Synthesis of 2-Aminophenols and Heterocycles by Ru Catalyzed Mono- and Double C-H Hydroxylation

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SUPPORTING INFORMATION

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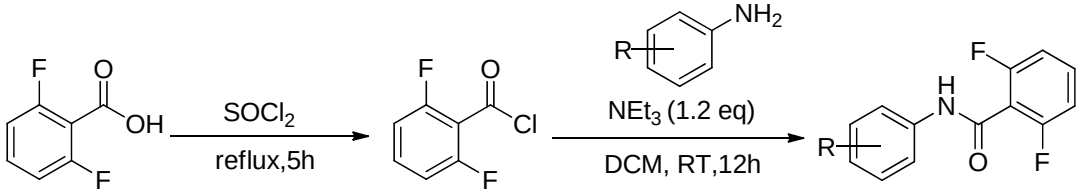
Materials and Methods

All commercial materials (Alfa Aesar, Aladdin, J&K Chemical LTD.) were used without further purification. All solvents were analytical grade. The ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker 400 MHz spectrometer in CD_3OD, DMSO-d6 or $CDCl_3$ using TMS or solvent peak as a standard. All ^{13}C-NMR spectra were recorded with complete proton decoupling. Low-resolution mass spectral analyses were performed with a Waters AQUITY UPLC^{TM}/MS. All reactions were carried out in oven-dried sealed tube. Analytical TLC was performed on Yantai Chemical Industry Research Institute silica gel 60 F254 plates and flash column chromatography was performed on Qingdao Haiyang Chemical Co. Ltd silica gel 60 (200-300 mesh). The rotavapor was BUCHI's Rotavapor R-3.

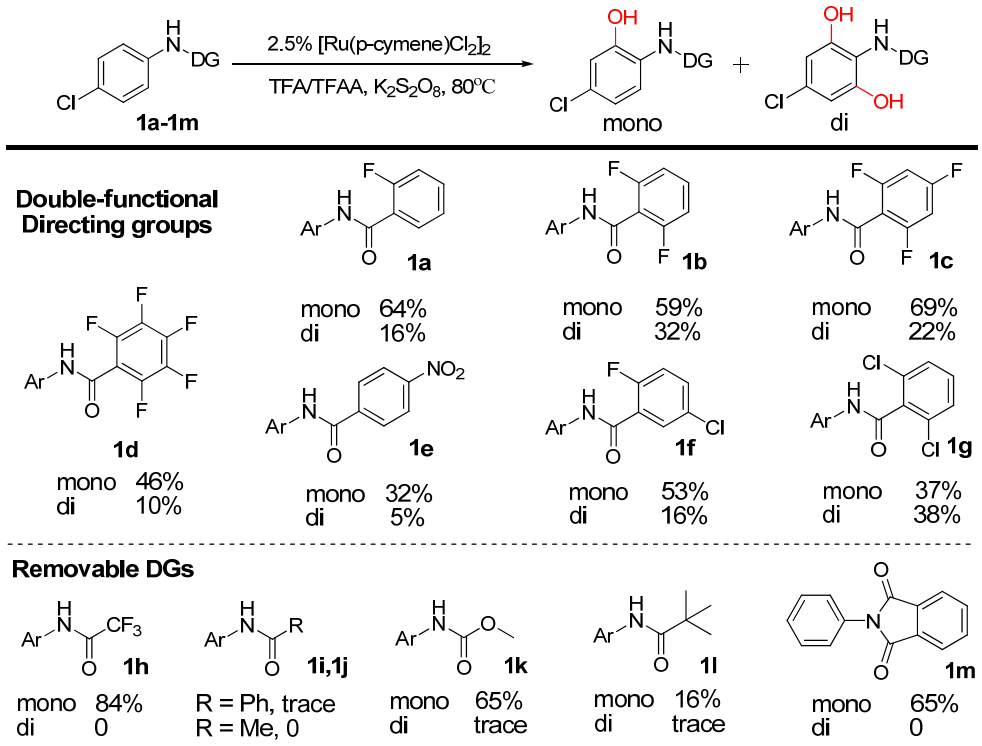
General procedure for substrates preparation:



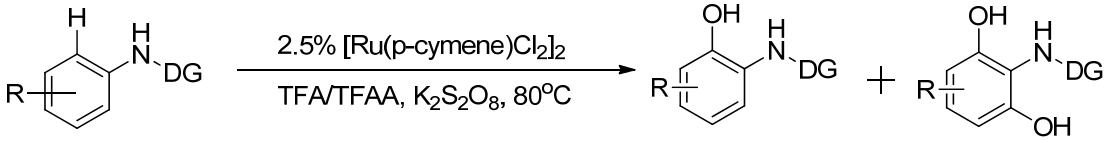
In room temperature, an acid chloride (20 mmol), prepared from 2,6-difluorobenzoic acid and thionyl chloride, was dissolved in 10 mL DCM, then NEt_3 (24.0 mol) and corresponding aniline were added solwly. The reaction mixture was stirred for 12h at room temperature. The product mixture was quenched with 1 mmol/L HCl, extracted with DCM, dried over Na_2SO_4, and evaporated in vacuum to afford the crude product, which was further purified by recrystallizing from DCM/hexane to give the amide.

Screening different directing groups

To a 15ml sealed-tubes were added protected 4-choloroaniline (0.1 mmol, 1.0 equiv), $K_2S_2O_8$ (54 mg, 2.0 equiv), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), TFAA (0.5 ml) and TFA (1.5ml). The tube was sealed and heated at 80 ^{o}C for 10-20h. The reaction was monitored by TLC (petroleum ether: ethyl acetate = 5:1). After completion of the reaction, dichloromethane was added to dilute the reaction mixture and saturated aqueous $NaHCO_3$ was added to neutralize TFA and TFAA. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography. Twelve directing groups were screened. The results were summarized in the following table.



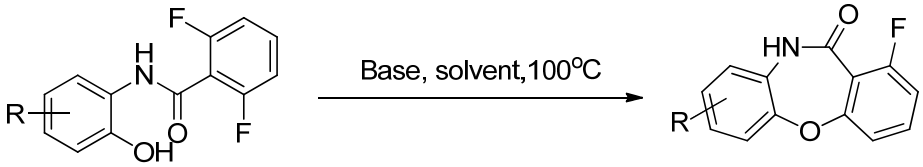
I General procedure for ruthenium catalyzed ortho hydroxylation of benzanilides



To a 15ml sealed-tube were added anilide (1.0 equiv), $K_2S_2O_8$ (1.2-4.0 equiv), [Ru(*p*-cymene)Cl_2]$_2$ (0.025 equiv), TFAA (0.5 ml) and TFA (1.5ml). The tube was sealed and heated at 80 °C. The reaction was monitored by TLC (petroleum ether: ethyl acetate = 5:1). After completion of the reaction, dichloromethane was added to dilute the reaction mixture and saturated aqueous $NaHCO_3$ was added to neutralize TFA and TFAA. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

Synthesis of dibenzoxazepines and benzoxazoles

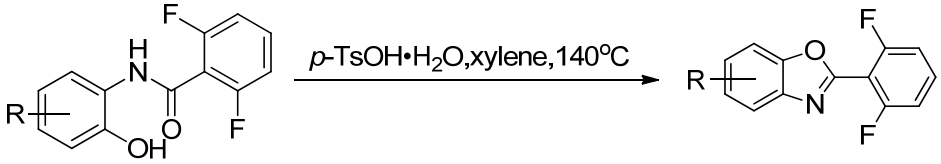
II General procedure for the synthesis of dibenzoxazepines



To a 15ml sealed-tube were added 2-hydroxy benzanilide (1.0equiv), K_2CO_3 (2-4 equiv) or NaOH (2-4 equiv) and 2ml of acetone or DMF at room temperature. The tube was sealed and heated at 100 °C for 2-12h. Then the resulting mixture was

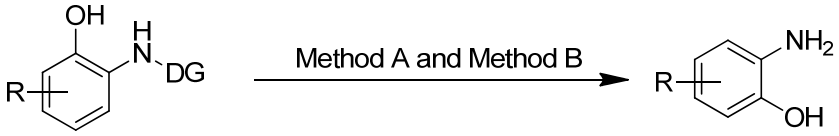
allowed to cool to room temperature and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

III General procedure for the synthesis of benzoxazoles



To a 15ml sealed-tube were added 2-hydroxy benzanilide (1.0equiv), *p*-TsOH.H_2O (5 equiv) and 2ml of *p*-xylene at room temperature. The tube was sealed and heated at 140 ^oC for 12h. Then the resulting mixture was allowed to cool to room temperature and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

General procedure for removal of directing group



Method A

To a 15ml sealed-tube were added 2-hydroxy benzanilide and 2ml of $NH_2NH_2.H_2O$ at room temperature. The tube was sealed and heated at 100 ^oC for 3h. Then the resulting mixture was allowed to cool to room temperature and neutralized with concentrated HCl to PH 7. Then the mixture was extracted with ethyl acetate, the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

Method B

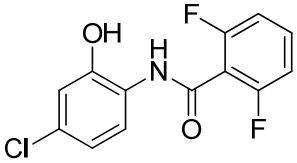
To a 15ml sealed-tube were added 2-hydroxy benzanilide, 17% HCl and THF (V:V=1:1) at room temperature. The tube was sealed and heated at 80 ^oC for 17h. Then the resulting mixture was allowed to cool to room temperature and neutralized with $NaHCO_3$ to PH 7. Then the mixture was extracted with ethyl acetate, the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

Note:

Benzanilides exhibit unique conformational properties. They can show E-form and Z-form in different solvents such as CD_3OD, $CDCl_3$ and DMSO-d6.[1,2] In this work, we observed that dihydroxylated benzanilides and some monohydroxylated benzanilides show trans and cis comformation with same polarity.

References:
[1] Azumaya, I.; Kagechika, H.; Fujiwara, Y.; Itoh, M.; Yamaguchi, K.; Shudo, K. *J. Am.Chem. Soc*. **1991**, *113*, 2833
[2] Okamoto I., Takahashi Y., Sawamura M., Matsumura M., Masu H., Katagiri K., Azumaya I., Nishino M., Kohama Y., Morita N., Tamura .O, Kagechika H., Tanatani H.*Tetrahedron* **2012**, *68*, 5346

Data of products



(1b')

N-(4-chloro-2-hydroxyphenyl)-2,6-difluorobenzamide (1b')

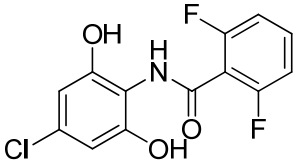
1) small-scale

Following the general procedure Ⅰ, N-(4-chlorophenyl)-2,6-difluorobenzamide (26.8 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 8h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(1b')** (22 mg, white solid) was isolated in 77% yield.

2) big-scale

Following the general procedure Ⅰ, N-(4-chlorophenyl)-2,6-difluorobenzamide (2.14 g, 8 mmol), $K_2S_2O_8$ (2.6 g, 9.6 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (49 mg, 0.08 mmol), 15ml TFA and 5ml TFAA were used. The reaction mixture was stirred at 90 ^oC for 15h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(1b')** (1.27 g, white solid) and compound **(1b")** (0.55 g, white solid) was isolated in 56% and 23% yield respectively.

^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.92 (d, *J* = 8.8 Hz, 1H), 7.55-7.48 (m, 1H), 7.09 (t, *J* = 8.2Hz, 2H), 7.91 (d, *J* = 2.0 Hz, 1H), 6.87 (dd, *J* = 8.8 Hz, 2.0 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.1 (dd, *J* = 249.3 Hz, 6.8 Hz), 161.2, 150.5, 133.4 (t, *J* = 10.1 Hz), 131.6, 125.7, 124.6, 120.3, 116.6, 115.9 (t, *J* = 20.8 Hz), 113.1-113.1 (m); LRMS (ESI) calcd for $C_{13}H_9ClF_2NO_2$ $[M+H]^+$: 284.02 found 284.00

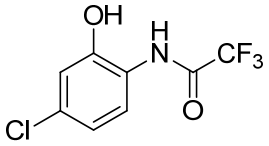


(1b")

N-(4-chloro-2,6-dihydroxyphenyl)-2,6-difluorobenzamide (1b")

Following the general procedure Ⅰ, N-(4-chlorophenyl)-2,6-difluorobenzamide (26.8

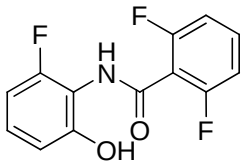
mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (3.0 mg, 0.005 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(1b")** (23 mg, white solid) was isolated in 77% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.55-7.48 (m, 1H), 7.08 (t, *J* = 8.2 Hz, 2H), 6.47 (s, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.3, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 156.2, 154.3, 133.9, 133.5 (t, *J* = 10.1 Hz), 129.8, 115.3 (t, *J* = 20.8 Hz), 113.2, 113.0-112.7 (m), 109.1, 107.8; LRMS (ESI) calcd for $C_{13}H_9ClF_2NO_3$ $[M+H]^+$: 300.02 found 299.96



(1h)

N-(4-chloro-2-hydroxyphenyl)-2,2,2-trifluoroacetamide (1h)

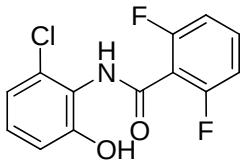
Following the general procedure Ⅰ, N-(4-chlorophenyl)-2,2,2-trifluoroacetamide (2.23g, 10.0 mmol), $K_2S_2O_8$ (5.4g, 20 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (60 mg, 0.1 mmol), 18ml TFA and 6ml TFAA were used. The reaction mixture was stirred at 90 ^oC for 50h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(1h)** (1.8g, white solid) was isolated in 75% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.66 (d, *J* = 8.6 Hz, 1H), 6.92 (d, *J* = 2.0 Hz, 1H), 6.86 (dd, *J* = 8.6 Hz, 1.9 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 156.8 (q, *J* = 37.4 Hz), 151.7, 133.2, 125.8, 123.3, 121.7, 120.4, 117.4 (q, *J* = 285.4 Hz), 116.6; LRMS (ESI) calcd for $C_8H_4ClF_3NO_2$ $[M-H]^-$: 238.00 found 237.91



(2)

2,6-difluoro-N-(2-fluoro-6-hydroxyphenyl)benzamide (2)

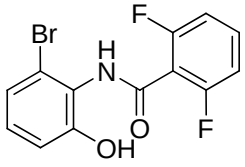
Following the general procedure Ⅰ, 2,6-difluoro-N-(2-fluorophenyl)benzamide (25.2 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 4h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(2)** (24.1 mg, white solid) was isolated in 90% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.54 – 7.50 (m, 1H), 7.17 – 7.07 (m, 3H), 6.75 (d, *J* = 8.28 Hz, 1H), 6.90 (t, *J* = 8.84 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.2, 161.2 (dd, *J* = 249.9 Hz, 7.1 Hz), 159.7 (d, *J* = 245.9 Hz), 155.4 (d, *J* = 4.3 Hz), 133.34 (t, *J* = 9.8 Hz), 129.3 (d, *J* = 10.3 Hz), 115.7 (t, *J* = 21.4 Hz), 113.7 (d, *J* = 15.2 Hz), 113.1 (d, *J* = 2.8 Hz), 113.0 – 112.1 (m), 107.5 (d, *J* = 20.4 Hz); LRMS (ESI) calcd for $C_{13}H_9F_3NO_2$ $[M+H]^+$: 268.05, found 268.01



(3)

N-(2-chloro-6-hydroxyphenyl)-2,6-difluorobenzamide (3)

Following the general procedure Ⅰ, N-(2-chlorophenyl)-2,6-difluorobenzamide (26.8 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 5h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(3)** (19.9 mg, white solid) was isolated in 70% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.54 – 7.48 (m, 1H), 7.17 – 7.07 (m, 3H), 6.98 (dd, *J* = 1.12 Hz, *J* = 8.04 Hz, 1H), 6.89 (dd, *J* = 1.12 Hz, *J* = 8.24 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.3, 161.3 (dd, *J* = 250.03 Hz, 7.1 Hz), 156.0, 133.9, 133.3 (t, *J* =10.0 Hz), 129.9, 123.0, 121.3, 116.0, 115.8-115.6 (m), 113.0-112.7 (m); LRMS (ESI) calcd for $C_{13}H_9F_2ClNO_2$ $[M+H]^+$: 284.02, found 284.00



(4)

N-(2-bromo-6-hydroxyphenyl)-2,6-difluorobenzamide (4)

1) small-scale

Following the general procedure Ⅰ, N-(2-bromophenyl)-2,6-difluorobenzamide (31.2 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 10h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(4)** (26.5 mg, white solid) was isolated in 81% yield.

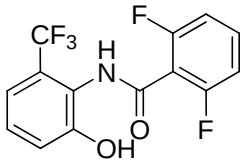
2) big-scale 1

Following the general procedure Ⅰ, N-(2-bromophenyl)-2,6-difluorobenzamide (1.284 g, 4 mmol), $K_2S_2O_8$ (1.296 g, 4.8 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (12.2 mg, 0.02 mmol), 15ml TFA and 5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 17 h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(4)** (0.88 g, white solid) was isolated in 67.4% yield.

3) big-scale 2

Following the general procedure Ⅰ, N-(2-bromophenyl)-2,6-difluorobenzamide (2.184 g, 7 mmol), $K_2S_2O_8$ (2.268 g, 8.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (4.5 mg, 0.007 mmol), 15ml TFA and 5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 55 h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(4)** (1.26 g, white solid) was isolated in 53.6% yield.

^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.55 – 7.48 (m, 1H), 7.15 (d, *J* = 7.96 Hz, 1H), 7.11 – 7.07 (m, 3H), 6.92 (d, *J* = 8.08 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.2, 161.4 (dd, *J* = 250.0 Hz, 7.1 Hz), 156.1, 133.2 (t, *J* = 10.0 Hz), 130.5, 124.5, 123.9, 116.6, 115.8 (t, *J* =21.1 Hz), 113.0 – 112.7 (m); LRMS (ESI) calcd for $C_{13}H_9F_2BrNO_2$ $[M+H]^+$: 327.97, found 327.97



(5)

2,6-difluoro-N-(2-hydroxy-6-(trifluoromethyl)phenyl)benzamide (5)

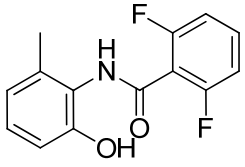
1) small-scale

Following the general procedure Ⅰ, 2,6-difluoro-N-(2-(trifluoromethyl)phenyl)benzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 5h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(5)** (26.3 mg, white solid) was isolated in 83% yield.

2) big-scale

Following the general procedure Ⅰ, 2,6-difluoro-N-(2-(trifluoromethyl)phenyl)benzamide (843 mg, 2.8 mmol), $K_2S_2O_8$ (907 mg, 3.36 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (17 mg, 0.028 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 24h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(5)** (0.48 g, white solid) was isolated in 57% yield.

^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.36 – 7.32 (m, 1H), 7.18 (t, *J* = 8.00 Hz, 1H), 7.03 (t, *J* = 7.40 Hz, 2H), 6.91 (t, *J* = 7.88 Hz, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 163.1, 161.0 (dd, *J* = 250.03 Hz, 7.1 Hz), 156.5, 133.2 (t, *J* =10.0 Hz), 130.8 (q, *J* = 29.6 Hz), 124.9 (q, *J* = 271.1 Hz), 122.3, 121.2, 117.7-117.5 (m), 115.9 (t, *J* = 24.2 Hz), 112.9-112.7 (m); LRMS (ESI) calcd for $C_{14}H_9F_5NO_2$ $[M+H]^+$: 318.05, found 318.00

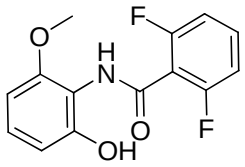


(6)

2,6-difluoro-N-(2-hydroxy-6-methylphenyl)benzamide (6)

Following the general procedure Ⅰ, 2,6-difluoro-N-(o-tolyl)benzamide (24.7 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), 5% $[(C_6H_5)_3P]_3RuCl_2$ (4.5 mg, 0.005 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 70 °C for 6.5h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(6)** (20

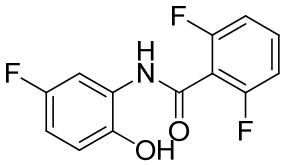
mg, white solid) was isolated in 75% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.52 – 7.50 (m, 1H), 7.12 – 7.04 (m, 3H), 6.77 (d, *J* = 7.84 Hz, 2H), 2.31 (s, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.2, 161.2 (dd, *J* = 249.2 Hz, 7.3 Hz), 154.1, 137.9, 133.1 (t, *J* =10.0 Hz), 129.7, 129.1, 123.7, 122.3, 116.2 (t, *J* =21.1 Hz), 114.8, 113.0-112.7 (m), 18.4; LRMS (ESI) calcd for $C_{14}H_{12}F_2NO_3$ $[M+H]^+$: 264.08, found 263.96



(7)

2,6-difluoro-N-(2-hydroxy-6-methoxyphenyl)benzamide (7)

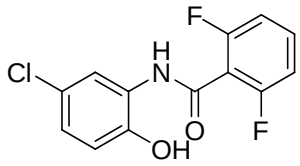
Following the general procedure Ⅰ, 2,6-difluoro-N-(2-methoxyphenyl)benzamide (26.4 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), 5% $[(C_6H_5)_3P]_3RuCl_2$ (4.5 mg, 0.005 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 70 ^{o}C for 4h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(7)** (15 mg, white solid) was isolated in 53% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.54 – 7.50 (m, 1H), 7.13 – 7.07 (m, 3H), 6.58 (d, *J* = 8.28 Hz, 2H), 3.84 (s, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.5, 161.3 (dd, *J* = 249.2 Hz, 7.3 Hz), 156.3, 153.9, 133.3 (t, *J* =10.0 Hz), 129.2, 115.8, 114.9, 113.0-112.7 (m), 111.1, 104.0, 56.5; LRMS (ESI) calcd for $C_{14}H_{12}F_2NO_3$ $[M+H]^+$: 280.07, found 280.07



(8)

2,6-difluoro-N-(5-fluoro-2-hydroxyphenyl)benzamide (8)

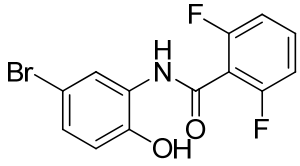
Following the general procedure Ⅰ, 2,6-difluoro-N-(3-fluorophenyl)benzamide (25.2 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^{o}C for 4h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(8)** (19.0 mg, white solid) was isolated in 71% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.88 (dd, *J* = 10.4 Hz, 2.8 Hz, 1H), 7.56-7.48 (m, 1H), 7.09 (t, *J* = 8.0 Hz, 2H), 6.87-6.83 (m, 1H), 6.79-6.74 (m, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.2(dd, *J* = 249.3 Hz, 11.2 Hz), 161.1, 157.2 (d, *J* = 233.1 Hz), 145.1 (d, *J* = 2.2 Hz), 133.5 (t, *J* = 10.1 Hz), 127.6 (d, *J* = 12.1 Hz), 116.6 (d, *J* = 8.8 Hz), 115.8 (t, *J* = 20.1 Hz), 113.1-112.9 (m), 112.2 (d, *J* = 23.2 Hz), 109.8 (d, *J* = 28.5 Hz); LRMS (ESI) calcd for $C_{13}H_9F_3NO_2$ $[M+H]^+$: 268.05, found 268.05



(9)

N-(5-chloro-2-hydroxyphenyl)-2,6-difluorobenzamide (9)

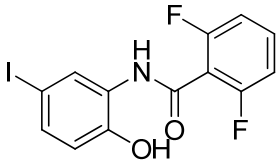
Following the general procedure Ⅰ, N-(3-chlorophenyl)-2,6-difluorobenzamide (26.8 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 5h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(9)** (20.5 mg, white solid) was isolated in 72% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 8.07 (d, *J* = 2.4 Hz, 1H), 7.56-7.49 (m, 1H), 7.10 (t, *J* = 8.0 Hz, 2H), 7.02 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 6.86 (d, *J* = 8.8 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.2(dd, *J* = 249.3 Hz, 11.2 Hz), 161.2, 148.0, 133.5 (t, *J* = 10.1 Hz), 127.9, 126.3, 125.0, 122.9, 117.3, 115.8 (t, *J* = 20.1 Hz), 113.1-112.8 (m); LRMS (ESI) calcd for $C_{13}H_9ClF_2NO_2$ $[M+H]^+$: 284.02, , found 283.97



(10)

N-(5-bromo-2-hydroxyphenyl)-2,6-difluorobenzamide (10)

Following the general procedure Ⅰ, N-(3-bromophenyl)-2,6-difluorobenzamide (31.2 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 10h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(10)** (26.5 mg, white solid) was isolated in 82% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 8.19 (d, *J* = 1.1 Hz, 1H), 7.53 (m, 1H), 7.15 (dd, *J* = 8.6 Hz, 1.1 Hz, 1H), 7.09 (t, *J* = 8.1 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.2(dd, *J* = 249.3 Hz, 6.8 Hz), 161.2, 148.5, 133.5 (t, *J* = 10.2 Hz), 129.2, 128.3, 125.8, 117.8, 115.8 (t, *J* = 20.8 Hz), 113.0 (m), 111.8; LRMS (ESI) calcd for $C_{13}H_9BrF_2NO_2$ $[M+H]^+$: 327.97 found 327.93

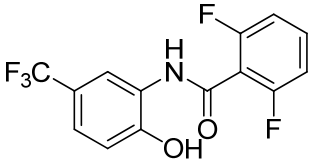


(11)

2,6-difluoro-N-(2-hydroxy-5-iodophenyl)benzamide (11)

Following the general procedure Ⅰ, 2,6-difluoro-N-(3-iodophenyl)benzamide (36.7 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 4h. After completion of the reaction, the residue was purified by silical gel

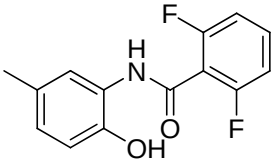
column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(11)** (31 mg, white solid) was isolated in 85% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 8.33 (d, *J* = 1.2 Hz, 1H), 7.56-7.49 (m, 1H), 7.33 (dd, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.09 (t, *J* = 8.1 Hz, 2H), 6.70 (d, *J* = 8.4 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.2(dd, *J* = 249.3 Hz, 6.8 Hz), 161.2, 149.4, 135.6, 133.5 (t, *J* = 10.2 Hz), 131.8, 128.5, 118.5, 115.8 (t, *J* = 20.8 Hz), 113.1-112.8 (m), 80.9; LRMS (ESI) calcd for $C_{13}H_7IF_2NO_2$ $[M-H]^-$: 373.96, found 373.90



(12)

2,6-difluoro-N-(2-hydroxy-5-(trifluoromethyl)phenyl)benzamide (12)

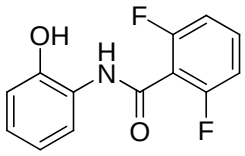
Following the general procedure Ⅰ, 2,6-difluoro-N-(3-(trifluoromethyl)phenyl)benzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 7h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(12)** (22 mg, white solid) was isolated in 79% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 8.40 (s, 1H), 7.56-7.49 (m, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 7.10 (t, *J* = 8.1 Hz, 2H), 7.02 (d, *J* = 8.4 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.2(dd, *J* = 249.3 Hz, 6.8 Hz), 161.4, 152.3, 133.5 (t, *J* = 10.2 Hz), 129.95-129.85 (m), 127.2, 124.6, 123.8-123.7 (m), 123.0-122.0 (m), 120.4-120.2 (m), 116.3, 115.8 (t, *J* = 20.8 Hz), 113.1-112.9 (m); LRMS (ESI) calcd for $C_{14}H_9F_5NO_2$ $[M+H]^+$: 318.05, found 318.04



(13)

2,6-difluoro-N-(2-hydroxy-5-methylphenyl)benzamide (13)

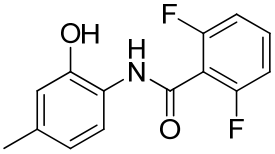
Following the general procedure Ⅰ, 2,6-difluoro-N-(m-tolyl)benzamide (24.7 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), 5% $[(C_6H_5)_3P]_3RuCl_2$ (4.5 mg, 0.005 mmol), 1.8ml TFA and 0.2ml TFAA were used. The reaction mixture was stirred at 80 °C for 4h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(13)** (10.5 mg, white solid) was isolated in 40% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.67 (s, 1H), 7.53-7.49 (m, 1H), 7.09 (t, *J* = 8.1 Hz, 2H), 6.87 (d, *J* = 8.0 Hz, 1H), 6.79 (d, *J* = 8.0 Hz, 1H), 2.27 (s, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.2(dd, *J* = 249.3 Hz, 6.8 Hz), 161.2, 147.3, 133.4 (t, *J* = 10.2 Hz), 130.1, 127.6, 126.3, 124.1, 116.9, 116.0 (t, *J* = 20.8 Hz), 113.1-112.8 (m), 20.8; LRMS (ESI) calcd for $C_{14}H_{12}F_2NO_2$ $[M+H]^+$: 264.08, found 264.06



(14)

2,6-difluoro-N-(2-hydroxyphenyl)benzamide (14)

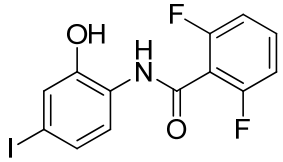
Following the general procedure Ⅰ, 2,6-difluoro-N-phenylbenzamide (22.3 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), 5% $[(C_6H_5)_3P]_3RuCl_2$ (4.5 mg, 0.005 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 70 °C for 6h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(14)** (15 mg, white solid) was isolated in 60% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.88 (d, *J* = 7.9 Hz, 1H), 7.55-7.47 (m, 1H), 7.11-7.03(m, 3H), 6.92-6.85(m, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 159.7(dd, *J* = 249.3 Hz, 6.8 Hz), 159.8, 148.2, 132.0 (t, *J* = 10.2 Hz), 125.8, 125.3, 122.3, 119.3, 115.5, 114.6 (t, *J* = 20.8 Hz), 111.7-111.4 (m); LRMS (ESI) calcd for $C_{13}H_8F_2NO_2$ $[M-H]^-$: 248.06, found 248.06



(15)

2,6-difluoro-N-(2-hydroxy-4-methylphenyl)benzamide (15)

Following the general procedure Ⅰ, 2,6-difluoro-N-(p-tolyl)benzamide (24.7 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), 5% $[(C_6H_5)_3P]_3RuCl_2$ (4.5 mg, 0.005 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 5h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(15)** (12.5 mg, white solid) was isolated in 50% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.68 (d, *J* = 8.4 Hz, 1H), 7.55-7.48 (m, 1H), 7.09(t, *J* = 8.1 Hz, 2H), 6.74 (s, 1H), 6.69 (d, *J* = 8.4 Hz, 1H), 2.27 (s, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.1 (dd, *J* = 249.3 Hz, 6.8 Hz), 161.2, 149.6, 137.5, 133.3 (t, *J* = 10.2 Hz), 124.1, 123.7, 121.1, 117.6, 116.1 (t, *J* = 20.8 Hz), 113.1-112.8 (m), 21.1; LRMS (ESI) calcd for $C_{14}H_{11}F_2NO_2$ $[M+H]^+$: 264.08, found 264.05

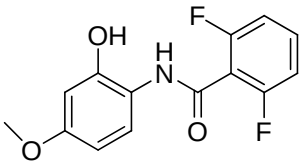


(16)

2,6-difluoro-N-(2-hydroxy-4-iodophenyl)benzamide (16)

Following the general procedure Ⅰ, 2,6-difluoro-N-(4-iodophenyl)benzamide (36.7 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 8h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 10:1). Finally, compound

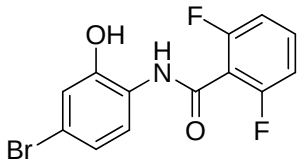
(16) (22.9 mg, white solid) was isolated in 61% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.75 (d, *J* = 8.4 Hz, 1H), 7.53-7.49 (m, 1H), 7.25 (s, 1H), 7.20 (d, *J* = 8.5 Hz, 1H), 7.08 (t, *J* = 8.2Hz, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 160.1 (dd, *J* = 249.3 Hz, 6.8 Hz), 161.1, 150.4, 133.4 (t, *J* = 10.1 Hz), 129.6, 126.9, 125.4, 125.0, 115.9 (t, *J* = 20.8 Hz), 113.1-112.8 (m), 89.4; LRMS (ESI) calcd for $C_{13}H_9F_2INO_2$ $[M+H]^+$: 375.96, found 375.94



(17)

2,6-difluoro-N-(2-hydroxy-4-methoxyphenyl)benzamide (17)

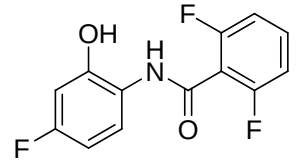
Following the general procedure Ⅰ, 2,6-difluoro-N-(4-methoxyphenyl)benzamide (26.4 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.8ml TFA and 0.2ml TFAA were used. The reaction mixture was stirred at 70 ^{o}C for 4h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(17)** (15 mg, white solid) was isolated in 52% yield. ^{1}H-NMR (400 MHz, Acetone-d6) δ (ppm) 9.51(s, 1H), 9.01 (s, 1H), 7.66 (d, *J* = 8.8 Hz, 1H), 7.61-7.54 (m, 1H), 7.16-7.11 (m, 2H), 6.55 (d, *J* = 2.8 Hz, 1H), 6.49 (dd, *J* = 8.8 Hz, 2.8Hz, 1H), 3.77 (s, 3H); ^{13}C-NMR (100 MHz, Acetone-d6) δ (ppm) 160.6 (dd, *J* = 249.3 Hz, 6.8 Hz), 159.8, 159.3, 150.3, 133.1 (t, *J* = 10.1 Hz), 123.9, 120.5, 115.8 (t, *J* = 20.8 Hz), 112.9-112.6 (m), 106.1, 103.8, 55.7; LRMS (ESI) calcd for $C_{14}H_{12}F_2NO_2$ $[M+H]^+$: 280.07, found 280.05



(18)

N-(4-bromo-2-hydroxyphenyl)-2,6-difluorobenzamide (18)

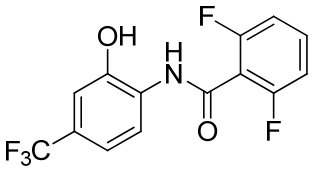
Following the general procedure Ⅰ, N-(4-bromophenyl)-2,6-difluorobenzamide (31.2 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^{o}C for 7h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(18)** (21 mg, white solid) was isolated in 64% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.89 (d, *J* = 8.6 Hz, 1H), 7.56-7.48 (m, 1H), 7.11-7.00 (m, 4H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.1 (dd, *J* = 249.3 Hz, 6.8 Hz), 161.2, 150.6, 133.4 (t, *J* = 10.1 Hz), 126.2, 124.8, 123.4, 119.5, 119.0, 116.0 (t, *J* = 20.8 Hz), 113.1-112.8 (m); LRMS (ESI) calcd for $C_{13}H_9ClF_2NO_2$ $[M+H]^+$: 327.97 found 327.98



(19)

2,6-difluoro-N-(4-fluoro-2-hydroxyphenyl)benzamide (19)

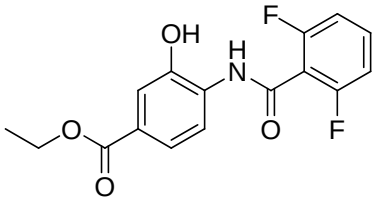
Following the general procedure Ⅰ, 2,6-difluoro-N-(4-fluorophenyl)benzamide (25.2 mg, 0.10 mmol), $K_2S_2O_8$ (54 mg, 0.20 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 50 ^oC for 20h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(19)** (17.0 mg, white solid) was isolated in 64% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.85-7.81 (m, 1H), 7.55-7.48 (m, 1H), 7.09 (t, *J* = 8.2Hz, 2H), 6.67-6.58 (m, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.0 (d, *J* = 241.4 Hz), 161.3, 161.1 (dd, *J* = 249.3 Hz, 6.8 Hz), 151.53 (d, *J* = 12.0 Hz), 133.3 (t, *J* = 10.1 Hz), 125.3 (d, *J* = 10.0 Hz), 123.0 (d, *J* = 3.0 Hz), 116.0 (t, *J* = 20.8 Hz), 113.0-112.8 (m), 106.6 (d, *J* = 22.4 Hz), 104.0 (d, *J* = 25.3 Hz); LRMS (ESI) calcd for $C_{13}H_9F_3NO_2$ $[M+H]^+$: 268.05, found 268.01



(20)

2,6-difluoro-N-(2-hydroxy-4-(trifluoromethyl)phenyl)benzamide (20)

Following the general procedure Ⅰ, 2,6-difluoro-N-(4-(trifluoromethyl)phenyl)benzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(20)** (23 mg, white solid) was isolated in 73% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 8.26 (d, *J* = 8.3 Hz, 1H), 7.57-7.49 (m, 1H), 7.18-7.08 (m, 4H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.2 (dd, *J* = 249.3 Hz, 6.8 Hz), 161.4, 149.3, 133.6 (t, *J* = 10.1 Hz), 130.4, 128.6 (q, *J* = 37.7 Hz), 125.5(q, *J* = 265.0 Hz), 123.1, 117.4-117.3 (m), 115.8 (t, *J* = 20.8 Hz), 113.1 (d, *J* = 5.3 Hz), 113.0-112.7 (m); LRMS (ESI) calcd for $C_{14}H_9F_5NO_2$ $[M+H]^+$: 318.05, found 318.03

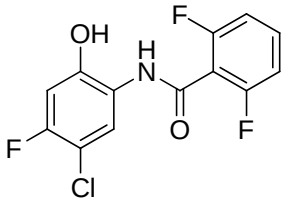


(21)

ethyl 4-(2,6-difluorobenzamido)-3-hydroxybenzoate (21)

Following the general procedure Ⅰ, ethyl 4-(2,6-difluorobenzamido)benzoate (35.8 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 22h. After completion of the reaction, the residue was purified by silical gel

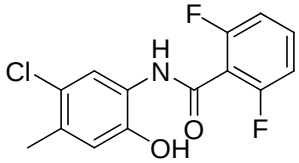
column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(21)** (20 mg, white solid) was isolated in 53% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 8.21 (d, *J* = 8.3 Hz, 1H), 7.57-7.49 (m, 3H), 7.08 (t, *J* = 8.2Hz, 2H), 4.34 (q, *J* = 7.1 Hz, 2H), 1.38 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 167.7, 161.21, 161.18 (dd, *J* = 249.3 Hz, 6.8 Hz), 148.6, 133.6 (t, *J* = 10.1 Hz), 131.5, 128.3, 122.3, 122.1, 116.9, 115.9 (t, *J* = 20.8 Hz), 113.1-112.9 (m), 62.1, 14.6; LRMS (ESI) calcd for $C_{16}H_{13}F_2NO_4$ $[M+H]^+$: 322.08, found 322.05



(22)

N-(5-chloro-4-fluoro-2-hydroxyphenyl)-2,6-difluorobenzamide (22)

Following the general procedure Ⅰ, N-(3-chloro-4-fluorophenyl)-2,6-difluorobenzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^{o}C for 17h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(22)** (21 mg, white solid) was isolated in 80% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 8.09 (d, *J* = 8.1 Hz, 1H), 7.56-7.48 (m, 1H), 7.09 (t, *J* = 8.2 Hz, 2H), 6.77 (d, *J* = 10.3 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 161.3, 161.1 (d, *J* = 249.3 Hz, 6.8 Hz), 156.5 (d, *J* = 243.6 Hz), 149.8 (d, *J* = 9.8 Hz), 133.5 (t, *J* = 10.1 Hz), 124.7, 123.9 (d, *J* = 3.3 Hz), 115.8 (t, *J* = 20.8 Hz), 113.1-112.8 (m), 110.8 (d, *J* = 18.5 Hz), 104.7 (d, *J* = 24.5 Hz,); LRMS (ESI) calcd for $C_{13}H_6ClF_3NO_2$ $[M-H]^-$: 301.01, 302.01, found 302.01

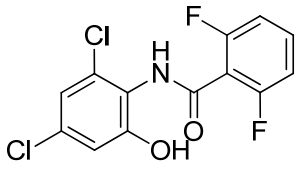


(23)

N-(5-chloro-2-hydroxy-4-methylphenyl)-2,6-difluorobenzamide (23)

Following the general procedure Ⅰ, N-(3-chloro-4-methylphenyl)-2,6-difluorobenzamide (28 mg, 0.10 mmol), $K_2S_2O_8$ (40.5 mg, 0.15 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^{o}C for 8h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(23)** (24 mg, white solid) was isolated in 80% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 10.07 (s, 1H), 10.06 (s, 1H), 7.97 (s, 1H), 7.56-7.52 (m, 1H), 7.18 (t, *J* = 8.0 Hz, 2H), 6.85 (s, 1H), 2.24 (s, 3H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 158.9 (d, *J* = 247.1 Hz, 7.9 Hz), 147.1, 132.0-131.7 (m), 124.5, 122.3, 122.1, 117.5, 115.3 (t, *J* = 22.0 Hz), 112.0-111.7 (m), 19.3; LRMS (ESI) calcd for $C_{14}H_{10}F_2NO_2$ $[M-H]^-$: 296.04,

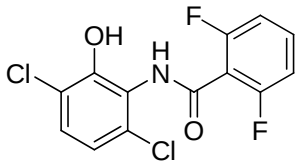
found 295.88



(24)

N-(2,4-dichloro-6-hydroxyphenyl)-2,6-difluorobenzamide (24)

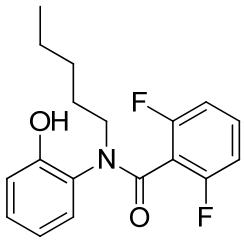
Following the general procedure Ⅰ, N-(2,4-dichlorophenyl)-2,6-difluorobenzamide (30 mg, 0.1 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 16h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(24)** (25 mg, white solid) was isolated in 79% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.50-7.46 (m, 1H), 7.05 (t, *J* = 8.1 Hz, 2H), 6.99 (d, *J* = 1.7 Hz, 1H), 6.87 (d, *J* = 1.7 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.2, 161.0 (dd, *J* = 249.3 Hz, 6.8 Hz), 156.6, 134.9, 134.7, 133.3 (t, *J* = 10.0 Hz), 122.2, 120.9, 116.0, 115.6 (t, *J* = 20.8 Hz), 113.0-112.9 (m); LRMS (ESI) calcd for $C_{13}H_6Cl_2F_2NO_2$ $[M-H]^-$: 315.98, found 315.83



(25)

N-(3,6-dichloro-2-hydroxyphenyl)-2,6-difluorobenzamide (25)

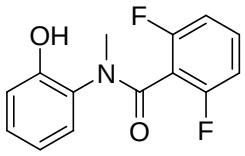
Following the general procedure Ⅰ, N-(2,5-dichlorophenyl)-2,6-difluorobenzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 90 ^oC for 16h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(25)** (17 mg, white solid) was isolated in 53% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.57-7.49 (m, 1H), 7.31 (d, *J* = 8.7 Hz, 1H), 7.10 (t, *J* = 8.1 Hz, 2H), 7.01 (d, *J* = 8.7 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.3, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 151.9, 133.5 (t, *J* = 10.1 Hz), 132.5, 130.0, 125.0, 121.8, 115.5 (t, *J* = 20.8 Hz), 113.0-112.8 (m); LRMS (ESI) calcd for $C_{13}H_8Cl_2F_2NO_2$ $[M+H]^+$: 317.98, found 318.00



(26)

2,6-difluoro-N-(2-hydroxyphenyl)-N-pentylbenzamide (26)

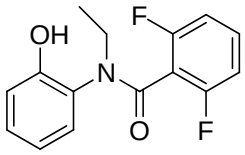
Following the general procedure Ⅰ, 2,6-difluoro-N-pentyl-N-phenylbenzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (54 mg, 0.2 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 26h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(26)** (17 mg, white solid) was isolated in 53% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 9.82 (s, 1H), 7.30-7.22 (m, 1H), 7.01-6.98 (m, 2H), 6.92-6.86 (m, 2H), 6.74 (d, *J* = 8.3 Hz, 1H), 6.63 (t, *J* = 7.5 Hz, 1H), 4.03-3.96 (m, 1H), 3.51-3.45 (m, 1H), 1.49-1.46 (m, 2H), 1.29-1.24 (m, 4H), 0.85 (t, *J* = 7.0 Hz, 3H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 161.1, 159.6, 159.0, 158.9, 157.1, 157.0, 156.5, 156.4, 153.5, 131.0 (t, *J* = 9.8 Hz), 129.2, 129.1, 127.2, 118.5, 116.6, 116.1, 115.3 (t, *J* = 23.2 Hz), 111.6-110.7 (m), 46.9, 28.4, 26.7, 21.8, 13.8; LRMS (ESI) calcd for $C_{18}H_{20}F_2NO_2$ $[M+H]^+$: 320.14, found 320.13



(27)

2,6-difluoro-N-(2-hydroxyphenyl)-N-methylbenzamide (27)

Following the general procedure Ⅰ, 2,6-difluoro-N-methyl-N-phenylbenzamide (25 mg, 0.10 mmol), $K_2S_2O_8$ (54 mg, 0.2 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(27)** (18.4 mg, white solid) was isolated in 70% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 9.85 (s, 1H), 7.32-7.24 (m, 1H), 7.05-6.98 (m, 2H), 6.91 (t, *J* = 8.2 Hz, 2H), 6.75 (d, *J* = 8.1 Hz, 1H), 6.63 (t, *J* = 7.5 Hz, 1H), 3.26 (s, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.5, 160.9, 160.2, 158.4, 157.7, 154.5, 132.4 (t, *J* = 10.1 Hz), 130.5, 130.2, 129.5, 120.0, 117.4, 116.3 (t, *J* = 20.8 Hz), 112.9-112.1 (m), 36.6; LRMS (ESI) calcd for $C_{14}H_{12}F_2NO_2$ $[M+H]^+$: 264.08, found 264.09

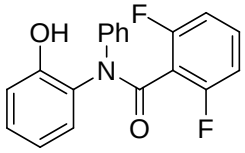


(28)

N-ethyl-2,6-difluoro-N-(2-hydroxyphenyl)benzamide (28)

Following the general procedure Ⅰ, N-ethyl-2,6-difluoro-N-phenylbenzamide (26 mg, 0.10 mmol), $K_2S_2O_8$ (54 mg, 0.2 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(28)** (19 mg, white solid) was isolated in 69% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 9.82 (s, 1H), 7.30-7.22 (m, 1H), 7.02-6.99 (m, 2H), 6.93-6.86 (m, 2H), 6.75 (d, *J* = 8.1 Hz, 1H), 6.64 (t, *J* = 7.5 Hz, 1H), 4.03-3.94 (m, 1H), 3.60-3.49 (m, 1H), 1.08 (t, *J*

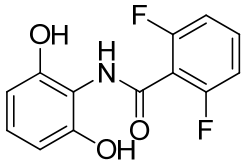
= 7.1 Hz, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 160.8, 159.7, 159.6, 159.0, 158.9, 157.2, 157.1, 156.6, 156.5, 153.6, 153.2, 131.0 (t, *J* = 10.1 Hz), 129.3, 127.1, 118.6, 116.1, 115.3 (t, *J* = 20.8 Hz), 111.6-110.8 (m), 42.2, 12.6; LRMS (ESI) calcd for $C_{15}H_{14}F_2NO_2$ $[M+H]^+$: 278.09, found 278.04



(29)

2,6-difluoro-N-(2-hydroxyphenyl)-N-phenylbenzamide (29)

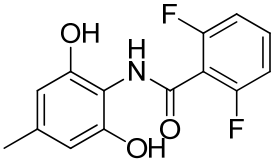
Following the general procedure Ⅰ, 2,6-difluoro-N,N-diphenylbenzamide (31 mg, 0.10 mmol), $K_2S_2O_8$ (54 mg, 0.2 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 5.5h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(29)** (25 mg, white solid) was isolated in 74% yield. LRMS (ESI) calcd for $C_{19}H_{14}F_2NO_2$ $[M+H]^+$: 326.09, found 326.11



(30)

N-(2,6-dihydroxyphenyl)-2,6-difluorobenzamide (30)

Following the general procedure Ⅰ, 2,6-difluoro-N-phenylbenzamide (22.3 mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 16h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(30)** (12 mg, white solid) was isolated in 45% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.56-7.49 (m, 1H), 7.09 (t, *J* = 8.2 Hz, 2H), 6.98 (t, *J* = 8.3 Hz, 1H), 6.46 (d, *J* = 8.1 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.2, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 133.5 (t, *J* = 10.1 Hz), 128.8, 115.3 (t, *J* = 20.8 Hz), 114.5, 113.0-112.8 (m), 109.1; LRMS (ESI) calcd for $C_{13}H_{10}F_2NO_3$ $[M+H]^+$: 266.06, found 266.02

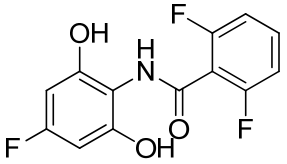


(31)

N-(2,6-dihydroxy-4-methylphenyl)-2,6-difluorobenzamide (31)

Following the general procedure Ⅰ, 2,6-difluoro-N-(p-tolyl)benzamide (24.7 mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), 5% $[(C_6H_5)_3P]_3RuCl_2$ (4.5 mg, 0.005 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 70 °C for 33h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(31)** (22

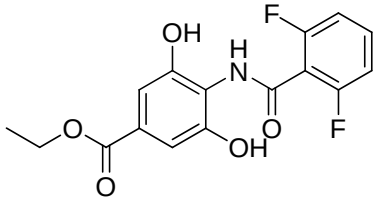
mg, white solid) was isolated in 80% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.54-7.50 (m, 1H), 7.08 (t, *J* = 8.2 Hz, 2H), 6.30 (s, 2H), 2,21 (s, 3H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.5, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 152.7, 139.3, 133.5 (t, *J* = 10.1 Hz), 115.4 (t, *J* = 20.8 Hz), 113.0-112.8 (m), 112.0, 109.8, 108.3, 21.4; LRMS (ESI) calcd for $C_{14}H_{12}F_2NO_3$ $[M+H]^+$: 280.07, found 280.05



(32)

2,6-difluoro-N-(4-fluoro-2,6-dihydroxyphenyl)benzamide (32)

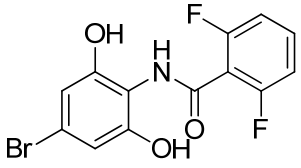
Following the general procedure Ⅰ, 2,6-difluoro-N-(4-fluorophenyl)benzamide (25.2 mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^{o}C for 14h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(32)** (19 mg, white solid) was isolated in 67% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.54-7.50 (m, 1H), 7.08 (t, *J* = 8.2 Hz, 2H), 6.20 (d, *J* = 10.0 Hz, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 163.7 (d, *J* = 256.4 Hz), 162.5, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 154.8 (d, *J* = 14.6 Hz), 133.4 (t, *J* = 10.1 Hz), 129.8, 115.5 (t, *J* = 20.8 Hz), 113.0-112.7 (m), 110.5 (d, *J* = 3.7 Hz), 96.0 (d, *J* = 25 Hz), 94.8 (d, *J* = 25 Hz); LRMS (ESI) calcd for $C_{13}H_9F_3NO_3$ $[M+H]^+$: 284.05 found 284.00



(33)

ethyl 4-(2,6-difluorobenzamido)-3,5-dihydroxybenzoate (33)

Following the general procedure Ⅰ, ethyl 4-(2,6-difluorobenzamido)benzoate (35.8 mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^{o}C for 36h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(33)** (21 mg, white solid) was isolated in 62% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 9.88 (s, 1H), 9.79 (s, 2H), 7.55-7.52 (m, 1H), 7.18 (t, *J* = 8.2Hz, 2H), 7.04 (s, 2H), 4.27 (q, *J* = 6.72 Hz, 2H), 1.30 (t, *J* = 7.04 Hz, 3H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 165.5, 159.1 (dd, *J* = 249.3 Hz, 6.8 Hz), 158.5, 153.6, 131.7 (t, *J* = 9.0 Hz), 128.6, 116.8, 115.4 (t, *J* = 22.1 Hz), 112.0-111.7 (m), 107.6, 60.7, 14.2; LRMS (ESI) calcd for $C_{16}H_{14}F_2NO_5$ $[M+H]^+$: 338.08 found 338.08



(34)

N-(4-bromo-2,6-dihydroxyphenyl)-2,6-difluorobenzamide (34)

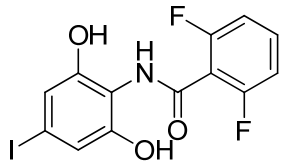
1) small-scale

Following the general procedure Ⅰ, N-(4-bromophenyl)-2,6-difluorobenzamide (31.2 mg, 0.10 mmol), $K_2S_2O_8$ (108 mg, 0.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(34)** (27 mg, white solid) was isolated in 78% yield.

2) big-scale

Following the general procedure Ⅰ, N-(4-bromophenyl)-2,6-difluorobenzamide (1.284 g, 4 mmol), $K_2S_2O_8$ (2.7 g, 10 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (49 mg, 0.0025 mmol), 15ml TFA and 5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 17h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(34)** (0.92 g, white solid) was isolated in 67.4% yield.

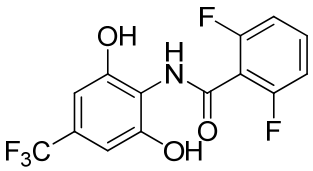
^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.55-7.48 (m, 1H), 7.08 (t, *J* = 8.2 Hz, 2H), 6.62 (s, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.3, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 156.3, 154.4, 133.5 (t, *J* = 10.1 Hz), 121.4, 115.3 (t, *J* = 20.8 Hz), 113.6, 113.0-112.7 (m), 112.0, 110.8; LRMS (ESI) calcd for $C_{13}H_9BrF_2NO_3$ $[M+H]^+$: 343.97 found 343.98



(35)

N-(2,6-dihydroxy-4-iodophenyl)-2,6-difluorobenzamide (35)

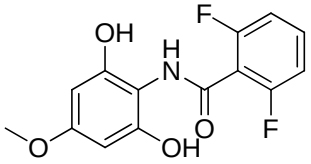
Following the general procedure Ⅰ, 2,6-difluoro-N-(4-iodophenyl)benzamide (36.7 mg, 0.10 mmol), $K_2S_2O_8$ (108 mg, 0.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 ^oC for 13h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 10:1). Finally, compound **(35)** (21 mg, white solid) was isolated in 54% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.55-7.48 (m, 1H), 7.08 (t, *J* = 8.2 Hz, 2H), 6.82 (s, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.3, 161.2 (dd, *J* = 249.3 Hz, 6.8 Hz), 154.2, 133.5 (t, *J* = 10.1 Hz), 118.2, 115.3 (t, *J* = 20.8 Hz), 114.5, 113.0-112.7 (m), 91.9; LRMS (ESI) calcd for $C_{13}H_9F_2INO_3$ $[M+H]^+$: 391.95 found 391.95



(36)

N-(2,6-dihydroxy-4-(trifluoromethyl)phenyl)-2,6-difluorobenzamide (36)

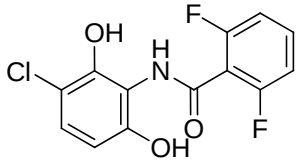
Following the general procedure Ⅰ, 2,6-difluoro-N-(4-(trifluoromethyl)phenyl)benzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 14h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(36)** (17 mg, white solid) was isolated in 51% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.57-7.50 (m, 1H), 7.10 (t, *J* = 8.2 Hz, 2H), 6.71 (s, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.4, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 154.1, 133.6 (t, *J* = 10.1 Hz), 132.3, 130.9, 130.6, 129.8, 126.7, 124.0, 117.3, 115.3 (t, *J* = 20.8 Hz), 113.0-112.8 (m), 105.5 (d, *J* = 3.7 Hz); LRMS (ESI) calcd for $C_{14}H_9F_5NO_3$ $[M+H]^+$: 334.04 found 334.02



(37)

N-(2,6-dihydroxy-4-methoxyphenyl)-2,6-difluorobenzamide (37)

Following the general procedure Ⅰ, 2,6-difluoro-N-(4-methoxyphenyl)benzamide (26.4 mg, 0.10 mmol), $K_2S_2O_8$ (81 mg, 0.3 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 8.5h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(37)** (16 mg, white solid) was isolated in 53% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.55-7.48 (m, 1H), 7.08 (t, *J* = 8.2 Hz, 2H), 6.01 (s, 2H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 162.5, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 161.2, 154.0, 133.4 (t, *J* = 10.1 Hz), 115.5 (t, *J* = 20.8 Hz), 113.0-112.7 (m), 107.9, 95.0, 93.6, 55.7, 55.5; LRMS (ESI) calcd for $C_{14}H_{12}F_2NO_4$ $[M+H]^+$: 296.07 found 296.08

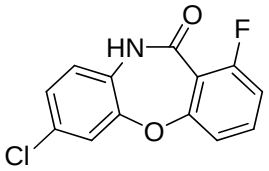


(38)

N-(3-chloro-2,6-dihydroxyphenyl)-2,6-difluorobenzamide (38)

Following the general procedure Ⅰ, N-(2,4-dichlorophenyl)-2,6-difluorobenzamide (30 mg, 0.10 mmol), $K_2S_2O_8$ (32.4 mg, 0.12 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (1.5 mg, 0.0025 mmol), 1.5ml TFA and 0.5ml TFAA were used. The reaction mixture was stirred at 80 °C for 16h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(38)** (25 mg, white solid) was isolated in 30% yield. ^{1}H-NMR (400 MHz, CD_3OD) δ (ppm) 7.50-7.46 (m, 1H), 7.05 (t, *J* = 8.1 Hz, 2H), 6.99 (d, *J* = 1.7 Hz, 1H), 6.87 (d, *J* = 1.7 Hz, 1H); ^{13}C-NMR (100 MHz, CD_3OD) δ (ppm) 169.3, 162.5, 161.3 (dd, *J* = 249.3 Hz, 6.8 Hz), 152.3, 149.1, 133.6 (t, *J* = 10.0 Hz), 132.3, 129.8, 128.7, 126.2, 122.9, 117.3, 116.0, 115.2, 113.9, 113.0-112.8 (m), 109.1; LRMS (ESI) calcd

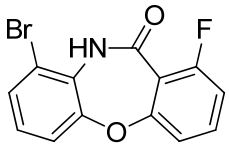
for $C_{13}H_8Cl_2F_2NO_2$ $[M+H]^+$: 300.02, found 299.94



(39)

7-chloro-1-fluorodibenzo[b,f][1,4]oxazepin-11(10H)-one (39)

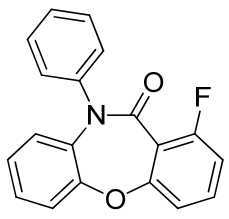
Following the general procedure II, N-(4-chloro-2-hydroxyphenyl)-2,6-difluorobenzamide (28.3 mg, 0.1 mmol), K_2CO_3 (27.6 mg, 0.2 mmol) and 1.5 ml DMF were used. The reaction mixture was stirred at 100 °C for 3h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(39)** (23 mg, white solid) was isolated in 90% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 11.08 (s, 1H), 7.72-7.68 (m, 2H), 7.52 (d, *J* = 2.3 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 7.29 (dd, *J* = 8.6 Hz, 2.3Hz, 1H), 7.17 (d, *J* = 8.6 Hz, 1H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 164.1, 157.0, 150.34, 134.17, 130.6, 130.0, 129.8, 127.1, 126.2, 122.88, 122.87, 121.5; LRMS (ESI) calcd for $C_{13}H_8ClFNO_2$ $[M+H]^+$: 264.01 found 264.05



(40)

9-bromo-1-fluorodibenzo[b,f][1,4]oxazepin-11(10H)-one (40)

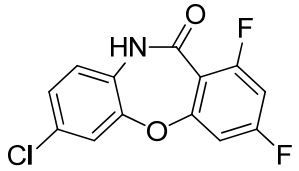
Following the general procedure II, N-(2-bromo-6-hydroxyphenyl)-2,6-difluorobenzamide (32.7 mg, 0.1 mmol), K_2CO_3 (27.6 mg, 0.2 mmol) and 1.5 ml acetone were used. The reaction mixture was stirred at 100 °C for 8h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(40)** (29 mg, white solid) was isolated in 95% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.70 (s, 1H), 7.49-7.41 (m, 2H), 7.23 (d, *J* = 8.1 Hz, 1H), 7.07 (d, *J* = 8.2 Hz, 1H), 7.02 (t, *J* = 8.4 Hz, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 162.2 (d, *J* = 261.0 Hz), 161.1 (d, *J* = 3.0 Hz), 160.5, (d, *J* = 4.0 Hz), 152.1, 134.13, 134.02, 130.07, 129.71, 126.54, 121.1, 116.5, 116.46, 115.04, 115.0, 114.9, 114.3, 114.1; LRMS (ESI) calcd for $C_{13}H_8BrFNO_2$ $[M+H]^+$: 307.96 found 308.15



(41)

9-bromo-1-fluoro-10-phenyldibenzo[b,f][1,4]oxazepin-11(10H)-one (41)

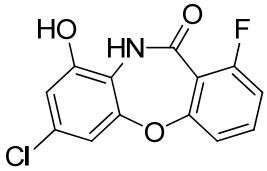
Following the general procedure II, 2,6-difluoro-N-(2-hydroxyphenyl)-N-phenylbenzamide (32.5 mg, 0.1 mmol), K_2CO_3 (27.6 mg, 0.2 mmol) and 1.5 ml acetone were used. The reaction mixture was stirred at 100 °C for 6h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(41)** (27 mg, white solid) was isolated in 87% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.48-7.34 (m, 6H), 7.29-7.25 (m, 1H), 7.13-6.95 (m, 4H), 6.84 (d, *J* = 8.0 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 163.5, 162.24, 162.20, 162.15, 162.13, 160.9, 154.8, 141.8, 135.5, 133.2, 133.1, 129.6, 128.9, 127.9, 126.7, 126.2, 126.0, 121.6, 116.7, 116.6, 115.7, 115.6, 113.9, 113.7, ; LRMS (ESI) calcd for $C_{19}H_{12}FNO_2$ $[M+H]^+$: 306.09 found 306.12



(42)

7-chloro-1,3-difluorodibenzo[b,f][1,4]oxazepin-11(10H)-one (42)

Following the general procedure II, N-(4-chloro-2-hydroxyphenyl)-2,4,6-trifluorobenzamide (30.1 mg, 0.1 mmol), K_2CO_3 (27.6 mg, 0.2 mmol) and 1.5 ml acetone were used. The reaction mixture was stirred at 100 °C for 10h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(42)** (23 mg, white solid) was isolated in 85% yield.^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 11.75 (s, 1H), 7.53 (d, *J* = 1.9 Hz, 1H), 7.32-7.27 (m, 3H), 7.15 (d, *J* = 8.6 Hz, 1H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 165.7, 163.6-163.2 (m), 161.4-160.8 (m), 151.4, 130.3, 129.1, 127.1, 123.7, 122.1, 112.9-112.7 (m), 105.7-105.4 (m), 103.9-103.4 (m); LRMS (ESI) calcd for $C_{13}H_6ClFNO_2$ $[M-H]^-$: 280.01 found 279.85

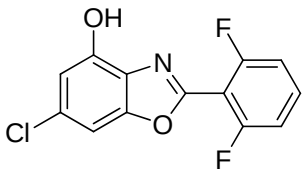


(43)

7-chloro-1-fluoro-9-hydroxydibenzo[b,f][1,4]oxazepin-11(10H)-one (43)

Following the general procedure II, N-(4-chloro-2,6-dihydroxyphenyl)-2,6-difluorobenzamide (30.0 mg, 0.1 mmol), NaOH (16 mg, 0.4 mmol) and 1.5 ml DMF were used. The reaction mixture was stirred at 100 °C for 2h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 1:1). Finally, compound **(43)** (23 mg, yellow solid) was isolated in 82% yield.^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 10.7 (s, 1H), 9.9 (s, 1H), 7.62-7.56 (m, 1H), 7.25-7.12 (m, 2H), 6.97 (d, *J* = 1.2 Hz, 1H), 6.76 (s, 1H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 166.9, 162.0, 161.4, 161.3, 160.2, 160.1, 159.4, 153.6, 150.6, 133.9, 133.8, 131.6,

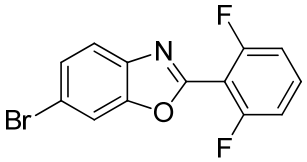
131.4, 128.8, 128.6, 118.2, 116.6, 116.5, 115.5, 115.4, 113.9, 113.7, 112.8, 111.8, 111.6; LRMS (ESI) calcd for $C_{13}H_8ClFNO_3$ $[M+H]^+$: 280.01 found 279.98



(44)

6-chloro-2-(2,6-difluorophenyl)benzo[d]oxazol-4-ol (44)

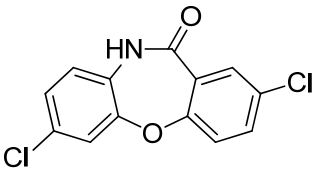
Following the general procedure III, N-(4-chloro-2,6-dihydroxyphenyl)-2,6-difluorobenzamide (30.0 mg, 0.1 mmol), *p*-TsOH.H_2O (95 mg, 0.5 mmol) and 2ml of p-xylene were used. The reaction mixture was stirred at 140 ^oC for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(44)** (25 mg, white solid) was isolated in 91% yield.^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 11.08 (s, 1H), 7.79-7.71 (m, 1H), 7.44-7.37 (m, 3H), 6.86 (d, *J* = 1.1 Hz, 1H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 160.2, 152.5, 151.8, 150.1, 134.4 (t, *J* = 10.6 Hz), 130.9, 129.1, 113.0-112.7 (m), 111.1, 105.1 (t, *J* = 16.0 Hz), 102.3; LRMS (ESI) calcd for $C_{13}H_5ClF_2NO_2$ $[M-H]^-$: 280.01 found 279.99



(45)

6-bromo-2-(2,6-difluorophenyl)benzo[d]oxazole (45)

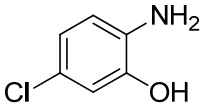
Following the general procedure III, N-(4-bromo-2-hydroxyphenyl)-2,6-difluorobenzamide (32.7 mg, 0.1 mmol), *p*-TsOH.H_2O (95 mg, 0.5 mmol) and 2ml of p-xylene were used. The reaction mixture was stirred at 140 ^oC for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(45)** (25 mg, white solid) was isolated in 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.82 (s, 1H), 7.73 (d, *J* = 8.5 Hz, 1H), 7.55-7.48 (m, 2H), 7.11 (t, *J* = 8.6 Hz, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 161.4 (dd, *J* = 257.9 Hz, 5.3 Hz), 155.5, 151.1, 140.7, 133.4 (t, *J* = 10.7 Hz), 128.4, 121.7, 119.1, 114.5, 112.8-112.5 (m), 106.1 (t, *J* = 14.6 Hz); LRMS (ESI) calcd for $C_{13}H_6BrF_2NO_2$ $[M+H]^+$: 309.96 found 309.97



(46)

2,7-dichlorodibenzo[b,f][1,4]oxazepin-11(10H)-one (46)

Following the general procedure II, 5-chloro-N-(4-chloro-2-hydroxyphenyl)-2-fluorobenzamide (30.0 mg, 0.1 mmol), K_2CO_3 (27.6 mg, 0.2 mmol) and 1.5 ml DMF were used. The reaction mixture was stirred at 100 °C for 3h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(46)** (26 mg, white solid) was isolated in 92% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 10.75 (s, 1H), 7.72-7.68 (m, 2H), 7.52 (d, *J* = 2.3 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 7.29 (dd, *J* = 8.6 Hz, 2.3Hz, 1H), 7.17 (d, *J* = 8.6 Hz, 1H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 162.1, 161.5, 159.7, 159.6, 159.5, 151.4, 134.2, 134.1, 131.5, 130.0, 128.6, 126.3, 123.1, 121.5, 116.7, 116.6, 115.2, 115.1, 114.1, 113.9; LRMS (ESI) calcd for $C_{13}H_8Cl_2NO_2$ $[M+H]^+$: 279.99 found 280.01



(47)

2-amino-5-chlorophenol (47)

Following the general procedure method A, N-(4-chloro-2-hydroxyphenyl)-2,6-difluorobenzamide (28.3 mg, 0.1 mmol) and 2ml of $NH_2NH_2.H_2O$ were used. The reaction mixture was stirred at 100 °C for 3h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(47)** (12 mg, yellow solid) was isolated in 85% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 9.44 (s, 1H), 6.63 (s, 1H), 6.55 (s, 2H), 6.64 (s, 2H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 144.8, 135.8, 118.9, 118.8, 114.7, 114.0; LRMS (ESI) calcd for C_6H_7ClNO $[M+H]^+$: 144.01 found 143.95

(48)

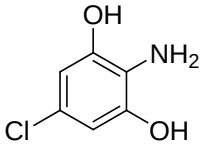
2-amino-3-bromophenol (48)

Following the general procedure method A, N-(2-bromo-6-hydroxyphenyl)-2,6-difluorobenzamide (900 mg, 2.75 mmol) and 4ml of $NH_2NH_2.H_2O$ were used. The reaction mixture was stirred at 100 °C for 4h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 5:1). Finally, compound **(48)** (465 mg, white solid) was isolated in 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.04 (dd, *J* = 8.1 Hz, 1.0 Hz, 1H), 6.67 (dd, *J* = 7.9 Hz, 1.0 Hz, 1H), 6.53 (t, *J* = 8.0 Hz, 1H), 4.26 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 144.1, 133.7, 125.0, 119.2, 114.0, 110.6; LRMS (ESI) calcd for C_6H_7ClNO $[M+H]^+$: 187.96 found 187.93

(49)

2-amino-3-(trifluoromethyl)phenol

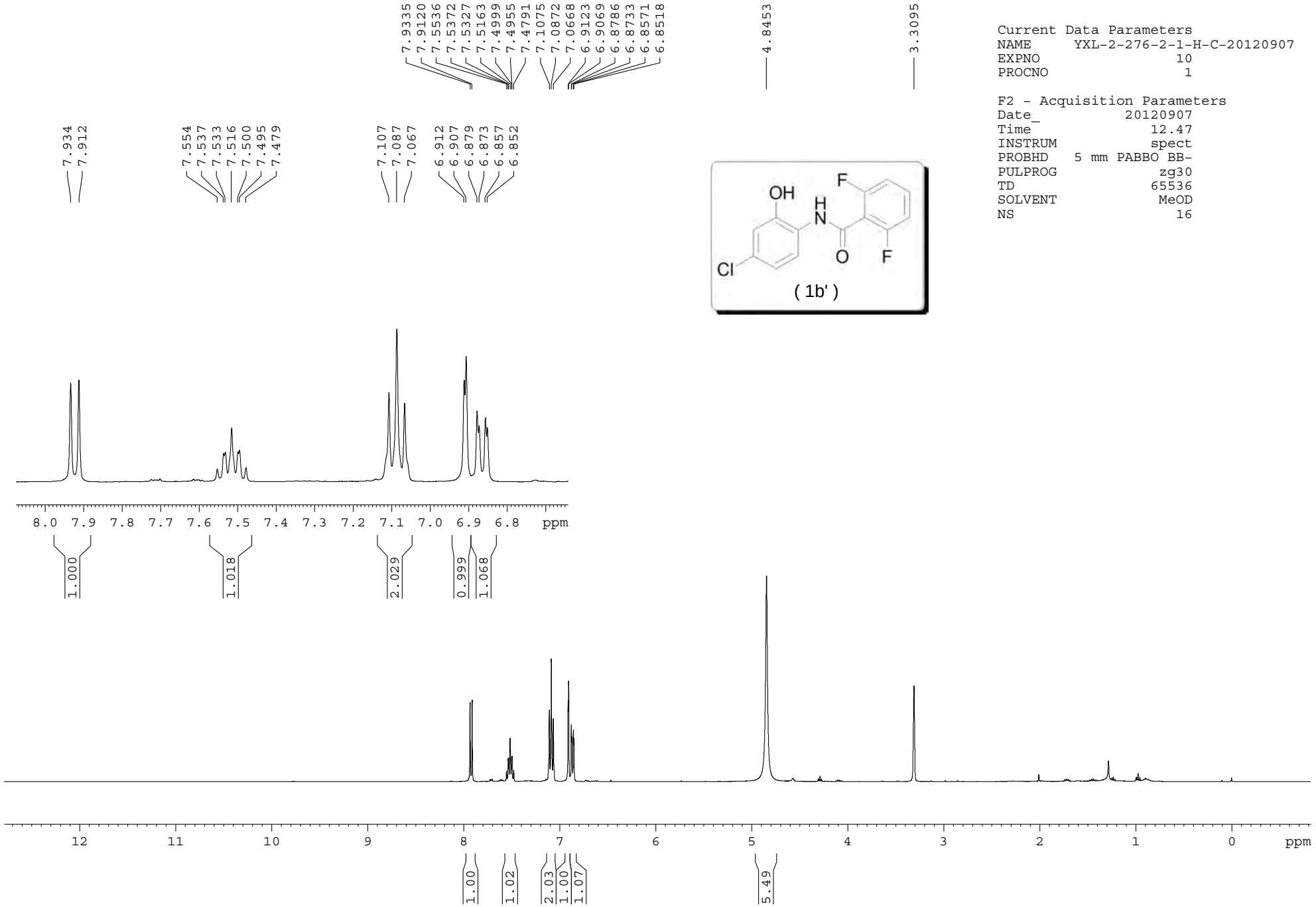
Following the general procedure method A, 2,6-difluoro-N-(2-hydroxy-6-(trifluoromethyl)phenyl)benzamide (380 mg, 1.2 mmol) and 5 ml of $NH_2NH_2.H_2O$ were used. The reaction mixture was stirred at 100 ^oC for 4 h. After workup, the crude NMR showed the convertion ratio was 100%. ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.04 (d, *J* = 8.0 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.64 (t, *J* = 7.9 Hz, 2H), 5.02 (s, 3H)



(50)

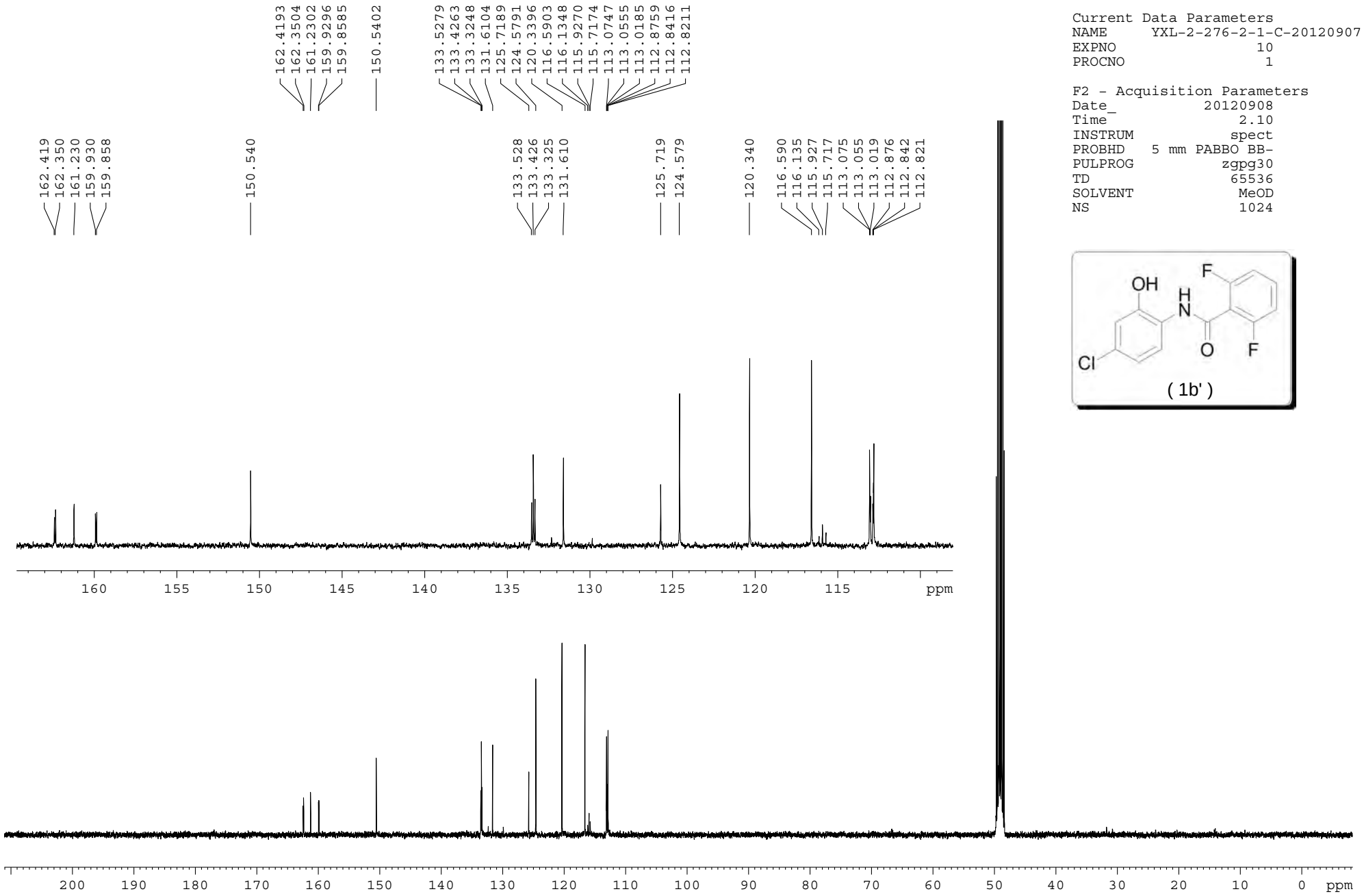
2-amino-5-chlorobenzene-1,3-diol (50)

Following the general procedure method A, N-(4-chloro-2,6-dihydroxyphenyl)-2,6-difluorobenzamide (210 mg, 0.7 mmol) and 4ml of $NH_2NH_2.H_2O$ were used. The reaction mixture was stirred at 100 ^oC for 4h. After completion of the reaction, the residue was purified by silical gel column chromatography (petroleum ether: ethyl acetate = 1:1). Finally, compound **(50)** (95mg, yellow solid) was isolated in 86% yield. ^{1}H-NMR (400 MHz, DMSO-d6) δ (ppm) 9.27 (s, 2H), 6.26 (s, 2H), 4.10 (s, 2H); ^{13}C-NMR (100 MHz, DMSO-d6) δ (ppm) 145.2, 123.0, 118.9, 106.6; LRMS (ESI) calcd for $C_6H_6ClNO_2$ $[M-H]^-$: 158.01 found 157.97



Current Data Parameters
NAME YXL-2-276-2-1-H-C-20120907
EXPNO 10
PROCNO 1

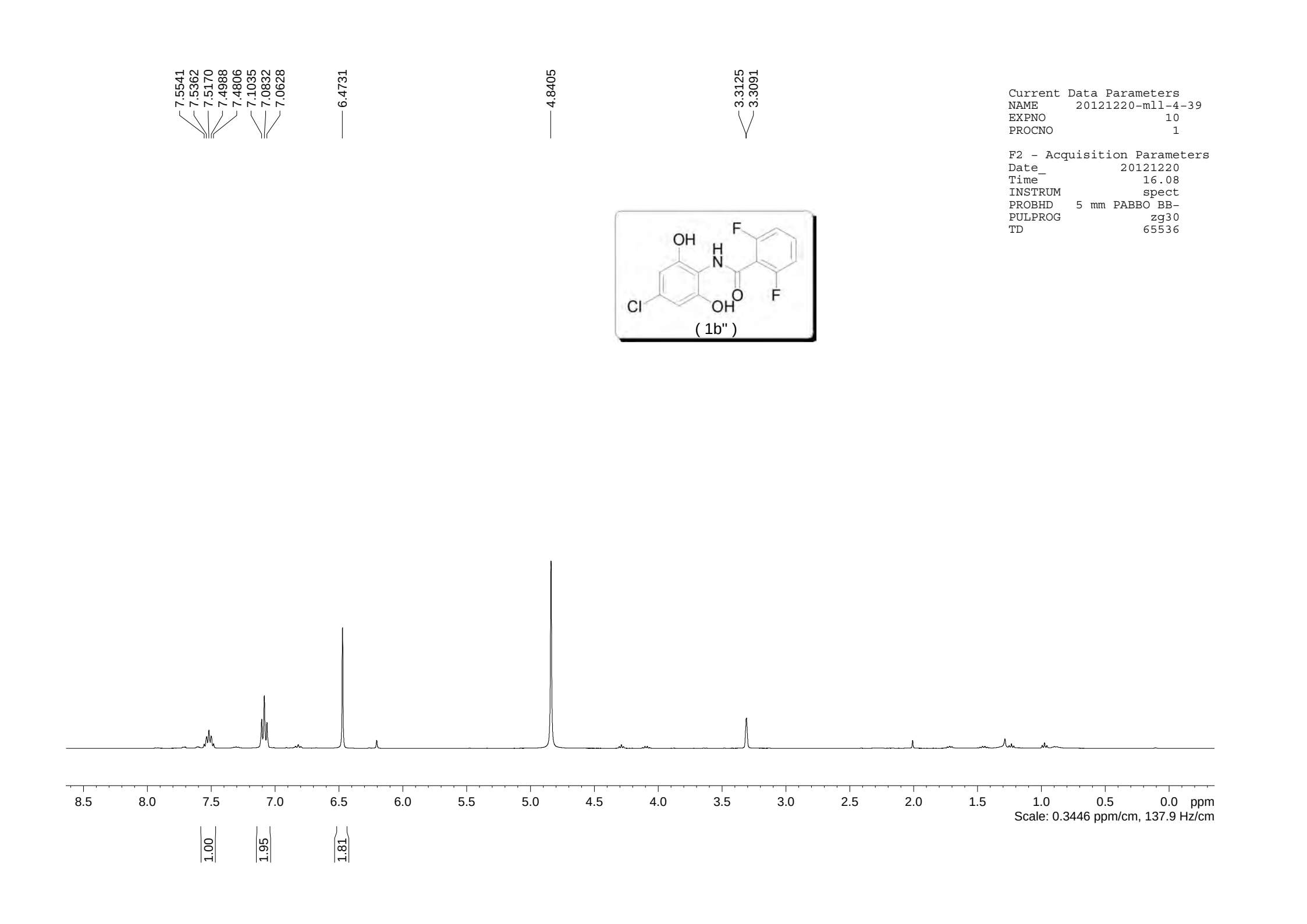
F2 - Acquisition Parameters
Date_ 20120907
Time 12.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16

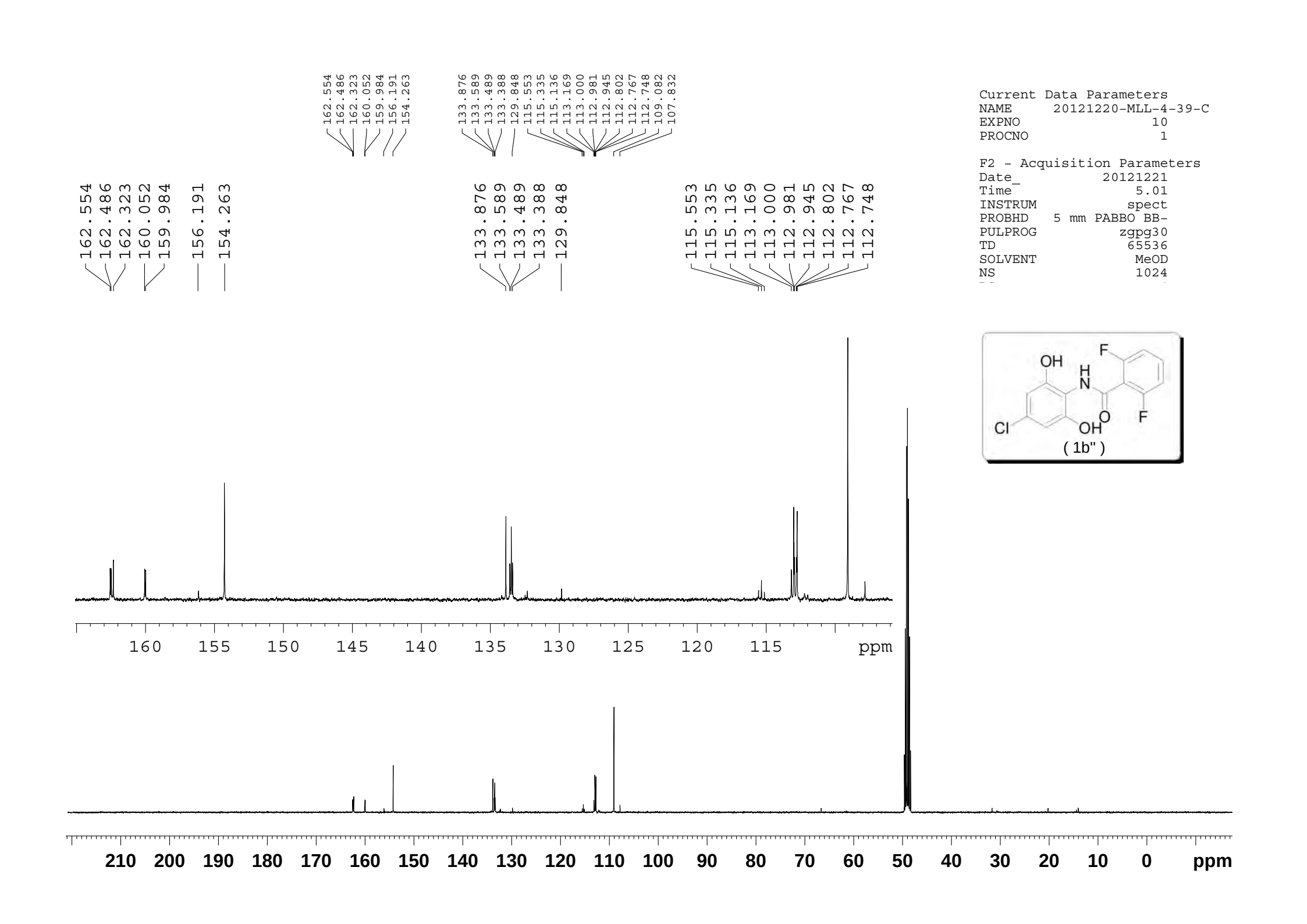


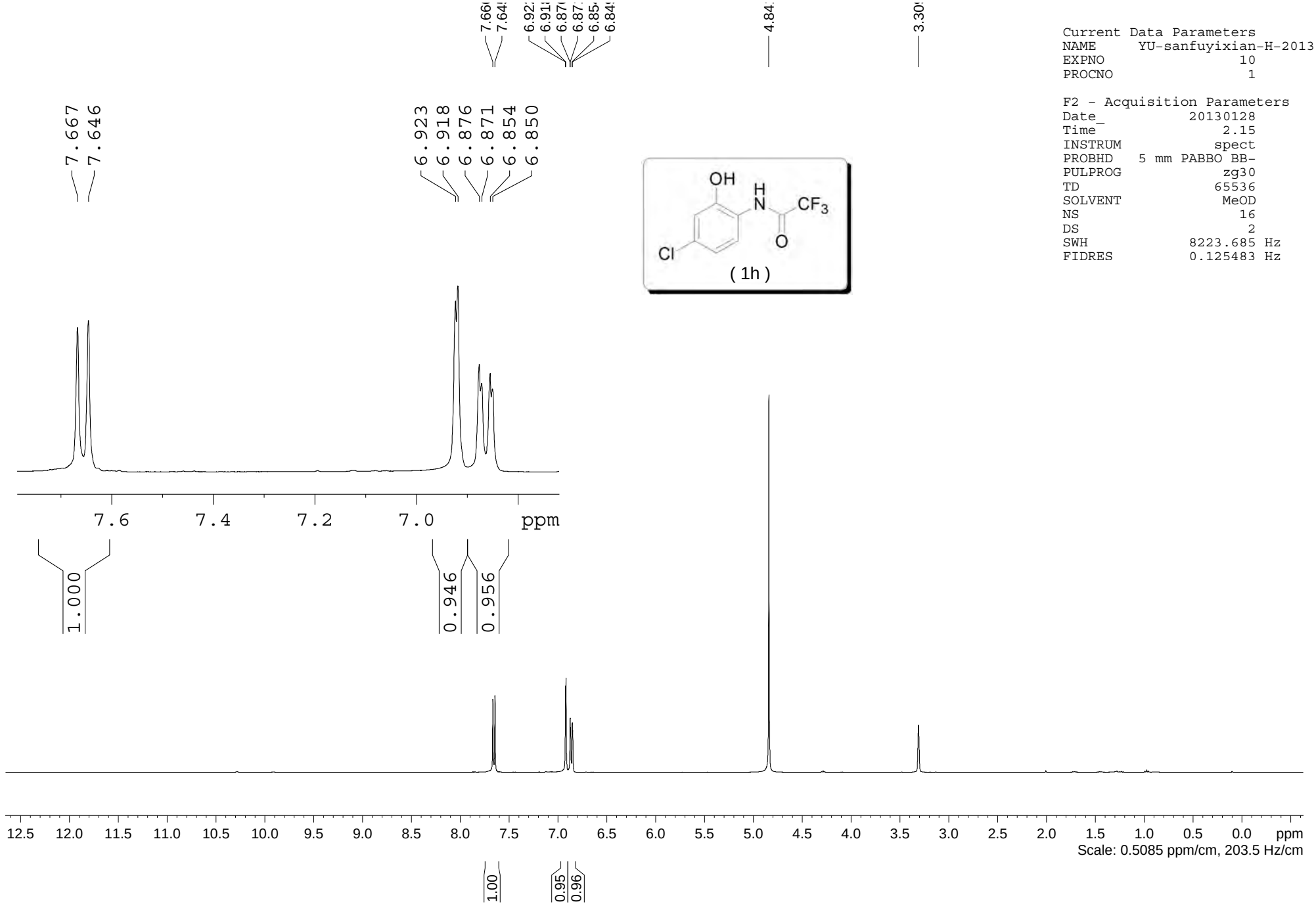
Current Data Parameters
NAME YXL-2-276-2-1-C-20120907
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120908
Time 2.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024

(1b')

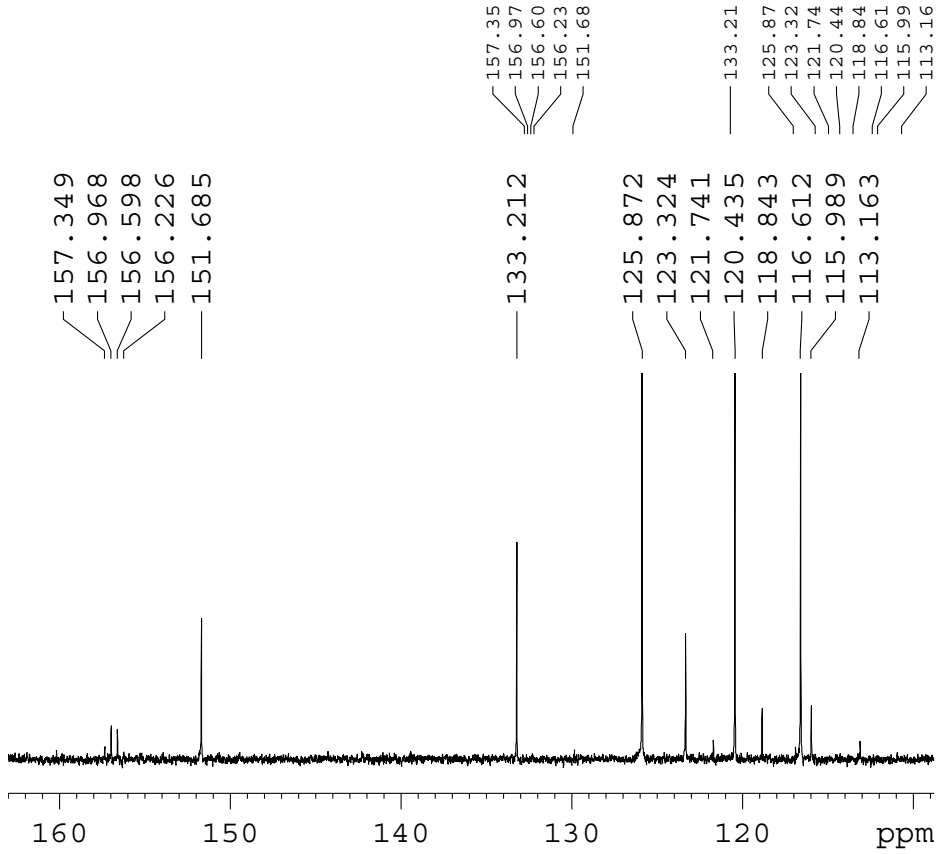

7.5541
7.5362
7.5170
7.4988
7.4806
7.1035
7.0832
7.0628
6.4731
4.8405
3.3125
3.3091
Current Data Parameters
NAME 20121220-mll-4-39
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121220
Time 16.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
OH
H
N
F
O
F
Cl
OH
(1b")
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.3446 ppm/cm, 137.9 Hz/cm
1.00
1.95
1.81

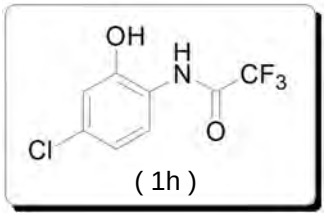

162.554
162.486
162.323
160.052
159.984
156.191
154.263
133.876
133.589
133.489
133.388
129.848
115.553
115.335
115.136
113.169
113.000
112.981
112.945
112.802
112.767
112.748
109.082
107.832
Current Data Parameters
NAME 20121220-MLL-4-39-C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121221
Time 5.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
OH
F
H
N
O
F
Cl
OH
(1b")
160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



Current Data Parameters
NAME YU-sanfuyixian-H-20130
EXPNO 10
PROCNO 1

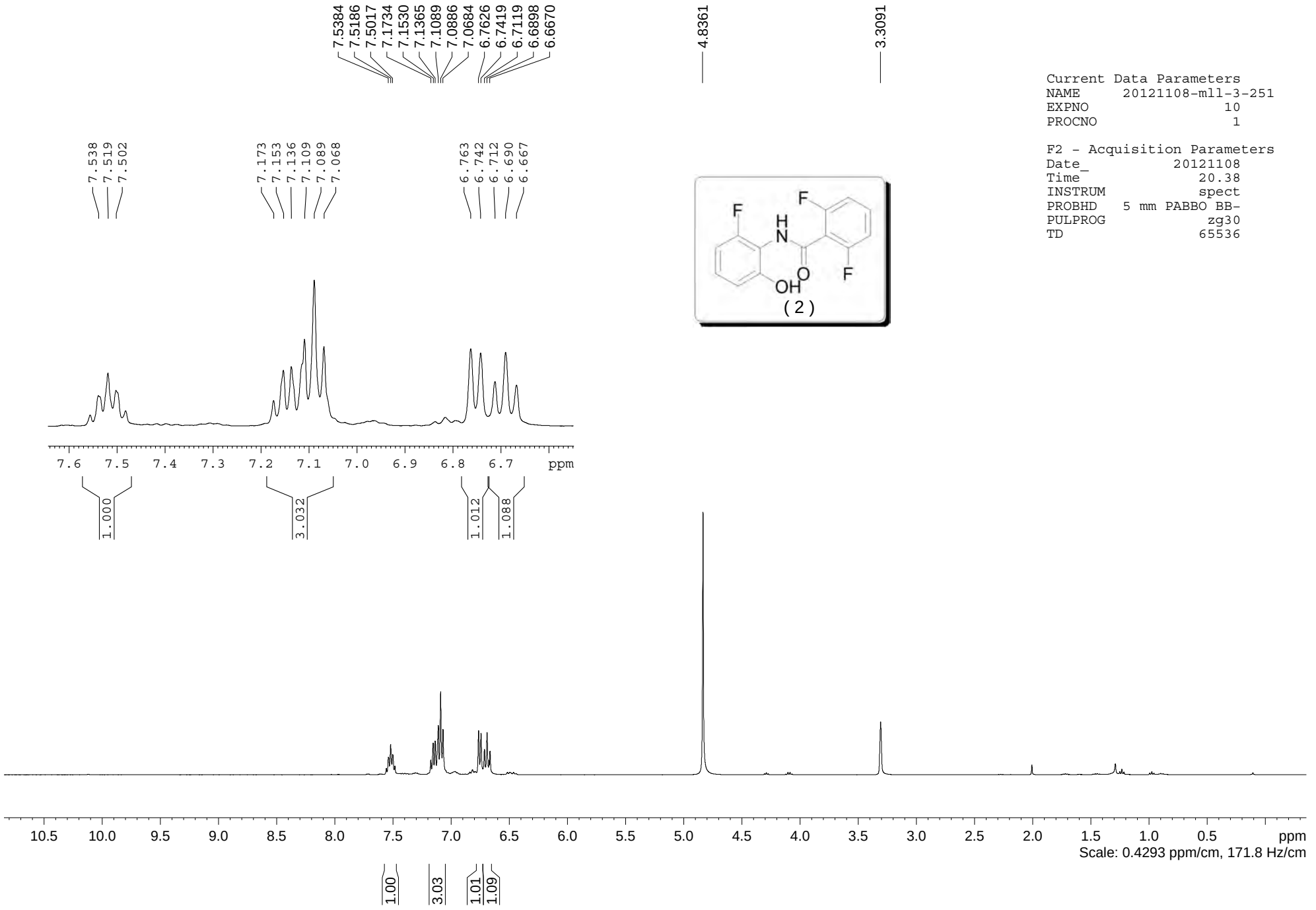
F2 - Acquisition Parameters
Date_ 20130128
Time 2.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz





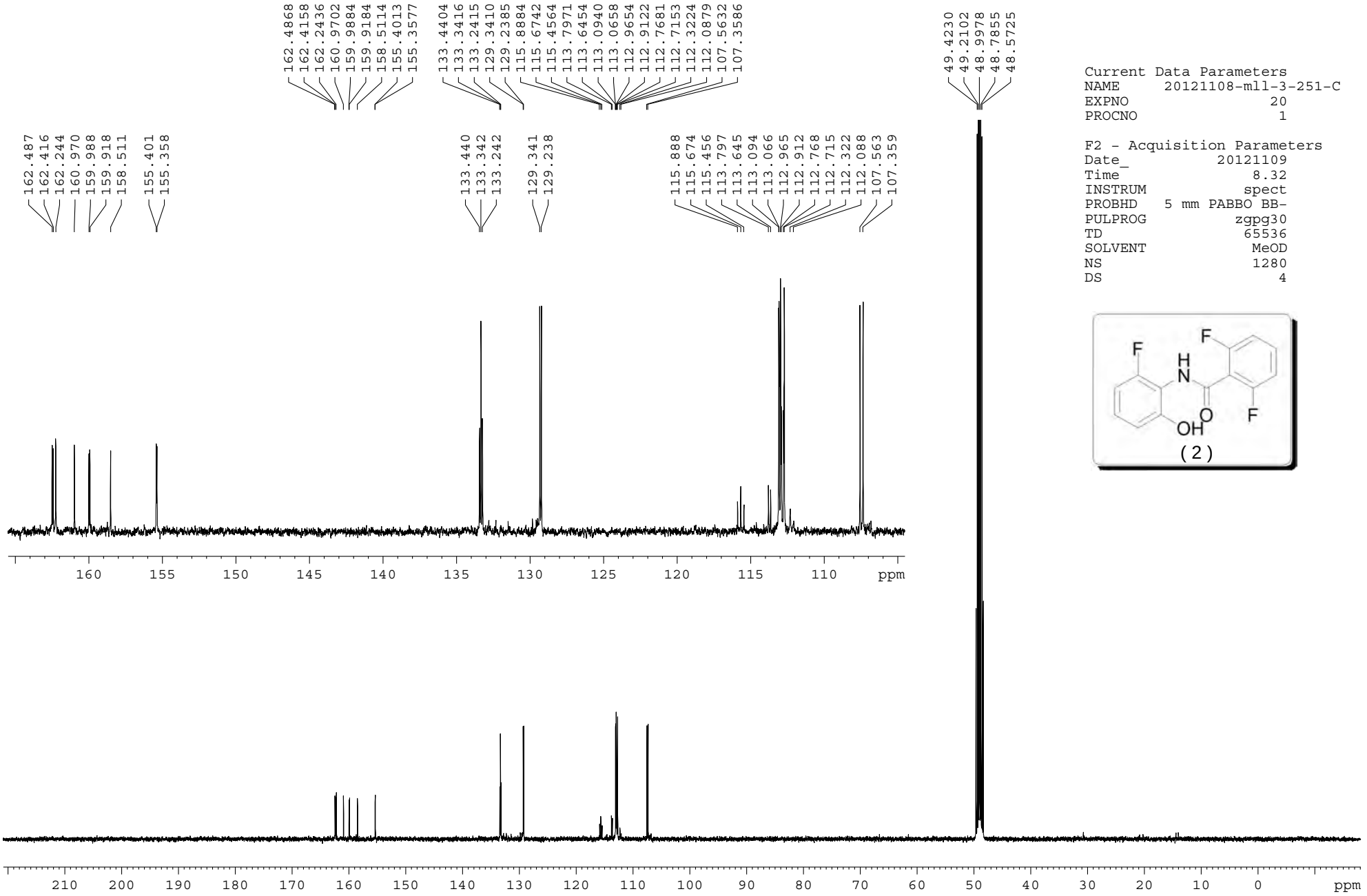
(1h)

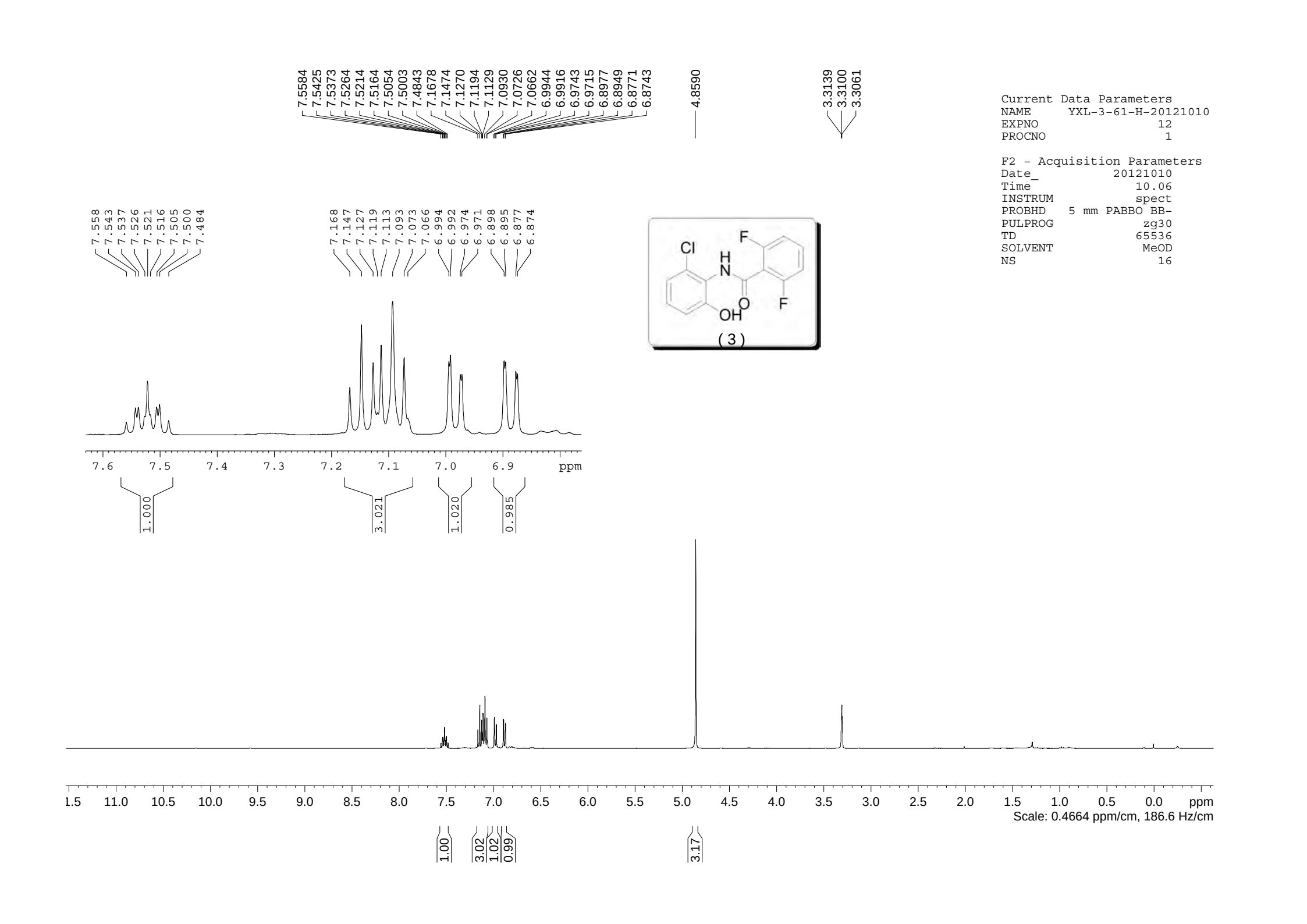

```
Current Data Parameters
NAME      YU-sanfuyixian-C-20130127
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_            20130128
Time                3.11
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              MeOD
NS                    950
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                 209.25
DW                 20.800 usec
DE                  6.50 usec
TE        0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.47 usec
PLW1         50.00299835 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00199986 W
PLW12         0.37380001 W
PLW13         0.30276999 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6126298 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

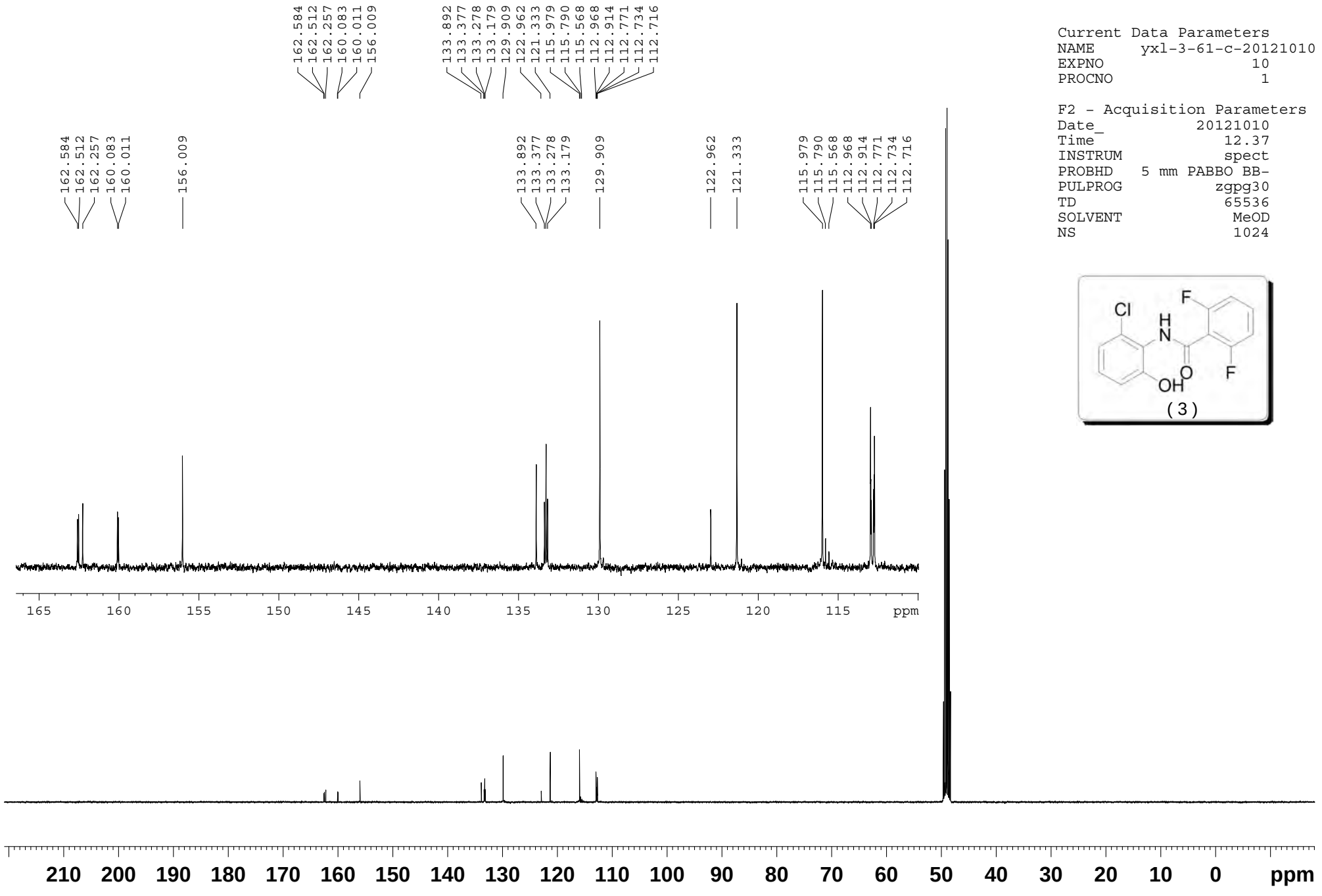


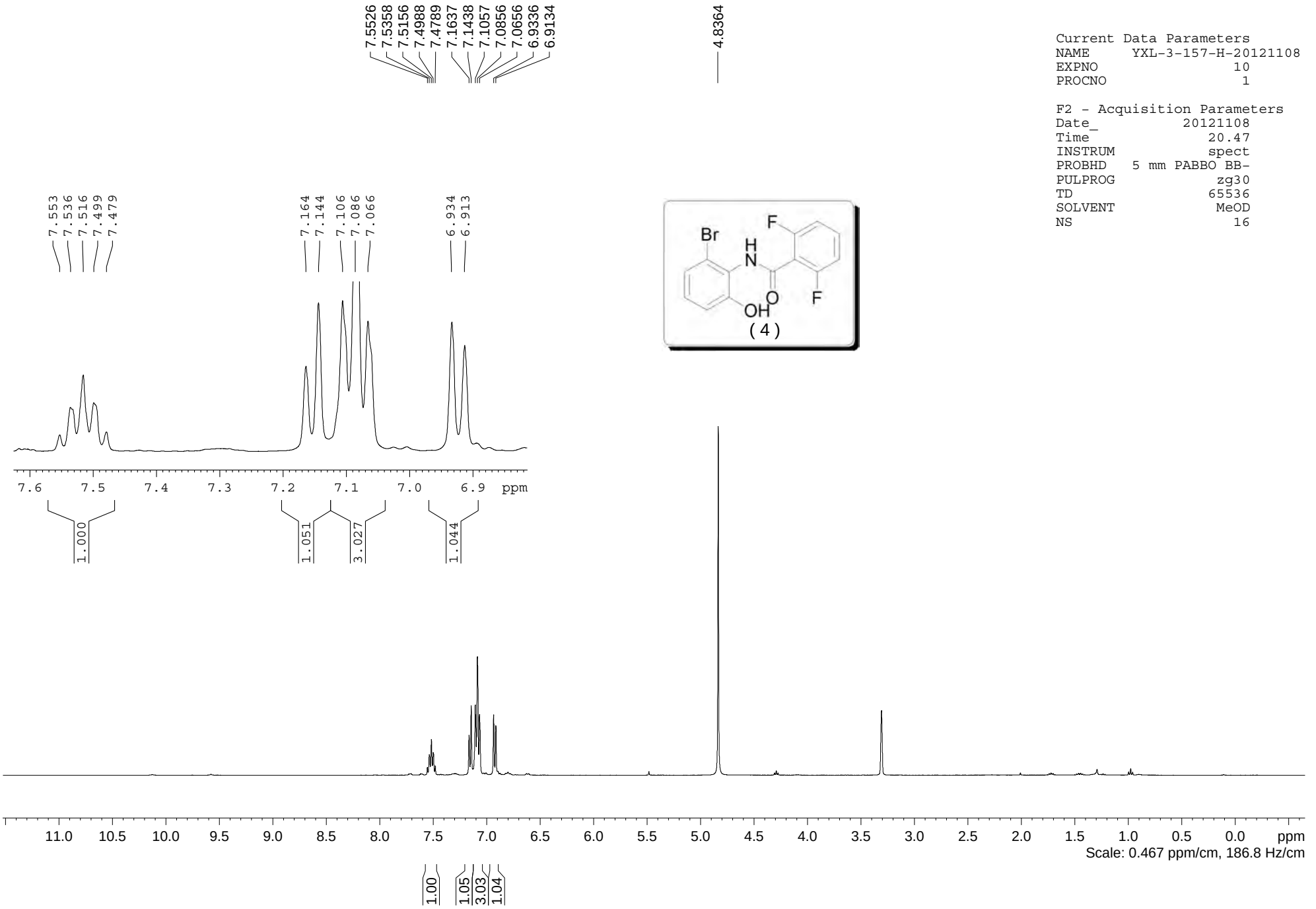
Current Data Parameters
NAME 20121108-mll-3-251
EXPNO 10
PROCNO 1

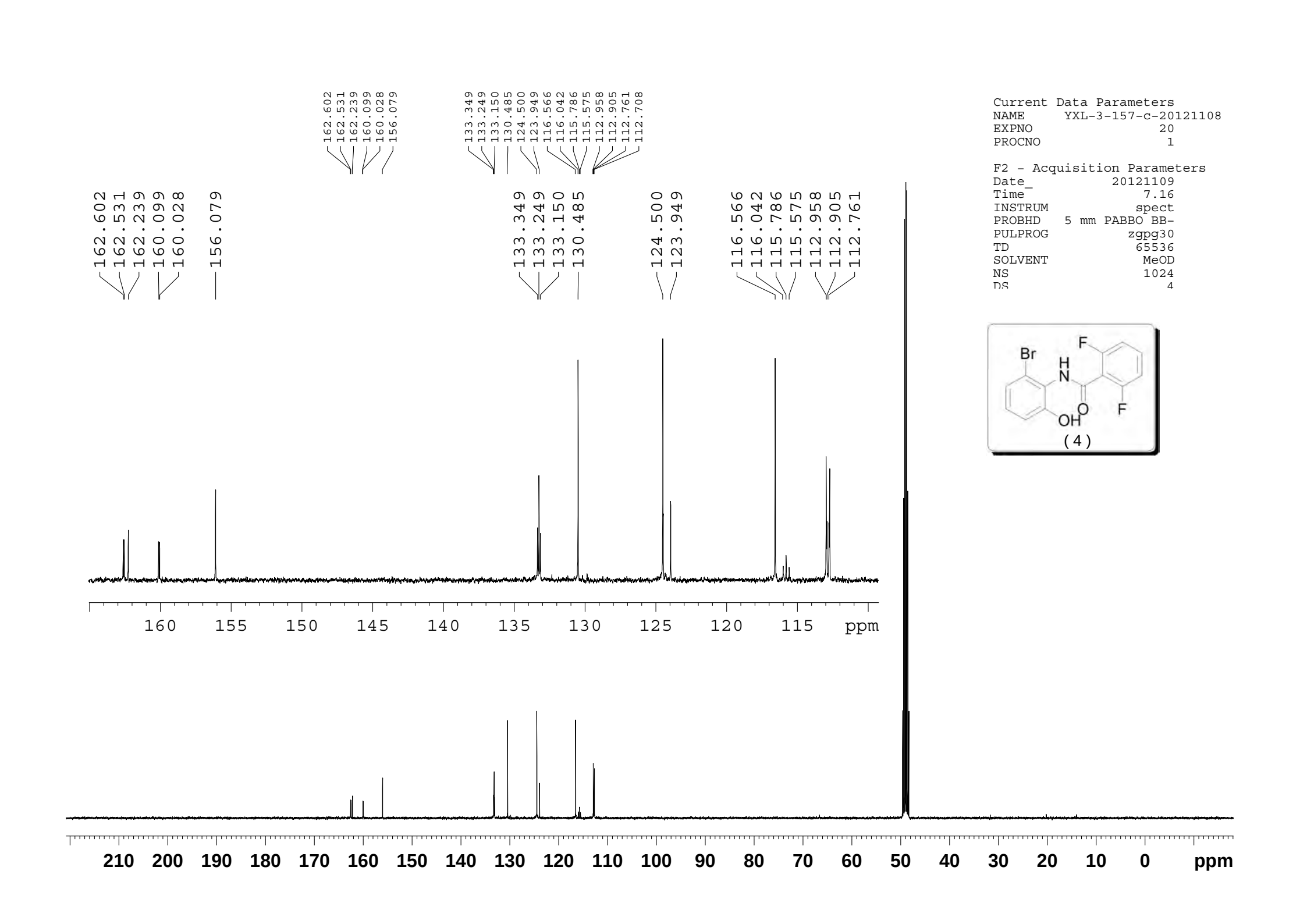
F2 - Acquisition Parameters
Date_ 20121108
Time 20.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536

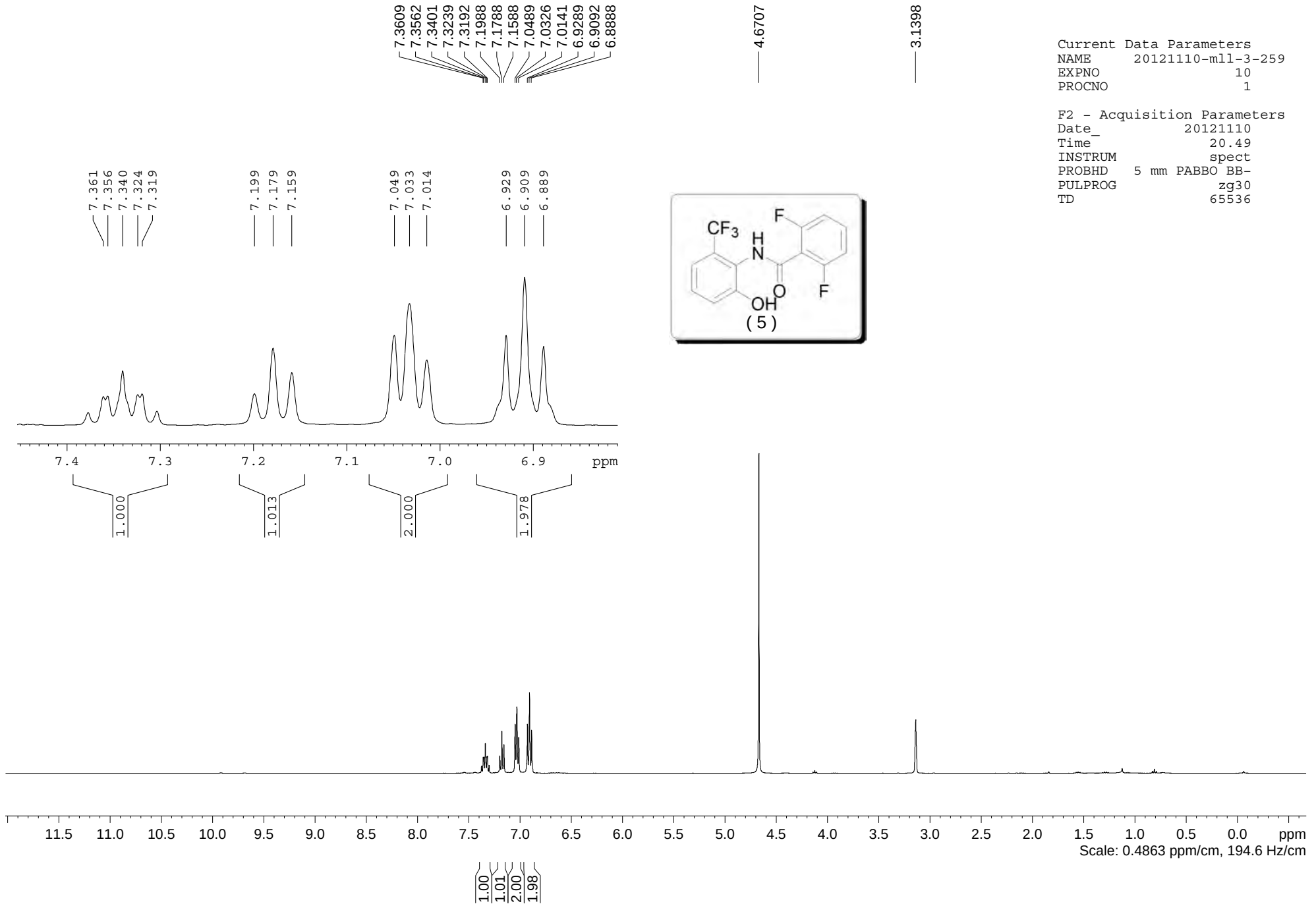

162.4868
162.4158
162.2436
160.9702
159.9884
159.9184
158.5114
155.4013
155.3577
133.4404
133.3416
133.2415
129.3410
129.2385
115.8884
115.6742
115.4564
113.7971
113.6454
113.0940
113.0658
112.9654
112.9122
112.7681
112.7153
112.3224
112.0879
107.5632
107.3586
49.4230
49.2102
48.9978
48.7855
48.5725
162.487
162.416
162.244
160.970
159.988
159.918
158.511
155.401
155.358
133.440
133.342
133.242
129.341
129.238
115.888
115.674
115.456
113.797
113.645
113.094
113.066
112.965
112.912
112.768
112.715
112.322
112.088
107.563
107.359
160
155
150
145
140
135
130
125
120
115
110
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
Current Data Parameters
NAME 20121108-mll-3-251-C
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121109
Time 8.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1280
DS 4
F
F
H
N
O
OH
F
(2)


7.5584
7.5425
7.5373
7.5264
7.5214
7.5164
7.5054
7.5003
7.4843
7.1678
7.1474
7.1270
7.1194
7.1129
7.0930
7.0726
7.0662
6.9944
6.9916
6.9743
6.9715
6.8977
6.8949
6.8771
6.8743
4.8590
3.3139
3.3100
3.3061
Current Data Parameters
NAME YXL-3-61-H-20121010
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121010
Time 10.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
7.558
7.543
7.537
7.526
7.521
7.516
7.505
7.500
7.484
7.168
7.147
7.127
7.119
7.113
7.093
7.073
7.066
6.994
6.992
6.974
6.971
6.898
6.895
6.877
6.874
Cl
F
H
N
O
OH
F
(3)
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
ppm
1.000
3.021
1.020
0.985
1.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4664 ppm/cm, 186.6 Hz/cm
1.00
3.02
1.02
0.99
3.17


162.584
162.512
162.257
160.083
160.011
156.009
133.892
133.377
133.278
133.179
129.909
122.962
121.333
115.979
115.790
115.568
112.968
112.914
112.771
112.734
112.716
Current Data Parameters
NAME yxl-3-61-c-20121010
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121010
Time 12.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
Cl
F
H
N
O
OH
F
(3)
165 160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

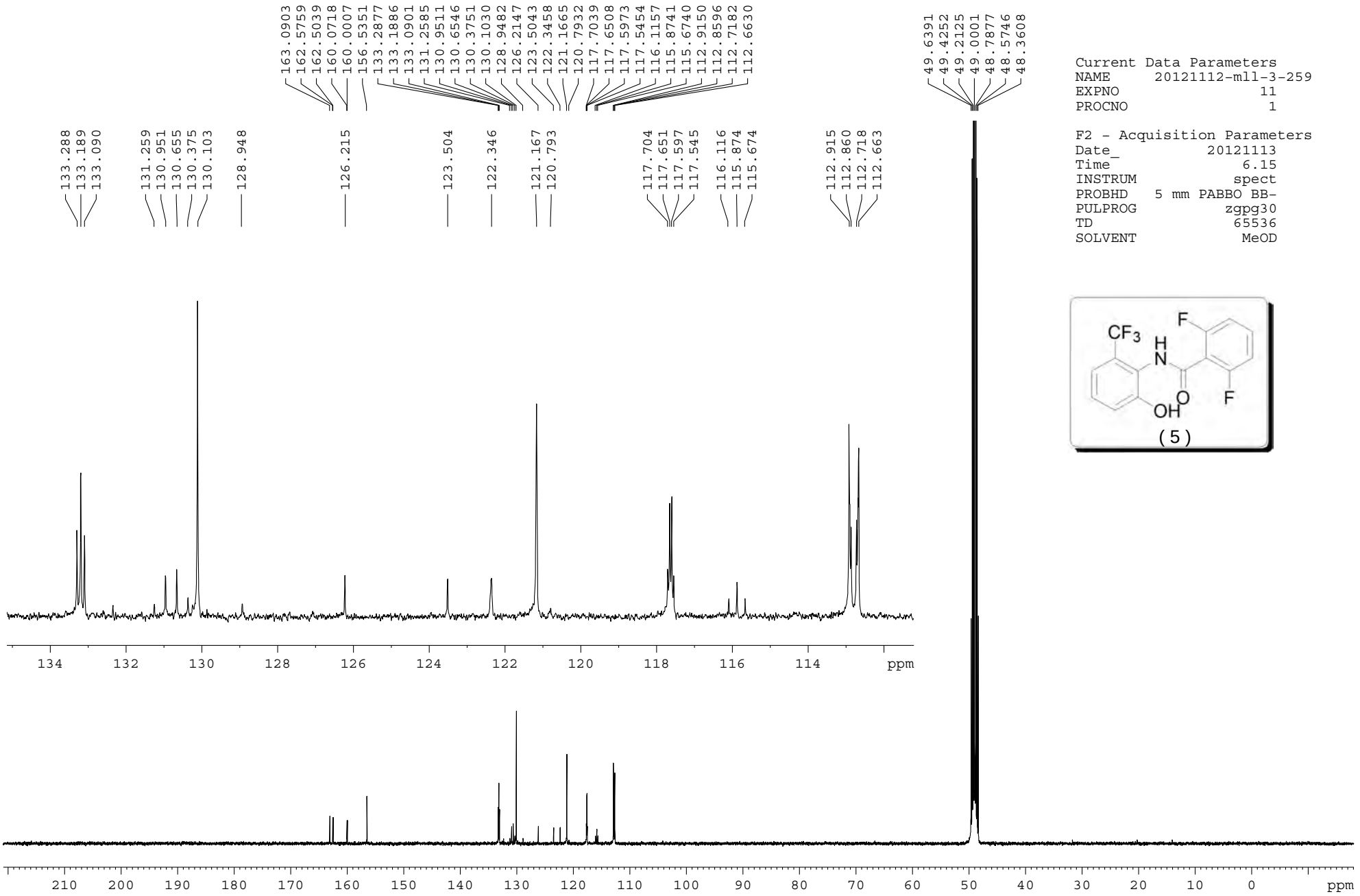

7.5526
7.5358
7.5156
7.4988
7.4789
7.1637
7.1438
7.1057
7.0856
7.0656
6.9336
6.9134
4.8364
Current Data Parameters
NAME YXL-3-157-H-20121108
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121108
Time 20.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
7.553
7.536
7.516
7.499
7.479
7.164
7.144
7.106
7.086
7.066
6.934
6.913
Br
F
H
N
O
OH
F
(4)
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
ppm
1.000
1.051
3.027
1.044
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.467 ppm/cm, 186.8 Hz/cm
1.00
1.05
3.03
1.04

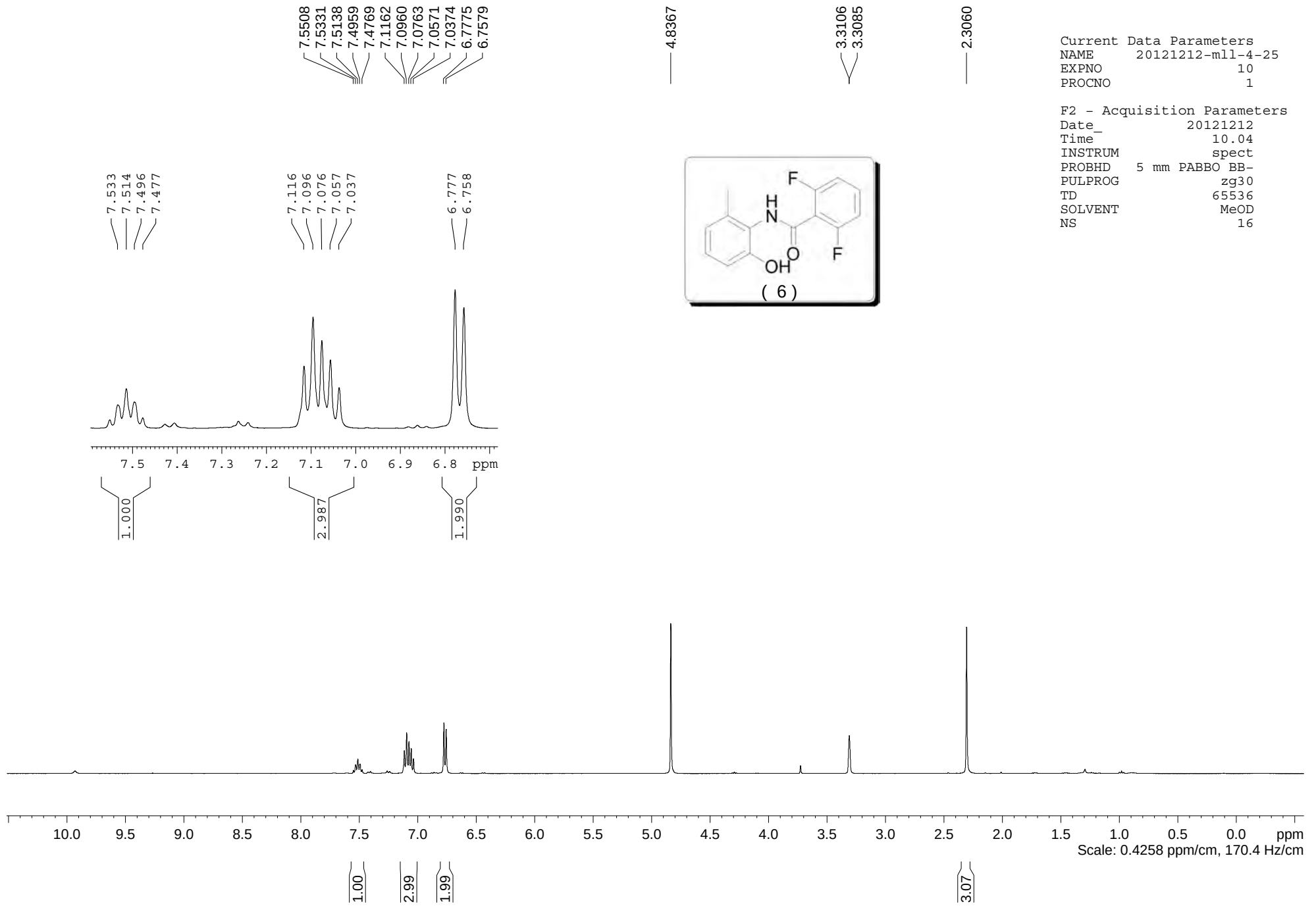

162.602
162.531
162.239
160.099
160.028
156.079
133.349
133.249
133.150
130.485
124.500
123.949
116.566
116.042
115.786
115.575
112.958
112.905
112.761
112.708
Current Data Parameters
NAME YXL-3-157-c-20121108
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121109
Time 7.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
Br
F
H
N
O
OH
F
(4)
160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

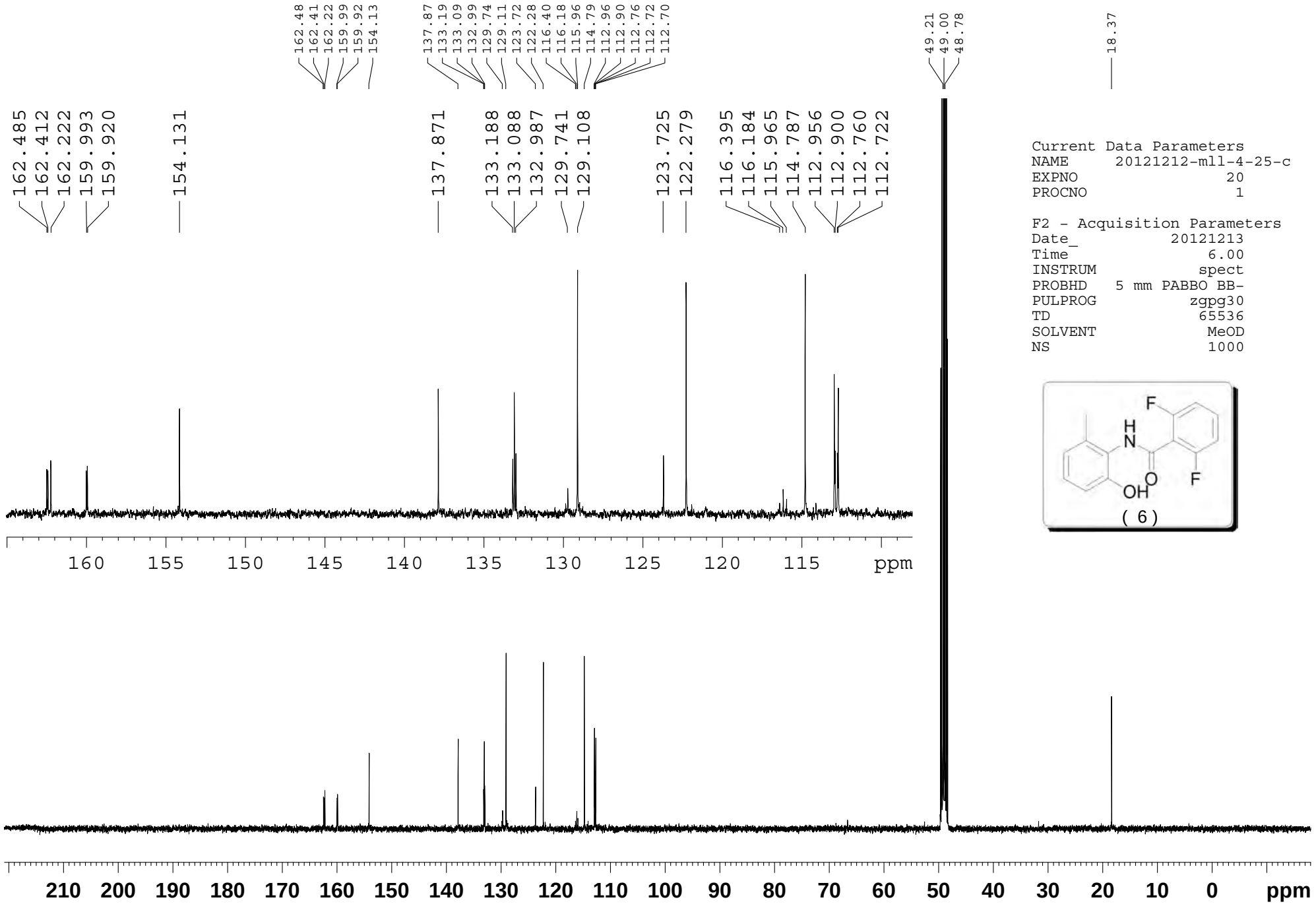


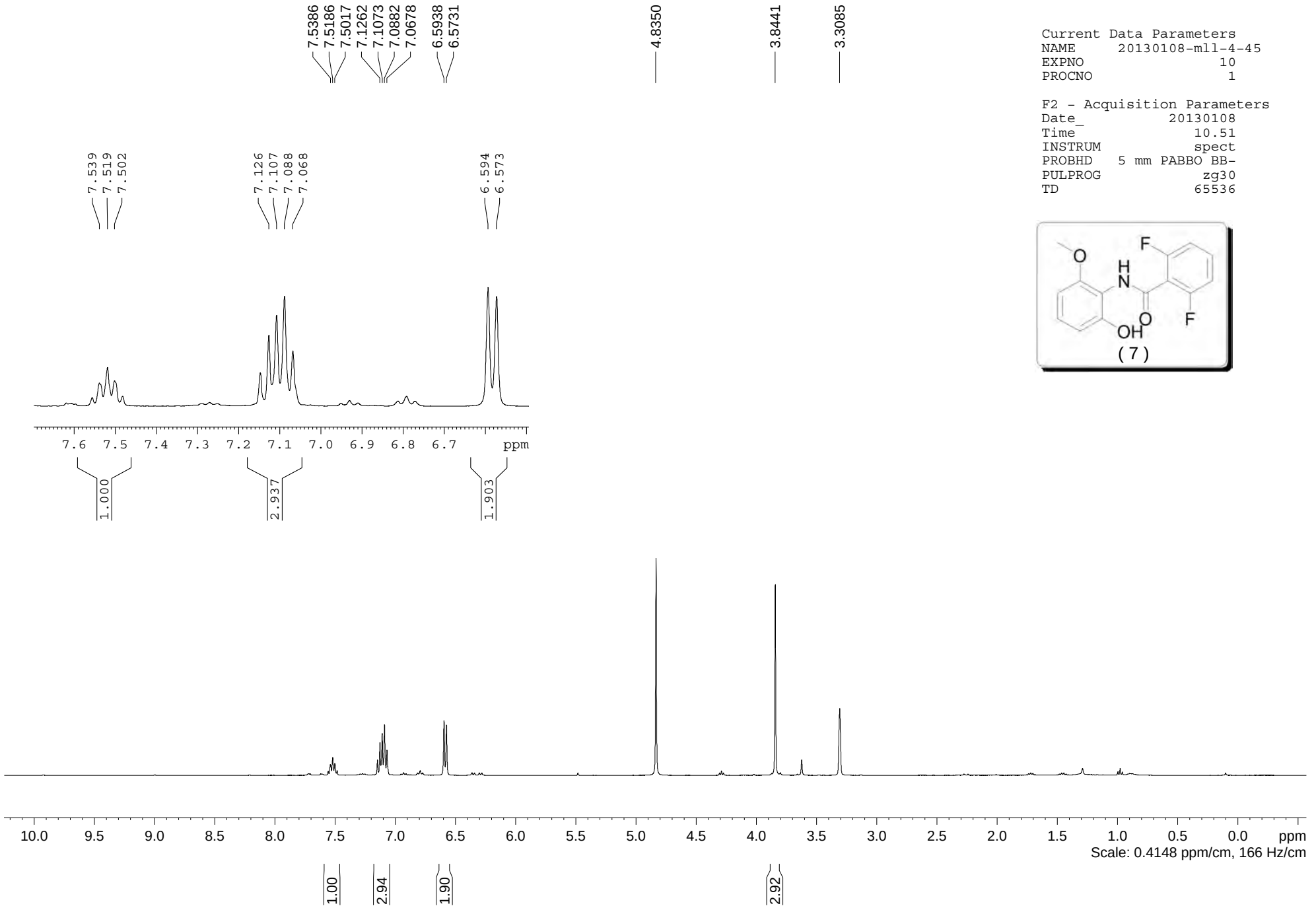
Current Data Parameters
NAME 20121110-mll-3-259
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121110
Time 20.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536


163.0903
162.5759
162.5039
160.0718
160.0007
156.5351
133.2877
133.1886
133.0901
131.2585
130.9511
130.6546
130.3751
130.1030
128.9482
126.2147
123.5043
122.3458
121.1665
120.7932
117.7039
117.6508
117.5973
117.5454
116.1157
115.8741
115.6740
112.9150
112.8596
112.7182
112.6630
49.6391
49.4252
49.2125
49.0001
48.7877
48.5746
48.3608
133.288
133.189
133.090
131.259
130.951
130.655
130.375
130.103
128.948
126.215
123.504
122.346
121.167
120.793
117.704
117.651
117.597
117.545
116.116
115.874
115.674
112.915
112.860
112.718
112.663
Current Data Parameters
NAME 20121112-mll-3-259
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121113
Time 6.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
CF3
F
H
N
O
OH
F
(5)
134
132
130
128
126
124
122
120
118
116
114
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.5508
7.5331
7.5138
7.4959
7.4769
7.1162
7.0960
7.0763
7.0571
7.0374
6.7775
6.7579
4.8367
3.3106
3.3085
2.3060
Current Data Parameters
NAME 20121212-mll-4-25
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121212
Time 10.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
7.533
7.514
7.496
7.477
7.116
7.096
7.076
7.057
7.037
6.777
6.758
F
H
N
O
OH
F
(6)
7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
1.000
2.987
1.990
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.4258 ppm/cm, 170.4 Hz/cm
1.00
2.99
1.99
3.07

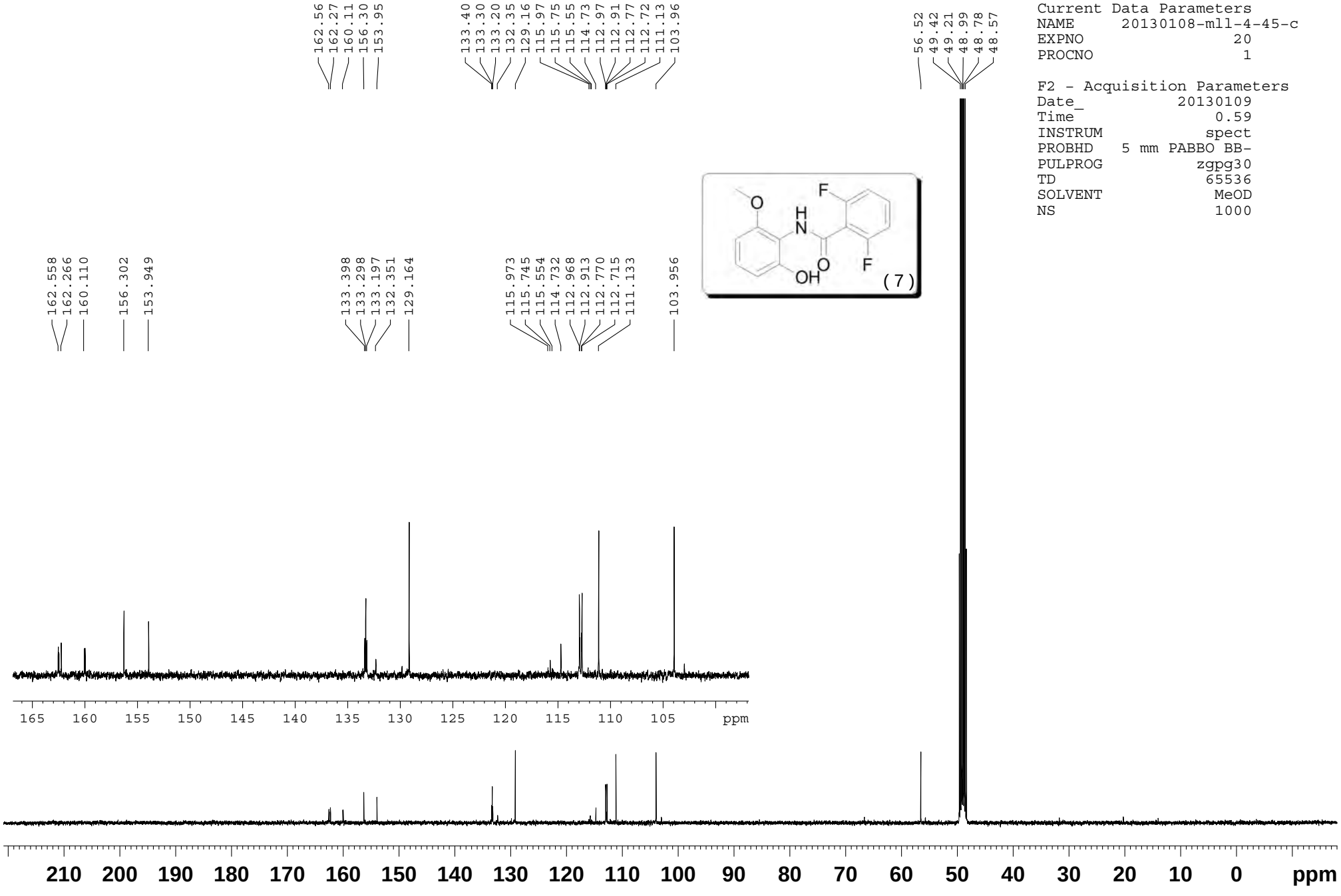

162.48
162.41
162.22
159.99
159.92
154.13
137.87
133.19
133.09
132.99
129.74
129.11
123.72
122.28
116.40
116.18
115.96
114.79
112.96
112.90
112.76
112.72
112.70
49.21
49.00
48.78
18.37
162.485
162.412
162.222
159.993
159.920
154.131
137.871
133.188
133.088
132.987
129.741
129.108
123.725
122.279
116.395
116.184
115.965
114.787
112.956
112.900
112.760
112.722
Current Data Parameters
NAME 20121212-mll-4-25-c
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121213
Time 6.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1000
F
H
N
O
OH
F
(6)
160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

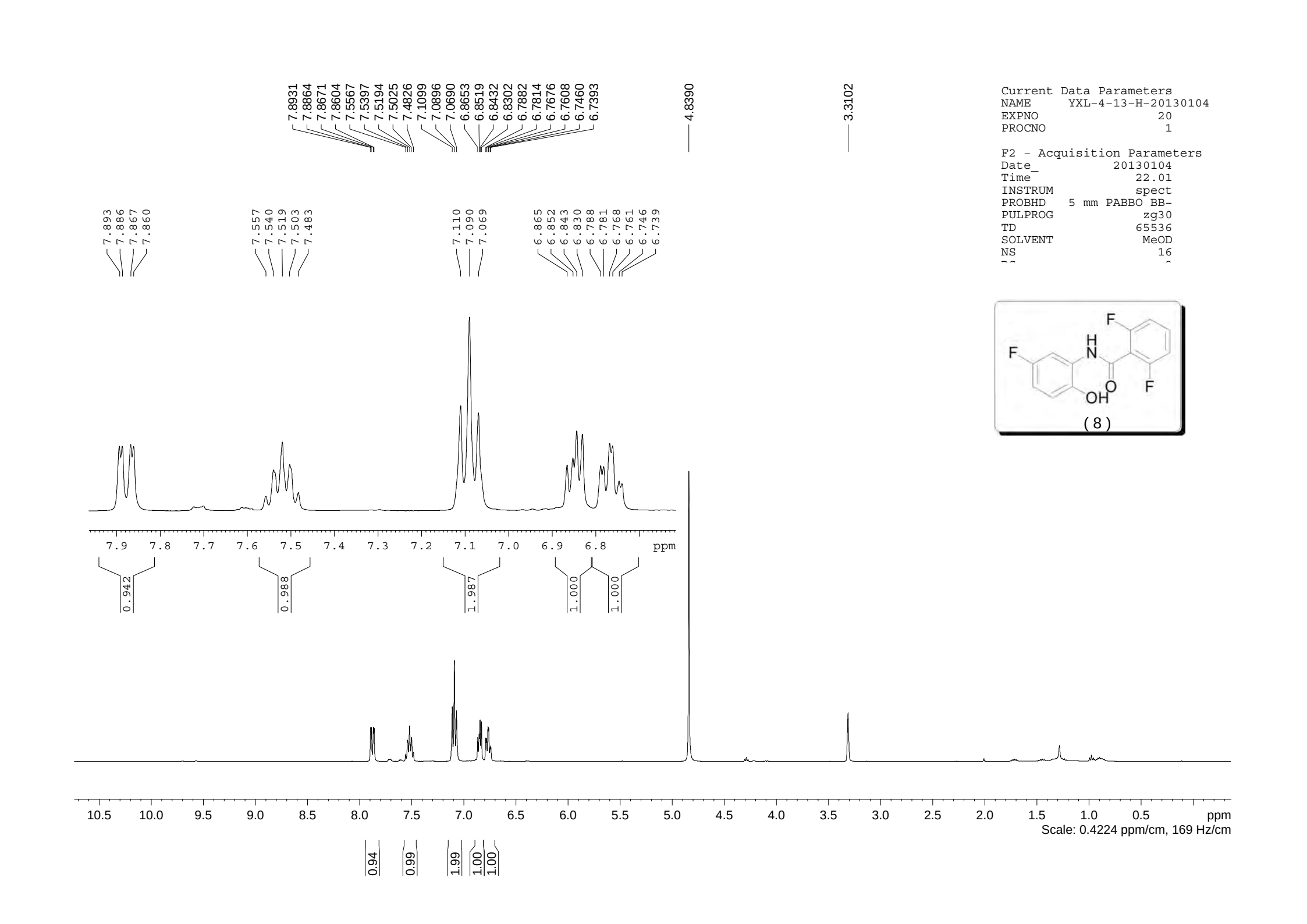


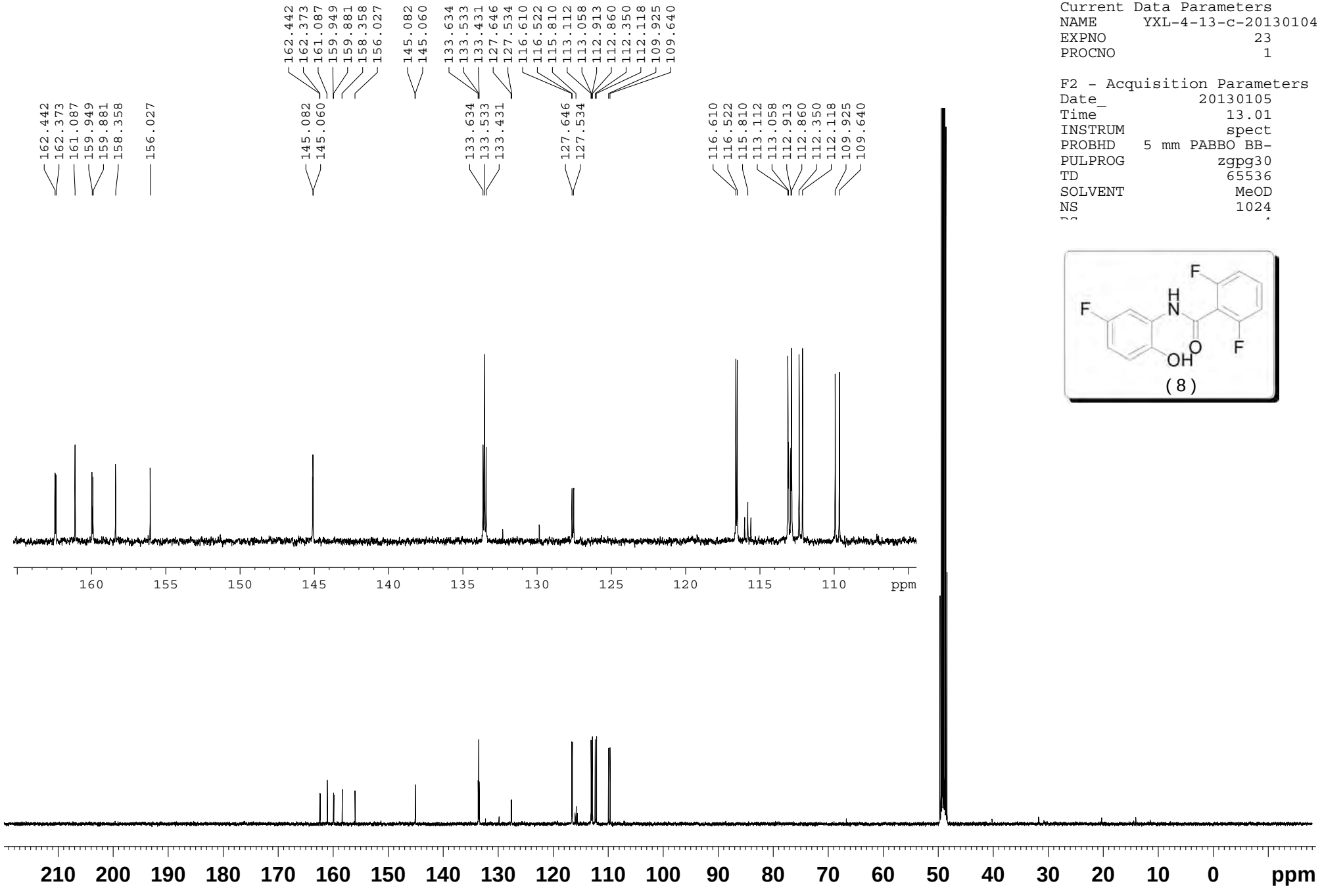
Current Data Parameters
NAME 20130108-mll-4-45
EXPNO 10
PROCNO 1

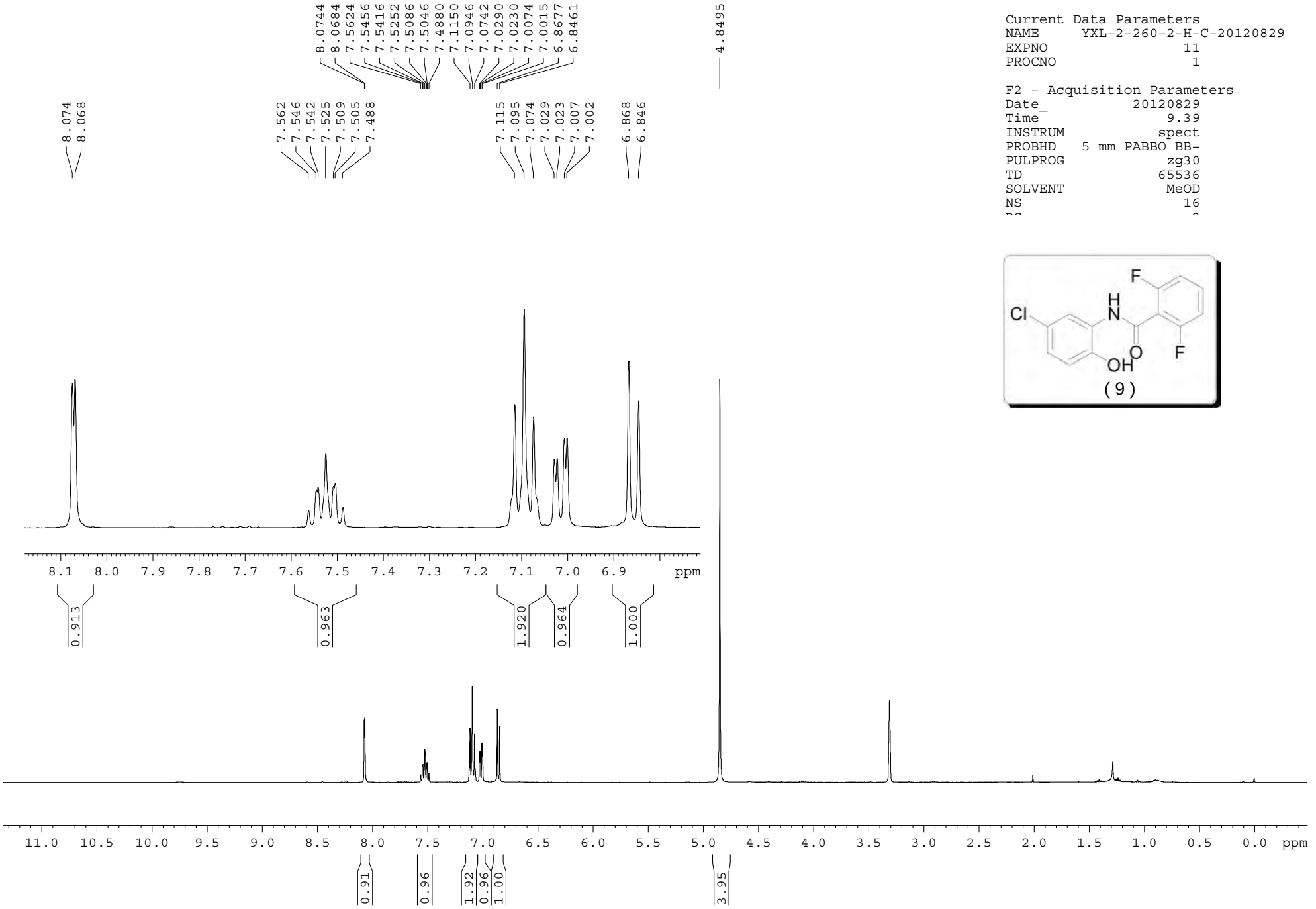
F2 - Acquisition Parameters
Date_ 20130108
Time 10.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536

(7)


162.56
162.27
160.11
156.30
153.95
133.40
133.30
133.20
132.35
129.16
115.97
115.75
115.55
114.73
112.97
112.91
112.77
112.72
111.13
103.96
56.52
49.42
49.21
48.99
48.78
48.57
Current Data Parameters
NAME 20130108-mll-4-45-c
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130109
Time 0.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1000
O
HN
F
O
OH
F
(7)
162.558
162.266
160.110
156.302
153.949
133.398
133.298
133.197
132.351
129.164
115.973
115.745
115.554
114.732
112.968
112.913
112.770
112.715
111.133
103.956
165 160 155 150 145 140 135 130 125 120 115 110 105 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.8931
7.8864
7.8671
7.8604
7.5567
7.5397
7.5194
7.5025
7.4826
7.1099
7.0896
7.0690
6.8653
6.8519
6.8432
6.8302
6.7882
6.7814
6.7676
6.7608
6.7460
6.7393
4.8390
3.3102
7.893
7.886
7.867
7.860
7.557
7.540
7.519
7.503
7.483
7.110
7.090
7.069
6.865
6.852
6.843
6.830
6.788
6.781
6.768
6.761
6.746
6.739
Current Data Parameters
NAME YXL-4-13-H-20130104
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130104
Time 22.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
F
H
N
F
O
F
OH
(8)
ppm
0.942
0.988
1.987
1.000
1.000
0.94
0.99
1.99
1.00
1.00
Scale: 0.4224 ppm/cm, 169 Hz/cm

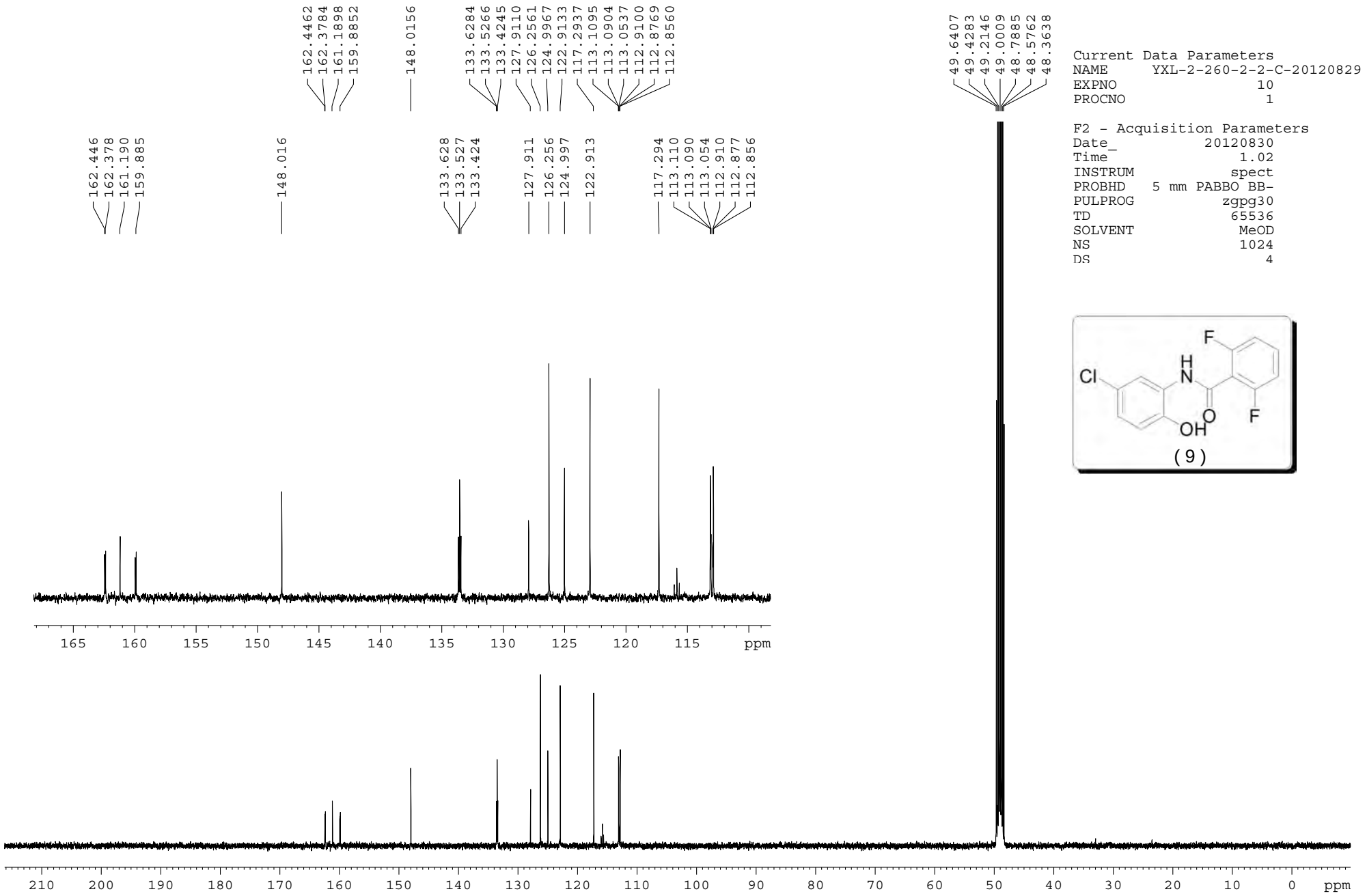

162.442
162.373
161.087
159.949
159.881
158.358
156.027
145.082
145.060
133.634
133.533
133.431
127.646
127.534
116.610
116.522
115.810
113.112
113.058
112.913
112.860
112.350
112.118
109.925
109.640
Current Data Parameters
NAME YXL-4-13-c-20130104
EXPNO 23
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130105
Time 13.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
F
H
N
F
O
OH
F
(8)
160 155 150 145 140 135 130 125 120 115 110 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

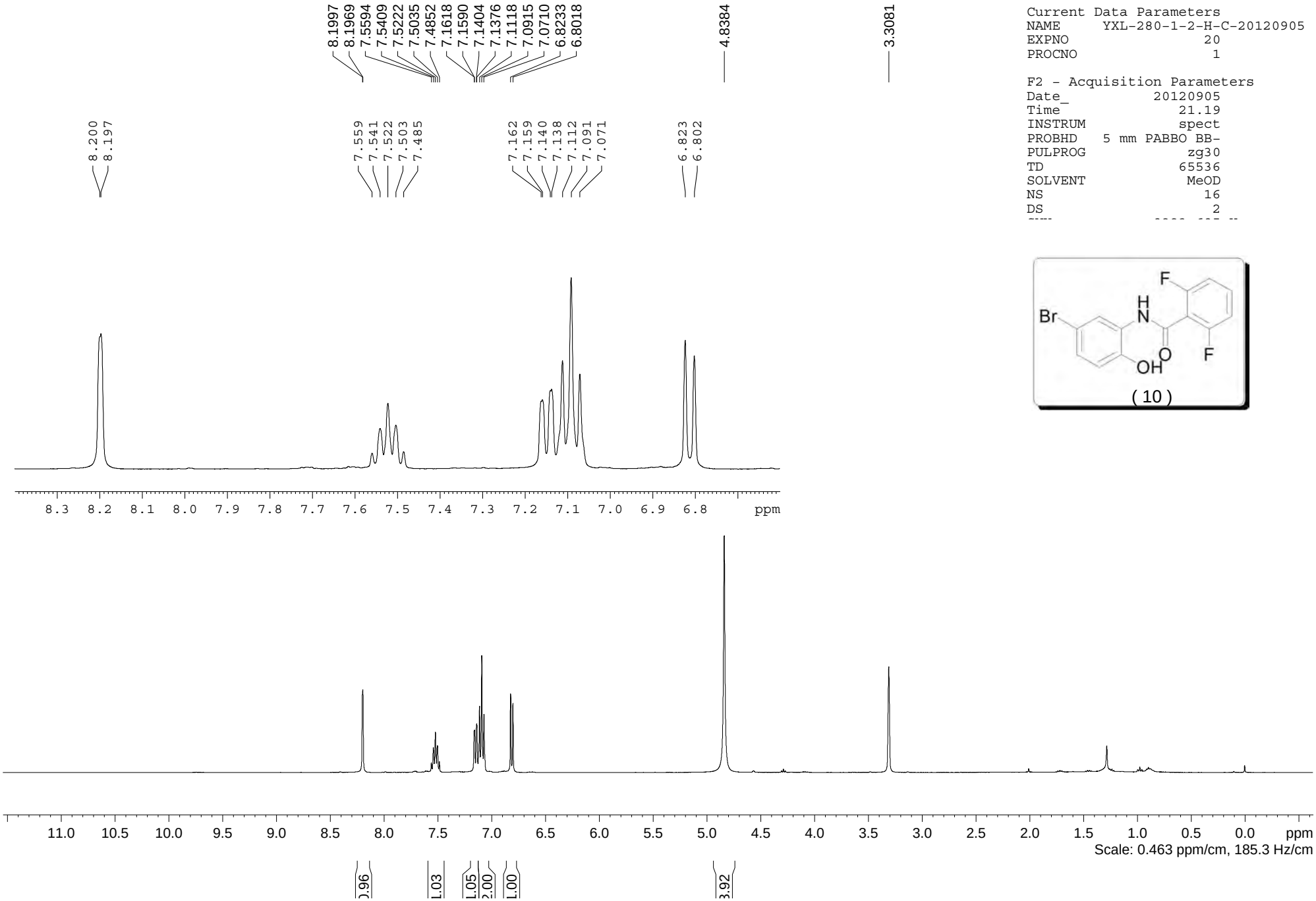


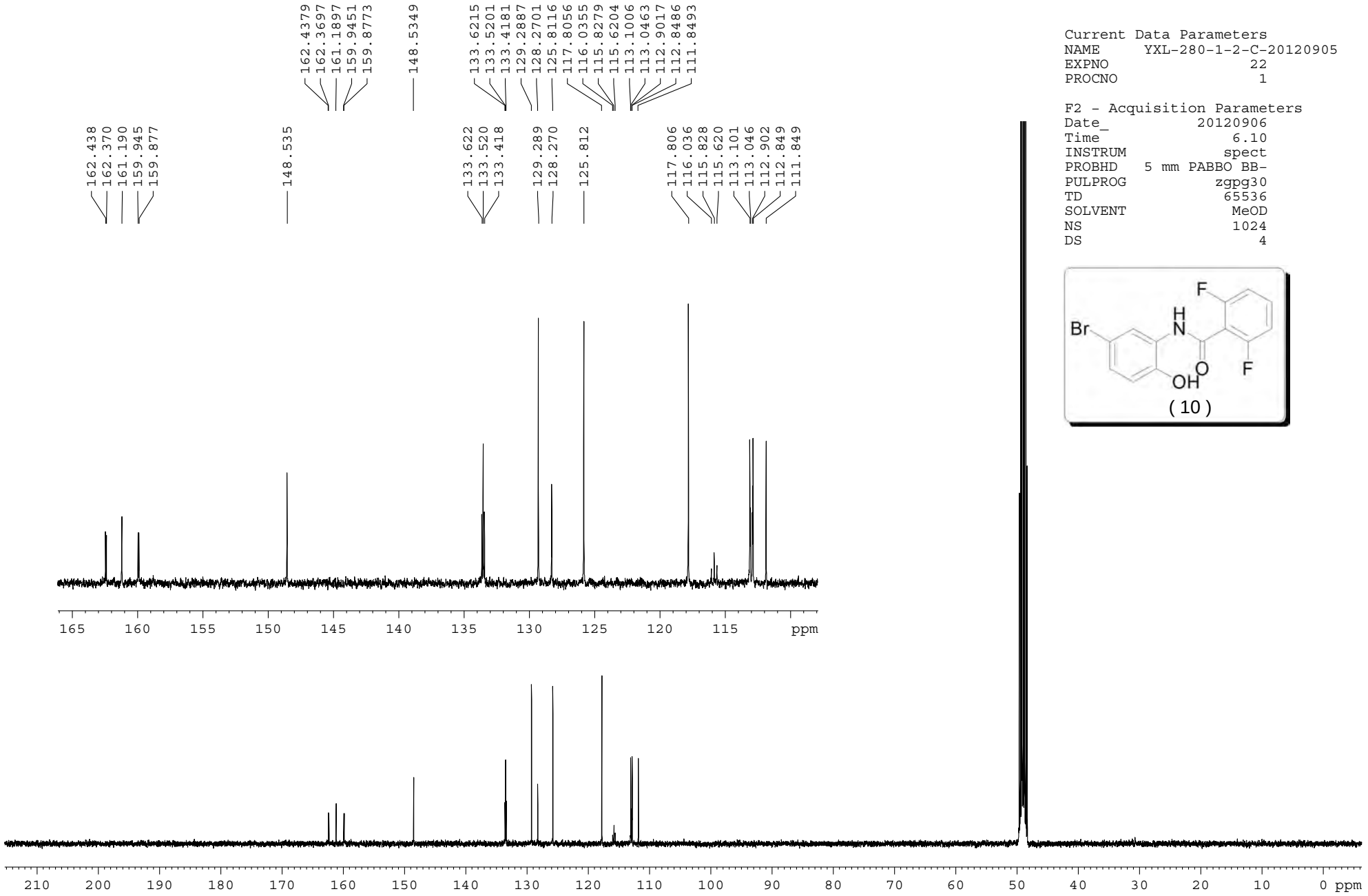
Current Data Parameters
NAME YXL-2-260-2-H-C-20120829
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120829
Time 9.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16

(9)


162.4462
162.3784
161.1898
159.8852
148.0156
133.6284
133.5266
133.4245
127.9110
126.2561
124.9967
122.9133
117.2937
113.1095
113.0904
113.0537
112.9100
112.8769
112.8560
49.6407
49.4283
49.2146
49.0009
48.7885
48.5762
48.3638
162.446
162.378
161.190
159.885
148.016
133.628
133.527
133.424
127.911
126.256
124.997
122.913
117.294
113.110
113.090
113.054
112.910
112.877
112.856
Current Data Parameters
NAME YXL-2-260-2-2-C-20120829
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120830
Time 1.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
F
H
N
Cl
O
OH
F
(9)
165 160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

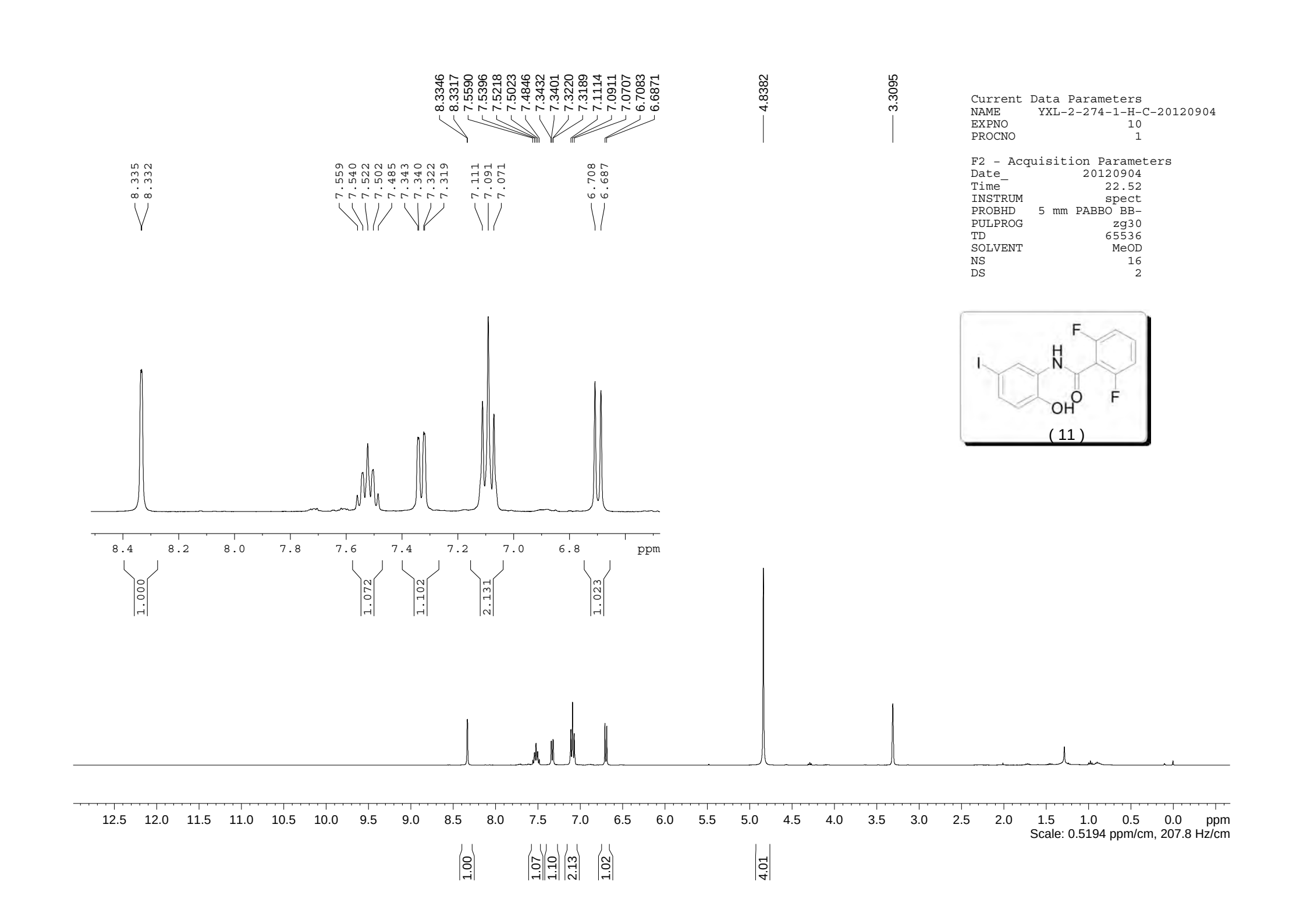

8.1997
8.1969
7.5594
7.5409
7.5222
7.5035
7.4852
7.1618
7.1590
7.1404
7.1376
7.1118
7.0915
7.0710
6.8233
6.8018
4.8384
3.3081
8.200
8.197
7.559
7.541
7.522
7.503
7.485
7.162
7.159
7.140
7.138
7.112
7.091
7.071
6.823
6.802
Current Data Parameters
NAME YXL-280-1-2-H-C-20120905
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120905
Time 21.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
F
H
N
Br
O
OH
F
(10)
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.463 ppm/cm, 185.3 Hz/cm
0.96
1.03
1.05
2.00
1.00
3.92

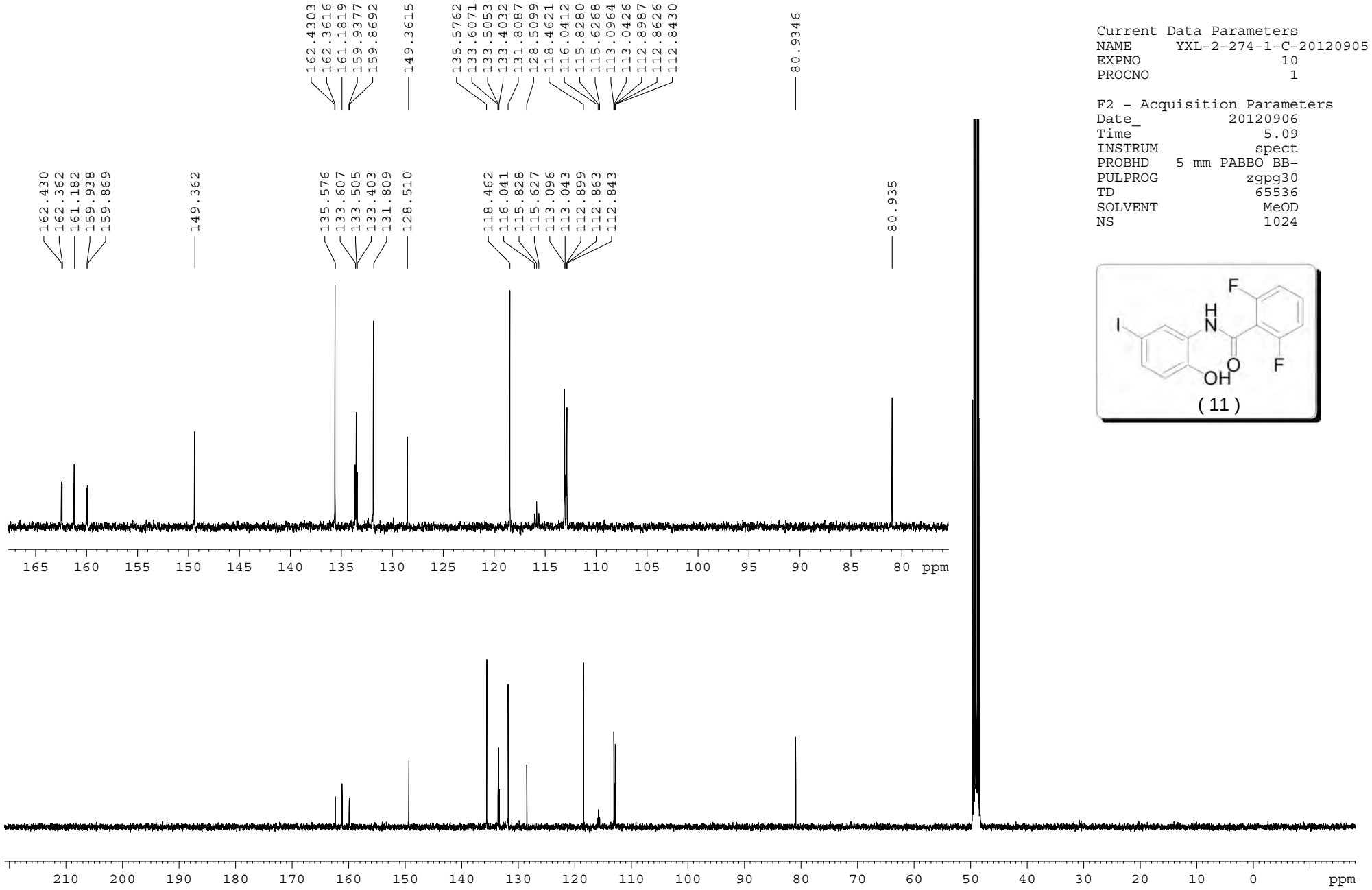


Current Data Parameters
NAME YXL-280-1-2-C-20120905
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120906
Time 6.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4

(10)

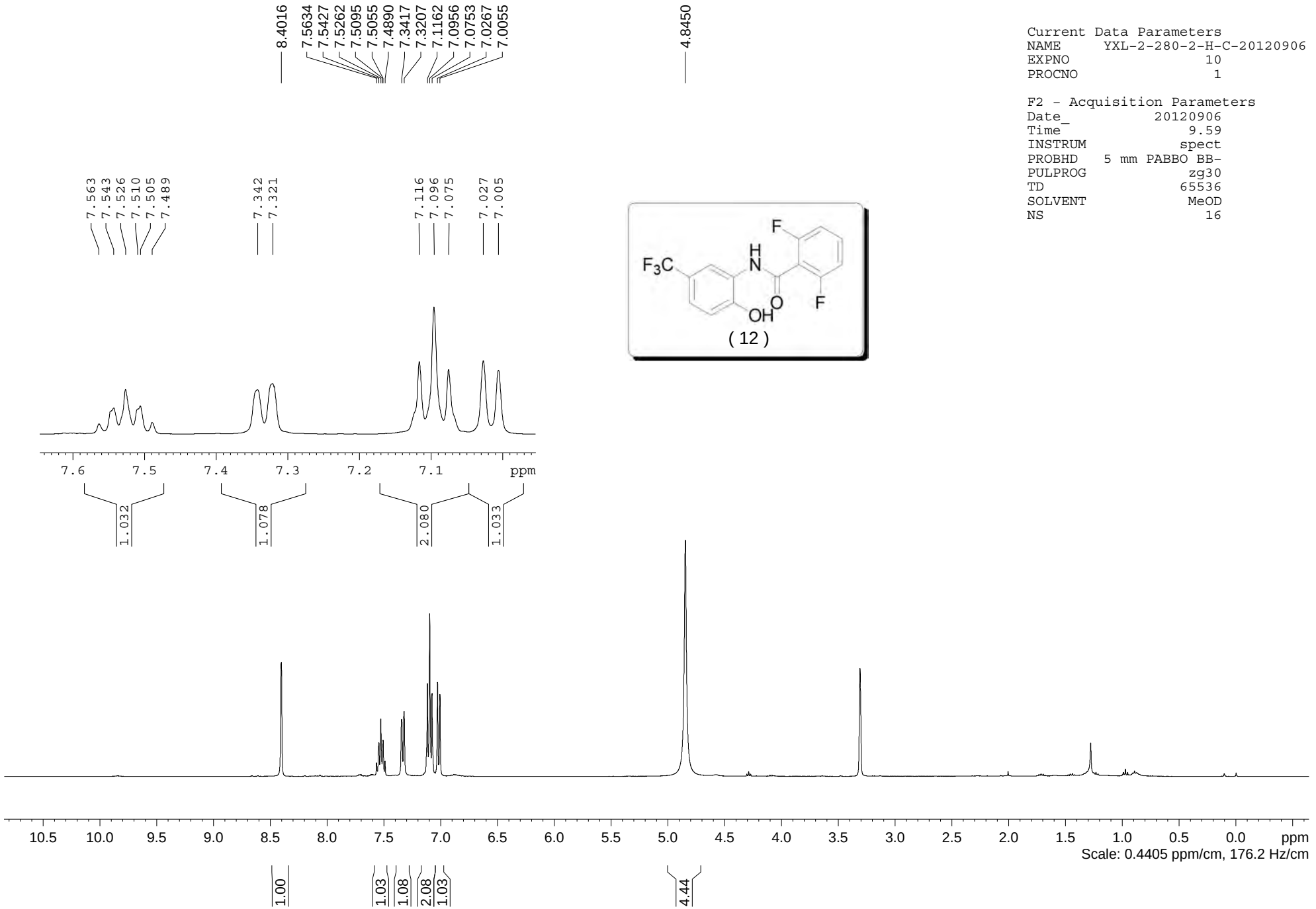

8.3346
8.3317
7.5590
7.5396
7.5218
7.5023
7.4846
7.3432
7.3401
7.3220
7.3189
7.1114
7.0911
7.0707
6.7083
6.6871
4.8382
3.3095
8.335
8.332
7.559
7.540
7.522
7.502
7.485
7.343
7.340
7.322
7.319
7.111
7.091
7.071
6.708
6.687
Current Data Parameters
NAME YXL-2-274-1-H-C-20120904
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120904
Time 22.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
F
H
N
I
O
OH
F
(11)
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
ppm
1.000
1.072
1.102
2.131
1.023
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.5194 ppm/cm, 207.8 Hz/cm
1.00
1.07
1.10
2.13
1.02
4.01



Current Data Parameters
NAME YXL-2-274-1-C-20120905
EXPNO 10
PROCNO 1

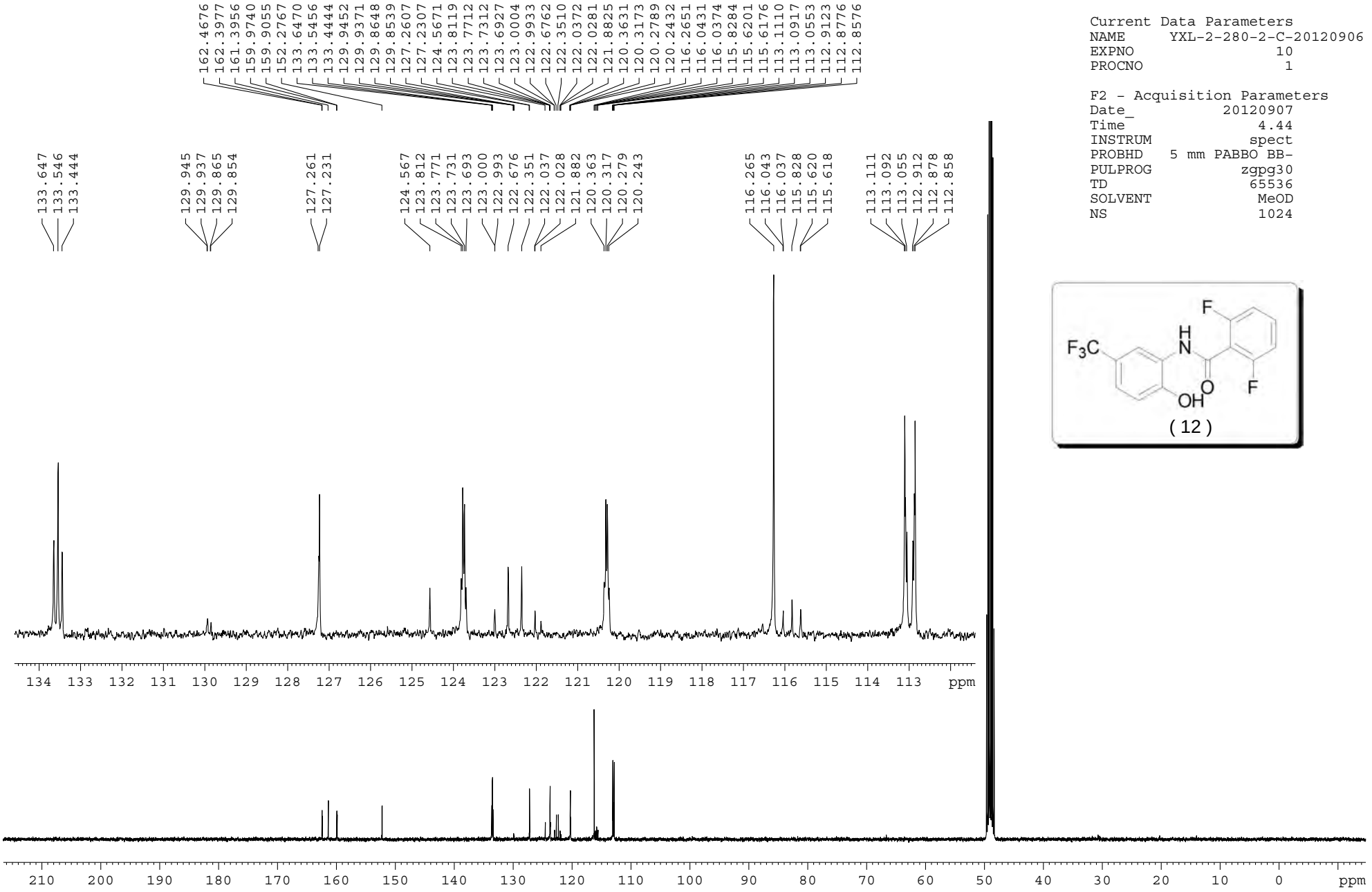
F2 - Acquisition Parameters
Date_ 20120906
Time 5.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024

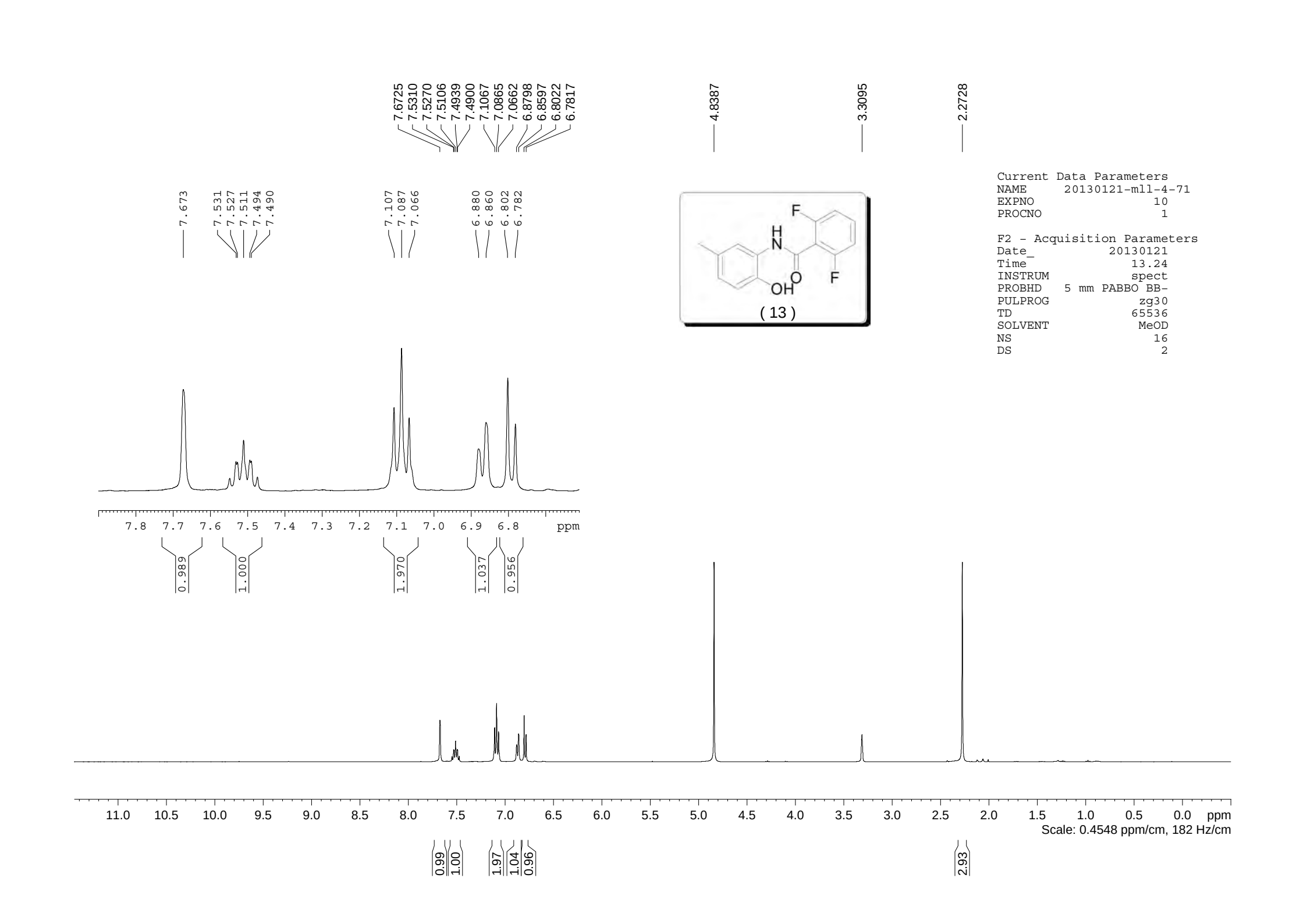
(11)

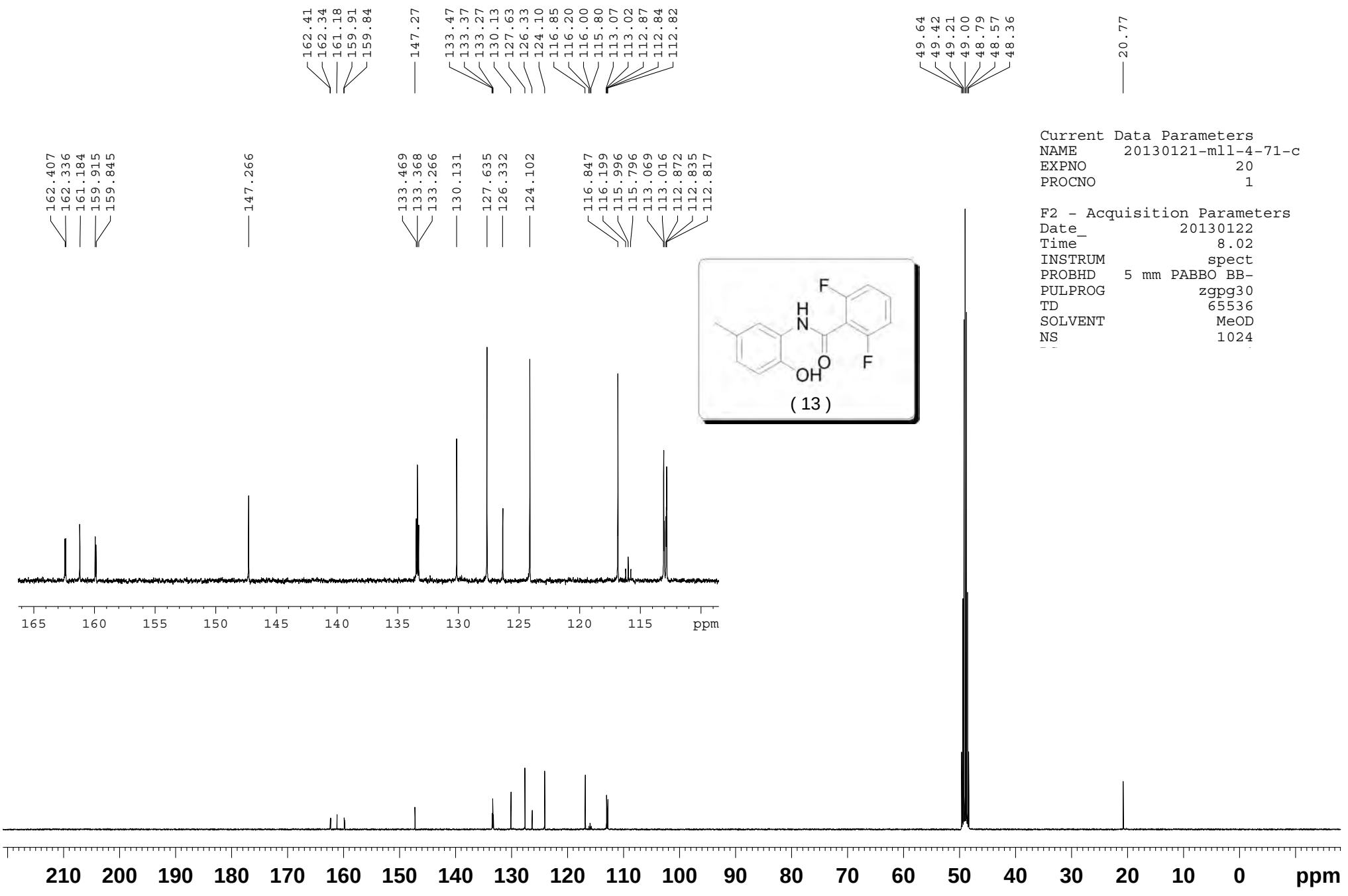


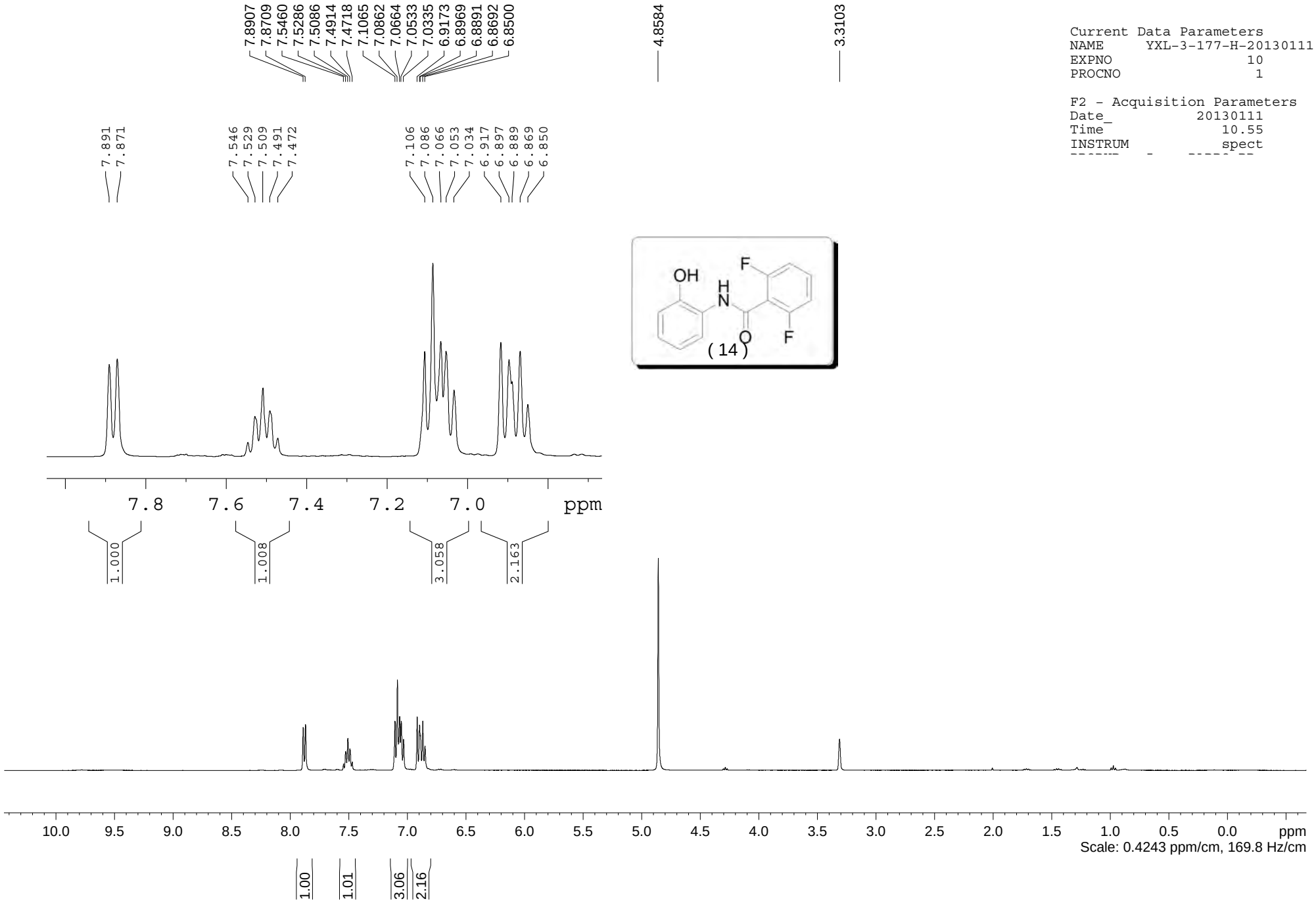
Current Data Parameters
NAME YXL-2-280-2-H-C-20120906
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120906
Time 9.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16


162.4676
162.3977
161.3956
159.9740
159.9055
152.2767
133.6470
133.5456
133.4444
129.9452
129.9371
129.8648
129.8539
127.2607
127.2307
124.5671
123.8119
123.7712
123.7312
123.6927
123.0004
122.9933
122.6762
122.3510
122.0372
122.0281
121.8825
120.3631
120.3173
120.2789
120.2432
116.2651
116.0431
116.0374
115.8284
115.6201
115.6176
113.1110
113.0917
113.0553
112.9123
112.8776
112.8576
133.647
133.546
133.444
129.945
129.937
129.865
129.854
127.261
127.231
124.567
123.812
123.771
123.731
123.693
123.000
122.993
122.676
122.351
122.037
122.028
121.882
120.363
120.317
120.279
120.243
116.265
116.043
116.037
115.828
115.620
115.618
113.111
113.092
113.055
112.912
112.878
112.858
Current Data Parameters
NAME YXL-2-280-2-C-20120906
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120907
Time 4.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
F
H
N
F3C
O
F
OH
(12)
134 133 132 131 130 129 128 127 126 125 124 123 122 121 120 119 118 117 116 115 114 113 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

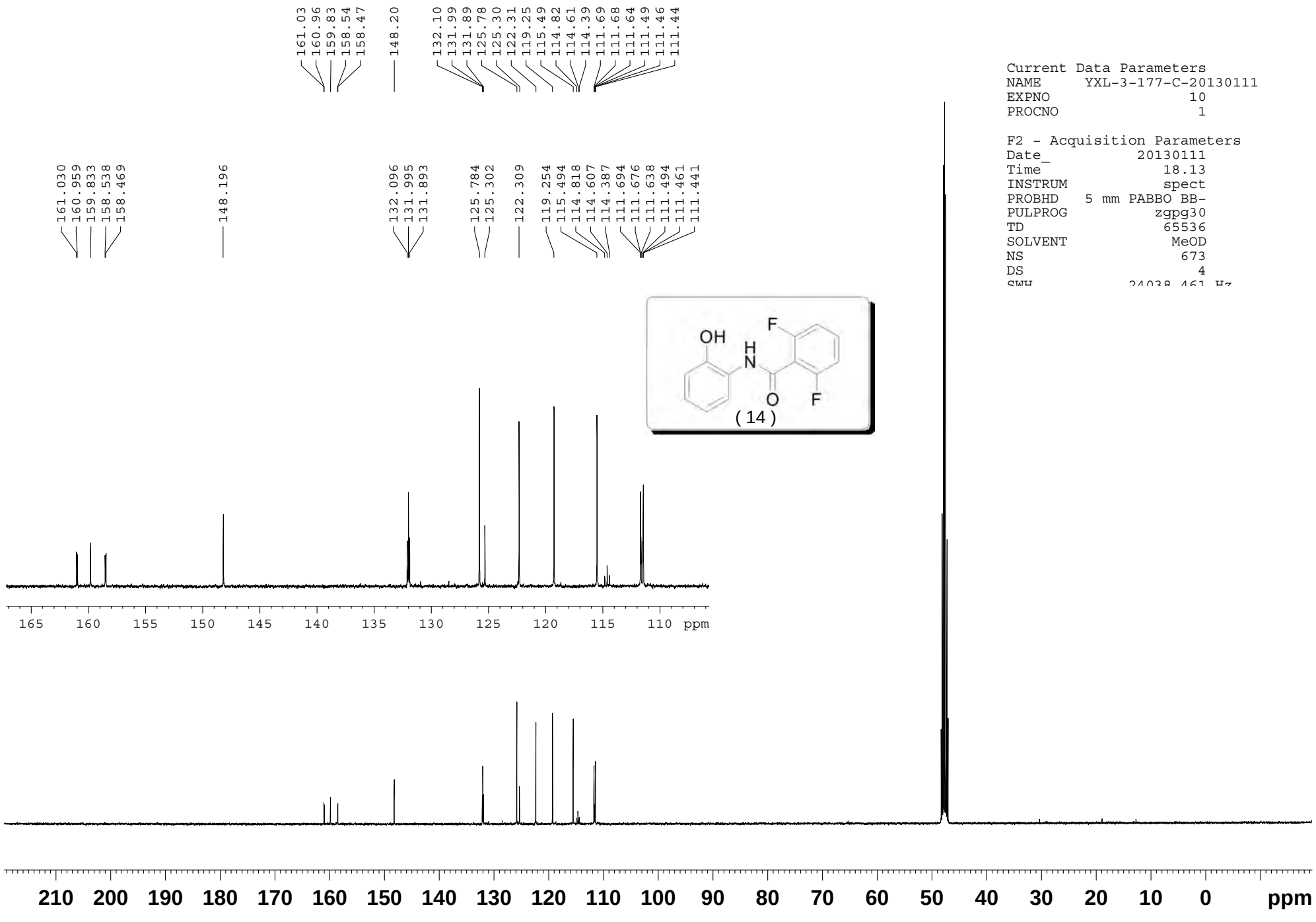

7.6725
7.5310
7.5270
7.5106
7.4939
7.4900
7.1067
7.0865
7.0662
6.8798
6.8597
6.8022
6.7817
4.8387
3.3095
2.2728
7.673
7.531
7.527
7.511
7.494
7.490
7.107
7.087
7.066
6.880
6.860
6.802
6.782
F
H
N
O
OH
F
(13)
Current Data Parameters
NAME 20130121-mll-4-71
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130121
Time 13.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
0.989
1.000
1.970
1.037
0.956
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.4548 ppm/cm, 182 Hz/cm
0.99
1.00
1.97
1.04
0.96
2.93

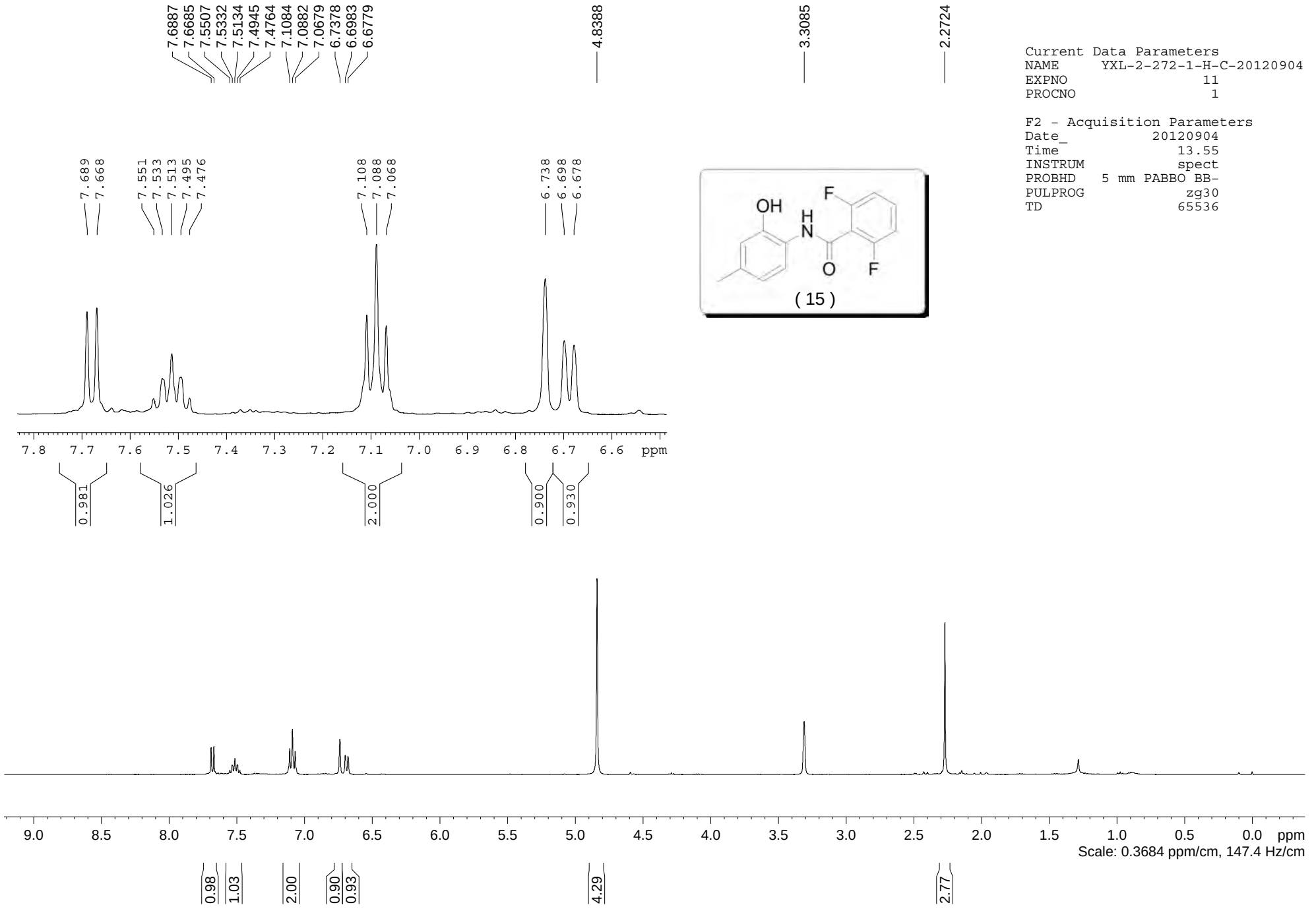

162.41
162.34
161.18
159.91
159.84
147.27
133.47
133.37
133.27
130.13
127.63
126.33
124.10
116.85
116.20
116.00
115.80
113.07
113.02
112.87
112.84
112.82
49.64
49.42
49.21
49.00
48.79
48.57
48.36
20.77
162.407
162.336
161.184
159.915
159.845
147.266
133.469
133.368
133.266
130.131
127.635
126.332
124.102
116.847
116.199
115.996
115.796
113.069
113.016
112.872
112.835
112.817
Current Data Parameters
NAME 20130121-mll-4-71-c
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130122
Time 8.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
F
H
N
O
OH
F
(13)
165 160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

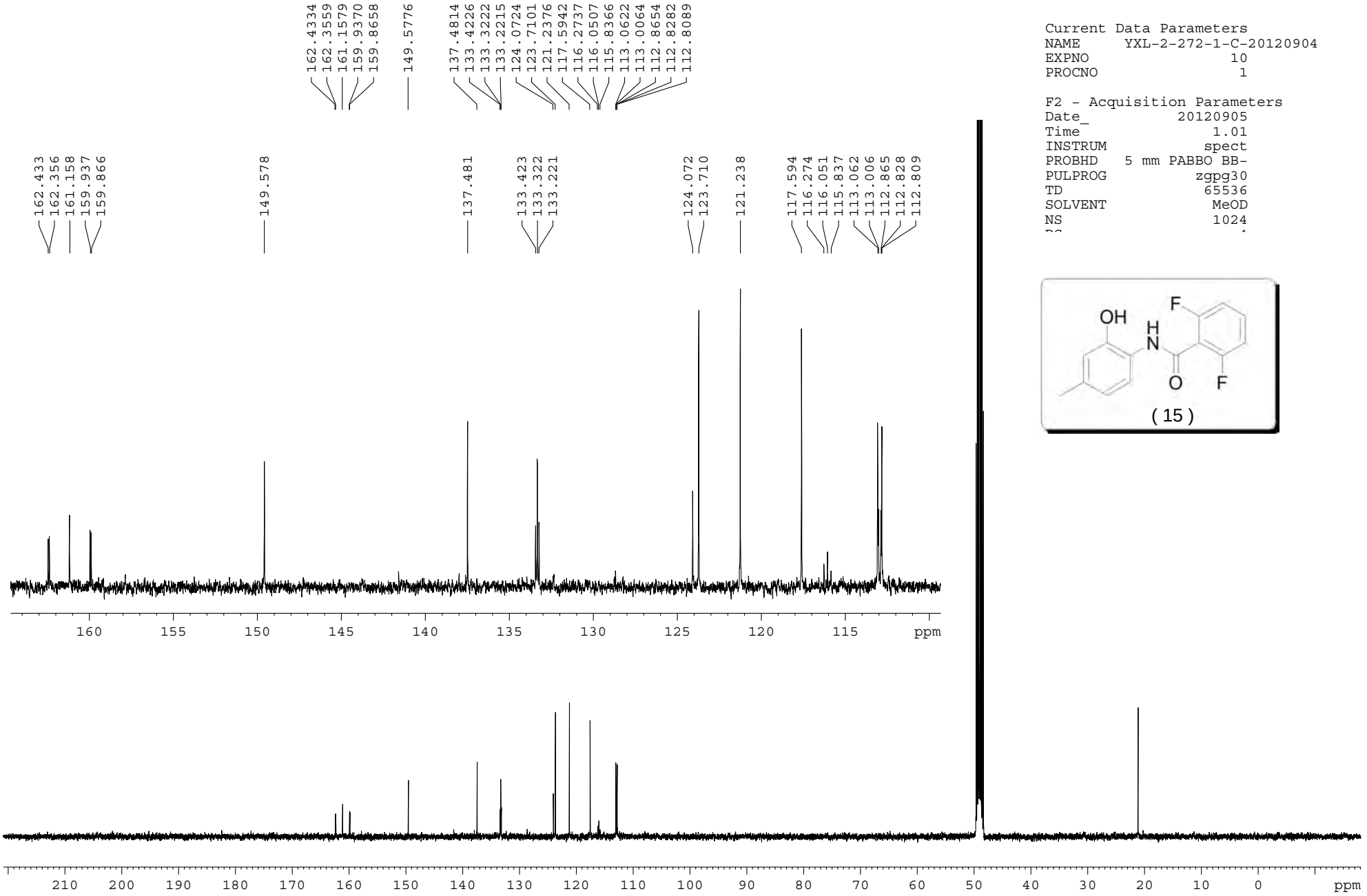


Current Data Parameters
NAME YXL-3-177-H-20130111
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130111
Time 10.55
INSTRUM spect


161.03
160.96
159.83
158.54
158.47
148.20
132.10
131.99
131.89
125.78
125.30
122.31
119.25
115.49
114.82
114.61
114.39
111.69
111.68
111.64
111.49
111.46
111.44
161.030
160.959
159.833
158.538
158.469
148.196
132.096
131.995
131.893
125.784
125.302
122.309
119.254
115.494
114.818
114.607
114.387
111.694
111.676
111.638
111.494
111.461
111.441
OH
H
N
F
O
F
(14)
Current Data Parameters
NAME YXL-3-177-C-20130111
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130111
Time 18.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 673
DS 4
165 160 155 150 145 140 135 130 125 120 115 110 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

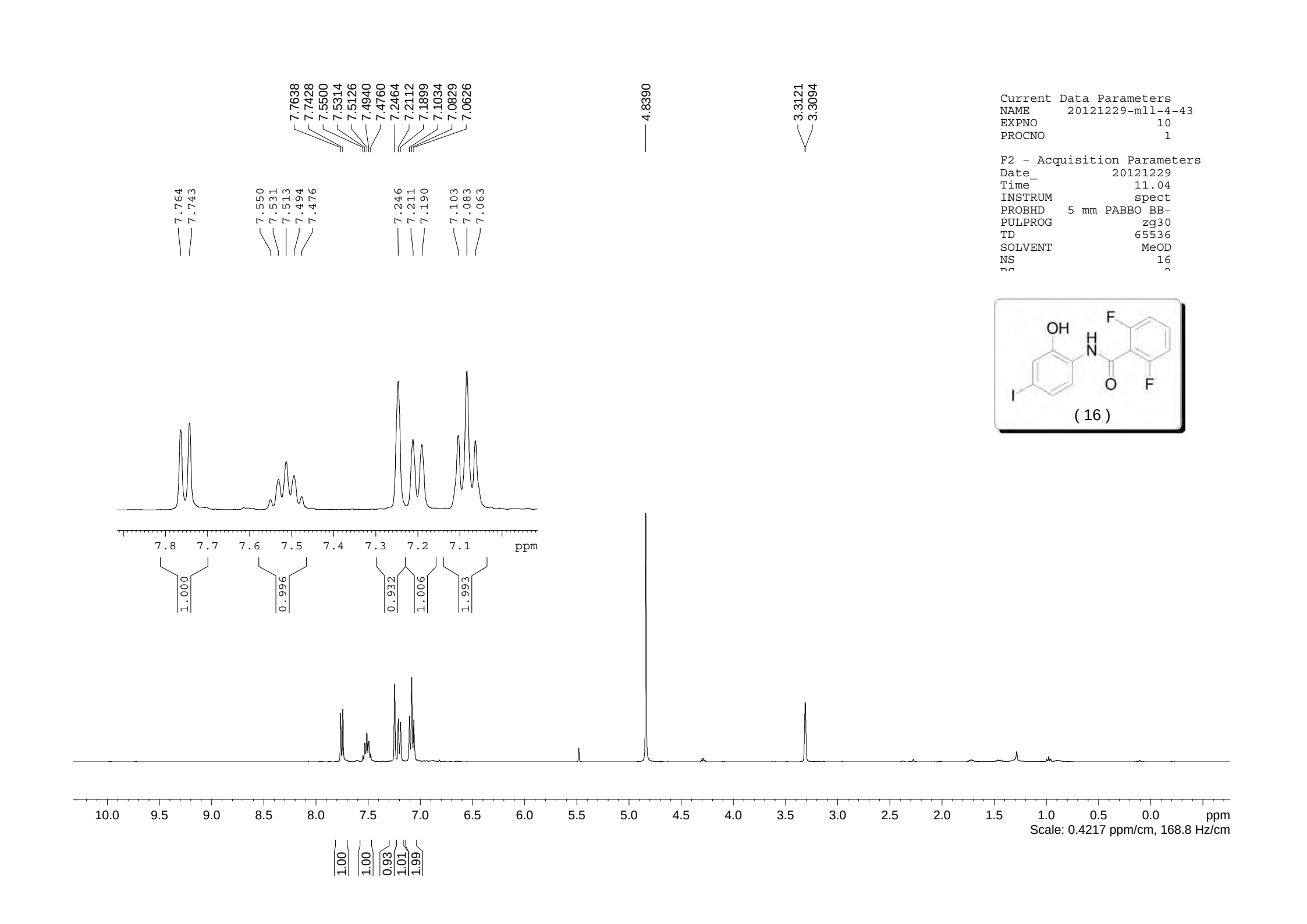

7.6887
7.6685
7.5507
7.5332
7.5134
7.4945
7.4764
7.1084
7.0882
7.0679
6.7378
6.6983
6.6779
4.8388
3.3085
2.2724
Current Data Parameters
NAME YXL-2-272-1-H-C-20120904
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120904
Time 13.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
7.689
7.668
7.551
7.533
7.513
7.495
7.476
7.108
7.088
7.068
6.738
6.698
6.678
OH
F
H
N
O
F
(15)
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 ppm
0.981
1.026
2.000
0.900
0.930
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.3684 ppm/cm, 147.4 Hz/cm
0.98
1.03
2.00
0.90
0.93
4.29
2.77

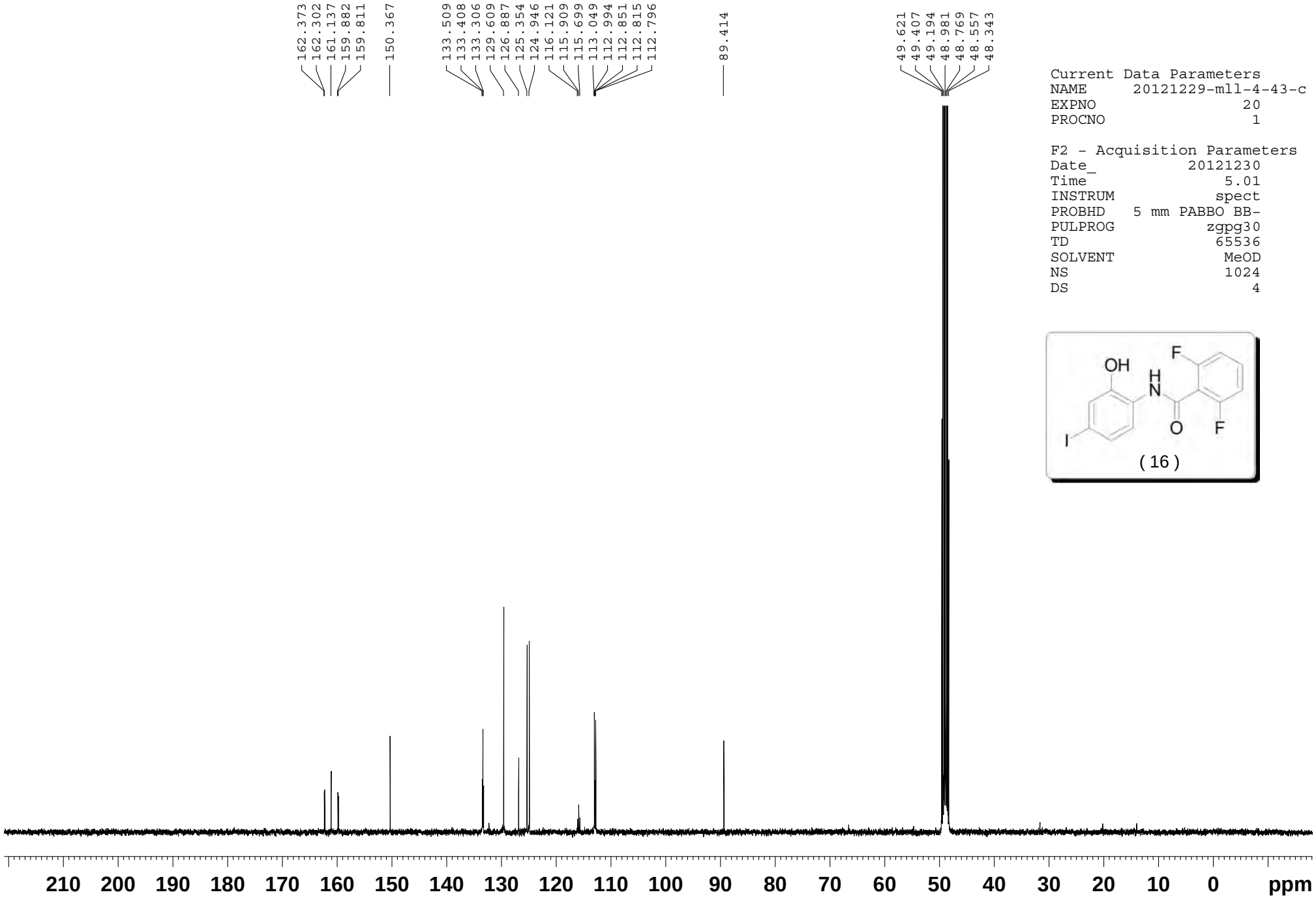


Current Data Parameters
NAME YXL-2-272-1-C-20120904
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120905
Time 1.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024

(15)


7.7638
7.7428
7.5500
7.5314
7.5126
7.4940
7.4760
7.2464
7.2112
7.1899
7.1034
7.0829
7.0626
4.8390
3.3121
3.3094
7.764
7.743
7.550
7.531
7.513
7.494
7.476
7.246
7.211
7.190
7.103
7.083
7.063
Current Data Parameters
NAME 20121229-mll-4-43
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121229
Time 11.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
OH
H
N
F
O
F
I
(16)
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
1.000
0.996
0.932
1.006
1.993
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.4217 ppm/cm, 168.8 Hz/cm
1.00
1.00
0.93
1.01
1.99

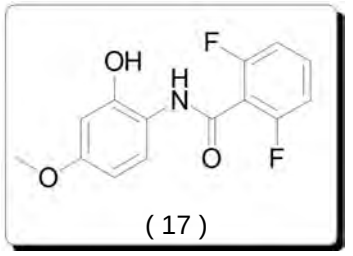

162.373
162.302
161.137
159.882
159.811
150.367
133.509
133.408
133.306
129.609
126.887
125.354
124.946
116.121
115.909
115.699
113.049
112.994
112.851
112.815
112.796
89.414
49.621
49.407
49.194
48.981
48.769
48.557
48.343
Current Data Parameters
NAME 20121229-mll-4-43-c
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121230
Time 5.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
OH
H
N
F
O
F
I
(16)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Data Parameters
NAME YXL-2-260-1-1-H-C-20120829
EXPNO 10
PROCNO 1

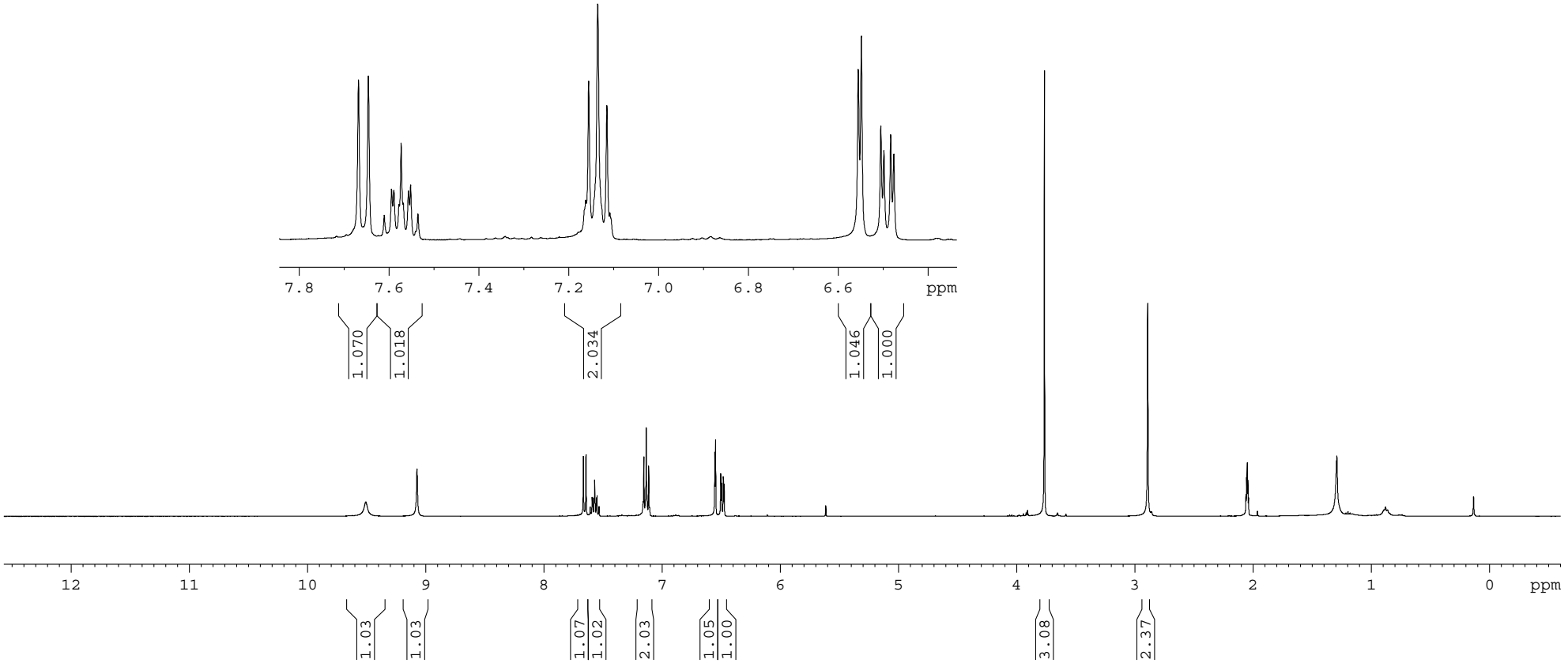
F2 - Acquisition Parameters
Date_ 20120829
Time 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2

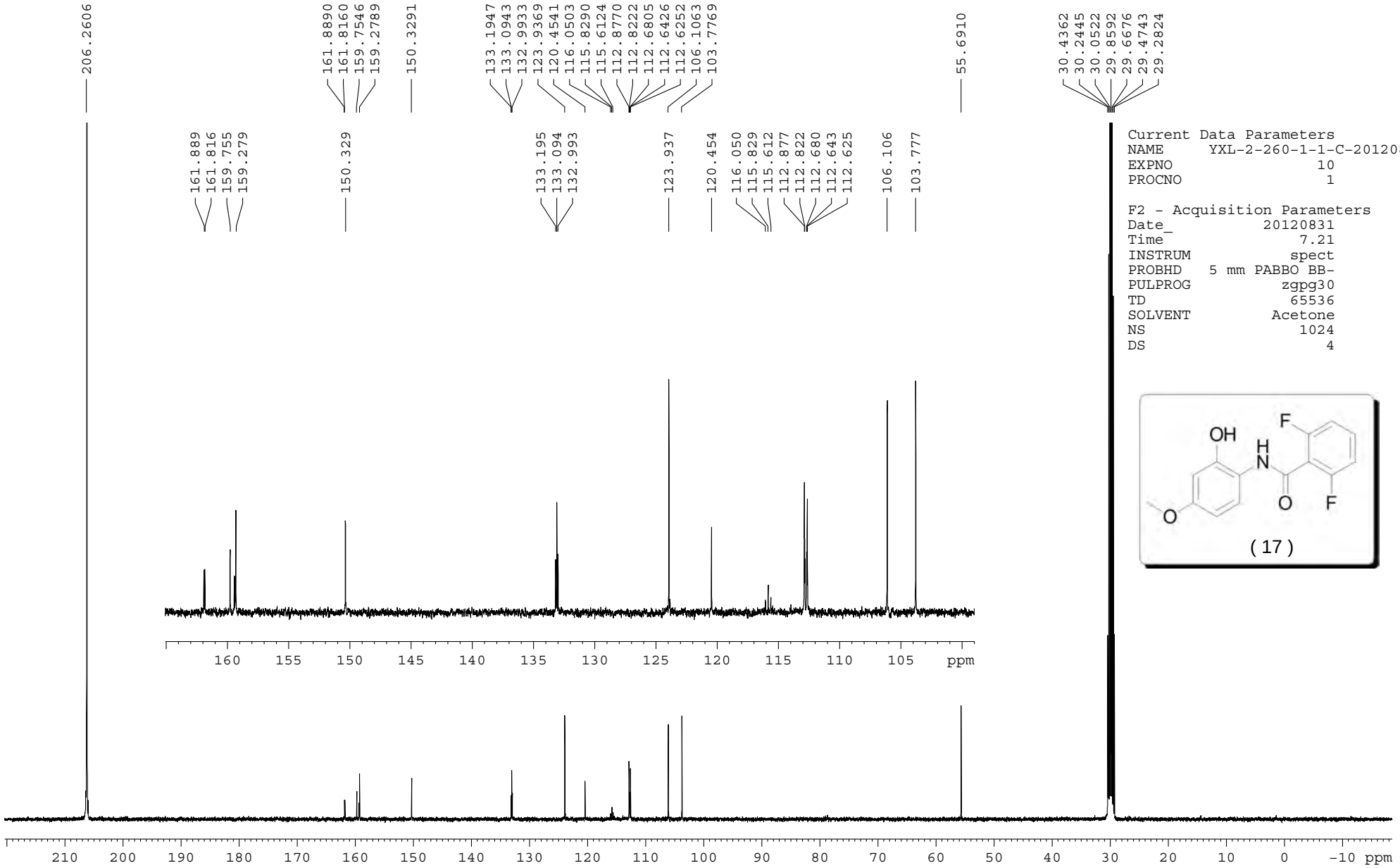
9.5088 9.0754 7.6675 7.6455 7.6101 7.5938 7.5889 7.5775 7.5726 7.5679 7.5563 7.5515 7.5352 7.1624 7.1559 7.1357 7.1151 7.1085 6.5556 6.5487 6.5053 6.4984 6.4832 6.4763 5.6166 3.7662 2.8921 2.0609 2.0554 2.0499 2.0444 2.0390

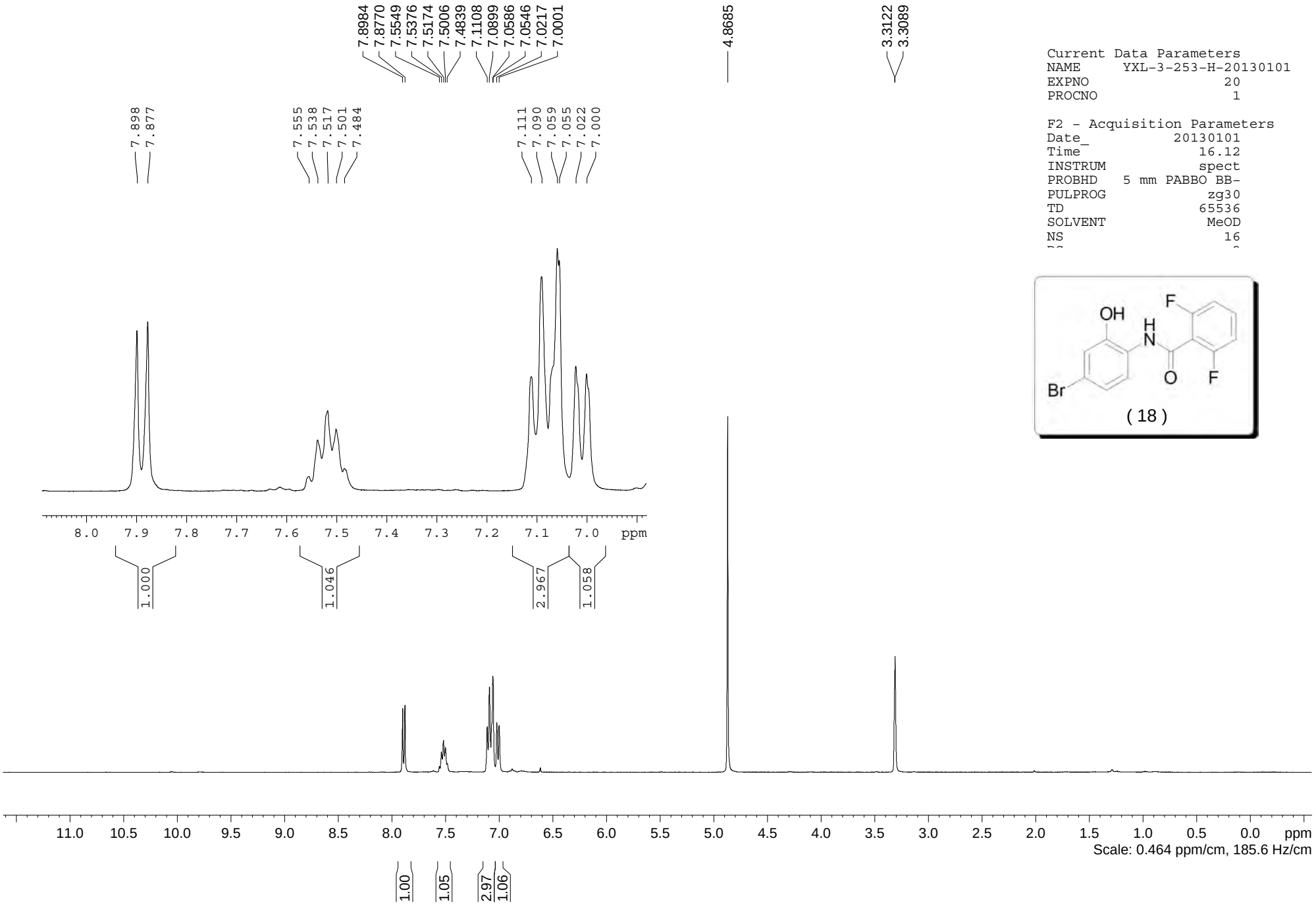
7.668 7.646 7.610 7.594 7.589 7.577 7.573 7.568 7.556 7.551 7.535 7.162 7.156 7.136 7.115 7.109 6.556 6.549 6.505 6.498 6.483 6.476

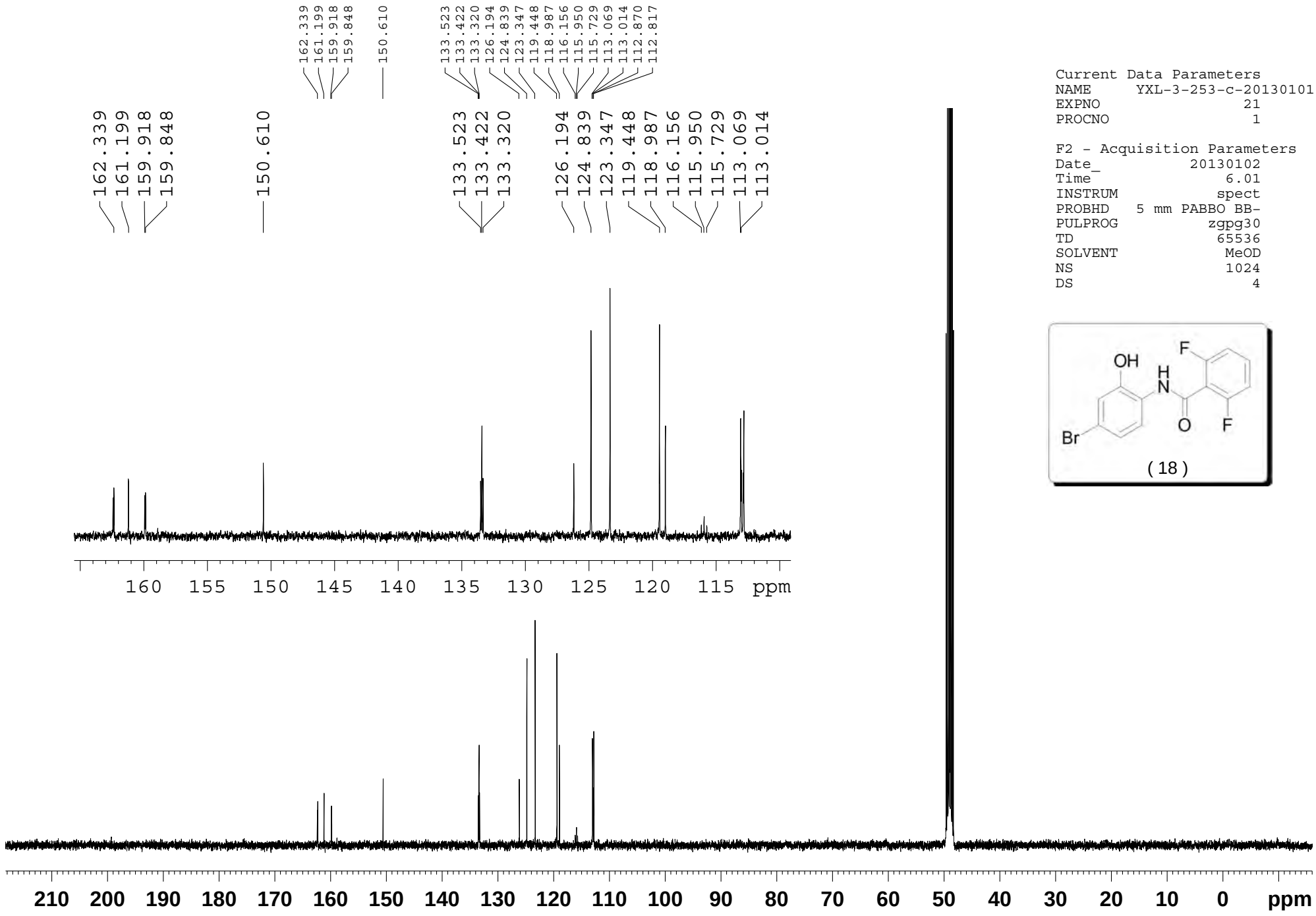


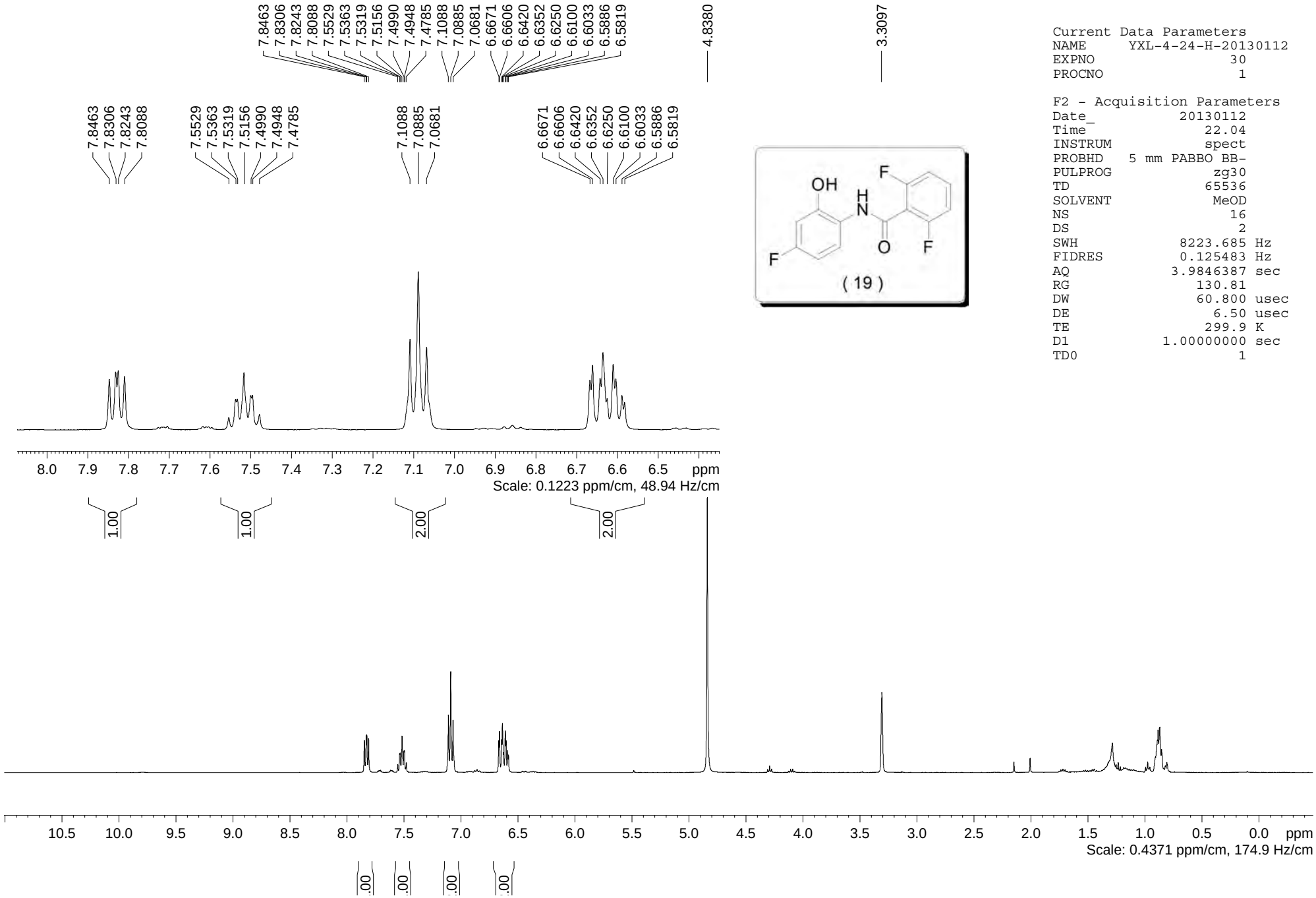
(17)



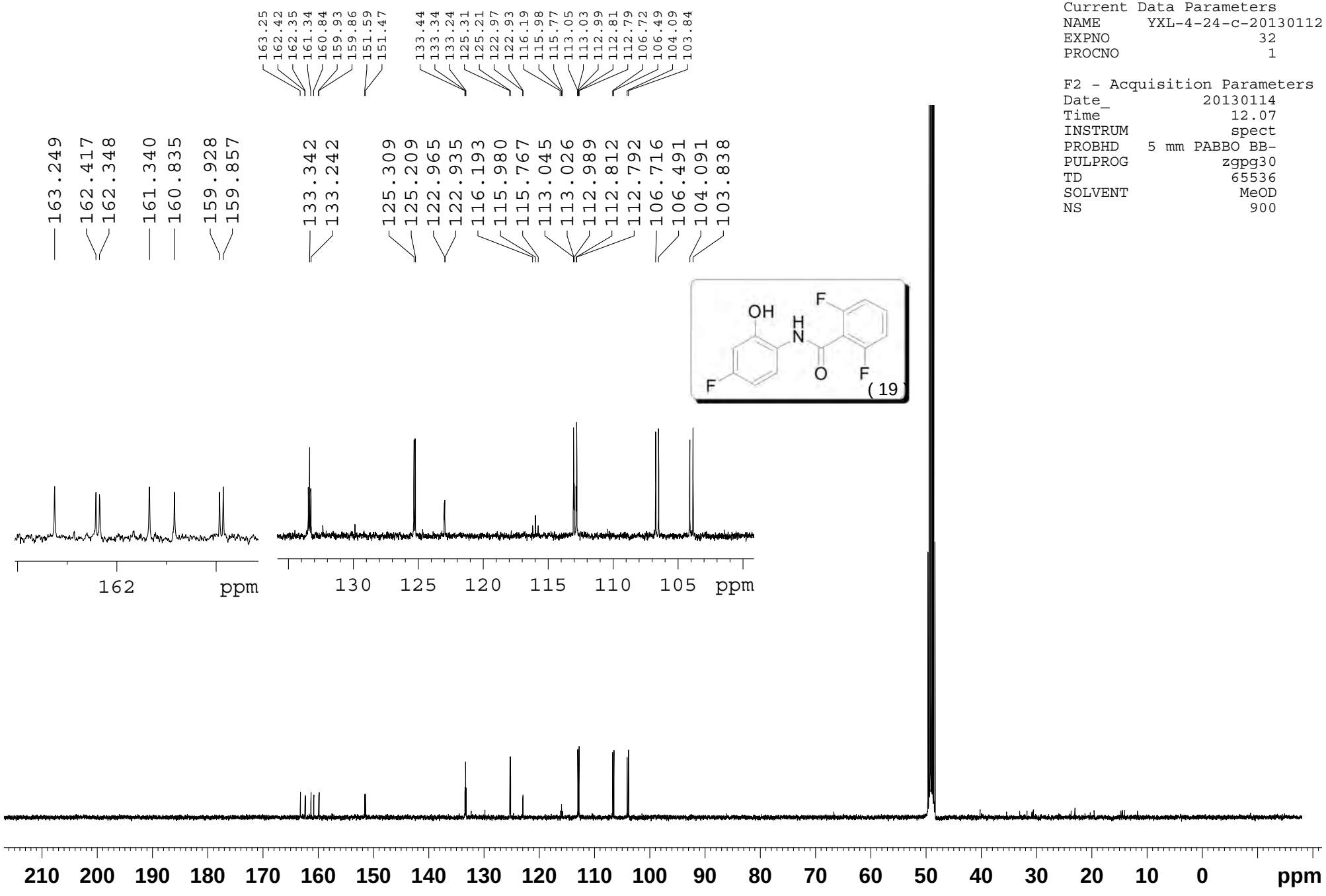

206.2606
161.8890
161.8160
159.7546
159.2789
150.3291
133.1947
133.0943
132.9933
123.9369
120.4541
116.0503
115.8290
115.6124
112.8770
112.8222
112.6805
112.6426
112.6252
106.1063
103.7769
55.6910
30.4362
30.2445
30.0522
29.8592
29.6676
29.4743
29.2824
161.889
161.816
159.755
159.279
150.329
133.195
133.094
132.993
123.937
120.454
116.050
115.829
115.612
112.877
112.822
112.680
112.643
112.625
106.106
103.777
160
155
150
145
140
135
130
125
120
115
110
105
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10 ppm
Current Data Parameters
NAME YXL-2-260-1-1-C-2012083
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120831
Time 7.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
OH
H
N
F
O
O
F
(17)


7.8984
7.8770
7.5549
7.5376
7.5174
7.5006
7.4839
7.1108
7.0899
7.0586
7.0546
7.0217
7.0001
4.8685
3.3122
3.3089
7.898
7.877
7.555
7.538
7.517
7.501
7.484
7.111
7.090
7.059
7.055
7.022
7.000
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
ppm
1.000
1.046
2.967
1.058
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.05
2.97
1.06
Scale: 0.464 ppm/cm, 185.6 Hz/cm
Current Data Parameters
NAME YXL-3-253-H-20130101
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130101
Time 16.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
OH
H
N
F
F
O
Br
(18)


162.339
161.199
159.918
159.848
150.610
133.523
133.422
133.320
126.194
124.839
123.347
119.448
118.987
116.156
115.950
115.729
113.069
113.014
112.870
112.817
Current Data Parameters
NAME YXL-3-253-c-20130101
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130102
Time 6.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
OH
H
N
F
O
F
Br
(18)
160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

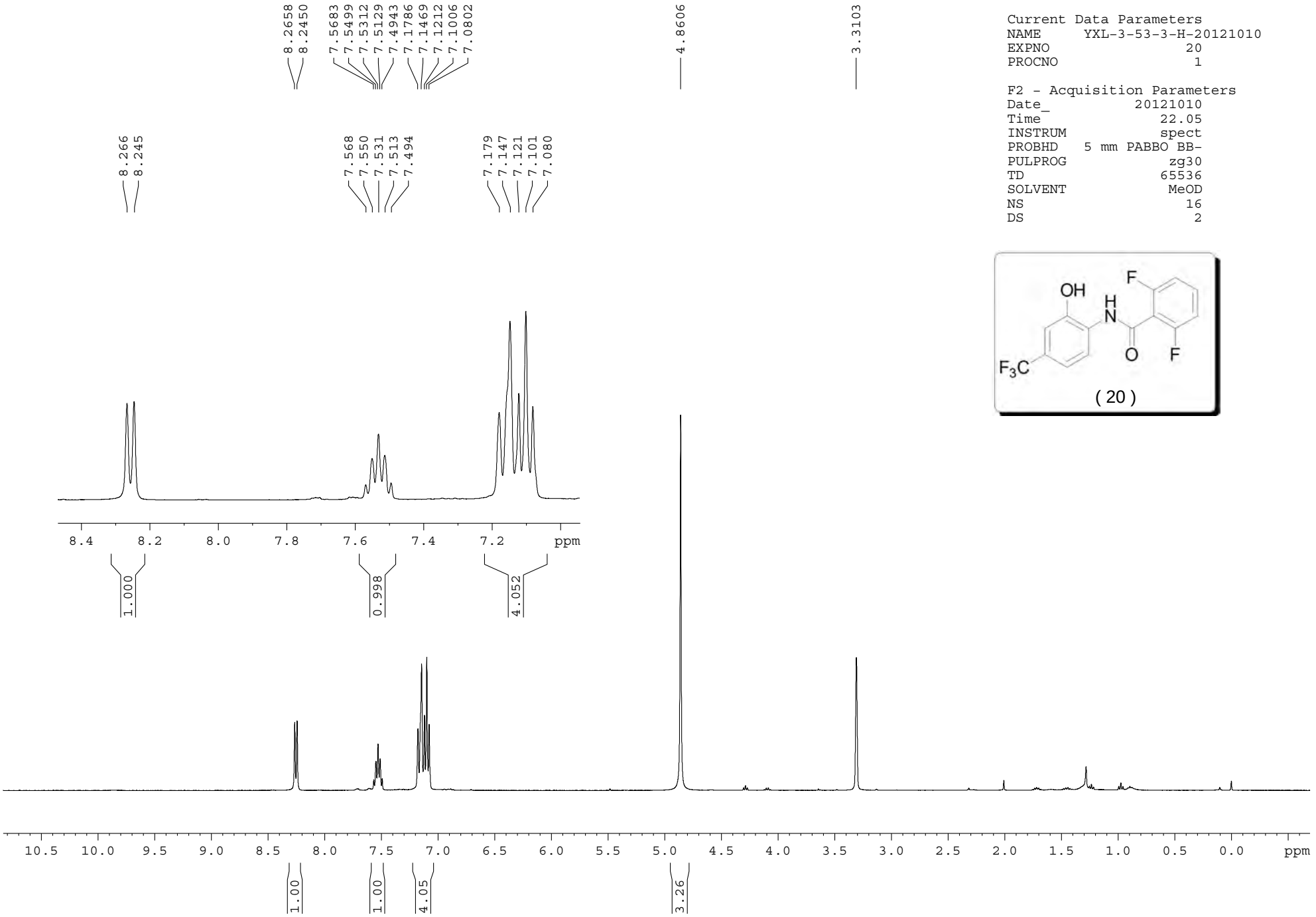



```
Current Data Parameters
NAME      YXL-4-24-H-20130112
EXPNO                      30
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130112
Time                  22.04
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                    130.81
DW                    60.800 usec
DE                     6.50 usec
TE                    299.9 K
D1              1.00000000 sec
TD0                       1
```



Current Data Parameters
NAME YXL-4-24-c-20130112
EXPNO 32
PROCNO 1

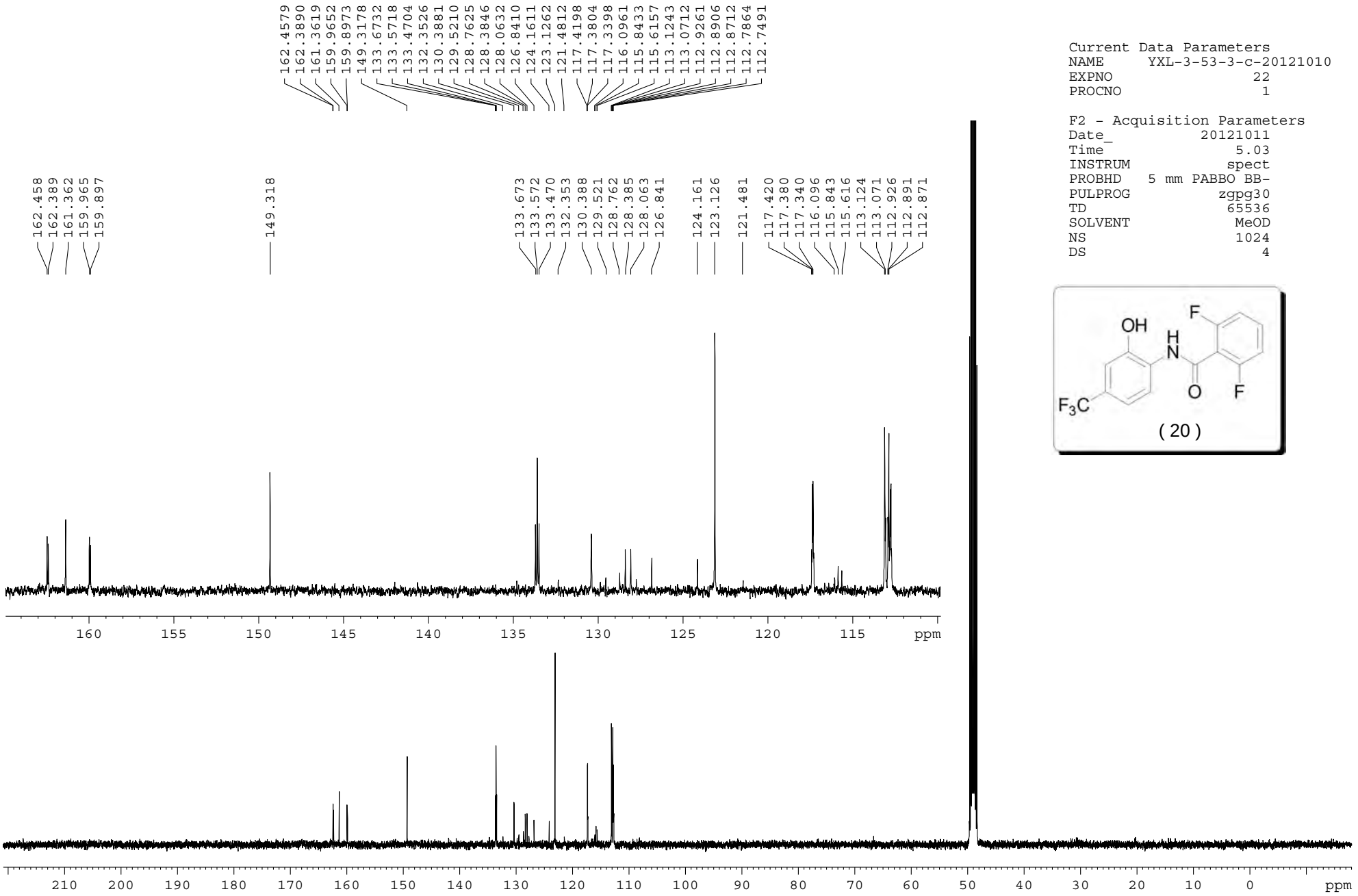
F2 - Acquisition Parameters
Date_ 20130114
Time 12.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 900



Current Data Parameters
NAME YXL-3-53-3-H-20121010
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121010
Time 22.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2

(20)

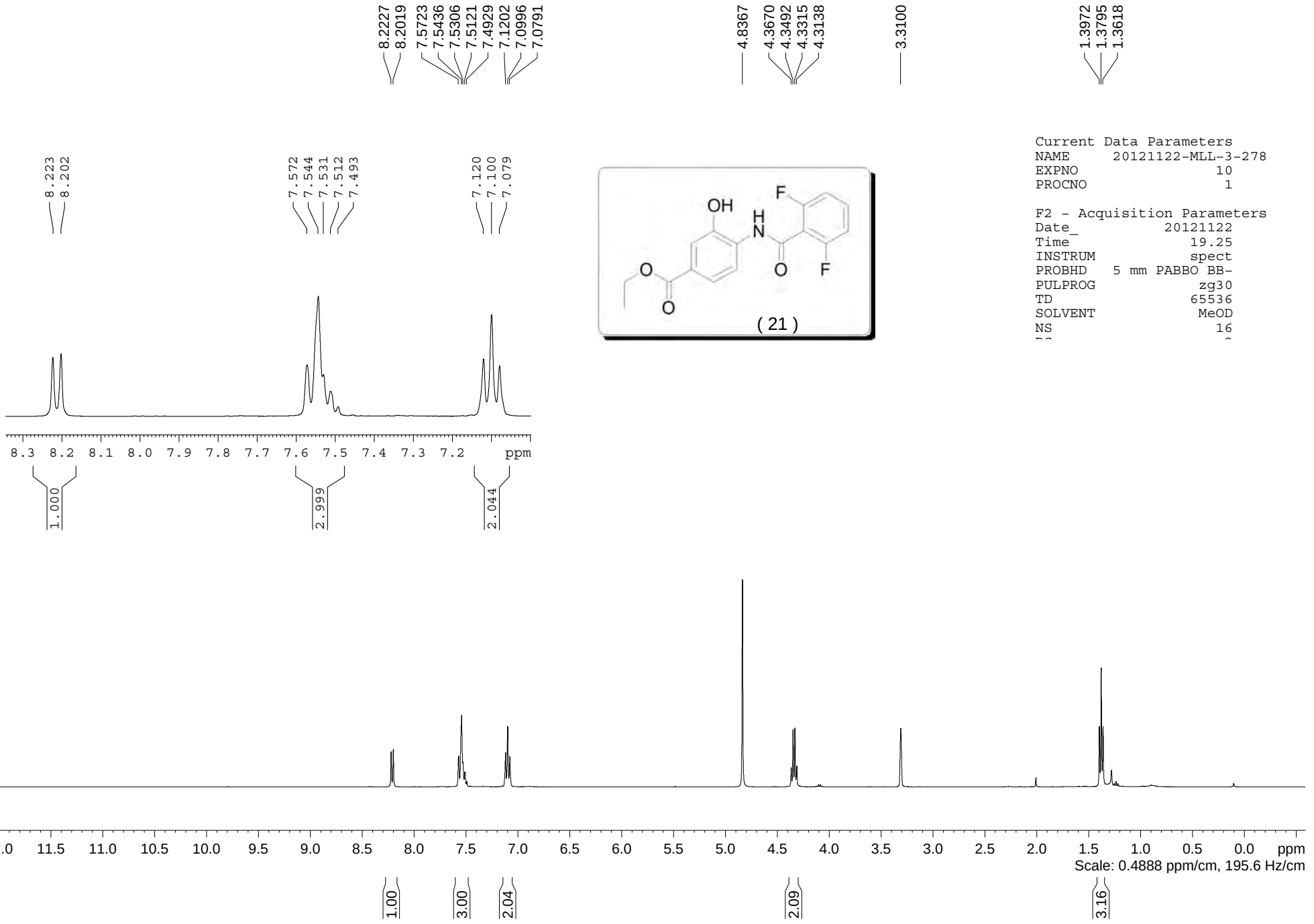


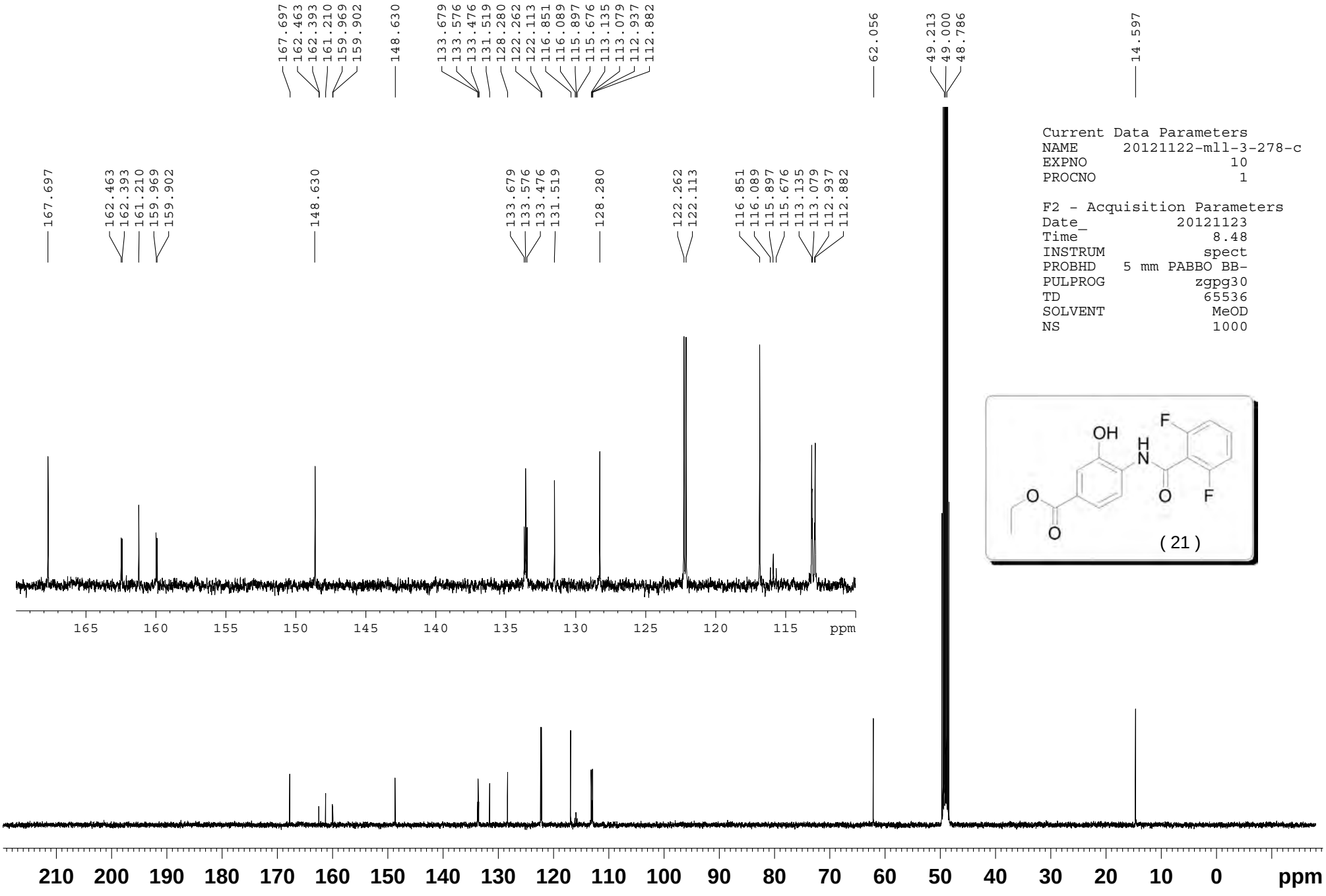
Current Data Parameters
NAME YXL-3-53-3-c-20121010
EXPNO 22
PROCNO 1

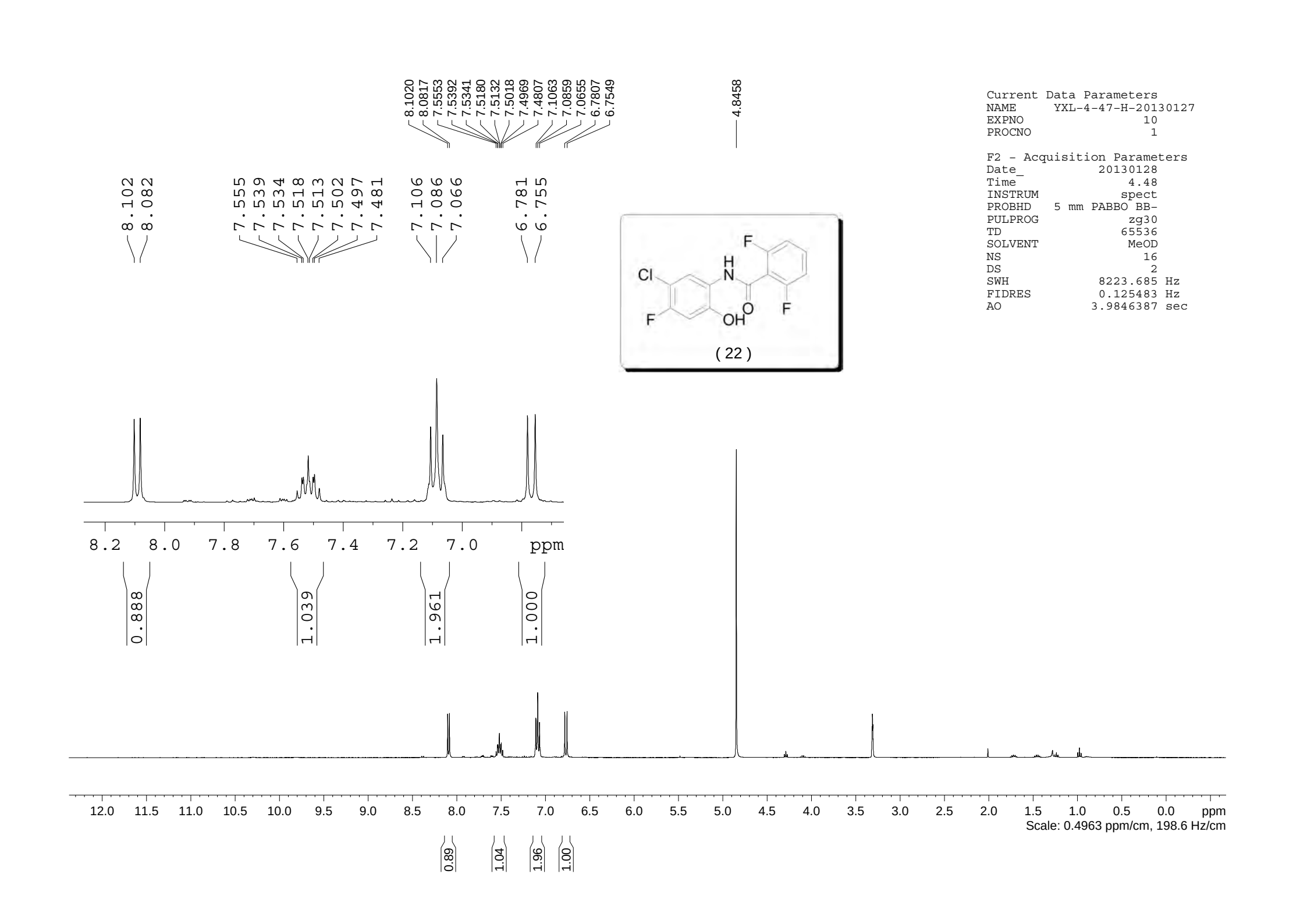
F2 - Acquisition Parameters
Date_ 20121011
Time 5.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4

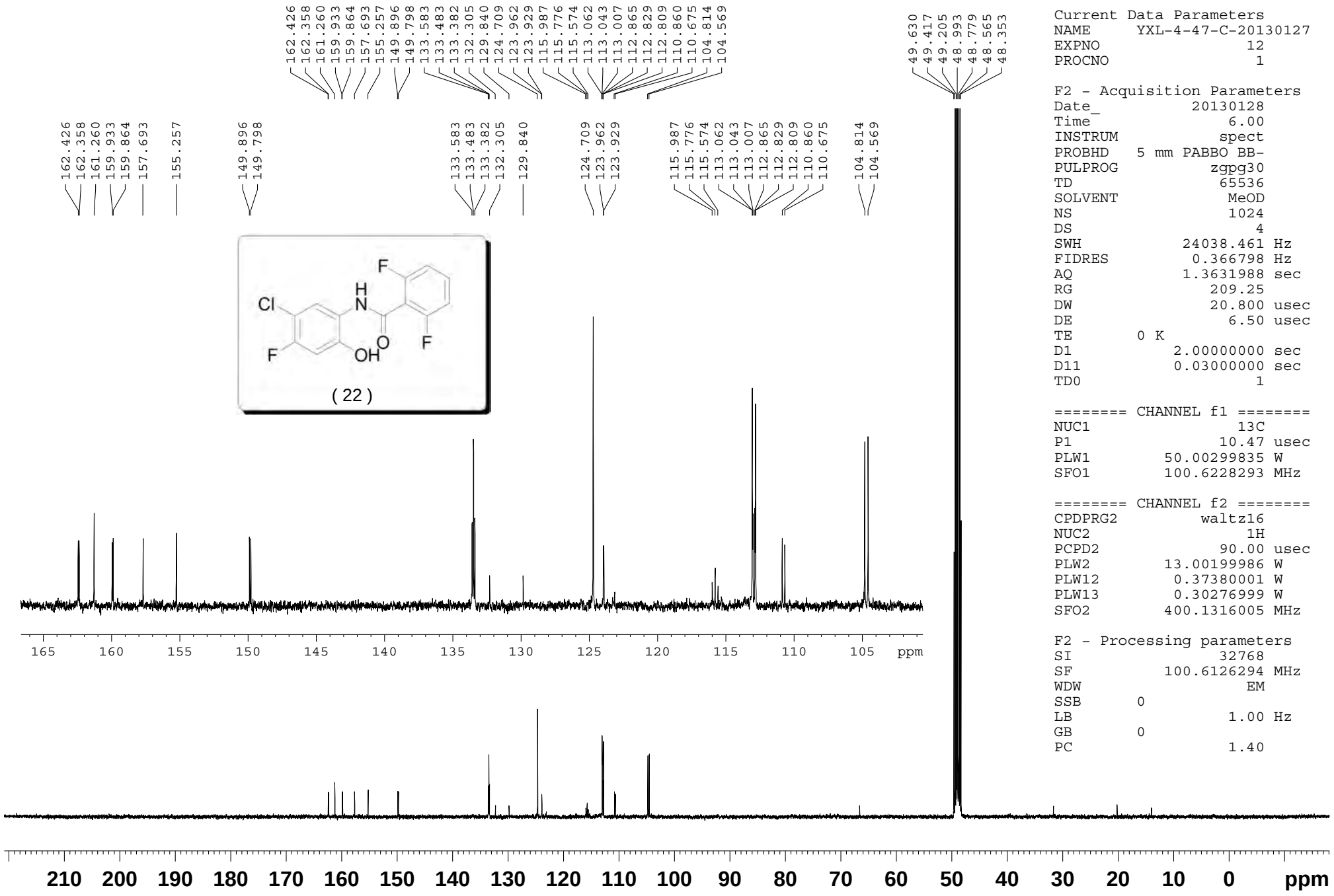
(20)

(21)

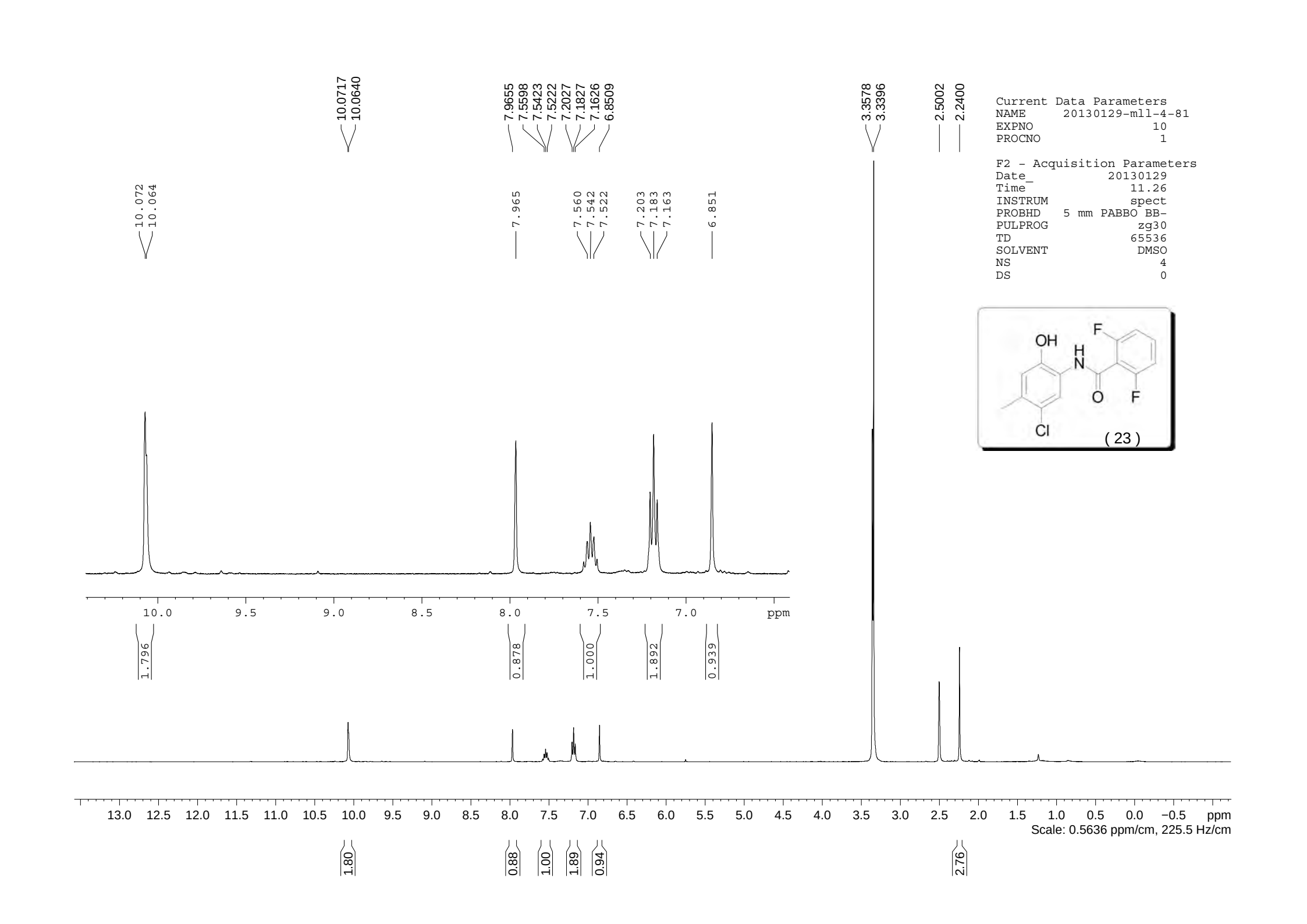

8.2227
8.2019
7.5723
7.5436
7.5306
7.5121
7.4929
7.1202
7.0996
7.0791
4.8367
4.3670
4.3492
4.3315
4.3138
3.3100
1.3972
1.3795
1.3618
8.223
8.202
7.572
7.544
7.531
7.512
7.493
7.120
7.100
7.079
OH
H
N
F
F
O
O
O
(21)
Current Data Parameters
NAME 20121122-MLL-3-278
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121122
Time 19.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
1.000
2.999
2.044
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.4888 ppm/cm, 195.6 Hz/cm
1.00
3.00
2.04
2.09
3.16

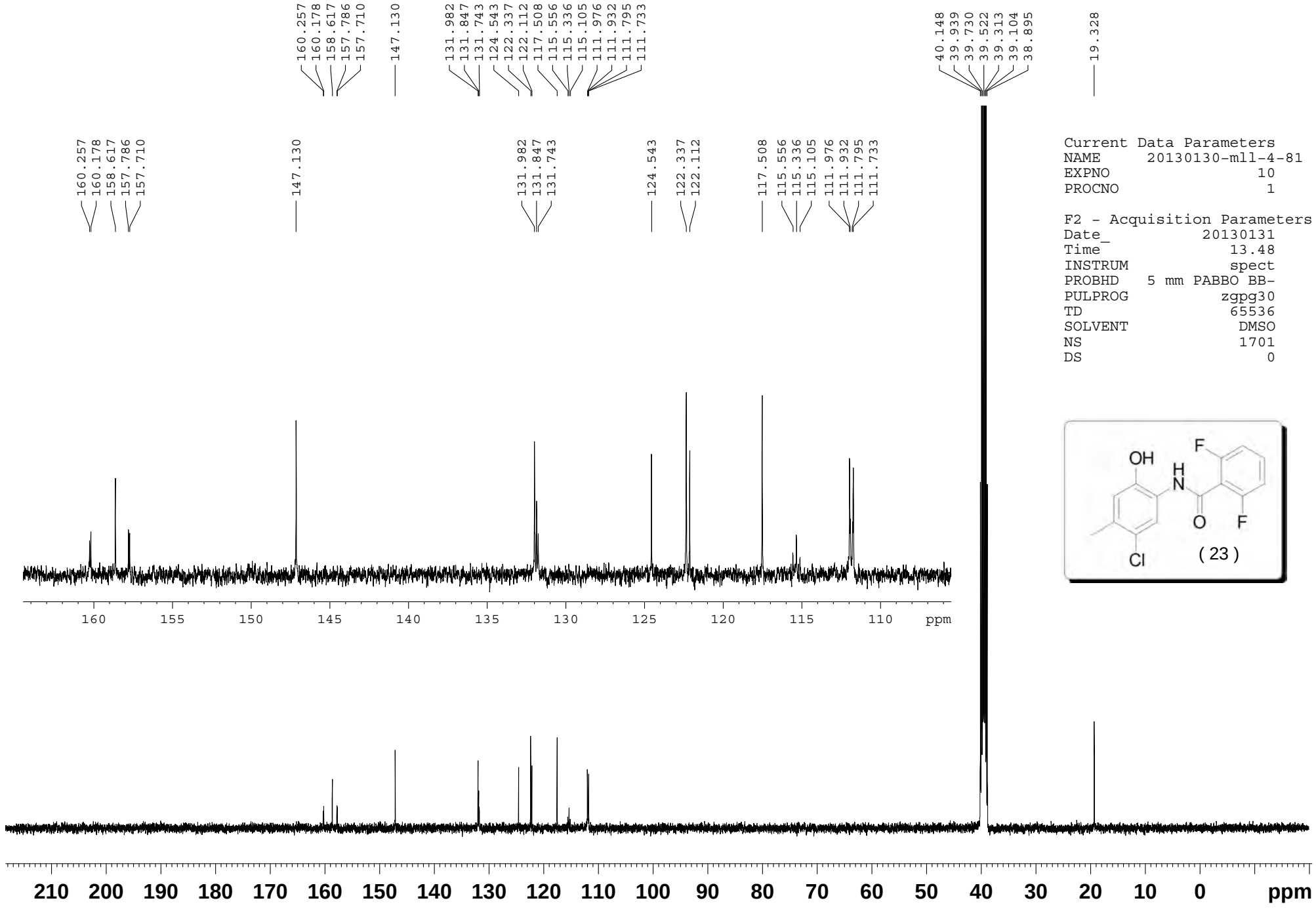

167.697
162.463
162.393
161.210
159.969
159.902
148.630
133.679
133.576
133.476
131.519
128.280
122.262
122.113
116.851
116.089
115.897
115.676
113.135
113.079
112.937
112.882
62.056
49.213
49.000
48.786
14.597
Current Data Parameters
NAME 20121122-mll-3-278-c
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121123
Time 8.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1000
OH
H
N
F
O
O
O
F
(21)
165 160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

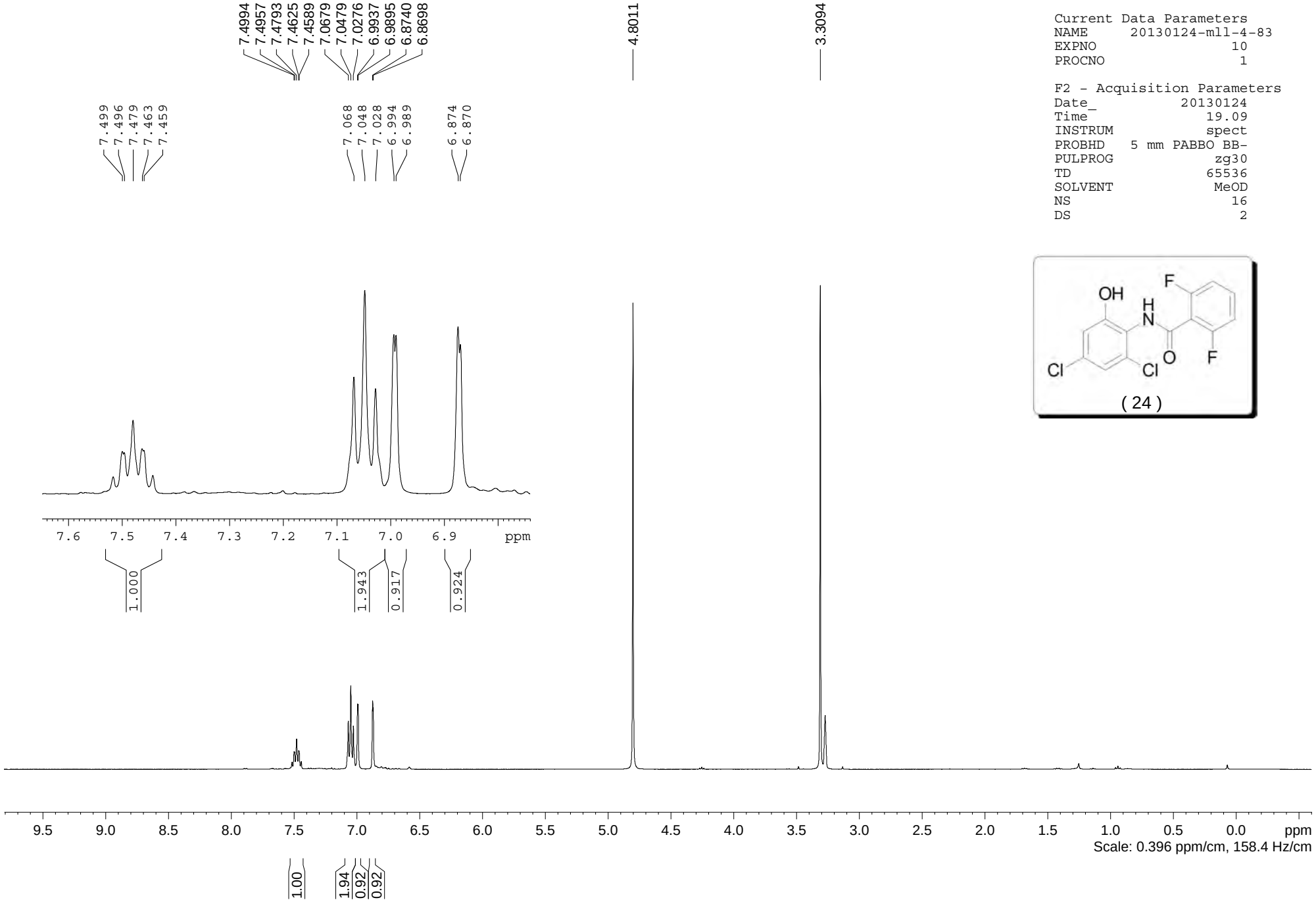

8.1020
8.0817
7.5553
7.5392
7.5341
7.5180
7.5132
7.5018
7.4969
7.4807
7.1063
7.0859
7.0655
6.7807
6.7549
4.8458
8.102
8.082
7.555
7.539
7.534
7.518
7.513
7.502
7.497
7.481
7.106
7.086
7.066
6.781
6.755
Current Data Parameters
NAME YXL-4-47-H-20130127
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130128
Time 4.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AO 3.9846387 sec
Cl
F
H
N
O
F
F
OH
(22)
8.2
8.0
7.8
7.6
7.4
7.2
7.0
ppm
0.888
1.039
1.961
1.000
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4963 ppm/cm, 198.6 Hz/cm
0.89
1.04
1.96
1.00




```
Current Data Parameters
NAME      YXL-4-47-C-20130127
EXPNO                     12
PROCNO                     1

F2 - Acquisition Parameters
Date_              20130128
Time                   6.00
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                MeOD
NS                     1024
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                   209.25
DW                   20.800 usec
DE                    6.50 usec
TE          0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.47 usec
PLW1          50.00299835 W
SFO1          100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2          13.00199986 W
PLW12          0.37380001 W
PLW13          0.30276999 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6126294 MHz
WDW                     EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```


10.0717
10.0640
7.9655
7.5598
7.5423
7.5222
7.2027
7.1827
7.1626
6.8509
3.3578
3.3396
2.5002
2.2400
10.072
10.064
7.965
7.560
7.542
7.522
7.203
7.183
7.163
6.851
1.796
0.878
1.000
1.892
0.939
1.80
0.88
1.00
1.89
0.94
2.76
ppm
Scale: 0.5636 ppm/cm, 225.5 Hz/cm
Current Data Parameters
NAME 20130129-mll-4-81
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130129
Time 11.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 4
DS 0
OH
H
N
F
O
F
Cl
(23)


160.257
160.178
158.617
157.786
157.710
147.130
131.982
131.847
131.743
124.543
122.337
122.112
117.508
115.556
115.336
115.105
111.976
111.932
111.795
111.733
40.148
39.939
39.730
39.522
39.313
39.104
38.895
19.328
Current Data Parameters
NAME 20130130-mll-4-81
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time 13.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1701
DS 0
OH
HN
O
F
F
Cl
(23)
ppm

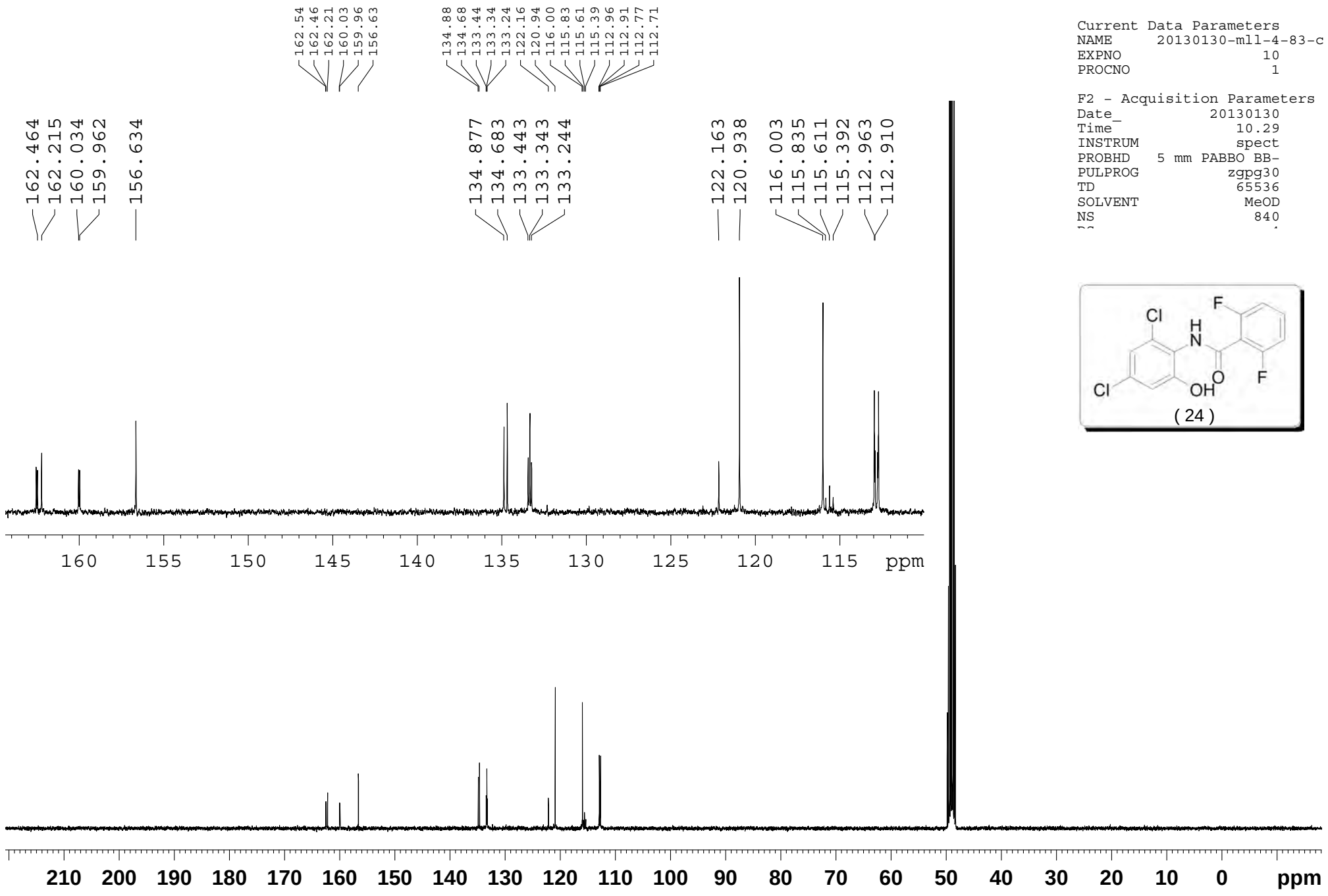


Current Data Parameters
NAME 20130124-mll-4-83
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130124
Time 19.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2

(24)

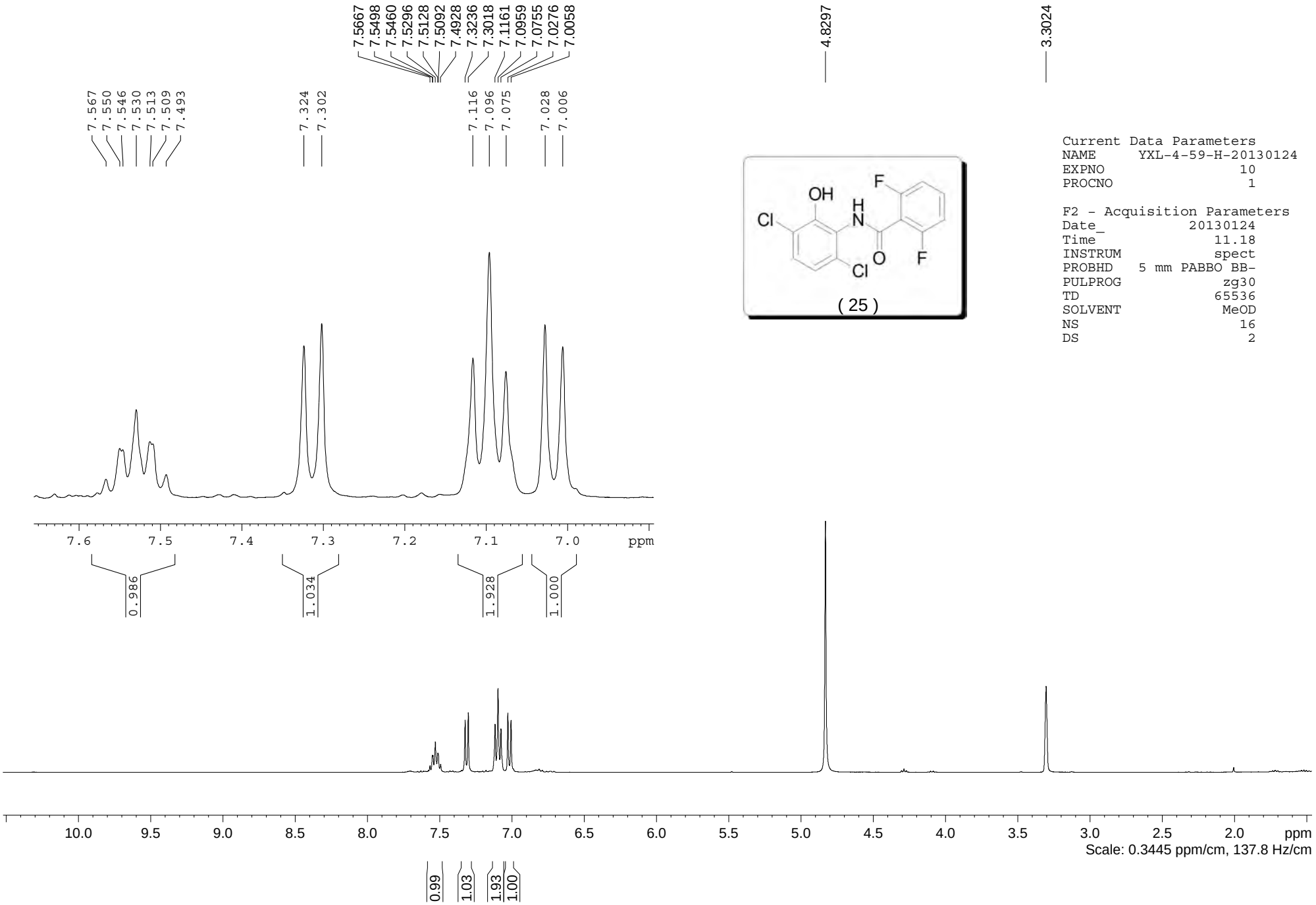
Scale: 0.396 ppm/cm, 158.4 Hz/cm

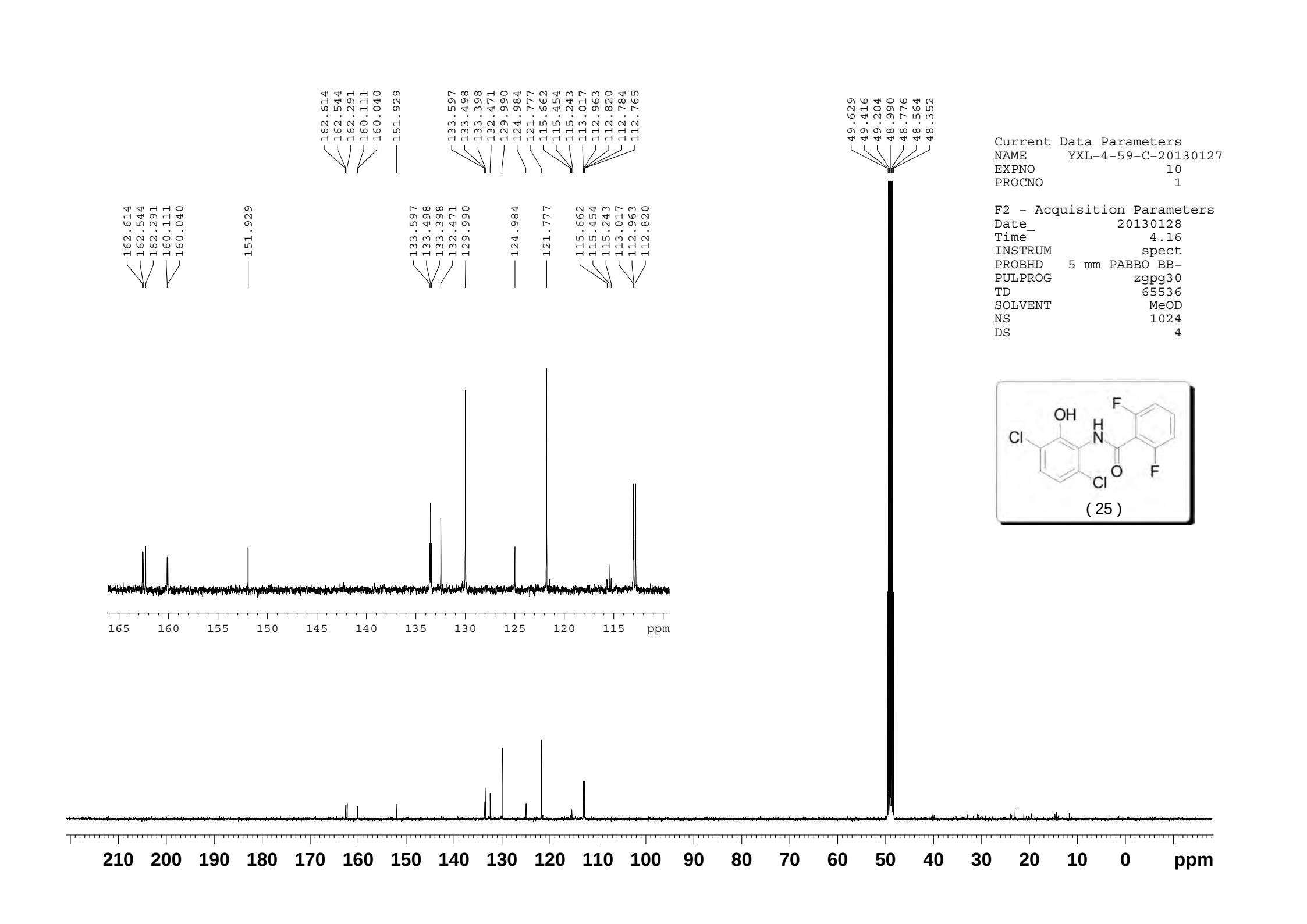


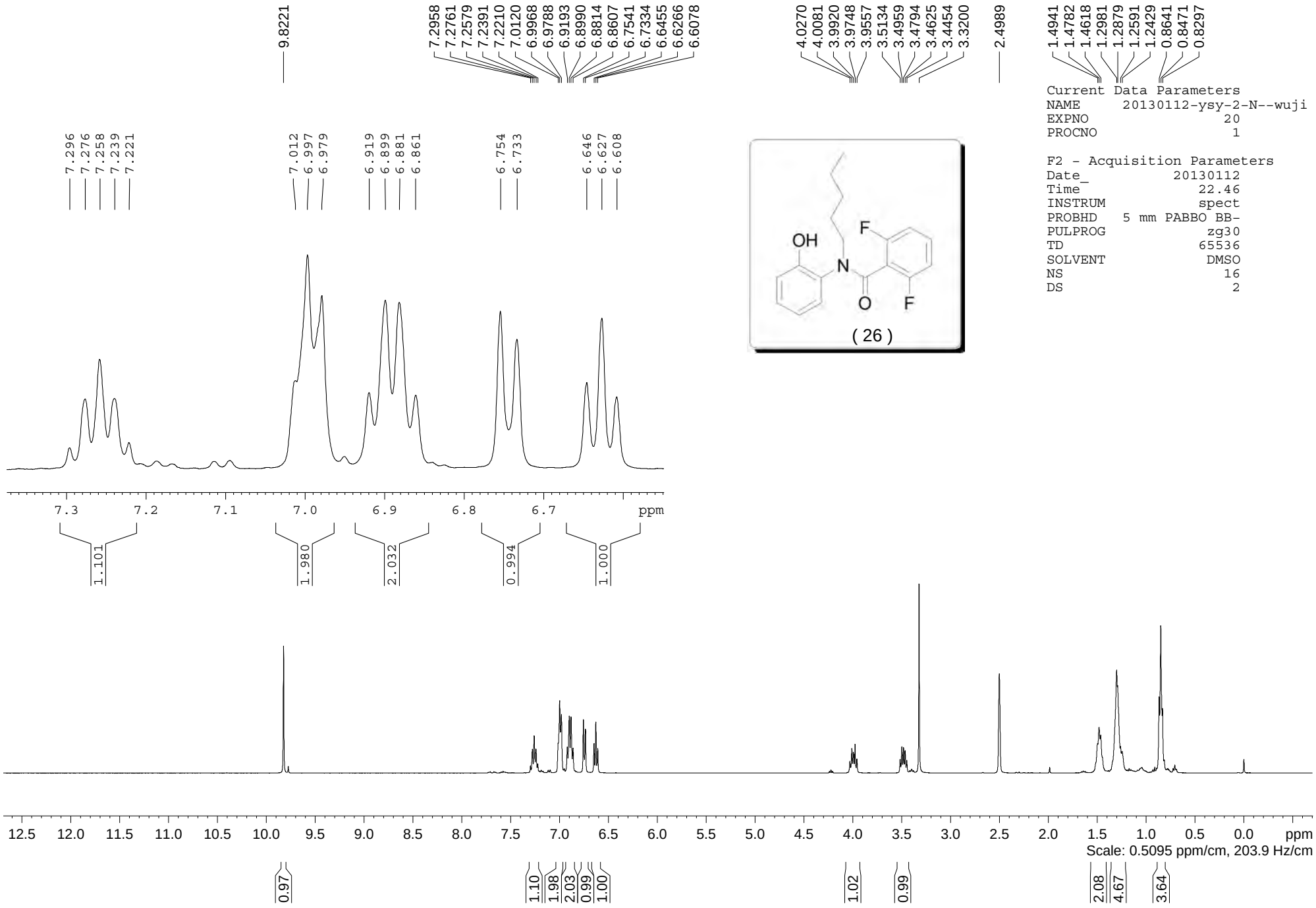
Current Data Parameters
NAME 20130130-mll-4-83-c
EXPNO 10
PROCNO 1

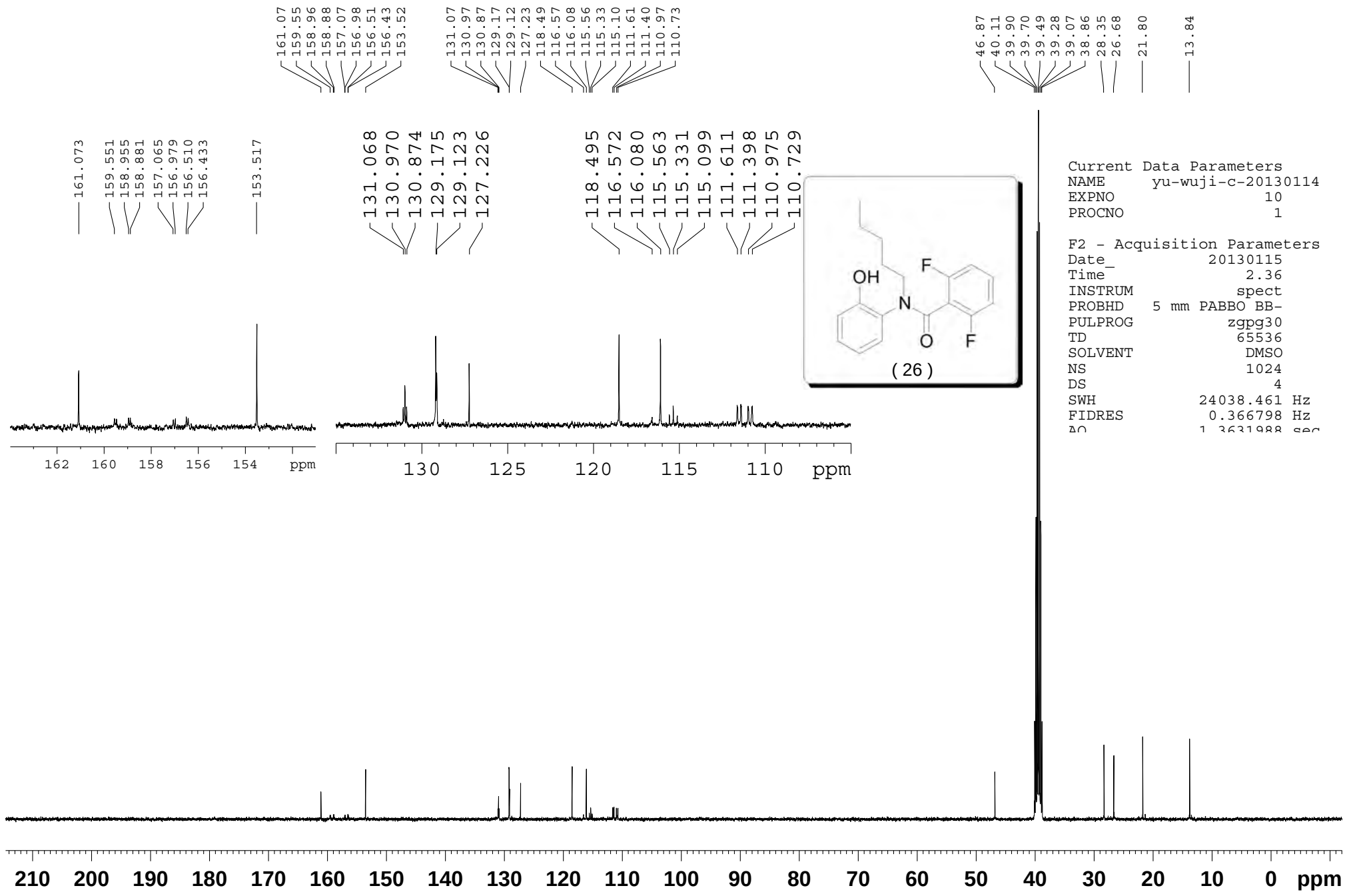
F2 - Acquisition Parameters
Date_ 20130130
Time 10.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 840

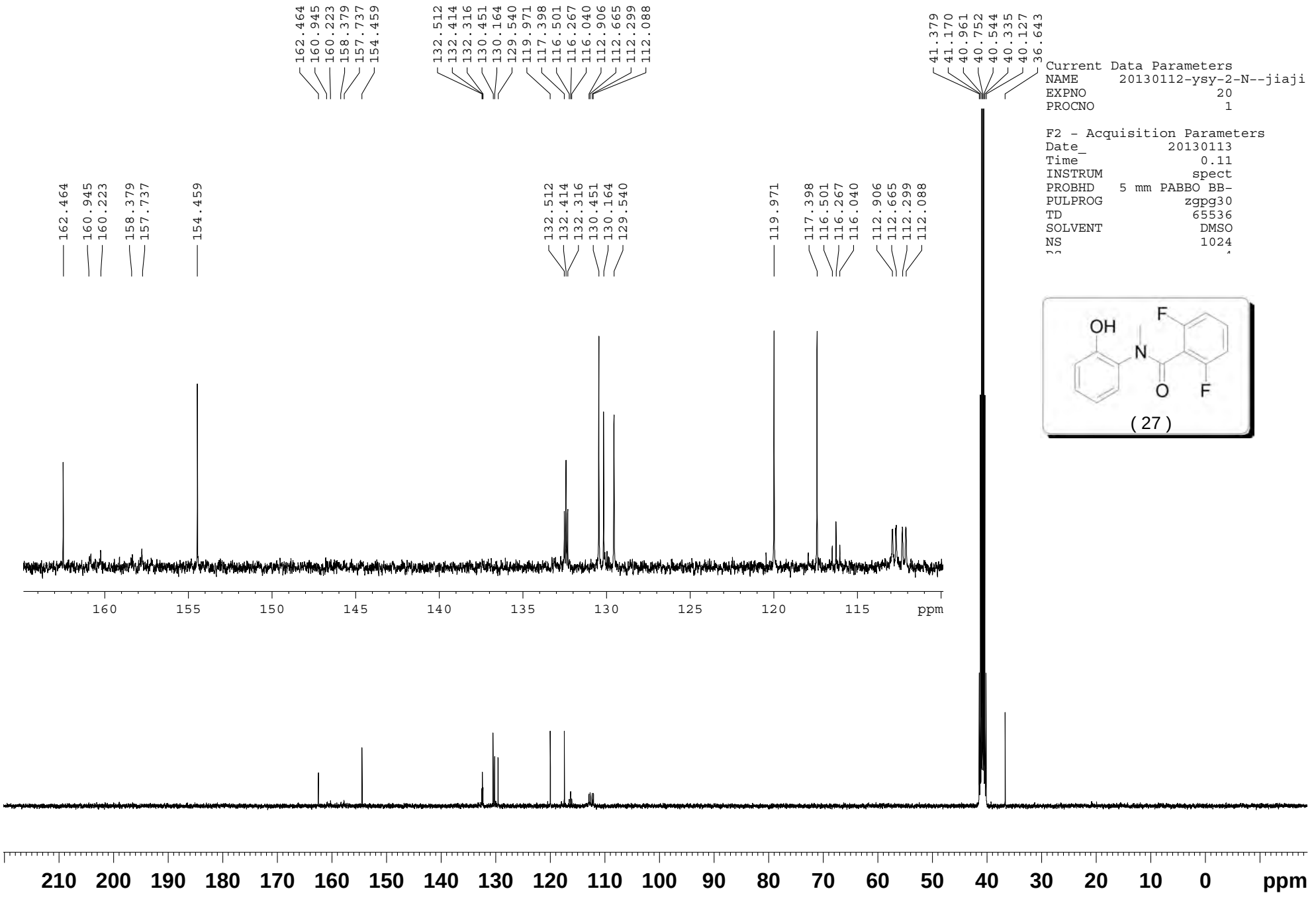
(24)

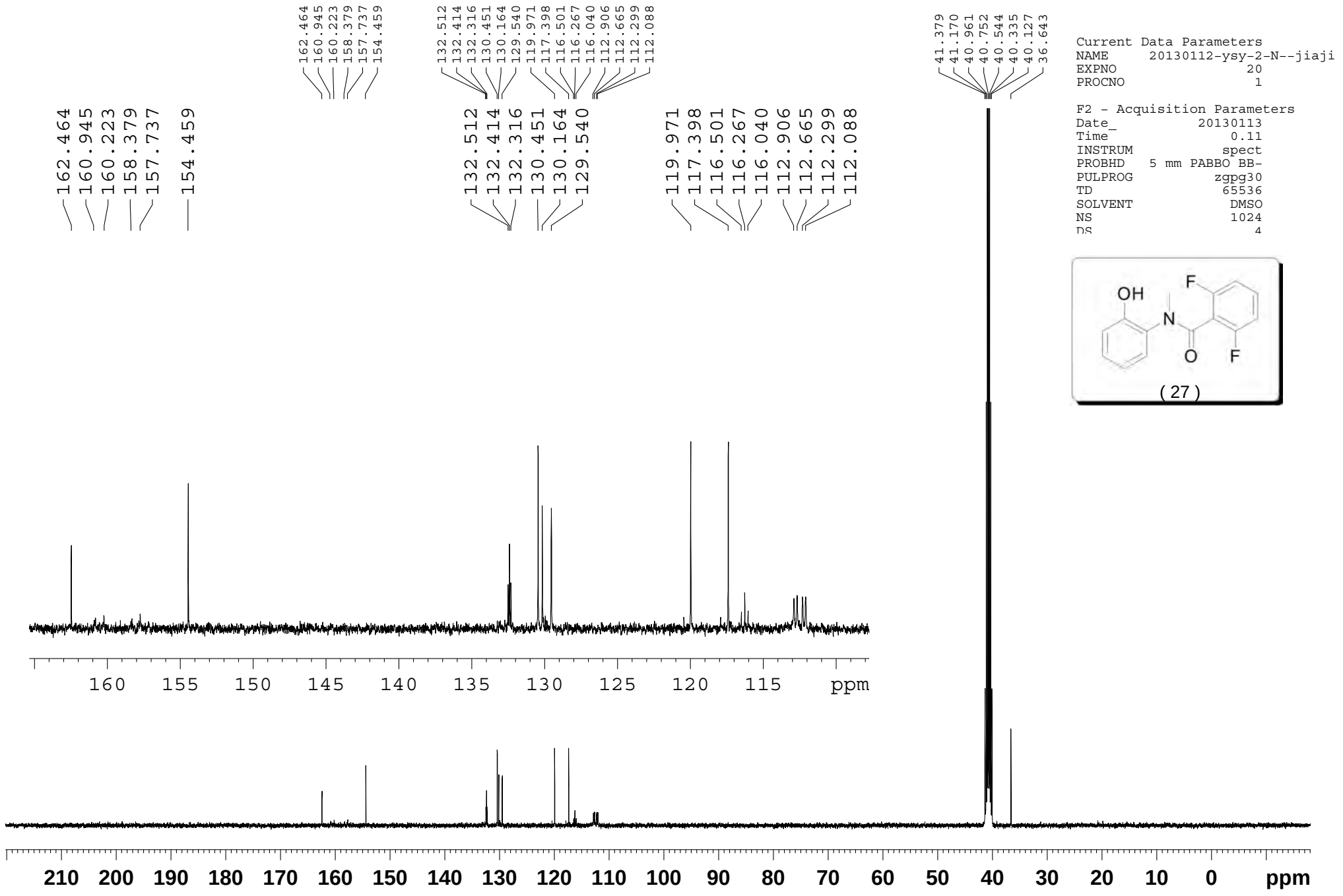

7.5667
7.5498
7.5460
7.5296
7.5128
7.5092
7.4928
7.3236
7.3018
7.1161
7.0959
7.0755
7.0276
7.0058
4.8297
3.3024
7.567
7.550
7.546
7.530
7.513
7.509
7.493
7.324
7.302
7.116
7.096
7.075
7.028
7.006
OH
Cl
H
N
O
F
F
Cl
(25)
Current Data Parameters
NAME YXL-4-59-H-20130124
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130124
Time 11.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
7.6
7.5
7.4
7.3
7.2
7.1
7.0
ppm
0.986
1.034
1.928
1.000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
0.99
1.03
1.93
1.00
Scale: 0.3445 ppm/cm, 137.8 Hz/cm

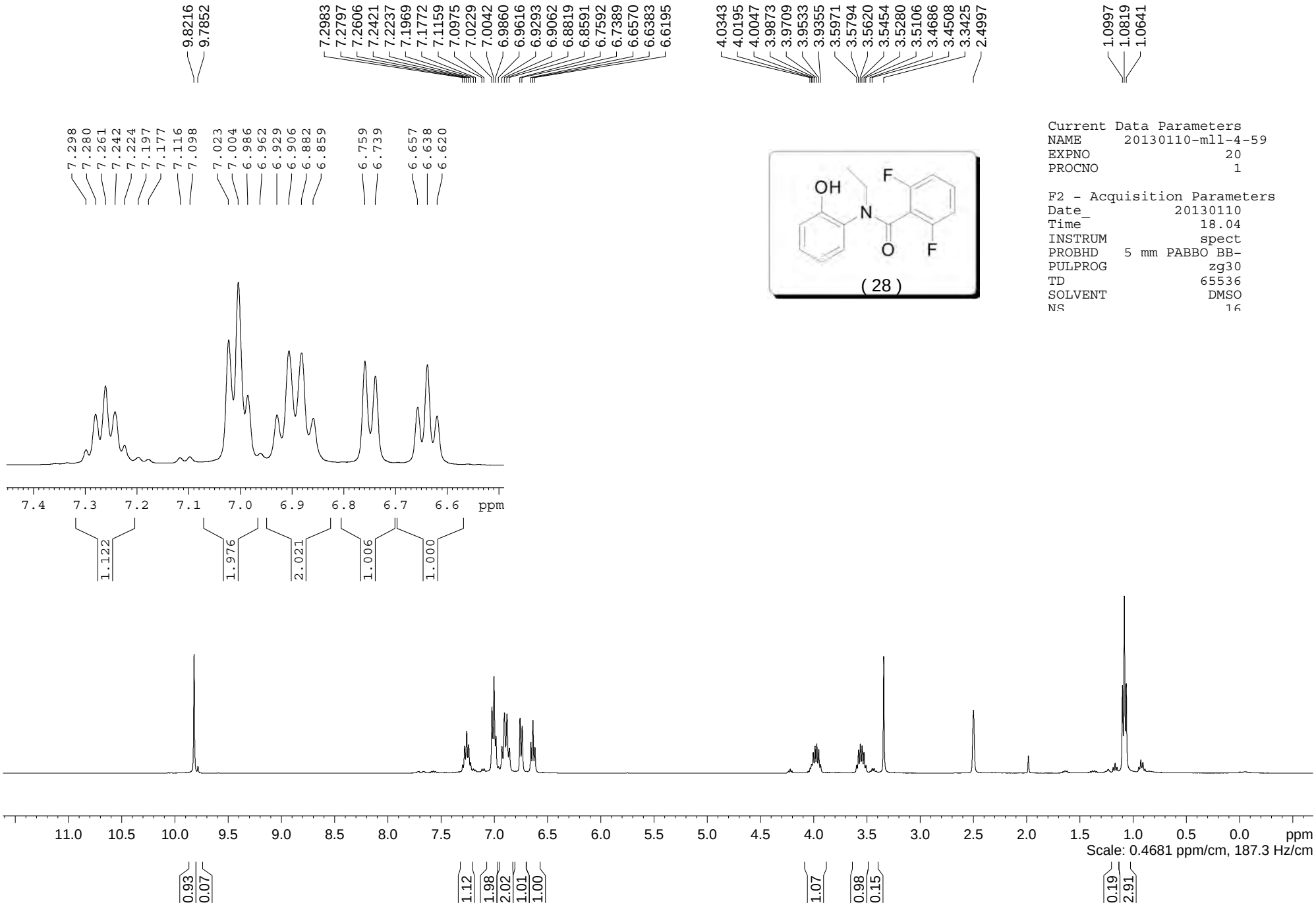

162.614
162.544
162.291
160.111
160.040
151.929
133.597
133.498
133.398
132.471
129.990
124.984
121.777
115.662
115.454
115.243
113.017
112.963
112.820
112.784
112.765
49.629
49.416
49.204
48.990
48.776
48.564
48.352
Current Data Parameters
NAME YXL-4-59-C-20130127
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130128
Time 4.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
OH
F
Cl
H
N
O
F
Cl
(25)
165 160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

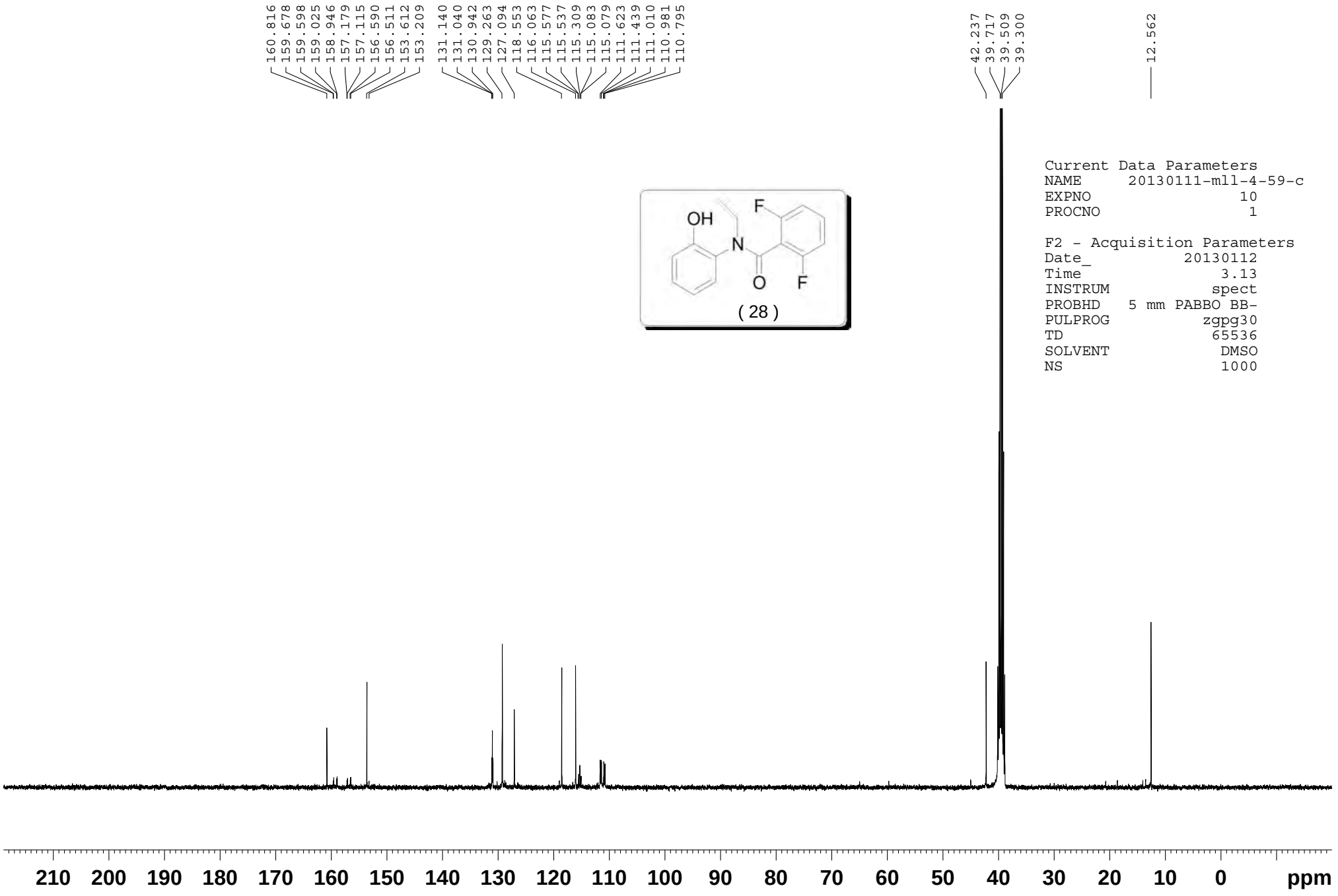

9.8221
7.2958
7.2761
7.2579
7.2391
7.2210
7.0120
6.9968
6.9788
6.9193
6.8990
6.8814
6.8607
6.7541
6.7334
6.6455
6.6266
6.6078
4.0270
4.0081
3.9920
3.9748
3.9557
3.5134
3.4959
3.4794
3.4625
3.4454
3.3200
2.4989
1.4941
1.4782
1.4618
1.2981
1.2879
1.2591
1.2429
0.8641
0.8471
0.8297
7.296
7.276
7.258
7.239
7.221
7.012
6.997
6.979
6.919
6.899
6.881
6.861
6.754
6.733
6.646
6.627
6.608
OH
F
N
O
F
(26)
Current Data Parameters
NAME 20130112-ysy-2-N--wuji
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130112
Time 22.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
7.3
7.2
7.1
7.0
6.9
6.8
6.7
ppm
1.101
1.980
2.032
0.994
1.000
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.5095 ppm/cm, 203.9 Hz/cm
0.97
1.10
1.98
2.03
0.99
1.00
1.02
0.99
2.08
4.67
3.64

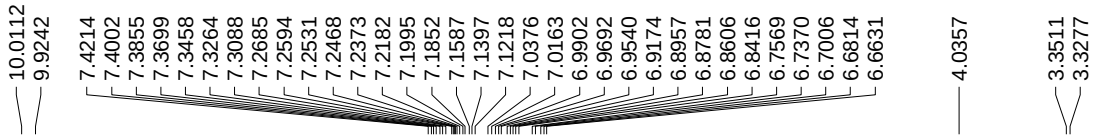

161.07
159.55
158.96
158.88
157.07
156.98
156.51
156.43
153.52
131.07
130.97
130.87
129.17
129.12
127.23
118.49
116.57
116.08
115.56
115.33
115.10
111.61
111.40
110.97
110.73
46.87
40.11
39.90
39.70
39.49
39.28
39.07
38.86
28.35
26.68
21.80
13.84
161.073
159.551
158.955
158.881
157.065
156.979
156.510
156.433
153.517
131.068
130.970
130.874
129.175
129.123
127.226
118.495
116.572
116.080
115.563
115.331
115.099
111.611
111.398
110.975
110.729
OH
N
F
O
F
(26)
162
160
158
156
154
ppm
130
125
120
115
110
ppm
Current Data Parameters
NAME yu-wuji-c-20130114
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130115
Time 2.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


162.464
160.945
160.223
158.379
157.737
154.459
132.512
132.414
132.316
130.451
130.164
129.540
119.971
117.398
116.501
116.267
116.040
112.906
112.665
112.299
112.088
41.379
41.170
40.961
40.752
40.544
40.335
40.127
36.643
Current Data Parameters
NAME 20130112-ysy-2-N--jiaji
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130113
Time 0.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
OH
F
N
O
F
(27)
160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


162.464
160.945
160.223
158.379
157.737
154.459
132.512
132.414
132.316
130.451
130.164
129.540
119.971
117.398
116.501
116.267
116.040
112.906
112.665
112.299
112.088
41.379
41.170
40.961
40.752
40.544
40.335
40.127
36.643
Current Data Parameters
NAME 20130112-ysy-2-N--jiaji
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130113
Time 0.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
OH
F
N
O
F
(27)
160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

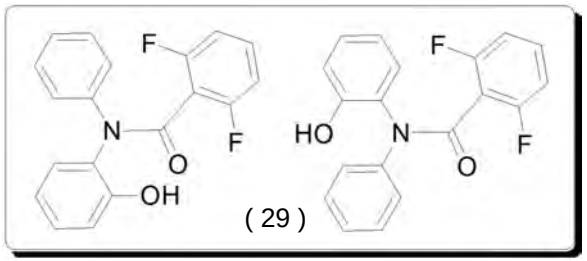

9.8216
9.7852
7.2983
7.2797
7.2606
7.2421
7.2237
7.1969
7.1772
7.1159
7.0975
7.0229
7.0042
6.9860
6.9616
6.9293
6.9062
6.8819
6.8591
6.7592
6.7389
6.6570
6.6383
6.6195
4.0343
4.0195
4.0047
3.9873
3.9709
3.9533
3.9355
3.5971
3.5794
3.5620
3.5454
3.5280
3.5106
3.4686
3.4508
3.3425
2.4997
1.0997
1.0819
1.0641
7.298
7.280
7.261
7.242
7.224
7.197
7.177
7.116
7.098
7.023
7.004
6.986
6.962
6.929
6.906
6.882
6.859
6.759
6.739
6.657
6.638
6.620
OH
F
N
O
F
(28)
Current Data Parameters
NAME 20130110-mll-4-59
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130110
Time 18.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
ppm
1.122
1.976
2.021
1.006
1.000
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4681 ppm/cm, 187.3 Hz/cm
0.93
0.07
1.12
1.98
2.02
1.01
1.00
1.07
0.98
0.15
0.19
2.91


160.816
159.678
159.598
159.025
158.946
157.179
157.115
156.590
156.511
153.612
153.209
131.140
131.040
130.942
129.263
127.094
118.553
116.063
115.577
115.537
115.309
115.083
115.079
111.623
111.439
111.010
110.981
110.795
42.237
39.717
39.509
39.300
12.562
OH
F
N
O
F
(28)
Current Data Parameters
NAME 20130111-mll-4-59-c
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130112
Time 3.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

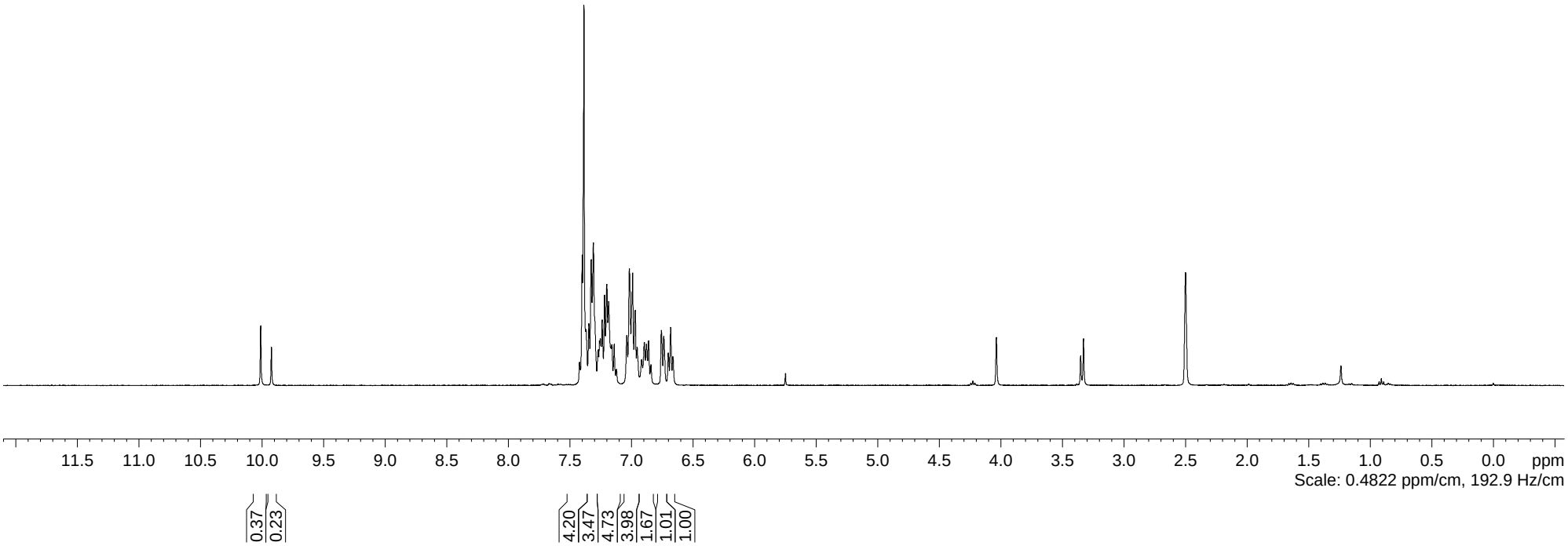


Current Data Parameters
NAME yxl-3-265-H-1220-300k
EXPNO 10
PROCNO 1

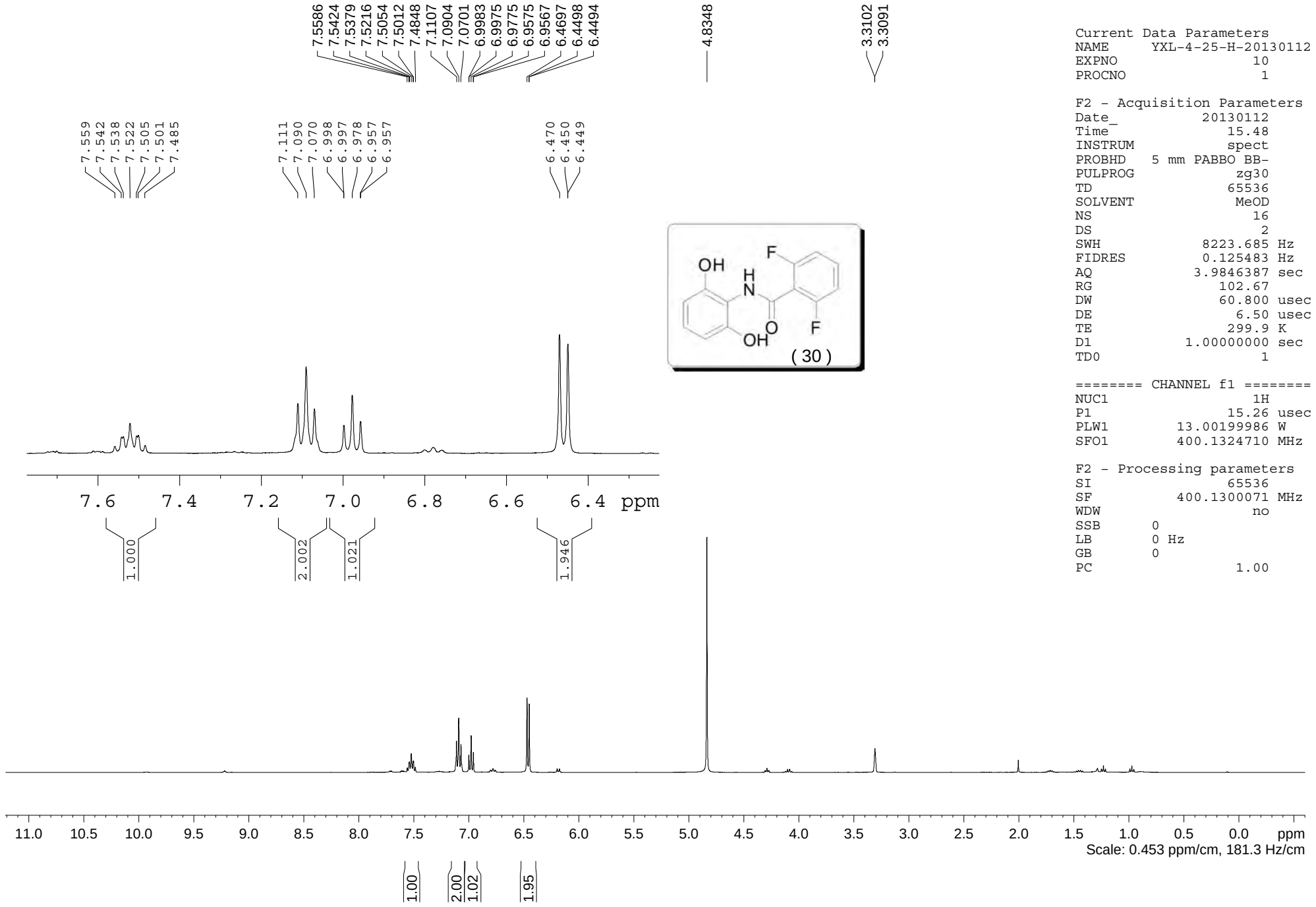
F2 - Acquisition Parameters
Date_ 20121220
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 5
DS 0



(29)

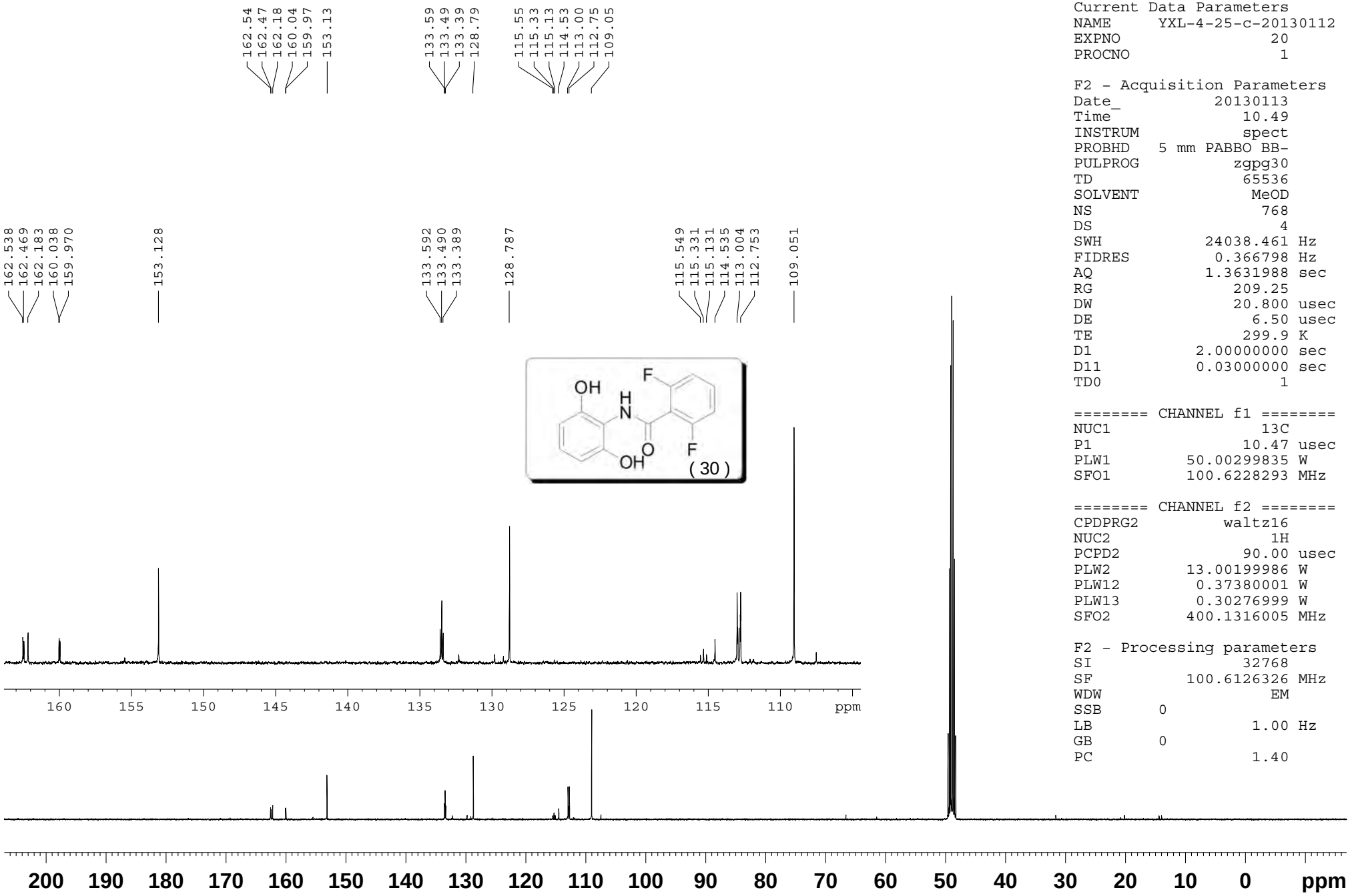


Scale: 0.4822 ppm/cm, 192.9 Hz/cm



Scale: 0.453 ppm/cm, 181.3 Hz/cm

```
Current Data Parameters
NAME      YXL-4-25-H-20130112
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130112
Time                  15.48
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                3.9846387 sec
RG                   102.67
DW                   60.800 usec
DE                    6.50 usec
TE                   299.9 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.26 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF             400.1300071 MHz
WDW                         no
SSB       0
LB        0 Hz
GB        0
PC                      1.00
```



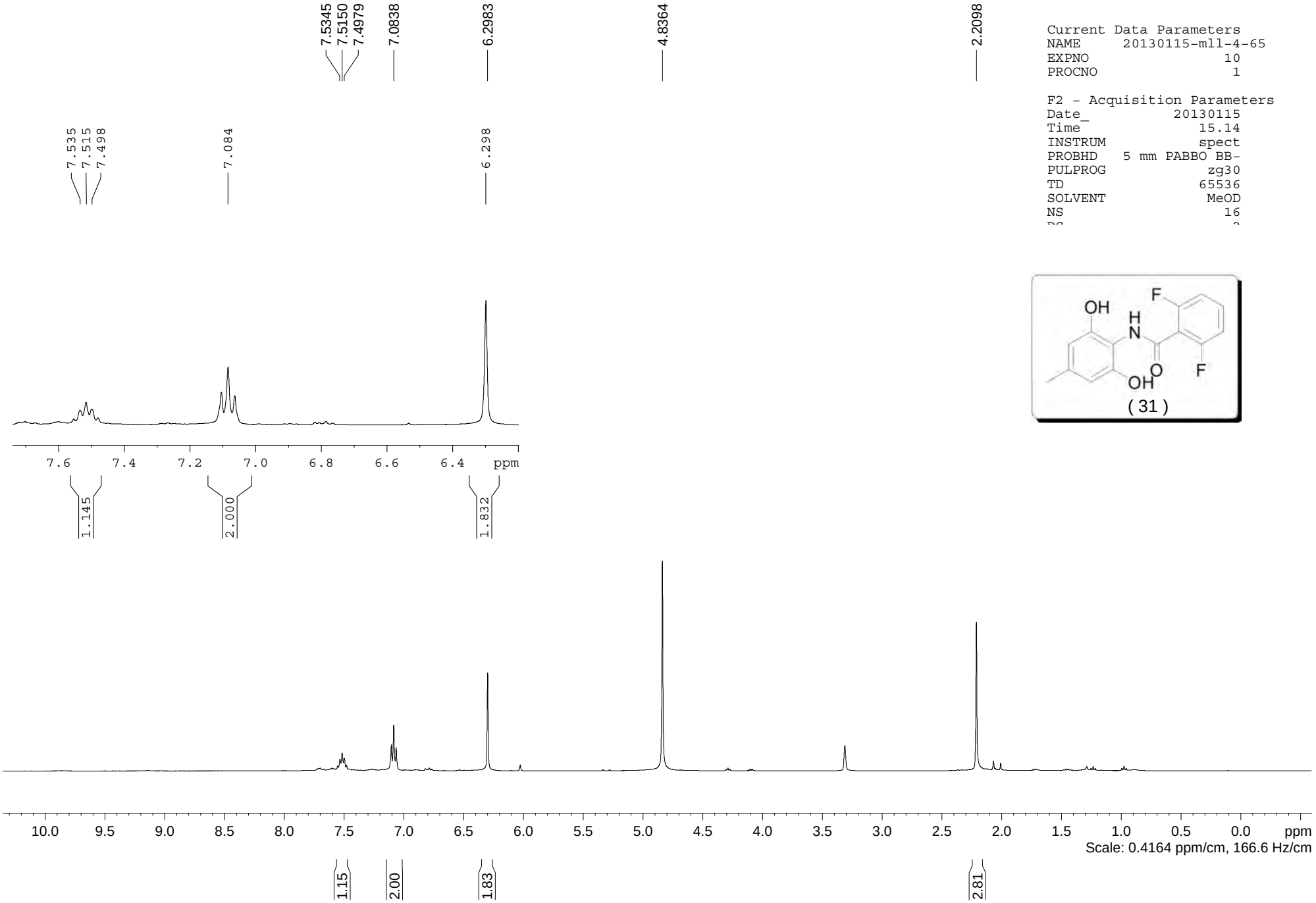
Current Data Parameters
NAME YXL-4-25-c-20130112
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130113
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 768
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 209.25
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.47 usec
PLW1 50.00299835 W
SFO1 100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.00199986 W
PLW12 0.37380001 W
PLW13 0.30276999 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6126326 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

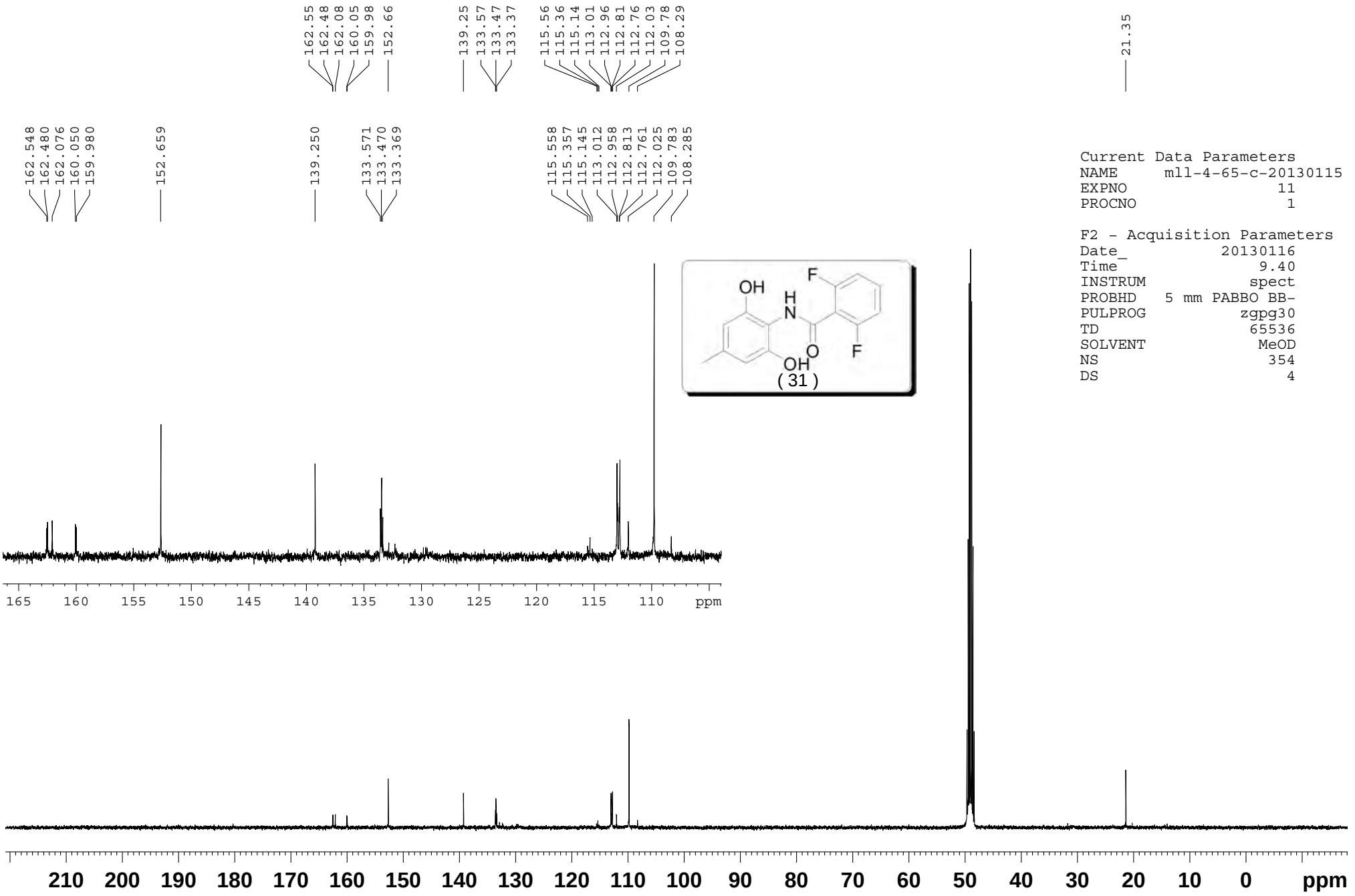


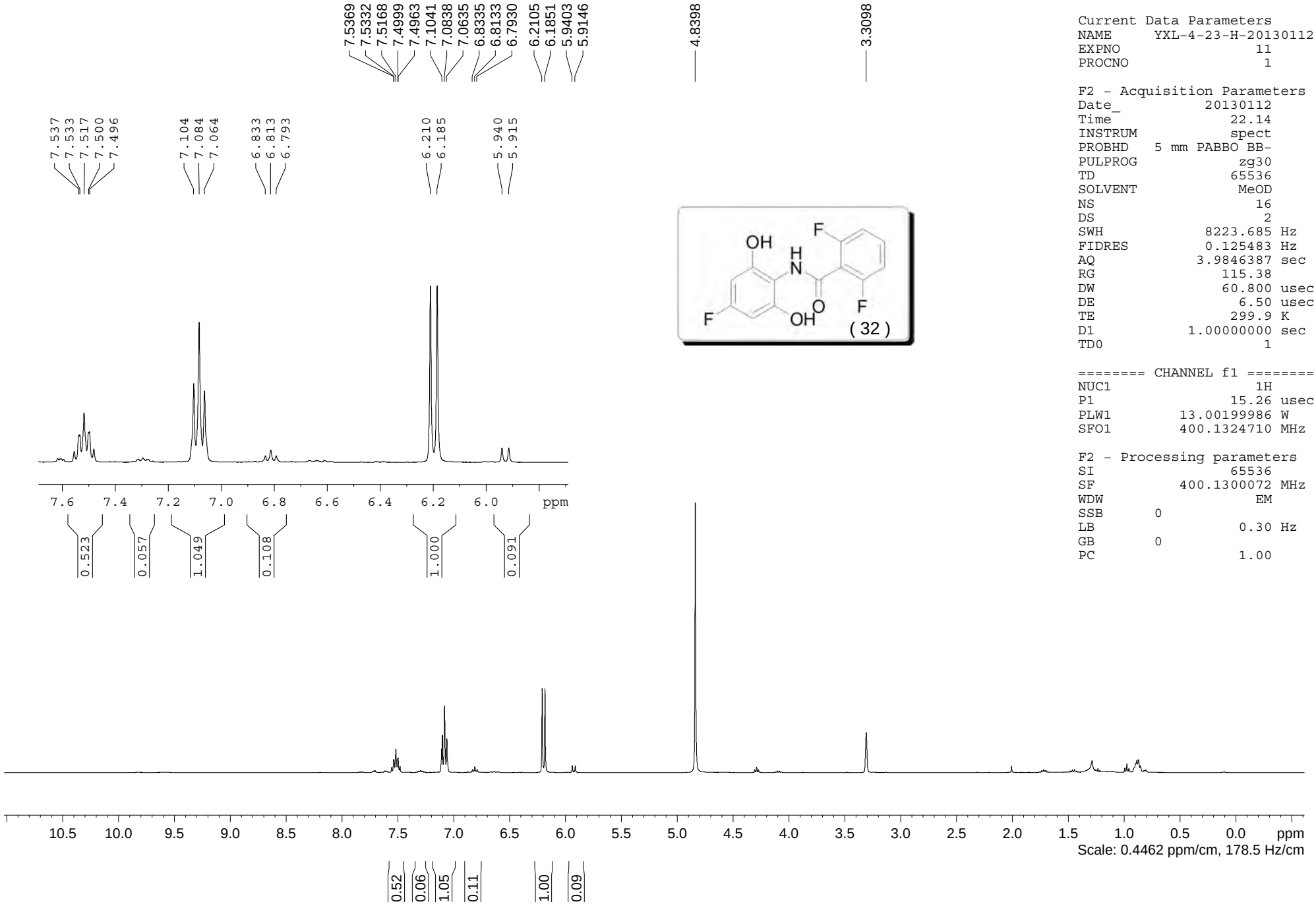
Current Data Parameters
NAME 20130115-mll-4-65
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130115
Time 15.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16

(31)

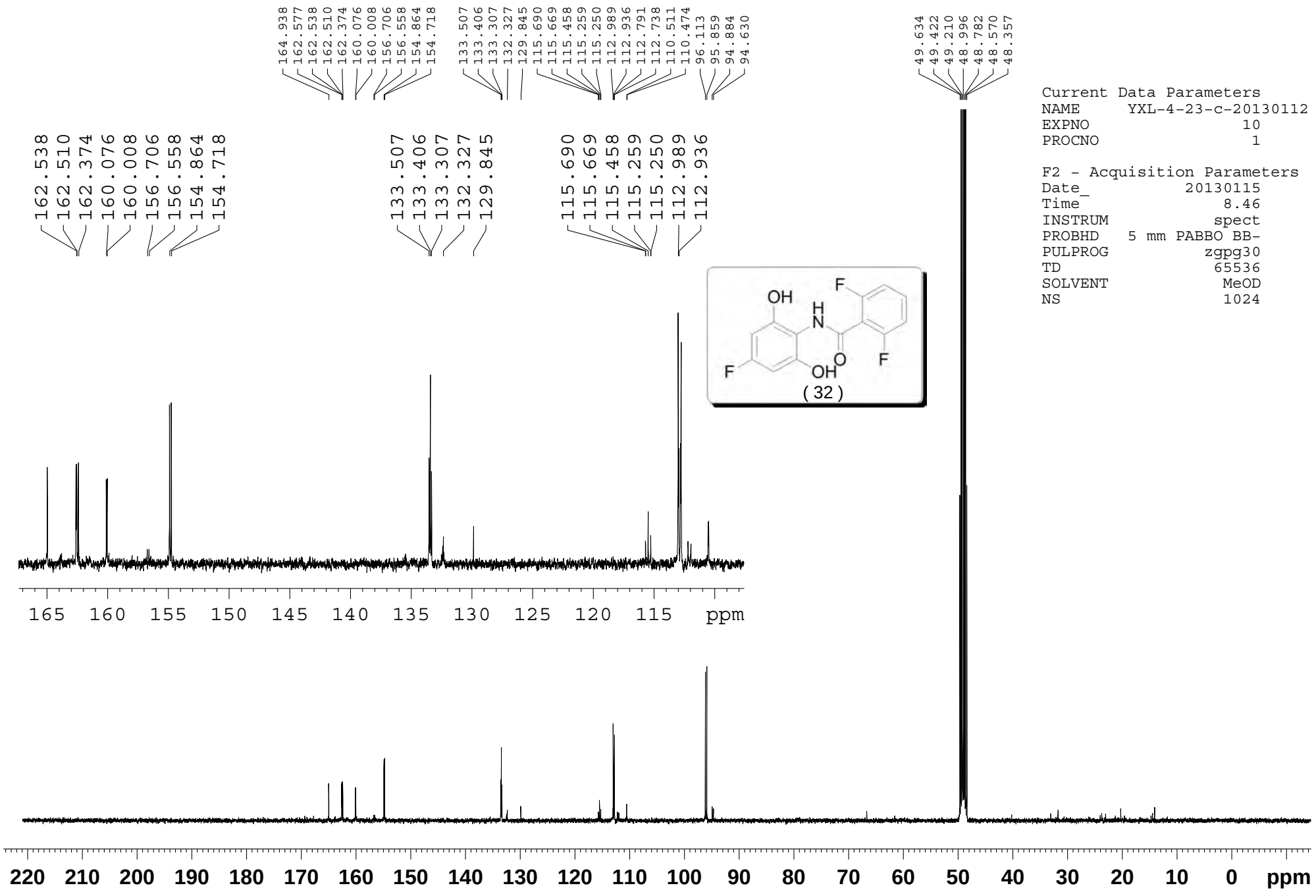
Scale: 0.4164 ppm/cm, 166.6 Hz/cm

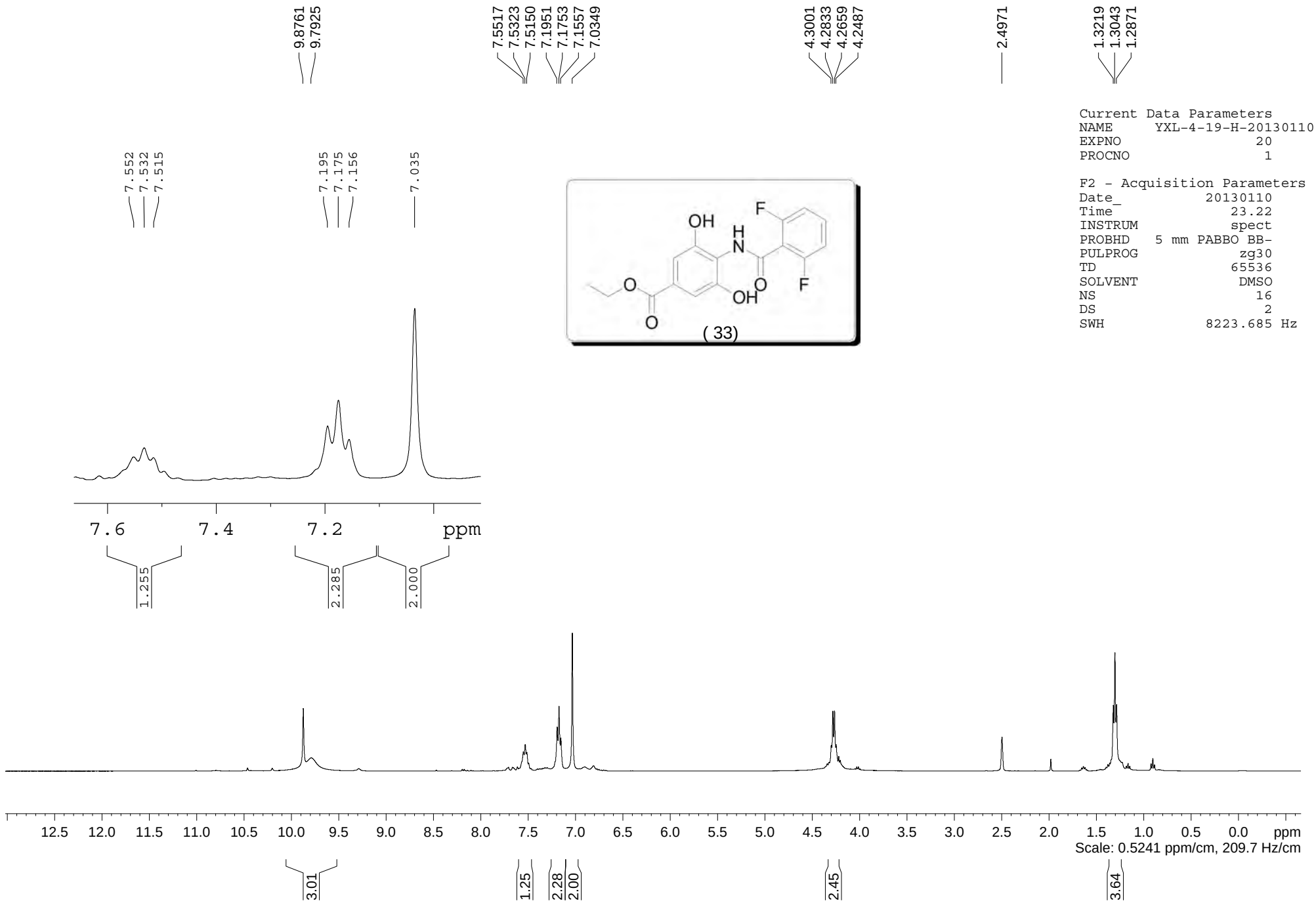

162.55
162.48
162.08
160.05
159.98
152.66
139.25
133.57
133.47
133.37
115.56
115.36
115.14
113.01
112.96
112.81
112.76
112.03
109.78
108.29
21.35
162.548
162.480
162.076
160.050
159.980
152.659
139.250
133.571
133.470
133.369
115.558
115.357
115.145
113.012
112.958
112.813
112.761
112.025
109.783
108.285
OH
H
N
O
OH
F
F
(31)
Current Data Parameters
NAME mll-4-65-c-20130115
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130116
Time 9.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 354
DS 4
165 160 155 150 145 140 135 130 125 120 115 110 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

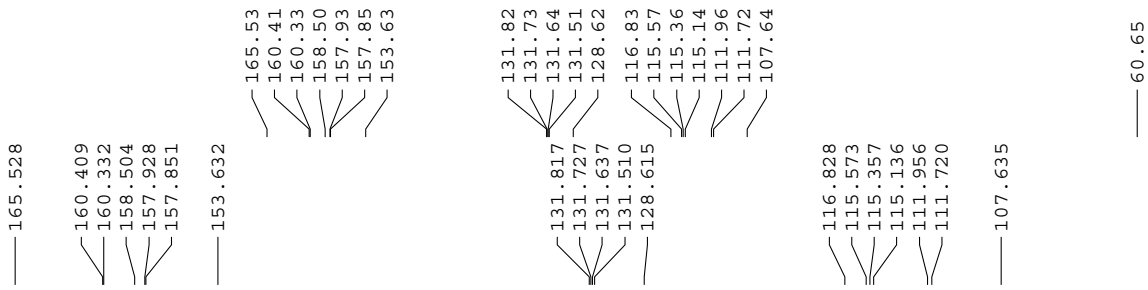



```
Current Data Parameters
NAME      YXL-4-23-H-20130112
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130112
Time                  22.14
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                3.9846387 sec
RG                   115.38
DW                   60.800 usec
DE                    6.50 usec
TE                   299.9 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.26 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF             400.1300072 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

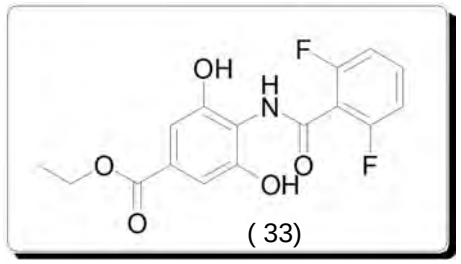
Scale: 0.4462 ppm/cm, 178.5 Hz/cm


164.938
162.577
162.538
162.510
162.374
160.076
160.008
156.706
156.558
154.864
154.718
133.507
133.406
133.307
132.327
129.845
115.690
115.669
115.458
115.259
115.250
112.989
112.936
112.791
112.738
110.511
110.474
96.113
95.859
94.884
94.630
49.634
49.422
49.210
48.996
48.782
48.570
48.357
Current Data Parameters
NAME YXL-4-23-c-20130112
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130115
Time 8.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
OH
H
N
F
O
F
F
OH
(32)
165 160 155 150 145 140 135 130 125 120 115 ppm
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

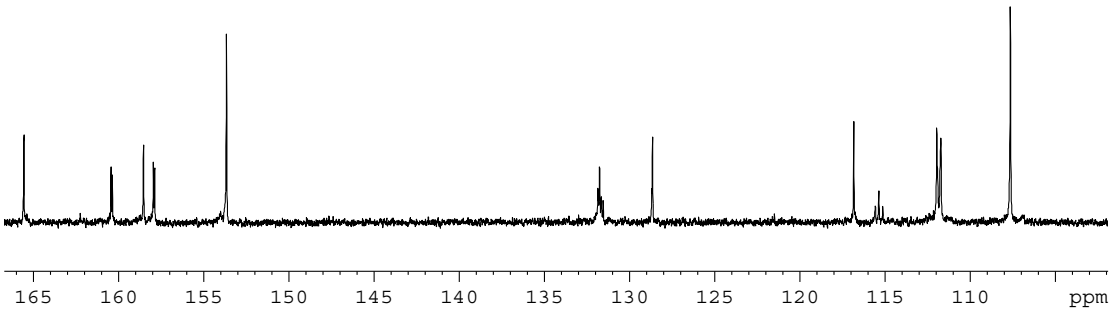

9.8761
9.7925
7.5517
7.5323
7.5150
7.1951
7.1753
7.1557
7.0349
4.3001
4.2833
4.2659
4.2487
2.4971
1.3219
1.3043
1.2871
7.552
7.532
7.515
7.195
7.175
7.156
7.035
OH
F
H
N
O
O
OH
F
O
(33)
Current Data Parameters
NAME YXL-4-19-H-20130110
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130110
Time 23.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
7.6
7.4
7.2
ppm
1.255
2.285
2.000
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.5241 ppm/cm, 209.7 Hz/cm
3.01
1.25
2.28
2.00
2.45
3.64

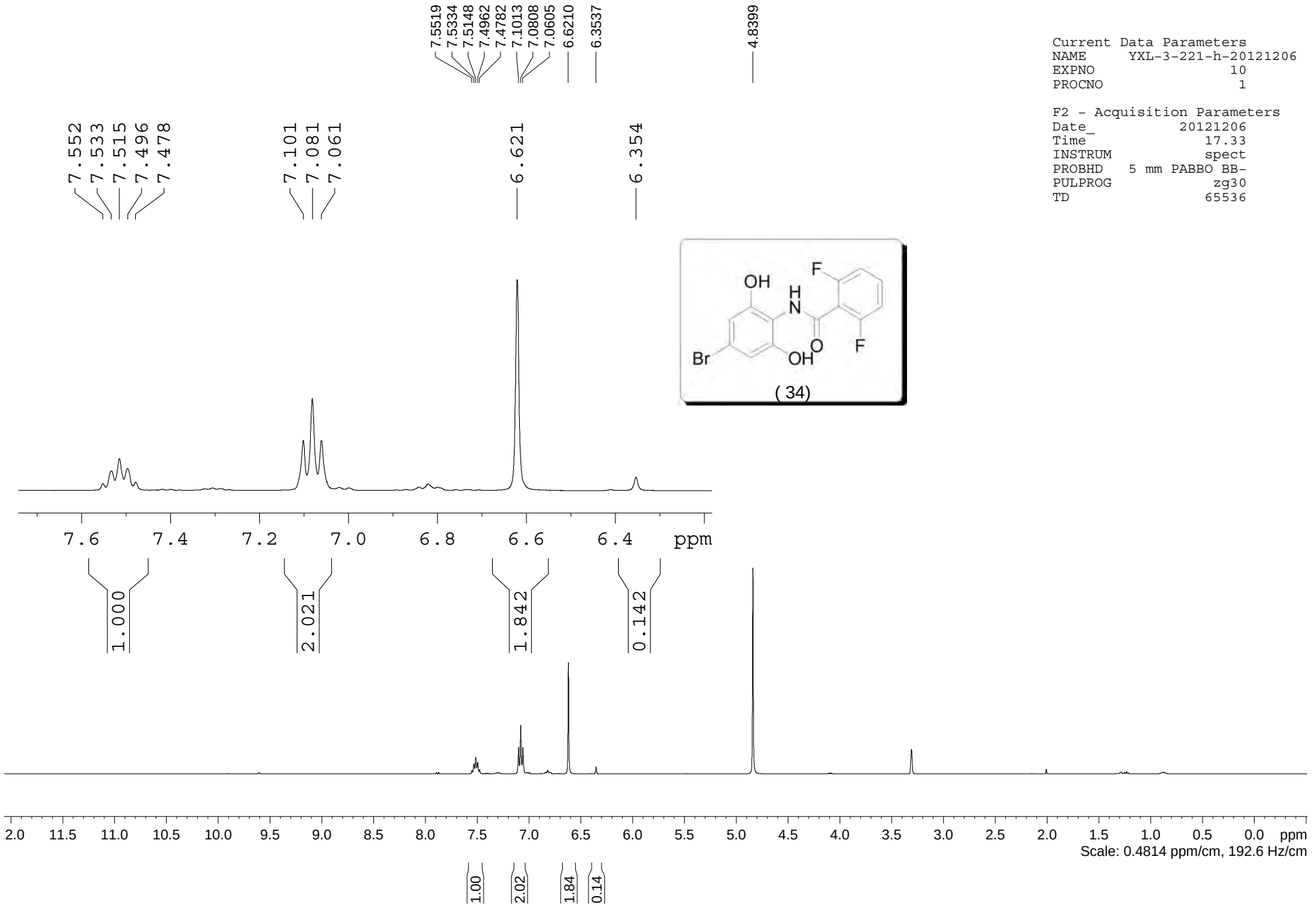


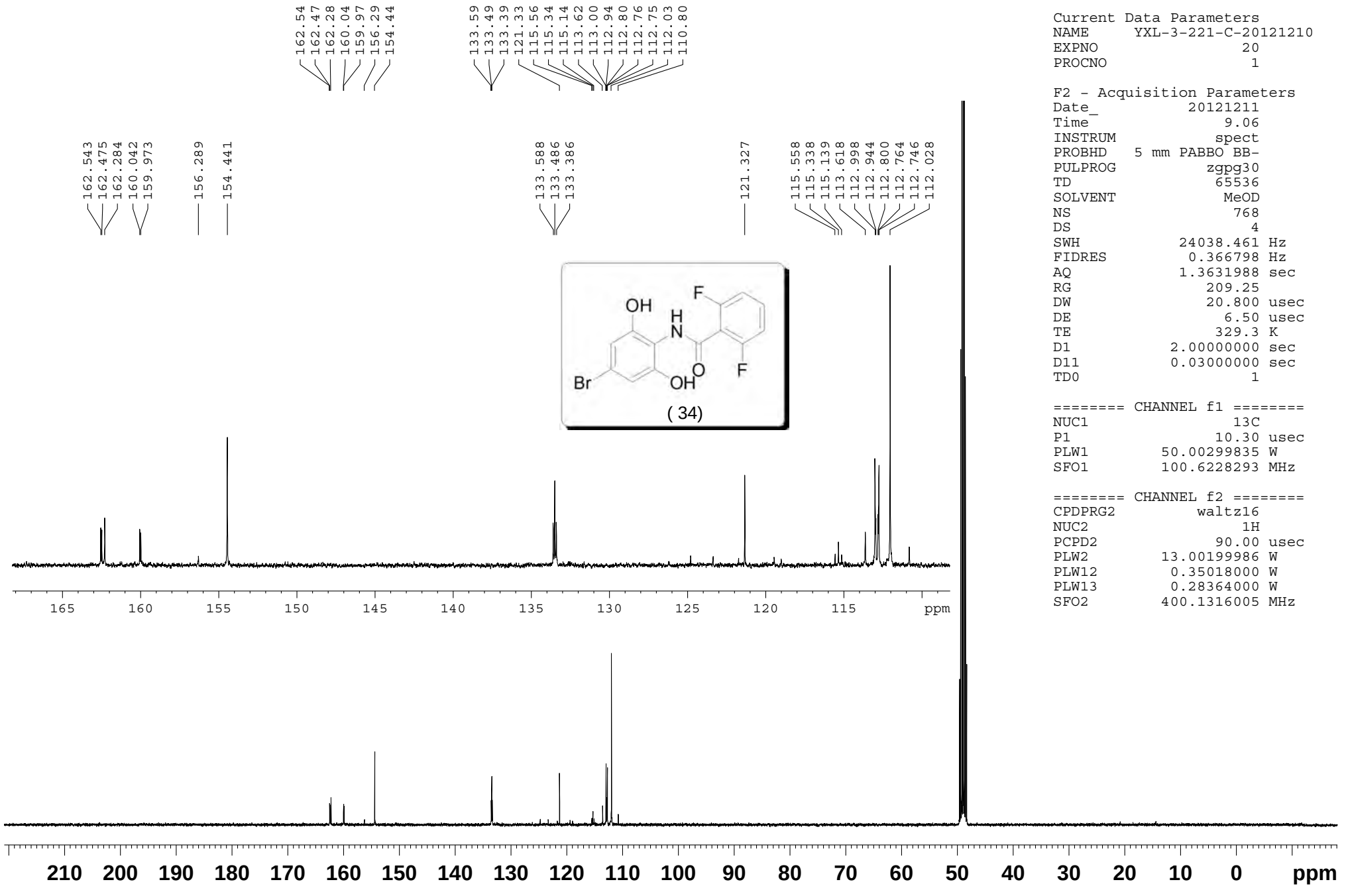

```
Current Data Parameters
NAME          YXL-4-19-C-20130111
EXPNO                          10
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20130112
Time                         9.46
INSTRUM                     spect
PROBHD       5 mm PABBO BB-
PULPROG                    zgpg30
TD                          65536
SOLVENT                       DMSO
NS                             768
```



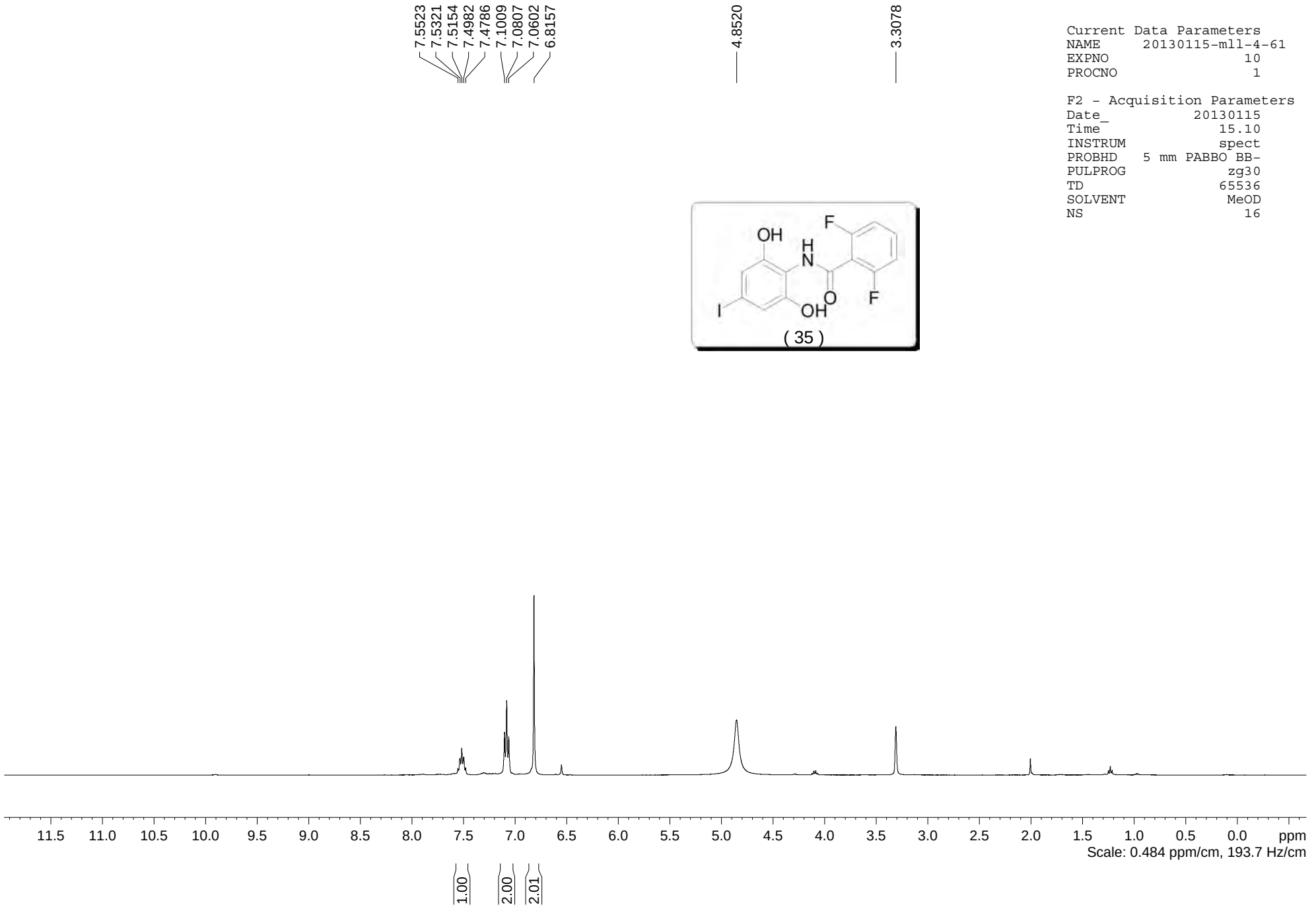
(33)

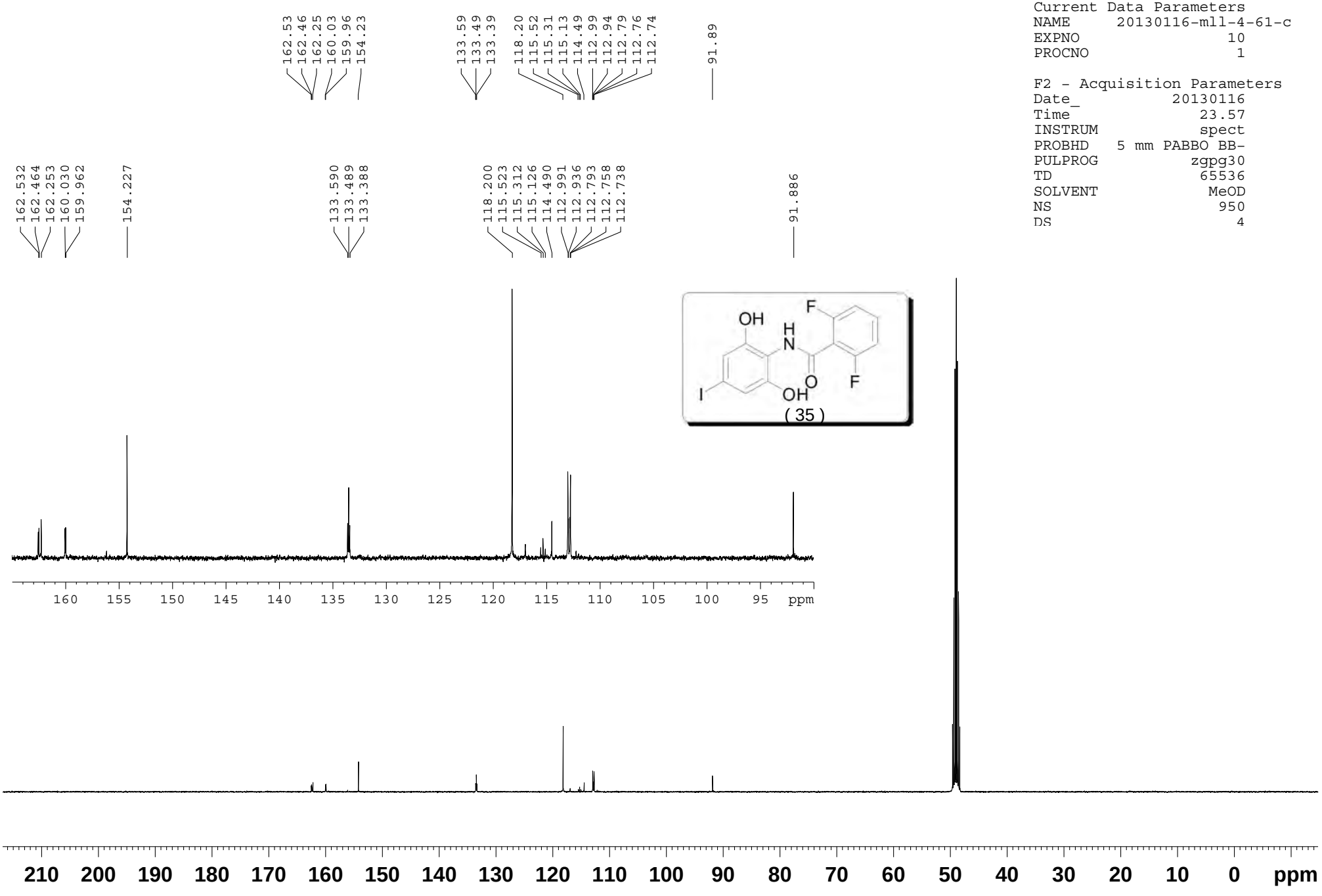


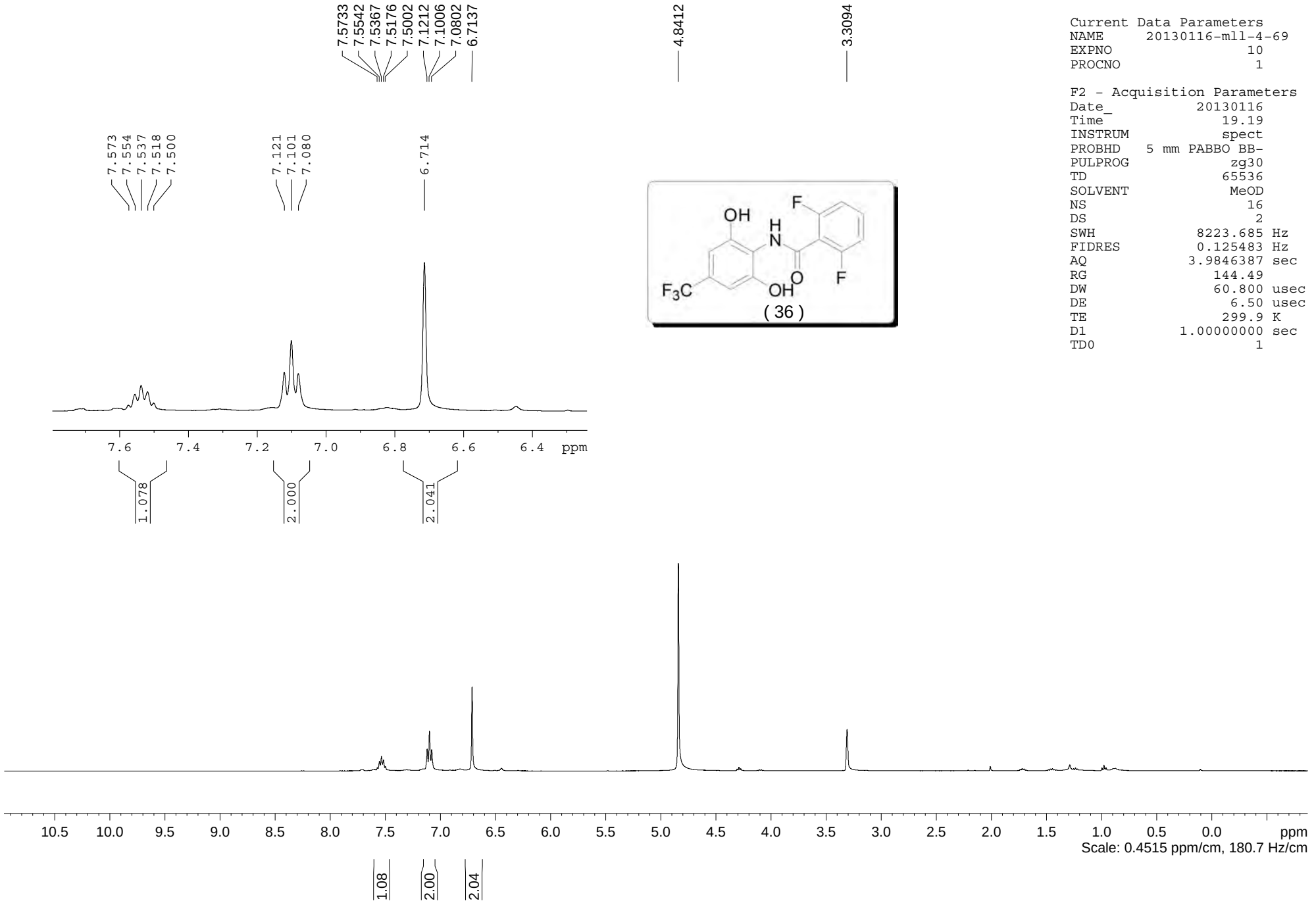

7.5519
7.5334
7.5148
7.4962
7.4782
7.1013
7.0808
7.0605
6.6210
6.3537
4.8399
7.552
7.533
7.515
7.496
7.478
7.101
7.081
7.061
6.621
6.354
Current Data Parameters
NAME YXL-3-221-h-20121206
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121206
Time 17.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
OH
H
N
F
O
F
Br
OH
(34)
7.6
7.4
7.2
7.0
6.8
6.6
6.4
ppm
1.000
2.021
1.842
0.142
2.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4814 ppm/cm, 192.6 Hz/cm
1.00
2.02
1.84
0.14




```
Current Data Parameters
NAME      YXL-3-221-C-20121210
EXPNO                      20
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121211
Time                   9.06
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                MeOD
NS                       768
DS                         4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                   209.25
DW                  20.800 usec
DE                   6.50 usec
TE                  329.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.30 usec
PLW1          50.00299835 W
SFO1         100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                90.00 usec
PLW2          13.00199986 W
PLW12          0.35018000 W
PLW13          0.28364000 W
SFO2         400.1316005 MHz
```


7.5523
7.5321
7.5154
7.4982
7.4786
7.1009
7.0807
7.0602
6.8157
4.8520
3.3078
Current Data Parameters
NAME 20130115-mll-4-61
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130115
Time 15.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
OH
F
H
N
O
F
I
OH
(35)
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.484 ppm/cm, 193.7 Hz/cm
1.00
2.00
2.01


162.53
162.46
162.25
160.03
159.96
154.23
133.59
133.49
133.39
118.20
115.52
115.31
115.13
114.49
112.99
112.94
112.79
112.76
112.74
91.89
162.532
162.464
162.253
160.030
159.962
154.227
133.590
133.489
133.388
118.200
115.523
115.312
115.126
114.490
112.991
112.936
112.793
112.758
112.738
91.886
Current Data Parameters
NAME 20130116-mll-4-61-c
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130116
Time 23.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 950
DS 4
OH
H
N
O
F
F
I
OH
(35)
160 155 150 145 140 135 130 125 120 115 110 105 100 95 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

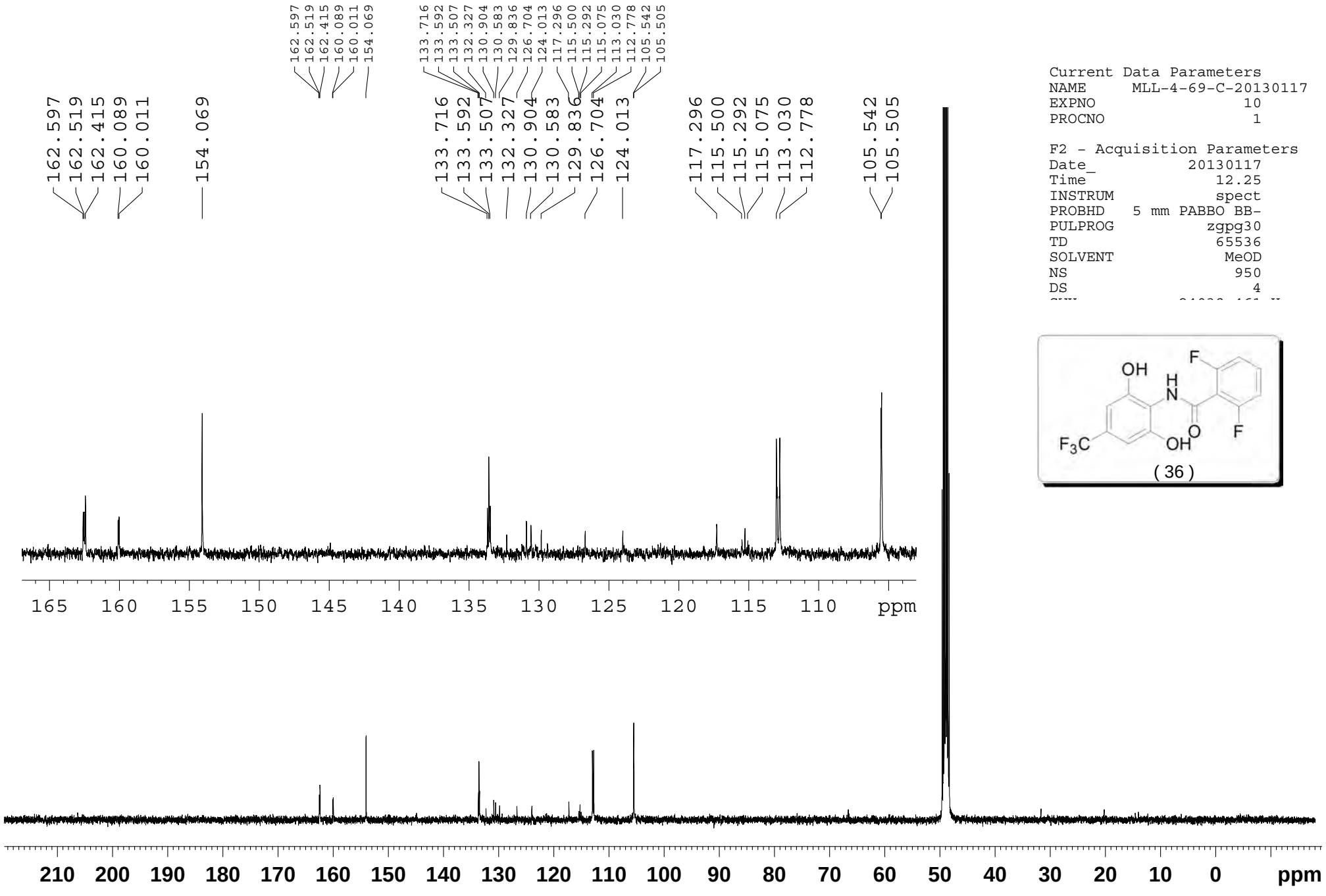


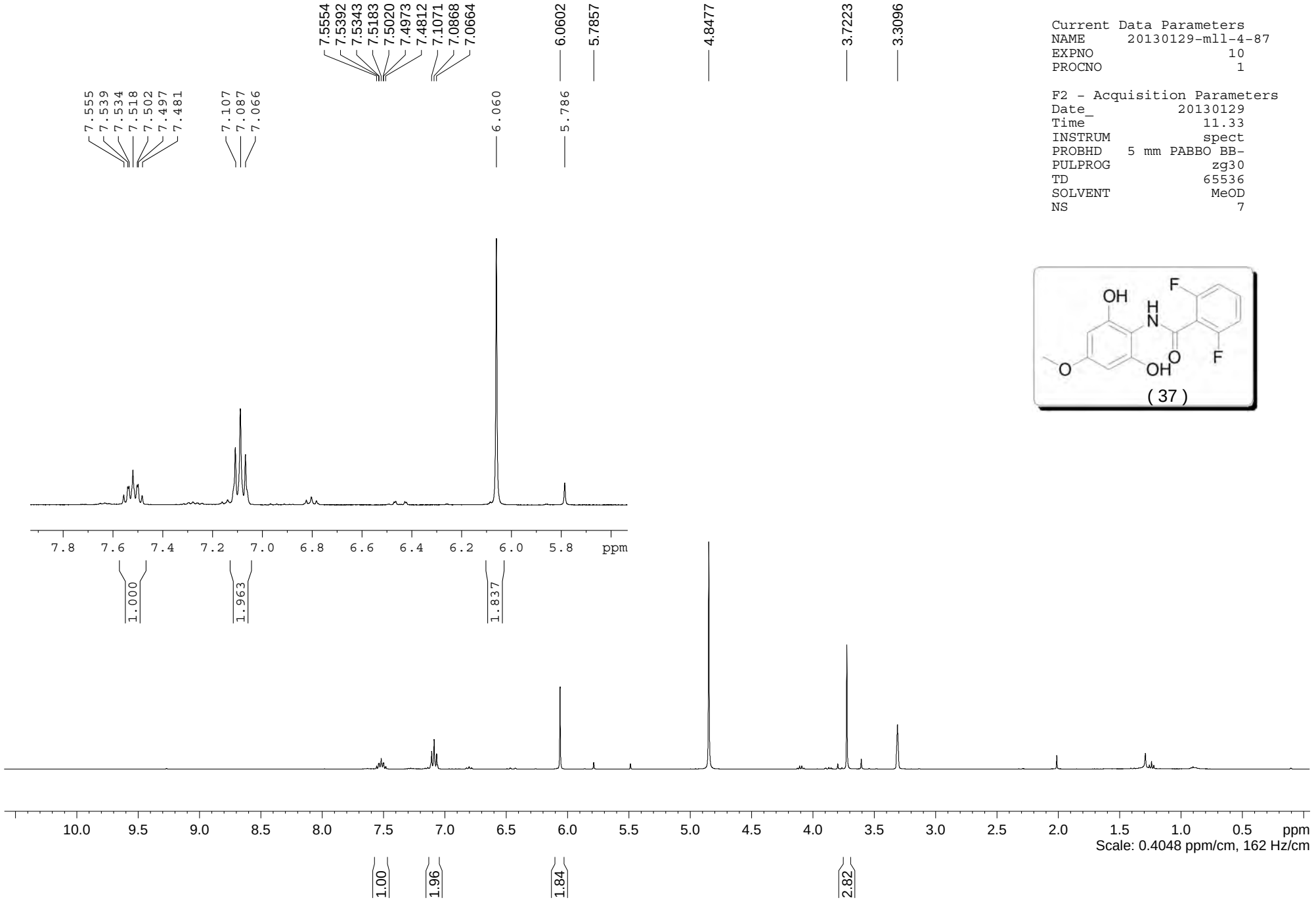
Current Data Parameters

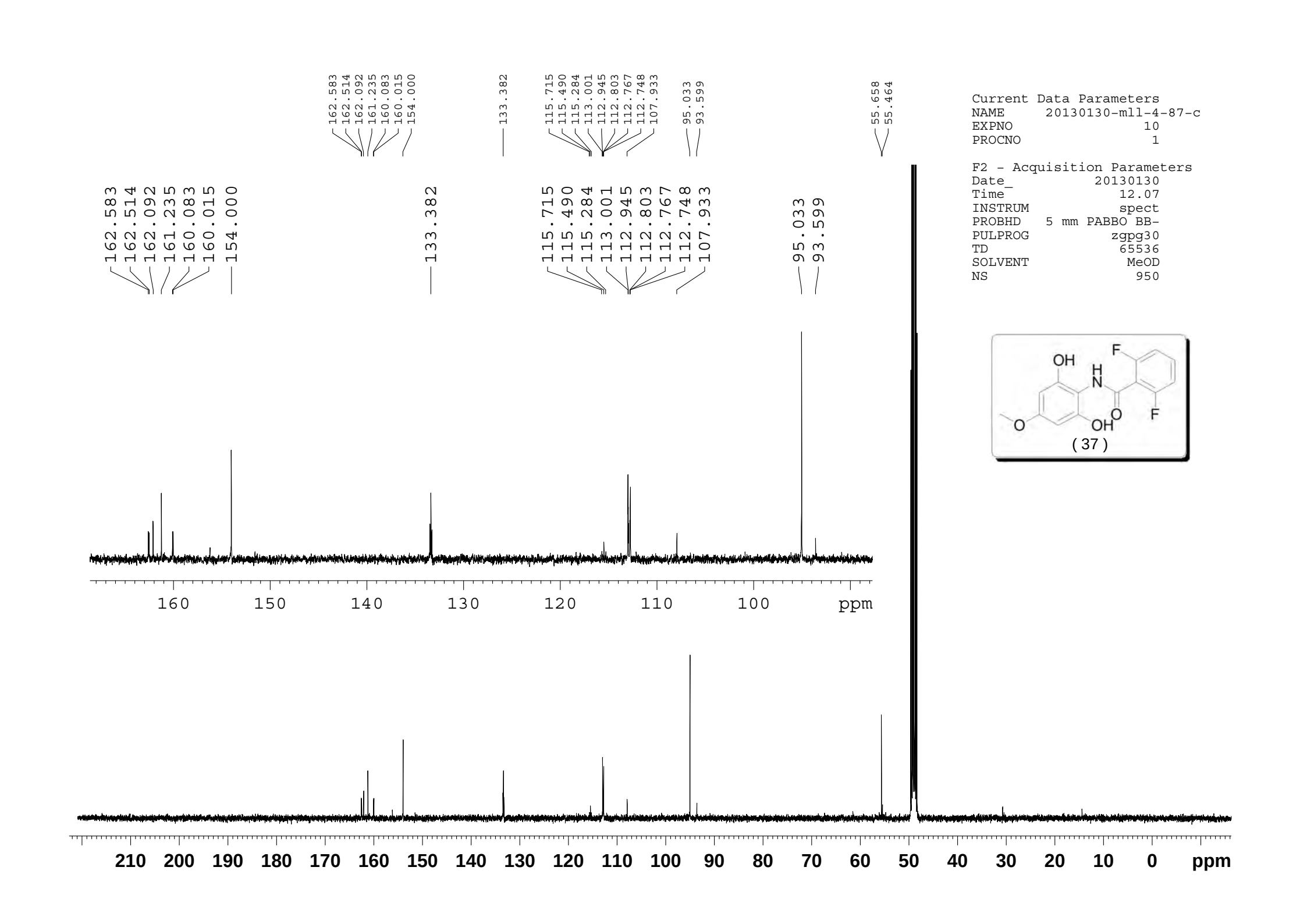
NAME	20130116-mll-4-69
EXPNO	10
PROCNO	1

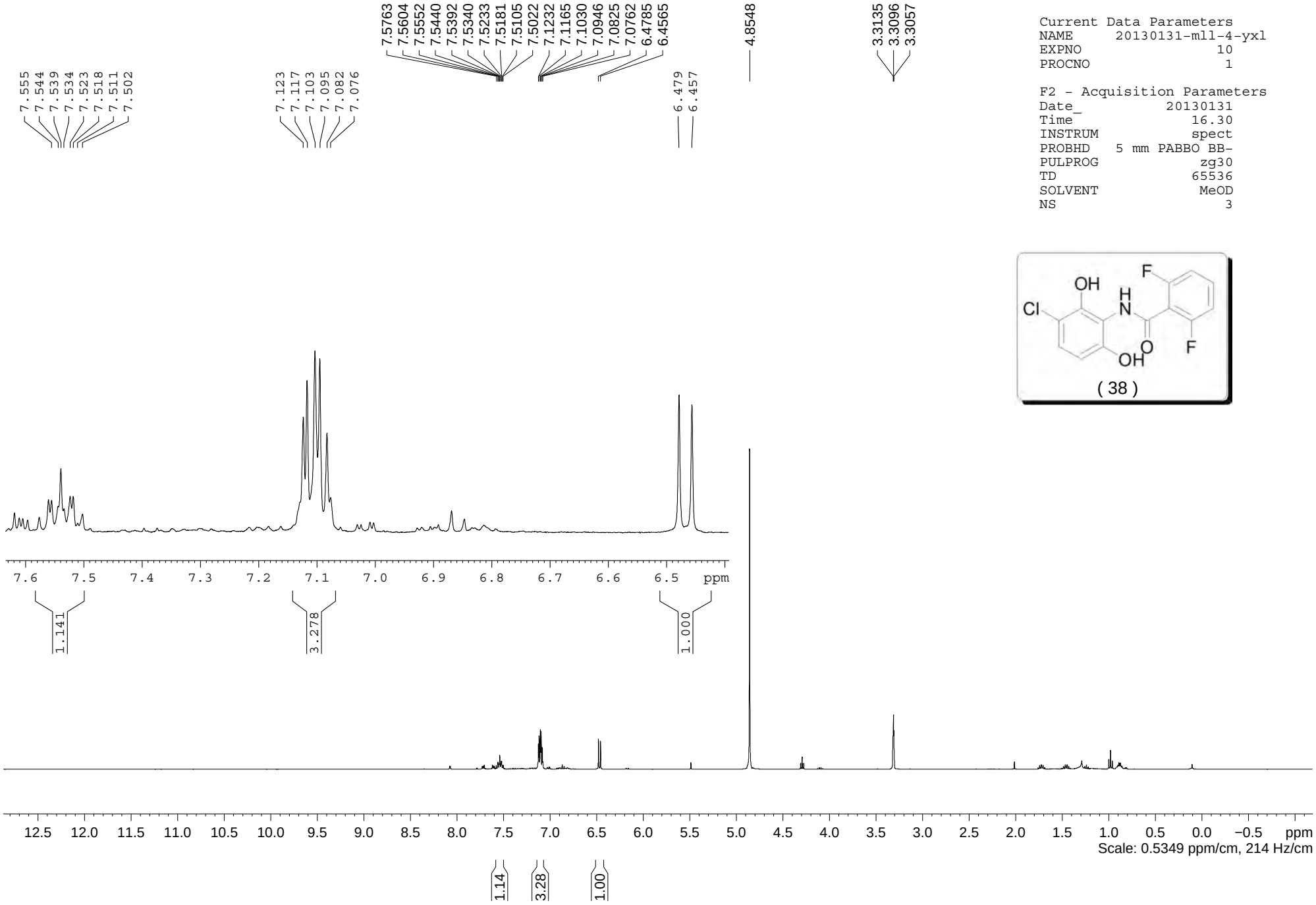
F2 - Acquisition Parameters

Date_	20130116
Time	19.19
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	144.49
DW	60.800 usec
DE	6.50 usec
TE	299.9 K
D1	1.00000000 sec
TD0	1


162.597
162.519
162.415
160.089
160.011
154.069
133.716
133.592
133.507
132.327
130.904
130.583
129.836
126.704
124.013
117.296
115.500
115.292
115.075
113.030
112.778
105.542
105.505
Current Data Parameters
NAME MLL-4-69-C-20130117
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130117
Time 12.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 950
DS 4
OH
F
H
N
O
F
F3C
OH
(36)
165 160 155 150 145 140 135 130 125 120 115 110 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.5554
7.5392
7.5343
7.5183
7.5020
7.4973
7.4812
7.1071
7.0868
7.0664
6.0602
5.7857
4.8477
3.7223
3.3096
7.555
7.539
7.534
7.518
7.502
7.497
7.481
7.107
7.087
7.066
6.060
5.786
Current Data Parameters
NAME 20130129-mll-4-87
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130129
Time 11.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 7
OH
F
H
N
O
F
O
OH
(37)
7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 ppm
1.000
1.963
1.837
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
Scale: 0.4048 ppm/cm, 162 Hz/cm
1.00
1.96
1.84
2.82

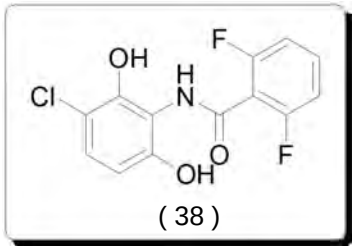

162.583
162.514
162.092
161.235
160.083
160.015
154.000
133.382
115.715
115.490
115.284
113.001
112.945
112.803
112.767
112.748
107.933
95.033
93.599
55.658
55.464
Current Data Parameters
NAME 20130130-mll-4-87-c
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130130
Time 12.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 950
OH
F
H
N
O
F
O
OH
(37)
160
150
140
130
120
110
100
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.555
7.544
7.539
7.534
7.523
7.518
7.511
7.502
7.123
7.117
7.103
7.095
7.082
7.076
7.5763
7.5604
7.5552
7.5440
7.5392
7.5340
7.5233
7.5181
7.5105
7.5022
7.1232
7.1165
7.1030
7.0946
7.0825
7.0762
6.4785
6.4565
6.479
6.457
4.8548
3.3135
3.3096
3.3057
Current Data Parameters
NAME 20130131-mll-4-yxl
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time 16.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 3
OH
H
N
Cl
F
O
F
OH
(38)
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
ppm
1.141
3.278
1.000
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
Scale: 0.5349 ppm/cm, 214 Hz/cm
1.14
3.28
1.00

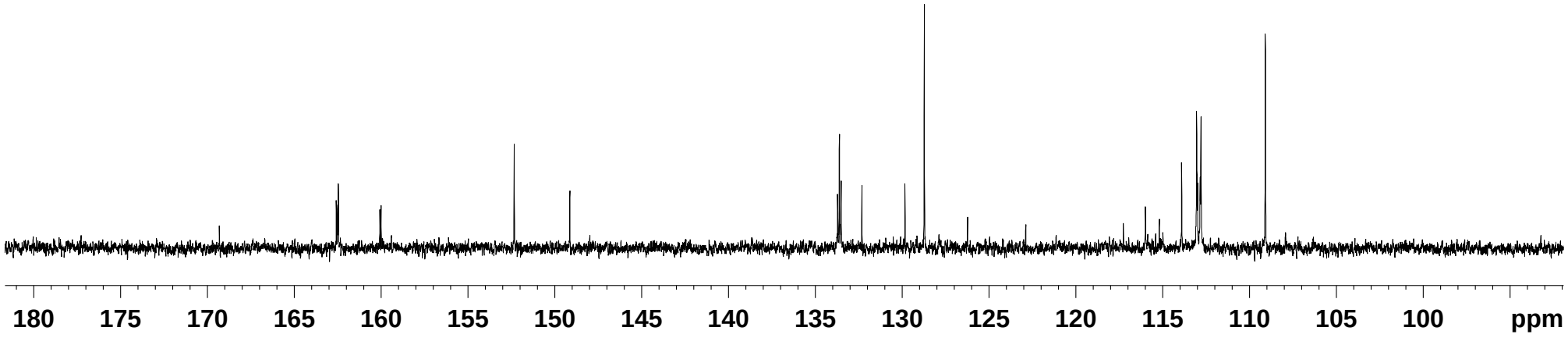
169.305
162.589
162.519
162.473
160.084
160.016
152.346
149.147
133.723
133.623
133.522
132.323
129.845
128.728
126.237
122.890
117.267
115.994
115.198
113.906
113.044
112.989
112.845
112.791
109.091

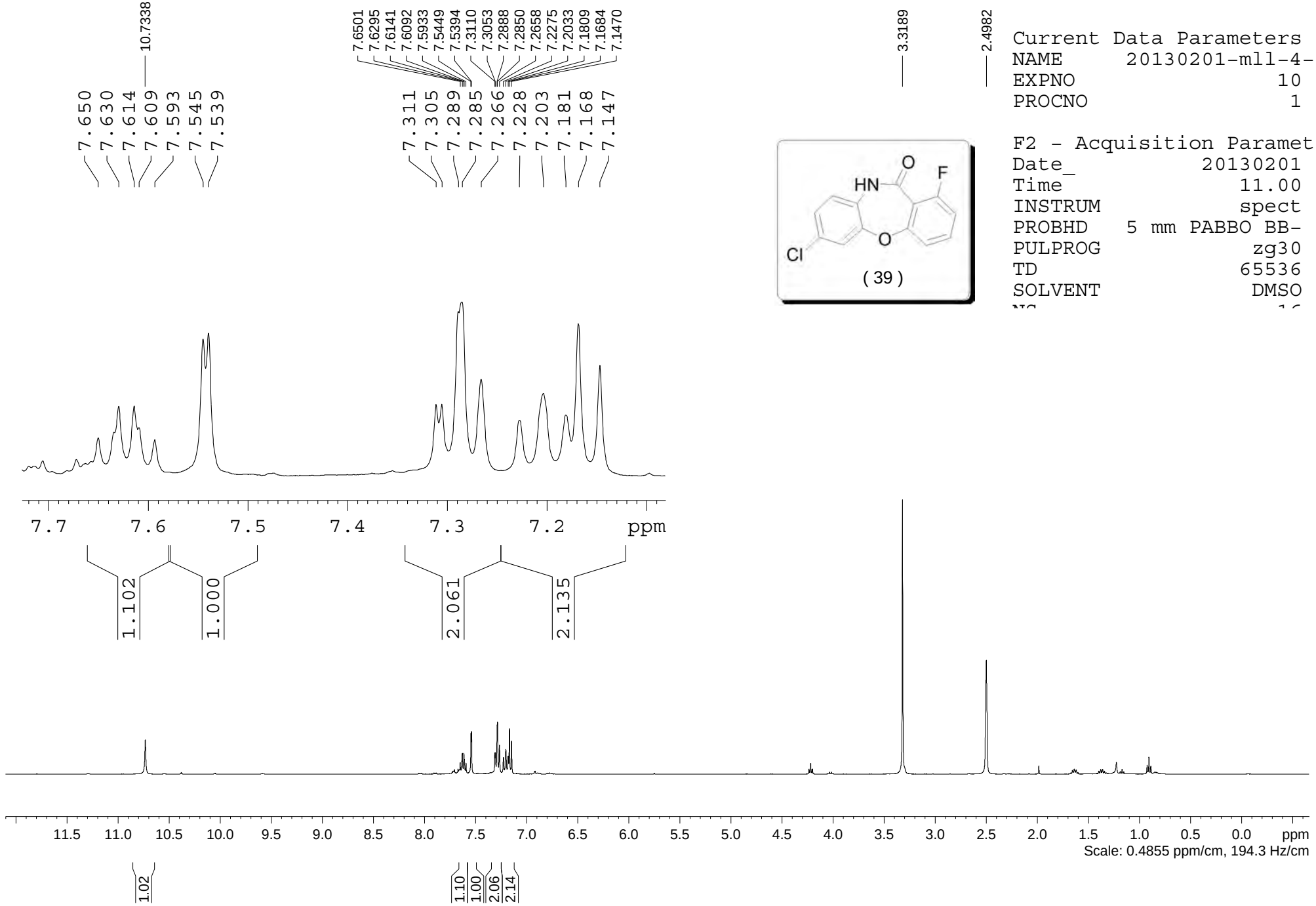
Current Data Parameters
NAME mll-90-yxl-c
EXPNO 10
PROCNO 1

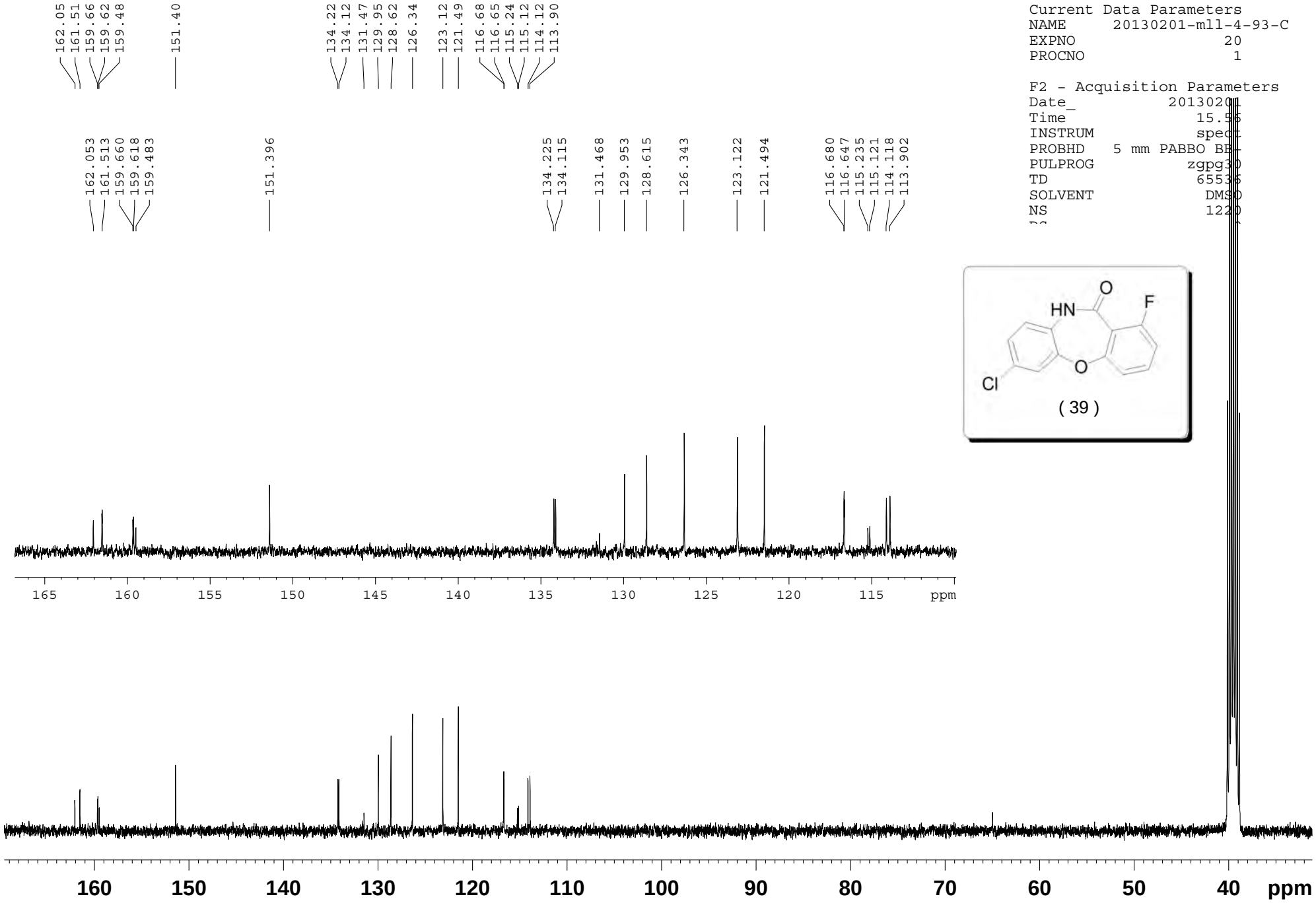
F2 - Acquisition Parameters
Date_ 20130201
Time 9.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024

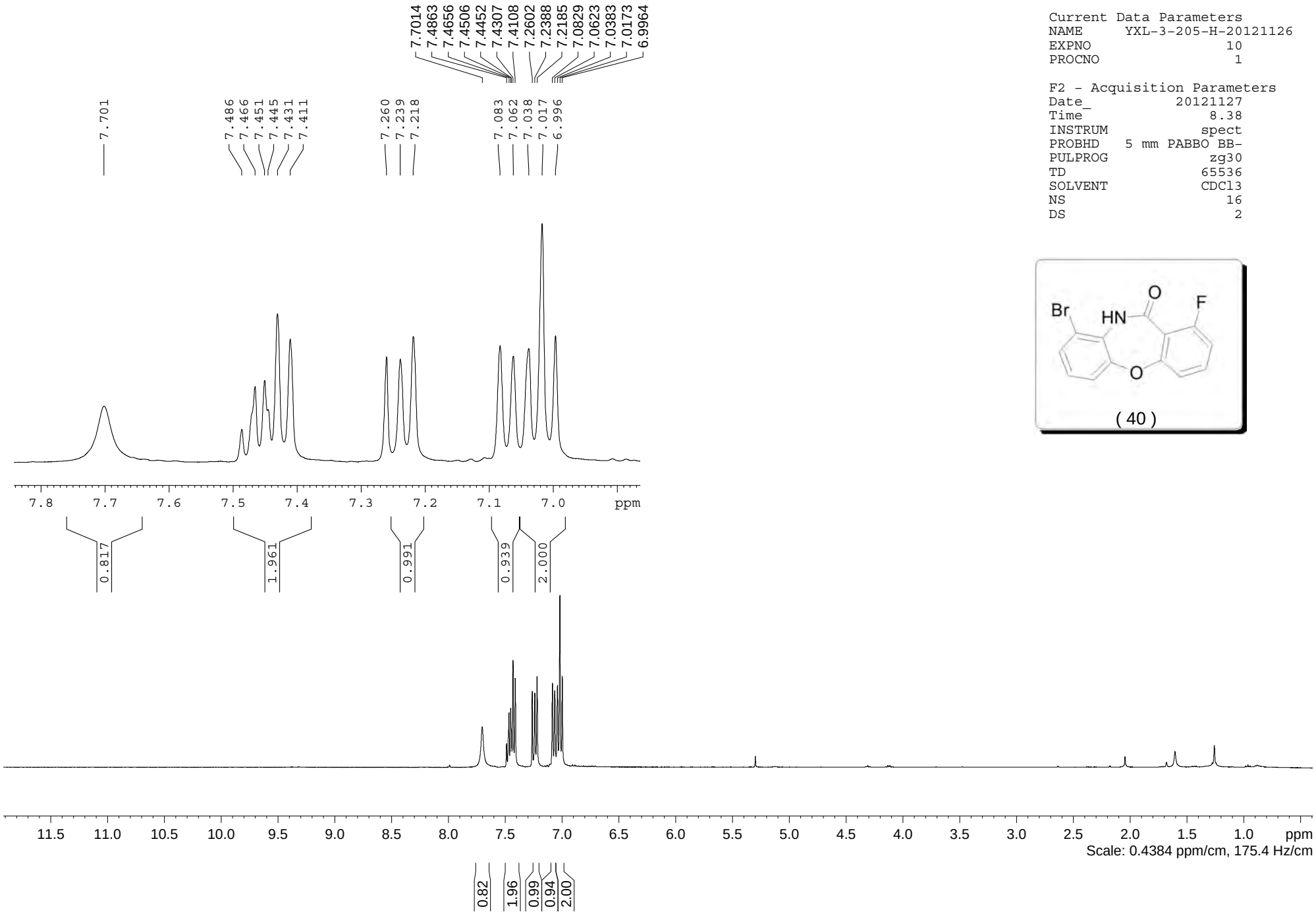


(38)



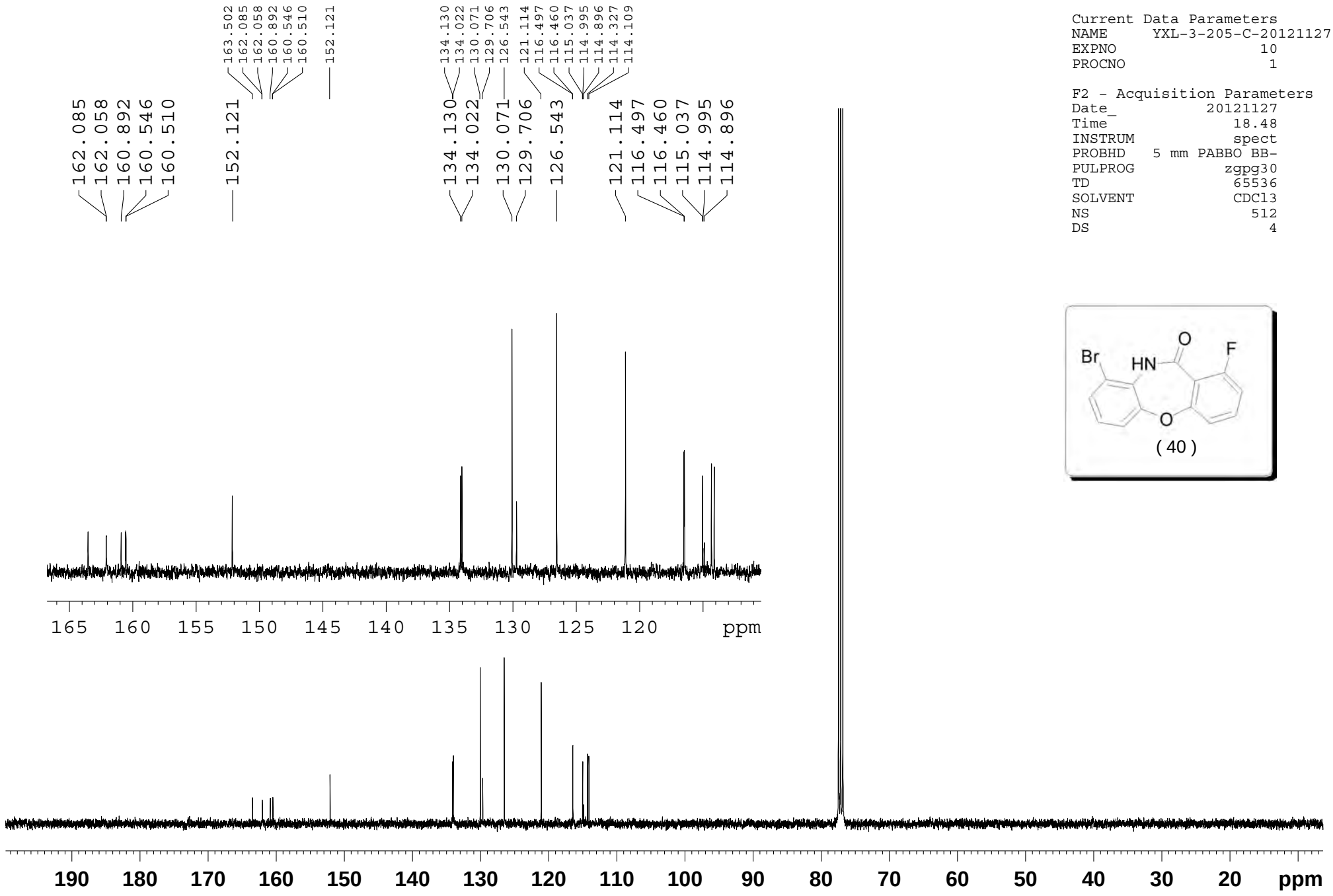

10.7338
7.650
7.630
7.614
7.609
7.593
7.545
7.539
7.6501
7.6295
7.6141
7.6092
7.5933
7.5449
7.5394
7.3110
7.3053
7.2888
7.2850
7.2658
7.2275
7.2033
7.1809
7.1684
7.1470
7.311
7.305
7.289
7.285
7.266
7.228
7.203
7.181
7.168
7.147
3.3189
2.4982
Current Data Parameters
NAME 20130201-mll-4-
EXPNO 10
PROCNO 1
F2 - Acquisition Paramet
Date_ 20130201
Time 11.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
HN
O
F
Cl
O
(39)
7.7
7.6
7.5
7.4
7.3
7.2
ppm
1.102
1.000
2.061
2.135
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4855 ppm/cm, 194.3 Hz/cm
1.02
1.10
1.00
2.06
2.14


162.05
161.51
159.66
159.62
159.48
151.40
134.22
134.12
131.47
129.95
128.62
126.34
123.12
121.49
116.68
116.65
115.24
115.12
114.12
113.90
162.053
161.513
159.660
159.618
159.483
151.396
134.225
134.115
131.468
129.953
128.615
126.343
123.122
121.494
116.680
116.647
115.235
115.121
114.118
113.902
Current Data Parameters
NAME 20130201-mll-4-93-C
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 2013020
Time 15.5
INSTRUM spec
PROBHD 5 mm PABBO BB
PULPROG zgpg3
TD 6553
SOLVENT DMSO
NS 122
HN
O
F
Cl
O
(39)
165
160
155
150
145
140
135
130
125
120
115
ppm
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm




```
Current Data Parameters
NAME      YXL-3-205-H-20121126
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_              20121127
Time                   8.38
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
```

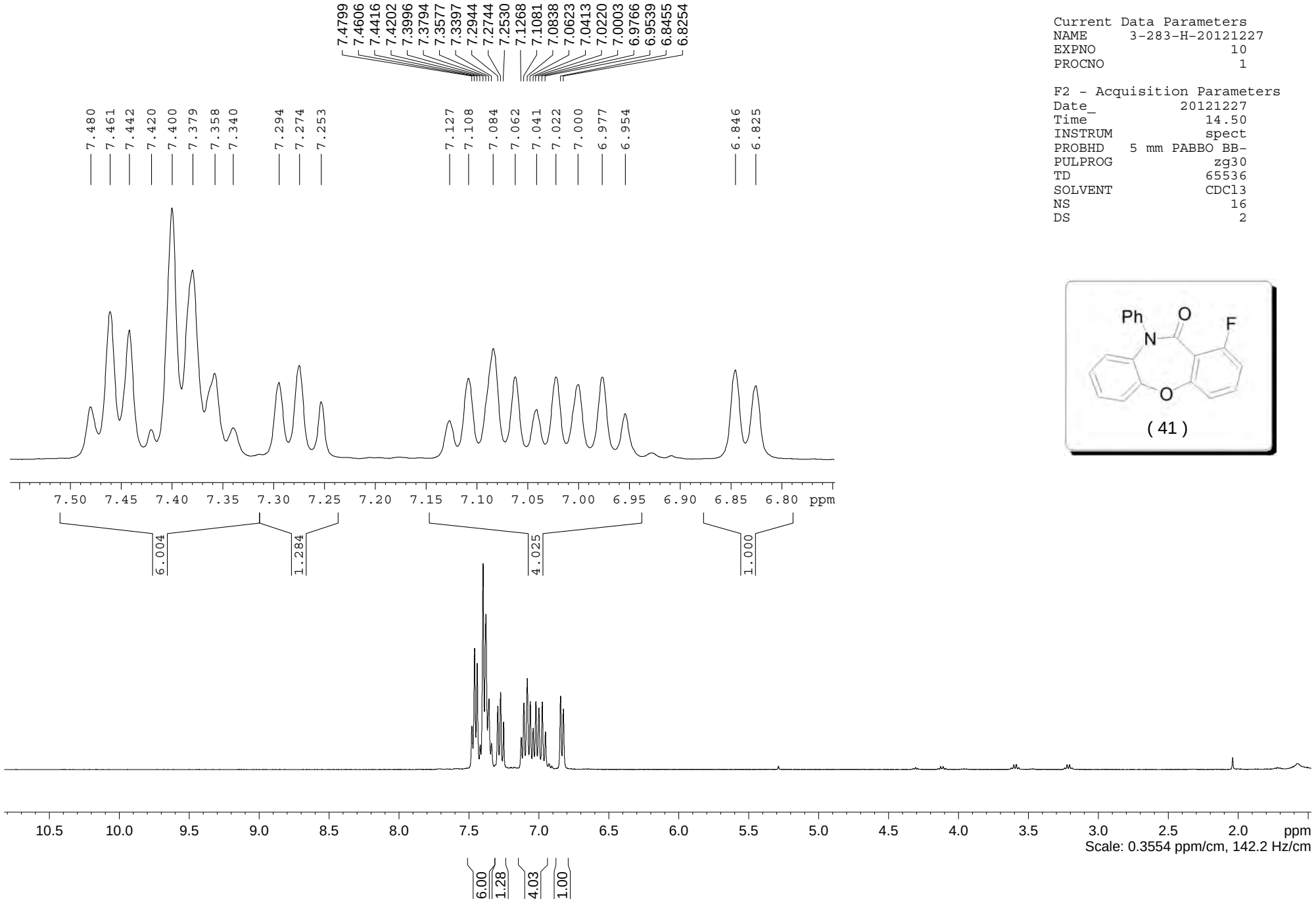

(40)

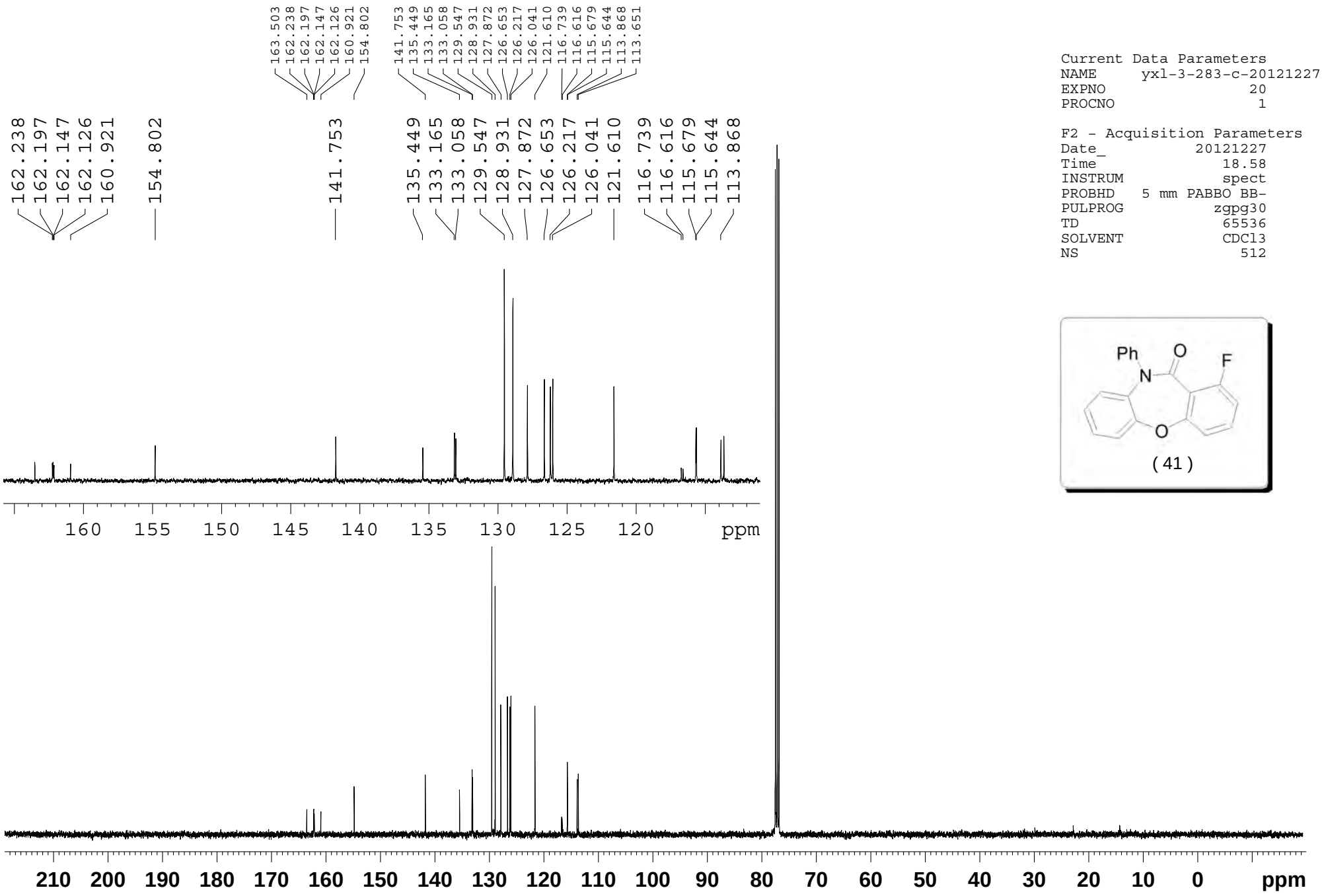


Current Data Parameters
NAME YXL-3-205-C-20121127
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121127
Time 18.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4

(40)

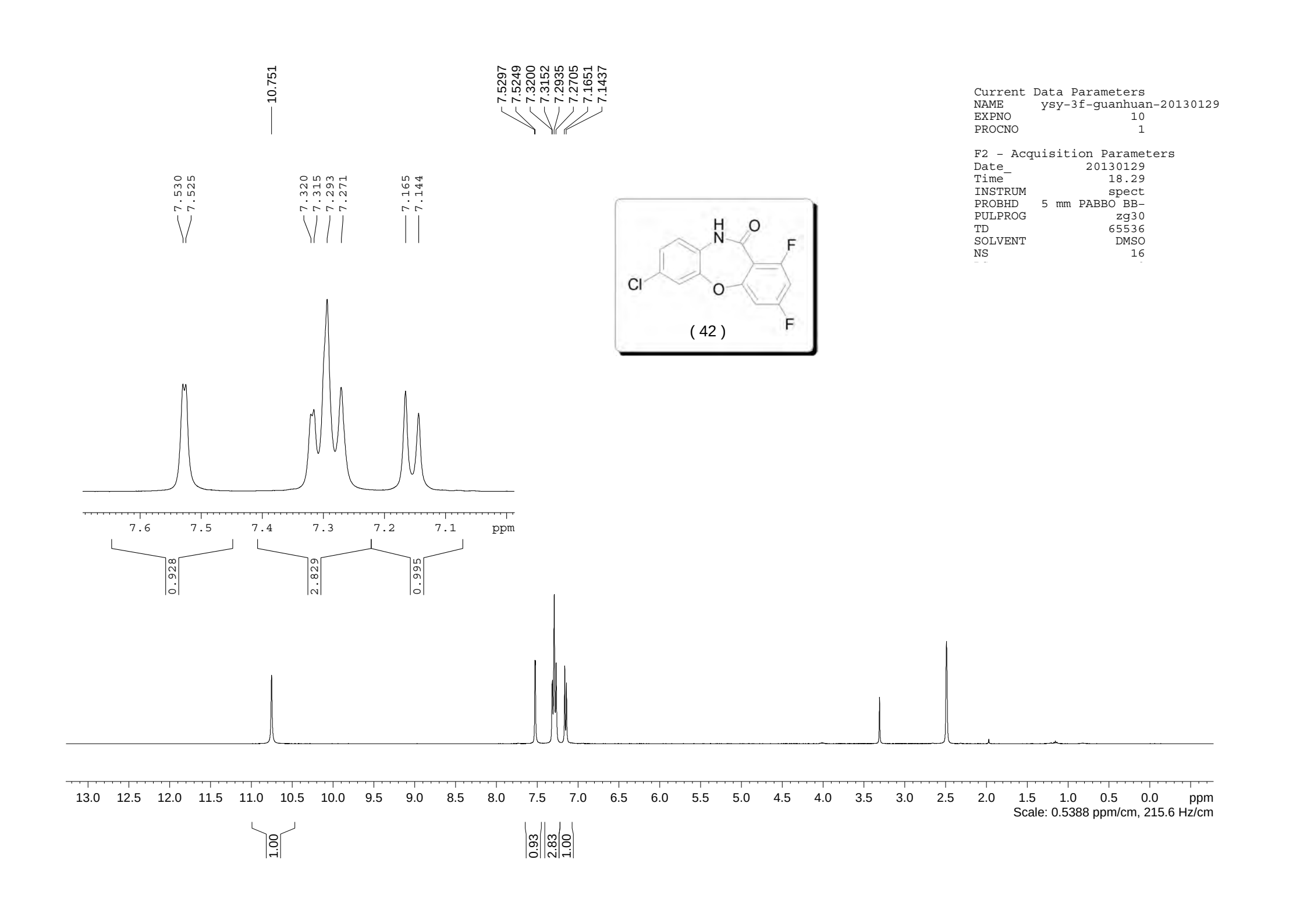

7.4799
7.4606
7.4416
7.4202
7.3996
7.3794
7.3577
7.3397
7.2944
7.2744
7.2530
7.1268
7.1081
7.0838
7.0623
7.0413
7.0220
7.0003
6.9766
6.9539
6.8455
6.8254
7.480
7.461
7.442
7.420
7.400
7.379
7.358
7.340
7.294
7.274
7.253
7.127
7.108
7.084
7.062
7.041
7.022
7.000
6.977
6.954
6.846
6.825
7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 ppm
6.004
1.284
4.025
1.000
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
6.00
1.28
4.03
1.00
Current Data Parameters
NAME 3-283-H-20121227
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121227
Time 14.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
Ph
O
N
F
O
(41)
Scale: 0.3554 ppm/cm, 142.2 Hz/cm

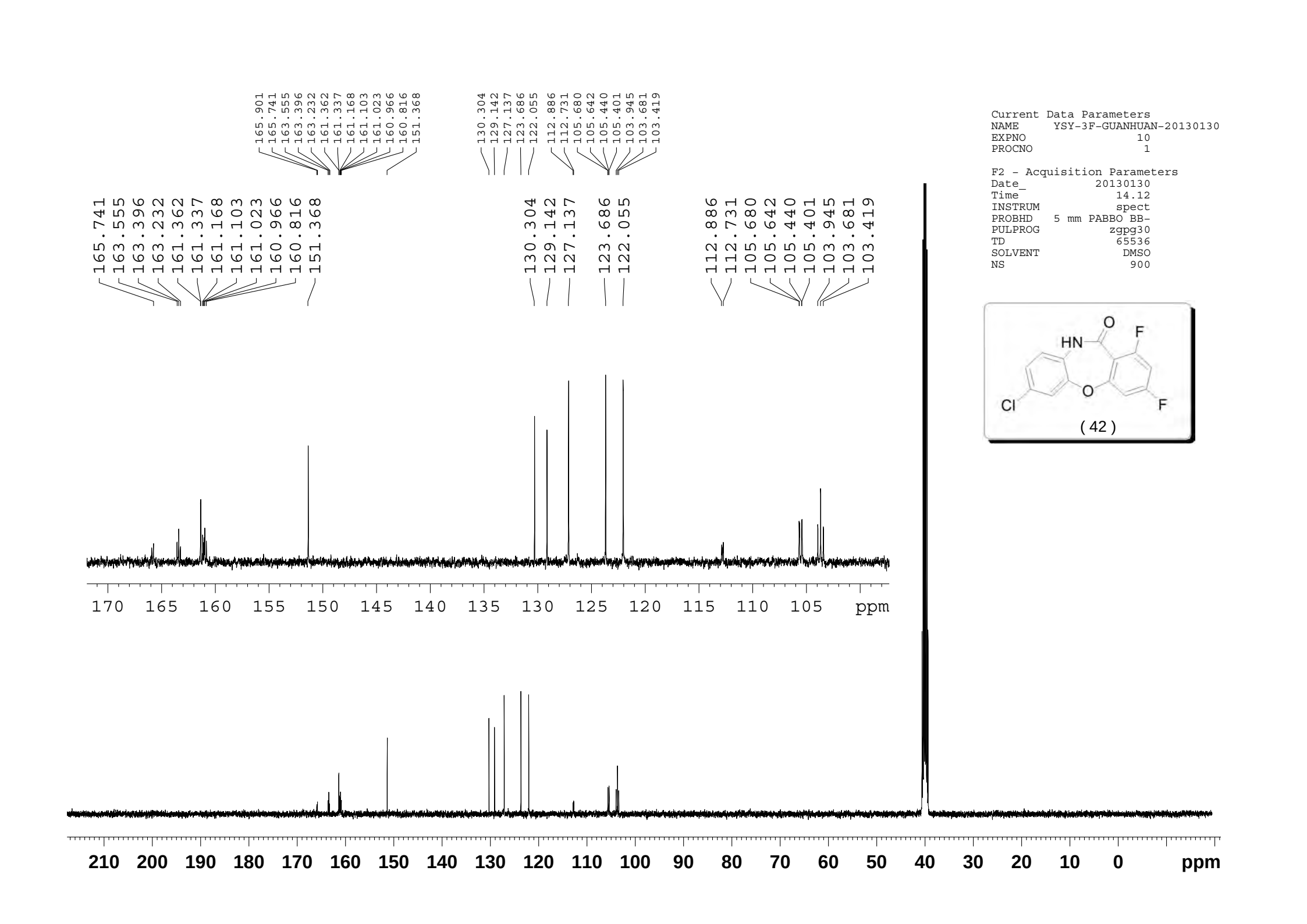


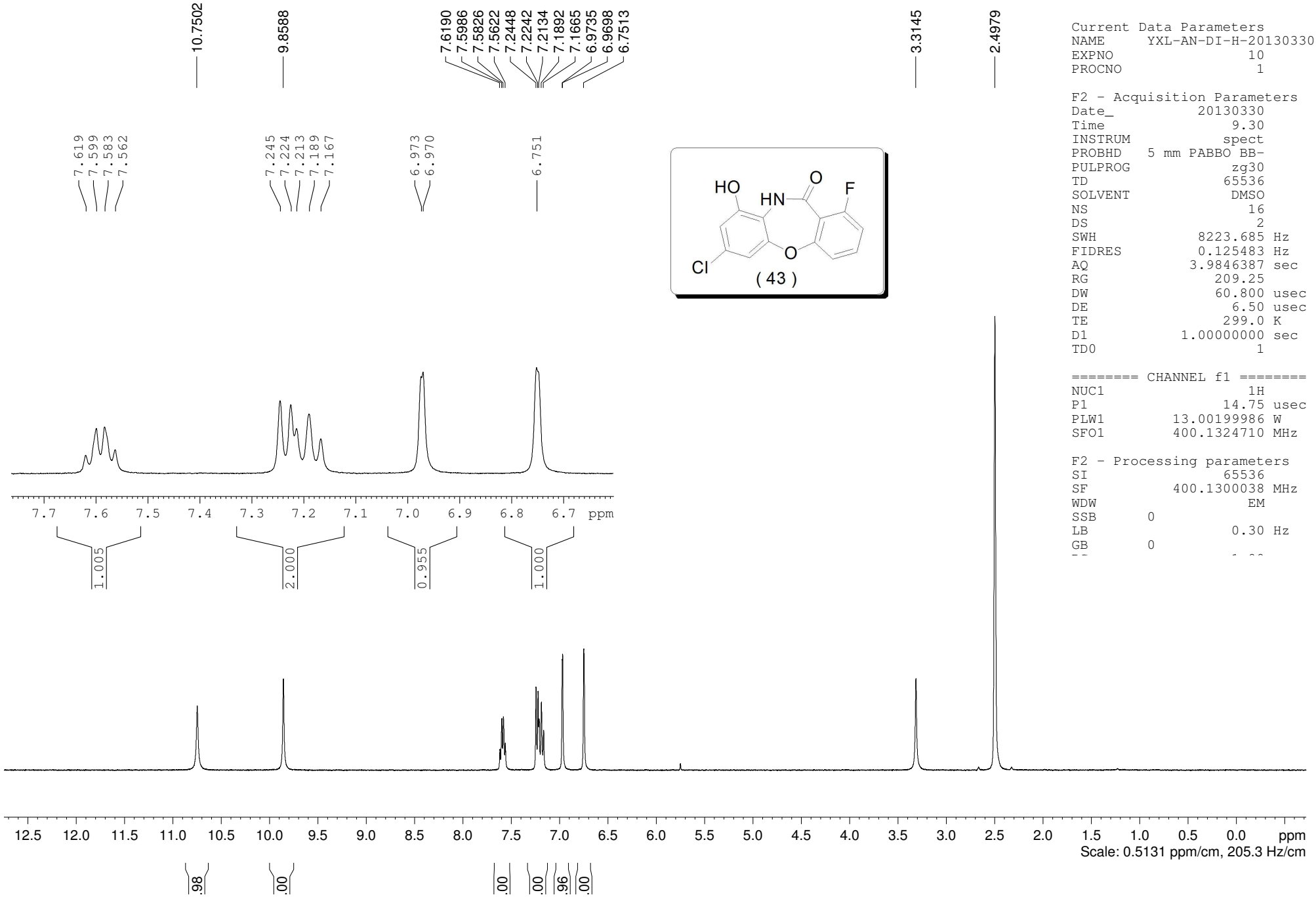
Current Data Parameters
NAME yxl-3-283-c-20121227
EXPNO 20
PROCNO 1

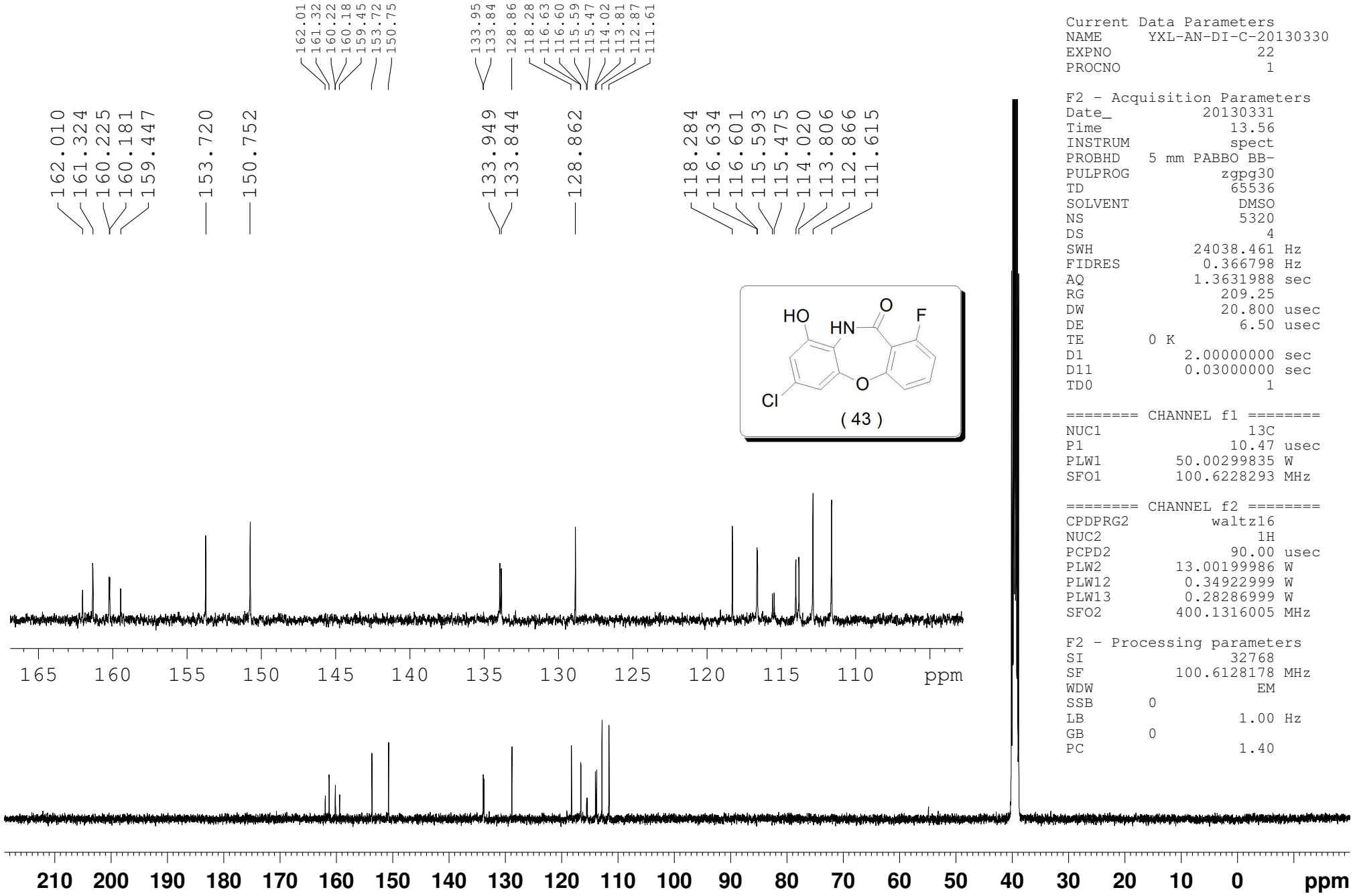
F2 - Acquisition Parameters
Date_ 20121227
Time 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512

(41)

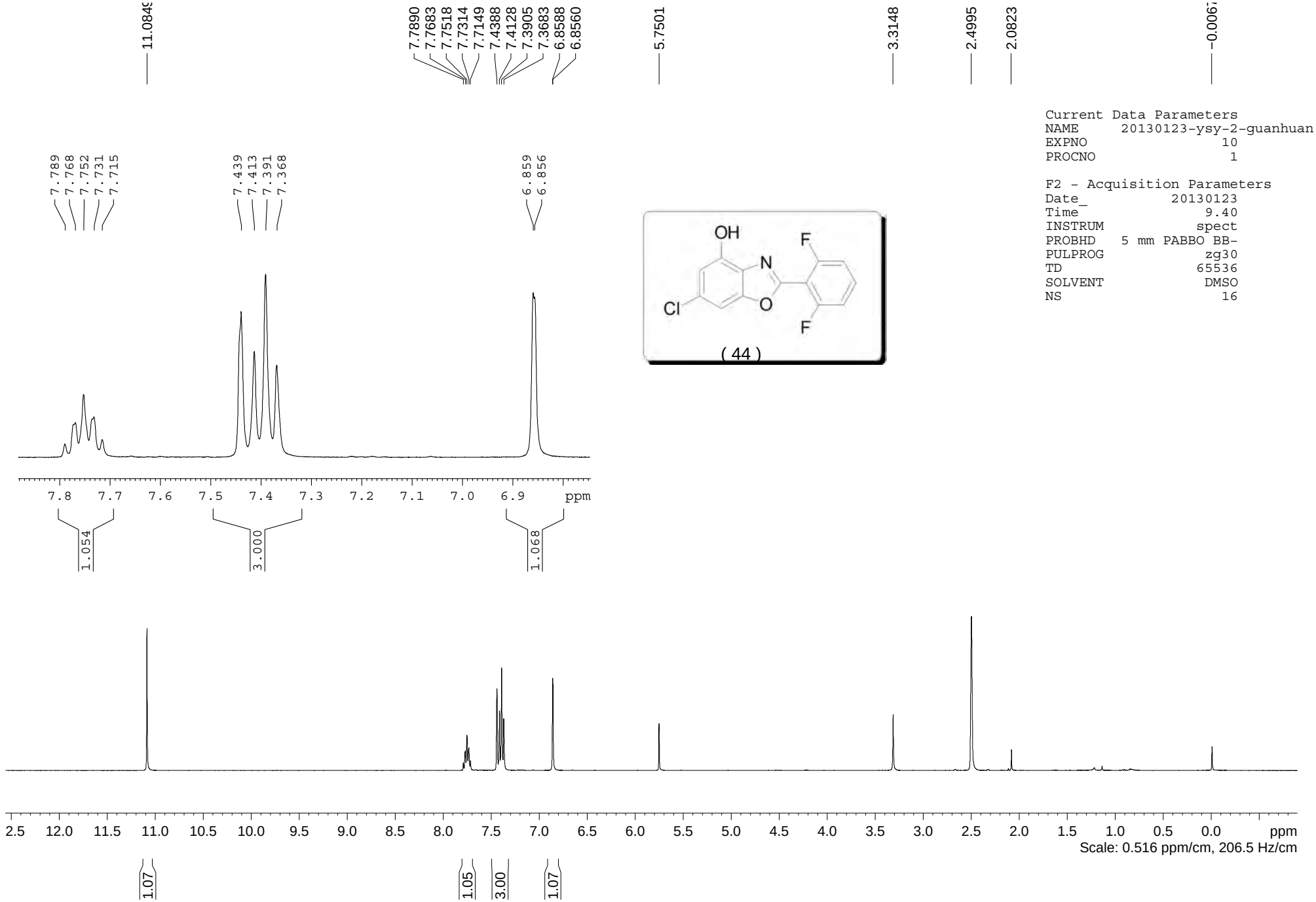

10.751
7.5297
7.5249
7.3200
7.3152
7.2935
7.2705
7.1651
7.1437
7.530
7.525
7.320
7.315
7.293
7.271
7.165
7.144
Current Data Parameters
NAME ysy-3f-guanhuan-20130129
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130129
Time 18.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
H
N
O
F
Cl
O
F
(42)
7.6
7.5
7.4
7.3
7.2
7.1
ppm
0.928
2.829
0.995
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.5388 ppm/cm, 215.6 Hz/cm
1.00
0.93
2.83
1.00

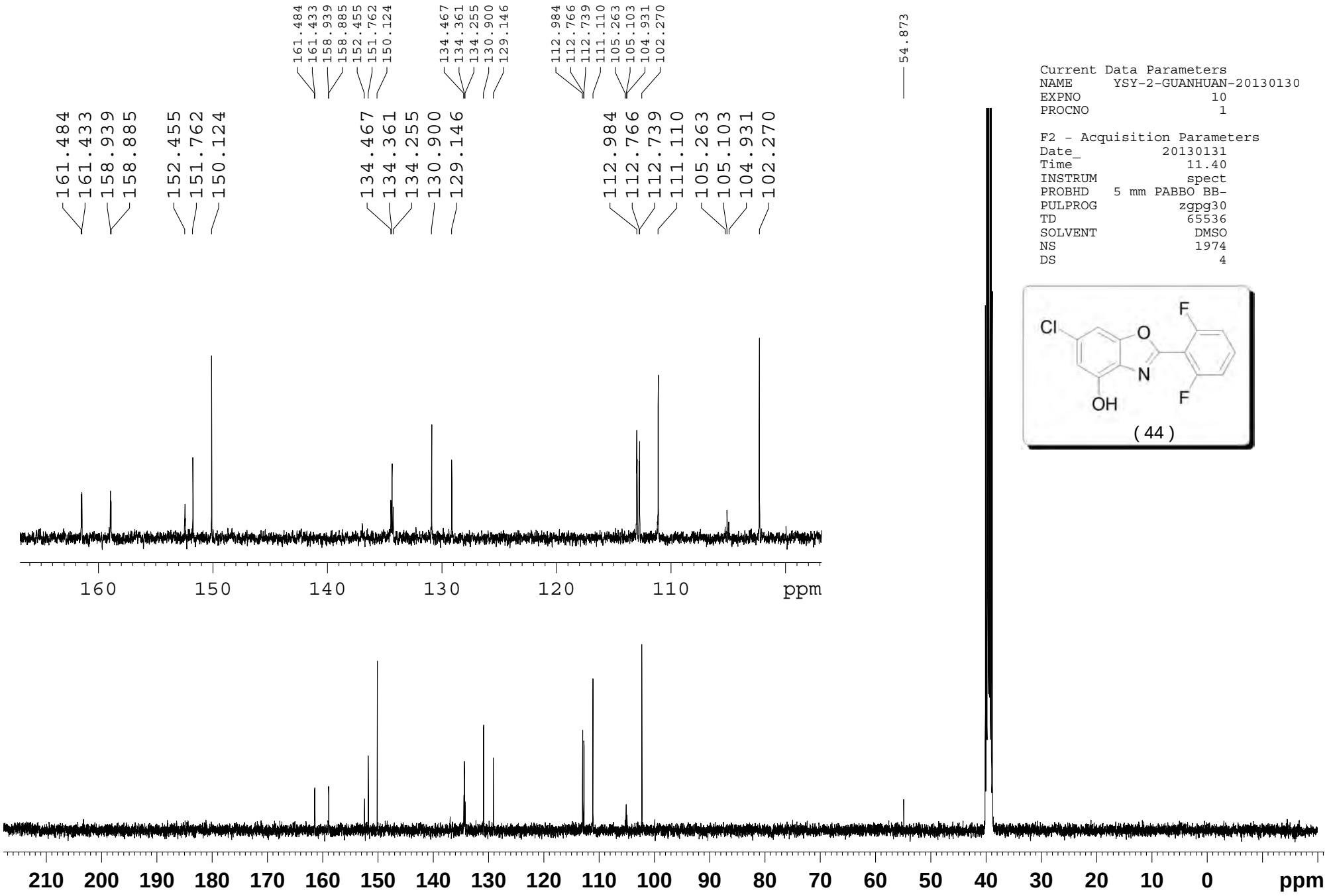

165.901
165.741
163.555
163.396
163.232
161.362
161.337
161.168
161.103
161.023
160.966
160.816
151.368
130.304
129.142
127.137
123.686
122.055
112.886
112.731
105.680
105.642
105.440
105.401
103.945
103.681
103.419
Current Data Parameters
NAME YSY-3F-GUANHUAN-20130130
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130130
Time 14.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 900
O
HN
F
Cl
O
F
(42)
170 165 160 155 150 145 140 135 130 125 120 115 110 105 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

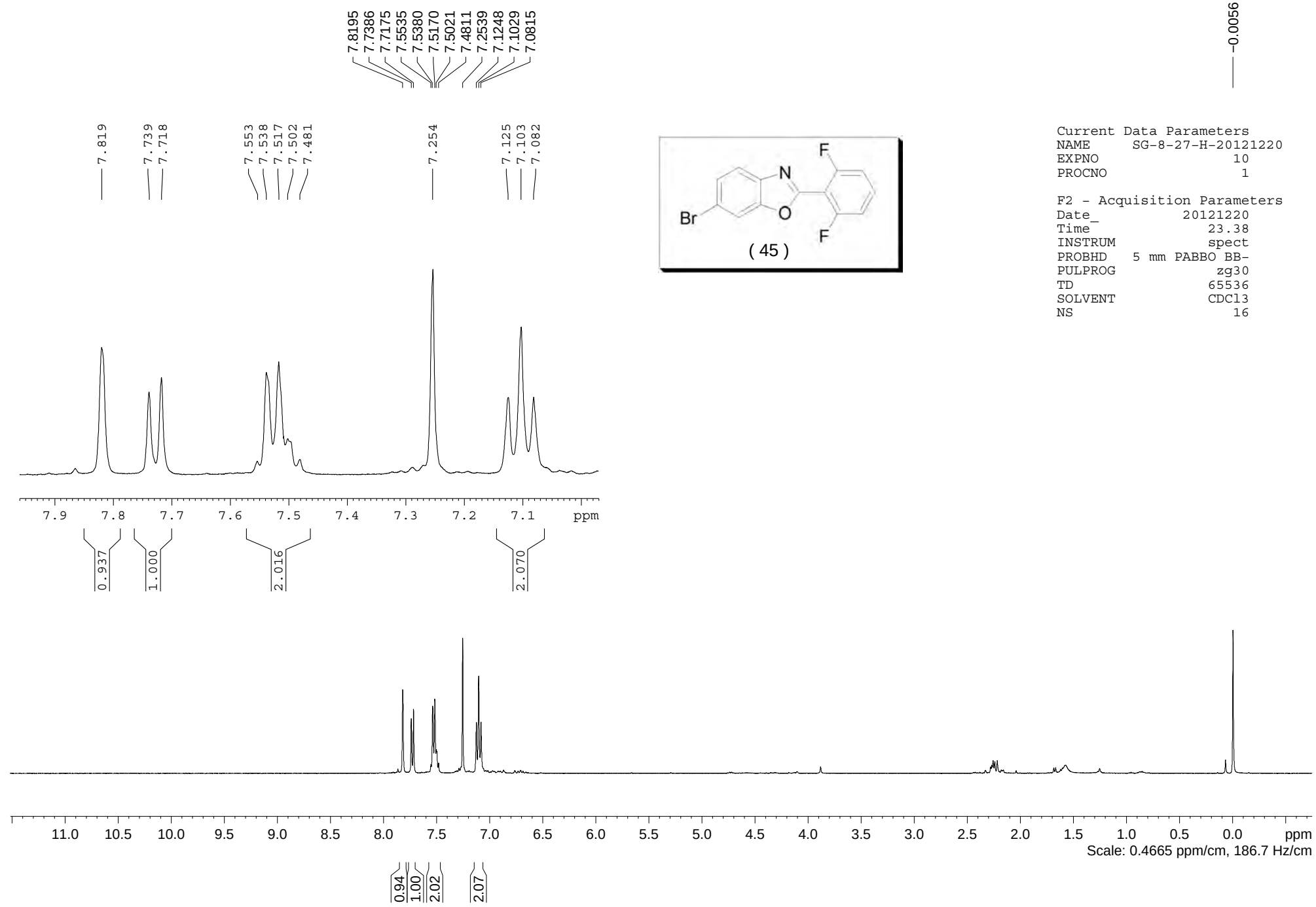

10.7502
9.8588
7.6190
7.5986
7.5826
7.5622
7.2448
7.2242
7.2134
7.1892
7.1665
6.9735
6.9698
6.7513
3.3145
2.4979
7.619
7.599
7.583
7.562
7.245
7.224
7.213
7.189
7.167
6.973
6.970
6.751
HO
HN
O
F
Cl
O
(43)
7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 ppm
1.005
2.000
0.955
1.000
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
.98
.00
.00
.00
.96
.00
Current Data Parameters
NAME YXL-AN-DI-H-20130330
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130330
Time 9.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 209.25
DW 60.800 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300038 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
Scale: 0.5131 ppm/cm, 205.3 Hz/cm

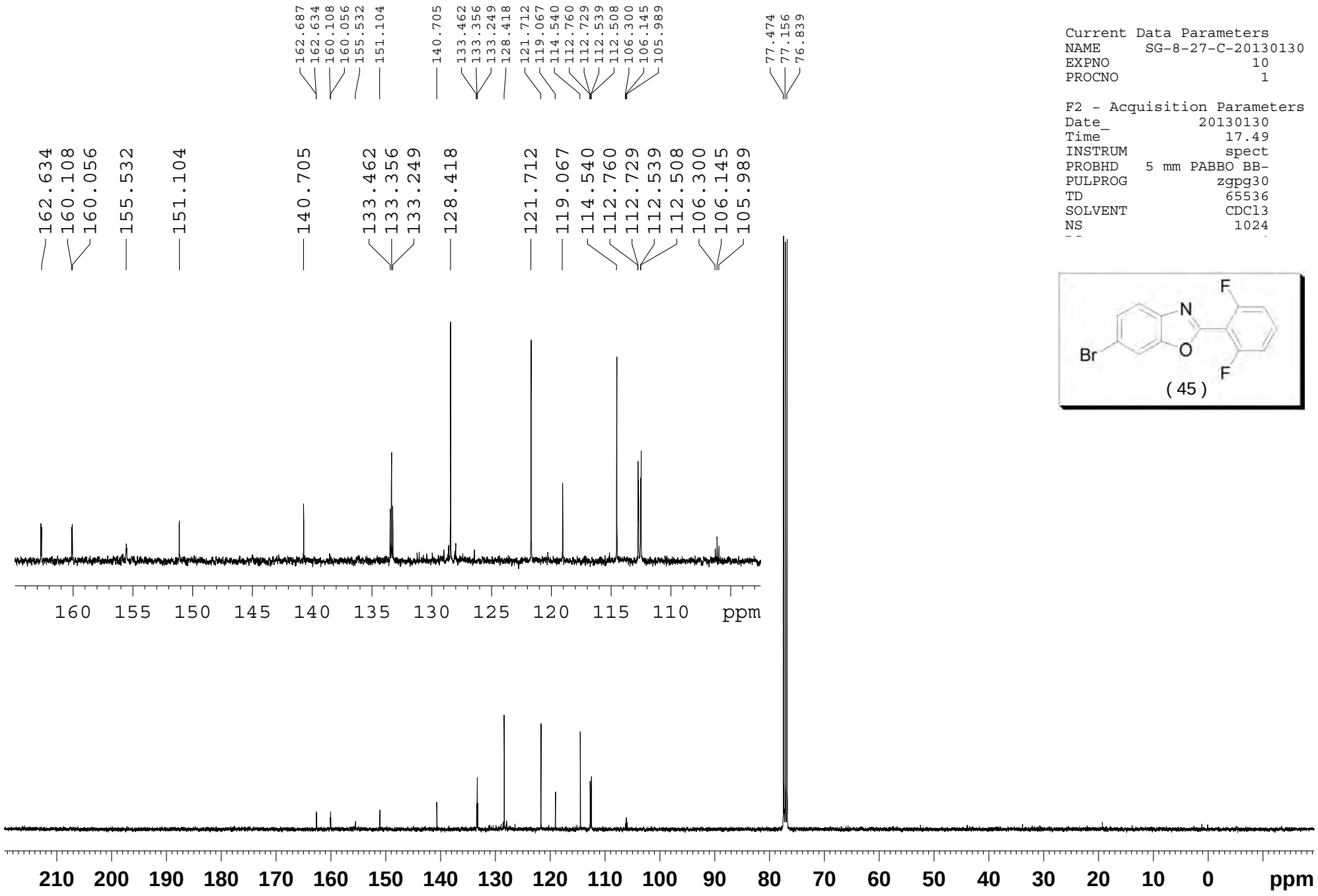


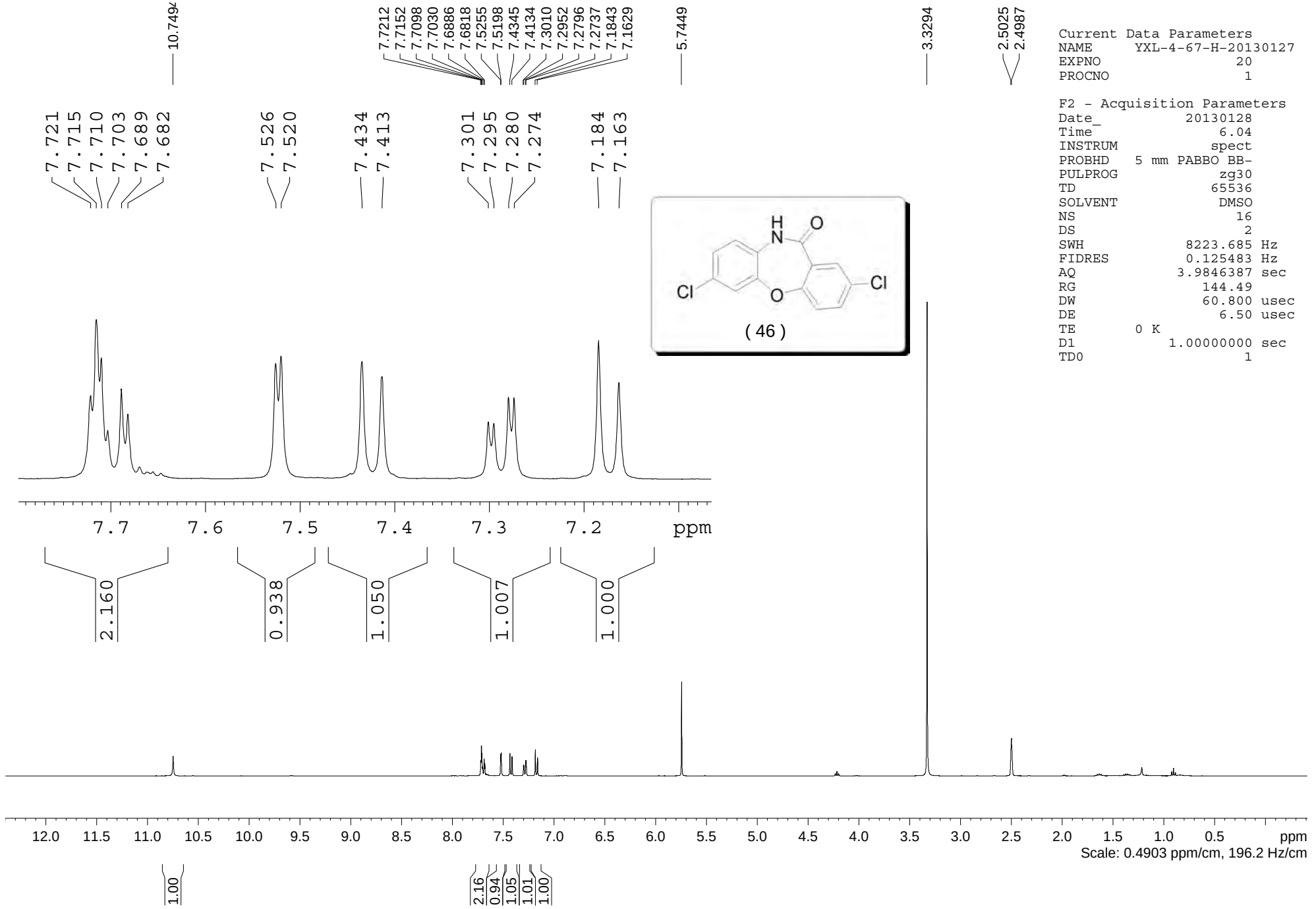

```
Current Data Parameters
NAME      YXL-AN-DI-C-20130330
EXPNO                      22
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130331
Time                  13.56
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                     5320
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                   209.25
DW                   20.800 usec
DE                    6.50 usec
TE          0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   10.47 usec
PLW1           50.00299835 W
SFO1          100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.34922999 W
PLW13           0.28286999 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6128178 MHz
WDW                      EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```


11.084
7.7890
7.7683
7.7518
7.7314
7.7149
7.4388
7.4128
7.3905
7.3683
6.8588
6.8560
5.7501
3.3148
2.4995
2.0823
-0.006
7.789
7.768
7.752
7.731
7.715
7.439
7.413
7.391
7.368
6.859
6.856
OH
F
N
Cl
O
F
(44)
Current Data Parameters
NAME 20130123-ysy-2-guanhuan
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130123
Time 9.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 ppm
1.054
3.000
1.068
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.516 ppm/cm, 206.5 Hz/cm
1.07
1.05
3.00
1.07


161.484
161.433
158.939
158.885
152.455
151.762
150.124
134.467
134.361
134.255
130.900
129.146
112.984
112.766
112.739
111.110
105.263
105.103
104.931
102.270
54.873
Current Data Parameters
NAME YSY-2-GUANHUAN-20130130
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time 11.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1974
DS 4
Cl
O
F
N
OH
F
(44)
160
150
140
130
120
110
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.8195
7.7386
7.7175
7.5535
7.5380
7.5170
7.5021
7.4811
7.2539
7.1248
7.1029
7.0815
-0.0056
7.819
7.739
7.718
7.553
7.538
7.517
7.502
7.481
7.254
7.125
7.103
7.082
F
N
Br
O
F
(45)
Current Data Parameters
NAME SG-8-27-H-20121220
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121220
Time 23.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
0.937
1.000
2.016
2.070
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.94
1.00
2.02
2.07
Scale: 0.4665 ppm/cm, 186.7 Hz/cm

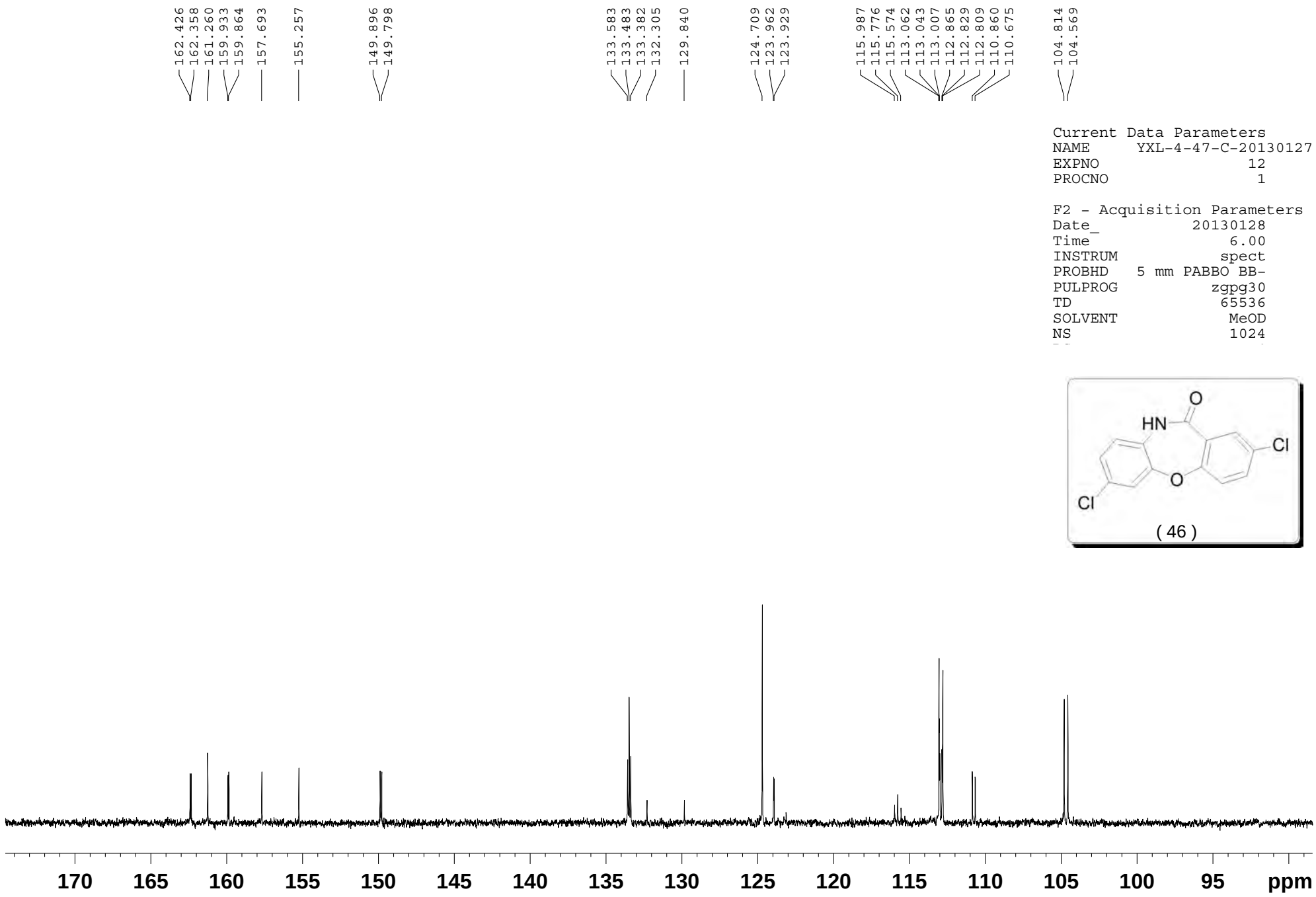

162.687
162.634
160.108
160.056
155.532
151.104
140.705
133.462
133.356
133.249
128.418
121.712
119.067
114.540
112.760
112.729
112.539
112.508
106.300
106.145
105.989
77.474
77.156
76.839
Current Data Parameters
NAME SG-8-27-C-20130130
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130130
Time 17.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
F
N
Br
O
F
(45)
160 155 150 145 140 135 130 125 120 115 110 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

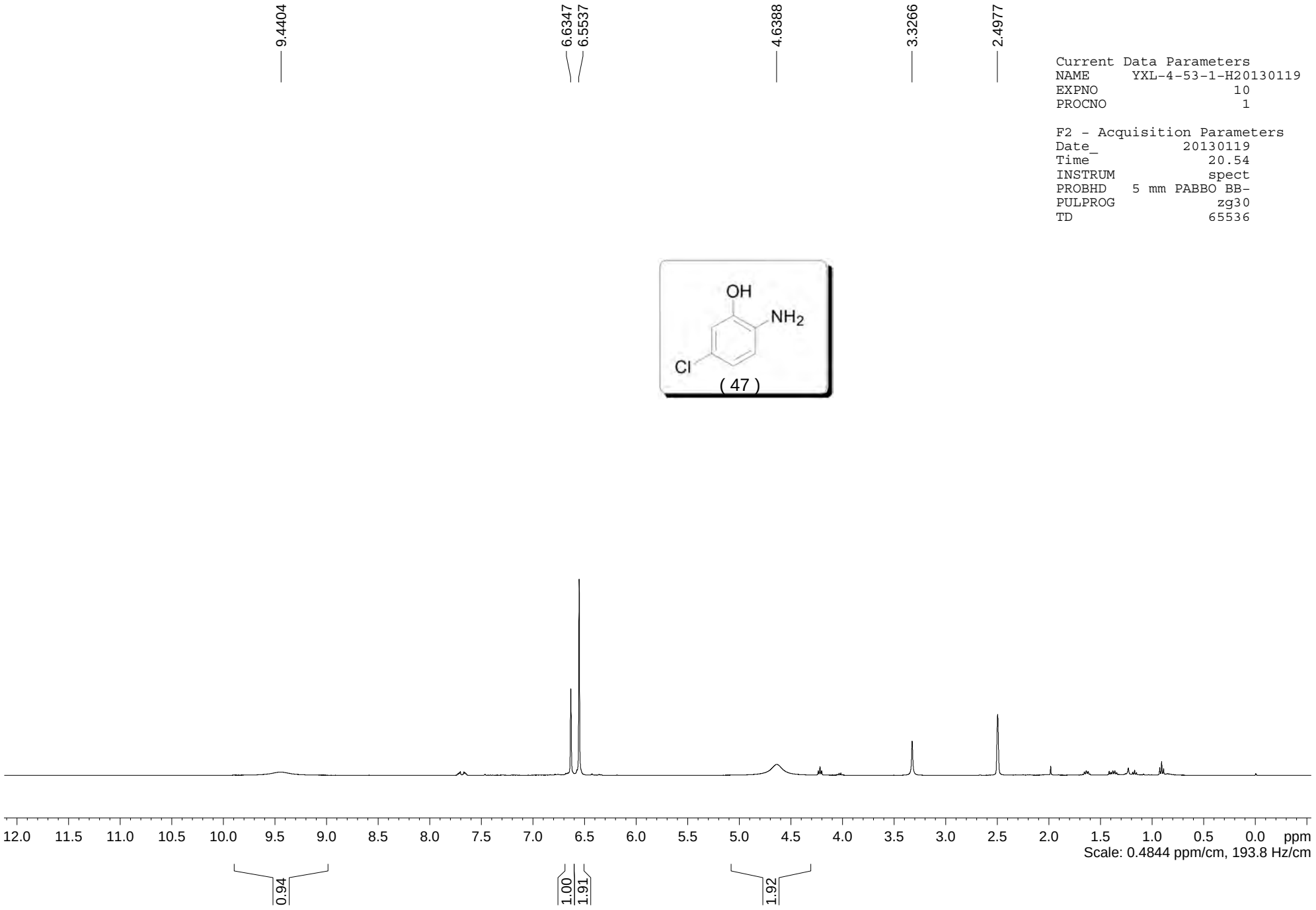


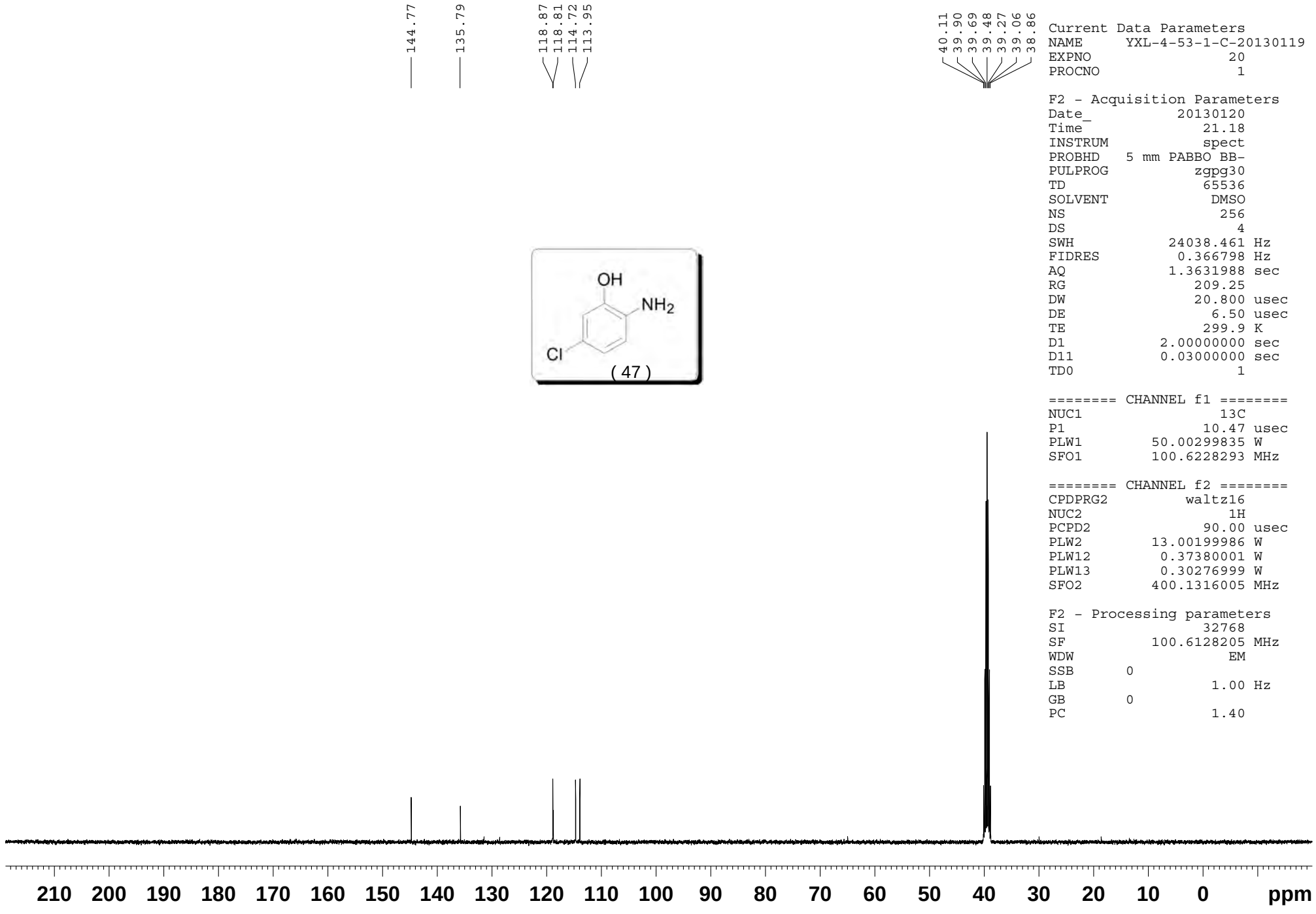
Current Data Parameters
NAME YXL-4-67-H-20130127
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130128
Time 6.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144.49
DW 60.800 usec
DE 6.50 usec
TE 0 K
D1 1.00000000 sec
TD0 1

Scale: 0.4903 ppm/cm, 196.2 Hz/cm


162.426
162.358
161.260
159.933
159.864
157.693
155.257
149.896
149.798
133.583
133.483
133.382
132.305
129.840
124.709
123.962
123.929
115.987
115.776
115.574
113.062
113.043
113.007
112.865
112.829
112.809
110.860
110.675
104.814
104.569
Current Data Parameters
NAME YXL-4-47-C-20130127
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130128
Time 6.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
O
HN
Cl
O
Cl
(46)
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
ppm


9.4404
6.6347
6.5537
4.6388
3.3266
2.4977
Current Data Parameters
NAME YXL-4-53-1-H20130119
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130119
Time 20.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
OH
NH2
Cl
(47)
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
Scale: 0.4844 ppm/cm, 193.8 Hz/cm
0.94
1.00
1.91
1.92



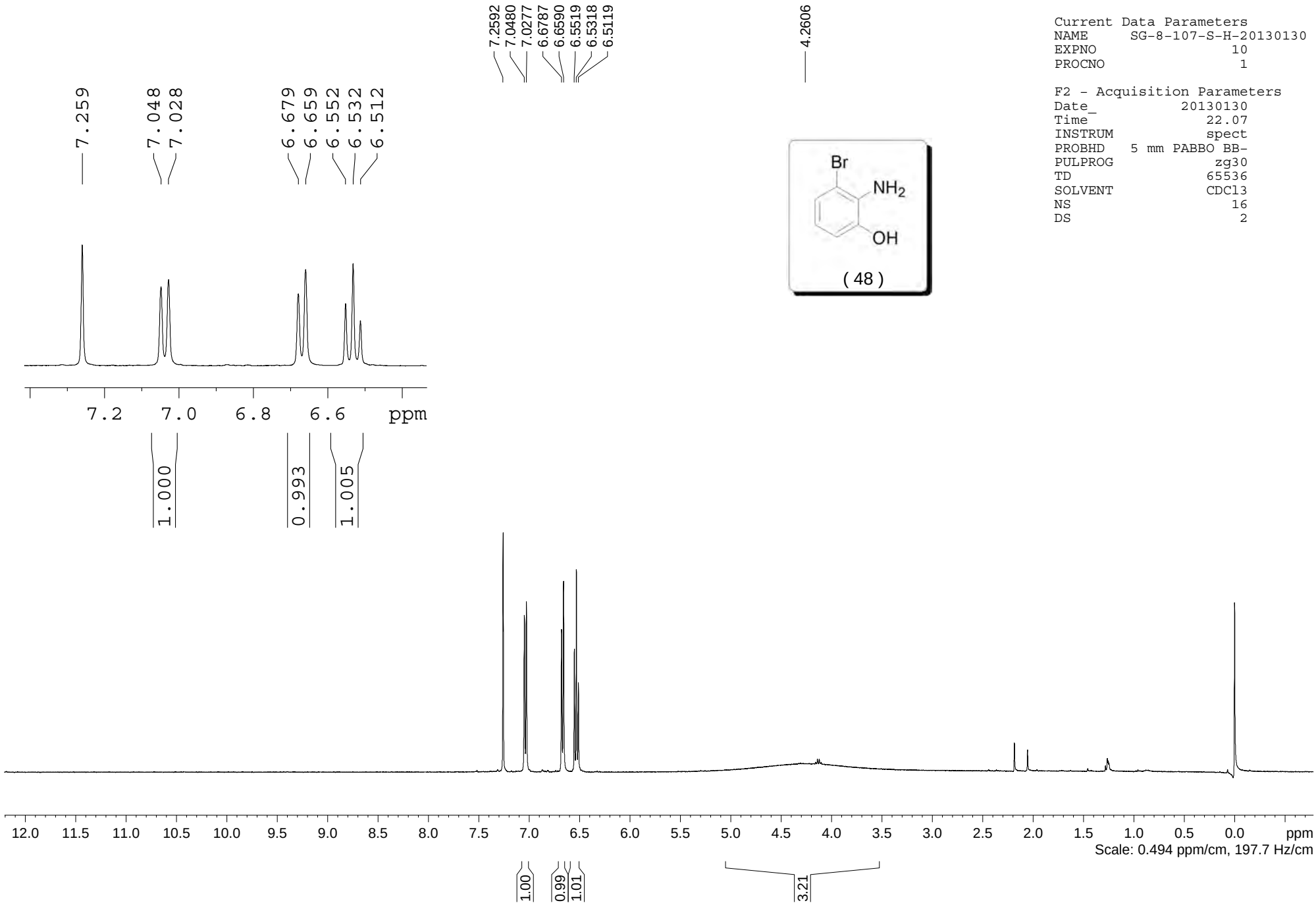
Current Data Parameters
NAME YXL-4-53-1-C-20130119
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130120
Time 21.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 209.25
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.47 usec
PLW1 50.00299835 W
SFO1 100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.00199986 W
PLW12 0.37380001 W
PLW13 0.30276999 W
SFO2 400.1316005 MHz

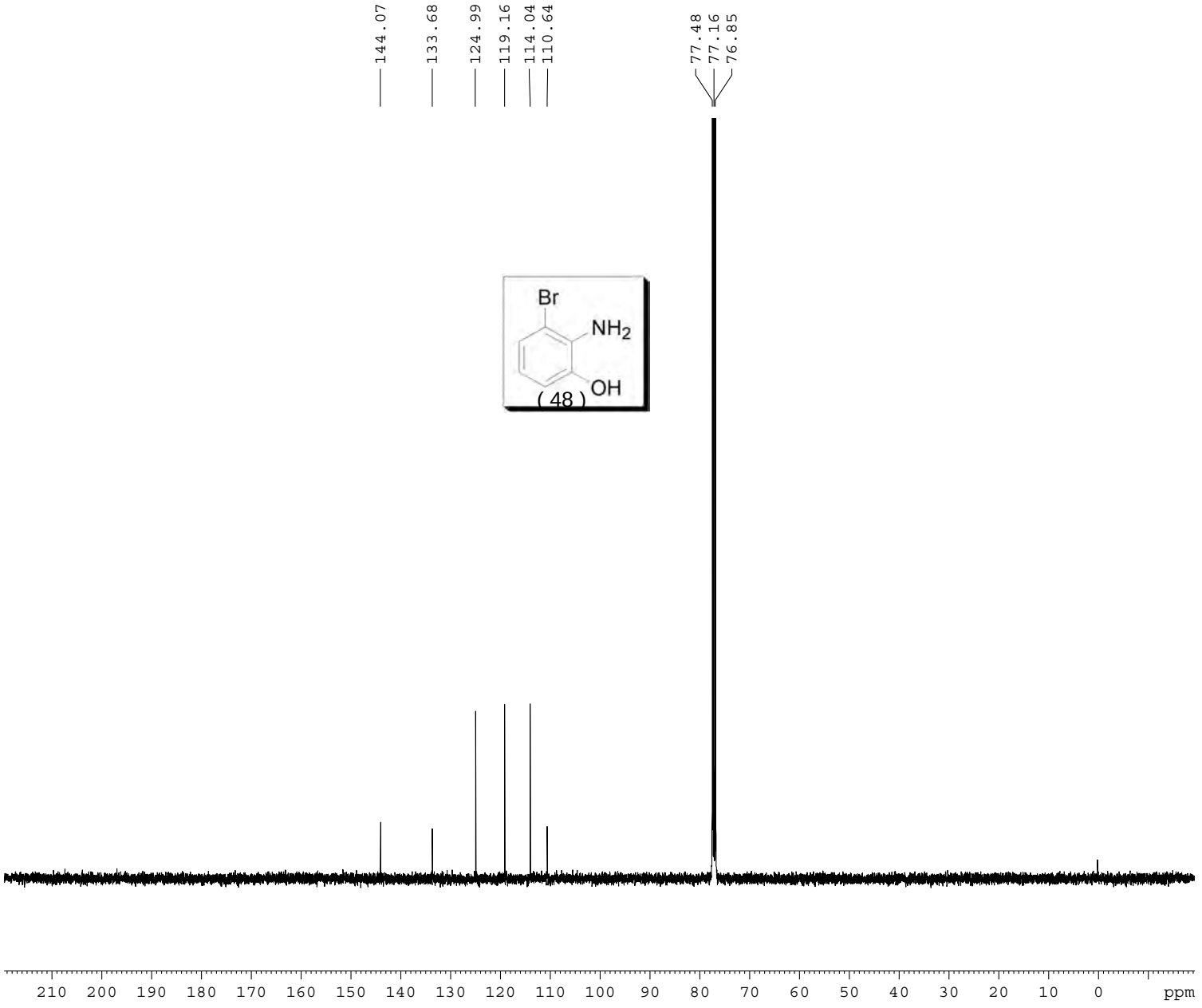
F2 - Processing parameters
SI 32768
SF 100.6128205 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



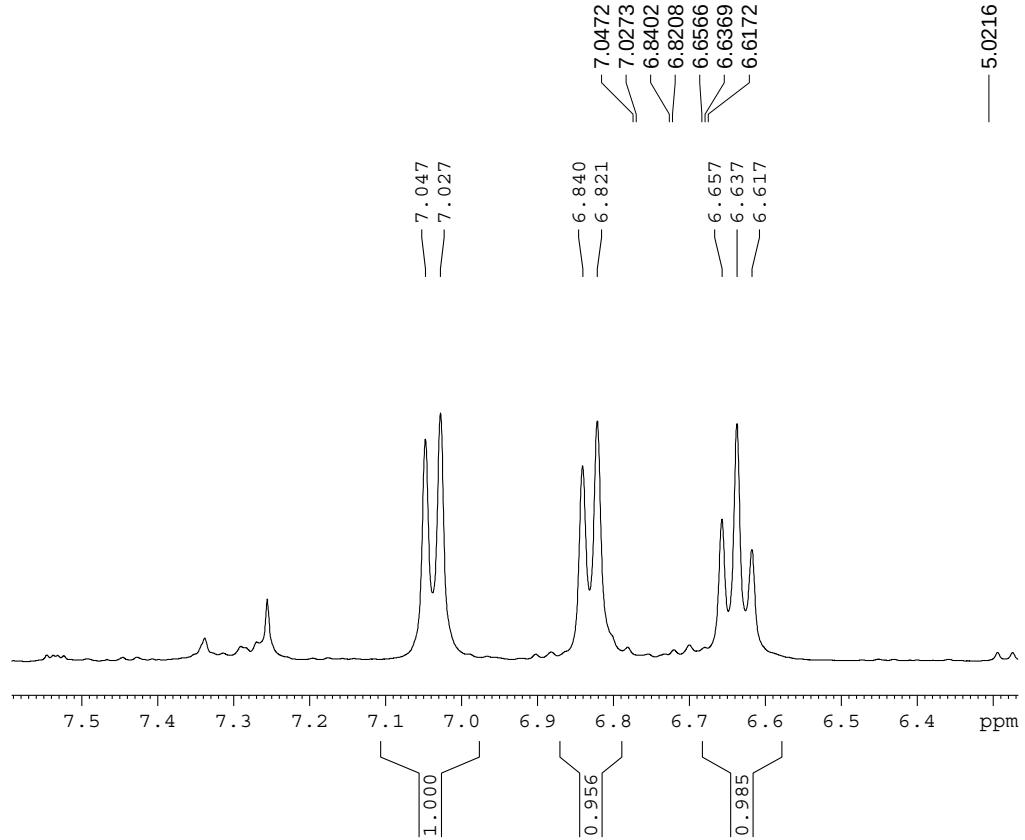

```
Current Data Parameters
NAME      SG-8-107-S-H-20130130
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_             20130130
Time                22.07
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
```

Scale: 0.494 ppm/cm, 197.7 Hz/cm

C13CPD CDCl3 {D:\NMR_DATA} RY 12

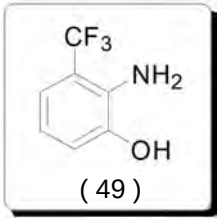



```
Current Data Parameters
NAME      SG-8-107-S-C-20130130
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130130
Time                  19.01
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  209.25
DW                  20.800 usec
DE                   6.50 usec
TE                  299.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   10.47 usec
PLW1          50.00299835 W
SFO1          100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2          13.00199986 W
PLW12          0.37380001 W
PLW13          0.30276999 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6127543 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

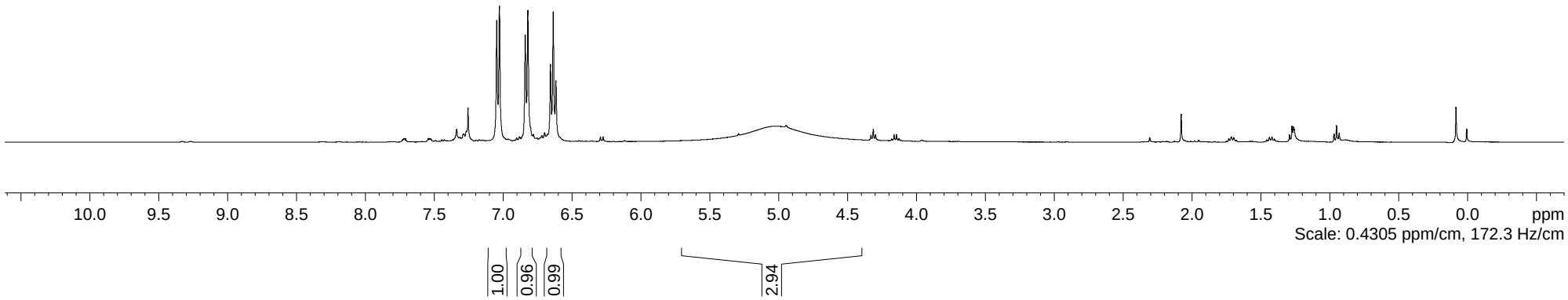


Current Data Parameters
NAME sg-3fjia-h-crude-20130201
EXPNO 10
PROCNO 1

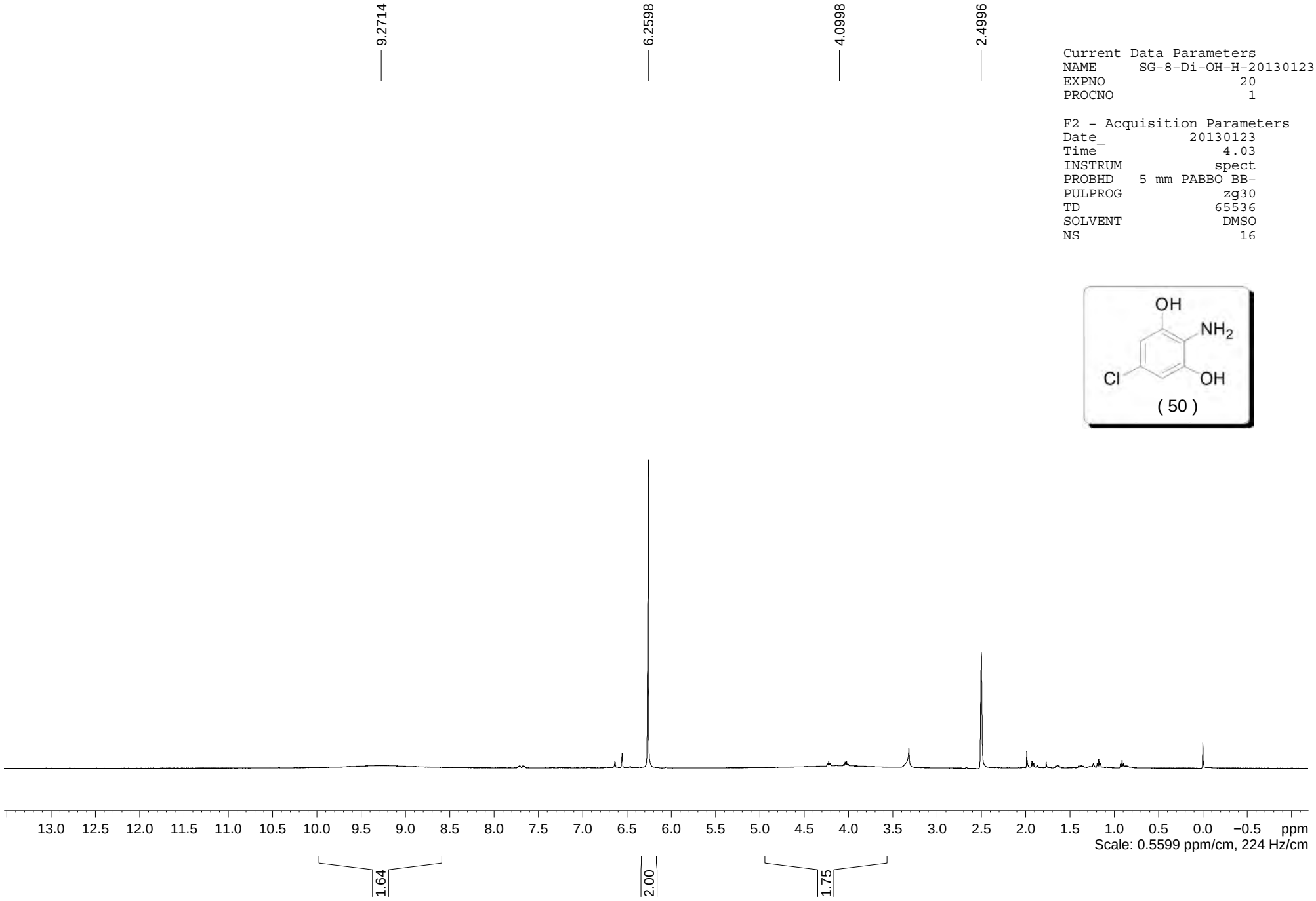
F2 - Acquisition Parameters
Date_ 20130201
Time 18.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2

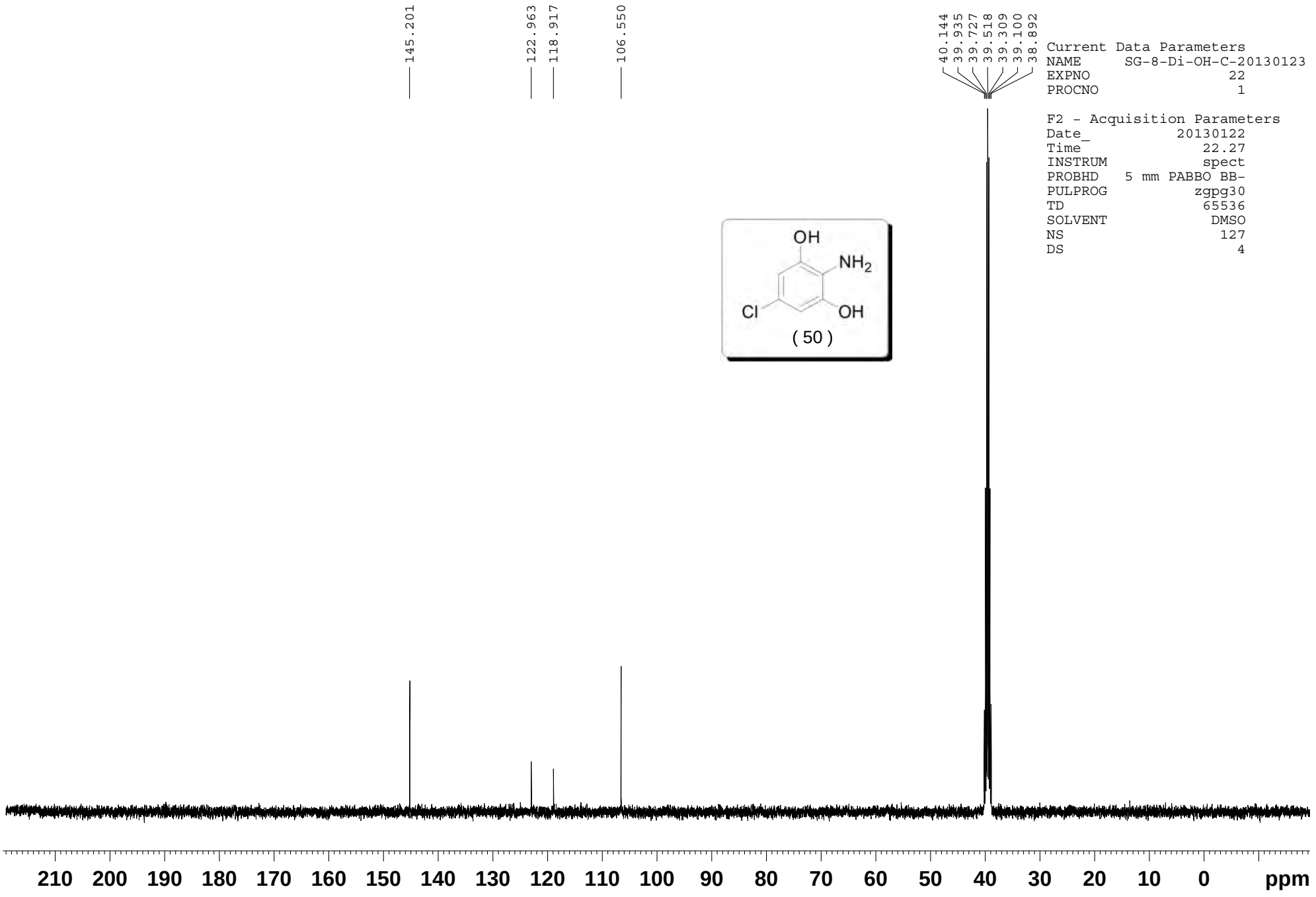


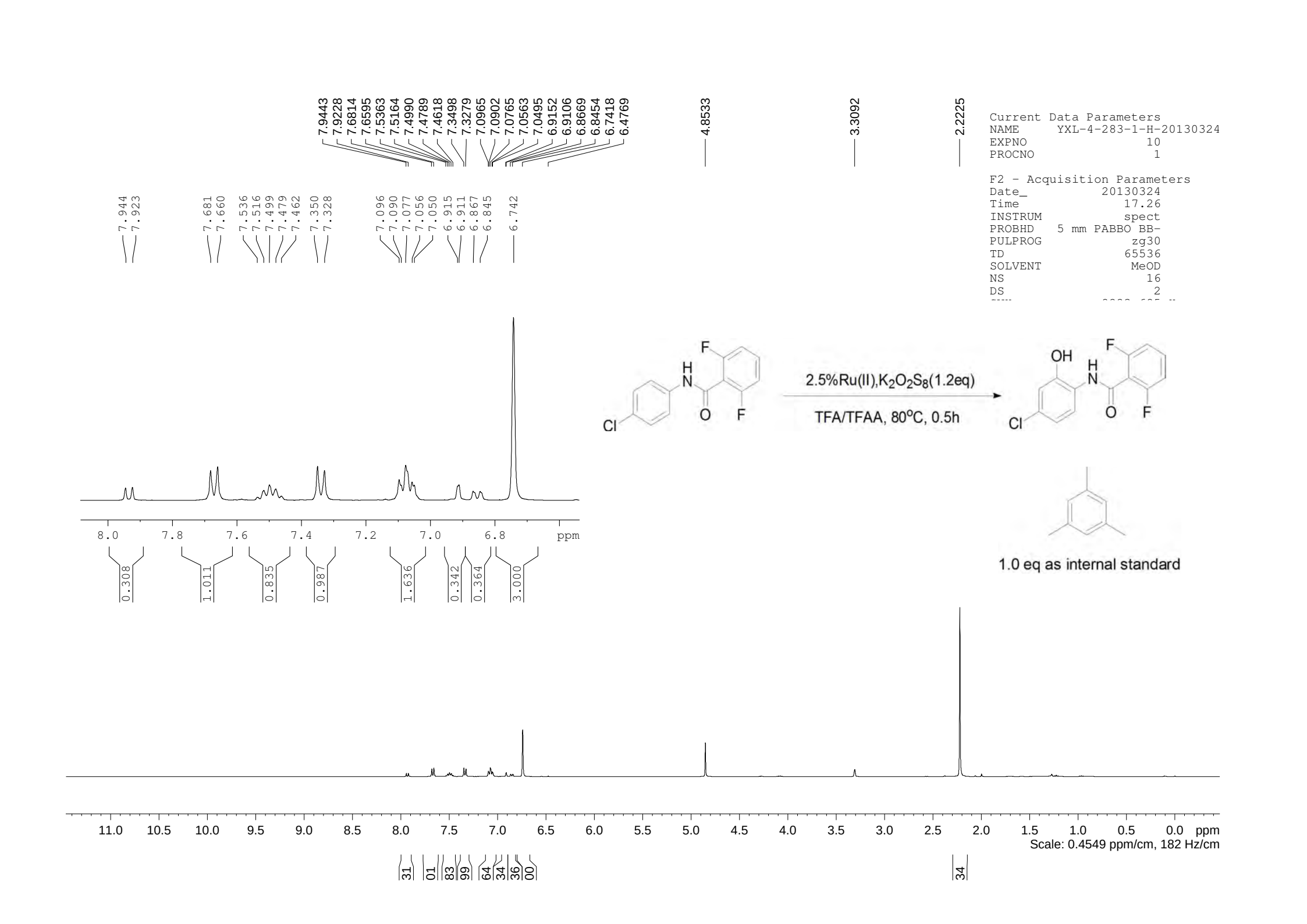
(49)

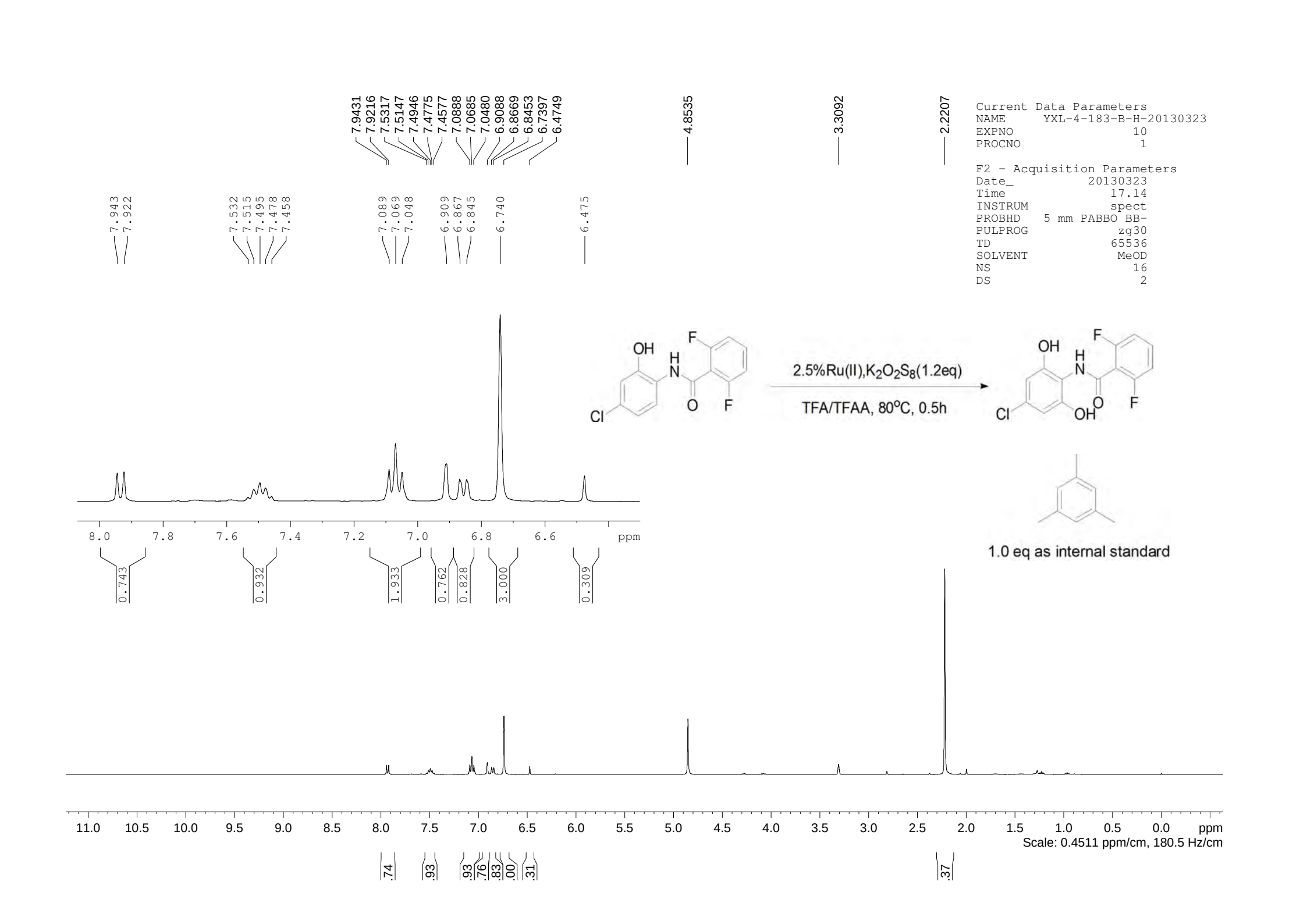


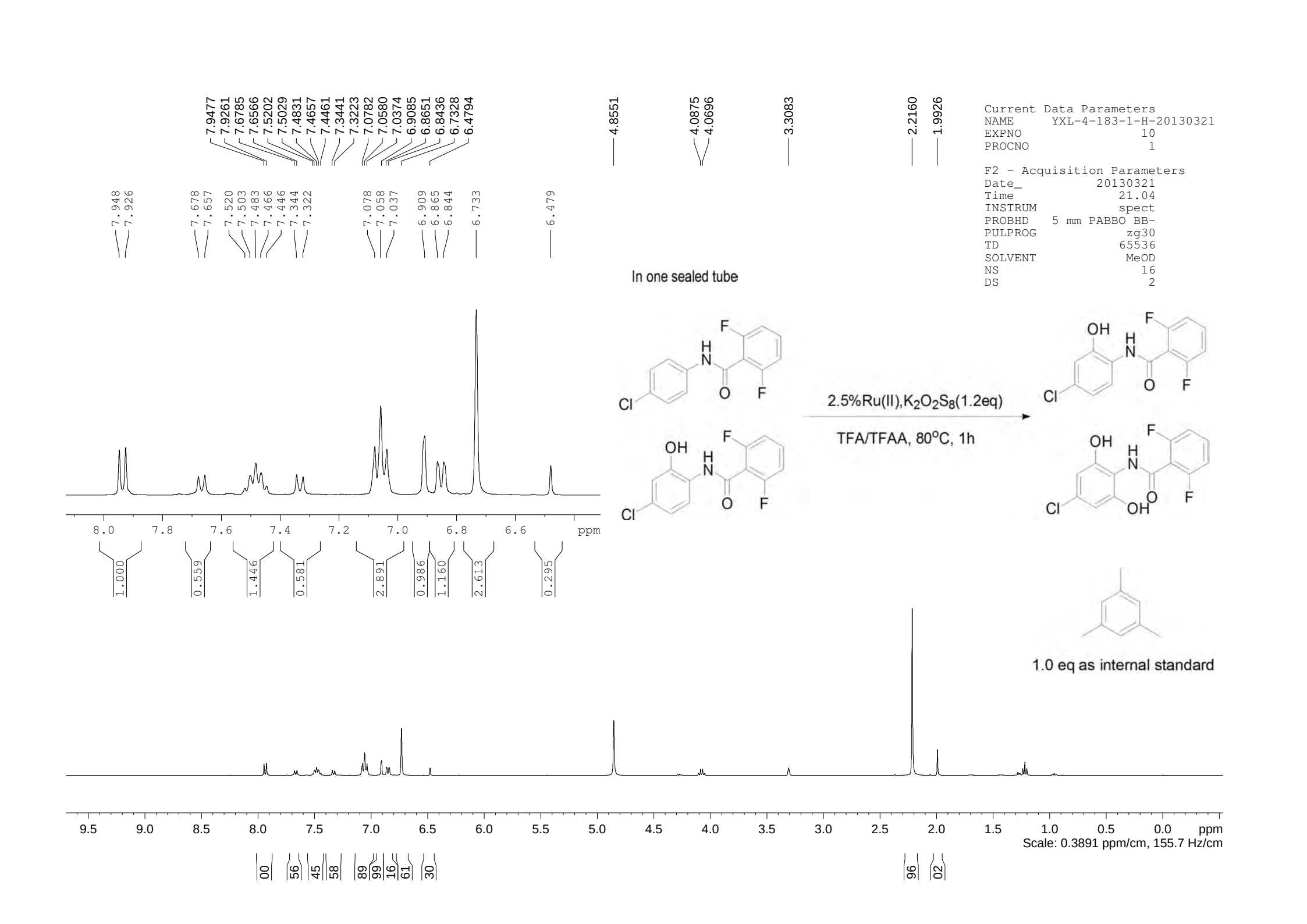
Scale: 0.4305 ppm/cm, 172.3 Hz/cm


9.2714
6.2598
4.0998
2.4996
Current Data Parameters
NAME SG-8-Di-OH-H-20130123
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130123
Time 4.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
OH
NH2
Cl
OH
(50)
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
Scale: 0.5599 ppm/cm, 224 Hz/cm
1.64
2.00
1.75


145.201
122.963
118.917
106.550
40.144
39.935
39.727
39.518
39.309
39.100
38.892
Current Data Parameters
NAME SG-8-Di-OH-C-20130123
EXPNO 22
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130122
Time 22.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 127
DS 4
OH
NH2
Cl
OH
(50)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.9443
7.9228
7.6814
7.6595
7.5363
7.5164
7.4990
7.4789
7.4618
7.3498
7.3279
7.0965
7.0902
7.0765
7.0563
7.0495
6.9152
6.9106
6.8669
6.8454
6.7418
6.4769
4.8533
3.3092
2.2225
Current Data Parameters
NAME YXL-4-283-1-H-20130324
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130324
Time 17.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
7.944
7.923
7.681
7.660
7.536
7.516
7.499
7.479
7.462
7.350
7.328
7.096
7.090
7.077
7.056
7.050
6.915
6.911
6.867
6.845
6.742
8.0
7.8
7.6
7.4
7.2
7.0
6.8
ppm
0.308
1.011
0.835
0.987
1.636
0.342
0.364
3.000
2.5%Ru(II),K2O2S8(1.2eq)
TFA/TFAA, 80°C, 0.5h
1.0 eq as internal standard
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4549 ppm/cm, 182 Hz/cm
31
01
83
99
64
34
36
00
34


7.9431
7.9216
7.5317
7.5147
7.4946
7.4775
7.4577
7.0888
7.0685
7.0480
6.9088
6.8669
6.8453
6.7397
6.4749
4.8535
3.3092
2.2207
Current Data Parameters
NAME YXL-4-183-B-H-20130323
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130323
Time 17.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
7.943
7.922
7.532
7.515
7.495
7.478
7.458
7.089
7.069
7.048
6.909
6.867
6.845
6.740
6.475
OH
F
H
N
O
F
Cl
2.5%Ru(II),K2O2S8(1.2eq)
TFA/TFAA, 80°C, 0.5h
1.0 eq as internal standard
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
ppm
0.743
0.932
1.933
0.762
0.828
3.000
0.309
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4511 ppm/cm, 180.5 Hz/cm
.74
.93
.93
.76
.83
.00
.31
.37


7.9477
7.9261
7.6785
7.6566
7.5202
7.5029
7.4831
7.4657
7.4461
7.3441
7.3223
7.0782
7.0580
7.0374
6.9085
6.8651
6.8436
6.7328
6.4794
4.8551
4.0875
4.0696
3.3083
2.2160
1.9926
Current Data Parameters
NAME YXL-4-183-1-H-20130321
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130321
Time 21.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
In one sealed tube
2.5%Ru(II),K2O2S8(1.2eq)
TFA/TFAA, 80°C, 1h
1.0 eq as internal standard
ppm
Scale: 0.3891 ppm/cm, 155.7 Hz/cm