Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

A general and direct synthesis of imidazolium ionic liquids using orthoester

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1. General remark

1. 1 Chemical sources and general procedure

Commercially available reagents were used without further purification. Chemical sources of chemicals are shown below

Chemicals from C-TRI company (http://www.c-tri.co.kr/ctri_eng).
[BMIM][BF_4], Batch. No. ILI04C-131113
Halide content: 10 ppm, water content: 31.9 ppm according to the certificate of analysis

Chemicals from Aldrich
1-Butylimidazole, 98%, Cat. No. 348414
Trimethyl orthoformate, 99%, Cat. No. 108456
Ammonium tetrafluoroborate, 97+%, Cat. No. 223727
Tetrafluoroboric acid solution 48 wt. % in H_2O, Cat. No. 207934
Imidazole ACS reagent, ≥99%, Cat. No. 436151
Ammonium hexafluorophosphate, >= 98.0 % , Cat. No. 0-9820
Ammonium iodide, >99%, Cat. No. 0-9874
Ammonium nitrate, 98+ %, Cat. No. 221244
Bis(trifluoromethane)sulfonimide ≥95.0%, Cat. No. 15220
Nitric acid ACS reagent, 70%, Cat. No. 438073
Hexafluorophosphoric acid ~55 wt. % in H_2O, Cat. No. 200956

Chemicals from TCI
1-Phenylimidazole >98.0%(GC), Cat. No. P2030
Trifluoromethanesulfonic acid >98.0%(T), Cat. No. T0751
Isoquinoline >95.0%(GC), Cat. No. I0182
1-Vinylimidazole >98.0%(GC)(T), Cat. No. V0045
Triethyl orthoformate >98%(GC), Cat. No. O0066
Triisopropyl orthoformate >97.0%(GC), Cat. No. O0215
Tributyl orthoformate >95.0%(GC), Cat. No. O0269

Chemical from Wako
Ammonium bromide >98%, Cat. No. 1294

Chemicals from Junsei
Ammonium chloride >98%, Cat. No. 9D1547
p-Toluenesulfonic acid (chemical pure), Cat. No. 811572

Chemicals from Daesung
Ethyl acetate 99%,
Dichloromethane 99.5%
Acetone 99.5%
Acetonitrile 99.5%
Methyl alcohol 99.5%

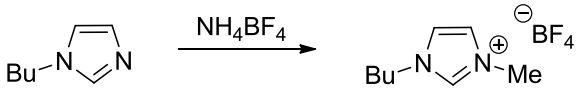
1-alkyl imidazole (1 eq) and ammonium salt (1.2 eq) were mixed with trialkyl orthoformate (5 eq) under N_2 atmosphere. In some reactions, protic acids (HX, 1 eq) were used to protonate imidazole. All reactions were monitored by ^{1}H NMR using DMSO-d6. After the reaction, solvent was removed under reduced pressure. Reaction mixture was dissolved in suitable solvent. The mixture was filtered through basic alumina. And then solvent was removed under reduced pressure to have the desired product.

1.2 Instrumentation

^{1}H NMR and ^{13}C NMR spectra were recorded in DMSO-d6 and $CDCl_3$ (Cambridge isotope) at a Varian Mercury Plus 300MHz spectrometers. ^{19}F NMR spectra were recorded in DMSO-d6 at Unity-Inova 500 MHz spectrometers. TG analysis were performed on DSC Q200 **(**TA Instruments Korea) and STA6000/8000 (Perkin Elmer). Mass spectra (FAB) were obtained using a Jeol JMS700 high-resolution mass spectrometer at the Korea Basic Science Center, Daegu, Korea. Mass spectra (ESI) were obtained using Agilent, Q-TOF 6530 at PNU Center for Research Facilities, Pusan, Korea. Ion chromatographic analysis was performed using Dionex (ICS-5000), equipped with an Dionex IonPacTM As15 column (4 x 250mm). Karl-Fisher test was performed using 831 KFC coulometer.

2. Experimental section

2.1 Screen of other alkylating agents



Entry	Alkylating agent	Temp. (^{o}C)	Time (h)	Yield[a] (%)
1	$CH_3(OCH_3)_3$	110	22	97
2	$(CH_3)_2(OCH_3)_2$	80	22	NR
3	$(CH_3)_2N(OCH_3)_3$	100	22	NR

[a] isolated yield

2.2 Synthesis of ionic liquids

1-butyl-3-methylimidazolium bromide [1]

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium bromide (2.18 mmol, 213 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.74 mmol, 383 mg) was collected in 96% yield.

^{1}H NMR δ0.83 (t, J=7.5 Hz, 3H) 1.19 (sextet, J=7.5 Hz, 2H) 1.74 (quintet, J=7.5 Hz. 2H) 3.88 (s, 3H) 4.21 (t, J=7.5 Hz, 2H) 7.82 (s, 1H) 7.91 (s, 1H) 9.45 (s, 1H) ^{13}C NMR δ13.70 19.17 31.85 36.25 48.83 122.67 123.94 137.98

1-butyl-3-methylimidazolium iodide [1]

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium iodide (2.18 mmol, 316 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.77 mmol, 472 mg) was collected in 97% yield.

^{1}H NMR δ0.89 (t, J=7.5 Hz, 3H) 1.24 (sextet, J=7.5 Hz, 2H) 1.76 (quintet, J=7.5 Hz. 2H) 3.85 (s, 3H) 4.17 (t, J=7.5 Hz, 2H) 7.72 (s, 1H) 7.80 (s, 1H) 9.16 (s, 1H) ^{13}C NMR δ13.99 19.45 32.03 36.56 49.18 122.95 124.27 137.16

1-butyl-3-methylimidazolium nitrate [1]

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium nitrate (2.18 mmol, 174 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 48 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.75 mmol, 353 mg) was collected in 96% yield.

^{1}H NMR δ0.86 (t, J=7.5 Hz, 3H) 1.21 (sextet, J=7.5 Hz, 2H) 1.74 (quintet, J=7.5Hz. 2H) 3.85 (s, 3H) 4.17 (t, J=7.5Hz, 2H) 7.72 (s, 1H) 7.80 (s, 1H) 9.24 (s, 1H) ^{13}C NMR δ13.85 19.43 32.05 36.27 49.15 122.95 124.25 137.37

1-butyl-3-methylimidazolium tetrafluoroborate [2]

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 17 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.76 mmol, 400 mg) was collected in 97% yield.

^{1}H NMR δ0.89 (t, J=7.5 Hz, 3H) 1.24 (sextet, J=7.5 Hz, 2H) 1.76 (quintet, J=7.5 Hz, 2H) 3.85 (s, 3H) 4.16 (d, J=7.5 Hz, 2H) 7.65 (s, 1H) 7.72 (s, 1H) 9.02 (s, 1H) ^{13}C NMR δ13.83 19.40 31.99 36.29 49.18 122.86 124.19 137.10

1-butyl-3-methylimidazolium hexafluorophosphate [2]

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 17 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.61 mmol, 460 mg) was collected in 88% yield.

^{1}H NMR δ0.90 (t, J=7.5 Hz, 3H) 1.28 (sextet, J=7.5 Hz, 2H) 1.77 (quintet, J=7.5 Hz, 2H) 3.84 (s, 3H) 4.15 (d, J=7.5 Hz, 2H) 7.65 (s, 1H) 7.71 (s, 1H) 9.05 (s, 1H) ^{13}C NMR δ13.80 19.40 31.97 36.28 49.20 122.83 124.19 137.12

1-butyl-3-methylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole (1.82 mmol, 0.24 mL) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then trimethyl orthoformate (9.1 mmol, 1 mL) was added to the residue. The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent removed under vacuum and then the resulting product (1.74 mmol, 394 mg) was collected in 96% yield.

^{1}H NMR δ0.89 (t, J=7.5 Hz, 3H) 1.24 (sextet, J=7.5 Hz, 2H) 1.76 (quintet, J=7.5 Hz, 2H) 3.85 (s, 3H) 4.16 (d, J=7.5 Hz, 2H) 7.65 (s, 1H) 7.72 (s, 1H) 9.02 (s, 1H) ^{13}C NMR δ13.83 19.40 31.99 36.29 49.18 122.86 124.19 137.10

1-butyl-3-methylimidazolium hexafluorophosphate

Aqueous hexafluorophosphoric acid (1.82 mmol, 0.247 mL) was added to 1-butylimidazole(1.82 mmol, 0.24 mL) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then trimethyl orthoformate (9.1 mmol, 1 mL) was added to the residue. The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent removed under reduced pressure and then the resulting product (1.73 mmol, 494 mg) was collected in 95% yield.

^{1}H NMR δ0.90 (t, J=7.5 Hz, 3H) 1.28 (sextet, J=7.5 Hz, 2H) 1.77 (quintet, J=7.5 Hz, 2H) 3.84 (s, 3H) 4.15 (d, J=7.5 Hz, 2H) 7.65 (s, 1H) 7.71 (s, 1H) 9.05 (s, 1H) ^{13}C NMR δ13.80 19.40 31.97 36.28 49.20 122.83 124.19 137.12

1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)amide [1]

1-butylimidazole (1.69 mml, 0.22 mL) and bis(trifluoromethane)sulfonimide (1.69 mmol, 475 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.66 mmol, 645 mg) was collected in 99% yield.

^{1}H NMR δ0.90 (t, J=7.5 Hz, 3H) 1.26 (sextet, J=7.5 Hz, 2H) 1.78 (quintet, J=7.5 Hz. 2H) 3.85 (s, 3H) 4.16 (t, J=7.5 Hz, 2H) 7.66 (s, 1H) 7.72 (s, 1H) 9.09 (s, 1H) ^{13}C NMR δ13.37 19.12 31.75 35.98 48.96 113.51 117.77 122.03 122.58 123.92 126.28 136.90

1-butyl-3-methylimidazolium trifluoromethanesulfonate [3]

1-butylimidazole (1.82 mml, 0.24 mL) and trifluoromethanesulfonic acid (1.82 mmol, 0.16 mL) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.72 mmol, 496 mg) was collected in 95% yield.

^{1}H NMR δ0.89 (t, J=7.5 Hz, 3H) 1.27 (sextet, J=7.5 Hz, 2H) 1.76 (quintet, J=7.5 Hz. 2H) 3.85 (s, 3H) 4.16 (t, J=7.5 Hz, 2H) 7.68 (s, 1H) 7.75 (s, 1H) 9.08 (s, 1H) ^{13}C NMR δ13.56 19.15 31.75 36.07 48.93 114.66 118.92 122.62 123.19 123.97 127.45 136.90

1-butyl-3-methylimidazolium 4-methylbezenensulfonate [4]

1-butylimidazole (1.82 mmol, 0.24 mL) and *p*-toluenesulfonic acid (1.82 mmol, 346 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in dichloromethane. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.71 mmol, 532 mg) was collected in 94% yield.

^{1}H NMR δ0.8 (t, J=7.5 Hz, 3H) 1.22 (sextet, J=7.5 Hz, 2H) 1.74 (quintet, J=7.5 Hz. 2H) 3.84 (s, 3H) 4.15 (t, J=7.5 Hz, 2H) 7.11 (d, J=7.8 Hz, 2H) 7.48 (d, J=7.8 Hz, 2H) 7.71 (s, 1H) 7.77 (s, 1H) 9.15 (s, 1H) ^{13}C NMR δ 13.73 19.21 21.23 31.82 36.15 48.90 122.74 124.05 125.92 128.52 137.01 138.09 146.18

1,3-dimethylimidazolium hexafluorophosphate [5]

1-methylimidazole (1.82 mmol, 0.145 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.79 mmol, 433 mg) was collected in 98% yield.

^{1}H NMR δ3.84 (s, 6H) 7.66 (s, 2H) 9.00 (s, 1H) ^{13}C NMR δ36.28 124.08 137.6

1,3-dimethylimidazolium tetrafluoroborate [6]

1-methylimidazole (1.82 mmol, 0.145 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.71 mmol, 314 mg) was collected in 94% yield.

^{1}H NMR δ3.84 (s, 6H) 7.66 (s, 2H) 9.00 (s, 1H) ^{13}C NMR δ36.09 123.89 137.46

1-allyl-3-methylimidazolium hexafluorophosphate

1-allylimidazole (1.82 mml, 0.195 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.81 mmol, 487 mg) was collected in 97% yield. HRMS (FAB+) calcd for $C_7H_{11}N_2$:123.0922; found, 123.0921

^{1}H NMR δ3.86 (s, 3H) 4.83 (d, J=6 Hz, 2H) 5.28 (d, J=17 Hz, 1H) 5.39 (d, J=11.5 Hz, 1H) 6.01 (m ,1H) 7.66 (s, 2H) 9.06 (s, 1H) ^{13}C NMR δ36.14 51.25 12.68 122.70 124.16 131.96 137.04

1-allyl-3-methylimidazolium tetrafluoroborate [7]

1-allylimidazole (1.82 mmol, 0.195 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl

acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.79 mmol, 376 mg) was collected in 96% yield.

^{1}H NMR δ3.85 (s, 3H) 4.83 (d, J=6 Hz, 2H) 5.28 (d, J=17 Hz, 1H) 5.39 (d, J=11.5 Hz,1H) 6.02 (m, 1H) 7.70 (s, 2H) 9.08 (s, 1H) ^{13}C NMR δ36.16 51.20 120.63 122.73 124.17 132.11 136.99

1-benzyl-3-methylimidazolium hexafluorophosphate [8]

1-benzylimidazole (1.82 mmol, 288 mg) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 19 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.80 mmol, 576 mg) was collected in 99% yield.

^{1}H NMR δ3.86 (s, 3H) 5.42 (s, 2H) 7.42 (s, 5H) 7.70 (s, 1H) 7.78 (s, 1H) 9.20 (s, 1H) ^{13}C NMR δ36.30 52.36 122.80 124.45 128.70 129.19 129.44 135.25 137.13

1-benzyl-3-methylimidazolium tetrafluoroborate [8]

1-benzylimidazole (1.82 mml, 288 mg) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 19 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.77 mmol, 461 mg) was collected in 97% yield.

^{1}H NMR δ3.85 (s, 3H) 5.41 (s, 2H) 7.42 (s, 5H) 7.71(s, 1H) 7.78 (s, 1H) 9.19 (s,1H) ^{13}C NMR δ36.29 52.30 122.78 124.44 128.73 129.19 129 45 135.32 137.07

3-methyl-1-phenylimidazolium hexafluorophosphate

1-phenylimidazole (1.03 mml, 0.13 mL) and ammonium hexafluorophosphoate (1.24 mmol, 201 mg) was mixed with trimethyl orthoformate (5.15 mmol, 0.56 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and acetone. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.00 mmol, 318 mg) was collected in 97% yield. HRMS (FAB+) calcd for $C_{10}H_{11}N_2$: 159.0922; found, 159.0923

^{1}H NMR δ3.94 (s, 1H) 7.59 (d, J=7.2 Hz, 1H) 7.67 (t, J=7.2 Hz, 2H) 7.77 (d, J=7.2 Hz, 2H) 7.93 (s, 1H) 8.28 (s, 1H) 9.73 (s, 1H) ^{13}C NMR δ36.56 121.47 122.30 124.88 130.23 130.67 135.21 136.41

3-methyl-1-phenylimidazolium tetrafluoroborate

1-phenylimidazole (1.03 mmol, 0.13 mL) and ammonium tetrafluoroborate (1.24 mmol, 130 mg) was mixed with trimethyl orthoformate (5.15 mmol, 0.56 mL). The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.00 mmol, 244 mg) was collected in 96% yield. HRMS (FAB+) calcd for $C_{10}H_{11}N_2$: 159.0922; found, 159.0925

^{1}H NMR δ3.94 (s, 1H) 7.59 (d, J=7.2 Hz, 1H) 7.69 (t , J=7.2 Hz, 2H) 7.76 (d, J=7.2 Hz, 2H) 7.94 (s, 1H) 8.29 (s, 1H) 9.73(s, 1H) ^{13}C NMR δ36.56 121.41 122.25 124.87 130.20 130.68 135.20 136.38

3-methyl-1-vinylimidazolium hexafluorophosphate

1-vinylimidazole (1.82 mmol, 0.165 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in acetonitrile. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.76 mmol, 452 mg) was collected in 97% yield. HRMS (FAB+) calcd for $C_6H_9N_2$: 109.0766; found, 109.0765

^{1}H NMR δ3.88 (s, 3H) 5.39 (d, J=8.8 Hz, 1H) 5.90 (d, J=15.7 Hz, 1H) 7.26 (dd, J_1=15.7 Hz, J_2=8.8 Hz) 7.82 (s, 1H) 8.15 (s, 1H) 9.38 (s, 1H) ^{13}C NMR δ36.42 108.86 119.27 124.83 129.28 136.50

3-methyl-1-vinylimidazolium tetrafluoroborate

1-vinylimidazole (1.82 mmol, 0.165 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.76 mmol, 346 mg) was collected in 96% yield. HRMS (FAB+) calcd for $C_6H_9N_2$:109.0766; found, 109.0765

^{1}H NMR δ3.85 (s, 3H) 5.39 (d, J=8.8 Hz,1H) 5.90 (d, J=15.7 Hz, 1H) 7.26 (dd, J_1=15.7 Hz, J_2=8.8 Hz) 7.78 (s, 1H) 8.12 (s, 1H) 9.35 (s, 1H) ^{13}C NMR δ36.42 108.86 119.27 124.83 129.28 136.50

1-butyl-3-ethyllimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole(1.82 mmol, 0.24 mL) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then triethyl orthoformate (9.1 mmol, 1.5 mL) was added to residue. The reaction mixture was heated to 120 ^{o}C for 25 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.66 mmol, 399 mg) was collected in 91% yield. HRMS (FAB+) calcd for $C_9H_{17}N_2$: 153.1392; found, 153.1391

^{1}H NMR δ0.89 (t, J=7.5 Hz, 3H) 1.25 (sextet, H=7.5 Hz, 2H) 1.41 (t, J=7.5 Hz, 3H) 1.78 (quintet, J=7.5 Hz, 2H) 4.17 (m, 4H) 7.77 (s, 2H) 9,12 (s, 1H) ^{13}C NMR δ13.62 15.34 19.22 31.72 44.66 49.02 122.51 122.79 136.02

1-butyl-3-propylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole(1.82 mmol, 0.24 mL) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then tripropyl orthoformate (9.1 mmol, 1.9 mL) was added to residue. The reaction mixture was heated to 130 ^{o}C for 28 h. After the reaction, tripropyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under vacuum and then the resulting product (1.68 mmol, 429 mg) was collected in 93% yield. HRMS (FAB+) calcd for $C_{10}H_{19}N_2$: 167.1548; found, 167.1546

^{1}H NMR δ0.87 (m, 6H) 1.25 (sextet, J=7.5 Hz, 2H) 1.82 (m, 4H) 4.14 (m, 4H) 7.79 (s, 2H) 9.17 (s, 1H) ^{13}C NMR δ10.90 13.80 19.42 23.43 31.93 49.27 51.02 123.05 136.53

1-butyl-3-isopropylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole(1.82 mmol, 0.24 mL) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then triisopropyl orthoformate (9.1 mmol, 1.9 mL) was added to the residue. The reaction mixture was heated to 130 ^{o}C for 24 h. After the reaction, triisopropyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.44 mmol, 366 mg) was collected in 79% yield. HRMS (FAB+) calcd for $C_{10}H_{19}N_2$: 167.1548; found, 167.1551

^{1}H NMR δ0.90 (t, J=7.5Hz 3H) 1.26 (sextet, J=7.5 Hz, 2H) 1.48 (d, J=6.0 Hz, 6H) 1.80 (quintet, J=7.5 Hz, 2H) 4.16 (t, J=7.5 Hz, 2H) 4.62 (septet, J=6.0 Hz, 1H) 7.77 (s, 1H) 7.87 (s, 1H) 9.16 (s, 1H) ^{13}C NMR δ13.62 19.26 31.71 49.06 52.71 120.97 122.88 135.06

1,3-dibutylimidazolium tetrafluoroborate [9]

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole(1.82 mmol, 0.24 mL) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then tributyl orthoformate (9.1 mmol, 2.4 mL) was added to the residue. The reaction mixture was heated to 140 ^{o}C for 24 h. After the reaction, tributyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.70 mmol, 457 mg) was collected in 94% yield.

^{1}H NMR 0.90 (t, J=7.5 Hz, 6H) 1.24 (sextet, J=7.5 Hz, 4H) 1.77 (t, J=7.5 Hz, 4H) 4.16 (t, J=7.5 Hz, 4H) 7.79 (s, 2H) 9.18 (s, 1H) ^{13}C NMR δ13.62 19.19 31.71 49.02 122.85 136.30

***N*-methylisoquinolinium tetrafluoroborate**

Isoquinoline (1.82 ml, 0.21 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 ^{o}C for 24 h. After the reaction, solvent was removed under reduced pressure. Ethyl acetate was added to the residue for recrystallization. The resulting product (1.61 mmol, 372 mg) was obtained as orange solid in 89% yield. HRMS (FAB+) calcd for $C_{10}H_{10}N$: 144.0813; found, 144.0811

^{1}H NMR 4.47 (s, 3H) 8.06 (t, J=7.8 Hz, 1H) 8.24 (t, J=7.5 Hz, 1H) 8.33 (d, J=8.1 Hz, 1H) 8.46 (d, J=8.1 Hz, 1H) 8.54 (d, J=6.6 Hz, 1H), 8.69 (d, J=6.6 Hz, 1H) 9.97 (s, 1H) ^{13}C NMR δ48.33 125.84 127.48 127.66 130.59 131.60 136.30 137.05 137.14 151.13

1,3-dimethylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to imidazole (1.82 mmol, 124 mg) in at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then trimethyl orthoformate (9.1 mmol, 1 mL) was added to the residue. The reaction mixture was heated to 110 ^{o}C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.54 mmol, 284 mg) was collected in 84% yield.

^{1}H NMR δ3.84 (s, 6H) 7.64 (s, 2H) 8.99 (s, 1H) ^{13}C NMR δ36.28 124.08 137.64

1,3-diethylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to imidazole (1.82 mmol, 124 mg) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then triethyl orthoformate (9.1 mmol, 1.5 mL) was added to the residue. The reaction mixture was heated to 130 ^{o}C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.54 mmol, 284 mg) was collected in 68% yield. HRMS (FAB+) calcd for $C_7H_{13}N_2$: 125.1079; found, 125.1079

^{1}H NMR 1.42 (t, J= 7.5 Hz, 6H), 4.17 (q, J=7.5 Hz, 4H) 7.80 (s, 2H) 9.16 (s, 1H) ^{13}C NMR δ15.45 44.62 122.54 135.80

1,3-diisopropylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to imidazole (1.82 mmol, 124 mg) at 0 ^{o}C and water was removed under reduced pressure using phosphorus pentoxide. And then triisopropyl orthoformate (9.1 mmol, 1.5 mL) was added to the residue. The reaction mixture was heated to 130 ^{o}C for 48 h. After the reaction, triisopropyl orthoformate was removed under reduced pressure. Recrystallization was performed in the presence of ethyl acetate and the resulting product (0.57 mmol, 137 mg) was collected in 31% yield. HRMS (FAB+) calcd for $C_9H_{17}N_2$: 153.1392; found, 153.1393

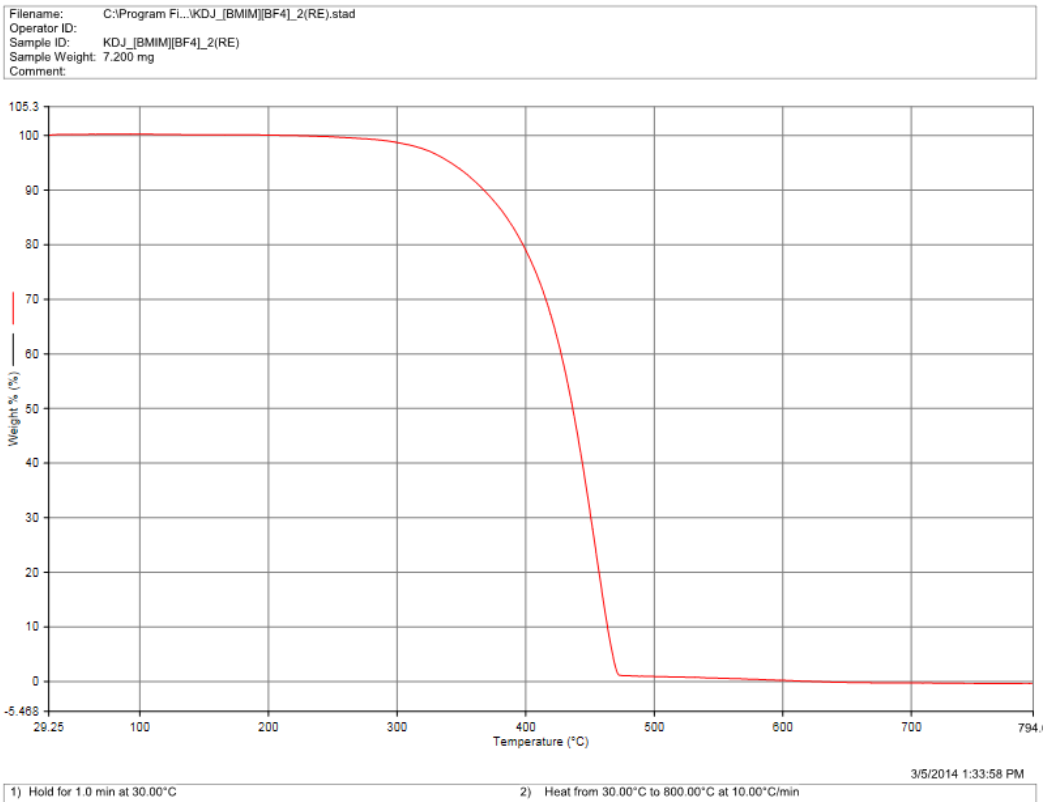
^{1}H NMR 1.47 (d, J= 6.9 Hz, 12H), 4.60 (septet, J=6.9 Hz, 2H) 7.91 (s, 2H) 9.23 (s, 1H) ^{13}C NMR δ22.73 52.69 121.08 133.94

1,3-diethylimidazolium 4-methylbenzenesulfonate

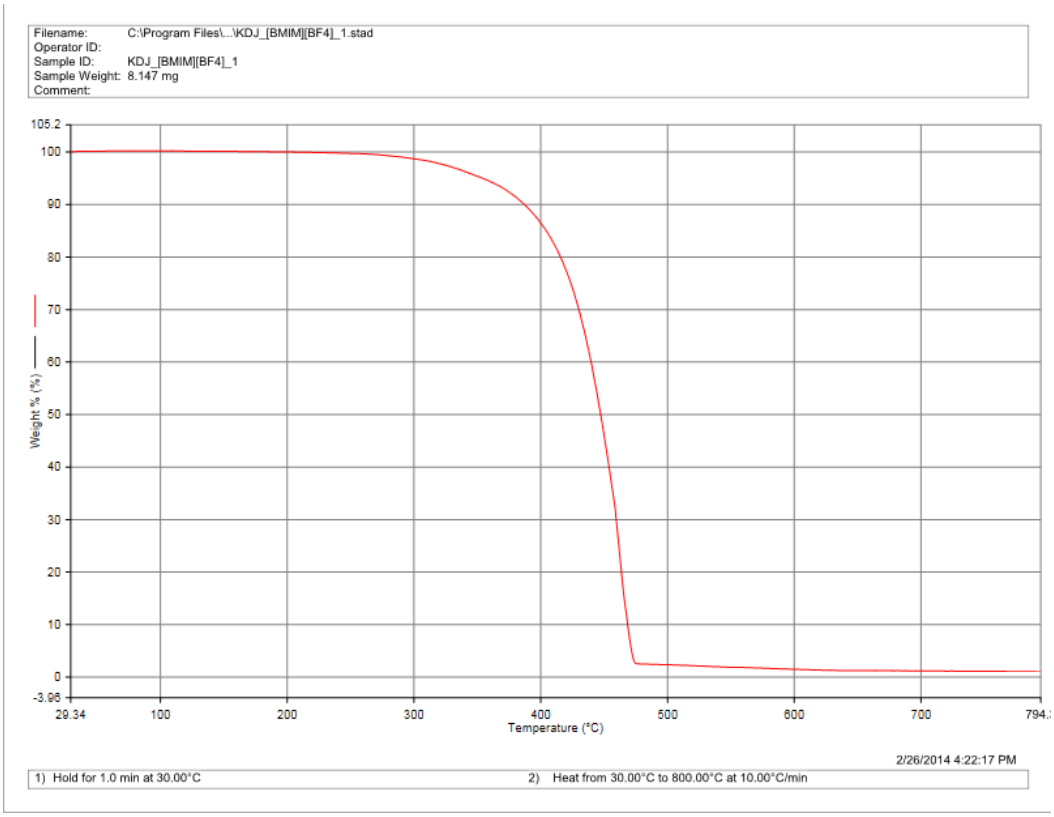
1-butylimidazole (1.82 mml, 124 mg) and *p*-toluenesulfonic acid (1.82 mmol, 346 mg) was mixed with triethyl orthoformate (9.1 mmol, 1.5 mL). The reaction mixture was heated to 130 ^{o}C for 24 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in dichloromethane and the mixture was filtered through basic alumina. Solvent removed under reduced pressure and then the resulting product (1.61 mmol, 479 mg) was collected in 89% yield. HRMS (FAB+) calcd for $C_7H_{13}N_2$: 125.1079; found, 125.1076

^{1}H NMR 1.38 (t, J= 7.5 Hz, 6H), 2.28 (s, 3H) 4.16 (q, J=7.5 Hz, 4H) 7.13 (d, J=6.9 Hz, 2H) 7.53 (d, J=6.9 Hz, 2H) 7.82 (s, 2H) 9.29 (s, 1H) ^{13}C NMR δ15.50 21.20 44.55 122.53 125.89 128.61 135.98 138.29 145.98

2.3 TGA data for [BMIM][BF_4]



Synthesized [BMIM][BF_4] from HBF_4



Commercial [BMIM][BF_4] (c-tri)

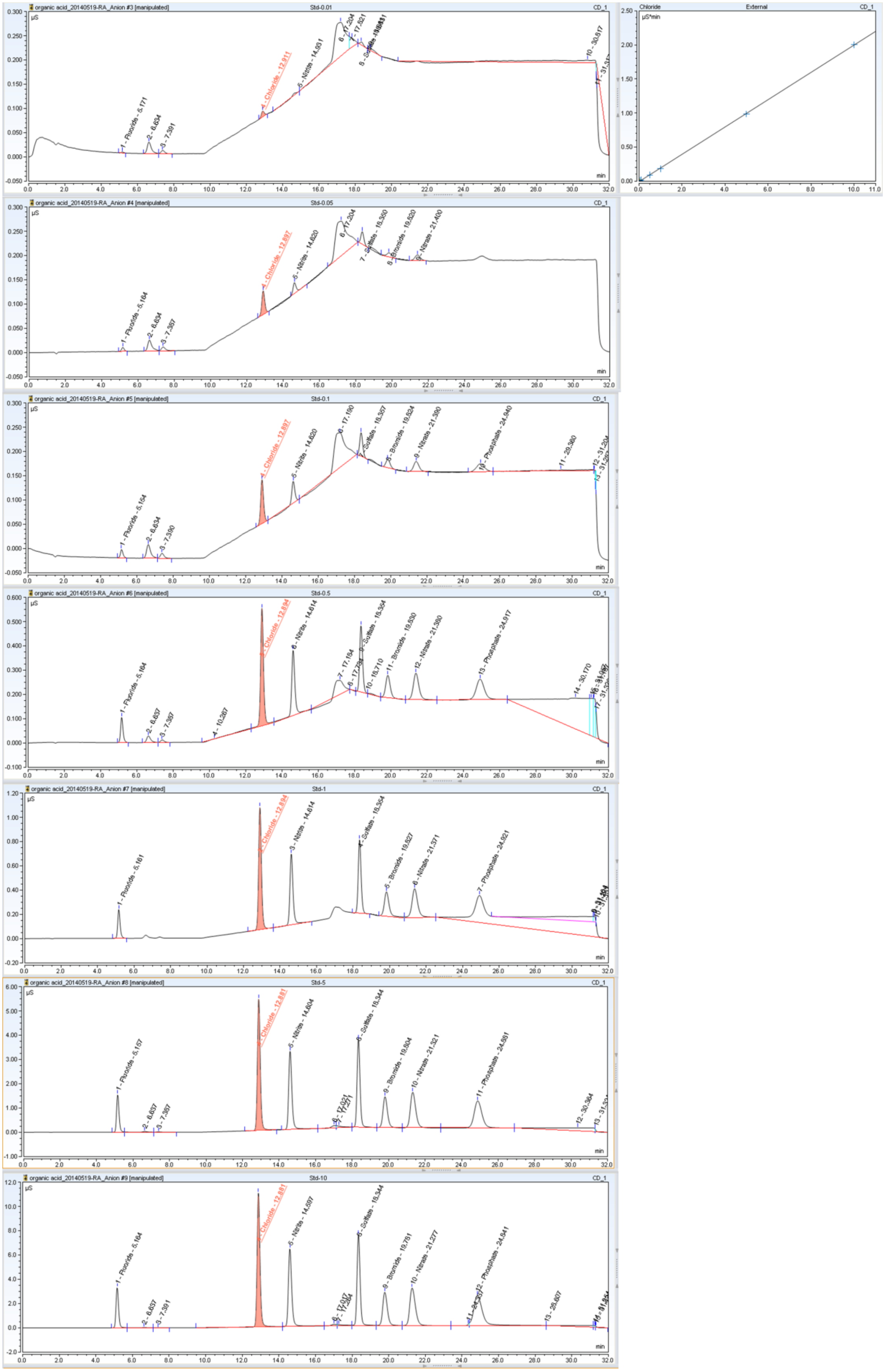
2.4 Ion chromatography

* Reagents and Standards: Deionized water, 18 MΩ-cm, Chloride Standard, 100 mg/L (seven anion standard II, Dionex)
* Instrument: Dionex ICS-5000 system
* Column: Analytical column AS15(Dionex, USA) (4x250mm), Guard column AS15 (Dionexm, USA) (4x50mm)
* Flow : 1.5mL/min
* Eluent 32mM KOH
* Temperature: 35 ^{o}C
* Sampling: [BMIM]Br (from NH_4Br): 0.1408 g
  [BMIM]I (from NH_4I): 0.1375 g
  [BMIM]BF_4 (from HBF_4): 0.9285 g
  [BMIM]BF_4 (from NH_4BF_4): 0.9312 g
  [BMIM]BF_4 (C-TRI): 0.9905 g
  [BMIM]NO_3 (from HNO_3): 0.7103 g
  [BMIM]NO_3 (from NH_4NO_3): 0.7092 g
  [BMIM]$N(Tf)_2$ (from $HN(Tf)_2$): 0.2871 g
  [BMIM]OTs (from TsOH): 0.1204 g
  [BMIM]OTf (from TfOH): 0.4962 g

(1) Calibration results for chloride and chromatogram of standard solution

***Cl calibration : 0.01, 0.05, 0.1, 0.5, 1, 5, 10 ppm**

	Calibration Batch Report						
Sequence:	organic acid_20140519-RA_Anion				Injection V	5,000.00	
Instrument Met	organic acid_20130613				Operator:	user	
Inj. Date / Time	19-5-2012	/ 22:14			Run Time:	32.00067	
Calibration Summary							
Peak Name	Eval.Type	Cal.Type	Points	Offset	Slope	Curve	Coeff.Det.
				(C0)	(C1)	(C2)	%
Fluoride	Area	Lin, WithOffset	7	-0.008	0.27	0	99.7963
***Chloride**	**Area**	**Lin, WithOffset**	**7**	**-0.007**	**0.201**	**0**	**99.9895**
Nitrite	Area	Lin, WithOffset	7	-0.009	0.136	0	99.9779
Sulfate	Area	Lin, WithOffset	7	-0.018	0.149	0	99.8193
Bromide	Area	Lin, WithOffset	6	-0.012	0.08	0	99.8572
Nitrate	Area	Lin, WithOffset	6	-0.018	0.111	0	99.4542
Phosphate	*Area*	*Lin, WithOffset*	*5*	*0.87*	*-0.051*	*0*	*55.1178*
		AVERAGE:		0.1136	0.1285	0	93.3577
Injection Name	Ret.Time	Area	Height	Amount			
	min	mS*min	mS				
Chloride	Chloride	Chloride	Chloride	Chloride			
	CD_1	CD_1	CD_1	CD_1			
Std-0.01	12.911	0.0023	0.012	0.047			
Std-0.05	12.897	0.0093	0.049	0.082			
Std-0.1	12.897	0.0167	0.091	0.119			
Std-0.5	12.894	0.0909	0.485	0.488			
Std-1	12.894	0.1812	1.011	0.939			
Std-5	12.881	0.9856	5.427	4.947			
Std-10	12.881	2.0041	11.061	10.023			
Average	12.893						
Rel. Std. Dev.	0.08%						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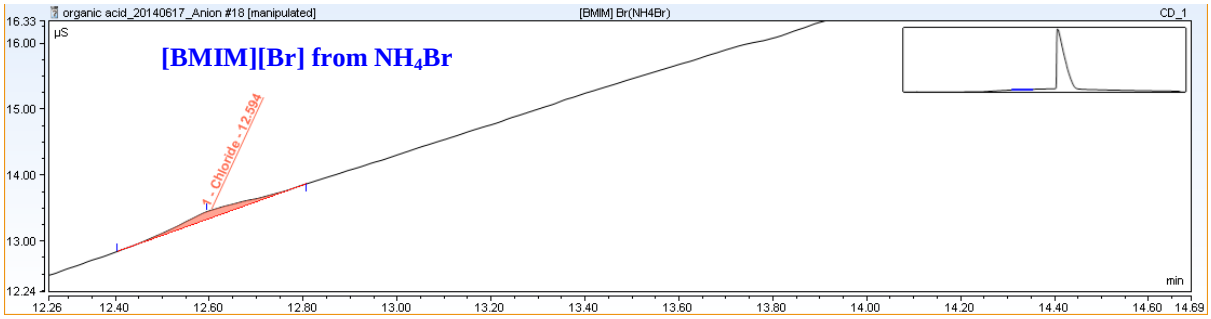
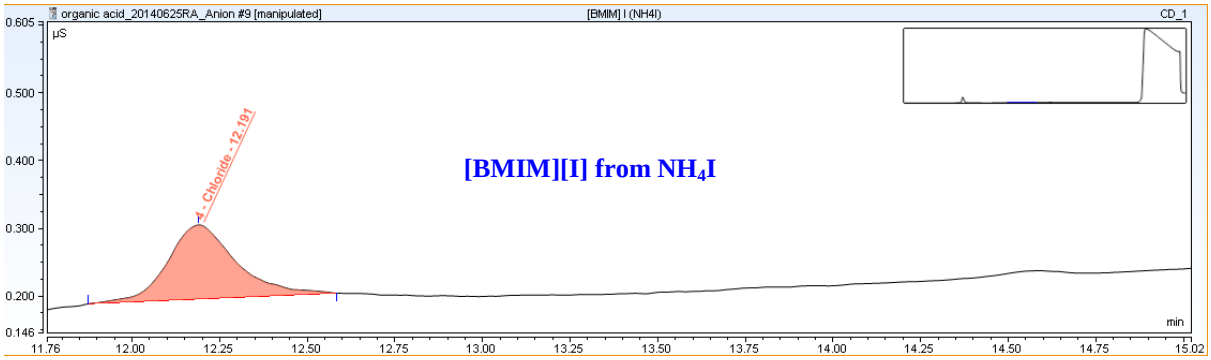
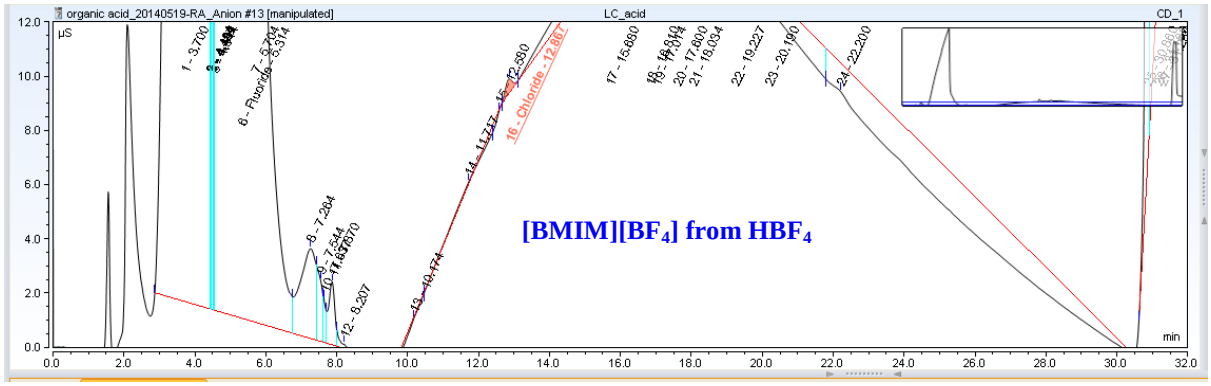
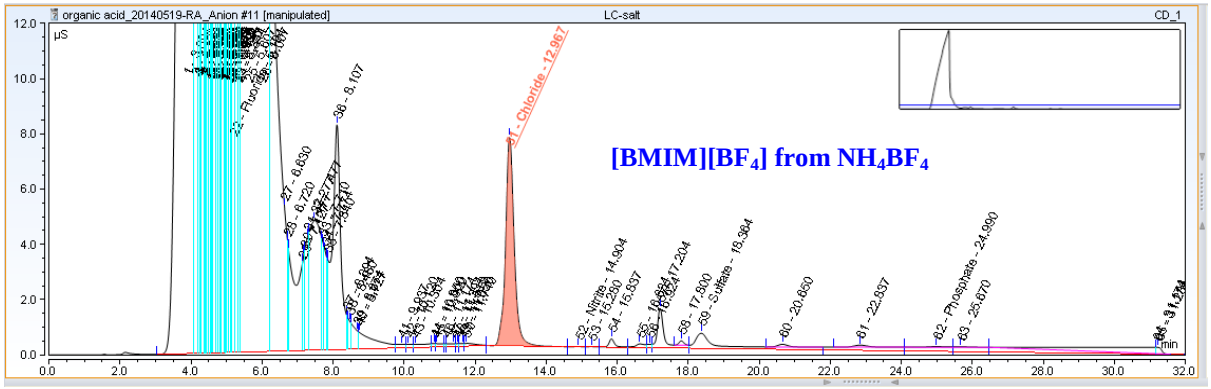
Std-0.01
CD_1
Chloride
External
organic acid_20140519-RA_Anion #4 [manipulated]
Std-0.05
organic acid_20140519-RA_Anion #5 [manipulated]
Std-0.1
organic acid_20140519-RA_Anion #6 [manipulated]
Std-0.5
organic acid_20140519-RA_Anion #7 [manipulated]
Std-1
organic acid_20140519-RA_Anion #8 [manipulated]
Std-5
organic acid_20140519-RA_Anion #9 [manipulated]
Std-10

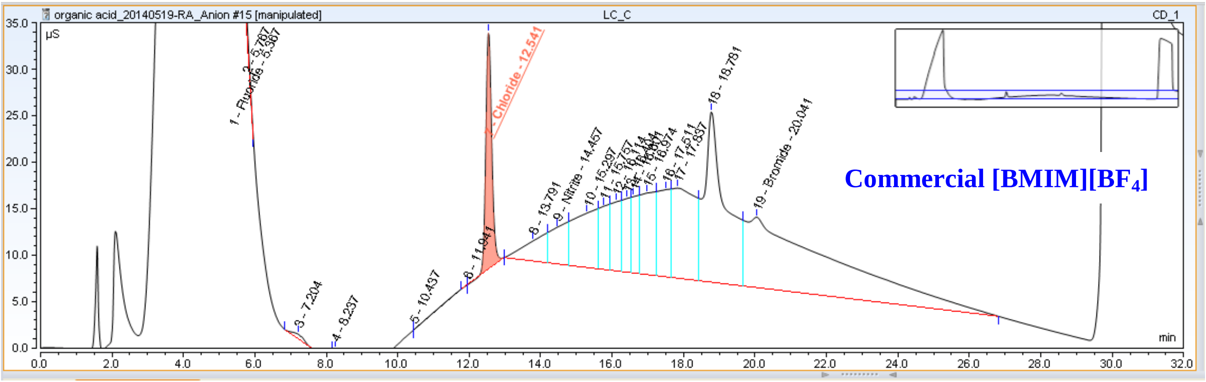
(2) Chromatogram







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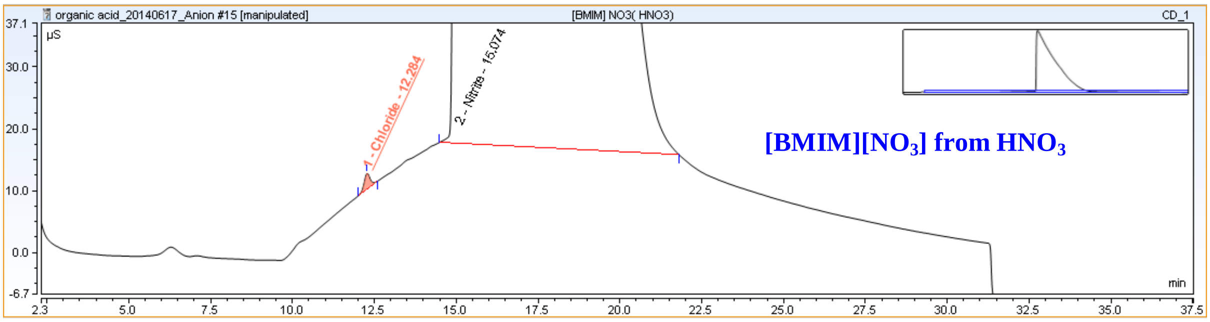
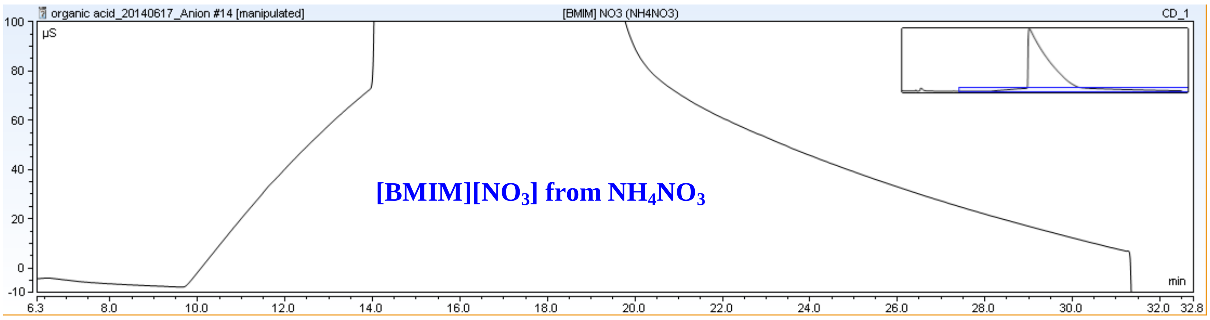
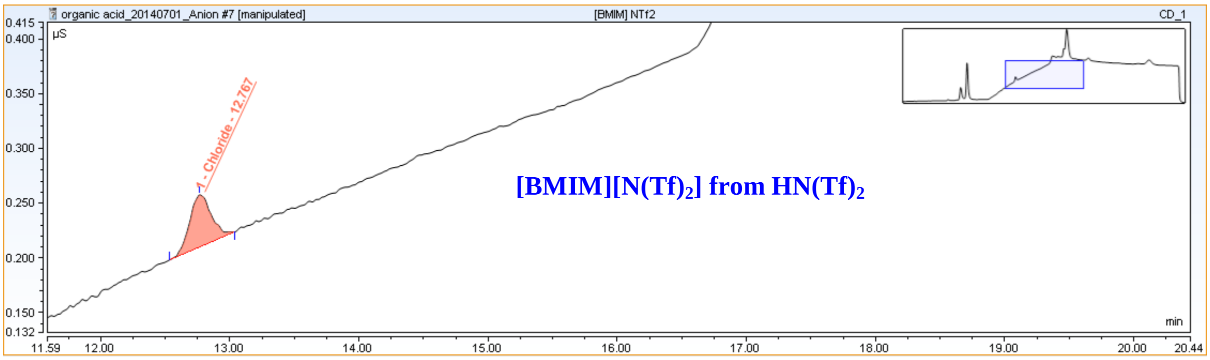
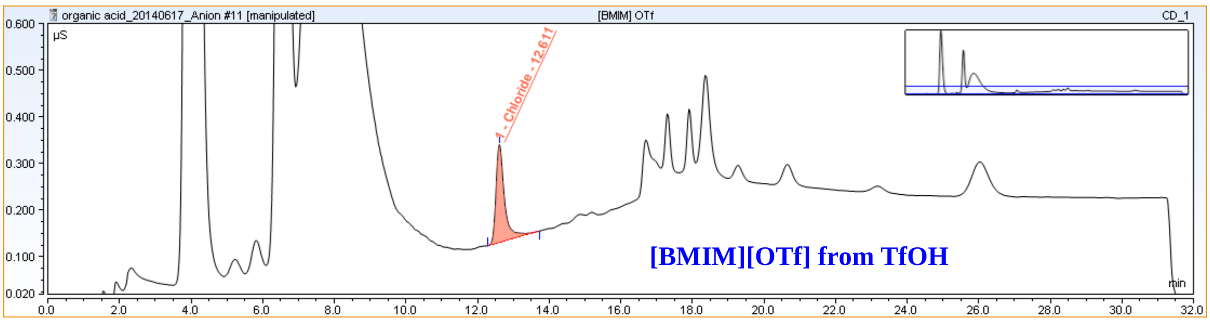
LC_C
CD_1
µS
min
1 - Fluoride - 5.787
2 - 5.787
3 - 7.204
4 - 8.257
5 - 10.437
6 - 11.941
7 - Chloride - 12.541
8 - 13.791
9 - Nitrite - 14.457
10 - 15.297
18 - 18.781
19 - Bromide - 20.041
Commercial [BMIM][BF4]


organic acid_20140617_Anion #15 [manipulated]
[BMIM] NO3(HNO3)
CD_1
µS
min
1 - Chloride - 12.284
2 - Nitrite - 15.074
[BMIM][NO3] from HNO3


organic acid_20140617_Anion #14 [manipulated]
[BMIM] NO3 (NH4NO3)
CD_1
µS
min
[BMIM][NO3] from NH4NO3


organic acid_20140701_Anion #7 [manipulated]
[BMIM] NTf2
CD_1
µS
min
1 - Chloride - 12.767
[BMIM][N(Tf)2] from HN(Tf)2


organic acid_20140617_Anion #11 [manipulated]
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CD_1
µS
min
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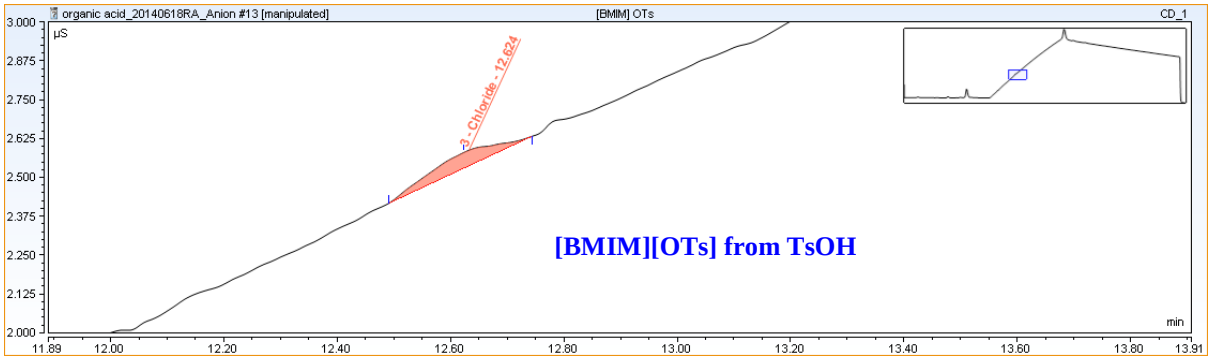
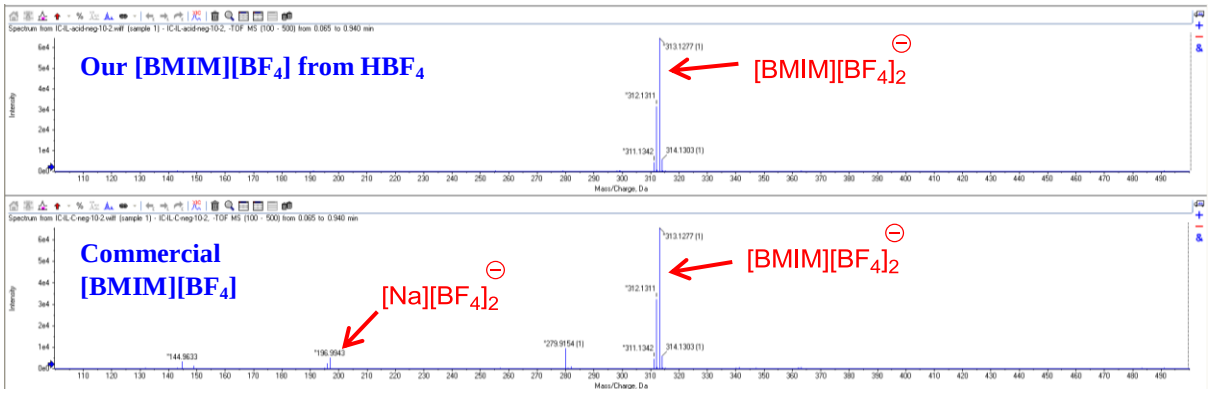


(3) Cl content table

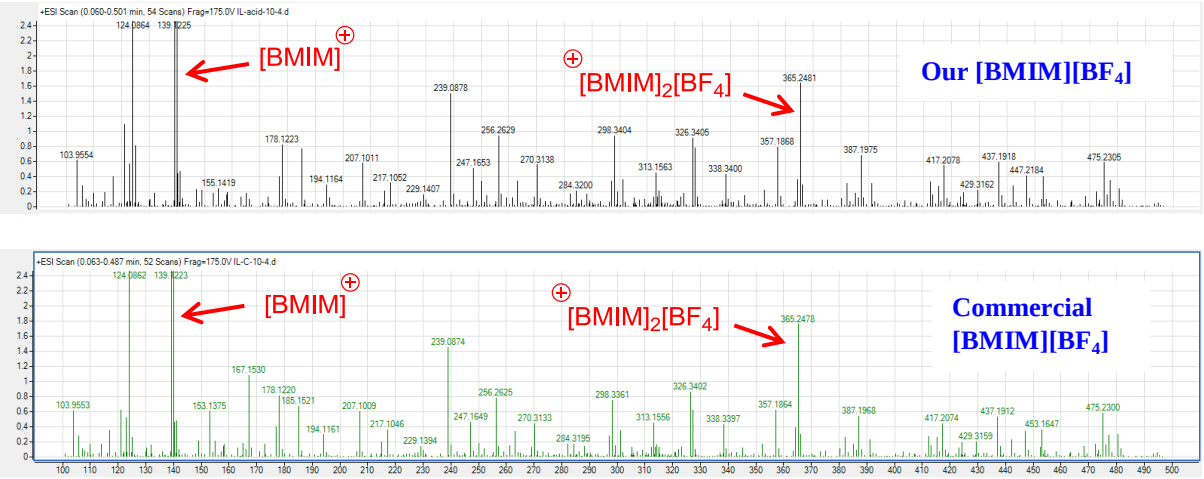
Name	Time	Area	Rel.Area	Height	Rel.Height	Amount	dilution factor	**ppm**
[BMIM]Br (NH4Br)	12.594	0.0194	100	0.12	100	0.1462	72.5447	**10.6082**
[BMIM]I (NH4I)	12.191	0.0247	0.96	0.11	0.87	0.1916	43.0938	**8.2568**
[BMIM]BF4 (HBF4)	12.867	0.1196	0.02	0.52	0.03	0.6314	10.6328	**6.7138**
[BMIM]BF4 (NH4BF4)	12.967	2.1158	0.57	7.57	0.17	10.5795	13.5888	**143.7632**
[BMIM]BF4 (C-TRI)	12.541	4.8312	3.67	25.45	8.45	24.1119	10.0617	**242.6070**
[BMIM]NO3 (HNO3)	12.284	0.5308	0.02	2.53	0.18	2.4603	10.6014	**26.0826**
[BMIM]NO3 (NH4NO3)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	**n.a.**
[BMIM]NTf2 (NTf2)	12.767	0.0092	8.74	0.05	12.74	0.0753	24.7739	**1.8664**
[BMIM]OTf (TfOH)	12.611	0.0254	1.06	0.08	1.05	0.1954	11.4913	**2.2453**
[BMIM]OTs (TsOH)	12.624	0.0072	1.98	0.05	3.76	0.0994	93.7987	**9.3236**

2.5 High resolution mass spectra

ESI-negative



ESI-positive



3. References

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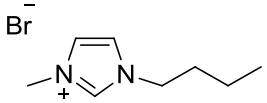
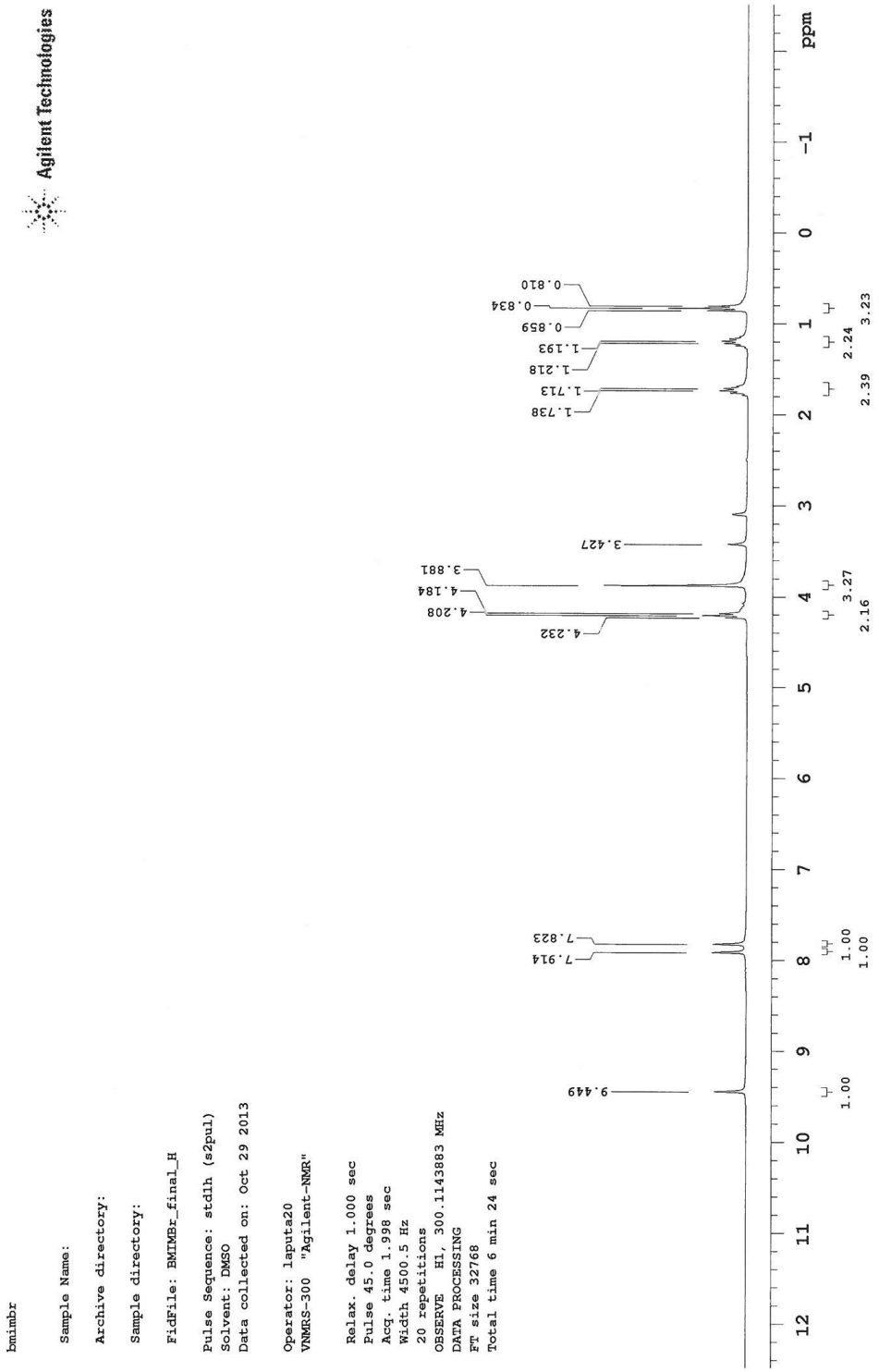
4. Spectral data

1-butyl-3-methylimidazolium bromide





BMIMBr

Sample Name:

Archive directory:

Sample directory:

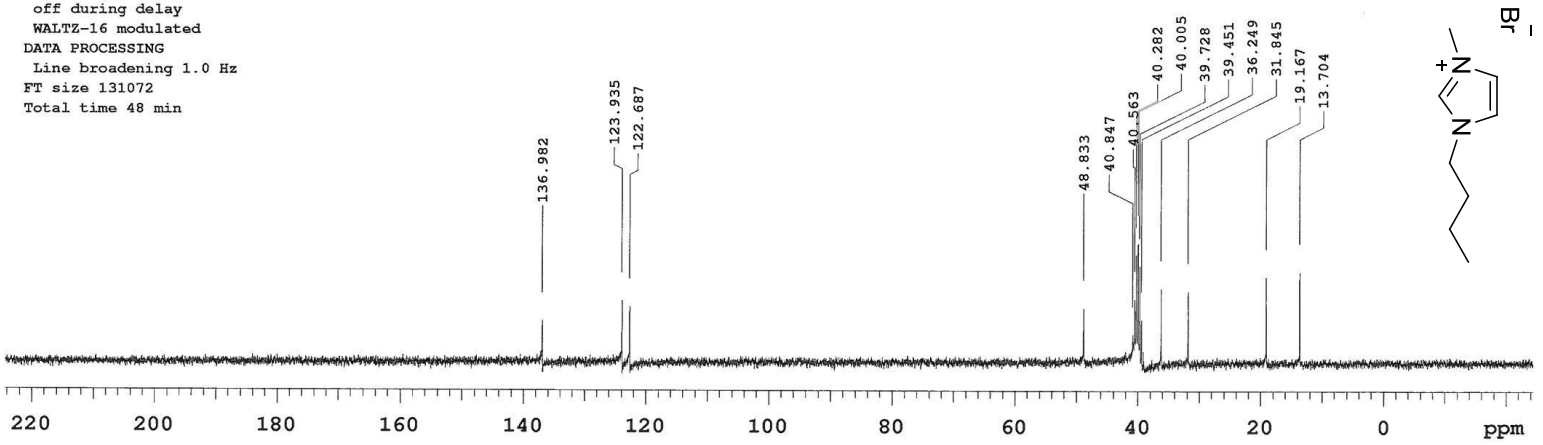
FidFile: BMIMBr_final_C

Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 29 2013

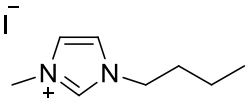
Operator: laputa20
VNMRS-300 "Agilent-NMR"

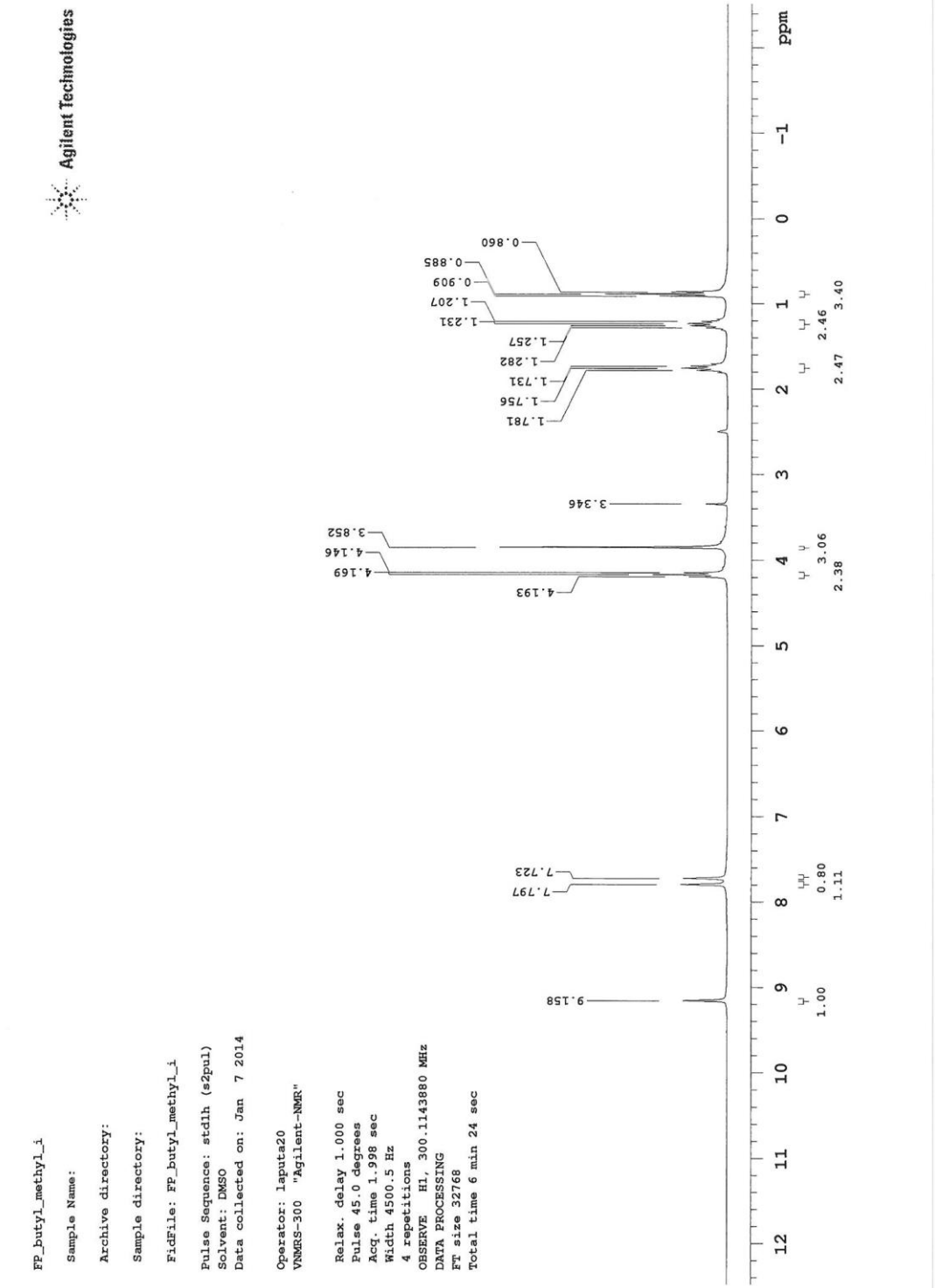
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
56 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min

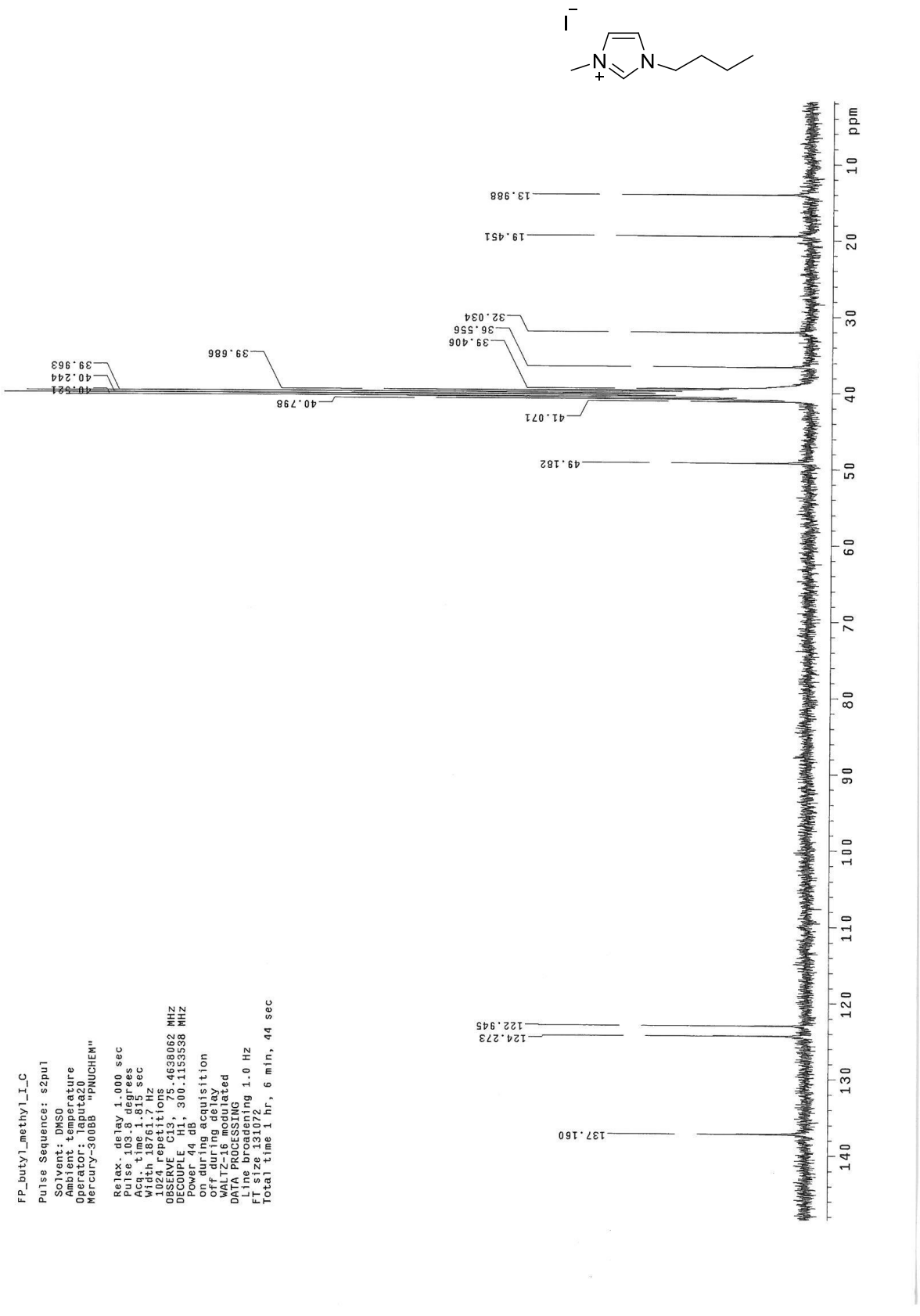
Agilent Technologies



1-butyl-3-methylimidazolium iodide






FP_butyl_methyl_I_C
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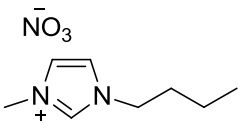
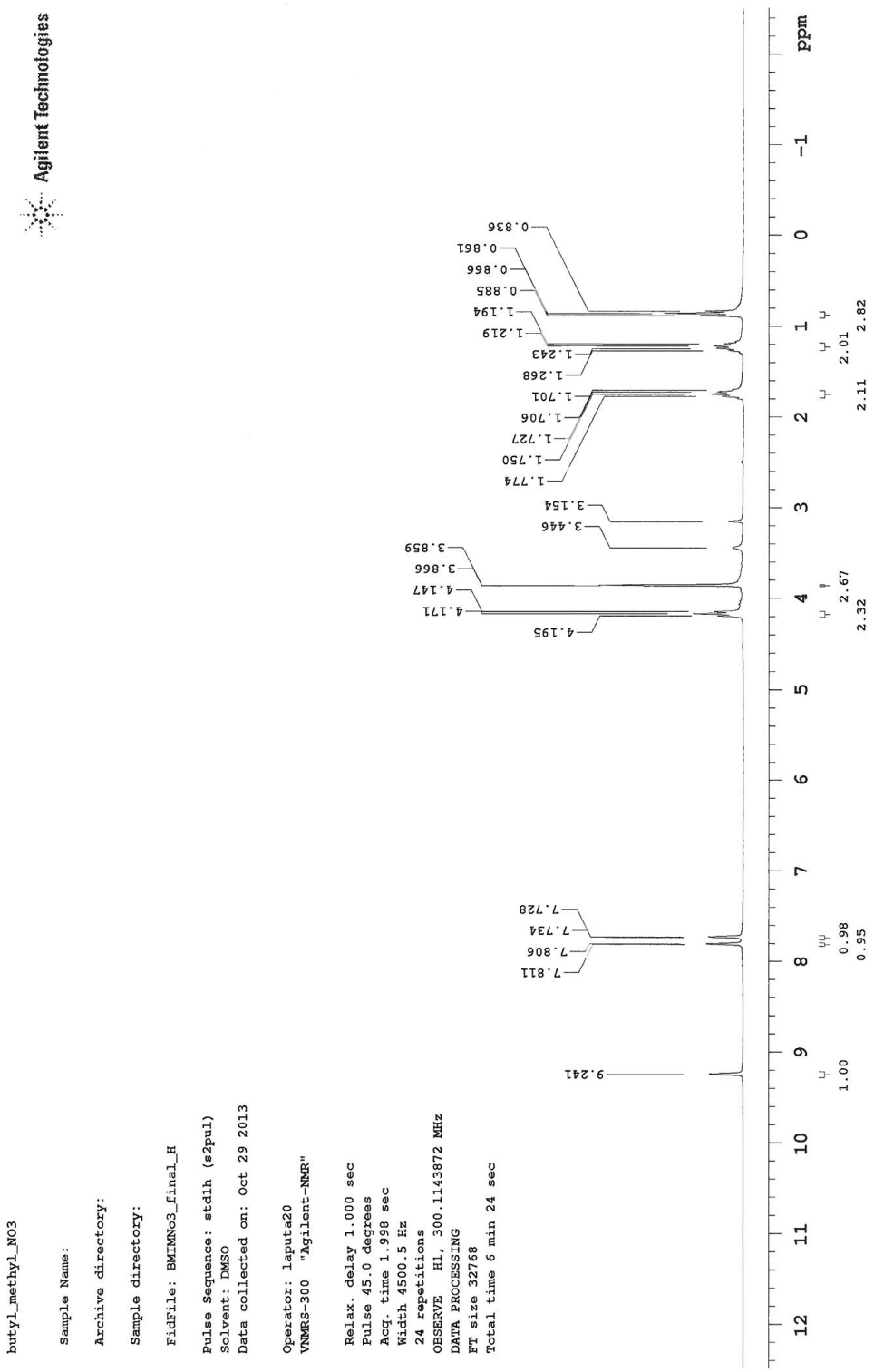
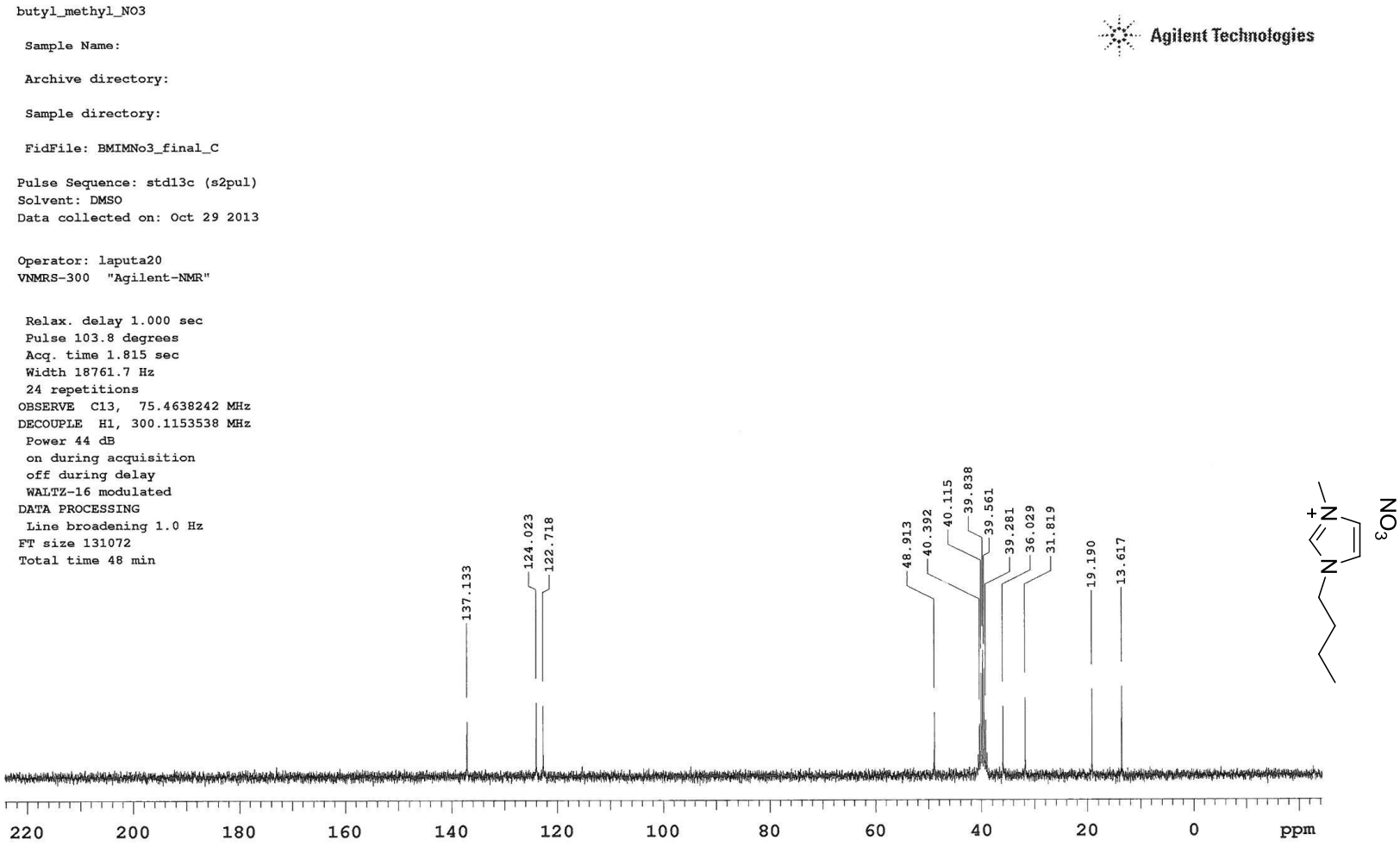
Solvent: DMSO
Ambient temperature
Operator: laputa20
Mercury-300BB "PNUCHEM"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4638062 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 6 min, 44 sec
137.160
124.273
122.945
49.182
41.071
40.798
39.686
39.406
36.556
32.034
19.451
13.988
39.963
40.244
40.521
ppm

1-butyl-3-methylimidazolium nitrate






butyl_methyl_NO3
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Archive directory:
Sample directory:
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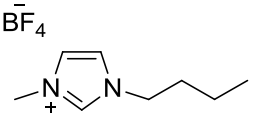
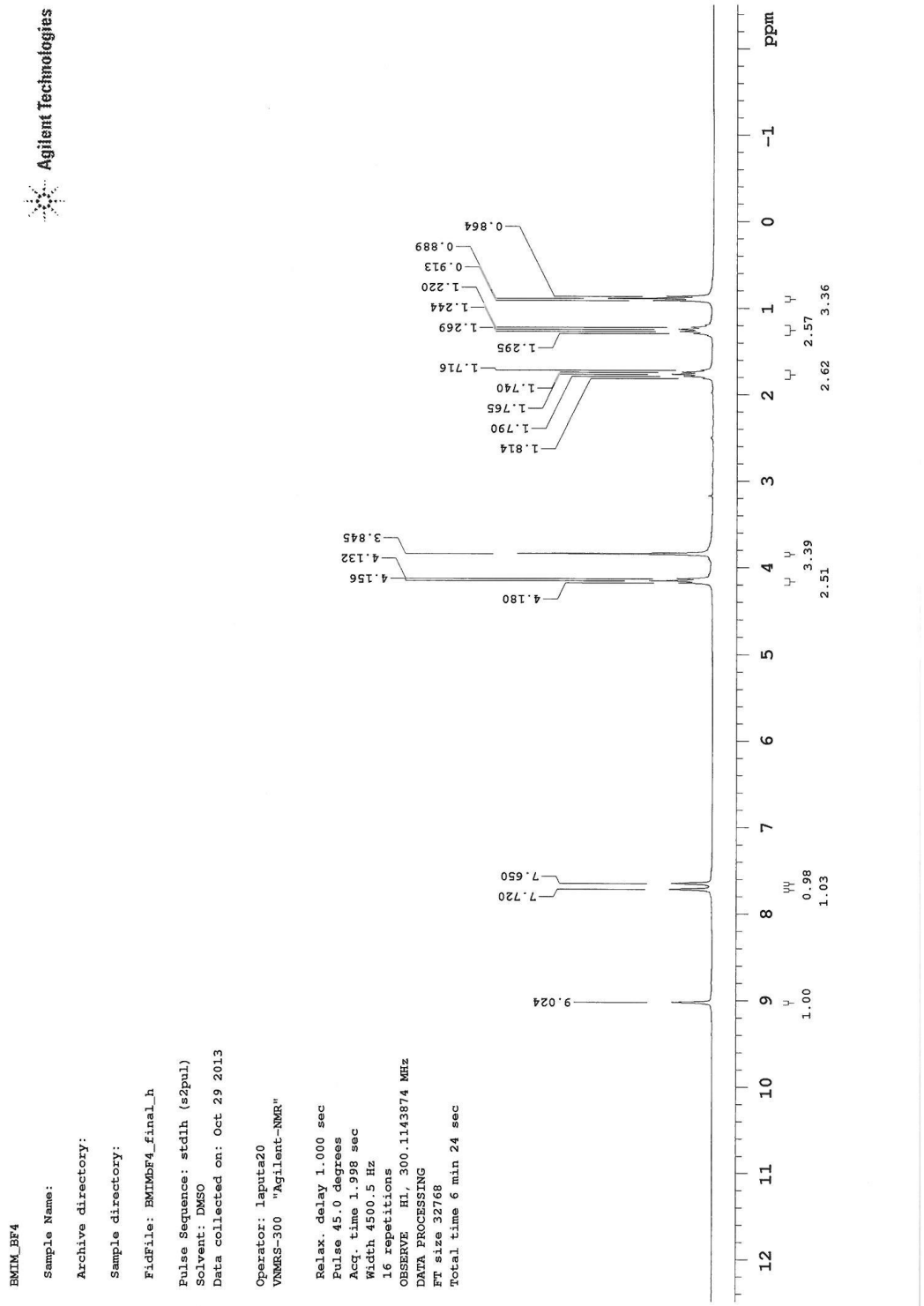
Solvent: DMSO
Data collected on: Oct 29 2013
Operator: laputa20
VNMRS-300 "Agilent-NMR"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
24 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min
Agilent Technologies
137.133
124.023
122.718
48.913
40.392
40.115
39.838
39.561
39.281
36.029
31.819
19.190
13.617
220
200
180
160
140
120
100
80
60
40
20
0
ppm
NO_3^-

1-butyl-3-methylimidazolium tetrafluoroborate





BMIM_BF4

Sample Name:

Archive directory:

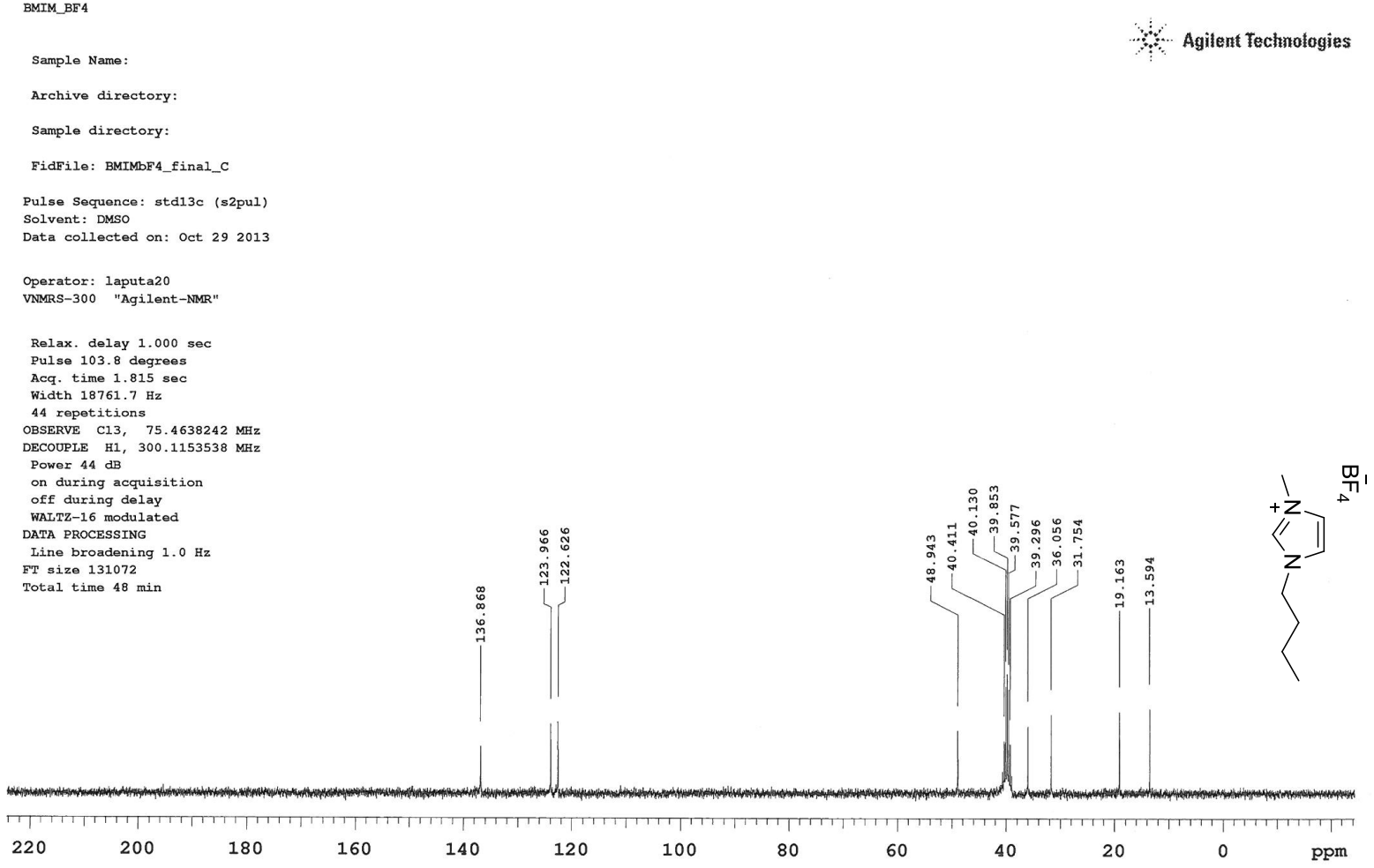
Sample directory:

FidFile: BMIMbF4_final_C

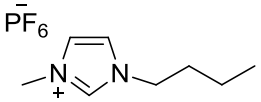
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 29 2013

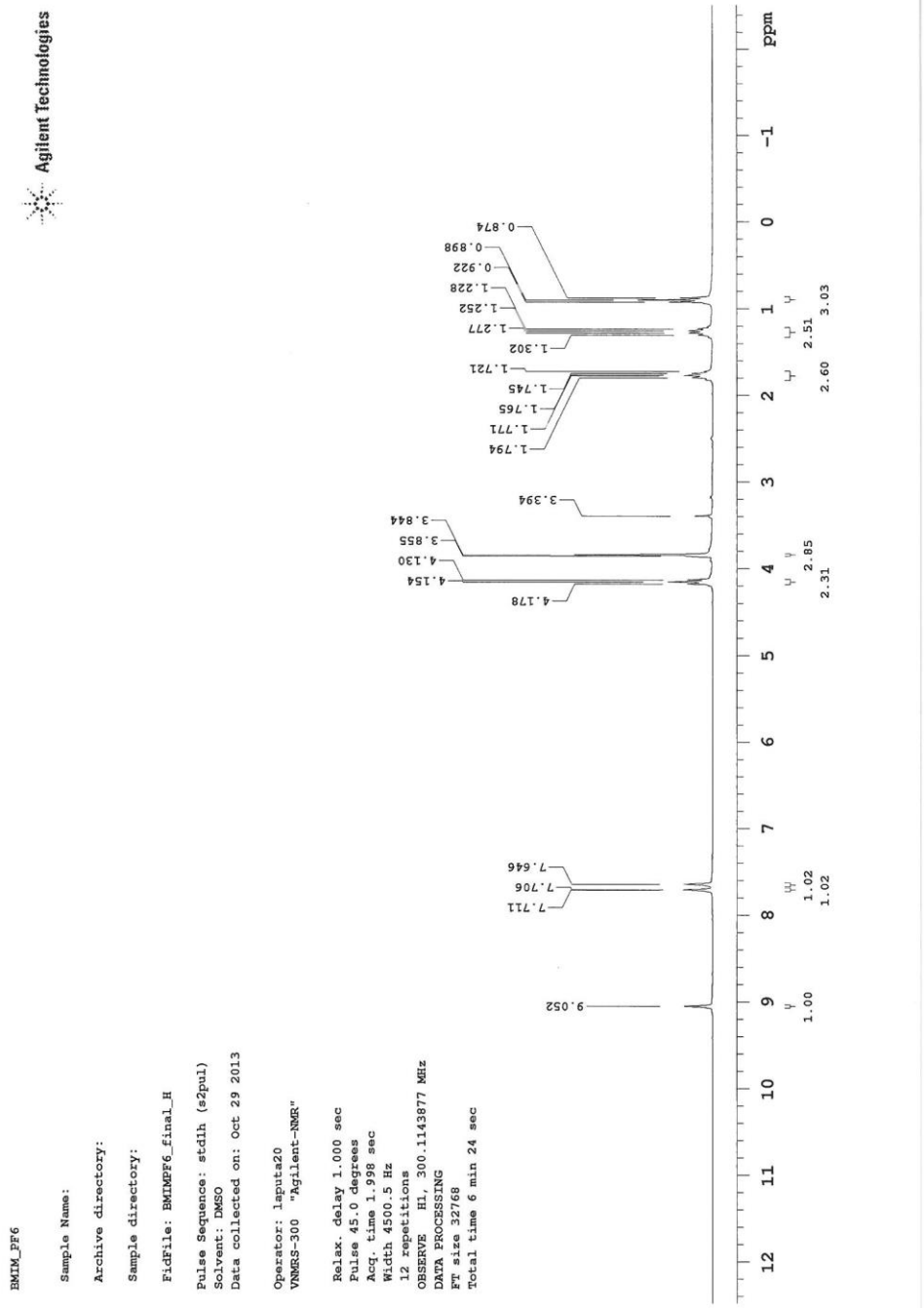
Operator: laputa20
VNMRS-300 "Agilent-NMR"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
44 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min



1-butyl-3-methylimidazolium hexafluorophosphate





13C OBSERVE

Sample Name:

Archive directory:

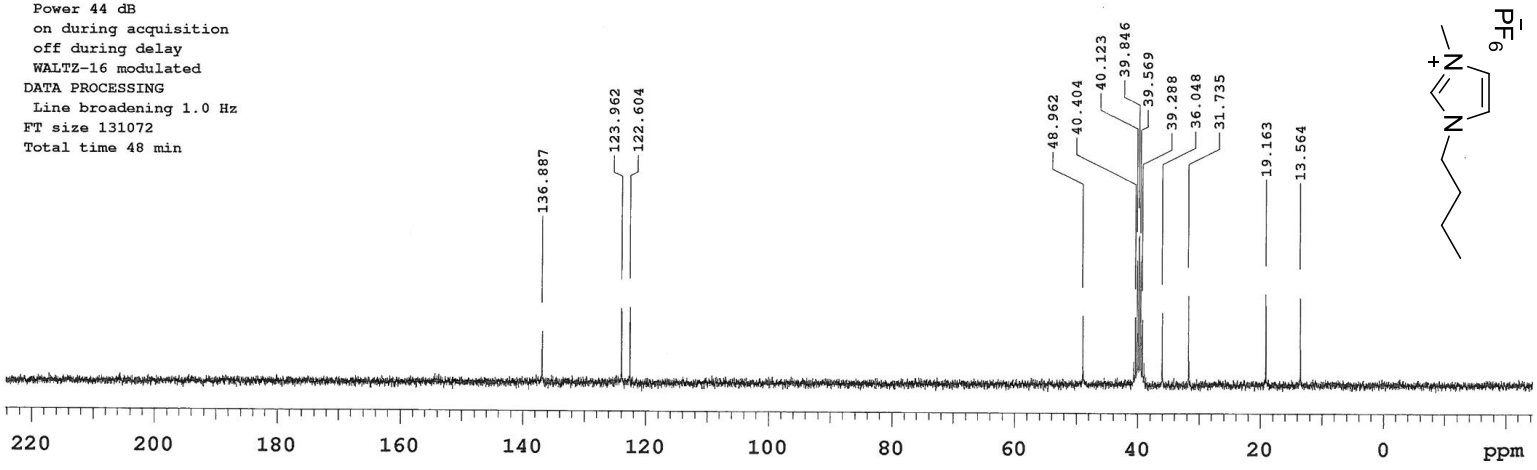
Sample directory:

FidFile: BMIMPF6_final_C

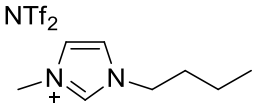
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 29 2013

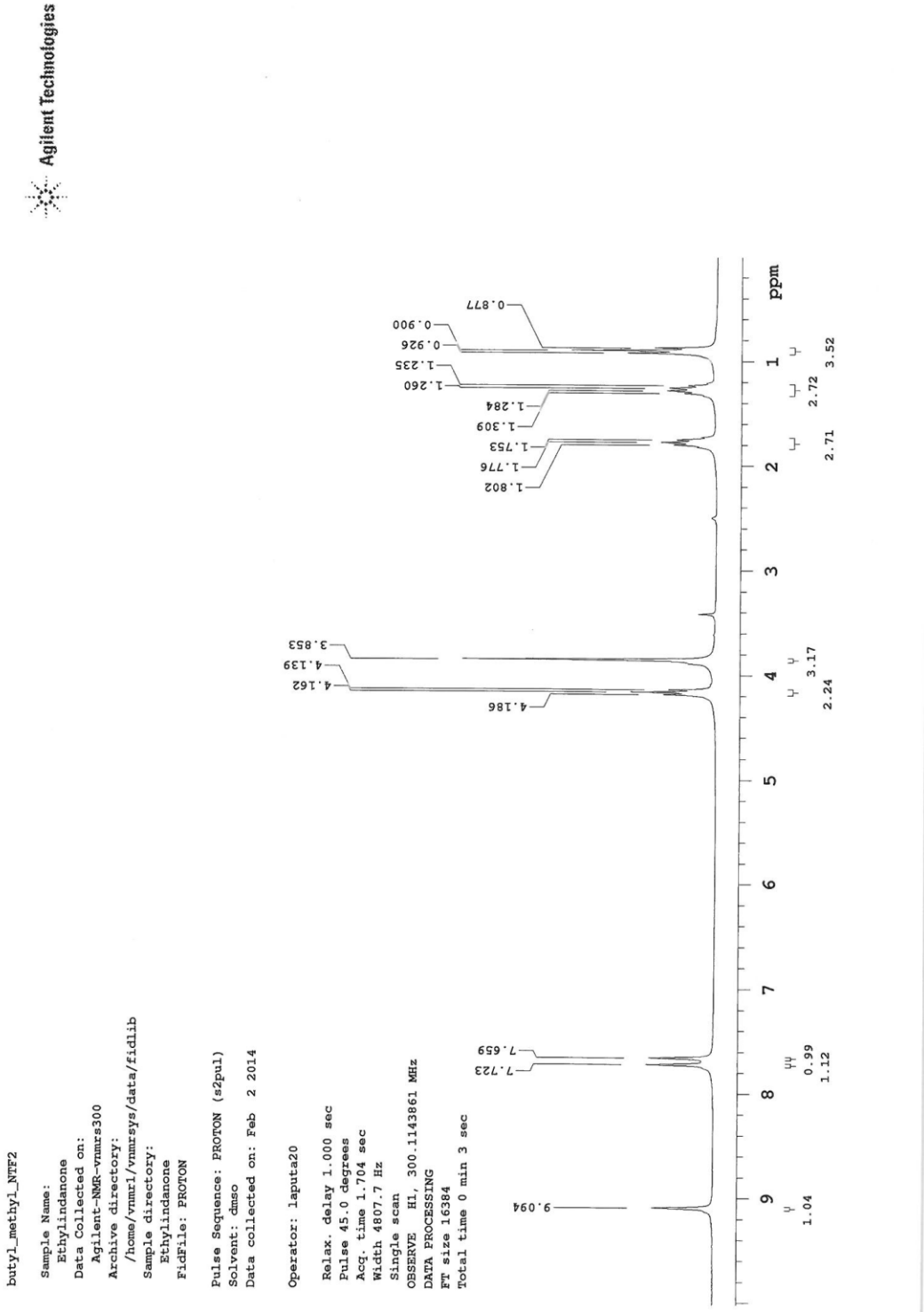
Operator: laputa20
VNMRS-300 "Agilent-NMR"

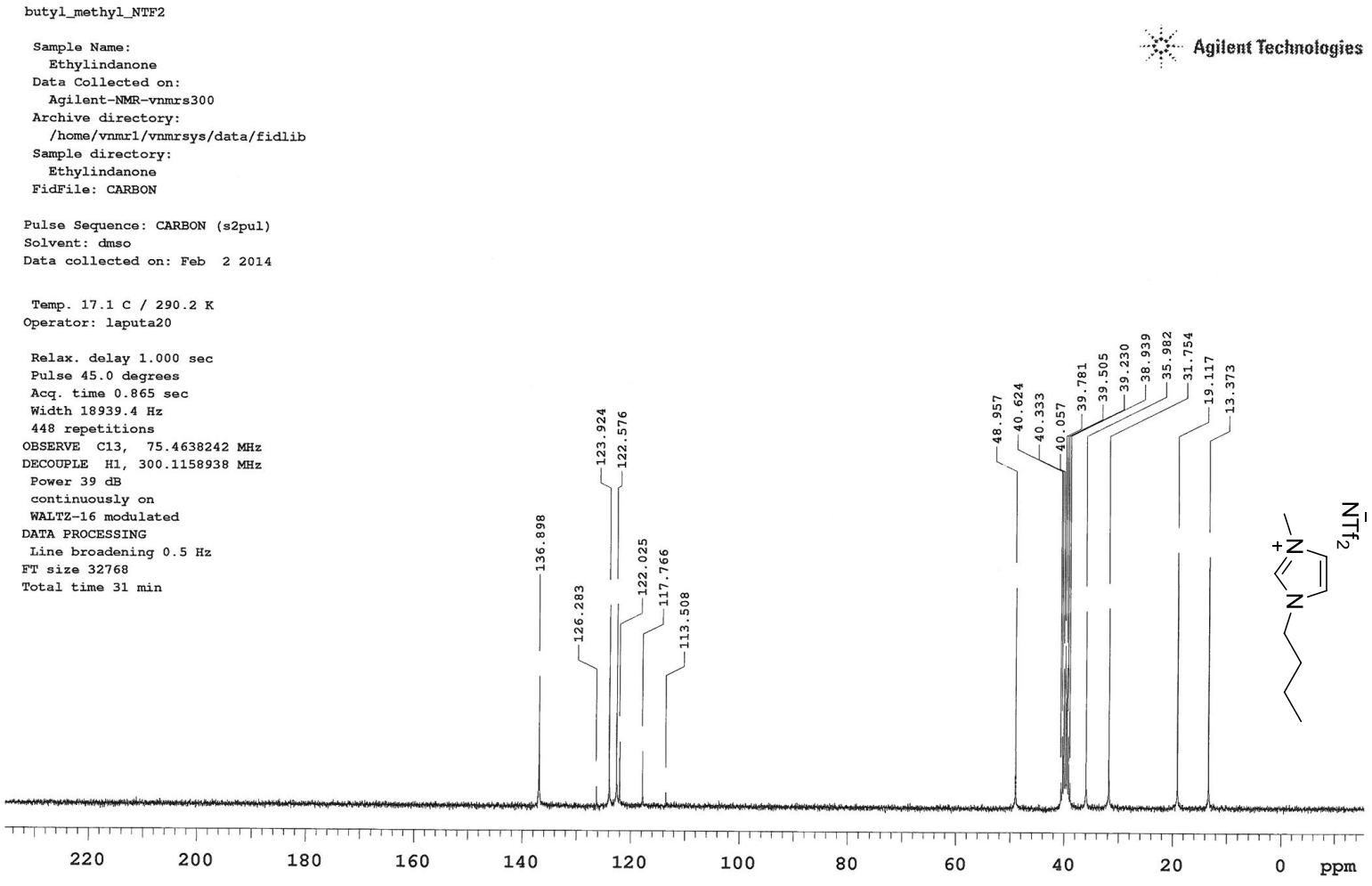
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
60 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min



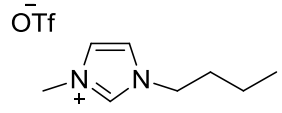
1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)amide

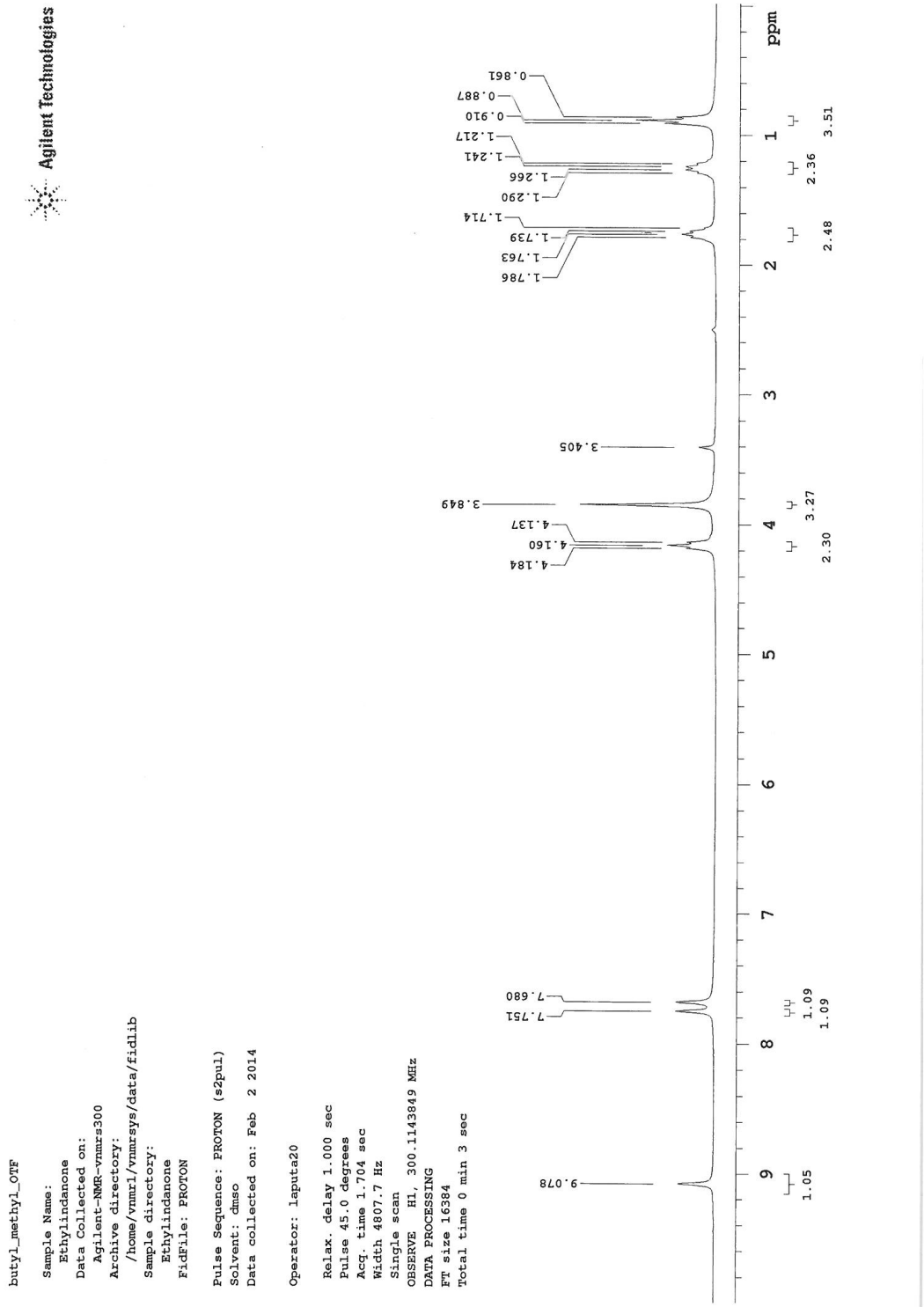





butyl_methyl_NTF2
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 2 2014
Temp. 17.1 C / 290.2 K
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
448 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
136.898
126.283
123.924
122.576
122.025
117.766
113.508
48.957
40.624
40.333
40.057
39.781
39.505
39.230
38.939
35.982
31.754
19.117
13.373
NTf2
220
200
180
160
140
120
100
80
60
40
20
0
ppm

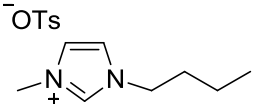
1-butyl-3-methylimidazolium trifluoromethanesulfonate

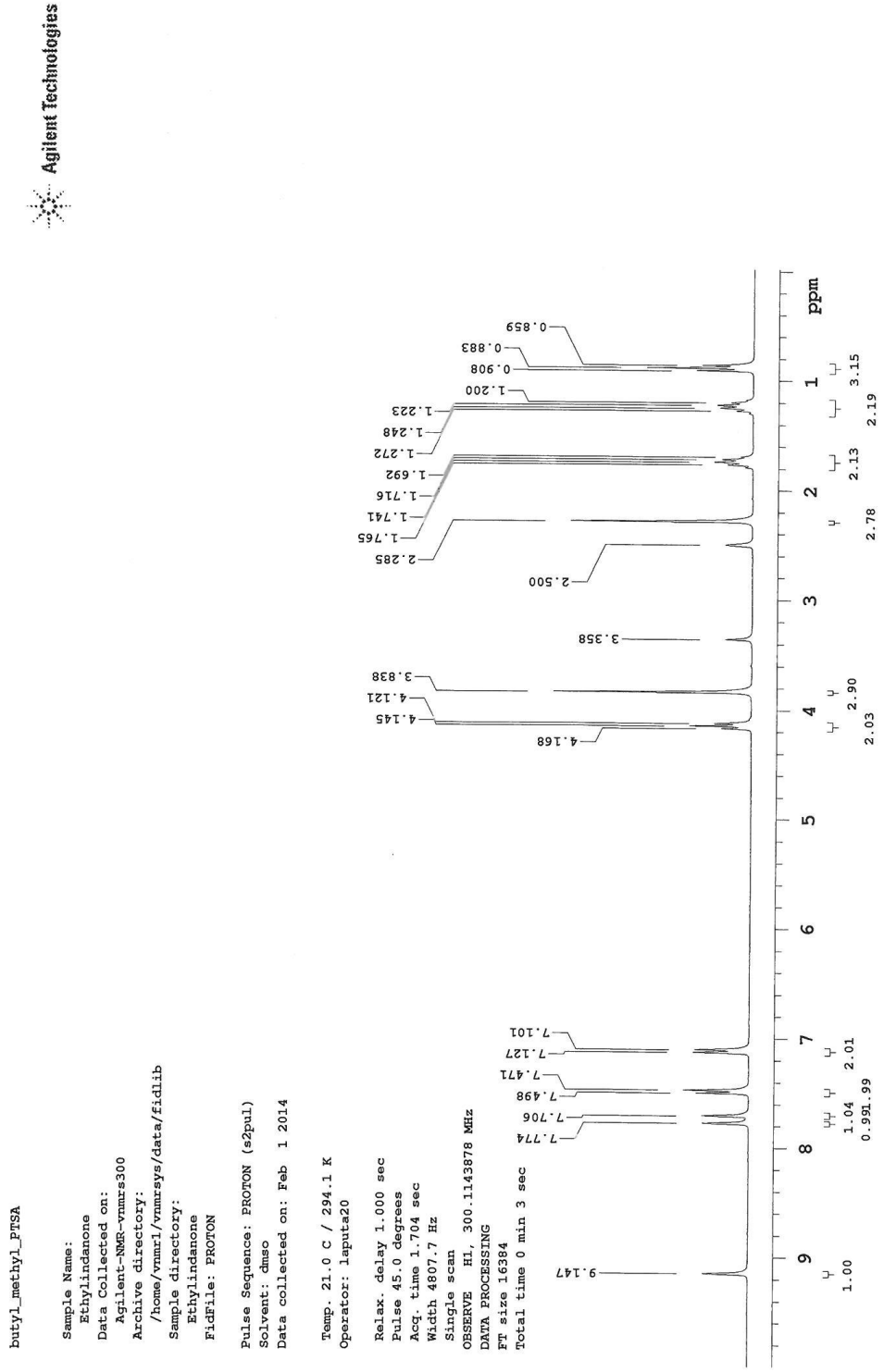


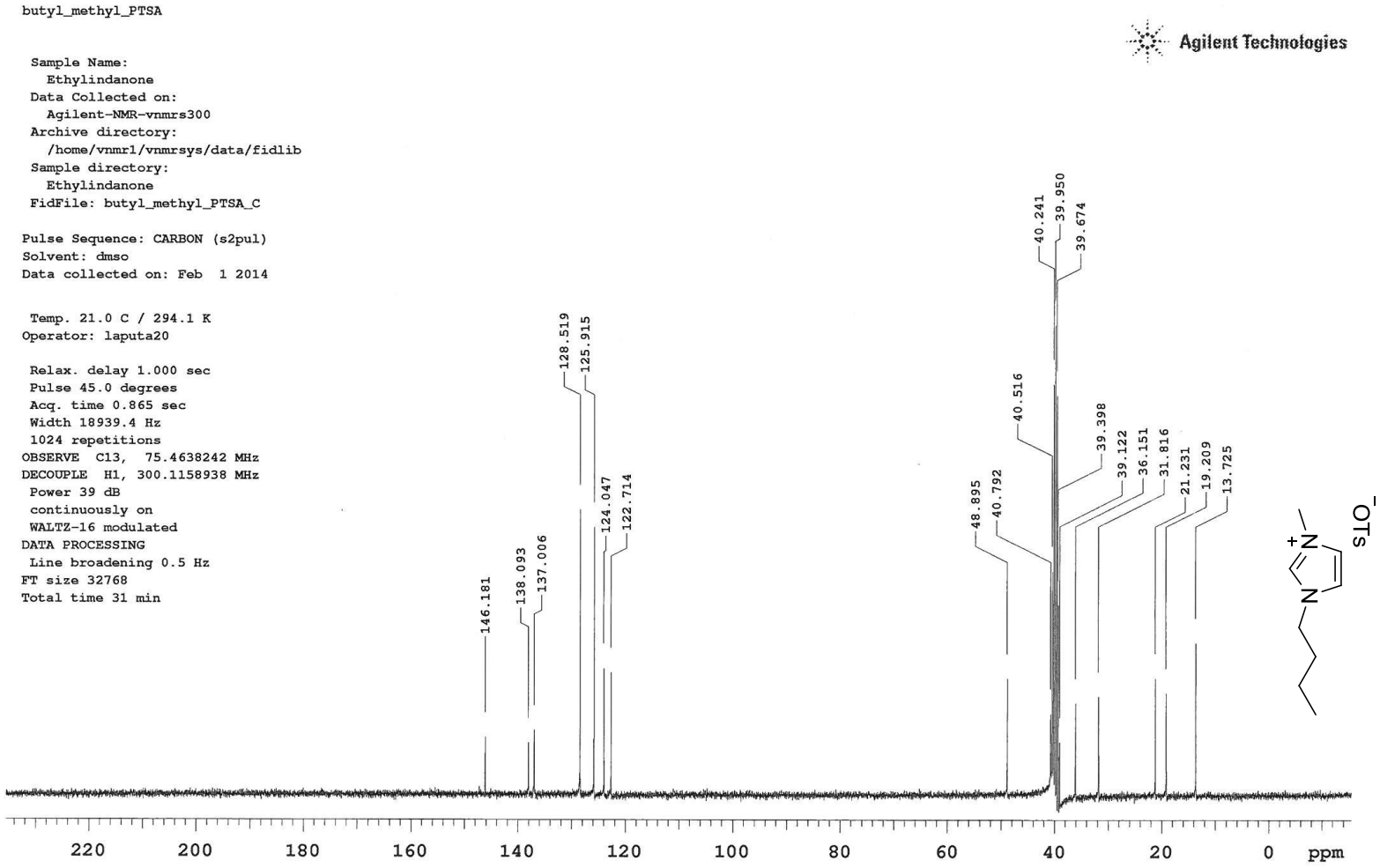


butyl_methyl_OTF
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: butyl_methyl_OFT_C
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 2 2014
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
OTf
13.556
19.148
31.754
36.074
39.000
39.276
39.551
39.827
40.103
40.379
40.654
48.926
114.656
118.915
122.622
123.189
123.970
127.447
136.898
ppm

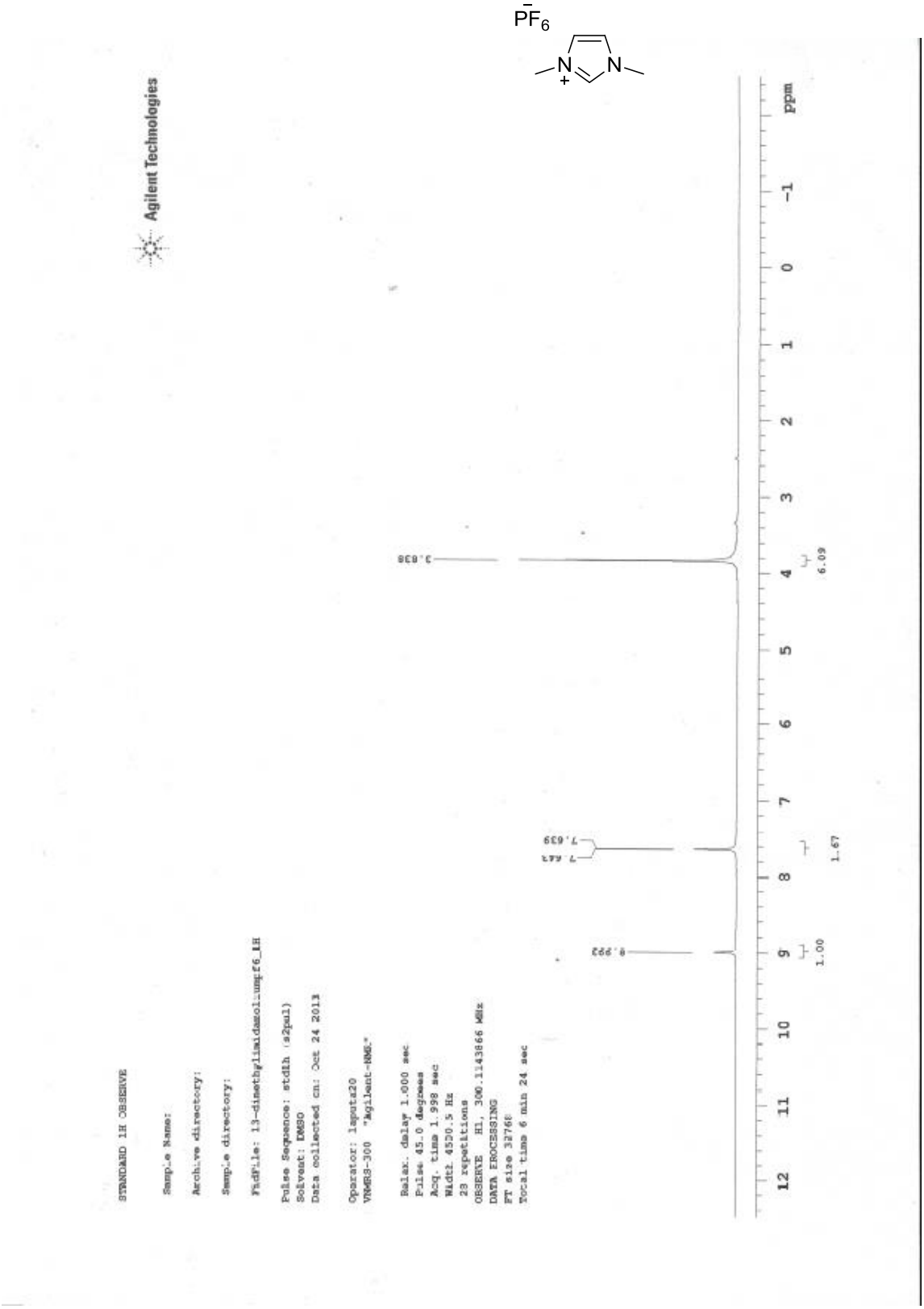
1-butyl-3-methylimidazolium 4-methylbezneznsulfonate






butyl_methyl_PTSA
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmrl/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: butyl_methyl_PTSA_C
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 1 2014
Temp. 21.0 C / 294.1 K
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
146.181
138.093
137.006
128.519
125.915
124.047
122.714
48.895
40.792
40.516
40.241
39.950
39.674
39.398
39.122
36.151
31.816
21.231
19.209
13.725
OTs
220
200
180
160
140
120
100
80
60
40
20
0
ppm

1,3-dimethylimidazolium hexafluorophosphate



methyl_methyl_PF6

Sample Name:

Archive directory:

Sample directory:

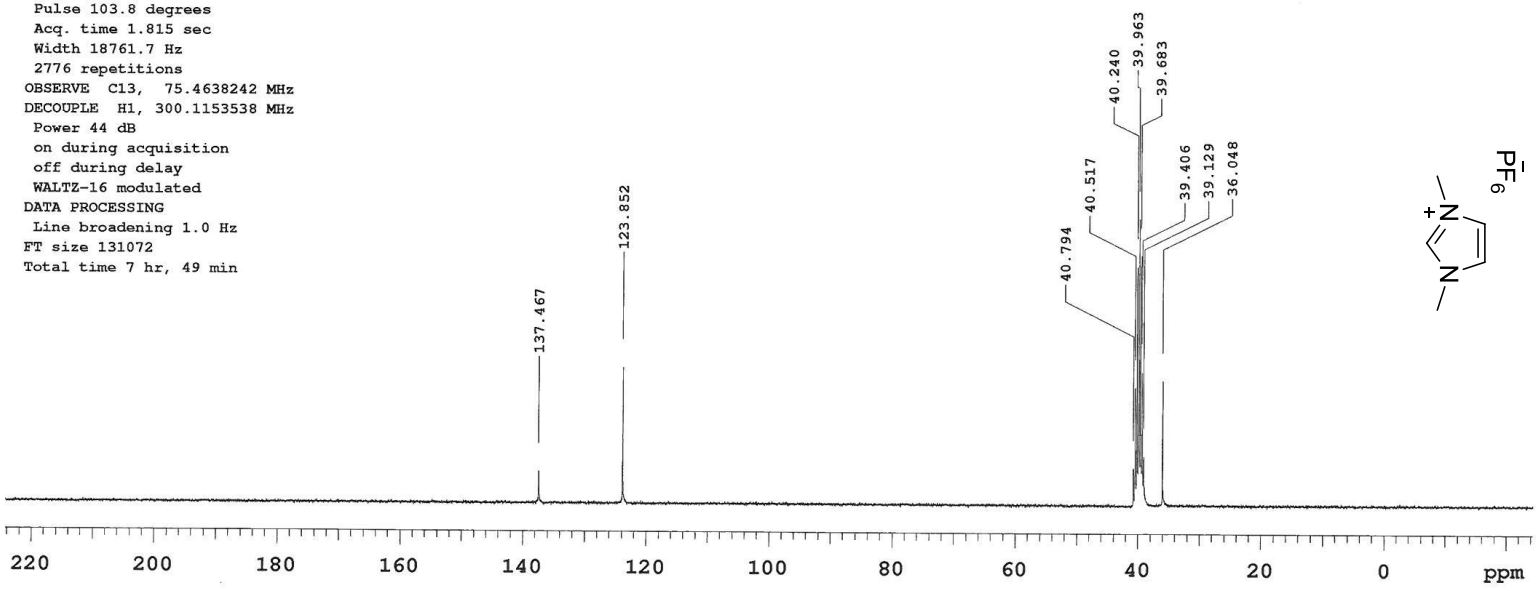
FidFile: 13-dimethylimidazoliumpf6

Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 24 2013

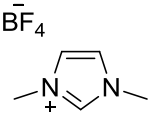
Operator: laputa20
VNMRS-300 "Agilent-NMR"

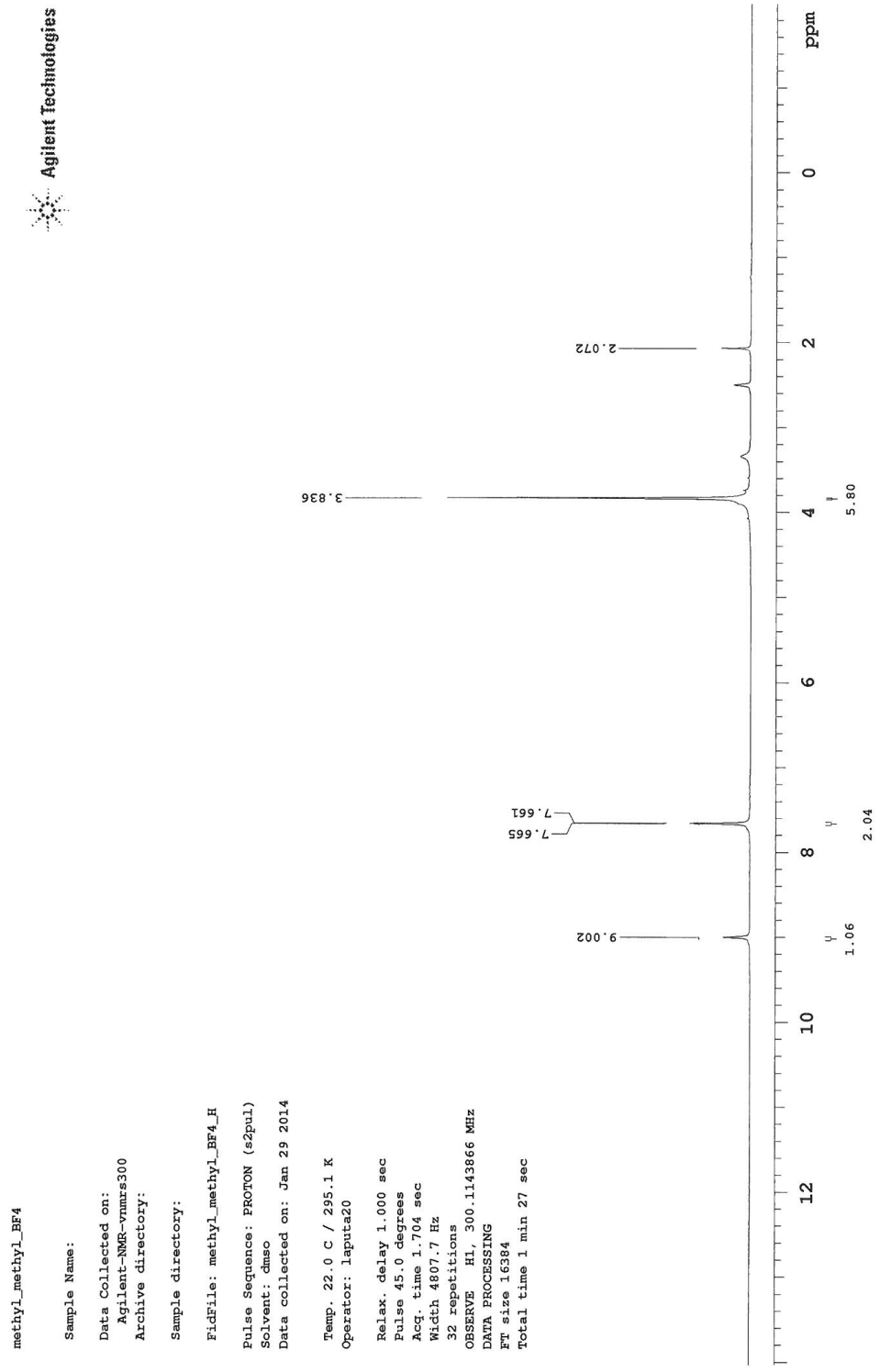
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
2776 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 49 min

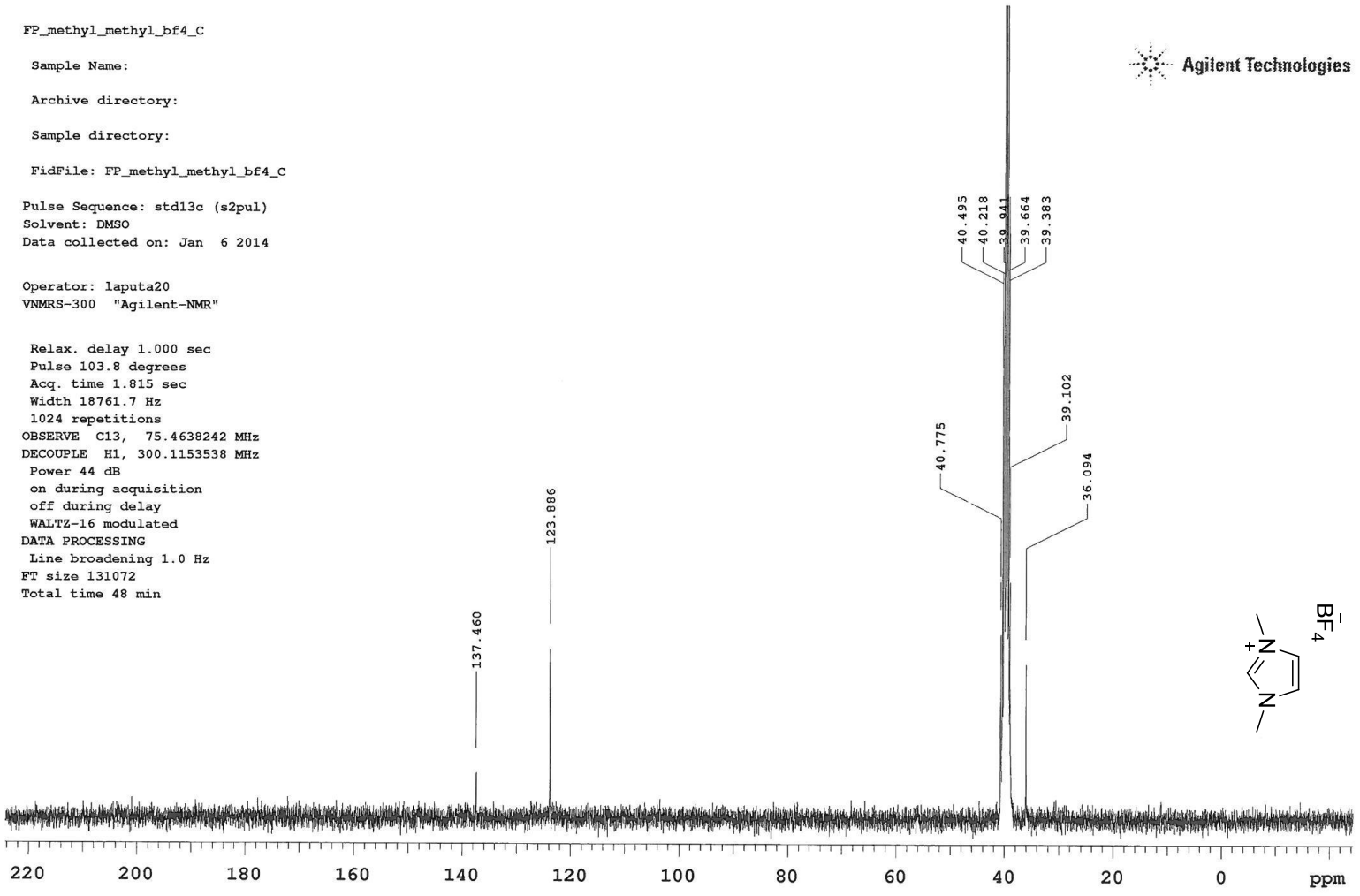
Agilent Technologies



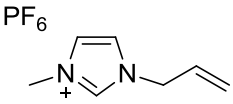
1,3-dimethylimidazolium tetrafluoroborate

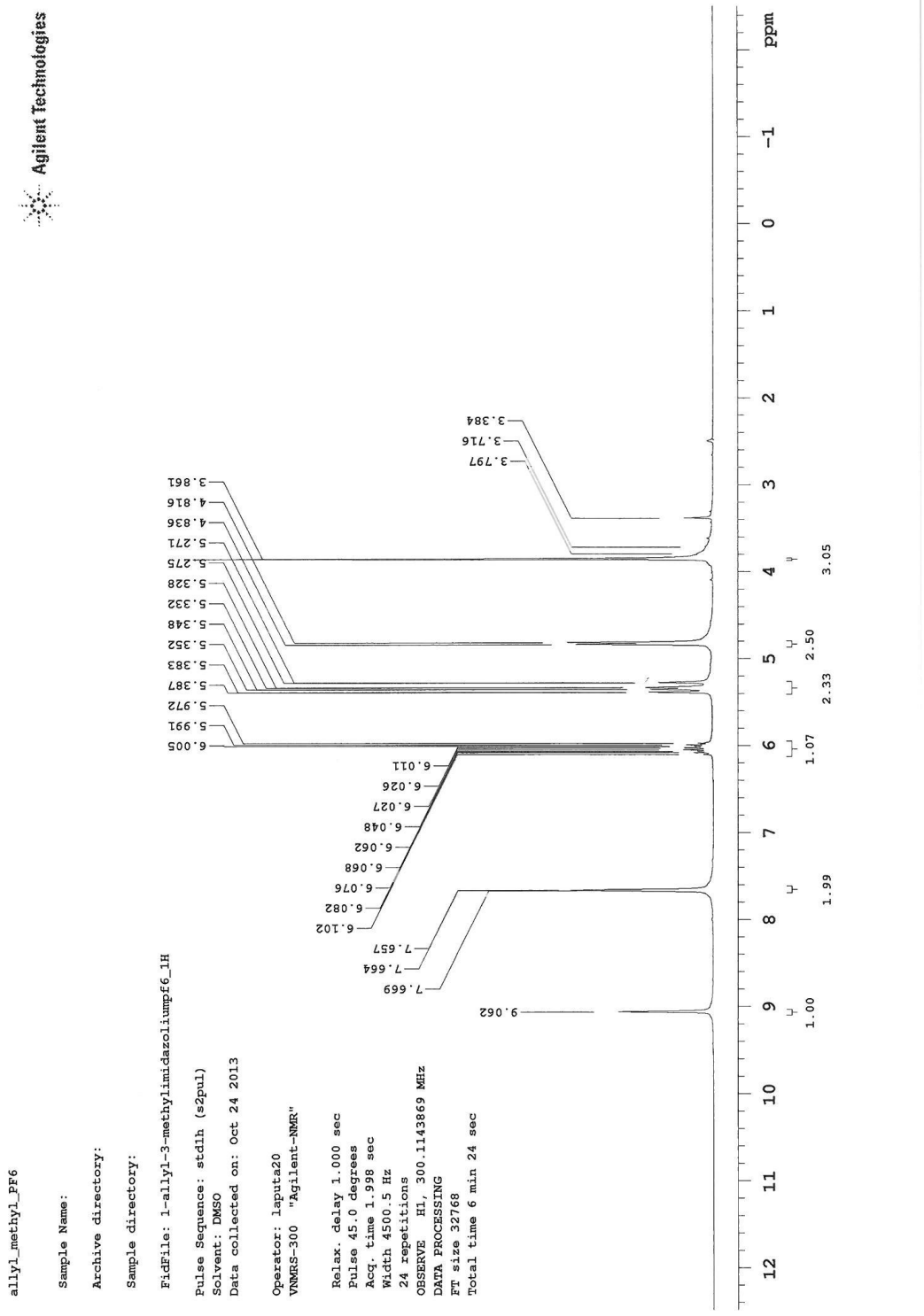





FP_methyl_methyl_bf4_C
Sample Name:
Archive directory:
Sample directory:
FidFile: FP_methyl_methyl_bf4_C
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Jan 6 2014
Operator: laputa20
VNMRS-300 "Agilent-NMR"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min
Agilent Technologies
137.460
123.886
40.775
40.495
40.218
39.941
39.664
39.383
39.102
36.094
220
200
180
160
140
120
100
80
60
40
20
0
ppm

1-allyl-3-methylimidazolium hexafluorophosphate





13C OBSERVE

Sample Name:

Archive directory:

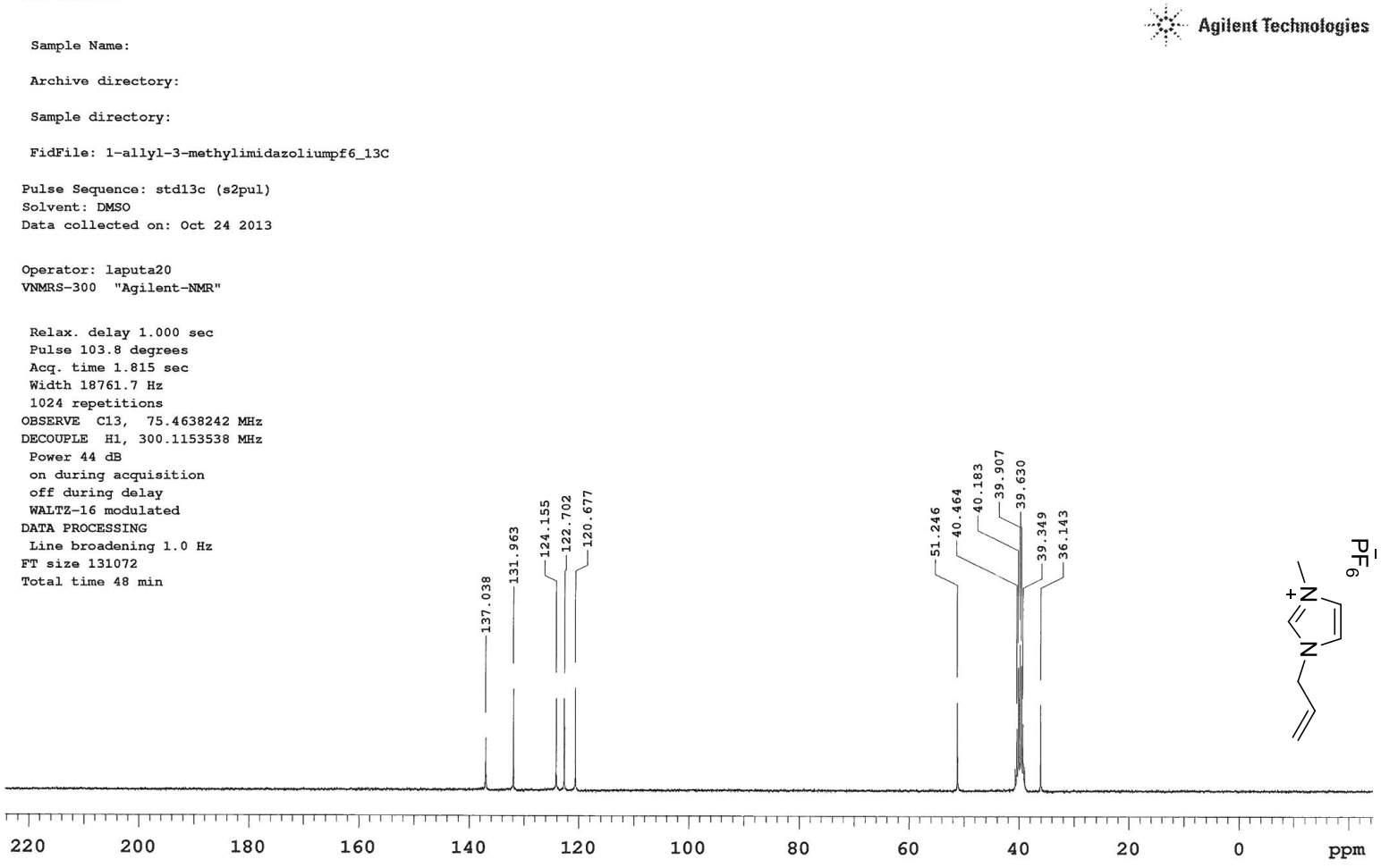
Sample directory:

FidFile: 1-allyl-3-methylimidazoliumpf6_13C

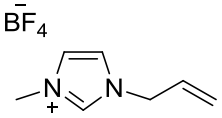
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 24 2013

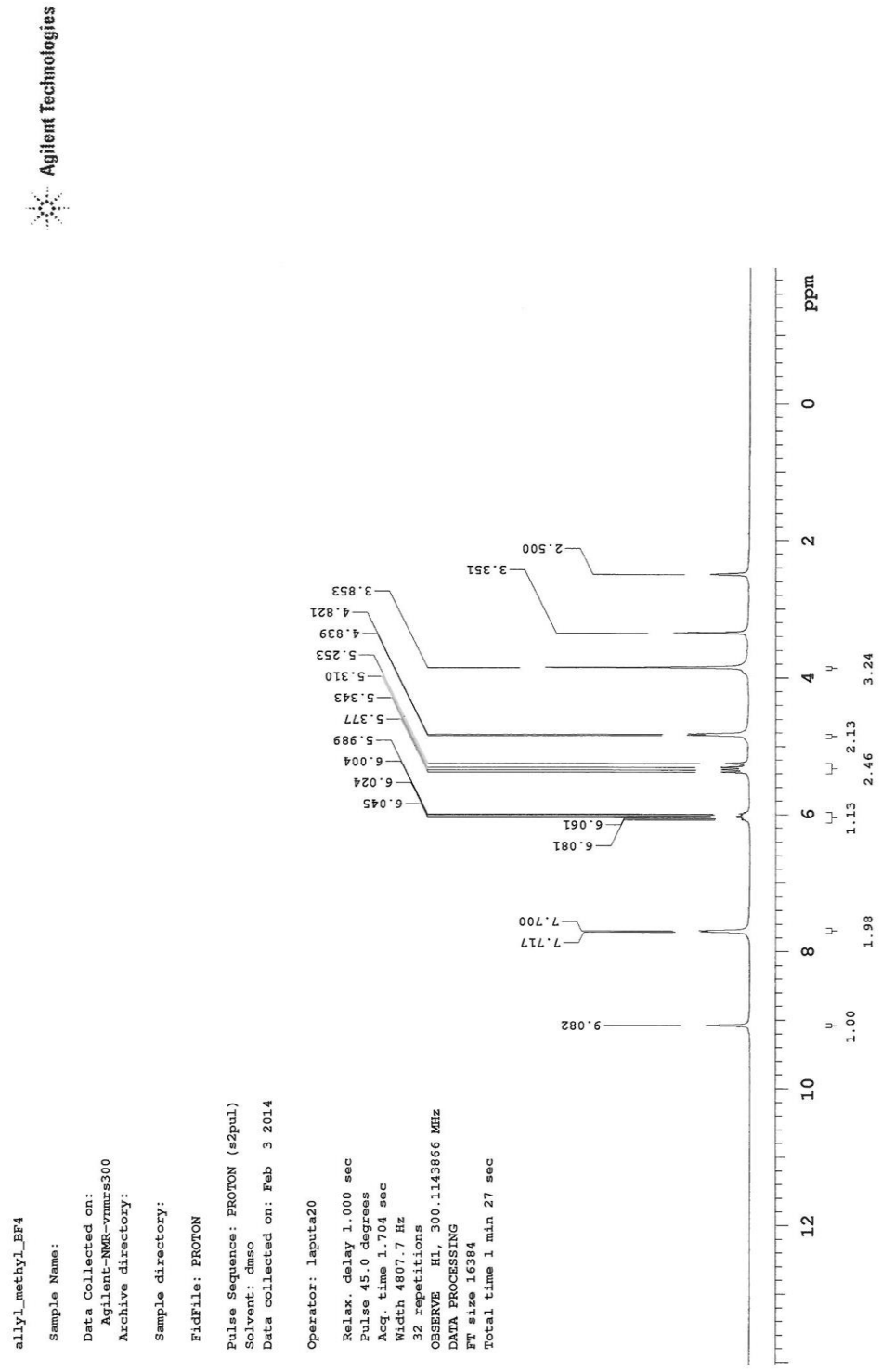
Operator: laputa20
VNMRS-300 "Agilent-NMR"

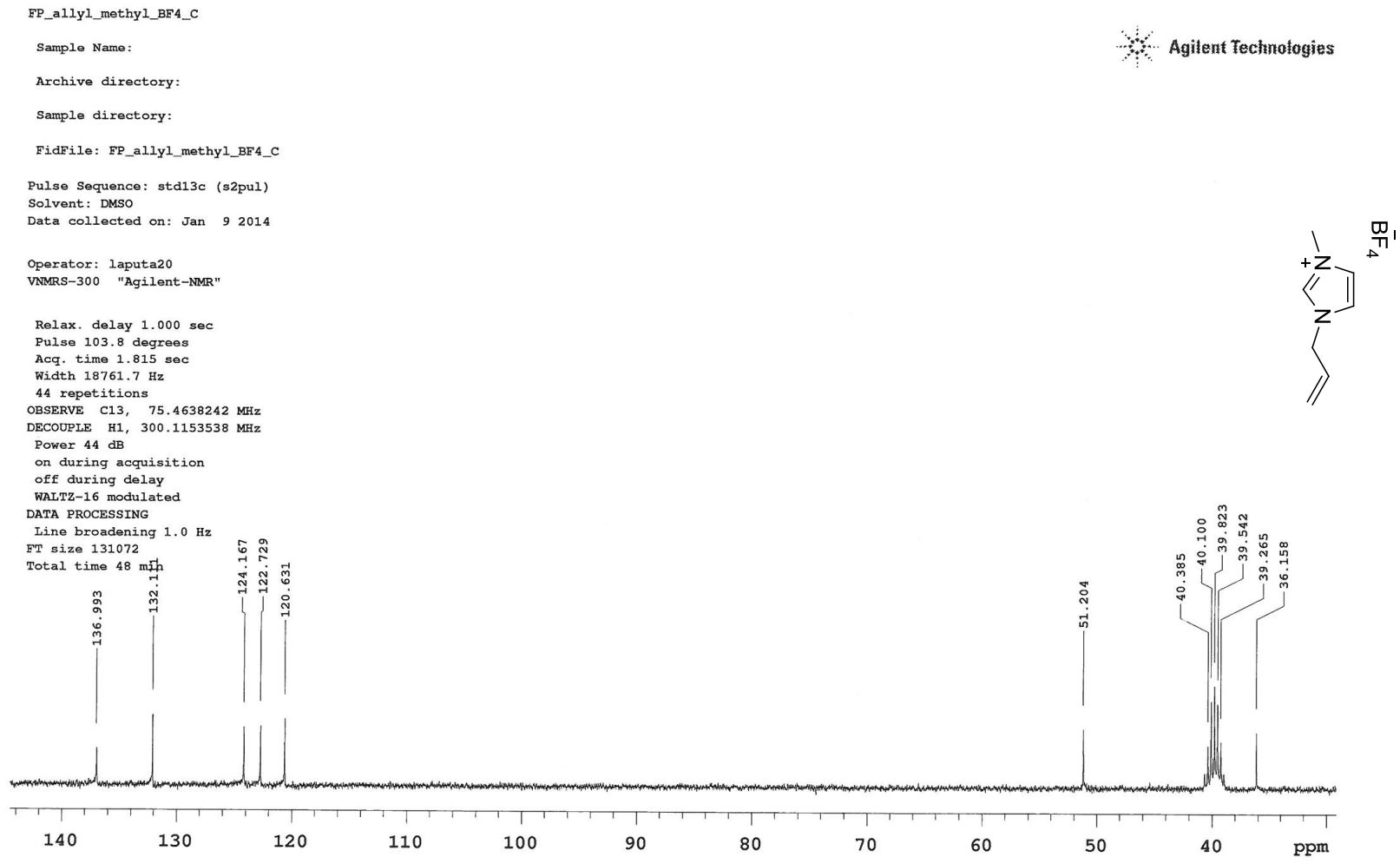
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min



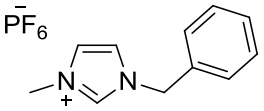
1-allyl-3-methylimidazolium tetrafluoroborate

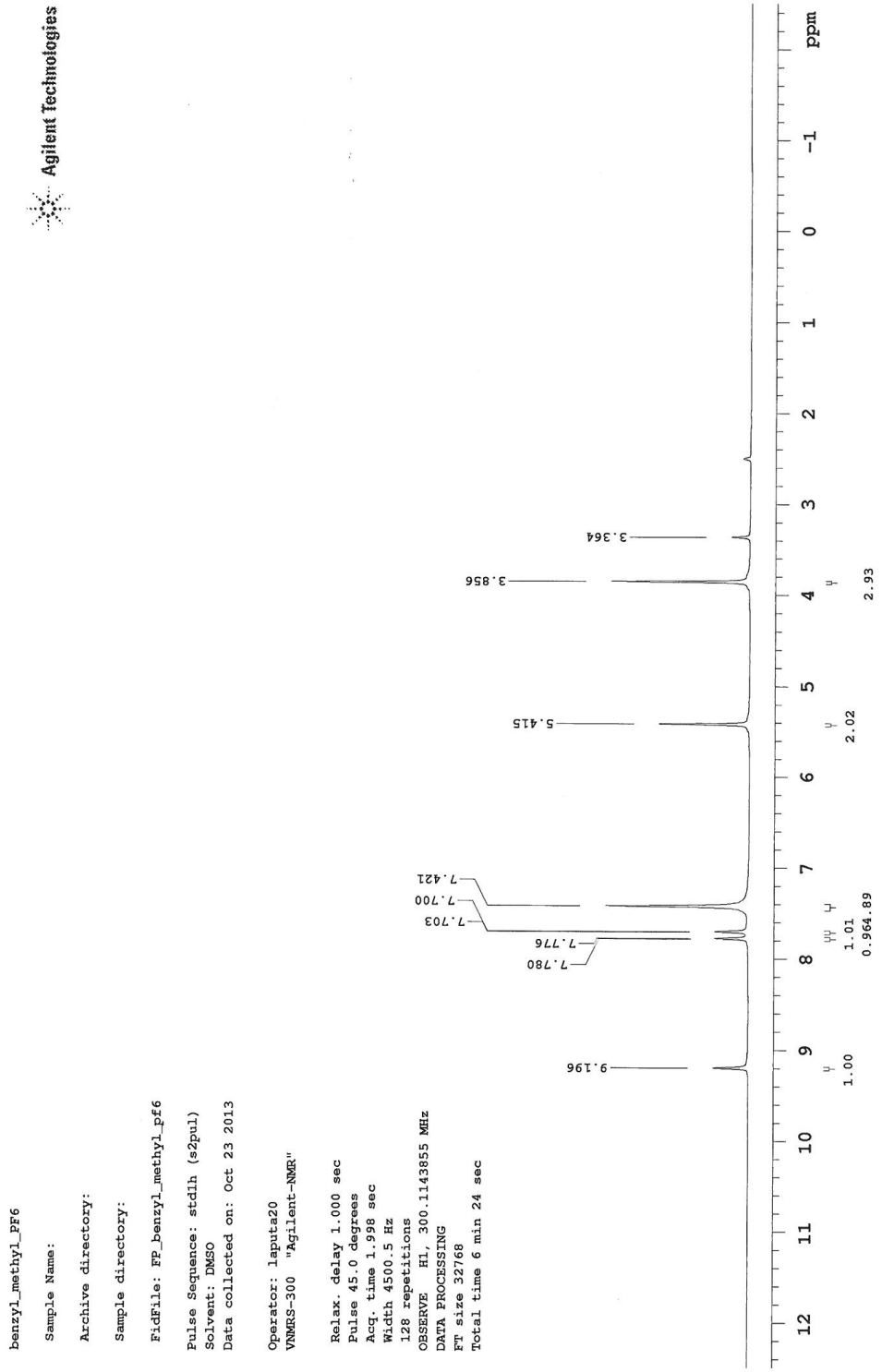





FP_allyl_methyl_BF4_C
Sample Name:
Archive directory:
Sample directory:
FidFile: FP_allyl_methyl_BF4_C
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Jan 9 2014
Operator: laputa20
VNMRS-300 "Agilent-NMR"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
44 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min
Agilent Technologies
136.993
132.111
124.167
122.729
120.631
51.204
40.385
40.100
39.823
39.542
39.265
36.158
140
130
120
110
100
90
80
70
60
50
40
ppm

1-benzyl-3-methylimidazolium hexafluorophosphate





benzyl_methyl_PF6

Agilent Technologies

Sample Name:

Archive directory:

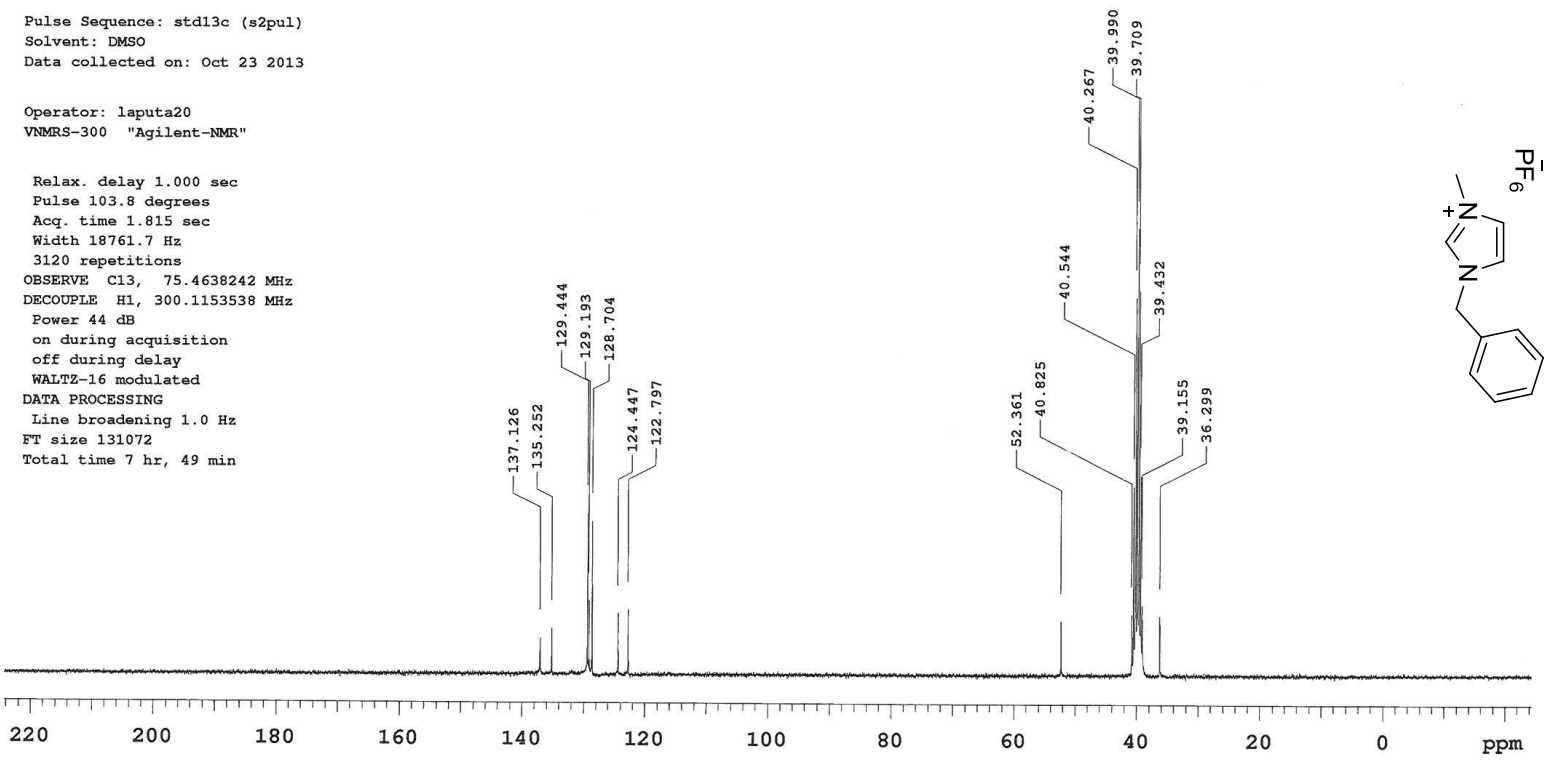
Sample directory:

FidFile: FP_benzyl_methyl_pf6_c

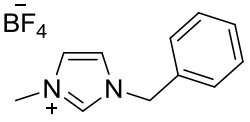
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 23 2013

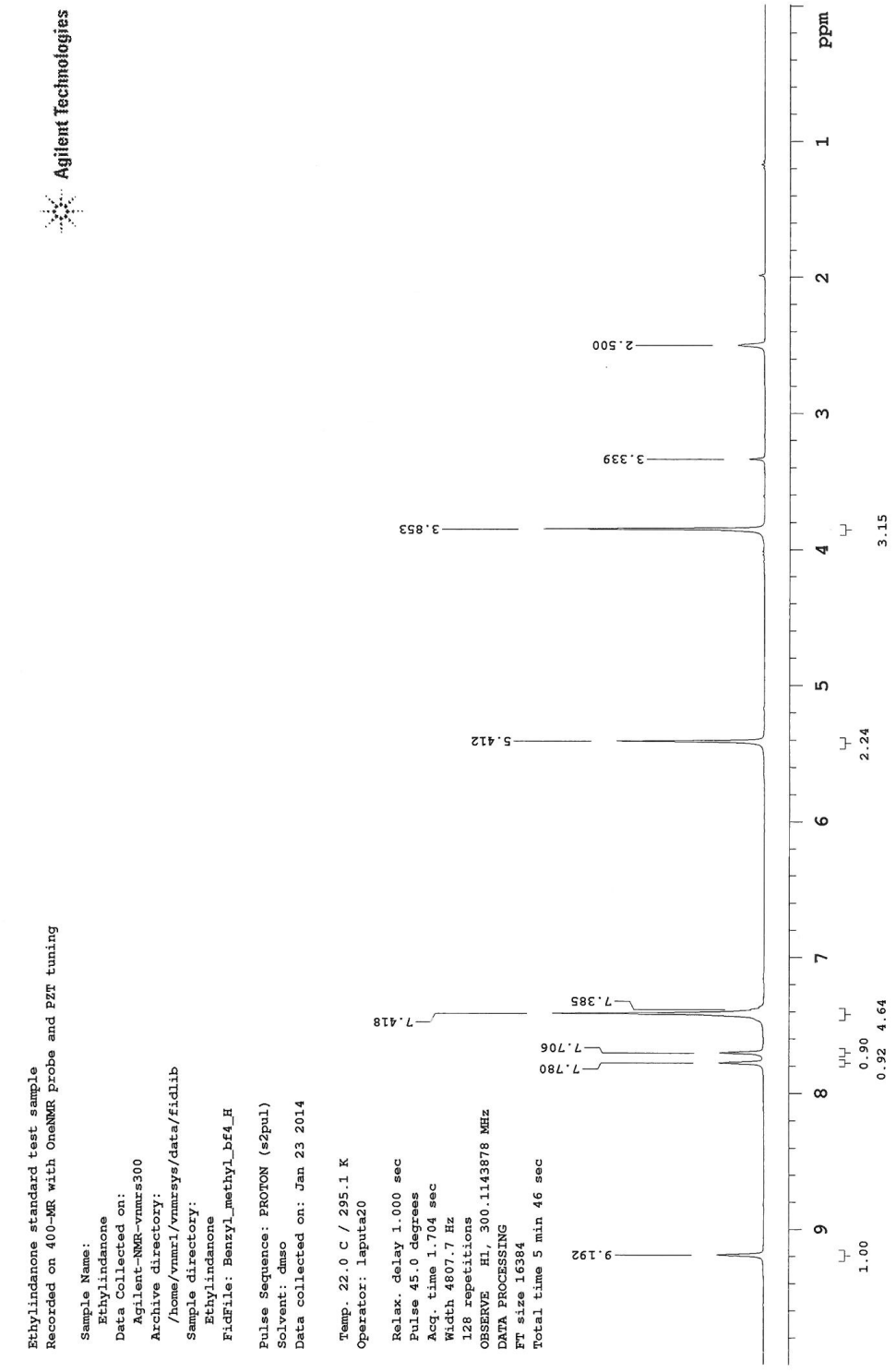
Operator: laputa20
VNMRS-300 "Agilent-NMR"

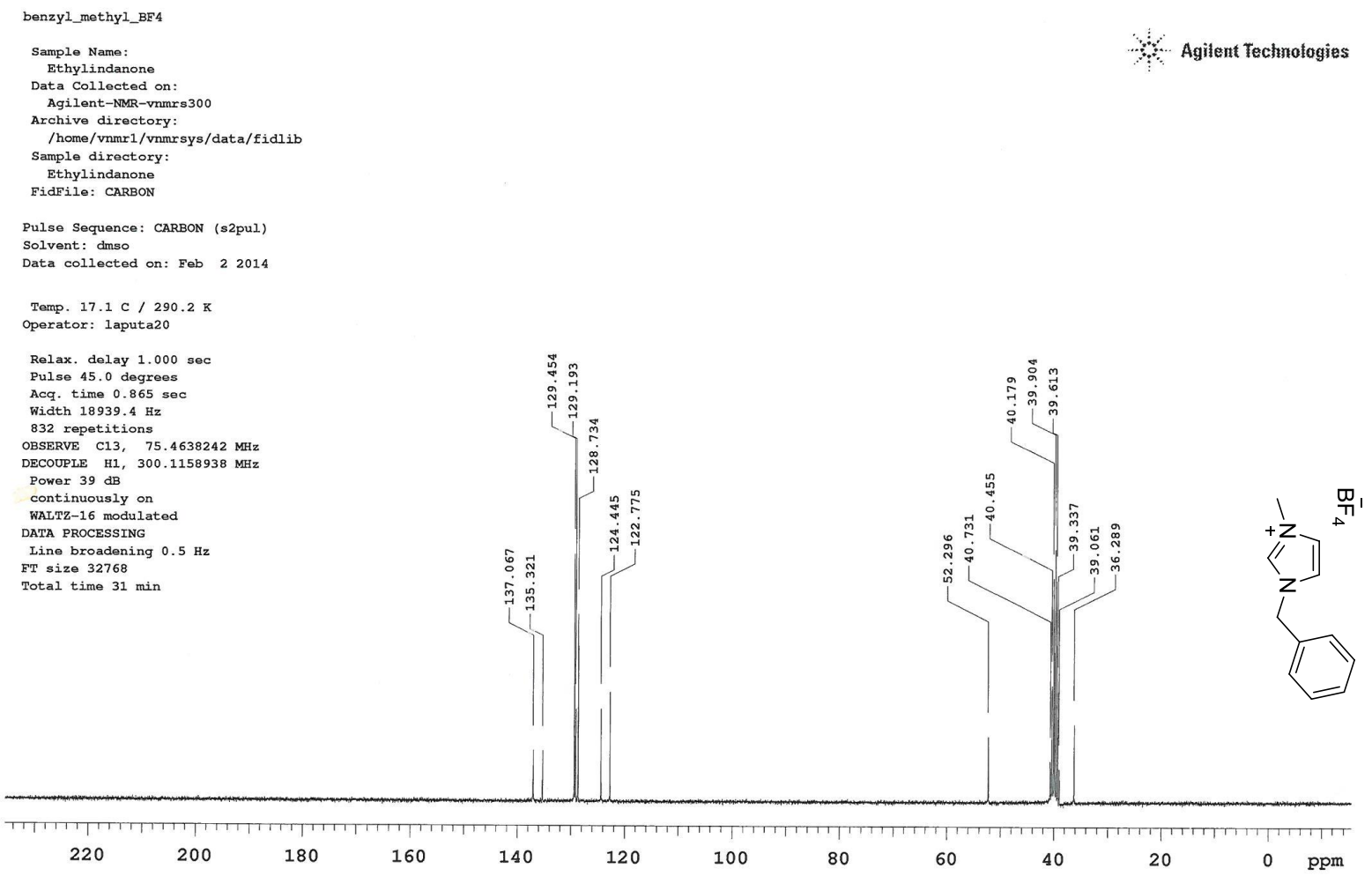
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
3120 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 49 min



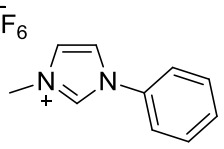
1-benzyl-3-methylimidazolium tetrafluoroborate

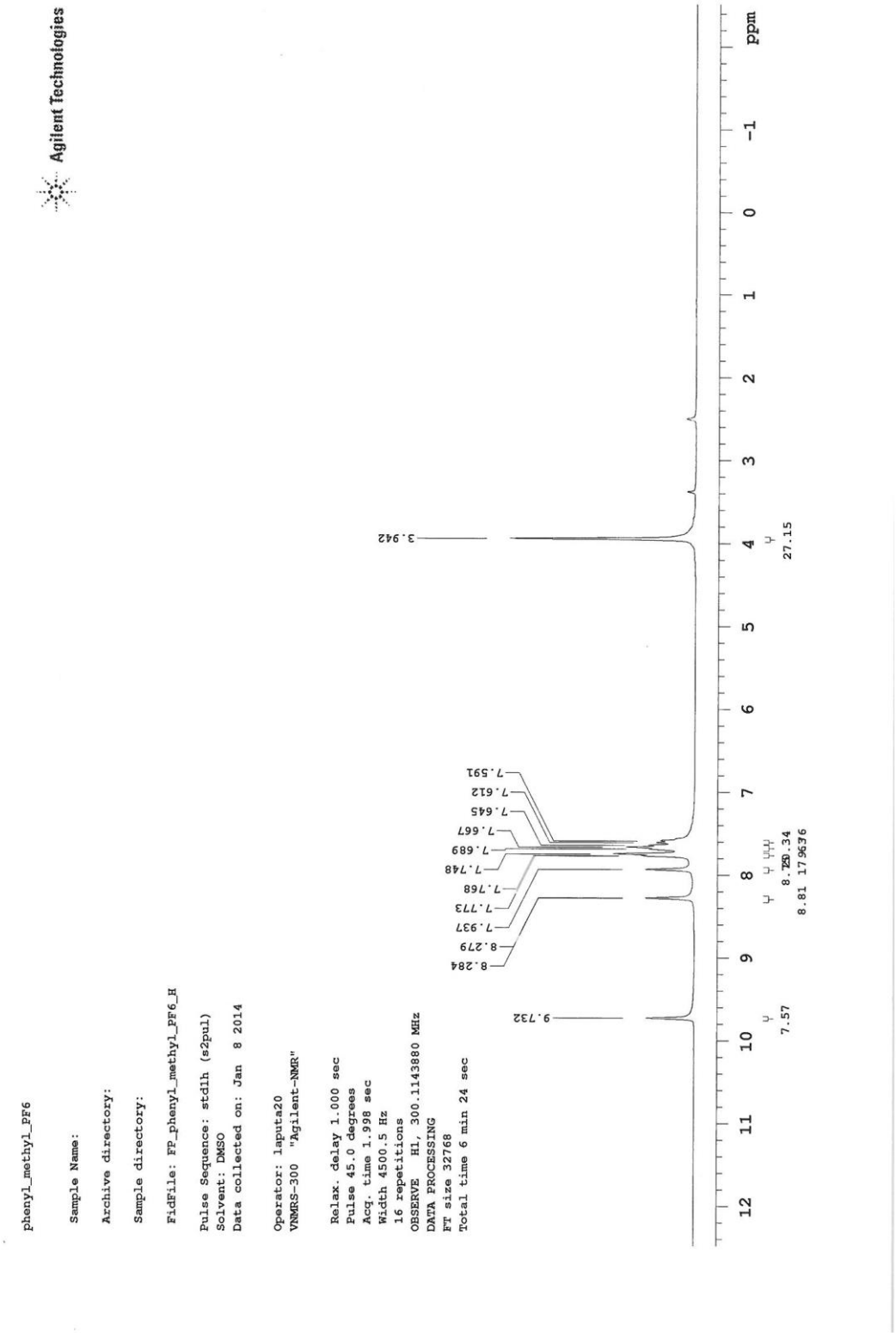


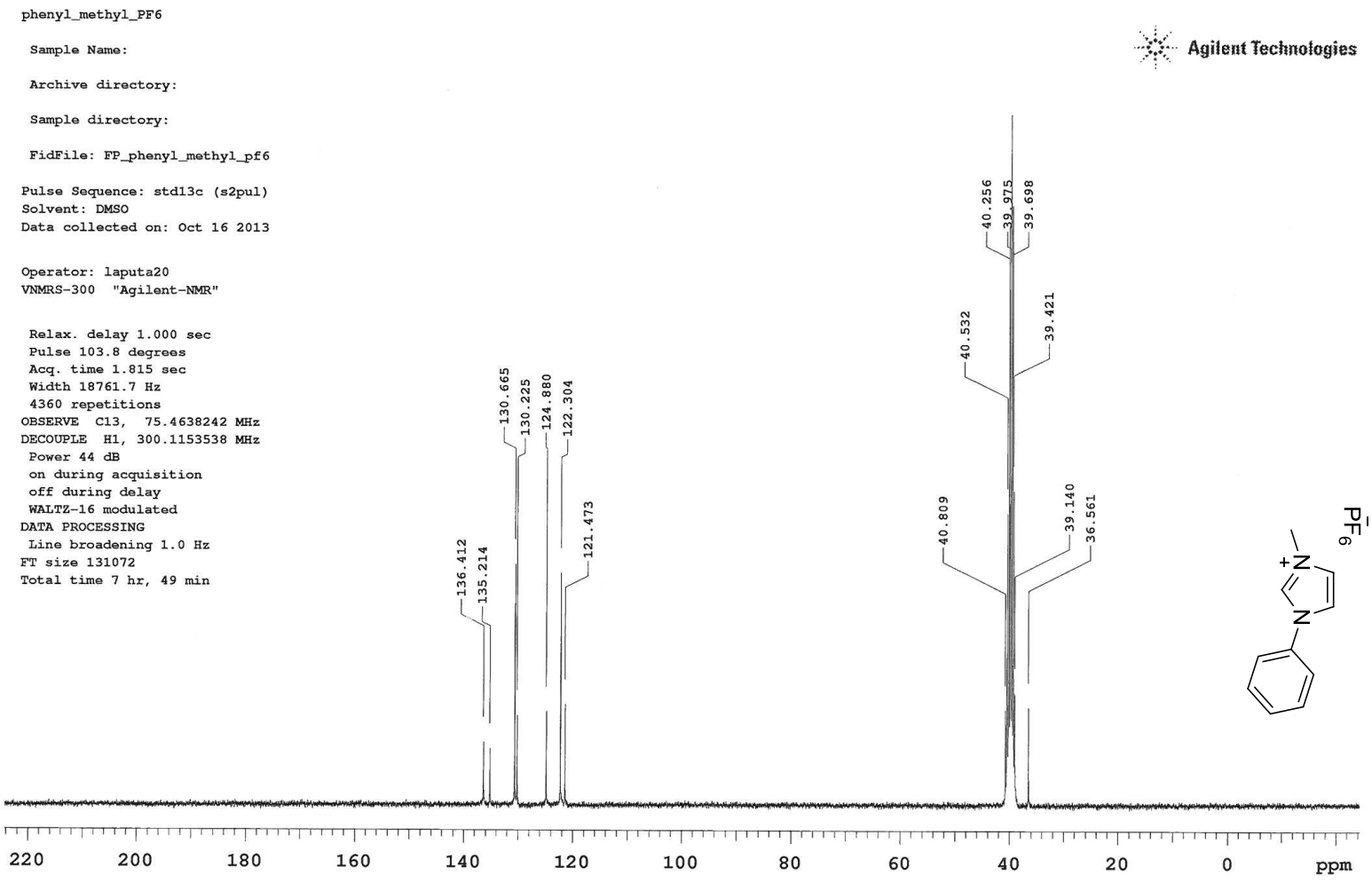



benzyl_methyl_BF4
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 2 2014
Temp. 17.1 C / 290.2 K
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
832 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
137.067
135.321
129.454
129.193
128.734
124.445
122.775
52.296
40.731
40.455
40.179
39.904
39.613
39.337
39.061
36.289
220
200
180
160
140
120
100
80
60
40
20
0
ppm

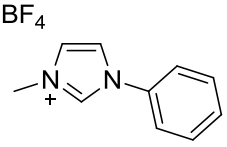
3-methyl-1-phenylimidazolium hexafluorophosphate

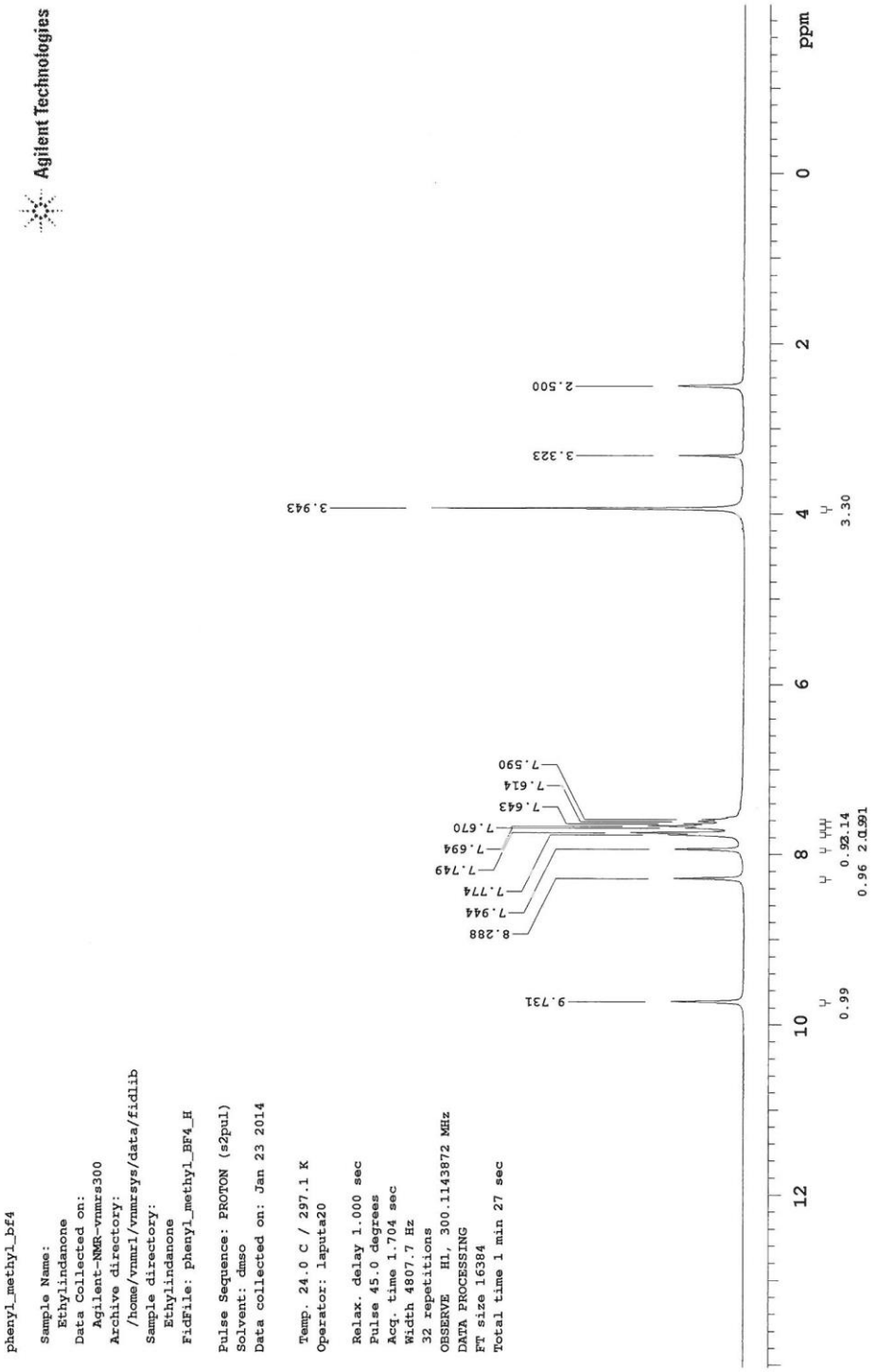





phenyl_methyl_PF6
Sample Name:
Archive directory:
Sample directory:
FidFile: FP_phenyl_methyl_pf6
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 16 2013
Operator: laputa20
VNMRS-300 "Agilent-NMR"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
4360 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 49 min
Agilent Technologies
136.412
135.214
130.665
130.225
124.880
122.304
121.473
40.809
40.532
40.256
39.975
39.698
39.421
39.140
36.561
220
200
180
160
140
120
100
80
60
40
20
0
ppm
PF6

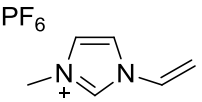
3-methyl-1-phenylimidazolium tetrafluoroborate

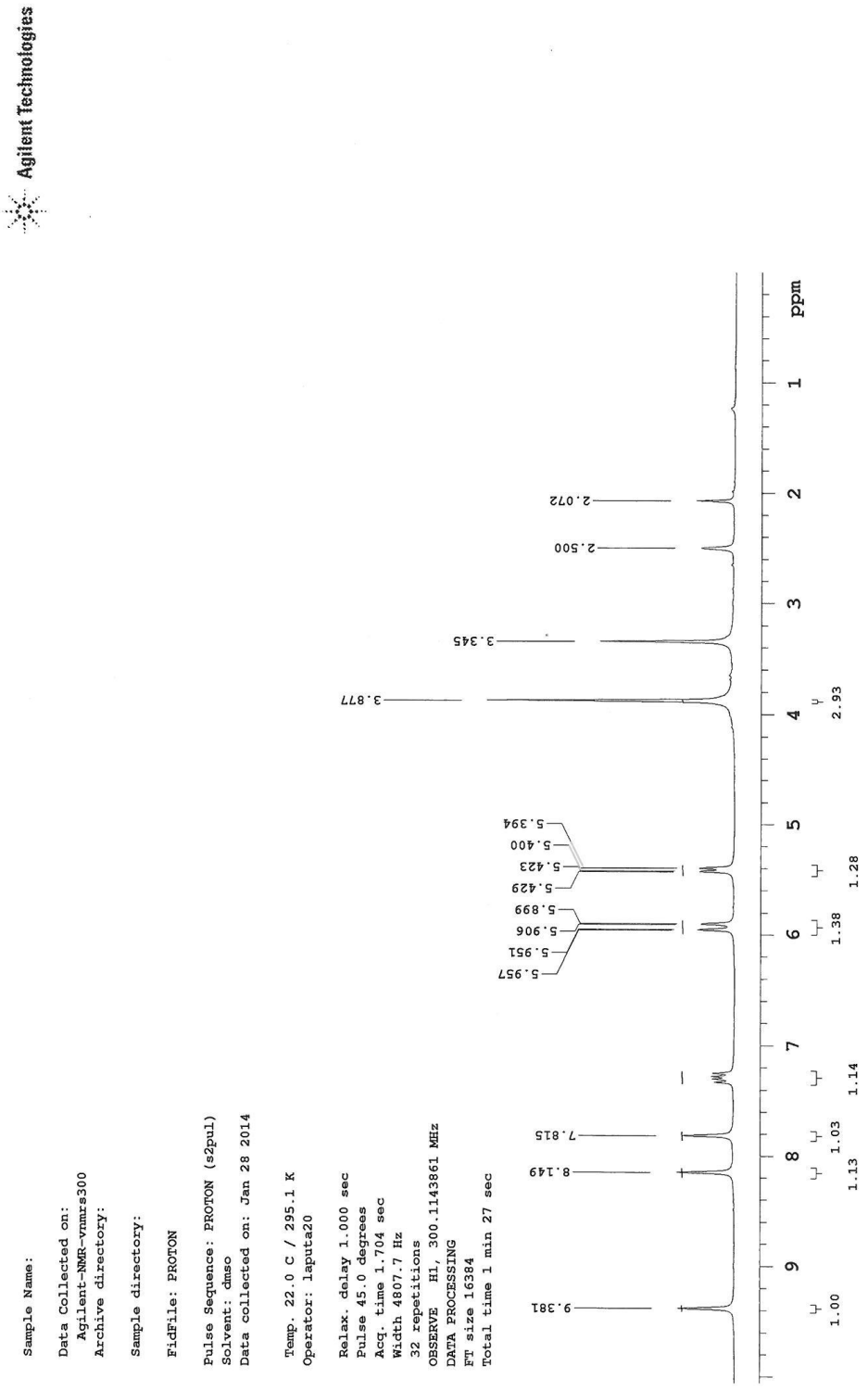


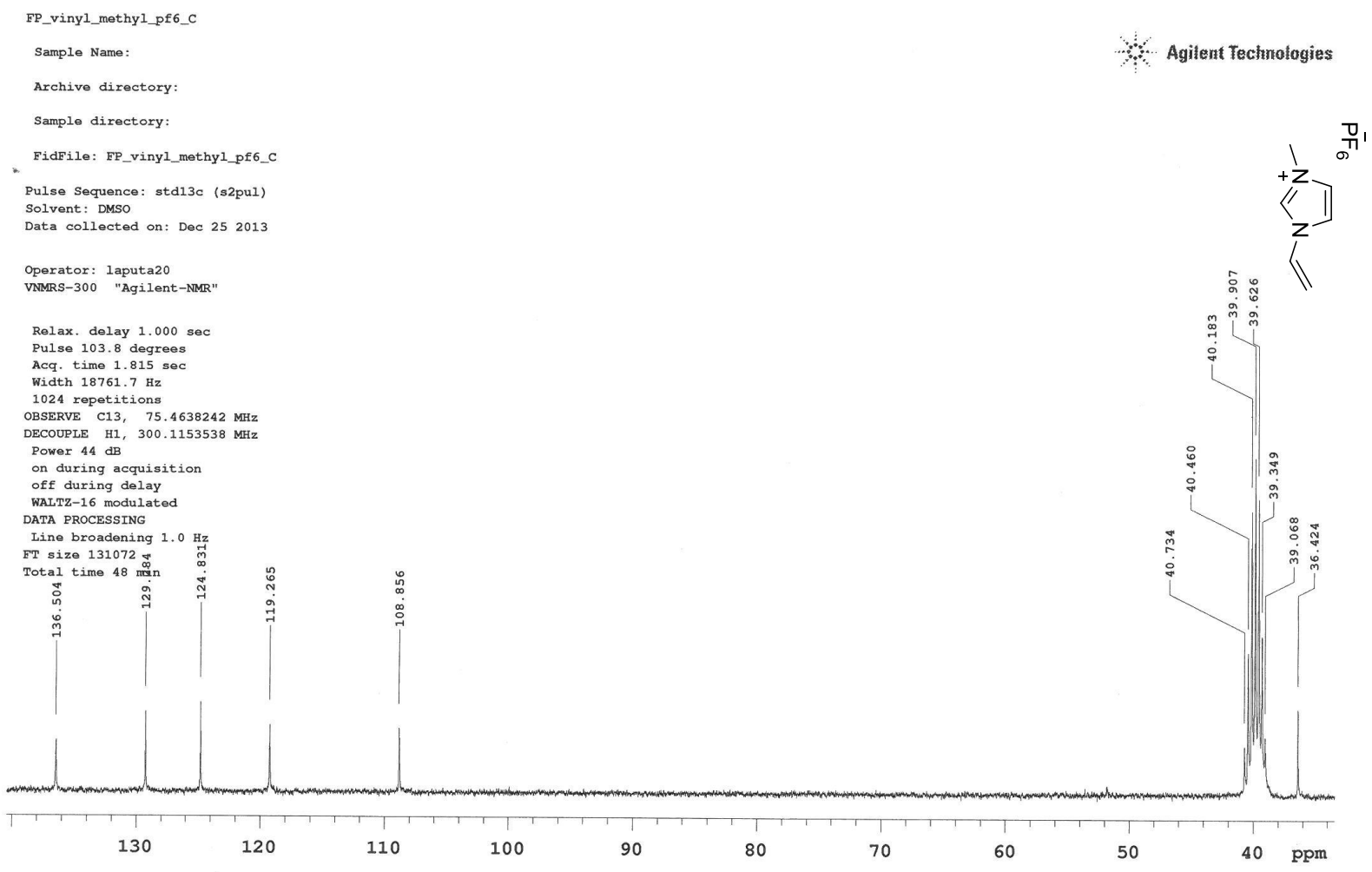


phenyl_methyl_BF4
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: phenyl_methyl_BF4_C
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 2 2014
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
BF4
N
N
36.564
39.061
39.337
39.613
39.904
40.179
40.455
40.731
121.412
122.254
124.874
130.204
130.679
135.198
136.378
220
200
180
160
140
120
100
80
60
40
20
0
ppm

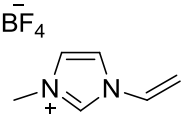
3-methyl-1-vinylimidazolium hexafluorophosphate

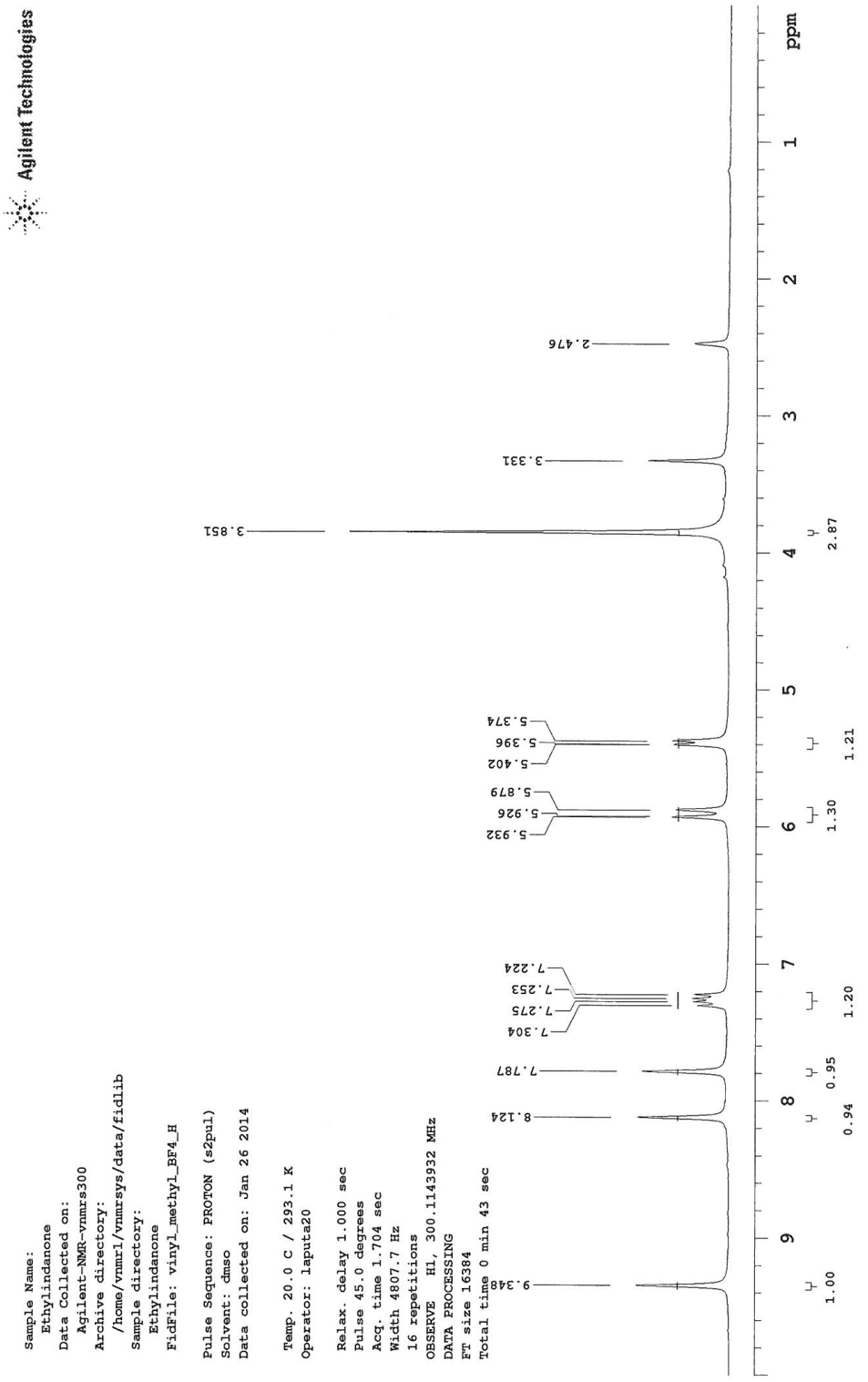


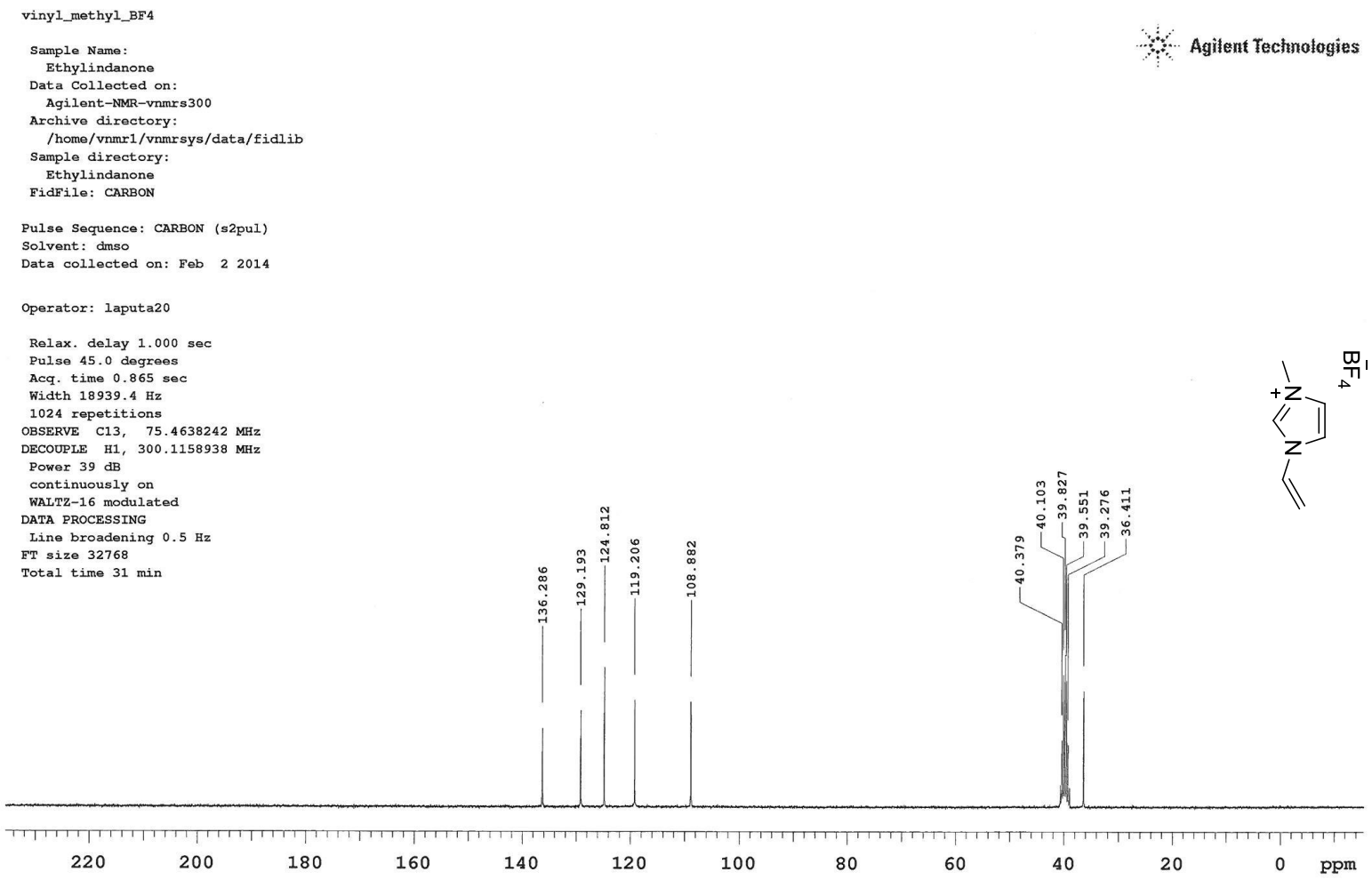



FP_vinyl_methyl_pf6_C
Sample Name:
Archive directory:
Sample directory:
FidFile: FP_vinyl_methyl_pf6_C
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Dec 25 2013
Operator: laputa20
VNMRS-300 "Agilent-NMR"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min
Agilent Technologies
136.504
129.184
124.831
119.265
108.856
40.734
40.460
40.183
39.907
39.626
39.349
39.068
36.424
130
120
110
100
90
80
70
60
50
40
ppm

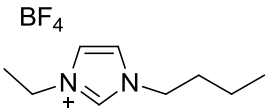
3-methyl-1-vinylimidazolium tetrafluoroborate

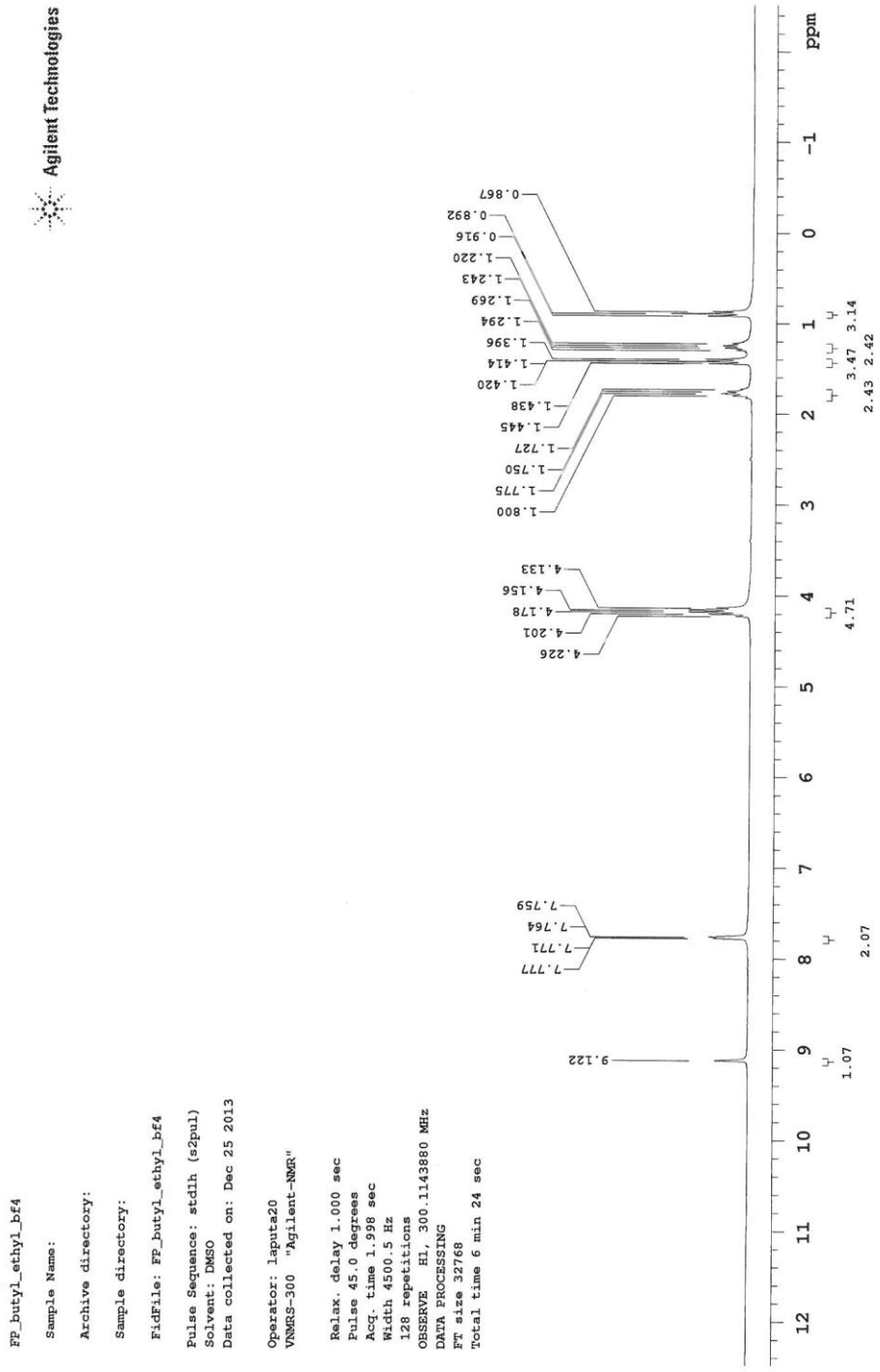


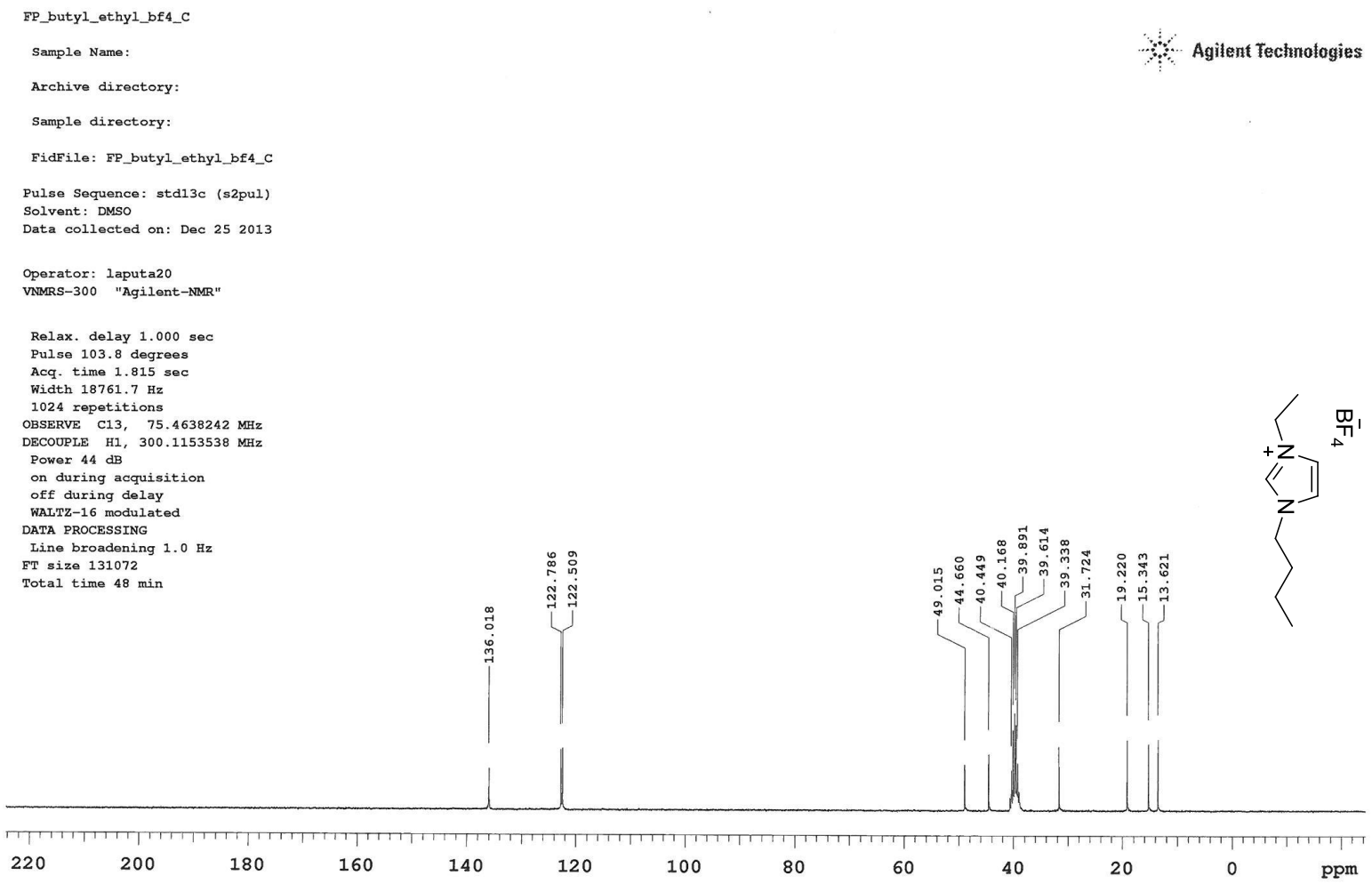



vinyl_methyl_BF4
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 2 2014
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
136.286
129.193
124.812
119.206
108.882
40.379
40.103
39.827
39.551
39.276
36.411
BF4
220
200
180
160
140
120
100
80
60
40
20
0
ppm

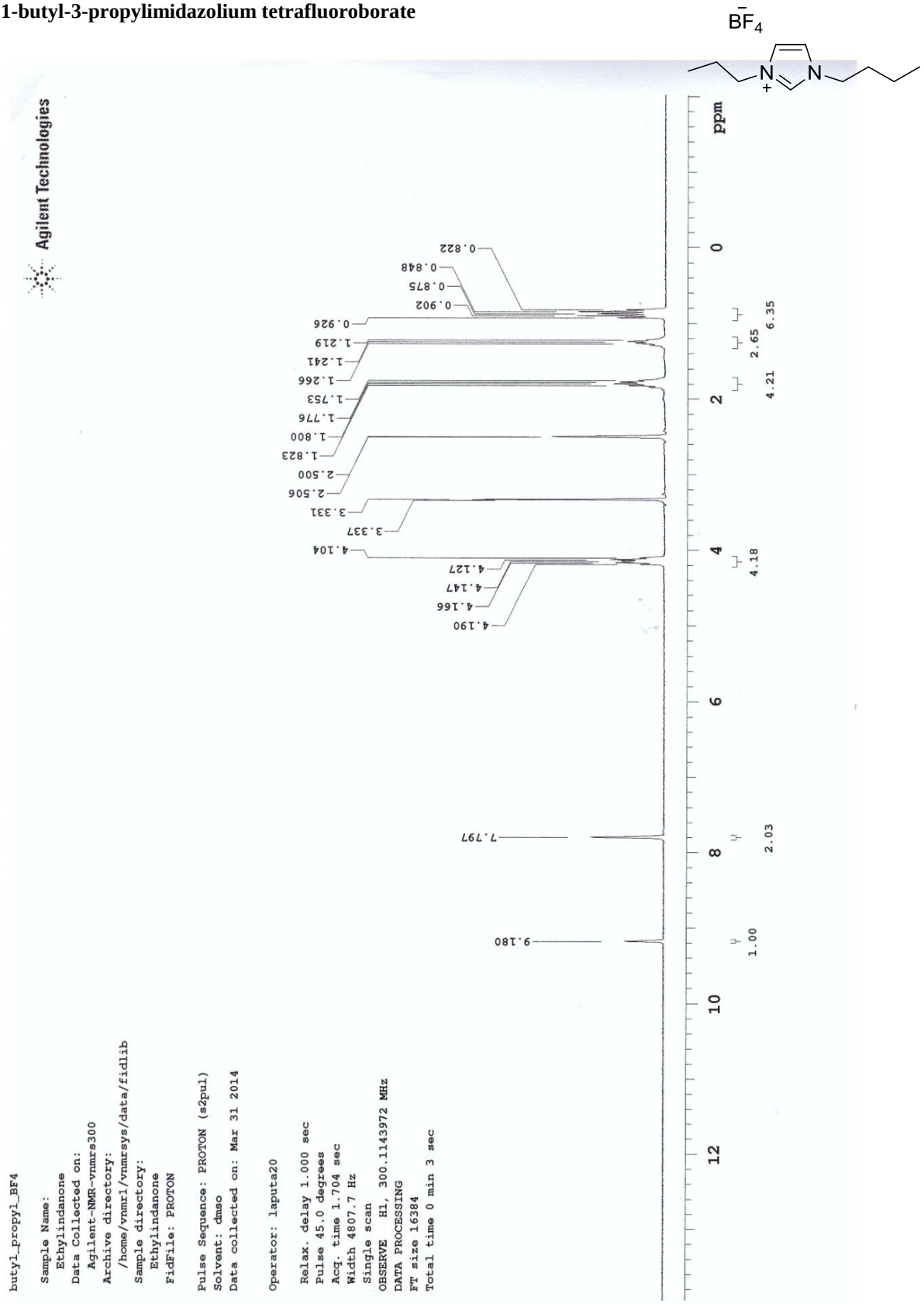
1-butyl-3-ethyllimidazolium tetrafluoroborate

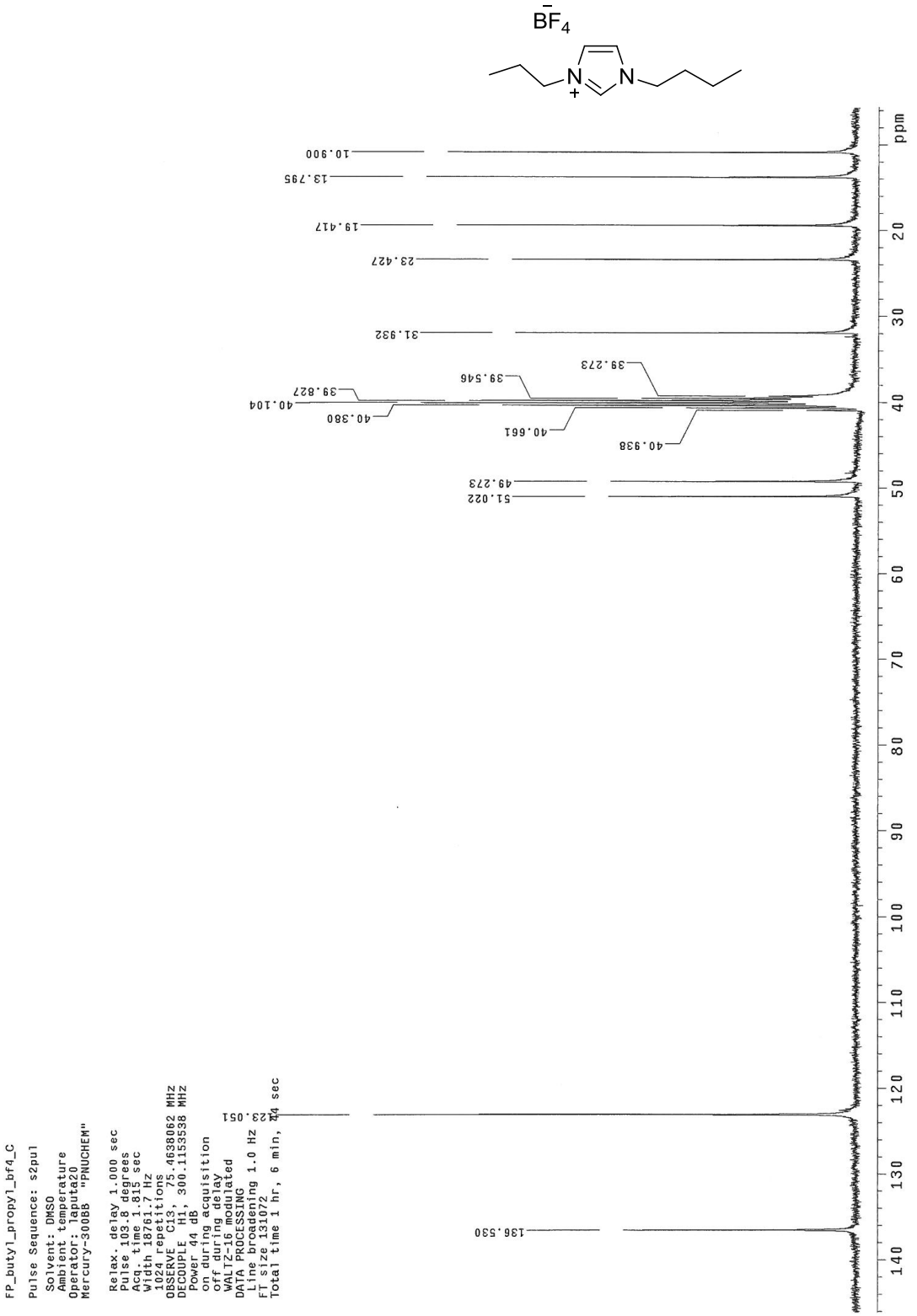




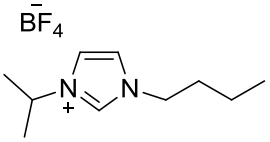

FP_butyl_ethyl_bf4_C
Sample Name:
Archive directory:
Sample directory:
FidFile: FP_butyl_ethyl_bf4_C
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Dec 25 2013
Operator: laputa20
VNMRS-300 "Agilent-NMR"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min
Agilent Technologies
136.018
122.786
122.509
49.015
44.660
40.449
40.168
39.891
39.614
39.338
31.724
19.220
15.343
13.621
220
200
180
160
140
120
100
80
60
40
20
0
ppm
BF4
N
N

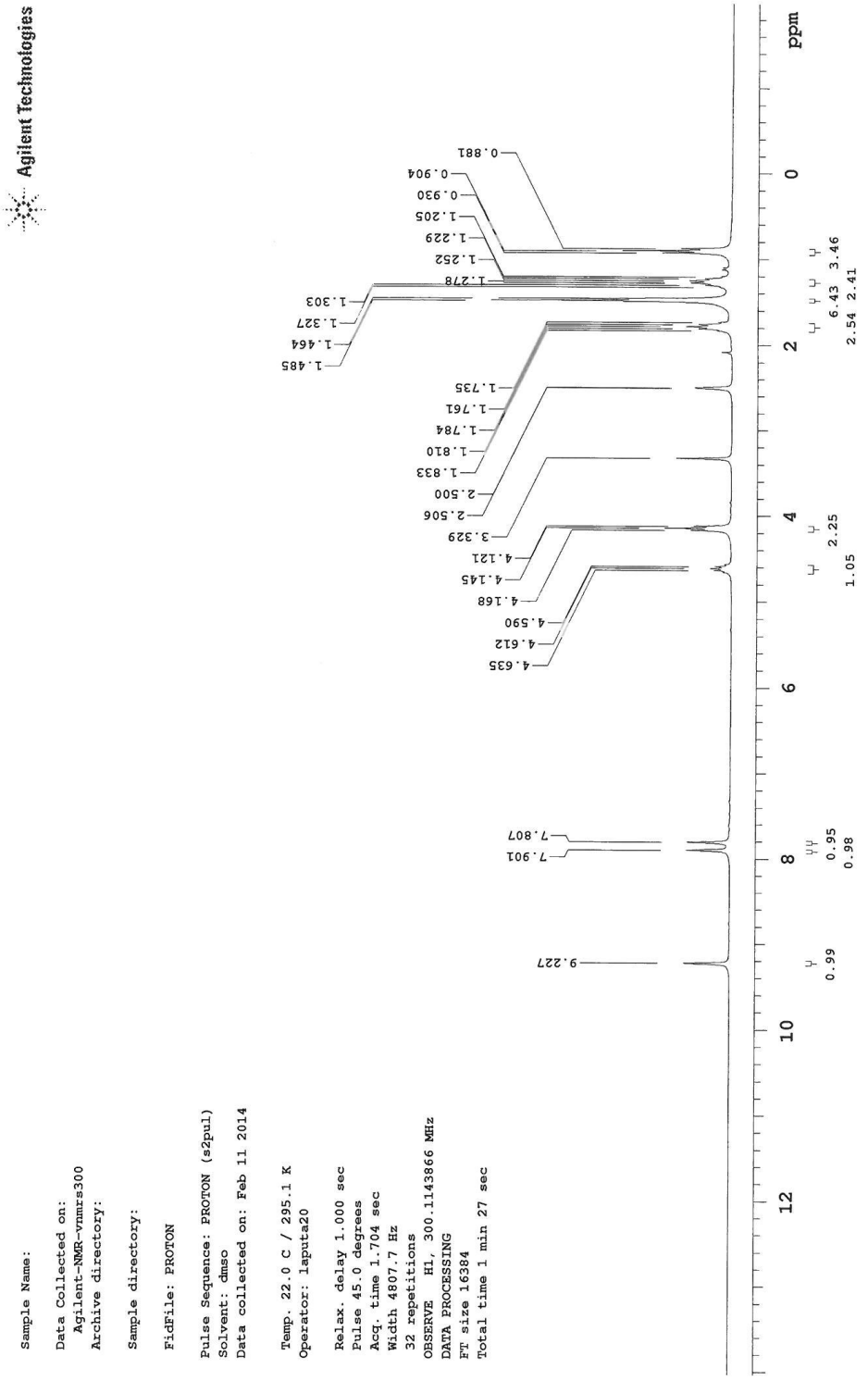
1-butyl-3-propylimidazolium tetrafluoroborate

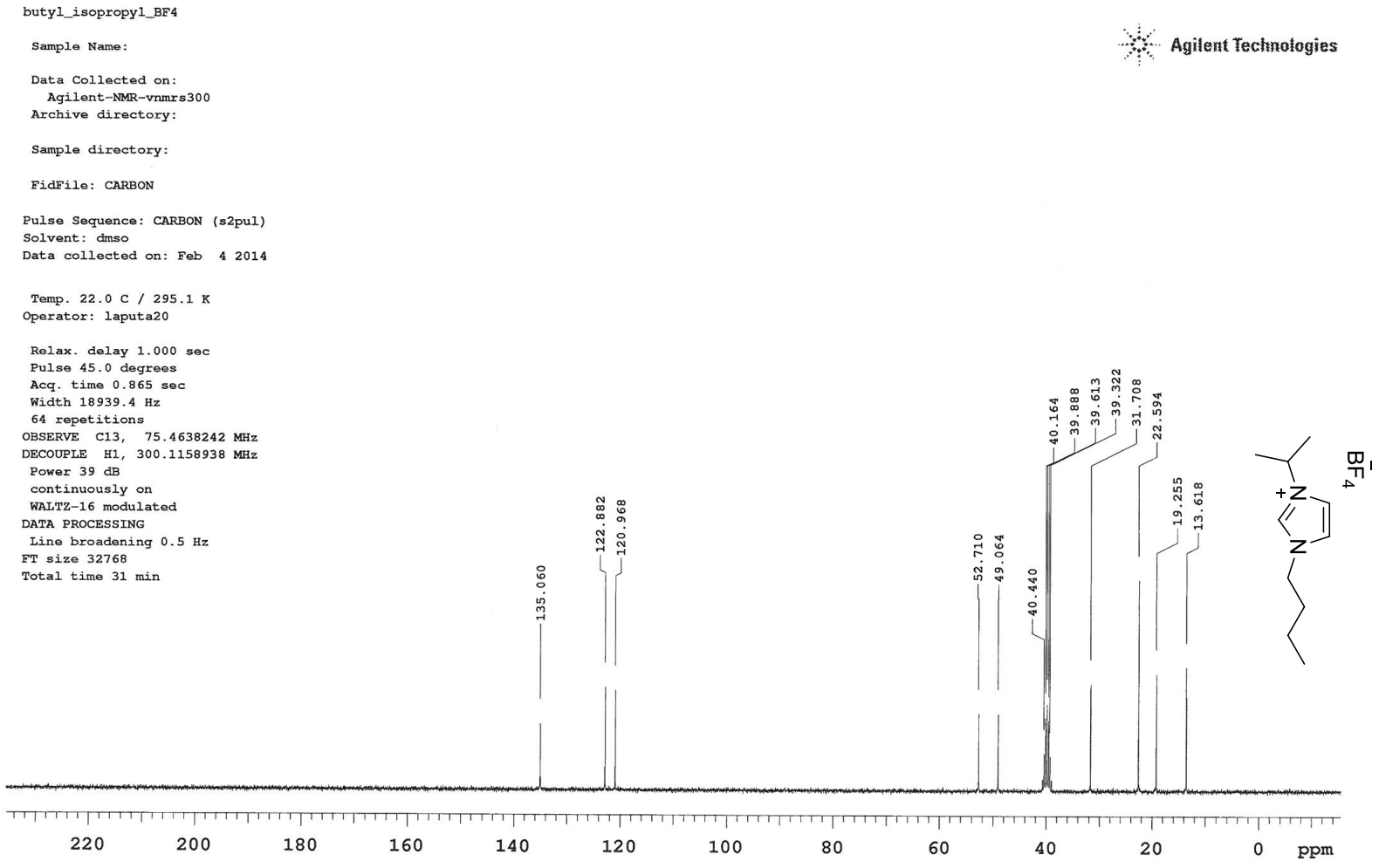



BF_4^-
N
N
+
FP_butyl_propyl_bf4_C
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
Operator: laputa20
Mercury-300BB "PNUCHEM"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4638062 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 6 min, 44 sec
136.530
123.051
49.273
51.022
40.938
40.661
40.380
40.104
39.827
39.546
39.273
31.932
23.427
19.417
13.795
10.900
ppm

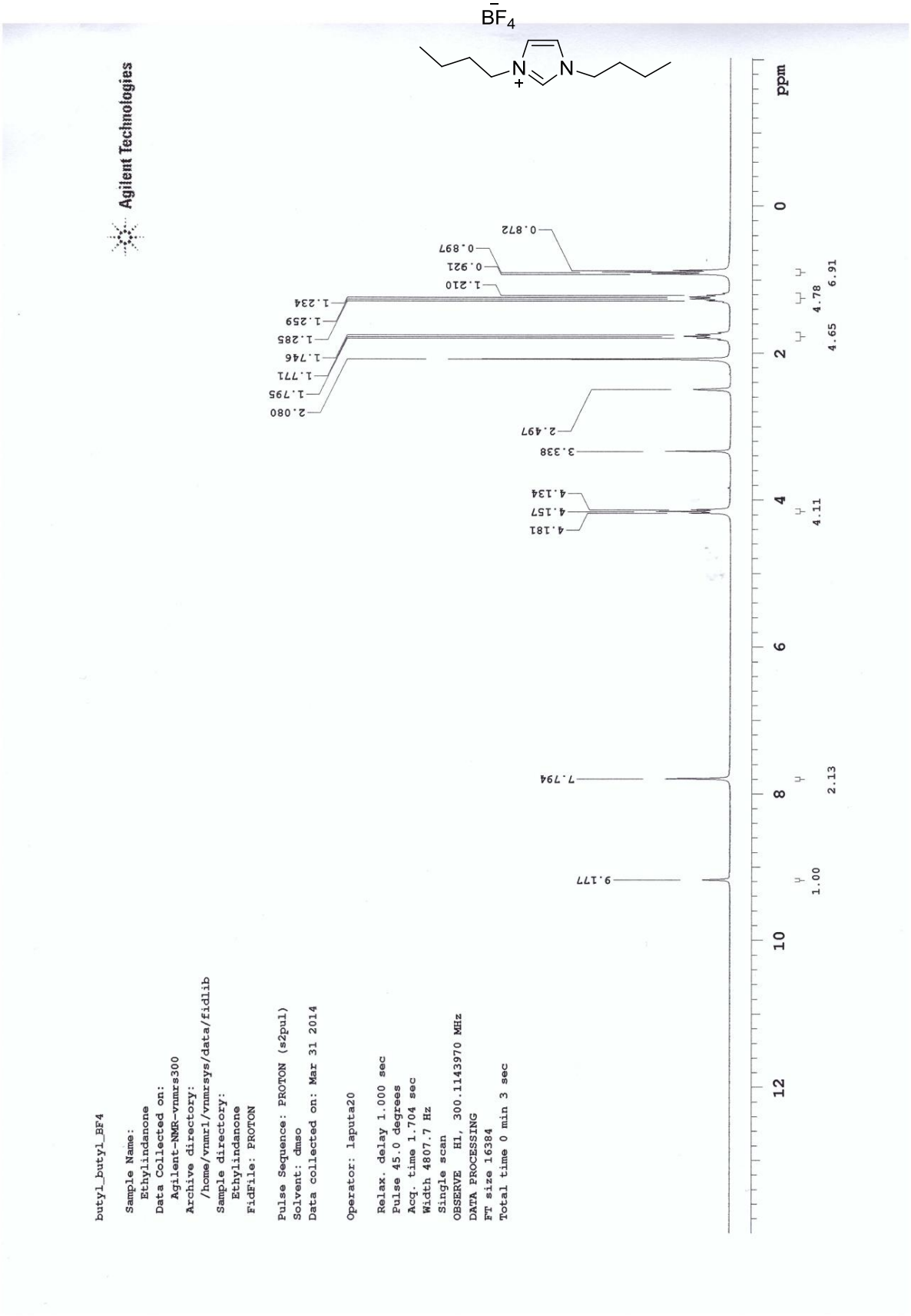
1-butyl-3-isopropylimidazolium tetrafluoroborate

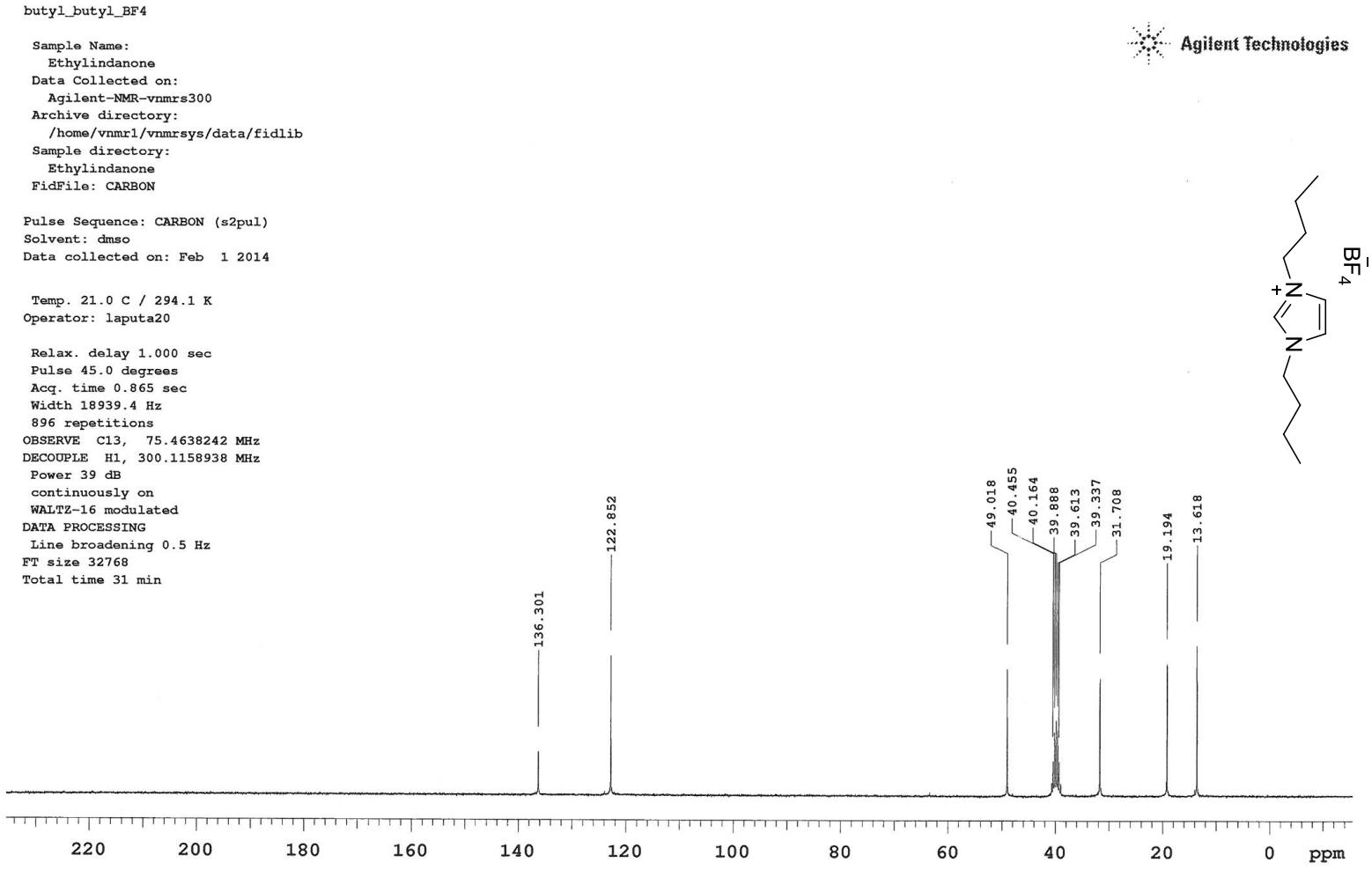




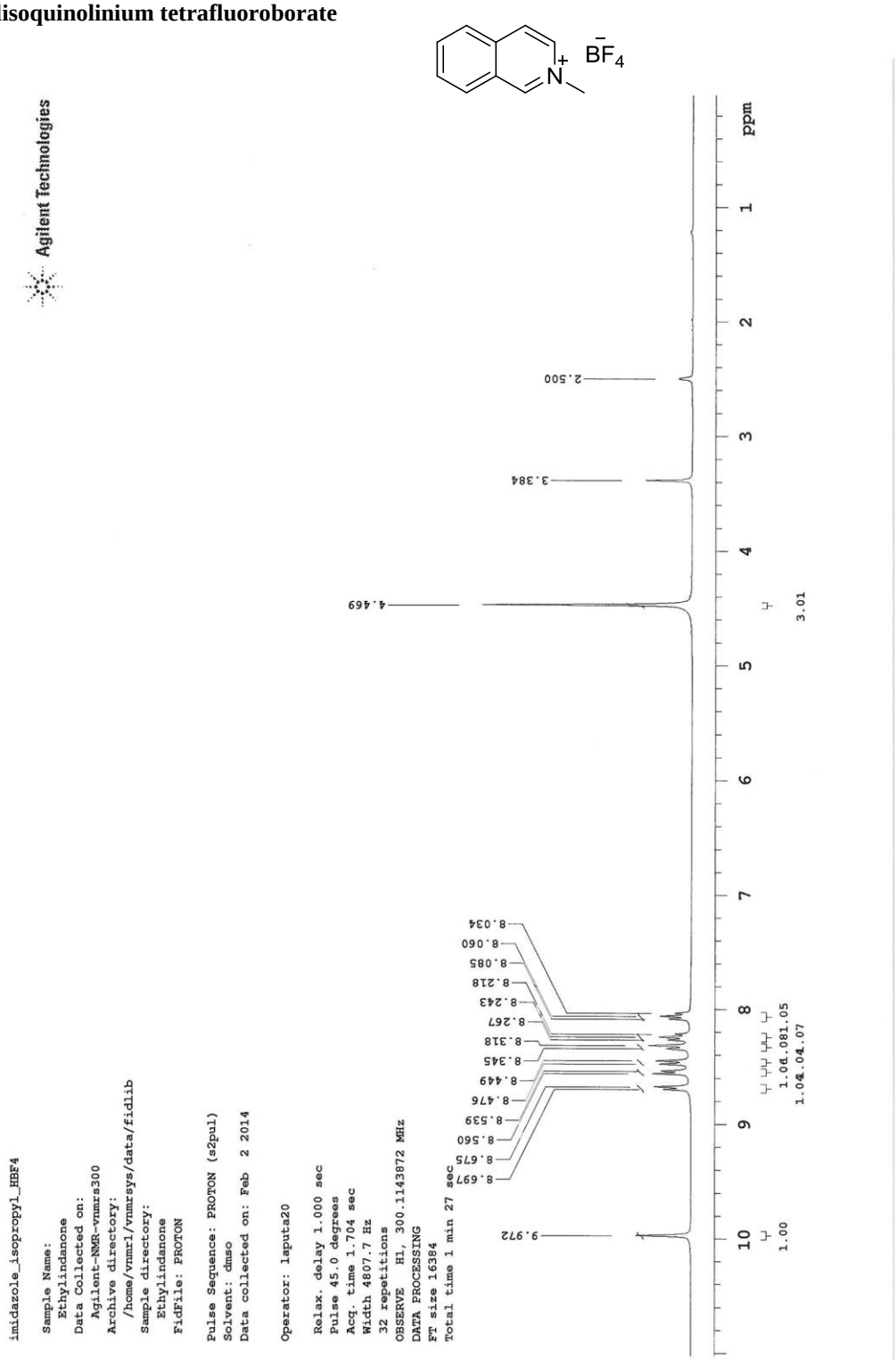

butyl_isopropyl_BF4
Sample Name:
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 4 2014
Temp. 22.0 C / 295.1 K
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
64 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
135.060
122.882
120.968
52.710
49.064
40.440
40.164
39.888
39.613
39.322
31.708
22.594
19.255
13.618
BF4
220
200
180
160
140
120
100
80
60
40
20
0
ppm

1,3-dibutylimidazolium tetrafluoroborate




butyl_butyl_BF4
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 1 2014
Temp. 21.0 C / 294.1 K
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
896 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
136.301
122.852
49.018
40.455
40.164
39.888
39.613
39.337
31.708
19.194
13.618
220
200
180
160
140
120
100
80
60
40
20
0
ppm

N-methylisoquinolinium tetrafluoroborate



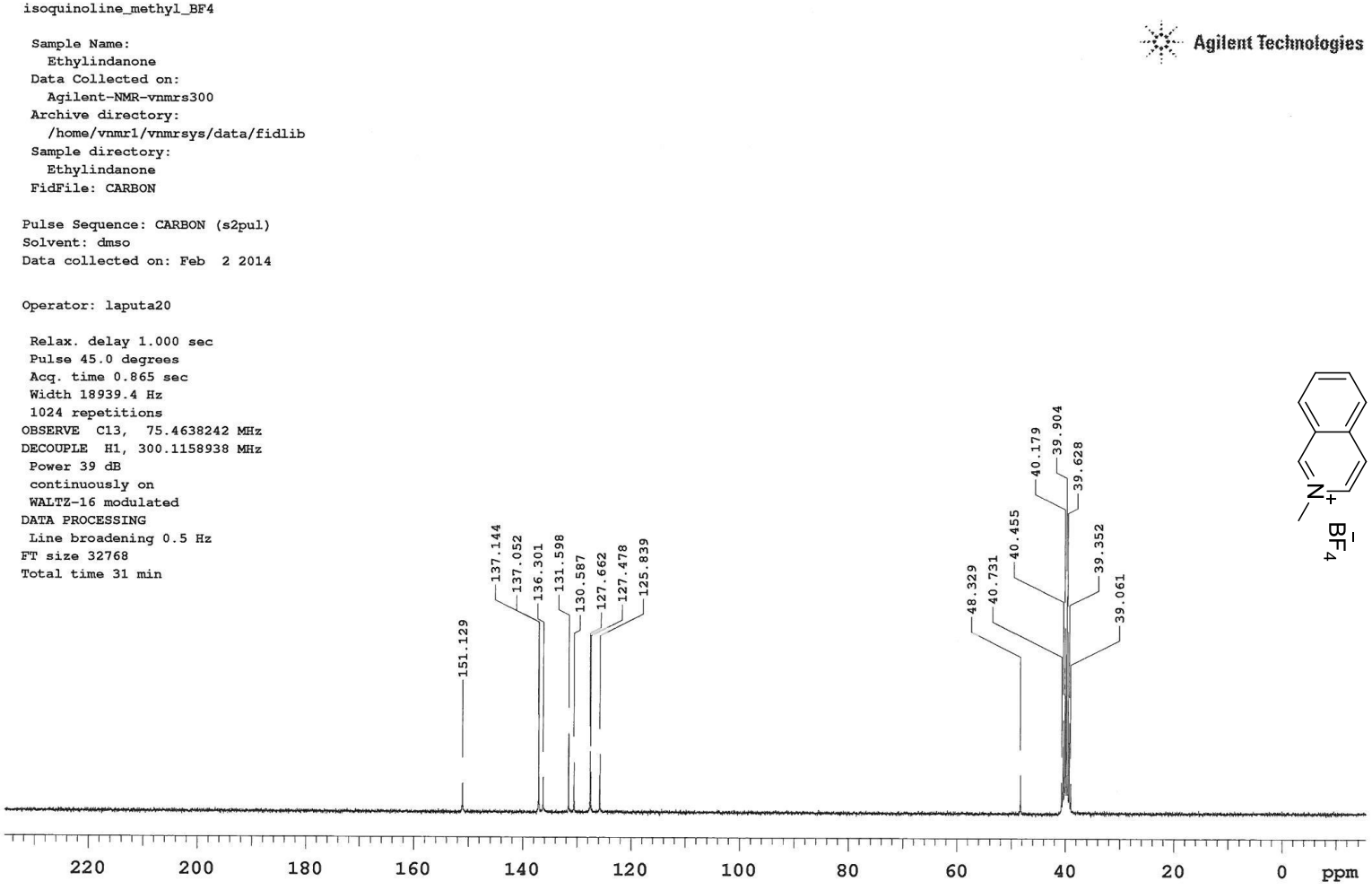
isoquinoline_methyl_BF4

Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: CARBON

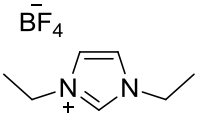
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 2 2014

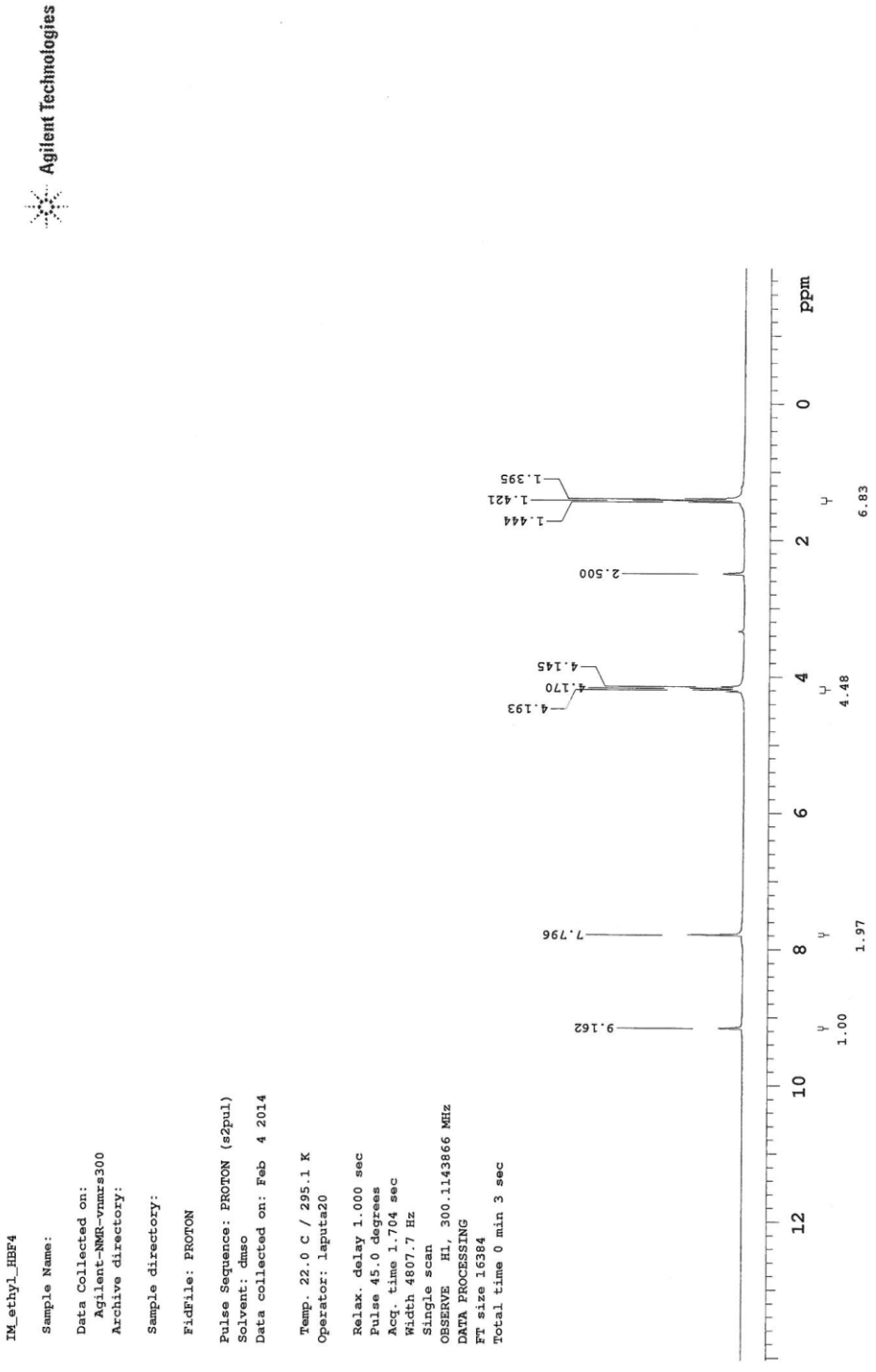
Operator: laputa20

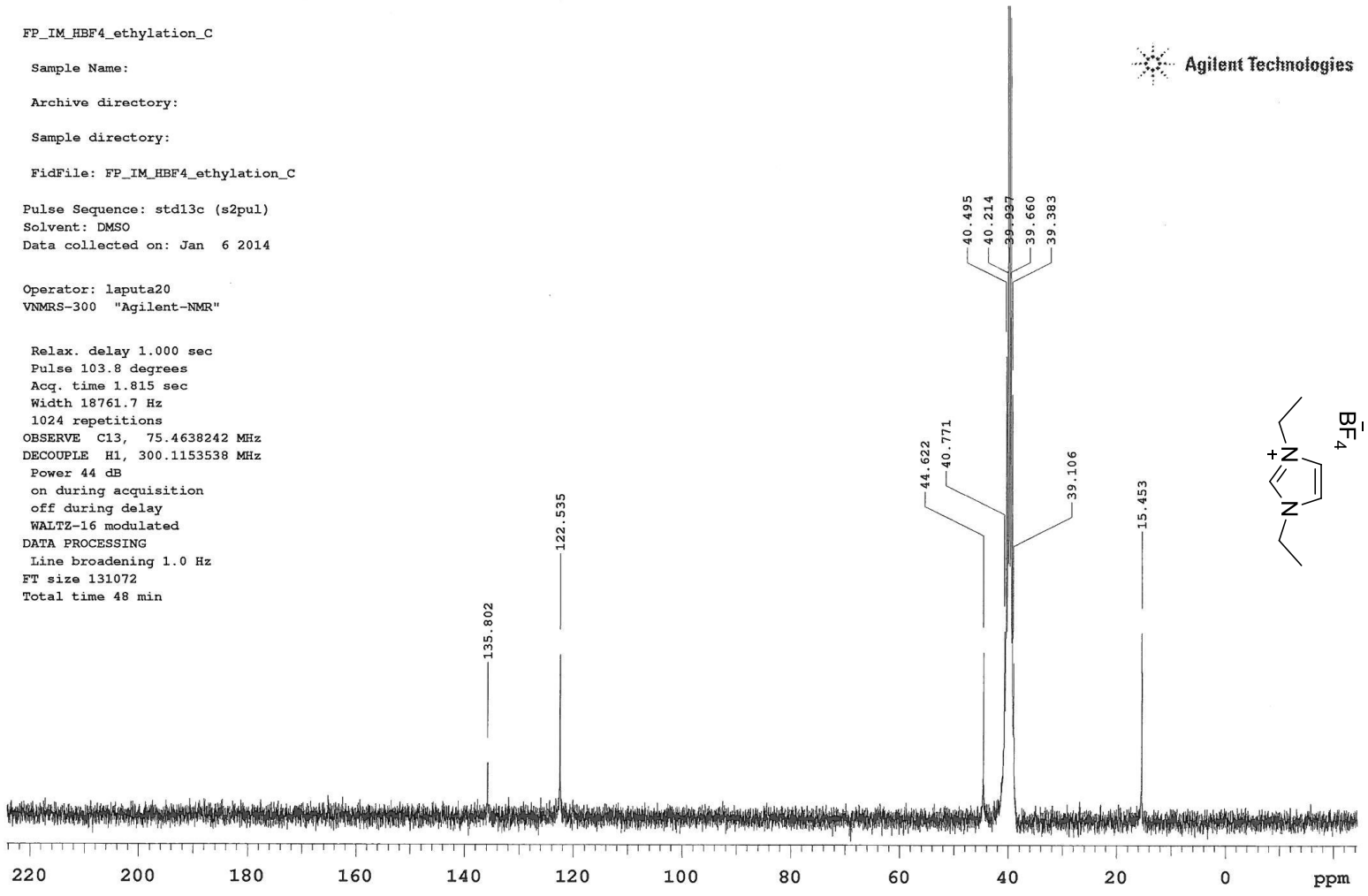
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min



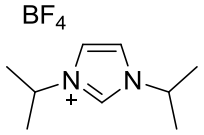
1,3-diethylimidazolium tetrafluoroborate

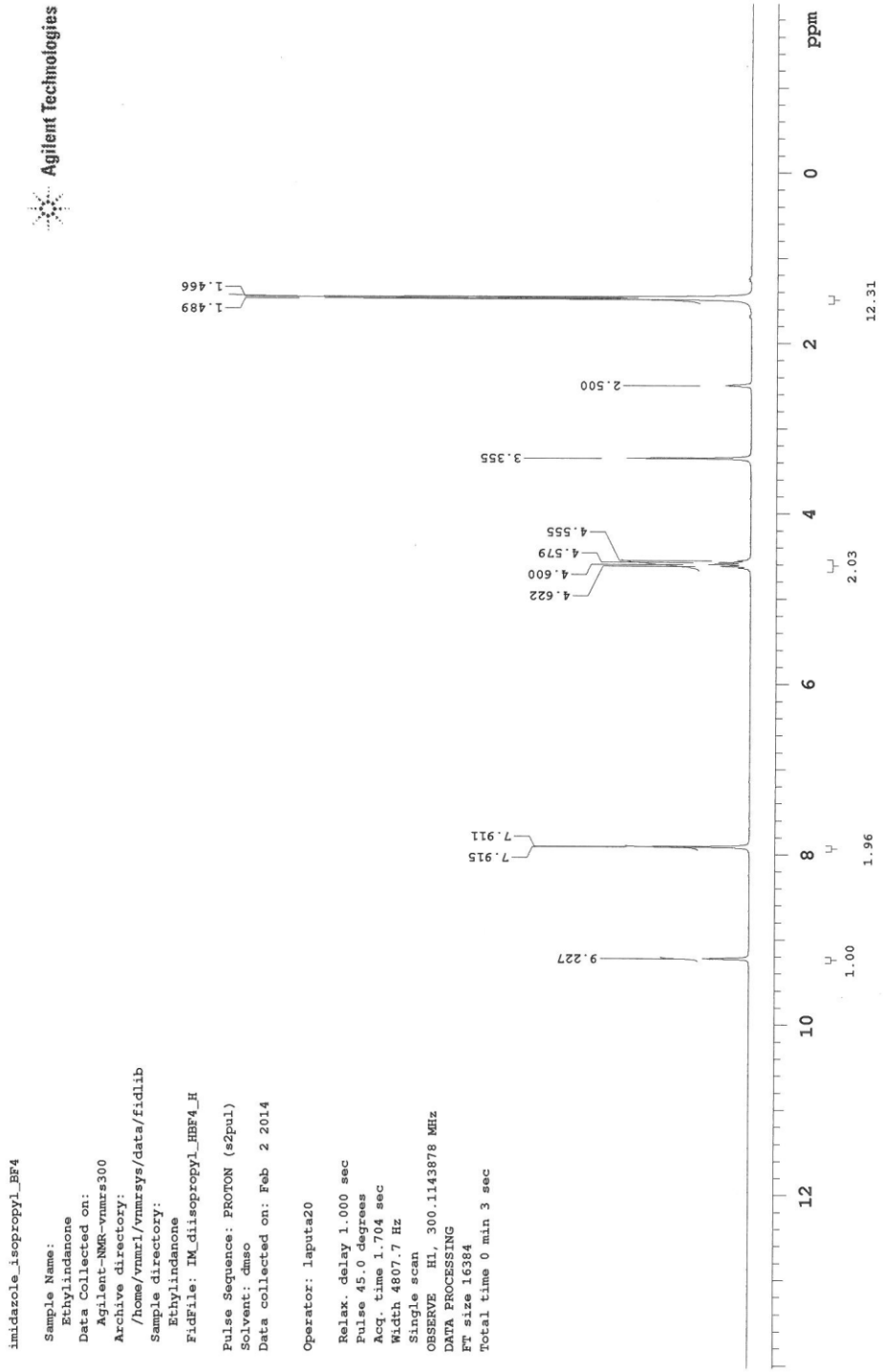


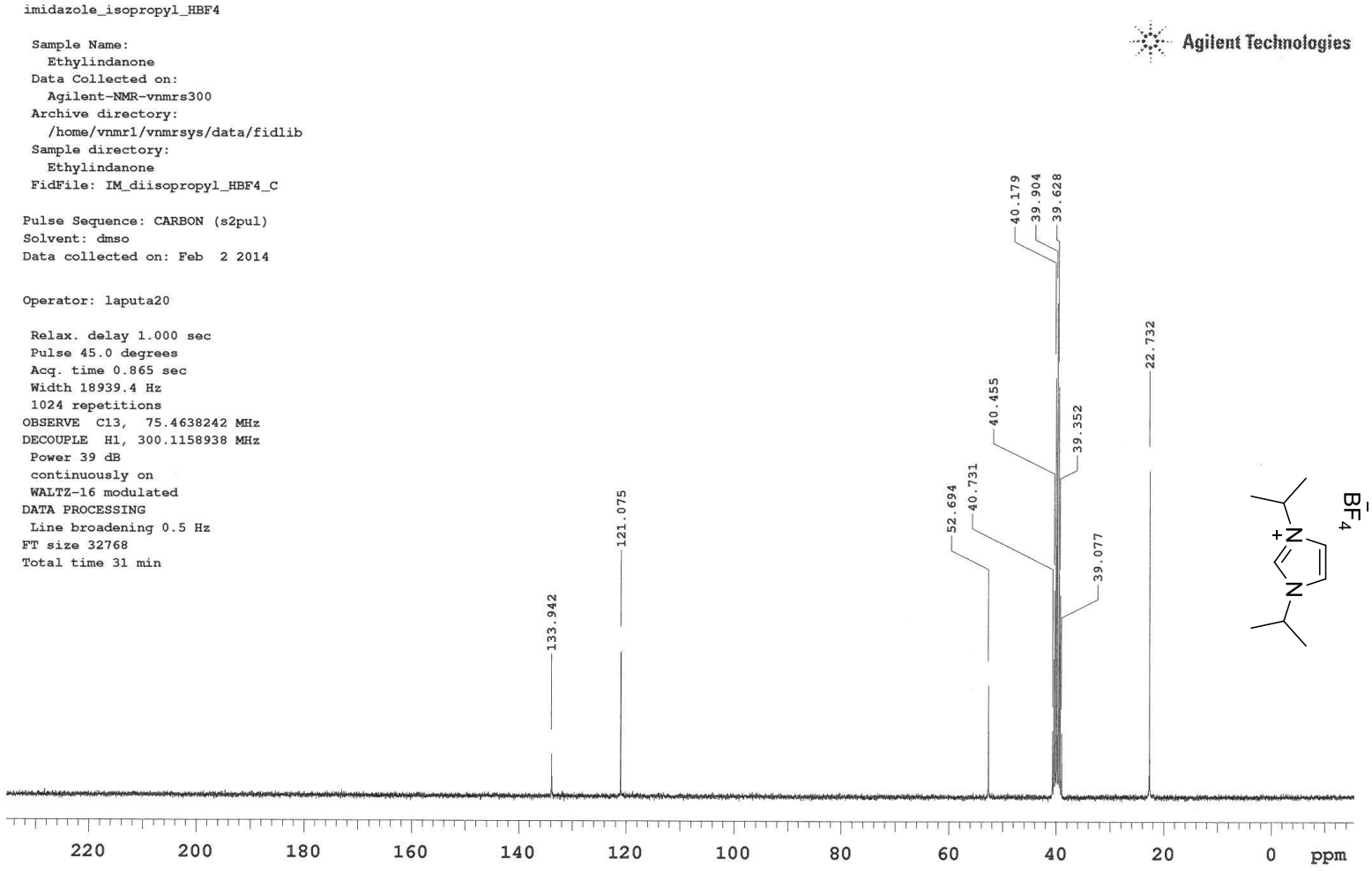



FP_IM_HBF4_ethylation_C
Sample Name:
Archive directory:
Sample directory:
FidFile: FP_IM_HBF4_ethylation_C
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Jan 6 2014
Operator: laputa20
VNMRS-300 "Agilent-NMR"
Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min
Agilent Technologies
135.802
122.535
44.622
40.771
40.495
40.214
39.937
39.660
39.383
39.106
15.453
220
200
180
160
140
120
100
80
60
40
20
0
ppm

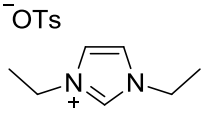
1,3-diisopropylimidazolium tetrafluoroborate

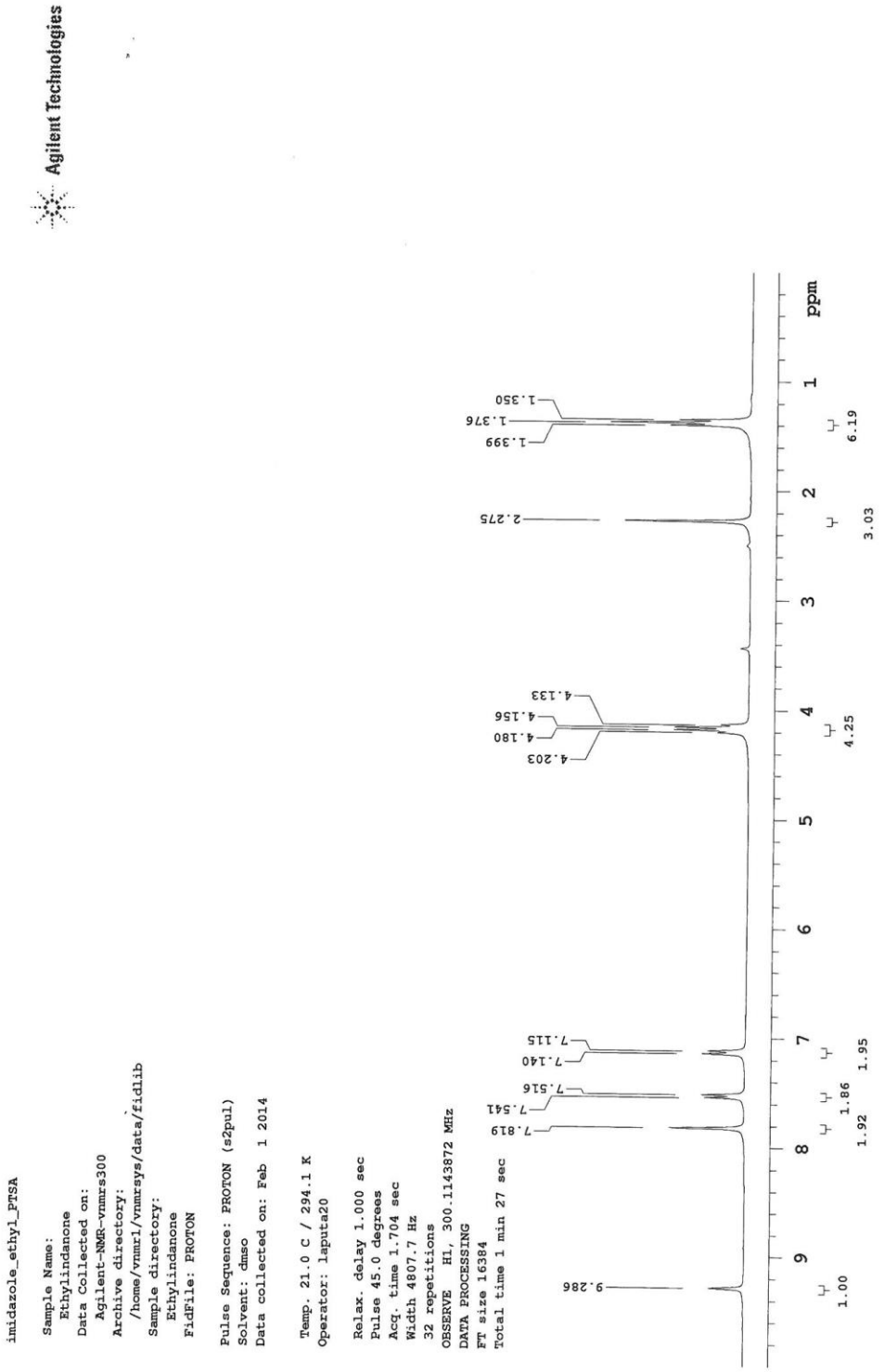


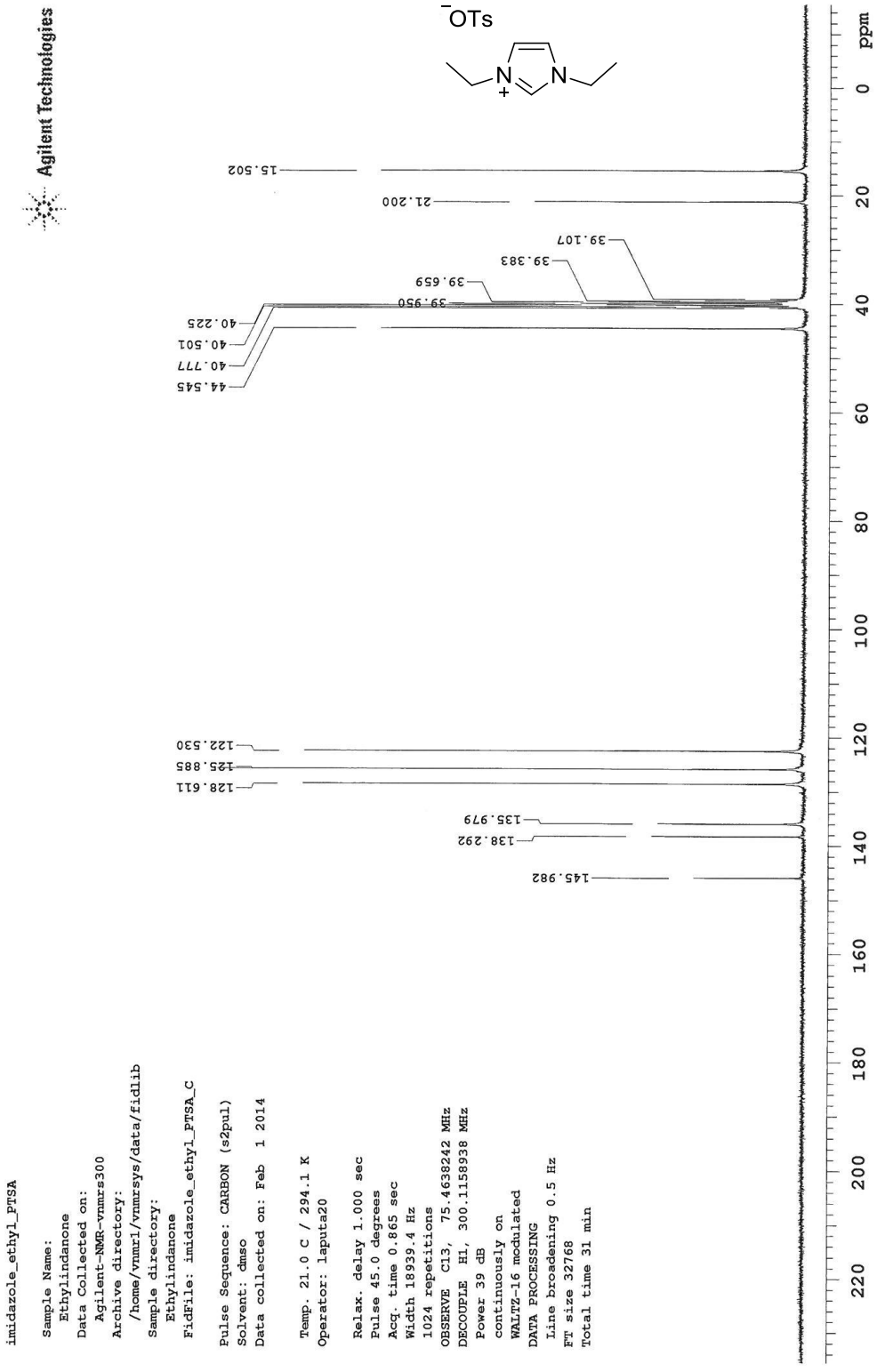



imidazole_isopropyl_HBF4
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: IM_diisopropyl_HBF4_C
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 2 2014
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
133.942
121.075
52.694
40.731
40.455
40.179
39.904
39.628
39.352
39.077
22.732
220
200
180
160
140
120
100
80
60
40
20
0
ppm

1,3-diethylimidazolium 4-methylbenzenesulfonate

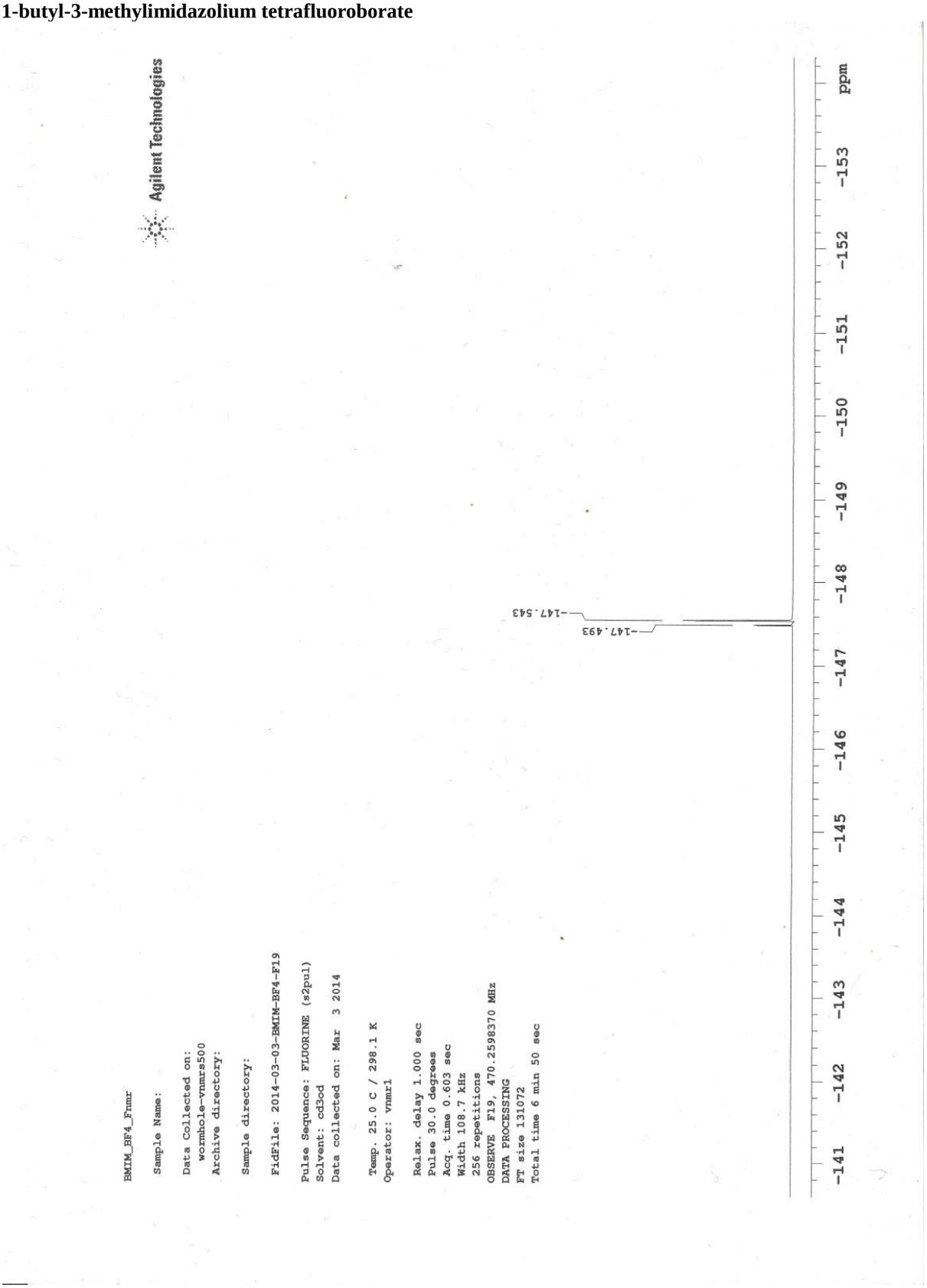





imidazole_ethyl_PTSA
Sample Name:
Ethylindanone
Data Collected on:
Agilent-NMR-vnmrs300
Archive directory:
/home/vnmr1/vnmrsys/data/fidlib
Sample directory:
Ethylindanone
FidFile: imidazole_ethyl_PTSA_C
Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 1 2014
Temp. 21.0 C / 294.1 K
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Agilent Technologies
OTs
15.502
21.200
39.107
39.383
39.659
39.950
40.225
40.501
40.777
44.545
122.530
125.885
128.611
135.979
138.292
145.982
220
200
180
160
140
120
100
80
60
40
20
0
ppm

^{19}F NMR spectrum

1-butyl-3-methylimidazolium tetrafluoroborate



1-butyl-3-methylimidazolium hexafluorophosphate