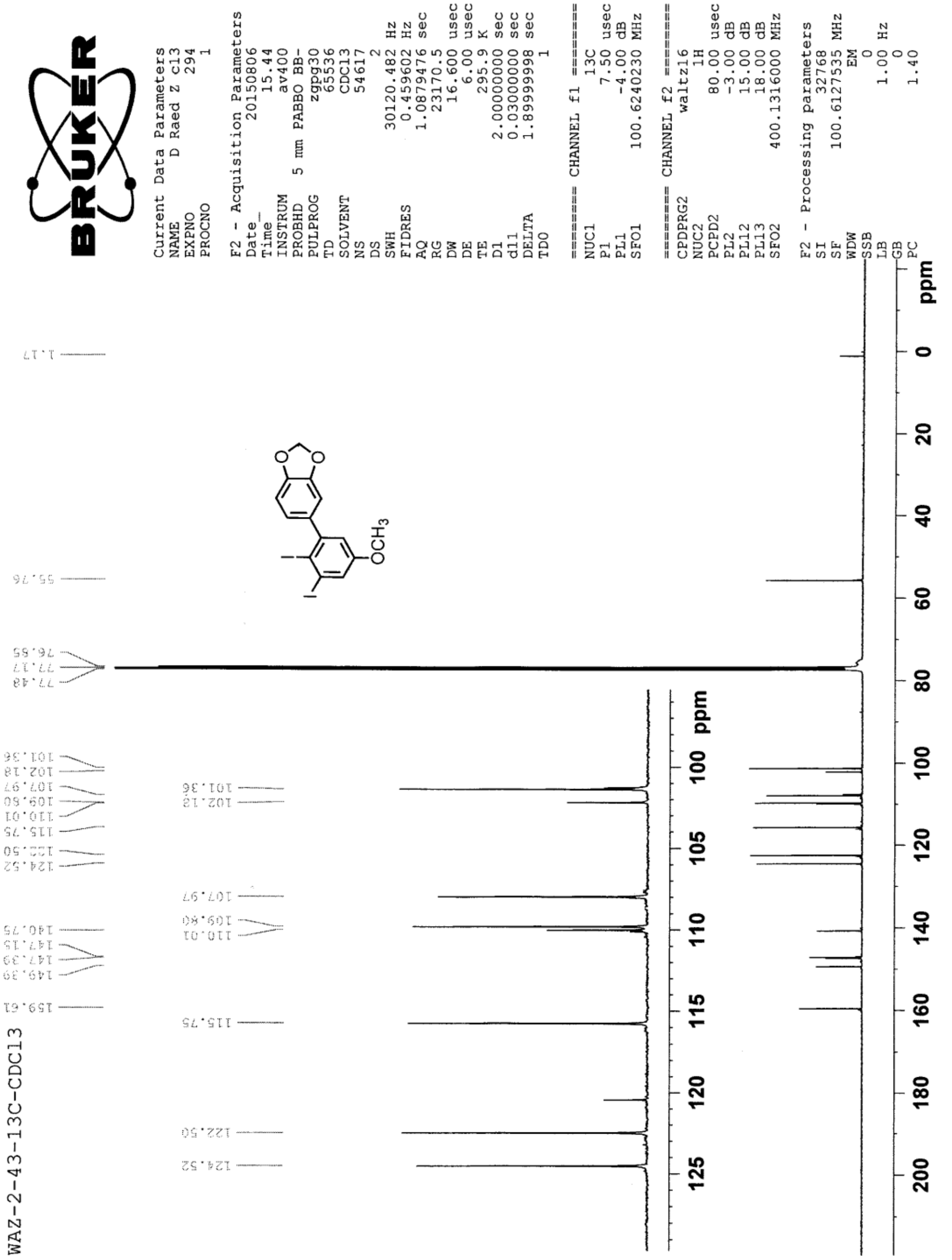
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 15.00 dB
PL13                 18.00 dB
SFO2           400.1316000 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127562 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

1.5.21 ^{1}H-NMR of 5-(2,3-diiodo-5-methoxyphenyl)benzo[d][1,3]dioxole (22) in CDCl$_3$ at 25 °C.



1.5.22 ^{13}C-NMR of 5-(2,3-diiodo-5-methoxyphenyl)benzo[d][1,3]dioxole (22) in $CDCl_3$ at 25 °C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  294
PROCNO                   1

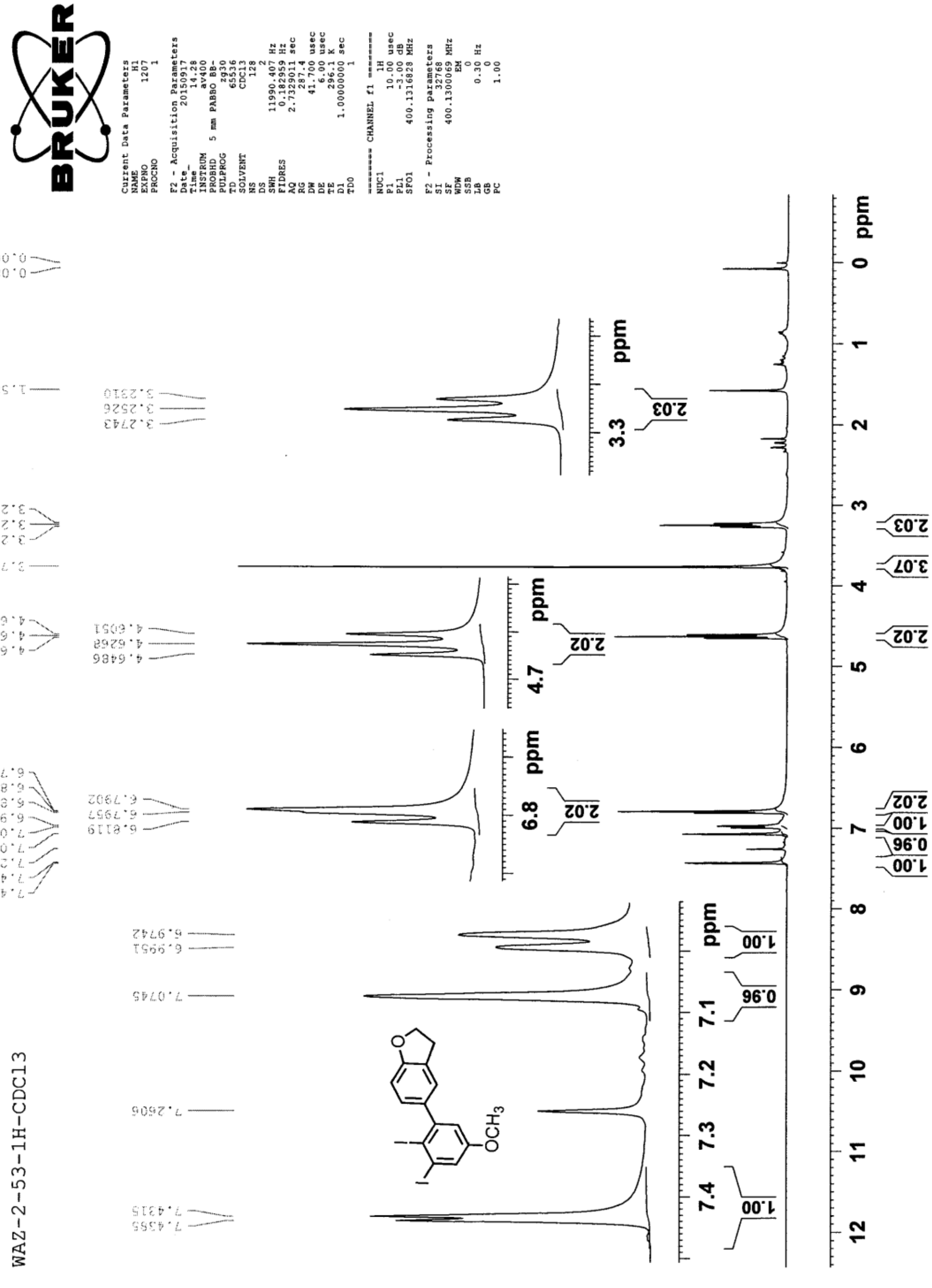
F2 - Acquisition Parameters
Date_             20150806
Time                 15.44
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                   54617
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   295.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                      1

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

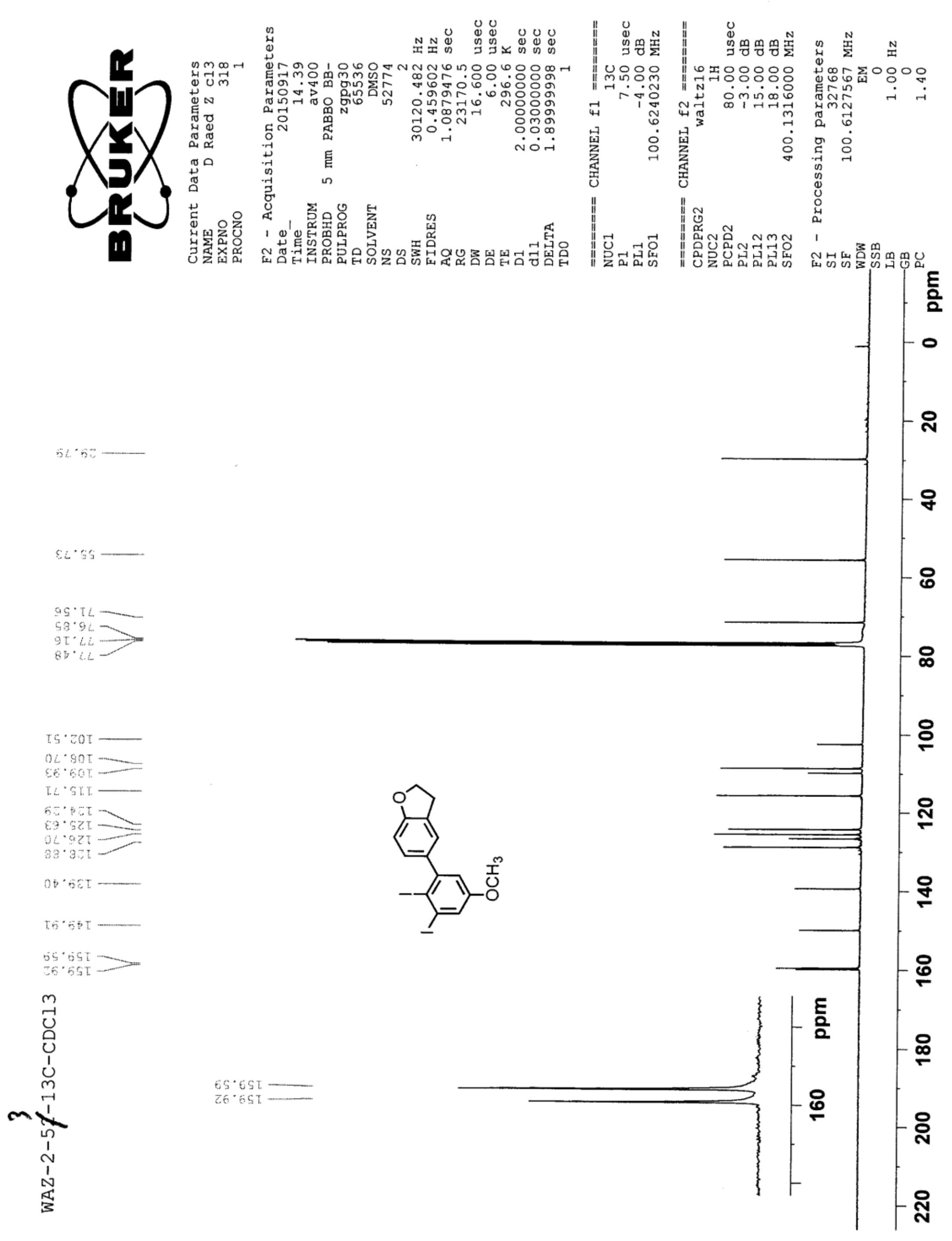
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 15.00 dB
PL13                 18.00 dB
SFO2          400.1316000 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127535 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

1.5.23 ^{1}H-NMR of 5-(2,3-diiodo-5-methoxyphenyl)-2,3-dihydrobenzofuran (23) in $CDCl_3$ at 25 °C.



1.5.24 ^{13}C-NMR of 5-(2,3-diiodo-5-methoxyphenyl)-2,3-dihydrobenzofuran (23) in CDCl$_3$ at 25 ^{o}C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  318
PROCNO                   1

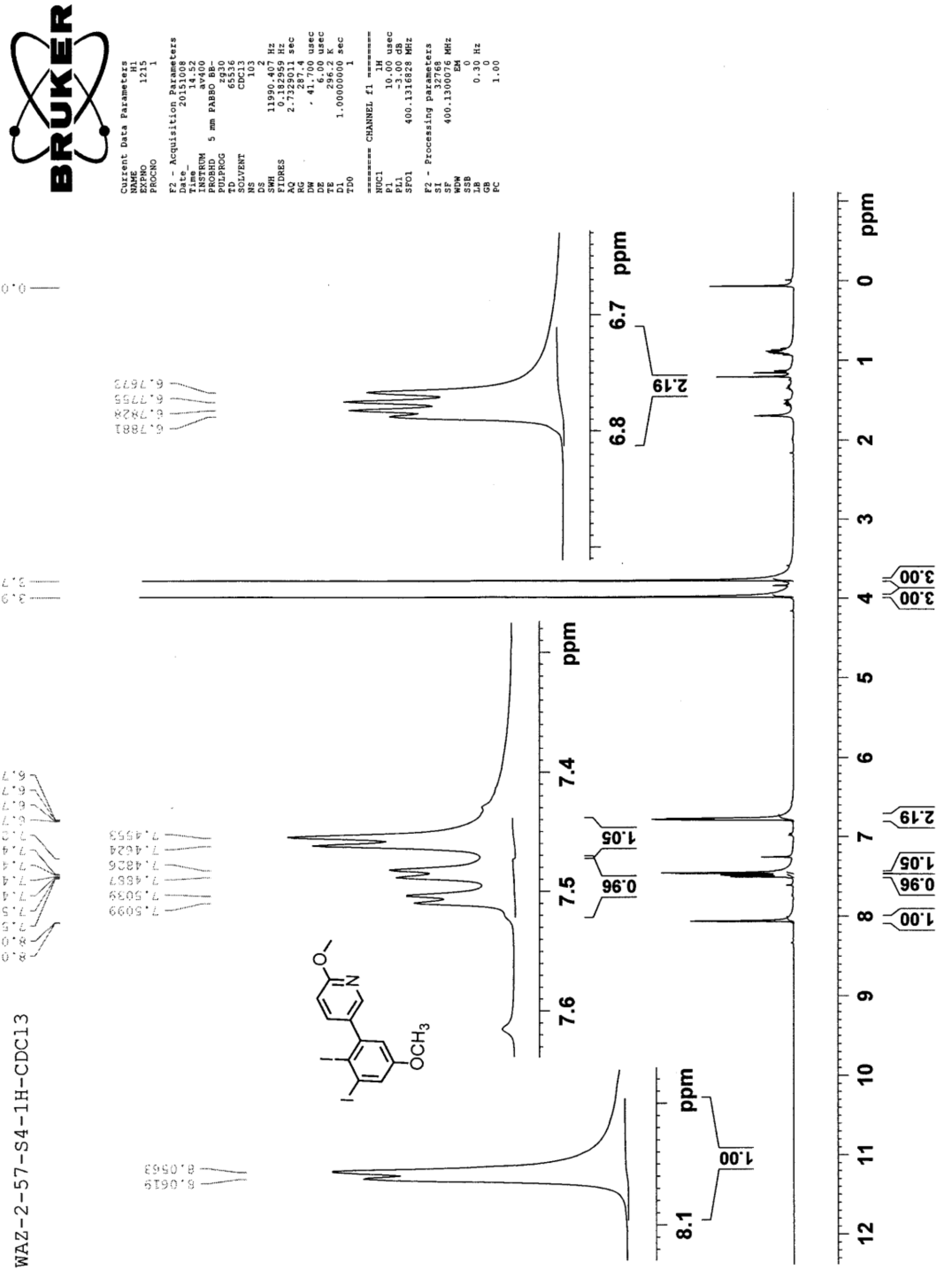
F2 - Acquisition Parameters
Date_             20150917
Time                 14.39
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               DMSO
NS                   52774
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   296.6 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

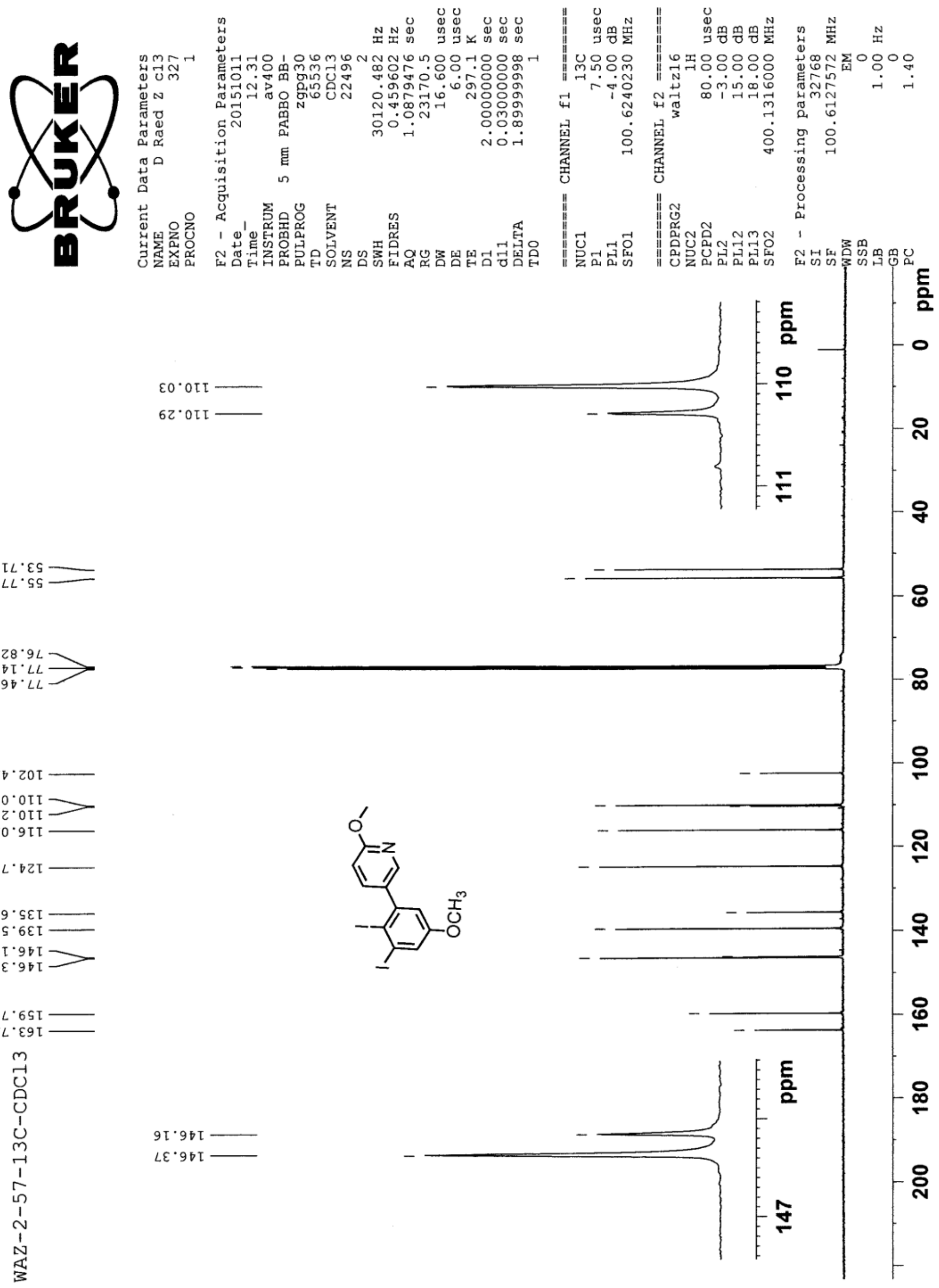
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 15.00 dB
PL13                 18.00 dB
SFO2          400.1316000 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127567 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

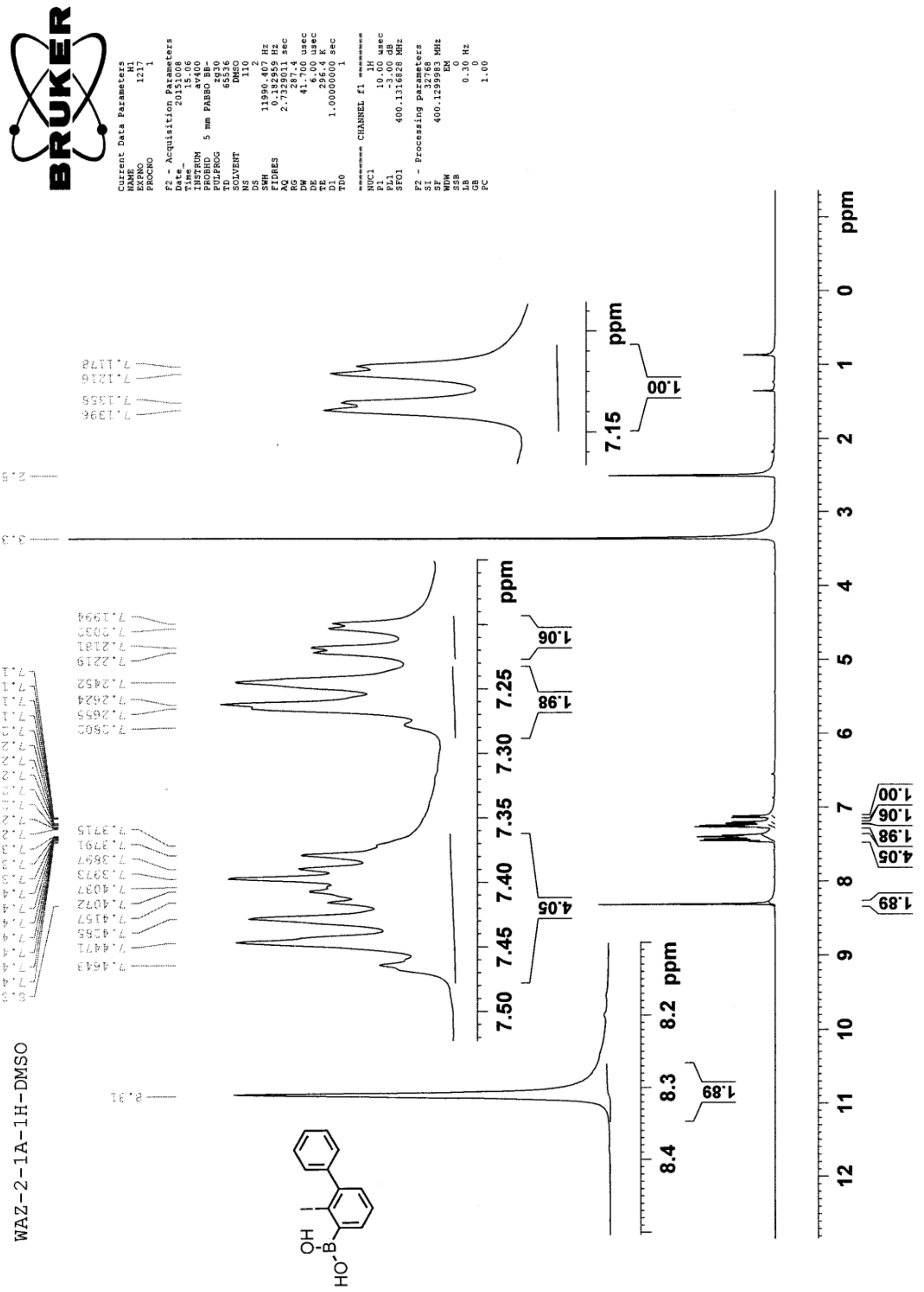
1.5.25 ^{1}H-NMR of 5-(2,3-diiodo-5-methoxyphenyl)-2-methoxypyridine (24) in $CDCl_3$ at 25 ^oC.



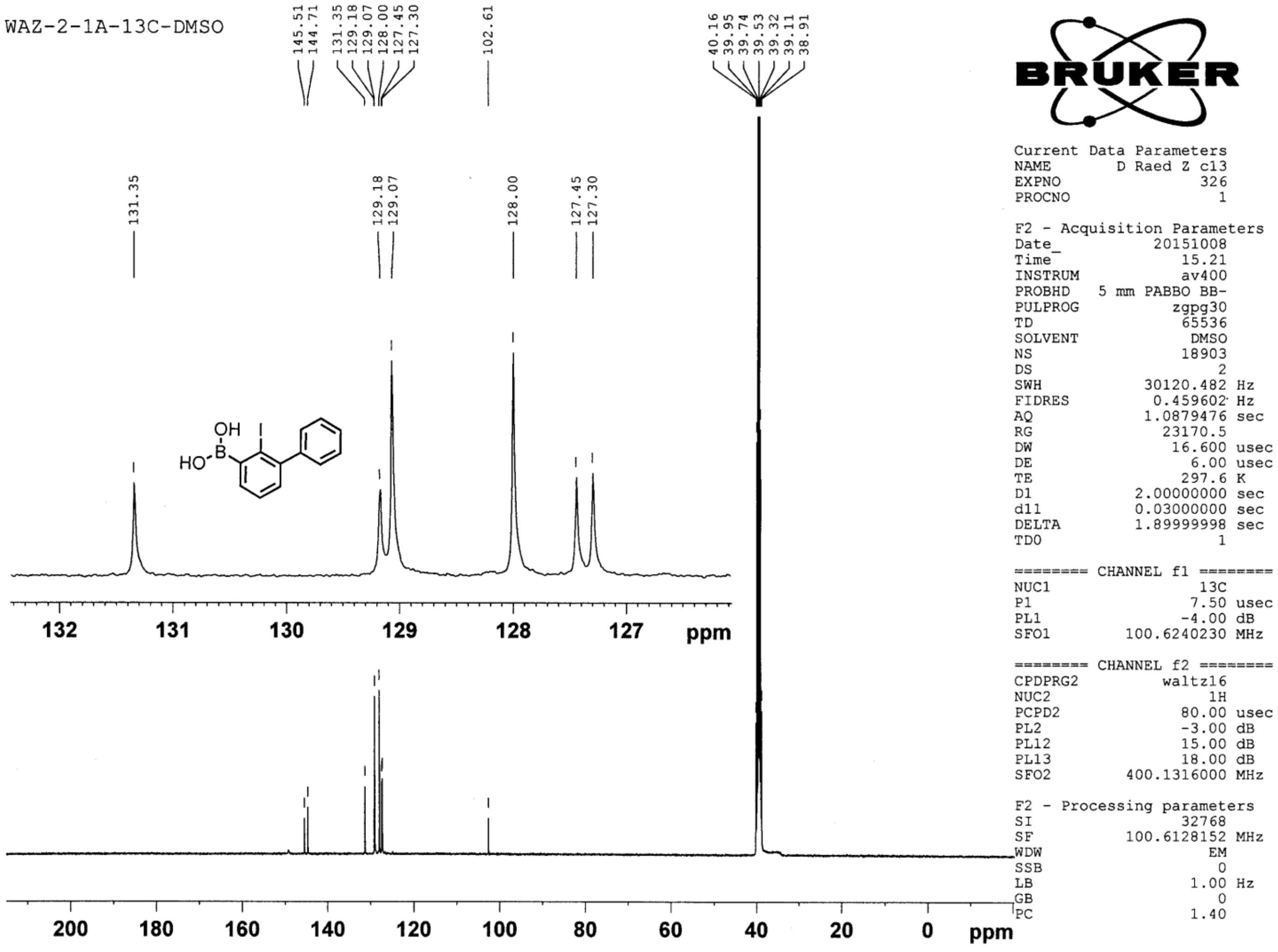
1.5.26 ^{13}C-NMR of 5-(2,3-diiodo-5-methoxyphenyl)-2-methoxypyridine (24) in $CDCl_3$ at 25 °C.



1.5.27 ^{1}H-NMR of (2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (5_A) in d^6-DMSO at 25 ^{o}C.



1.5.28 ^{13}C–NMR of (2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (5_A) in d^6-DMSO at 25 °C.



```
Current Data Parameters
NAME        D Raed Z c13
EXPNO                326
PROCNO                 1

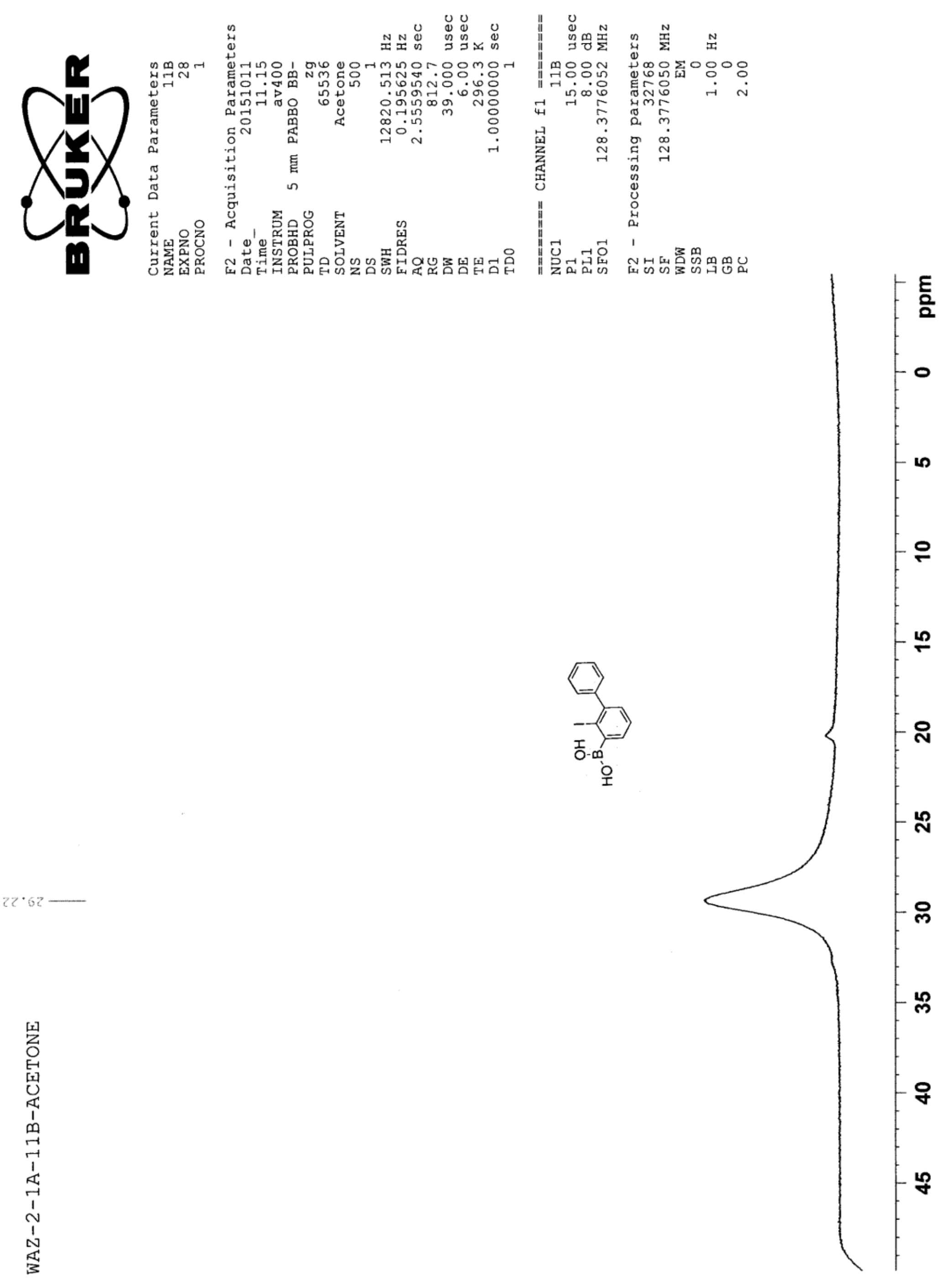
F2 - Acquisition Parameters
Date_           20151008
Time               15.21
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                 18903
DS                     2
SWH           30120.482 Hz
FIDRES         0.459602 Hz
AQ            1.0879476 sec
RG               23170.5
DW                16.600 usec
DE                  6.00 usec
TE                 297.6 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    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
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

1.5.29 ^{11}B-NMR of (2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (5_A) in d^6-acetone at 25 ^{o}C



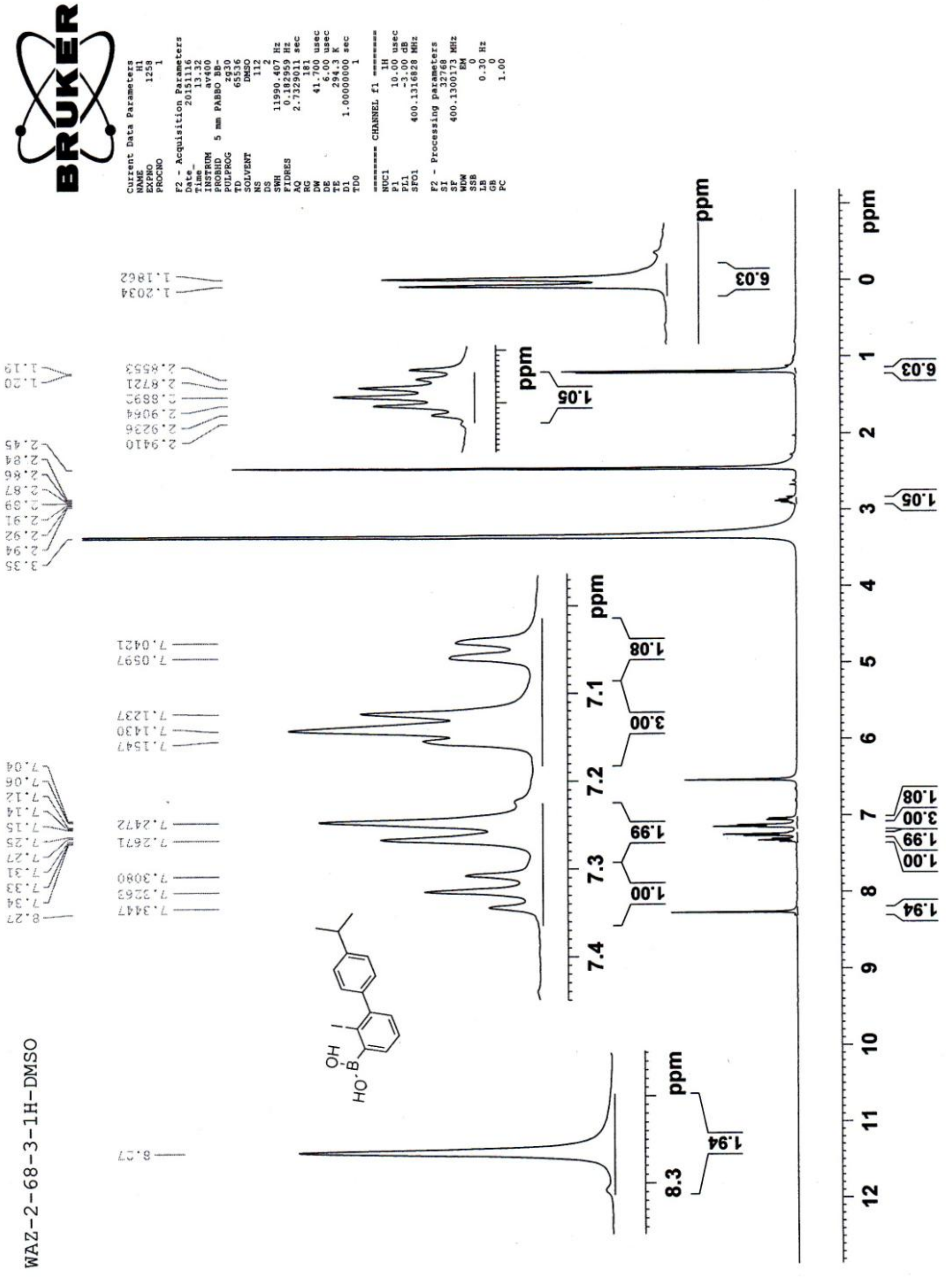
```
Current Data Parameters
NAME              11B
EXPNO              28
PROCNO              1

F2 - Acquisition Parameters
Date_        20151011
Time            11.15
INSTRUM         av400
PROBHD   5 mm PABBO BB-
PULPROG            zg
TD              65536
SOLVENT       Acetone
NS                500
DS                  1
SWH         12820.513 Hz
FIDRES       0.195625 Hz
AQ          2.5559540 sec
RG              812.7
DW             39.000 usec
DE               6.00 usec
TE              296.3 K
D1         1.00000000 sec
TD0                 1

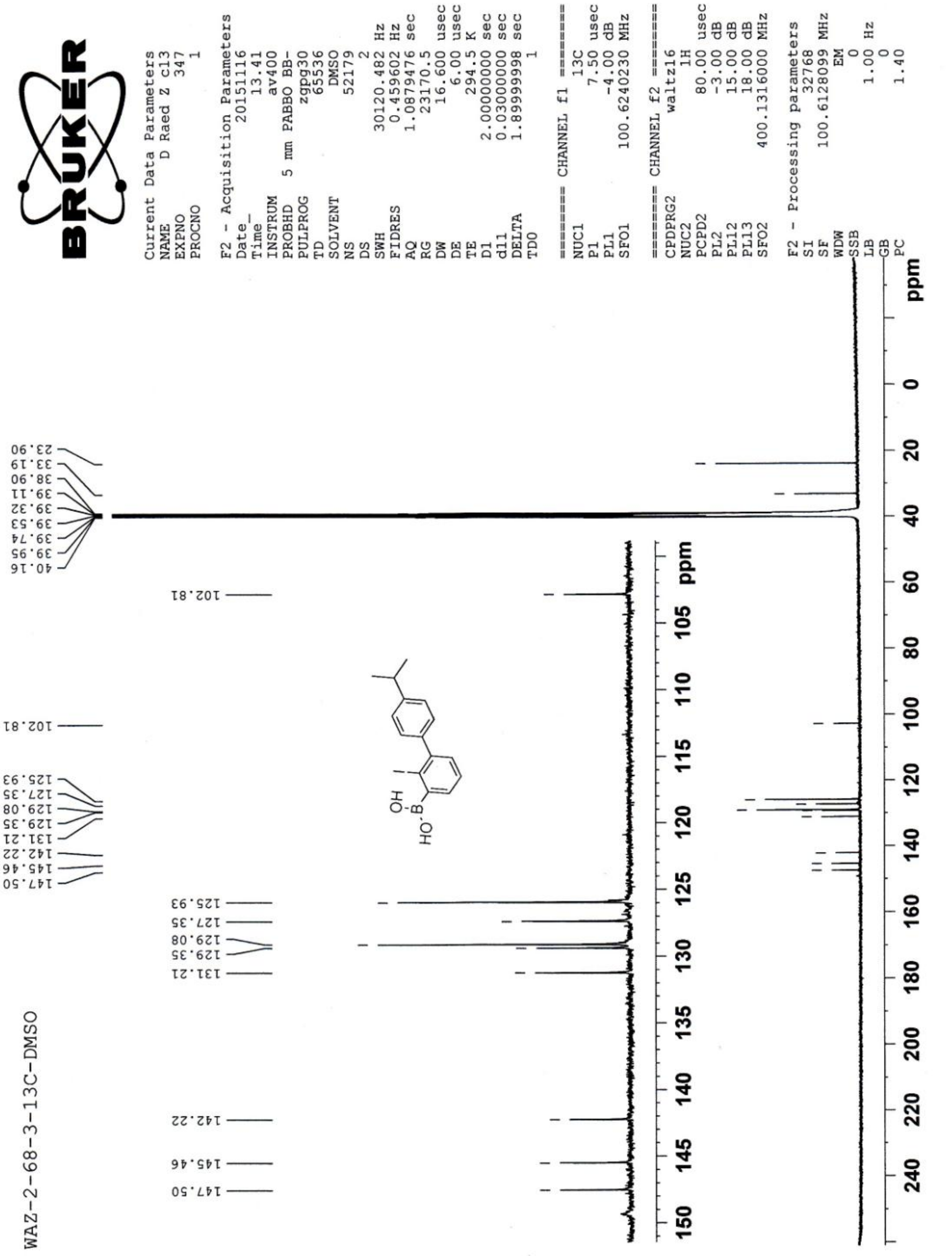
======== CHANNEL f1 ========
NUC1              11B
P1              15.00 usec
PL1              8.00 dB
SFO1      128.3776052 MHz

F2 - Processing parameters
SI              32768
SF        128.3776050 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               2.00
```

1.5.30 ¹H-NMR of (2-iodo-4'-isopropyl-[1,1'-biphenyl]-3-yl)boronic acid (6_A) in d^6-DMSO at 25 °C.



1.5.31 ^{13}C–NMR of (2-iodo-4'-isopropyl-[1,1'-biphenyl]-3-yl)boronic acid (6_A) in d^{6}-DMSO at 25 ^{o}C.



```
Current Data Parameters
NAME          D Raeed Z c13
EXPNO                   347
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151116
Time                  13.41
INSTRUM               av400
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT                DMSO
NS                    52179
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                  294.5 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                       1

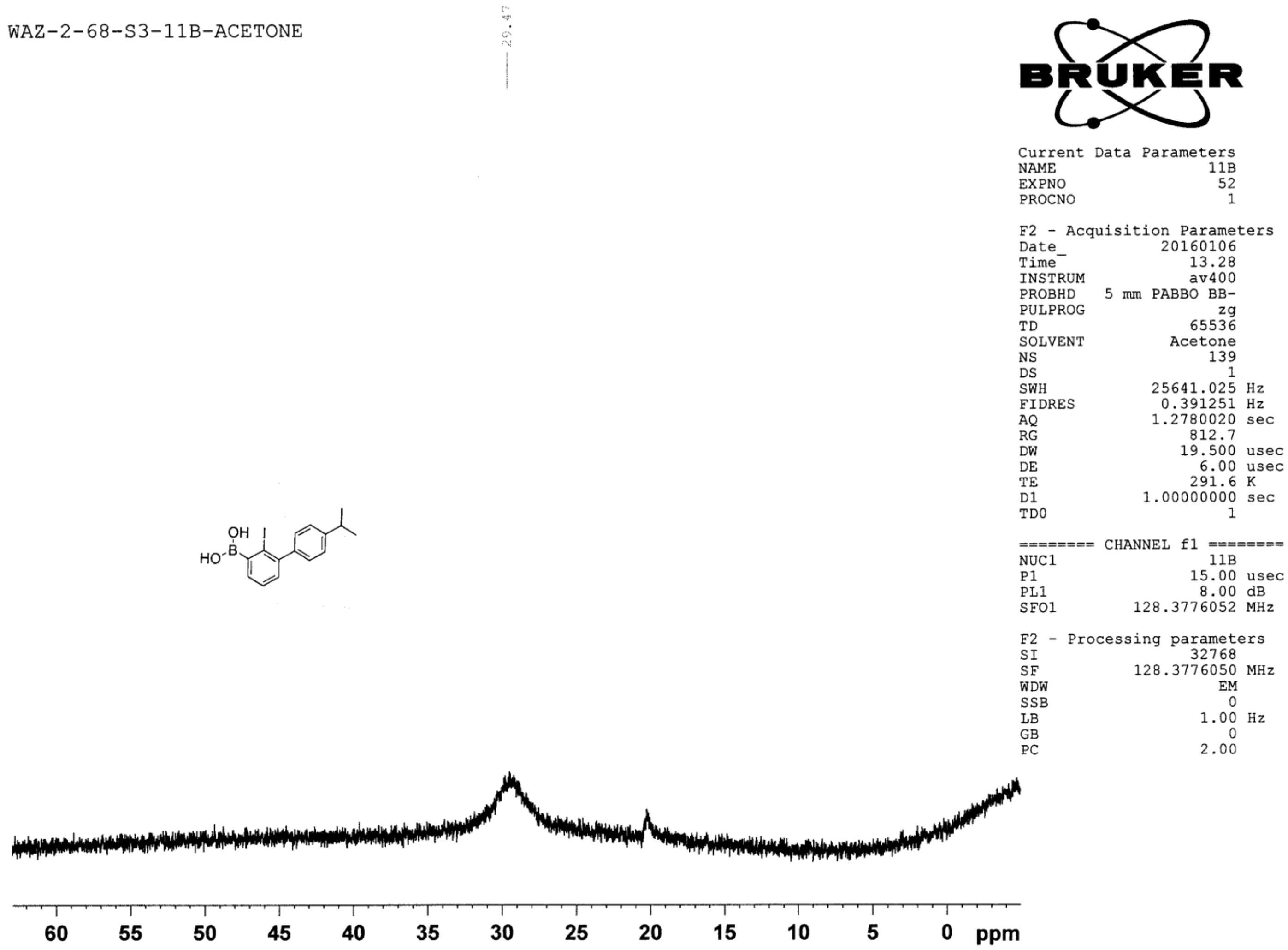
======== CHANNEL f1 ========
NUC1                    13C
P1                 7.50 usec
PL1                -4.00 dB
SFO1         100.6240230 MHz

======== 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           100.6128099 MHz
WDW                      EM
SSB                       0
LB                  1.00 Hz
GB                        0
PC                     1.40
```

1.5.32 ^{11}B-NMR of (2-iodo-4'-isopropyl-[1,1'-biphenyl]-3-yl)boronic acid (6$_A$) in d^6-acetone at 25 °C

.



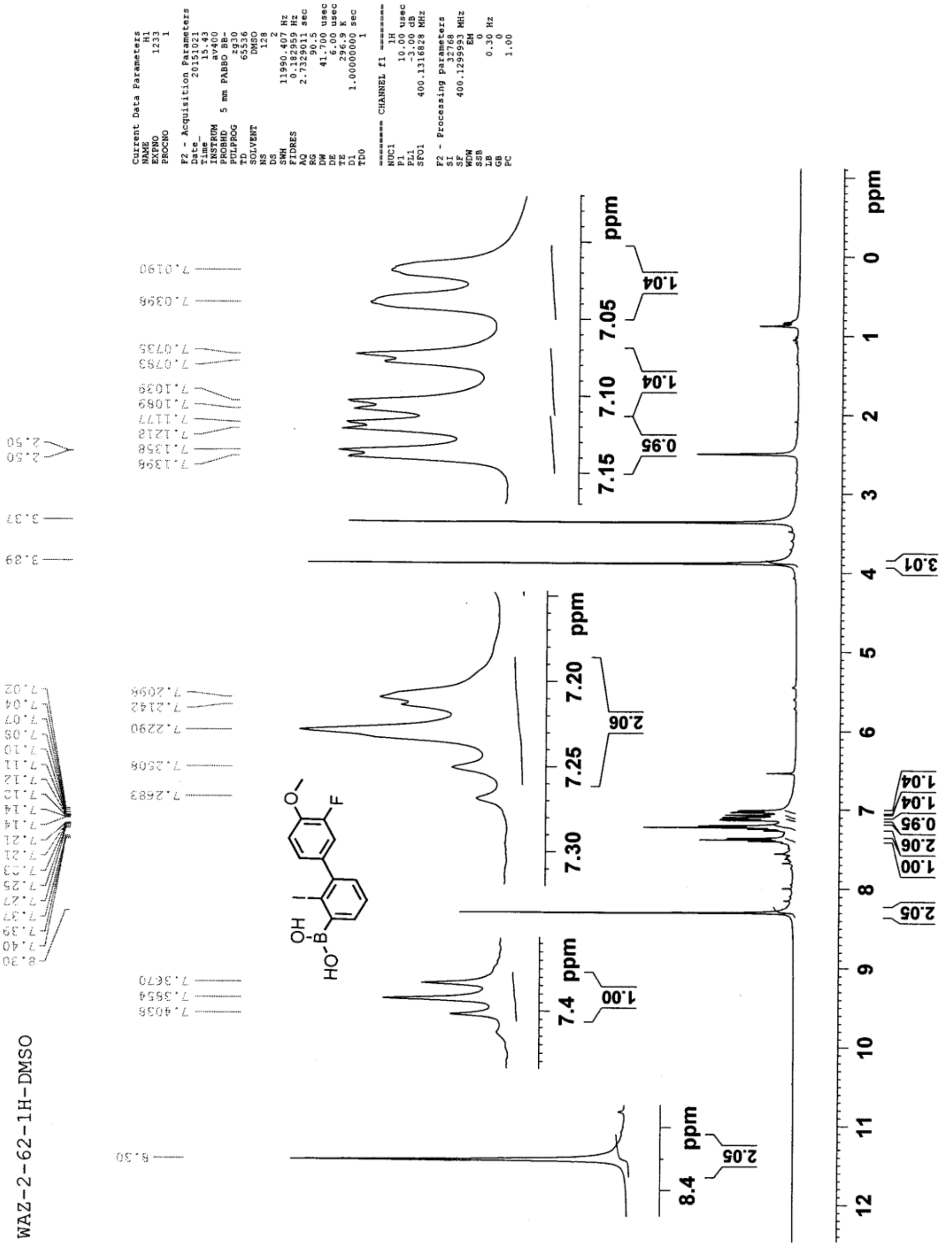
```
Current Data Parameters
NAME                 11B
EXPNO                 52
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160106
Time               13.28
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 65536
SOLVENT          Acetone
NS                   139
DS                     1
SWH            25641.025 Hz
FIDRES          0.391251 Hz
AQ             1.2780020 sec
RG                 812.7
DW                19.500 usec
DE                  6.00 usec
TE                 291.6 K
D1            1.00000000 sec
TD0                    1

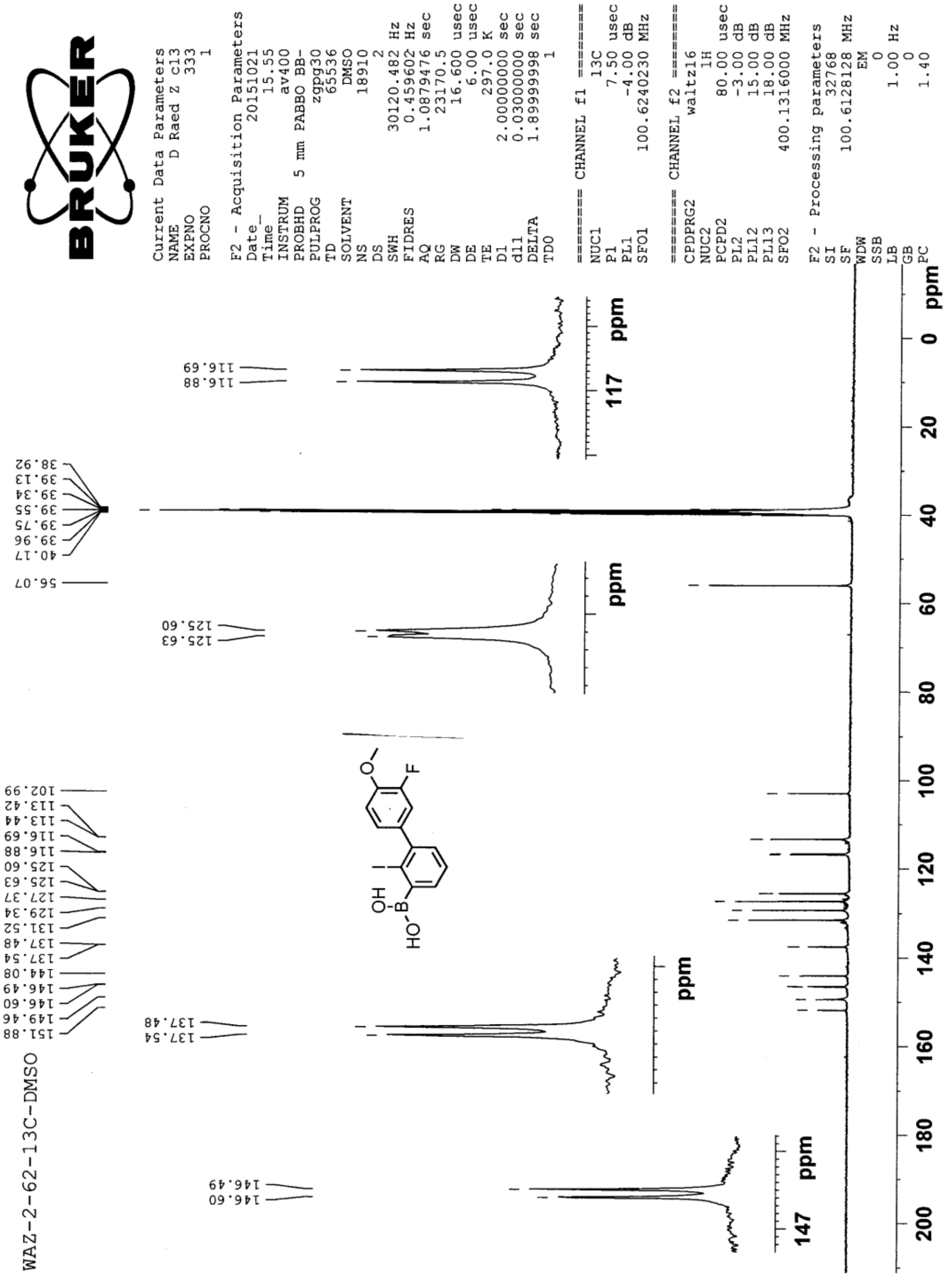
======== CHANNEL f1 ========
NUC1                 11B
P1                 15.00 usec
PL1                 8.00 dB
SFO1         128.3776052 MHz

F2 - Processing parameters
SI                 32768
SF           128.3776050 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00
```

1.5.33 ^{1}H-NMR of (3'-fluoro-2-iodo-4'-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (7$_A$) in d^6-DMSO at 25 ^{o}C.



1.5.34 ^{13}C–NMR of (3'-fluoro-2-iodo-4'-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (7$_A$) in d^{6-}DMSO at 25 ^{o}C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  333
PROCNO                   1

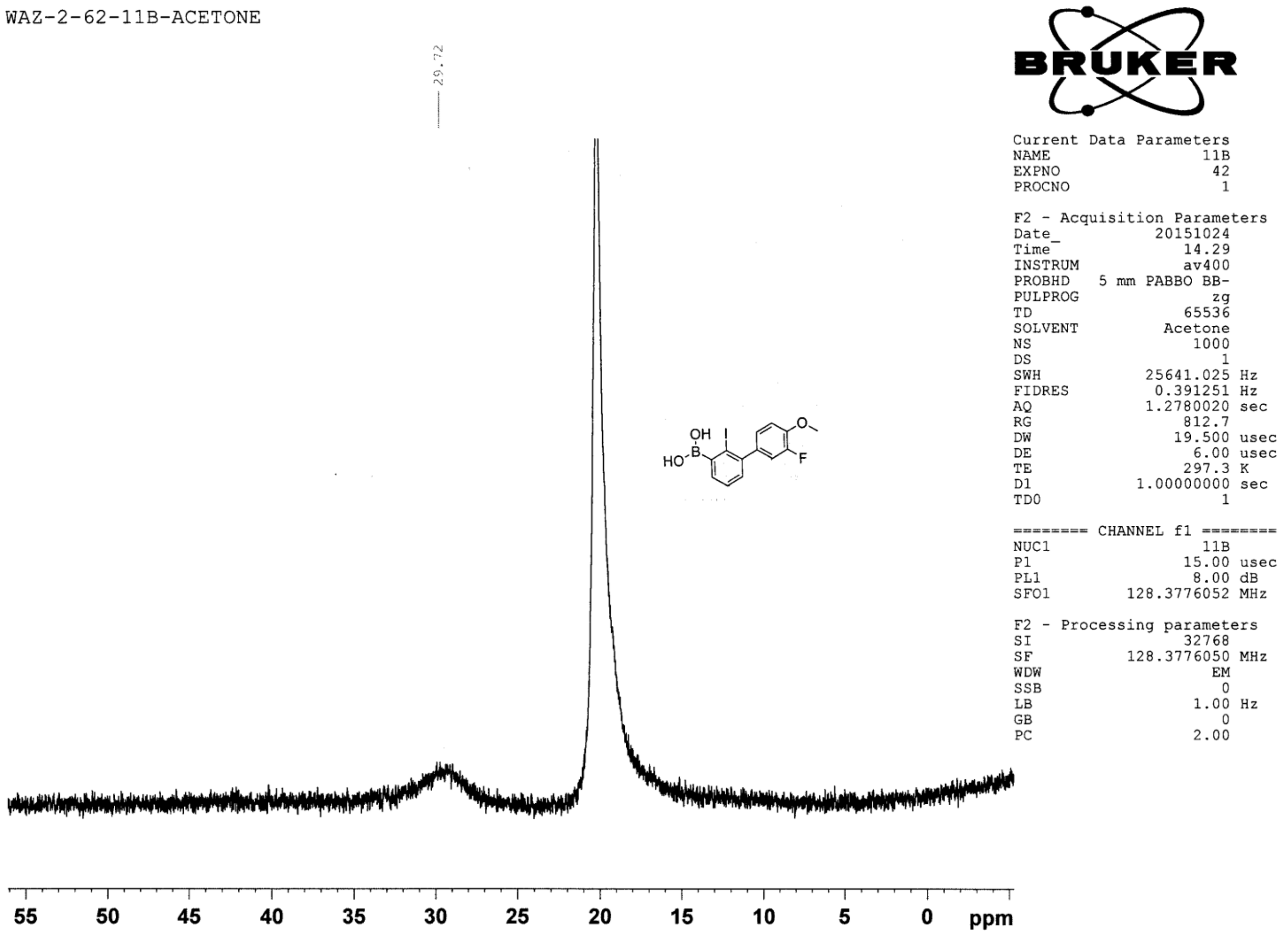
F2 - Acquisition Parameters
Date_             20151021
Time                 15.55
INSTRUM              av400
PROBHD      5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                   18910
DS                       2
SWH          30120.482 Hz
FIDRES        0.459602 Hz
AQ           1.0879476 sec
RG                 23170.5
DW              16.600 usec
DE                6.00 usec
TE               297.0 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                      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         100.6128128 MHz
WDW                     EM
SSB                      0
LB                1.00 Hz
GB                       0
PC                    1.40
```

1.5.35 ^{11}B-NMR of (3'-fluoro-2-iodo-4'-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (7_A) in d^6-acetone at 25 °C



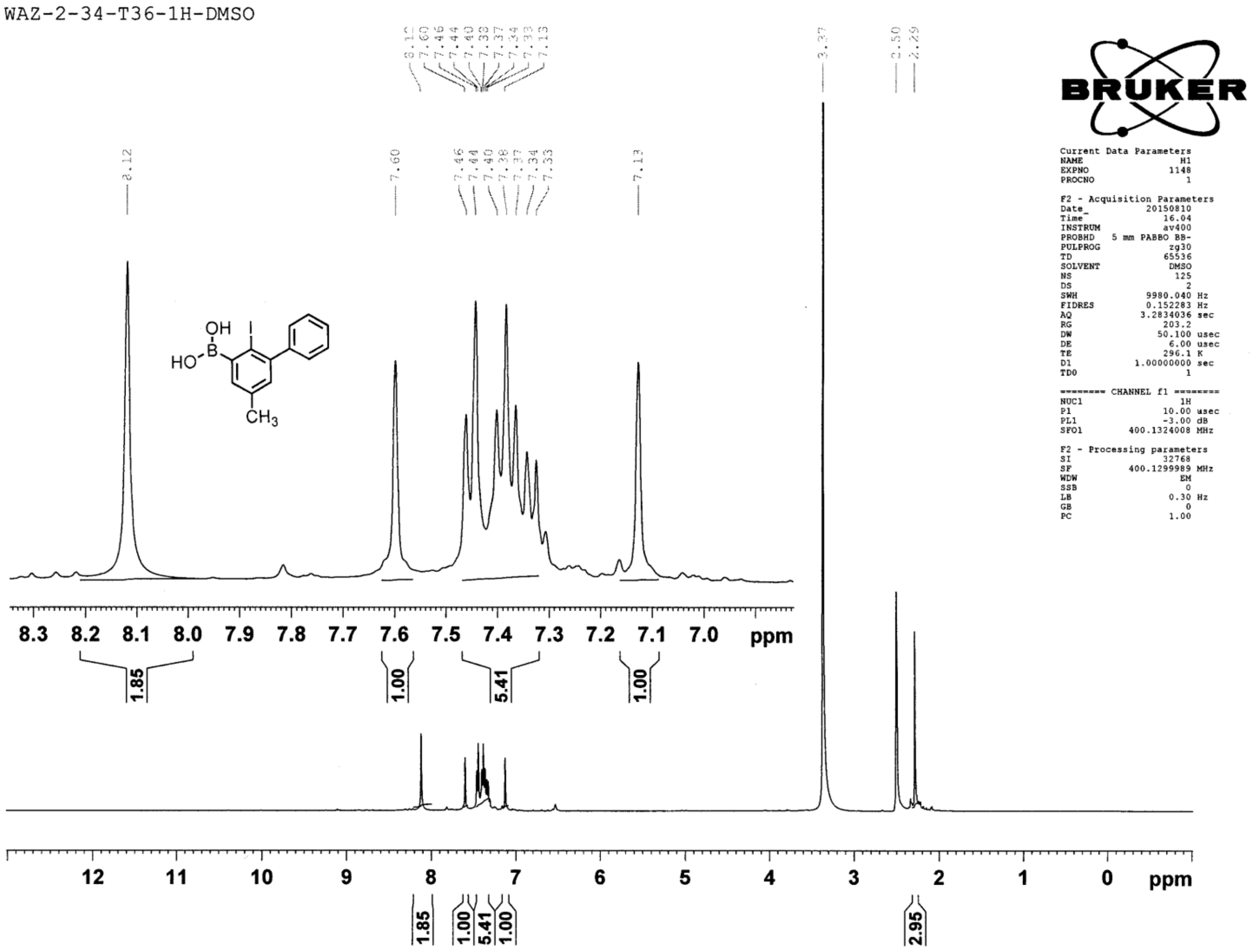
```
Current Data Parameters
NAME                 11B
EXPNO                 42
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151024
Time               14.29
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 65536
SOLVENT          Acetone
NS                  1000
DS                     1
SWH            25641.025 Hz
FIDRES          0.391251 Hz
AQ             1.2780020 sec
RG                 812.7
DW                19.500 usec
DE                  6.00 usec
TE                 297.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 11B
P1                 15.00 usec
PL1                 8.00 dB
SFO1         128.3776052 MHz

F2 - Processing parameters
SI                 32768
SF           128.3776050 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00
```

1.5.36 ^{1}H-NMR of (2-iodo-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (8$_A$) in d^6-DMSO at 25 °C.



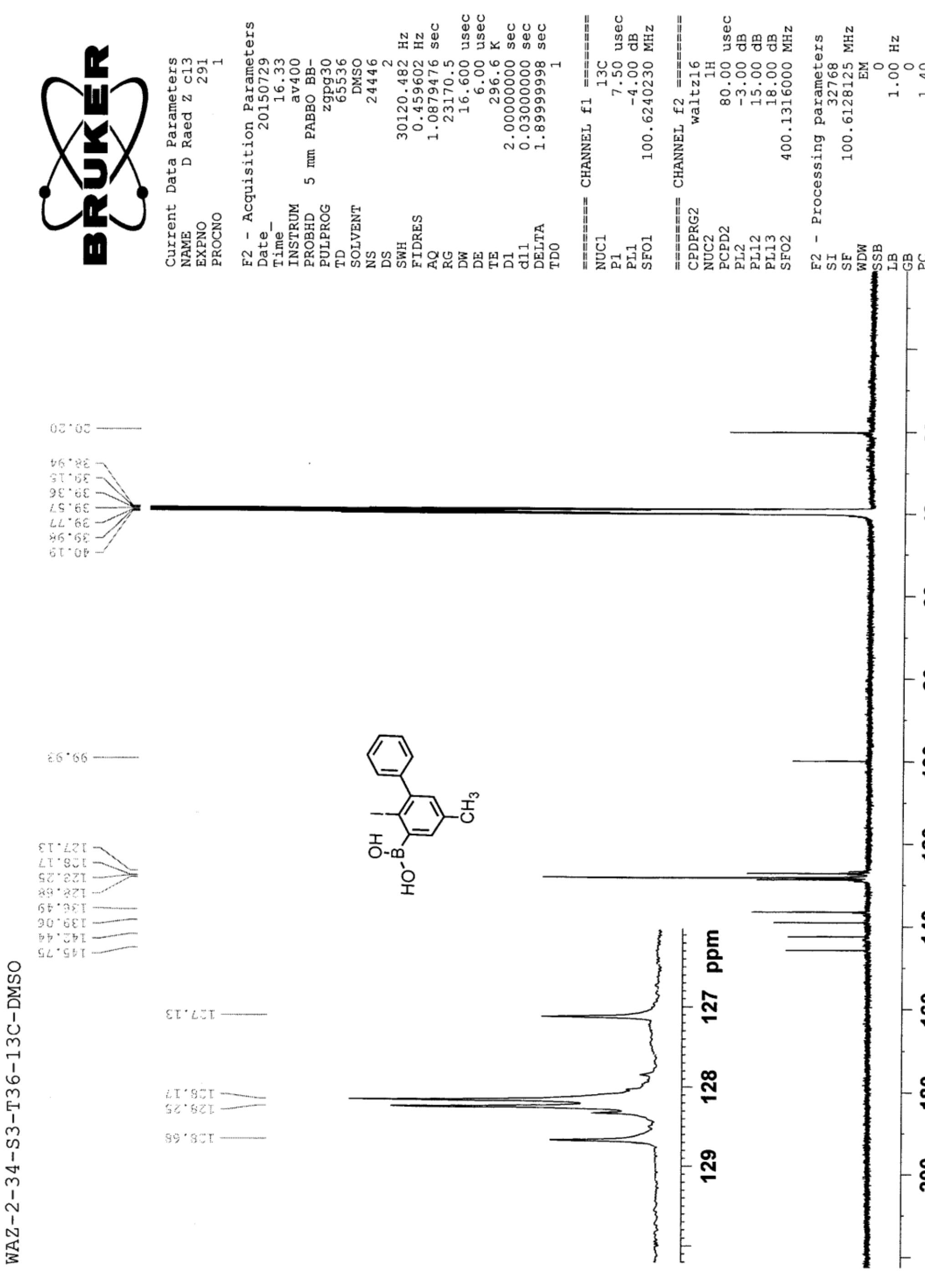
```
Current Data Parameters
NAME                  H1
EXPNO               1148
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150810
Time               16.04
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                   125
DS                     2
SWH             9980.040 Hz
FIDRES          0.152283 Hz
AQ             3.2834036 sec
RG                 203.2
DW                50.100 usec
DE                  6.00 usec
TE                 296.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -3.00 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                 32768
SF           400.1299989 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

1.5.37 ^{13}C–NMR of (2-iodo-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (8$_A$) in d^{6-} DMSO at 25 °C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  291
PROCNO                   1

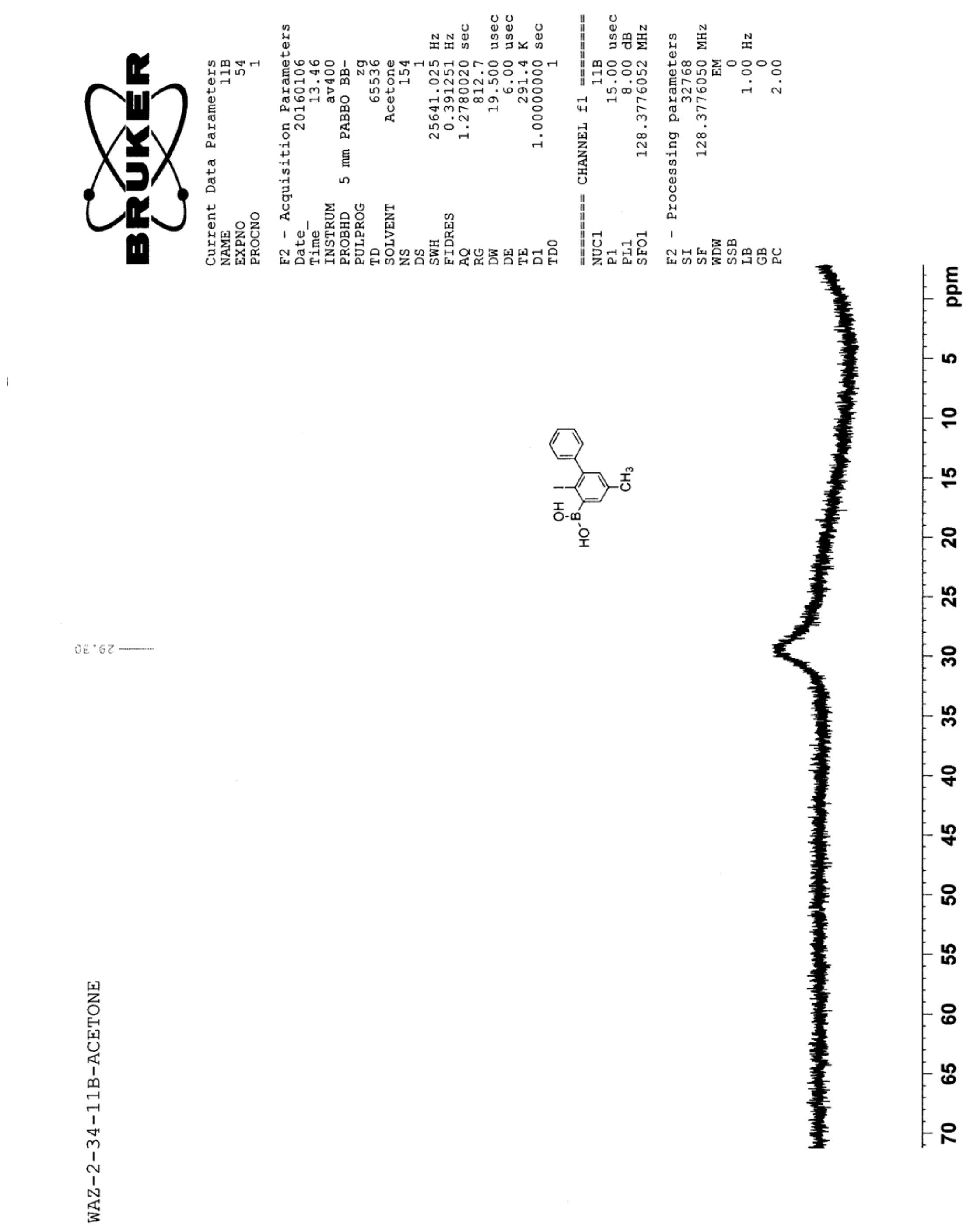
F2 - Acquisition Parameters
Date_             20150729
Time                 16.33
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               DMSO
NS                   24446
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   296.6 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      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             100.6128125 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

1.5.38 ^{11}B-NMR of (2-iodo-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (8_A) in d^6-acetone at 25 ^{o}C



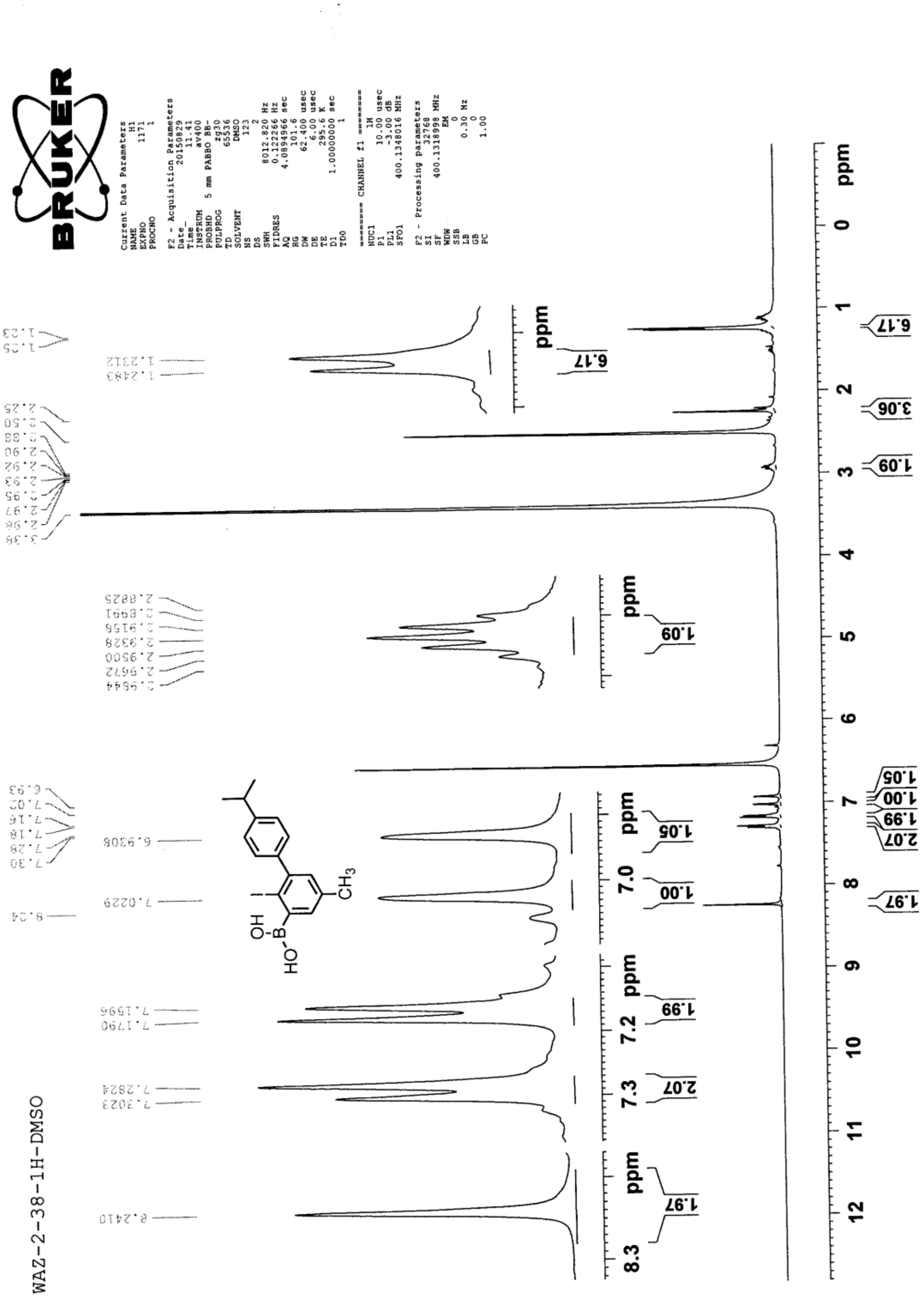
Current Data Parameters
NAME 11B
EXPNO 54
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160106
Time 13.46
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 65536
SOLVENT Acetone
NS 154
DS 1
SWH 25641.025 Hz
FIDRES 0.391251 Hz
AQ 1.2780020 sec
RG 812.7
DW 19.500 usec
DE 6.00 usec
TE 291.4 K
D1 1.00000000 sec
TD0 1

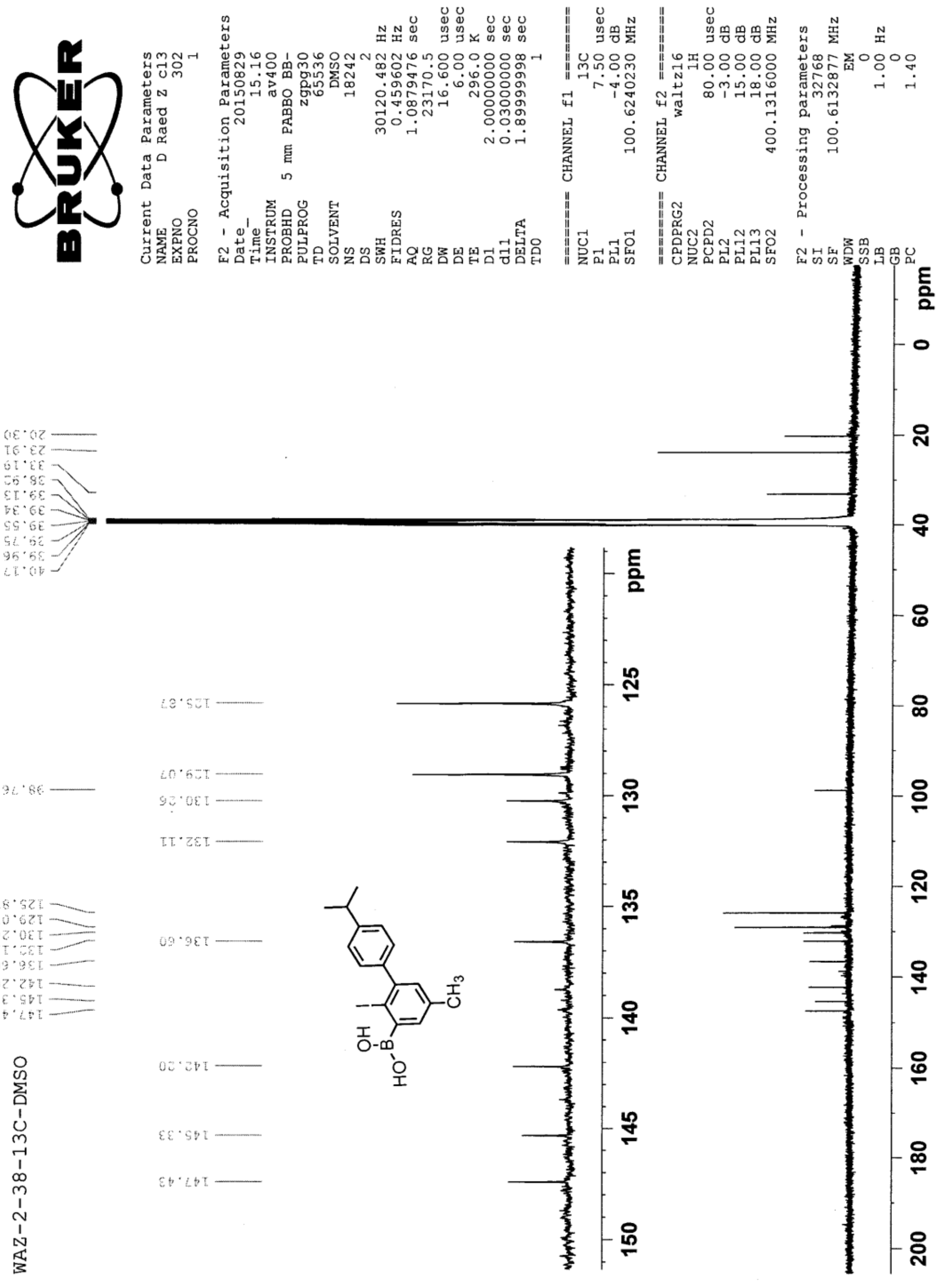
======== CHANNEL f1 ========
NUC1 11B
P1 15.00 usec
PL1 8.00 dB
SFO1 128.3776052 MHz

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1.5.39 ^{1}H-NMR of (2-iodo-4'-isopropyl-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (9_A) in d^{6}-DMSO at 25 °C.



1.5.40 ^{13}C–NMR of (2-iodo-4'-isopropyl-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (9_A) in d^6-DMSO at 25 °C.



```
Current Data Parameters
NAME             D Raed Z c13
EXPNO                     302
PROCNO                      1

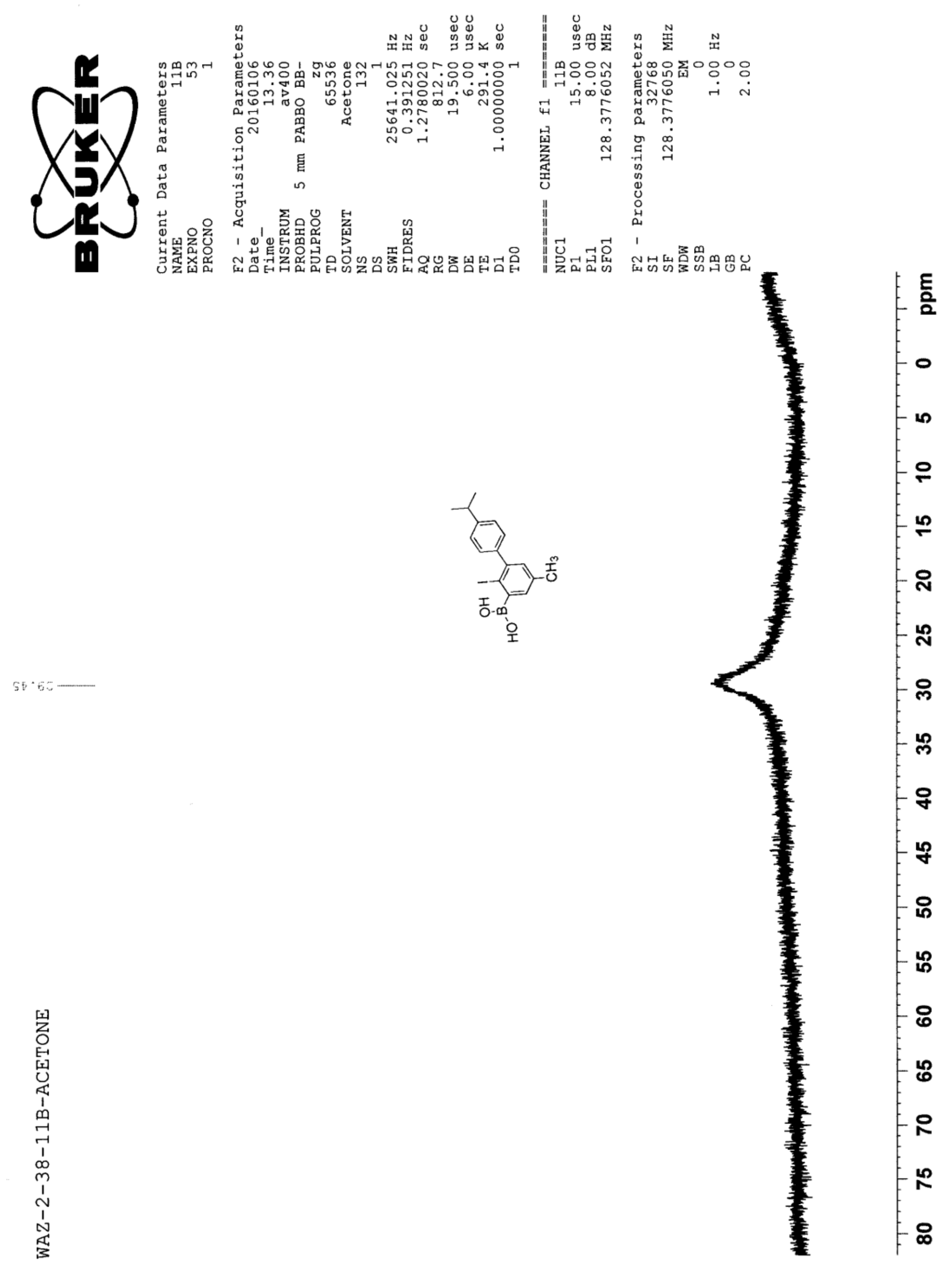
F2 - Acquisition Parameters
Date_                20150829
Time                    15.16
INSTRUM                 av400
PROBHD       5 mm PABBO BB-
PULPROG               zgpg30
TD                      65536
SOLVENT                  DMSO
NS                      18242
DS                          2
SWH               30120.482 Hz
FIDRES             0.459602 Hz
AQ                1.0879476 sec
RG                    23170.5
DW                     16.600 usec
DE                       6.00 usec
TE                      296.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                         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               100.6132877 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

1.5.41 ^{11}B-NMR of (2-iodo-4'-isopropyl-5-methyl-[1,1'-biphenyl]-3-yl)boronic acid (9_A) in d^6-acetone at 25 ^{o}C



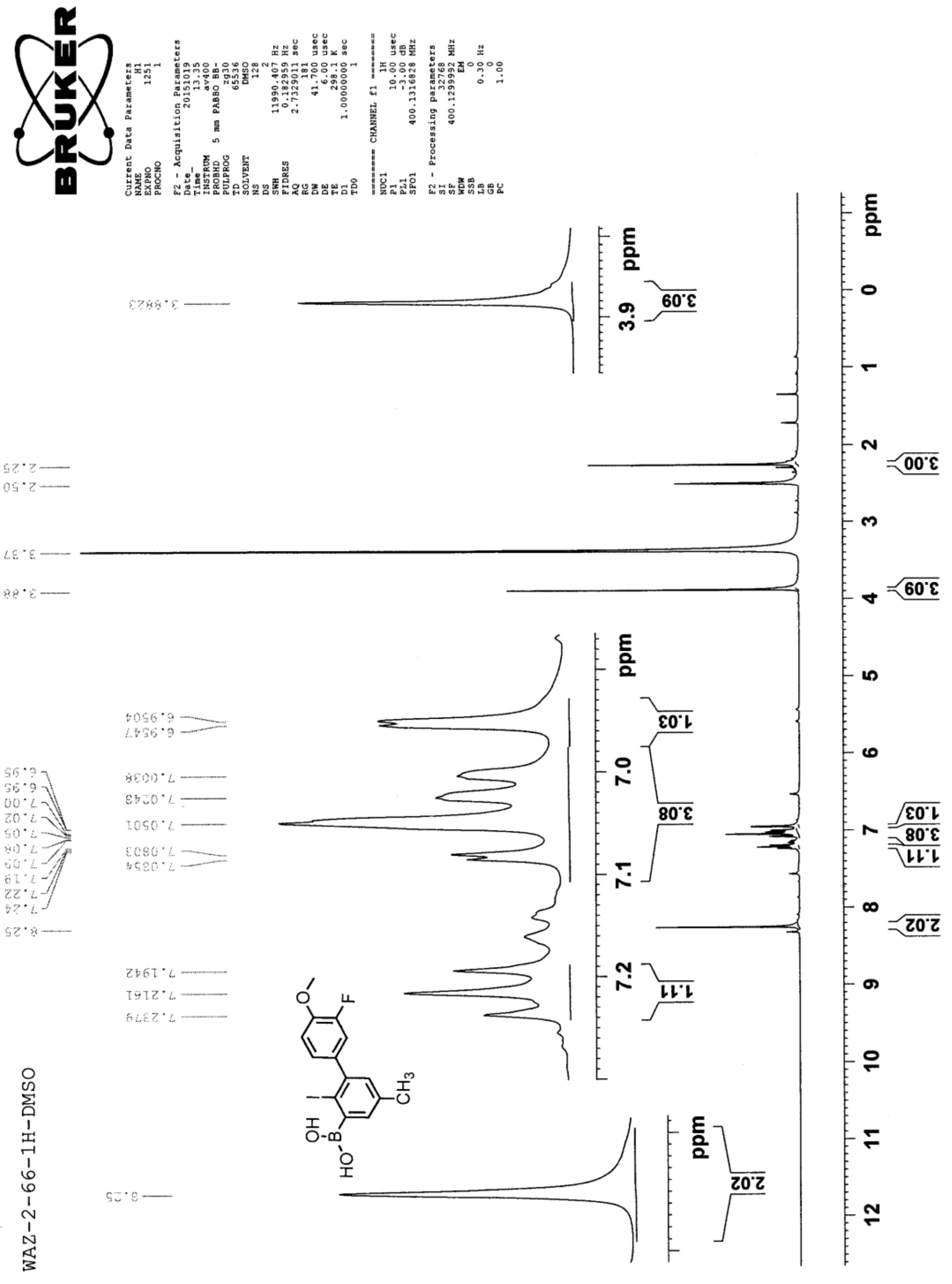
```
Current Data Parameters
NAME               11B
EXPNO               53
PROCNO               1

F2 - Acquisition Parameters
Date_         20160106
Time_            13.36
INSTRUM          av400
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD               65536
SOLVENT        Acetone
NS                 132
DS                   1
SWH          25641.025 Hz
FIDRES        0.391251 Hz
AQ           1.2780020 sec
RG               812.7
DW              19.500 usec
DE                6.00 usec
TE               291.4 K
D1          1.00000000 sec
TD0                  1

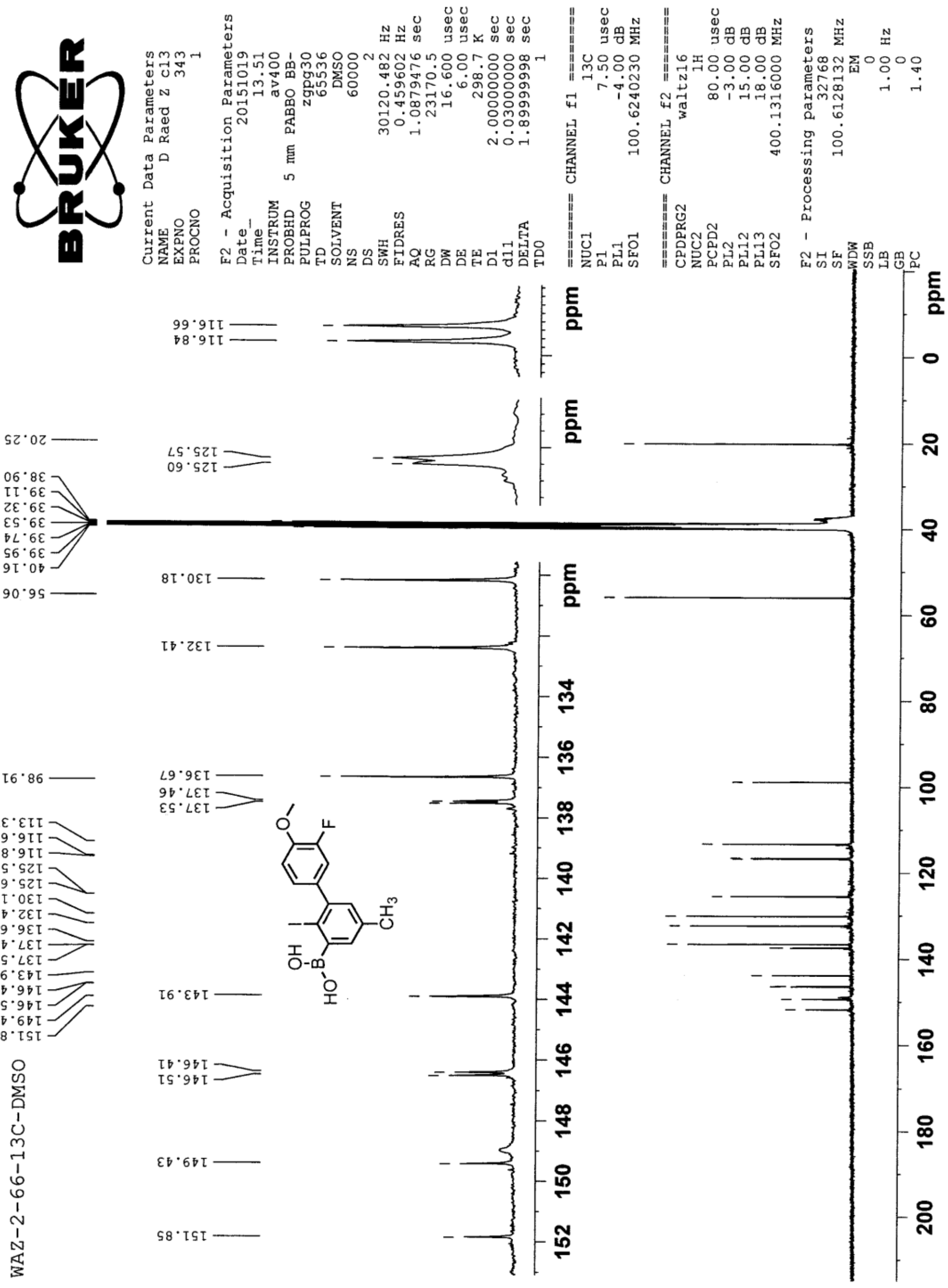
======== CHANNEL f1 ========
NUC1               11B
P1               15.00 usec
PL1               8.00 dB
SFO1       128.3776052 MHz

F2 - Processing parameters
SI               32768
SF         128.3776050 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                2.00
```

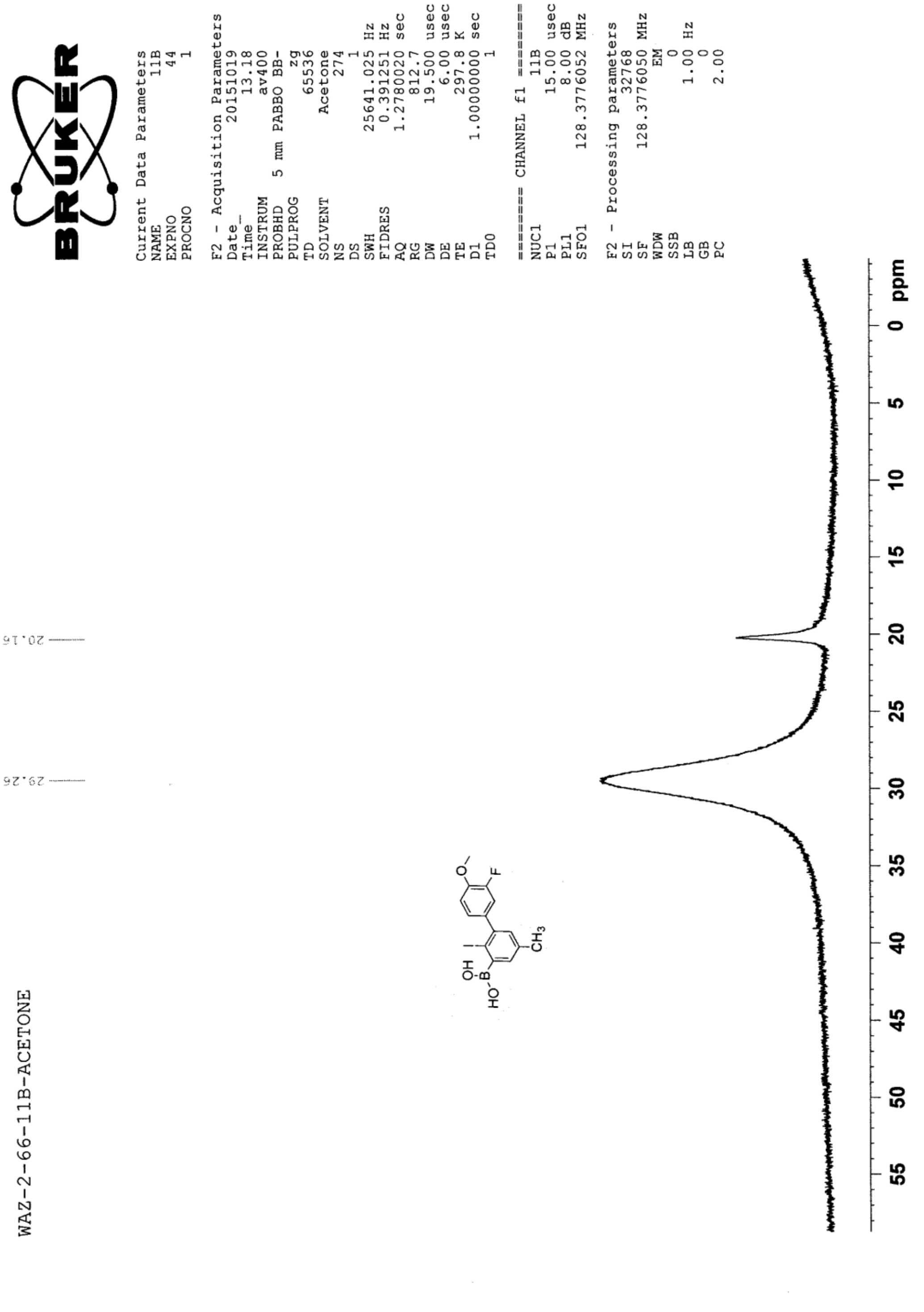
1.5.42 ^{1}H-NMR of (3'-fluoro-2-iodo-4'-methoxy-5-methyl-[1,1'-biphenyl]-3-yl) boronic acid (10_A) in d^6-DMSO at 25 ^{o}C.



1.5.43 ^{13}C–NMR of (3'-fluoro-2-iodo-4'-methoxy-5-methyl-[1,1'-biphenyl]-3-yl) boronic acid (10$_A$) in d^6-DMSO at 25 °C.



1.5.44 ^{11}B-NMR of (3'-fluoro-2-iodo-4'-methoxy-5-methyl-[1,1'-biphenyl]-3-yl) boronic acid (10$_A$) in d^6-acetone at 25 °C



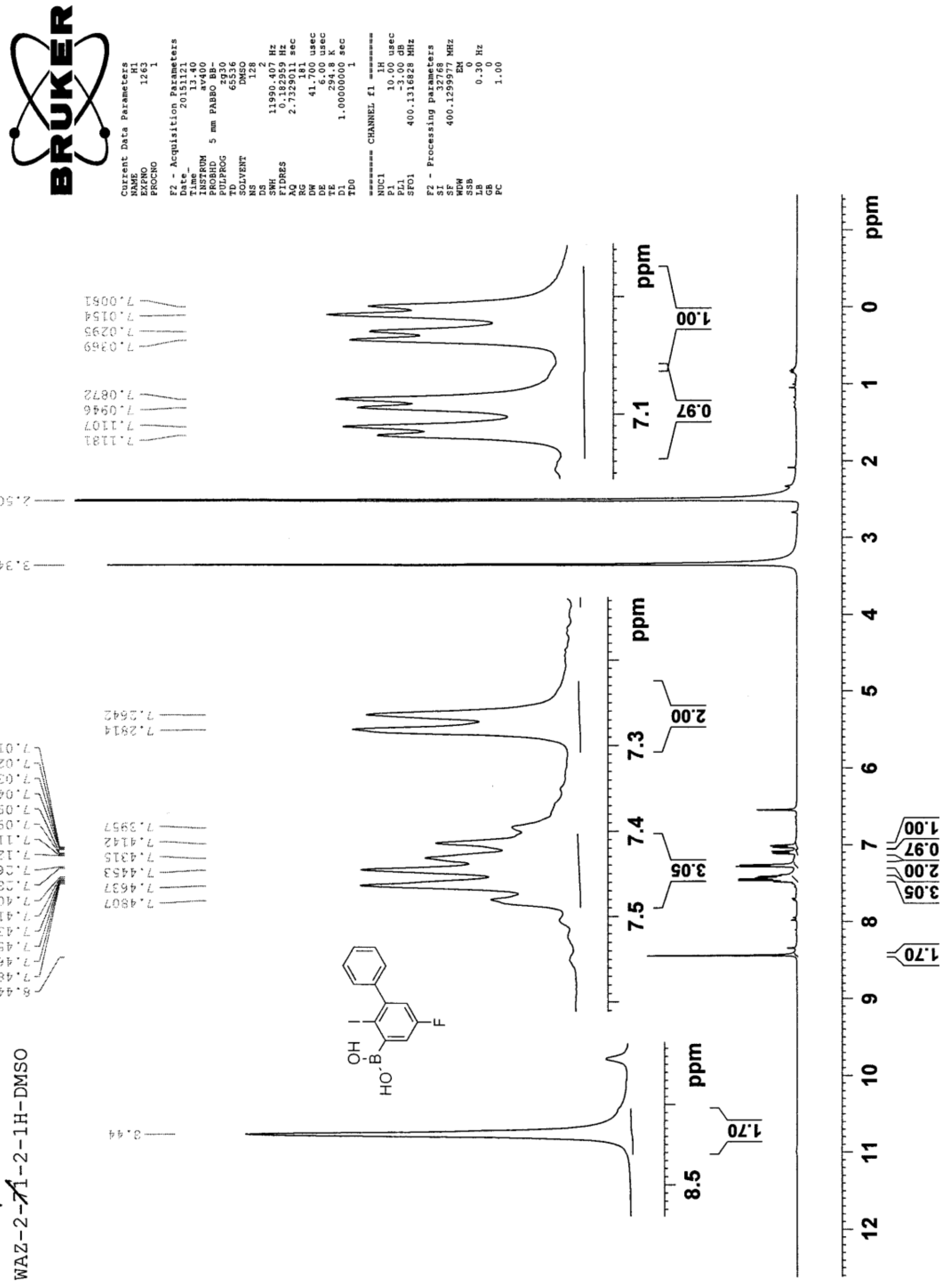
```
Current Data Parameters
NAME                 11B
EXPNO                 44
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151019
Time               13.18
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 65536
SOLVENT          Acetone
NS                   274
DS                     1
SWH            25641.025 Hz
FIDRES          0.391251 Hz
AQ             1.2780020 sec
RG                 812.7
DW                19.500 usec
DE                  6.00 usec
TE                 297.8 K
D1            1.00000000 sec
TD0                    1

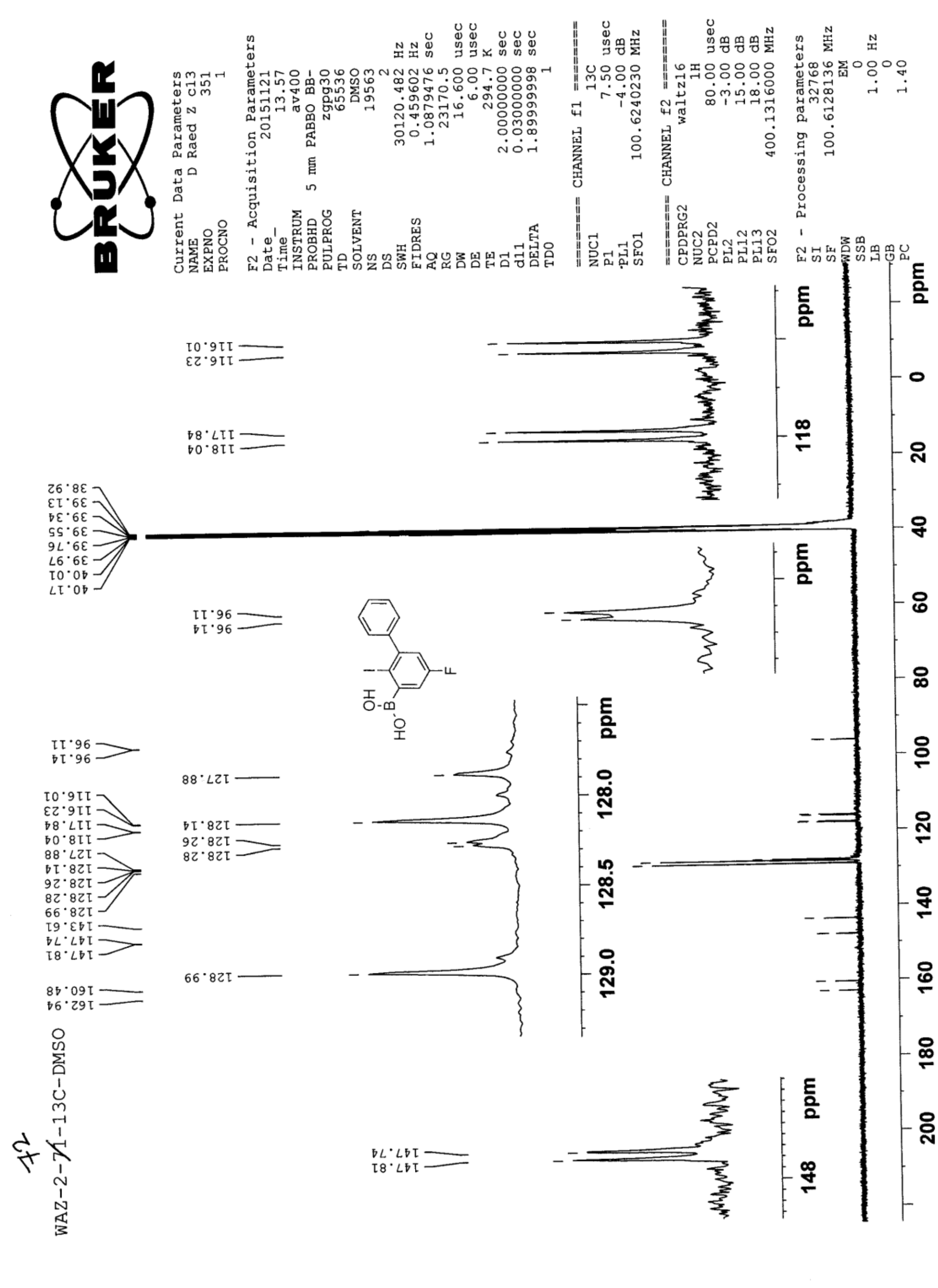
======== CHANNEL f1 ========
NUC1                 11B
P1                 15.00 usec
PL1                 8.00 dB
SFO1         128.3776052 MHz

F2 - Processing parameters
SI                 32768
SF           128.3776050 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00
```

1.5.45 ¹H-NMR of (5-fluoro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (11_A) in d^6-DMSO at 25 °C.



1.5.46 ^{13}C–NMR of (5-fluoro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (11$_A$) in d^{6-}DMSO at 25 ^{o}C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  351
PROCNO                   1

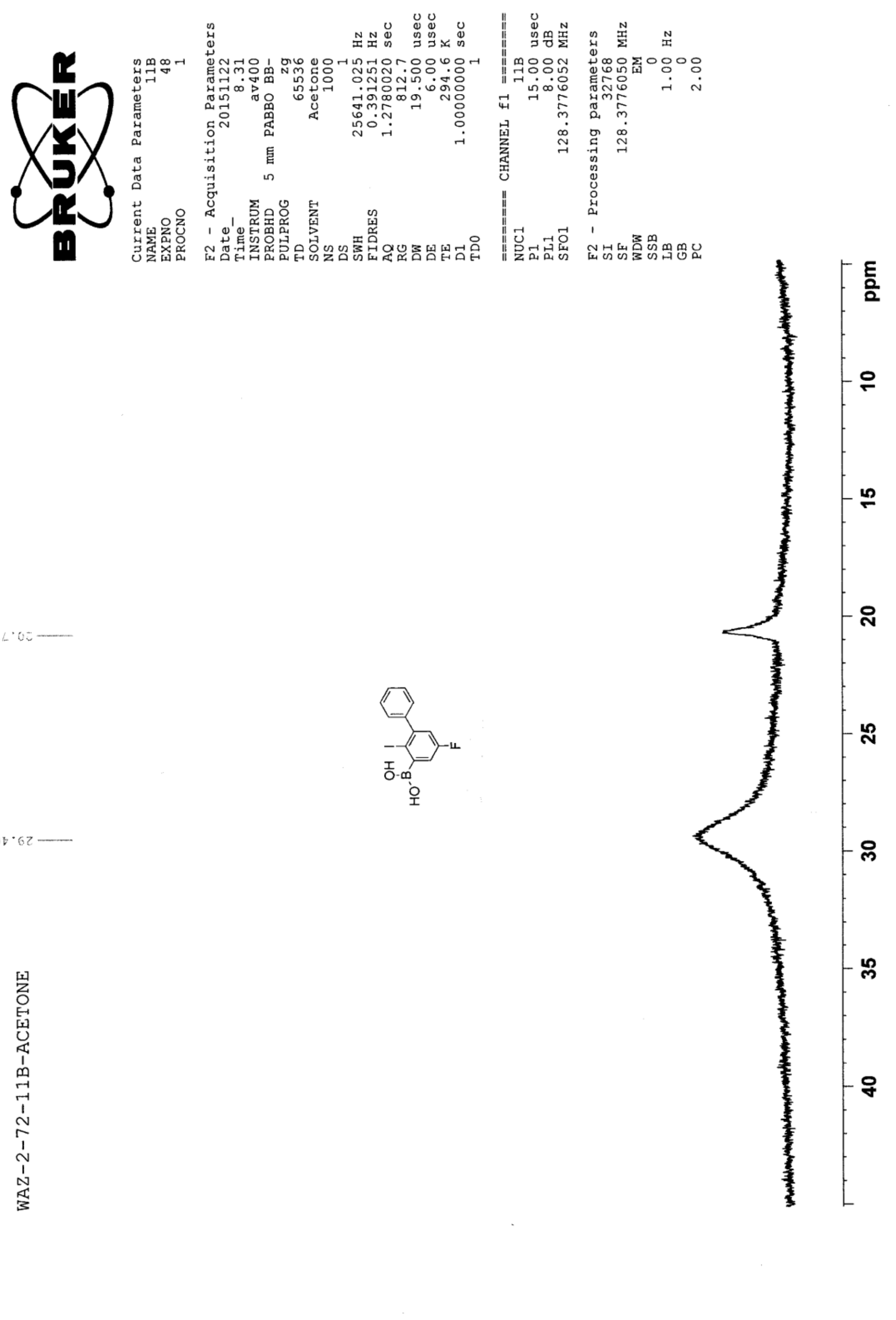
F2 - Acquisition Parameters
Date_             20151121
Time_                13.57
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               DMSO
NS                   19563
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   294.7 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

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

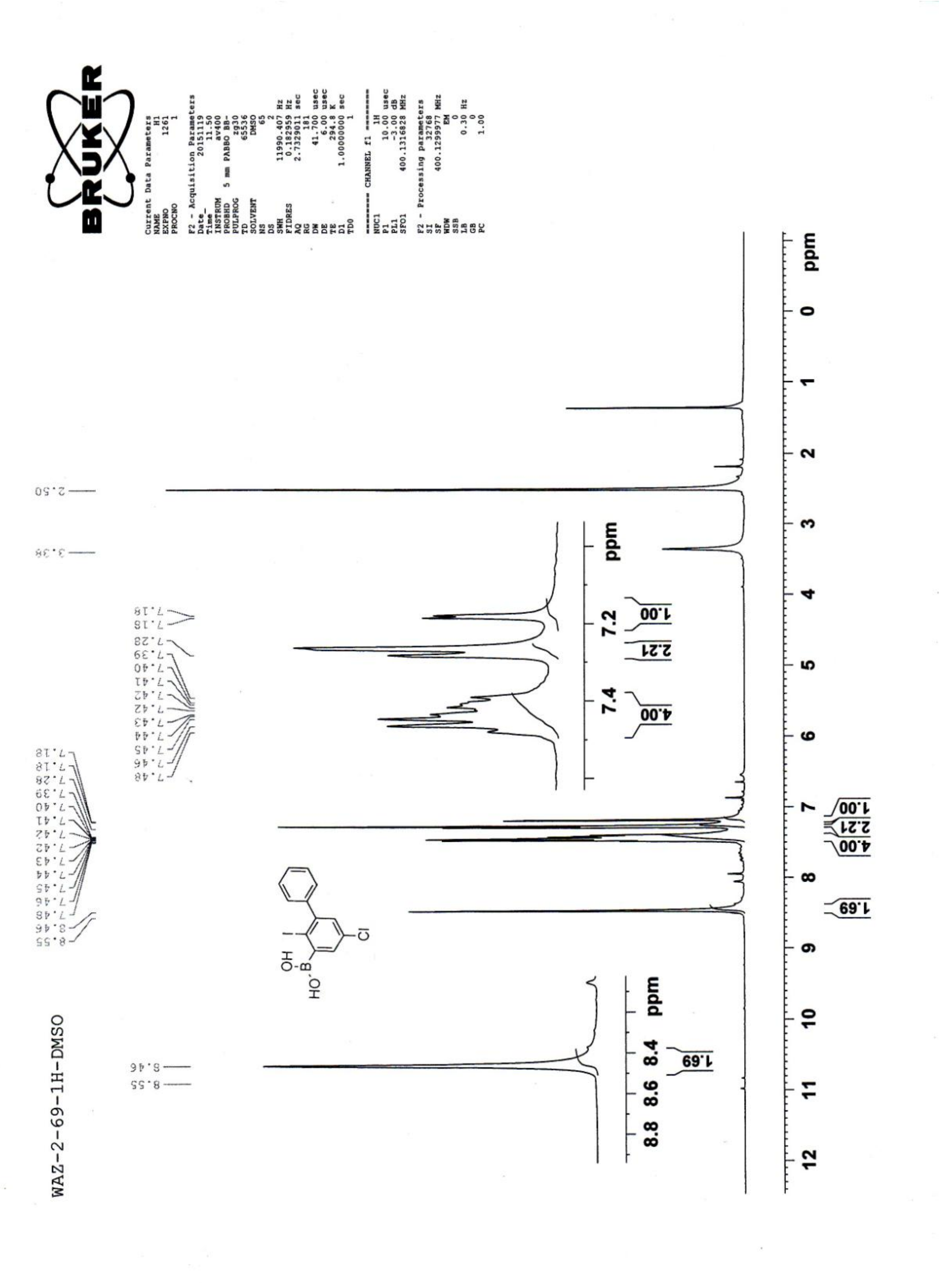
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 15.00 dB
PL13                 18.00 dB
SFO2         400.1316000 MHz

F2 - Processing parameters
SI                   32768
SF           100.6128136 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

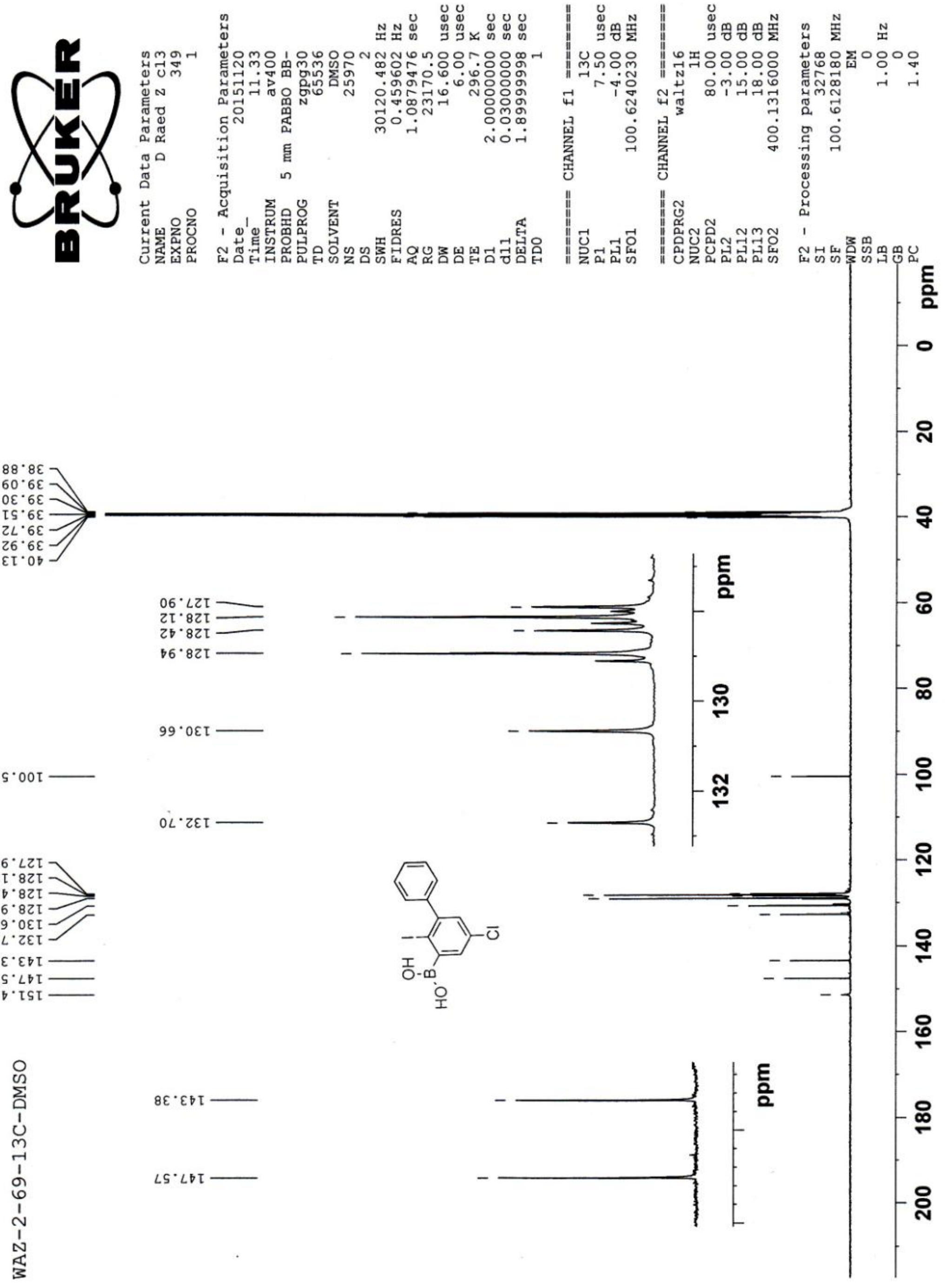
1.5.47 ^{11}B-NMR of (5-fluoro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (11$_A$) in d^6-acetone at 25 °C



1.5.48 ^{1}H-NMR of (5-chloro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (12_A) in d^6-DMSO at 25 ^{o}C.


WAZ-2-69-1H-DMSO
Current Data Parameters
NAME H1
EXPNO 1261
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151119
Time 11.50
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 65
DS 2
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 181
DW 41.700 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -3.00 dB
SFO1 400.1316828 MHz
F2 - Processing parameters
SI 32768
SF 400.1299977 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.50
3.38
7.18
7.18
7.28
7.39
7.40
7.41
7.42
7.42
7.43
7.44
7.45
7.46
7.48
8.46
8.55
OH
HO
B
I
Cl
1.69
4.00
2.21
1.00
ppm
12
11
10
9
8
7
6
5
4
3
2
1
0
8.8
8.6
8.4
7.4
7.2

1.5.49 ^{13}C–NMR of (5-chloro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (12_A) in d^{6}-DMSO at 25 ^{o}C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  349
PROCNO                   1

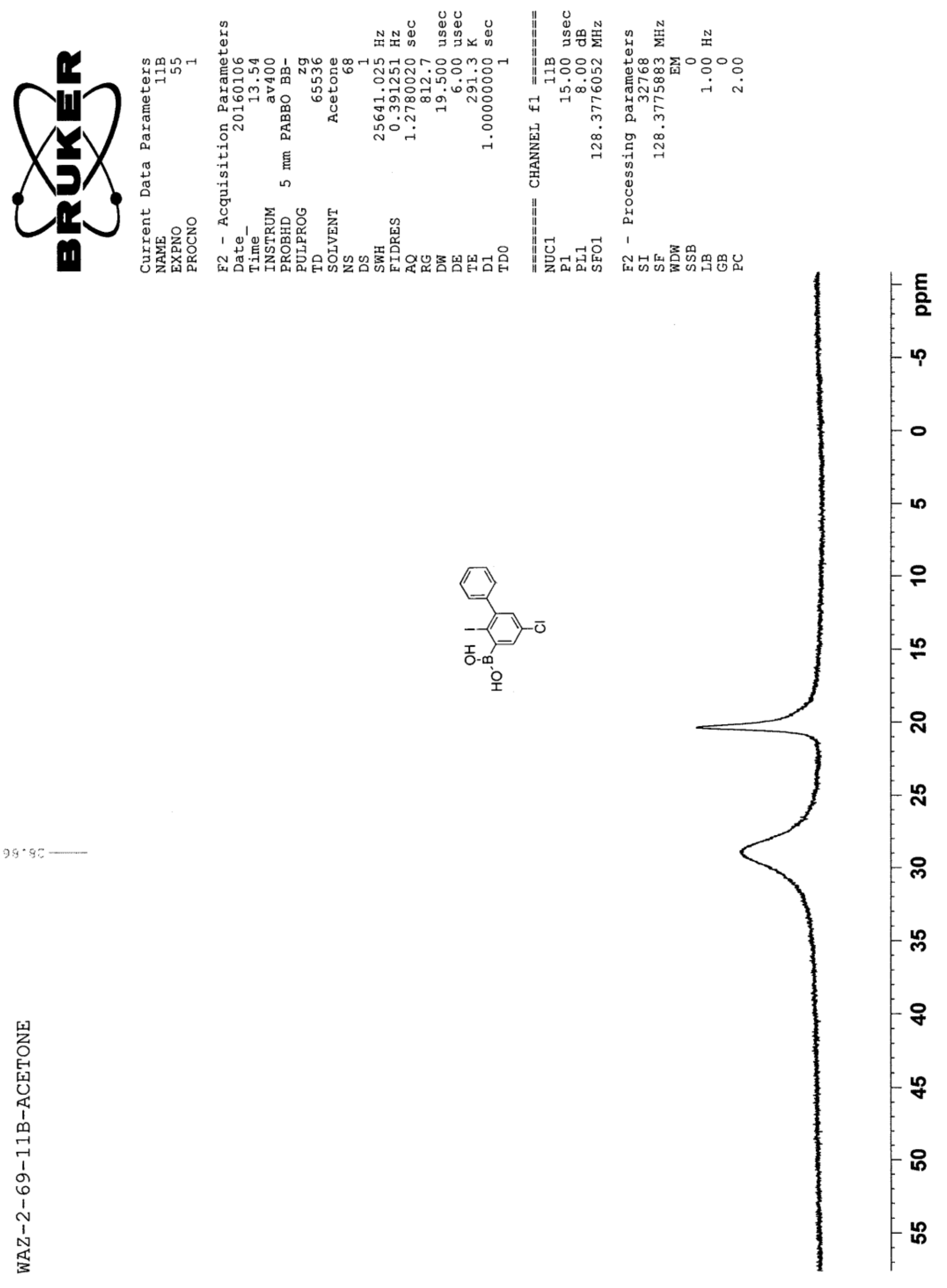
F2 - Acquisition Parameters
Date_             20151120
Time_                11.33
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                  25970
DS                      2
SWH             30120.482 Hz
FIDRES           0.459602 Hz
AQ              1.0879476 sec
RG                23170.5
DW                 16.600 usec
DE                   6.00 usec
TE                  296.7 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     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            100.6128180 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

1.5.50 ^{11}B-NMR of (5-chloro-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (12_A) in d^6-acetone at 25 °C



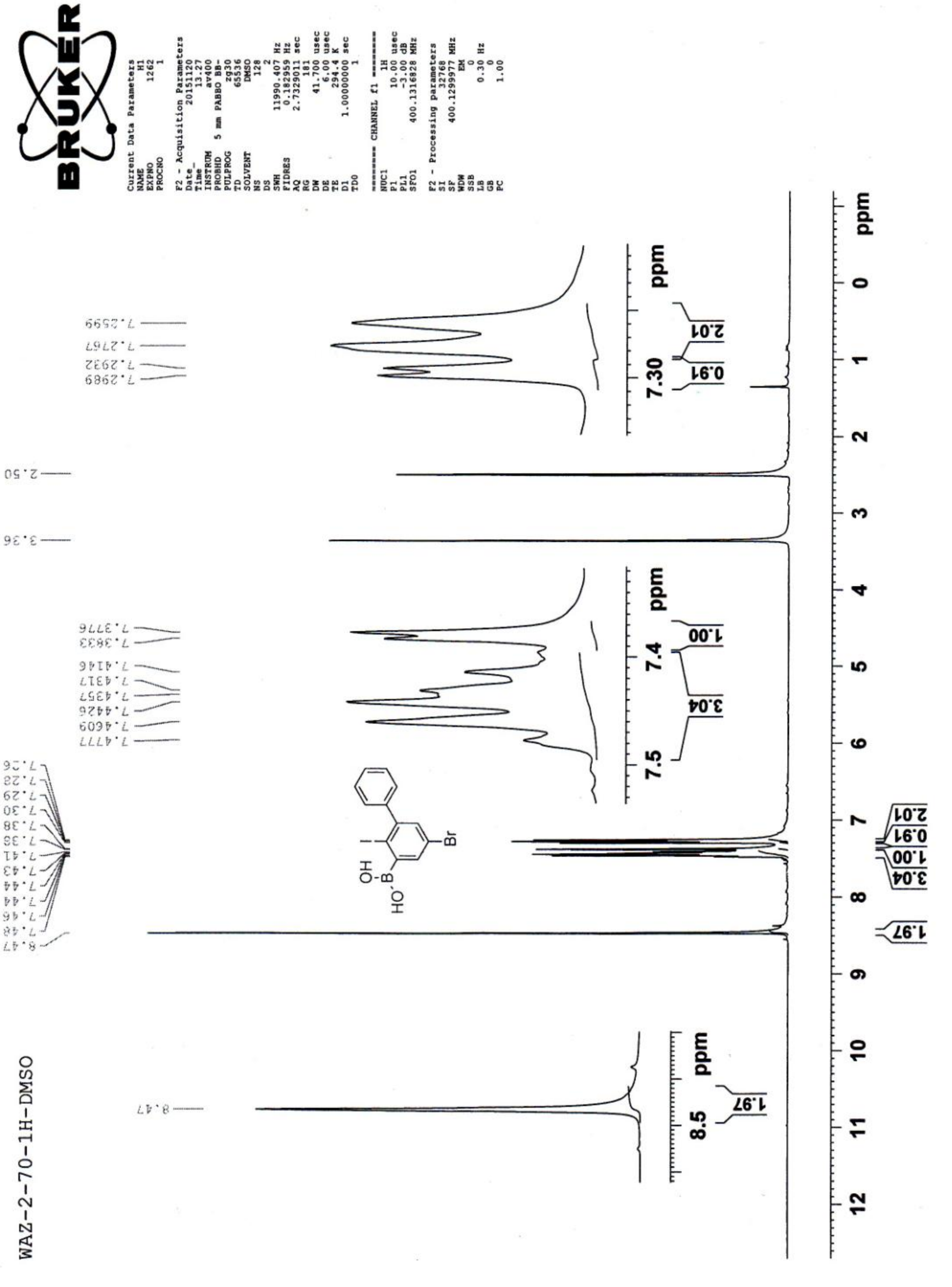
```
Current Data Parameters
NAME              11B
EXPNO              55
PROCNO              1

F2 - Acquisition Parameters
Date_        20160106
Time            13.54
INSTRUM         av400
PROBHD   5 mm PABBO BB-
PULPROG            zg
TD              65536
SOLVENT       Acetone
NS                 68
DS                  1
SWH         25641.025 Hz
FIDRES       0.391251 Hz
AQ          1.2780020 sec
RG              812.7
DW             19.500 usec
DE               6.00 usec
TE              291.3 K
D1         1.00000000 sec
TD0                 1

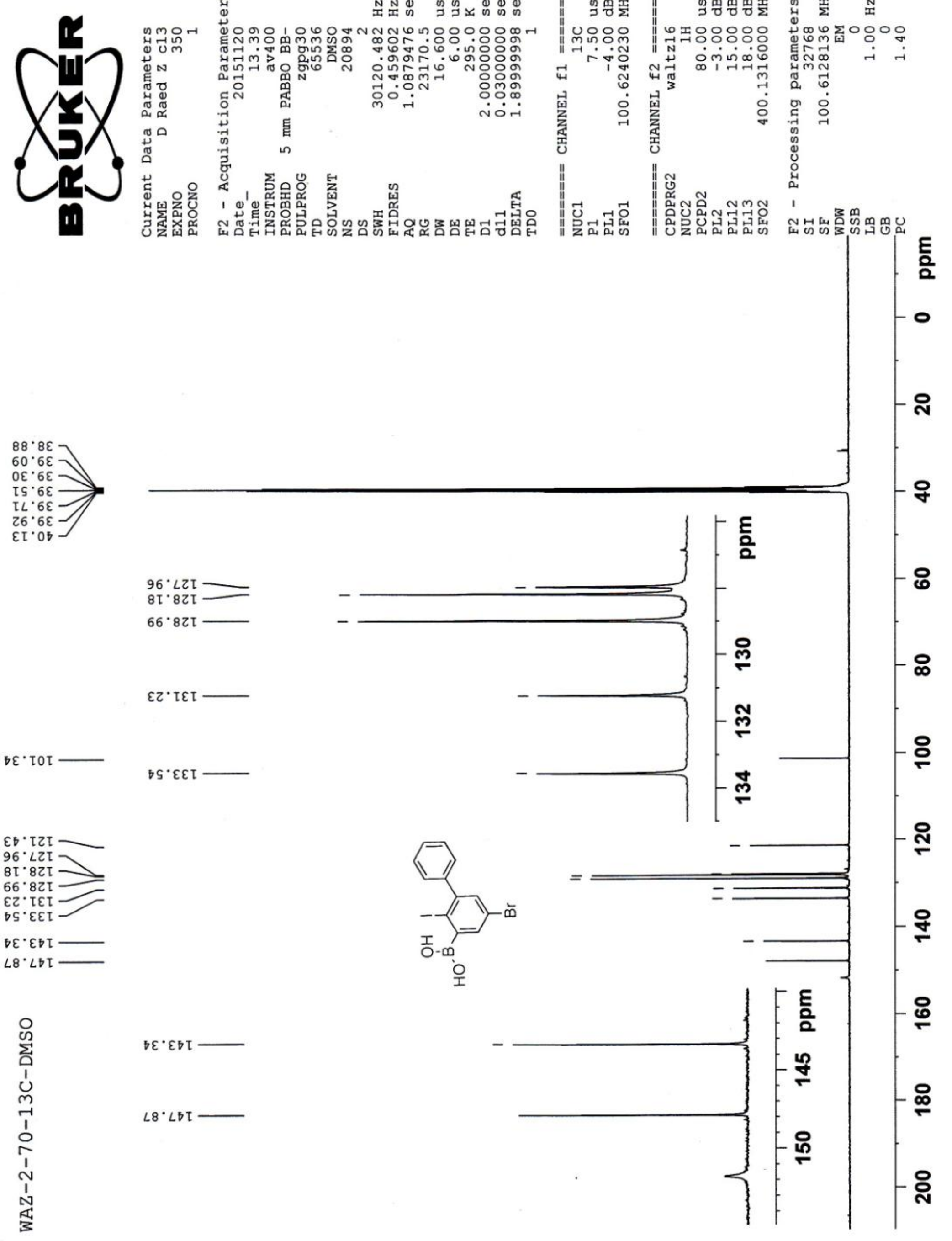
======== CHANNEL f1 ========
NUC1              11B
P1              15.00 usec
PL1              8.00 dB
SFO1     128.3776052 MHz

F2 - Processing parameters
SI              32768
SF        128.3775883 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               2.00
```

1.5.51 ^{1}H-NMR of (5-bromo-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (13_A) in d^{6-}DMSO at 25 ^{o}C.



1.5.52 ^{13}C–NMR of (5-bromo-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (13_A) in d^6-DMSO at 25 °C.



```
Current Data Parameters
NAME           D Raed Z c13
EXPNO                   350
PROCNO                    1

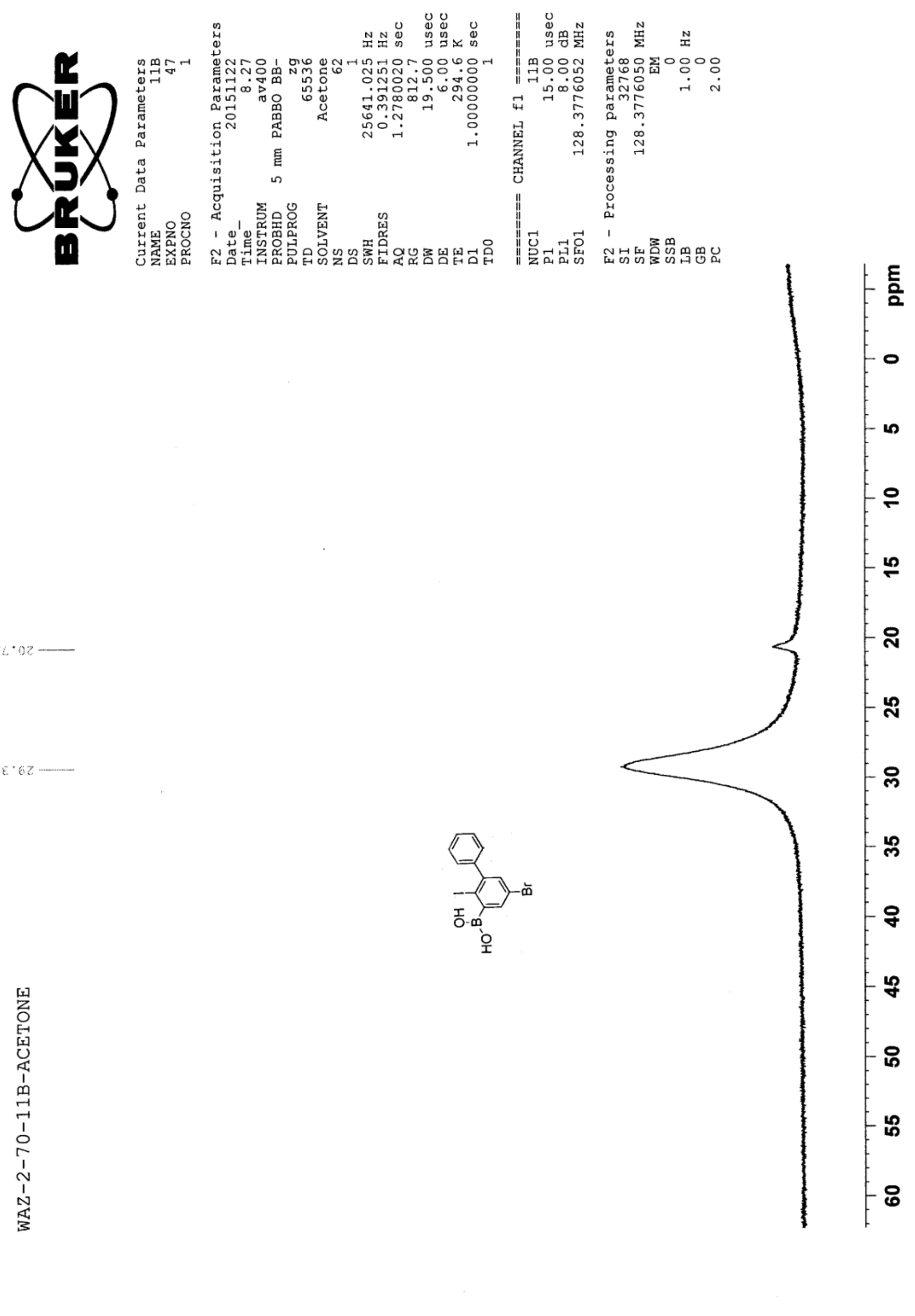
F2 - Acquisition Parameters
Date_              20151120
Time_                 13.39
INSTRUM               av400
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT                DMSO
NS                    20894
DS                        2
SWH               30120.482 Hz
FIDRES             0.459602 Hz
AQ                1.0879476 sec
RG                  23170.5
DW                   16.600 usec
DE                     6.00 usec
TE                    295.0 K
D1               2.00000000 sec
d11              0.03000000 sec
DELTA            1.89999998 sec
TD0                       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              100.6128136 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

1.5.53 ^{11}B-NMR of (5-bromo-2-iodo-[1,1'-biphenyl]-3-yl)boronic acid (13$_A$) in d^6-acetone at 25 ^{o}C



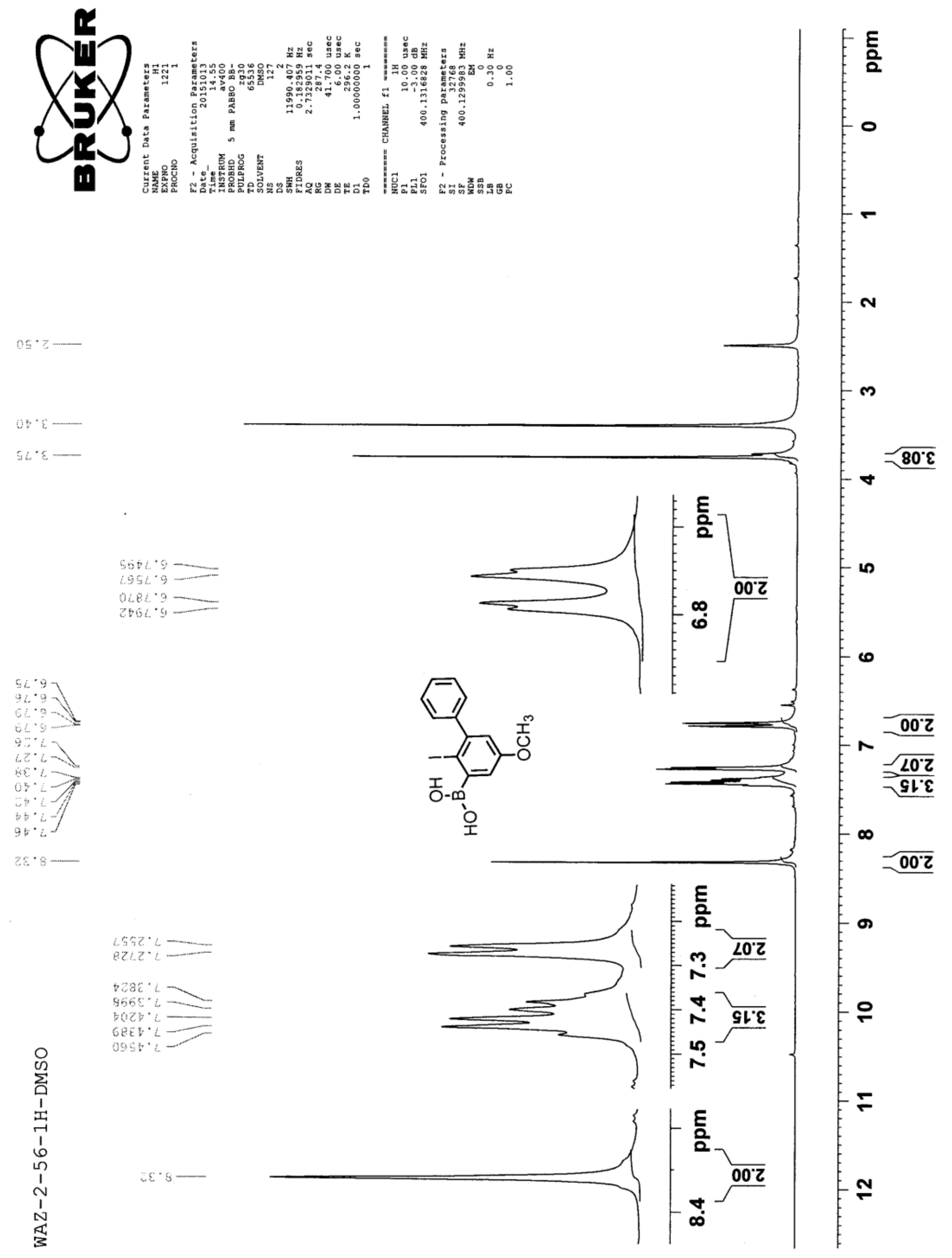
```
Current Data Parameters
NAME                11B
EXPNO                47
PROCNO                1

F2 - Acquisition Parameters
Date_          20151122
Time               8.27
INSTRUM           av400
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                65536
SOLVENT         Acetone
NS                   62
DS                    1
SWH           25641.025 Hz
FIDRES         0.391251 Hz
AQ            1.2780020 sec
RG                812.7
DW               19.500 usec
DE                 6.00 usec
TE                294.6 K
D1           1.00000000 sec
TD0                   1

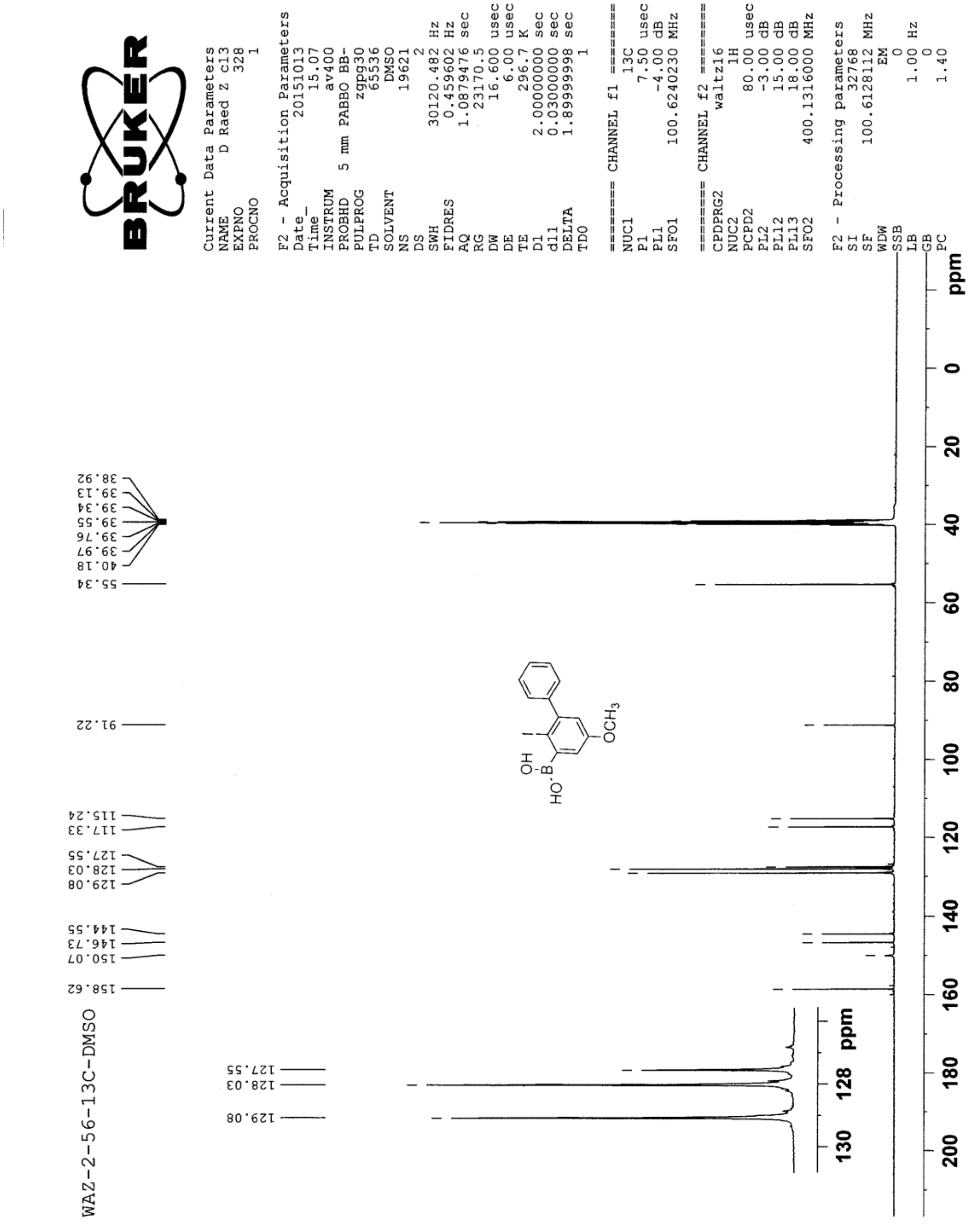
======== CHANNEL f1 ========
NUC1                11B
P1                15.00 usec
PL1                8.00 dB
SFO1        128.3776052 MHz

F2 - Processing parameters
SI                32768
SF          128.3776050 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00
```

1.5.54 ^{1}H-NMR of (2-iodo-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (14$_A$) in d^{6}-DMSO at 25 ^{o}C.



1.5.55 ^{13}C–NMR of (2-iodo-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (14$_A$) in d^{6}-DMSO at 25 °C.



```
Current Data Parameters
NAME       D Raed Z c13
EXPNO               328
PROCNO                1

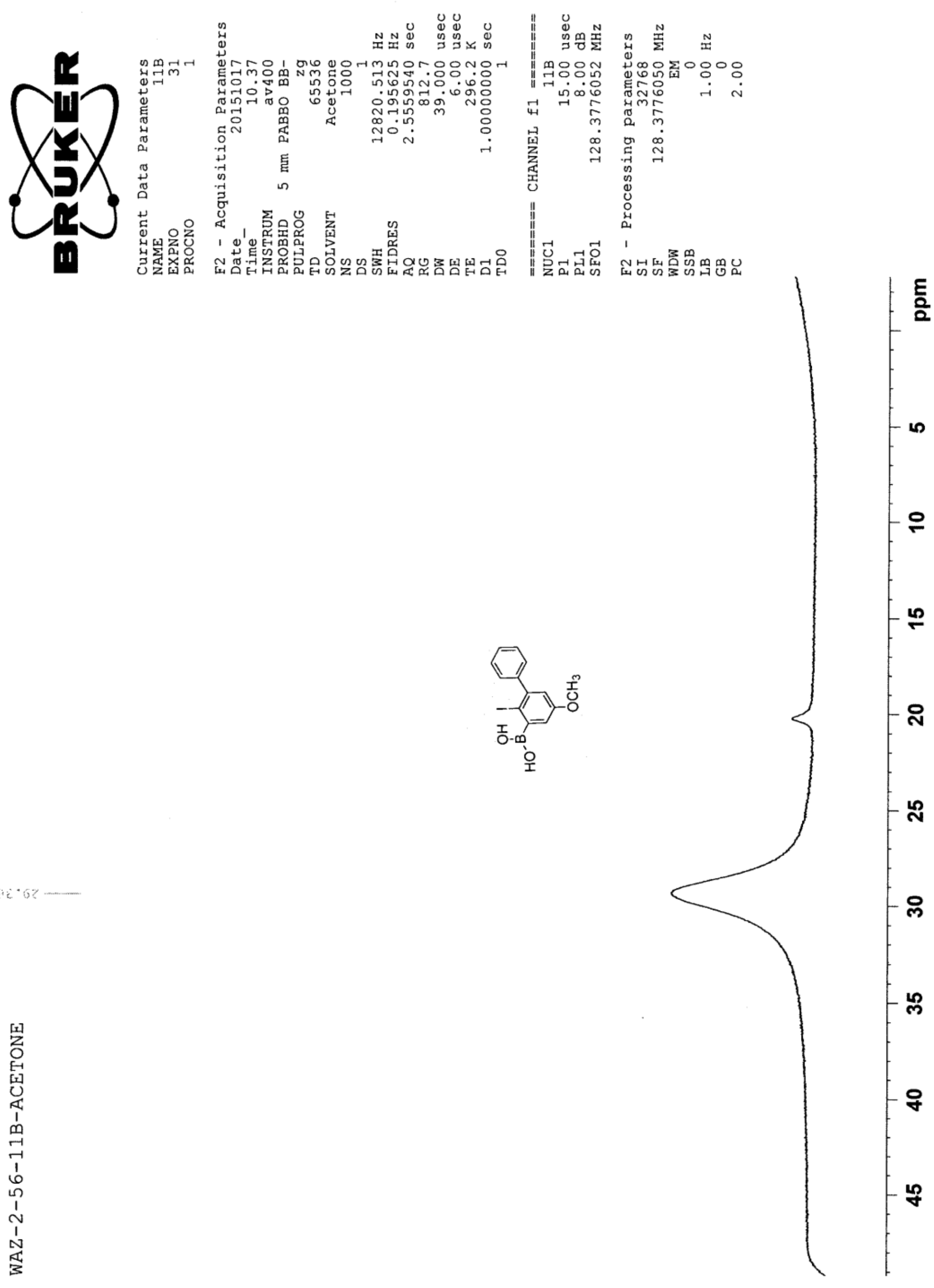
F2 - Acquisition Parameters
Date_          20151013
Time              15.07
INSTRUM           av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                19621
DS                    2
SWH           30120.482 Hz
FIDRES         0.459602 Hz
AQ            1.0879476 sec
RG              23170.5
DW               16.600 usec
DE                 6.00 usec
TE                296.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   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          100.6128112 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

1.5.56 ^{11}B-NMR of (2-iodo-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (14_A) in d^6-acetone at 25 ^{o}C



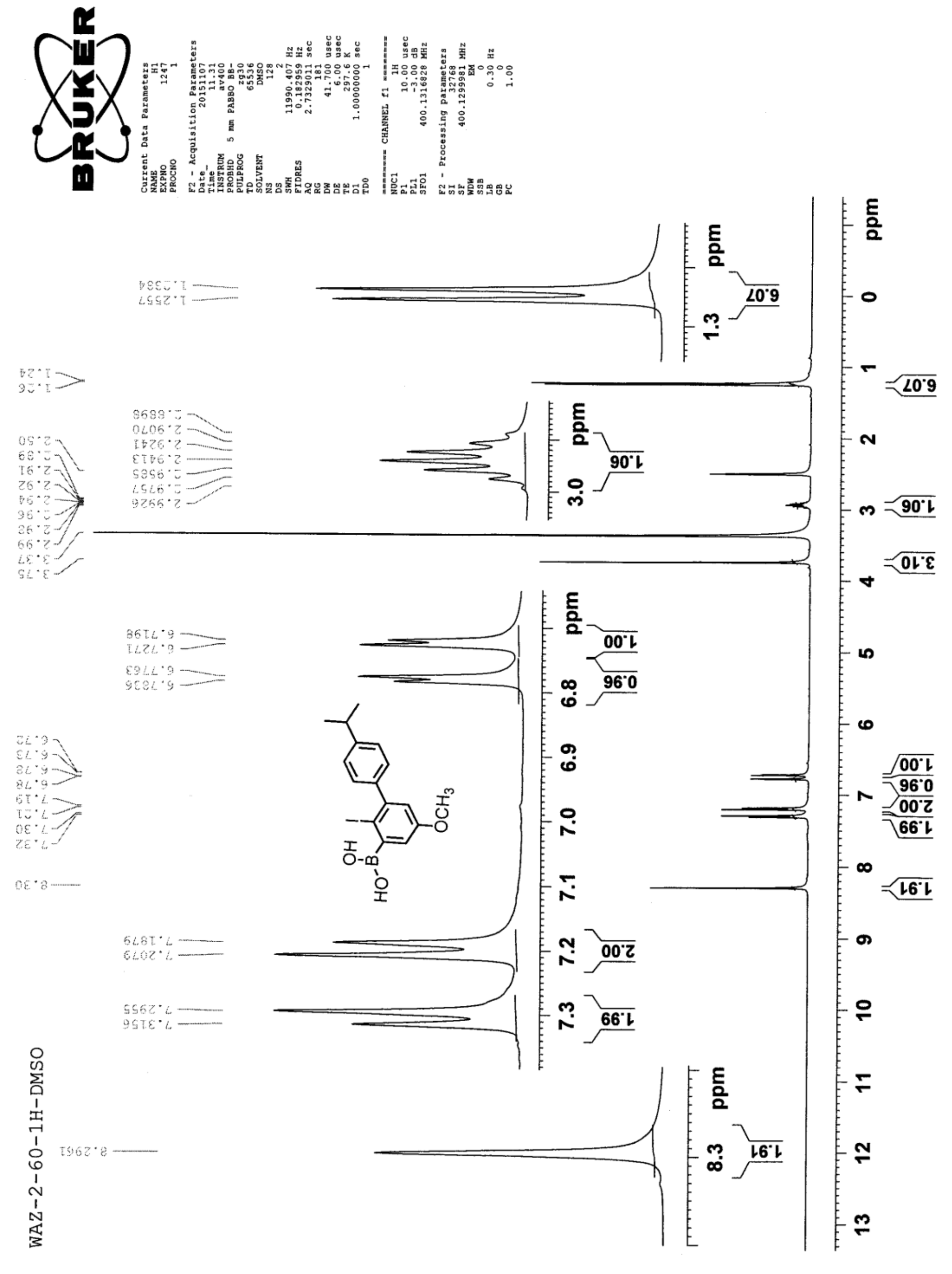
```
Current Data Parameters
NAME               11B
EXPNO               31
PROCNO               1

F2 - Acquisition Parameters
Date_          20151017
Time             10.37
INSTRUM          av400
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD               65536
SOLVENT        Acetone
NS                1000
DS                   1
SWH          12820.513 Hz
FIDRES        0.195625 Hz
AQ           2.5559540 sec
RG               812.7
DW              39.000 usec
DE                6.00 usec
TE               296.2 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               11B
P1               15.00 usec
PL1               8.00 dB
SFO1       128.3776052 MHz

F2 - Processing parameters
SI               32768
SF         128.3776050 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                2.00
```

1.5.57 ^{1}H-NMR of (2-iodo-4'-isopropyl-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (15$_A$) in d^6-DMSO at 25 ^{o}C.



```
Current Data Parameters
NAME                 H1
EXPNO              1247
PROCNO                1

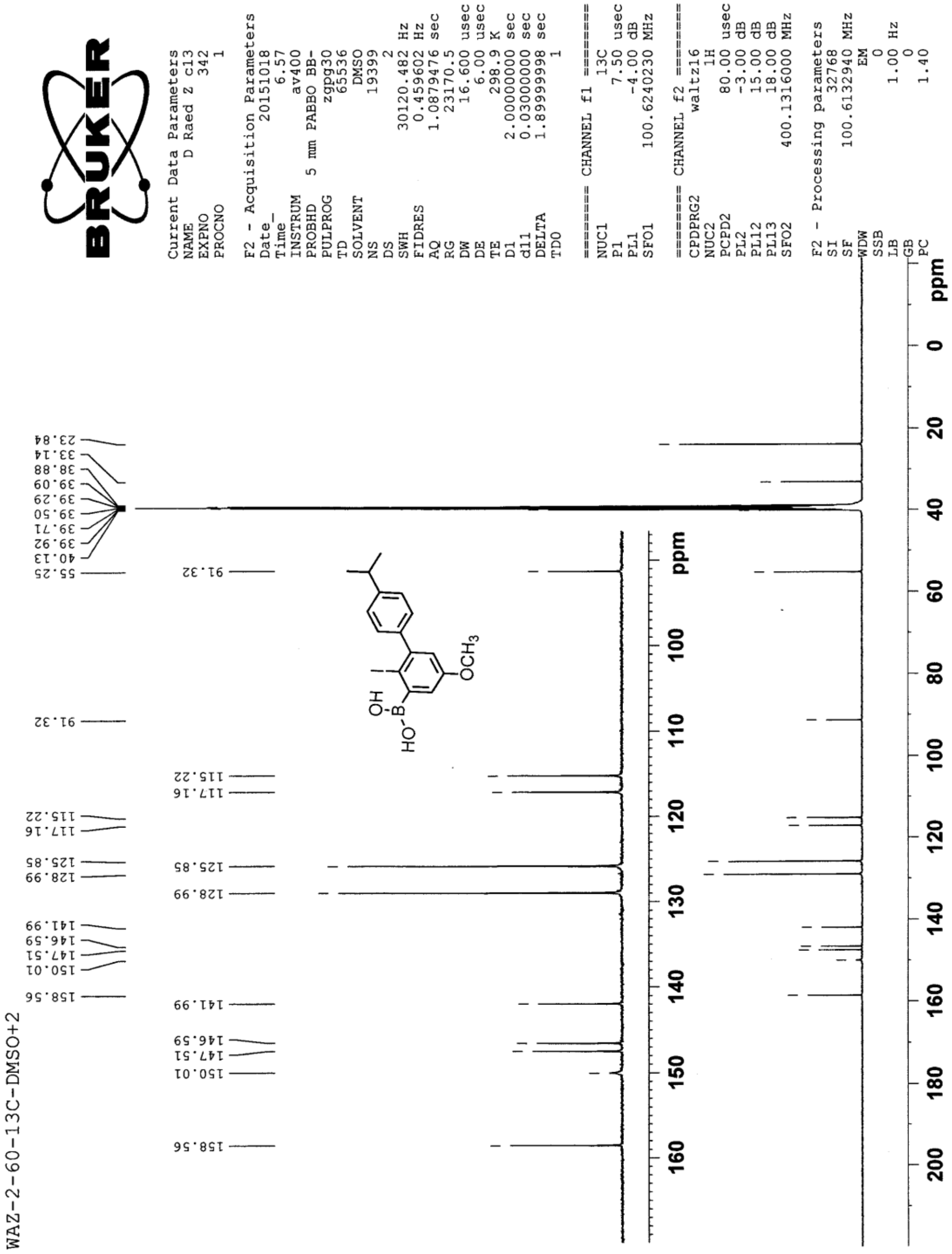
F2 - Acquisition Parameters
Date_          20151107
Time              11.31
INSTRUM           av400
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                  128
DS                    2
SWH          11990.407 Hz
FIDRES        0.182959 Hz
AQ           2.7329011 sec
RG                  181
DW              41.700 usec
DE                6.00 usec
TE               297.6 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -3.00 dB
SFO1        400.1316828 MHz

F2 - Processing parameters
SI                32768
SF          400.1299981 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

.

1.5.58 ^{13}C–NMR of (2-iodo-4'-isopropyl-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (15$_A$) in d^{6}-DMSO at 25 ^{o}C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  342
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151018
Time                  6.57
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               DMSO
NS                   19399
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                298.9 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                      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          100.6132940 MHz
WDW                     EM
SSB                      0
LB                1.00 Hz
GB                       0
PC                    1.40
```

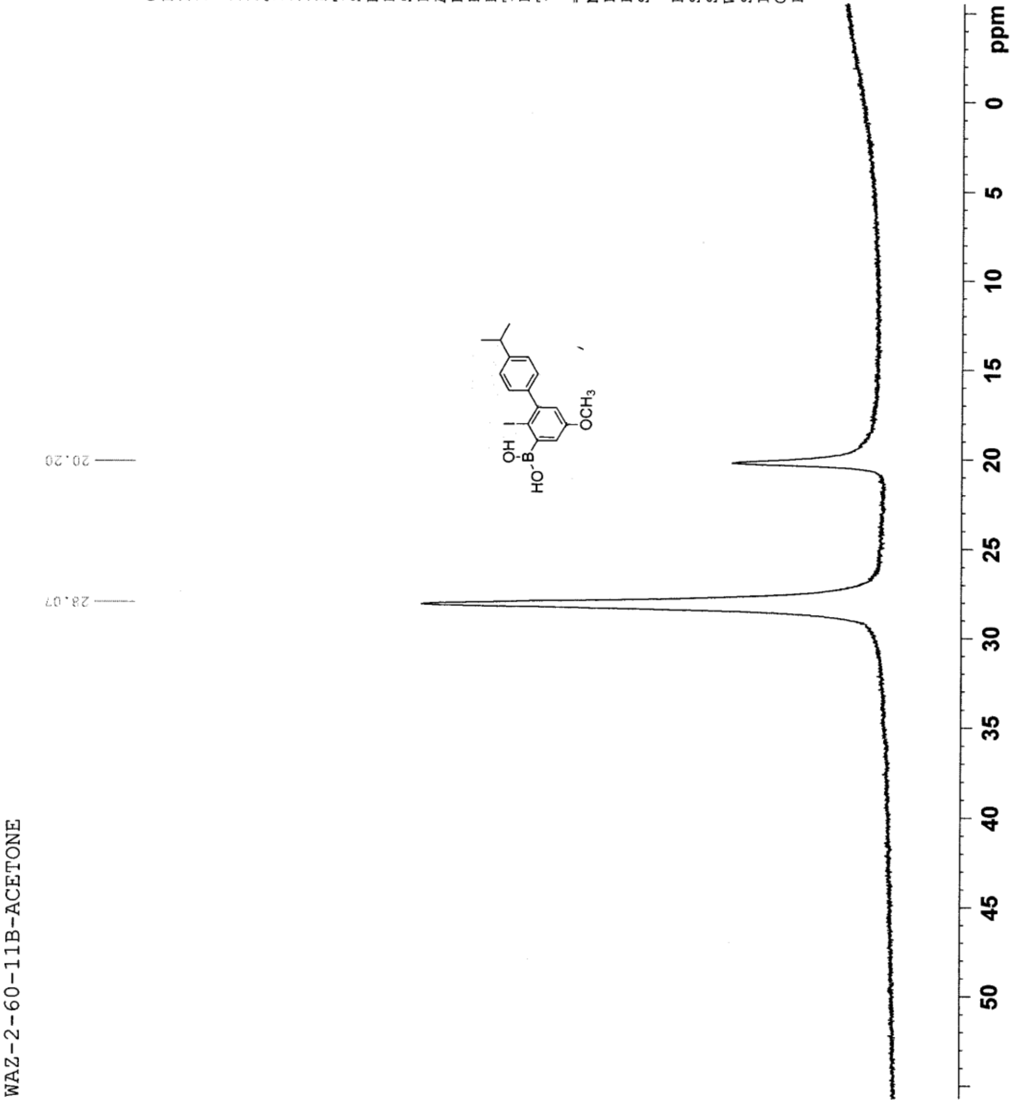
1.5.59 ^{11}B-NMR of (2-iodo-4'-isopropyl-5-methoxy-[1,1'-biphenyl]-3-yl)boronic acid (15_A) in d^6-acetone at 25 °C

```
Current Data Parameters
NAME              11B
EXPNO              43
PROCNO              1

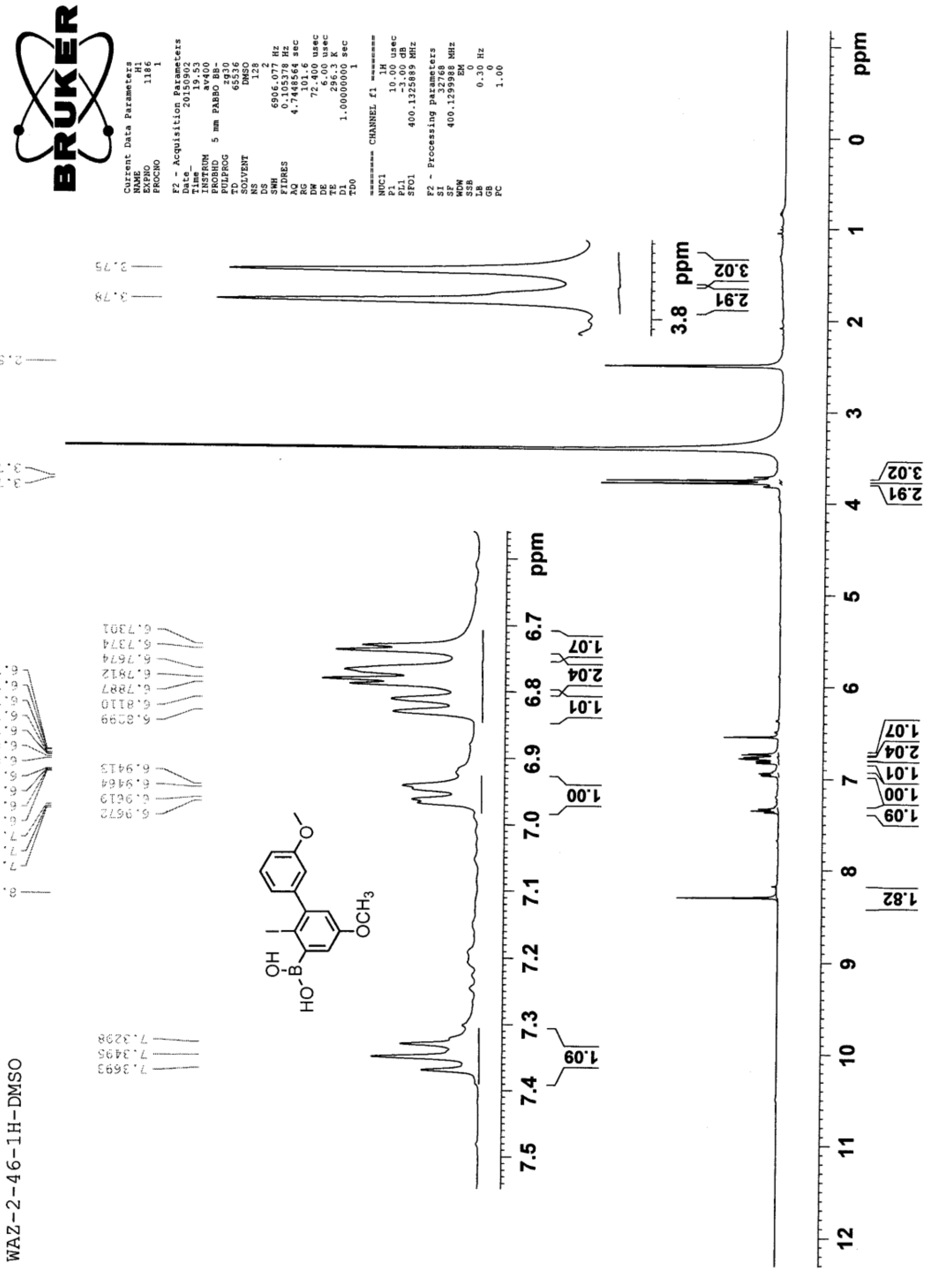
F2 - Acquisition Parameters
Date_        20151107
Time            11.57
INSTRUM         av400
PROBHD   5 mm PABBO BB-
PULPROG            zg
TD              65536
SOLVENT       Acetone
NS                237
DS                  1
SWH         25641.025 Hz
FIDRES       0.391251 Hz
AQ          1.2780020 sec
RG              812.7
DW             19.500 usec
DE               6.00 usec
TE              296.8 K
D1         1.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1              11B
P1              15.00 usec
PL1              8.00 dB
SFO1      128.3776052 MHz

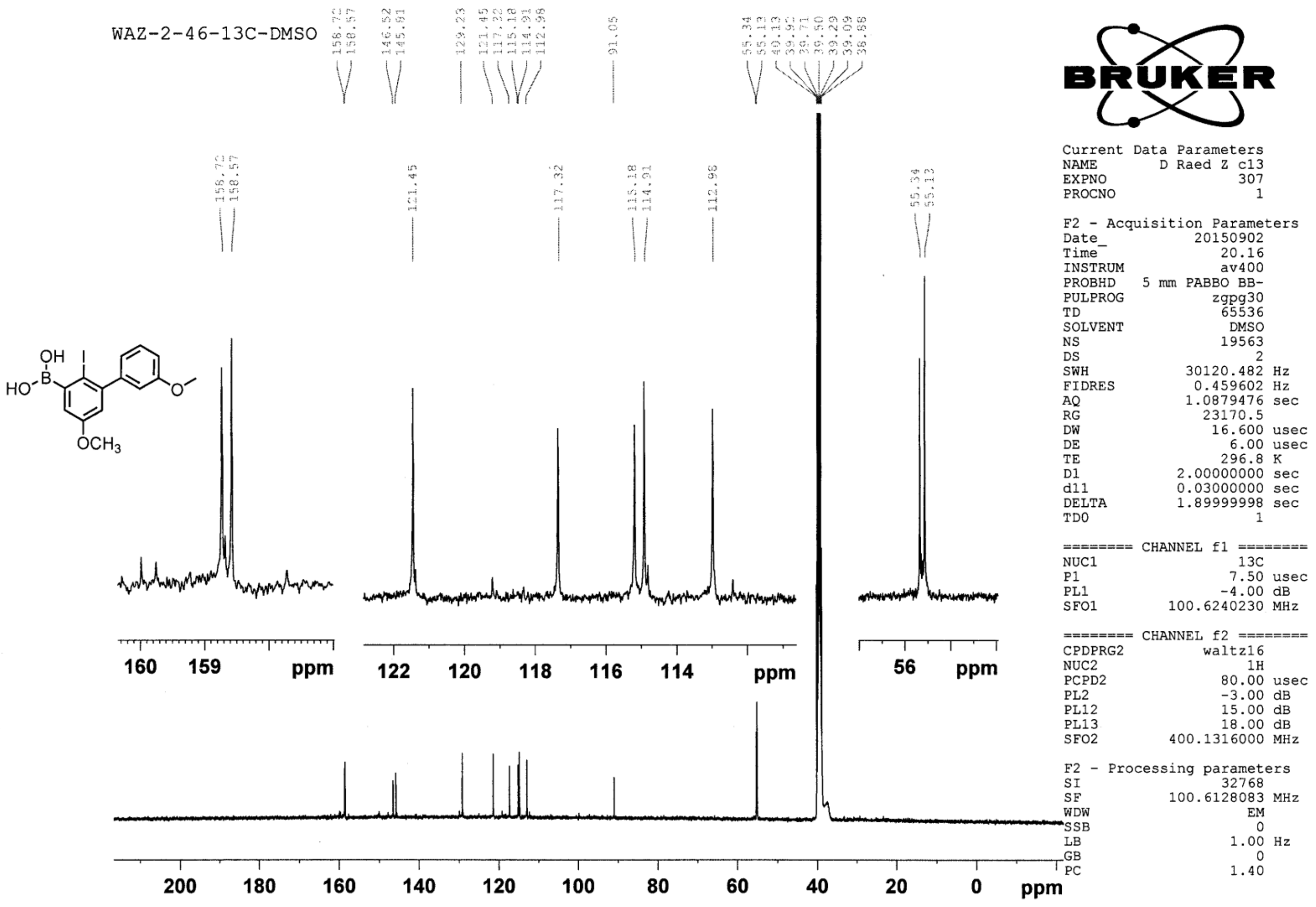
F2 - Processing parameters
SI              32768
SF        128.3776050 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               2.00
```



1.5.60 ^{1}H-NMR of (2-iodo-3',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (16_A) in d^{6-}DMSO at 25 ^{o}C.



1.5.61 ^{13}C–NMR of (2-iodo-3',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (16_A) in d^6-DMSO at 25 °C.



```
Current Data Parameters
NAME        D Raed Z c13
EXPNO                307
PROCNO                 1

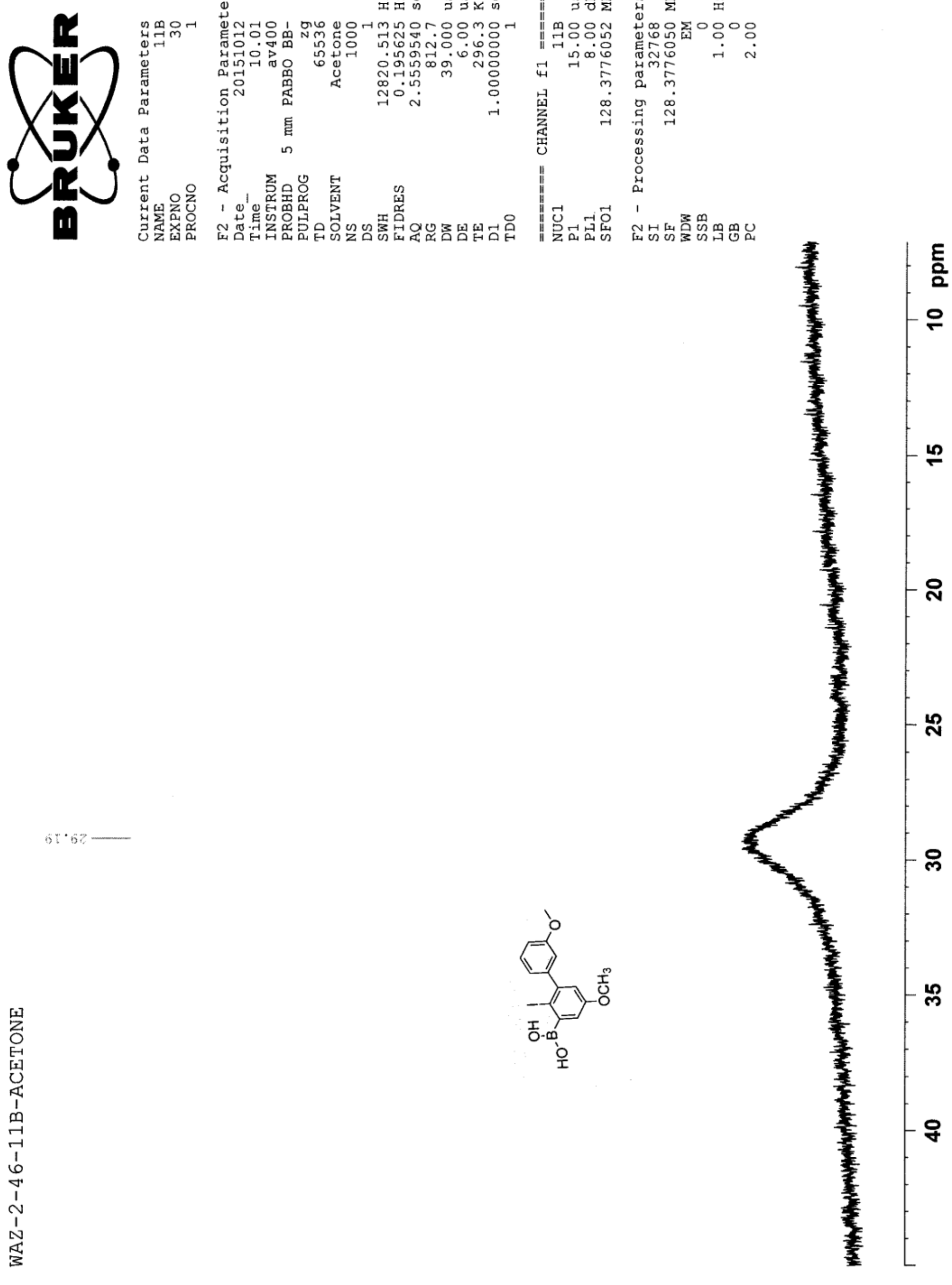
F2 - Acquisition Parameters
Date_           20150902
Time               20.16
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                19563
DS                    2
SWH           30120.482 Hz
FIDRES         0.459602 Hz
AQ            1.0879476 sec
RG              23170.5
DW               16.600 usec
DE                 6.00 usec
TE                296.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   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           100.6128083 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

1.5.62 ^{11}B-NMR of (2-iodo-3',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (16_A) in d^6-acetone at 25 ^{o}C



WAZ-2-46-11B-ACETONE

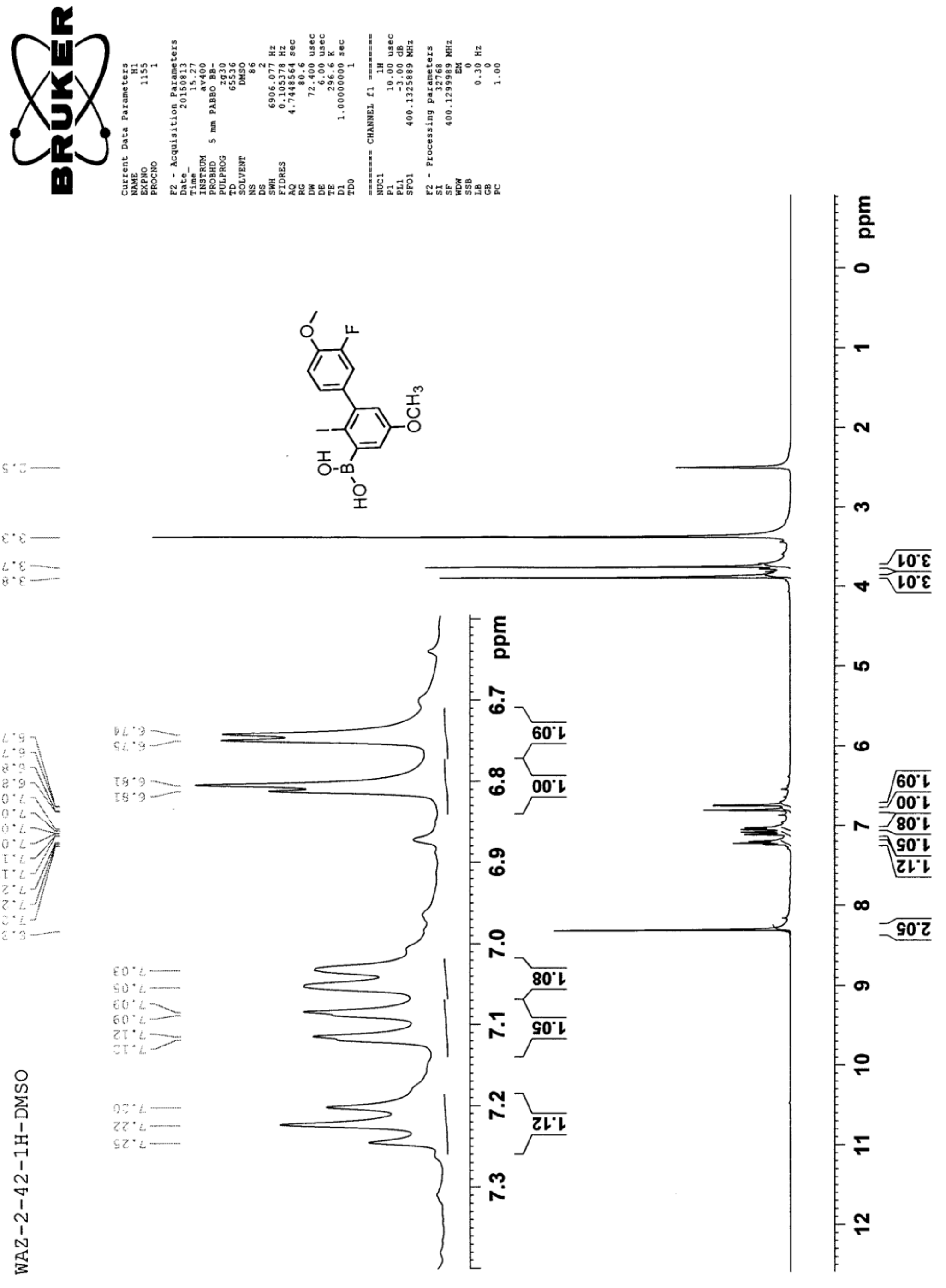
```
Current Data Parameters
NAME                 11B
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151012
Time               10.01
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 65536
SOLVENT          Acetone
NS                  1000
DS                     1
SWH            12820.513 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                 812.7
DW                39.000 usec
DE                  6.00 usec
TE                 296.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 11B
P1                 15.00 usec
PL1                 8.00 dB
SFO1         128.3776052 MHz

F2 - Processing parameters
SI                 32768
SF           128.3776050 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00
```

1.5.63 ^{1}H-NMR of (3'-fluoro-2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (17_A) in d^6-DMSO at 25 ^{o}C.



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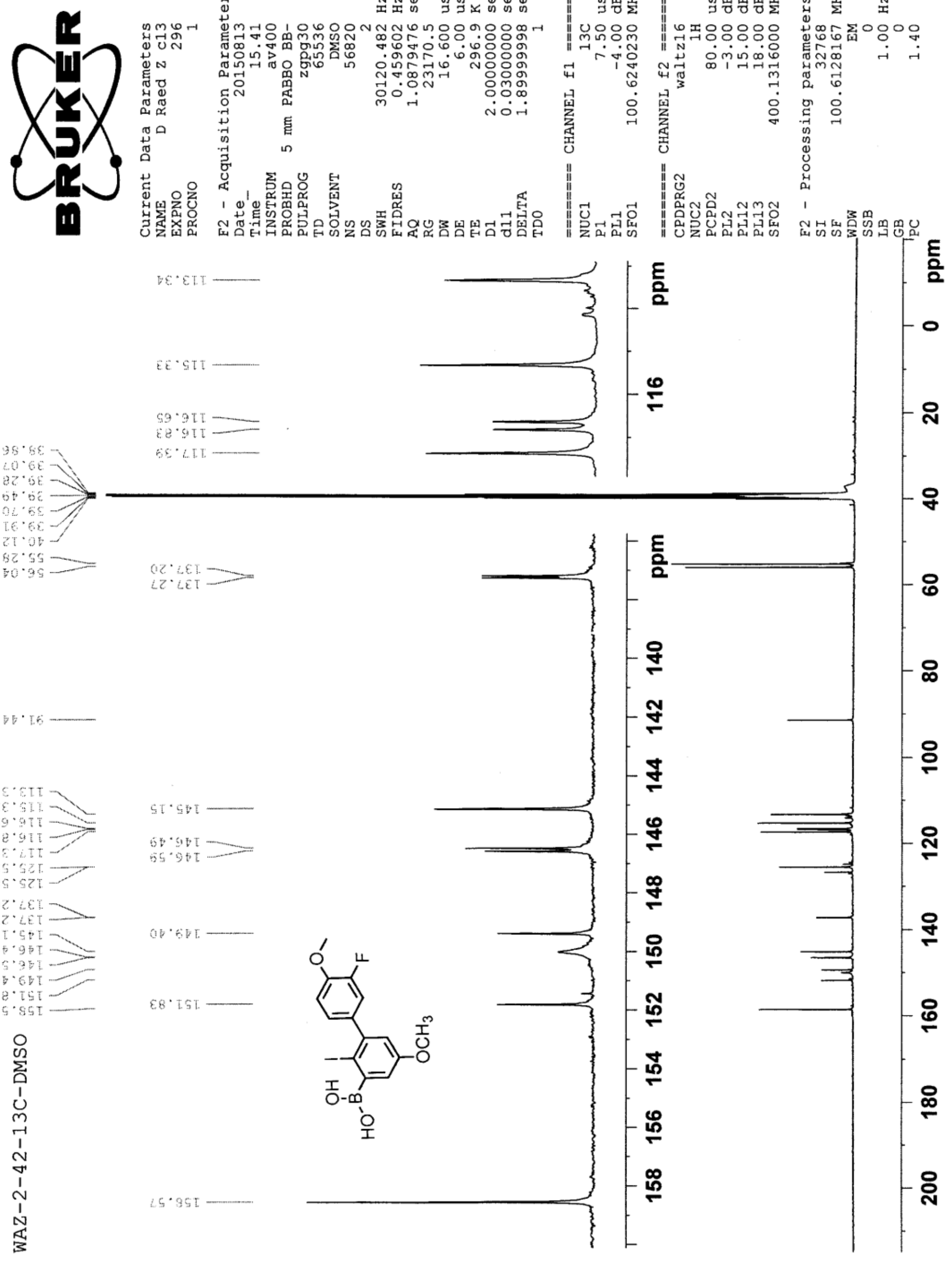
1.5.64 ^{13}C–NMR of (3'-fluoro-2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (17$_A$) in d^6-DMSO at 25 °C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  296
PROCNO                   1

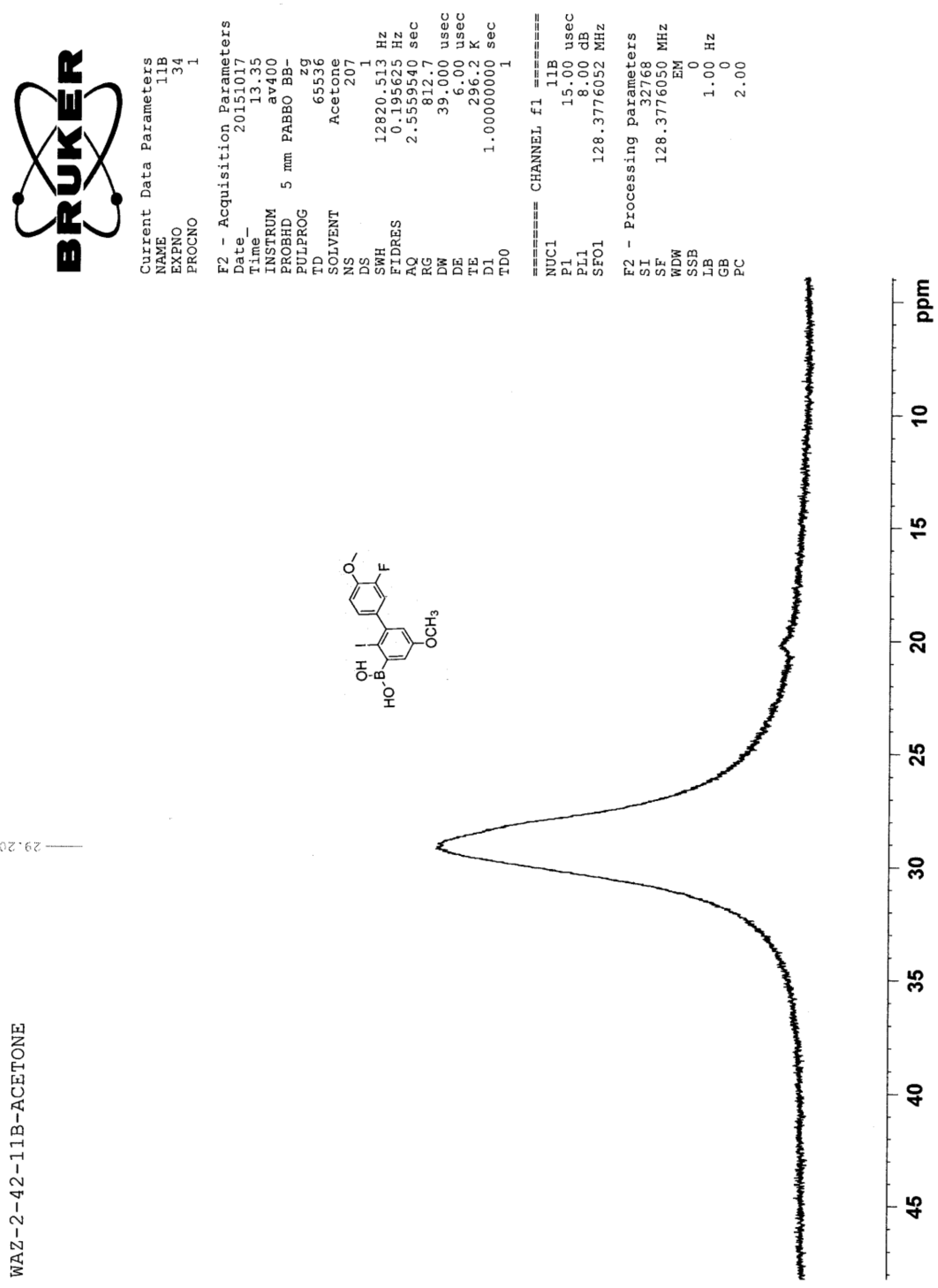
F2 - Acquisition Parameters
Date_             20150813
Time                 15.41
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               DMSO
NS                   56820
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   296.9 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      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             100.6128167 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

1.5.65 ^{11}B-NMR of (3'-fluoro-2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (17_A) in d^6-acetone at 25 ^{o}C



```
Current Data Parameters
NAME                11B
EXPNO                34
PROCNO                1

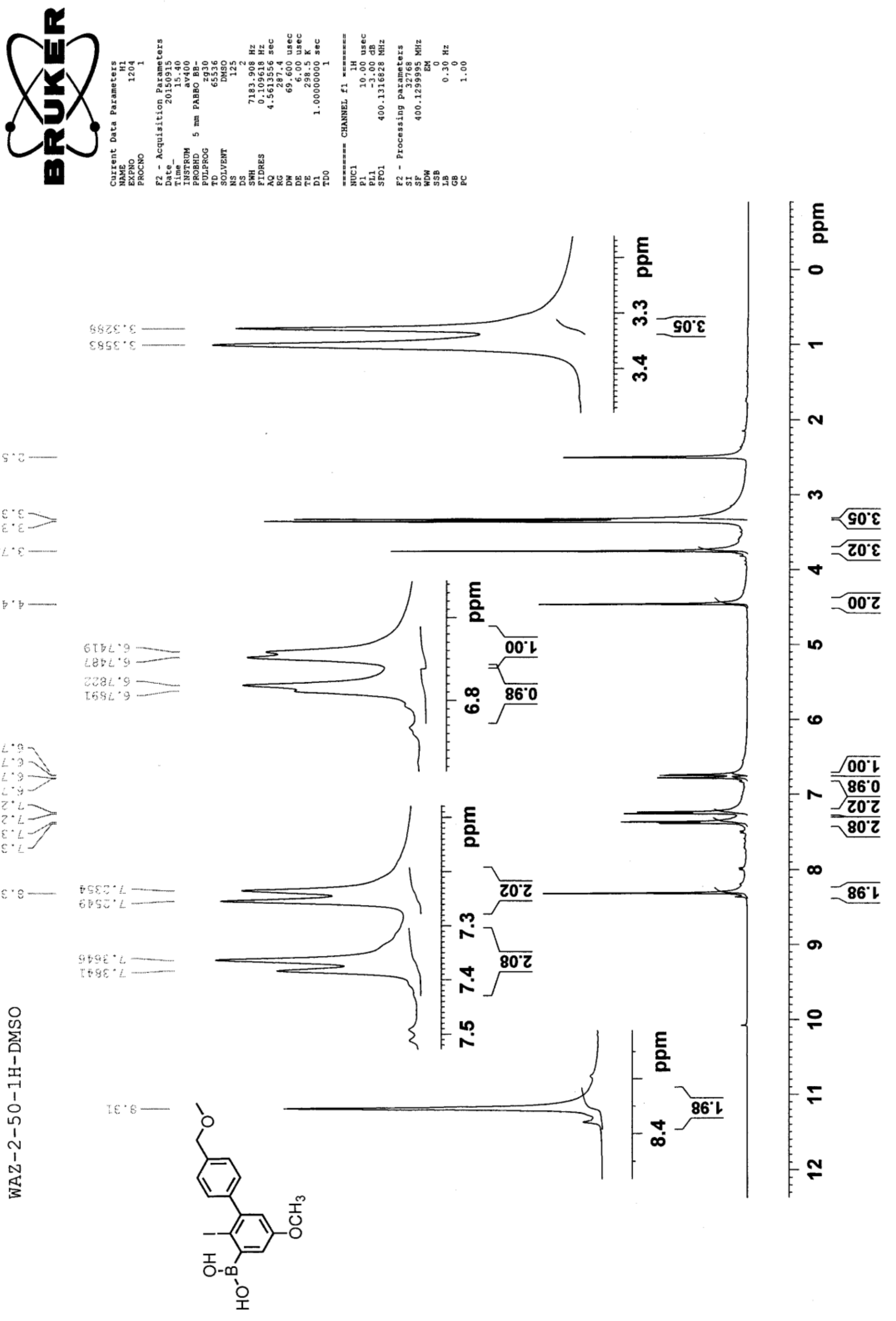
F2 - Acquisition Parameters
Date_          20151017
Time_             13.35
INSTRUM           av400
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                65536
SOLVENT         Acetone
NS                  207
DS                    1
SWH           12820.513 Hz
FIDRES         0.195625 Hz
AQ            2.5559540 sec
RG                812.7
DW               39.000 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                11B
P1                15.00 usec
PL1                8.00 dB
SFO1        128.3776052 MHz

F2 - Processing parameters
SI                32768
SF          128.3776050 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00
```

1.5.66

1.5.67 ^{1}H-NMR of (2-iodo-5-methoxy-4'-(methoxymethyl)-[1,1'-biphenyl]-3-yl) boronic acid (18$_A$) in d^6-DMSO at 25 °C.



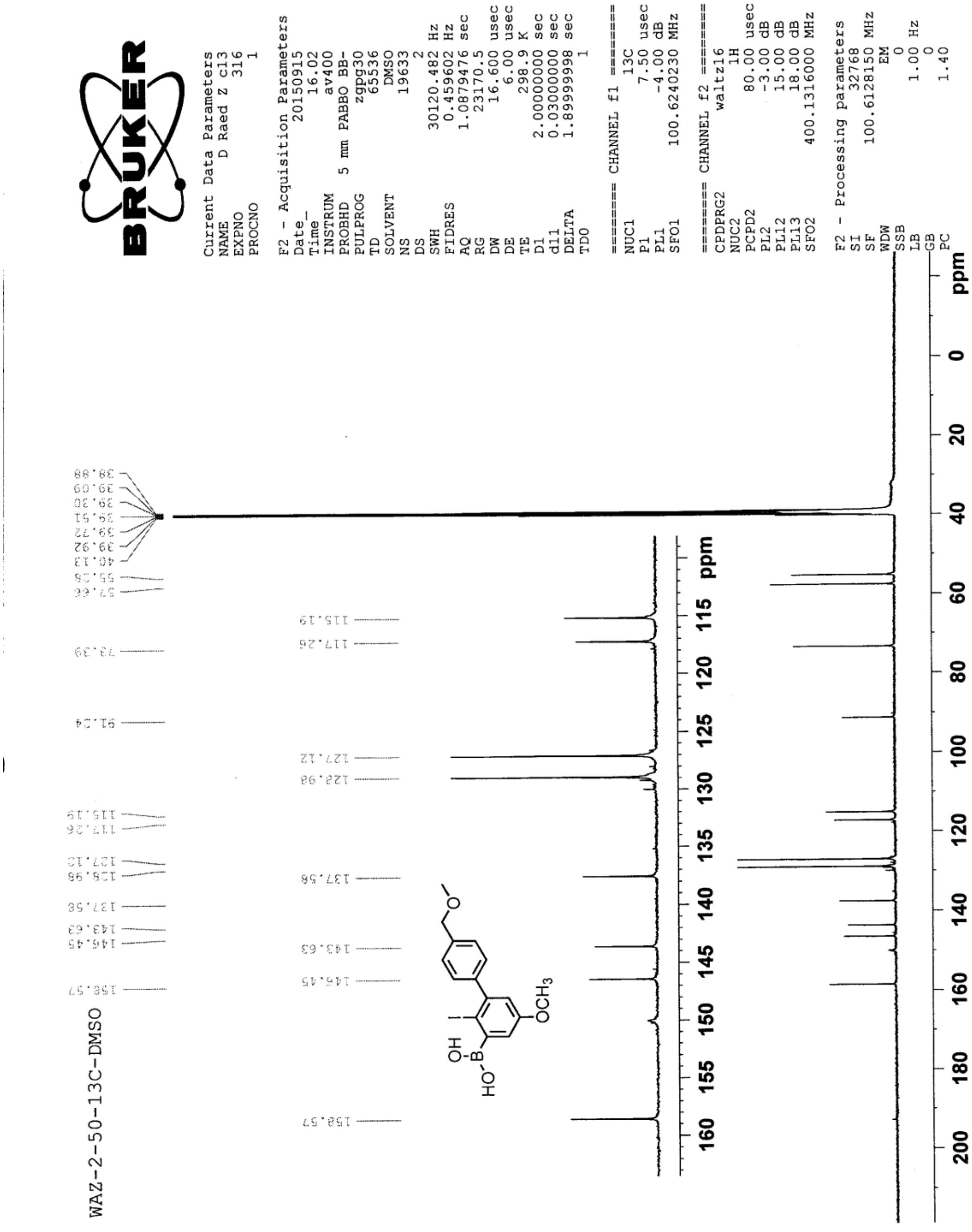
```
Current Data Parameters
NAME                 H1
EXPNO              1204
PROCNO                1

F2 - Acquisition Parameters
Date_          20150915
Time              15.40
INSTRUM           av400
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                  125
DS                    2
SWH            7183.908 Hz
FIDRES         0.109618 Hz
AQ            4.5613556 sec
RG                287.4
DW               69.600 usec
DE                 6.00 usec
TE                298.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -3.00 dB
SFO1        400.1316828 MHz

F2 - Processing parameters
SI                32768
SF          400.1299995 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

1.5.68 ^{13}C–NMR of (2-iodo-5-methoxy-4'-(methoxymethyl)-[1,1'-biphenyl]-3-yl) boronic acid (18$_A$) in d^6-DMSO at 25 ^{o}C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  316
PROCNO                   1

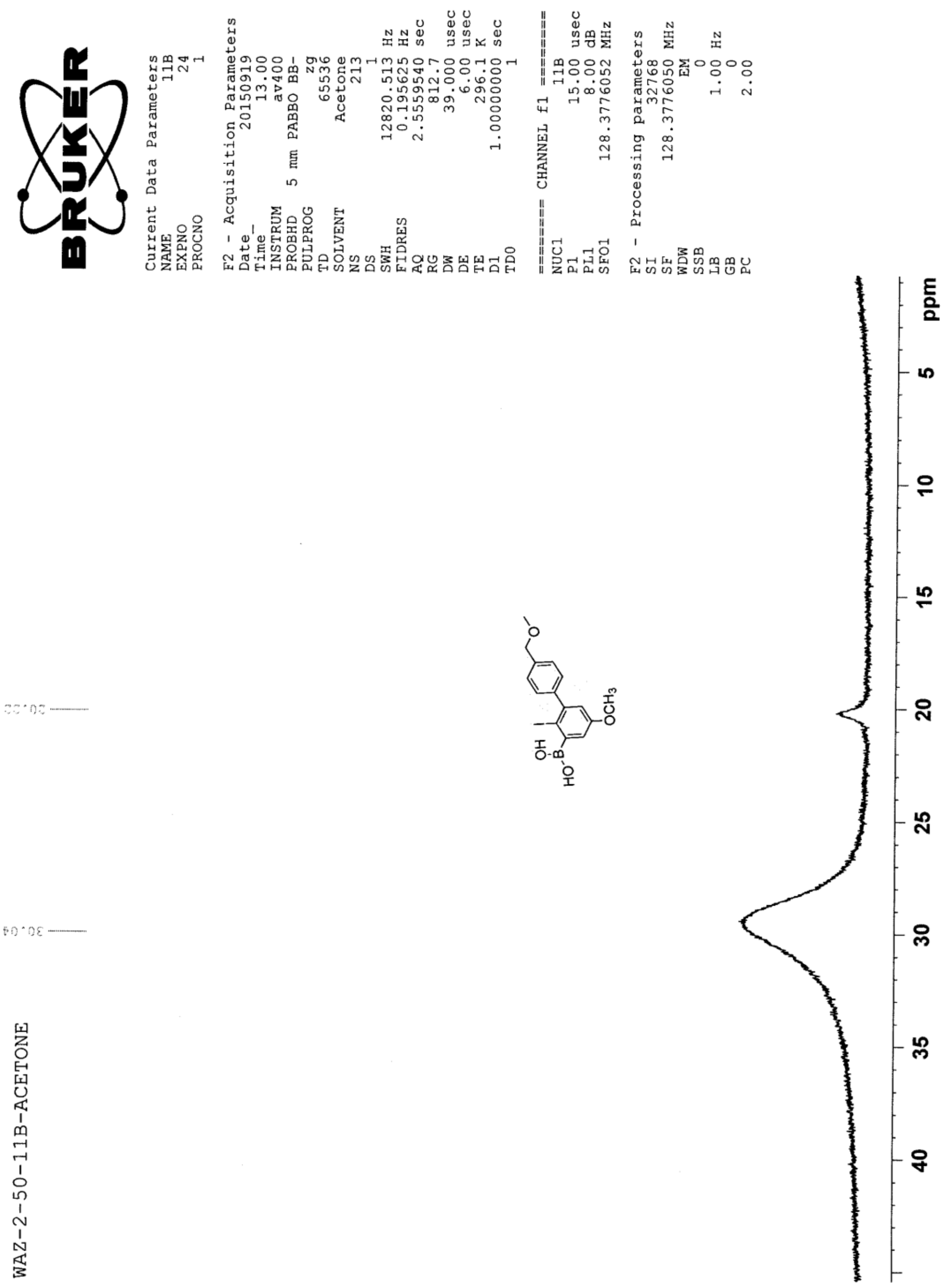
F2 - Acquisition Parameters
Date_             20150915
Time                 16.02
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                   19633
DS                       2
SWH          30120.482 Hz
FIDRES        0.459602 Hz
AQ          1.0879476 sec
RG                 23170.5
DW              16.600 usec
DE                6.00 usec
TE                 298.9 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0                      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          100.6128150 MHz
WDW                     EM
SSB                      0
LB                 1.00 Hz
GB                       0
PC                    1.40
```

1.5.69 ^{11}B-NMR of (2-iodo-5-methoxy-4'-(methoxymethyl)-[1,1'-biphenyl]-3-yl) boronic acid (18$_A$) in d^6-acetone at 25 ^{o}C


Current Data Parameters
NAME 11B
EXPNO 24
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150919
Time_ 13.00
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 65536
SOLVENT Acetone
NS 213
DS 1
SWH 12820.513 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 812.7
DW 39.000 usec
DE 6.00 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1

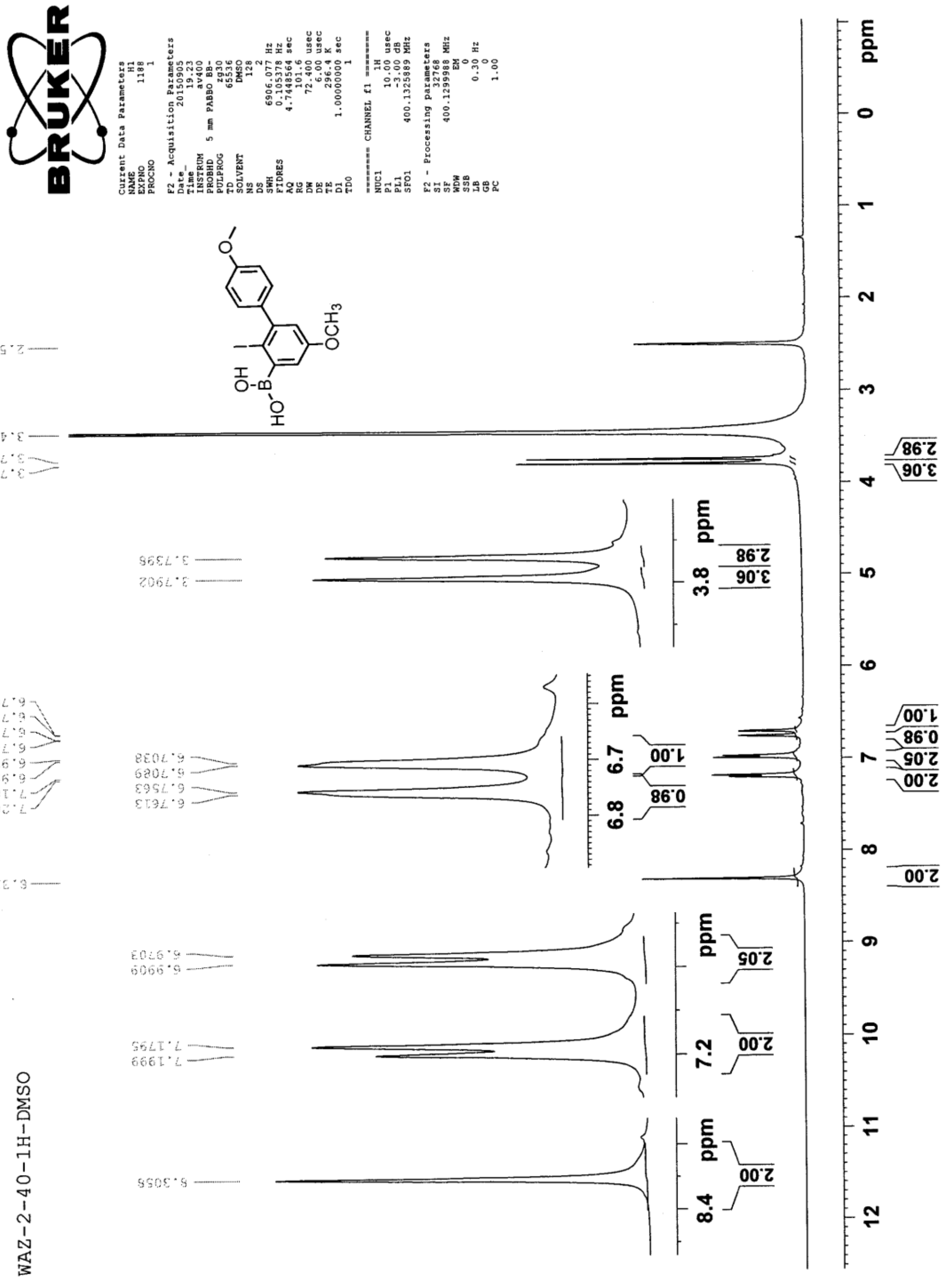
======== CHANNEL f1 ========
NUC1 11B
P1 15.00 usec
PL1 8.00 dB
SFO1 128.3776052 MHz

F2 - Processing parameters
SI 32768
SF 128.3776050 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

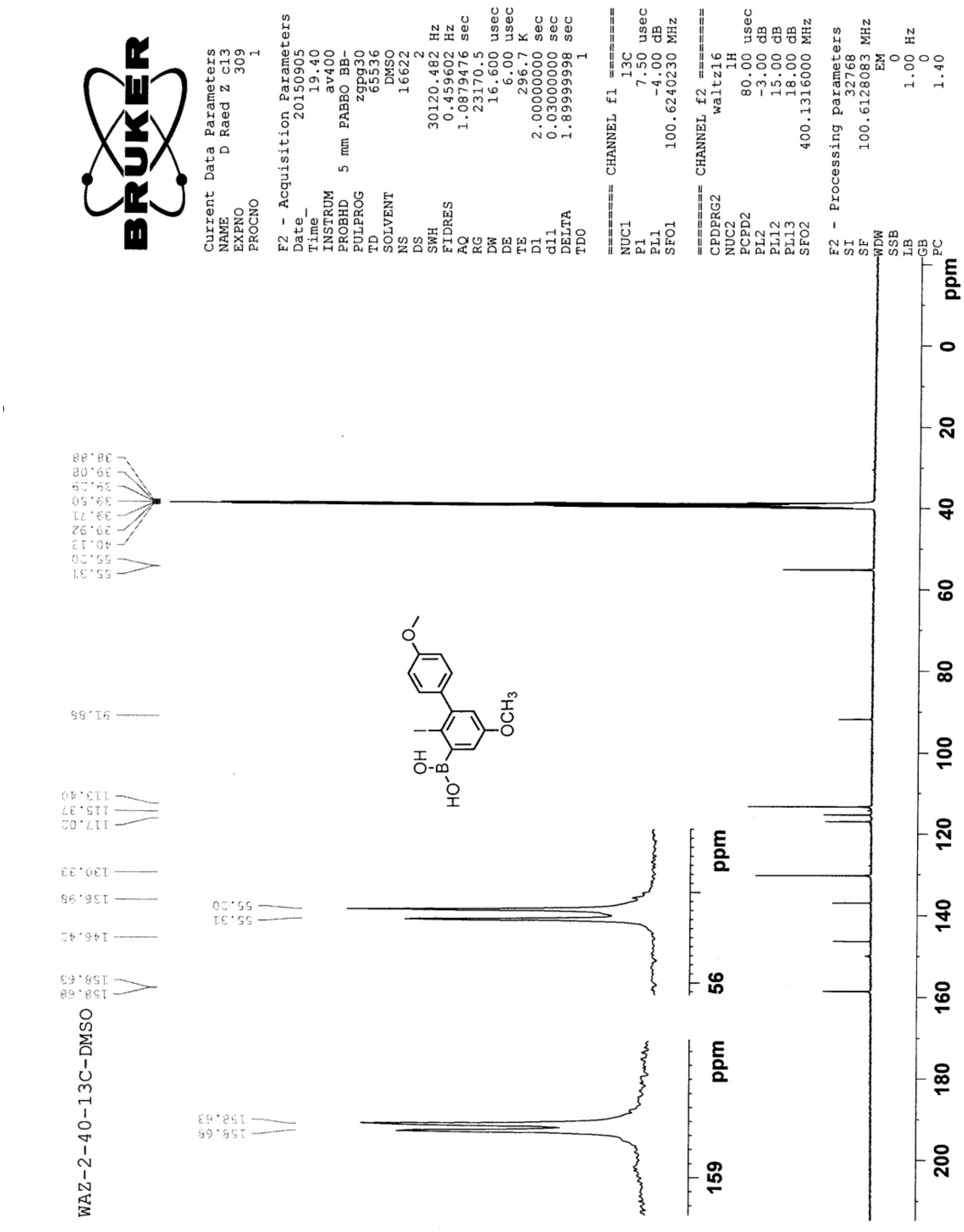
WAZ-2-50-11B-ACETONE

1.5.70 .

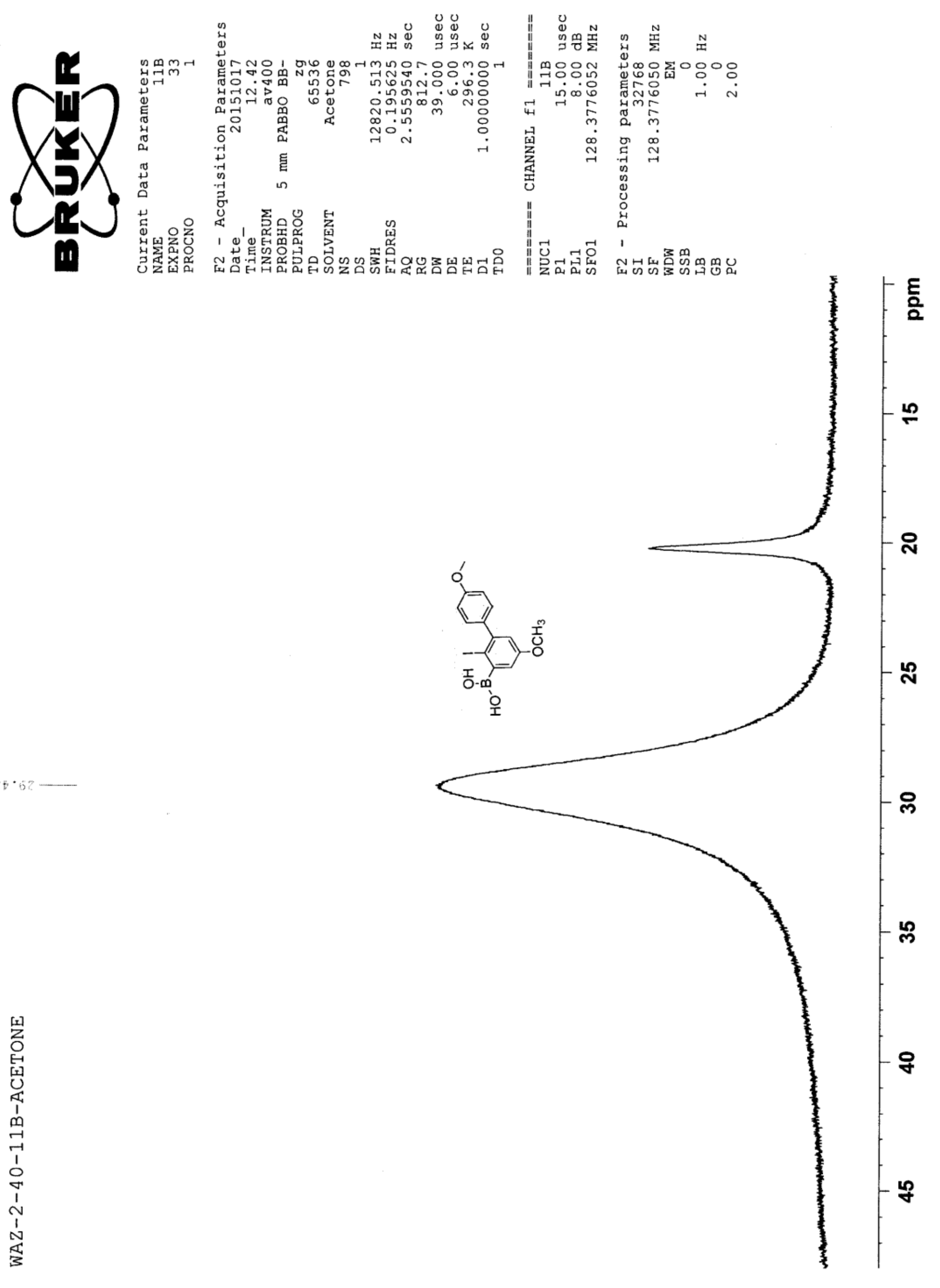
1.5.71 ^{1}H-NMR of (2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (19$_A$) in d^6-DMSO at 25 ^{o}C.



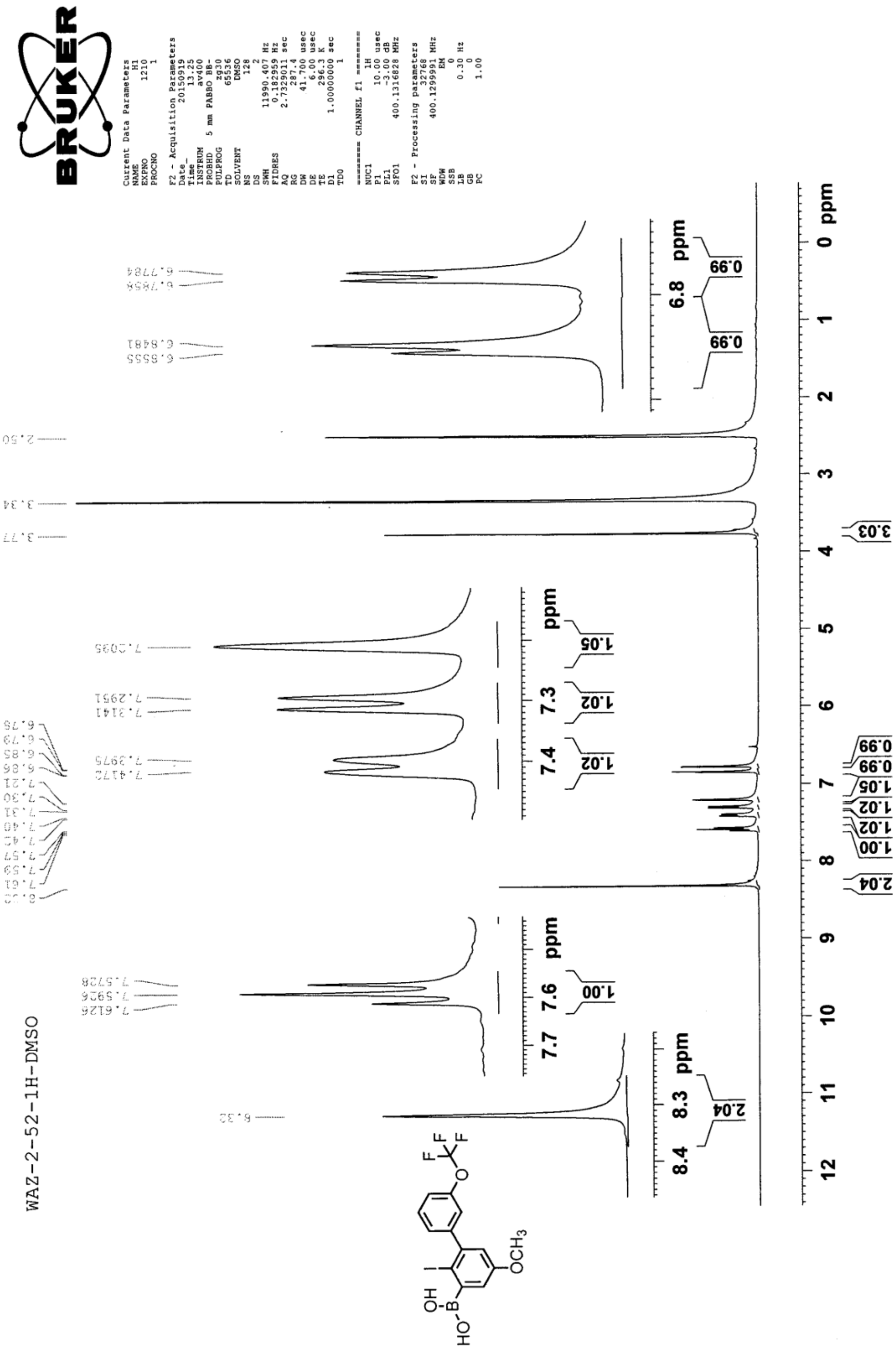
1.5.72 ^{13}C–NMR of (2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (19_A) in d^{6}-DMSO at 25 ^{o}C.



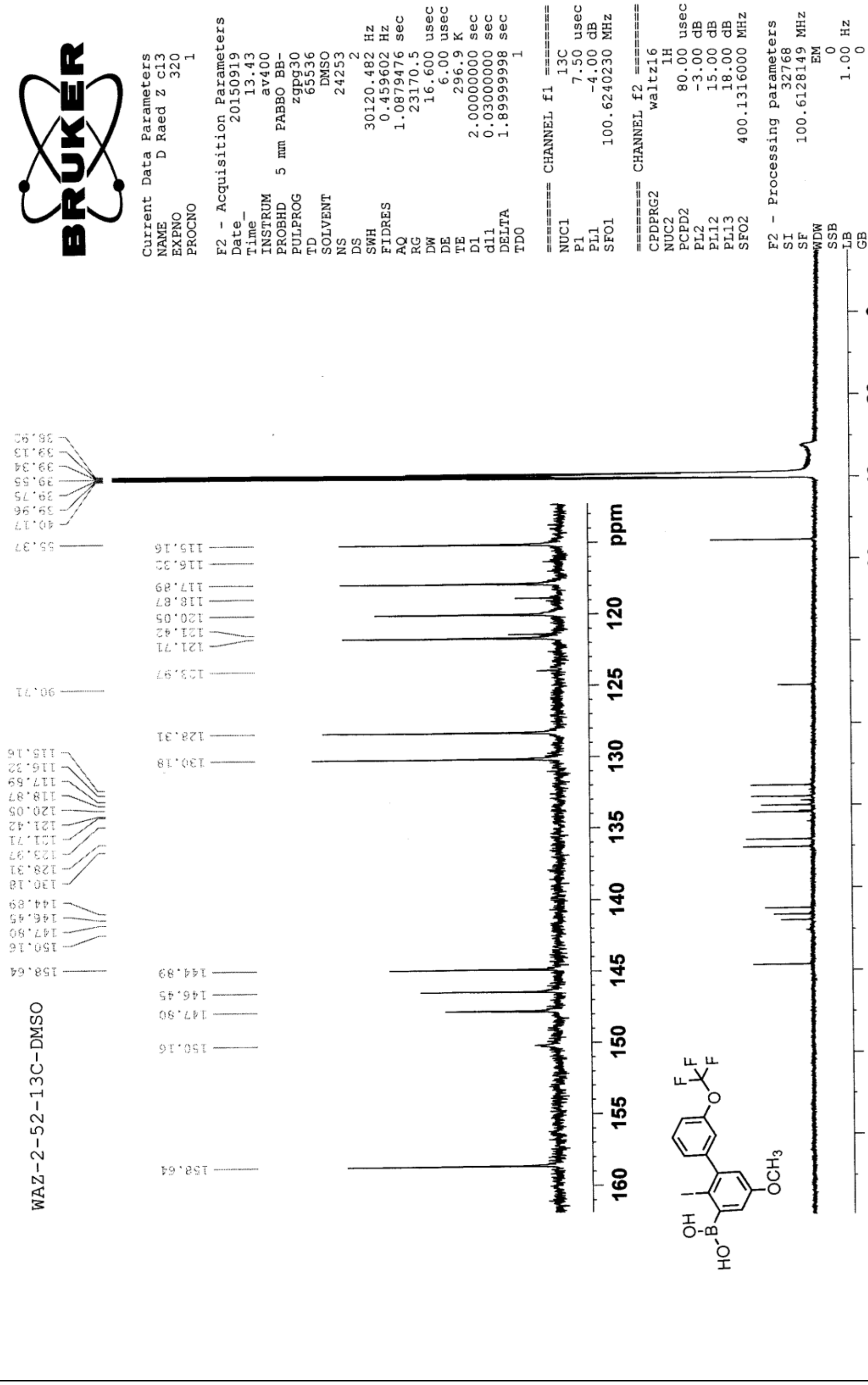
1.5.73 ^{11}B-NMR of (2-iodo-4',5-dimethoxy-[1,1'-biphenyl]-3-yl)boronic acid (19_A) in d^6-acetone at 25 °C



1.5.74 ^{1}H-NMR of (2-iodo-5-methoxy-3'-(trifluoromethoxy)-[1,1'-biphenyl]-3-yl)boronic acid (20_A) in d^{6-}DMSO at 25 ^{o}C.



1.5.75 ^{13}C–NMR of (2-iodo-5-methoxy-3'-(trifluoromethoxy)-[1,1'-biphenyl]-3-yl)boronic acid (20_A) in d^6-DMSO at 25 °C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  320
PROCNO                   1

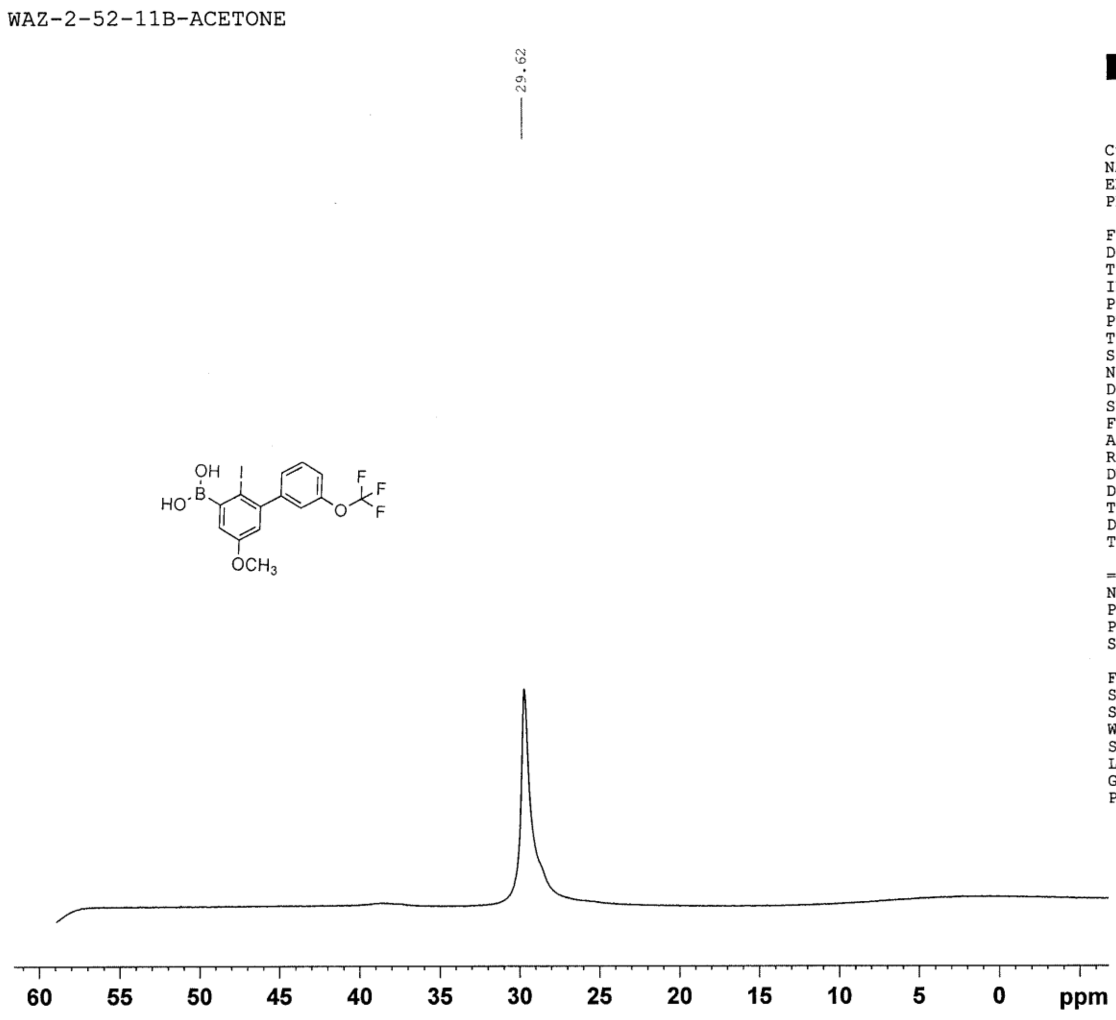
F2 - Acquisition Parameters
Date_             20150919
Time                 13.43
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               DMSO
NS                   24253
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   296.9 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      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             100.6128149 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

1.5.76 ^{11}B-NMR of (2-iodo-5-methoxy-3'-(trifluoromethoxy)-[1,1'-biphenyl]-3-yl) boronic acid (20_A) in d^6-acetone at 25 °C



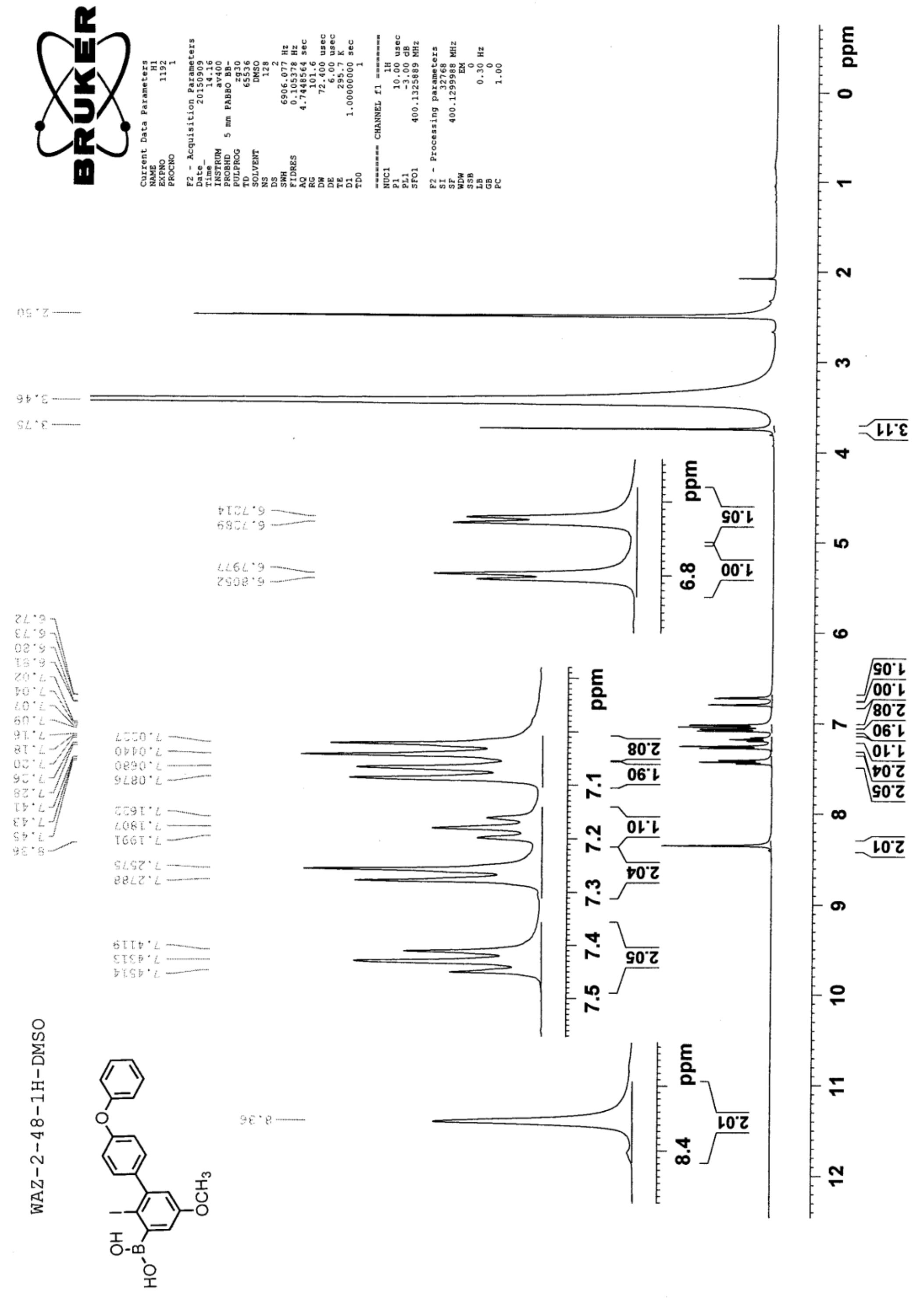
```
Current Data Parameters
NAME                 11B
EXPNO                 25
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150920
Time               12.49
INSTRUM            av400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 65536
SOLVENT          Acetone
NS                   500
DS                     1
SWH            12820.513 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                 812.7
DW                39.000 usec
DE                  6.00 usec
TE                 296.3 K
D1            1.00000000 sec
TD0                    1

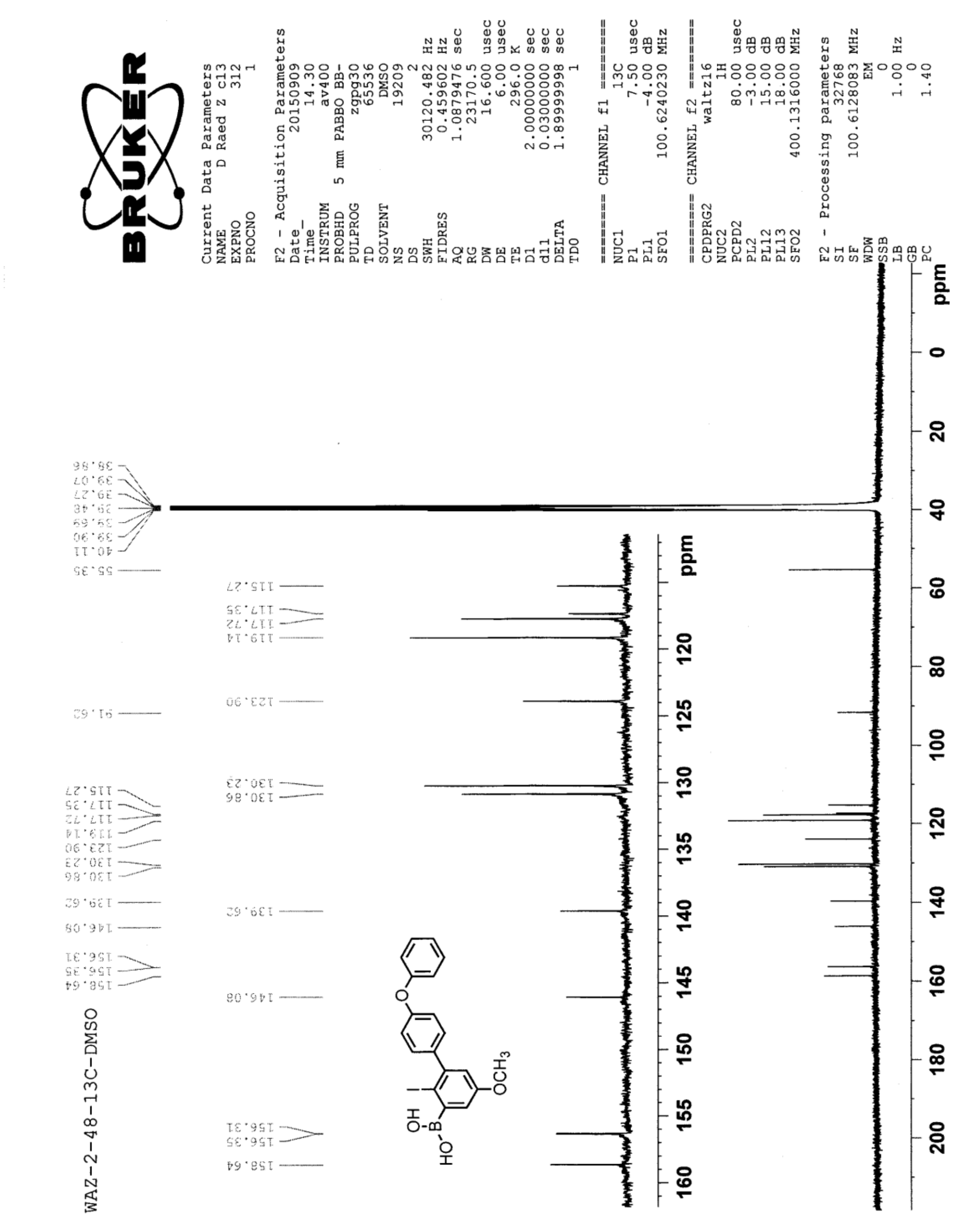
======== CHANNEL f1 ========
NUC1                 11B
P1                 15.00 usec
PL1                 8.00 dB
SFO1         128.3776052 MHz

F2 - Processing parameters
SI                 32768
SF           128.3776050 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00
```

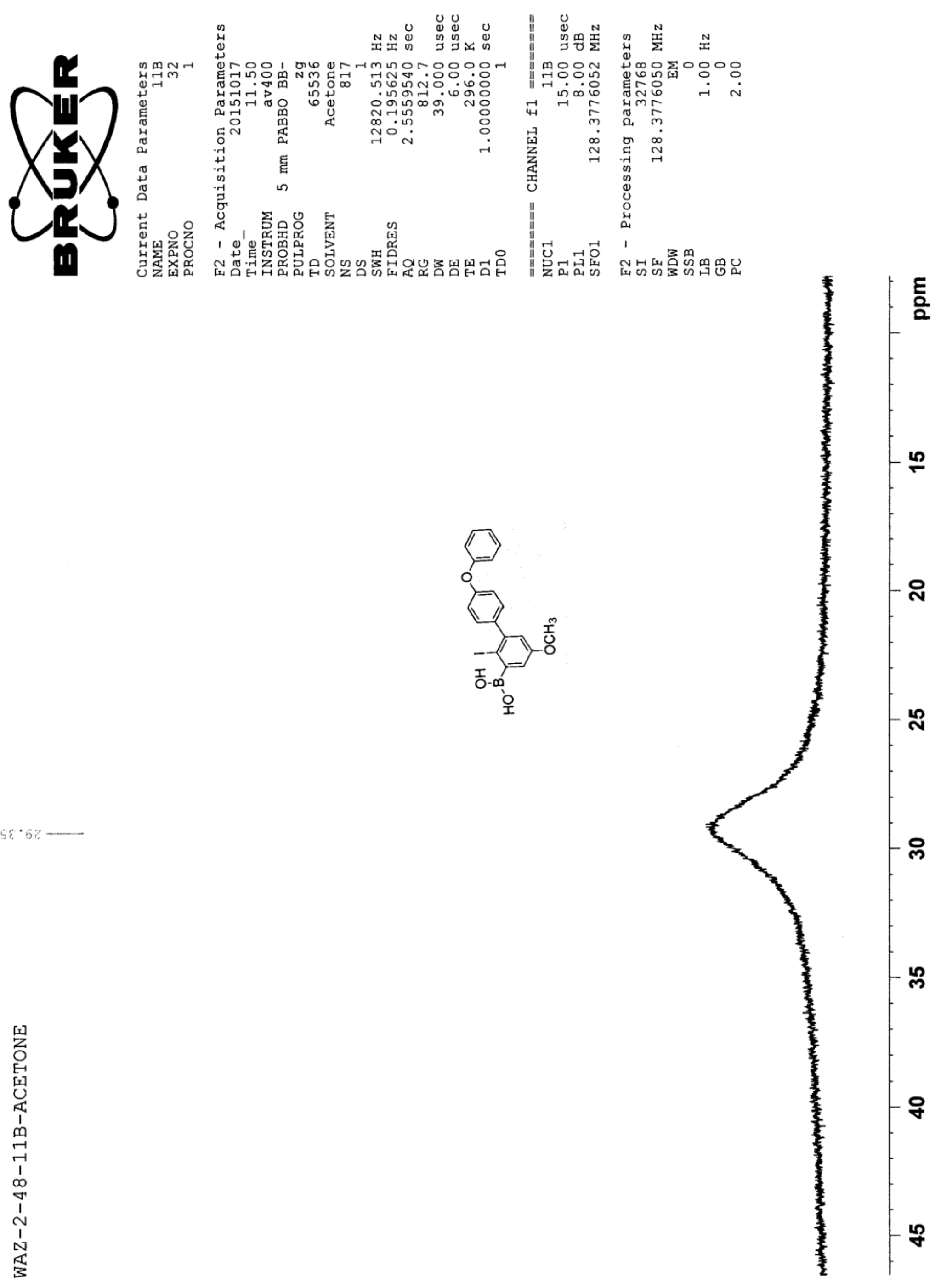
1.5.77 ^{1}H-NMR of (2-iodo-5-methoxy-4'-phenoxy-[1,1'-biphenyl]-3-yl)boronic acid (21$_A$) in d^{6-}DMSO at 25 ^{o}C.



1.5.78 ^{13}C–NMR of (2-iodo-5-methoxy-4'-phenoxy-[1,1'-biphenyl]-3-yl)boronic acid (21_A) in d^6-DMSO at 25 °C.

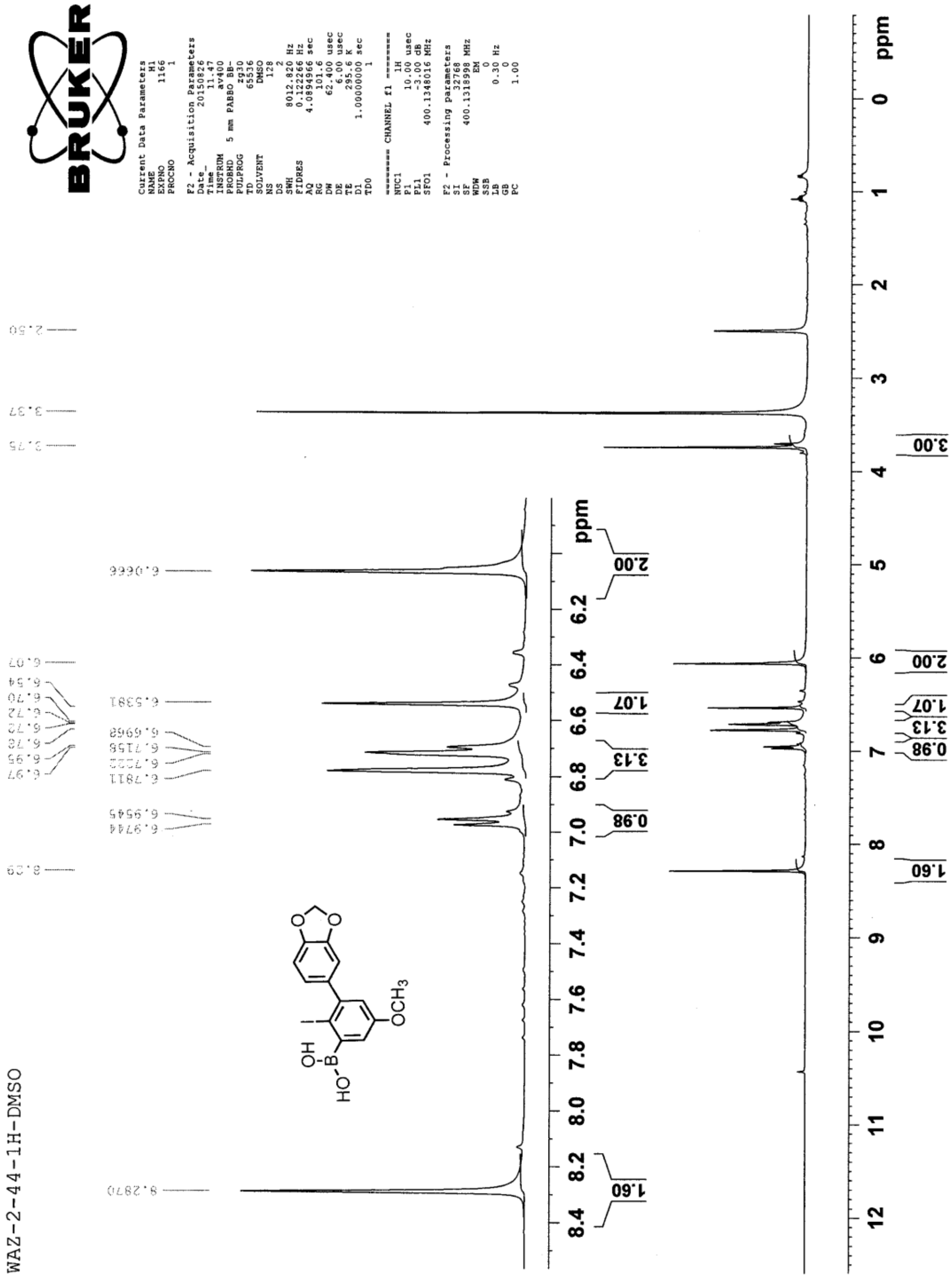


1.5.79 ^{11}B-NMR of (2-iodo-5-methoxy-4'-phenoxy-[1,1'-biphenyl]-3-yl)boronic acid (21$_A$) in d^6-acetone at 25 ^{o}C

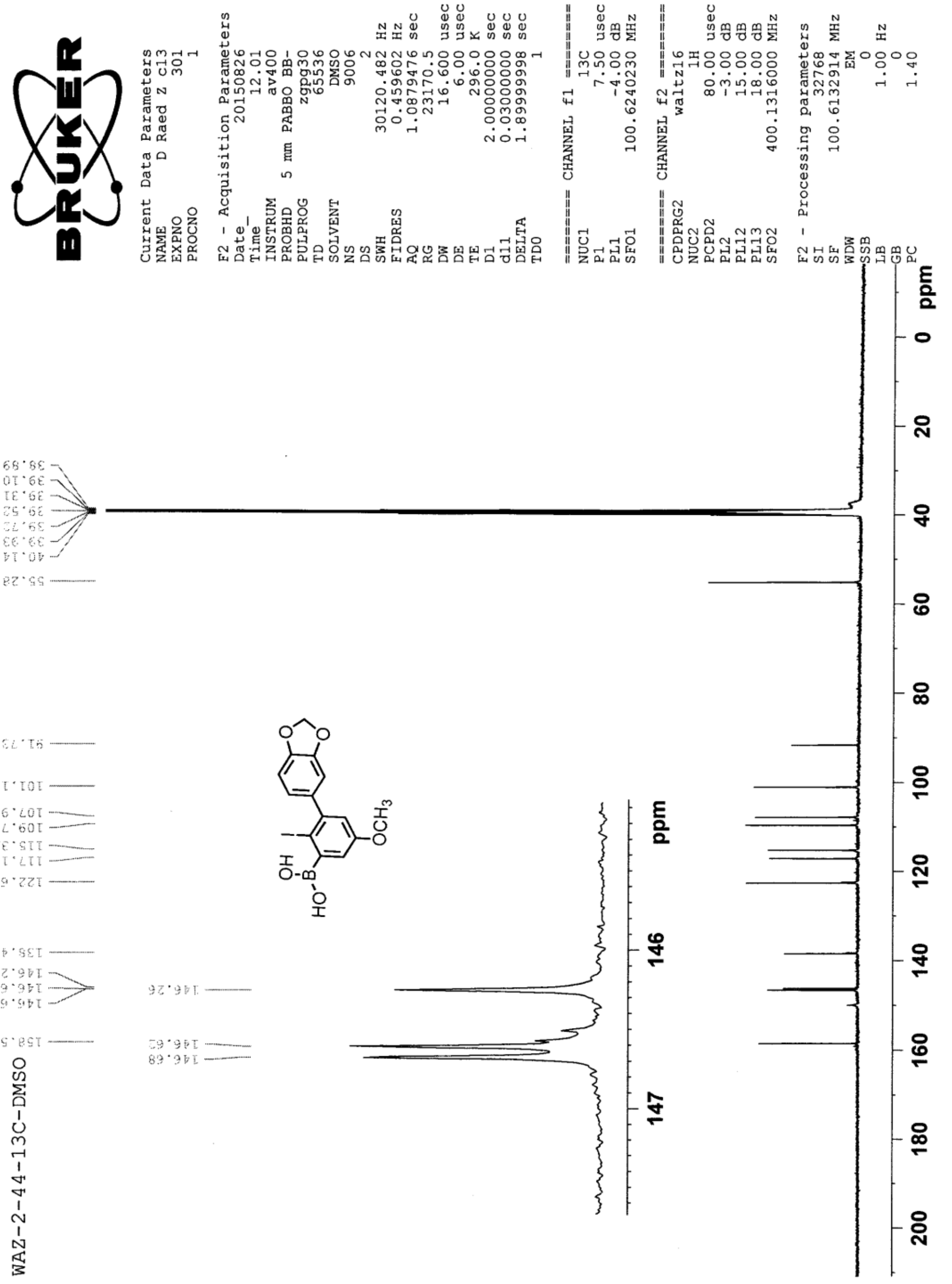


1.5.80

1.5.81 ^{1}H-NMR of (3-(benzo[d][1,3]dioxol-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (22_A) in d^6-DMSO at 25 °C.



1.5.82 ^{13}C–NMR of (3-(benzo[d][1,3]dioxol-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (22$_A$) in d^6-DMSO at 25 °C.



```
Current Data Parameters
NAME          D Raed Z c13
EXPNO                  301
PROCNO                   1

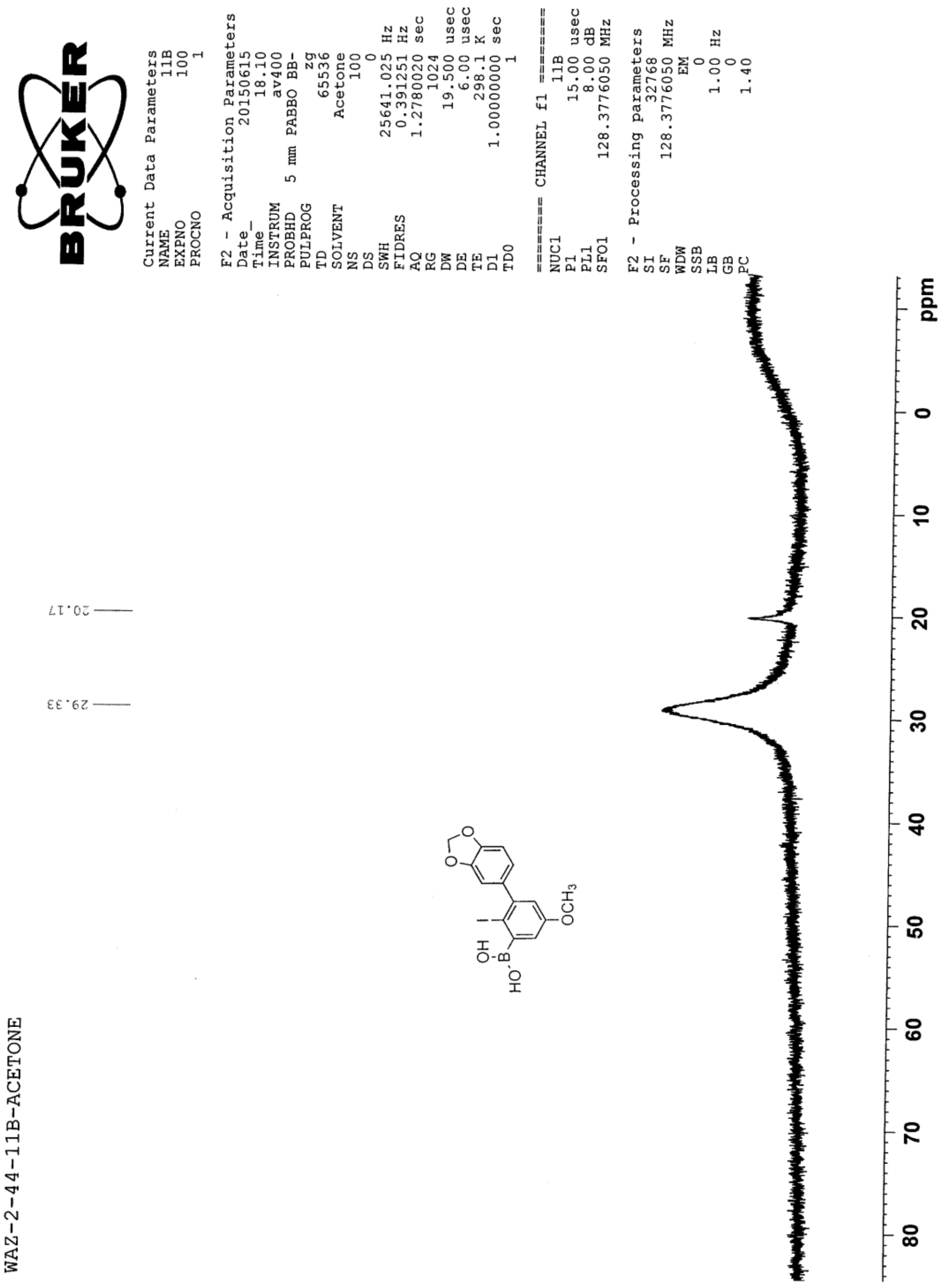
F2 - Acquisition Parameters
Date_             20150826
Time                 12.01
INSTRUM              av400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT               DMSO
NS                    9006
DS                       2
SWH              30120.482 Hz
FIDRES            0.459602 Hz
AQ               1.0879476 sec
RG                 23170.5
DW                  16.600 usec
DE                    6.00 usec
TE                   296.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      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             100.6132914 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

1.5.83 ^{11}B-NMR of (3-(benzo[d][1,3]dioxol-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (22$_A$) in d^6-acetone at 25 °C



```
Current Data Parameters
NAME                 11B
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150615
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INSTRUM            av400
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PULPROG               zg
TD                 65536
SOLVENT          Acetone
NS                   100
DS                     0
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FIDRES          0.391251 Hz
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RG                  1024
DW                19.500 usec
DE                  6.00 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                    1

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P1                 15.00 usec
PL1                 8.00 dB
SFO1         128.3776050 MHz

F2 - Processing parameters
SI                 32768
SF           128.3776050 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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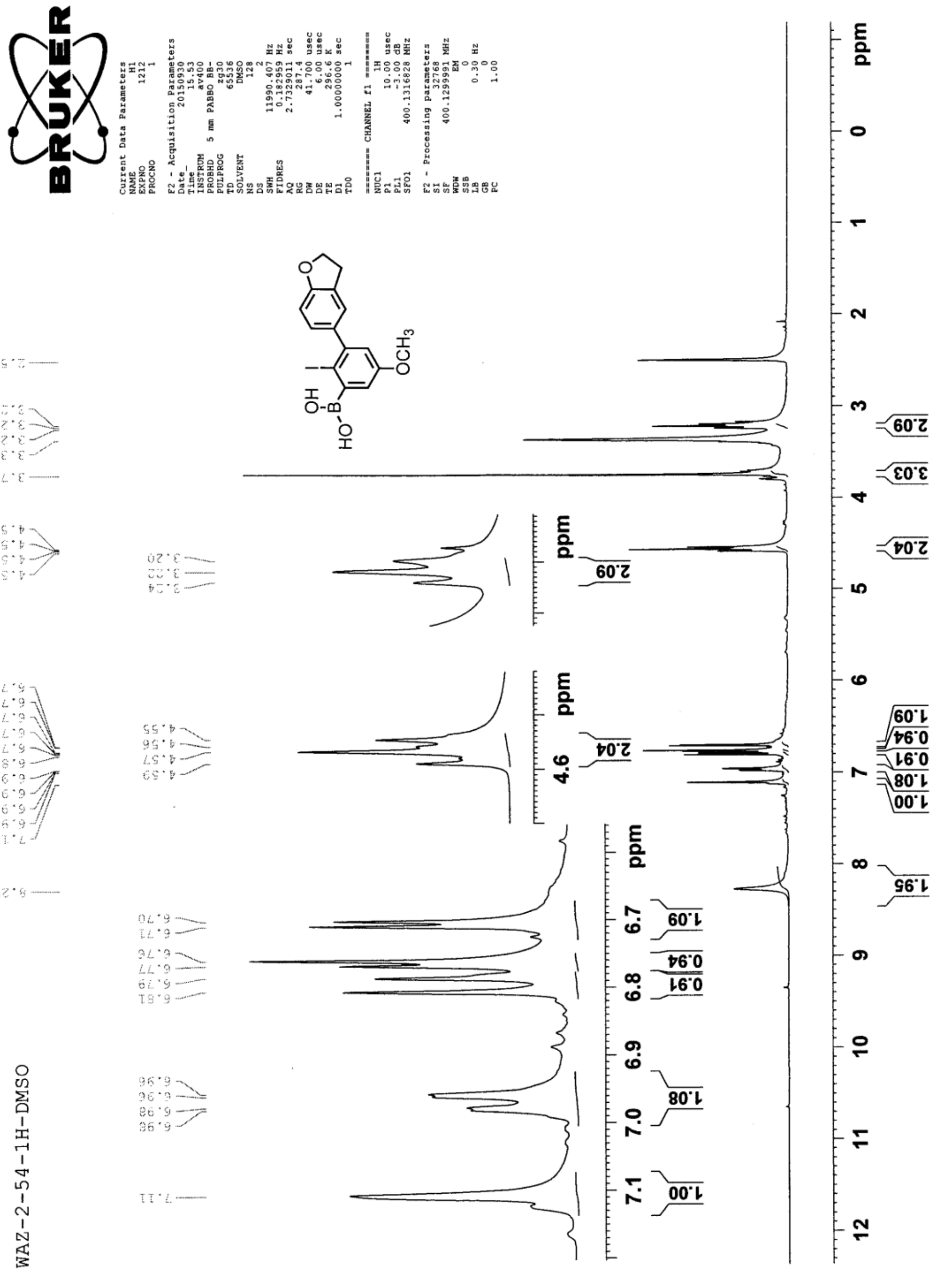
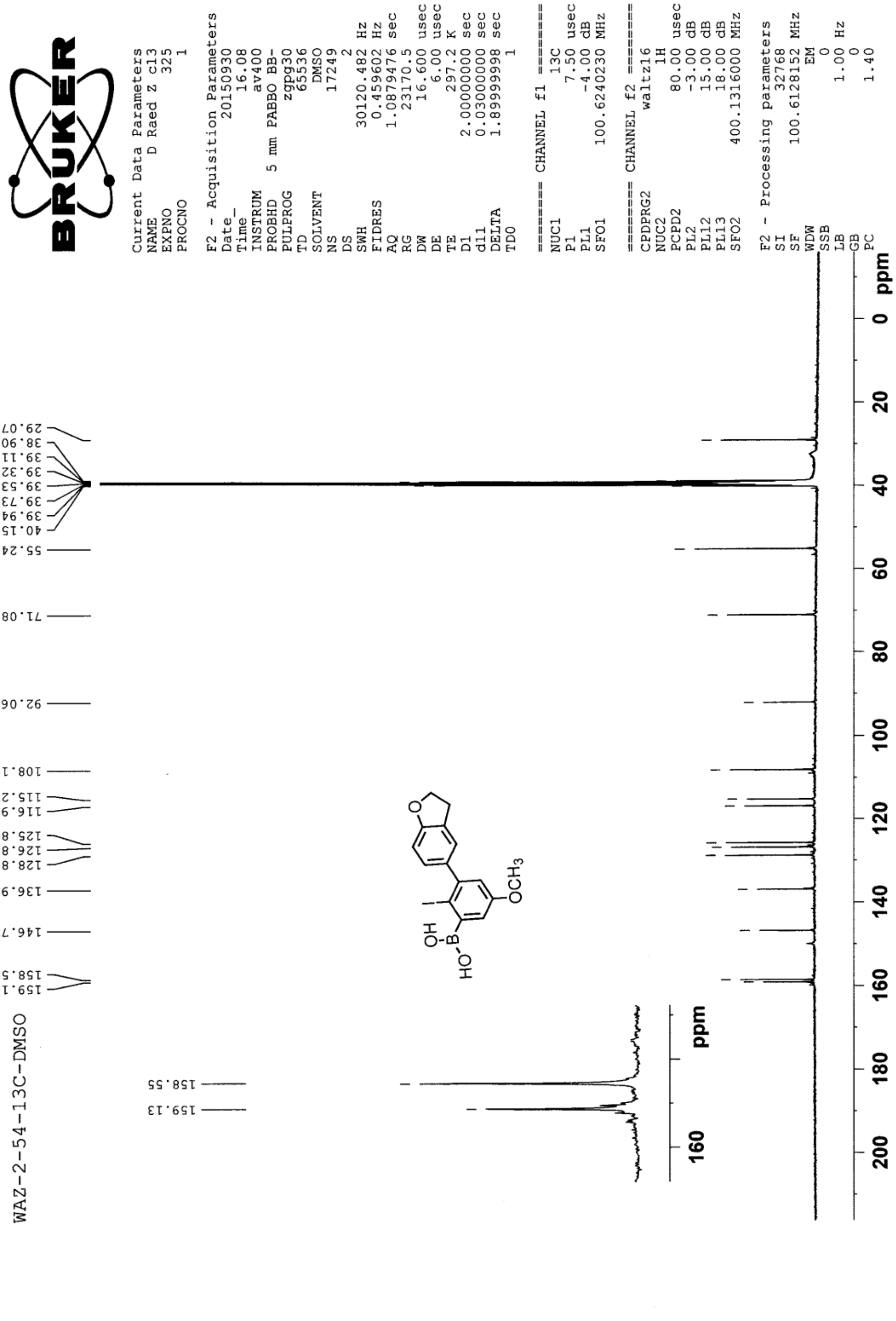
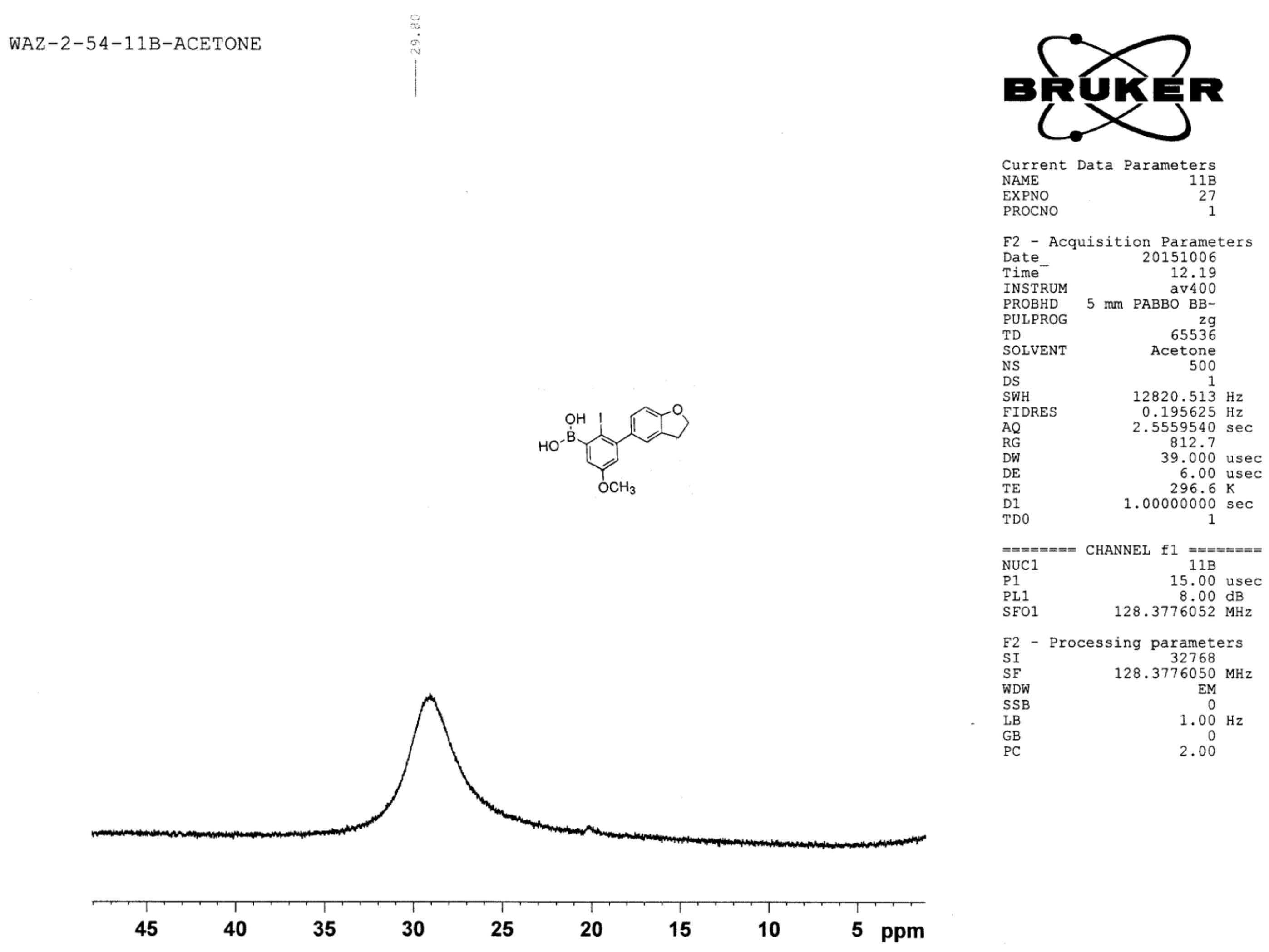
1.5.84 ^{1}H-NMR of (3-(2,3-dihydrobenzofuran-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (23_A) in d^6-DMSO at 25 °C.



1.5.85 ^{13}C–NMR of (3-(2,3-dihydrobenzofuran-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (23$_A$) in d^{6-}DMSO at 25 ^{o}C.



1.5.86 ^{11}B-NMR of (3-(2,3-dihydrobenzofuran-5-yl)-2-iodo-5-methoxyphenyl) boronic acid (23_A) in d^6-acetone at 25 °C



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EXPNO                 27
PROCNO                 1

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PULPROG               zg
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DS                     1
SWH            12820.513 Hz
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AQ             2.5559540 sec
RG                 812.7
DW                39.000 usec
DE                  6.00 usec
TE                 296.6 K
D1            1.00000000 sec
TD0                    1

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SFO1         128.3776052 MHz

F2 - Processing parameters
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WDW                   EM
SSB                    0
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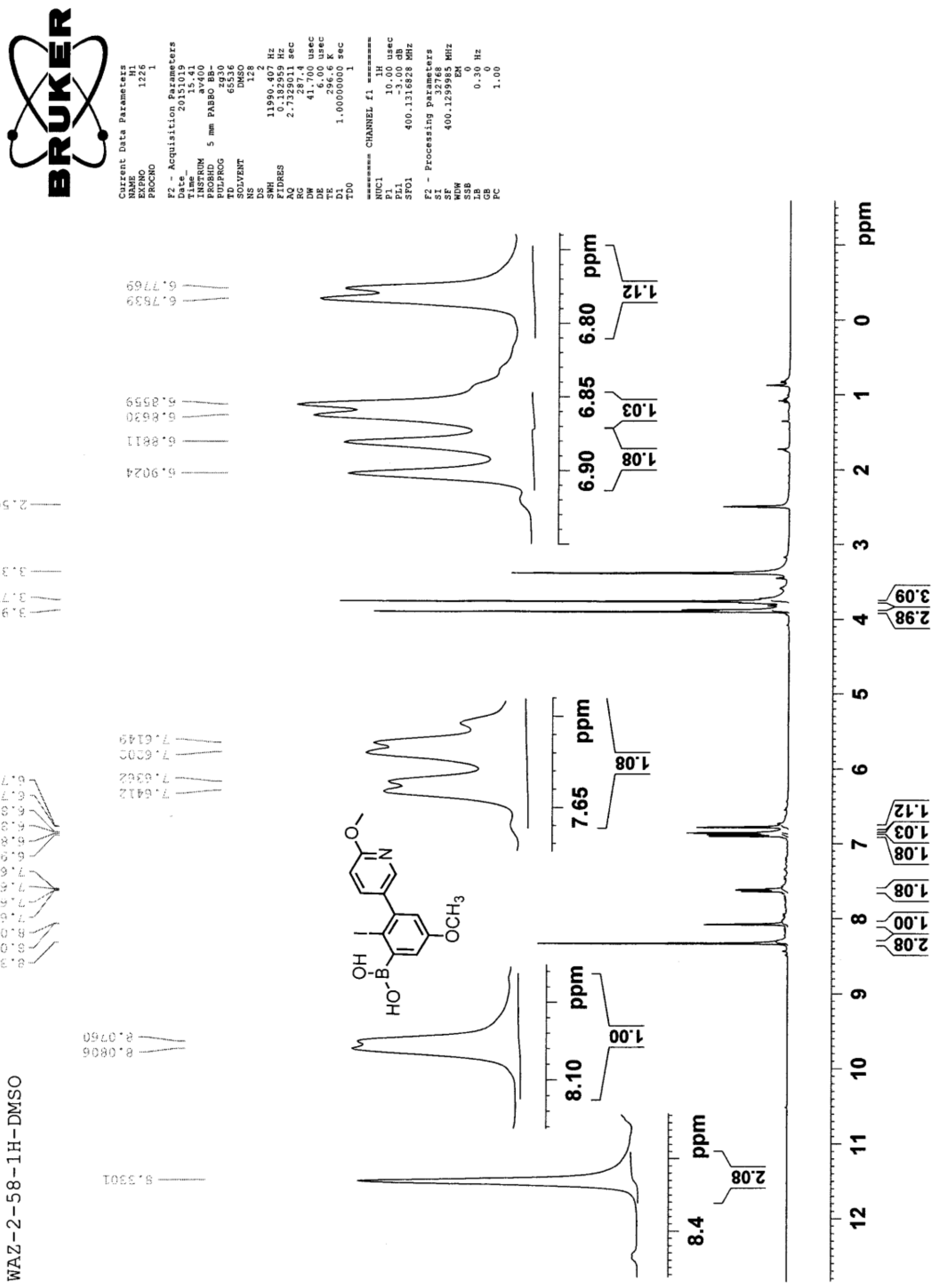
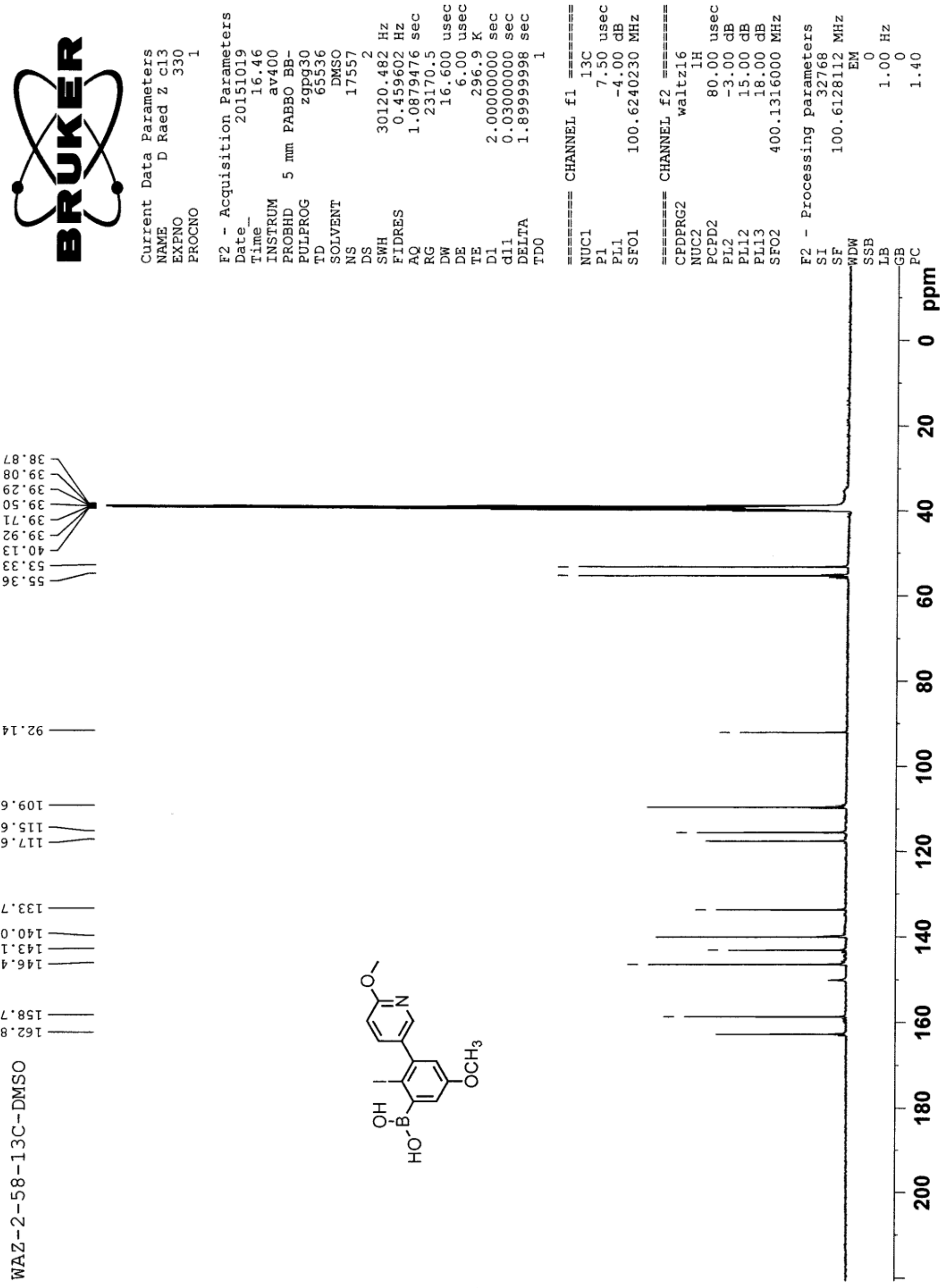
GB                     0
PC                  2.00
```

1.5.87 ^{1}H-NMR of 2-iodo-5-methoxy-3-(6-methoxypyridin-3-yl)phenyl) boronic acid (24_A) in d^6-DMSO at 25 ^{o}C.



1.5.88 ^{13}C–NMR of 2-iodo-5-methoxy-3-(6-methoxypyridin-3-yl)phenyl) boronic acid (24$_A$) in d^{6-}DMSO at 25 ^{o}C.



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EXPNO                330
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PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                 17557
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               296.9 K
D1        2.00000000 sec
d11       0.03000000 sec
DELTA     1.89999998 sec
TD0                    1

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P1              7.50 usec
PL1             -4.00 dB
SFO1     100.6240230 MHz

======== CHANNEL f2 ========
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NUC2                  1H
PCPD2          80.00 usec
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PL13            18.00 dB
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F2 - Processing parameters
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WDW                   EM
SSB                    0
LB               1.00 Hz
GB                     0
PC                  1.40
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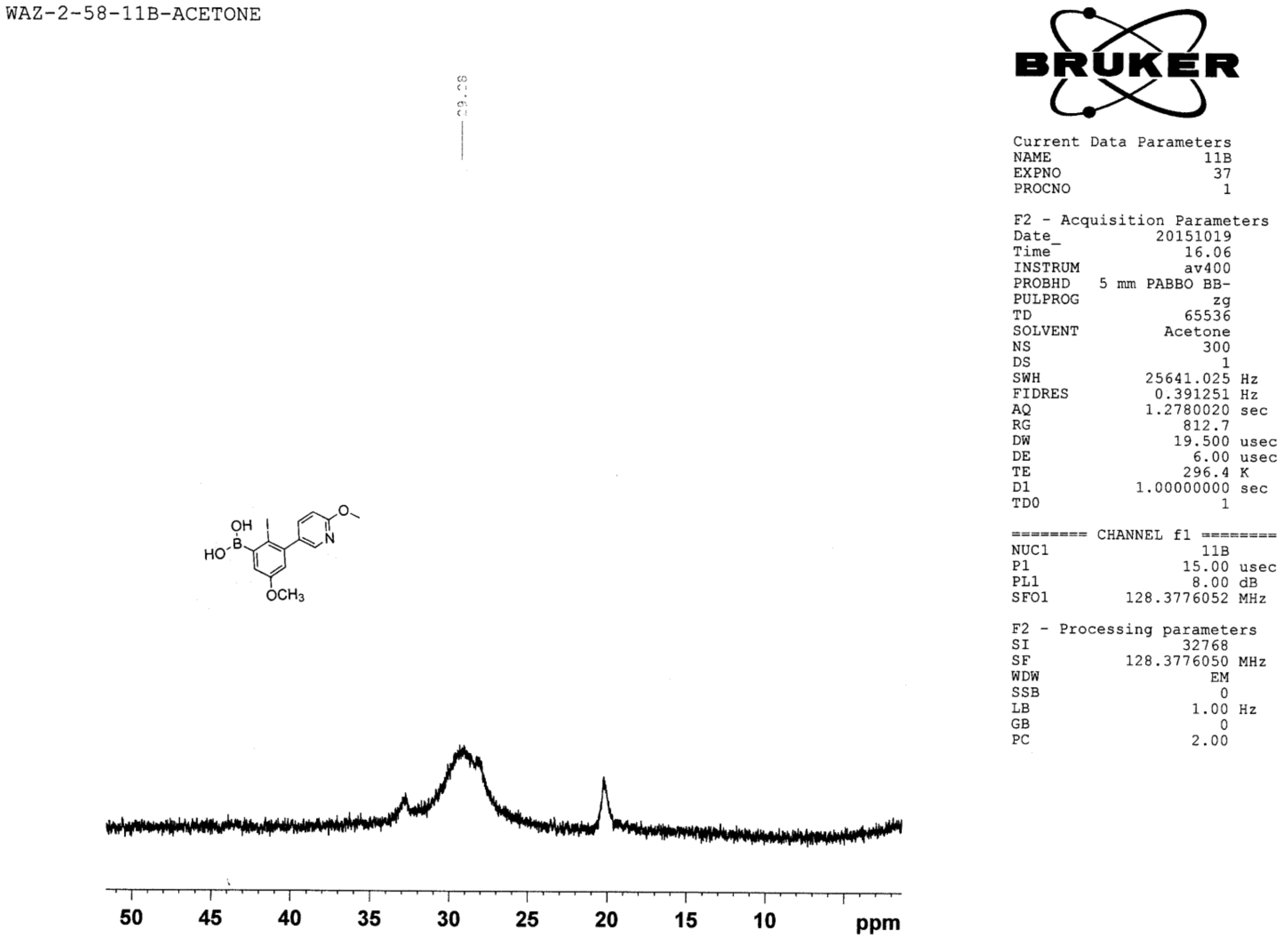
1.5.89 ^{11}B-NMR of 2-iodo-5-methoxy-3-(6-methoxypyridin-3-yl)phenyl) boronic acid (24_A) in d^6-acetone at 25 °C



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EXPNO                 37
PROCNO                 1

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PULPROG               zg
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SOLVENT          Acetone
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SWH            25641.025 Hz
FIDRES          0.391251 Hz
AQ             1.2780020 sec
RG                 812.7
DW                19.500 usec
DE                  6.00 usec
TE                 296.4 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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PL1                 8.00 dB
SFO1         128.3776052 MHz

F2 - Processing parameters
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SF           128.3776050 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00
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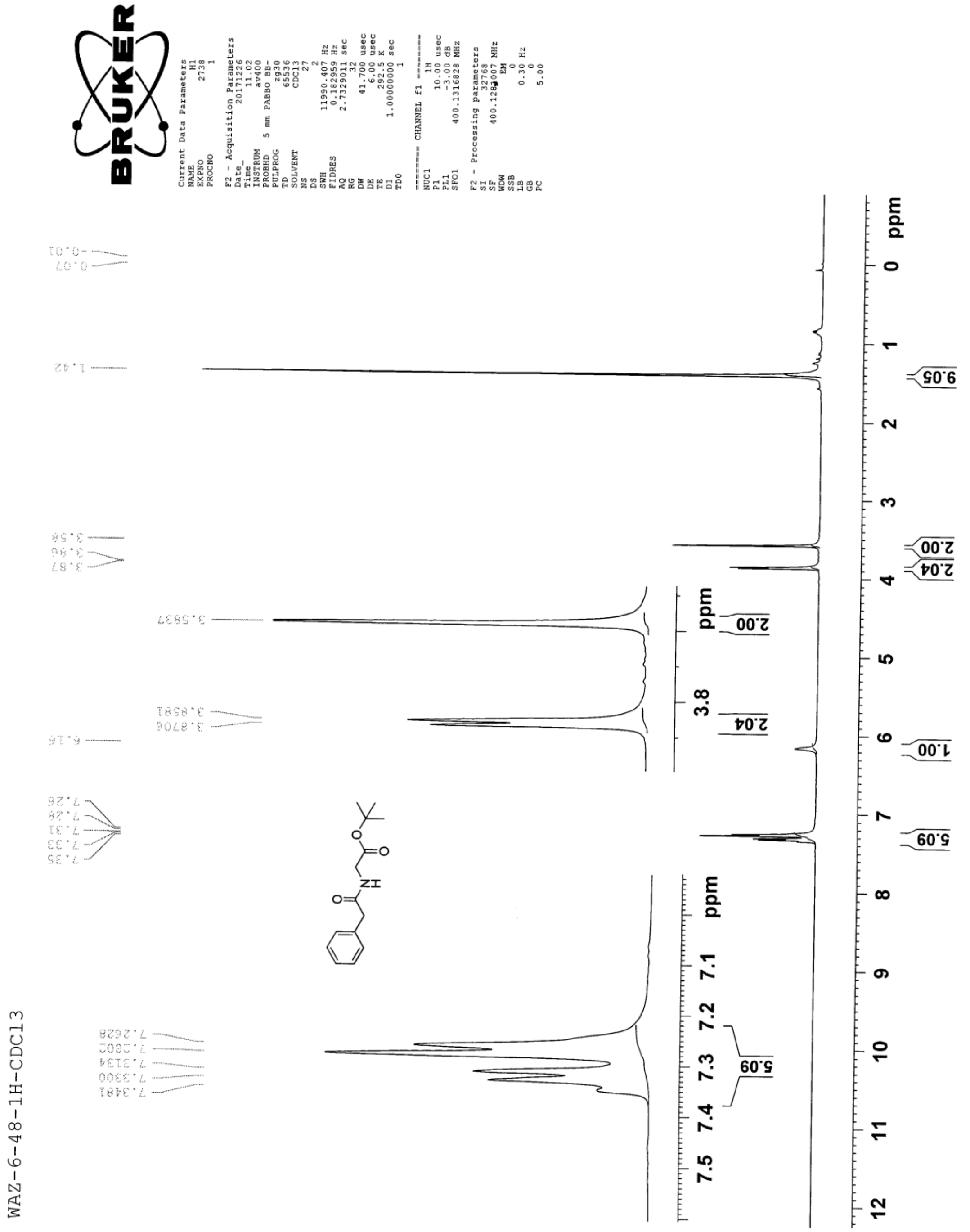
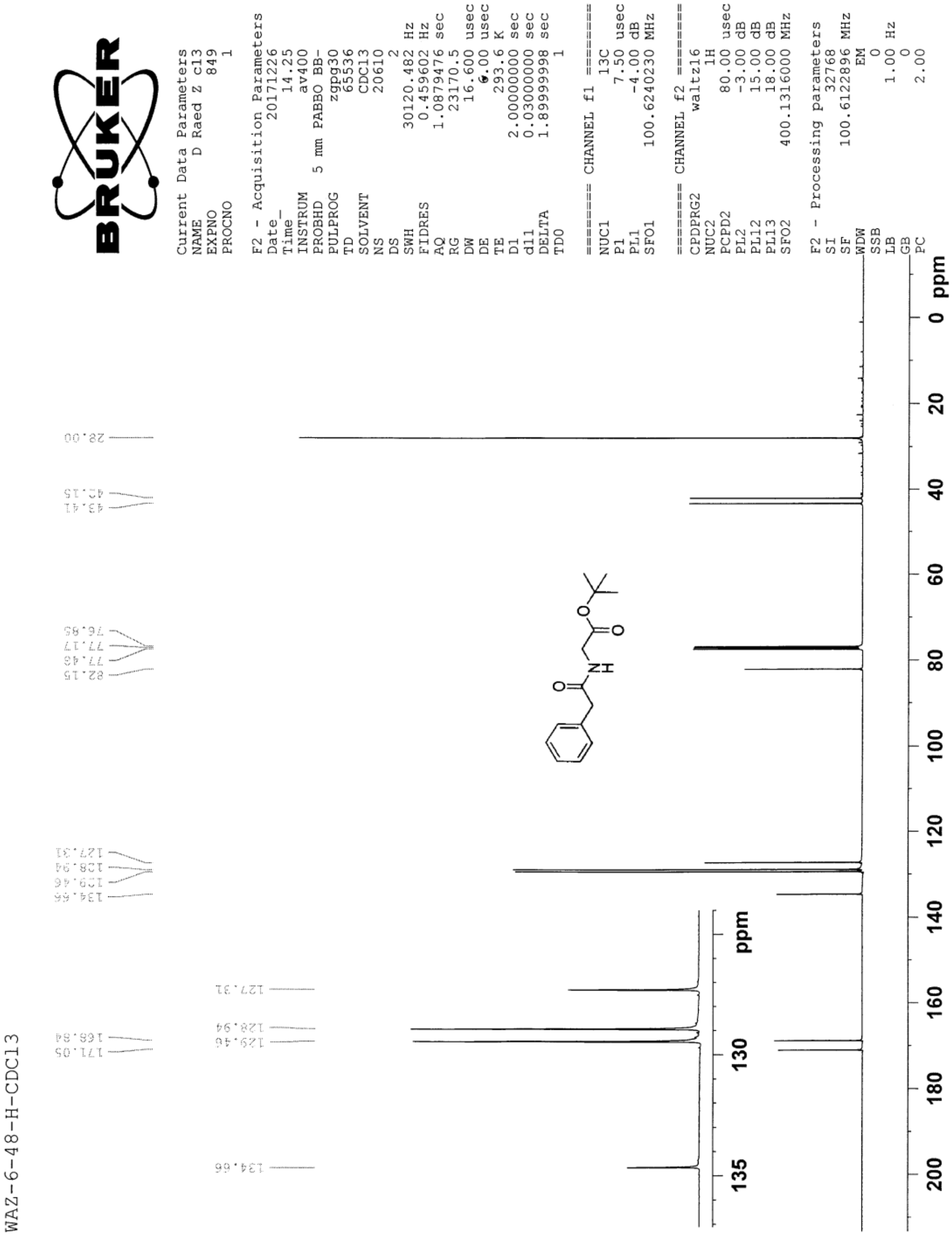
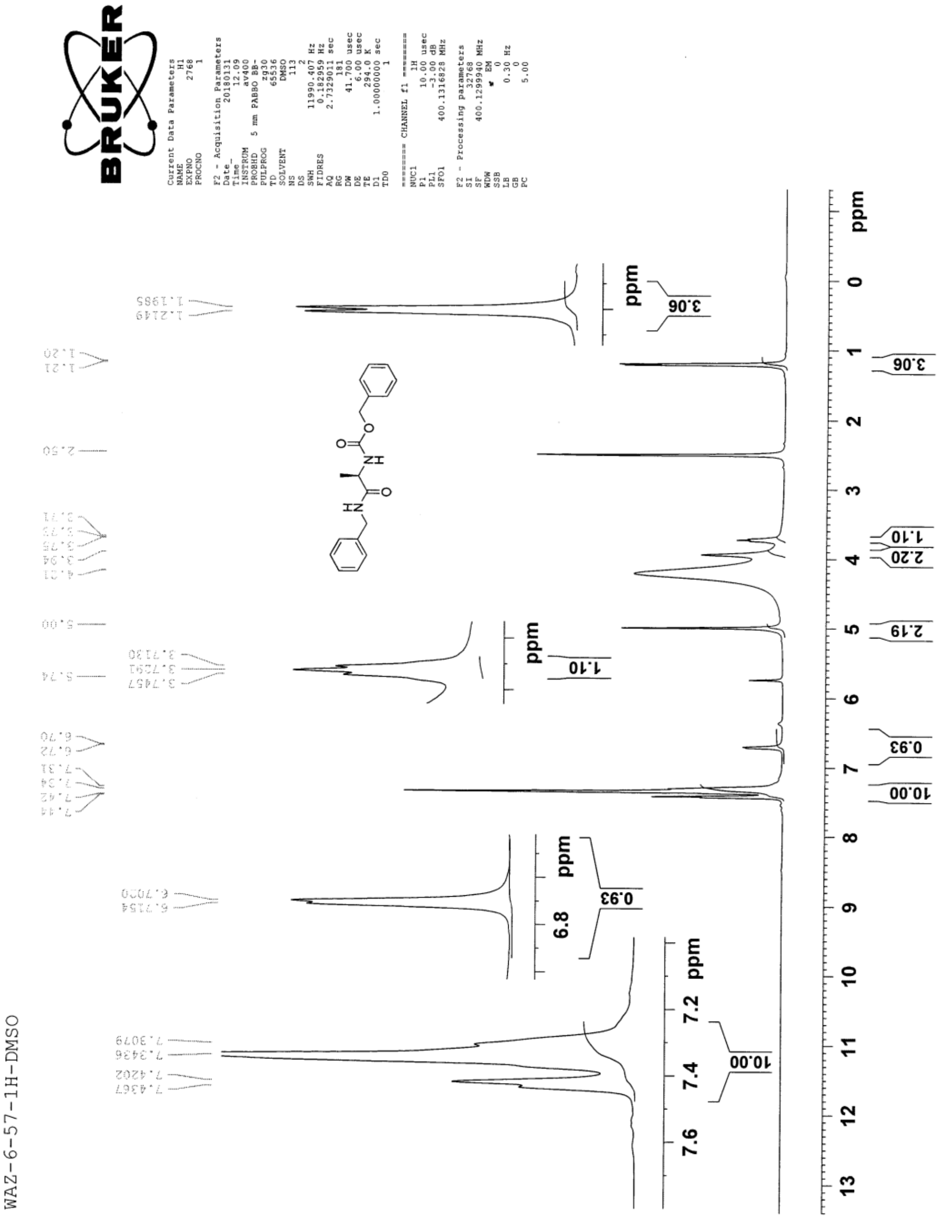
1.5.90 ^{1}H-NMR of tert-butyl (2-phenylacetyl)glycinate (26) in d-CDCl$_3$ at 25 ^{o}C



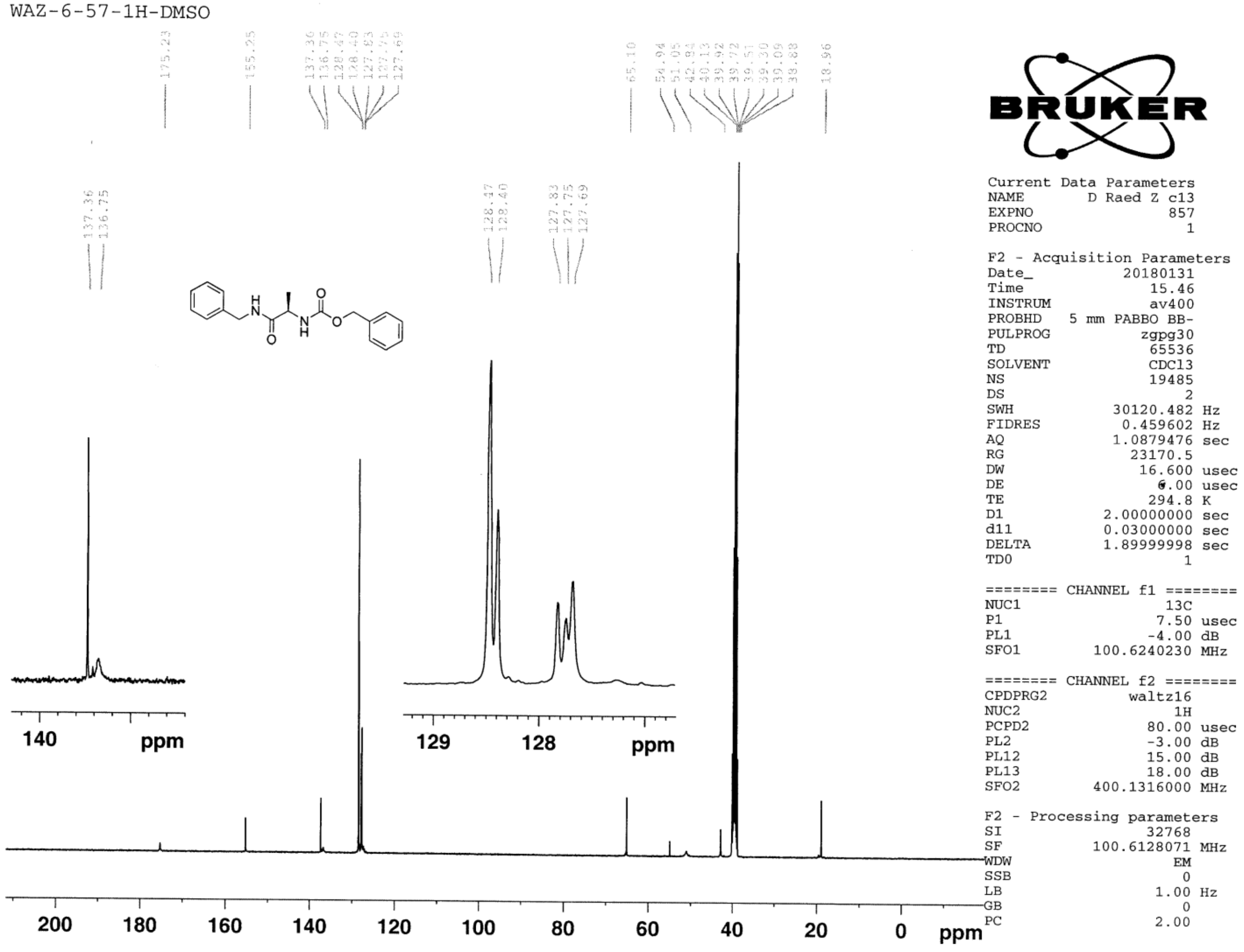
1.5.91 ^{13}C–NMR of tert-butyl (2-phenylacetyl)glycinate (26) in d-CDCl$_3$ at 25 ^{o}C.



1.5.92 ^{1}H-NMR of benzyl (S)-(1-(benzylamino)-1-oxopropan-2-yl)carbamate (28) in d^6- DMSO at 25 ^{o}C



1.5.93 ^{13}C–NMR of benzyl (S)-(1-(benzylamino)-1-oxopropan-2-yl)carbamate (28) in d^{6}-DMSO at 25 °C.



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PROCNO                 1

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TD                65536
SOLVENT           CDCl3
NS                19485
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                294.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   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          100.6128071 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00
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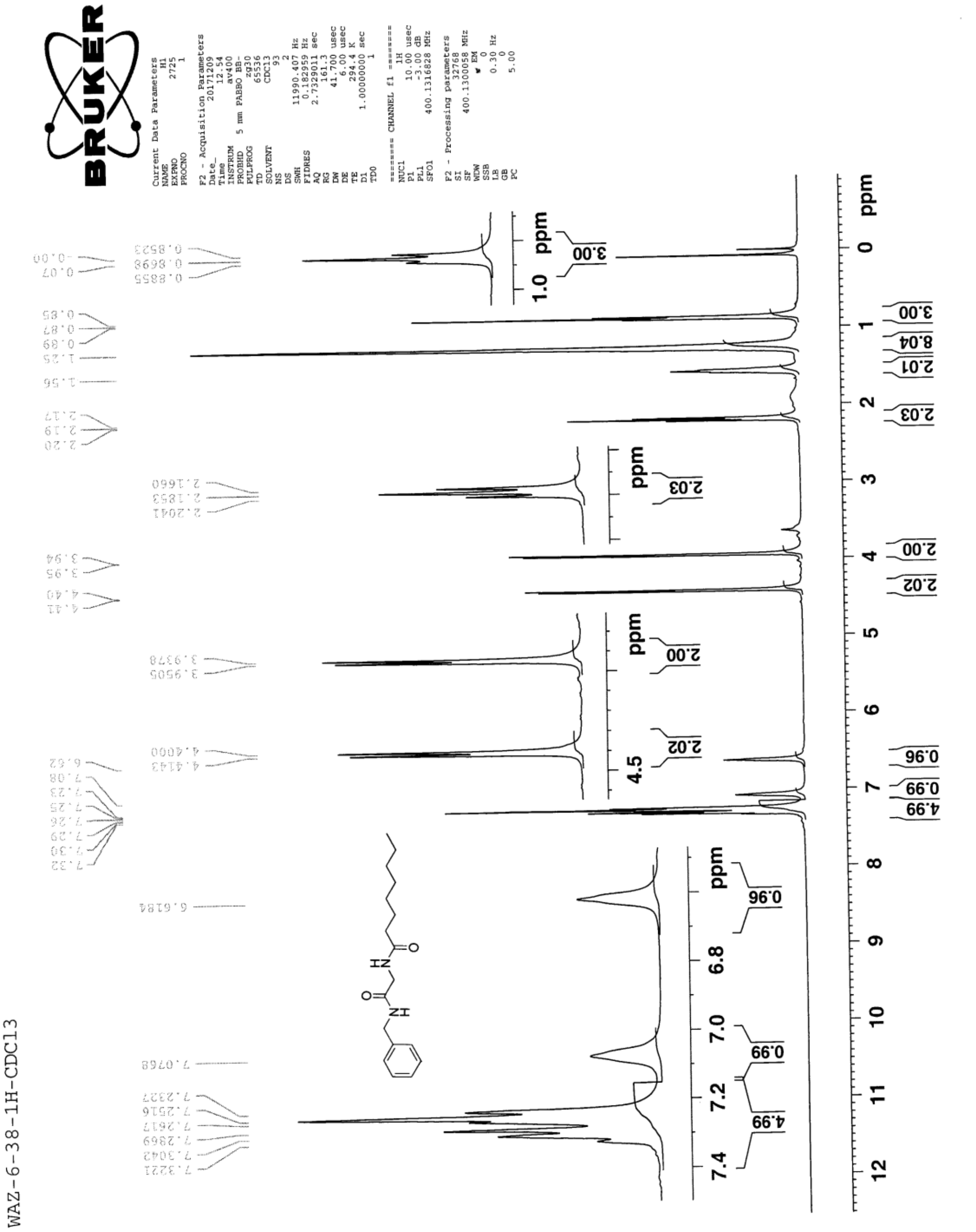
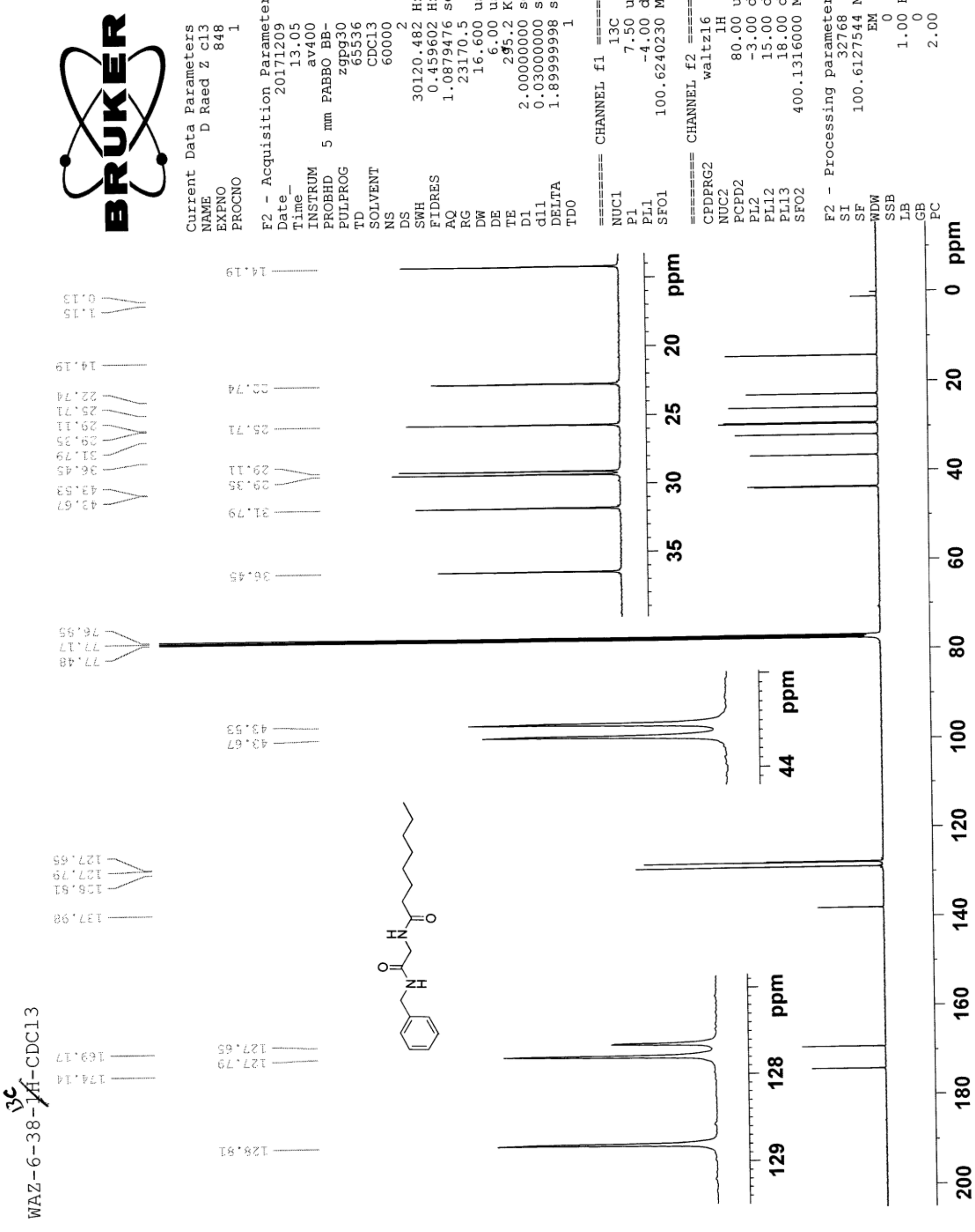
1.5.94 ¹H-NMR of N-(2-(benzylamino)-2-oxoethyl)octanamide (29) in d-CDCl₃ at 25 ᵒC



1.5.95 ^{13}C–NMR of N-(2-(benzylamino)-2-oxoethyl)octanamide (29) in d-$CDCl_3$ at 25 °C.



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PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                    60000
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
TD0                       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
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F2 - Processing parameters
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SSB                       0
LB                  1.00 Hz
GB                        0
PC                     2.00
```

1.5 X-ray of new compounds

1.5.1 X-ray data of (2-Iodobiphenyl-3-yl)boronic acid (**5_A**)

STRUCTURE REPORT

XCL Code: JUS1502 **Date:** 22 June 2015

Compound: (2-Iodobiphenyl-3-yl)boronic acid
Formula: $C_{12}H_{10}BIO_2$

Supervisor: R. M. Al-Zoubi, Jordan University of Science and Technology
Crystallographer: R. McDonald

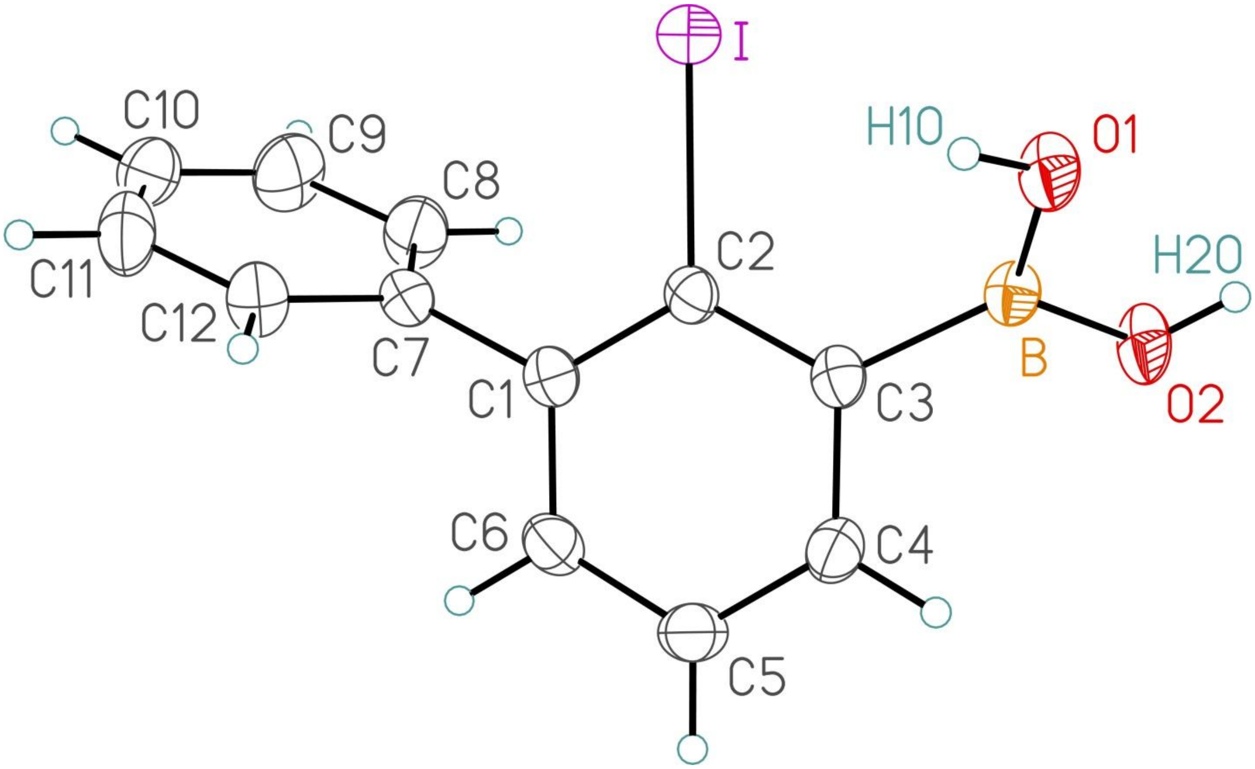
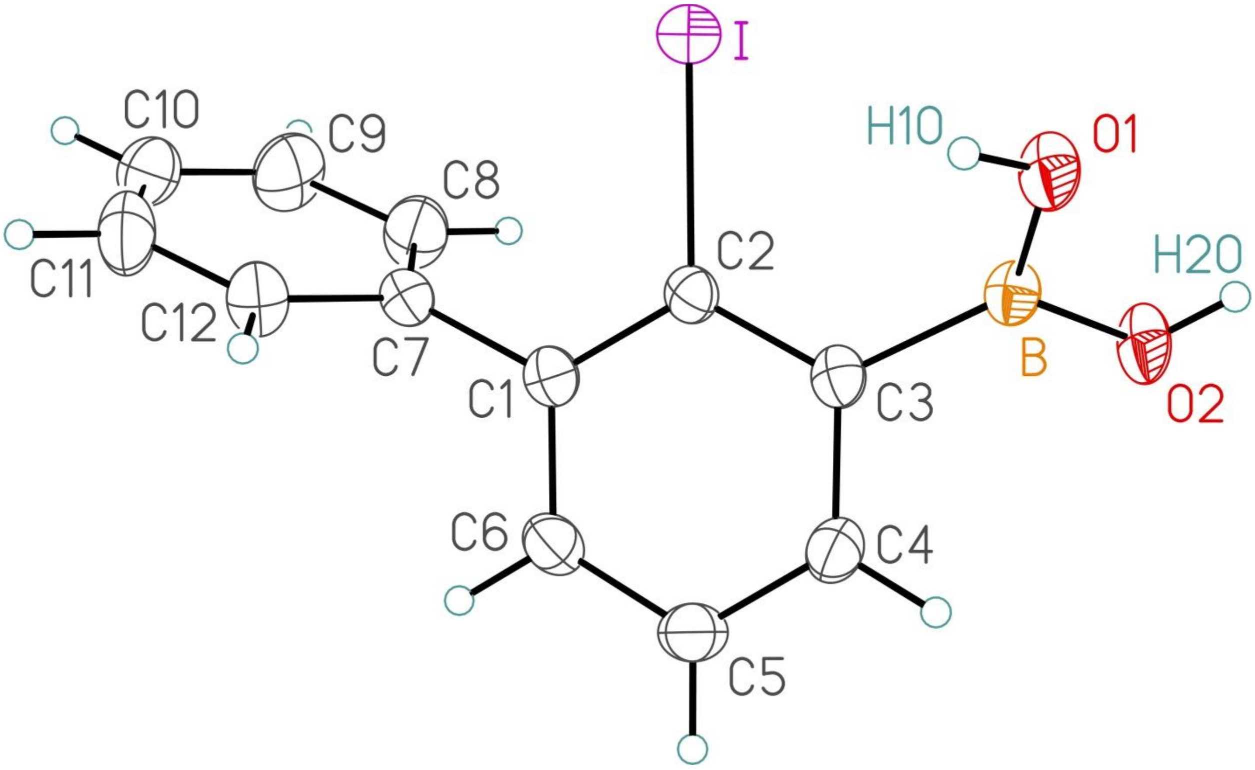
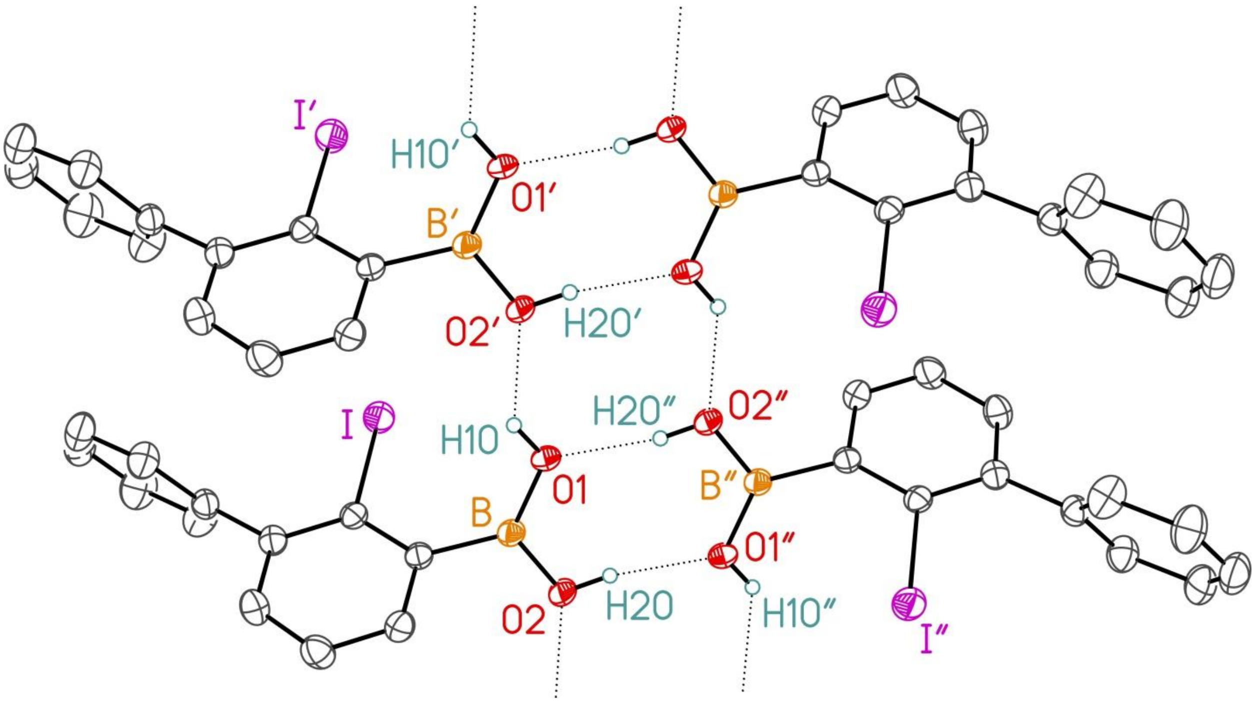


Figure Legends

Figure 1. Perspective view of the (2-iodobiphenyl-3-yl)boronic acid molecule showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Illustration of hydrogen-bonded interactions between adjacent (2-iodobiphenyl-3-yl)boronic acid molecules in the crystal lattice. Primed atoms are related to unprimed ones via the crystallographic symmetry operation (1+*x*, *y*, *z*). Double-primed atoms are related to unprimed ones via the crystallographic inversion center (0, 0, 0). The chain propagates in a direction parallel to the crystal unit cell's *a* axis.


I
H1O
O1
C10
C9
C8
C2
H2O
C11
C12
B
O2
C7
C1
C3
C6
C4
C5


I′
H1O′
O1′
B′
O2′
H2O′
I
H1O
H2O″
O2″
O1
B″
B
O1″
O2
H2O
H1O″
I″

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data

formula	$C_{12}H_{10}BIO_2$
formula weight	323.91
crystal dimensions (mm)	0.27 × 0.08 × 0.02
crystal system	monoclinic
space group	$P2_1/n$ (an alternate setting of $P2_1/c$ [No. 14])
unit cell parameters[a]	
a (Å)	4.9683 (12)
b (Å)	13.968 (3)
c (Å)	17.534 (4)
β (deg)	90.619 (3)
V (Å^3)	1216.8 (5)
Z	4
ρ_{calcd} (g cm^{-3})	1.768
μ (mm^{-1})	2.612

B. Data Collection and Refinement Conditions

diffractometer	Bruker PLATFORM/APEX II CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ω scans (0.3°) (25 s exposures)
data collection 2θ limit (deg)	56.85
total data collected	10367 ($-6 \le h \le 6$, $-18 \le k \le 18$, $-23 \le l \le 23$)
independent reflections	3014 ($R_{\text{int}} = 0.0494$)
number of observed reflections (*NO*)	2052 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	intrinsic phasing (*SHELXT-2014*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	0.9691–0.6365
data/restraints/parameters	3014 / 2[e] / 153
goodness-of-fit (S)[f] [all data]	0.990
final R indices[g]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0361
wR_2 [all data]	0.0790
largest difference peak and hole	0.712 and –0.581 e Å^{-3}

[a] Obtained from least-squares refinement of 2092 reflections with $5.48° < 2\theta < 41.30°$.

[b] Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[c] Sheldrick, G. M. *Acta Crystallogr.* **2015**, *A71*, 3–8.

[d] Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[e] The O1–H1O and O2–H2O distances were constrained to be 0.84(1) Å during refinement.

[f] $S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + 0.0314P)^2]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[g] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	x	y	z	U_{eq}, Å^2
I	0.29538(6)	0.18552(2)	0.21803(2)	0.05336(12)*
O1	0.2153(5)	0.0143(2)	0.07543(16)	0.0482(7)*
O2	-0.2468(5)	-0.0022(2)	0.07963(16)	0.0498(7)*
C1	0.0750(7)	0.0516(2)	0.3382(2)	0.0380(8)*
C2	0.0932(6)	0.0633(2)	0.25977(19)	0.0357(8)*
C3	-0.0182(6)	-0.0018(2)	0.2075(2)	0.0364(8)*
C4	-0.1548(7)	-0.0802(3)	0.2376(2)	0.0450(9)*
C5	-0.1668(8)	-0.0959(3)	0.3146(2)	0.0508(10)*
C6	-0.0503(8)	-0.0304(3)	0.3645(2)	0.0471(9)*
C7	0.1707(7)	0.1225(2)	0.3958(2)	0.0406(8)*
C8	0.0370(10)	0.2085(3)	0.4051(3)	0.0637(13)*
C9	0.1198(11)	0.2723(3)	0.4619(3)	0.0742(14)*
C10	0.3315(10)	0.2510(3)	0.5083(3)	0.0672(13)*
C11	0.4635(10)	0.1668(3)	0.4996(3)	0.0685(13)*
C12	0.3848(8)	0.1024(3)	0.4437(2)	0.0553(11)*
B	-0.0102(8)	0.0055(3)	0.1186(2)	0.0360(9)*
H1O	0.357(6)	0.009(3)	0.101(2)	0.090(18)
H2O	-0.223(10)	0.000(4)	0.0325(8)	0.10(2)

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
I	C2	2.115(3)	C3	B	1.563(5)
O1	B	1.365(5)	C4	C5	1.371(5)
O1	H1O	0.830(10)[a]	C5	C6	1.388(5)
O2	B	1.357(5)	C7	C8	1.383(5)
O2	H2O	0.836(10)[a]	C7	C12	1.377(5)
C1	C2	1.389(5)	C8	C9	1.394(6)
C1	C6	1.385(5)	C9	C10	1.356(7)
C1	C7	1.488(5)	C10	C11	1.356(6)
C2	C3	1.401(5)	C11	C12	1.384(6)
C3	C4	1.396(5)			

[a] Distance constrained during refinement.

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
B	O1	H1O	113(4)[b]	C1	C6	C5	121.5(4)
B	O2	H2O	111(4)[b]	C1	C7	C8	120.5(3)
C2	C1	C6	117.5(3)	C1	C7	C12	121.2(3)
C2	C1	C7	124.7(3)	C8	C7	C12	118.2(4)
C6	C1	C7	117.7(3)	C7	C8	C9	120.1(4)
I	C2	C1	118.3(3)	C8	C9	C10	120.6(4)
I	C2	C3	118.9(2)	C9	C10	C11	119.7(4)
C1	C2	C3	122.8(3)	C10	C11	C12	120.6(4)
C2	C3	C4	116.9(3)	C7	C12	C11	120.7(4)
C2	C3	B	126.6(3)	O1	B	O2	116.1(3)
C4	C3	B	116.5(3)	O1	B	C3	126.1(3)
C3	C4	C5	121.7(3)	O2	B	C3	117.7(3)
C4	C5	C6	119.5(4)				

[b] Angle includes O–H distance constrained during refinement.

Table 5. Hydrogen-Bonded Interactions

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A (deg)
O1–H1O···O2[a]	0.830(10)[c]	2.01(4)	2.683(3)	137(5)
O2–H2O···O1[b]	0.836(10)[c]	1.904(14)	2.730(4)	170(5)

[a]At 1+x, y, z. [b]At $\bar{x}$, $\bar{y}$, $\bar{z}$. [c]Distance constrained during refinement.

Table 6. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C6	C1	C2	I	-177.9(3)	B	C3	C4	C5	177.6(3)
C6	C1	C2	C3	2.6(5)	C2	C3	B	O1	55.5(5)
C7	C1	C2	I	4.6(4)	C2	C3	B	O2	-128.0(4)
C7	C1	C2	C3	-174.9(3)	C4	C3	B	O1	-125.8(4)
C2	C1	C6	C5	-3.5(5)	C4	C3	B	O2	50.7(5)
C7	C1	C6	C5	174.2(3)	C3	C4	C5	C6	2.7(6)
C2	C1	C7	C8	70.4(5)	C4	C5	C6	C1	0.9(6)
C2	C1	C7	C12	-113.0(4)	C1	C7	C8	C9	176.7(4)
C6	C1	C7	C8	-107.1(4)	C12	C7	C8	C9	0.0(7)
C6	C1	C7	C12	69.5(5)	C1	C7	C12	C11	-176.7(4)
I	C2	C3	C4	-178.6(2)	C8	C7	C12	C11	0.1(6)
I	C2	C3	B	0.1(4)	C7	C8	C9	C10	0.1(8)
C1	C2	C3	C4	0.8(5)	C8	C9	C10	C11	-0.2(8)
C1	C2	C3	B	179.5(3)	C9	C10	C11	C12	0.2(8)
C2	C3	C4	C5	-3.5(5)	C10	C11	C12	C7	-0.1(7)

Table 7. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I	0.05845(19)	0.05436(18)	0.04720(18)	0.00588(13)	-0.00248(13)	-0.01494(13)
O1	0.0210(13)	0.0824(19)	0.0413(16)	-0.0040(14)	-0.0021(12)	-0.0025(12)
O2	0.0217(12)	0.090(2)	0.0377(17)	-0.0062(15)	-0.0055(12)	0.0008(12)
C1	0.0373(19)	0.0400(19)	0.037(2)	-0.0001(16)	-0.0003(16)	0.0055(15)
C2	0.0296(18)	0.0411(19)	0.036(2)	0.0032(16)	-0.0029(15)	0.0019(14)
C3	0.0269(17)	0.047(2)	0.035(2)	-0.0006(16)	-0.0008(15)	0.0037(15)
C4	0.045(2)	0.048(2)	0.042(2)	-0.0077(18)	-0.0012(18)	-0.0052(17)
C5	0.061(3)	0.042(2)	0.050(3)	-0.0001(18)	0.009(2)	-0.0069(18)
C6	0.059(3)	0.045(2)	0.037(2)	0.0039(17)	0.0027(19)	0.0025(18)
C7	0.045(2)	0.043(2)	0.035(2)	0.0024(16)	-0.0007(17)	-0.0008(16)
C8	0.072(3)	0.049(2)	0.070(3)	-0.003(2)	-0.017(3)	0.008(2)
C9	0.097(4)	0.051(3)	0.075(4)	-0.023(3)	-0.006(3)	0.003(3)
C10	0.089(4)	0.062(3)	0.051(3)	-0.013(2)	-0.006(3)	-0.017(3)
C11	0.065(3)	0.093(4)	0.047(3)	-0.006(2)	-0.013(2)	-0.002(3)
C12	0.057(3)	0.059(2)	0.049(3)	-0.003(2)	-0.006(2)	0.005(2)
B	0.027(2)	0.044(2)	0.037(2)	-0.0047(18)	-0.0009(17)	0.0036(16)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 8. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	x	y	z	U_{eq}, Å^2
H4	-0.2416	-0.1238	0.2038	0.054
H5	-0.2542	-0.1513	0.3338	0.061
H6	-0.0566	-0.0421	0.4178	0.056
H8	-0.1114	0.2241	0.3729	0.076
H9	0.0270	0.3312	0.4680	0.089
H10	0.3871	0.2949	0.5467	0.081
H11	0.6115	0.1518	0.5322	0.082
H12	0.4796	0.0438	0.4382	0.066

1.5.2 X-ray data of (2-Iodo-5-methoxybiphenyl-3-yl)boronic acid (**14_A**)

STRUCTURE REPORT

XCL Code: JUS1511 **Date:** 11 December 2015

Compound: (2-Iodo-5-methoxybiphenyl-3-yl)boronic acid
Formula: $C_{13}H_{12}BIO_3$

Supervisor: R. M. Al-Zoubi, Jordan University of Science and Technology
Crystallographer: R. McDonald

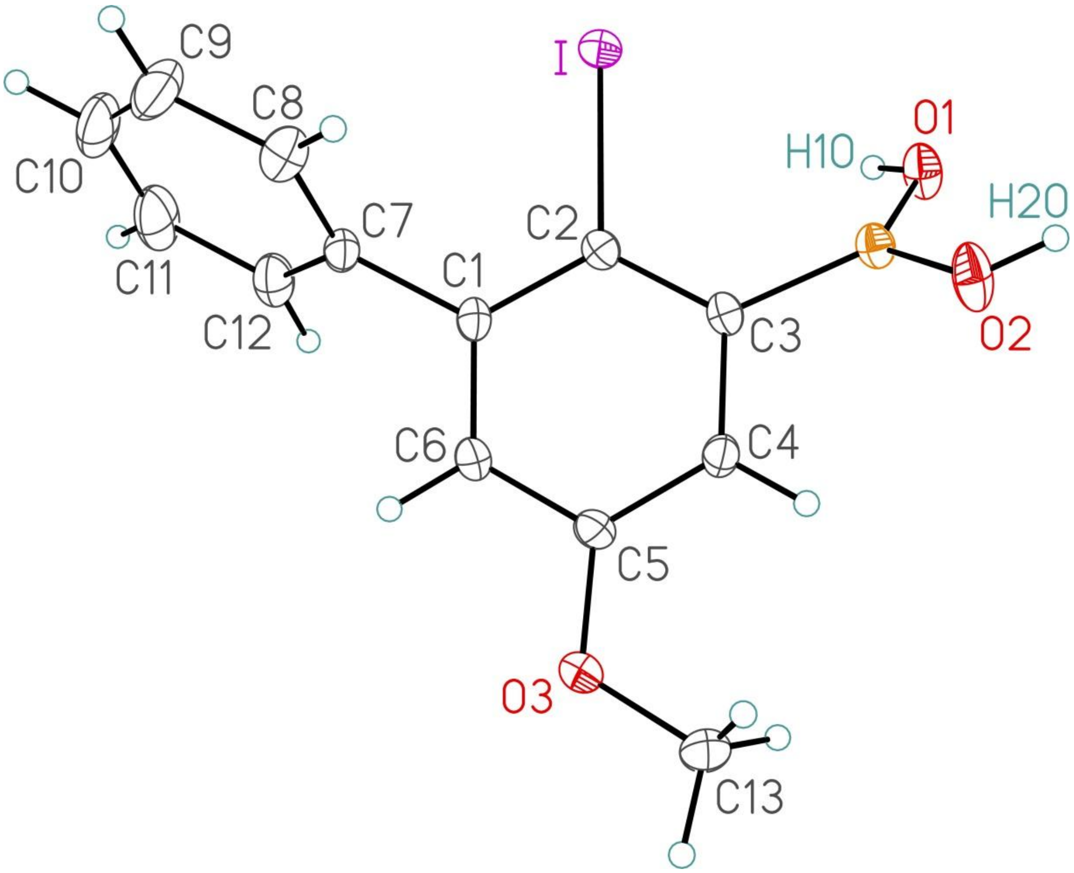
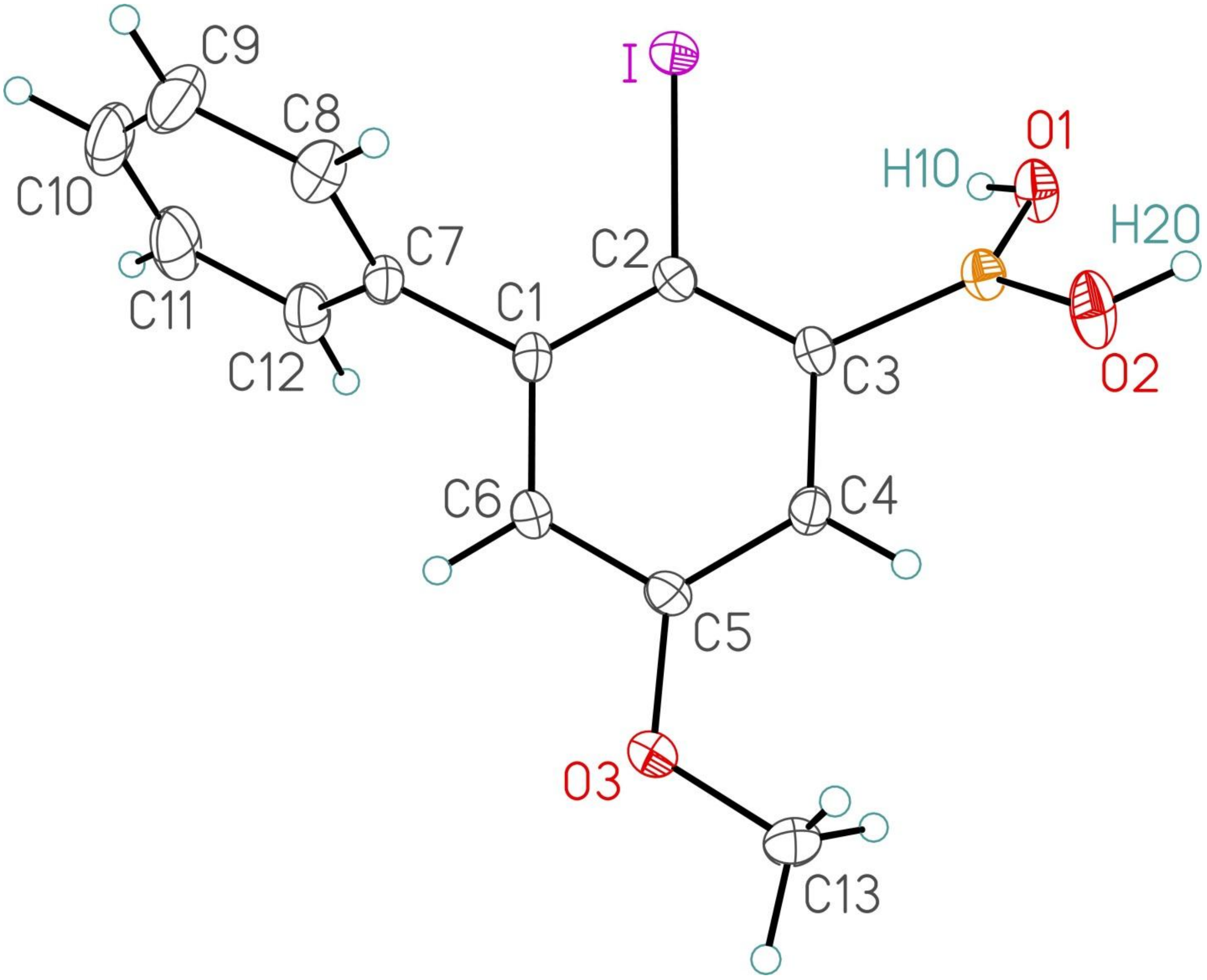
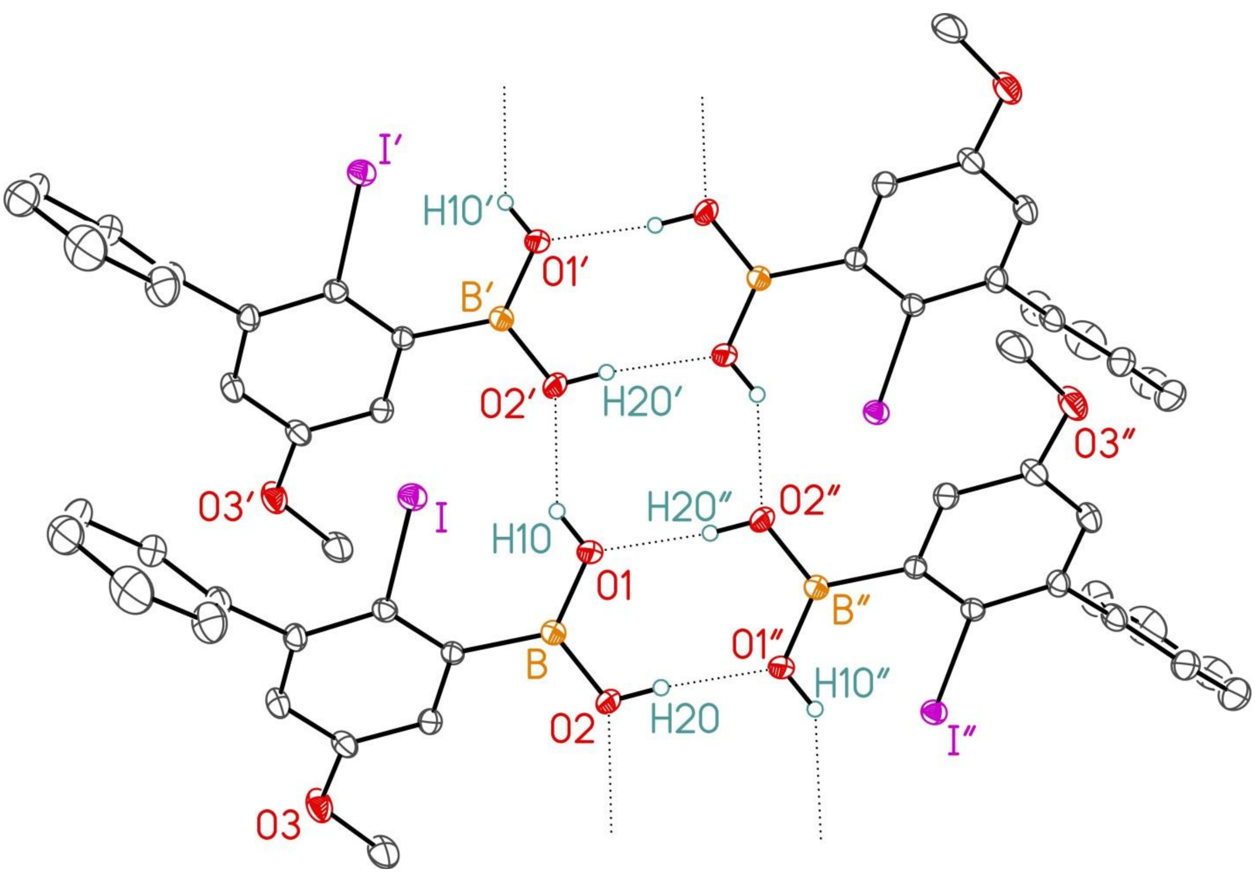


Figure Legends

Figure 1. Perspective view of the (2-iodo-5-methoxybiphenyl-3-yl)boronic acid molecule showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Illustration of hydrogen-bonded interactions (shown by dotted lines) between adjacent molecules in the crystal lattice. Primed atoms are related to unprimed ones via the crystallographic symmetry operation (x, $1+y$, z) (translation in a direction parallel to the crystallographic unit's cell's b axis). Double-primed atoms are related to unprimed ones via the crystallographic inversion center ($^{1}/4$, $^{1}/4$, $^{1}/4$).


C9
I
C8
O1
H1O
C10
H2O
C7
C2
C1
C11
C12
C3
O2
C6
C4
C5
O3
C13


I′
H1O′
O1′
B′
O2′
H2O′
O3′
I
H1O
O1
H2O″
O2″
O3″
B″
B
O1″
H1O″
O2
H2O
O3
I″

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data

formula	$C_{13}H_{12}BIO_3$
formula weight	353.94
crystal dimensions (mm)	0.56 × 0.06 × 0.03
crystal system	monoclinic
space group	*I*2/*a* (an alternate setting of *C*2/*c* [No. 15])
unit cell parameters[a]	
a (Å)	19.7878 (14)
b (Å)	5.0588 (4)
c (Å)	27.336 (2)
β (deg)	93.9416 (10)
V (Å^3)	2729.9 (4)
Z	8
ρ_{calcd} (g cm^{-3})	1.722
μ (mm^{-1})	2.342

B. Data Collection and Refinement Conditions

diffractometer	Bruker PLATFORM/APEX II CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ω scans (0.3°) (25 s exposures)
data collection 2θ limit (deg)	55.14
total data collected	22880 ($-25 \le h \le 25$, $-6 \le k \le 6$, $-35 \le l \le 35$)
independent reflections	3157 (R_{int} = 0.0370)
number of observed reflections (*NO*)	2747 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	Patterson/structure expansion (*DIRDIF–2008*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[d])
absorption correction method	multi-scan (*TWINABS*)
range of transmission factors	0.7967–0.6605
data/restraints/parameters	3157 / 2[e] / 173
goodness-of-fit (S)[f] [all data]	1.033
final R indices[g]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0264
wR_2 [all data]	0.0625
largest difference peak and hole	0.976 and –0.482 e Å^{-3}

[a] Obtained from least-squares refinement of 8347 reflections with 4.92° < 2θ < 55.12°.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[b] Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker. The crystal used for data collection was found to display non-merohedral twinning. Both components of the twin were indexed with the program *CELL_NOW* (Bruker AXS Inc., Madison, WI, 2004). The second twin component can be related to the first component by 180º rotation about the [1 0 0] axis in both real space and reciprocal space. Integrated intensities for the reflections from the two components were written into a *SHELXL-2014* HKLF 5 reflection file with the data integration program *SAINT* (version 8.34A), using all reflection data (exactly overlapped, partially overlapped and non-overlapped). The refined value of the twin fraction (*SHELXL-2014* BASF parameter) was 0.1934(9).

[c] Beurskens, P. T.; Beurskens, G.; de Gelder, R.; Smits, J. M. M.; Garcia-Granda, S.; Gould, R. O. (2008). The *DIRDIF-2008* program system. Crystallography Laboratory, Radboud University Nijmegen, The Netherlands.

[d] Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[e] The O1–H1O and O2–H2O distances were constrained to be 0.84(1) Å during refinement.

[f] $S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0303P)^2 + 4.1548P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[g] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	*x*	*y*	*z*	U_{eq}, Å^2
I	0.02560(2)	0.63092(4)	0.20447(2)	0.02817(7)*
O1	0.19356(12)	0.4661(4)	0.21985(10)	0.0357(6)*
O2	0.19887(13)	0.0117(5)	0.21119(10)	0.0404(6)*
O3	0.09875(13)	-0.1070(5)	0.03209(9)	0.0425(6)*
C1	0.00555(16)	0.3339(6)	0.10785(11)	0.0264(6)*
C2	0.05223(15)	0.3677(5)	0.14882(11)	0.0236(6)*
C3	0.11399(15)	0.2328(6)	0.15281(11)	0.0228(6)*
C4	0.12944(15)	0.0651(6)	0.11418(12)	0.0265(6)*
C5	0.08609(16)	0.0442(6)	0.07214(12)	0.0294(7)*
C6	0.02471(16)	0.1790(6)	0.06936(12)	0.0305(7)*
C7	-0.06452(16)	0.4419(7)	0.10540(12)	0.0314(7)*
C8	-0.1113(2)	0.3375(8)	0.13557(16)	0.0482(10)*
C9	-0.1774(2)	0.4298(11)	0.1333(2)	0.0665(14)*
C10	-0.1961(2)	0.6293(10)	0.1011(2)	0.0664(15)*
C11	-0.1508(2)	0.7343(9)	0.07061(19)	0.0600(13)*
C12	-0.08481(19)	0.6378(7)	0.07230(15)	0.0417(9)*
C13	0.1575(2)	-0.2689(9)	0.03591(16)	0.0515(11)*
B	0.16917(18)	0.2426(7)	0.19709(13)	0.0268(7)*
H1O	0.181(2)	0.610(5)	0.2080(16)	0.063(15)
H2O	0.2283(17)	0.026(9)	0.2345(11)	0.065(15)

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
I	C2	2.116(3)	C3	B	1.574(4)
O1	B	1.363(4)	C4	C5	1.390(4)
O2	B	1.352(4)	C5	C6	1.390(5)
O3	C5	1.373(4)	C7	C8	1.386(5)
O3	C13	1.420(4)	C7	C12	1.383(5)
C1	C2	1.412(4)	C8	C9	1.385(6)
C1	C6	1.386(4)	C9	C10	1.373(7)
C1	C7	1.488(4)	C10	C11	1.373(7)
C2	C3	1.397(4)	C11	C12	1.391(6)
C3	C4	1.404(4)			

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C5	O3	C13	117.1(3)	C4	C5	C6	119.7(3)
C2	C1	C6	118.2(3)	C1	C6	C5	121.3(3)
C2	C1	C7	123.2(3)	C1	C7	C8	119.5(3)
C6	C1	C7	118.4(3)	C1	C7	C12	121.4(3)
I	C2	C1	117.7(2)	C8	C7	C12	119.1(3)
I	C2	C3	120.7(2)	C7	C8	C9	120.8(4)
C1	C2	C3	121.6(3)	C8	C9	C10	119.3(5)
C2	C3	C4	118.1(3)	C9	C10	C11	120.9(4)
C2	C3	B	127.0(3)	C10	C11	C12	119.7(4)
C4	C3	B	115.0(3)	C7	C12	C11	120.2(4)
C3	C4	C5	120.9(3)	O1	B	O2	117.0(3)
O3	C5	C4	124.5(3)	O1	B	C3	125.6(3)
O3	C5	C6	115.9(3)	O2	B	C3	117.2(3)

Table 5. Hydrogen-Bonded Interactions

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A (deg)
O1–H1O···O2[a]	0.830(10)[c]	2.06(3)	2.773(3)	143(4)
O2–H2O···O1[b]	0.836(10)[c]	1.920(13)	2.747(3)	170(5)

[a] At *x*, 1+*y*, *z*. [b] At 1/2–*x*, 1/2–*y*, 1/2–*z*. [c] Distance constrained during refinement.

Table 6. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C13	O3	C5	C4	5.5(5)	B	C3	C4	C5	-178.0(3)
C13	O3	C5	C6	-173.4(3)	C2	C3	B	O1	-47.4(5)
C6	C1	C2	I	174.8(2)	C2	C3	B	O2	138.2(3)
C6	C1	C2	C3	-5.2(4)	C4	C3	B	O1	133.8(3)
C7	C1	C2	I	-9.2(4)	C4	C3	B	O2	-40.5(4)
C7	C1	C2	C3	170.8(3)	C3	C4	C5	O3	177.4(3)
C2	C1	C6	C5	4.6(5)	C3	C4	C5	C6	-3.8(5)
C7	C1	C6	C5	-171.6(3)	O3	C5	C6	C1	178.7(3)
C2	C1	C7	C8	-68.4(4)	C4	C5	C6	C1	-0.2(5)
C2	C1	C7	C12	114.1(4)	C1	C7	C8	C9	-178.5(4)
C6	C1	C7	C8	107.5(4)	C12	C7	C8	C9	-0.9(6)
C6	C1	C7	C12	-70.0(4)	C1	C7	C12	C11	179.7(3)
I	C2	C3	C4	-178.6(2)	C8	C7	C12	C11	2.2(6)
I	C2	C3	B	2.6(4)	C7	C8	C9	C10	-0.8(7)
C1	C2	C3	C4	1.4(4)	C8	C9	C10	C11	1.3(7)
C1	C2	C3	B	-177.4(3)	C9	C10	C11	C12	-0.1(7)
C2	C3	C4	C5	3.2(5)	C10	C11	C12	C7	-1.7(6)

Table 7. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I	0.03153(11)	0.02701(10)	0.02615(10)	-0.00192(8)	0.00338(8)	-0.00053(9)
O1	0.0363(13)	0.0208(11)	0.0474(15)	-0.0045(10)	-0.0148(11)	0.0009(10)
O2	0.0431(14)	0.0196(11)	0.0550(16)	0.0050(11)	-0.0223(13)	0.0000(10)
O3	0.0358(14)	0.0569(16)	0.0337(13)	-0.0186(12)	-0.0057(10)	0.0098(12)
C1	0.0201(15)	0.0295(15)	0.0290(15)	0.0013(12)	-0.0019(12)	-0.0020(12)
C2	0.0251(15)	0.0210(13)	0.0247(14)	0.0004(12)	0.0003(11)	-0.0040(12)
C3	0.0236(15)	0.0193(13)	0.0249(15)	0.0026(12)	-0.0027(12)	-0.0046(12)
C4	0.0211(15)	0.0271(14)	0.0309(16)	-0.0012(12)	-0.0006(12)	0.0010(12)
C5	0.0283(16)	0.0323(16)	0.0272(16)	-0.0081(13)	0.0000(13)	-0.0027(13)
C6	0.0236(16)	0.0391(18)	0.0282(16)	-0.0043(13)	-0.0031(12)	-0.0023(13)
C7	0.0221(15)	0.0367(17)	0.0348(18)	-0.0081(14)	-0.0024(13)	0.0007(13)
C8	0.033(2)	0.060(3)	0.052(2)	-0.001(2)	0.0053(17)	-0.0013(18)
C9	0.031(2)	0.088(4)	0.083(4)	-0.021(3)	0.016(2)	-0.005(2)
C10	0.031(2)	0.069(3)	0.098(4)	-0.036(3)	-0.008(2)	0.017(2)
C11	0.049(3)	0.049(2)	0.078(3)	-0.010(2)	-0.023(2)	0.020(2)
C12	0.036(2)	0.0376(19)	0.050(2)	-0.0021(17)	-0.0089(16)	0.0040(16)
C13	0.043(2)	0.057(2)	0.053(2)	-0.030(2)	-0.0047(19)	0.015(2)
B	0.0273(17)	0.0230(16)	0.0293(19)	0.0003(14)	-0.0030(14)	-0.0019(14)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 8. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	x	y	z	U_{eq}, Å^2
H4	0.1701	-0.0354	0.1168	0.032
H6	-0.0047	0.1645	0.0405	0.037
H8	-0.0979	0.2011	0.1581	0.058
H9	-0.2093	0.3557	0.1537	0.080
H10	-0.2411	0.6956	0.0999	0.080
H11	-0.1643	0.8722	0.0485	0.072
H12	-0.0536	0.7066	0.0506	0.050
H13A	0.1979	-0.1575	0.0412	0.062
H13B	0.1603	-0.3702	0.0056	0.062
H13C	0.1549	-0.3909	0.0636	0.062

1.5.3 X-ray data of (2-Iodo-4’,5-dimethoxybiphenyl-3-yl)boronic acid (**19**$_{\mathbf{A}}$)

STRUCTURE REPORT

XCL Code: JUS1508 **Date:** 11 December 2015

Compound: (2-Iodo-4’,5-dimethoxybiphenyl-3-yl)boronic acid
Formula: $C_{14}H_{14}BIO_4$

Supervisor: R. M. Al-Zoubi, Jordan University of Science and Technology
Crystallographer: R. McDonald

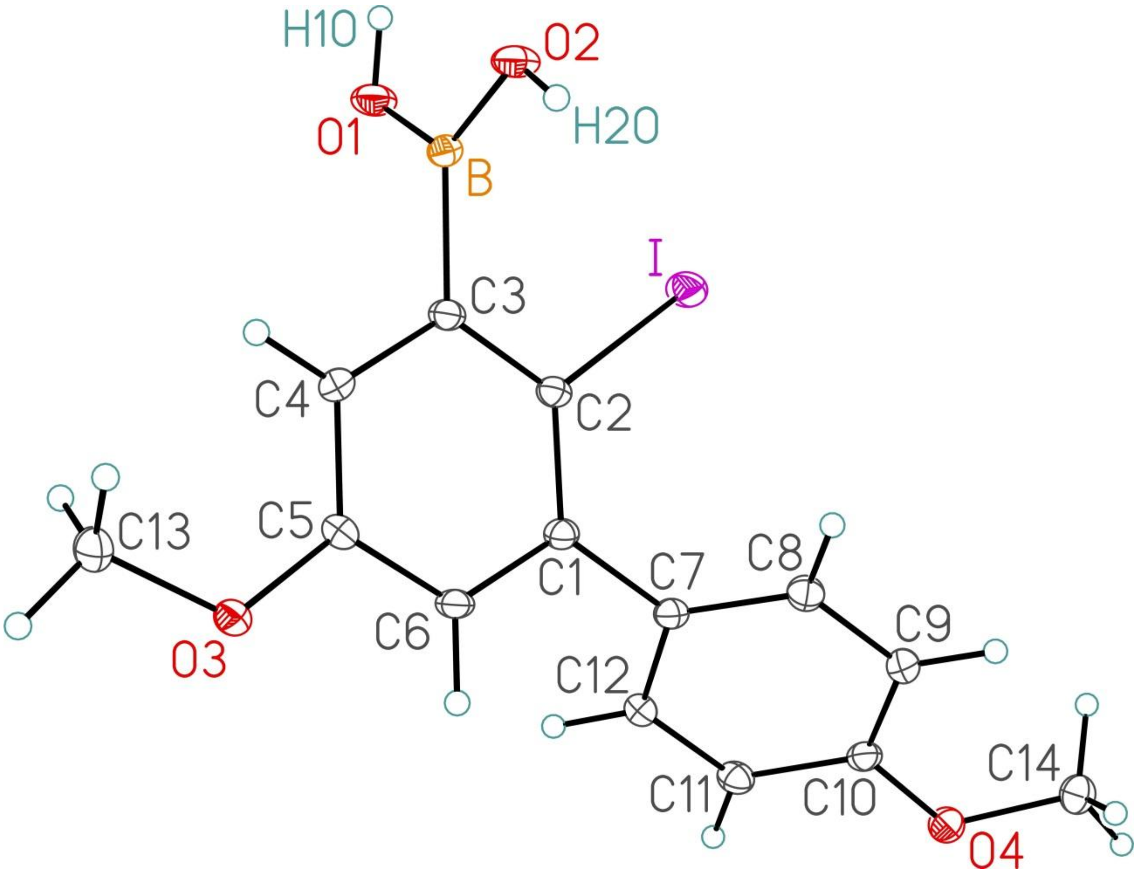
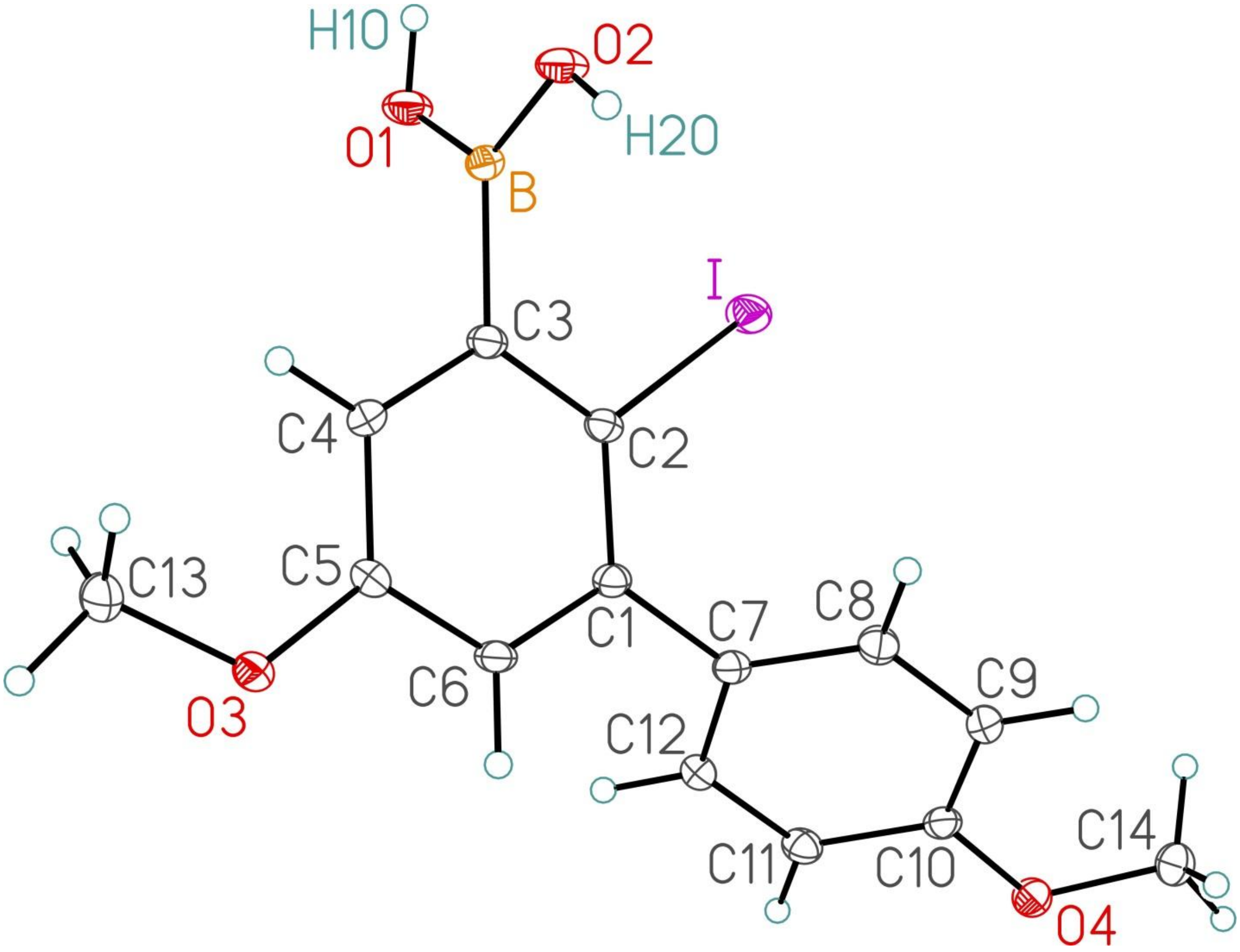


Figure Legends

Figure 1. Perspective view of the (2-iodo-4’,5-dimethoxybiphenyl-3-yl)boronic acid molecule showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Illustration of hydrogen-bonded interactions (shown by dotted lines) between adjacent molecules in the crystal lattice. Primed atoms are related to unprimed ones via the crystallographic inversion center (1, $^1/_2$, 0). Double-primed atoms are related to unprimed ones via the crystallographic inversion center ($^1/_2$, 0, 0).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H2O
B
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C3
C4
C2
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C14
C11
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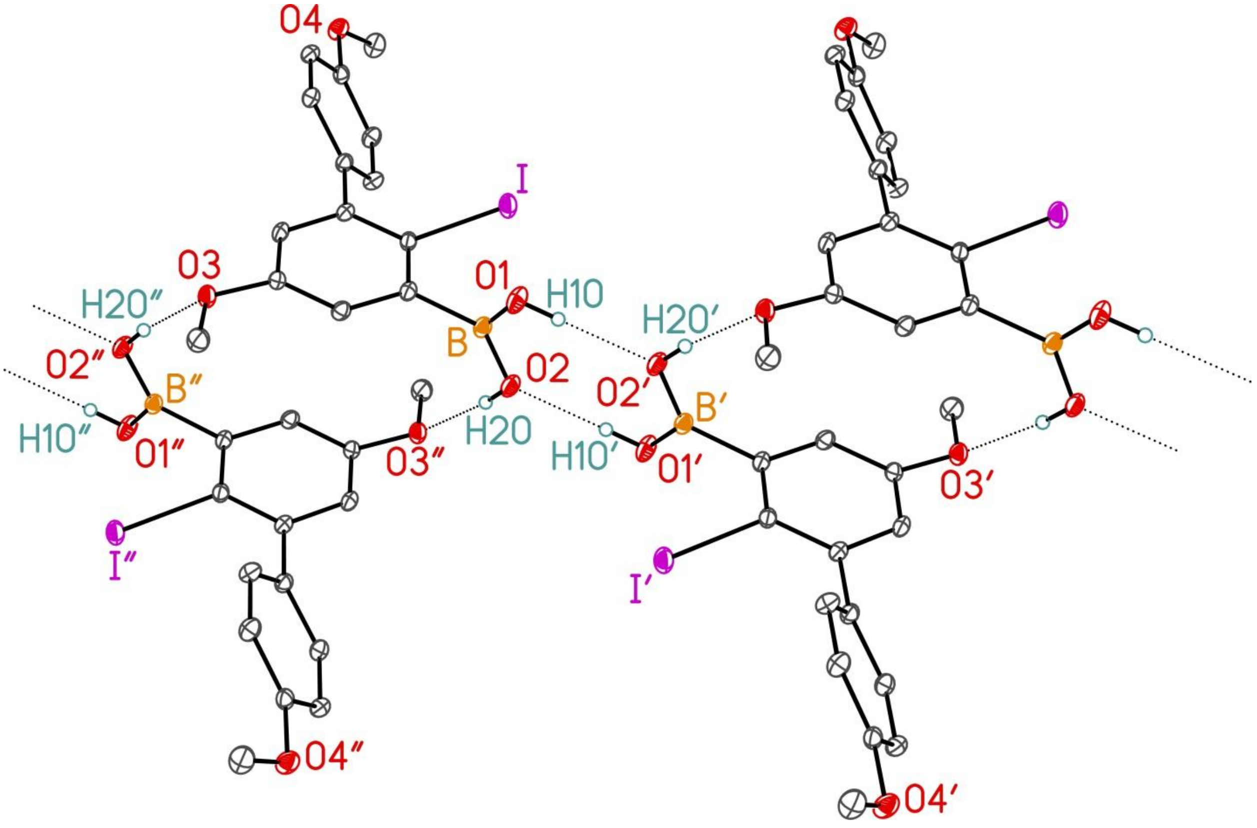
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B
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O3″
H2O
H1O′
O1′
O3′
I″
I′
O4″
O4′

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data	
formula	$C_{14}H_{14}BIO_4$
formula weight	383.96
crystal dimensions (mm)	0.17 × 0.14 × 0.04
crystal system	triclinic
space group	$P\bar{1}$ (No. 2)
unit cell parameters[a]	
a (Å)	7.4048 (3)
b (Å)	9.6801 (4)
c (Å)	10.5728 (4)
α (deg)	101.6750 (4)
β (deg)	90.6702 (5)
γ (deg)	105.7630 (4)
V (Å^3)	712.47 (5)
Z	2
ρ_{calcd} (g cm^{-3})	1.790
μ (mm^{-1})	2.256
B. Data Collection and Refinement Conditions	
diffractometer	Bruker PLATFORM/APEX II CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–100
scan type	ω scans (0.3°) (30 s exposures)
data collection 2θ limit (deg)	56.69
total data collected	6867 ($-9 \le h \le 9$, $-12 \le k \le 12$, $-14 \le l \le 14$)
independent reflections	3500 ($R_{\text{int}} = 0.0144$)
number of observed reflections (*NO*)	3235 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	Patterson/structure expansion (*DIRDIF–2008*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	0.9660–0.7538
data/restraints/parameters	3500 / 2[e] / 191
goodness-of-fit (S)[f] [all data]	1.070
final R indices[g]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0208
wR_2 [all data]	0.0499
largest difference peak and hole	0.924 and –0.445 e Å^{-3}

[a] Obtained from least-squares refinement of 7784 reflections with $4.48° < 2\theta < 56.14°$.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[b] Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

[c] Beurskens, P. T.; Beurskens, G.; de Gelder, R.; Smits, J. M. M.; Garcia-Granda, S.; Gould, R. O. (2008). The *DIRDIF-2008* program system. Crystallography Laboratory, Radboud University Nijmegen, The Netherlands.

[d] Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[e] The O1–H1O and O2–H2O distances were constrained to an idealized distance of 0.84(1) Å.

[f] $S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0229P)^2 + 0.3304P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[g] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	x	y	z	U_{eq}, Å^2
I	0.59205(2)	0.46716(2)	0.29533(2)	0.03040(5)*
O1	1.0144(2)	0.39467(17)	0.13198(15)	0.0309(3)*
O2	0.7784(2)	0.37270(16)	-0.02707(14)	0.0272(3)*
O3	0.58086(19)	-0.19229(14)	0.12745(13)	0.0241(3)*
O4	-0.0925(2)	0.24448(16)	0.63368(14)	0.0284(3)*
C1	0.4447(3)	0.1335(2)	0.27893(17)	0.0195(4)*
C2	0.5718(3)	0.2419(2)	0.22811(17)	0.0195(4)*
C3	0.7016(3)	0.2085(2)	0.14057(18)	0.0201(4)*
C4	0.7085(3)	0.0626(2)	0.10565(18)	0.0207(4)*
C5	0.5866(3)	-0.0458(2)	0.15679(17)	0.0198(4)*
C6	0.4550(3)	-0.0106(2)	0.24106(18)	0.0210(4)*
C7	0.3010(3)	0.1641(2)	0.37029(18)	0.0203(4)*
C8	0.1675(3)	0.2303(2)	0.33859(18)	0.0243(4)*
C9	0.0323(3)	0.2582(2)	0.42277(19)	0.0243(4)*
C10	0.0313(3)	0.2195(2)	0.54251(18)	0.0213(4)*
C11	0.1613(3)	0.1503(2)	0.57504(18)	0.0236(4)*
C12	0.2942(3)	0.1224(2)	0.49017(18)	0.0217(4)*
C13	0.7291(3)	-0.2329(2)	0.0558(2)	0.0304(4)*
C14	-0.2118(3)	0.3307(3)	0.6094(2)	0.0330(5)*
B	0.8383(3)	0.3329(2)	0.0798(2)	0.0219(4)*
H1O	1.078(4)	0.463(2)	0.100(3)	0.059(9)
H2O	0.672(2)	0.325(3)	-0.061(3)	0.047(8)

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
I	C2	2.1157(18)	C3	C4	1.401(3)
O1	B	1.337(3)	C3	B	1.598(3)
O2	B	1.371(3)	C4	C5	1.391(2)
O3	C5	1.378(2)	C5	C6	1.392(3)
O3	C13	1.437(2)	C7	C8	1.386(3)
O4	C10	1.369(2)	C7	C12	1.403(3)
O4	C14	1.423(2)	C8	C9	1.392(3)
C1	C2	1.407(2)	C9	C10	1.390(3)
C1	C6	1.395(3)	C10	C11	1.390(3)
C1	C7	1.494(3)	C11	C12	1.383(3)
C2	C3	1.398(3)			

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C5	O3	C13	117.85(15)	C1	C6	C5	121.28(17)
C10	O4	C14	117.06(16)	C1	C7	C8	121.55(16)
C2	C1	C6	117.43(17)	C1	C7	C12	120.51(16)
C2	C1	C7	123.91(17)	C8	C7	C12	117.91(17)
C6	C1	C7	118.66(16)	C7	C8	C9	121.91(17)
I	C2	C1	120.85(13)	C8	C9	C10	119.22(18)
I	C2	C3	116.64(13)	O4	C10	C9	124.15(17)
C1	C2	C3	122.26(17)	O4	C10	C11	116.09(17)
C2	C3	C4	118.57(16)	C9	C10	C11	119.76(17)
C2	C3	B	121.14(16)	C10	C11	C12	120.41(17)
C4	C3	B	120.29(16)	C7	C12	C11	120.75(17)
C3	C4	C5	120.07(17)	O1	B	O2	120.18(18)
O3	C5	C4	124.49(17)	O1	B	C3	119.14(17)
O3	C5	C6	115.15(16)	O2	B	C3	120.65(17)
C4	C5	C6	120.34(17)				

Table 5. Hydrogen-Bonded Interactions

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A (deg)
O1–H1O···O2[a]	0.832(10)[c]	1.966(10)	2.7979(19)	178(3)
O2–H2O···O3[b]	0.832(10)[c]	1.987(11)	2.813(2)	172(3)

[a] At 2–x, 1–y, $\bar{z}$. [b] At 1–x, $\bar{y}$, $\bar{z}$. [c] Distance constrained during refinement.

Table 6. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C13	O3	C5	C4	10.3(3)	B	C3	C4	C5	-178.66(17)
C13	O3	C5	C6	-171.52(17)	C2	C3	B	O1	97.9(2)
C14	O4	C10	C9	-7.7(3)	C2	C3	B	O2	-84.2(2)
C14	O4	C10	C11	172.62(18)	C4	C3	B	O1	-82.8(2)
C6	C1	C2	I	-172.62(13)	C4	C3	B	O2	95.1(2)
C6	C1	C2	C3	1.5(3)	C3	C4	C5	O3	179.24(17)
C7	C1	C2	I	7.4(2)	C3	C4	C5	C6	1.1(3)
C7	C1	C2	C3	-178.55(17)	O3	C5	C6	C1	-179.92(16)
C2	C1	C6	C5	0.3(3)	C4	C5	C6	C1	-1.6(3)
C7	C1	C6	C5	-179.63(17)	C1	C7	C8	C9	179.51(18)
C2	C1	C7	C8	57.2(3)	C12	C7	C8	C9	1.3(3)
C2	C1	C7	C12	-124.6(2)	C1	C7	C12	C11	-179.94(17)
C6	C1	C7	C8	-122.8(2)	C8	C7	C12	C11	-1.7(3)
C6	C1	C7	C12	55.3(2)	C7	C8	C9	C10	0.4(3)
I	C2	C3	C4	172.36(13)	C8	C9	C10	O4	178.62(18)
I	C2	C3	B	-8.3(2)	C8	C9	C10	C11	-1.7(3)
C1	C2	C3	C4	-2.0(3)	O4	C10	C11	C12	-178.98(18)
C1	C2	C3	B	177.33(17)	C9	C10	C11	C12	1.4(3)
C2	C3	C4	C5	0.6(3)	C10	C11	C12	C7	0.4(3)

Table 7. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I	0.03558(9)	0.01838(8)	0.03727(9)	0.00611(5)	0.01223(6)	0.00709(6)
O1	0.0231(7)	0.0318(8)	0.0393(8)	0.0206(7)	0.0022(6)	0.0006(6)
O2	0.0253(7)	0.0275(8)	0.0280(7)	0.0145(6)	0.0023(6)	-0.0004(6)
O3	0.0266(7)	0.0177(7)	0.0293(7)	0.0066(5)	0.0078(6)	0.0073(6)
O4	0.0309(8)	0.0304(8)	0.0300(7)	0.0118(6)	0.0140(6)	0.0141(6)
C1	0.0191(8)	0.0210(9)	0.0194(8)	0.0079(7)	0.0024(7)	0.0046(7)
C2	0.0203(9)	0.0163(9)	0.0216(9)	0.0057(7)	0.0025(7)	0.0034(7)
C3	0.0202(9)	0.0183(9)	0.0218(9)	0.0076(7)	0.0025(7)	0.0033(7)
C4	0.0194(9)	0.0227(9)	0.0213(9)	0.0071(7)	0.0060(7)	0.0062(7)
C5	0.0237(9)	0.0165(9)	0.0198(8)	0.0049(7)	0.0008(7)	0.0056(7)
C6	0.0207(9)	0.0194(9)	0.0232(9)	0.0097(7)	0.0037(7)	0.0023(7)
C7	0.0207(9)	0.0195(9)	0.0214(9)	0.0069(7)	0.0046(7)	0.0047(7)
C8	0.0264(10)	0.0284(10)	0.0214(9)	0.0101(8)	0.0046(7)	0.0097(8)
C9	0.0220(9)	0.0265(10)	0.0285(10)	0.0113(8)	0.0040(7)	0.0092(8)
C10	0.0207(9)	0.0171(9)	0.0248(9)	0.0045(7)	0.0059(7)	0.0030(7)
C11	0.0284(10)	0.0235(10)	0.0205(9)	0.0081(7)	0.0041(7)	0.0073(8)
C12	0.0219(9)	0.0206(9)	0.0246(9)	0.0083(7)	0.0023(7)	0.0069(7)
C13	0.0360(11)	0.0270(11)	0.0326(11)	0.0077(9)	0.0109(9)	0.0150(9)
C14	0.0313(11)	0.0325(12)	0.0415(12)	0.0118(10)	0.0141(9)	0.0161(9)
B	0.0237(10)	0.0197(10)	0.0248(10)	0.0079(8)	0.0091(8)	0.0075(8)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 8. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	x	y	z	U_{eq}, Å^2
H4	0.7966	0.0377	0.0470	0.025
H6	0.3705	-0.0863	0.2734	0.025
H8	0.1684	0.2574	0.2571	0.029
H9	-0.0583	0.3031	0.3987	0.029
H11	0.1590	0.1220	0.6561	0.028
H12	0.3819	0.0744	0.5133	0.026
H13A	0.8511	-0.1756	0.1007	0.036
H13B	0.7223	-0.2128	-0.0311	0.036
H13C	0.7149	-0.3378	0.0488	0.036
H14A	-0.2948	0.2795	0.5314	0.040
H14B	-0.1349	0.4260	0.5966	0.040
H14C	-0.2879	0.3460	0.6834	0.040

1.5.4 X-ray data of (2-Iodo-5-methoxy-4'-phenoxybiphenyl-3-yl)boronic acid (**21_A**)

STRUCTURE REPORT

XCL Code: JUS1509 **Date:** 11 December 2015

Compound: (2-Iodo-5-methoxy-4'-phenoxybiphenyl-3-yl)boronic acid
Formula: $C_{19}H_{16}BIO_4$

Supervisor: R. M. Al-Zoubi, Jordan University of Science and Technology
Crystallographer: R. McDonald

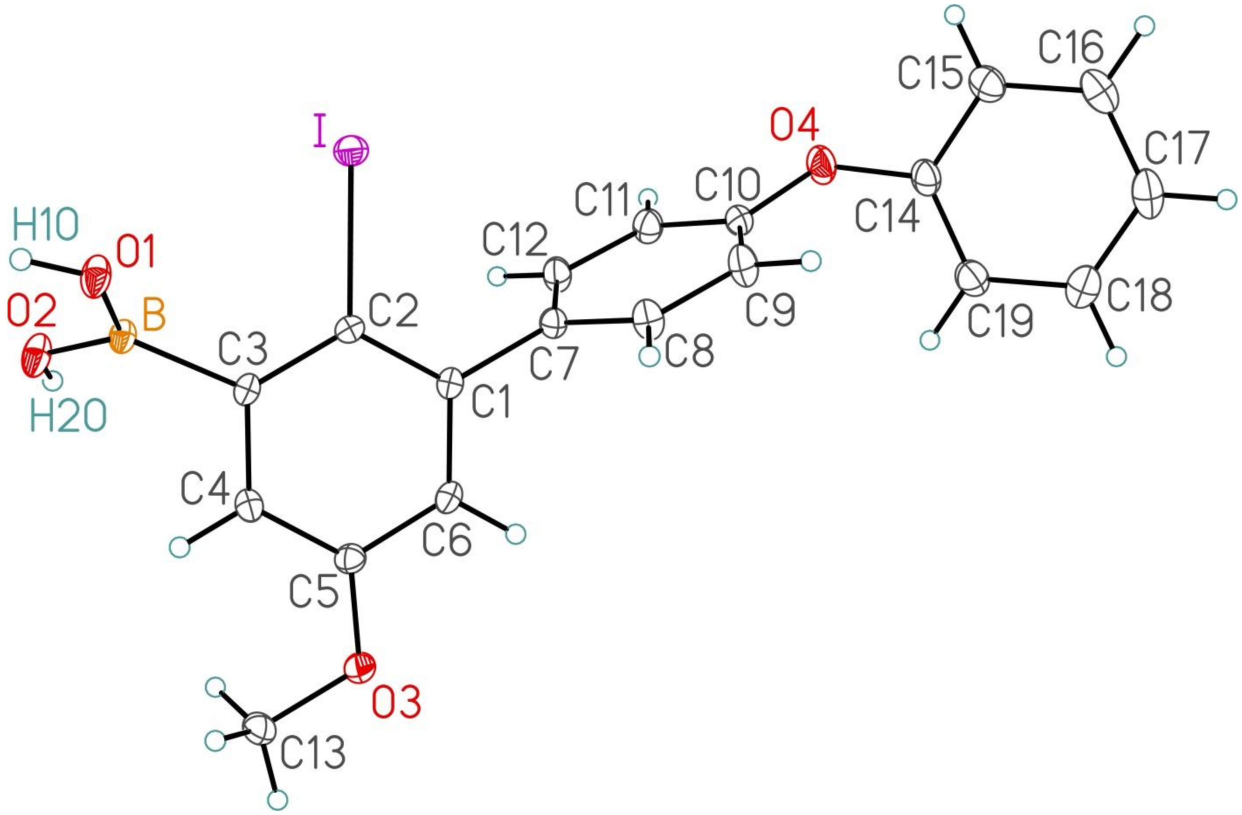
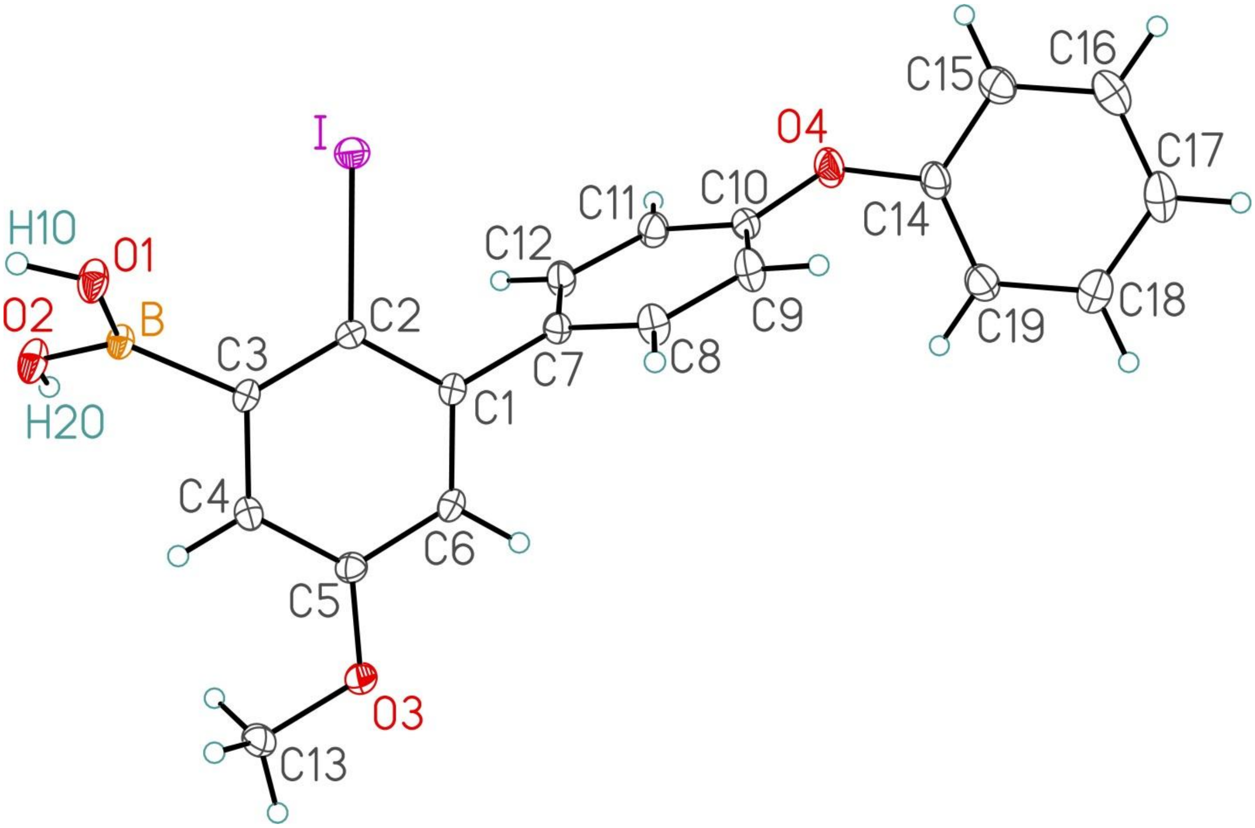


Figure Legends

Figure 1. Perspective view of the (2-iodo-5-methoxy-4’-phenoxybiphenyl-3-yl)boronic acid molecule showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Illustration of hydrogen-bonded interactions (shown by dotted lines) between adjacent molecules in the crystal lattice. Primed atoms are related to unprimed ones via the crystallographic inversion center ($^1/_2$, 1, $^1/_2$). Double-primed atoms are related to unprimed ones via the crystallographic symmetry operation (1+*x*, *y*, *z*) (translation in a direction parallel to the crystallographic unit’s cell’s *a* axis).


I
H10
O1
O2
B
H20
C3
C2
C1
C4
C5
C6
O3
C13
C12
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C7
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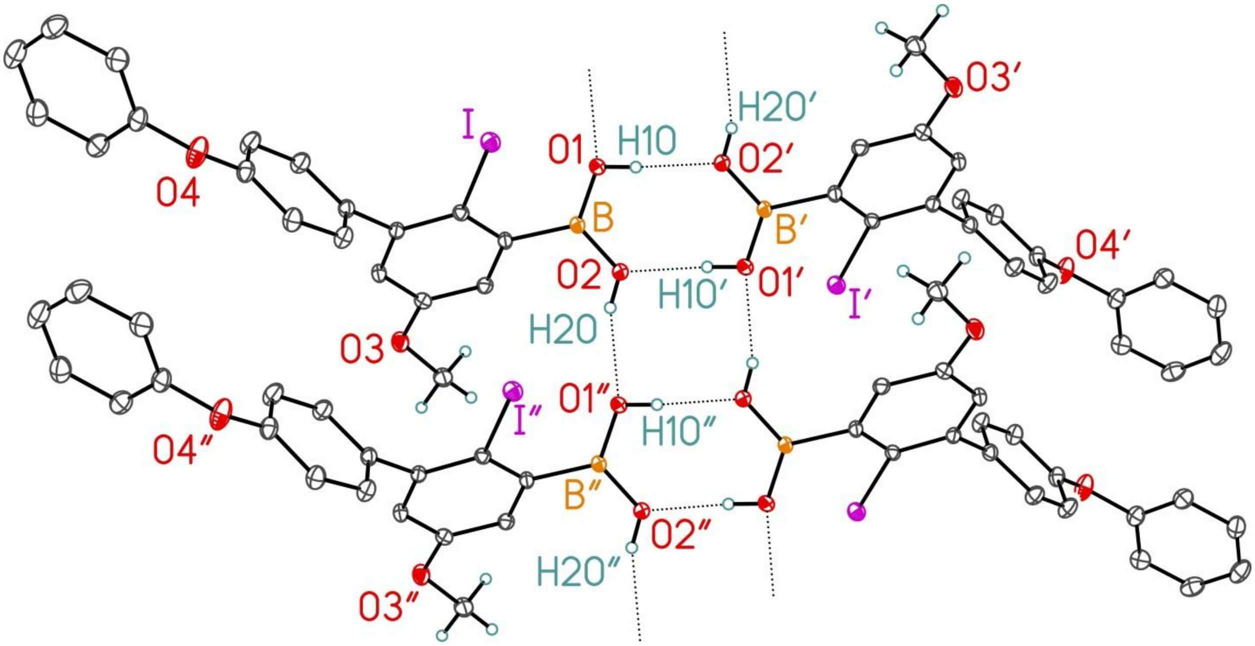
C9
O4
C14
C15
C16
C17
C18
C19


I
O1
H1O
H2O′
O2′
O3′
O4
B
B′
O2
H1O′
O1′
O4′
I′
H2O
O3
O1″
I″
H1O″
O4″
B″
O2″
H2O″
O3″

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data

formula	$C_{19}H_{16}BIO_4$
formula weight	446.03
crystal dimensions (mm)	0.21 × 0.05 × 0.02
crystal system	triclinic
space group	$P\bar{1}$ (No. 2)
unit cell parameters[a]	
a (Å)	5.1433 (2)
b (Å)	13.2175 (4)
c (Å)	14.1071 (4)
α (deg)	114.1398 (17)
β (deg)	94.4519 (16)
γ (deg)	93.3392 (16)
V (Å^3)	868.24 (5)
Z	2
ρ_{calcd} (g cm^{-3})	1.706
μ (mm^{-1})	14.66

B. Data Collection and Refinement Conditions

diffractometer	Bruker D8/APEX II CCD[b]
radiation (λ [Å])	Cu Kα (1.54178) (microfocus source)
temperature (°C)	–100
scan type	ω and ϕ scans (1.0°) (5 s exposures)
data collection 2θ limit (deg)	147.89
total data collected	6177 ($-6 \le h \le 6$, $-16 \le k \le 16$, $-17 \le l \le 17$)
independent reflections	3396 ($R_{\text{int}} = 0.0382$)
number of observed reflections (*NO*)	3147 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	intrinsic phasing (*SHELXT-2014*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	0.8683–0.2320
data/restraints/parameters	3396 / 2[e] / 235
goodness-of-fit (S)[f] [all data]	0.998
final R indices[g]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0252
wR_2 [all data]	0.0610
largest difference peak and hole	0.799 and –0.713 e Å^{-3}

[a] Obtained from least-squares refinement of 9848 reflections with 6.90° < 2θ < 147.34°.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[b] Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

[c] Sheldrick, G. M. *Acta Crystallogr.* **2015**, *A71*, 3–8.

[d] Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[e] The O1–H1O and O2–H2O distances were constrained to be 0.84(1) Å during refinement.

[f] $S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0309P)^2]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[g] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	x	y	z	U_{eq}, Å^2
I	0.09245(3)	0.63235(2)	0.45180(2)	0.02620(7)*
O1	0.2300(4)	0.87928(15)	0.42428(18)	0.0298(4)*
O2	0.6861(4)	0.89361(16)	0.45499(18)	0.0314(4)*
O3	0.7652(4)	0.55925(17)	0.08337(17)	0.0317(4)*
O4	-0.0433(5)	0.11239(19)	0.28855(18)	0.0393(5)*
C1	0.3394(5)	0.5041(2)	0.2529(2)	0.0206(5)*
C2	0.3217(5)	0.6120(2)	0.32880(19)	0.0192(4)*
C3	0.4588(5)	0.7050(2)	0.3255(2)	0.0206(5)*
C4	0.6144(5)	0.6887(2)	0.2445(2)	0.0225(5)*
C5	0.6226(5)	0.5830(2)	0.1660(2)	0.0235(5)*
C6	0.4822(5)	0.4919(2)	0.1702(2)	0.0233(5)*
C7	0.2276(5)	0.4013(2)	0.2606(2)	0.0224(5)*
C8	0.0030(6)	0.3386(2)	0.1989(2)	0.0295(6)*
C9	-0.0935(6)	0.2411(2)	0.2055(3)	0.0337(7)*
C10	0.0414(6)	0.2051(2)	0.2727(2)	0.0268(5)*
C11	0.2685(6)	0.2656(2)	0.3341(2)	0.0295(6)*
C12	0.3594(6)	0.3639(2)	0.3287(2)	0.0284(6)*
C13	0.9483(6)	0.6463(2)	0.0858(2)	0.0314(6)*
C14	-0.2206(6)	0.0305(2)	0.2142(2)	0.0302(6)*
C15	-0.4343(6)	-0.0062(3)	0.2498(3)	0.0338(6)*
C16	-0.6078(7)	-0.0940(3)	0.1794(3)	0.0402(8)*
C17	-0.5682(7)	-0.1433(3)	0.0750(3)	0.0426(8)*
C18	-0.3535(7)	-0.1063(3)	0.0406(3)	0.0385(7)*
C19	-0.1773(6)	-0.0191(3)	0.1102(3)	0.0344(6)*
B	0.4553(6)	0.8303(2)	0.4069(2)	0.0213(5)*
H1O	0.263(8)	0.9469(12)	0.464(3)	0.046(12)
H2O	0.826(4)	0.864(3)	0.453(3)	0.039(10)

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
I	C2	2.109(3)	C5	C6	1.393(4)
O1	B	1.350(4)	C7	C8	1.386(3)
O2	B	1.365(3)	C7	C12	1.397(4)
O3	C5	1.362(3)	C8	C9	1.395(4)
O3	C13	1.432(3)	C9	C10	1.384(4)
O4	C10	1.384(3)	C10	C11	1.387(4)
O4	C14	1.382(3)	C11	C12	1.389(4)
C1	C2	1.404(3)	C14	C15	1.385(4)
C1	C6	1.386(4)	C14	C19	1.383(5)
C1	C7	1.492(3)	C15	C16	1.389(4)
C2	C3	1.401(3)	C16	C17	1.382(6)
C3	C4	1.398(4)	C17	C18	1.382(5)
C3	B	1.585(4)	C18	C19	1.389(4)
C4	C5	1.388(4)			

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C5	O3	C13	117.3(2)	C7	C8	C9	121.1(3)
C10	O4	C14	119.8(2)	C8	C9	C10	119.3(3)
C2	C1	C6	118.4(2)	O4	C10	C9	123.9(3)
C2	C1	C7	123.3(2)	O4	C10	C11	115.4(2)
C6	C1	C7	118.2(2)	C9	C10	C11	120.6(2)
I	C2	C1	118.83(18)	C10	C11	C12	119.6(2)
I	C2	C3	119.96(19)	C7	C12	C11	120.8(2)
C1	C2	C3	121.2(2)	O4	C14	C15	116.9(3)
C2	C3	C4	118.7(2)	O4	C14	C19	121.8(3)
C2	C3	B	125.7(2)	C15	C14	C19	121.1(3)
C4	C3	B	115.7(2)	C14	C15	C16	119.1(3)
C3	C4	C5	120.7(2)	C15	C16	C17	120.3(3)
O3	C5	C4	124.8(2)	C16	C17	C18	120.0(3)
O3	C5	C6	115.5(2)	C17	C18	C19	120.4(3)
C4	C5	C6	119.6(3)	C14	C19	C18	119.1(3)
C1	C6	C5	121.2(2)	O1	B	O2	118.5(2)
C1	C7	C8	122.1(2)	O1	B	C3	121.6(2)
C1	C7	C12	119.1(2)	O2	B	C3	119.7(2)
C8	C7	C12	118.7(2)				

Table 5. Hydrogen-Bonded Interactions

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A (deg)
O1–H1O···O2[a]	0.835(10)[c]	1.934(11)	2.766(3)	174(4)
O2–H2O···O1[b]	0.836(10)[c]	2.16(3)	2.869(3)	143(4)

[a]At 1–*x*, 2–*y*, 1–*z*. [b]At 1+*x*, *y*, *z*. [c]Distance constrained during refinement.

Table 6. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C13	O3	C5	C4	9.5(4)	C4	C3	B	O1	121.5(3)
C13	O3	C5	C6	-169.0(2)	C4	C3	B	O2	-53.3(3)
C14	O4	C10	C9	21.2(5)	C3	C4	C5	O3	179.1(2)
C14	O4	C10	C11	-162.4(3)	C3	C4	C5	C6	-2.5(4)
C10	O4	C14	C15	-132.1(3)	O3	C5	C6	C1	176.8(2)
C10	O4	C14	C19	52.8(4)	C4	C5	C6	C1	-1.7(4)
C6	C1	C2	I	177.53(18)	C1	C7	C8	C9	178.2(3)
C6	C1	C2	C3	-4.3(4)	C12	C7	C8	C9	1.0(5)
C7	C1	C2	I	-6.8(3)	C1	C7	C12	C11	-176.8(3)
C7	C1	C2	C3	171.4(2)	C8	C7	C12	C11	0.5(5)
C2	C1	C6	C5	5.0(4)	C7	C8	C9	C10	-1.6(5)
C7	C1	C6	C5	-170.9(2)	C8	C9	C10	O4	177.0(3)
C2	C1	C7	C8	106.3(3)	C8	C9	C10	C11	0.8(5)
C2	C1	C7	C12	-76.5(4)	O4	C10	C11	C12	-175.9(3)
C6	C1	C7	C8	-78.0(4)	C9	C10	C11	C12	0.7(5)
C6	C1	C7	C12	99.2(3)	C10	C11	C12	C7	-1.3(5)
I	C2	C3	C4	178.40(18)	O4	C14	C15	C16	-175.3(3)
I	C2	C3	B	-1.4(3)	C19	C14	C15	C16	-0.2(5)
C1	C2	C3	C4	0.2(4)	O4	C14	C19	C18	175.5(3)
C1	C2	C3	B	-179.6(2)	C15	C14	C19	C18	0.6(5)
C2	C3	C4	C5	3.2(4)	C14	C15	C16	C17	-0.5(5)
B	C3	C4	C5	-176.9(2)	C15	C16	C17	C18	0.8(5)
C2	C3	B	O1	-58.6(4)	C16	C17	C18	C19	-0.4(5)
C2	C3	B	O2	126.6(3)	C17	C18	C19	C14	-0.3(5)

Table 7. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I	0.02873(10)	0.02727(9)	0.02379(10)	0.01104(7)	0.00630(6)	0.00406(6)
O1	0.0205(9)	0.0187(8)	0.0414(12)	0.0031(8)	0.0054(8)	0.0034(7)
O2	0.0203(9)	0.0207(8)	0.0409(12)	0.0008(8)	0.0006(8)	0.0029(7)
O3	0.0347(11)	0.0262(9)	0.0286(11)	0.0045(8)	0.0139(8)	-0.0014(8)
O4	0.0560(14)	0.0312(10)	0.0311(12)	0.0174(10)	-0.0051(10)	-0.0152(10)
C1	0.0205(11)	0.0177(11)	0.0212(13)	0.0061(10)	-0.0002(9)	0.0014(9)
C2	0.0195(11)	0.0205(11)	0.0171(12)	0.0070(9)	0.0023(8)	0.0033(9)
C3	0.0220(12)	0.0162(10)	0.0199(12)	0.0041(9)	0.0005(9)	0.0025(9)
C4	0.0205(11)	0.0192(11)	0.0277(14)	0.0099(10)	0.0023(10)	0.0012(9)
C5	0.0220(12)	0.0239(12)	0.0236(13)	0.0081(10)	0.0052(10)	0.0037(9)
C6	0.0260(13)	0.0178(10)	0.0212(13)	0.0032(10)	0.0019(9)	0.0024(9)
C7	0.0244(12)	0.0182(11)	0.0238(13)	0.0078(10)	0.0031(10)	0.0013(9)
C8	0.0271(13)	0.0258(12)	0.0363(16)	0.0155(12)	-0.0047(11)	-0.0005(10)
C9	0.0299(14)	0.0283(13)	0.0408(18)	0.0153(13)	-0.0067(12)	-0.0068(11)
C10	0.0332(14)	0.0215(11)	0.0251(14)	0.0097(11)	0.0041(11)	-0.0027(10)
C11	0.0360(15)	0.0250(12)	0.0274(15)	0.0122(11)	-0.0023(11)	-0.0002(11)
C12	0.0307(14)	0.0243(12)	0.0284(15)	0.0111(11)	-0.0033(11)	-0.0035(10)
C13	0.0311(14)	0.0284(13)	0.0353(16)	0.0125(12)	0.0128(12)	0.0013(11)
C14	0.0353(15)	0.0228(12)	0.0341(16)	0.0148(12)	0.0002(12)	-0.0020(11)
C15	0.0378(16)	0.0305(14)	0.0395(17)	0.0207(13)	0.0059(13)	0.0036(12)
C16	0.0316(15)	0.0409(16)	0.057(2)	0.0309(17)	0.0001(14)	-0.0027(13)
C17	0.0430(18)	0.0320(15)	0.051(2)	0.0206(15)	-0.0129(15)	-0.0090(13)
C18	0.0454(18)	0.0308(14)	0.0348(17)	0.0107(13)	-0.0043(13)	0.0025(13)
C19	0.0341(15)	0.0337(14)	0.0372(17)	0.0176(13)	0.0026(12)	-0.0018(12)
B	0.0209(13)	0.0161(12)	0.0241(15)	0.0054(11)	0.0017(10)	0.0031(10)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 8. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	x	y	z	U_{eq}, Å^2
H4	0.7156	0.7504	0.2431	0.027
H6	0.4844	0.4201	0.1153	0.028
H8	-0.0868	0.3625	0.1515	0.035
H9	-0.2502	0.1999	0.1641	0.040
H11	0.3613	0.2401	0.3796	0.035
H12	0.5130	0.4063	0.3718	0.034
H13A	0.8529	0.7056	0.0792	0.038
H13B	1.0551	0.6163	0.0276	0.038
H13C	1.0622	0.6767	0.1521	0.038
H15	-0.4619	0.0283	0.3215	0.041
H16	-0.7542	-0.1202	0.2030	0.048
H17	-0.6887	-0.2027	0.0269	0.051
H18	-0.3263	-0.1407	-0.0311	0.046
H19	-0.0291	0.0062	0.0867	0.041

Methods:

Antimicrobial activity assay

Inhibition zone determination

The antimicrobial activity of the $\mathbf{5_A}$, $\mathbf{6_A}$, $\mathbf{9_A}$, $\mathbf{14_A}$, $\mathbf{15_A}$, $\mathbf{17_A}$ - $\mathbf{22_A}$ compounds was tested for antimicrobial activity against the Gram-negative bacterial strains *Escherichia coli (ATCC 25922)* and Gram-positive bacterial strains *Staphylococcus aureus (ATCC 29213), Micrococcus luteus (ATCC 9341*) and *Bacillus cereus (ATCC 11778).* Moreover, the antifungal activity was performed against *Candida albicans (clinical sample supplied by King Abdullah University Hospital-KAUH).*

The different bacterial strains were inoculated into the nutrient broth, and on nutrient agar media, the resulting pure colonies were used to prepare bacterial suspensions. A loop full of the tested bacteria was used to inoculate nutrient broth and was incubated for 24 hrs at 30 °C. The bacterial suspension turbidity was adjusted to 0.5 McFarland standards. One hundred microliters of each test organism was inoculated into solidified agar plates.

Well-diffusion method was performed for the assessment of the antimicrobial activity according to the recommendations of the National Committee for Clinical Laboratory Standards (NCCLS) [1, 2]. Briefly, 8 mm wells were generated after inoculation of the nutrient agar plate with the appropriate bacterial strain. Fifty microliters of each DMSO-dissolved compound were added to each well followed by overnight incubation at 30 ^{o}C. The inhibition zones were recorded (Table 1) and the antimicrobial activities of the tested compounds against the tested microorganisms were analyzed.

Minimum inhibitory concentration

The minimum inhibitory concentration of $\mathbf{5_A}$, $\mathbf{6_A}$, $\mathbf{9_A}$, $\mathbf{14_A}$, $\mathbf{15_A}$, $\mathbf{17_A}$ - $\mathbf{22_A}$ compounds was assessed with modified techniques by Satyajit D. Sarker *et al* [3]. The tested bacterial strains were cultured in 10 ml of nutrient broth tube and incubated for 24 hrs at 30˚C. The different volume of the tested compounds was added into a separate well of sterile 96 well microplate. Ten µl of prepared nutrient broth cultures in a concentration of 5 × 105 CFU/mL of *Escherichia coli (ATCC 25922), Staphylococcus aureus (ATCC 29213), Micrococcus luteus (ATCC 9341*) and *Bacillus cereus (ATCC 11778).* microorganisms, and 10 µl of resazurin were added different wells. The nutrient broth was added into well up to 120 µl final volume. 96 well microplate was incubated for 24h at 30˚C, and then the color change was assessed visually. Any color changes from purple to pink or colorless were recorded as positive growth (Table 2). All experiments were performed in triplicates.

Results:

Antimicrobial activity assay:

Inhibition zone determination

The zone of inhibition diameter for the tested compounds against the growth of the selected microorganisms is presented below. Control experiment results showed that the tested microorganisms were able to grow in the presence of DMSO alone.

The inhibition zone exhibited by synthetic iodinated biphenylboronic acids **5_A** - **6_A**, **9_A**, **14_A** - **15_A** and **17_A** - **22_A** against two gram-positive and two gram-negative bacteria strains as well as one fungus strain using well-diffusion method

Microbiological Species	**Synthetic Iodinated biphenylboronic acid compounds**										
	5_A	**6_A**	**9_A**	**14_A**	**15_A**	**17_A**	**18_A**	**19_A**	**20_A**	**21_A**	**22_A**
mg/mL	3	1	1	12	7	6	5	5	6	3	5
Micrococcus Luteus	20 (±4.2)	20 (±2.1)	12 (±4.9)	15 (±3.5)	10 (±6.3)	NI	NI	13 (±4.9)	13 (±4.9)	18 (±4.9)	20 (±4.9)
Bacillus Cereus	20 (±3.5)	25 (±4.2)	19 (±4.8)	20 (±7.7)	13 (± 5.0)	21 (±4.2)	15 (±4.2)	23 (±4.2)	17 (±4.2)	20 (±4.2)	22 (±4.2)
Escherichia Coli	20 (±3.5)	NI	NI	NI	NI	NI	NI	NI	NI	NI	10 (±4.2)
Staphylococcus aureus	22 (±3.5)	20 (±1.5)	11 (±3.5)	20 (±4.2)	NI	15 (±1.5)	10 (±1.5)	18 (±1.5)	15 (±1.5)	18 (±1.5)	22 (±1.5)
Candida albicans	20 (±2.5)	12 (±2.8)	NI	17 (±3.5)	NI	NI	NI	NI	NI	NI	15 (±4.4)

Zone of inhibition in mm
NI: No Inhibition

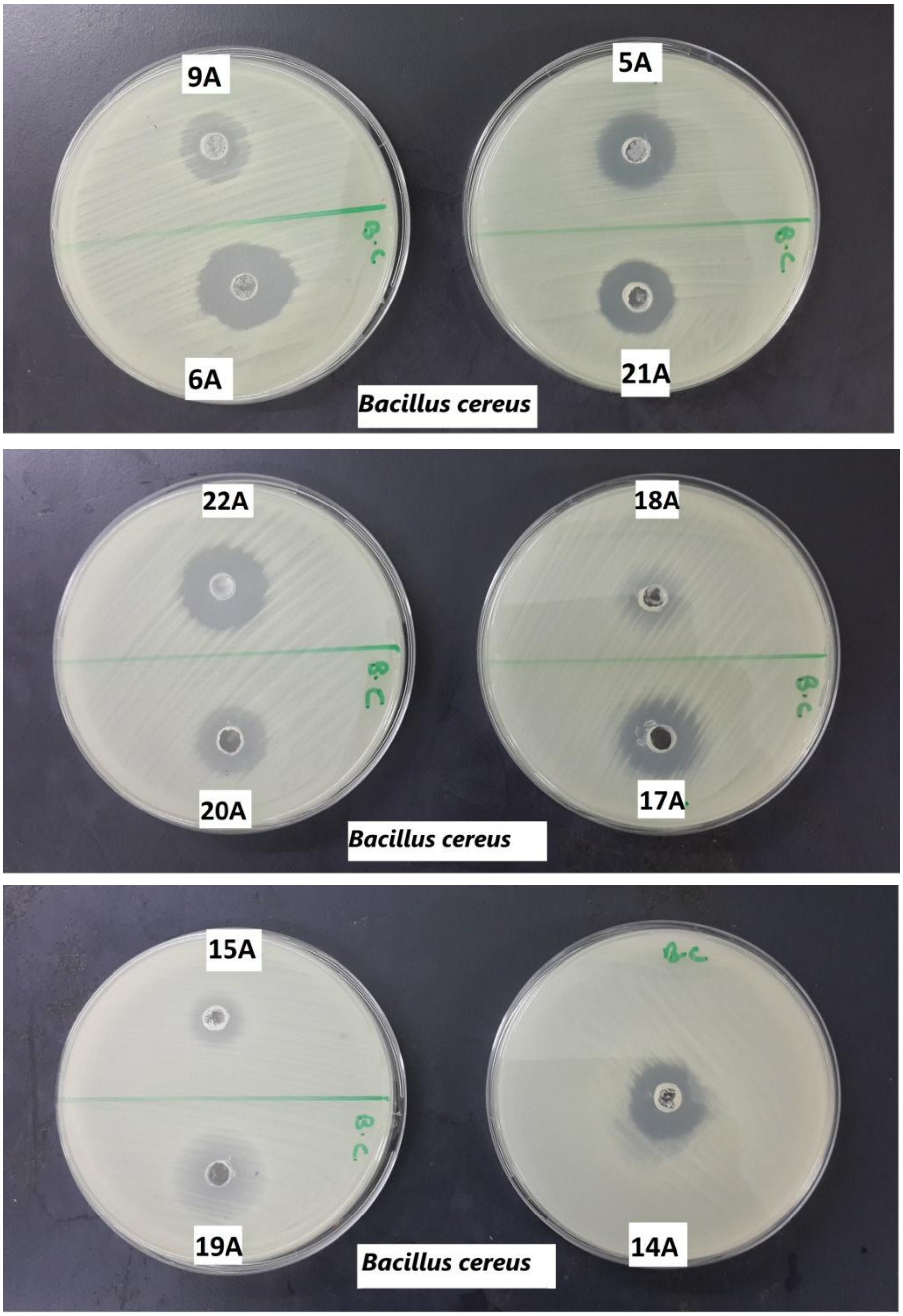


Figure 1. Anti *Bacillus cereus* activity for iodinated biphenylboronic acids. No inhibition for the negative control (DMSO).

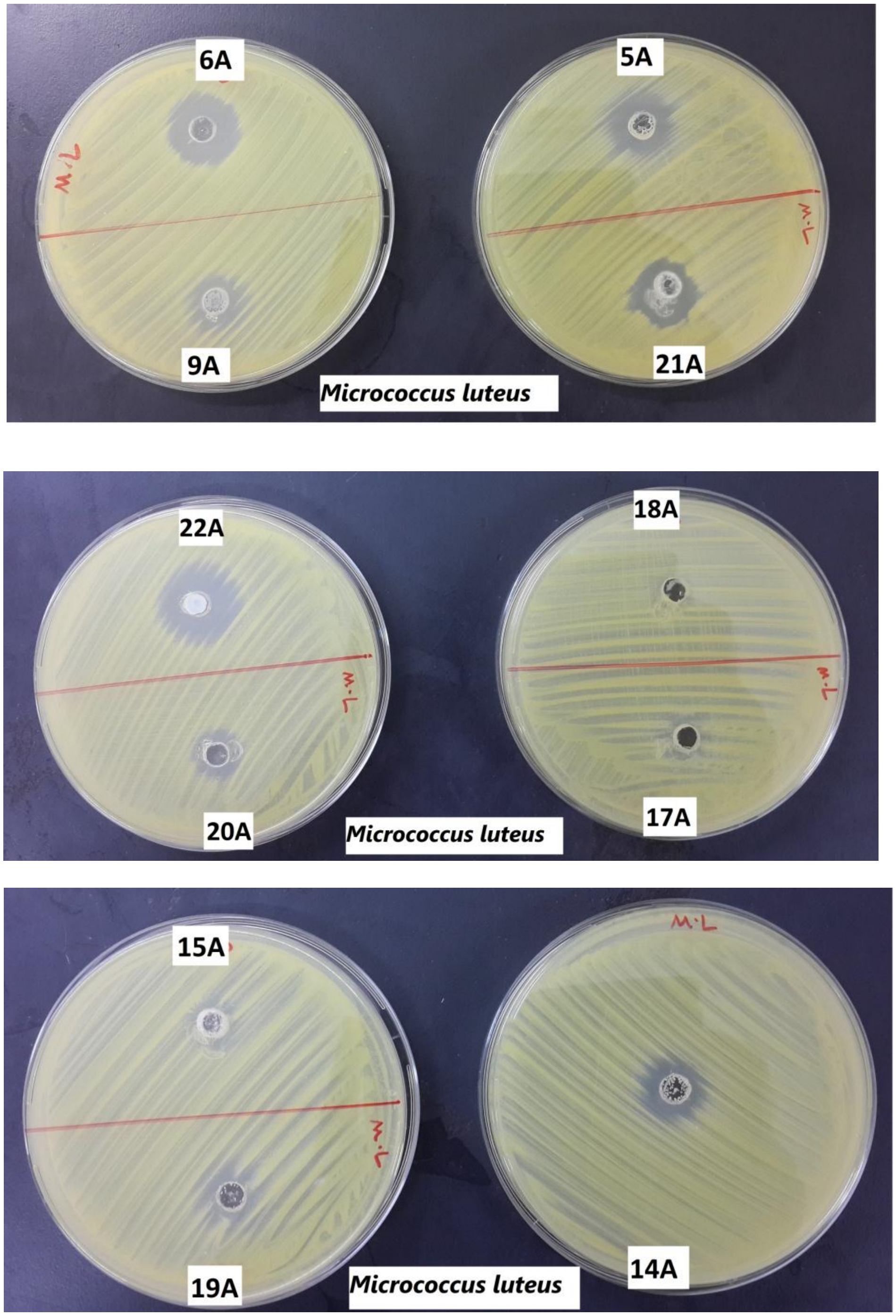


Figure 2. **Anti *Micrococcus luteus* activity for iodinated biphenylboronic acids. No inhibition for the negative control (DMSO).**

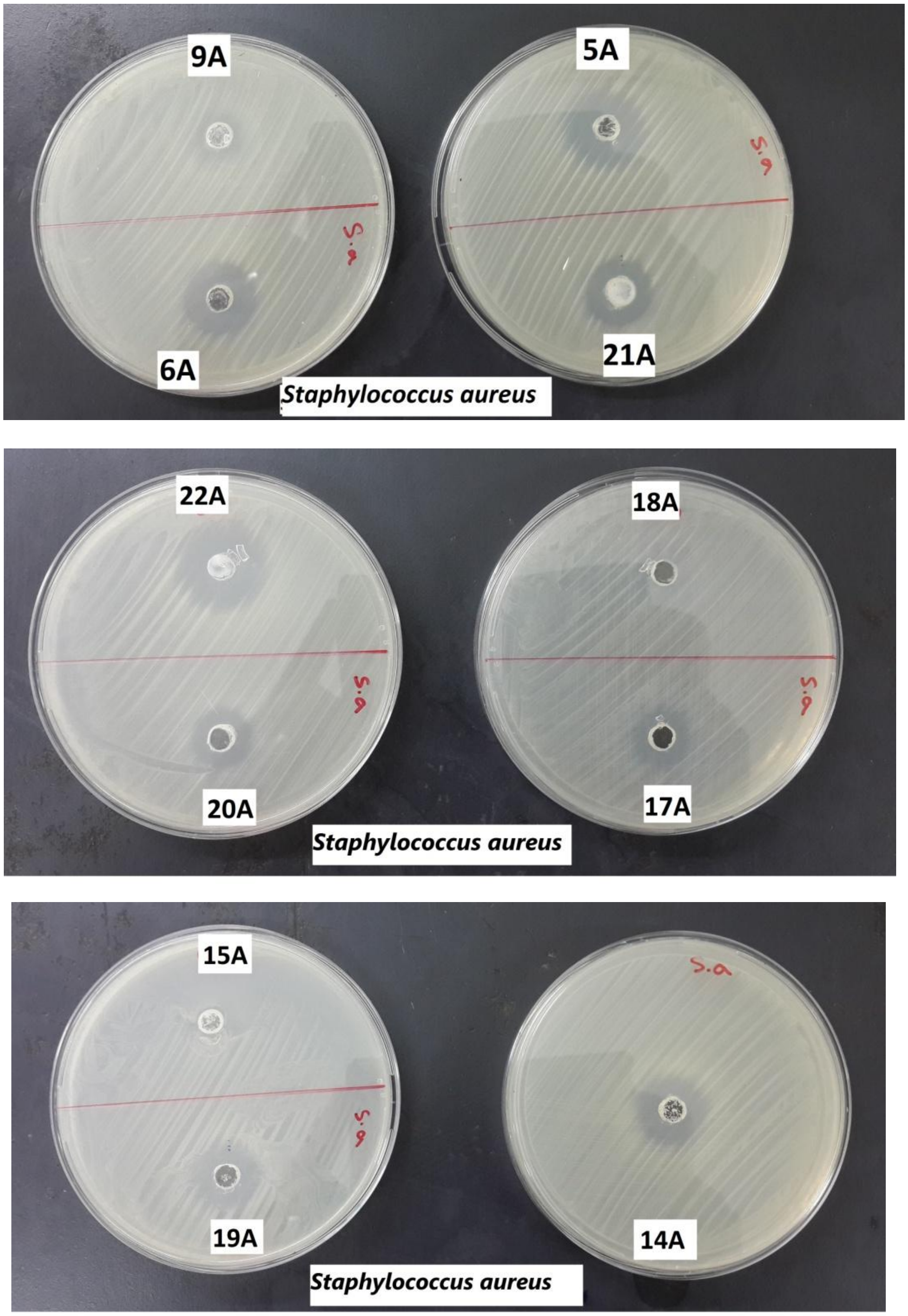


Figure 3. Anti *Staphylococcus aureus* activity for iodinated biphenylboronic acids. No inhibition for the negative control (DMSO).

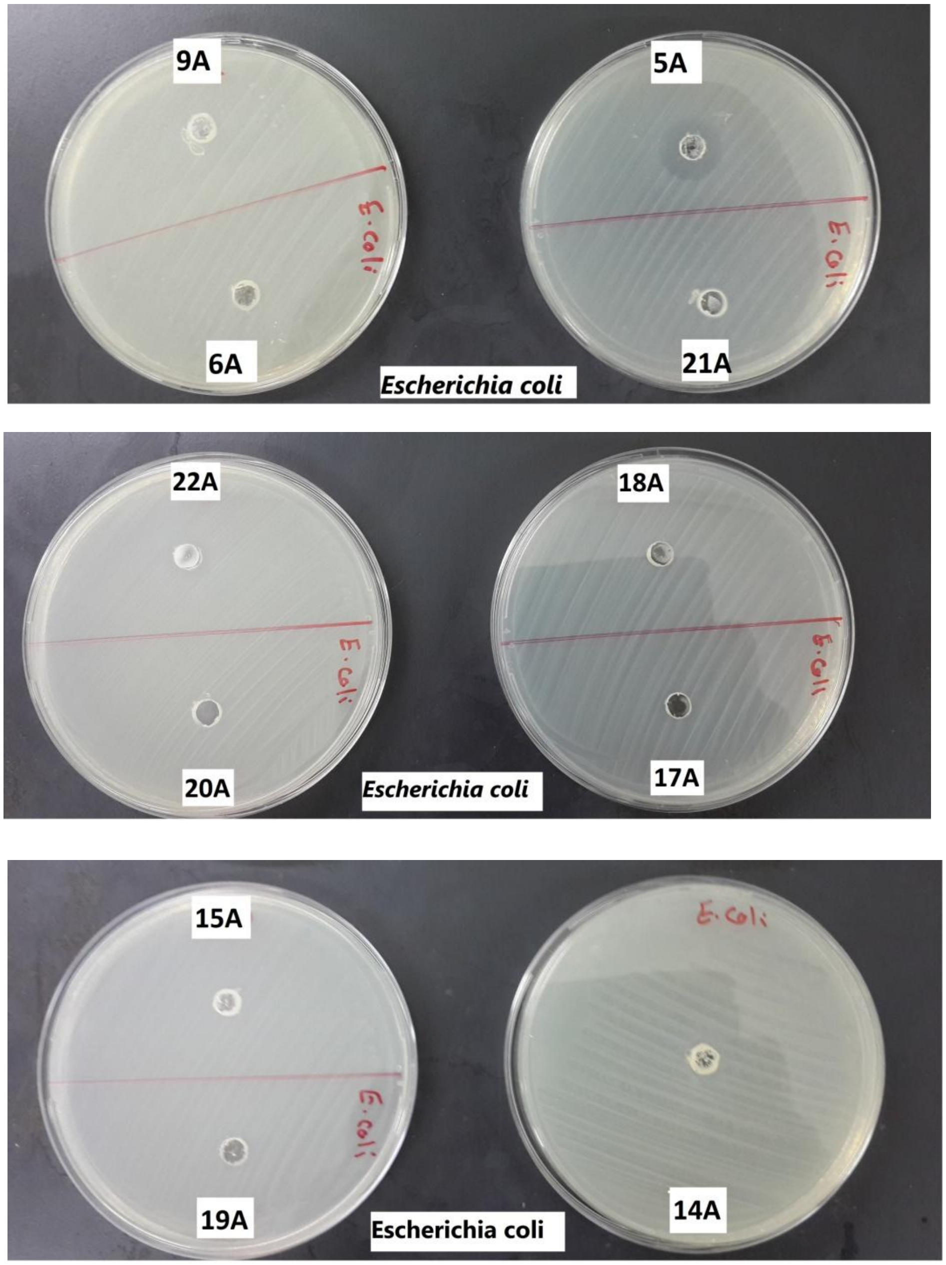


Figure 4. Anti *Escherichia coli* activity for iodinated biphenylboronic acids. No inhibition for the negative control (DMSO).

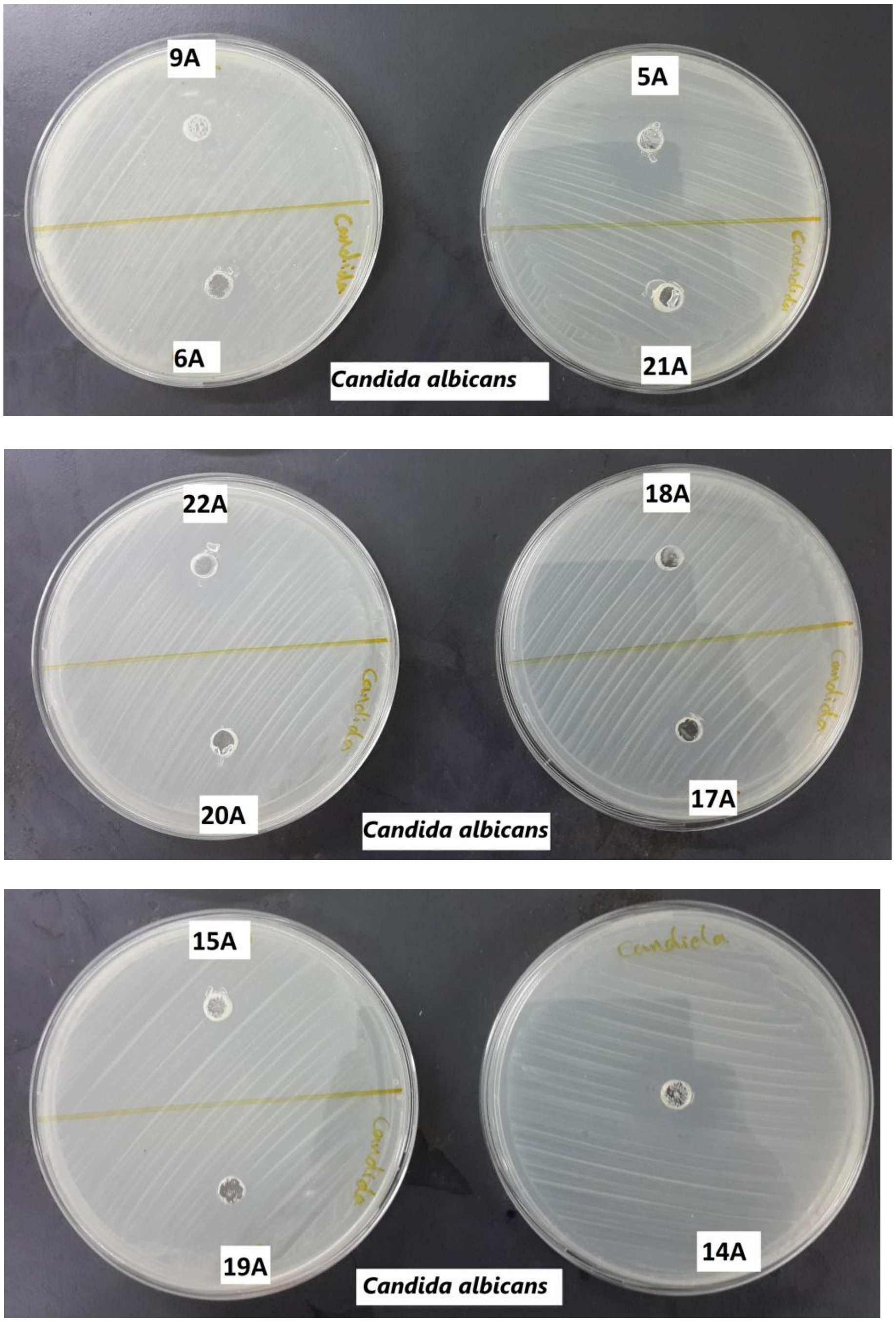


Figure 5. Anti *Candida albicans* activity for iodinated biphenylboronic acids. No inhibition for the negative control (DMSO).

Minimum inhibitory concentration (MIC)

The MIC value is the minimum concentration of the **5_A, 6_A, 9_A, 14_A, 15_A, 17_A - 22_A** compounds at which no microbial growth is recognized. The MIC of tested 11-compounds against the selected microorganisms is shown in next Table. In the current study, only 4 compounds exhibited anti-fungal activity, moreover, all of the tested compounds showed anti-microbial activity against gram positive bacteria. Compounds **6_A** and **22_A** exhibited a highly potent antimicrobial and anti-fungal activity by inhibition all tested microorganisms.

Minimum inhibitory concentration (MIC, mg/mL) for synthesized iodobiphenyl boronic acid derivatives against *Micrococcus Luteus, Bacillus cereus, Staphylococcus aureus* and *Candida albicans*

Iodobiphenyl boronic acid Compounds	**R_1 Group**	**Microbial Species**			
		M. luteus	*B. cereus*	*S. aureus*	*C. albicans*
5_A	H	0.45	0.48	0.37	0.9
6_A	H	0.15	0.10	0.16	0.48
9_A	Me	0.80	0.16	0.26	NT
14_A	OMe	3.2	1.6	1.4	3.0
15_A	OMe	3.0	1.4	NT	NT
17_A	Me	NT	0.75	0.8	NT
18_A	OMe	NT	0.62	0.7	NT
19_A	OMe	1.5	0.96	0.78	NT
20_A	OMe	0.8	0.49	0.75	NT
21_A	OMe	0.6	0.4	0.48	NT
22_A	OMe	0.2	0.15	0.15	0.3

References:

1. Allen, K.L., P.C. Molan, and G.M. Reid, *A survey of the antibacterial activity of some New Zealand honeys.* J Pharm Pharmacol, 1991. **43**(12): p. 817-22.
2. Holder, I.A. and S.T. Boyce, *Agar well diffusion assay testing of bacterial susceptibility to various antimicrobials in concentrations non-toxic for human cells in culture.* Burns, 1994. **20**(5): p. 426-9.
3. Sarker, S.D., L. Nahar, and Y. Kumarasamy, *Microtitre plate-based antibacterial assay incorporating resazurin as an indicator of cell growth, and its application in the in vitro antibacterial screening of phytochemicals.* Methods, 2007. **42**(4): p. 321-4.