Supporting information for:

Improving alkynyl(aryl)iodonium salts: 2-anisyl as a superior aryl group

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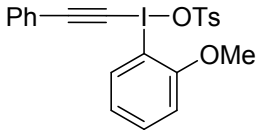
Contents

General

^{1}H NMR and ^{13}C NMR spectra were recorded in ppm from tetramethylsilane with the solvent resonance as the internal standard. Mass spectrometry (m/z) was performed in ESI mode (qTOF), with only molecular ions being reported. Infrared (IR) spectra ν_{max} are reported in cm^{-1}. All purchased reagents were used as received without further purification. Petroleum ether refers to the fraction boiling between 40 – 60 ^{o}C. Iodoarene diacetates were synthesized from the corresponding iodoarenes via published methodology.[1] Analytical data of the compounds produced matched that of the published literature. Iodosoarenes were synthesized from the corresponding iodoarene diacetates via published methodology.[2] Due to the insoluble, polymeric nature of these products, no analysis was attempted. Analytical data for compounds **1a**,[3] **1e**[4] and **5a**[5] matched the literature values. 1-Ethyl-2-ethynylbenzene was synthesized via published methodology.[6]

Synthesis of phenylethynyl(2-methoxyphenyl)iodonium tosylate (1b).



To a solution of 2-iodoanisole (1.15 g, 4.90 mmol), $TsOH{\cdot}H_2O$ (0.93 g, 4.90 mmol) and phenylacetylene (1.00 g, 9.80 mmol) in CH_2Cl_2 (25 mL) at 0 °C, was added *m*-CPBA (70-75% purity, 1.22 g, 4.90 mmol) slowly over a period of 1 hour. The resulting solution was slowly warmed to room temperature and stirred for 24 h, after

[1] (a) Kazmierczak, P.; Skulski, L.; Kraszkiewicz, L. *Molecules* **2001**, *6*, 881. (b) Hossain, D.; Kitamura, T. *J. Org. Chem.* **2005**, *70*, 6984.
[2] Saltzman, H.; Sharefkin, J. G. *Org. Synth.* **1963**, *43*, 60.
[3] Merritt, E. A.; Olofsson, B. *Eur. J. Org. Chem.* **2011**, 3690.
[4] Bouma, M. J.; Olofsson, B. *Chem. Eur. J.* **2012**, *18*, 14242.
[5] Ochiai, M.; Kunishima, M.; Tani, S.; Nagao, Y. *J. Am. Chem. Soc.* **1991**, *113*, 3135.
[6] Koumbis, A. E.; Kyzas, C. M.; Savva, A.; Varvoglis, A. *Molecules,* **2005**, *10,* 1340.

which it was reduced to half volume (approx. 12 ml) *in vacuo* at ambient temperature. 12 mL of diethyl ether was added to this solution in a drop wise manner, with gentle swirling. The resulting homogeneous solution was again reduced to half volume *in vacuo* (rota evaporator with no water bath), upon which time crystals formed. Over a period of one hour, 75 mL of diethyl ether was added with gentle swirling, and the resulting suspension was allowed to stand for 1 h. The precipitate was filtered, washed with diethyl ether and dried under vacuum, yielding the product as an off white amorphous powder (1.49 g, 61% yield). Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 114-116 °C.

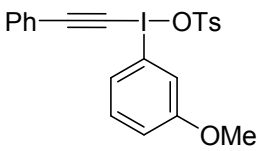
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (d, 1H, *J* = 8.1 Hz), 7.60 (d, 2H, *J* = 8.0 Hz), 7.56-7.52 (m, 1H), 7.40-7.37 (m, 3H), 7.33-7.29 (m, 2H) 7.07-7.04 (m, 3H), 6.99-6.96 (m, 1H), 3.95 (s, 3H), 2.32 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 156.3, 142.0, 140.3, 137.4, 135.1, 133.3, 131.0, 129.0, 128.8, 126.4, 124.0, 120.8, 113.0, 110.1, 103.3, 57.4, 38.5, 21.7.

IR (film): 2164 (w), 1472 (m), 1151 (m), 1118 (m), 1003 (s) cm^{-1}.

HRMS: cald. for $C_{15}H_{12}IO$ [M - OTs] 334.9927; found 334.9916. Cald. for $C_{22}H_{20}IO_4S$ $[MH]^+$ 507.0121; found 507.0140.

Synthesis of phenylethynyl(3-methoxyphenyl)iodonium tosylate (1c).



Product synthesized following the general procedure for **1b**. A cream amorphous powder (1.41 g, 57% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 91-94 °C.

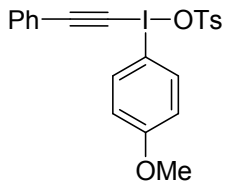
^{1}H NMR (400 MHz, *d*-Chloroform): δ 8.02-6.93 (m, 13H), 3.78 (s, 3H), 2.31 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 161.6, 146.5, 142.0, 140.4, 133.3, 132.5, 131.3, 129.1, 128.9, 126.4, 126.0, 119.1, 105.9, 94.2, 56.3, 39.5, 21.7.

IR (film): 3053 (w), 2159 cm^{-1} (m), 1592 (m), 1476 (m).

HRMS: cald. for $C_{15}H_{12}IO$ [M - OTs] 334.9927; found 334.9916. Cald. for: $C_{22}H_{20}IO_4S$ $[MH]^+$ 507.0121; found 507.0126.

Synthesis of phenylethynyl(4-methoxyphenyl)iodonium tosylate (1d).



Product synthesized following the general procedure for **1b**. A pale yellow amorphous powder (1.29 g, 52% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting Point: 101-103 °C.

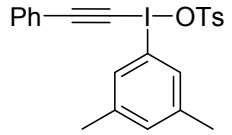
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, 2H, *J* = 8.5 Hz), 7.60 (d, 2H, *J* = 7.8 Hz), 7.42-7.26 (m, 5H), 7.06 (d, 2H, *J* = 7.8 Hz), 6.89 (d, 2H, *J* = 8.8 Hz), 3.82 (s, 3H), 2.31 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 162.7, 142.0, 140.4, 136.3, 133.3, 130.4, 129.0, 128.9, 126.4, 120.6, 117.9, 107.9, 104.9, 56.1, 39.3, 21.7.

IR (film): 3055 (b), 2160 (m), 1574 (m), 1485 (m) cm^{-1}.

HRMS: cald. for $C_{15}H_{12}IO$ [M - OTs] 334.9914; found 334.9912. Cald. for $C_{22}H_{20}IO_4S$ $[MH]^+$ 507.0121; found 507.0125.

Synthesis of phenylethynyl(3,5-dimethylphenyl)iodonium tosylate (1f).



Product synthesized according to the procedure for **1b**. A white amorphous powder (1.31g, 47% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 88-90 °C.

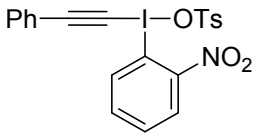
^1H NMR (400 MHz, $CDCl_3$): δ 7.70 (s, 2H), 7.64 (d, 2H, *J* = 8.1 Hz), 7.46-7.41 (m, 3H), 7.47-7.33 (m, 2H), 7.16 (s, 1H), 7.11-7.09 (m, 2H), 2.35-2.34 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 142.7, 141.9, 140.5, 134.1, 133.3, 131.6, 131.3, 129.1, 129.0, 126.4, 120.5, 119.5, 105.6, 39.4, 21.8, 21.7.

IR (film): 2161 (m), 1603 (w), 1486 (w), 996 (s) cm^{-1}.

HRMS: cald. for $C_{16}H_{14}I$ [M - OTs] 333.0135; found 333.0112. Cald. for $C_{22}H_{20}IO_4S$ $[MH]^+$ 505.0329; found 505.0321.

Synthesis of phenylethynyl(2-nitrophenyl)iodonium tosylate (1g).



To a solution of 2-iodonitrobenzene (1.22 g, 4.90 mmol), $TsOH{\cdot}H_2O$ (0.93 g, 4.90 mmol) in CH_2Cl_2 (25 mL) was added *m*-CPBA (70-70% purity, 1.22 g, 4.90 mmol) in

one portion. The solution was allowed to stir for 24 hours, upon which time phenylacetylene (1.00 g, 9.80 mmol) was added, followed by a further 24 hours stirring. Removal of the volatiles *in vacuo* at ambient temperature yielded a yellow residue, which was dissolved in the minimum amount of CH_2Cl_2 and added dropwise to diethyl ether (75 mL). After 2 h, the precipitate was filtered, washed with diethyl ether and dried under vacuum, yielding the product as a white amorphous powder (0.92 g, 36% yield). Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting Point: 111-113 °C.

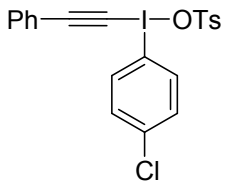
^{1}H NMR (400 MHz, *d*^{6}-DMSO): δ 8.58 (d, 1H, *J* = 7.8 Hz) 8.11-7.12 (m, 12H) 2.29 (s, 3H).

^{13}C NMR (100 MHz, *d*^{6}-DMSO): δ 160.1, 147.3, 145.8, 138.5, 134.7, 133.3, 132.7, 132.0, 130.7, 129.3, 128.62, 128.57, 128.3, 126.0, 108.7, 94.2, 21.3.

IR (film): 3053 (w), 2359 (m), 2164 (w), 1530 (m), 1370 (m) cm^{-1}.

HRMS: cald. for $C_{14}H_9INO_2$ [M - OTs] 349.9672; found 349.9648. Cald. for: $C_{21}H_{17}INO_5S$ [MH]$^+$ 521.9867; found 521.9853.

Synthesis of phenylethynyl(4-chlorophenyl)iodonium tosylate (1h).



Product synthesized following the general procedure for **1g**. A white amorphous powder (1.87 g, 75% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 119-120 °C (decomposes).

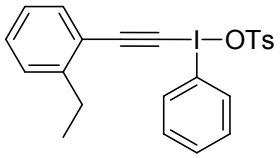
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05-7.06 (m, 13H) 2.33 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 140.2, 138.1, 136.2, 135.4, 133.0, 131.8, 131.0, 130.0, 129.4, 128.7, 126.0, 120.1, 113.4, 94.7, 21.3.

IR (film): 3056 (w), 2168 (w), 1376 (w) cm^{-1}.

HRMS: cald. for $C_{21}H_{17}ClIO_3S$ $[MH]^+$ 510.9626; found 510.9606.

Synthesis of 1-ethyl-2-ethynylbenzene(phenyl)iodonium tosylate (2a).



Product synthesized following the general procedure for **1b**. A white amorphous powder (1.55g, 63% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 109-110 °C

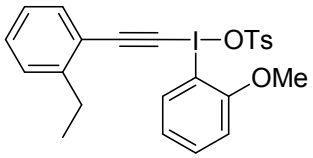
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.16-8.14 (m, 2H), 7.65 (d, 2H, *J* = 8.1 Hz), 7.56 (m, 1H), 7.45-7.40 (m, 3H), 7.36-7.33 (m, 1H), 7.23 (d, 1H, *J* = 7.7 Hz), 7.16 (m, 1H), 7.08 (d, 2H, *J* = 7.9 Hz), 2.69 (q, 2H, *J* = 7.7 Hz), 2.31 (s, 3H), 1.3 (t, 3H, *J* = 7.5 Hz).

^{13}C NMR (125 MHz, $CDCl_3$): δ 148.6, 141.6, 140.6, 134.2, 134.1, 132.3, 132.1, 131.5, 129.1, 128.6, 126.4, 126.2, 119.8, 119.6, 105.0, 42.0, 27.9, 21.7, 15.3.

IR (film): 3067 (b), 2155 (m), 1379 (m) cm^{-1}.

HRMS: cald. for $C_{16}H_{14}I$ [M - OTs] 333.0135; found 333.0112. Cald. for $C_{23}H_{22}IO_3S$ $[MH]^+$ 505.0329; found 505.0320.

Synthesis of 1-ethyl-2-ethynylbenzene(2-methoxyphenyl)iodonium tosylate (2b).



Product synthesized following the general procedure for **1b**. A cream amorphous powder (1.70g, 65% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 106-107 °C.

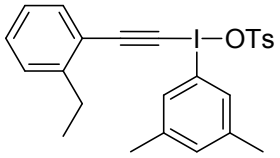
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.14 (dd, 1H, *J* = 8.0 Hz, *J* = 1.5 Hz), 7.62 (d, 2H, *J* = 8.2 Hz), 7.59-7.55 (m, 1H), 7.37 (dd, 1H, *J* = 7.7 Hz, *J* = 1.1 Hz), 7.33 (m, 1H), 7.21 (d, 1H, *J* = 7.5 Hz), 7.16 (m, 1H), 7.10 (d, 2H, *J* = 7.5 Hz), 7.08 (dd, 1H, *J* = 8.4 Hz, *J* = 1.3 Hz), 7.02-6.99 (m, 1H), 3.97 (s, 3H), 2.67 (q, 2H, *J* = 7.5 Hz), 2.33 (s, 3H) 1.11 (t, 3H, *J* = 7.5 Hz).

^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 148.6, 142.0, 140.5, 137.3, 135.2, 133.9, 131.3, 129.0, 128.6, 126.4, 126.2, 124.1, 119.8, 113.0, 110.7, 102.9, 57.4, 41.1, 27.9, 21.7, 15.2.

IR (film): 3061 (w), 2979 (w), 2157 (m), 1473 (m) cm^{-1}.

HRMS: cald. for $C_{17}H_{16}IO$ [M - OTs] 363.0240; found 363.0215.

Synthesis of 1-ethyl-2-ethynylbenzene(3,5-dimethylphenyl)iodonium tosylate (2c).



Product synthesized following the general procedure for **1b**. A white amorphous powder (1.22 g, 47% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 100-102 °C.

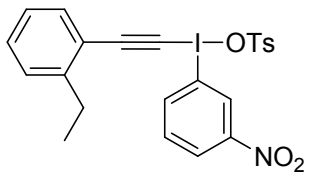
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (s, 2H), 7.65 (d, 2H, *J* = 8.2 Hz), 7.41 (dd, 1H, *J* = 7.7 Hz, *J* = 1.1 Hz), 7.37-7.33 (m, 1H), 7.23 (d, 1H, *J* = 7.6 Hz), 7.20-7.17 (m, 1H), 7.16 (s, 1H), 7.11 (d, 2H, *J* = 8.0 Hz), 2.70 (q, 2H, *J* = 7.6 Hz), 2.33 (s, 3H), 2.32 (s, 6H), 1.14 (t, 3H, *J* = 7.7 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ 148.5, 142.4, 141.7, 140.5, 134.0, 133.9, 131.7, 131.3, 129.0, 128.5, 126.4, 126.2, 119.8, 119.5, 104.4, 41.6, 27.8, 21.7, 15.6, 15.2.

IR (film): 3045 (w), 2163 (m) cm^{-1}.

HRMS: cald. for $C_{25}H_{26}IO_3S$ $[MH]^+$ 533.0642; found 533.0623.

Synthesis of 1-ethyl-2-ethynylbenzene(3-nitrophenyl)iodonium tosylate (2d).



Product synthesized following the general procedure for **1g**. A white amorphous powder (1.50 g, 56% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 101-103 °C.

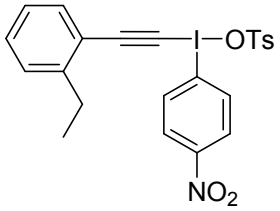
^{1}H NMR (400 MHz, $CDCl_3$, *d*^{6}-DMSO): δ 8.92-8.91 (m, 1H), 8.52 (d, 1H, *J* = 8.2 Hz), 8.32 (dd, 1H, *J* = 8.3 Hz, *J* = 1.3 Hz), 7.61-7.57 (m, 3H), 7.37-7.31 (m, 2H), 7.19 (d, 1H, *J* = 7.6 Hz), 7.14-7.11 (m, 1H), 7.01 (d, 2H, 8.0 Hz), 2.65 (t, 2H, *J* = 7.6 Hz), 2.28 (s, 3H) 1.08 (t, 3H, *J* = 7.5 Hz).

^{13}C NMR (100 MHz, $CDCl_3$,): δ 148.8, 148.4, 140.7, 140.6, 139.9, 133.9, 132.0, 131.3, 129.1, 128.8, 128.1, 126.2, 126.0, 125.8, 119.0, 118.6, 105.6, 41.9, 27.5, 21.3, 14.8.

IR (film): 3084 (b), 2169 (w), 1529 (m), 1343 (m) cm^{-1}.

HRMS: cald. for $C_{16}H_{13}INO_2$ [M - OTs] 377.9985; found 377.9974. Cald. for $C_{23}H_{21}INO_5S$ $[MH]^+$ 550.0180; found 550.0186.

Synthesis of 1-ethyl-2-ethynylbenzene(4-nitrophenyl)iodonium tosylate (2e).



Product synthesized following the general procedure for **1g**. A white amorphous powder (1.72 g, 64% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals. Melting point: 116-117 °C (decomposes).

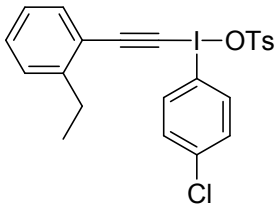
^{1}H NMR (400 MHz, *d*^{6}-DMSO): δ 8.64 (d, 2H, *J* = 8.9 Hz), 8.37 (d, 2H, *J* = 8.8 Hz), 7.49 (d, 2H, *J* = 7.9 Hz), 7.35-7.33 (m, 1H), 7.29-7.25 (m, 1H), 7.11 (d, 2H, *J* = 7.9 Hz) 2.65 (q, 2H, *J* = 7.5 Hz), 2.29 (s, 3H) 1.03 (t, 3H, *J* = 7.5 Hz).

^{13}C NMR (100 MHz, *d*^{6}-DMSO): δ 149.4, 147.7, 145.2, 138.8, 137.9, 136.1, 133.1, 131.4, 128.6, 126.4, 125.7, 125.5, 125.0, 118.6, 102.1, 44.8, 26.9, 20.8, 15.0.

IR (film): 3074 (b), 2162 (w), 1523 (m), 1352 (m) cm^{-1}.

HRMS: cald. for $C_{16}H_{13}INO_2$ [M - OTs] 377.9985; found: 377.9979. Cald. for $C_{23}H_{21}INO_5S$ $[MH]^+$ 550.0180; found 550.0175.

Synthesis of 1-ethyl-2-ethynylbenzene(4-chlorophenyl)iodonium tosylate (2f).



Product synthesized following the general procedure for **1g**. A white amorphous powder (1.21 g, 46% yield) was obtained. Analytically pure material was obtained by recrystallization in CH_2Cl_2 and ether, which yielded white crystals.

Melting point: 98-99 °C.

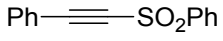
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, 2H, *J* = 8.7 Hz), 7.56 (d, 2H, *J* = 8.1 Hz), 7.3-7.21 (m, 3H), 7.21-7.19, (m, 1H), 7.16-7.12 (m, 2H), 7.05 (d, 2H *J* = 8.0 Hz) 2.65 (q, 2H, *J* = 7.5 Hz), 2.31 (s, 3H), 1.19 (t, 3H, *J* = 7.6 Hz).

^{13}C NMR (100 MHz, $CDCl_3$): δ 148.6, 140.9, 138.9, 136.7, 135.8, 134.1, 132.1, 129.1, 128.5, 126.3, 126.1, 119.6, 116.3, 104.9, 57.9, 41.5, 27.8, 21.7, 15.2.

IR (film): 3061 (w), 2969 (b), 2150 (m), 1379 (m) cm^{-1}.

HRMS: cald. for $C_{23}H_{21}ClIO_3S$ $[MH]^+$ 538.9939; found 538.9947.

General Synthesis of 2-(benzenesulfonyl)ethynylbenzene (3) from trimethyl(phenylethynyl)silane (1).[5]



To a suspension of an iodosylarene (fine powder, 0.5 mmol) in CH_2Cl_2 (5 mL) under an inert atmosphere was added trimethyl(phenylethynyl)silane **7** (0.052 g, 0.3 mmol), then, over a period of 5 min, boron trifluoride etherate (0.5 mmol). [For 2-(iodosyl)anisole, the suspension was cooled to -78 °C and boron trifluoride was added over a period of 1 h.] The reaction was allowed to stir for 16 h, which yielded a bright yellow homogenous solution. An aqueous solution of a sodium or lithium salt

(2.5 M, 1.8 mmol) was charged and the resulting emulsion was stirred vigorously for 15 min. The aqueous layer was separated and extracted with CH_2Cl_2 (2 x 5 ml). The combined organic layers were washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo,* yielding a viscous yellow oil, the crude alkynyl(aryl)iodonium salt. The crude residue was dissolved in CH_2Cl_2 (2 mL) and a solution of benzenesulfinic acid sodium salt (0.019 g, 0.116 mmol) and triethylbenzyl ammonium chloride (TEBA) (0.0024 g, 0.011 mmol) in water (1 mL) was added. The reaction mixture was stirred at room temperature for 1 hour, after which time the aqueous layer was separated and extracted with CH_2Cl_2 (2 x 5 ml). The combined organic layers were washed with water, then brine, and dried over anhydrous $MgSO_4$, filtered and concentrated *in vacuo*, yielding a viscous yellow oil. Purification by flash column chromatography (9:1 petroleum ether/ethyl acetate) yielded a pale yellow solid.

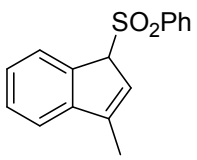
Melting point: 70-71 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (dd, 2H, *J* = 7.3 Hz, *J* = 1.3 Hz), 7.73-7.70 (m, 1H), 7.64-7.61 (m, 2H), 7.55 (dd, 2H, *J* = 7.1 Hz, *J* = 1.3 Hz), 7.52-7.48 (m, 1H), 7.41-7.38 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 141.7, 134.1, 132.7, 131.6, 129.3, 128.7, 127.4, 117.8, 93.5, 85.3.

HRMS: cald. for $C_{14}H_{10}NaO_2S$ $[MNa]^+$ 265.0294; found 265.0303.

General Synthesis of 1-benzenesulfonyl-3-methyl-indene (4) from 1-ethyl-2-ethynylbenzene(aryl)iodonium tosylates (2).[10c]



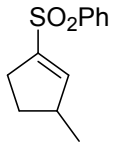
To a solution of 1-ethyl-2-ethynylbenzene(aryl)iodonium tosylate **2** (0.099 mmol) in CH_2Cl_2 (2 mL) was added benzenesulfinic acid sodium salt (0.018 g, 0.011 mmol). The suspension was stirred at room temperature for one hour upon which time deionized water (2 mL) was added. After an additional 5 min the aqueous layer was separated and extracted with CH_2Cl_2 (2 x 5 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo,* which yielded the crude product as a yellow oil. Purification by flash column chromatography (9:1 petroleum ether/ethyl acetate) yielded a pale yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, 1H, *J* = 6.7 Hz), 7.41-7.35 (m, 3H), 7.26-7.15 (m, 4H), 6.97 (d, 1H, *J* = 6.3 Hz), 6.05 (s, 1H), 4.91 (s, 1H), 1.88 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 146.5, 145.6, 136.0, 135.0, 133.4, 129.1, 128.9, 127.8, 126.3, 125.6, 122.5, 119.5, 77.2, 76.7, 72.4, 12.8.

HRMS: cald. for $C_{16}H_{14}SO_2Na$ $[MNa]^+$ 293.0607; found 293.0609.

General Synthesis of (3-methylcyclopenten-1-yl)sulfonylbenzene (10) from alkynylsilane 8.[10c]



To a suspension of an iodosylarene (fine powder, 0.5 mmol) in CH_2Cl_2 (5 mL) under an inert atmosphere was added hex-1-yn-1-yltrimethylsilane **8** (0.046 g, 0.3 mmol), then, over a period of 5 min, boron trifluoride etherate (0.5 mmol). [For 2-(iodosyl)anisole, the suspension was cooled to -78 °C and boron trifluoride was added over a period of 1 h.] The reaction was allowed to stir for 16 h, which yielded a bright yellow homogenous solution. An aqueous solution of sodium tetrafluoroborate

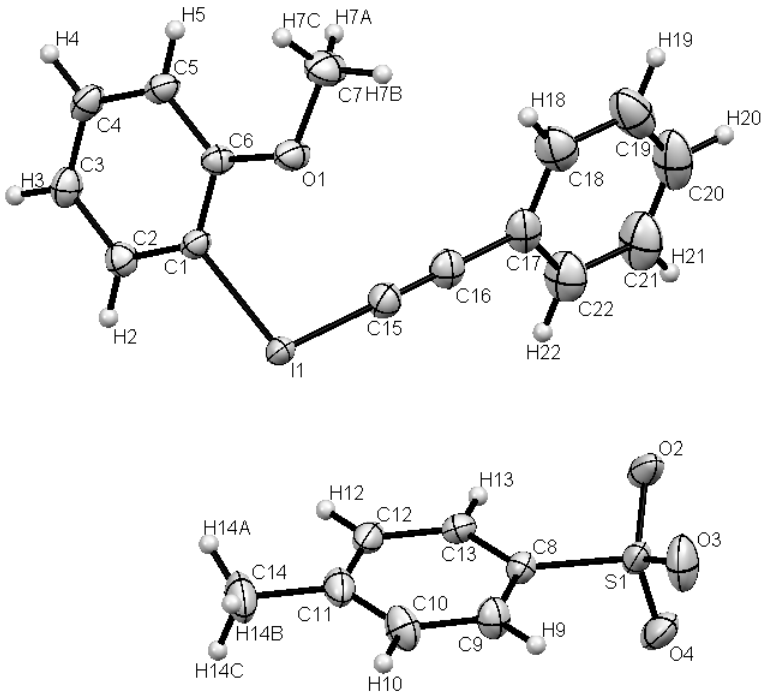
(2.5 M, 1.8 mmol) was charged and the resulting emulsion was stirred vigorously for 15 min. The aqueous layer was separated and extracted with CH_2Cl_2 (2 x 5 ml). The combined organic layers were washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* at ambient temperature, yielding a viscous yellow oil, which was dissolved in CH_2Cl_2 (3 mL). To this solution was added benzenesulfinic acid sodium salt (0.059 g, 0.36 mmol). The suspension was held at room temperature for 20 minutes upon which time deionized water (3 ml) was added. After an additional five min the aqueous layer was separated and extracted with CH_2Cl_2 (2 x 5 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo,* yielding the crude product, a yellow oil. Purification by flash column chromatography (9:1 petroleum ether/ethyl acetate) yielded a clear and colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, 2H, *J* = 7.4 Hz), 7.67-7.55 (m, 3H), 6.66 (d, 1H, *J* = 1.8 Hz), 3.01-2.92 (m, 1H), 2.62-2.56 (m, 1H), 2.54-2.45 (m, 1H), 2.30-2.22 (m, 1H), 1.59-1.53 (m, 1H), 1.11 (d, 3H, *J* = 7.0 Hz).

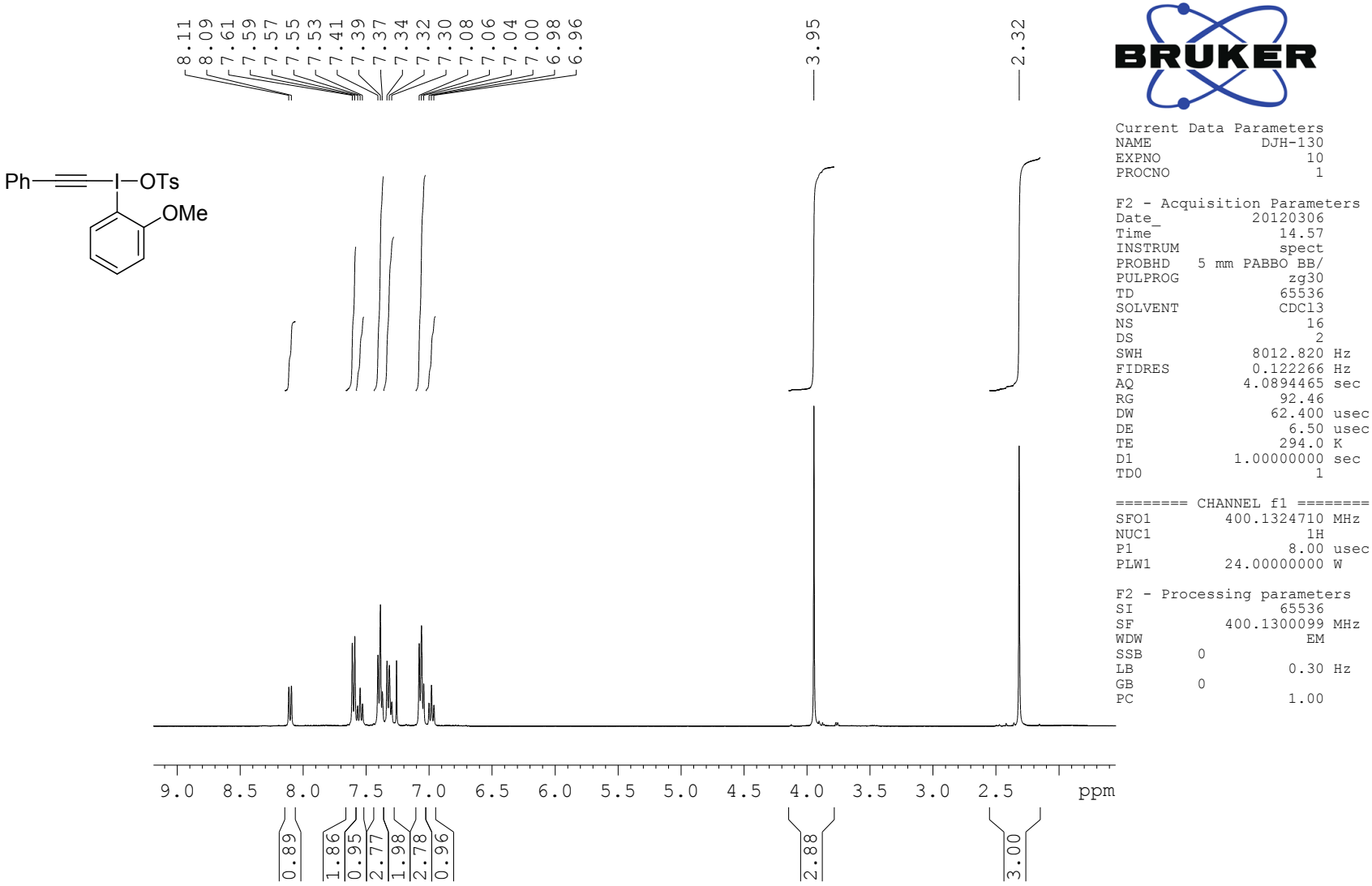
^{13}C NMR (100 MHz, $CDCl_3$): δ 148.1, 143.5, 139.6, 133.3, 129.1, 127.9, 40.6, 32.4, 30.4, 19.5.

HRMS: cald. for $C_{12}H_{14}NaO_2S$ $[MNa]^+$ 245.0607; found 245.0618.

Thermal ellipsoid plot for **1b**



^{1}H NMR spectra (400 MHz, $CDCl_3$) of phenylethynyl(2-methoxyphenyl)iodonium tosylate (**1b**)

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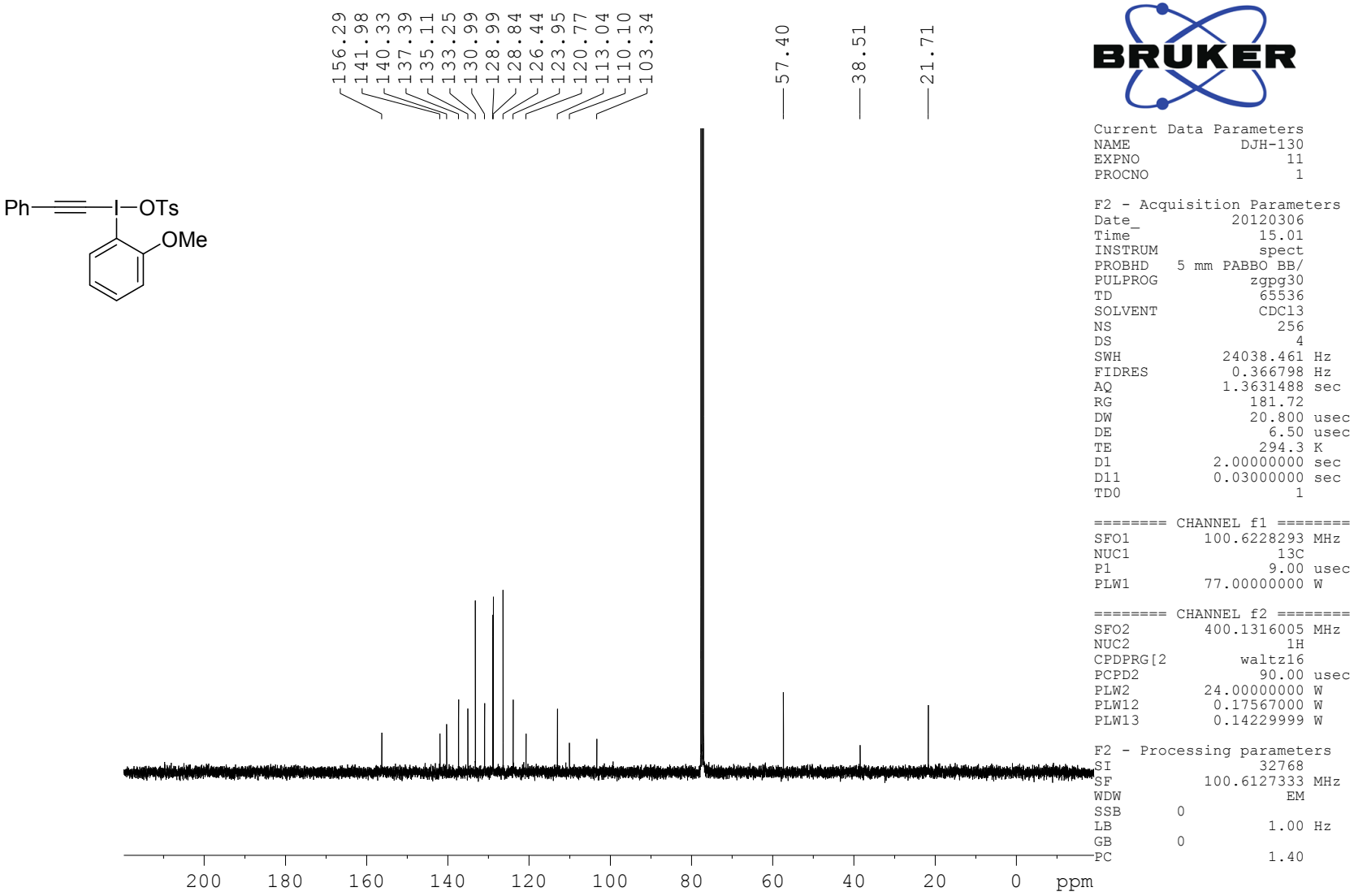
Current Data Parameters
NAME                  DJH-130
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20120306
Time                   14.57
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                     92.46
DW                     62.400 usec
DE                      6.50 usec
TE                     294.0 K
D1                  1.00000000 sec
TD0                        1

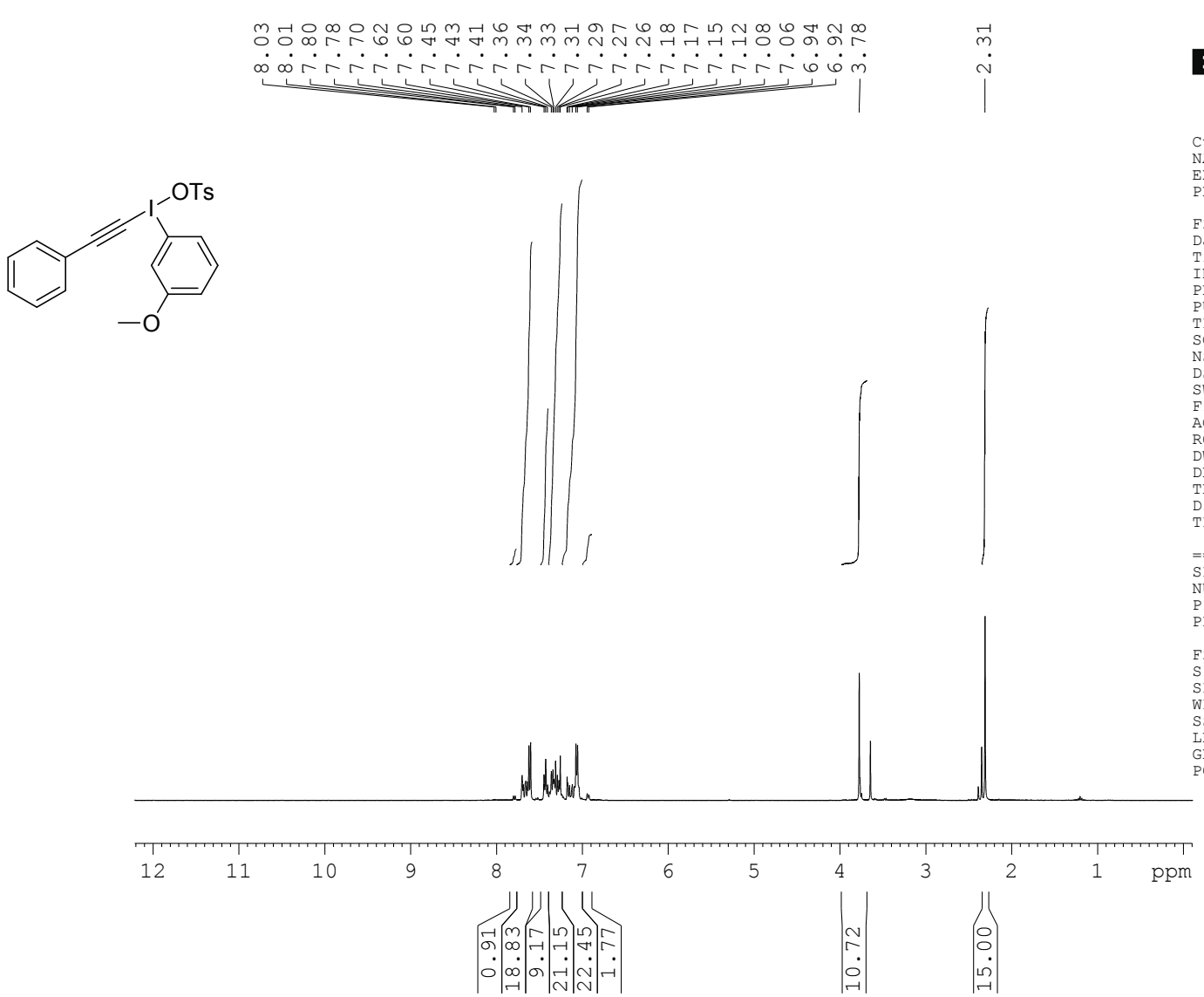
======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                       1H
P1                      8.00 usec
PLW1            24.00000000 W

F2 - Processing parameters
SI                     65536
SF              400.1300099 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of phenylethynyl(2-methoxyphenyl)iodonium tosylate (**1b**)



^{1}H NMR spectra (400 MHz, $CDCl_3$) of phenylethynyl(3-methoxyphenyl)iodonium tosylate (**1c**)



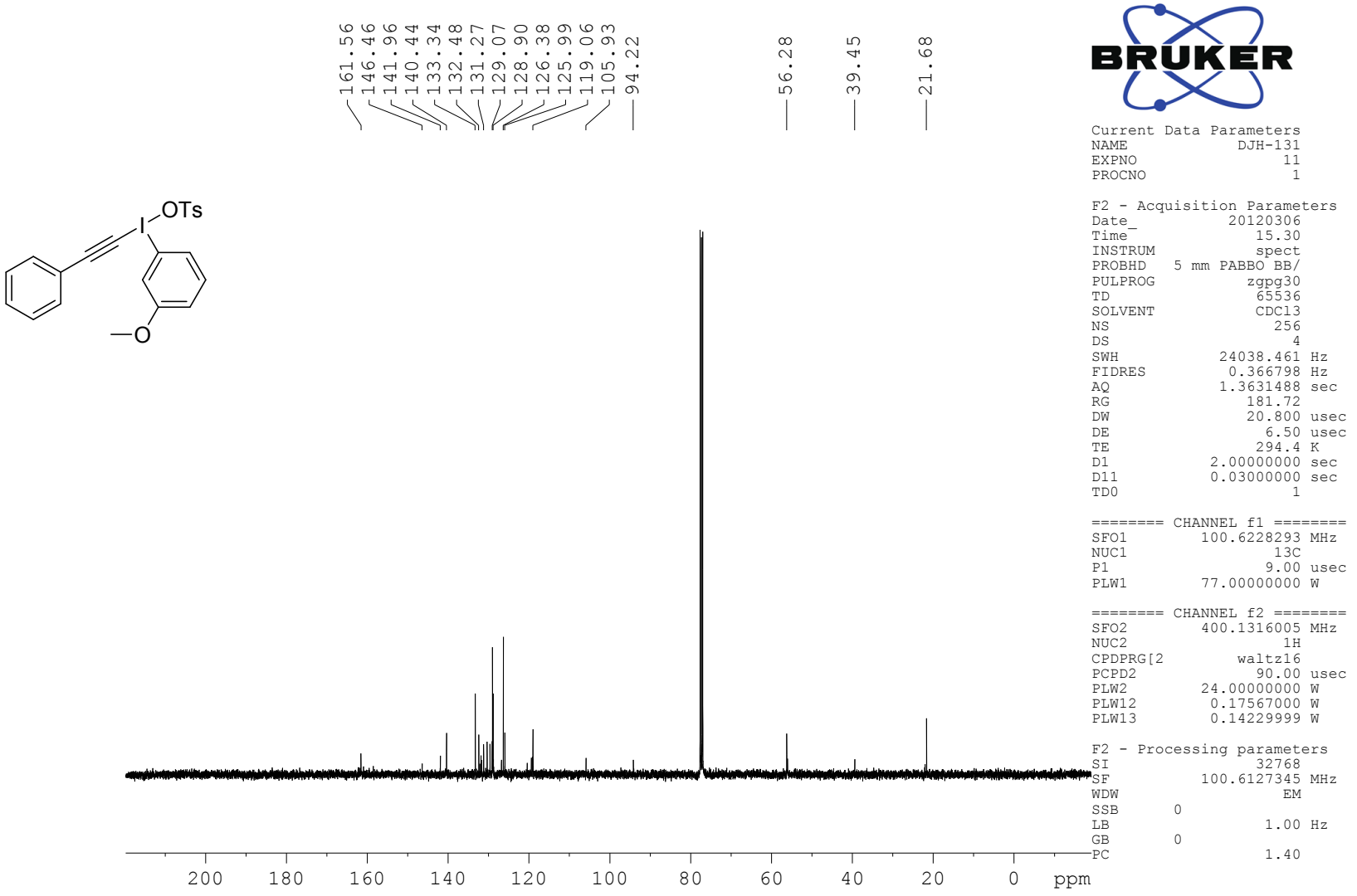

```
Current Data Parameters
NAME                  DJH-131
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_                20120306
Time                    15.17
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                     81.67
DW                     62.400 usec
DE                      6.50 usec
TE                     294.0 K
D1                  1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                      8.00 usec
PLW1            24.00000000 W

F2 - Processing parameters
SI                     65536
SF               400.1300101 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of phenylethynyl(3-methoxyphenyl)iodonium tosylate (**1c**)




```
Current Data Parameters
NAME                  DJH-131
EXPNO                      11
PROCNO                      1

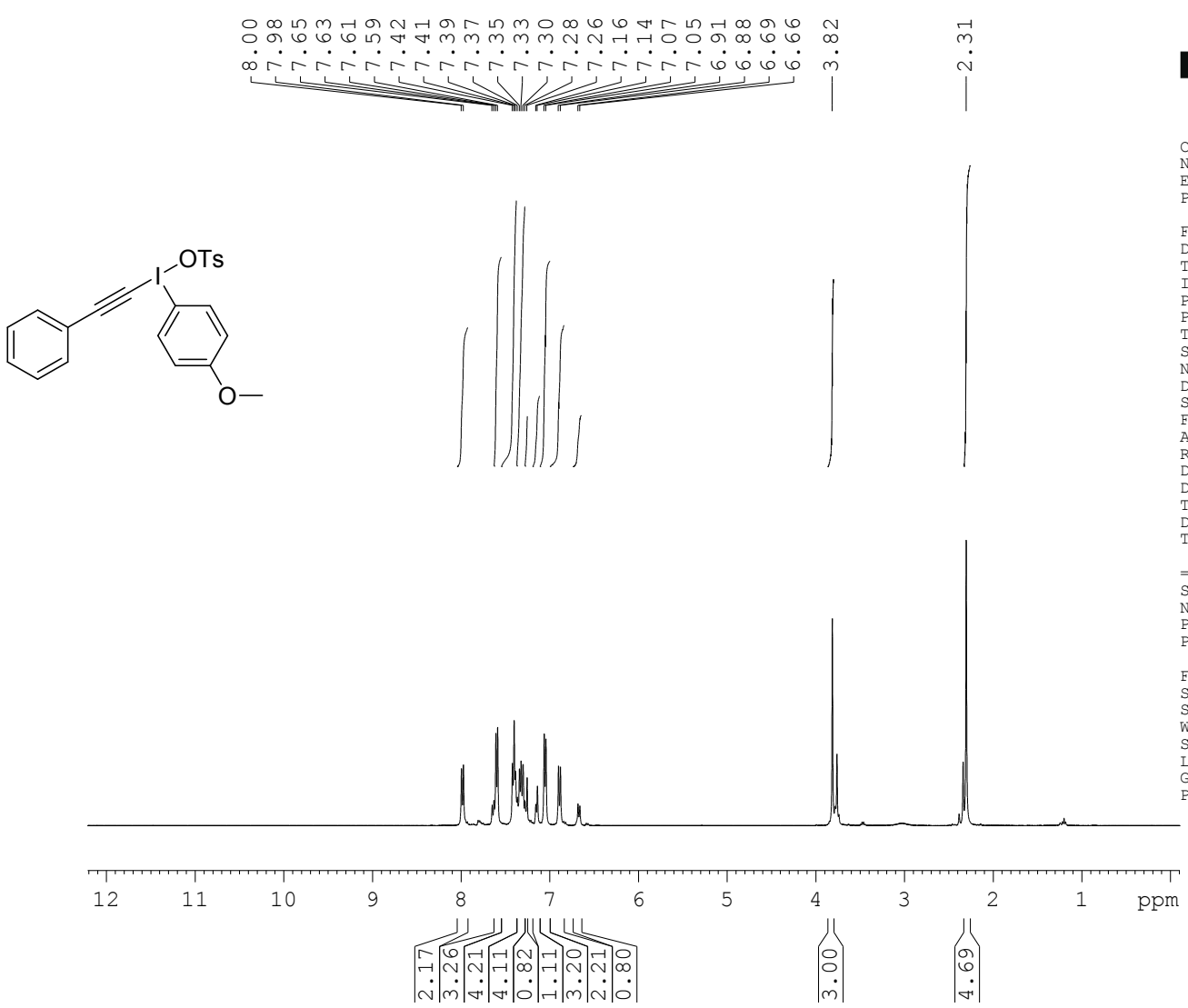
F2 - Acquisition Parameters
Date_               20120306
Time                   15.30
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        256
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                 1.3631488 sec
RG                    181.72
DW                    20.800 usec
DE                     6.50 usec
TE                    294.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             100.6228293 MHz
NUC1                     13C
P1                      9.00 usec
PLW1              77.00000000 W

======== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2              24.00000000 W
PLW12              0.17567000 W
PLW13              0.14229999 W

F2 - Processing parameters
SI                      32768
SF               100.6127345 MHz
WDW                        EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of phenylethynyl(4-methoxyphenyl)iodonium tosylate (**1d**)



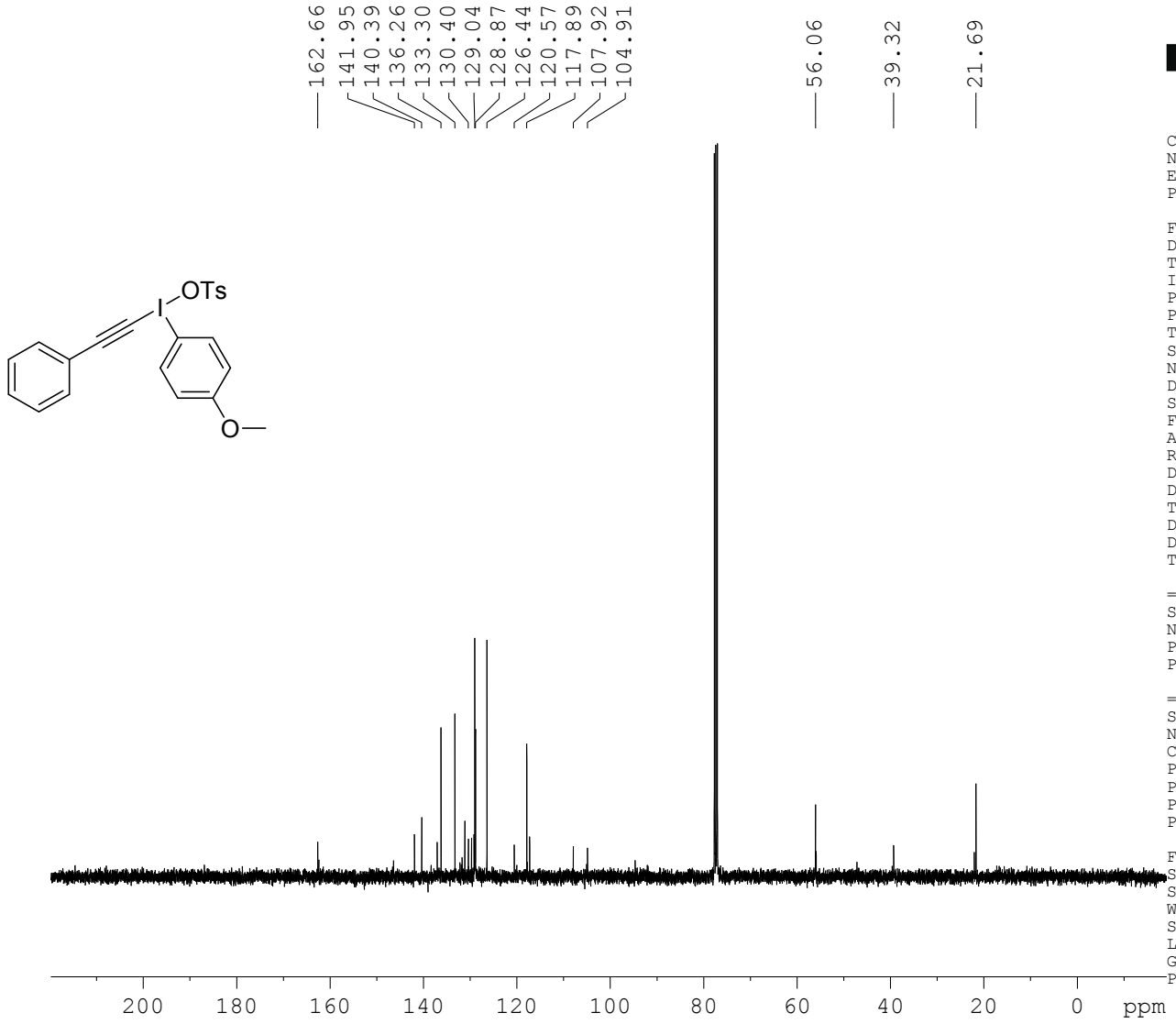

```
Current Data Parameters
NAME             DJH-132
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120306
Time               15.37
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                 81.67
DW                62.400 usec
DE                 6.50 usec
TE                294.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 8.00 usec
PLW1         24.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300102 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of phenylethynyl(3-methoxyphenyl)iodonium tosylate (**1d**)




```
Current Data Parameters
NAME            DJH-132
EXPNO                11
PROCNO                1

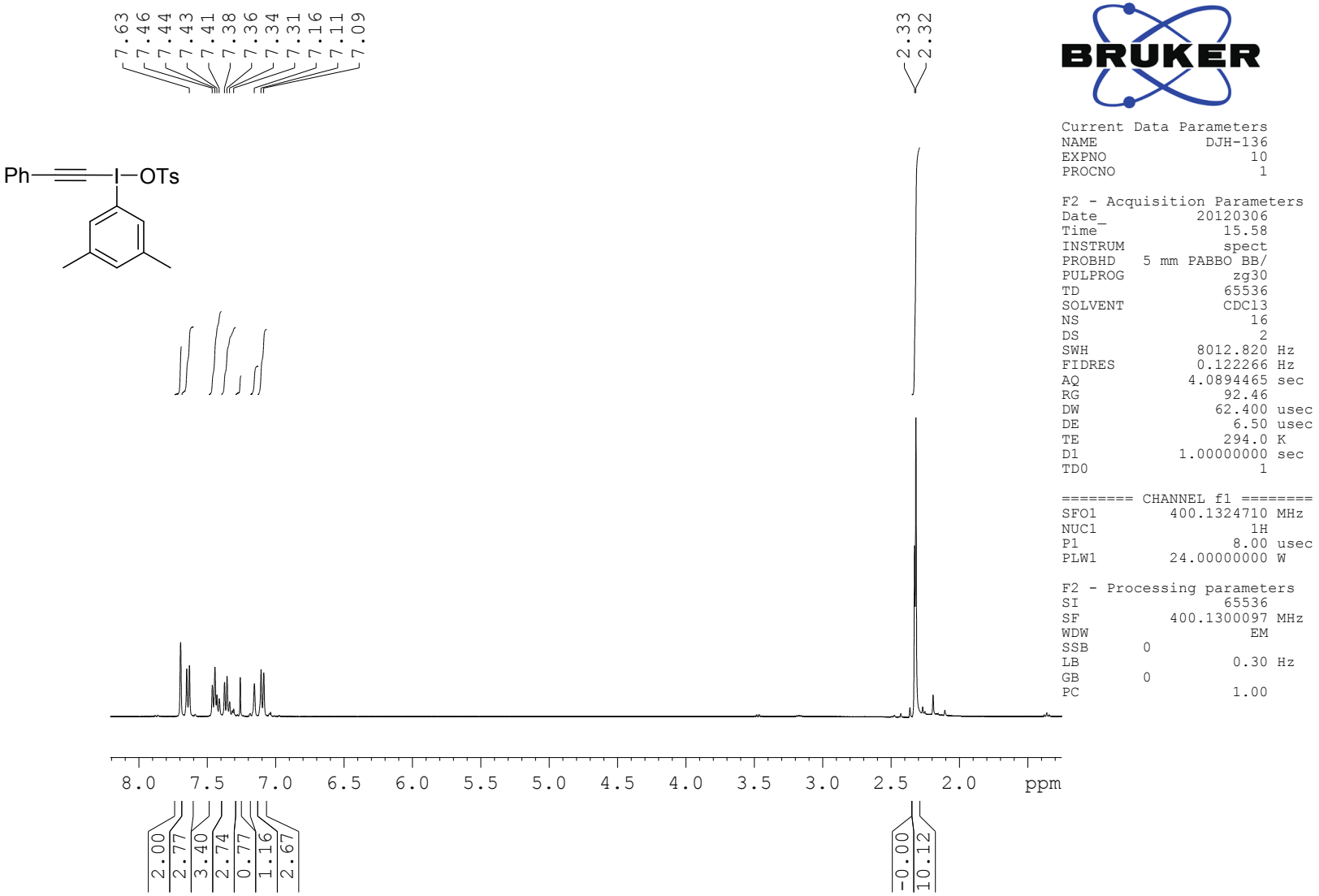
F2 - Acquisition Parameters
Date_          20120306
Time               15.53
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               181.72
DW               20.800 usec
DE                6.50 usec
TE               294.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                 9.00 usec
PLW1         77.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         24.00000000 W
PLW12         0.17567000 W
PLW13         0.14229999 W

F2 - Processing parameters
SI                 32768
SF           100.6127336 MHz
WDW                EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of phenylethynyl(2,5-dimethylphenyl)iodonium tosylate (**1f**)



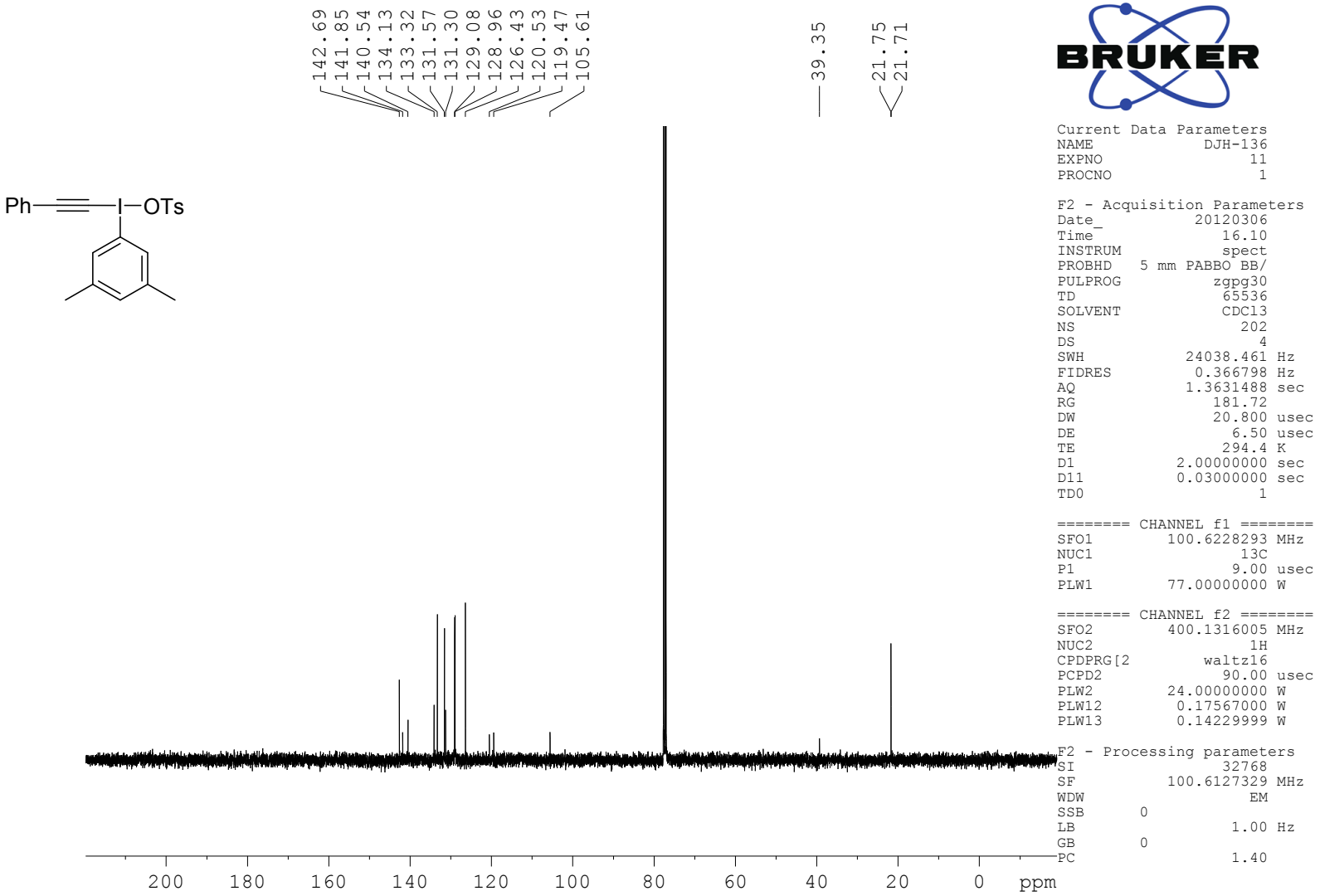

```
Current Data Parameters
NAME            DJH-136
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20120306
Time              15.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                92.46
DW               62.400 usec
DE                6.50 usec
TE                294.0 K
D1           1.00000000 sec
TD0                   1

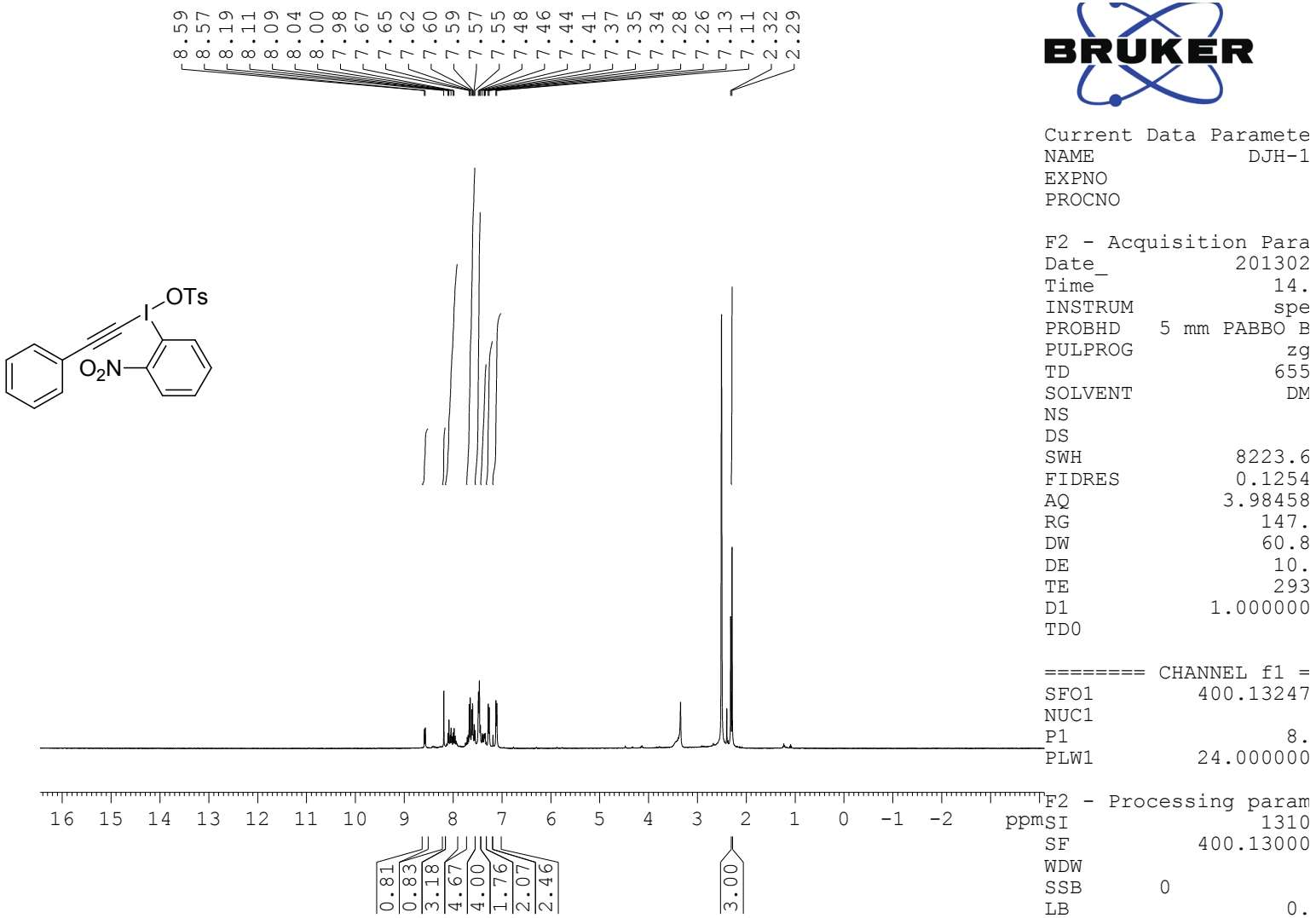
======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                8.00 usec
PLW1        24.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300097 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of phenylethynyl(2,5-dimethylphenyl)iodonium tosylate (**1f**)



^{1}H NMR spectra (400 MHz, *d*^{6}-DMSO) of phenylethynyl(2-nitrophenyl)iodonium tosylate (**1g**)



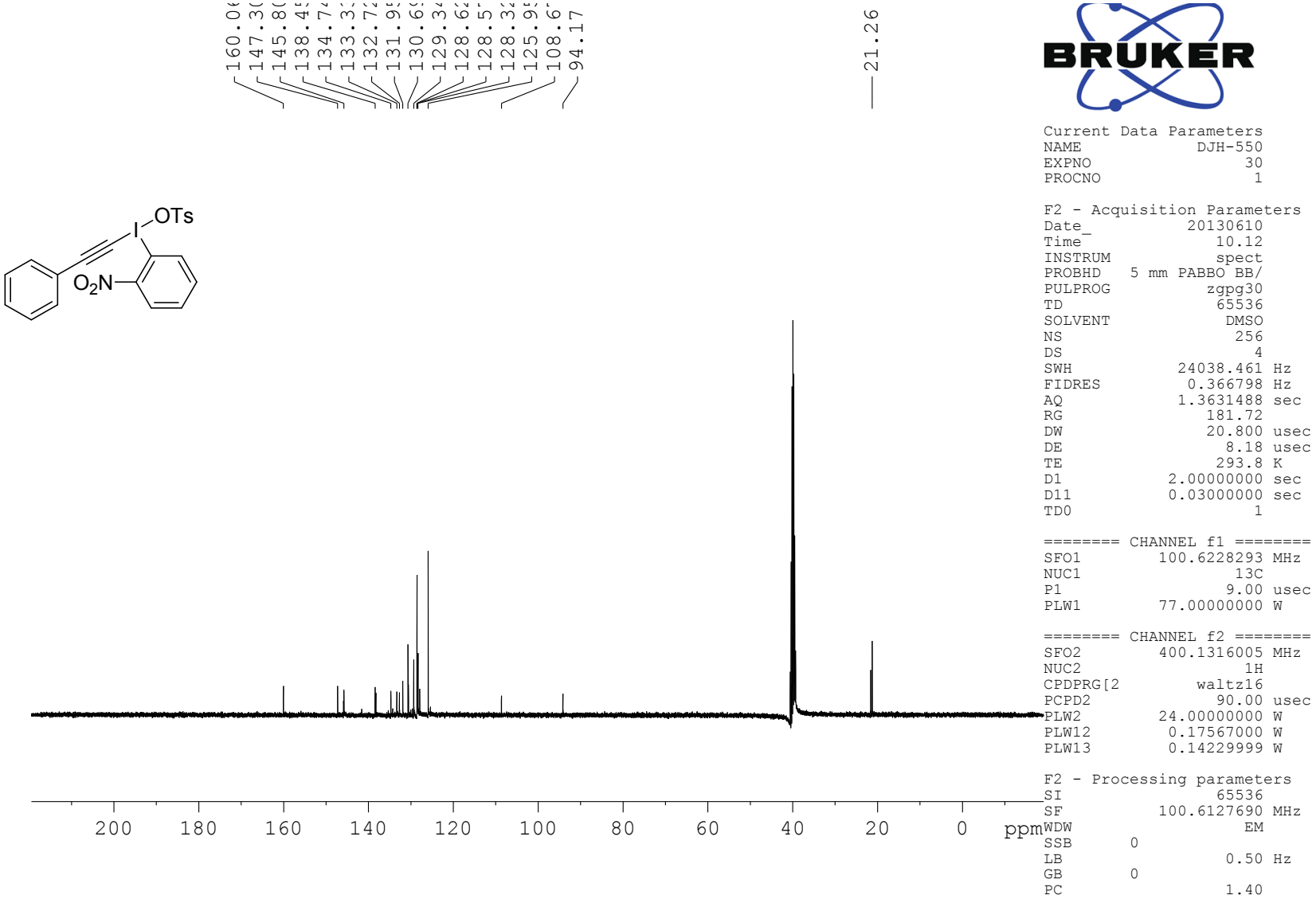

```
Current Data Paramete
NAME              DJH-1
EXPNO
PROCNO

F2 - Acquisition Para
Date_           201302
Time               14.
INSTRUM             spe
PROBHD   5 mm PABBO B
PULPROG             zg
TD                 655
SOLVENT             DM
NS
DS
SWH             8223.6
FIDRES          0.1254
AQ              3.98458
RG                 147.
DW                60.8
DE                 10.
TE                 293
D1             1.000000
TD0

======== CHANNEL f1 =
SFO1         400.13247
NUC1
P1                  8.
PLW1         24.000000

F2 - Processing param
SI                 1310
SF           400.13000
WDW
SSB       0
LB                  0.
```

^{13}C NMR spectra (100 MHz, *d*^{6}-DMSO) of phenylethynyl(2-nitrophenyl)iodonium tosylate (**1g**)




```
Current Data Parameters
NAME            DJH-550
EXPNO                30
PROCNO                1

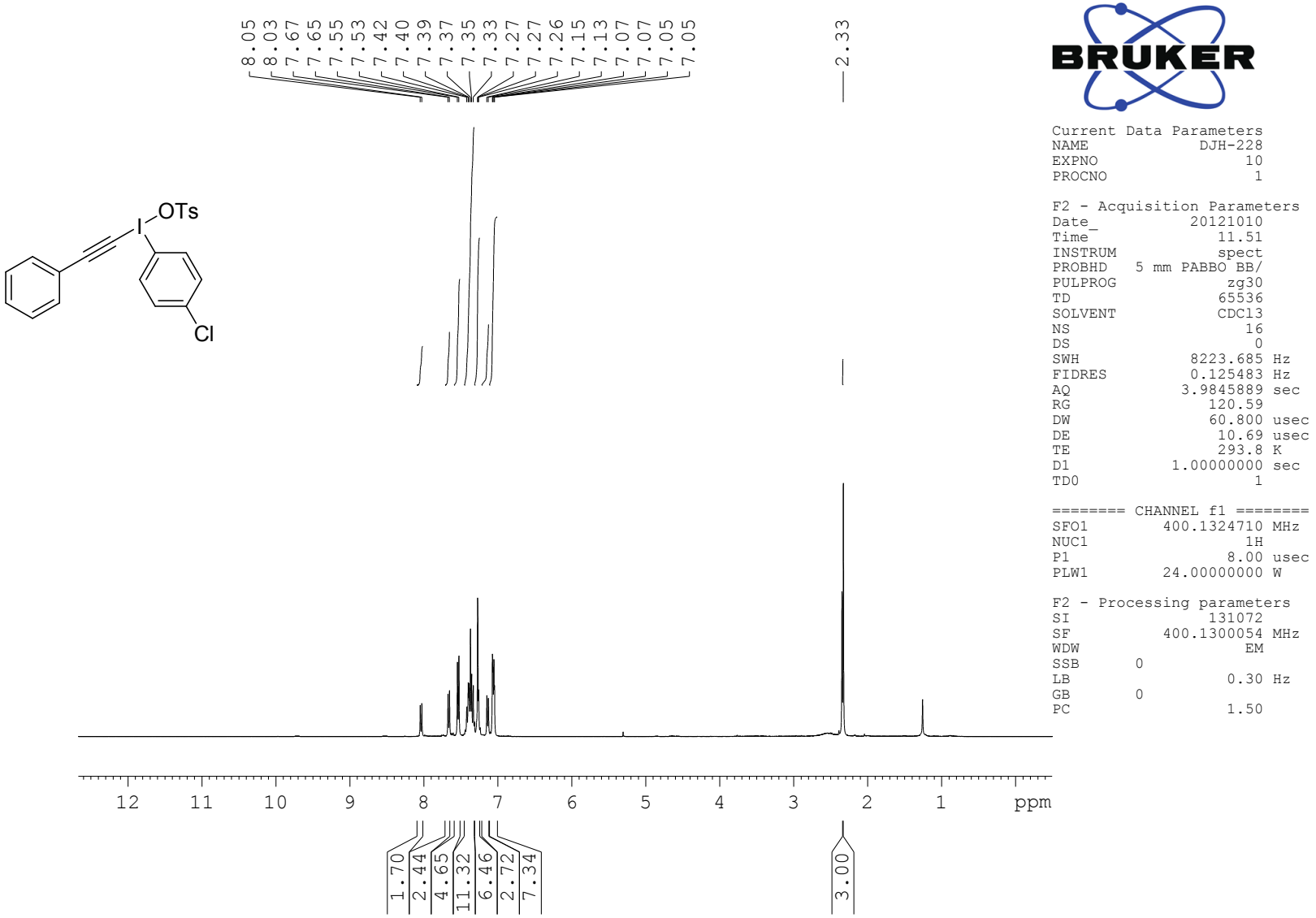
F2 - Acquisition Parameters
Date_          20130610
Time              10.12
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT             DMSO
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                181.72
DW                20.800 usec
DE                 8.18 usec
TE                293.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                 9.00 usec
PLW1         77.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         24.00000000 W
PLW12         0.17567000 W
PLW13         0.14229999 W

F2 - Processing parameters
SI                65536
SF           100.6127690 MHz
WDW                  EM
SSB       0
LB                 0.50 Hz
GB        0
PC                 1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of phenylethynyl(4-chlorophenyl)iodonium tosylate (**1h**)



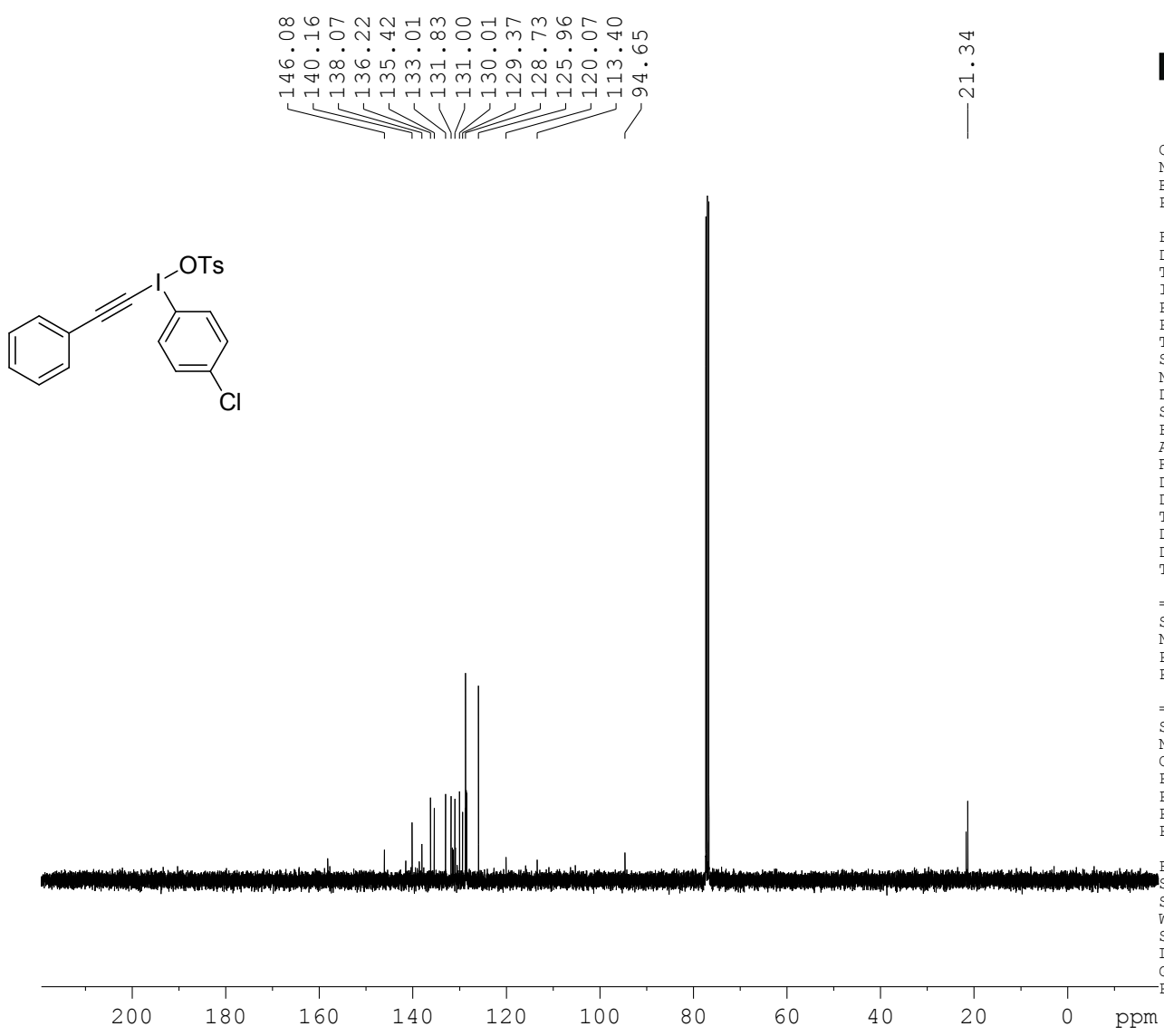

```
Current Data Parameters
NAME                  DJH-228
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20121010
Time                   11.51
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDC13
NS                        16
DS                         0
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9845889 sec
RG                     120.59
DW                     60.800 usec
DE                     10.69 usec
TE                     293.8 K
D1                  1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                      8.00 usec
PLW1            24.00000000 W

F2 - Processing parameters
SI                     131072
SF               400.1300054 MHz
WDW                        EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.50
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of phenylethynyl(4-chlorophenyl)iodonium tosylate (**1h**)




```
Current Data Parameters
NAME                  DJH-228
EXPNO                      11
PROCNO                      1

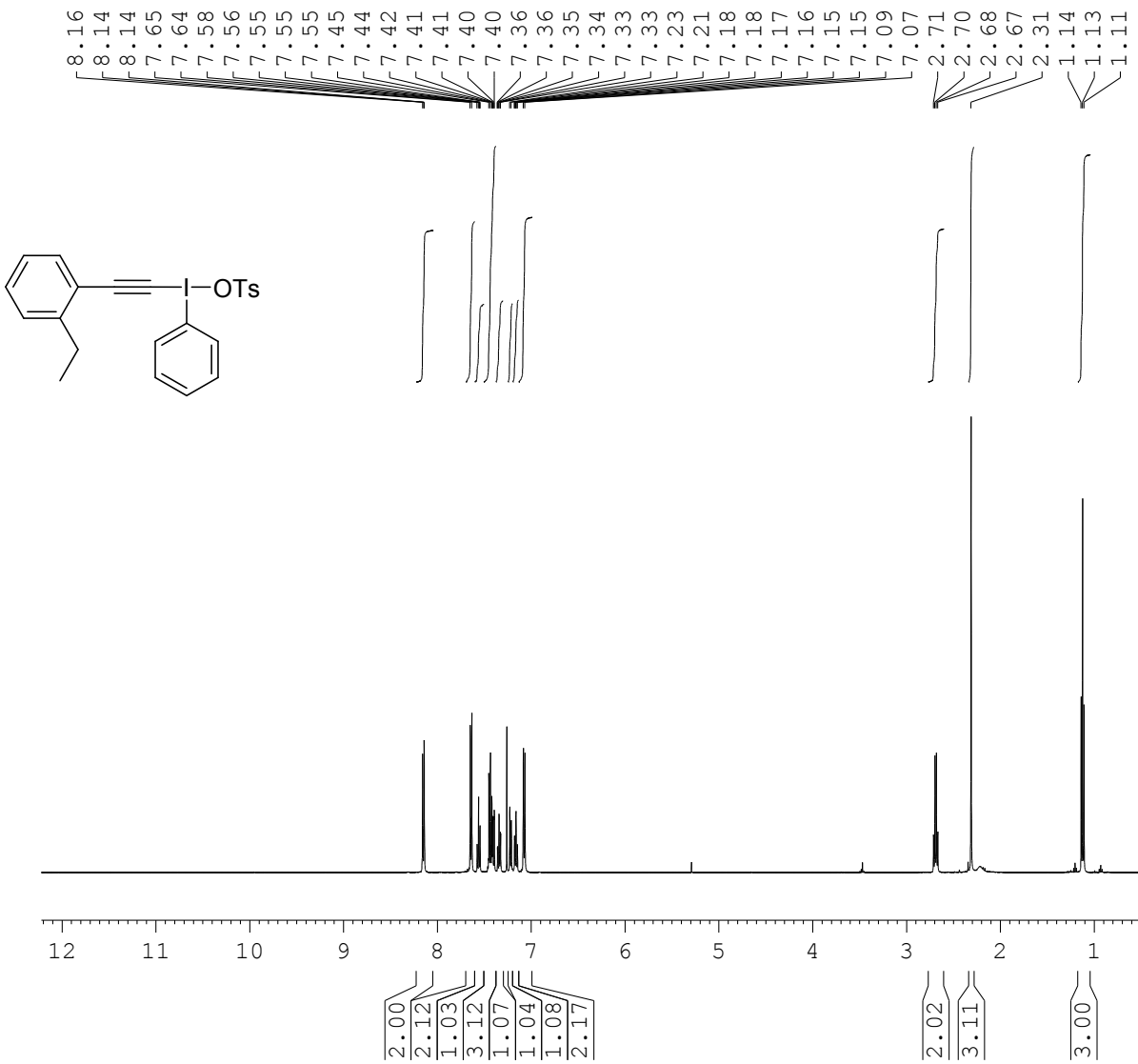
F2 - Acquisition Parameters
Date_               20121010
Time                   11.59
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        256
DS                          4
SWH                24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                    181.72
DW                    20.800 usec
DE                     8.18 usec
TE                    294.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1              100.6228293 MHz
NUC1                      13C
P1                       9.00 usec
PLW1              77.00000000 W

======== CHANNEL f2 ========
SFO2              400.1316005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                   90.00 usec
PLW2              24.00000000 W
PLW12              0.17567000 W
PLW13              0.14229999 W

F2 - Processing parameters
SI                      65536
SF                100.6127690 MHz
WDW                        EM
SSB       0
LB                       0.50 Hz
GB        0
PC                       1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(phenyl)iodonium tosylate (**2a**)



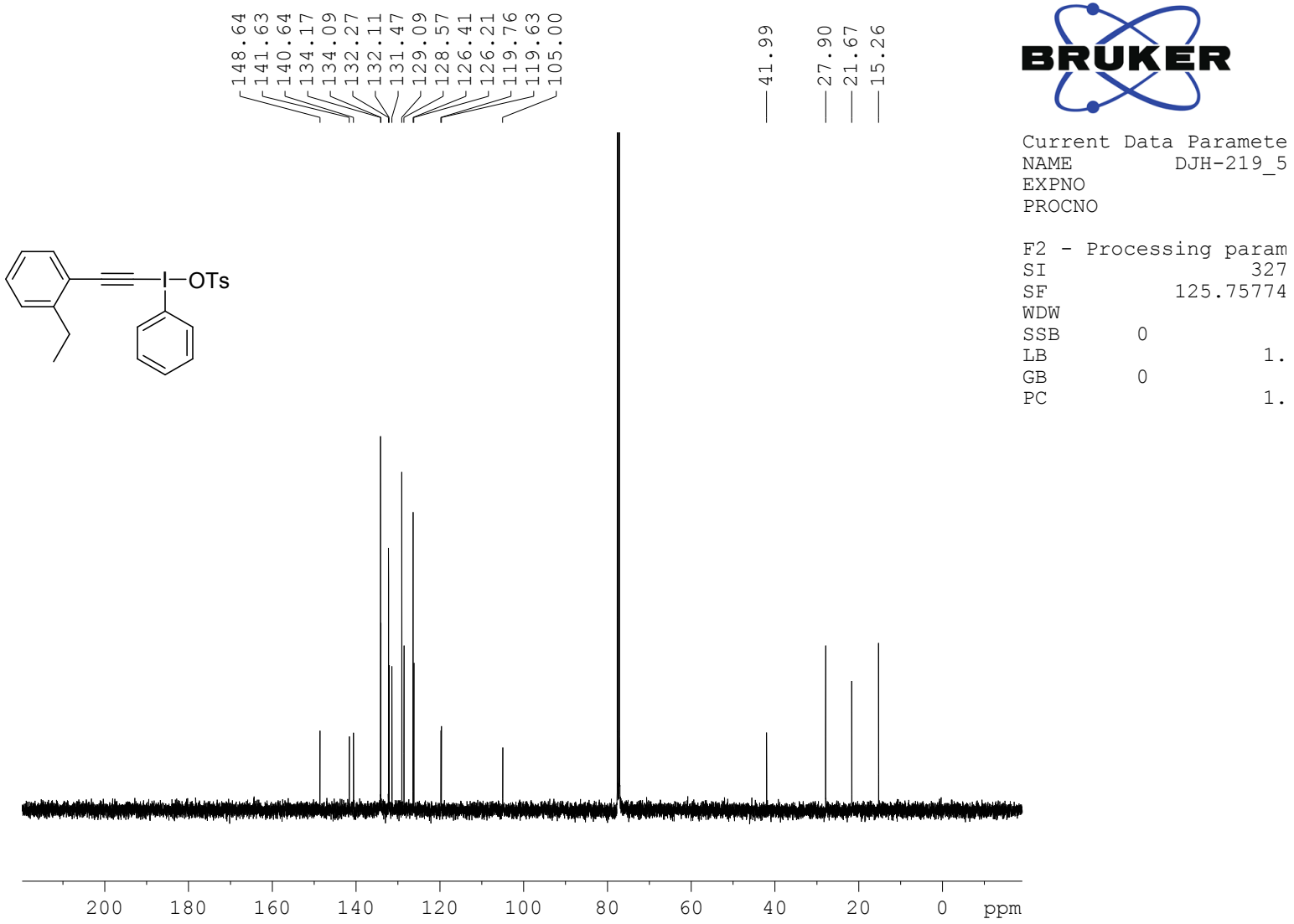

```
Current Data Parameters
NAME         DJH-219_500
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130425
Time              12.51
INSTRUM         av500sgu
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719909 sec
RG                   406
DW                48.400 usec
DE                 6.00 usec
TE                298.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1                0.10 dB
PL1W        22.82879257 W
SFO1        500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300118 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

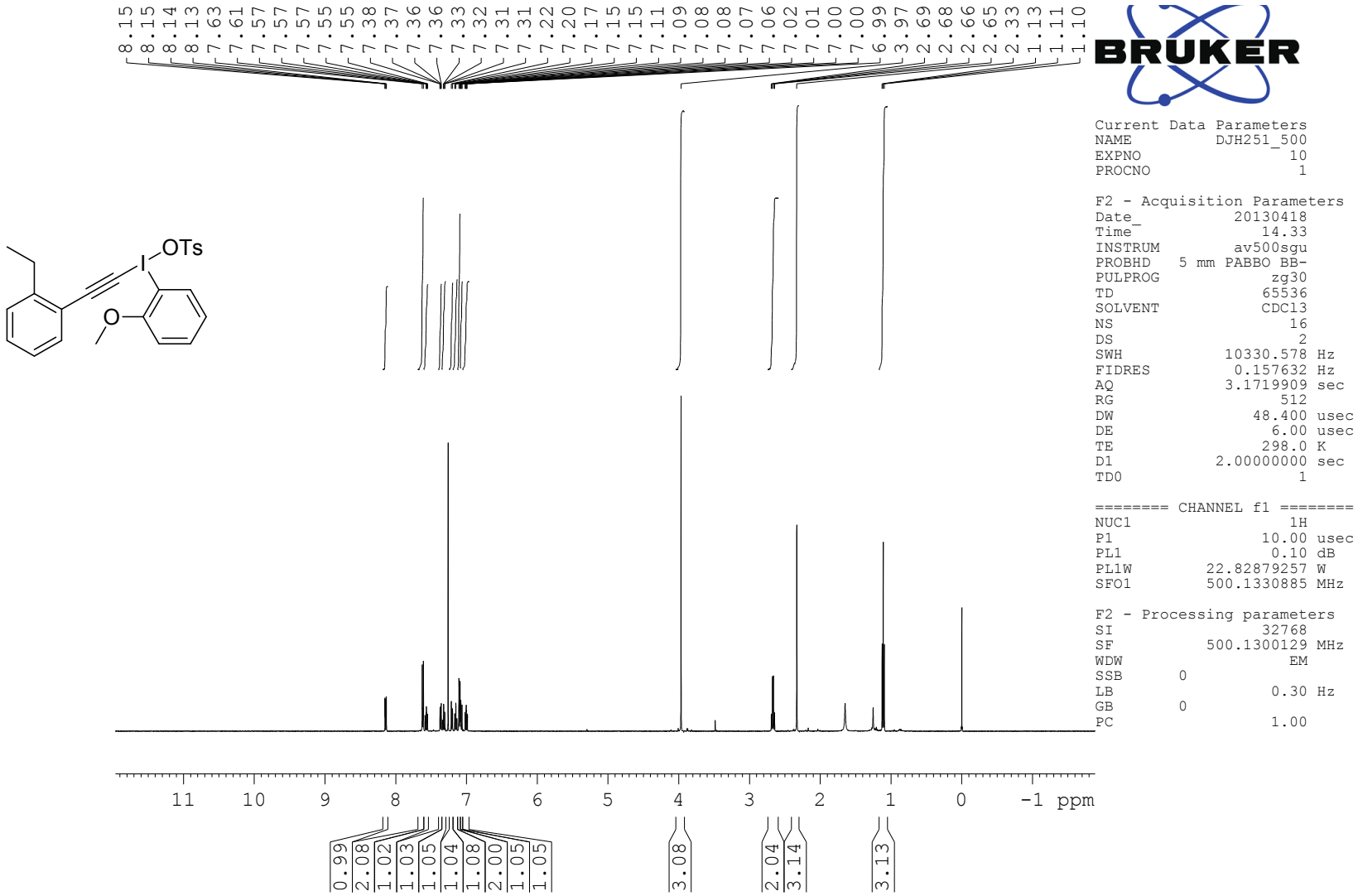
^{13}C NMR spectra (100 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(phenyl)iodonium tosylate (**2a**)




```
Current Data Paramete
NAME         DJH-219_5
EXPNO
PROCNO

F2 - Processing param
SI                 327
SF          125.75774
WDW
SSB        0
LB                  1.
GB         0
PC                  1.
```

^{1}H NMR spectra (500 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(2-methoxyphenyl)iodonium tosylate (**2b**)



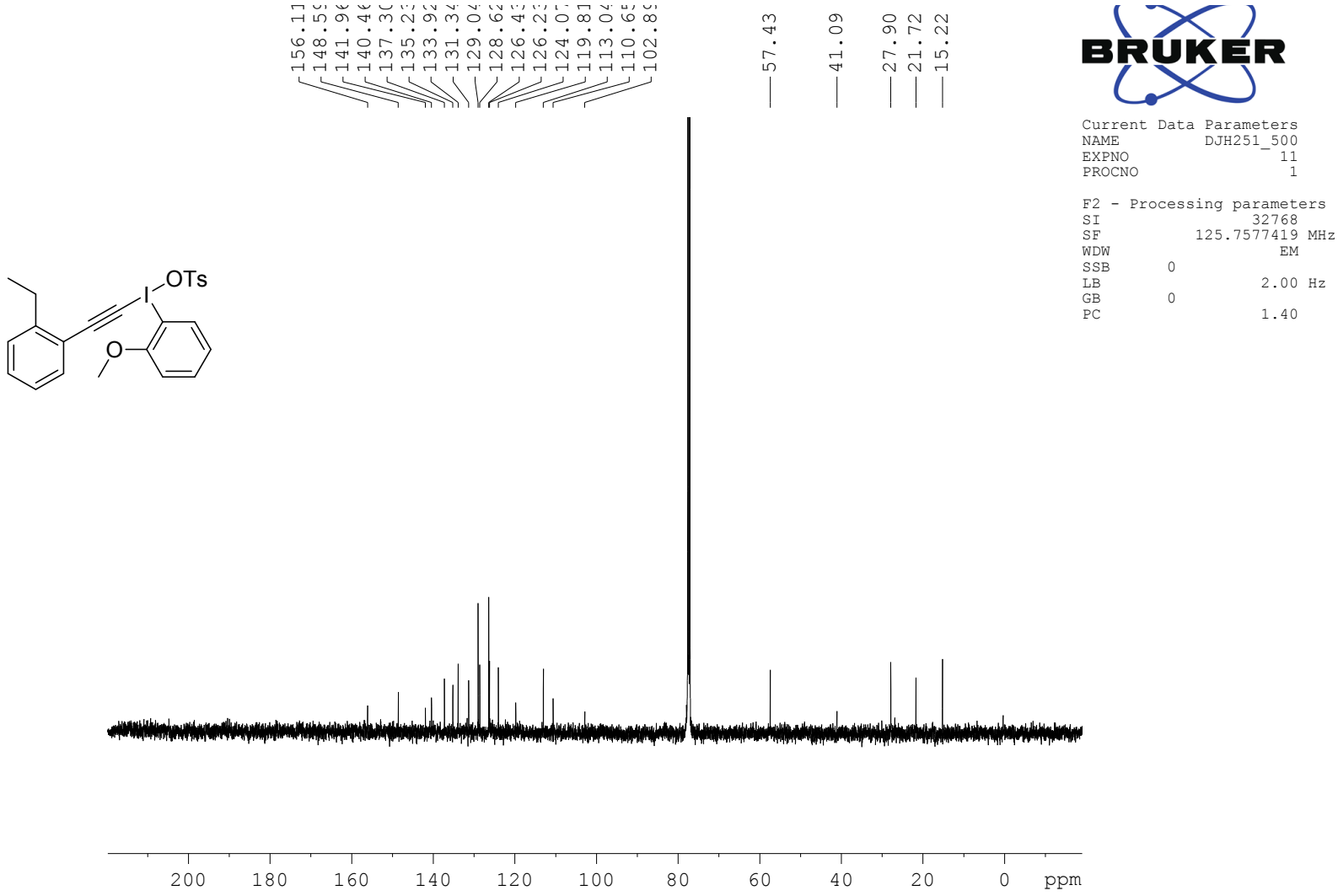

```
Current Data Parameters
NAME          DJH251_500
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130418
Time              14.33
INSTRUM         av500sgu
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719909 sec
RG                   512
DW                48.400 usec
DE                 6.00 usec
TE                298.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1                0.10 dB
PL1W        22.82879257 W
SFO1        500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300129 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

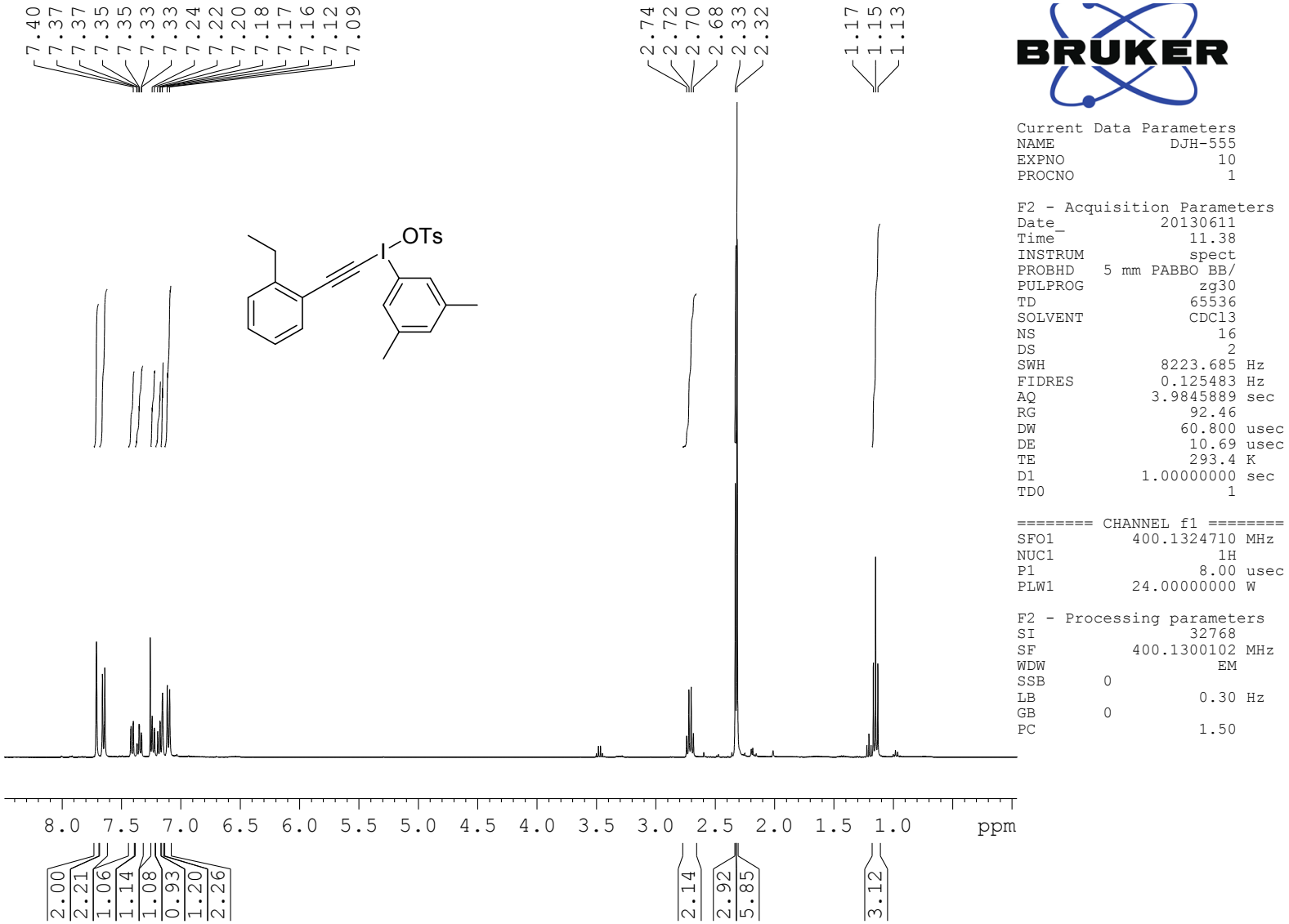
^{13}C NMR spectra (125 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(2-methoxyphenyl)iodonium tosylate (**2b**)




```
Current Data Parameters
NAME          DJH251_500
EXPNO                 11
PROCNO                 1

F2 - Processing parameters
SI                 32768
SF          125.7577419 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(3,5-dimethylphenyl)iodonium tosylate (**2c**)



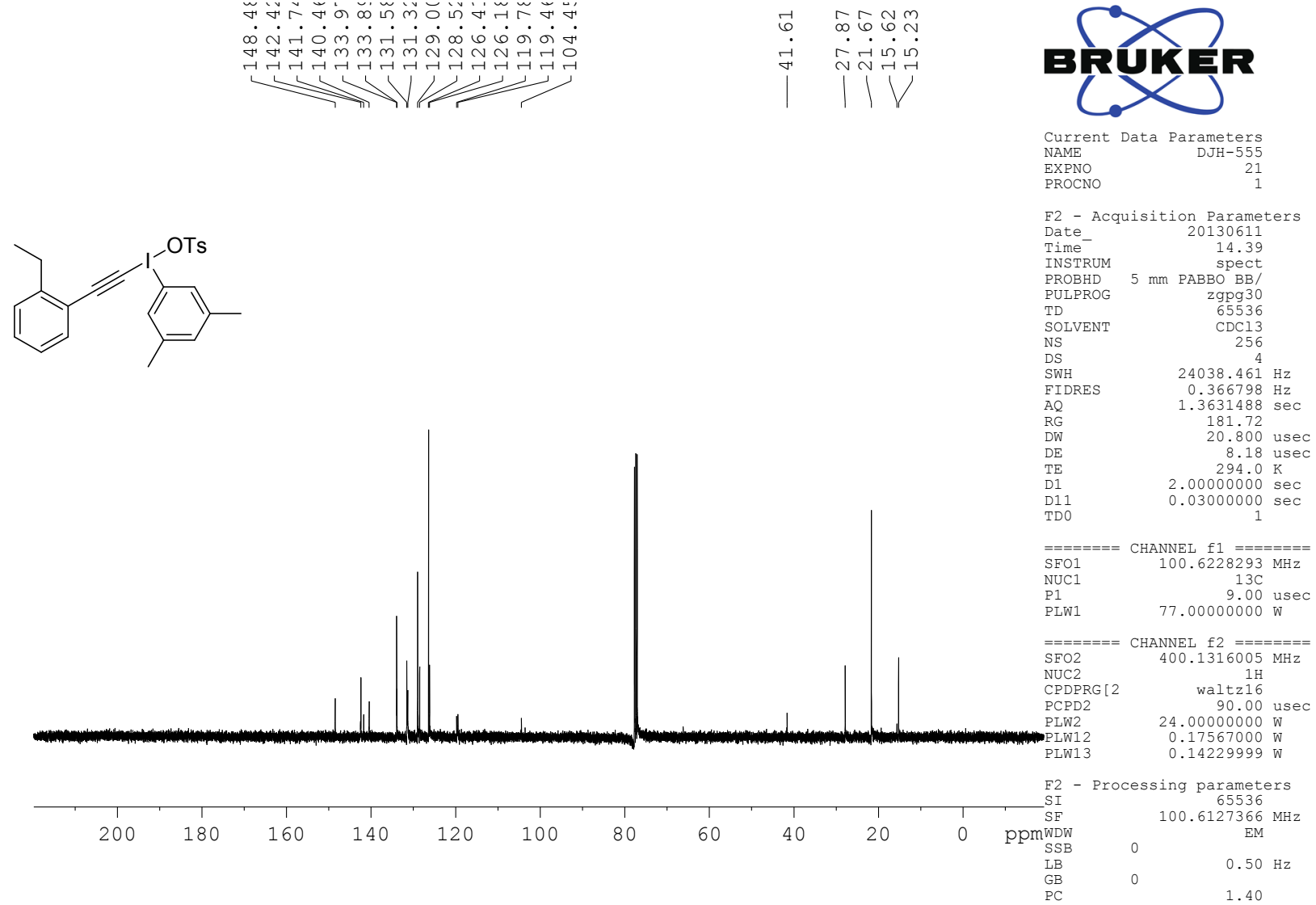

```
Current Data Parameters
NAME                  DJH-555
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20130611
Time                   11.38
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9845889 sec
RG                     92.46
DW                    60.800 usec
DE                    10.69 usec
TE                    293.4 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                       1H
P1                      8.00 usec
PLW1            24.00000000 W

F2 - Processing parameters
SI                        32768
SF              400.1300102 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.50
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(3,5-dimethylphenyl)iodonium tosylate (**2c**)




```
Current Data Parameters
NAME             DJH-555
EXPNO                 21
PROCNO                 1

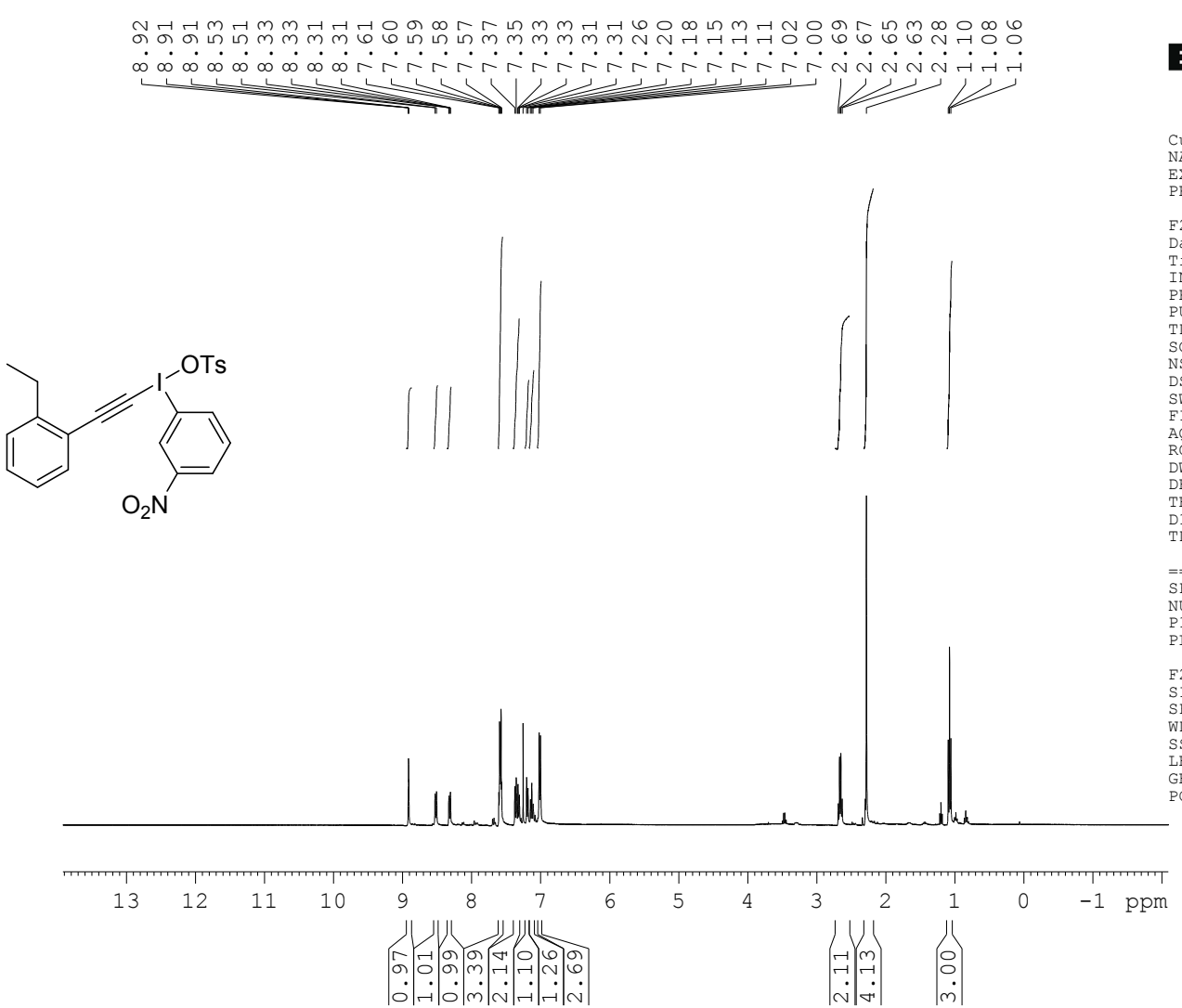
F2 - Acquisition Parameters
Date_           20130611
Time               14.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                181.72
DW                20.800 usec
DE                 8.18 usec
TE                294.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                 9.00 usec
PLW1         77.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         24.00000000 W
PLW12         0.17567000 W
PLW13         0.14229999 W

F2 - Processing parameters
SI                 65536
SF           100.6127366 MHz
WDW                   EM
SSB       0
LB                 0.50 Hz
GB        0
PC                 1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(3-nitrophenyl)iodonium tosylate (**2d**)



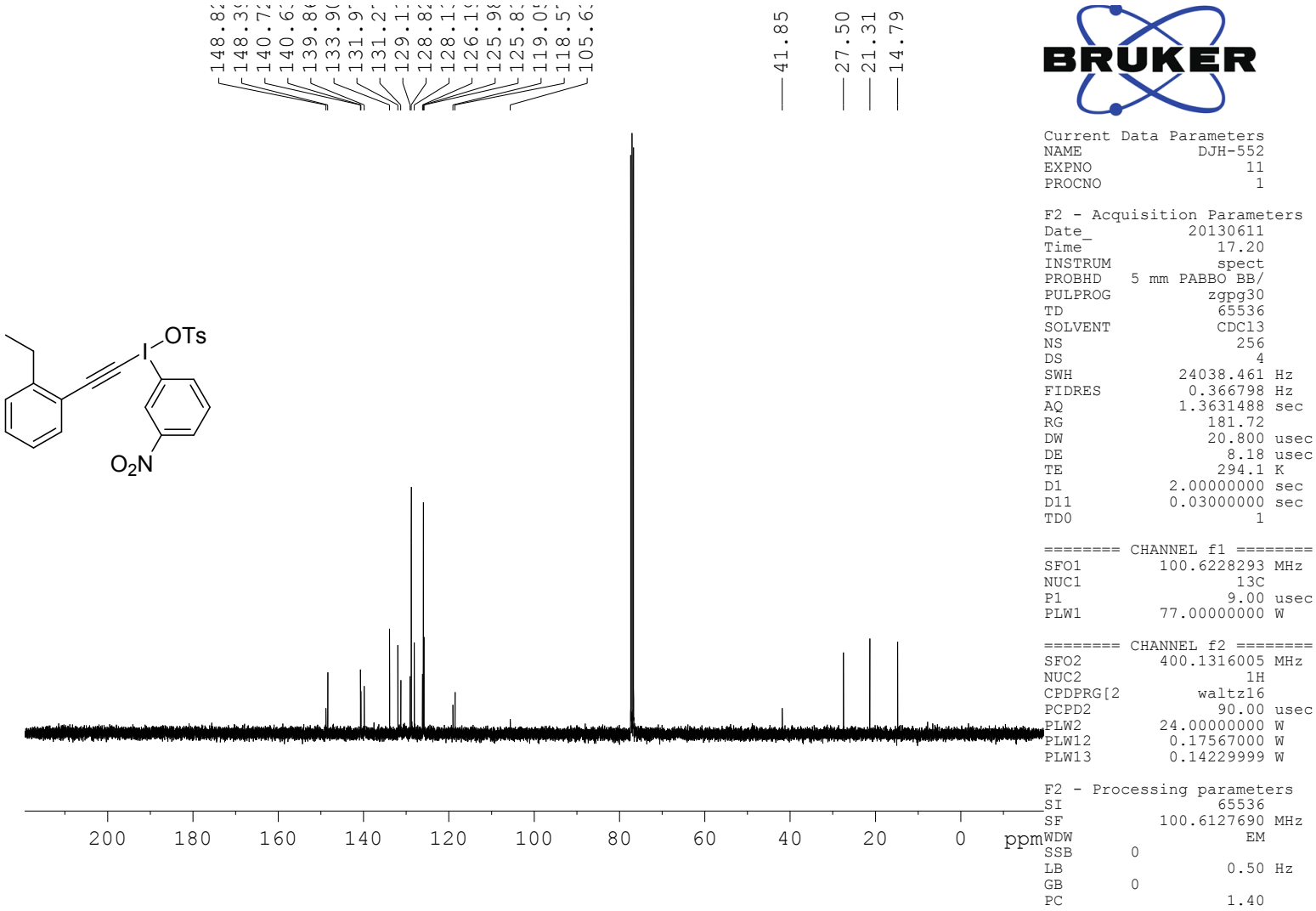

```
Current Data Parameters
NAME              DJH-552
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130611
Time                17.04
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9845889 sec
RG                  81.67
DW                 60.800 usec
DE                 10.69 usec
TE                 293.6 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  8.00 usec
PLW1         24.00000000 W

F2 - Processing parameters
SI                  32768
SF           400.1300114 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.50
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(3-nitrophenyl)iodonium tosylate (**2d**)




```
Current Data Parameters
NAME             DJH-552
EXPNO                 11
PROCNO                 1

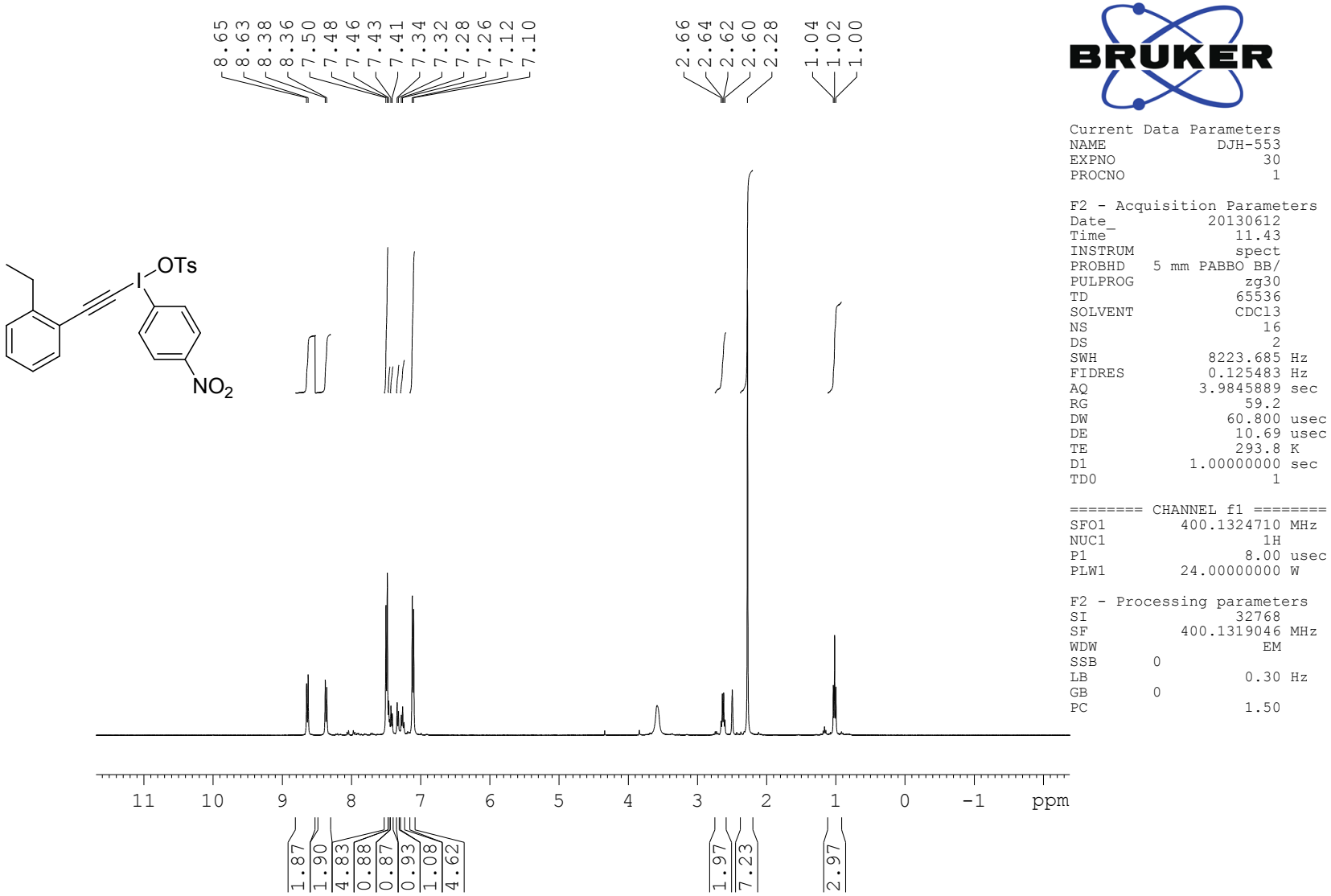
F2 - Acquisition Parameters
Date_           20130611
Time               17.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               181.72
DW               20.800 usec
DE                8.18 usec
TE               294.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                 9.00 usec
PLW1        77.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        24.00000000 W
PLW12        0.17567000 W
PLW13        0.14229999 W

F2 - Processing parameters
SI                65536
SF          100.6127690 MHz
WDW                  EM
SSB       0
LB                0.50 Hz
GB        0
PC                1.40
```

^{1}H NMR spectra (400 MHz, *d*^{6}-DMSO) of 1-ethyl-2-ethynylbezene(4-nitrophenyl)iodonium tosylate (**2e**)



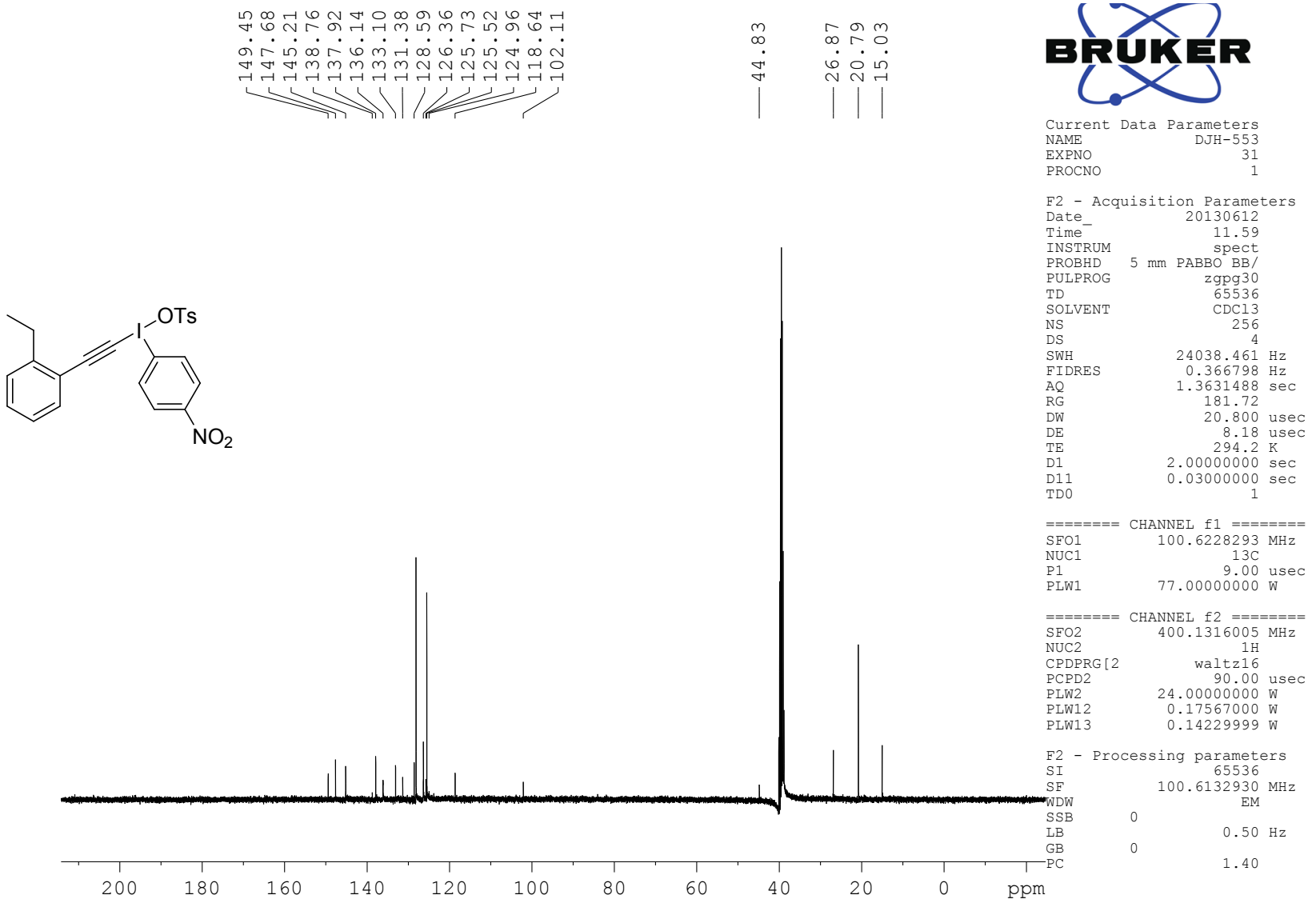

```
Current Data Parameters
NAME            DJH-553
EXPNO                30
PROCNO                1

F2 - Acquisition Parameters
Date_          20130612
Time              11.43
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9845889 sec
RG                59.2
DW               60.800 usec
DE               10.69 usec
TE               293.8 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                8.00 usec
PLW1         24.00000000 W

F2 - Processing parameters
SI                32768
SF           400.1319046 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.50
```

^{13}C NMR spectra (100 MHz, d^6-DMSO) of 1-ethyl-2-ethynylbezene(4-nitrophenyl)iodonium tosylate (**2e**)




```
Current Data Parameters
NAME            DJH-553
EXPNO                31
PROCNO                1

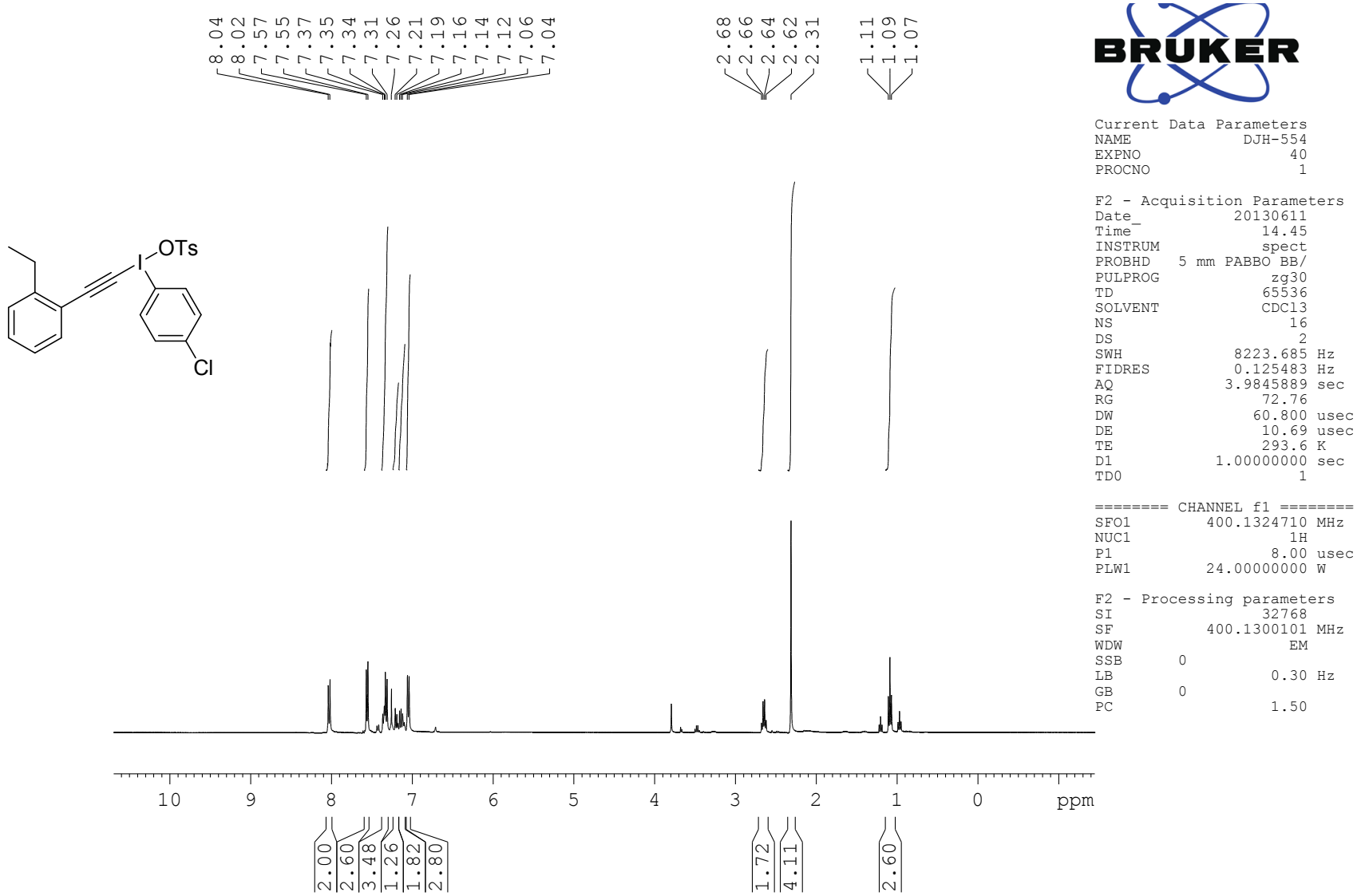
F2 - Acquisition Parameters
Date_          20130612
Time              11.59
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                181.72
DW                20.800 usec
DE                 8.18 usec
TE                294.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                 9.00 usec
PLW1         77.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         24.00000000 W
PLW12         0.17567000 W
PLW13         0.14229999 W

F2 - Processing parameters
SI                 65536
SF           100.6132930 MHz
WDW                  EM
SSB       0
LB                 0.50 Hz
GB        0
PC                 1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(4-chlorophenyl)iodonium tosylate (**2f**)



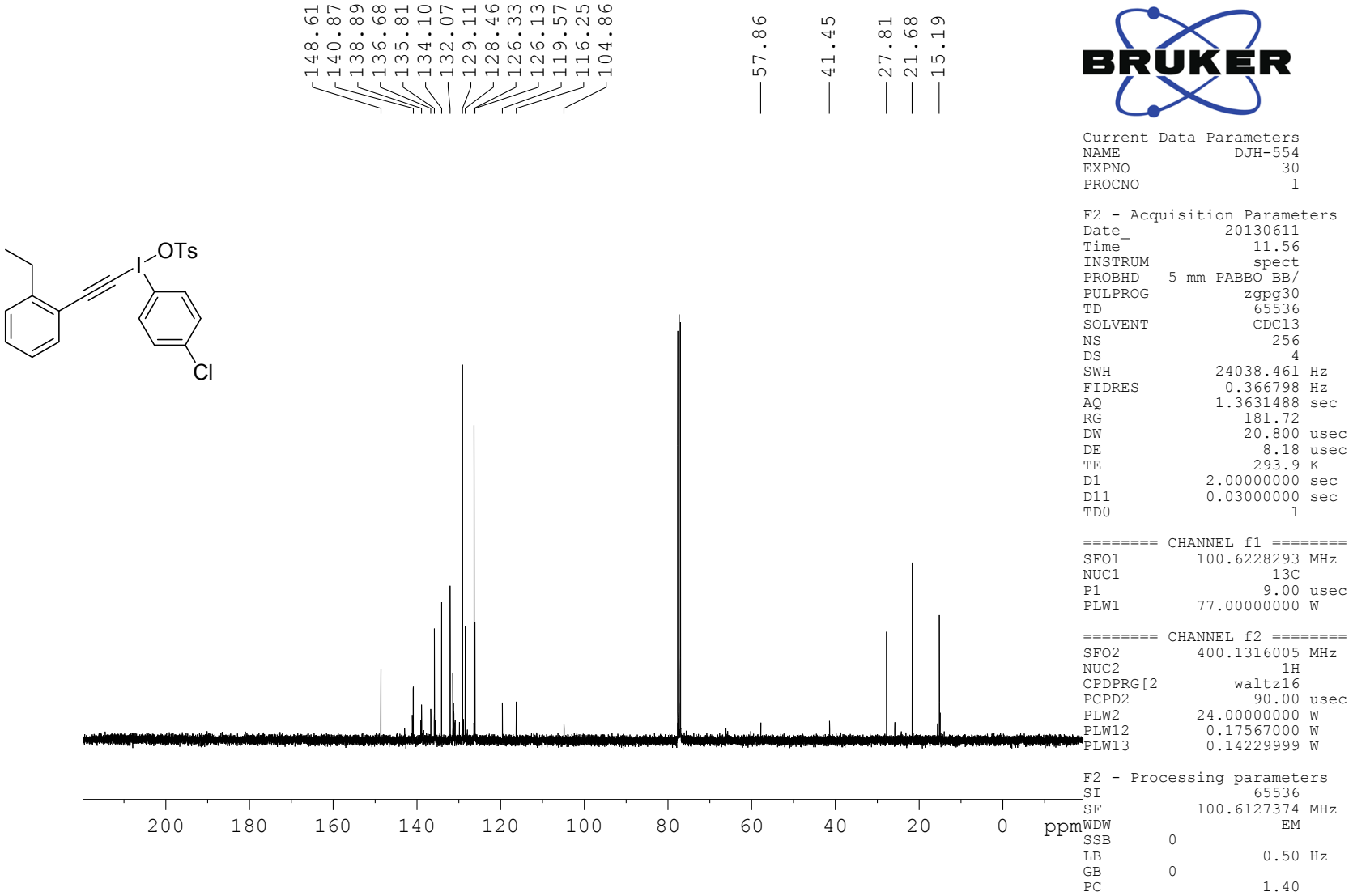

```
Current Data Parameters
NAME            DJH-554
EXPNO                40
PROCNO                1

F2 - Acquisition Parameters
Date_          20130611
Time              14.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9845889 sec
RG                72.76
DW               60.800 usec
DE               10.69 usec
TE                293.6 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                8.00 usec
PLW1        24.00000000 W

F2 - Processing parameters
SI                32768
SF           400.1300101 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.50
```

^{13}C NMR spectra (100 MHz, $CDCl_3$) of 1-ethyl-2-ethynylbezene(4-chlorophenyl)iodonium tosylate (**2f**)




```
Current Data Parameters
NAME             DJH-554
EXPNO                 30
PROCNO                 1

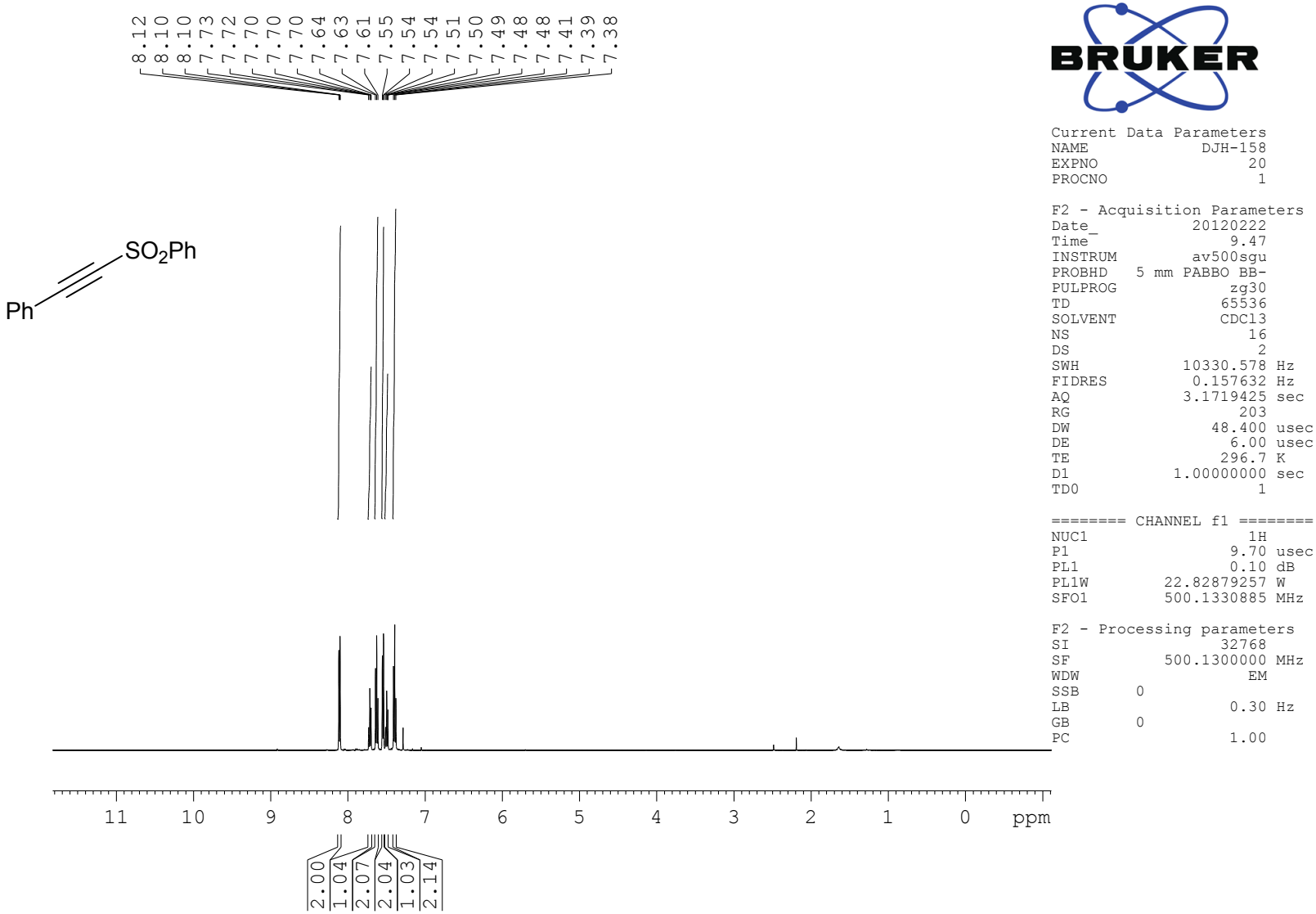
F2 - Acquisition Parameters
Date_           20130611
Time               11.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                181.72
DW                20.800 usec
DE                 8.18 usec
TE                293.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                 9.00 usec
PLW1         77.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         24.00000000 W
PLW12         0.17567000 W
PLW13         0.14229999 W

F2 - Processing parameters
SI                 65536
SF           100.6127374 MHz
WDW                   EM
SSB       0
LB                 0.50 Hz
GB        0
PC                 1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of 1-benzenesulfonyl-3-methyl-indene (**3**)



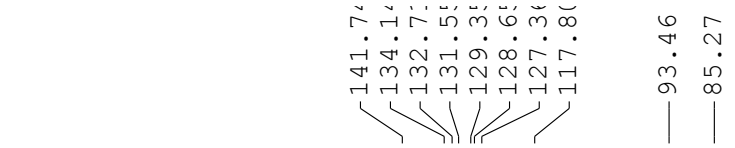

```
Current Data Parameters
NAME                 DJH-158
EXPNO                     20
PROCNO                     1

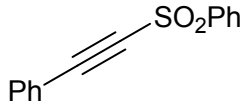
F2 - Acquisition Parameters
Date_               20120222
Time                    9.47
INSTRUM              av500sgu
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                10330.578 Hz
FIDRES              0.157632 Hz
AQ                  3.1719425 sec
RG                       203
DW                    48.400 usec
DE                     6.00 usec
TE                    296.7 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.70 usec
PL1                    0.10 dB
PL1W            22.82879257 W
SFO1           500.1330885 MHz

F2 - Processing parameters
SI                     32768
SF               500.1300000 MHz
WDW                       EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```

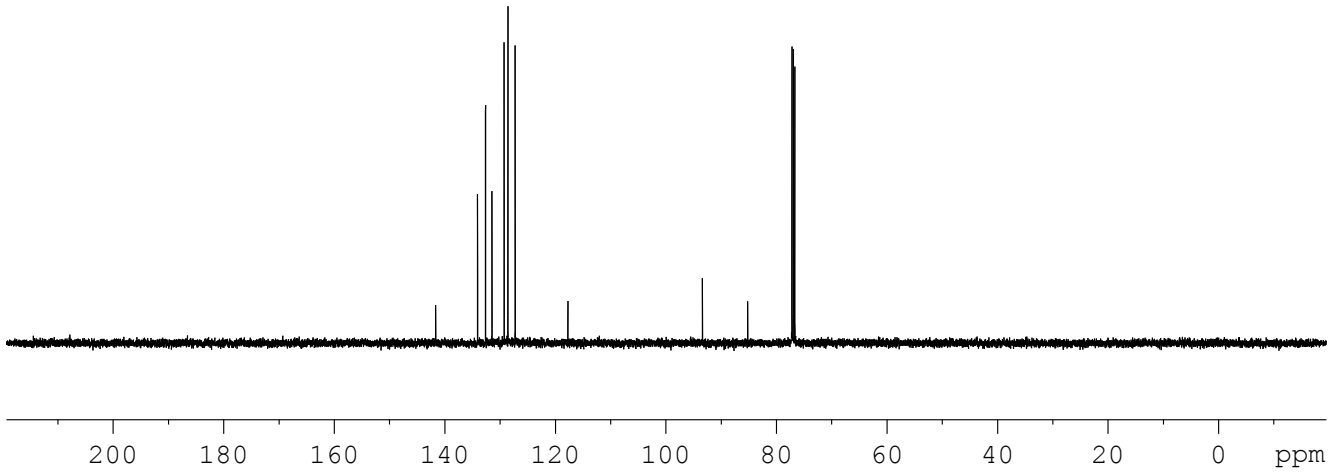
^{13}C NMR spectra (100 MHz, $CDCl_3$) of 1-benzenesulfonyl-3-methyl-indene (**3**)



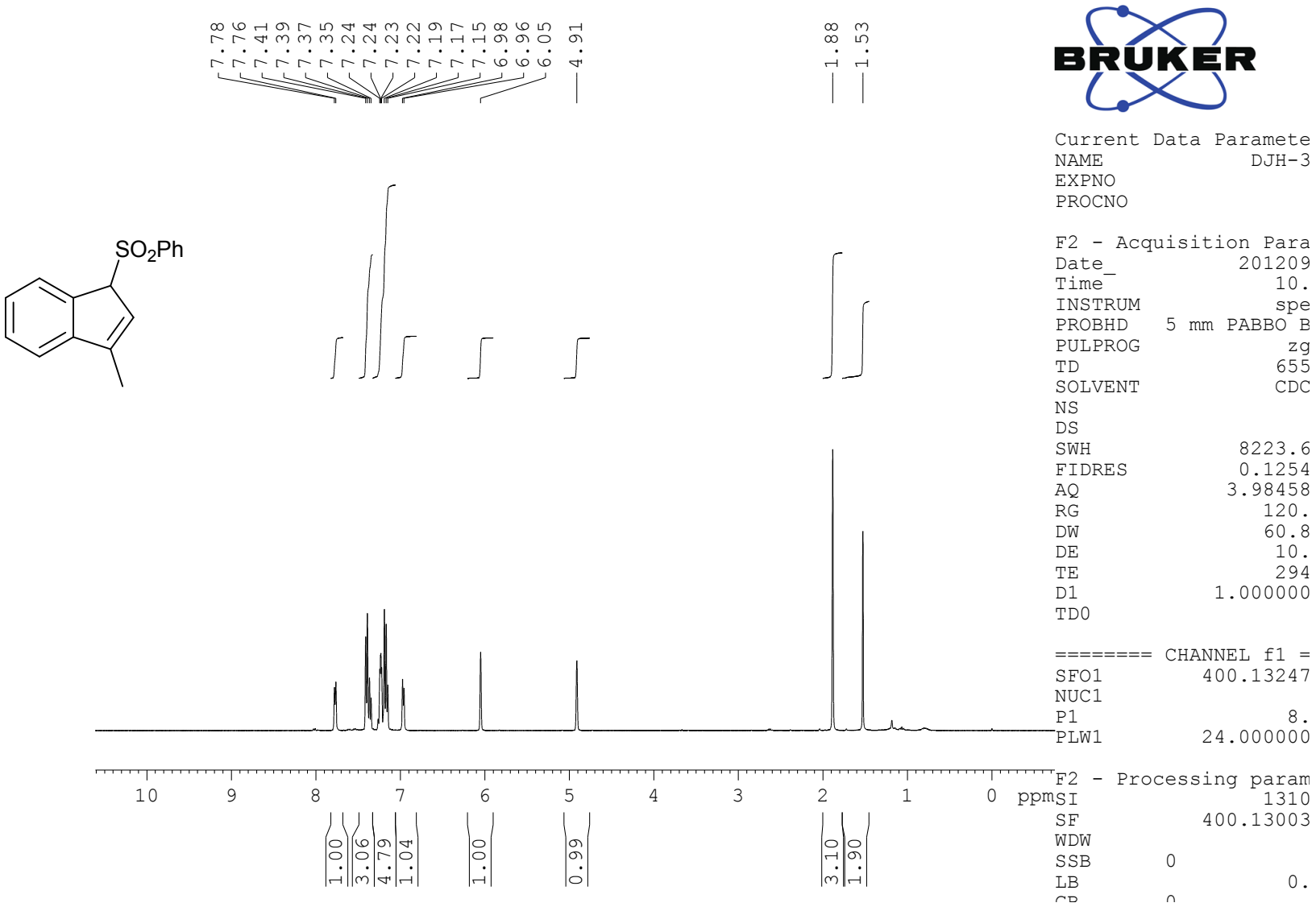



```
Current Data Parameters
NAME                  DJH-158
EXPNO                      21
PROCNO                      1

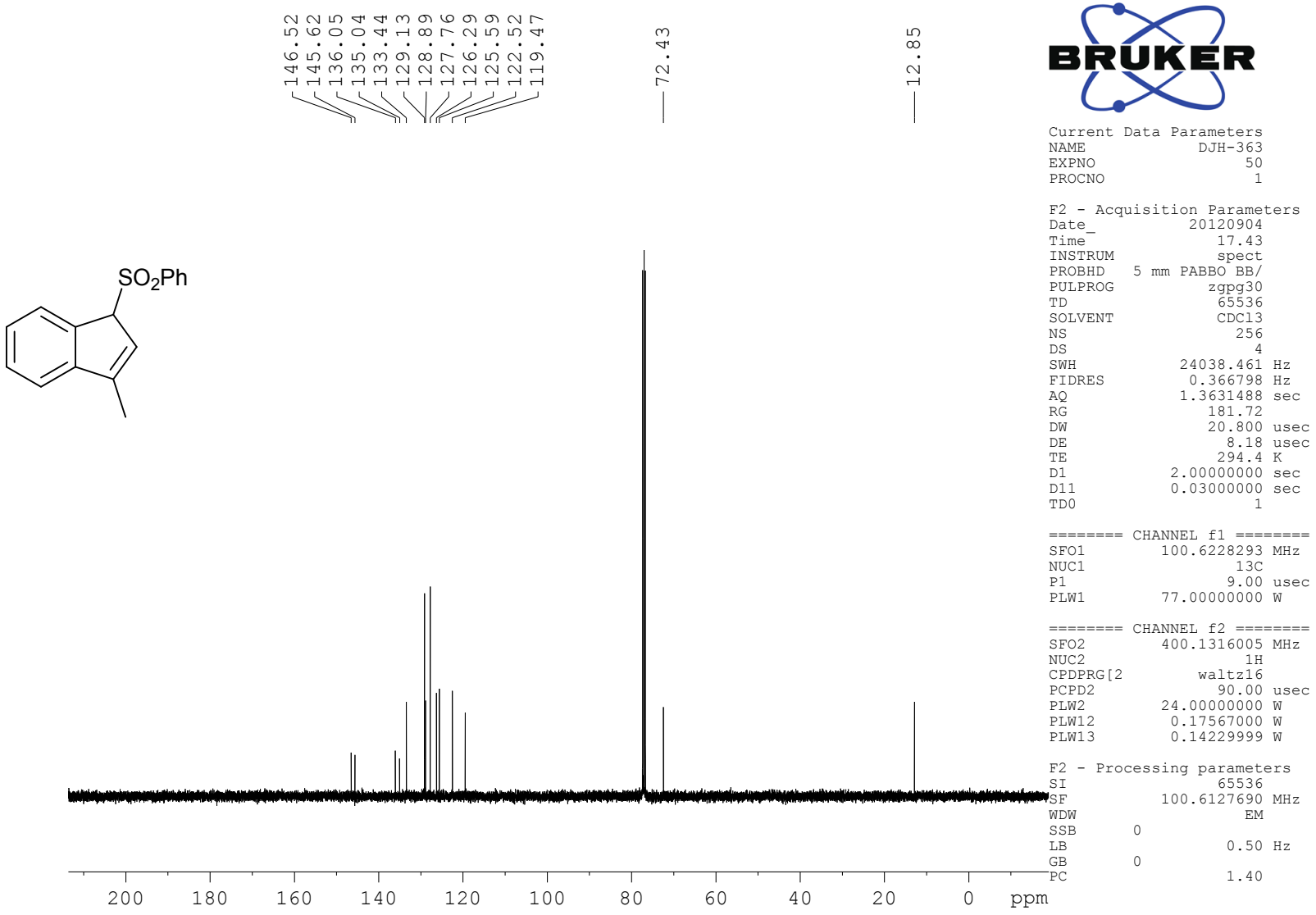
F2 - Processing parameters
SI                       32768
SF           125.7577976 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```



^{1}H NMR spectra (400 MHz, $CDCl_3$) of 1-benzenesulfonyl-3-methyl-indene (**4**)



^{13}C NMR spectra (100 MHz, $CDCl_3$) of 1-benzenesulfonyl-3-methyl-indene (4)




```
Current Data Parameters
NAME                  DJH-363
EXPNO                      50
PROCNO                      1

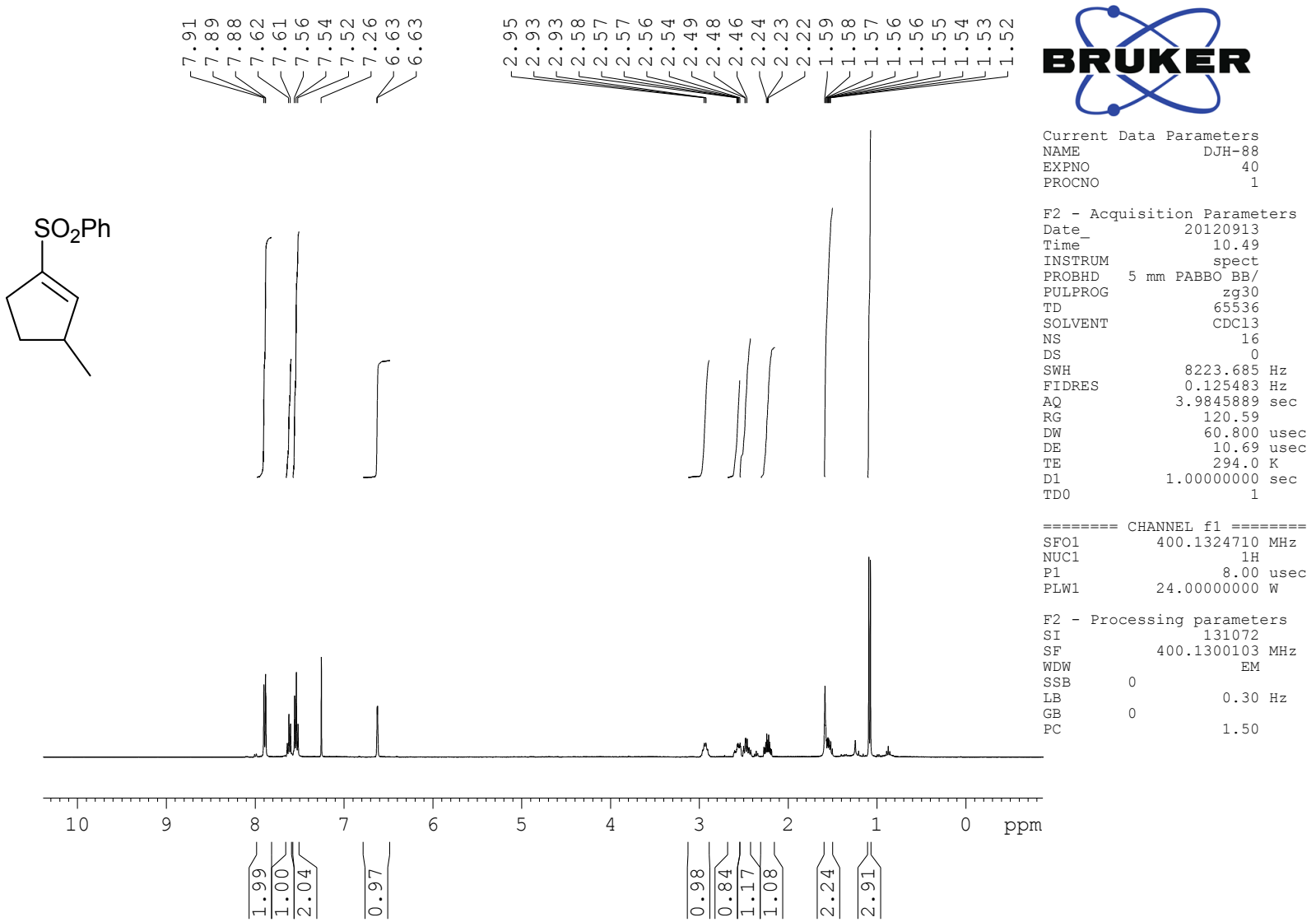
F2 - Acquisition Parameters
Date_                20120904
Time                    17.43
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       181.72
DW                       20.800 usec
DE                        8.18 usec
TE                       294.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               100.6228293 MHz
NUC1                       13C
P1                        9.00 usec
PLW1               77.00000000 W

======== CHANNEL f2 ========
SFO2               400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               24.00000000 W
PLW12               0.17567000 W
PLW13               0.14229999 W

F2 - Processing parameters
SI                       65536
SF                 100.6127690 MHz
WDW                        EM
SSB          0
LB                        0.50 Hz
GB           0
PC                        1.40
```

^{1}H NMR spectra (400 MHz, $CDCl_3$) of (3-methylcyclopenten-1-yl)sulfonylbenzene (**10**)



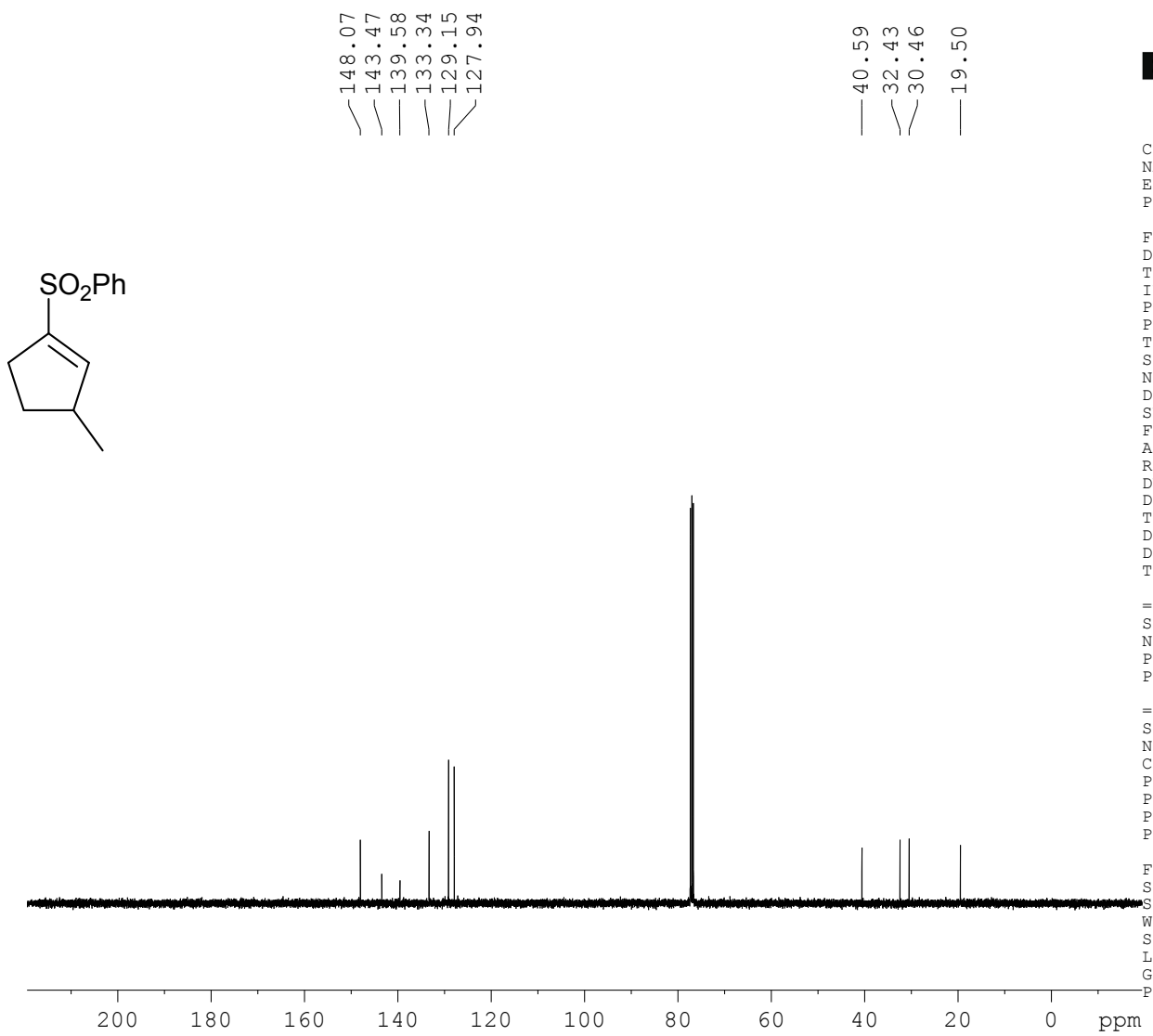
Current Data Parameters
NAME DJH-88
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120913
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 120.59
DW 60.800 usec
DE 10.69 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 8.00 usec
PLW1 24.00000000 W

F2 - Processing parameters
SI 131072
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.50

^{13}C NMR spectra (100 MHz, $CDCl_3$) of (3-methylcyclopenten-1-yl)sulfonylbenzene (**10**)




```
Current Data Parameters
NAME                  DJH-88
EXPNO                     41
PROCNO                     1

F2 - Acquisition Parameters
Date_               20120913
Time                   11.05
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        256
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     181.72
DW                     20.800 usec
DE                      8.18 usec
TE                     294.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             100.6228293 MHz
NUC1                     13C
P1                      9.00 usec
PLW1              77.00000000 W

======== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2              24.00000000 W
PLW12              0.17567000 W
PLW13              0.14229999 W

F2 - Processing parameters
SI                      65536
SF               100.6127690 MHz
WDW                        EM
SSB       0
LB                      0.50 Hz
GB        0
PC                      1.40
```