Supporting Information for:

Reverse fluorous solid phase extraction: A new technique for rapid separation of fluorous compounds

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General: All melting points are uncorrected. Reagents were used as they were received from Aldrich. ^{1}H and ^{19}F NMR spectra were measured in $CDCl_3$ with TMS or $CHCl_3$ as the internal standard. 2-Methylallyltributyltin[1] and 2-phenylallyltributyltin[2] were prepared by known procedure. Fluorous benzoates **1a-c**[3] were prepared by condensation of the corresponding fluoroalcohols and benzoyl chloride. Fluorous alkenes **2a-b**[4], **2d**[4], **3a-b**[4], **3d**[4], **4a**[5], fluorous ester **7c**[6] and fluorous amides **9c**[7] were known compounds. The purities of **2a-d** and **3a-d** were determined by GC. The purities of **4a-d** were determined by HPLC.

Benzoic acid 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-nonadecafluorodecyl ester 1a.[3]
Colorless solid; mp 52.5-53.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.84 (t, 2H, J = 13.3 Hz), 7.50 (t, 2H, J = 7.9 Hz), 7.64 (t, 1H, J = 7.9 Hz), 8.08 (d, 2H, J = 7.2 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) –124.9 (2F), –121.9 (2F), –121.5 (2F), –120.6 (8F), –118.0 (2F), –79.5 (3F).

Benzoic acid 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluorooctyl ester 1b.[3]
Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.84 (t, 2H, J = 13.3 Hz), 7.50 (t, 2H, J = 7.6 Hz), 7.64 (t, 1H, J = 7.6 Hz), 8.08 (d, 2H, J = 7.3 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –121.9 (2F), –121.5 (2F), –120.7 (4F), –118.0 (2F), –79.6 (3F).

Benzoic acid 2,2,3,3,4,4,4-heptafluorobutyl ester 1c.[3]
Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.82 (t, 2H, J = 13.2 Hz), 7.49 (t, 2H, J = 7.5 Hz), 7.63 (t, 1H, J = 7.5 Hz), 8.08 (d, 2H, J = 7.4 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) δ –126.3 (2F), –119.1 (2F), –79.6 (3F).

Typical procedure for a preparation of 3-(perfluoroalkyl)prop-1-enes by rfspe
Under argon atmosphere, perfluorooctyl iodide (272 mg, 0.5 mmol), tributylallylstannane (330 mg, 1.0 mmol) and AIBN (9 mg, 10 mol%) were dissolved in 5 mL of hexane. After stirring at 80 °C for 5 h, the reaction mixture was cooled, concentrated and charged to a column containing 6 g of standard silica gel. The column was eluted with 20 mL FC-72/ diethylether (2/1), and the solvent was evaporated to provide the **2a** (189 mg, 82%) as a

colorless oil.

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundec-1-ene 2a.[4]
Colorless oil (82% yield, 95.1% GC purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.86 (dt, 2H, *J* = 18.2, 6.7 Hz), 5.35 (m, 2H), 5.80 (m, 2H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –125.2 (2F), –122.4 (2F), –121.9 (2F), –120.7 (6F), –112.1 (2F), –79.4 (3F).

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,13-Heneicosafluorotridec-1-ene 2b.[4]
Colorless oil (97% yield, 97.0% purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.86 (dt, 2H, *J* = 18.2, 6.7 Hz), 5.36 (m, 2H), 5.81 (m, 2H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.8 (2F), –121.9 (2F), –121.6 (2F), –120.6 (10F), –112.1 (2F), –79.5 (3F).

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,14,14,15,15,15-Pentacosafluoropentadec-1-ene 2c.
colorless solid (89% yield, 94.5% purity); mp 74.5 – 75.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.86 (dt, 2H, *J* = 18.3, 6.9 Hz), 5.35 (m, 2H), 5.81 (m, 2H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –121.9 (2F), –121.5 (2F), –120.5 (14F), –112.0 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{15}H_5F_{25}$ (M^+): 659.9992. Found: 659.9996.

4,4,5,5,6,6,7,7,8,8,9,9,10,11,11,11-Hexadecafluoro-10-trifluoromethylundec-1-ene 2d.[4]
Colorless oil (86% yield, 92.2% purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.86 (dt, 2H, *J* = 18.3, 6.9 Hz), 5.36 (m, 2H), 5.81 (m, 2H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –184.8 (1F), –121.9 (2F), –120.3 (4F), –119.6 (2F), –113.8 (2F), –112.1 (2F), –70.8 (6F).

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-2-methylundec-1-ene 3a.[4]
Colorless oil (69% yield, purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.96 (s, 3H), 2.94 (t, 2H, *J* = 19.1 Hz), 5.06 (s, 1H), 5.19 (s, 1H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –125.1 (2F), –122.2 (2F), –121.5 (2F), –120.7 (6F), –111.5 (2F), –79.5 (3F).

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,13-Heneicosafluoro-2-methyltridec-1-ene

3b.[4]

Colorless solid (89% yield, 92.0% purity); mp 49.5-51.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.88 (s, 3H), 2.79 (t, 2H, *J* = 19.4 Hz), 4.98 (s, 1H), 5.11 (s, 1H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.0 (2F), –121.7 (2F), –120.6 (10F), –111.7 (2F), –79.5 (3F).

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,14,14,15,15,15-Pentacosafluoro-2-methylpentadec-1-ene 3c.

Colorless amorphous (75% yield, 91.3% purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.88 (s, 3H), 2.79 (t, 2H, *J* = 19.1 Hz), 4.98 (s, 1H), 5.11 (s, 1H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.8 (2F), –122.0 (2F), –121.5 (2F), –120.5 (14F), –111.5 (2F), –79.5 (3F).

4,4,5,5,6,6,7,7,8,8,9,9,10,11,11,11-Hexadecafluoro-2-methyl-10-trifluoromethylundec-1-ene 3d.[4]

Colorless amorphous (84% yield, 92.0% purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.88 (s, 3H), 2.79 (t, 2H, *J* = 19.3 Hz), 4.97 (s, 1H), 5.11 (s, 1H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –185.0 (1F), –122.4 (2F), –120.5 (4F), –119.6 (2F), –113.9 (2F), –111.8 (2F), –70.8 (6F).

[1-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-Heptadecafluorononyl)vinyl]benzene 4a.[5]

Colorless amorphous (93% yield, 97.5% purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.29 (t, 2H, *J* = 18.6 Hz), 5.39 (s, 1H), 5.65 (s, 1H), 7.29-7.42 (m, 5H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.1 (2F), –121.5 (2F), –120.7 (4F), –120.4 (2F), –111.2 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{17}H_9F_{17}$ (M^+): 536.0432. Found: 536.0408.

[1-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heneicosafluoroundecyl)vinyl]benzene 4b.

Colorless solid (93% yield, 97.5% purity); mp 57.0-58.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.30 (t, 2H, *J* = 18.6 Hz), 5.39 (s, 1H), 5.65 (s, 1H), 7.27-7.43 (m, 5H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –125.3 (2F), –122.1 (2F), –121.5 (2F), –120.6 (10F), –111.2 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{19}H_9F_{21}$ (M^+): 636.0369. Found: 636.0344.

[1-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,13-Pentacosafluorotridecyl)vinyl]benzene 4c.

Colorless solid (90% yield, 90.8% purity); mp 81.5-82.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.30 (t, 2H, *J* = 18.7 Hz), 5.39 (s, 1H), 5.65 (s, 1H), 7.31-7.42 (m, 5H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.4 (2F), –121.8 (2F), –120.5 (14F), –111.2 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{21}H_9F_{25}$ (M^+): 736.0305. Found: 736.0342.

[1-(2,2,3,3,4,4,5,5,6,6,7,7,8,9,9,9-Hexadecafluoro-8-trifluoromethylnonyl)vinyl]benzene 4d.

Colorless amorphous (86% yield, 99.4% purity); ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.29 (t, 2H, *J* = 18.5 Hz), 5.39 (s, 1H), 5.65 (s, 1H), 7.29-7.43 (m, 5H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –184.9 (1F), –122.3 (2F), –120.3 (4F), –119.6 (2F), –113.8 (2F), –111.2 (2F), –70.7 (6F); HRMS (EI) Calcd for $C_{18}H_9F_{19}$ (M^+): 586.0401. Found: 586.0401.

Typical procedure for a preparation of 5 by rfspe

Under argon atmosphere, perfluorooctyl iodide (272 mg, 0.5 mmol), tributylallylstannane (330 mg, 1.0 mmol) and AIBN (9 mg, 10 mol%) were dissolved in 5 mL of hexane. After stirring at 80 °C for 5 h, the reaction mixture was cooled, concentrated and added diethylether (10 ml). To the reaction mixture, benzaldehide oxime (363 mg, 3.0 mmol) and sodium hypochlorite solution (10 ml, available chlorine 10-13%) were added at –10 °C and stirred vigorously at 23°C for 24 h. After the organic layer was separated and concentrated *in vacuo*, the residue was charged to a column containing 8 g of standard silica gel. The column was eluted with 70 mL FC-72/diethylether (3/1), and the solvent was evaporated to provide the **5b** (197 mg, 68%) as a colorless solid.

5-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-Pentadecafluorooctyl)-3-phenyl-4,5-dihydro-isoxazole 5a.

Colorless solid (62% yield); mp 91.0-92.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.45 (m, 1H), 2.76 (m, 1H), 3.19 (m, 1H), 3.62 (m, 1H), 5.14 (m, 1H), 7.43 (m, 3H), 7.69 (dd, 2H, *J* = 7.5, 1.9 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) δ –125.0 (2F), –122.3 (2F), –121.5 (2F), –120.9 (2F),

–120.4 (2F), –111.4 (2F), –79.6 (3F); HRMS (EI) Calcd for $C_{17}H_{10}F_{15}NO$ (M^+): 529.0520. Found: 529.0523.

5-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-Heptadecafluorononyl)-3-phenyl-4,5-dihydroisoxazole 5b.

Colorless solid (68% yield); mp 100.5-101.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.45 (m, 1H), 2.78 (m, 1H), 3.22 (m, 1H), 3.60 (m, 1H), 5.11 (m, 1H), 7.44 (m, 3H), 7.69 (d, 2H, J = 7.5 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.2 (2F), –121.5 (2F), –120.7 (4F), –120.4 (2F), –111.3 (2F), –79.5 (3F); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.3, 41.0, 74.2, 105-120 (m, C_8F_{17}), 126.8, 129.0, 130.6, 156.8.

5-(2,2,3,3,4,4,5,5,6,6,7,7,8,9,9,9-Hexadecafluoro-8-trifluoromethylnonyl)-3-phenyl-4,5-dihydroisoxazole 5c.

Colorless solid (63% yield); mp 89.0-90.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.46 (m, 1H), 2.80 (m, 1H), 3.20 (m, 1H), 3.65 (m, 1H), 5.11 (m, 1H), 7.45 (m, 3H), 7.69 (m, 2H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –184.9 (1F), –122.2 (2F), –120.3 (4F), –119.5 (2F), –113.8 (2F), –111.4 (2F), –70.6 (6F); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.2, 41.0, 74.2, 105-120 (m, C_8F_{17}), 126.8, 128.9, 130.6, 156.8; HRMS (EI) Calcd for $C_{19}H_{10}F_{19}NO$ (M^+): 629.0486. Found: 629.0459.

5-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heneicosafluoroundecyl)-3-phenyl-4,5-dihydroisoxazole 5d.

Colorless solid (55% yield); mp 120.0-121.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.46 (m, 1H), 2.80 (m, 1H), 3.20 (m, 1H), 3.60 (m, 1H), 5.10 (m, 1H), 7.44 (m, 3H), 7.68 (m, 2H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.2 (2F), –121.5 (2F), –120.5 (10F), –111.4 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{20}H_{10}F_{21}NO$ (M^+): 679.0452. Found: 679.0427.

5-(2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,13-Pentacosafluorotridecyl)-3-phenyl-4,5-dihydroisoxazole 5e.

Colorless solid (55% yield); mp 144.0-144.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.45 (m,

1H), 2.79 (m, 1H), 3.20 (m, 1H), 3.61 (m, 1H), 5.11 (m, 1H), 7.44 (m, 3H), 7.69 (m, 2H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.2 (2F), –121.5 (2F), –120.5 (14F), –111.3 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{22}H_{10}F_{25}NO$ (M^+): 779.0359. Found: 779.0363.

Typical procedure for a preparation of 7 by rfspe

Under argon atmosphere, 3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooctan-1-ol (182 mg, 0.5 mmol), butyric acid (66 mg, 0.75 mmol), triphenylphospine (197 mg, 0.75 mmol) and Aldrithiol^{TM}-2 (165 mg, 0.75 mmol) were dissolved in 5 mL of benzene. After stirring at 80 °C for 24 h, the reaction mixture was cooled, concentrated and charged to a column containing 6 g of standard silica gel. The column was eluted with 20 mL FC-72/diethylether (2/1), and the solvent was evaporated to provide the **7a** (185 mg, 85%) as a colorless oil.

Butyric acid 3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl ester 7a.

Colorless oil (85% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.96 (t, 3H, *J* = 7.4 Hz), 1.66 (m, 2H), 2.32 (t, 2H, *J* = 7.4 Hz), 2.50 (m, 2H), 4.39 (t, 2H, *J* = 6.5 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) δ –125.0 (2F), –122.4 (2F), –121.7 (2F), –120.7 (2F), –112.5 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{12}H_{11}F_{13}O_2$ (M^+): 434.0541. Found: 434.0551.

Butyric acid 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluorooctyl ester 7b.

Colorless oil (62% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, 3H, *J* = 7.4 Hz), 1.67 (m, 2H), 2.41 (t, 2H, *J* = 7.4 Hz), 4.60 (t, 2H, *J* = 13.6 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.1 (2F), –121.5 (2F), –120.8 (4F), –118.3 (2F), –79.5 (3F); HRMS (EI) Calcd for $C_{12}H_9F_{15}O_2$ (M^+): 470.0383. Found: 470.0363.

Butyric acid 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-nonadecafluorodecyl ester 7c.[7]

Colorless oil (63% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, 3H, *J* = 7.4 Hz), 1.67 (m, 2H), 2.41 (t, 2H, *J* = 7.4 Hz), 4.60 (t, 2H, *J* = 13.6 Hz); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.1 (2F), –121.5 (2F), –120.7 (8F), –118.3 (2F), –79.5 (3F).

Butyric acid 2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11-eicosafluoroundecyl ester 7d.
Colorless solid (66% yield); mp 32.0-33.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, 3H, *J* = 7.4 Hz), 1.70 (m, 2H), 4.60 (t, 2H, *J* = 13.7 Hz), 6.07 (m, 1H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –135.8 (2F), –128.0 (2F), –122.1 (4F), –120.6 (10F), –118.3 (2F); HRMS (EI) Calcd for $C_{15}H_{10}F_{20}O_2$ (M^+): 602.0369. Found: 602.0361.

Typical procedure for a preparation of 9 by rfspe
Under argon atmosphere, piperidine-1,4-dicarboxylic acid mono(4,4,5,5,6,6,7,7,7-nonafluoro-1,1-dimethylheptyl) ester (27.7 mg, 0.06 mmol), EDCI (17.3 mg, 0.09 mmol), HOBT (12.2 mg, 0.09 mmol) and triethylamine (12.5 μl, 0.09 mmol) were dissolved in 1 mL of chloroform. After stirring at 23 °C for 16 h, the reaction mixture was concentrated and charged to a column containing 1 g of standard silica gel. The column was eluted with 5 mL FC-72/hexafluoroisopropanol (5/1), and the solvent was evaporated to provide the **9a** (27.0 mg, 81%) as a colorless solid.

4-(3,4-Dihydro-1*H*-isoquinoline-2-carbonyl)piperidine-1-carboxylic acid 4,4,5,5,6,6,7,7,7-nonafluoro-1,1-dimethylheptyl ester 9a.
Colorless solid (81% yield, 96.0% purity); mp 83.5-84.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.55 (s, 6H), 1.74 (bs, 4H), 2.05-2.18 (m, 4H), 2.78-3.00 (m, 5H), 3.74 (t, 1H, *J* = 5.9 Hz), 3.84 (bs, 1H), 4.15 (m, 2H), 4.69 (s, 1H), 4.71 (s, 1H), 7.15-7.27 (m, 4H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.8 (2F), –123.0 (2F), –113.3 (2F), –79.8 (3F).

4-(3,4-Dihydro-1*H*-isoquinoline-2-carbonyl)piperidine-1-carboxylic acid 4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluoro-1,1-dimethylnonyl ester 9b.
Colorless solid (74% yield, 96.2% purity); mp 97.0-97.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.51 (s, 6H), 1.74 (bs, 4H), 2.06-2.18 (m, 4H), 2.84-2.95 (m, 5H), 3.74 (t, 1H, *J* = 5.9 Hz), 3.85 (bs, 1H), 4.16 (m, 2H), 4.69 (s, 1H), 4.71 (s, 1H), 7.17-7.27 (m, 4H); ^{19}F NMR (272 MHz, $CDCl_3$) δ –124.9 (2F), –122.0 (2F), –121.6 (2F), –120.7 (2F), –113.1 (2F), –79.6 (3F).

4-(3,4-Dihydro-1*H*-isoquinoline-2-carbonyl)piperidine-1-carboxylic acid 4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-heptadecafluoro-1,1-dimethyl-undecyl ester 9c.[6]

Colorless solid (72% yield, 93.0% purity); mp 111.5-112.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.54 (s, 6H), 1.76 (bs, 4H), 2.19-2.25 (m, 4H), 2.80-3.00 (m, 5H), 3.74 (t, 1H, J = 6.0 Hz), 3.85 (bs, 1H), 4.15 (m, 2H), 4.71 (s, 1H), 4.75 (s, 1H), 7.18-7.30 (m, 4H).

Reference

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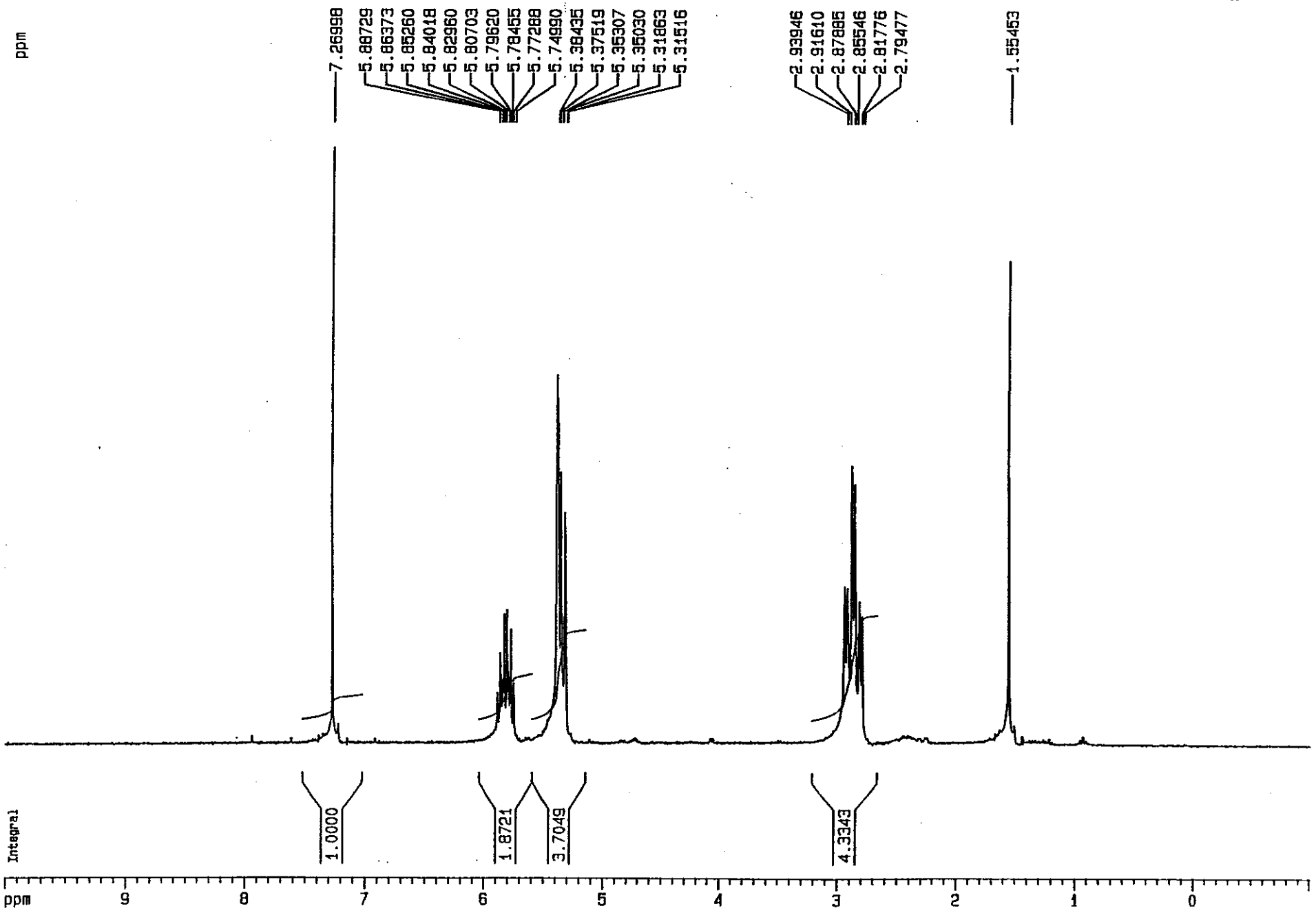
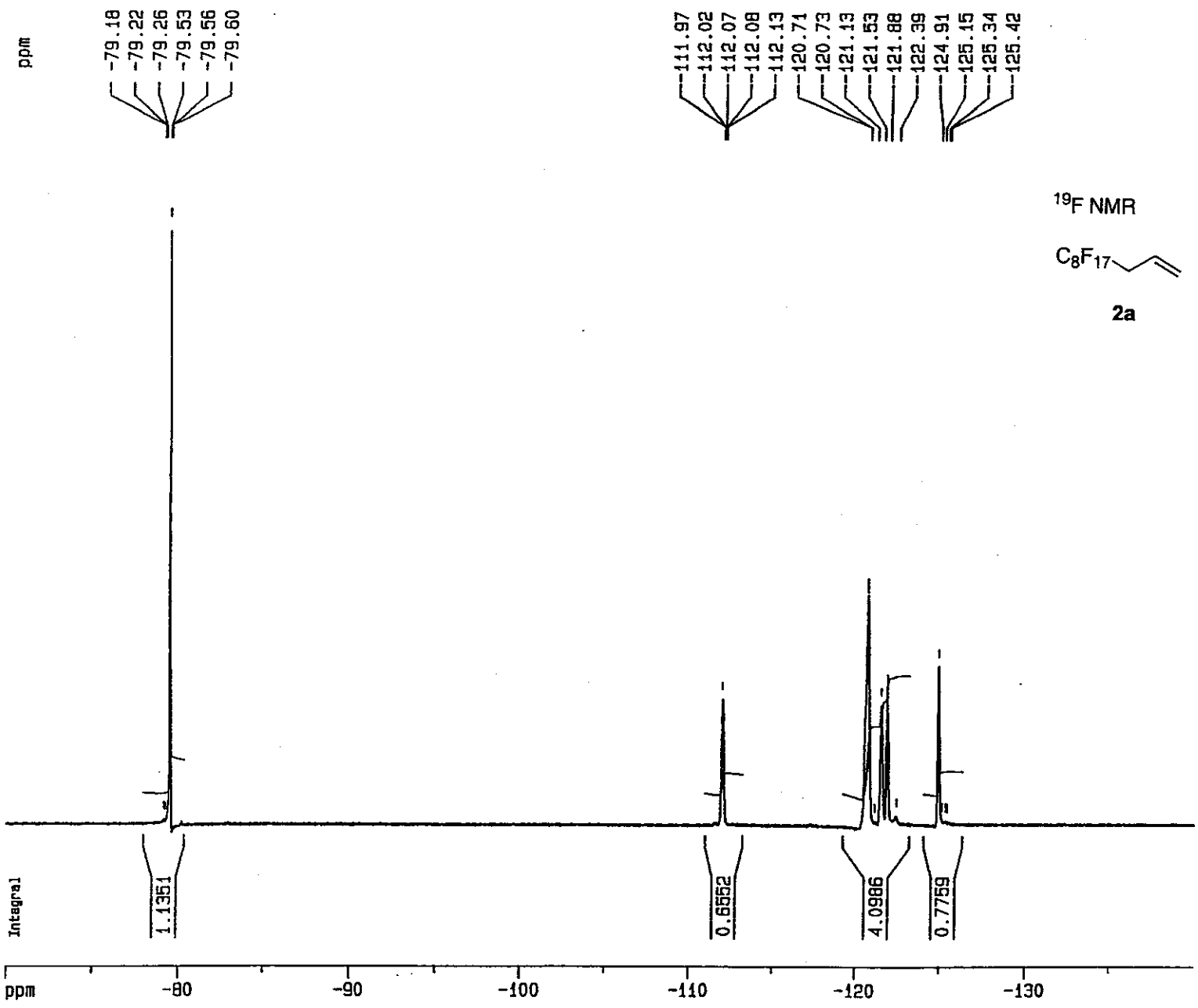
^{1}H NMR

C_8F_{17}

2a



C8H17allyl 19F



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EXPNO	2160
PROCNO	1

F2 - Acquisition Parameters

Date_	20030609
Time	18.29
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	c13wonoe
TD	32768
SOLVENT	Aceton
NS	20
DS	0
SWH	70422.539 Hz
FIDRES	2.149125 Hz
AQ	0.2327028 sec
RG	512
DW	7.100 usec
DE	5.04 usec
TE	300.0 K
D1	6.00000000 sec
D3	0.00100000 sec

CHANNEL f1

NUC1	19F
P1	8.00 usec
PL1	-6.00 dB
SFO1	282.3833007 MHz

CHANNEL f2

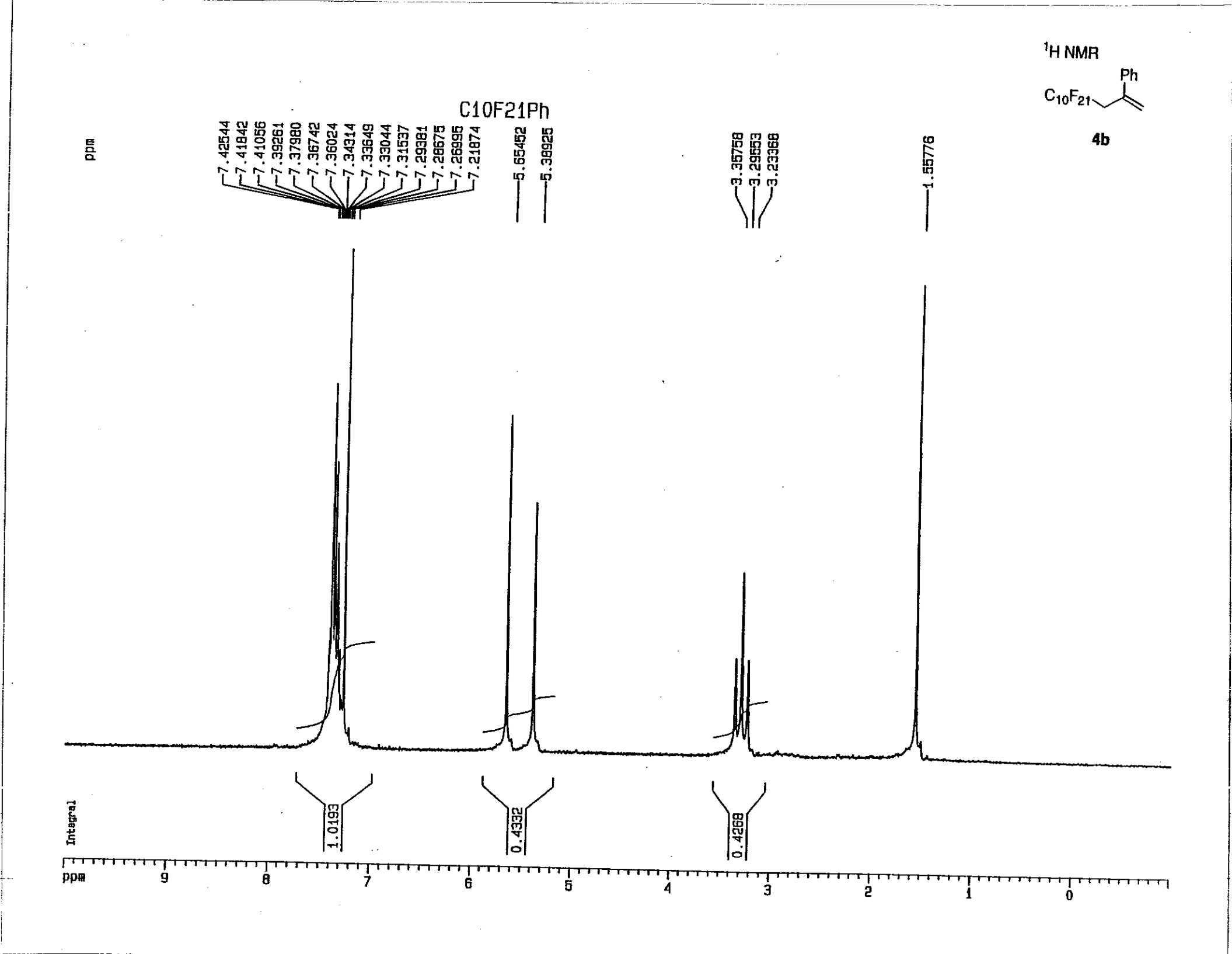
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	21.00 dB
PL12	70.00 dB
SFO2	300.1318008 MHz

F2 - Processing parameters

SI	65536
SF	282.4040236 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	-70.000 ppm
F1	-19768.28 Hz
F2P	-140.000 ppm
F2	-39536.56 Hz
PPMCM	3.50000 ppm/cm
HZCM	988.41406 Hz/cm


^{1}H NMR
Ph
$C_{10}F_{21}$
4b
C10F21Ph
ppm
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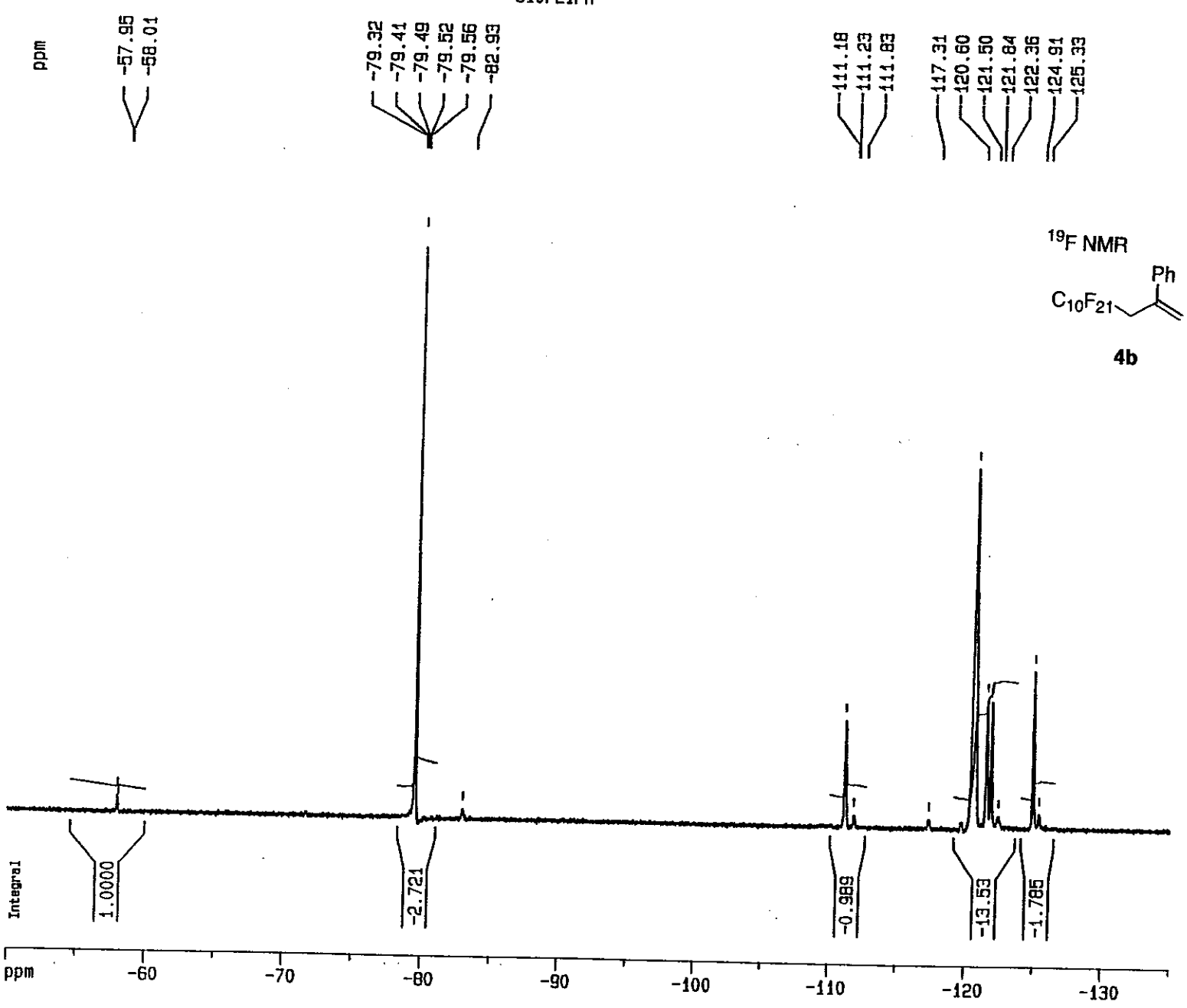
7.41842
7.41056
7.39261
7.37980
7.36742
7.36024
7.34314
7.33649
7.33044
7.31537
7.29381
7.28675
7.26995
7.21874
5.65462
5.38925
3.35758
3.29653
3.23368
1.55776
Integral
1.0193
0.4332
0.4268
ppm
9
8
7
6
5
4
3
2
1
0

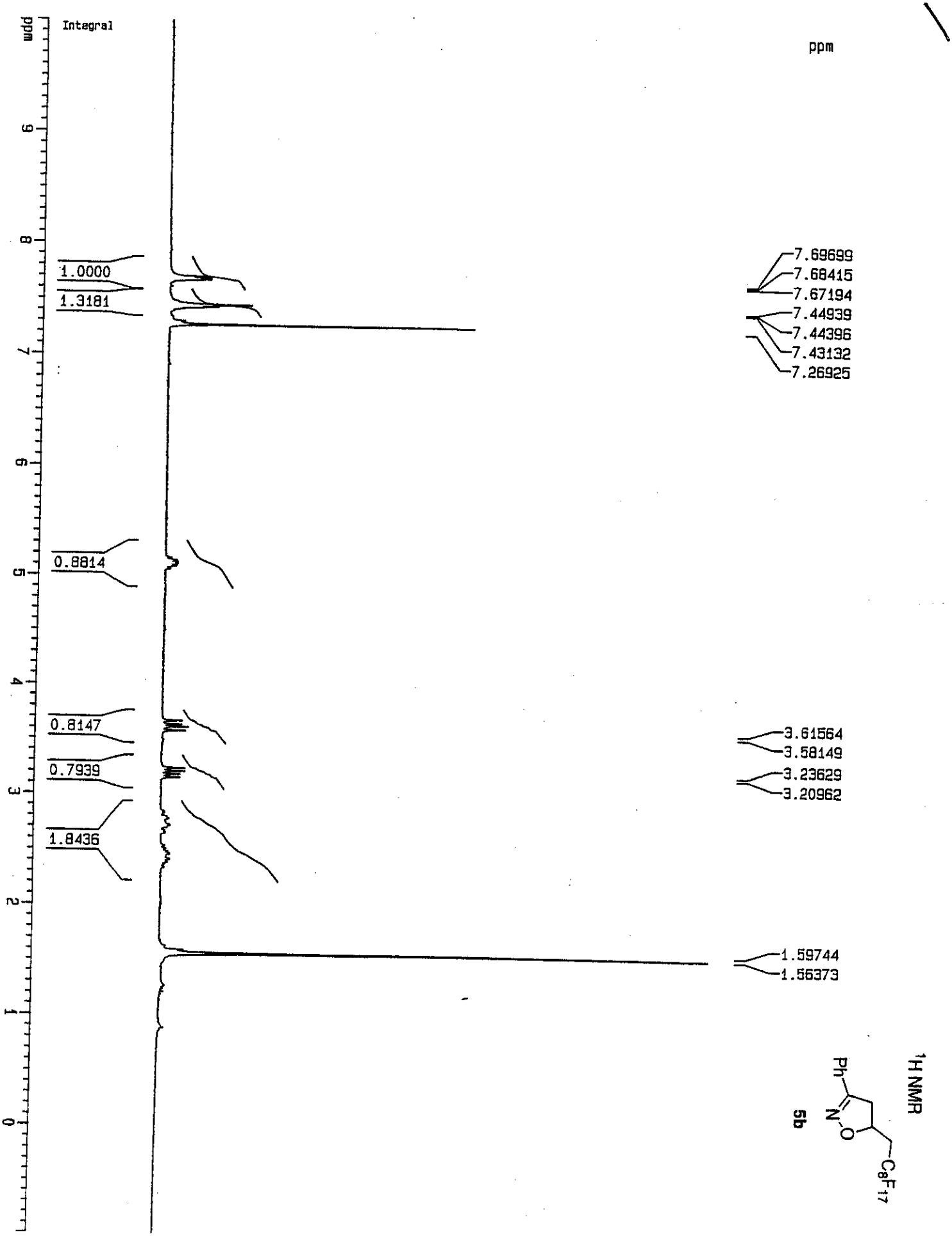
C10F21Ph



^{19}F NMR

4b

```
Current Data Parameters
NAME                  C10F21Ph
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20030604
Time                   15.38
INSTRUM                spect
PROBHD     5 mm QNP  1H
PULPROG                zi3wonoe
TD                     32768
SOLVENT                Aceton
NS                        16
DS                         0
SWH                70422.539 Hz
FIDRES             2.149125 Hz
AQ                 0.2327028 sec
RG                      1024
DW                   7.100 usec
DE                   5.04 usec
TE                   300.0 K
D1                 6.00000000 sec
D3                 0.00100000 sec

========== CHANNEL f1 =======
NUC1                     19F
P1                    8.00 usec
PL1                  -6.00 dB
SFO1           282.3833007 MHz

========== CHANNEL f2 =======
CPDPRG2                waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   21.00 dB
PL12                  70.00 dB
SFO2           300.1318008 MHz

F2 - Processing parameters
SI                       65536
SF             282.4040236 MHz
WDW                      EM
SSB                       0
LB                     0.20 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
F1P                 -50.000 ppm
F1                -14120.20 Hz
F2P                 -135.000 ppm
F2                -38124.54 Hz
PPMCM                4.25000 ppm/cm
HZCM              1200.21704 Hz/cm
```


Integral
ppm
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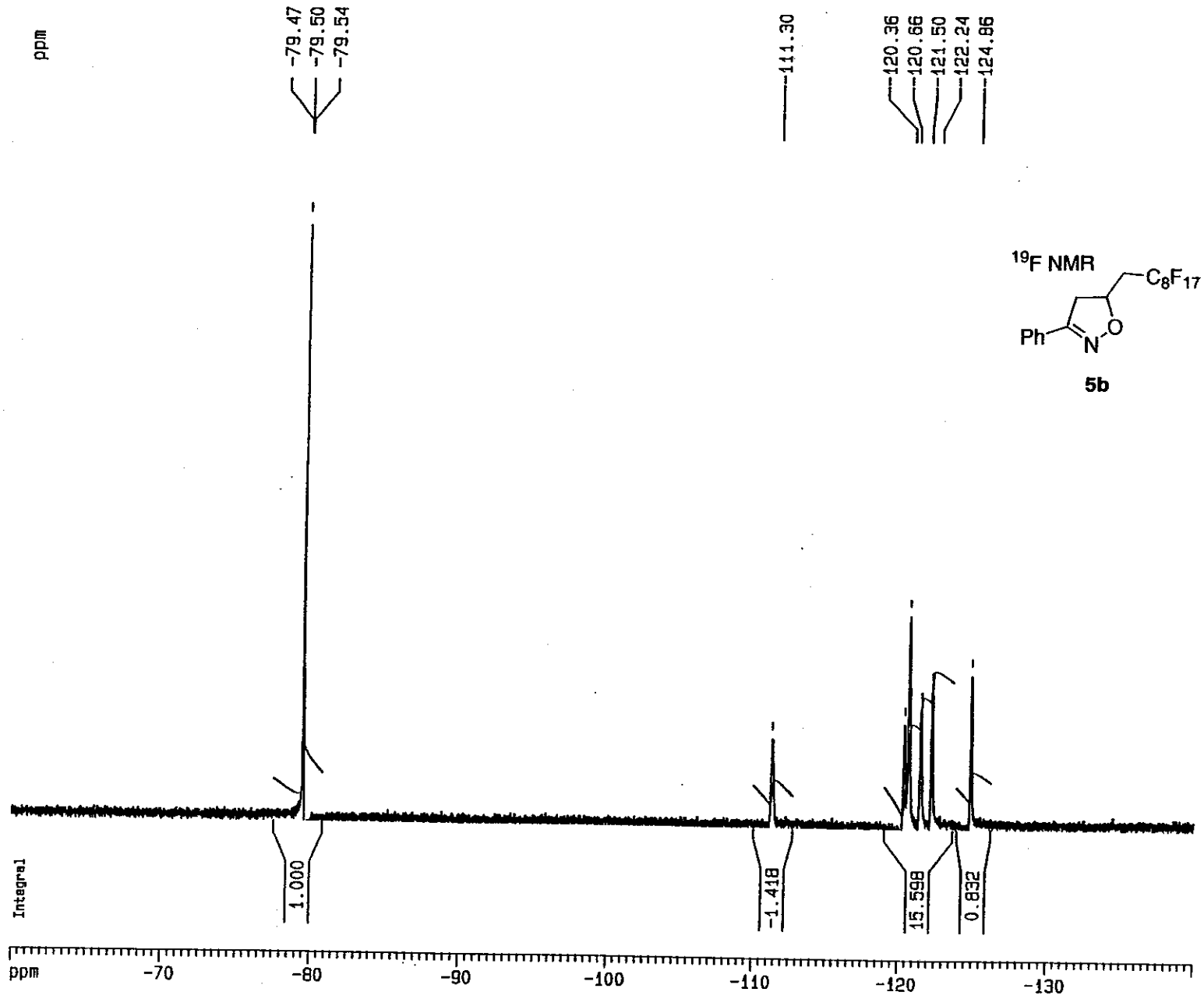
1.3181
0.8814
0.8147
0.7939
1.8436
7.69699
7.68415
7.67194
7.44939
7.44396
7.43132
7.26925
3.61564
3.58149
3.23629
3.20962
1.59744
1.56373
0
1
2
3
4
5
6
7
8
9
ppm

^{1}H NMR

5b

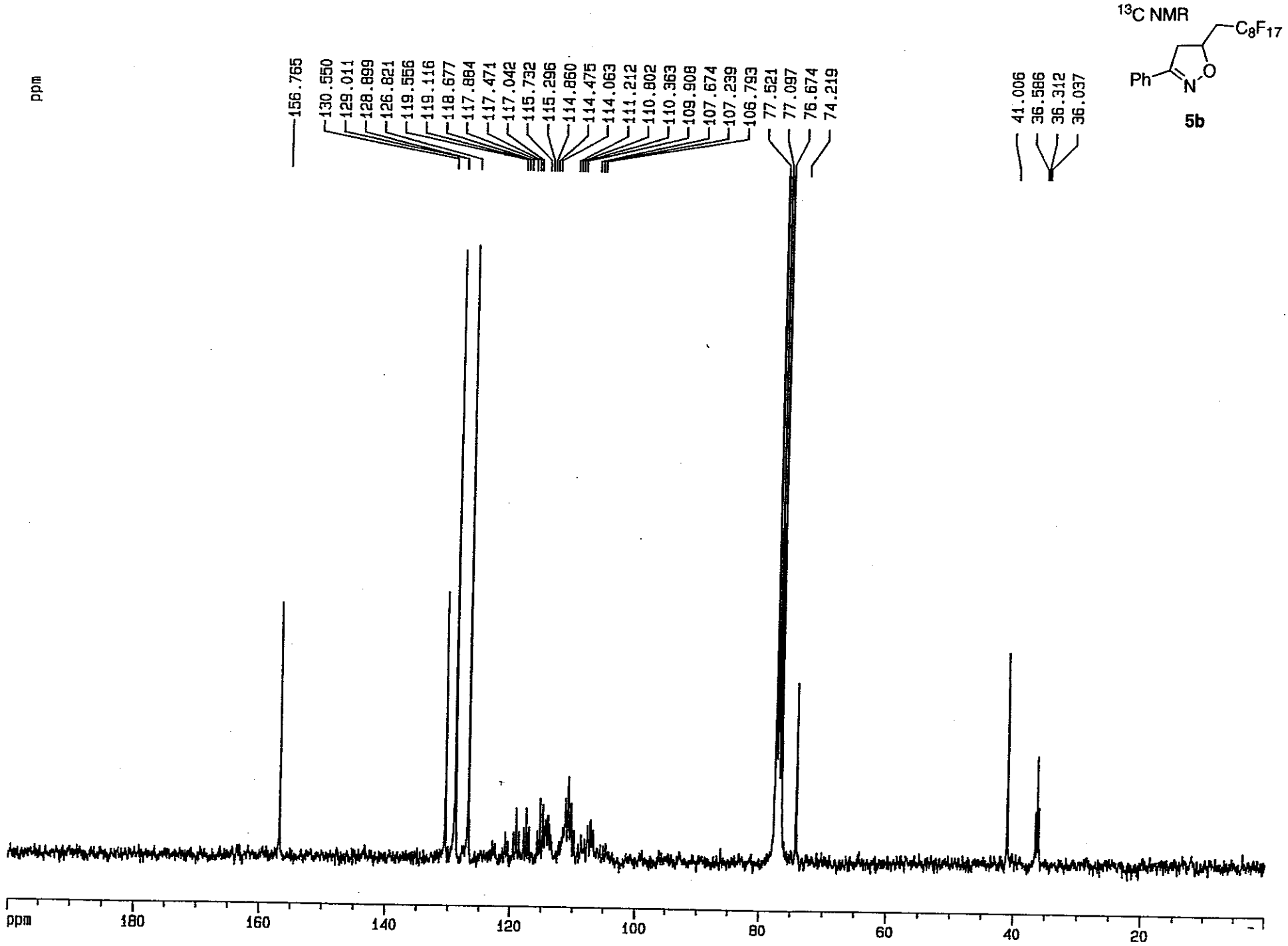
Ph
N
O
C8F17

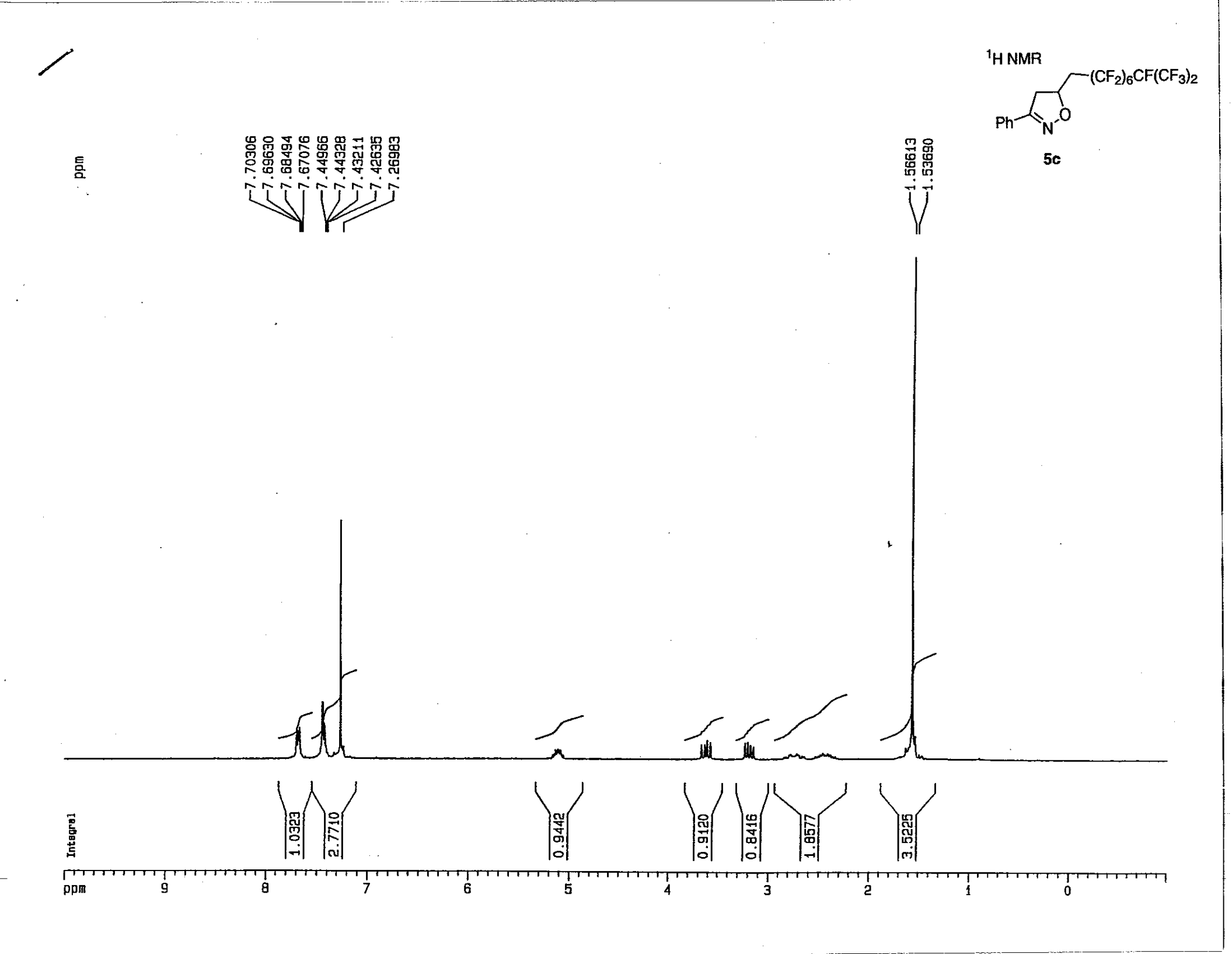


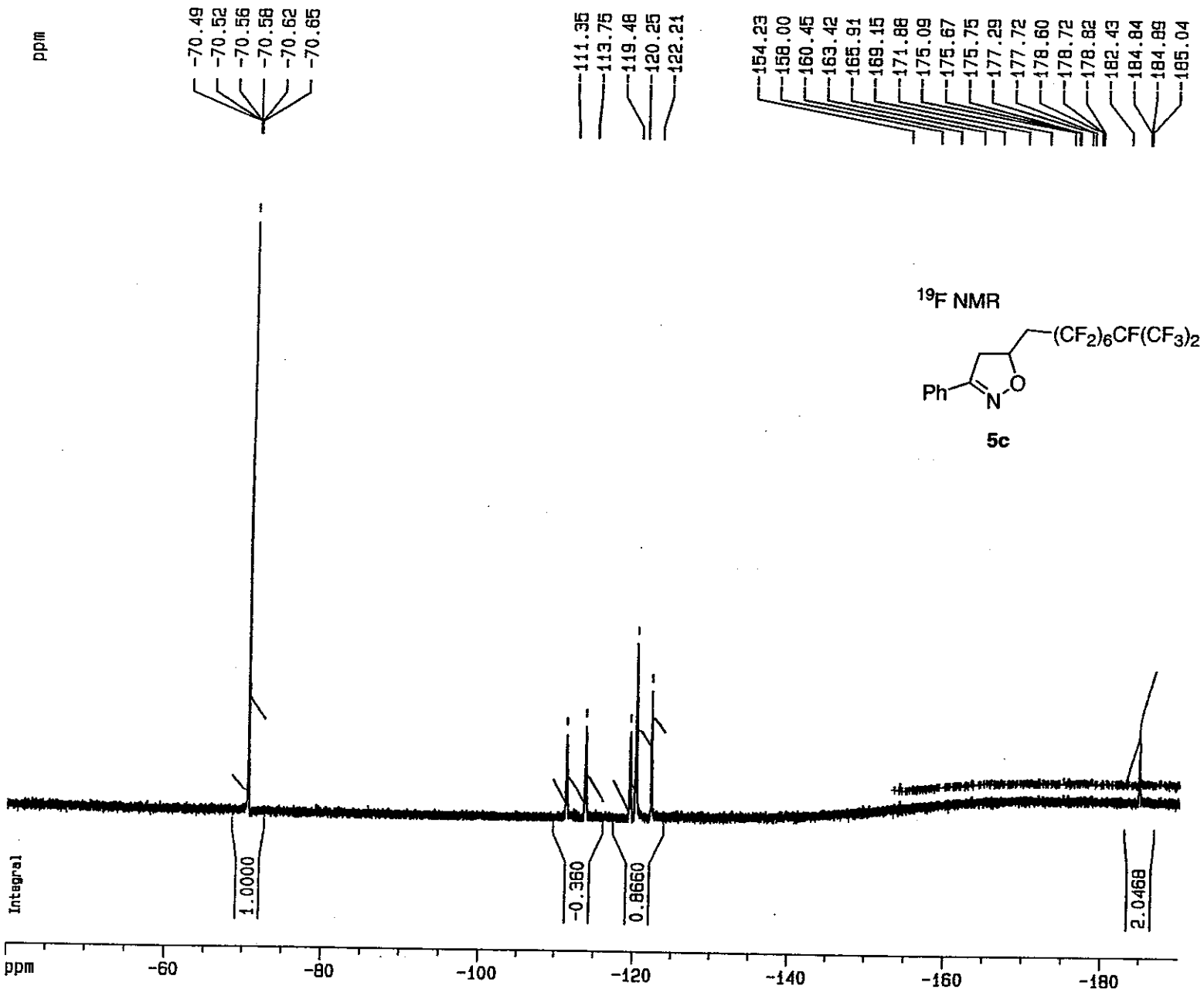
^{19}F NMR

5b

```
Current Data Parameters
NAME            cycloadduct
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20030807
Time                  11.07
INSTRUM               spect
PROBHD     5 mm QNP  1H
PULPROG           c13wonoe
TD                   32768
SOLVENT              Aceton
NS                       35
DS                        0
SWH           70422.539 Hz
FIDRES         2.149125 Hz
AQ             0.2327028 sec
RG                    1024
DW                7.100 usec
DE                5.04 usec
TE                300.0 K
D1             6.00000000 sec
D3             0.00100000 sec

============ CHANNEL f1 =======
NUC1                  19F
P1                 8.00 usec
PL1               -6.00 dB
SFO1          282.3833007 MHz

============ CHANNEL f2 =======
CPDPRG2           waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2               21.00 dB
PL12              70.00 dB
SFO2          300.1318008 MHz

F2 - Processing parameters
SI                   65536
SF            282.4040236 MHz
WDW                  EM
SSB                   0
LB                 0.20 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P              -60.000 ppm
F1             -16944.24 Hz
F2P             -140.000 ppm
F2             -39536.56 Hz
PPMCM             4.00000 ppm/cm
HZCM           1129.61609 Hz/cm
```


^{13}C NMR
C_8F_{17}
Ph
N
O
5b
ppm
156.765
130.550
129.011
128.899
126.821
119.556
119.116
118.677
117.884
117.471
117.042
115.732
115.296
114.860
114.475
114.063
111.212
110.802
110.363
109.908
107.674
107.239
106.793
77.521
77.097
76.674
74.219
41.006
36.586
36.312
36.037
ppm
180
160
140
120
100
80
60
40
20


^{1}H NMR
$(CF_2)_6CF(CF_3)_2$
Ph
N
O
5c
ppm
7.70306
7.69630
7.68494
7.67076
7.44966
7.44328
7.43211
7.42635
7.26983
1.56613
1.53690
Integral
1.0323
2.7710
0.9442
0.9120
0.8416
1.8577
3.5225
ppm
9
8
7
6
5
4
3
2
1
0

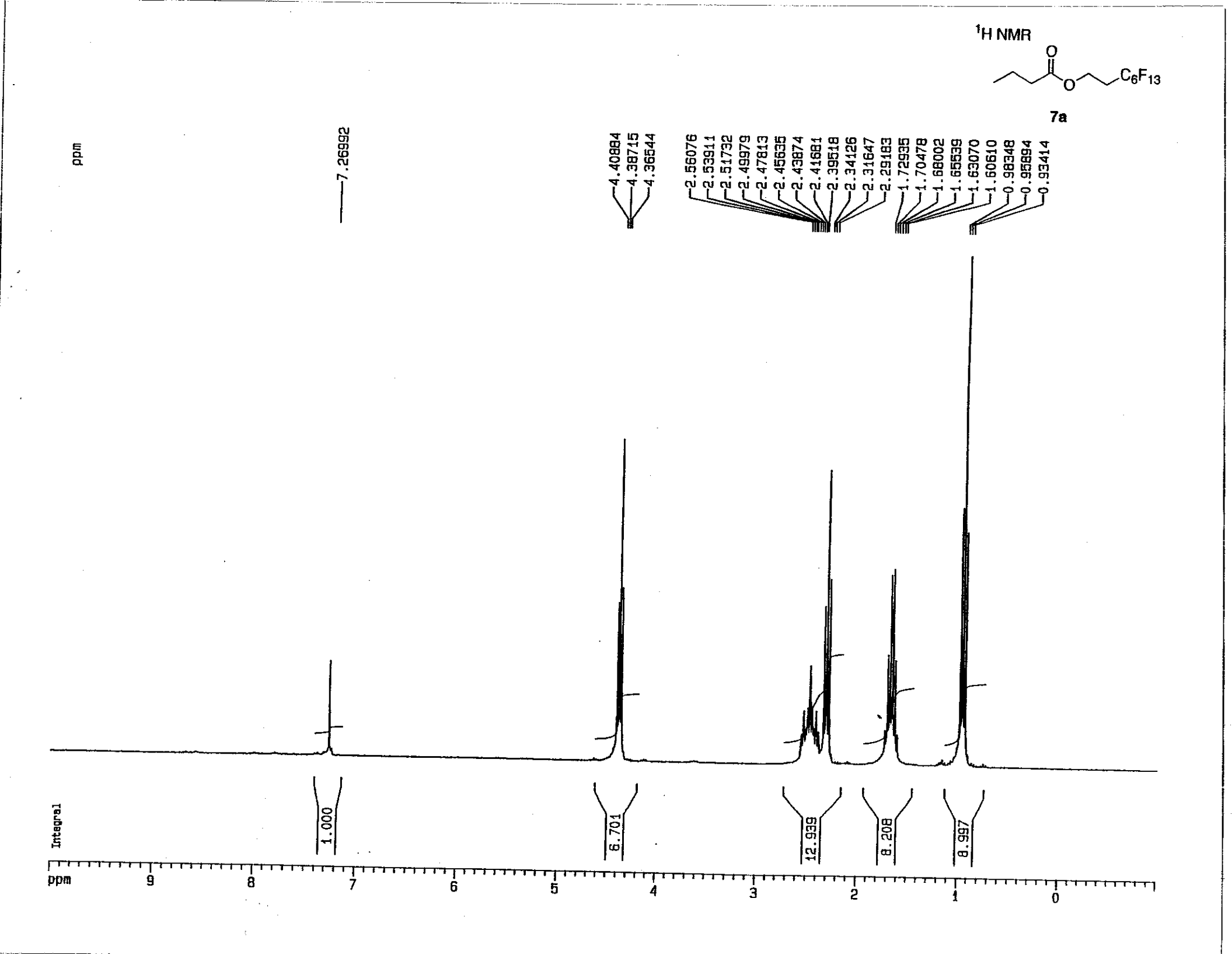


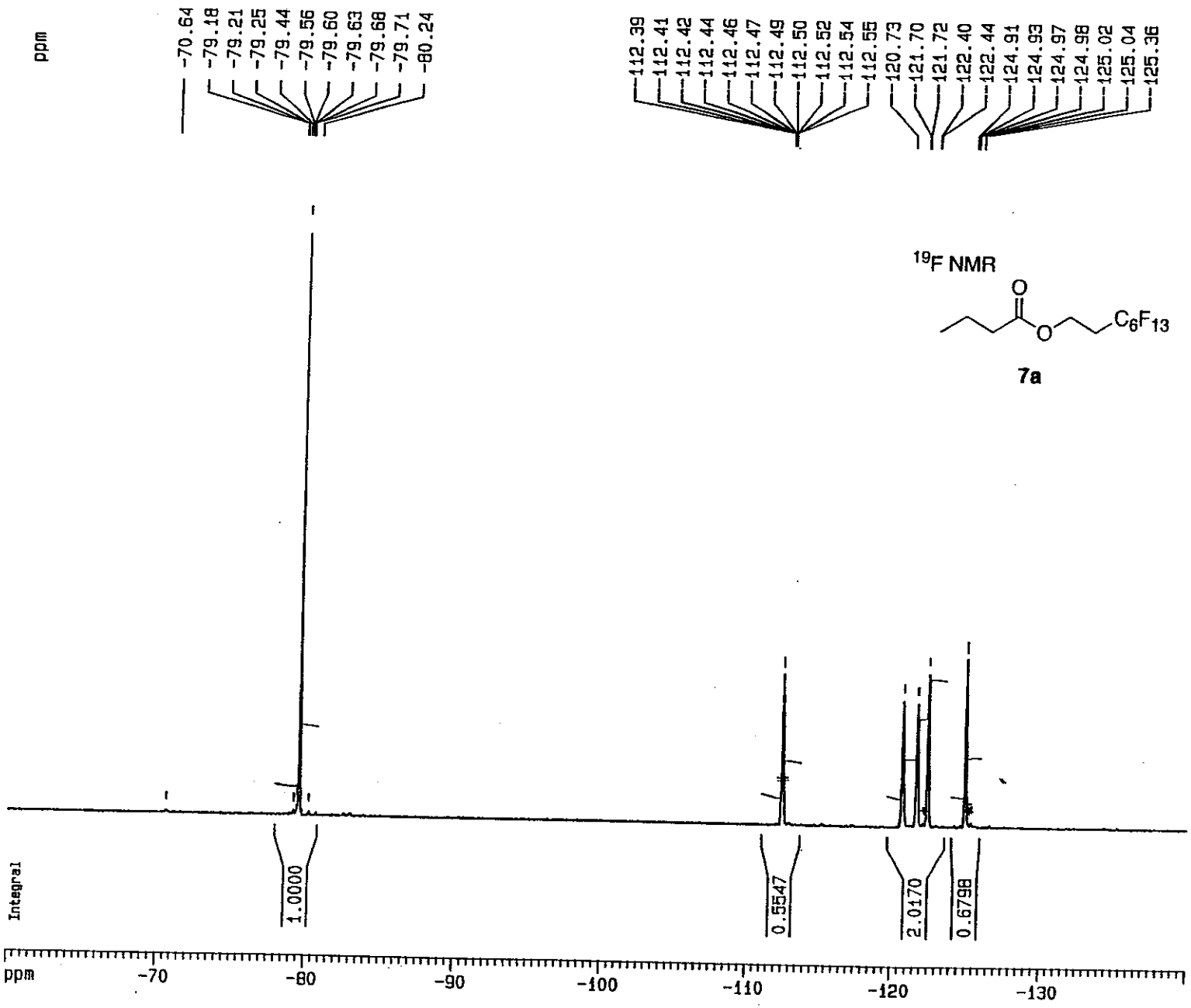
^{19}F NMR

5c

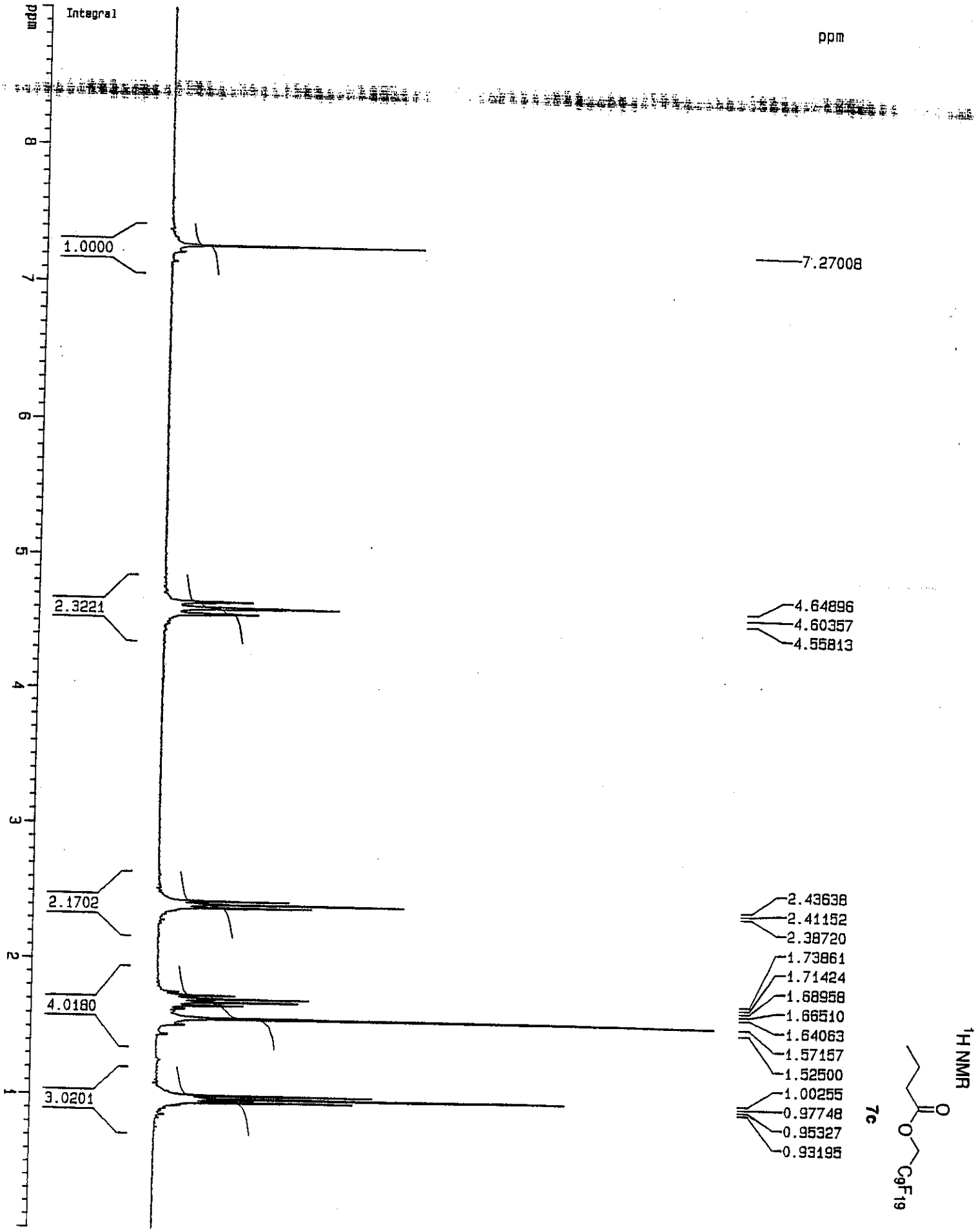
```
Current Data Parameters
NAME        jrsII-8-30-03a
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20030830
Time                  12.31
INSTRUM               spect
PROBHD     5 mm GNP  1H
PULPROG           c13wonoe
TD                   32768
SOLVENT              Aceton
NS                       35
DS                        0
SWH           70422.539 Hz
FIDRES         2.149125 Hz
AQ             0.2327028 sec
RG                    1024
DW                7.100 usec
DE                5.04 usec
TE                300.0 K
D1            6.00000000 sec
D3            0.00100000 sec

========== CHANNEL f1 =======
NUC1                    19F
P1                 8.00 usec
PL1                -6.00 dB
SFO1        282.3833007 MHz

========== CHANNEL f2 =======
CPDPRG2             waltz16
NUC2                    1H
PCPD2             100.00 usec
PL2                21.00 dB
PL12               70.00 dB
SFO2        300.1318008 MHz

F2 - Processing parameters
SI                     65536
SF          282.4040236 MHz
WDW                       EM
SSB                        0
LB                  0.20 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                 20.00 cm
F1P              -40.000 ppm
F1              -11296.16 Hz
F2P              -190.000 ppm
F2              -53656.76 Hz
PPMCM           7.50000 ppm/cm
HZCM         2118.03003 Hz/cm
```

ppm
160
140
120
100
80
60
40
ppm
156.780
130.586
128.926
126.839
120.785
120.434
116.931
116.580
112.742
110.783
77.536
77.113
76.689
74.229
41.035
36.323
36.046
13C NMR
Ph
N
O
(CF2)6CF(CF3)2
5c


^{1}H NMR
7a
ppm
7.26992
4.40884
4.38715
4.36544
2.56076
2.53911
2.51732
2.49979
2.47813
2.45635
2.43874
2.41681
2.39518
2.34126
2.31647
2.29183
1.72935
1.70478
1.68002
1.65539
1.63070
1.60610
0.98348
0.95894
0.93414
Integral
1.000
6.701
12.939
8.208
8.997
ppm
9
8
7
6
5
4
3
2
1
0




```
Current Data Parameters
NAME                  jun960
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20030927
Time                   15.20
INSTRUM                spect
PROBHD     5 mm QNP  1H
PULPROG             c13wonoe
TD                     32768
SOLVENT                Aceton
NS                         15
DS                          0
SWH                70422.539 Hz
FIDRES             2.149125 Hz
AQ                 0.2327028 sec
RG                      1024
DW                   7.100 usec
DE                   5.04 usec
TE                   300.0 K
D1                 6.00000000 sec
D3                 0.00100000 sec

============ CHANNEL f1 =======
NUC1                      19F
P1                     8.00 usec
PL1                   -6.00 dB
SFO1               282.3833007 MHz

============ CHANNEL f2 =======
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   21.00 dB
PL12                  70.00 dB
SFO2               300.1318008 MHz

F2 - Processing parameters
SI                        65536
SF                 282.4040236 MHz
WDW                        EM
SSB                         0
LB                     0.20 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                     20.00 cm
F1P                  -60.000 ppm
F1                  -16944.24 Hz
F2P                  -140.000 ppm
F2                  -39536.56 Hz
PPMCM                4.00000 ppm/cm
HZCM               1129.61609 Hz/cm
```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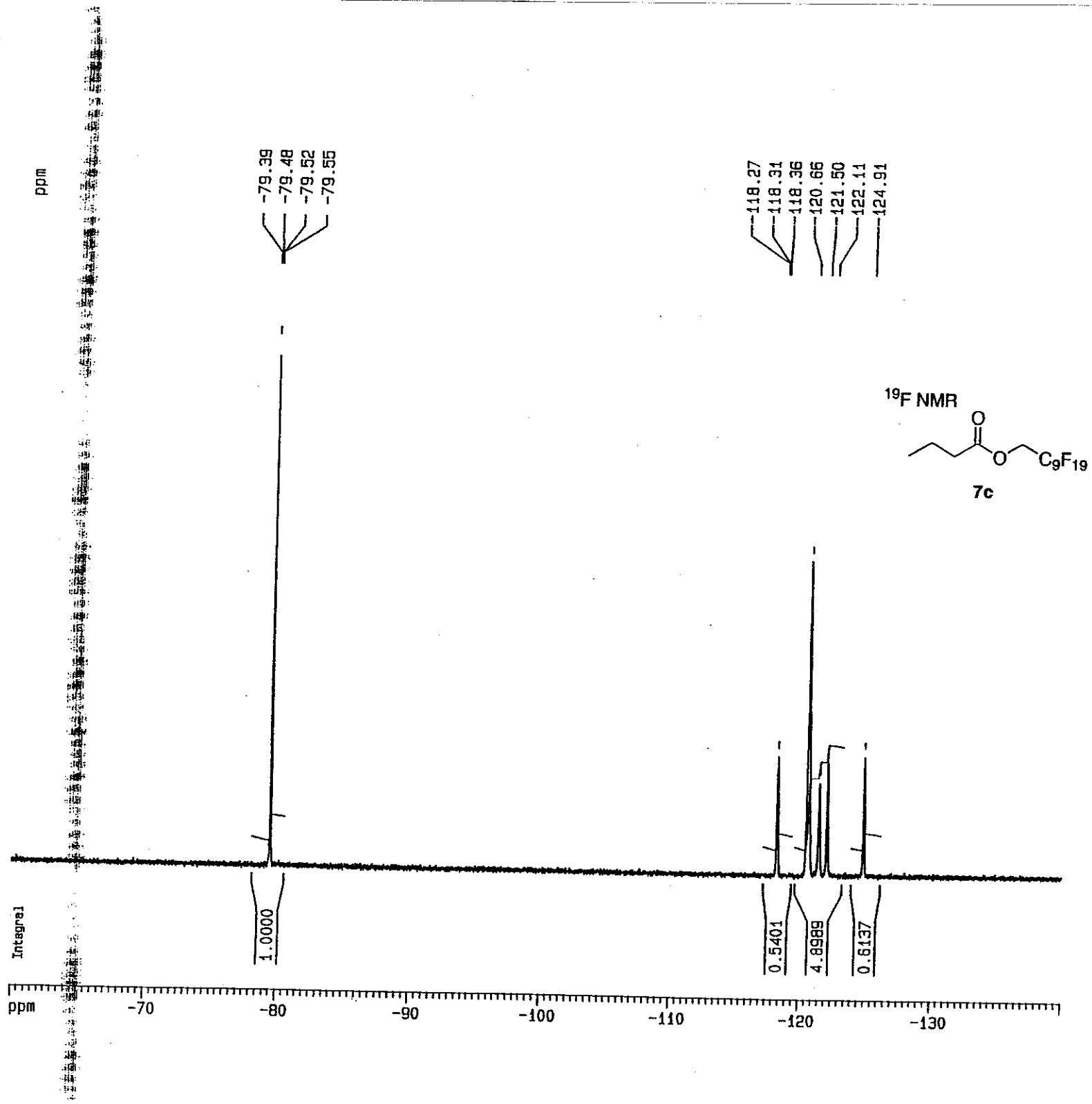
4.60357
4.55813
2.43638
2.41152
2.38720
1.73861
1.71424
1.68958
1.66510
1.64063
1.57157
1.52500
1.00255
0.97748
0.95327
0.93195
^{1}H NMR
7c
C_9F_{19}



Current Data Parameters

NAME	Magneticrecor
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030917
Time	14.15
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	c13wonoe
TD	32768
SOLVENT	Aceton
NS	6
DS	0
SWH	70422.539 Hz
FIDRES	2.149125 Hz
AQ	0.2327028 sec
RG	1024
DW	7.100 usec
DE	5.04 usec
TE	300.0 K
D1	6.00000000 sec
D3	0.00100000 sec

========== CHANNEL f1 =======

NUC1	19F
P1	8.00 usec
PL1	-6.00 dB
SFO1	282.3833007 MHz

========== CHANNEL f2 =======

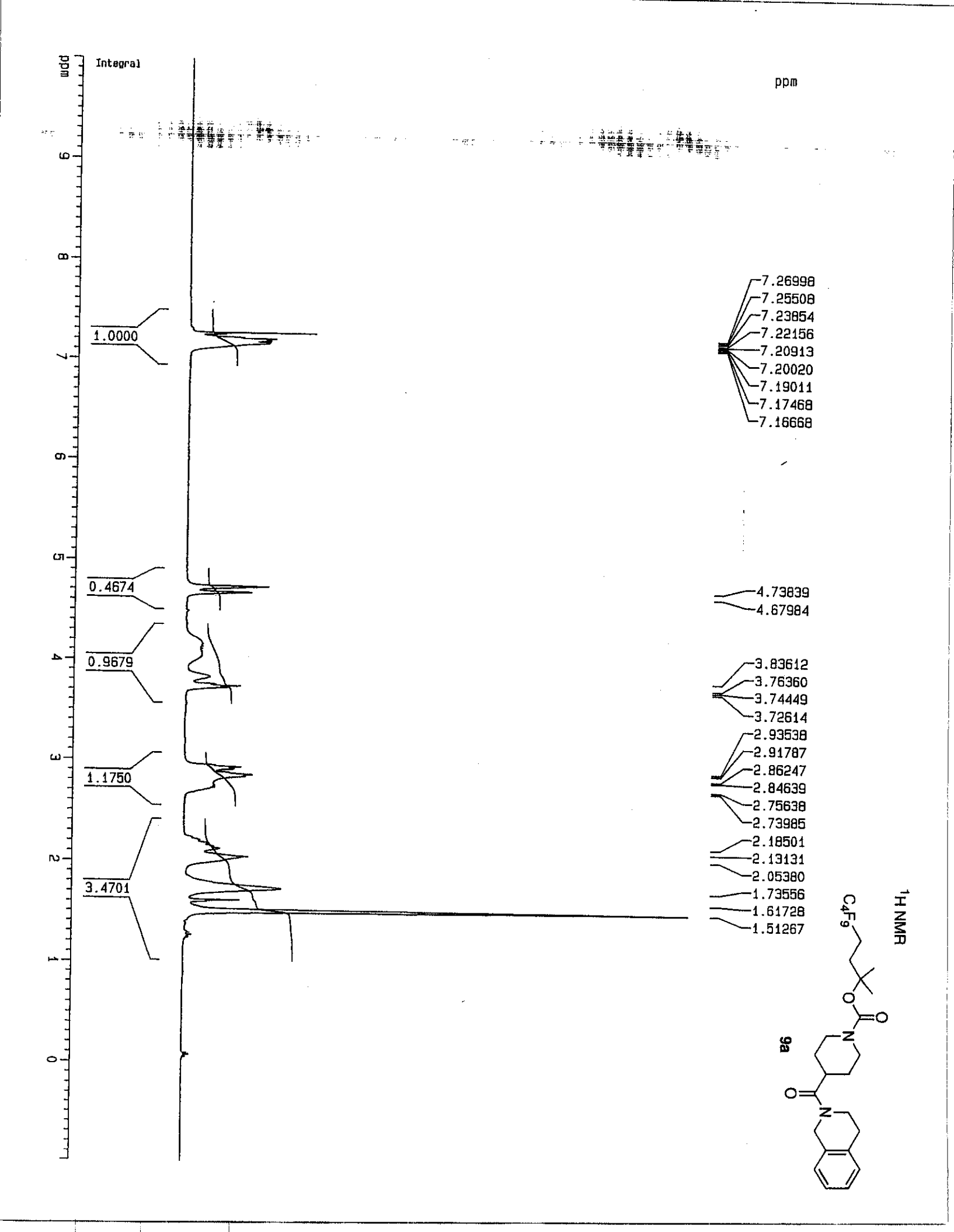
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	21.00 dB
PL12	70.00 dB
SFO2	300.1318008 MHz

F2 - Processing parameters

SI	65536
SF	282.4040236 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	-60.000 ppm
F1	-16944.24 Hz
F2P	-140.000 ppm
F2	-39536.56 Hz
PPMCM	4.00000 ppm/cm
HZCM	1129.61609 Hz/cm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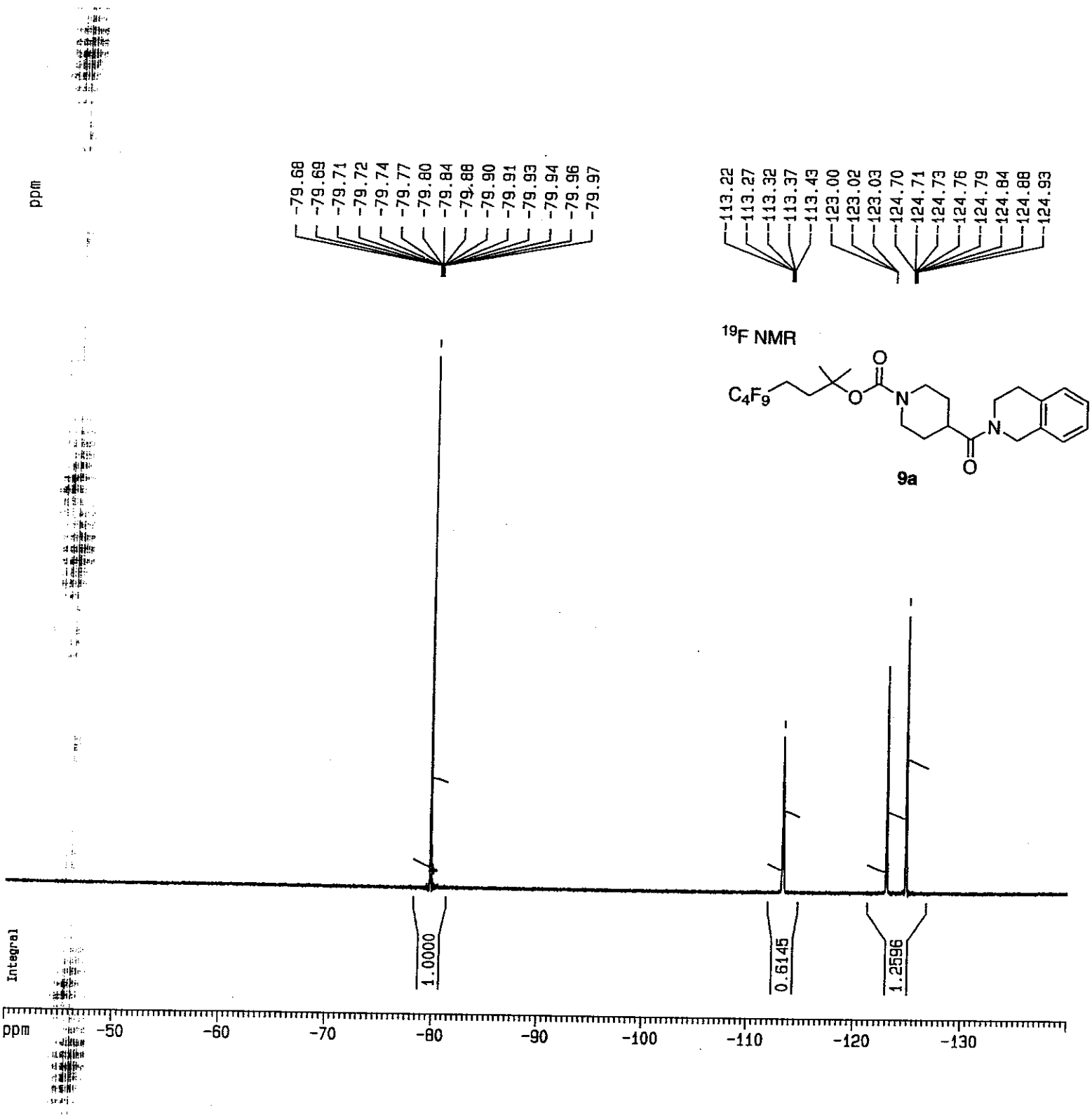
7.25508
7.23854
7.22156
7.20913
7.20020
7.19011
7.17468
7.16668
4.73839
4.67984
3.83612
3.76360
3.74449
3.72614
2.93538
2.91787
2.86247
2.84639
2.75638
2.73985
2.18501
2.13131
2.05380
1.73556
1.61728
1.51267
^{1}H NMR
C_4F_9
O
O
N
O
N
9a



Current Data Parameters

NAME	f9
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040520
Time	9.55
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	c13wonoe
TD	32768
SOLVENT	Aceton
NS	13
DS	0
SWH	70422.539 Hz
FIDRES	2.149125 Hz
AQ	0.2327028 sec
RG	2048
DW	7.100 usec
DE	5.04 usec
TE	300.0 K
D1	6.00000000 sec
D3	0.00100000 sec

========== CHANNEL f1 =======

NUC1	19F
P1	8.00 usec
PL1	-6.00 dB
SFO1	282.3833007 MHz

========== CHANNEL f2 =======

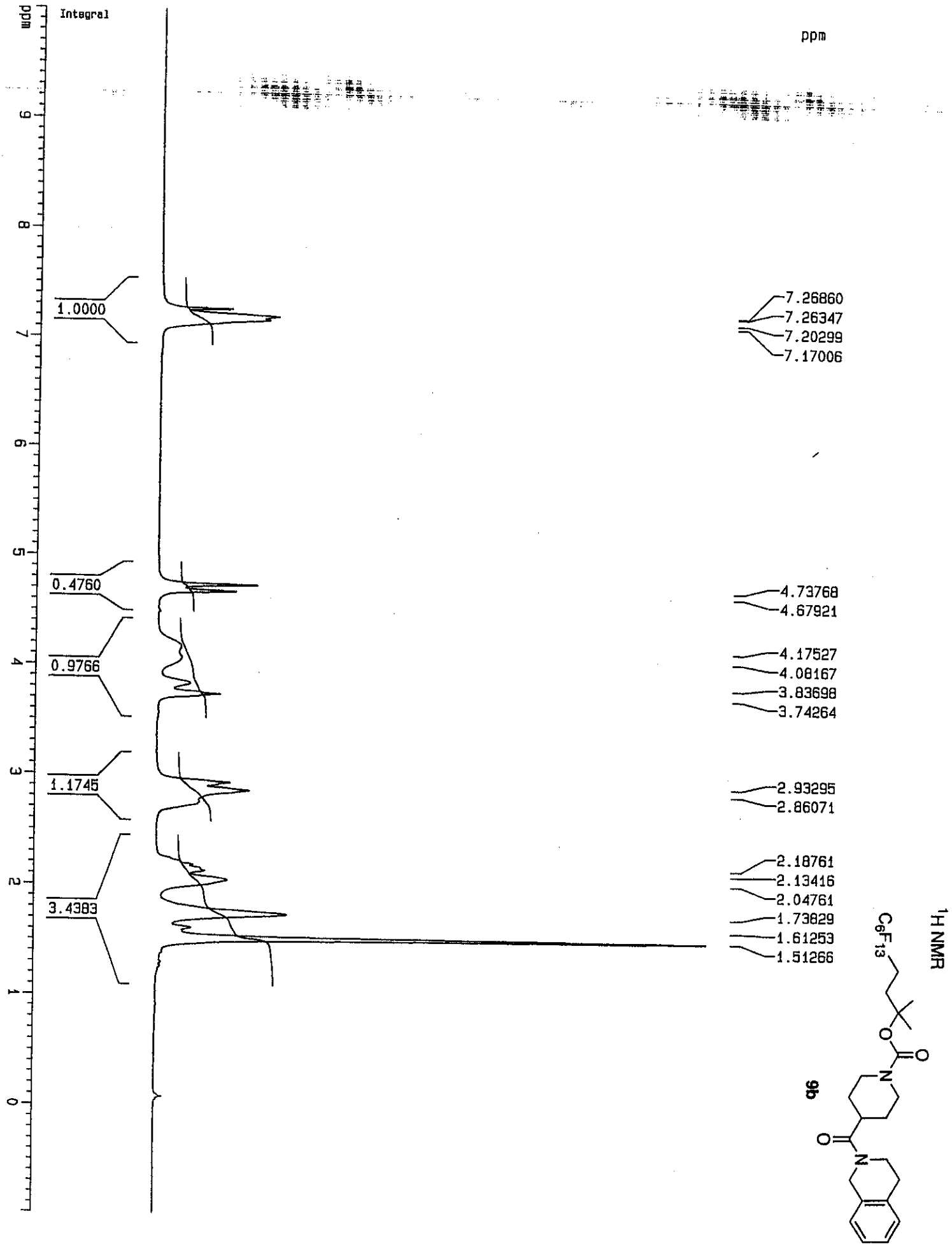
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	21.00 dB
PL12	70.00 dB
SFO2	300.1318008 MHz

F2 - Processing parameters

SI	65536
SF	282.4040236 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	-40.000 ppm
F1	-11296.16 Hz
F2P	-140.000 ppm
F2	-39536.56 Hz
PPMCM	5.00000 ppm/cm
HZCM	1412.02014 Hz/cm


Integral
ppm
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0.4760
0.9766
1.1745
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7.26860
7.26347
7.20299
7.17006
4.73768
4.67921
4.17527
4.08167
3.83698
3.74264
2.93295
2.86071
2.18761
2.13416
2.04761
1.73829
1.61253
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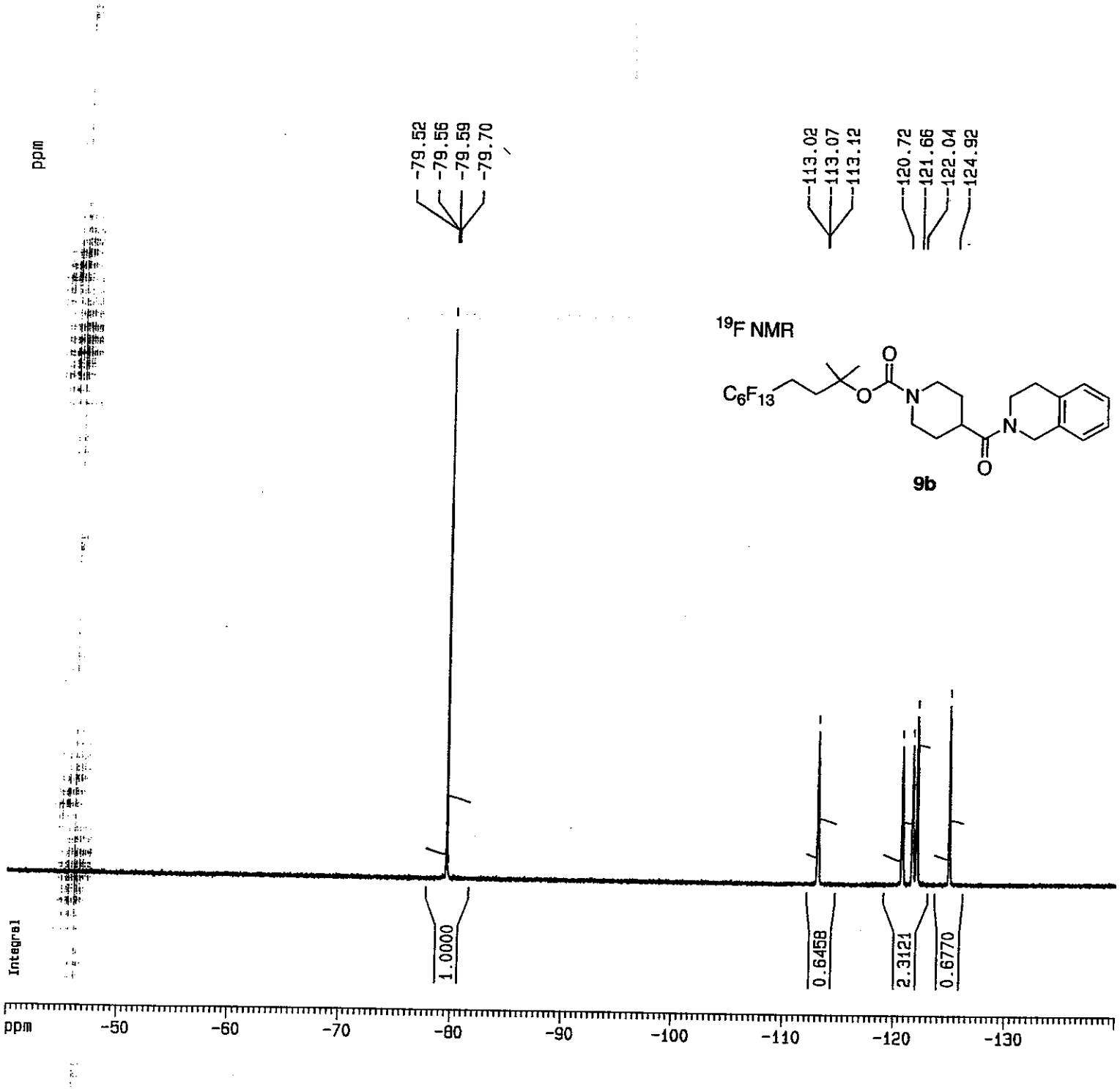
^{1}H NMR
C_6F_{13}
O
O
N
O
N
9g



Current Data Parameters

NAME	f13
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040520
Time	9.58
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	c13wonoe
TD	32768
SOLVENT	Aceton
NS	16
DS	0
SWH	70422.539 Hz
FIDRES	2.149125 Hz
AQ	0.2327028 sec
RG	2048
DW	7.100 usec
DE	5.04 usec
TE	300.0 K
D1	6.00000000 sec
D3	0.00100000 sec

========== CHANNEL f1 ======

NUC1	19F
P1	8.00 usec
PL1	-6.00 dB
SFO1	282.3833007 MHz

========== CHANNEL f2 ======

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	21.00 dB
PL12	70.00 dB
SFO2	300.1318008 MHz

F2 - Processing parameters

SI	65536
SF	282.4040236 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	-40.000 ppm
F1	-11296.16 Hz
F2P	-140.000 ppm
F2	-39536.56 Hz
PPMCM	5.00000 ppm/cm
HZCM	1412.02014 Hz/cm