Supporting Information

For

A one-pot Hypoiodite catalysed oxidative cycloetherification approach to benoxazoles

Siva Senthil Kumar Boominathan,[a] Wan-Ping Hu,[b] Gopal Chandru Senadi,[a] Jaya Kishore Vandavasi,[a] Jeh-Jeng Wang *[a]

[a] Department of medicinal and applied chemistry, Kaohsiung Medical University, Kaohsiung, Taiwan.
[b] Department of Biotechnology, Kaohsiung Medical University, Kaohsiung, Taiwan.

Email: jjwang@kmu.edu.tw

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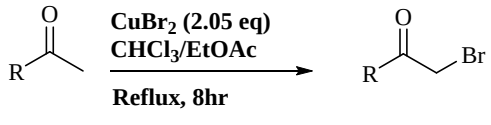
General information:

All chemicals were purchased form best grade in commercially available and are used without further purification. All solvents were purchased as HPLC grade and used without any purification or distillation. Analytical thin layer chromatography was performed on aluminium plate coated with silica gel (Merck). Gravity column chromatography was performed using 100-200 mesh silica gel and mixtures of hexane-ethyl acetate were used for elution. Visualization was accomplished using ultraviolet light (254 nm) and chemical staining with acidic potassium permanganate solution and Iodine.

Melting points was determined using "Mel-Temp" melting point apparatus and were uncorrected. Proton nuclear magnetic resonance (^{1}H NMR) and Carbon nuclear magnetic resonance (^{13}C NMR) spectra's were recorded using a Varian (400 MHz) or a *J*EOL (200 MHz) spectrometer. All spectra were recorded at ambient temperature (298 K). Chemical shifts (δ) are quoted in ppm relative to residual solvent (CHCl3: δ = 7.26 ppm for ^{1}H and δ = 77.0 for 13C; $(CD_3)_2SO$: δ = 2.50 ppm for ^{1}H). Coupling constants (*J*) are corrected and quoted to the nearest 0.1 Hz. The following abbreviations are used to indicate the multiplicity of the signals: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; bs = broad singlet; dt = doublet of triplet; td = triplet of doublet. High resolution mass spectra (HRMS) were measured on a Micromass Q-TOF spectrometer using EI (electron impact, 70 eV) at the *J*oint Center for High valued Instruments, NSYS University, Kaohsiung, Taiwan.

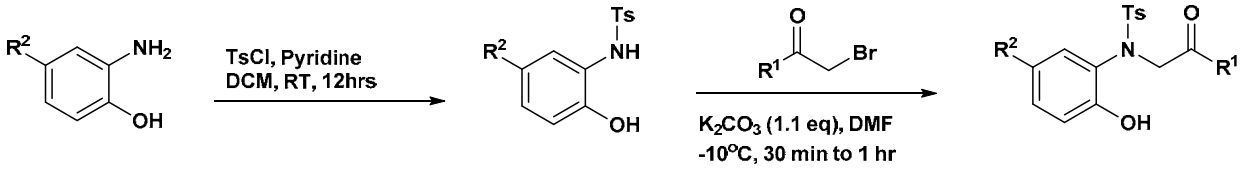
Typical experimental procedure for α- bromination of ketone derivatives: [1]



R = Ph, 4-Me-Ph, 4-OMe, 2-OMe-Ph, 4-CN-Ph,2-F-Ph, 3-F-Ph, 4-F-Ph, 3,4-Cl-Ph, 2-OMe-5-Cl, 2-OMe-5-Br, Thiophene, Furan

To a solution of Acetophenone (1 mmol) in EtOAc/$CHCl_3$ (5/5 ml) was added with copper (II) bromide (2.05 mmol) and refluxed for 8 hr. After reaction completed (monitored by TLC), solvent was evaporated and the resulted residue was partitioned between water and EtOAc. The combined organic layer was washed with sat. $Na_2S_2O_3$ and brine solution, dried and evaporated under vacuo. The obtained crude material was purified by flash column chromatography yielded the pure product (85%, off- white low melting solid).

General experiment procedure for synthesis of starting materials: (1a to 1w)

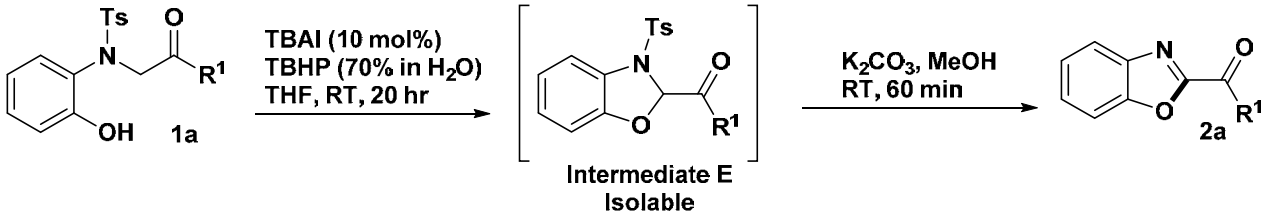


To a solution of 2-amino phenol (2mmol) in Dichloromethane (20 ml) was added with pyridine (3 equiv) followed by tosyl chloride (1.3 eq) at 0°C over a period of 10 min. After addition completes, reaction stirred at room temperature (~28°C) for 12 hours. After reaction completed (monitored by TLC), reaction mass quenched with ice-cold water and extracted into Dichloromethane. The combined organic layer was washed with 1N HCl and brine solution, and dried and evaporated to give crude residue. The obtained crude material passed through flash column chromatography to give pure product as brown solid (88% yield).

The N-tosyl-2-amino phenol (1 mmol) and potassium carbonate (1.1 equiv) in DMF (5 ml) was charged in a RB flask at -10°C (Ice-salt mixture) and stirred at same temperature for 30 min. After phenacyl bromide (1.05 equiv) in DMF (2 ml) was added drop wise over a period of 15 min and the reaction was stirred at same temperature till completion (~30-60 min). After reaction completed (monitored by TLC), reaction mass quenched with ice-cold water and extracted into EtOAc. The combined organic layer was washed with ice-cold water, sat. NH_4Cl and brine solution, and dried and evaporated to give the crude residue. The crude material was triturated with EtOAc/Hexane to afford the pure compound **1a** as a white solid (63% yield).

[***Note for N-Alkylation:*** ***i)****The moderate yield in this alkylation step is due to the less competitive O-Alkylation reaction with phenolic OH.* ***ii)*** *The crystallization (or trituration) technique is preferred method for purification of* ***1a-1u*** *rather than column chromatography, since the compounds are sparingly soluble in EtOAc.]*

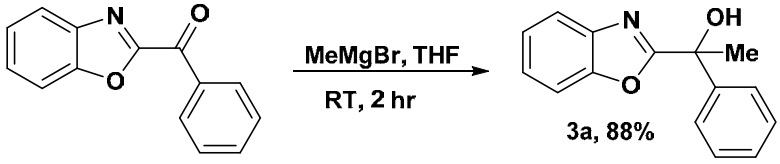
General Experimental procedure for α-keto benzoxazoles (2a-2u):



To a solution of **1a** (380 mg, 1 mmol) in THF (5 ml) was added with TBAI (37 mg, 10 mol %) and TBHP (180 mg, 2equiv, 70% aqueous solution) and stirred at RT for 20 hours. After first step was completed (monitored by TLC), potassium carbonate (138 mg, 1 equiv) and MeOH (2 ml) was added and stirred at RT for further 60 min. After reaction completed, reaction mass evaporated to remove the solvent and the crude was passed through the flash column chromatography to afford the title compound as white solid (210 mg, 94%).

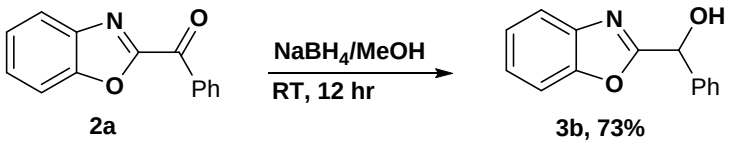
Experimental procedure for Synthetic Applications:

1-(benzo[d]oxazol-2-yl)-1-phenylethan-1-ol (3a):



To ketone **2a** (100mg, 0.44 mmol) in THF (3.0 mL) at 0 °C was added MeMgBr (836 µL, 0.49 mmol, 0.59 M in THF) drop wise and the mixture was stirred for 2 hours. The reaction mixture was quenched with NH_4Cl (4 mL, sat. aq.) and extracted into EtOAc. The combined organic layer was separated and the brine solution and concentrated *in vacuo*. The obtained crude passed through Flash column chromatography on silica gel provided the title compound (94 mg, 88%) as a beige solid..

benzo[d]oxazol-2-yl(phenyl)methanol (3b):

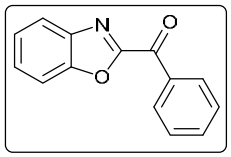


2a **3b, 73%**

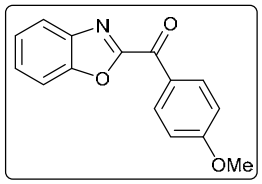
To ketone 2a (100 mg, 0.44 mmol) in MeOH (4 ml) at 0^{o}C was added sodium borohydride (25 mg, 0.66 mmol) over a period of 15 min and stirred at RT for 12 hours. After reaction finished, reaction mass quenched with NH_4CL solution and extracted into EtOAc. The combined organic layer was washed with brine solution and dried, evaporated under *vacuo*. The resulted crude passed through a Flash column chromatography on silica gel provided the title compound (73 mg, 73%) as pale-yellow solid.

Analytical data:

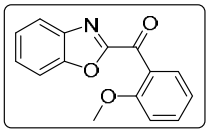
3.1 For products:



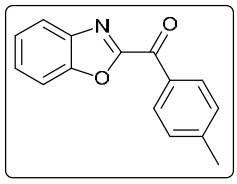
benzo[d]oxazol-2-yl(phenyl)methanone (2a):[2] Yield: 94%; white solid; m. p. 65-68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56-8.54 (2H, m), 7.96 (1H, d), 7.72 (1H, d, *J* = 8.4 Hz), 7.69 (1H, dt, *J* = 7.2, 1.2 Hz), 7.60-7.54 (2H, m), 7.48 (1H, td, *J* = 8.0, 0.8 Hz). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 180.5, 157.1, 150.4, 140.4, 135.7, 134.3, 131.3, 131.3, 128.6, 128.4, 125.7, 122.4, 111.8.



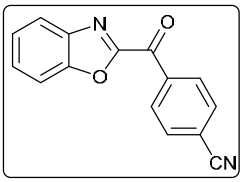
benzo[d]oxazol-2-yl(4-methoxyphenyl)methanone (2b):[2] Yield: 92%; white solid; m. p. 78-80 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (s, *J* = 9.2 Hz, 2H), 7.9-7.92 (m, 1H), 7.71 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.54 (td, *J* = 8.0, 1.Hz, 1H), 7.04 (d, *J* = 7.2 Hz, 2H), 3.92 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 178.7 164.6 157.4, 150.3, 140.7, 133.5, 128.1 127.9, 125.5, 122.5, 113.9 111.9, 55.5.



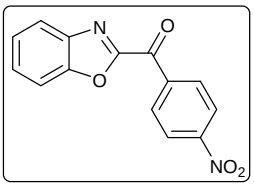
benzo[d]oxazol-2-yl(2-methoxyphenyl)methanone (2c):[3] Yield: 88%; white solid; m. p. 74-76 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (tt, *J* = 8.0, 0.8 Hz, 1H),7.75 (dd, *J* = 7.6 Hz, 1H), 7.68-7.65 (m, 1H), 7.60-7.55 (m, 1H), 7.54-7.50 (m, 1H), 7.46-7.41 (m, 1H), 7.11 (td, *J* = 7.8, 0.8 Hz, 1H), 7.05 (d, *J* = 8.4 Hz, 1H), 3.77 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 159.0, 158.4, 150.5, 134.3, 130.9, 128.0, 125.4, 122.2, 120.6, 112.0, 111.7, 55.9.



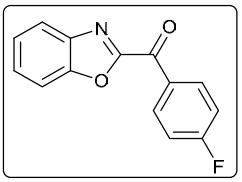
benzo[d]oxazol-2-yl(p-tolyl)methanone (2d): Yield: 91%; white solid; m. p. 80-82°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.0 Hz, 2H), 7.96-7.93 (m, 1H), 7.73-7.70 (m, 1H), 7.55 (td, *J* = 7.2, 1.2 Hz, 1H), 7.47 (td, *J* = 8.4, 1.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 2H), 2.47 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 180.1, 157.2, 150.3, 145.5, 131.1, 129.3, 128.2, 125.6, 122.3, 111.8, 26.6. HRMS-ESI (m/z): calcd for $C_{15}H_{11}NNaO_2$ $[M + Na]^+$: 260.0687, found 260.0685.



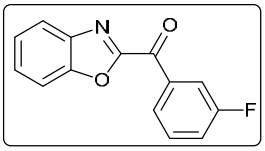
4-(benzo[d]oxazole-2-carbonyl)benzonitrile (2e): Yield: 87%; beige solid; m. p. 132-134°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 (d, *J* = 8.8, 2H), 7.96 (d, *J* = 8.0, 1H), 7.87 (d, *J* = 8.8 Hz, 2H), 7.74 (d, *J* = 8.4, 1H), 7.60 (td, *J* = 7.2, 1.2 Hz, 1H), 7.51 (td, *J* = 8.0, 1.2 Hz, 1H). ^{13}CNMR (CDCl3, 100 MHz): δ 178.93, 156.43, 150.48, 137.98, 137.98, 132.28, 131.37, 129.1, 126.1, 122.58, 117.2, 111.9. HRMS-ESI (m/z): calcd for $C_{15}H_8N_2NaO_2$ $[M + Na]^+$: 271.0483, found 271.0477.



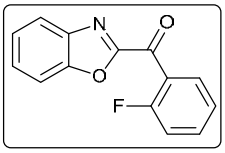
benzo[d]oxazol-2-yl(4-nitrophenyl)methanone (2f):[5] Yield: 90%; pale-yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (d, *J* = 9.2 Hz, 2H), 8.42 (d, *J* = 9.2 Hz, 2H), 7.99-7.97 (m, 1H), 7.76-7.73 (m, 1H), 7.63-7.73 (m, 1H), 7.63-7.59 (m, 1H), 7.54-7.50 (m, 1H). ^{13}CNMR (CDCl3, 100 MHz): δ 178.76, 156.47, 150.55, 140.64, 139.49, 132.13, 129.21, 126.18, 123.63, 122.66, 112.00. HRMS-ESI (m/z): calcd for $C_{14}H_8N_2NaO_4$ $[M + Na]^+$: 291.0382, found 291.03786.



benzo[d]oxazol-2-yl(4-fluorophenyl)methanone (2g): [2] Yield: 90%; white solid; m. p. 108-110°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69-8.64 (2H, m), 7.96-7.94 (m, 1H), 7.73-7.71 (m, 1H), 7.57 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.49 (dt, *J* = 8.4, 1.2 Hz, 1H), 7.28-7.22 (m, 2H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 178.7, 167.8, 165.3, 140.6, 133.96 (J_F = 9.8 Hz), 131.34 (J_F = 3.1 Hz), 128.5, 125.8, 122.3, 115.9 (J_F = 22 Hz), 111.88. HRMS-ESI (m/z): calcd for $C_{14}H_8FNNaO_2$ $[M + Na]^+$: 264.0437, found 264.0433.

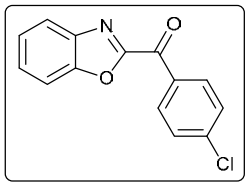


benzo[d]oxazol-2-yl(3-fluorophenyl)methanone (2h): Yield: 86%; off-white solid; m. p. 80-82°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (dt, *J* = 8.0, 1.2 Hz, 1H), 8.33-8.29 (m, 1H), 7.97-7.95 (m, 1H), 7.74-7.17 (m, 1H), 7.60-7.53 (m, 2H), 7.50 (td, *J* = 7.2, 1.2 Hz, 1H), 7.42-7.37 (m, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 179.07 (J_F = 3.0 Hz), 163.7, 156.6, 150.4 140.6, 136.7 (J_F = 6.8 Hz), 130.2 (J_F = 7.5 Hz), 128.7, 126.8 (J_F = 3.0 Hz), 125.8, 122.4 121.3 (J_F = 22 Hz), 117.7 (J_F = 22.7), 111.8. HRMS-ESI (m/z): calcd for $C_{14}H_8FNNaO_2$ $[M + Na]^+$: 264.0437, found 264.0433.

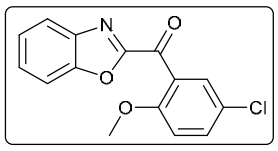


benzo[d]oxazol-2-yl(2-fluorophenyl)methanone(2i): Yield: 92%; beige solid; m. p. 88-90°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (td, *J* = 7.2, 1.6 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.66-7.61 (m, 1H), 7.56 (td, *J* = 8.0, 1.22 Hz, 1H), 7.34 (td, *J* = 7.6, 0.8 Hz, 1H), 7.26-7.21 (m, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 179.7, 162.5, 160.0, 157.2, 150.6, 140.7, 135.0 (J_F = 9.1 Hz), 131.74 (J_F = 1.5 Hz), 128.6,

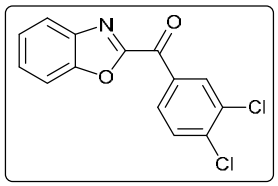
125.6, 125.8, 124.2 (J_F = 3.8 Hz), 116.7 (J_F = 21.1 Hz), 111.8. HRMS-ESI (m/z): calcd for $C_{14}H_8FNNaO_2$ $[M + Na]^+$: 264.0437, found 264.0433.



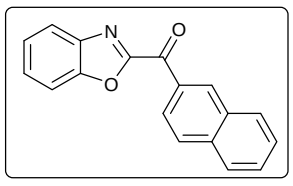
benzo[d]oxazol-2-yl(4-chlorophenyl)methanone (2j):[4] Yield: 86%; beige solid; m. p. 88-90°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58-8.54 (m, 2H), 7.96-7.94 (m, 1H), 7.72 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.59-7.54 (m, 3H), 7.49 (td, *J* = 8.4, 1.2 Hz, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 179.1, 156.8, 150.3, 141.0, 140.6, 133.2, 132.4, 128.9, 128.6, 125.8, 122.4, 111.8. HRMS-ESI (m/z): calcd for $C_{14}H_8ClNNaO_2$ $[M + Na]^+$: 280.0141, found xxx.



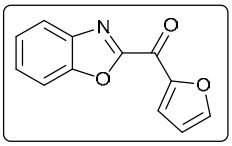
benzo[d]oxazol-2-yl(5-chloro-2-methoxyphenyl)methanone (2k): Yield: 82%; white crystalline solid; m. p. 138-141°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.88 (m, 1H), 7.69-7.66 (m, 2H), 7.56-7.50 (m, 2H), 7.45 (td, *J* = 7.2, 1.2 Hz, 1H), 6.99 (d, *J* = 8.8 Hz, 1H), 3.76 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 181.7, 157.8, 157.4, 150.6, 140.7, 133.7, 130.3, 128.3, 127.4, 125.8, 125.6, 122.3, 113.4, 111.8, 56.3. HRMS-ESI (m/z): calcd for $C_{15}H_{10}ClNNaO_3$ $[M + Na]^+$: 310.0247, found 310.0241.



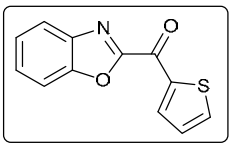
benzo[d]oxazol-2-yl(3,4-dichlorophenyl)methanone (2l): Yield: 92%; white solid; m. p. 112-114°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 2.0 Hz), 8.49 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.97 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 1H), 7.58 (td, *J* = 7.6, 1.2 Hz, 1H), 7.50 (td, *J* = 8.4, 1.2 Hz, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 177.9, 156.4, 150.4, 140.5, 139.1, 134.3, 133.3, 132.8, 130.7, 130.0, 128.9, 125.9, 122.5, 111.9. HRMS-ESI (m/z): calcd for $C_{14}H_7Cl_2NNaO_2$ $[M + Na]^+$: 313.9752, found 313.9346.



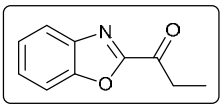
benzo[d]oxazol-2-yl(naphthalen-2-yl)methanone (2m): Yield: 86%; off-white solid; m. p. 110-112°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.32 (s, 1H), 8.44 (dd, *J* = 8.8, 1.6 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 7.98 (t, *J* = 8.0 Hz, 1H), 7.91 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.65 (td, *J* = 6.8, 1.2 Hz, 1H), 7.60-7.54 (m, 2H), 7.49 (dt, *J* = 6.8, 1.2 Hz, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 180.2, 157.2, 150.4, 136.0, 134.2, 132.3, 132.2, 130.2, 129.2, 128.4, 127.7, 126.8, 125.7, 125.3, 122.3, 111.8. HRMS-ESI (m/z): calcd for $C_{18}H_{11}NNaO_2$ $[M + Na]^+$: 296.0687, found 296.0679.



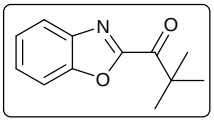
benzo[d]oxazol-2-yl(furan-2-yl)methanone (2n): Yield: 90%; off-white solid; m. p. 116-118°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (dd, *J* = 2.7, 0.8 Hz, 1H), 7.95-7.92 (m, 1H), 7.85-7.84 (m, 1H), 7.72-7.70 (m, 1H), 7.53 (td, *J* = 7.4, 1.2 Hz, 1H), 7.48 (td, *J* = 6.8, 2.4 Hz, 1H), 6.70 (dd, *J* = 1.6 Hz, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 167.1, 156.5, 149.3, 140.6, 128.3, 125.8, 124.9, 122.2, 113.0, 111.8. HRMS-ESI (m/z): calcd for $C_{12}H_7NNaO_3$ $[M+Na]^+$: 236.0324, found 236.0316.



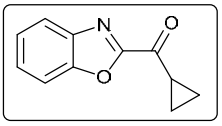
benzo[d]oxazol-2-yl(thiophen-2-yl)methanone (2o): Yield: 82%; white solid; m. p. 105-107°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (dd, *J* = 7.6, 0.8 Hz, 1H), 7.96-7.94 (m, 1H), 7.86 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.55 (td, *J* = 7.2, 1.2 Hz, 1H), 7.48 (td, *J* = 8.4, 1.2 Hz, 1H), 7.29-7.26 (m, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 172.1, 156.8, 150.5, 140.6, 140.5, 137.6, 137.0, 128.7, 128.3, 125.7, 122.2, 111.8. HRMS-ESI (m/z): calcd for $C_{12}H_7NNaO_2S$ $[M]^+$: 252.0095, found 252.00908.



1-(benzo[d]oxazol-2-yl)propan-1-one (2p): Yield: 76%; white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.88 (m, 1H), 7.68-7.65 (m, 1H), 7.55-7.51 (m, 1H), 7.48-7.44 (m, 1H), 3.25 (q, *J* = 7.6 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 128.4, 125.7, 122.2, 111.9, 32.9, 29.6. HRMS-ESI (m/z): calcd for $C_{10}H_9NNaO_2$ $[M]^+$: 198.0531, found 198.0523.

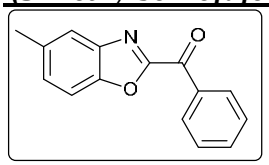


1-(benzo[d]oxazol-2-yl)-2,2-dimethylpropan-1-one (2q): Yield: 92%; white solid; m. p. 78-80°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (m, 1H), 7.65 (m, 1H), 7.53-7.49 (m, 1H), 7.44 (m, 1H), 1.52 (s, 9H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 195.1, 155.6, 149.8, 140.5, 128.1, 125.4, 122.2, 111.7, 44.3, 26.8. HRMS-ESI (m/z): calcd for $C_{12}H_{13}NNaO_2$ $[M + Na]^+$: 226.0844, found 226.0831.

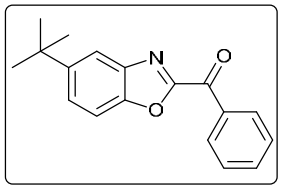


benzo[d]oxazol-2-yl(cyclopropyl)methanone (2r): Yield: 89%; white solid; m. p.86-88°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.90 (m, 1H), 7.66 (dt, *J* = 8.0, 0.8 Hz, 1H), 7.53 (td, *J* = 7.6, 1.6 Hz, 1H), 7.47 (td, *J* = 7.2, 1.2 Hz, 1H), 3.29-.23 (m. 1H), 1.43-1.32 (m, 1H), 1.28-1.22 (m, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 189.8, 157.7, 150.7, 140.6, 128.3, 125.6, 122.1, 111.9, 18.6, 13.5. HRMS-ESI (m/z): calcd for $C_{11}H_9NNaO_2$ $[M + Na]^+$: 210.0531, found 210.0524.

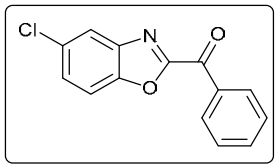
(5-methylbenzo[d]oxazol-2-yl)(phenyl)methanone (2s): Yield: 94%; white solid; m. p. 91-93 °C;



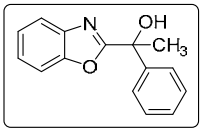
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55-8.52 (m, 2H), 7.72-7.71 (m, 1H), 7.74 (tt, *J* = 5.1, 1.2 Hz, 1H), 7.59-7.54 (m, 3H), 7.37-7.35 (m, 1H), 2.52 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 180.6, 157.2, 148.7, 140.9, 135.7, 134.2, 130.9, 129.8, 128.5, 121.9, 111.1, 21.5. HRMS-ESI (m/z): calcd for $C_{15}H_11NNaO_2$ $[M + Na]^+$: 260.0687, found 260.0683.



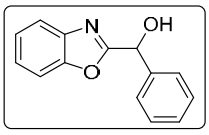
(5-(tert-butyl)benzo[d]oxazol-2-yl)(phenyl)methanone (2t): Yield: 96%; white crystalline solid; m. p. 70-72°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57-8.54 (m, 2H), 7.95 (t, *J* = 1.2 Hz, 1H), 7.68 (1H, tt, *J* = 6.8, 1.2 Hz), 7.63-7.62 (m, 2H), 7.58-7.54 (m, 2H),1.41 (s, 9H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 180.5, 157.3, 149.4, 148.4, 140.7, 134.1, 130.9, 128.5, 126.5, 118.4, 110.0, 31.6. HRMS-ESI (m/z): calcd for $C_{18}H_{17}NNaO_2$ $[M + Na]^+$: 302.1157, found 302.1154.



(5-chlorobenzo[d]oxazol-2-yl)(phenyl)methanone (2u)*:* Yield: 86%; beige solid; m. p. 98-100°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54-8.52 (2H, m), 7.94 (d, 1H, *J* = 2 Hz), 7.71 (1H, td, *J* = 6.8, 1.2 Hz),7.65 (1H, d, *J* = 8.8 Hz), 7.60-7.56 (m, 2H), 7.53 (1H, dd, *J* = 2.4 Hz). ^{13}CNMR (CDCl3, 100 MHz): δ 180.17, 158.08, 148.97, 141.75, 134.76, 134.55, 131.32, 131.02, 128.90, 128.70, 122.12, 112.71. HRMS-ESI (m/z): calcd for $C_{14}H_8ClNNaO_2$ $[M + Na]^+$: 280.0141, found 280.0137

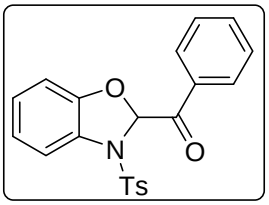


1-(benzo[d]oxazol-2-yl)-1-phenylethan-1-ol (3a):[4] Yield: 88%; beige solid; m. p. 66-68°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.70 (m, 1H), 7.54 (d, *J* = 7.2 Hz, 2H), 7.49-7.47 (m, 1H), 7.37-7.26 (m, 5H), 2.08 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 169.2, 151.1, 143.7, 140.4, 128.4, 127.9, 125.2, 124.9, 124.5, 120.2, 110.8, 73.6, 28.6.

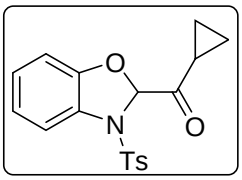


benzo[d]oxazol-2-yl(phenyl)methanol (3b):[4] Yield: 73%; pale-yellow solid; m. p. 106-108°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67-7.63 (m, 1H), 7.54-7.51(m, 2H), 7.47-7.43 (m, 1H), 7.39-7.33 (m, 3H), 7.31-7.29 (dd, *J* = 3.2 Hz, 2H), 6.04 (d, *J* = 4.8 Hz, 1H), 4.40 (d, *J* = 5.6 Hz, 1H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 166.6, 150.9, 140.2, 138.8, 128.77, 128.74, 126.8, 125.2, 124.5, 120.0, 110.8, 70.5.

3.2 Isolated Intermediates:



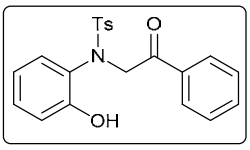
phenyl(3-tosyl-2,3-dihydrobenzo[d]oxazol-2-yl)methanone(1a'): Yield: 96%; white solid; m. p. 140-142°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21-8.19 (m, 2H), 7.63 (tt, J = 6.8 Hz, 1H),7.54-7.48 (m, 5H), 7.17 (d, J = 8.0 Hz, 2H), 7.06 (td, J = 8.0, 1.2 Hz, 1H), 7.00 (s, 1H), 6.95 (td, J = 7.6, 1.2 Hz, 1H), 6.73 (dd, J = 8.0, 1.6 Hz, 1H), 2.36 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 188.4,, 152.3, 145.1, 134.2, 132.9, 132.5, 129.6, 129.5, 128.5, 127.8, 127.6, 122.1, 119.0, 109.8, 91.4, 21.6.



cyclopropyl(3-tosyl-2,3-dihydrobenzo[d]oxazol-2-yl)methanone(1r'): Yield: 92%; white solid; m. p. 102-104 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, J = 7.6, 1.2 Hz, 1H), 7.46 (d, J = 8.4 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 7.06 (td, J = 8.0, 1.6 Hz, 1H), 6.97 (td, J = 7.6, 0.8 Hz, 1H), 6.75 (dd, J = 7.6, 0.8 Hz, 1H), 6.17 (s, 1H), 2.54-2.48 (m, 1H), 2.36 (s, 3H), 1.55-1.04 (m, 4H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 201.3, 151.9, 145.2, 132.1, 129.7, 128.0, 127.6, 127.5, 122.1, 118.5, 109.9, 93.9, 21.5, 16.1, 13.2, 12.4.

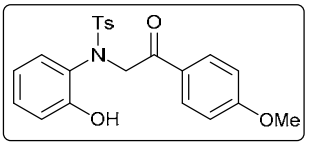
3.3 For Starting Materials:

N-(2-hydroxyphenyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1a) Yield: 63%;



Off-white solid; m. p. 148-150°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (s, 1H), 7.96 (d, *J* = 7.2 Hz, 2H), 7.62-7.60 (m, 3H), 7.51-7.47 (m, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.23-7.18 (m, 1H), 6.94 (dd, *J* = 8.0, 1.2 Hz, 2H), 6.88 (dd, *J* = 7.8, 2.4 Hz, 1H), 6.77-6.73 (m, 1H), 2.44 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 196.5, 144.2, 135.5, 134.5, 133.8 130.9, 129.9, 129.5, 128.9, 128.3, 128.0, 126.1, 120.1, 117.7, 58.4, 21.6. HRMS-ESI (m/z): calcd for $C_{21}H_{19}NO_4S$ $[M + Na]^+$: 404.0938, found 404.0927.

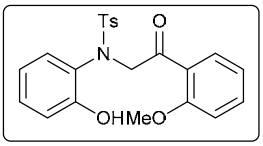
N-(2-hydroxyphenyl)-N-(2-(4-methoxyphenyl)-2-oxoethyl)-4-methylbenzenesulfonamide(1b):



Yield: 62%; white solid; m. p. 150-158°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.94 (s, 1H), 7.94 (d, *J* = 8.8 Hz, 2H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 7.6 Hz, 2H), 7.22-7.17 (m, 1H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.19-6.89 (m, 2H), 6.77-6.72 (m, 1H), 4.8-5.4 (bs, 2H), 3.88 (s, 3H), 2.43 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 194.9, 164.6, 156.0, 144.0, 135.7, 130.8, 130.7, 130.1, 129.4, 128.0, 126.7, 126.2,

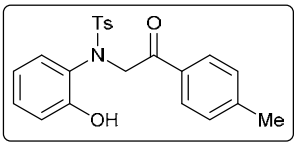
120.0, 117.7, 114.1, 58.0, 55.5, 21.6. HRMS-ESI (m/z): calcd for $C_{22}H_{21}NNaO_5S$ $[M + Na]^+$: 434.1038, found 434.1033.

N-(2-hydroxyphenyl)-N-(2-(2-methoxyphenyl)-2-oxoethyl)-4-methylbenzenesulfonamide(1c):



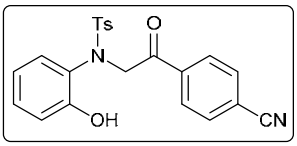
Yield: 66%; White solid; m. p. 118-120°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (s, 1H), 7.97 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.62-7.59 (m, 2H), 7.54 (dddd, *J* = 7.2, 1.6 Hz, 1H), 7.27-7.25 (m, 2H), 7.22-7.17 (m, 1H), 7.06-7.02 (m, 1H), 7.06-7.02 (m, 1H), 6.98 (bs, *J* = 8.4 Hz, 1H), 6.94-6.91 (m, 1H), 6.75 (m, 1H), 4.4-5.4 (bs, 2H), 3.93 (s, 3H), 2.43 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 198.1, 159.7, 156.0, 143.9, 135.9, 135.6, 131.6, 130.6, 129.4, 128.0, 126.5, 124.0, 121.1, 119.9, 117.5, 111.5, 62.7, 55.6, 21.6. HRMS-ESI (m/z): calcd for $C_{22}H_{21}NNaO_5S$ $[M + Na]^+$: 434.1038, found 434.1033.

N-(2-hydroxyphenyl)-4-methyl-N-(2-oxo-2-(p-tolyl)ethyl)benzenesulfonamide(1d): Yield: 71%;



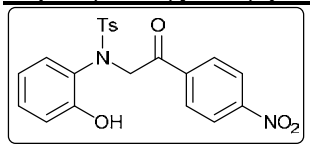
white solid; 150-152°C; ^{1}H NMR (400 MHz,$CDCl_3$) δ 8.81 (s, 1H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.29-7.26 (m, 4H), 7.22-7.18 (m, 1H), 6.93 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.88 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.77-6.73 (m, 1H), 5.6-4.6 (bs, 2H), 2.44 (s, 3H), 2.42 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 196.1, 155.9, 145.7, 131.2, 130.8, 130.0, 129.6, 129.5, 128.4, 128.0, 126.1, 120.1, 117.7, 58.2, 21.8, 21.6. HRMS-ESI (m/z): calcd for $C_{22}H_{21}NNaO_4S$ $[M + Na]^+$: 418.1089, found 418.1081.

N-(2-(4-cyanophenyl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (1e):[5]



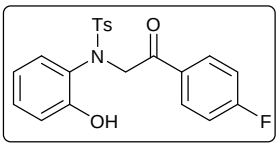
Yield: ~60%; brown solid; The crude has taken to next step without purification. HRMS-ESI (m/z): calcd for $C_{22}H_{21}NNaO_4S$ $[M + Na]^+$: 449.0885, found 449.0877.

N-(2-hydroxyphenyl)-4-methyl-N-(2-(4-nitrophenyl)-2-oxoethyl)benzenesulfonamide (1f):[5]



Yield: ~60%; orange-yellow solid; The crude has taken to next step without purification. HRMS-ESI (m/z): calcd for $C_{22}H_{21}NNaO_4S$ $[M + Na]^+$: 449.0783, found 449.0776.

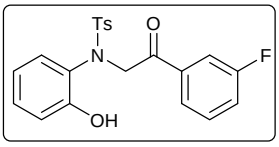
N-(2-(4-fluorophenyl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (1g):[6]



Yield: 51%; beige solid; m. p. 144-146°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 8.02-7.98 (m, 2H), 7.61-7.58 (m, 2H), 7.28-7.26 (m, 2H), 7.22-7.14 (m, 3H), 6.93 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.85 (dd, *J* = 8.0, 1.6 Hz,

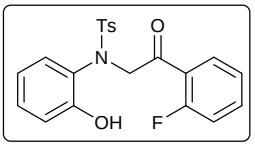
1H), 6.75 (td, J = 8.0, 1.6 Hz), 4.6-5.2 (bs, 2H), 2.44 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 195.0, 167.8 165.2 155.8, 144.2, 135.4, 131.1 (d, J_F = 9.1 Hz), 130.9, 130.3 (d, J_F = 2.0 Hz), 129.5, 128.5, 126.0, 120.1, 117.7, 116.2 (d, J_F = 22 Hz), 58.2, 21.6. HRMS-ESI (m/z): calcd for $C_{21}H_{18}FNNaO_4S$ $[M + Na]^+$: 422.0838, found 422.0832.

N-(2-(3-fluorophenyl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (1h):



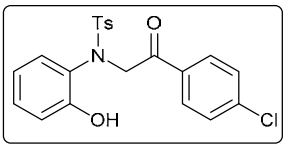
Yield: 58%; beige solid; m. p. 136-138ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (s, 1H), 7.74 (d, J = 7.6 Hz, 1H), 7.68-7.63 (m, 1H), 7.59 (d, J = 8.0 Hz, 2H), 7.51-7.46 (m, 1H), 7.36-7.31 (m, 1H), 7.28 (d, J = 8.0 Hz, 2H), 7.23-7.19 (m, 1H), 6.94 (dd, J = 8.4, 1.2 Hz, 1H), 6.83 (dd, J = 8.0, 1.6 Hz, 1H), 6.76-6.72 (m, 1H), 5.4-4.8 (bs, 2H), 2.44 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 195.4, 164.0, 155.7, 144.3, 135.2, 130.9, 130.7 (J_F = 8.3 Hz), 129.8, 129.5, 128.0, 124.0 (J_F = 3 Hz), 121.6 (J_F = 21.2 Hz), 120.2, 117.7, 115.1 (J_F = 22.7), 58.5, 21.6. HRMS-ESI (m/z): calcd for $C_{21}H_{18}FNNaO_4S$ $[M + Na]^+$: 422.0838, found 422.0832.

N-(2-(2-fluorophenyl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (1i):

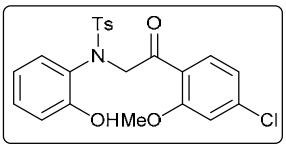


Yield: 60%; brown solid; m. p. 133-135ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (s, 1H), 8.04 (td, J = 7.6, 1.6 Hz, 1H), 7.63-7.58 (m, 3H), 7.30-7.23 (m, 3H), 7.22-7.14 (m, 2H), 6.97 (dd, J = 8.0, 1.2 Hz, 1H), 6.77-6.70 (m, 2H), 4.6-5.2 (bs, 2H), 2.44 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 195.0 (d, J_F = 61 Hz), 163.8,161.3, 155.9, 144.1, 136.(d, J_F = 9.1Hz), 136.2, 135.5, 131.3 (d, J_F = 2.3 Hz), 130.8, 129.6, 129.5, 127.9, 126.2, 125.0 (d, J_F = 3.1 Hz), 122.1 (d, J_F = 13.7 Hz), 120.1, 117.6, 116.8 (d, J_F = 23.4), 62.2 (d, J_F = 12.8 Hz), 21.6. HRMS-ESI (m/z): calcd for $C_{21}H_{18}FNNaO_4S$ $[M + Na]^+$: 422.0838, found 422.0832.

N-(2-(4-chlorophenyl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide(1j):

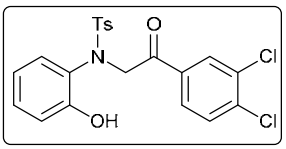


Yield: 67%; beige solid; m. p. 180-182ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 8.4 Hz, 2H), 7.67-7.64 (m, 1H), 7.60 (d, J = 8.4 Hz, 2H), 7.44 (dd, J = 7.6, 2.0 Hz, 1H), 7.40-7.29 (m, 4H), 5.22 (s, 3H), 2.25 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ192.6, 143.5, 139.98, 139.94, 136.81, 133.3, 133.1, 131.4, 129.5, 129.4, 129.0, 128.7, 128.4, 128.2, 127.6, 122.8, 122.5, 94.5, 85.8, 56.2, 21.4. HRMS-ESI (m/z): calcd for $C_{21}H_{18}ClNNaO_4S$ $[M + Na]^+$: 438.0543, found 438.0536.



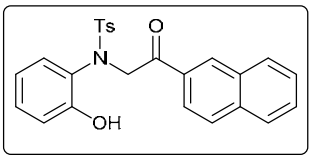
N-(2-(5-chloro-2-methoxyphenyl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (1k): [6] Yield: 63%; beige solid; m. p. 146-148°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (s, 1H), 7.92 (d, *J* = 2.4 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.49 (dd, *J* = 8.8, 2.8 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.22-7.18 (m, 1H), 6.95-7.18 (m, 3H), 6.77-6.73 (m, 1H), 4.8-5.4 (bs, 2H), 3.93 (s, 3H), 2.44 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ ^{13}CNMR ($CDCl_3$, 100 MHz): δ 197.1, 158.2, 155.8, 144.0 135.6, 135.1, 131.1 131.0, 130.7, 130.1, 129.4, 128.0, 126.6, 126.3, 125.1, 120.0, 117.5, 113.1, 62.6, 56.1, 21.6. HRMS-ESI (m/z): calcd for $C_{22}H_{20}ClNNaO_5S$ 468.0468, found 468.0462.

N-(2-(3,4-dichlorophenyl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide



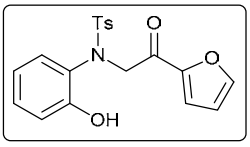
(1l): [6] Yield: 63%; beige solid; m. p. 189-191°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 8.04 (d, J = 2.0 Hz, 1H), 7.79 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.29-7.27 (m, 2H), 7.23-7.19 (m, 1H), 6.94 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.83 (dd, *J* = 8.0, 1.6 Hz), 6.77-6.72 (m, 1H) 2.44 (s, 3H).HRMS-ESI (m/z): calcd for $C_{21}H_{17}Cl_2NO_4S$ $[M + Na]^+$: 472.0153, found 472.01448.

N-(2-hydroxyphenyl)-4-methyl-N-(2-(naphthalen-2-yl)-2-oxoethyl)benzenesulfonamide (1m):



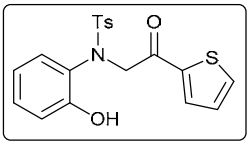
Yield: 66%; white solid; m. p. 158-160°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (s, 1H), 8.47 (s, 1H), 8.00-7.86 (m, 4H), 7.65-7.62 (m, 2H), 7.59-7.55 (m, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.23-7.19 (m, 1H), 6.94 (td, *J* = 8.0, 1.6 Hz, 2H), 6.76 (td, *J* = 7.6, 1.6 Hz, 1H), 5.0-5.4 (bs, 2H), 2.44 (s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 196.5, 155.9, 144.2, 131.2, 130.9, 130.4, 130.0, 129.6, 129.5, 129.2, 128.9, 128.0, 127.9, 127.2, 126.2, 123.4 120.1, 117.7, 58.4, 21.6. HRMS-ESI (m/z): calcd for $C_{25}H_{21}NNaO_4S$ $[M + Na]^+$: 454.1089, found 454.1081.

N-(2-(furan-2-yl)-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (1n): Yield:

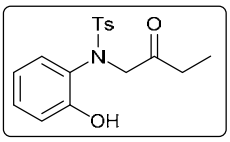


52%; brown solid; m. p. 156-158°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (s, 1H), 7.64-7.59 (m, 3H), 7.36 (dd, *J* = 3.6, 0.8 Hz, 1H), 7.28-7.26 (m, 2H), 7.23-7.18 (m, 1H), 6.93 (dd, *J* = 1.2 Hz, 1H), 6.86 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.76-6.72 (m, 1H), 6.61 (dd, *J* = 3.6, 1.2 Hz, 1H), 4.6-5.2 (2H, bs), 2.43 (3H, s). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 185.5, 155.8, 147.5 144.2, 135.5, 130.9, 129.9, 129.5, 126.1, 120.1, 119.2, 117.7, 112.9, 57.8, 21.6. HRMS-ESI (m/z): calcd for $C_{19}H_{17}NO_5S$ $[M + Na]^+$: 394.0497, found 394.0718.

N-(2-hydroxyphenyl)-4-methyl-N-(2-oxo-2-(thiophen-3-yl)ethyl)benzenesulfonamide (1o):



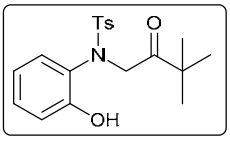
Yield: 56%; white solid; m. p. 166-168^{o}C; ^{1}H NMR (400 MHz, DMSO-d6) δ 9.62 (s, 1H), 8.08-8.04 (m, 2H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.35 d, *J* = 8.0 Hz, 2H), 7.27-7.24 (dd, *J* = 8.8, 3.6 Hz), 7.20 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.13-7.09 (m, 1H), 6.78-6.71 (m, 2H), 5.09 (s, 2H), 2.38 (s, 3H). ^{13}CNMR (DMSO-d6, 100 MHz): δ 189.1, 154.3, 143.1, 140.7, 137.0, 135.6, 133.9, 132.6, 129.6, 129.4, 128.9, 127.3, 125.3, 118.7, 116.4, 55.8, 21.0. HRMS-ESI (m/z): calcd for $C_{19}H_{17}NNaO_4S_2$ $[M + Na]^+$: 410.0497, found 410.0489.



N-(2-hydroxyphenyl)-4-methyl-N-(2-oxobutyl)benzenesulfonamide (1p):

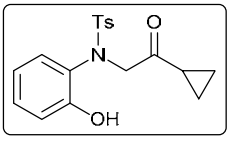
Yield: 46%; Off-white solid; m. p. 108-110^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (bs, 1H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.23-7.19 (m, 1H), 6.97-6.94 (m, 1H), 6.73-6.72 (m, 2H), 4.27 (q, *J* = 7.6 Hz, 2H), 3.8-4.4 (bs, 2H), 2.43 (s, 2H), 1.30 (t, *J* = 7.2 Hz, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 171.8, 155.5, 144.2, 135.3, 130.9, 129.5, 129.4, 127.9, 126.0, 120.2, 117.6, 62.6, 53.4, 21.6, 13.9. HRMS-ESI (m/z): calcd for $C_{17}H_{19}NNaO_4S$ $[M + Na]^+$: 356.0932, found 356.0925.

N-(3,3-dimethyl-2-oxobutyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide(1q): Yield:



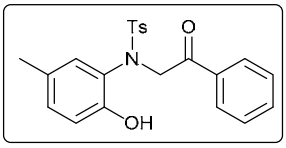
54%; white solid; m. p. 170-172^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H), 7.55 (d, *J* = 8.4 Hz, 2H), 7.26-7.24 (m, 3H), 6.91 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.86 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.76-6.71 (m, 1H), 2.42 (s, 3H), 1.21 (s, 9H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 213.6 155.8, 144.8, 135.1, 135.5, 130.8, 130.0, 129.4, 127.9, 126.1, 120.0, 117.5, 56.9, 43.2, 26.2, 21.6. HRMS-ESI (m/z): calcd for $C_{19}H_{23}NNaO_4S$ $[M + Na]^+$: 384.1245, found 384.1239.

N-(2-cyclopropyl-2-oxoethyl)-N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide (1r): [6] Yield:



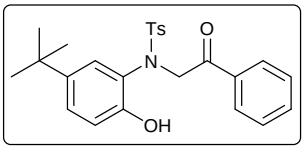
49%; white solid; m. p. 138-140^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (s, 1H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 7.6 Hz, 2H), 7.22-7.17 (m, 1H), 6.91 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.80 (dd, *J* = 8.0, 2.4 Hz. 1H), 6.74 (m, 1H), 4.2-4.6 (bs, 2H), 2.42 (s, 3H), 1.95-1.89 (m, 1H), 1.29-1.19 (m, 2H), 1.06-1.04 (m, 2H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 208.6155.7, 144.1, 135.5, 130.8, 129.9, 129.4, 127.9, 126.01, 126.06, 117.7, 61.2, 21.6, 18.1, 12.6. HRMS-ESI (m/z): calcd for $C_{18}H_{19}NNaO_4S$ $[M + Na]^+$: 368.0932, found 368.0924.

N-(2-hydroxy-5-methylphenyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide(1s):



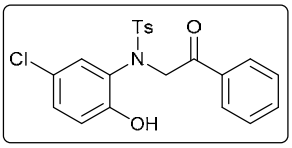
Yield: 64%; White solid; m. p. 156-158ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.97-7.94 (m, 2H), 7.65-7.60 (m, 3H), 7.51-7.46 (m, 2H), 7.29-7.27 (m, 2H), 7.01 dd, *J* = 8.4, 2.4 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.70 (d, *J* = 1.6 Hz, 1H), 4.6-5.4 (bs, 2H), 2.44 (s, 3H), 2.15 s, 3H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 195.5, 153.3, 144.1, 135.6, 134.5, 133.9, 131.5, 130.1, 129.5, 129.4, 128.9, 128.3, 128.0, 125.7, 117.3, 58.3, 21.6, 20.2. HRMS-ESI (m/z): calcd for $C_{22}H_{21}NNaO_4S$ $[M + Na]^+$: 418.1089, found 418.1081.

N-(5-(tert-butyl)-2-hydroxyphenyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1t):



Yield: 64%; white solid; m. p. 185-187ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (s, 1H), 7.97 (d, *J* = 7.2 Hz, 2H), 7.64-7.59 (m, 3H), 7.51-7.47 (m, 2H), 7.29-7.27 (m, 2H), 7.22 (dd, *J* = 8.8, 2.4 Hz), 6.91 (d, *J* = 8.4 Hz, 1H), 9.61 (d, *J* = 2.4 Hz, 1H), 2.44 (s, 3H), 1.11 (s, 9H). ^{13}CNMR ($CDCl_3$, 100 MHz): δ 196.3, 153.5, 144.0, 143.0 135.7, 134.4, 133.9, 129.5, 128.9, 128.3, 128.0, 127.8 126.2, 125.3, 117.1, 58.7, 33.8 31.2, 21.5. HRMS-ESI (m/z): calcd for $C_{25}H_{27}NO_4S$ $[M + Na]^+$: 460.1158, found 460.1553.

N-(5-chloro-2-hydroxyphenyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1u):[5]



Yield: 41%; pale-yellow solid; Without purification crude was taken to next step. HRMS-ESI (m/z): calcd for $C_{25}H_{27}NO_4S$ $[M + Na]^+$: 438.0543, found 438.0535

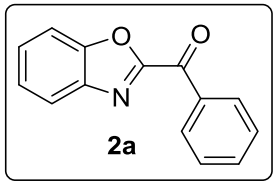
References:

1. L. Carroll King, K. Ostrum, *J. Org. Chem.*, 1964, **29**, 3459.
2. P. Anbarasan, M. Beller, H. Neumann, X. –F. Wu, *Angew. Chem. Int. Ed.*, 2010, **49**, 7316
3. P. Lassalas, F. Marsais, C. Hoarau, *Synlett*, 2013, **24**, 2223;
4. N. Erdmann, M. J. Guant, A. McNally, Q. Y. Toh, S. Vera. *J. Am. Chem. Soc.*, 2013, **135**, 3772;
5. HRMS has been attached.
6. Compounds containing small amount of impurities.

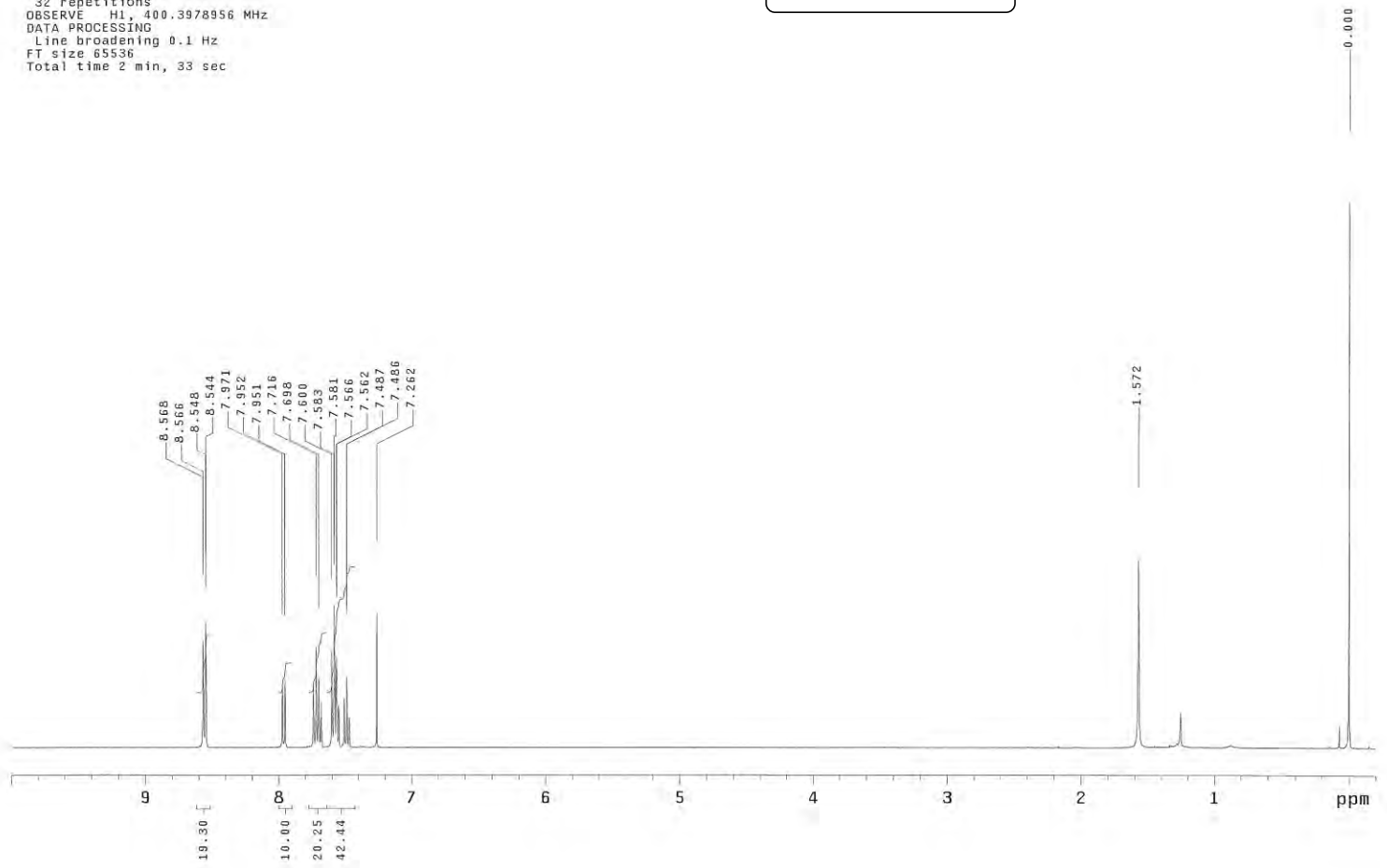
SIVA-RP3-30

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

 Pulse 48.1 degrees
 Acq. time 4.002 sec
 Width 5995.2 Hz
 32 repetitions
OBSERVE H1, 400.3978956 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 65536
Total time 2 min, 33 sec



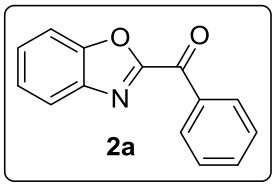
2a



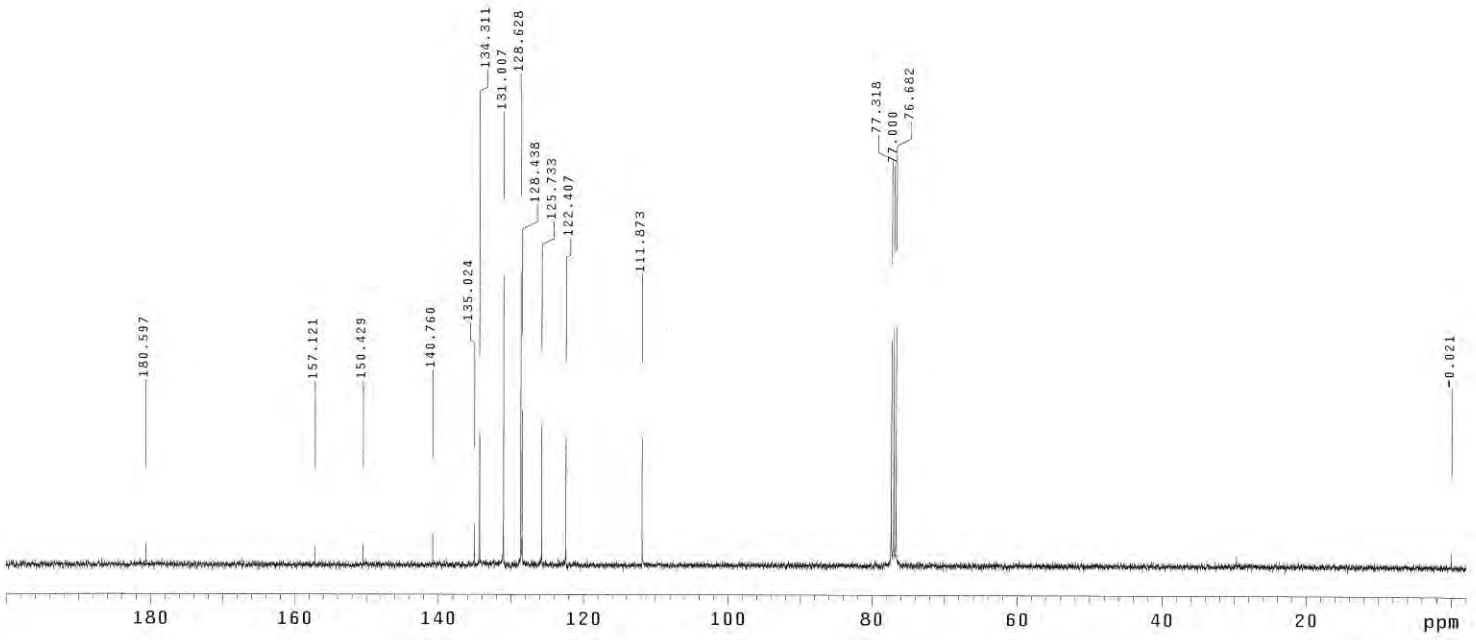
SIVA-RP3-30

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 4416 repetitions
OBSERVE C13, 100.6801315 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



2a

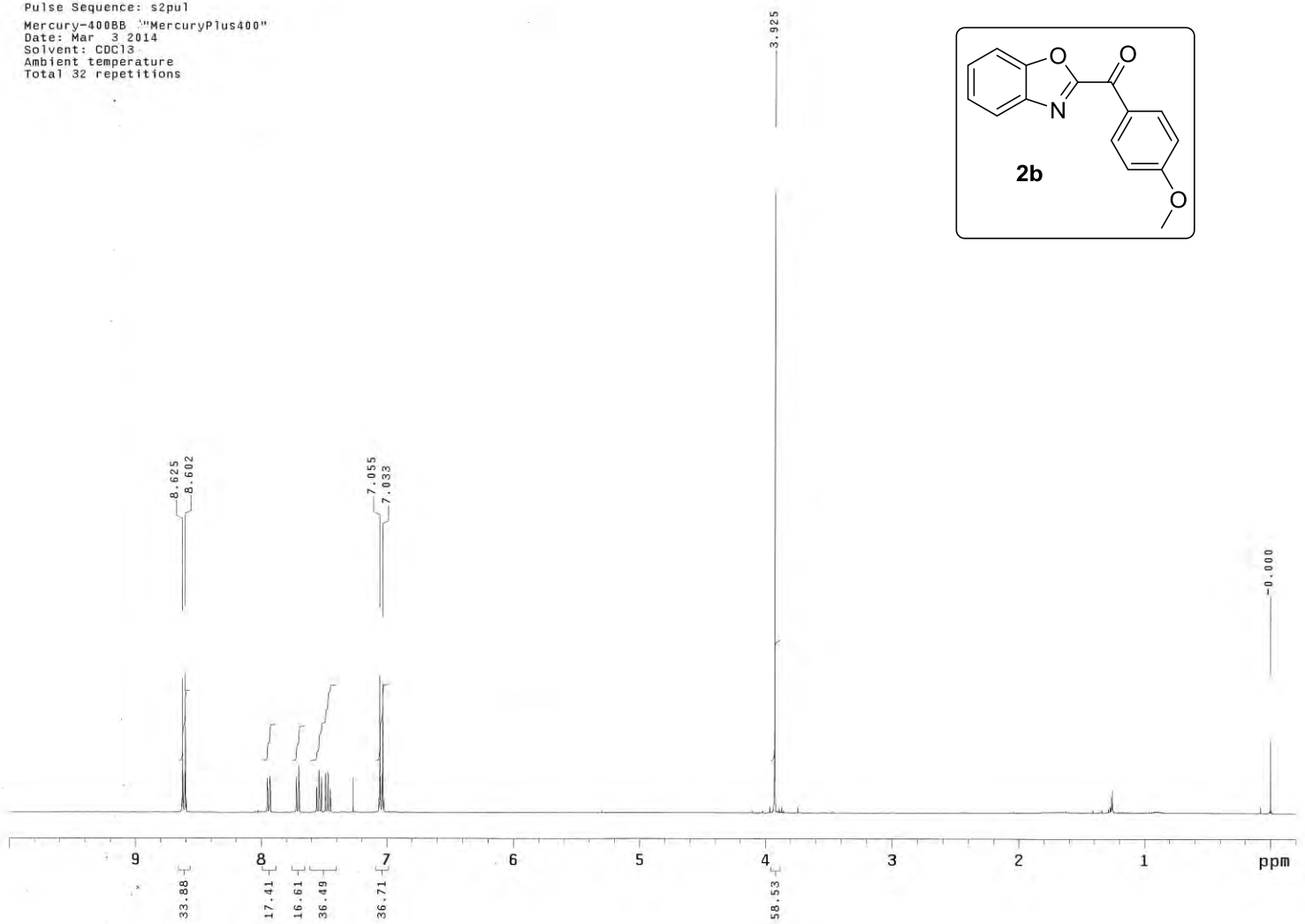


SIVA-RP4-188

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

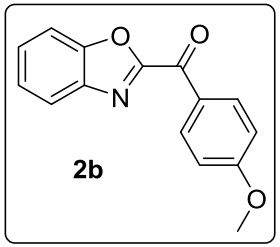
2b



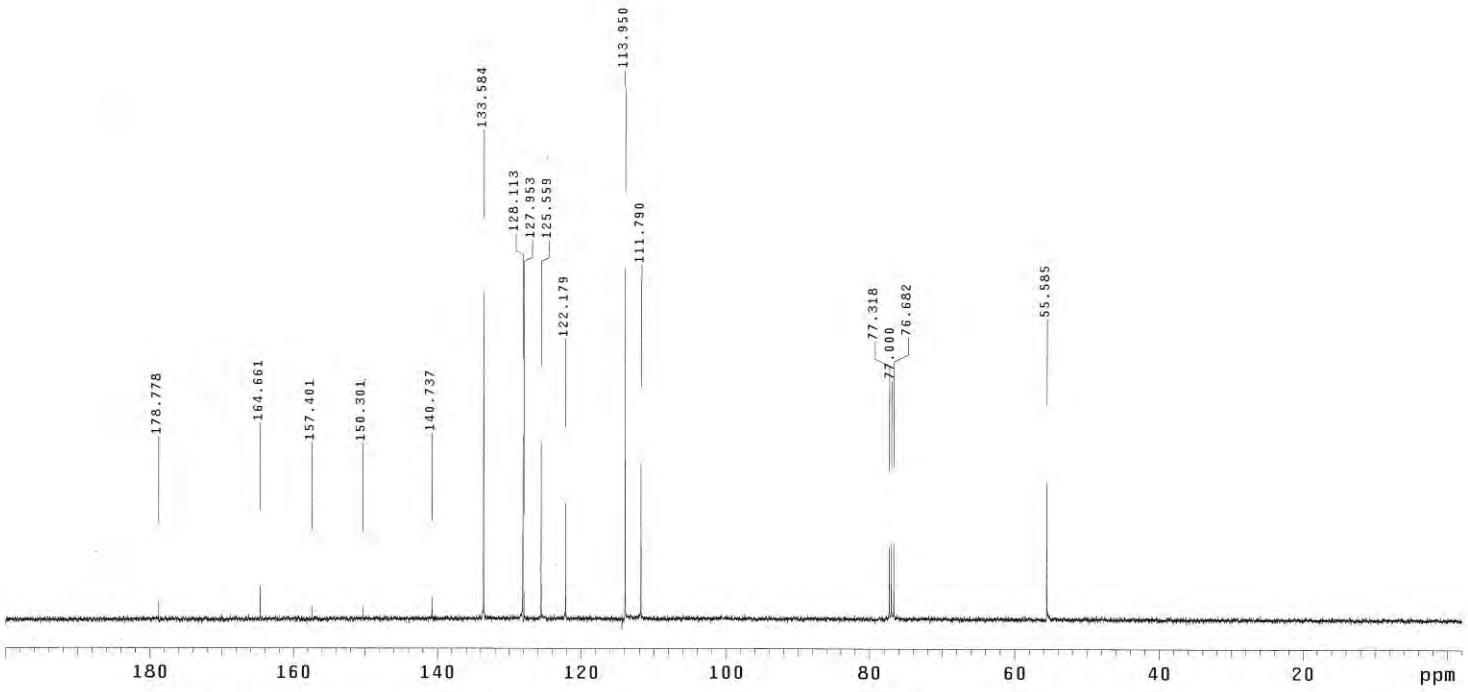
SIVA-RP4-188

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 2144 repetitions



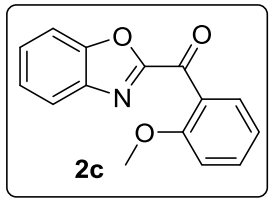
2b



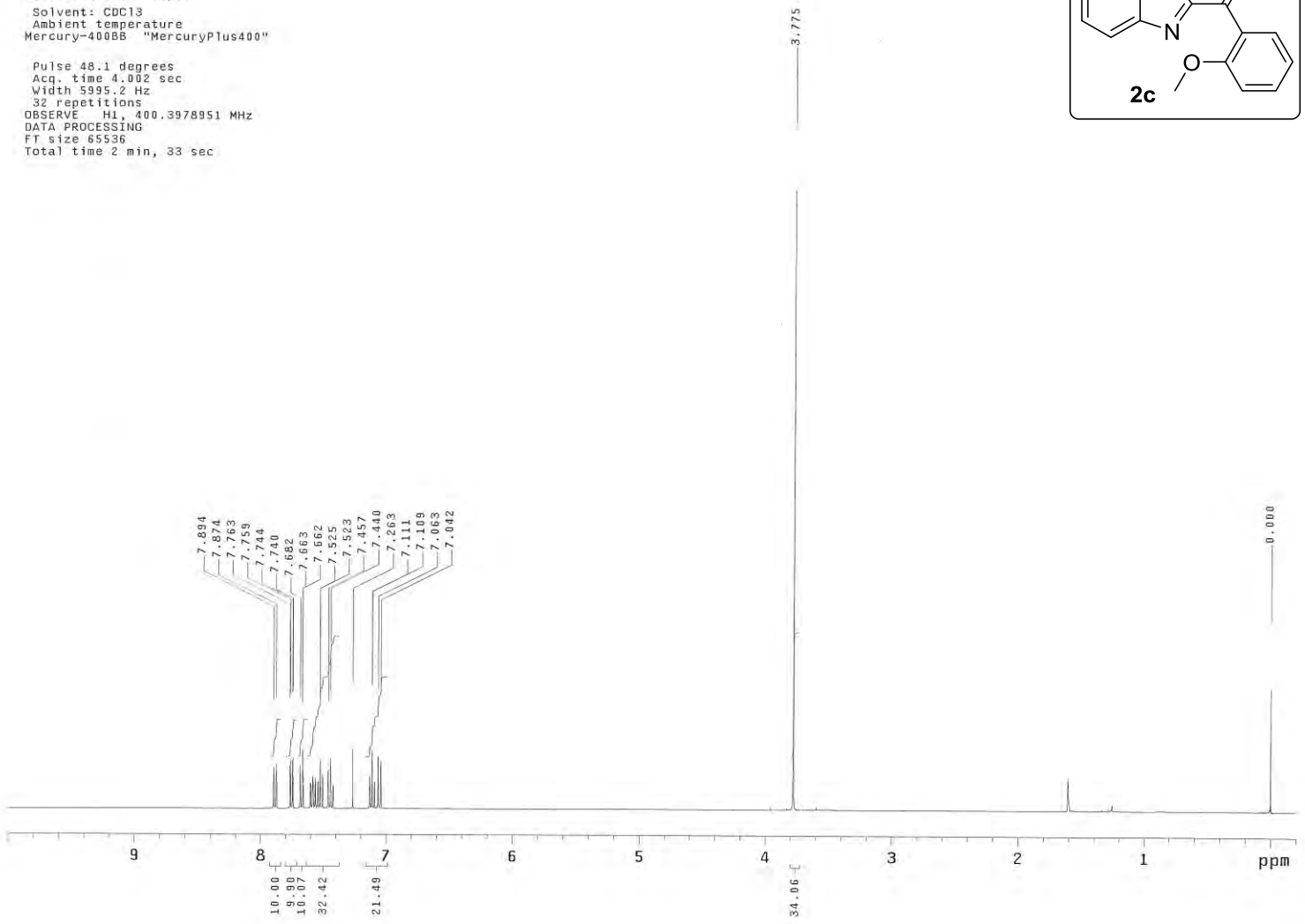
SIVA-RP3-116

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978951 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec



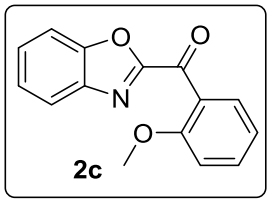
2c

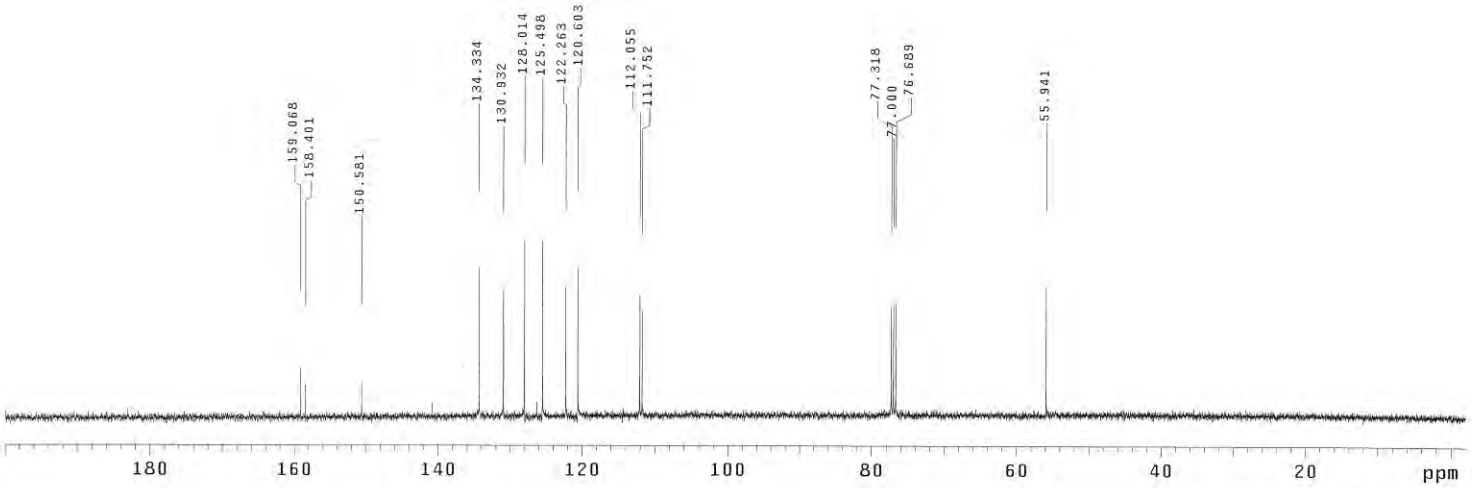


SIVA-RP3-116

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 832 repetitions
OBSERVE C13, 100.6801323 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 4 hr, 22 min, 18 sec

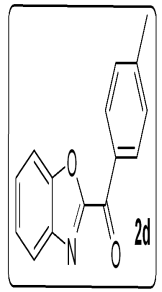


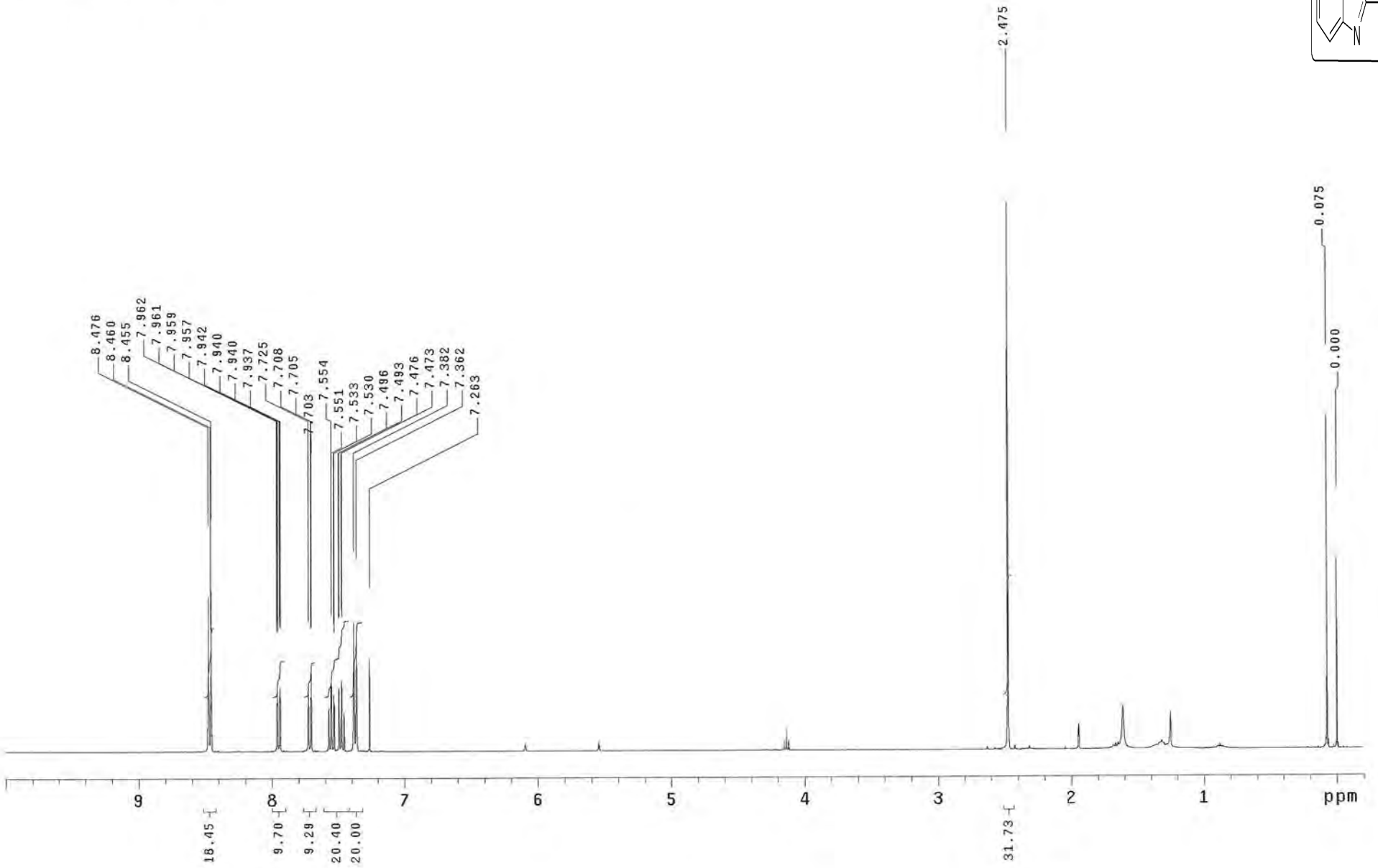


SIVA-RP3-20

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 28 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

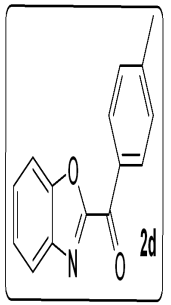


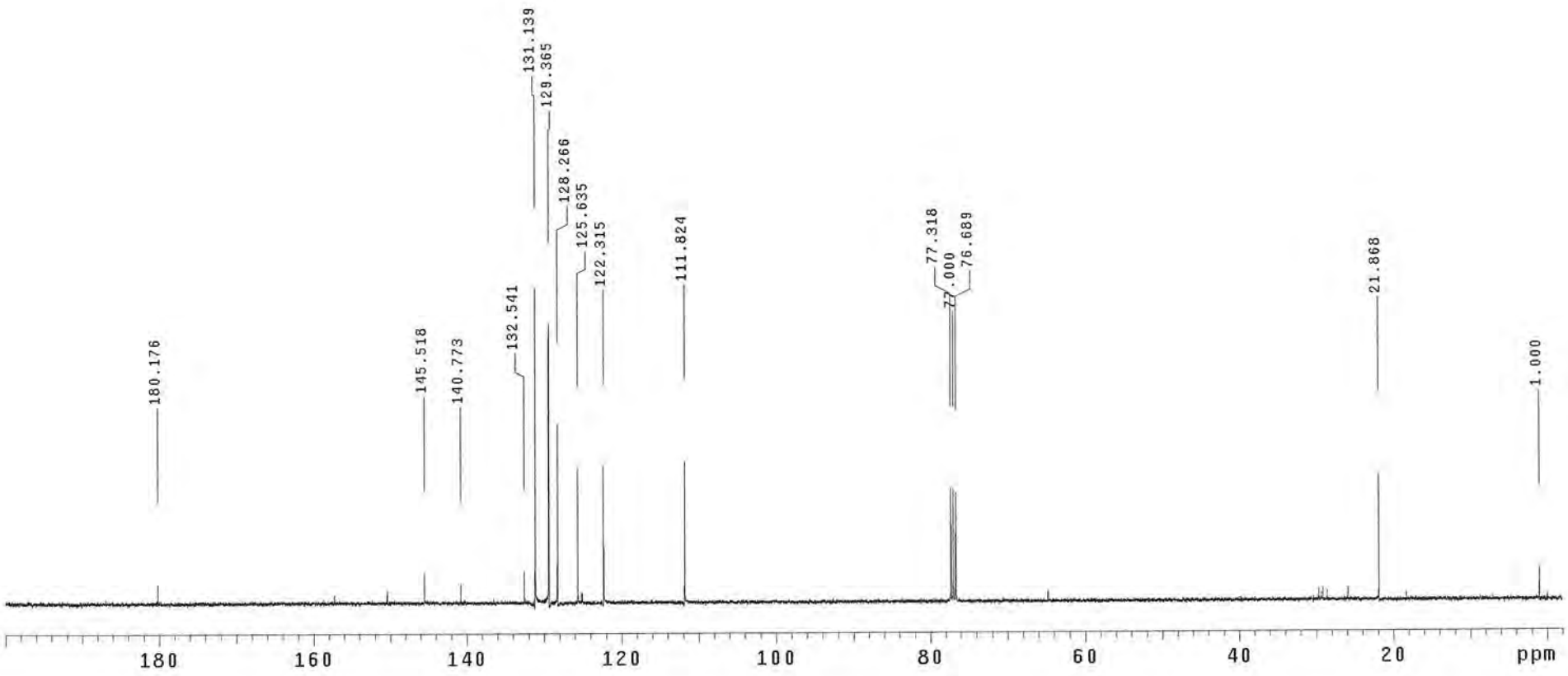


SIVA-RP3-20

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 28 2014
Solvent: CDCl3
Ambient temperature
Total 7376 repetitions

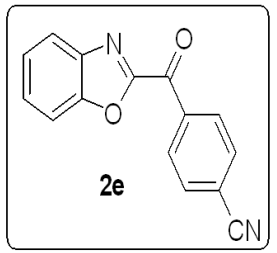


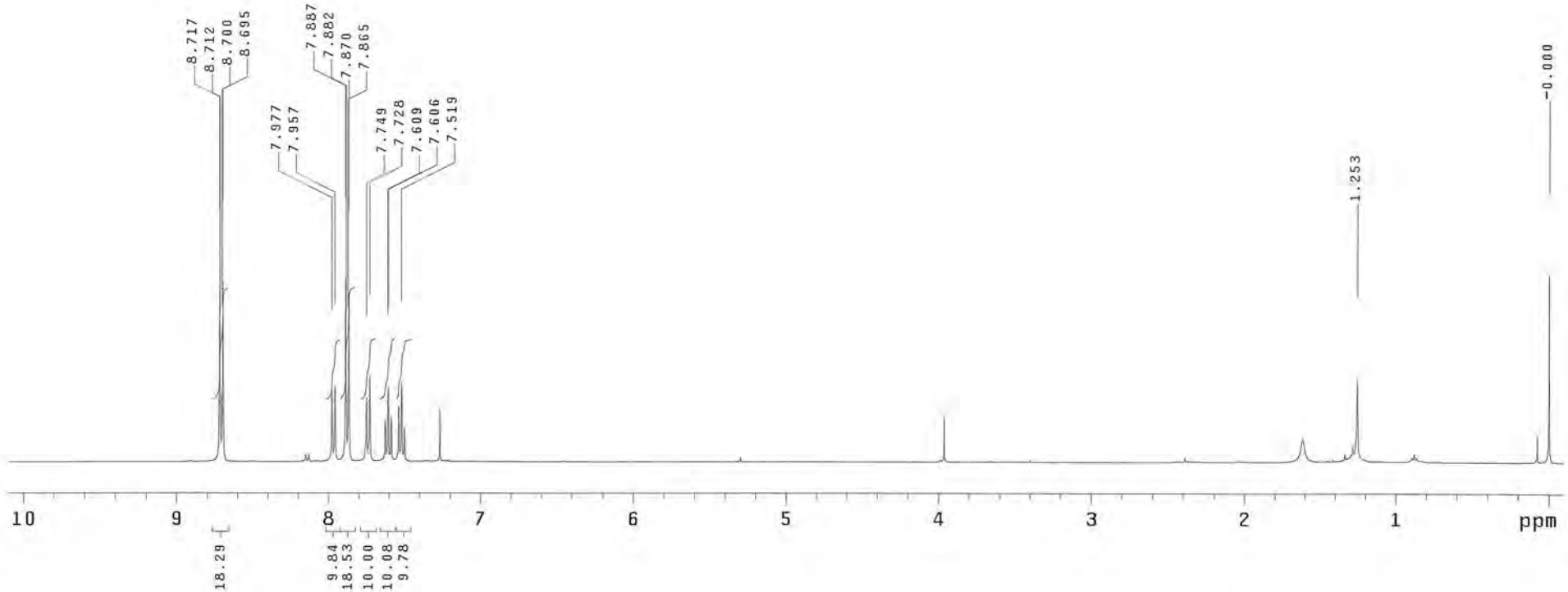


SIVA-RP3-28

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978932 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec

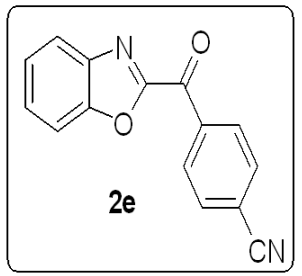


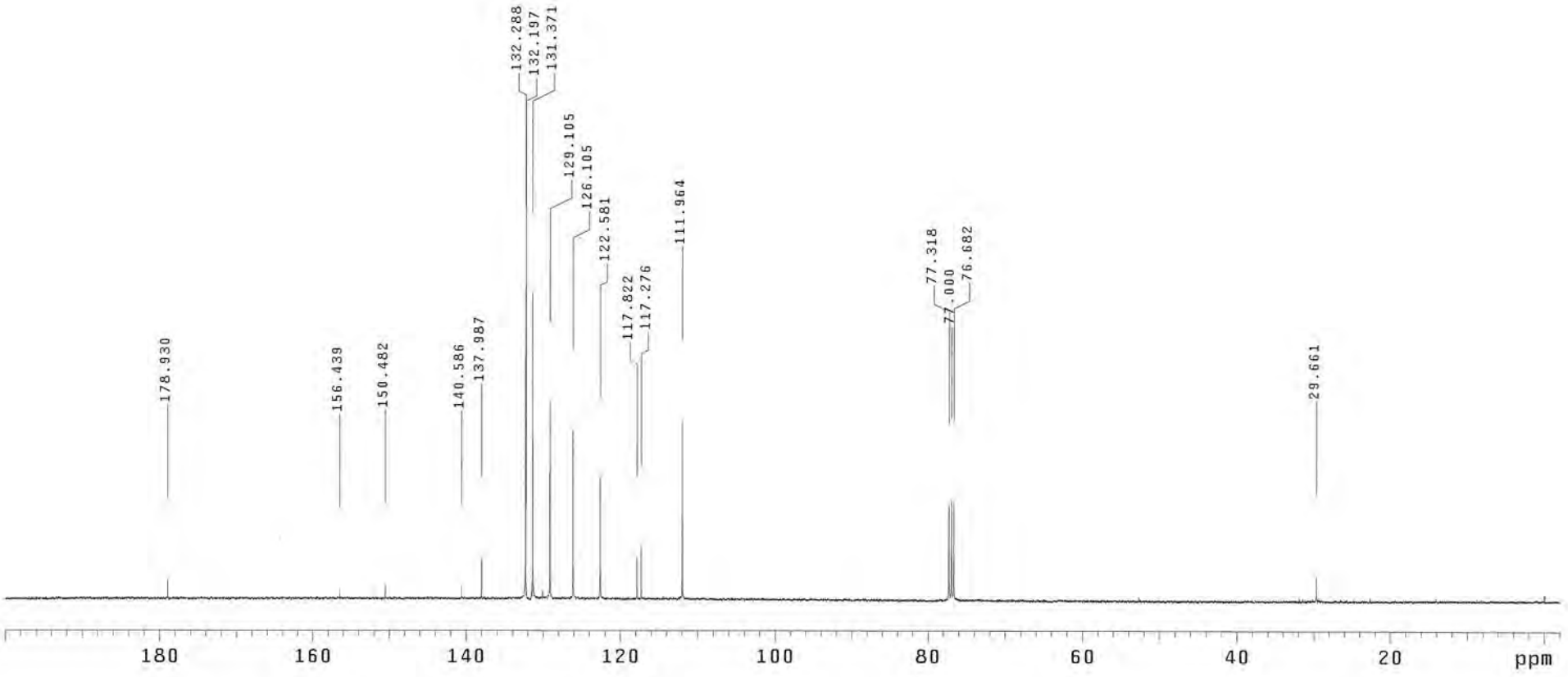


SIVA-RP3-28

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
6176 repetitions
OBSERVE C13, 100.6801330 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec

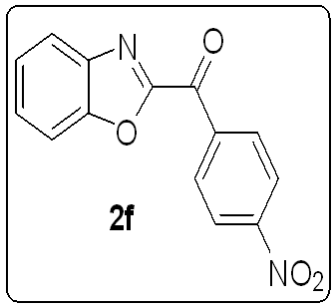


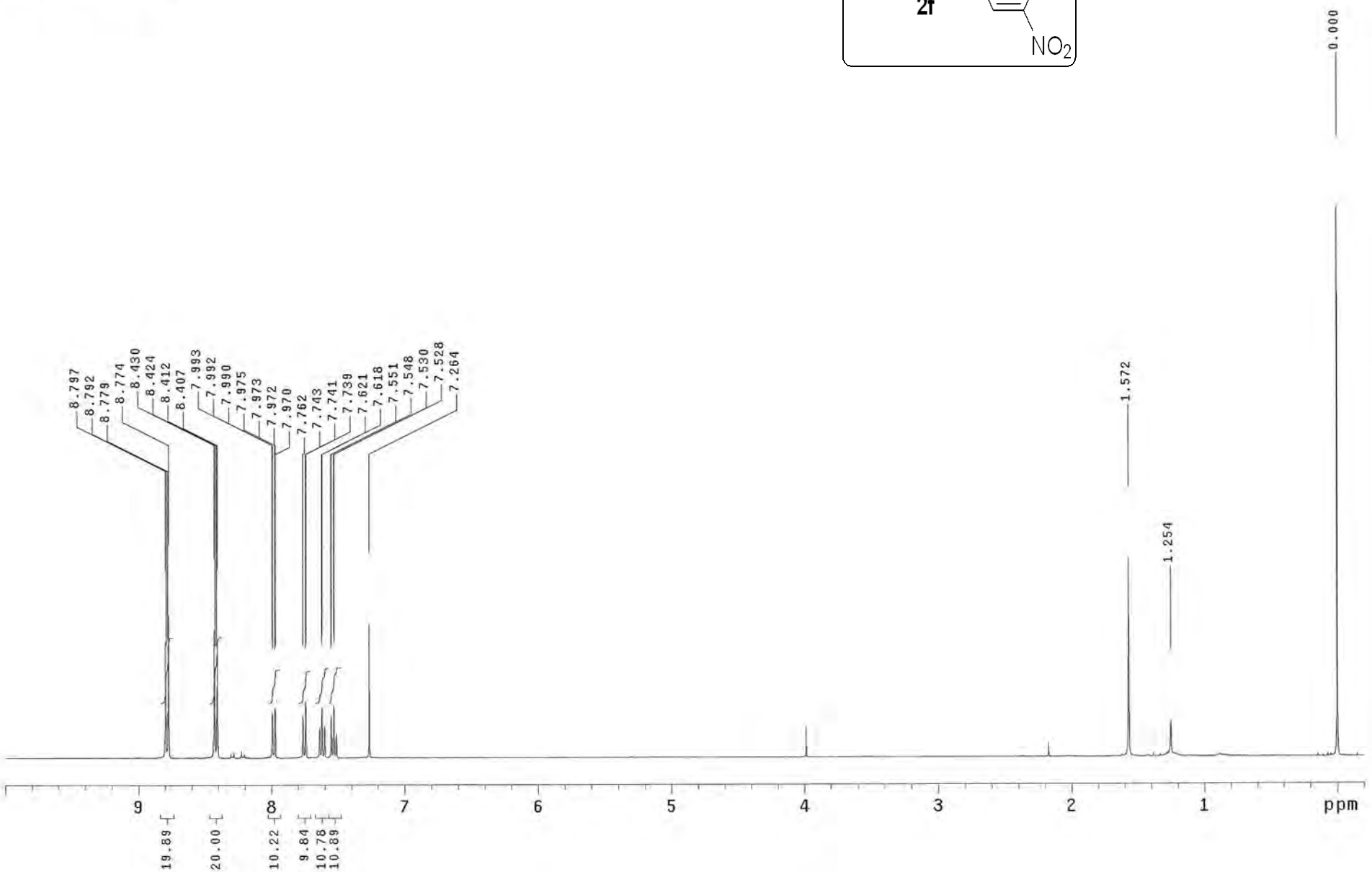


SIVA-RP3-44

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 21 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

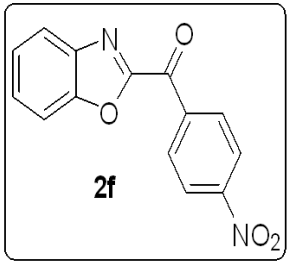


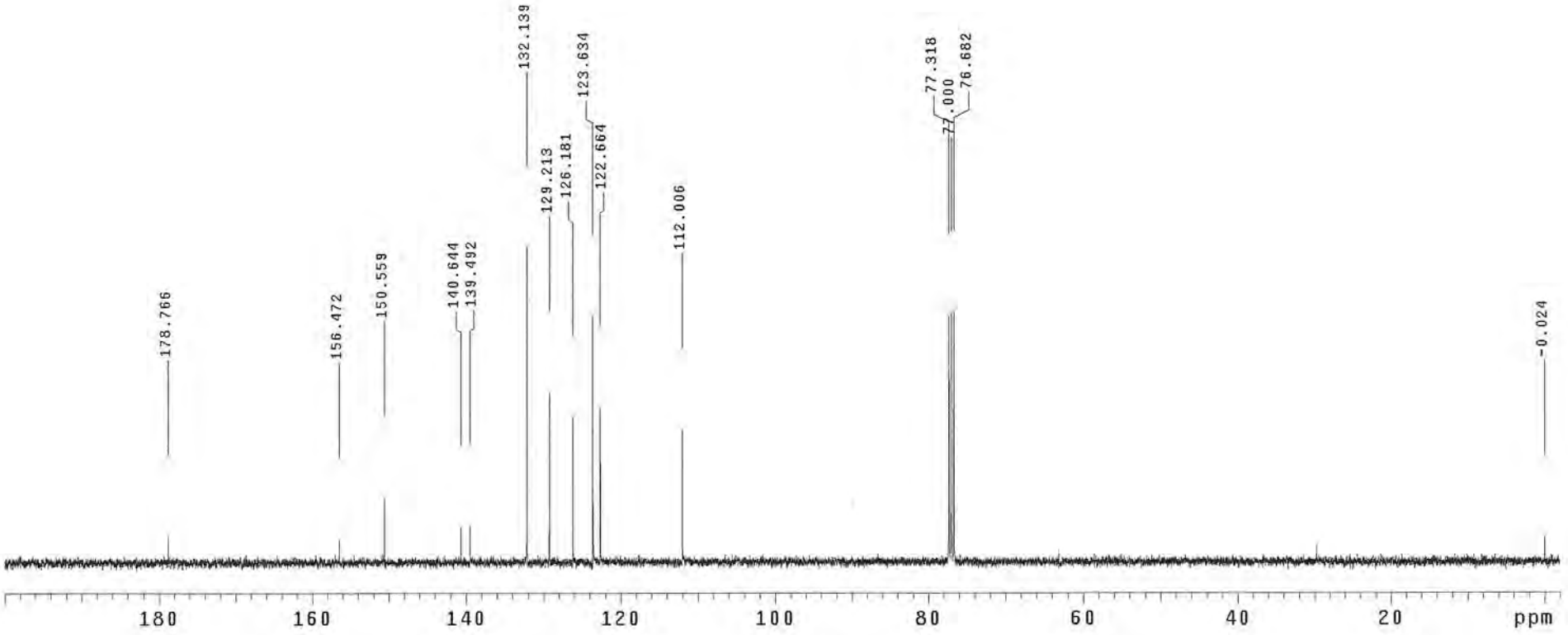


SIVA-RP3-44

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 21 2013
Solvent: CDCl3
Ambient temperature
Total 8016 repetitions

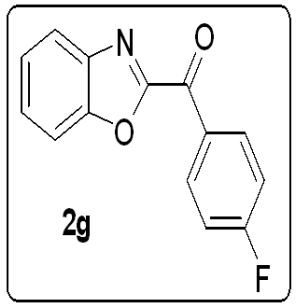


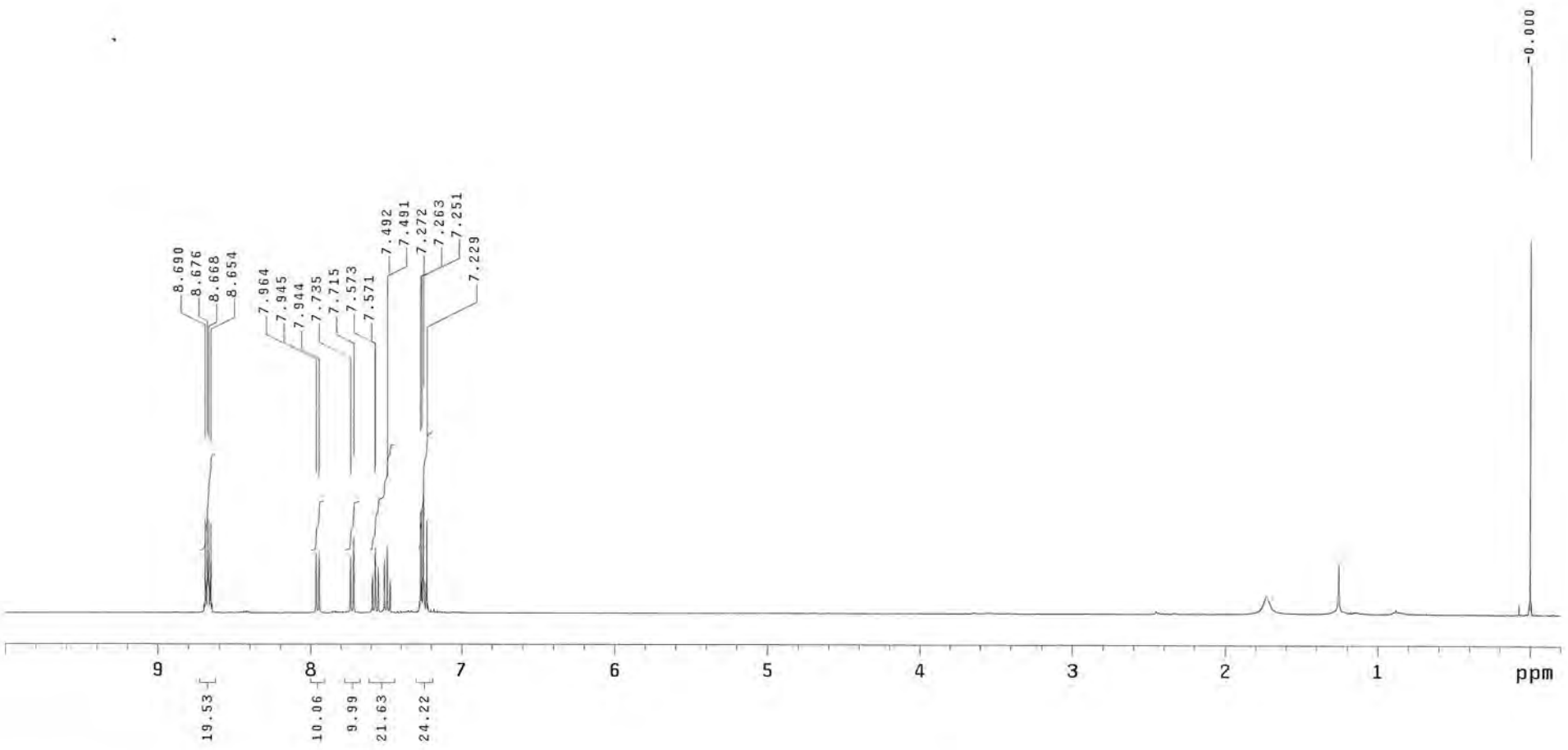


SIVA-RP3-66

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
24 repetitions
OBSERVE H1, 400.3978949 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec

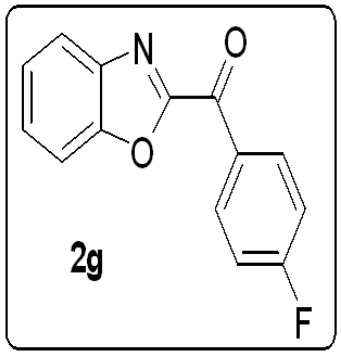




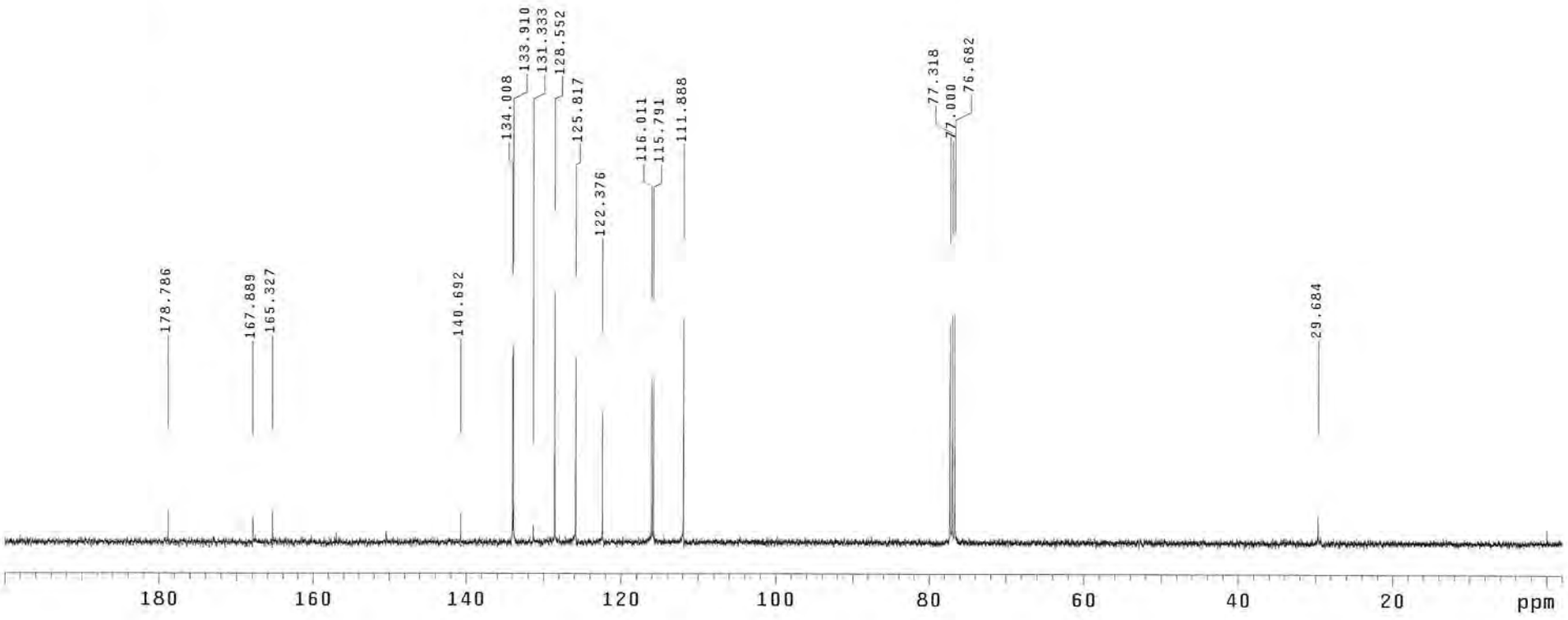
SIVA-RP3-66

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
4064 repetitions
OBSERVE C13, 100.6801315 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



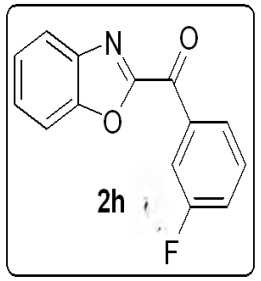
2g

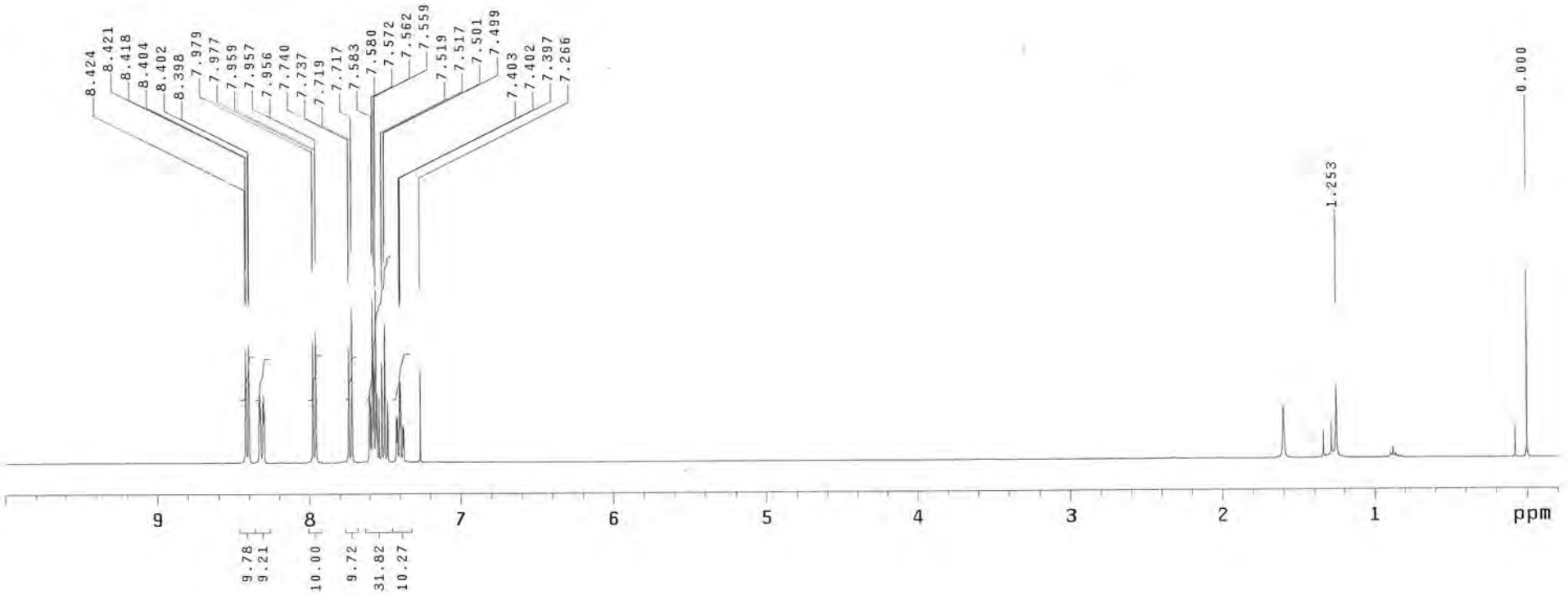


SIVA-RP4-122

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Dec 24 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

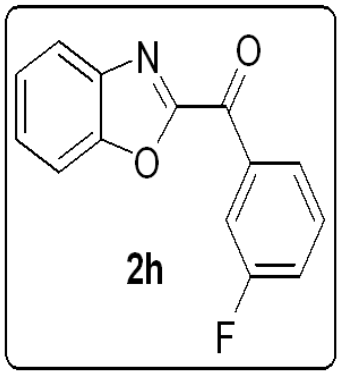


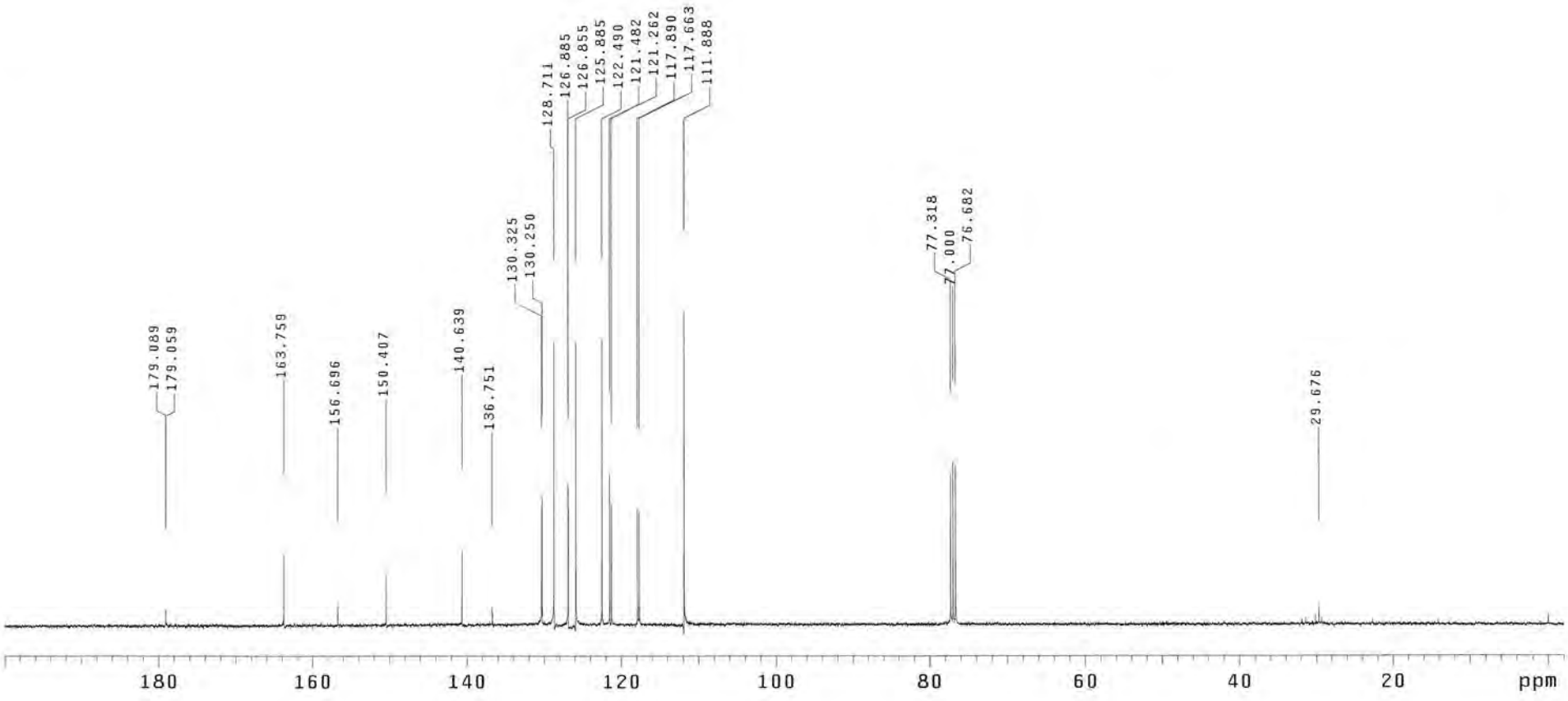


SIVA-RP4-122

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Dec 24 2013
Solvent: CDCl3
Ambient temperature
Total 4432 repetitions

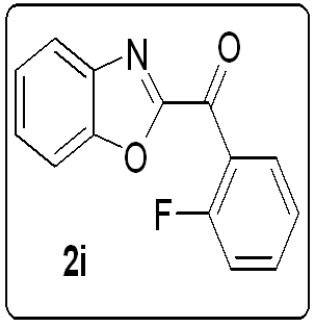




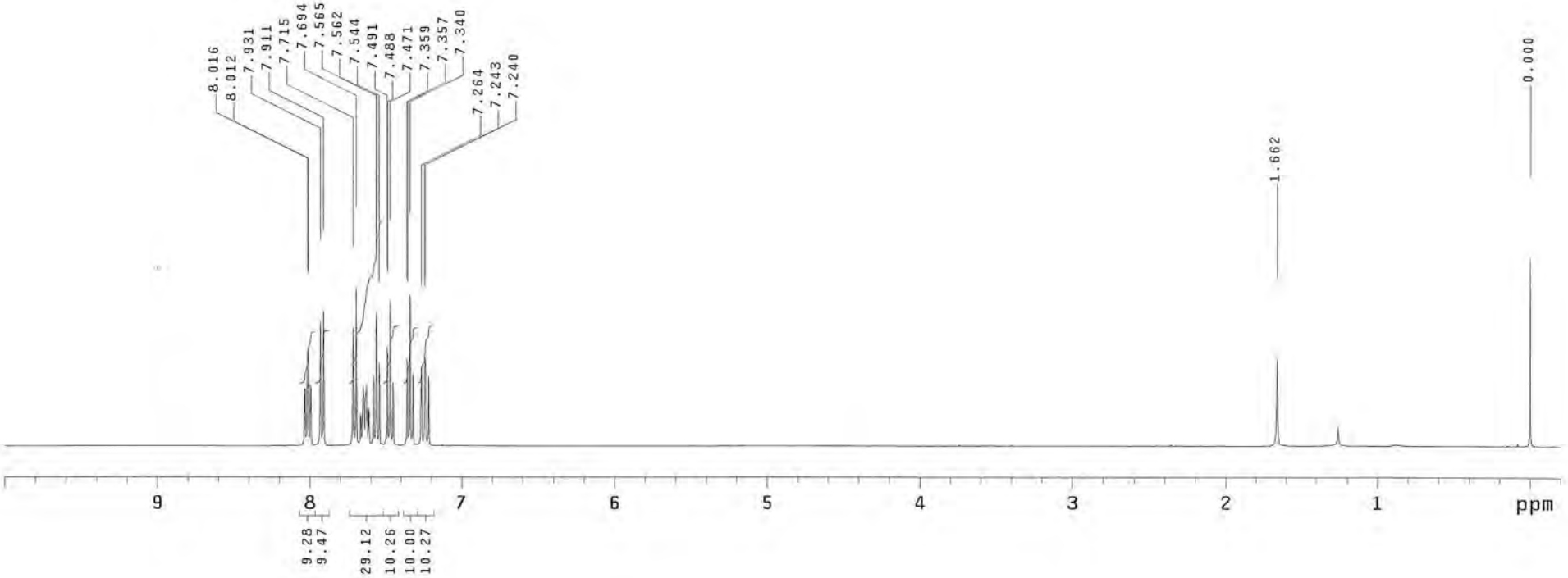
SIVA-RP3-39

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978927 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec



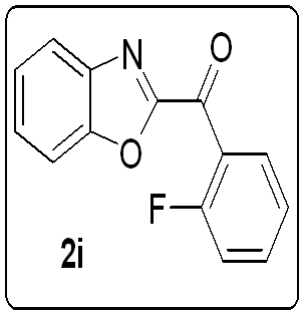
2i



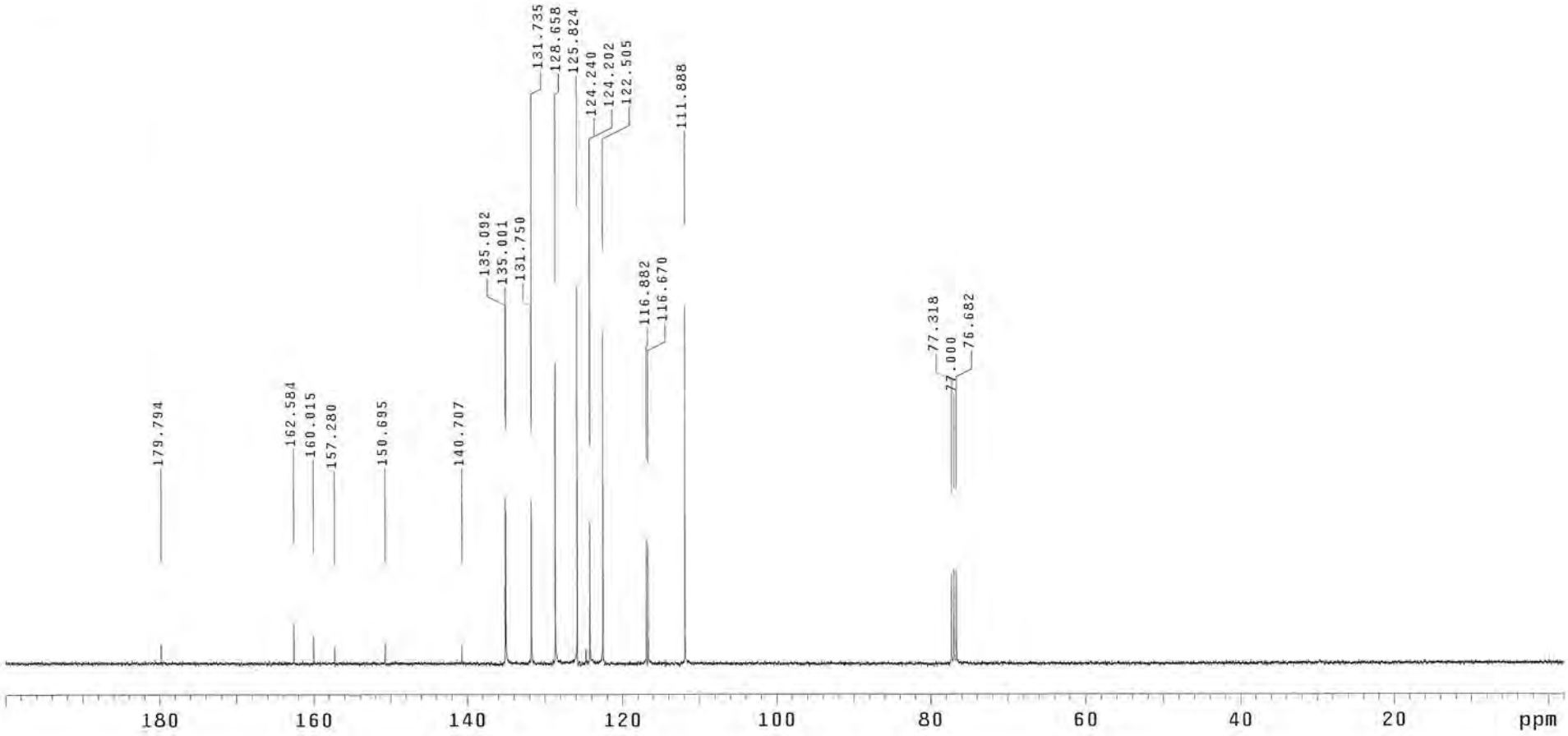
SIVA-RP3-39

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 2464 repetitions
OBSERVE C13, 100.6801346 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



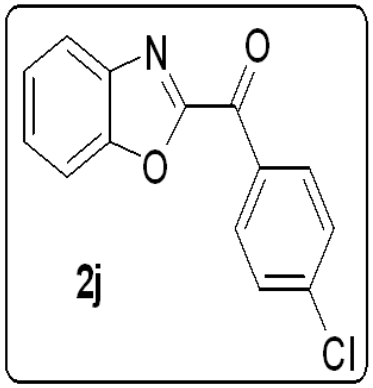
2i



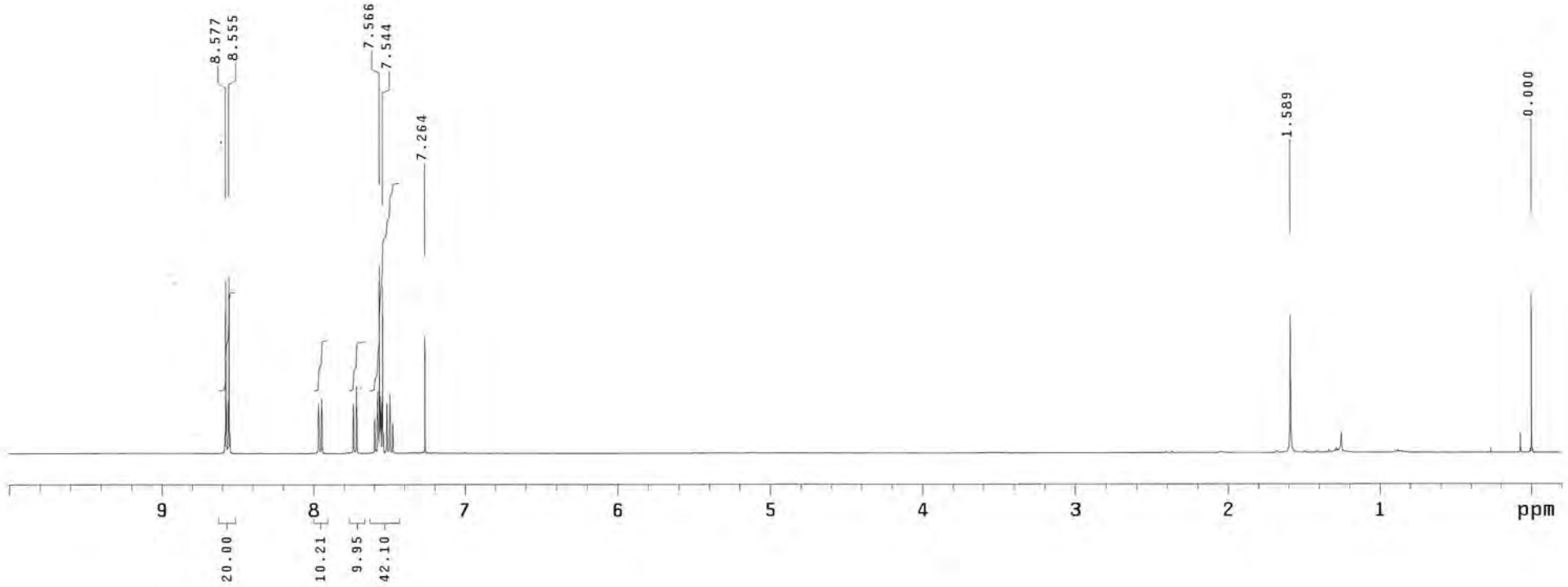
SIVA-RP4-81

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 18 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions



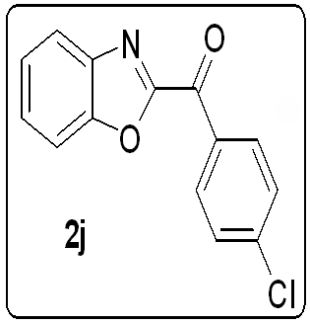
2j

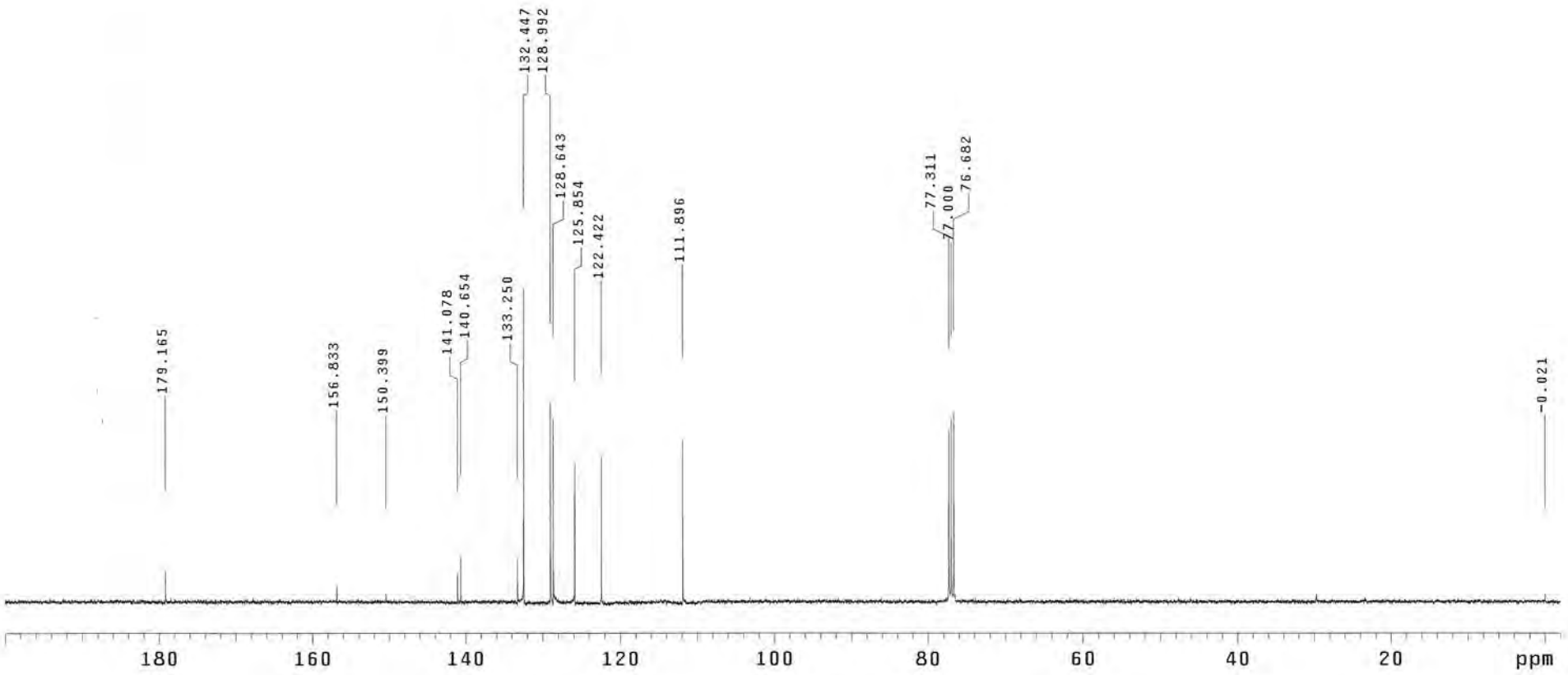


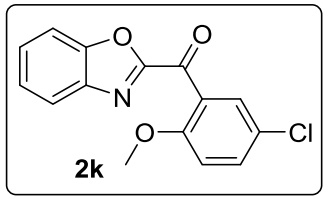
SIVA-RP4-81

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 18 2014
Solvent: CDCl3
Ambient temperature
Total 6160 repetitions



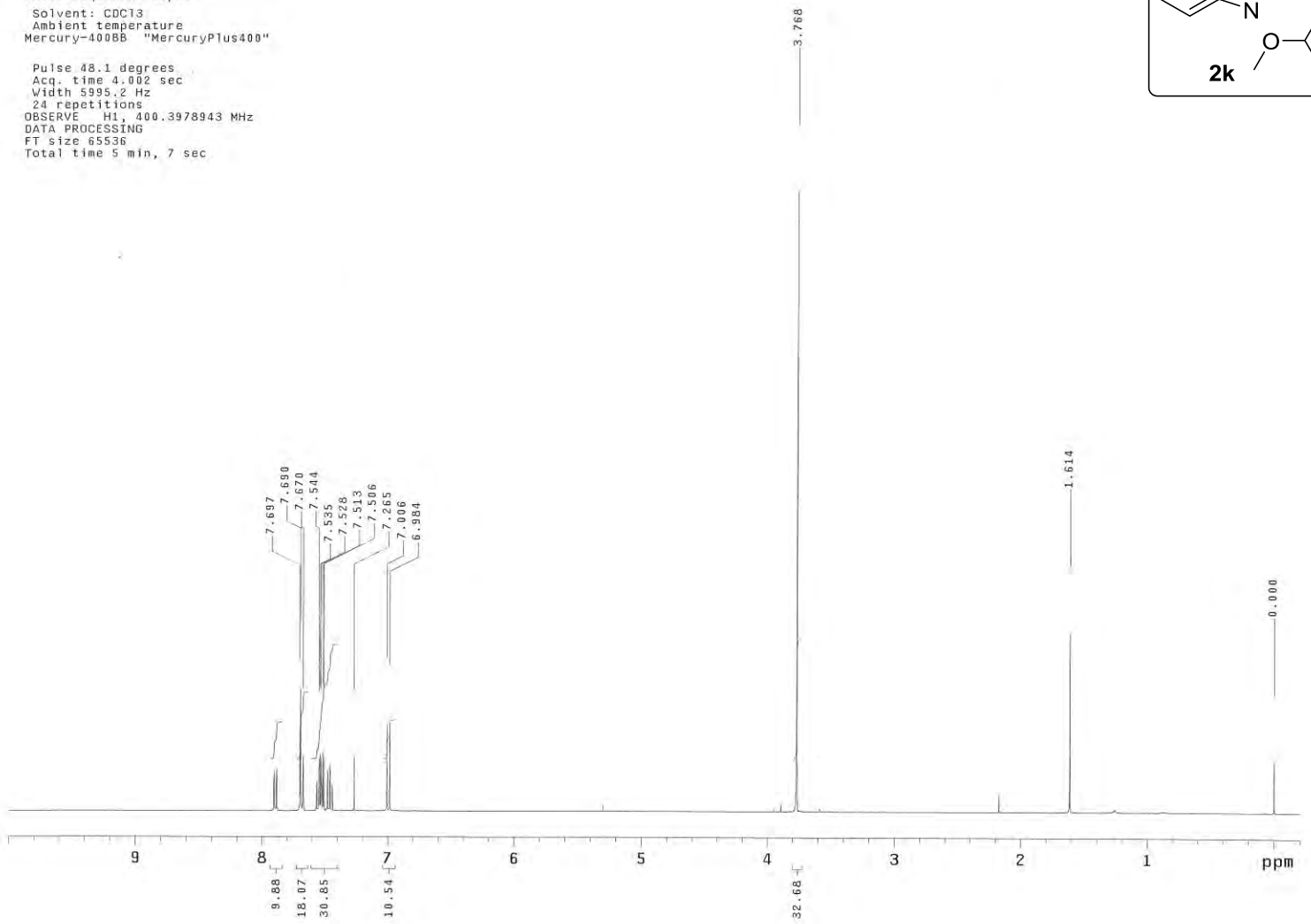


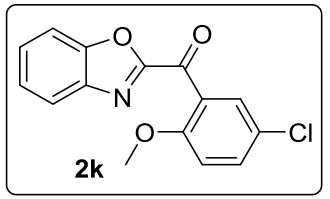


SIVA-RP4-59

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
24 repetitions
OBSERVE H1, 400.3978943 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec

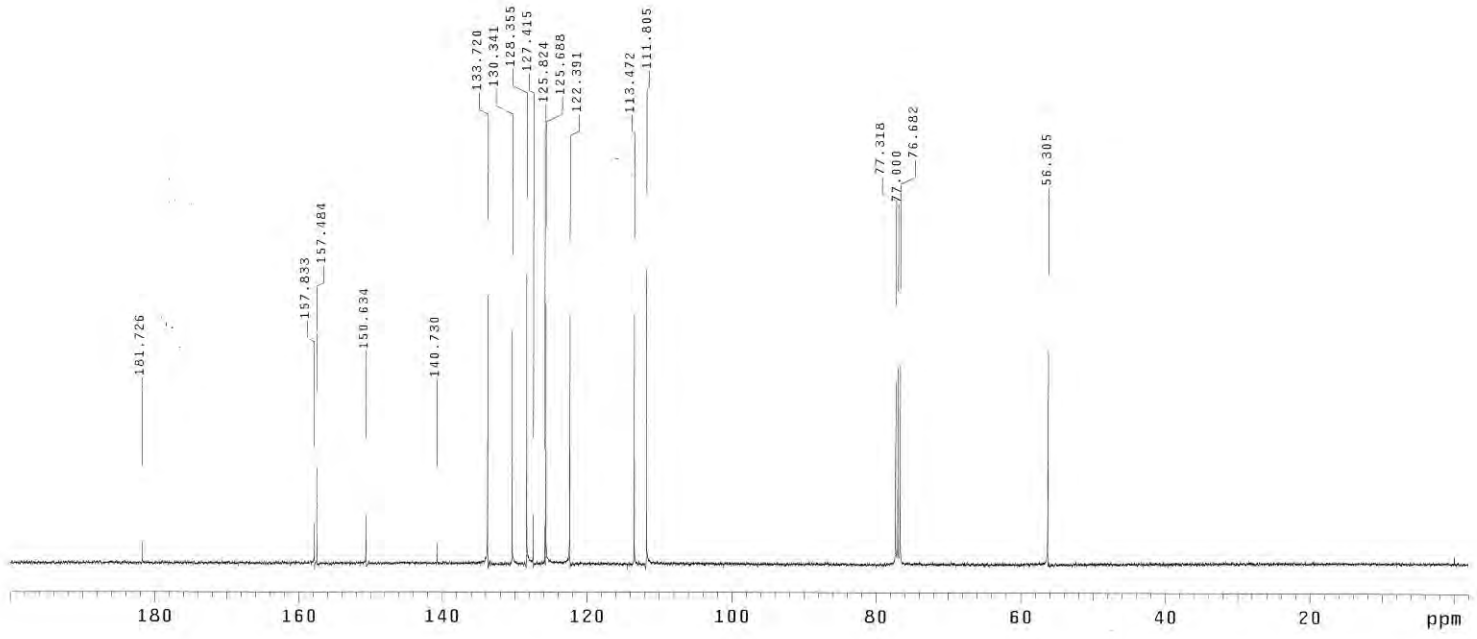




SIVA-RP4-59

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

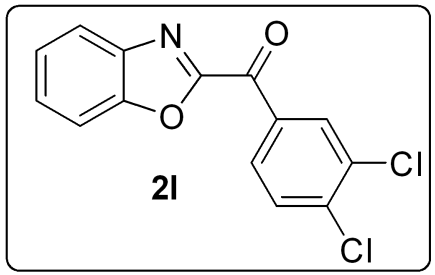
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
7008 repetitions
OBSERVE C13, 100.6801330 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



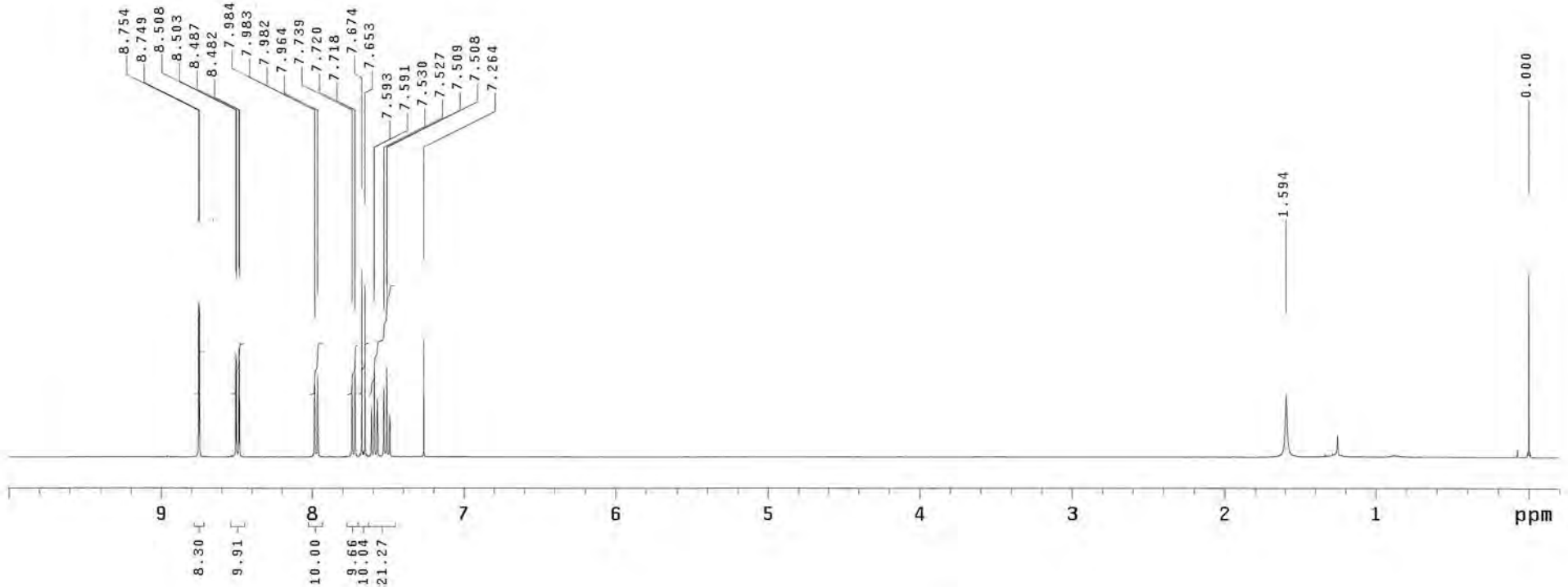
SIVA-RP3-60

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 17 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions



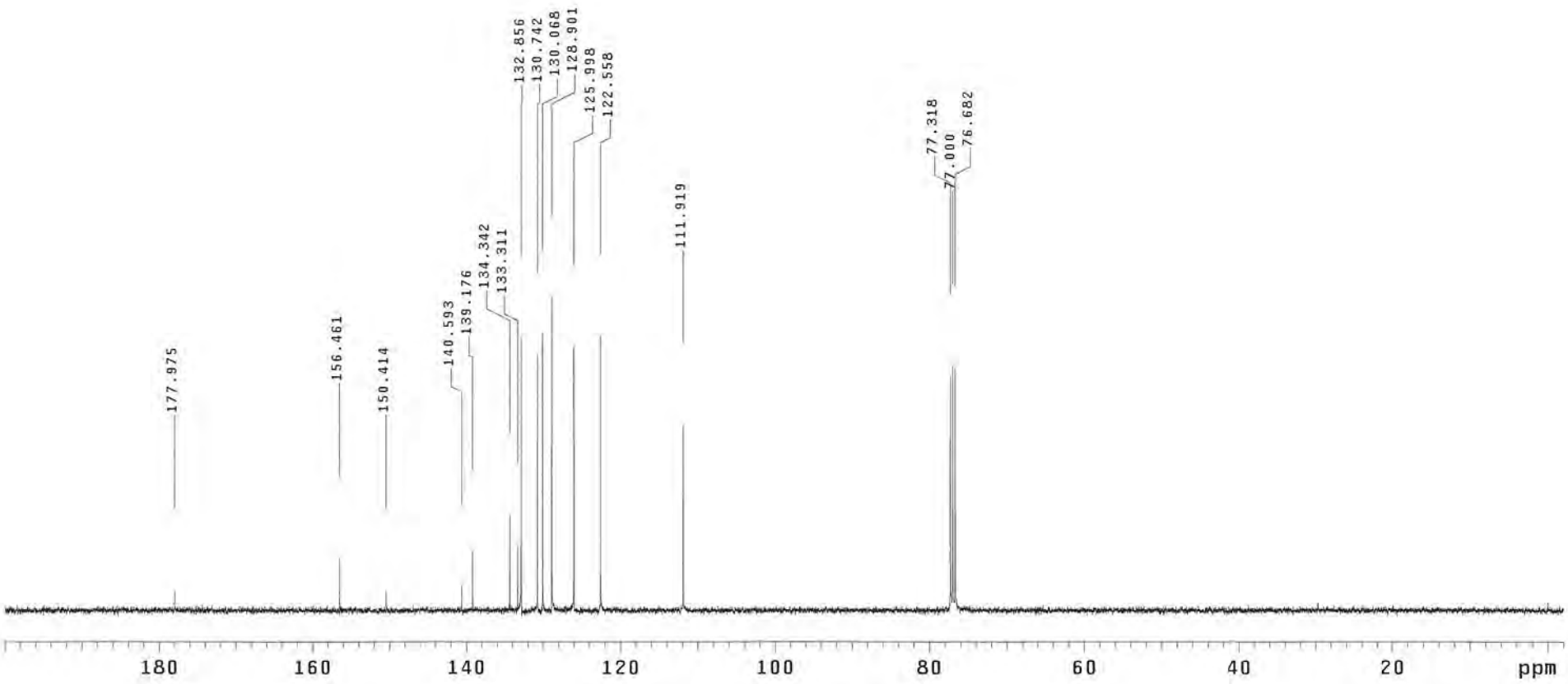
2l



SIVA-RP3-60

Pulse Sequence: s2pul

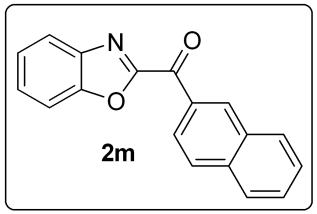
Mercury-400BB "MercuryPlus400"
Date: Mar 17 2014
Solvent: CDCl3
Ambient temperature
Total 4752 repetitions



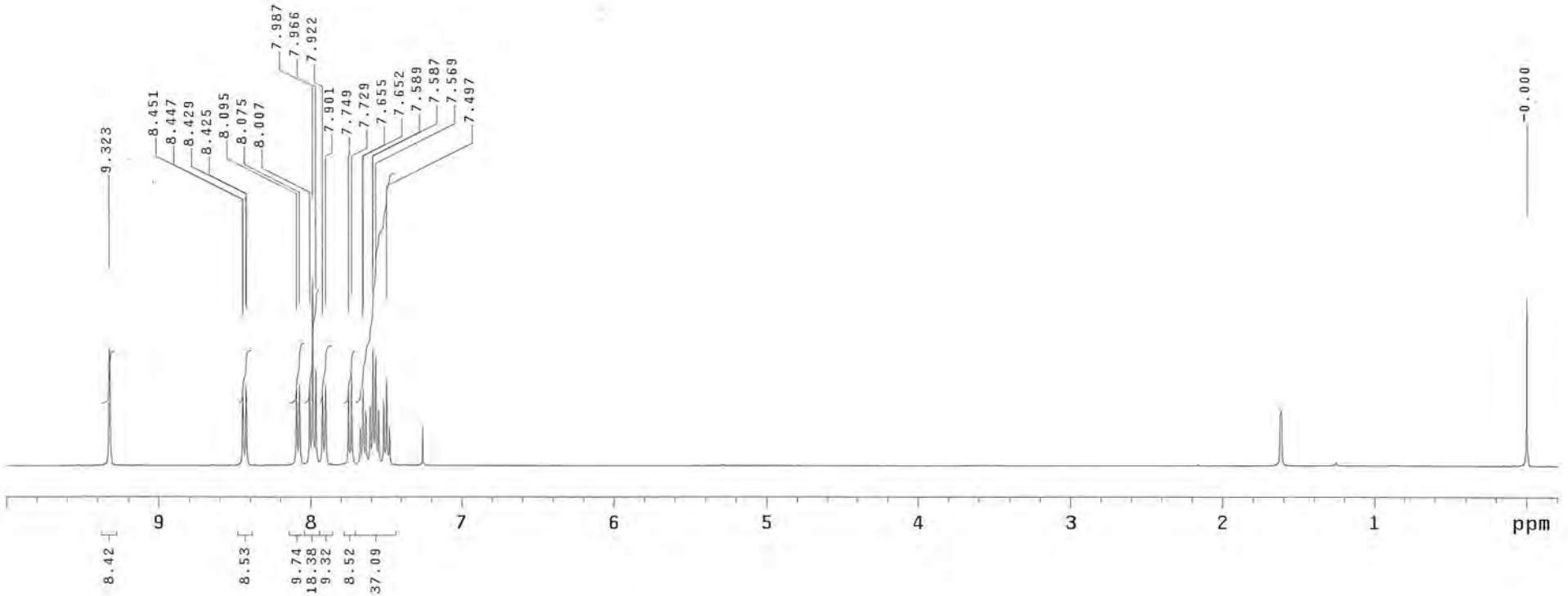
SIVA-RP3-35

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
24 repetitions
OBSERVE H1, 400.3978969 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec



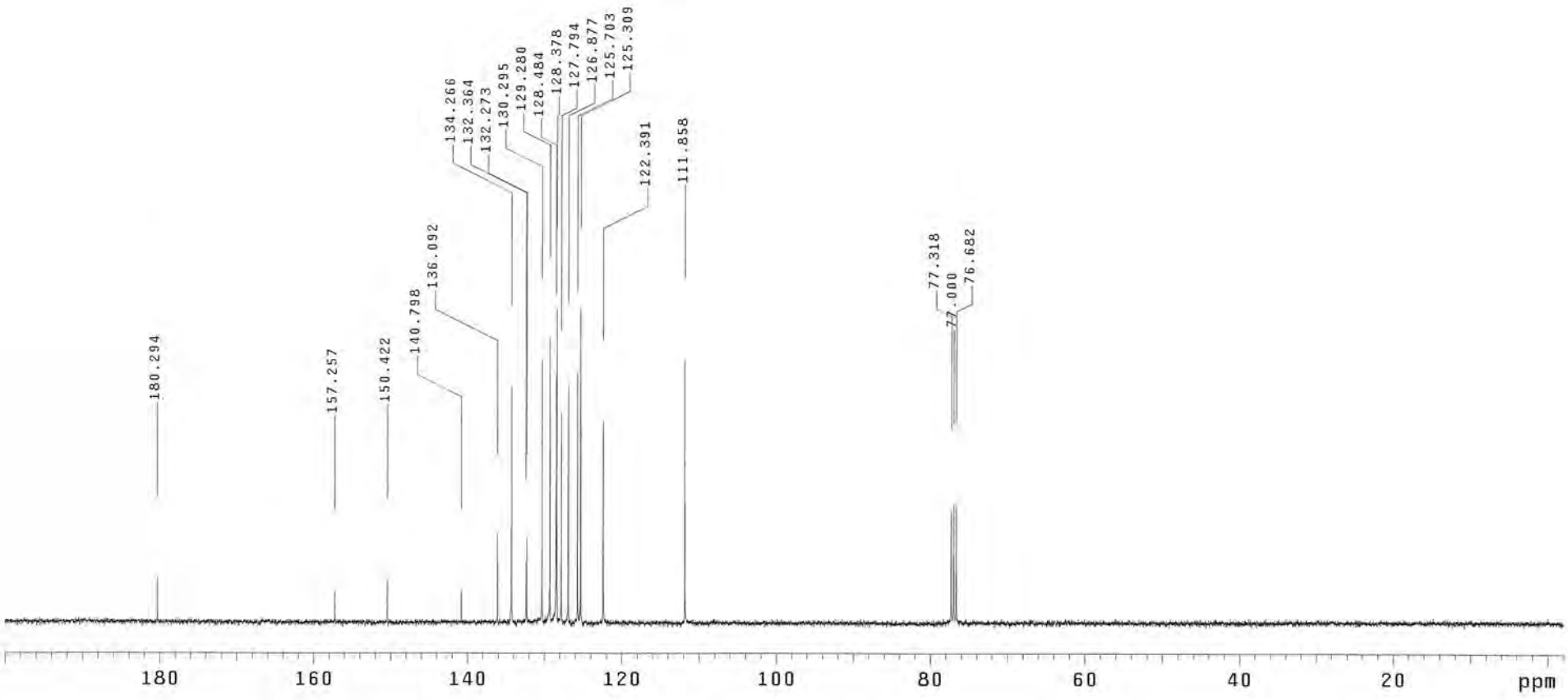
2m



SIVA-RP3-35

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

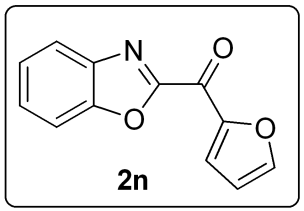
 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 2144 repetitions
OBSERVE C13, 100.6801338 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



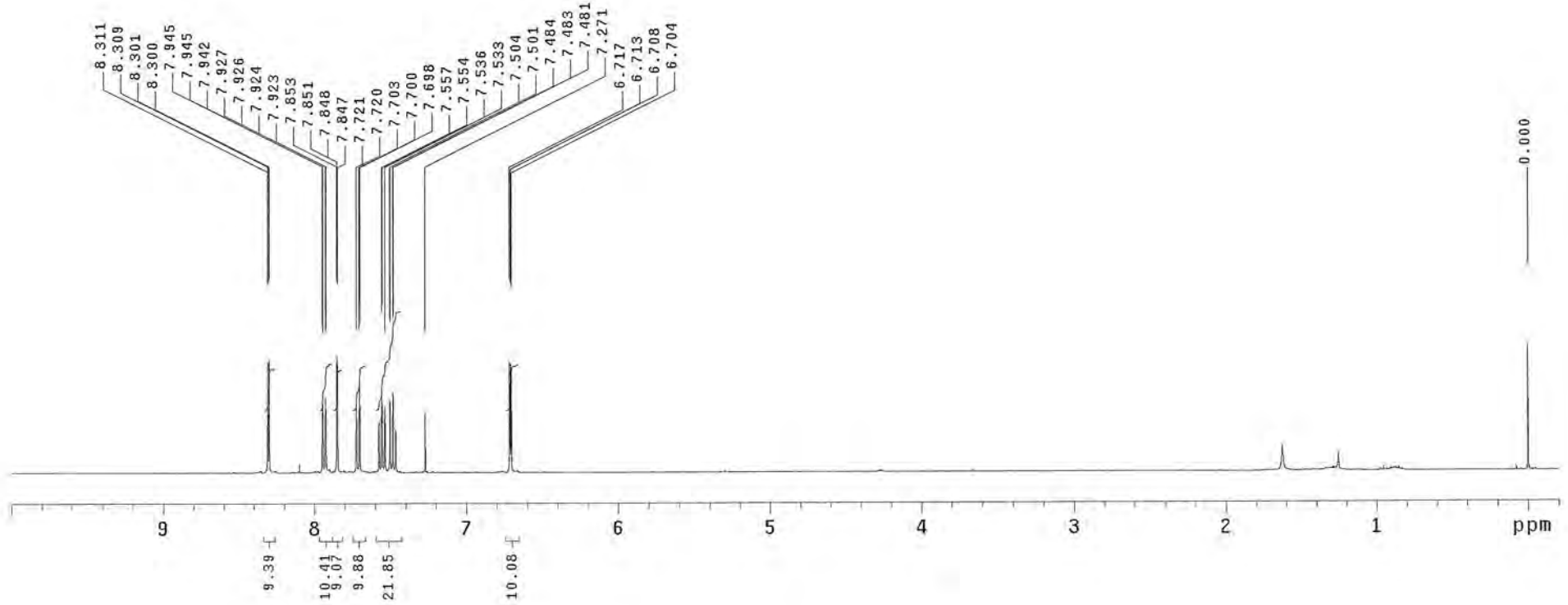
SIVA-RP3-33

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 16 2013
Solvent: CDCl3
Ambient temperature
Total 56 repetitions



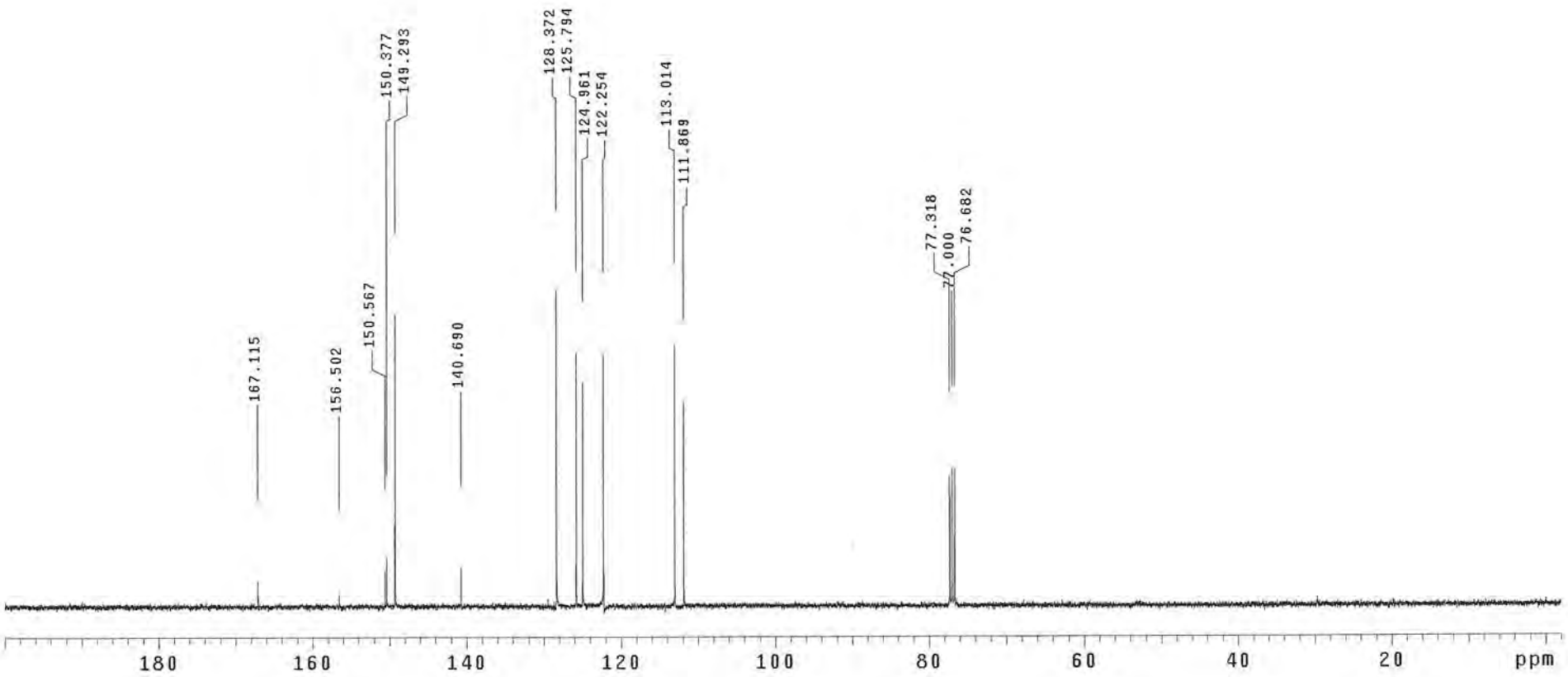
2n



SIVA-RP3-33

Pulse Sequence: s2pul

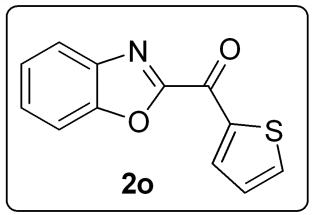
UNITYplus-400 "unity400"
Date: Oct 16 2013
Solvent: CDCl3
Ambient temperature
Total 5504 repetitions



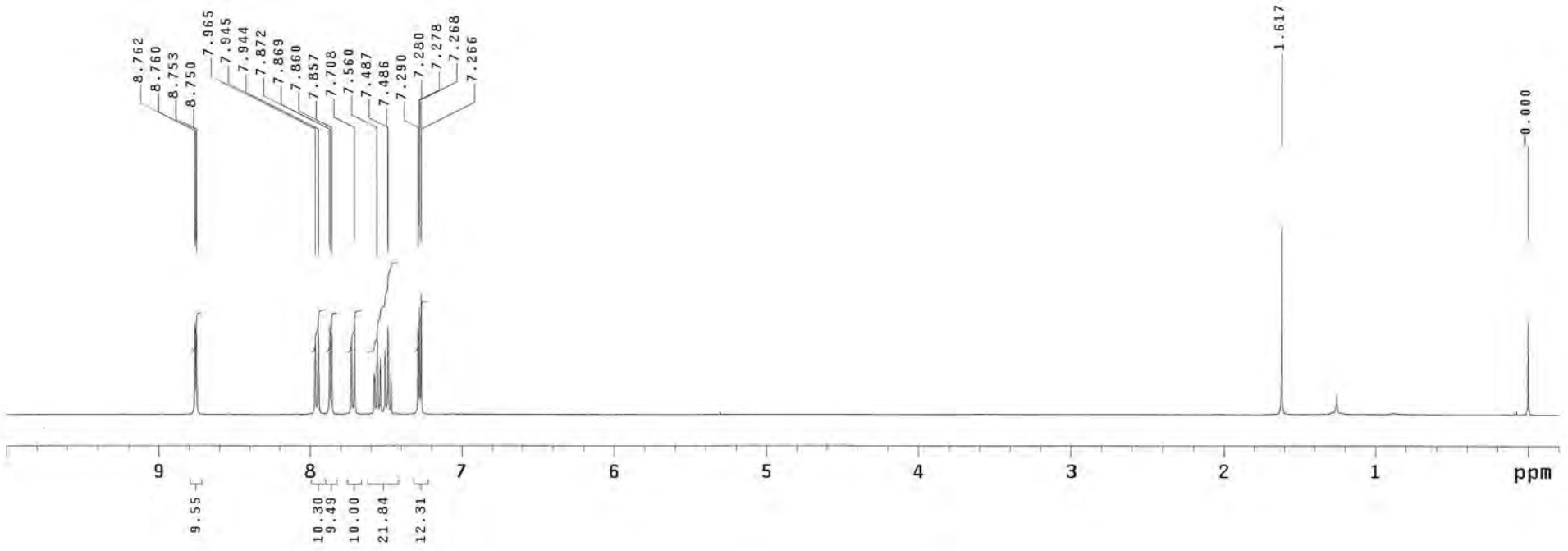
SIVA-RP3-47

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 17 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions



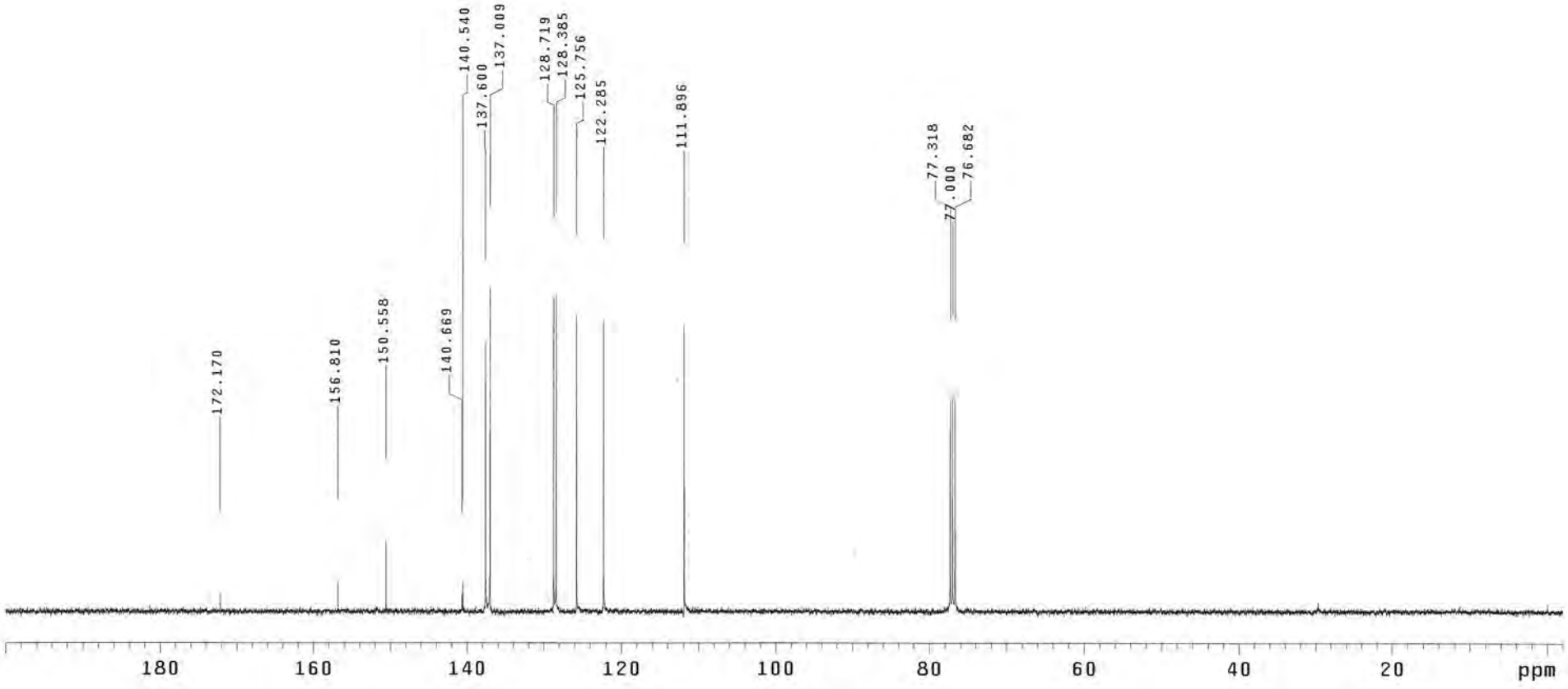
2o



SIVA-RP3-47

Pulse Sequence: s2pul

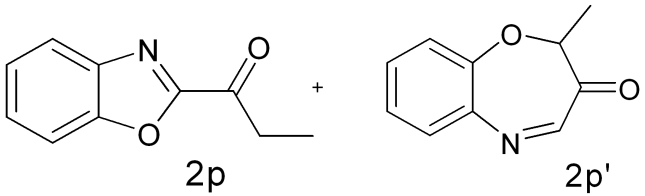
Mercury-400BB "MercuryPlus400"
Date: Mar 17 2014
Solvent: CDCl3
Ambient temperature
Total 4400 repetitions



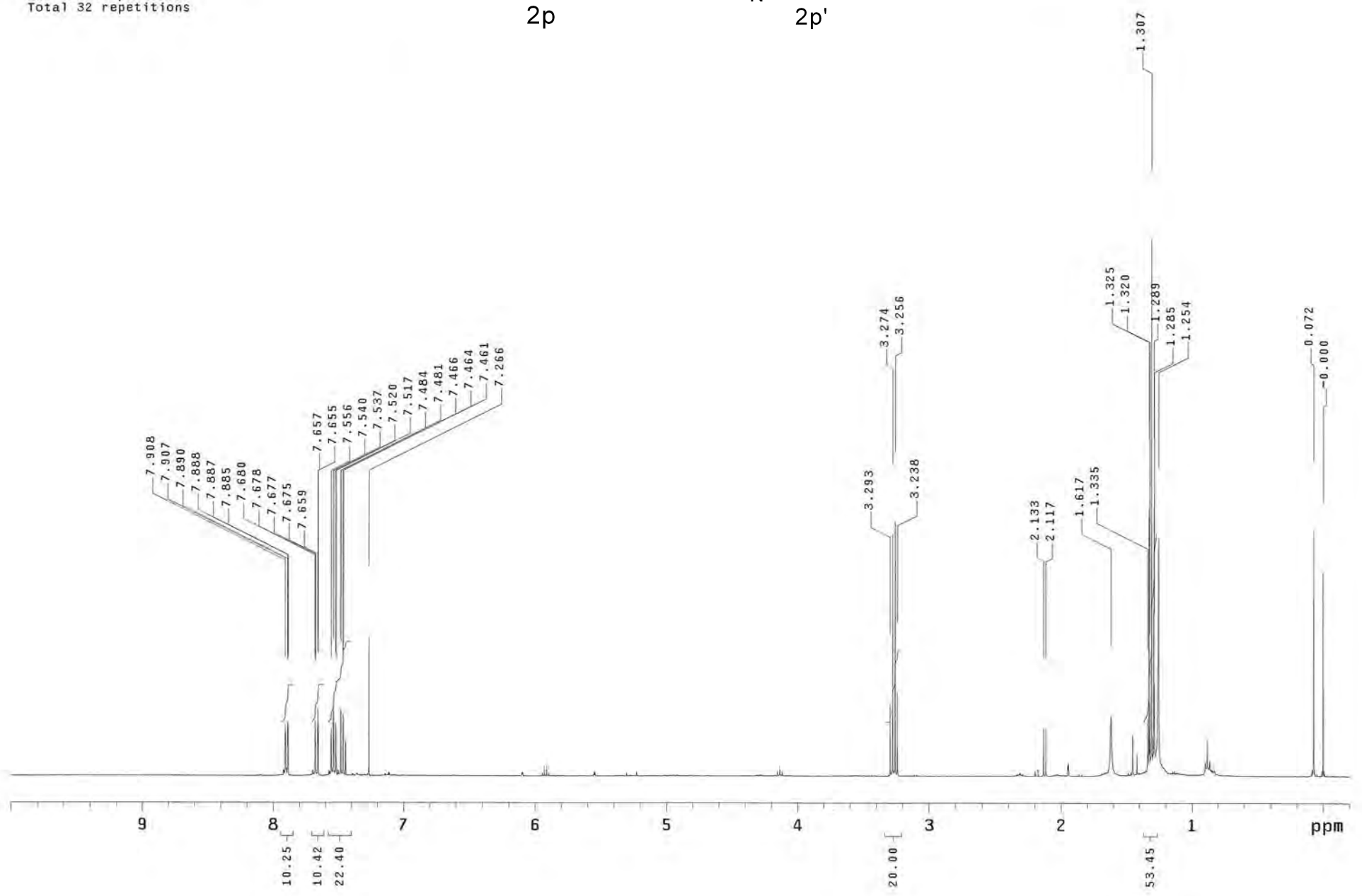
SIVA-RP3-77

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 18 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



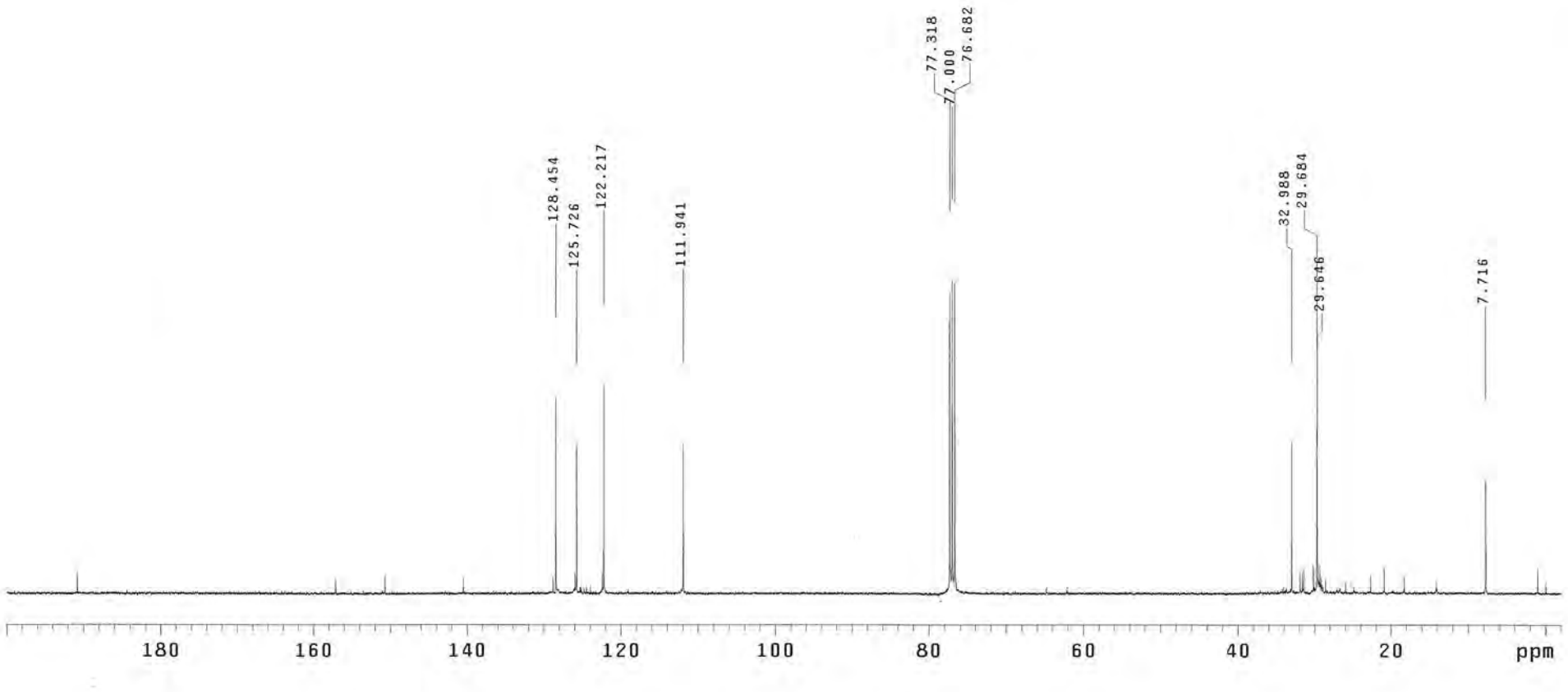
2p 2p'



SIVA-RP3-77

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 17 2014
Solvent: CDCl3
Ambient temperature
Total 62672 repetitions

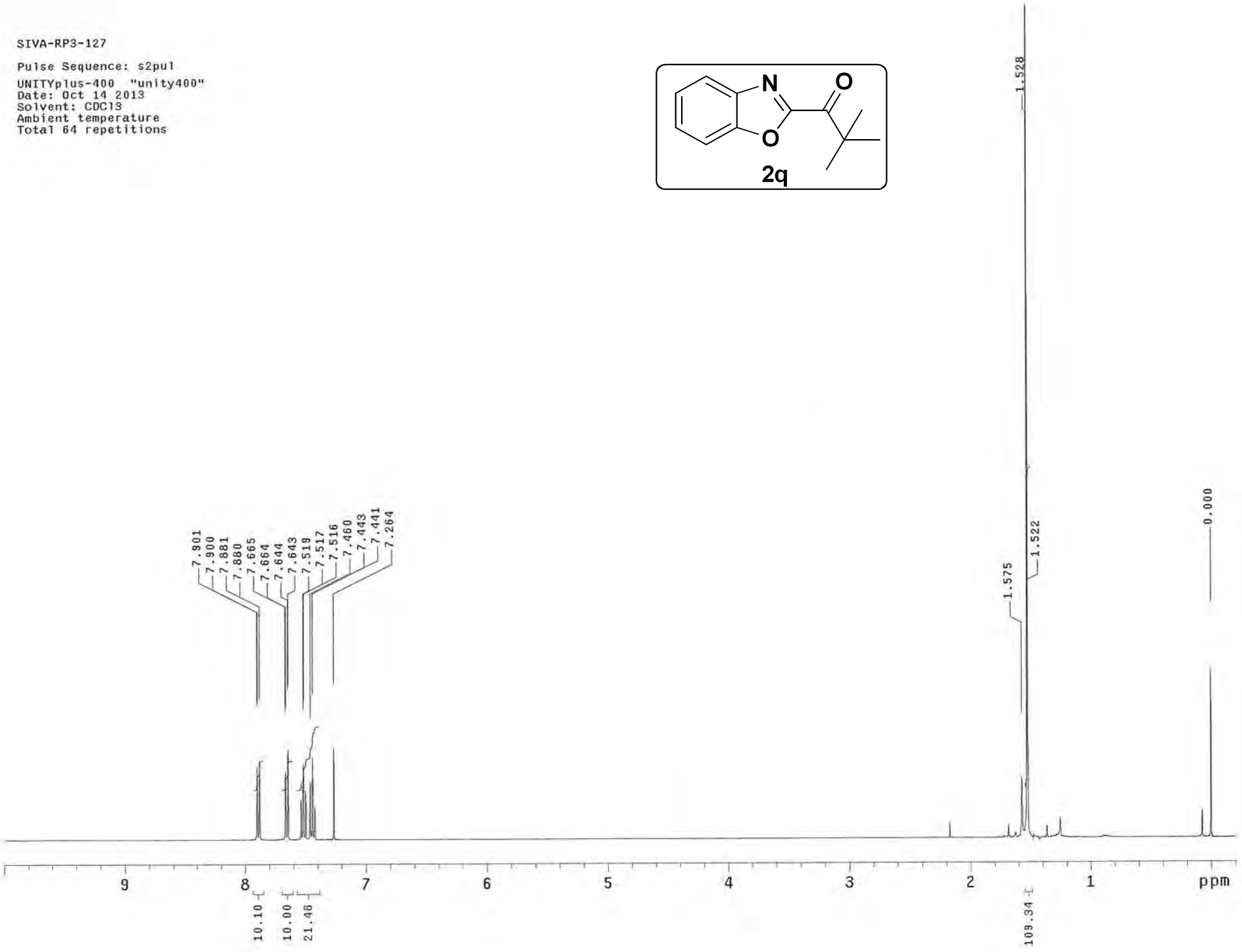


SIVA-RP3-127

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 14 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

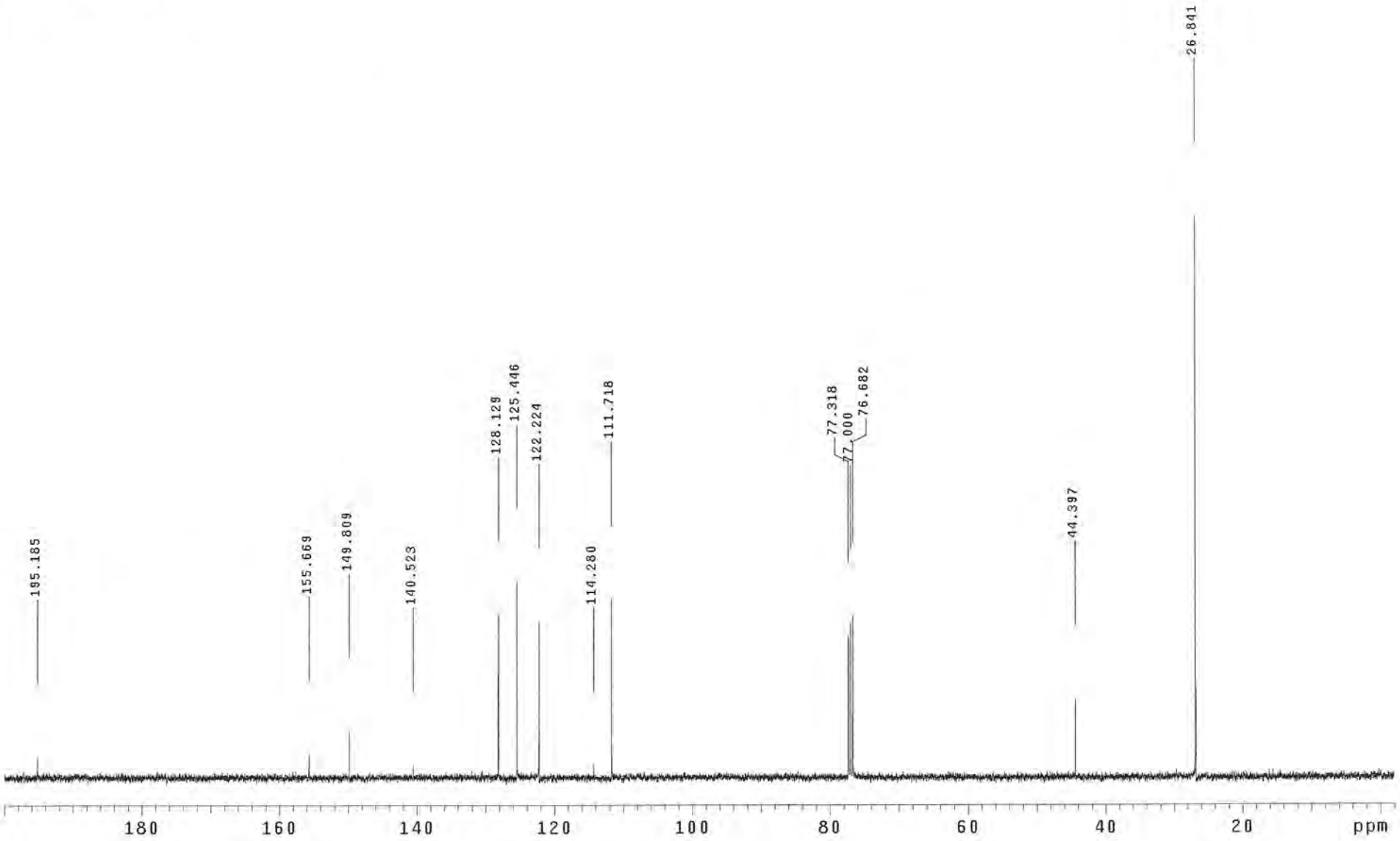
2q



SIVA-RP3-127

Pulse Sequence: s2pul

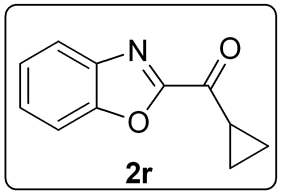
UNITYplus-400 "unity400"
Date: Oct 14 2013
Solvent: CDCl3
Ambient temperature
Total 3760 repetitions



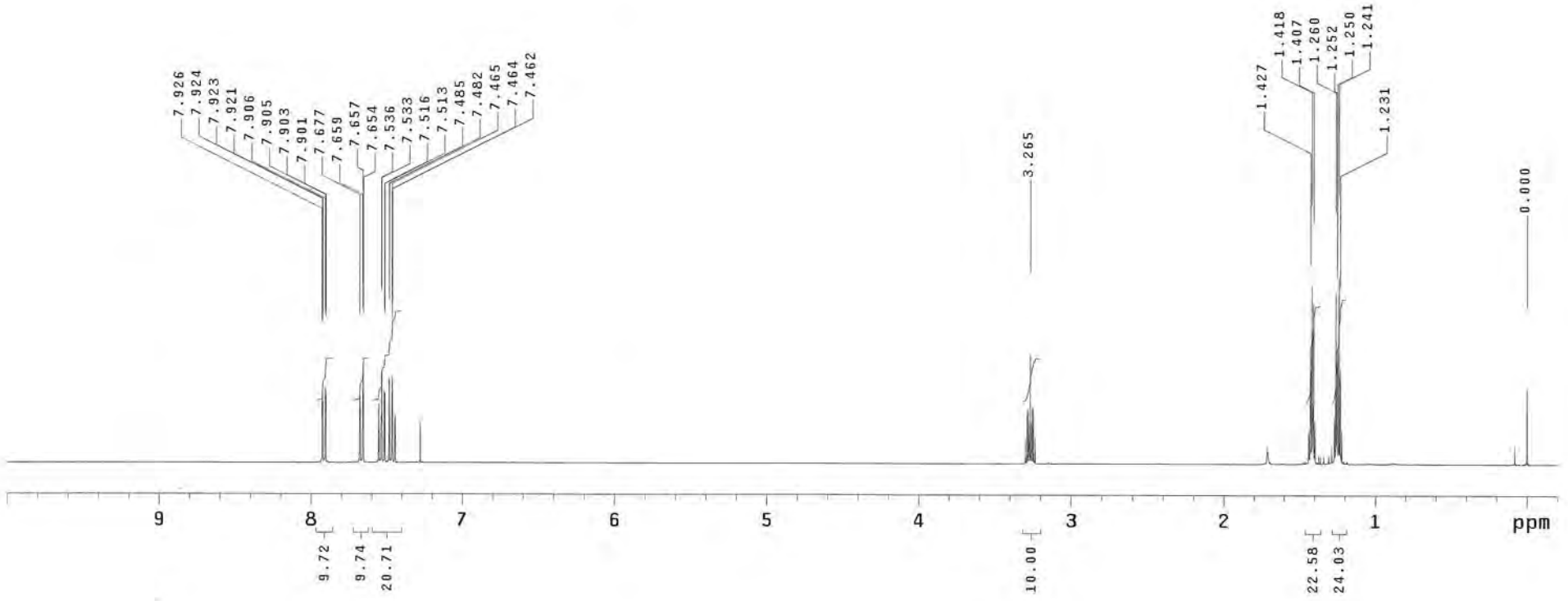
SIVA-RP4-158

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



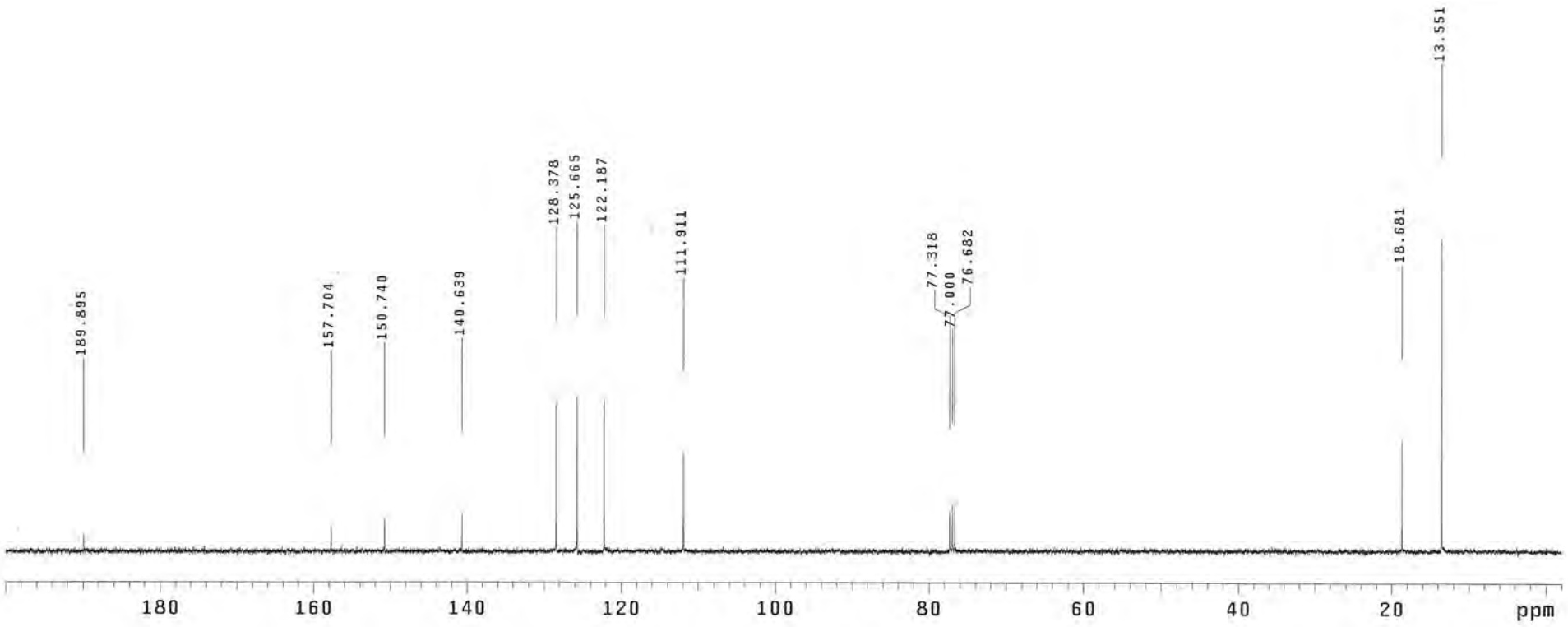
2r



SIVA-RP4-158

Pulse Sequence: s2pul

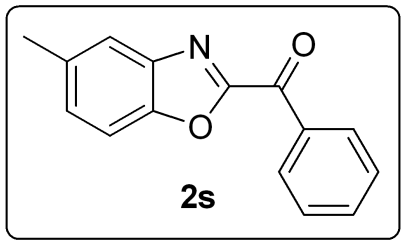
Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 320 repetitions



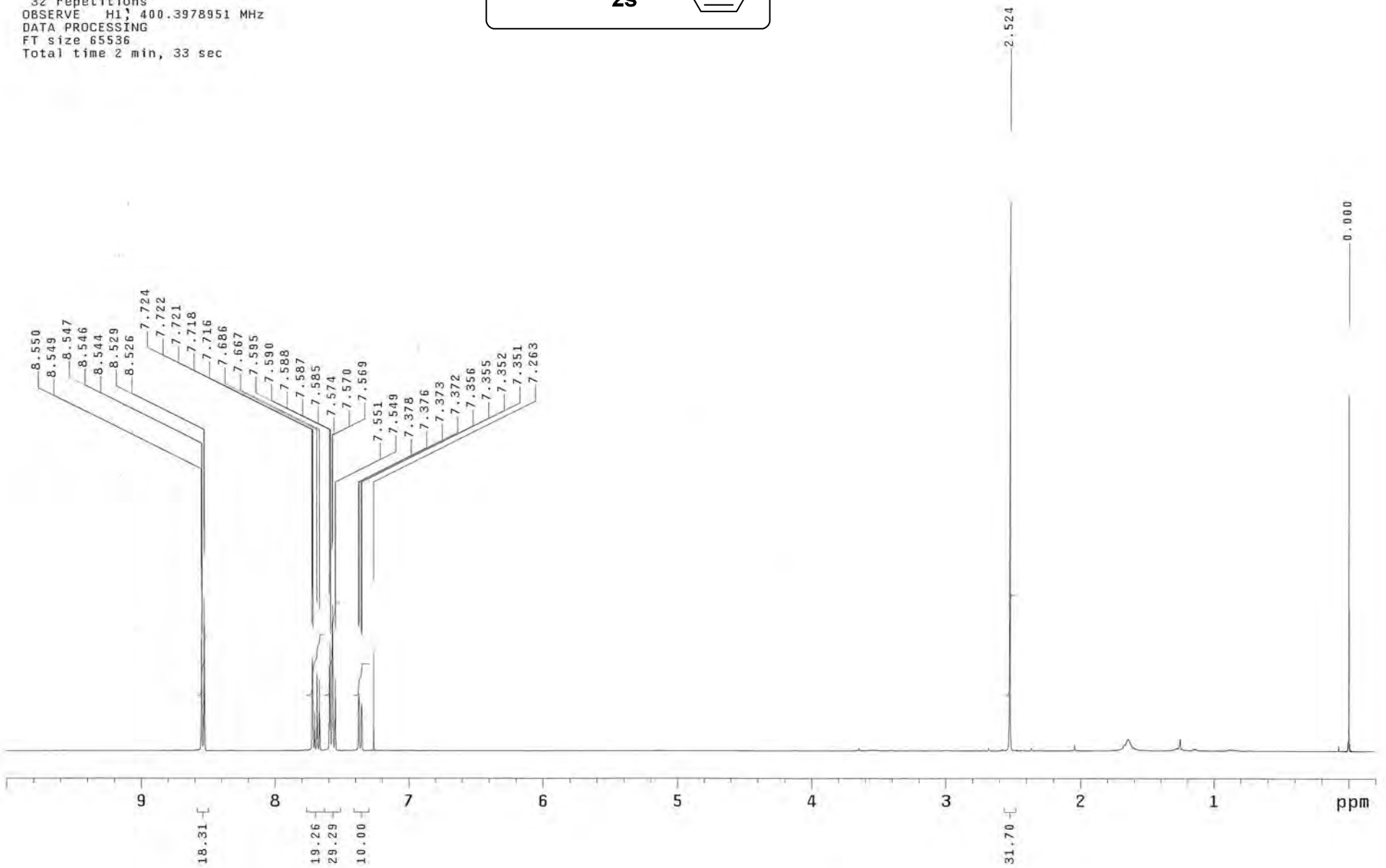
SIVA-RP57

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.000 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978951 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec



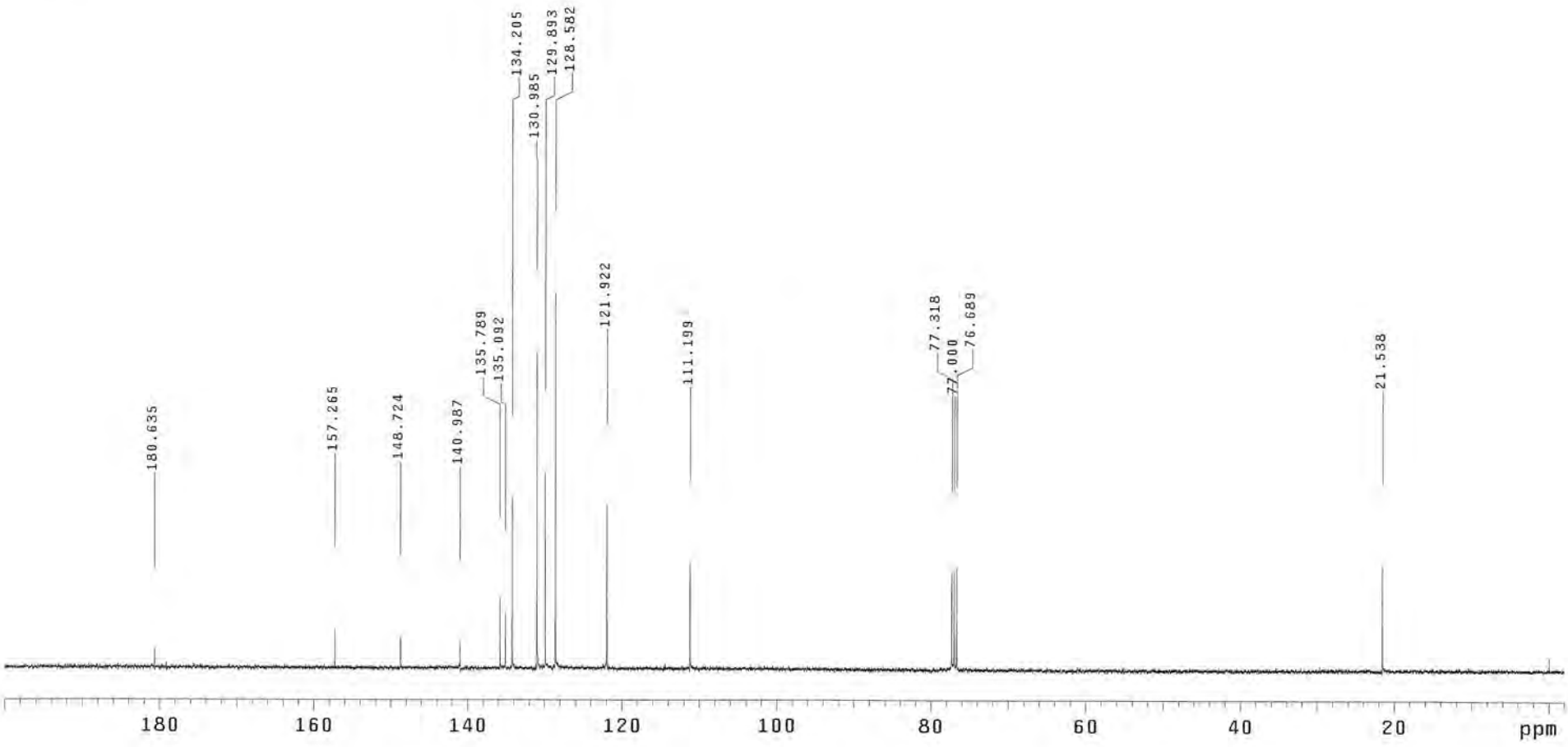
2s



SIVA-RP3-57

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 2064 repetitions
OBSERVE C13, 100.6801323 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 10 hr, 55 min, 47 sec

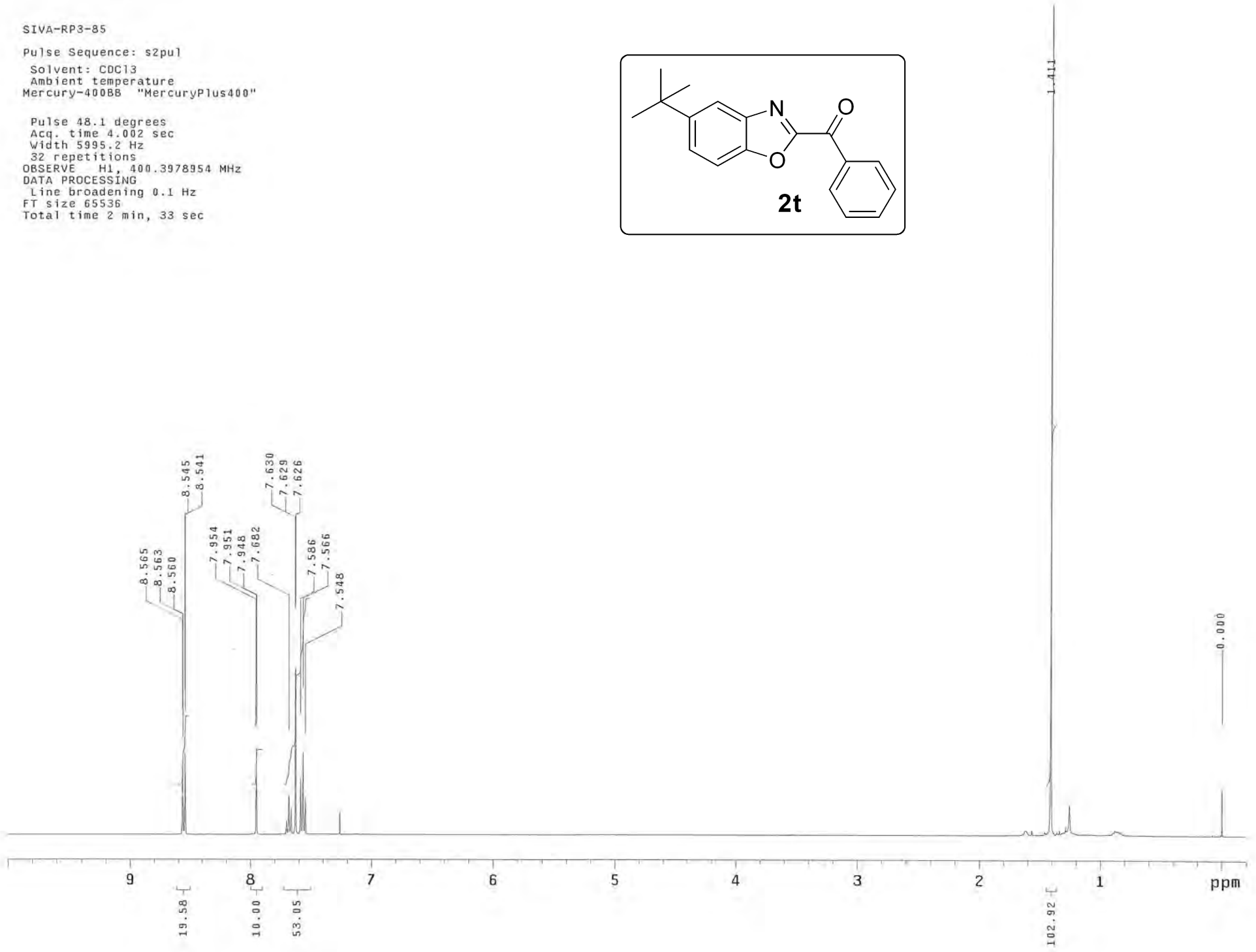


SIVA-RP3-85

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978954 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 2 min, 33 sec

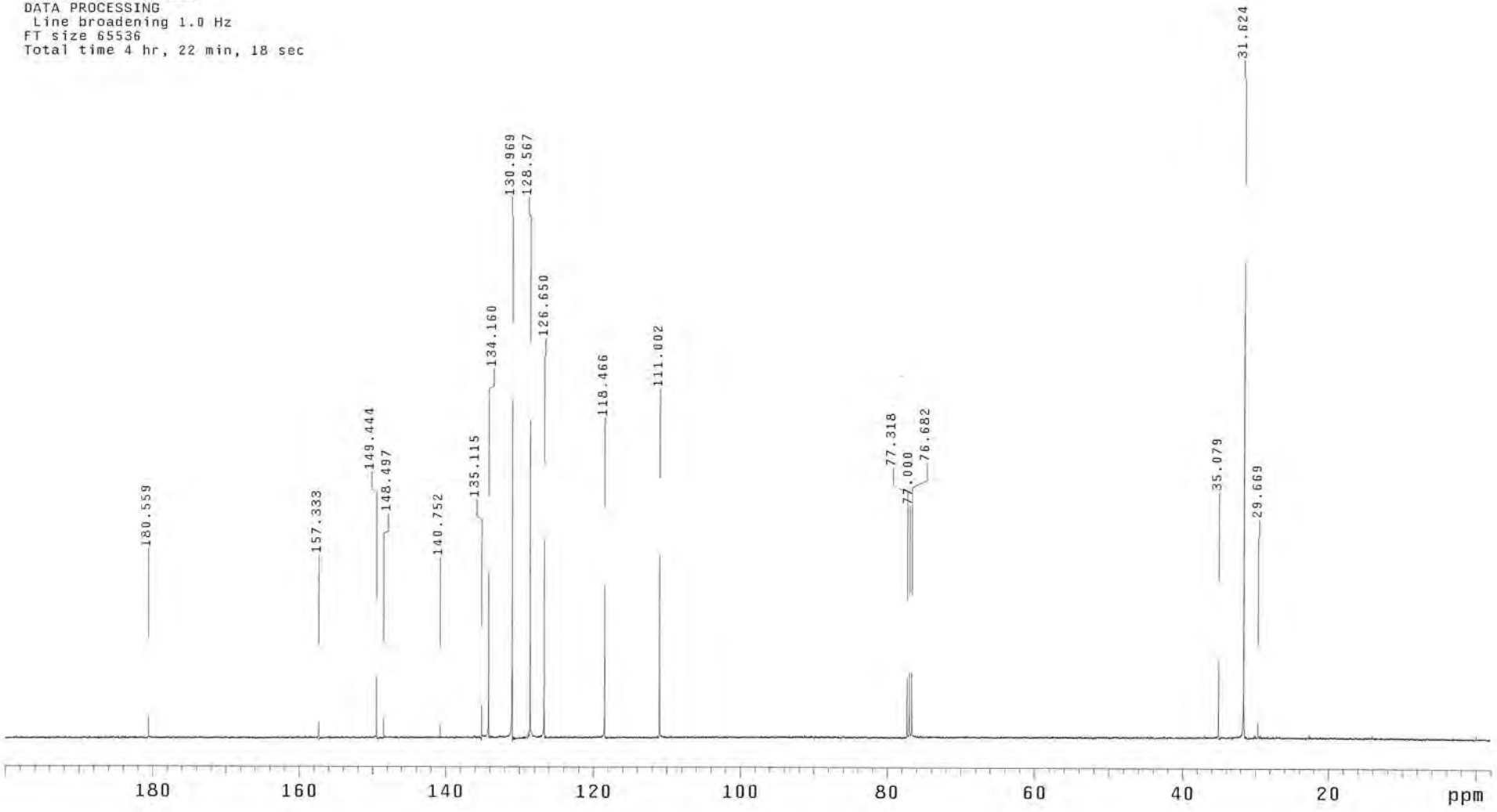
2t



SIVA-RP3-85

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

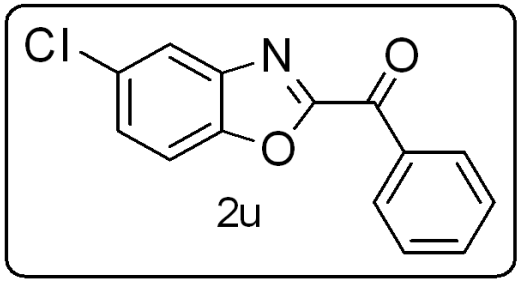
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
3328 repetitions
OBSERVE C13, 100.6801330 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 4 hr, 22 min, 18 sec

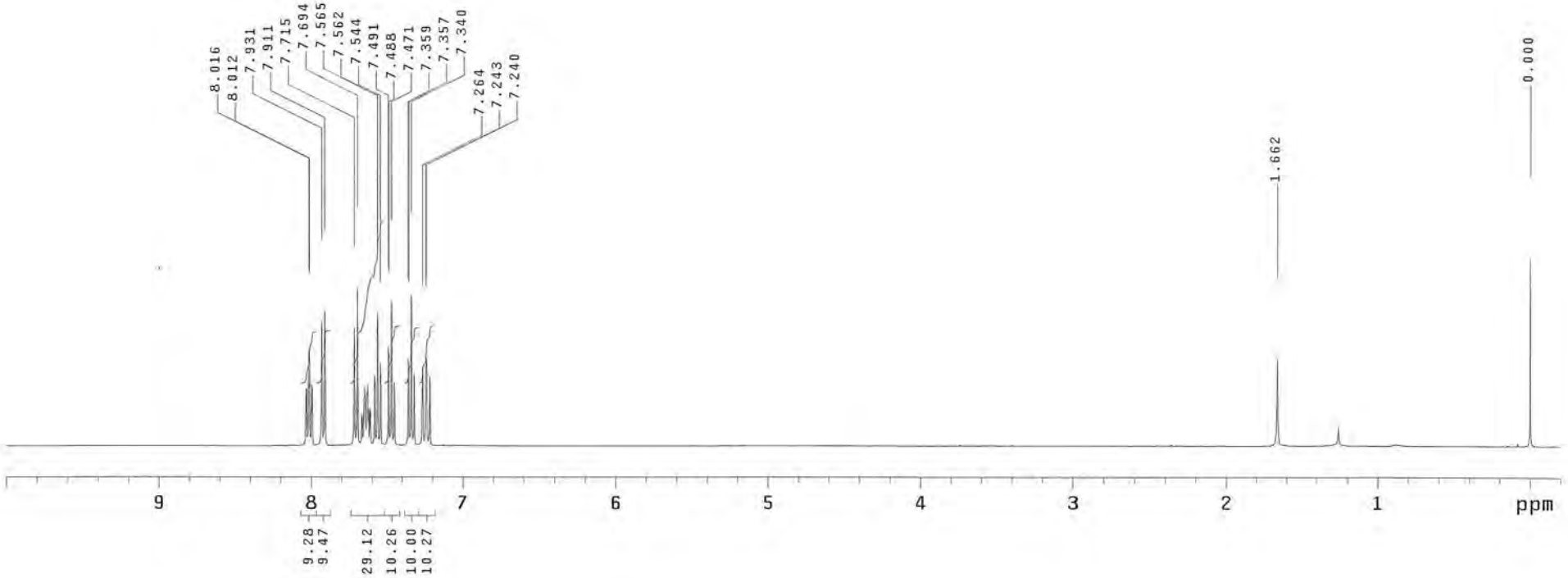


SIVA-RP3-39

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978927 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec

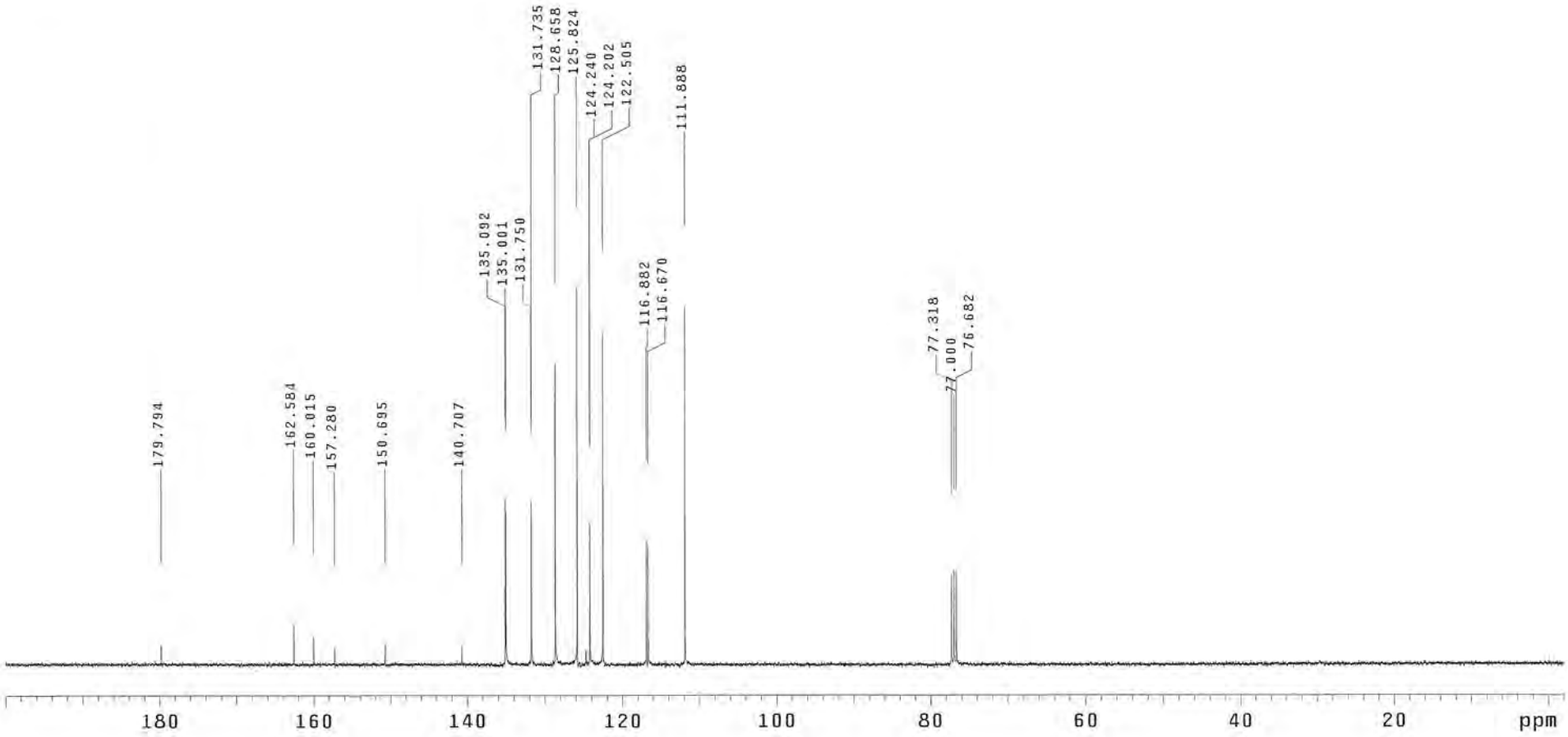




SIVA-RP3-39

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

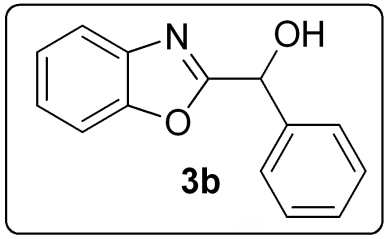
 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 2464 repetitions
OBSERVE C13, 100.6801346 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



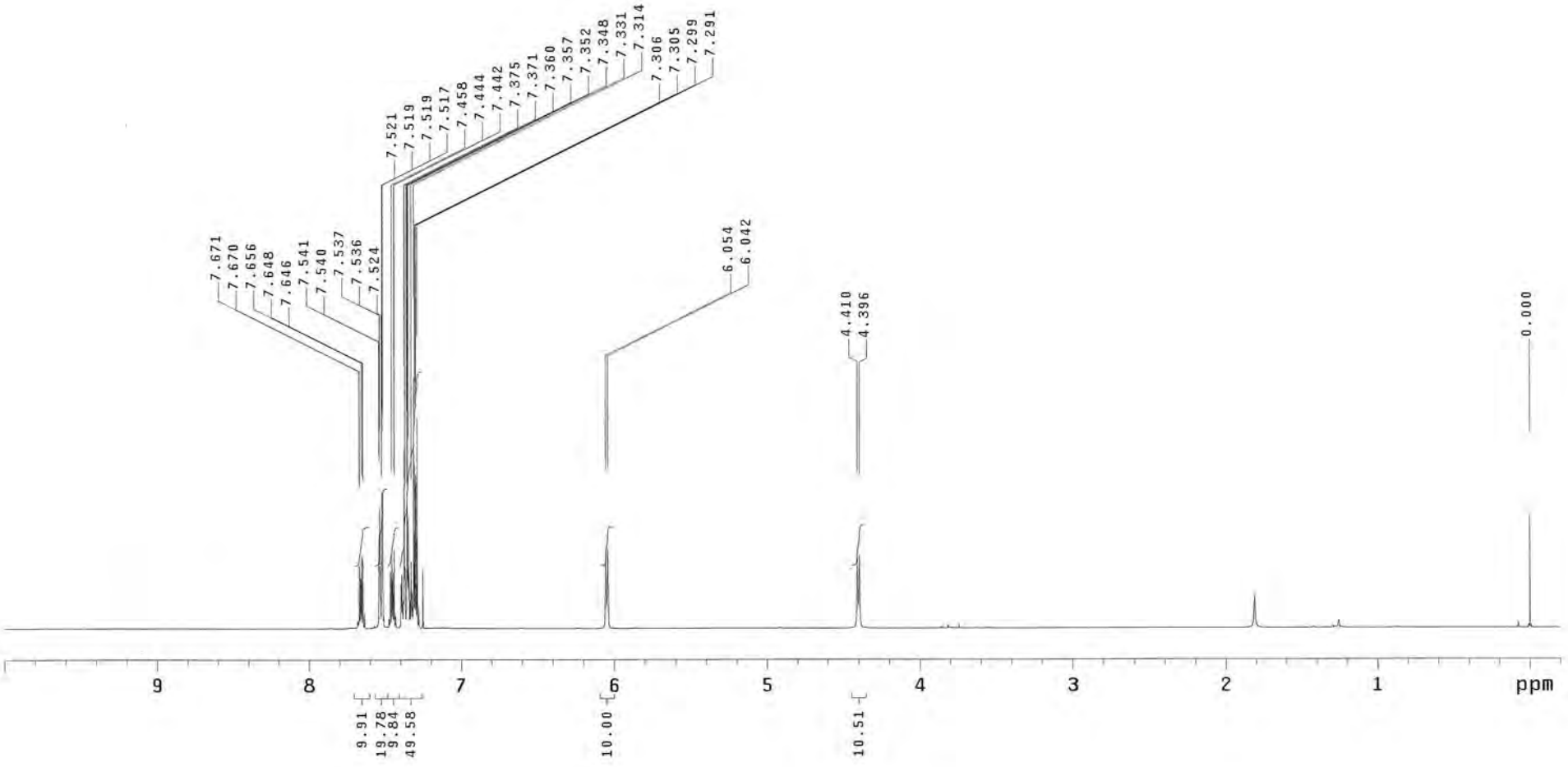
SIVA-RP5-10

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 4 2014
Solvent: CDCl3
Ambient temperature
Total 28 repetitions



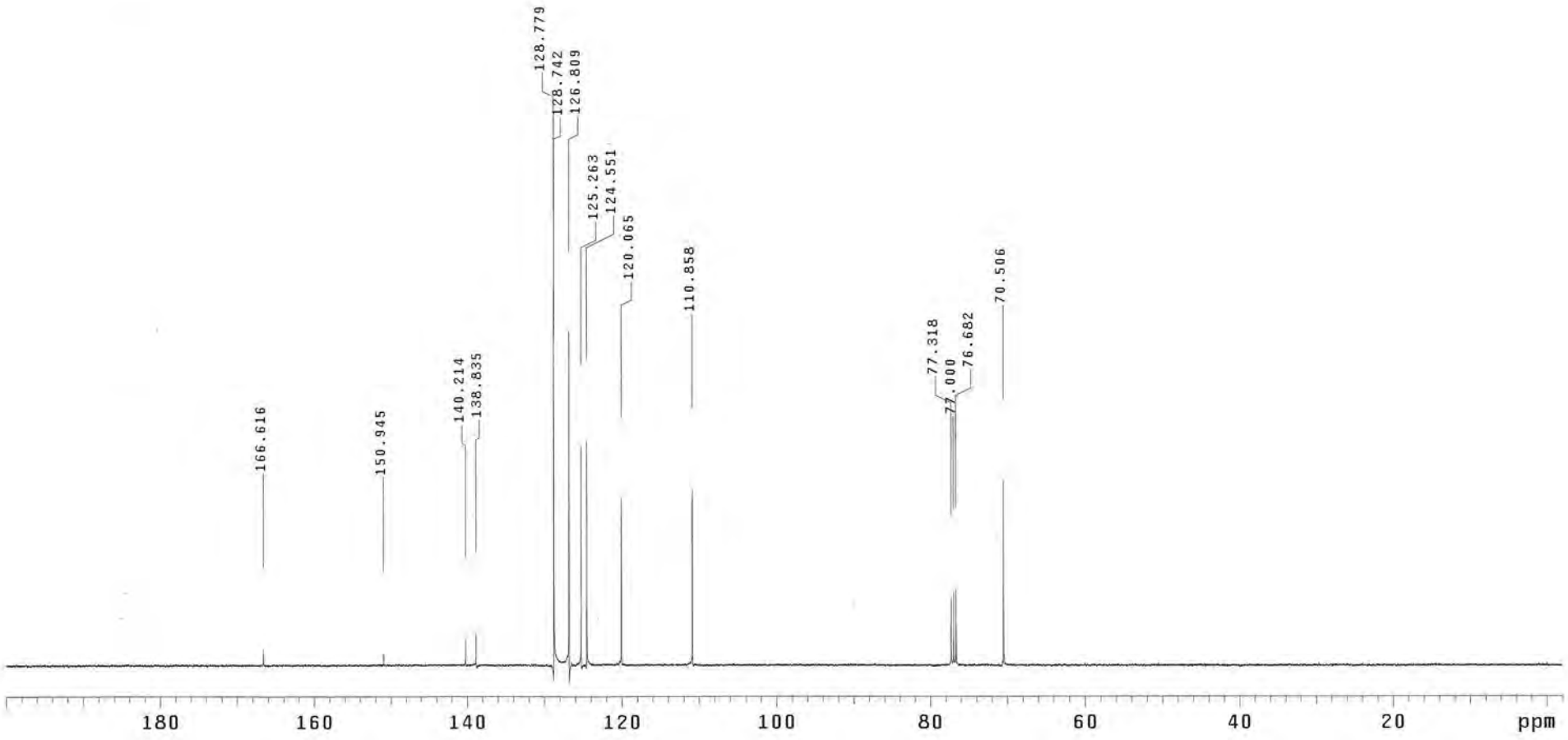
3b



SIVA-RP5-10

Pulse Sequence: s2pul

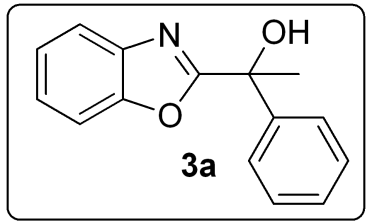
Mercury-400BB "MercuryPlus400"
Date: Mar 4 2014
Solvent: CDCl3
Ambient temperature
Total 2848 repetitions

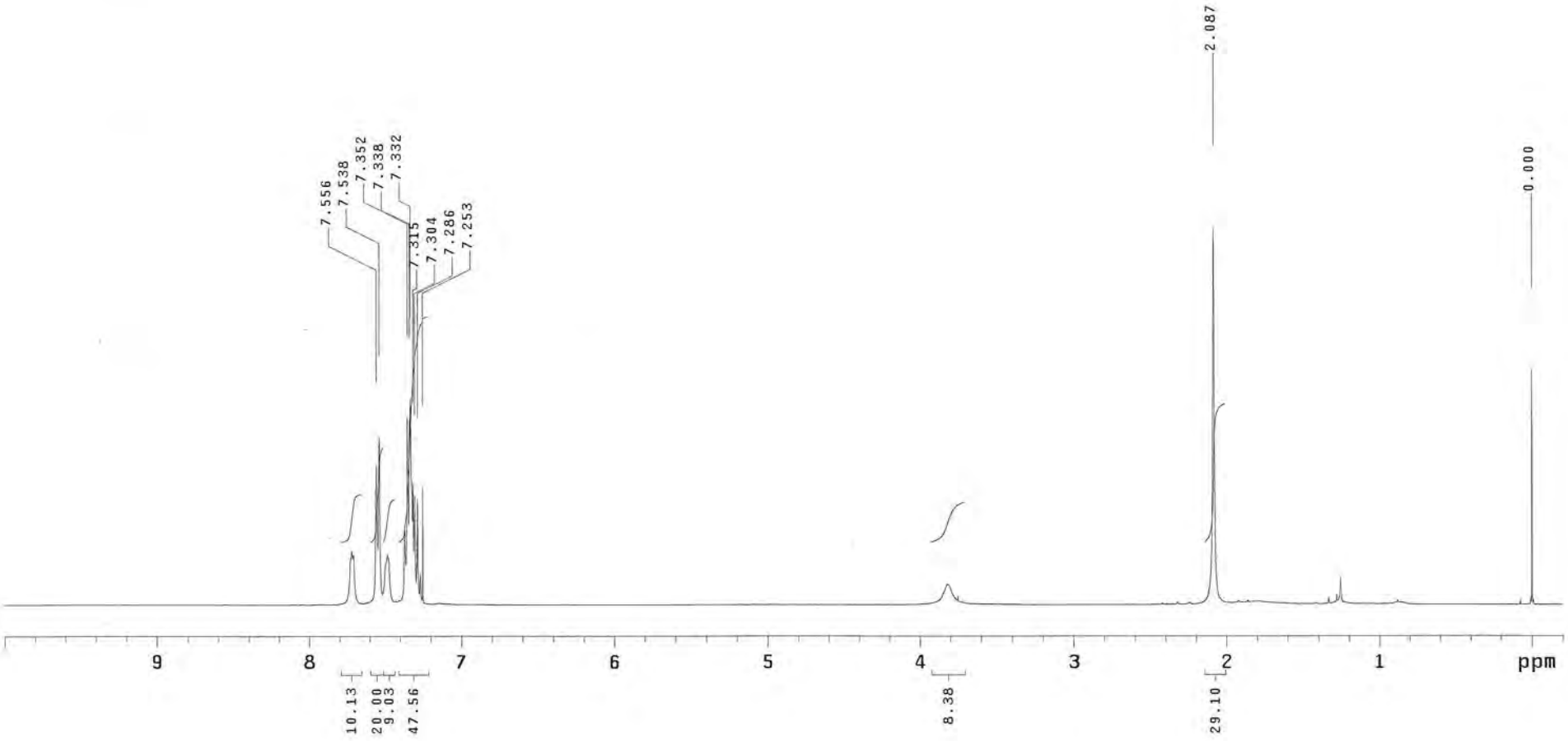


SIVA-RP4-183

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

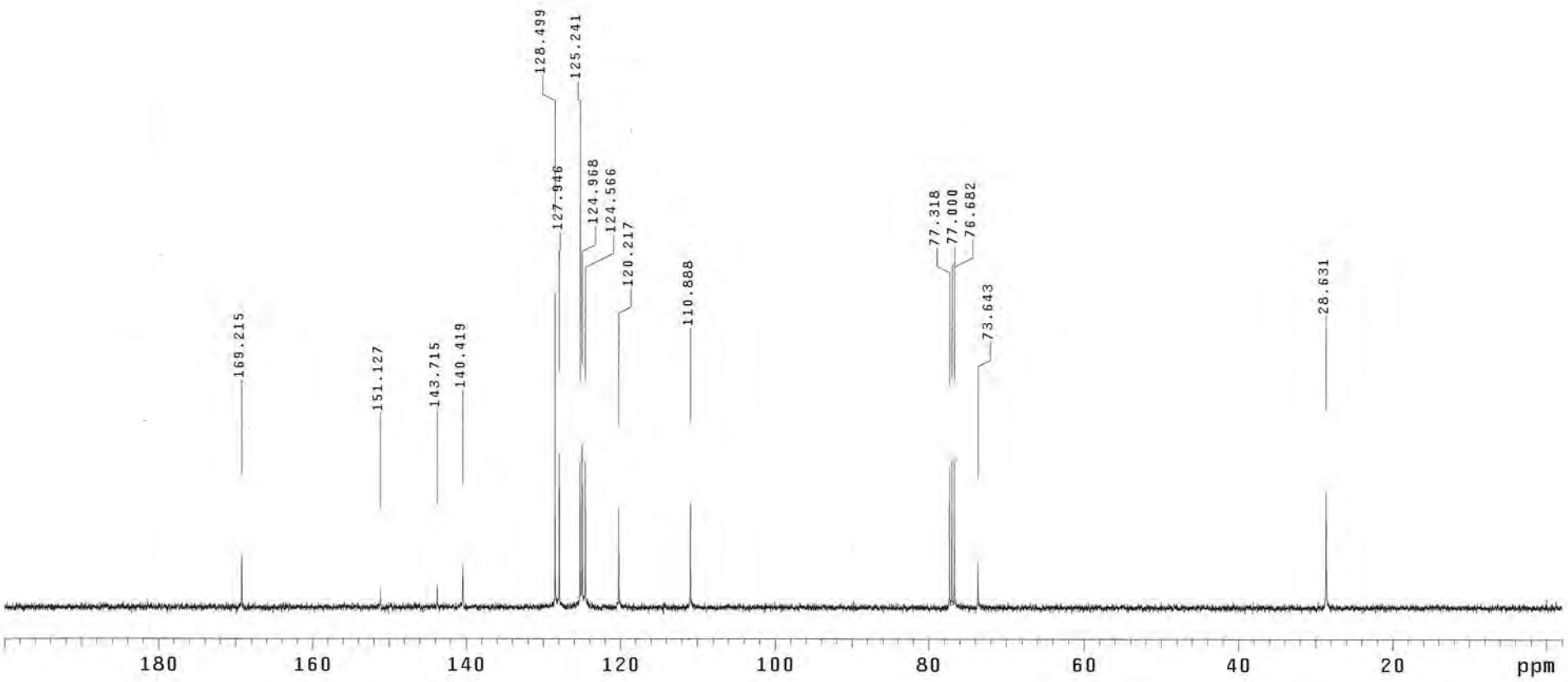




SIVA-RP4-183

Pulse Sequence: s2pul

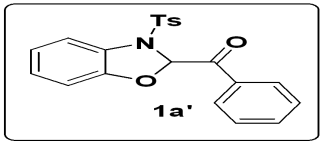
Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 3040 repetitions

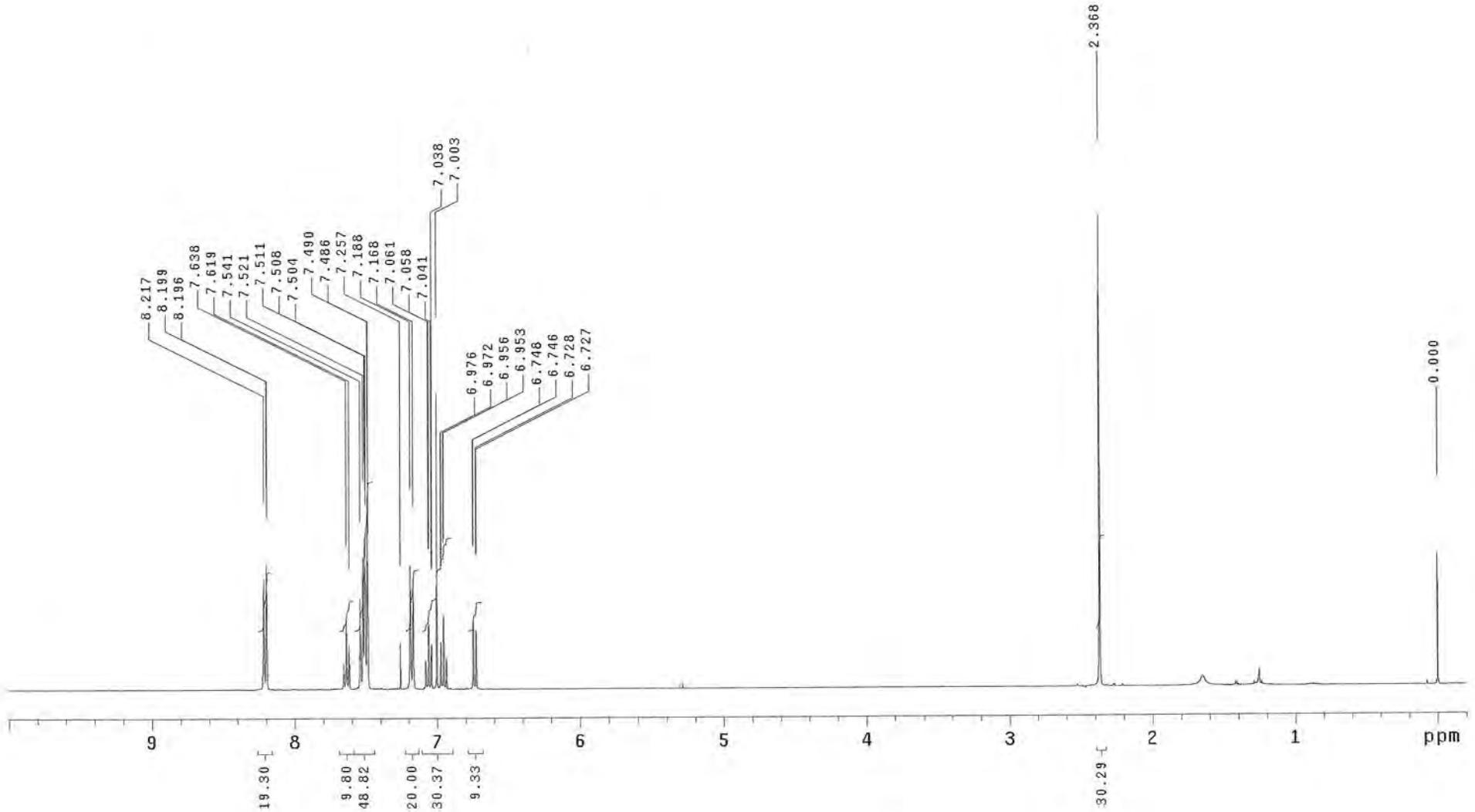


SIVA-RP5-121B

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 29 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

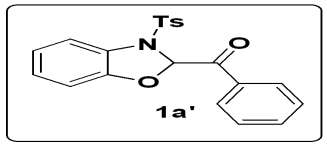


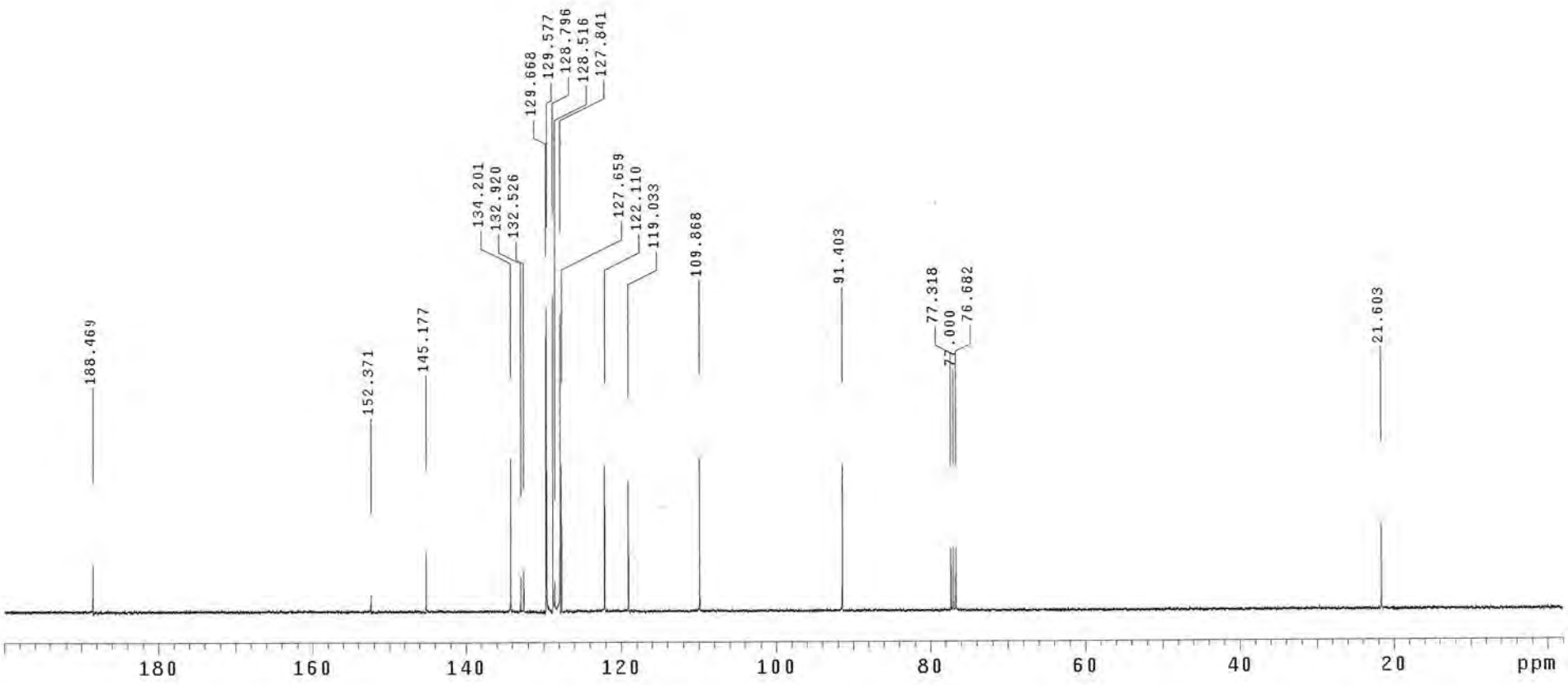


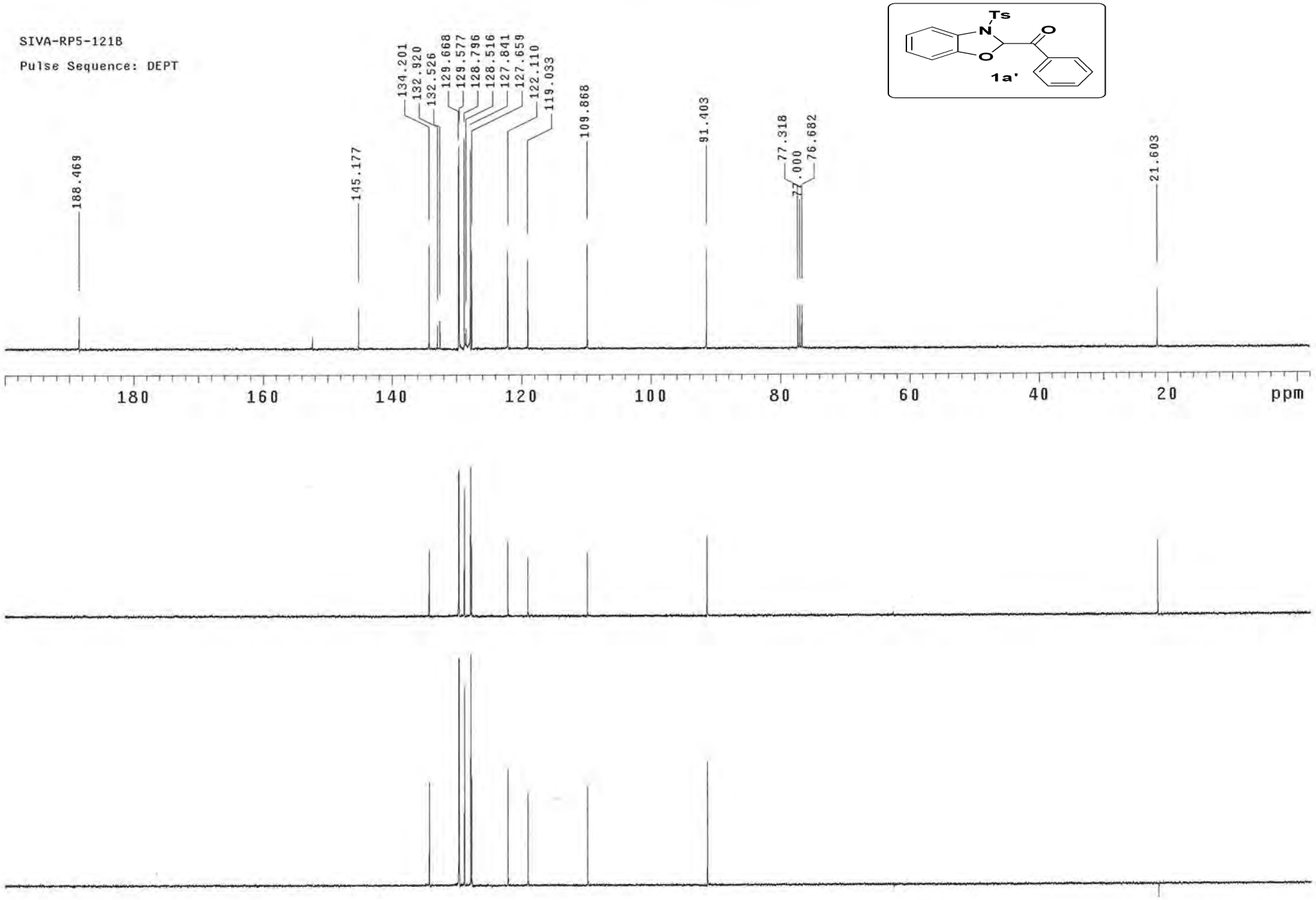
SIVA-RP5-121B

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 29 2014
Solvent: CDCl3
Ambient temperature
Total 3008 repetitions



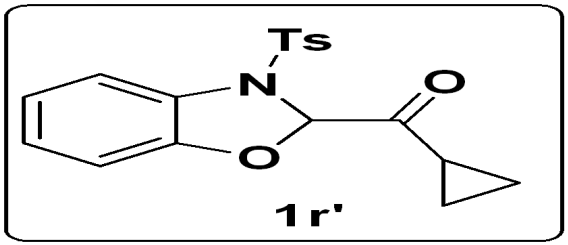


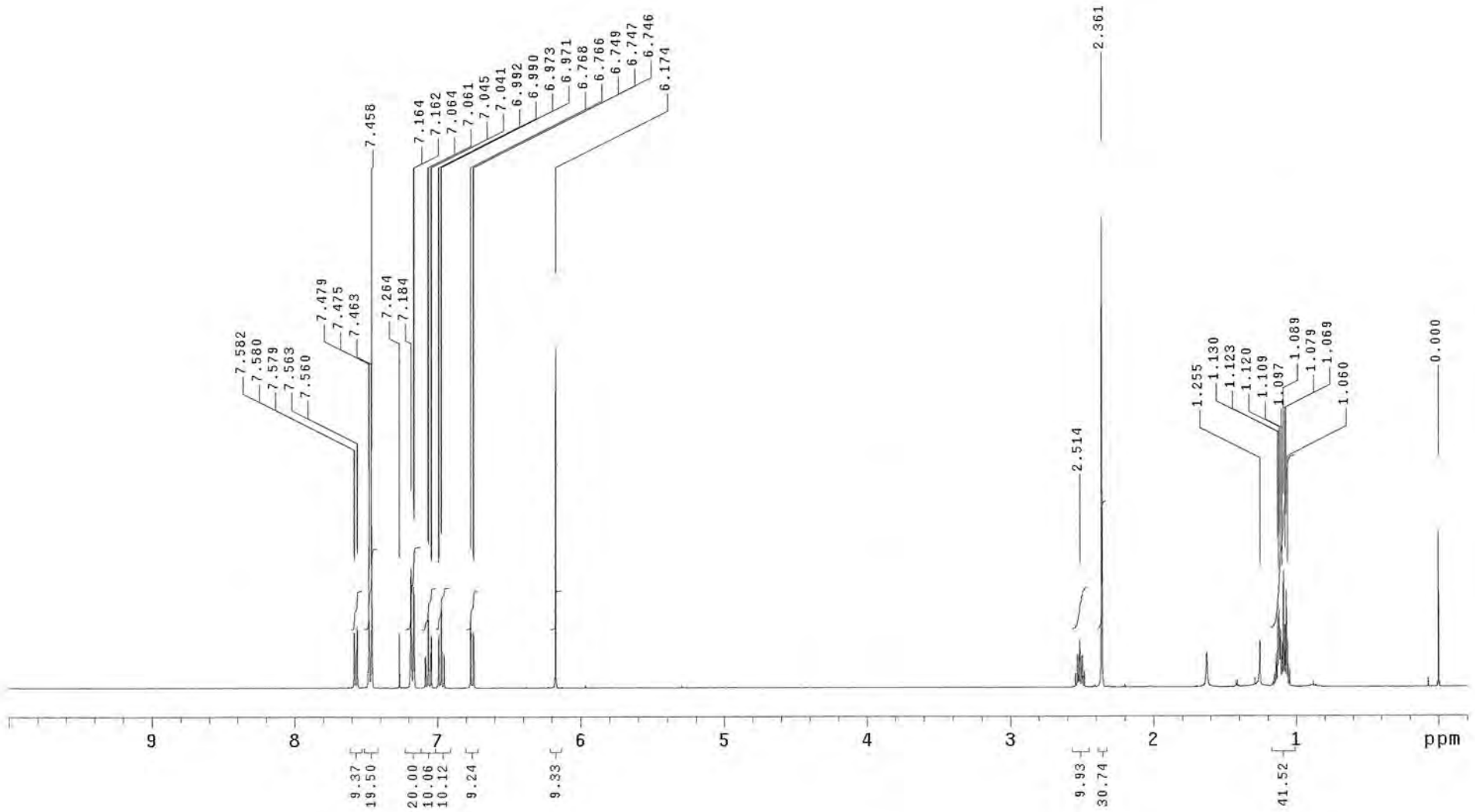

SIVA-RP5-121B
Pulse Sequence: DEPT
Ts
N
O
O
1a'
188.469
145.177
134.201
132.920
132.526
129.668
129.577
128.796
128.516
127.841
127.659
122.110
119.033
109.868
91.403
77.318
77.000
76.682
21.603
180
160
140
120
100
80
60
40
20
ppm

SIVA-RP5-122-A

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 29 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

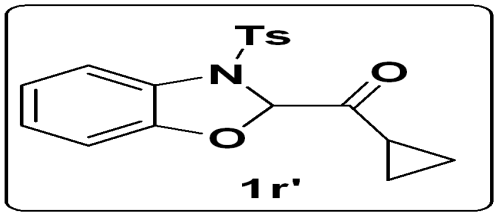




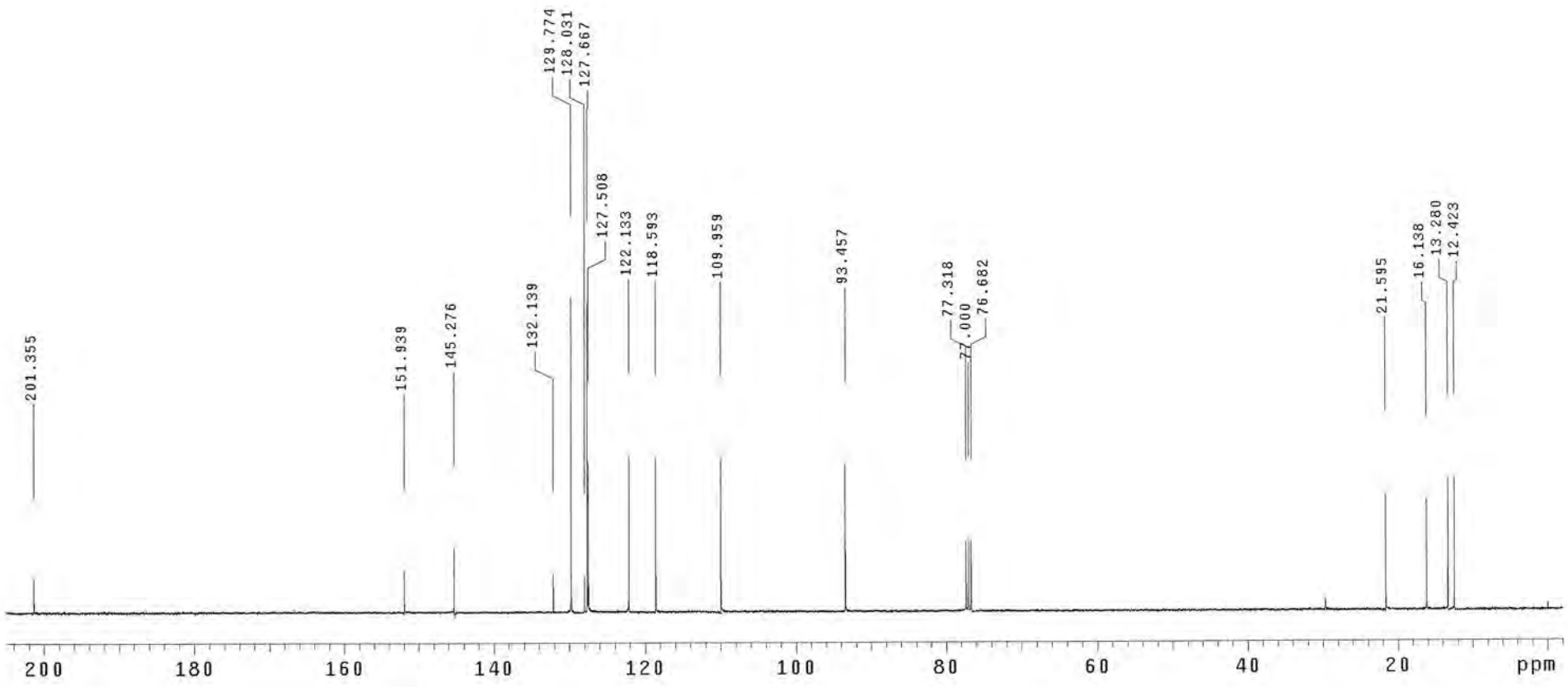
SIVA-RP5-122-A

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Apr 29 2014
Solvent: CDCl3
Ambient temperature
Total 6272 repetitions



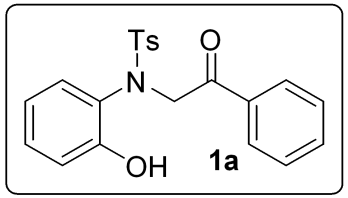
1r'



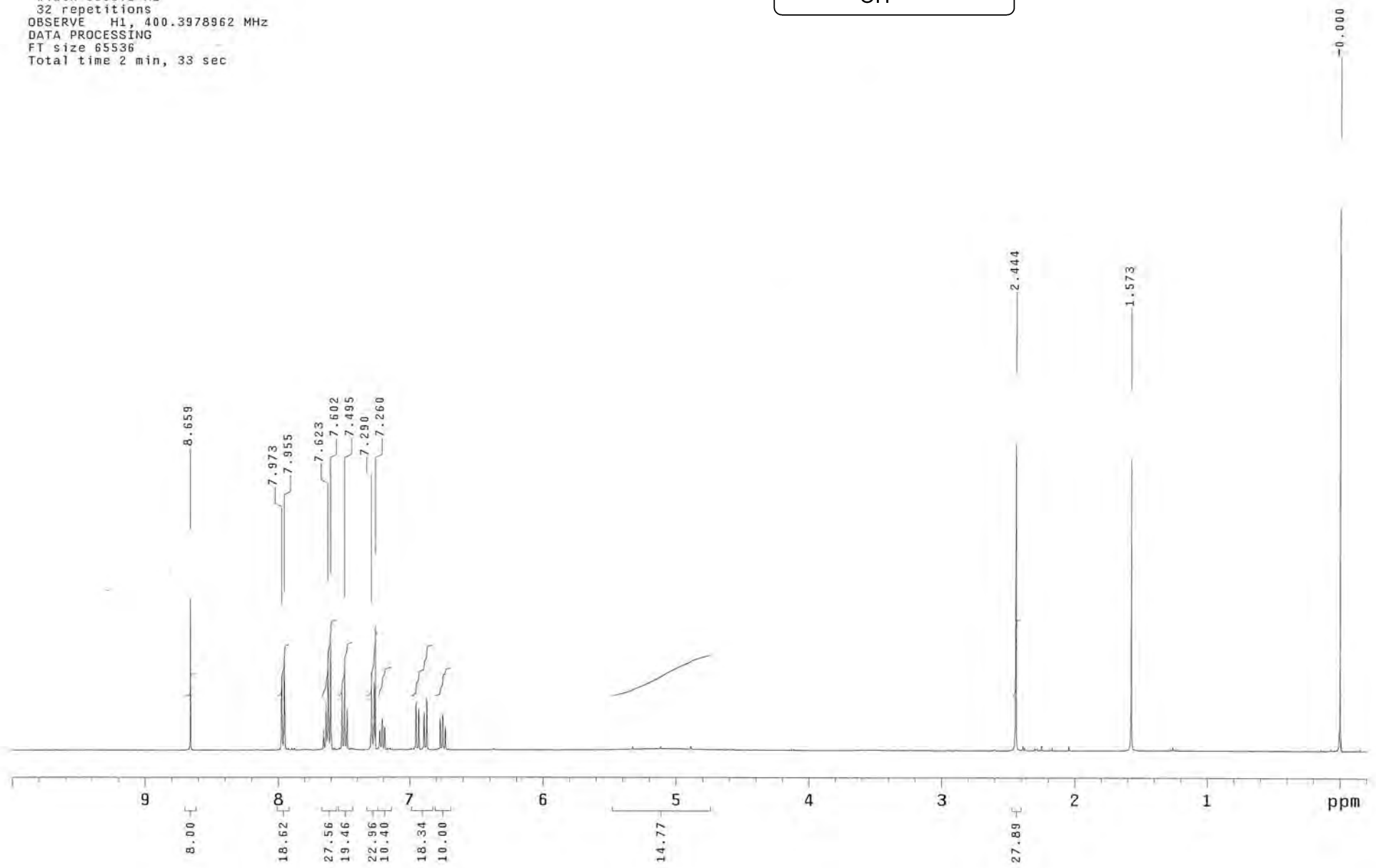
SIVA-RP3-29

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.000 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978962 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec



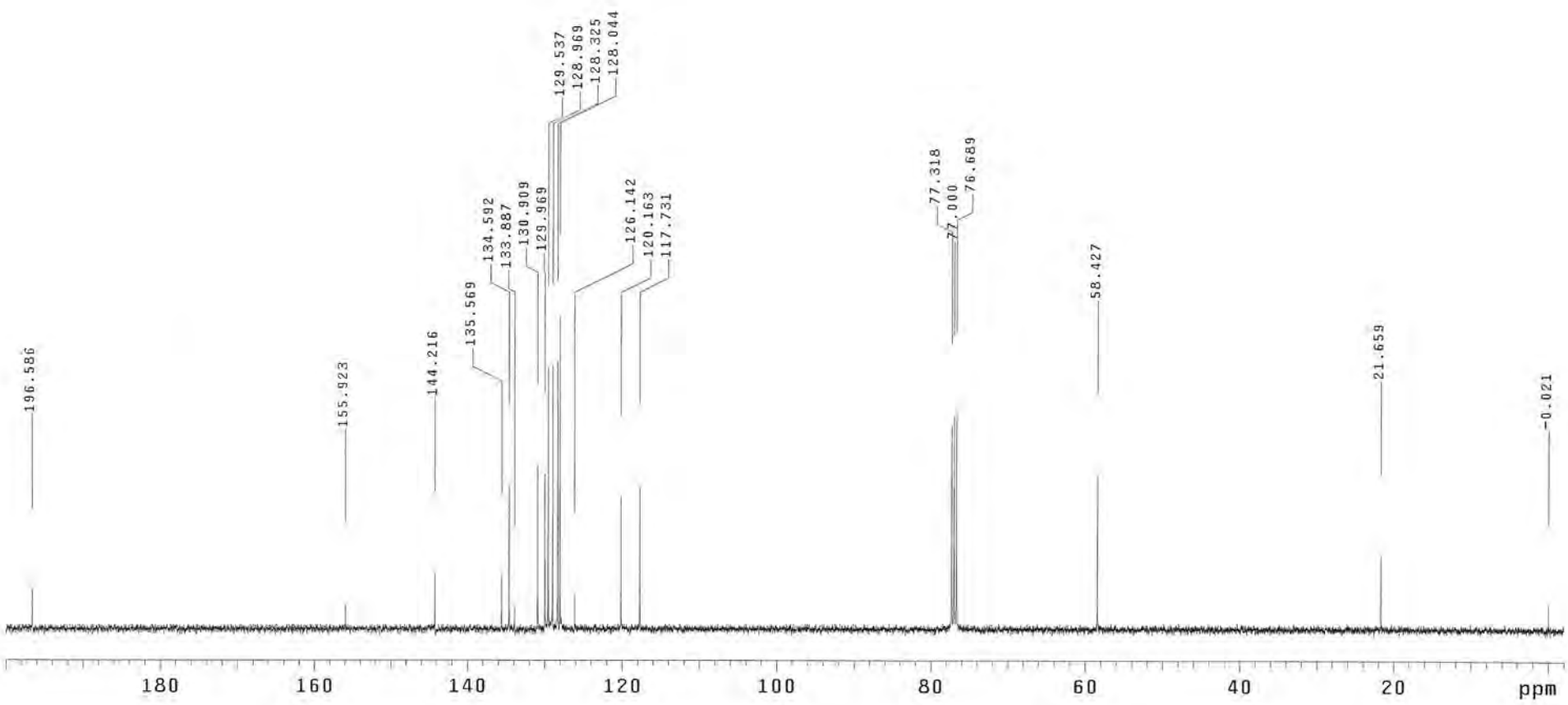
1a



SIVA-RP3-29

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

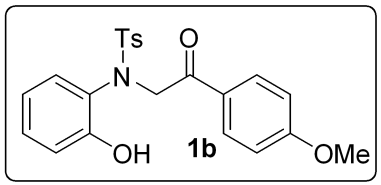
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
3376 repetitions
OBSERVE C13, 100.6801315 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec

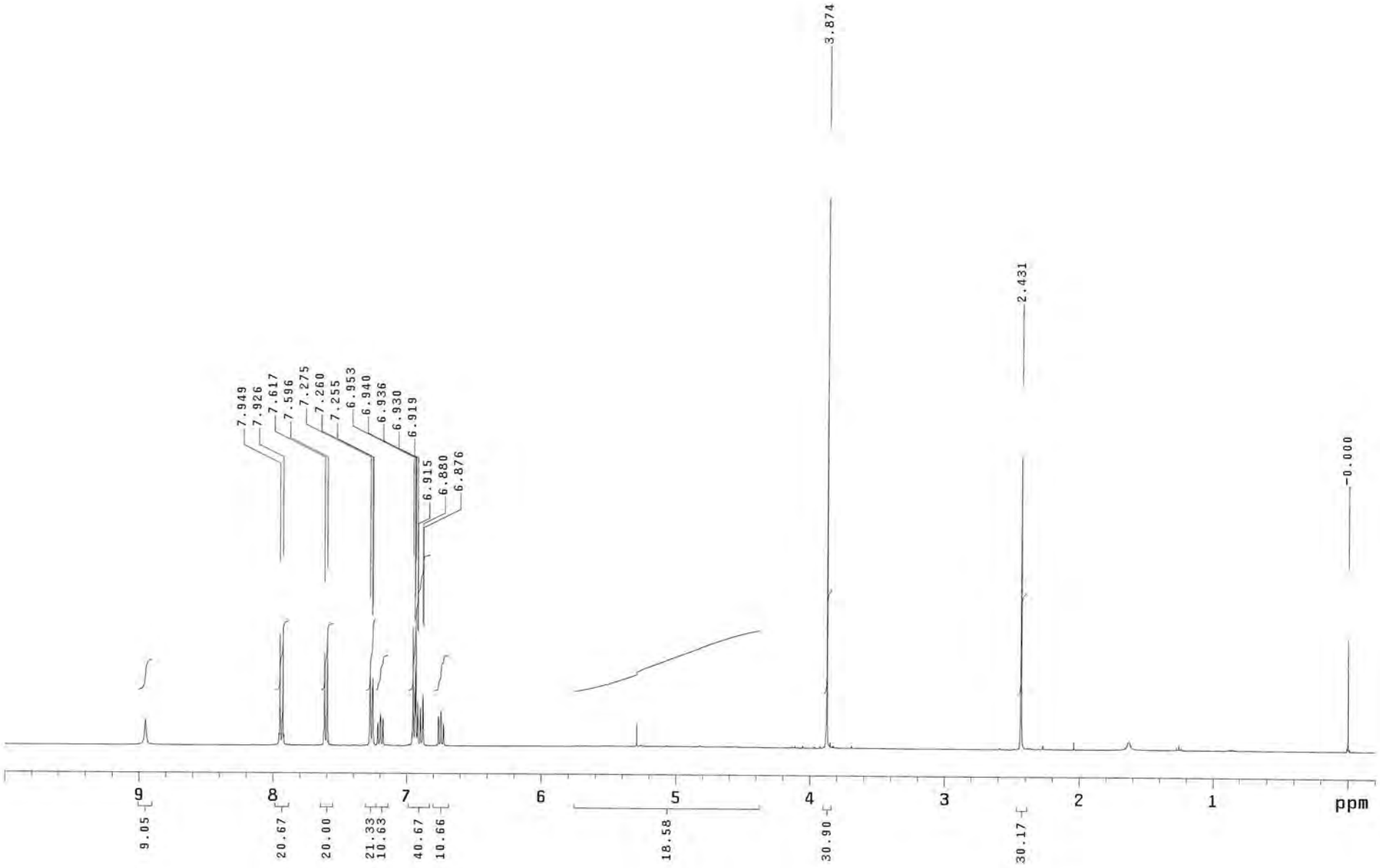


SIVA-RP4-186

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: May 2 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

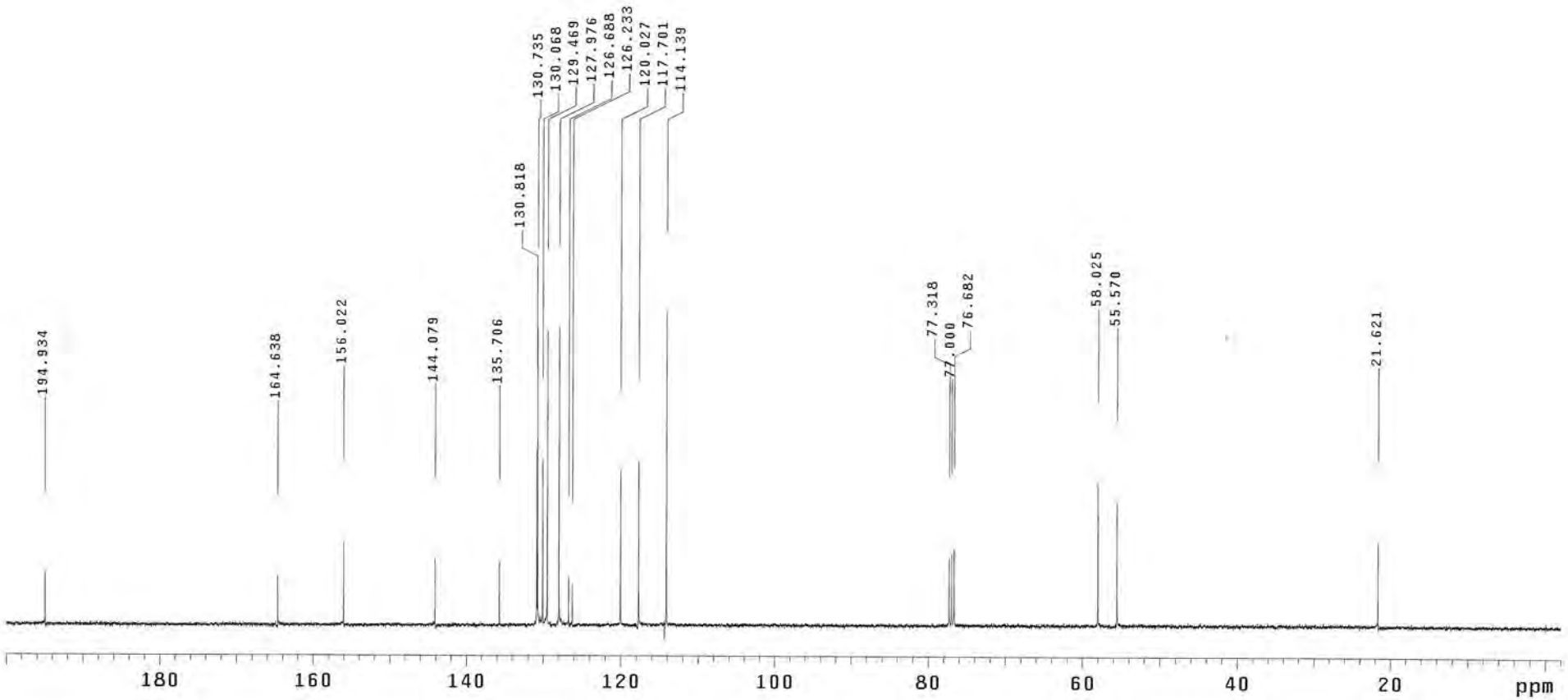




SIVA-RP4-186

Pulse Sequence: s2pul

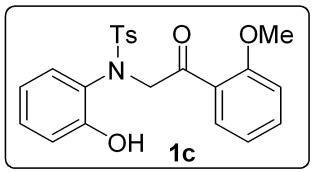
Mercury-400BB "MercuryPlus400"
Date: May 2 2014
Solvent: CDCl3
Ambient temperature
Total 1344 repetitions



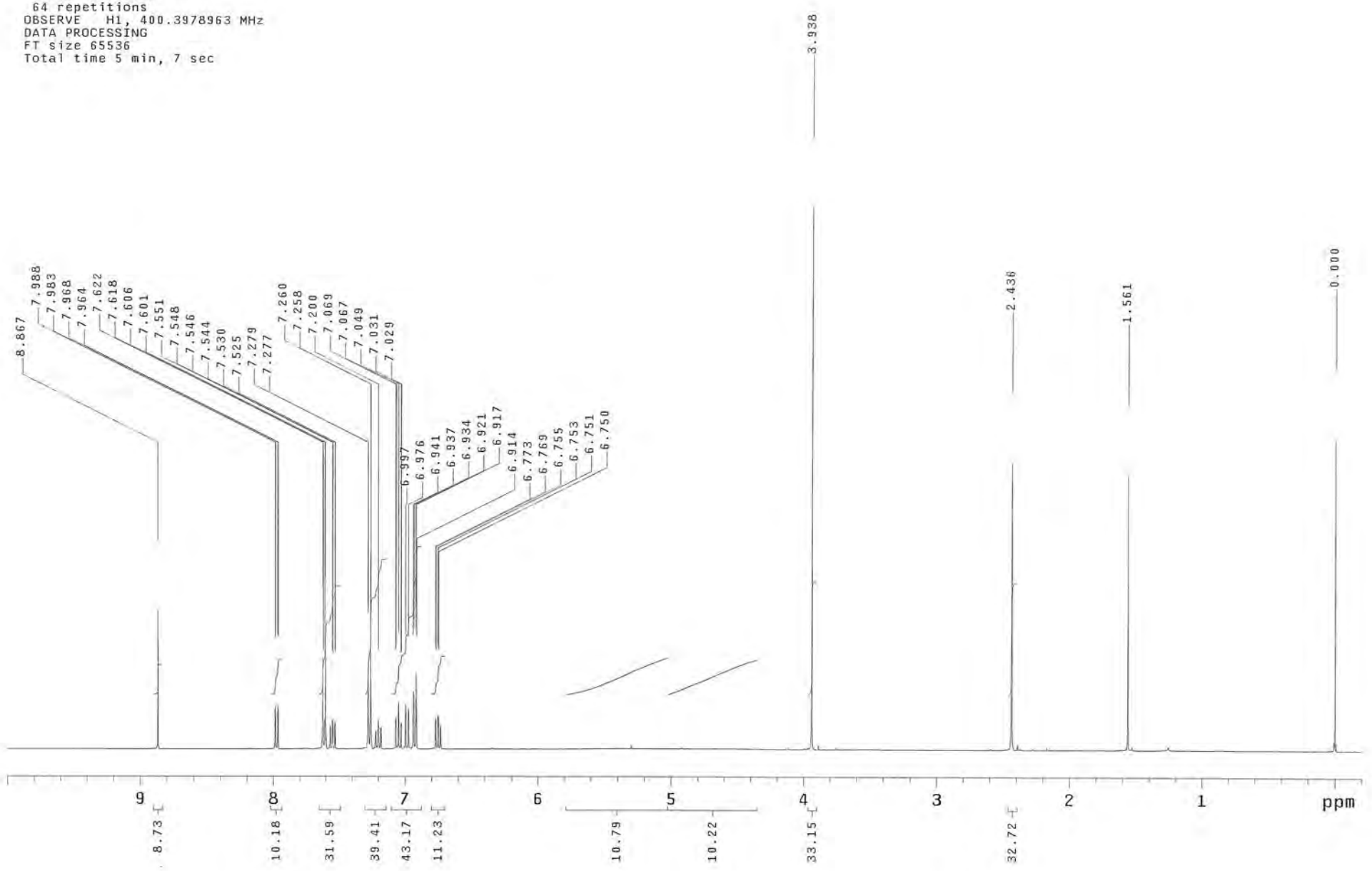
SIVA-RP3-114

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature.
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
64 repetitions
OBSERVE H1, 400.3978963 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec



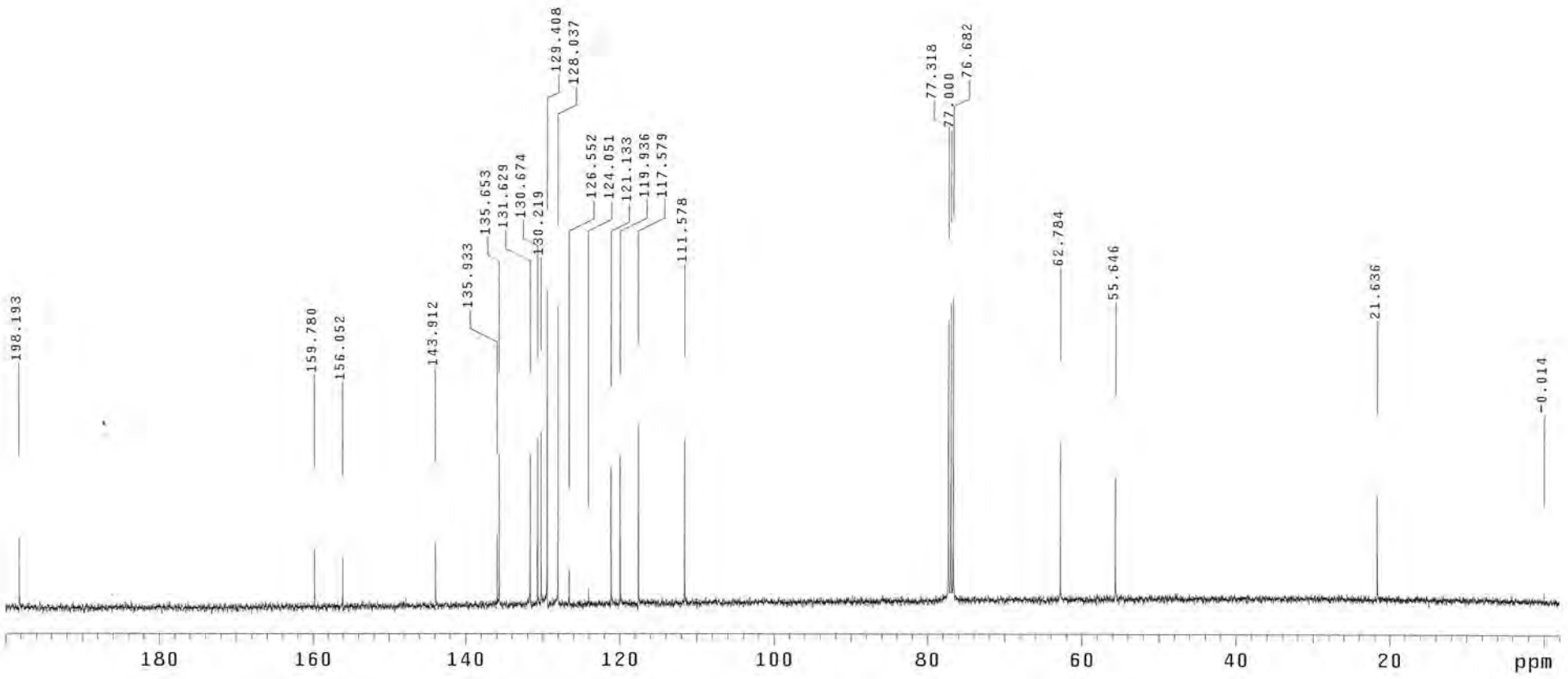
1c



SIVA-RP3-114

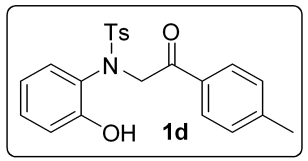
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

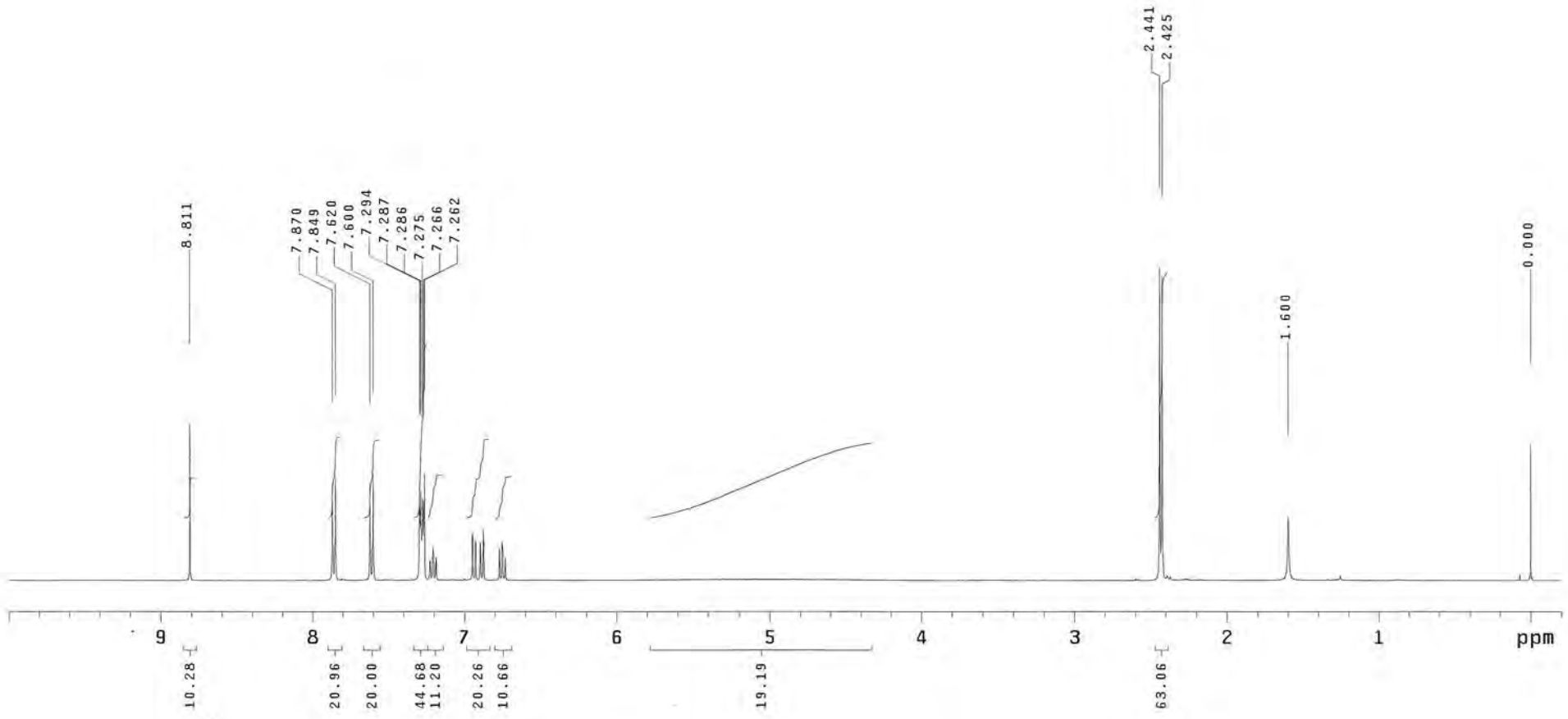
 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 5312 repetitions
OBSERVE C13, 100.6801315 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



SIVA-RP3-009

Pulse Sequence: s2pul
Mercury-400BB "MercuryPlus400"
Date: Mar 17 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

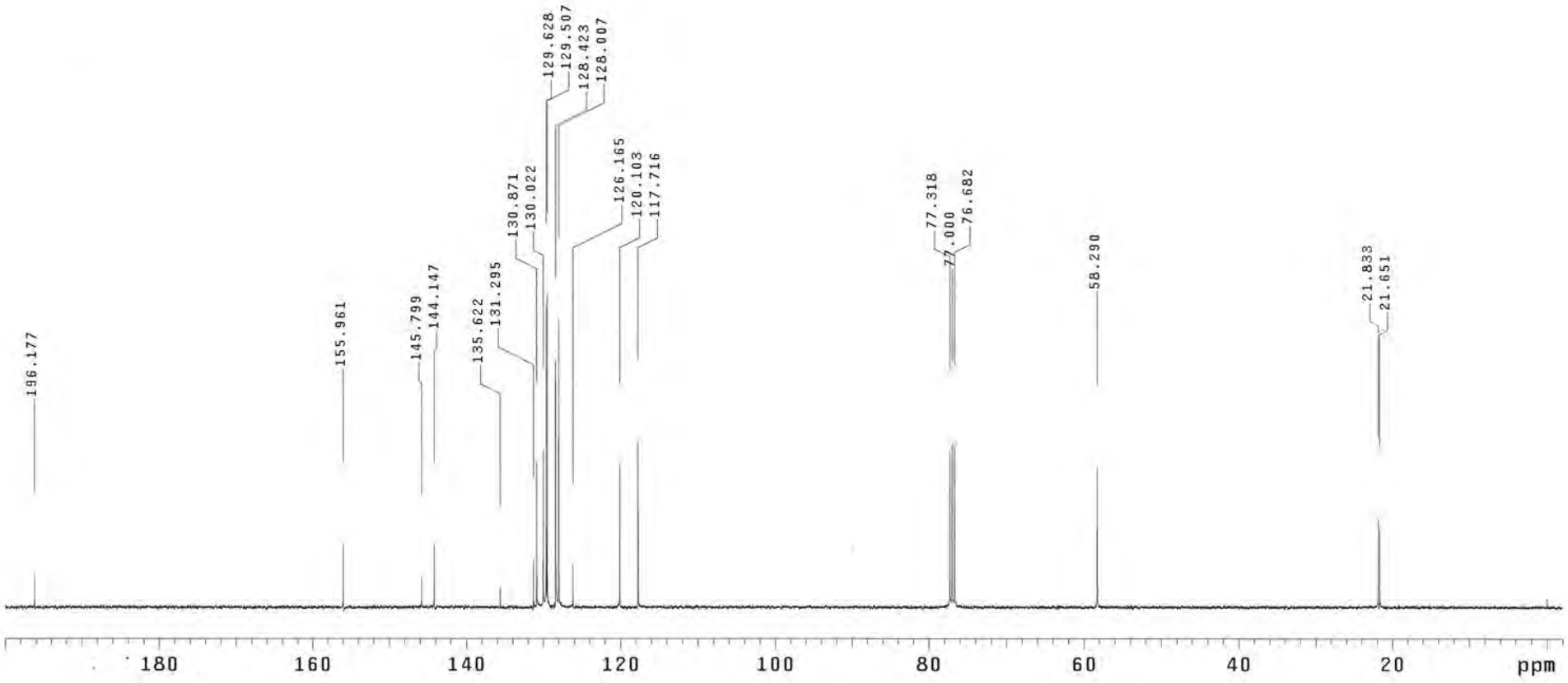




SIVA-RP3-009

Pulse Sequence: s2pul

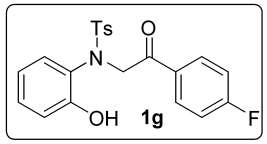
Mercury-400BB "MercuryPlus400"
Date: Mar 17 2014
Solvent: CDCl3
Ambient temperature
Total 6176 repetitions

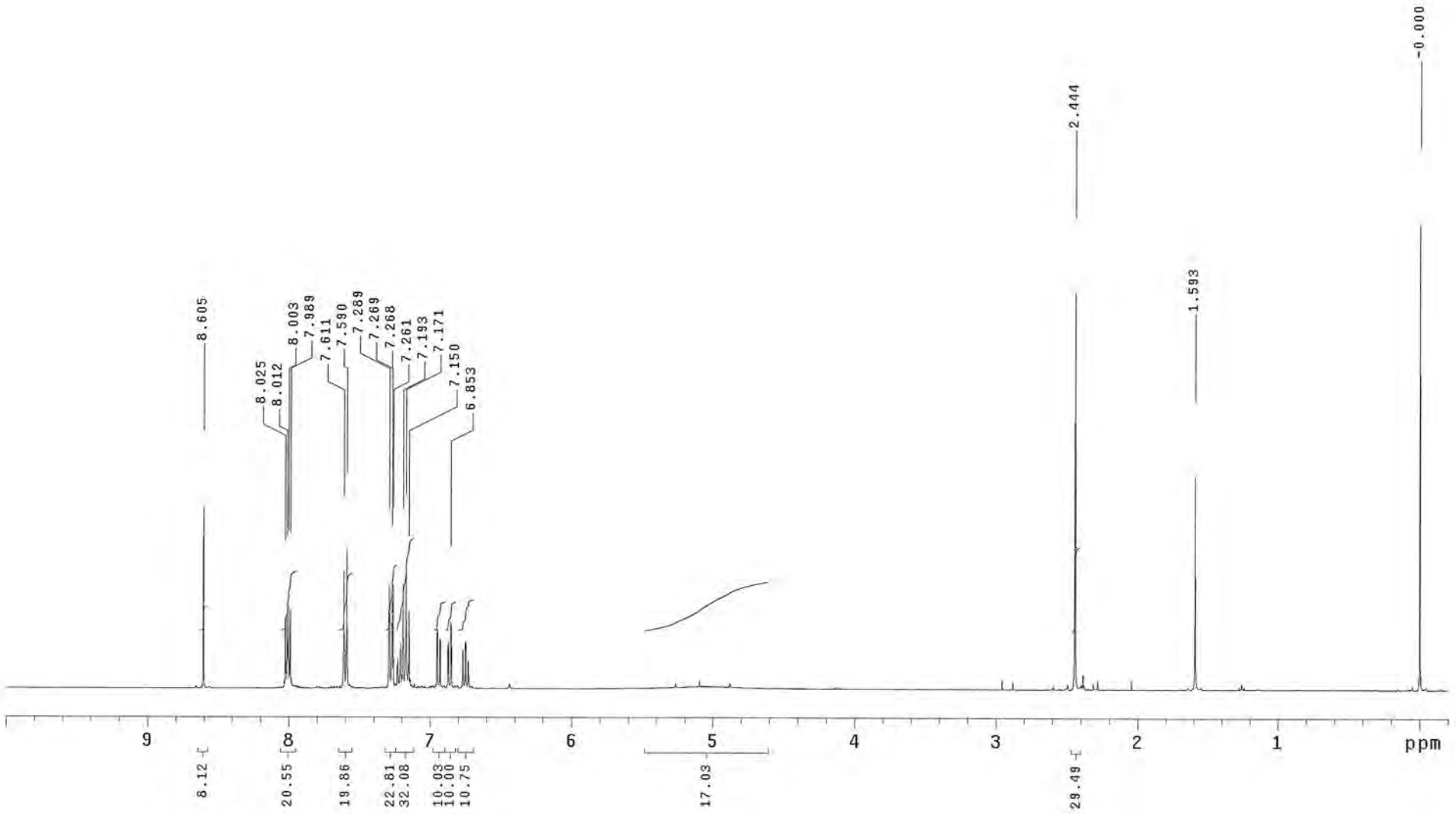


SIVA-RP3-64

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 19 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

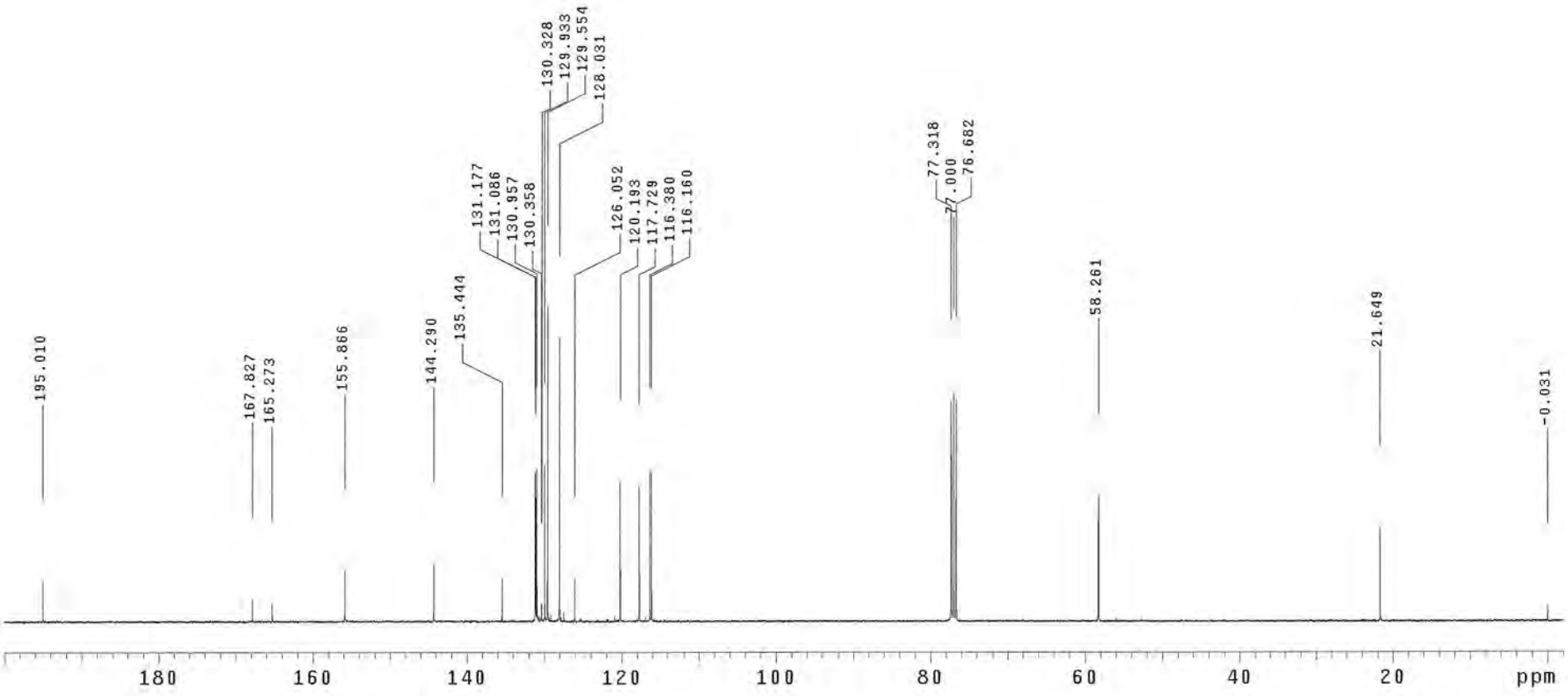




SIVA-RP3-64

Pulse Sequence: s2pul

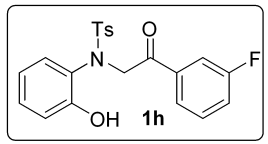
UNITYplus-400 "unity400"
Date: Aug 19 2013
Solvent: CDCl3
Ambient temperature
Total 32000 repetitions

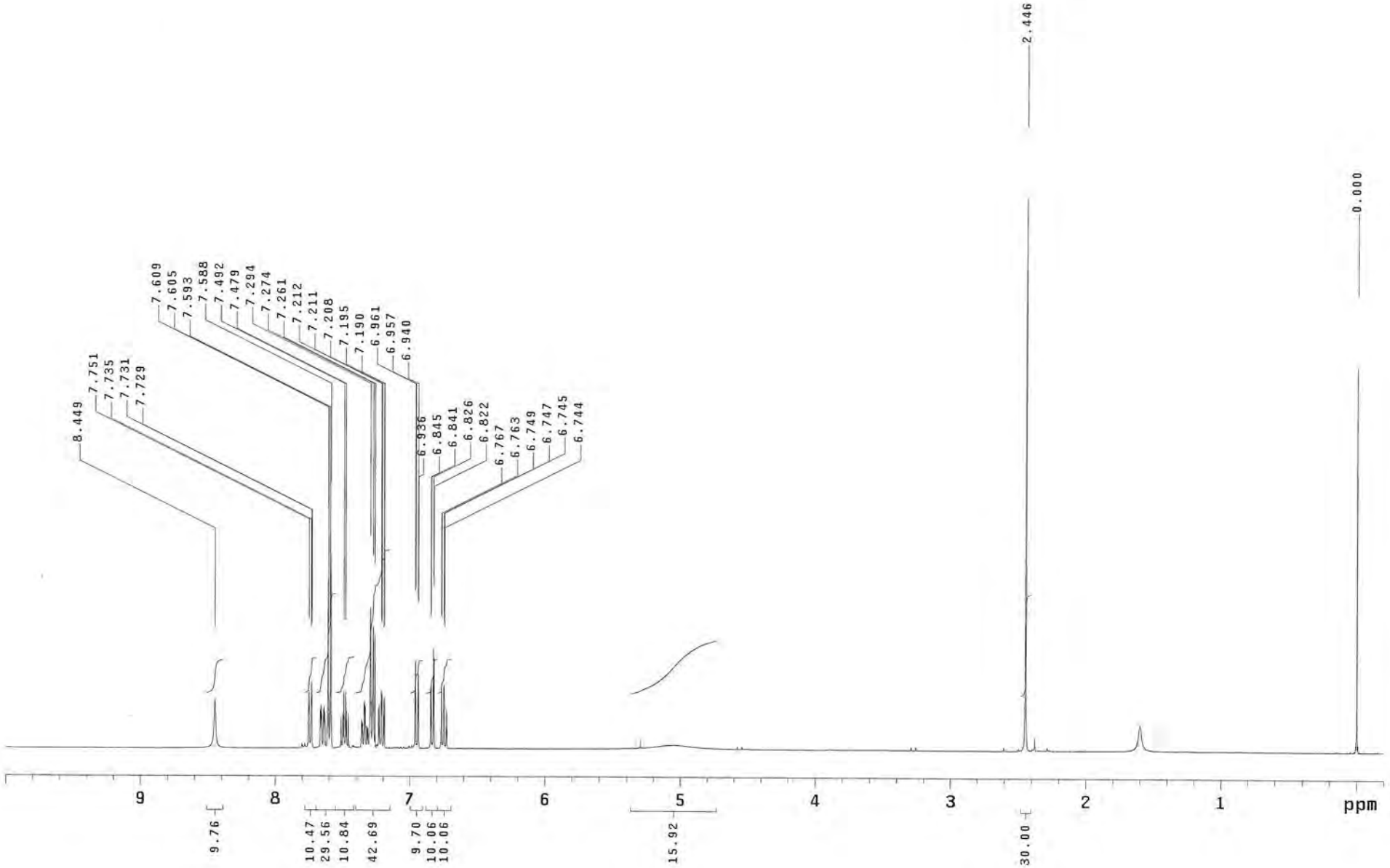


SIVA-RP4-121

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: May 2 2014
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

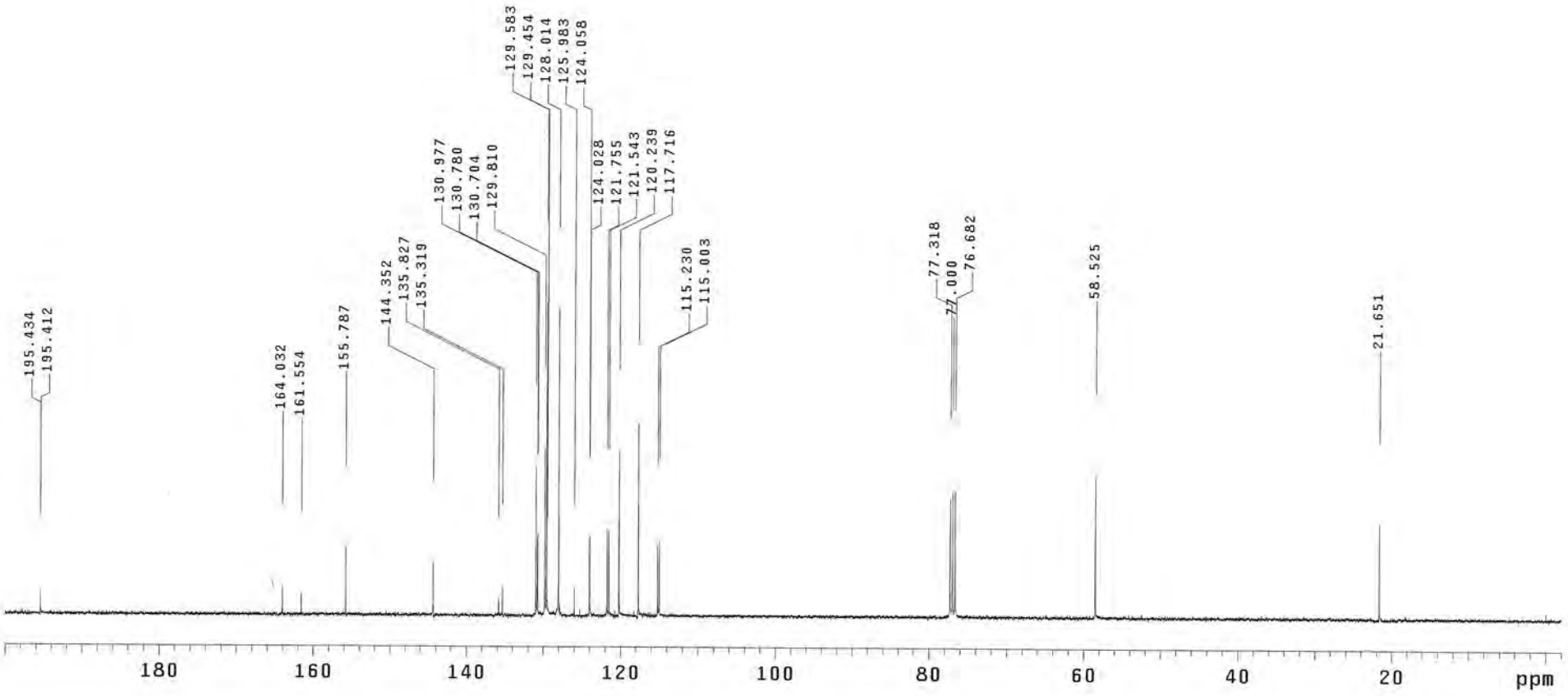




SIVA-RP4-121

Pulse Sequence: s2pul

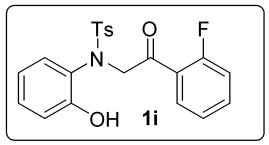
Mercury-400BB "MercuryPlus400"
Date: May 2 2014
Solvent: CDCl3
Ambient temperature
Total 7760 repetitions



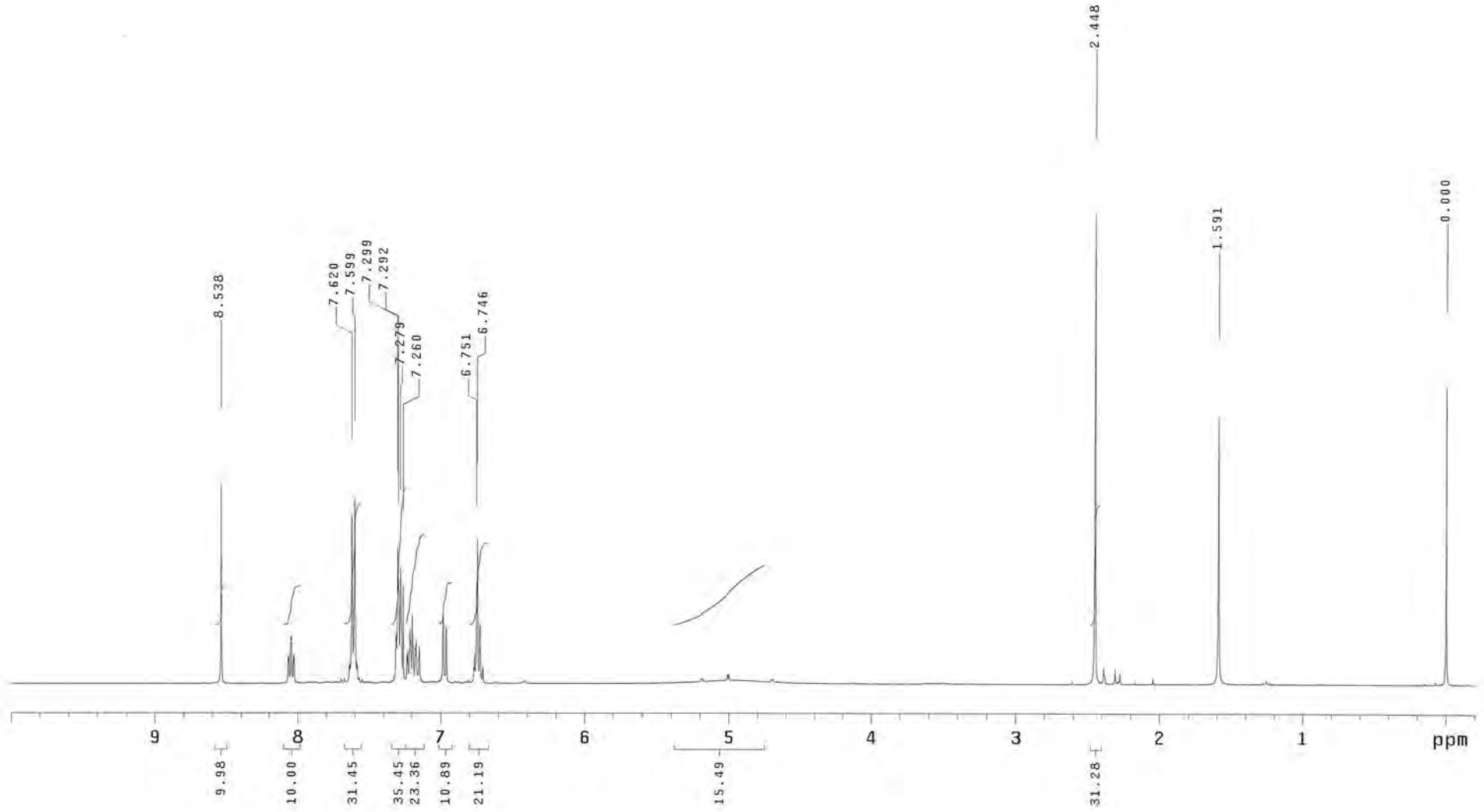
SIVA-RP3-37

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
64 repetitions
OBSERVE H1, 400.3978960 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec



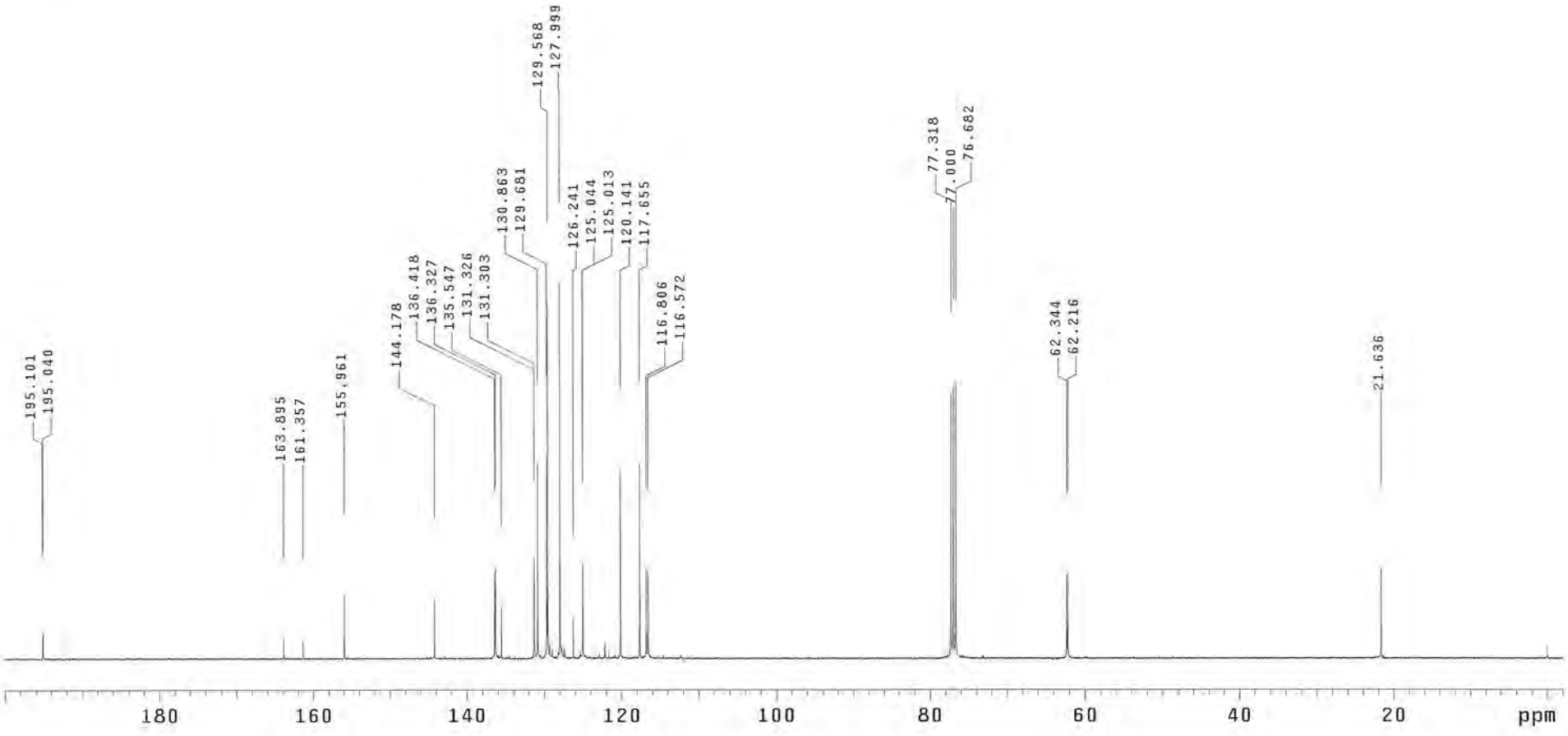
1i



SIVA-RP3-37

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

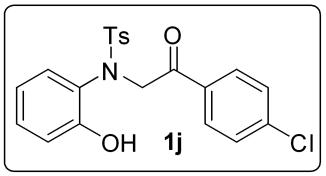
Relax. delay 0.500 sec
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
80 repetitions
OBSERVE C13, 100.6801323 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 30 hr, 44 min, 54 sec

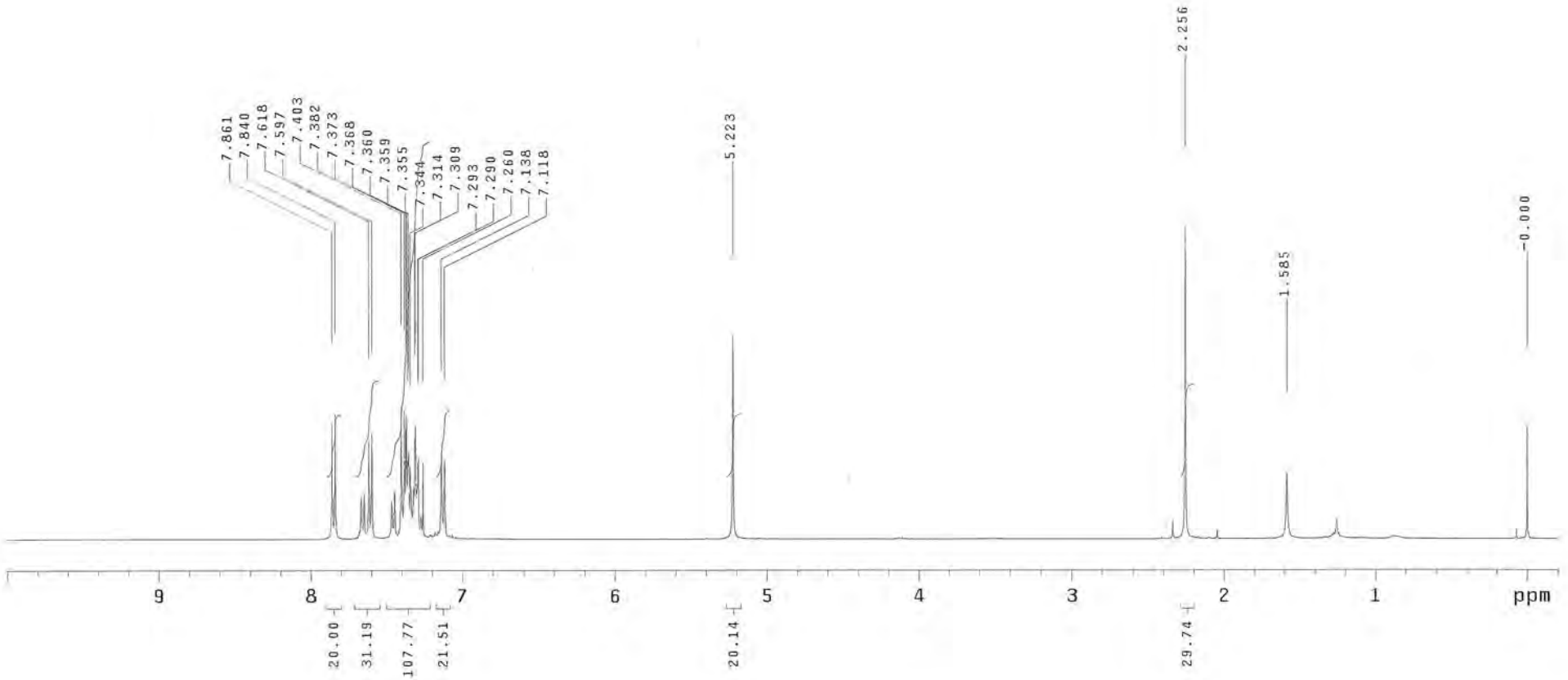


SIVA-RP4-080

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
64 repetitions
OBSERVE H1, 400.3978962 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec

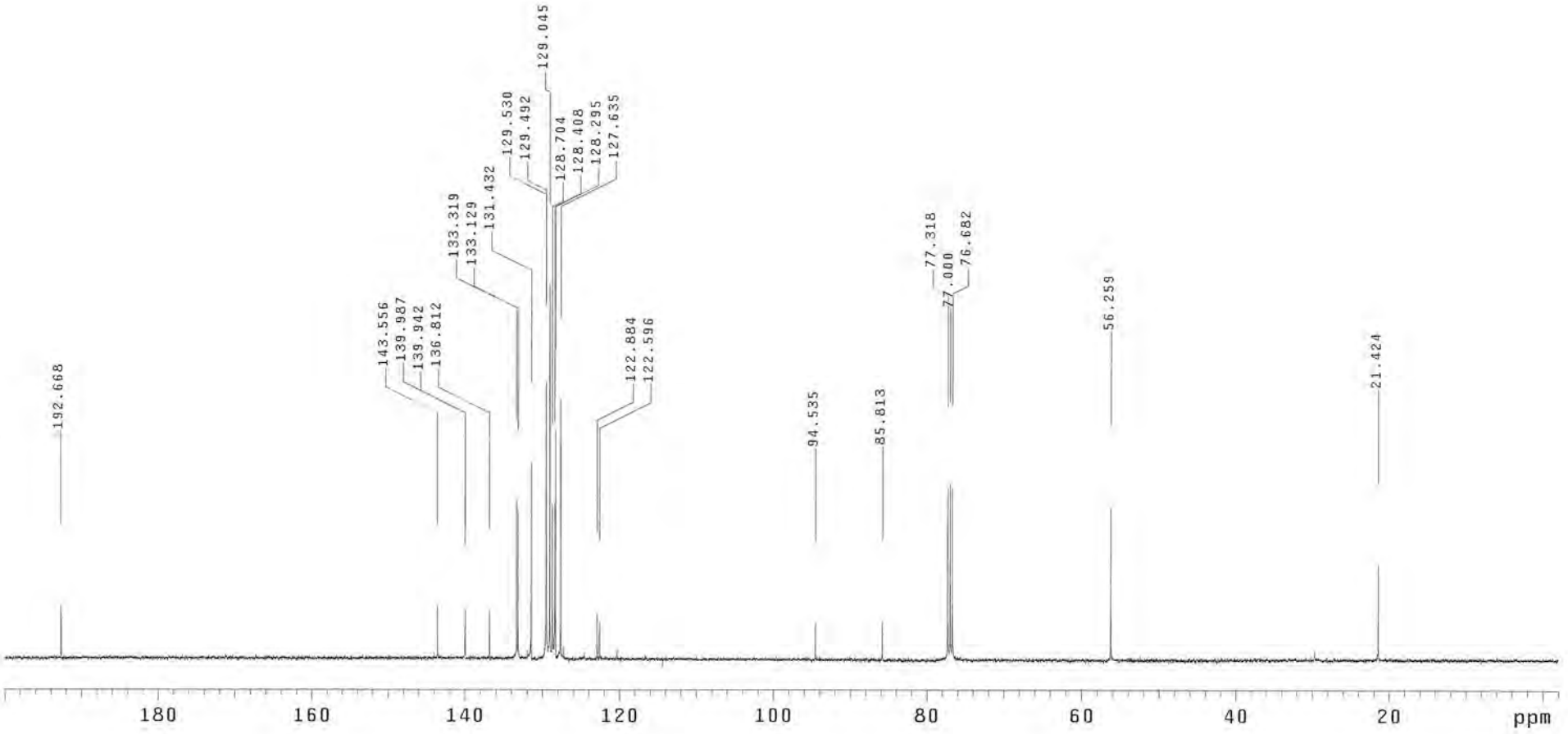




SIVA-RP4-080

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

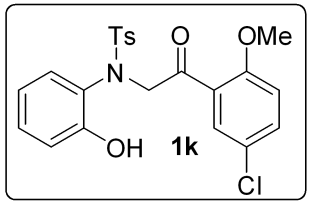
 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 336 repetitions
OBSERVE C13, 100.6801330 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec

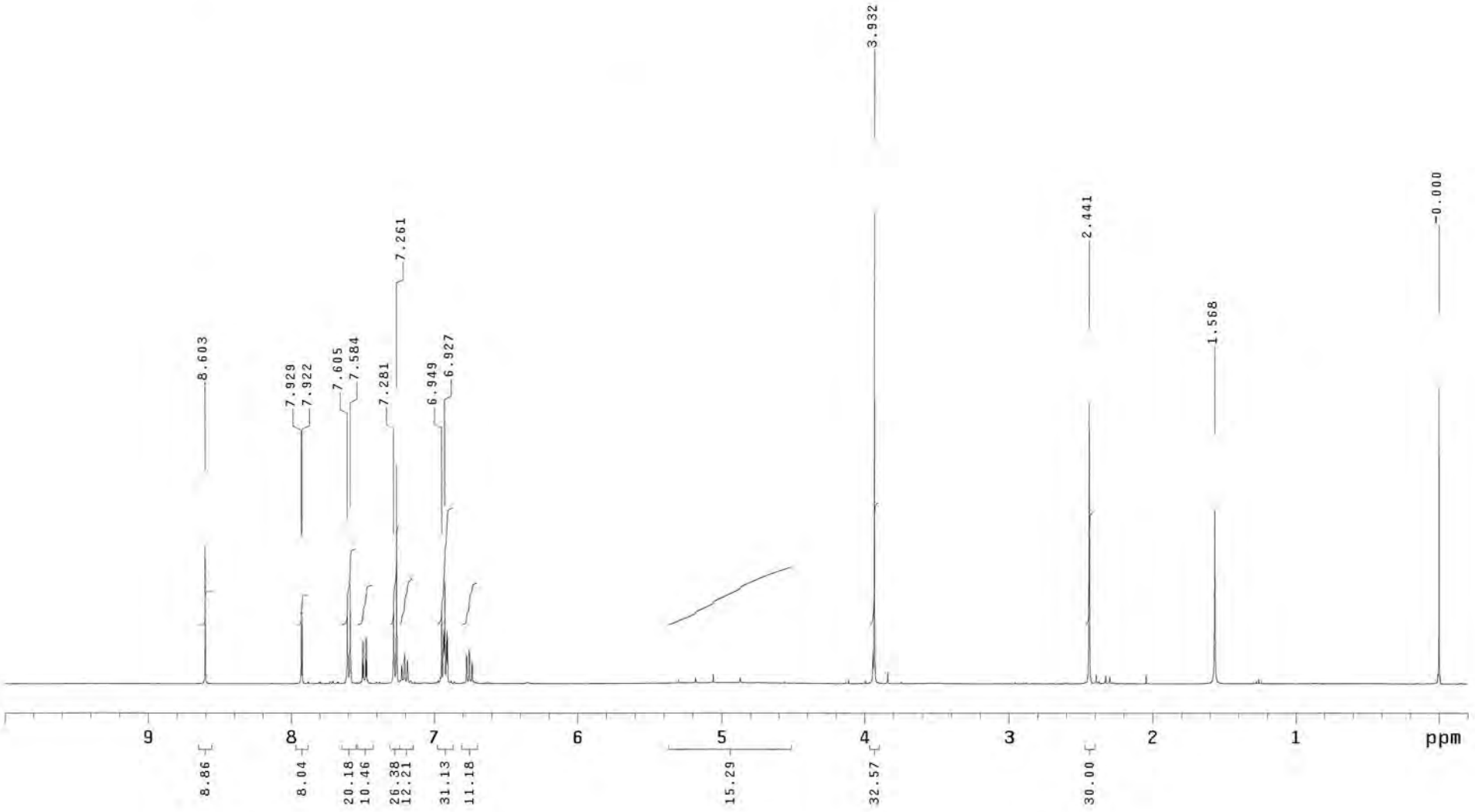


SIVA-RP4-057

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

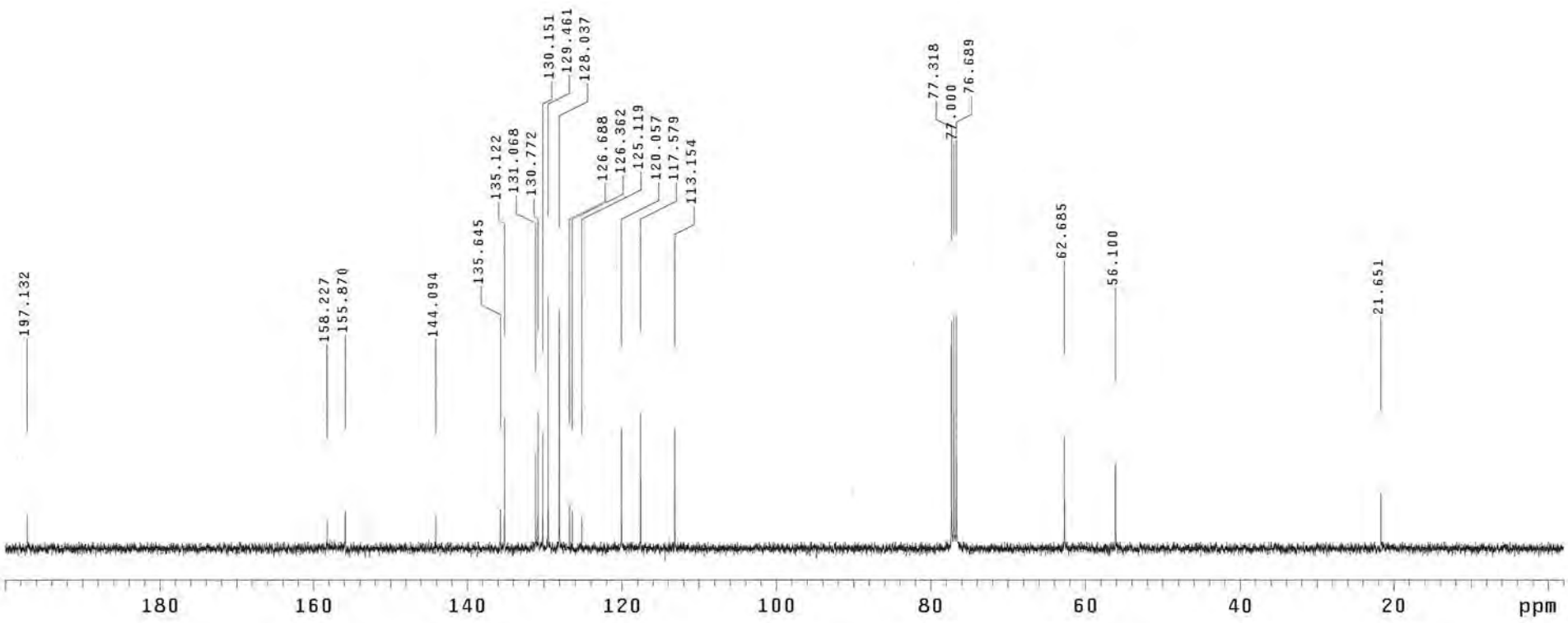




SIVA-RP4-057

Pulse Sequence: s2pul

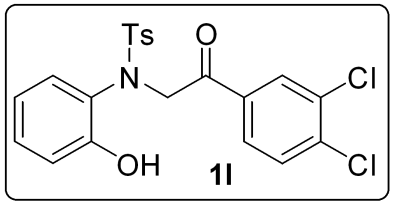
Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 3344 repetitions



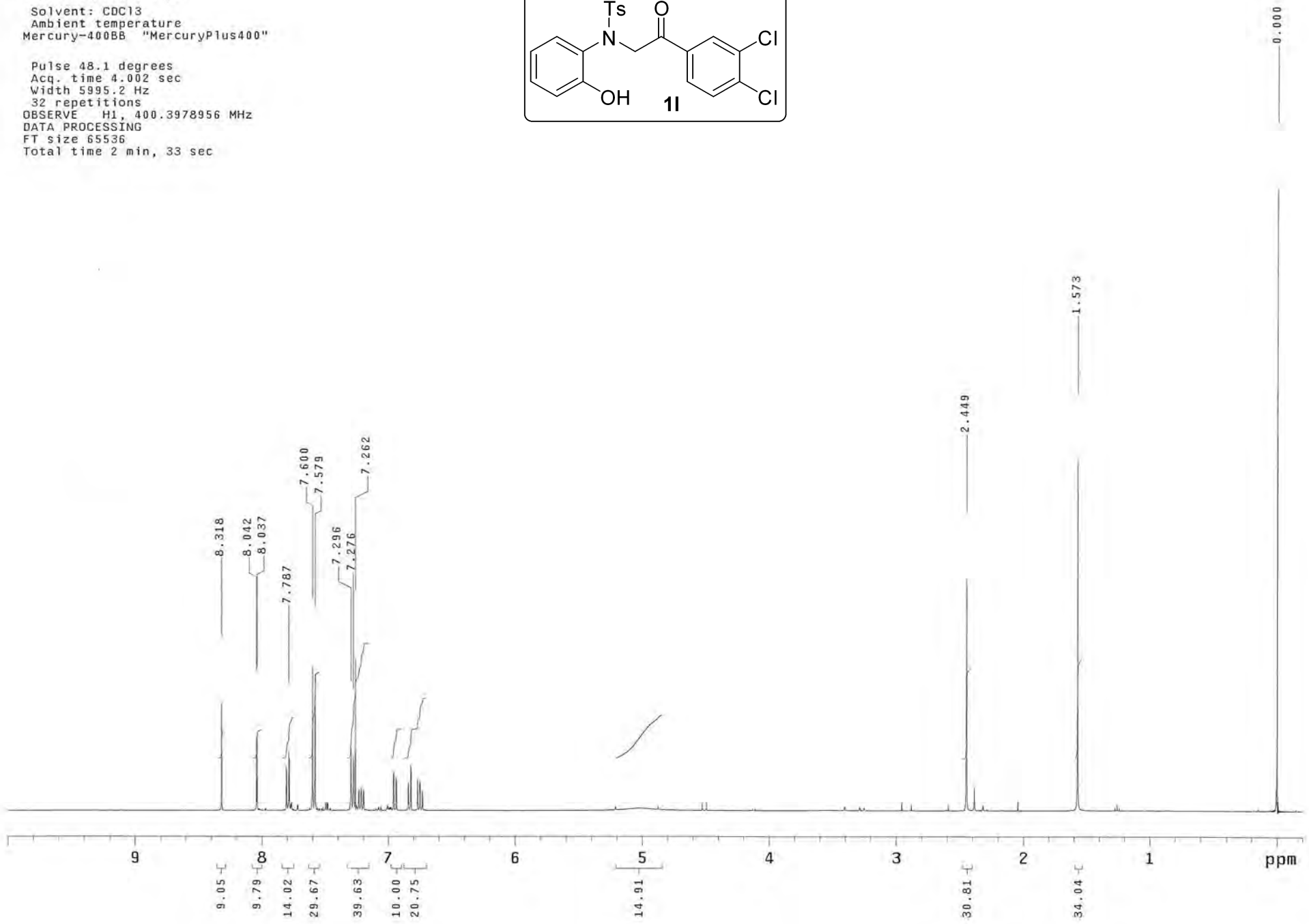
SIVA-RP3-58

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978956 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec



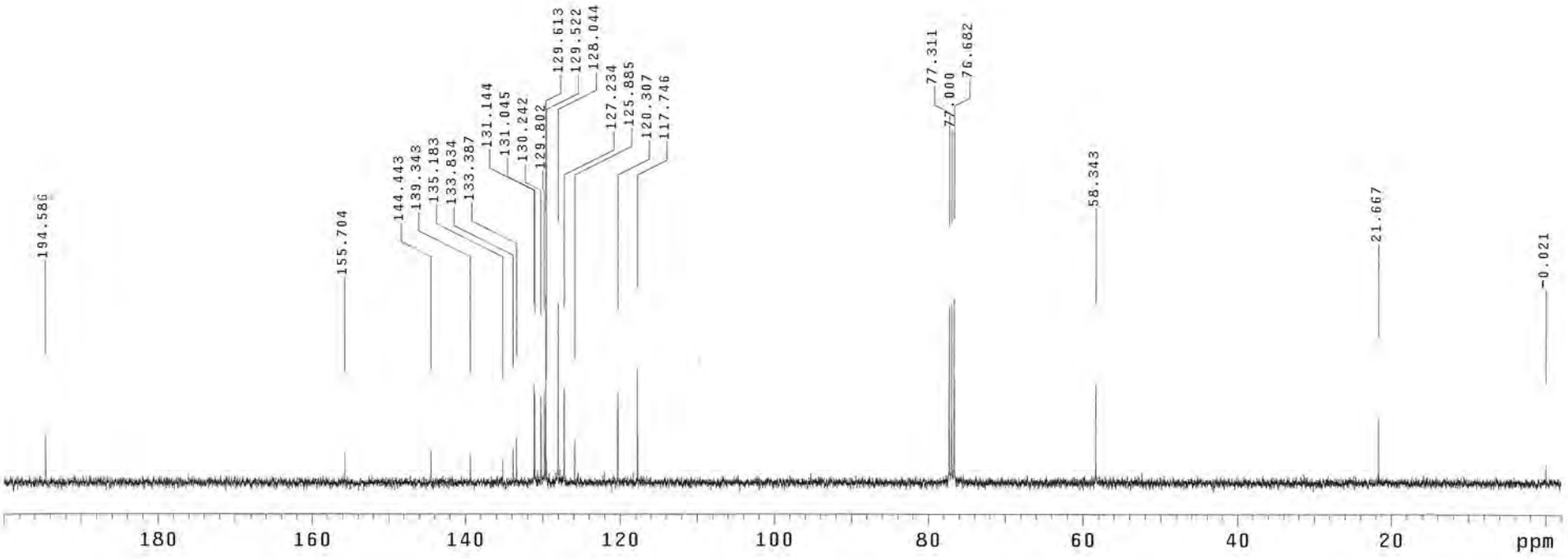
1l



SIVA-RP3-58

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

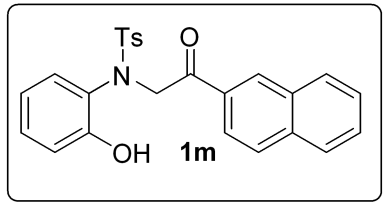
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
2048 repetitions
OBSERVE C13, 100.6801315 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



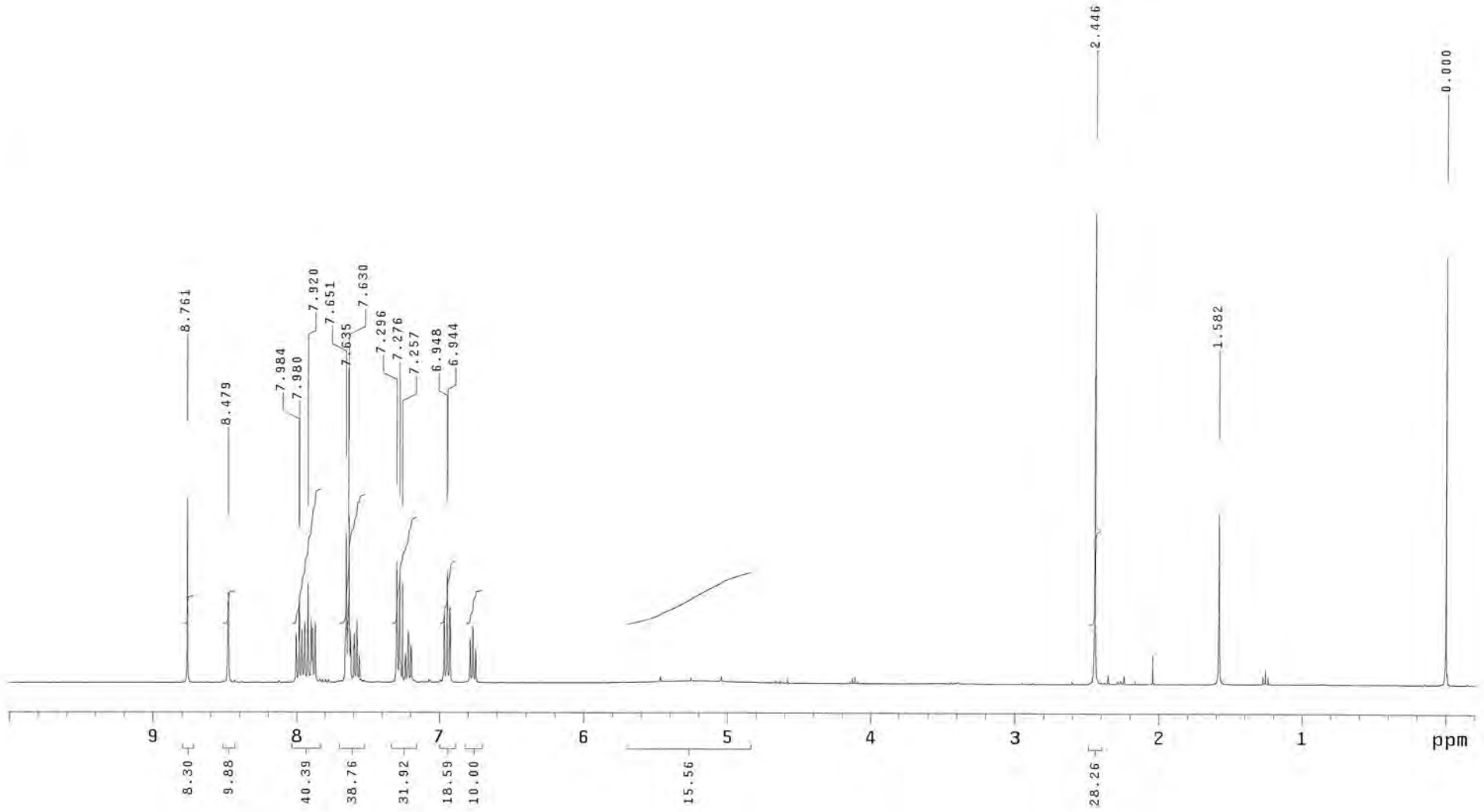
SIVA-RP3-34

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978973 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec



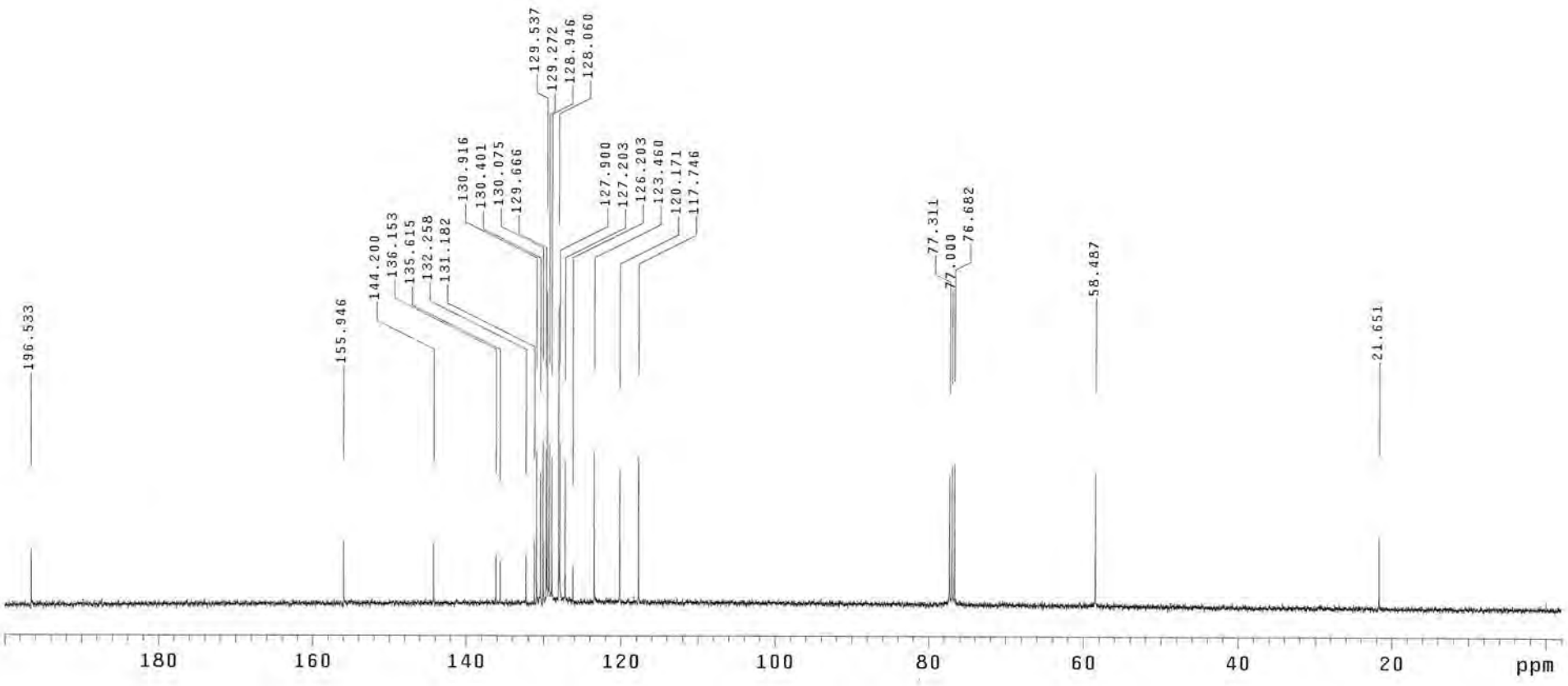
1m



SIVA-RP3-34

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
Mercury-400BB "MercuryPlus400"

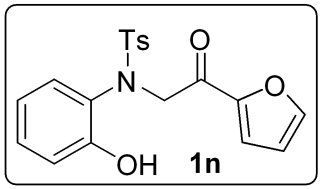
 Pulse 68.7 degrees
 Acq. time 1.000 sec
 Width 25000.0 Hz
 2304 repetitions
OBSERVE C13, 100.6801330 MHz
DECOUPLE H1, 400.3999572 MHz
 Power 38 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec



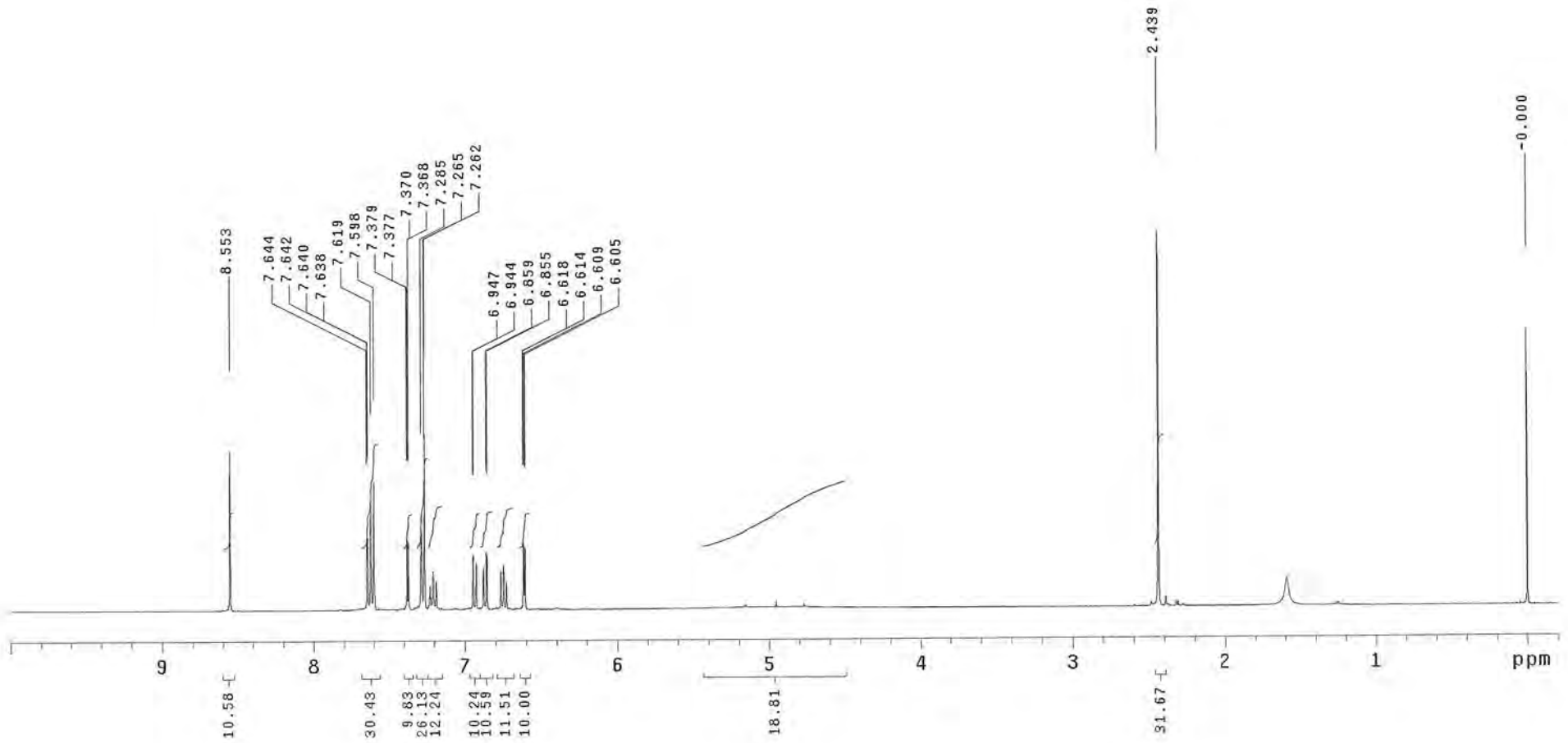
SIVA-RP3-31

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 15 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions



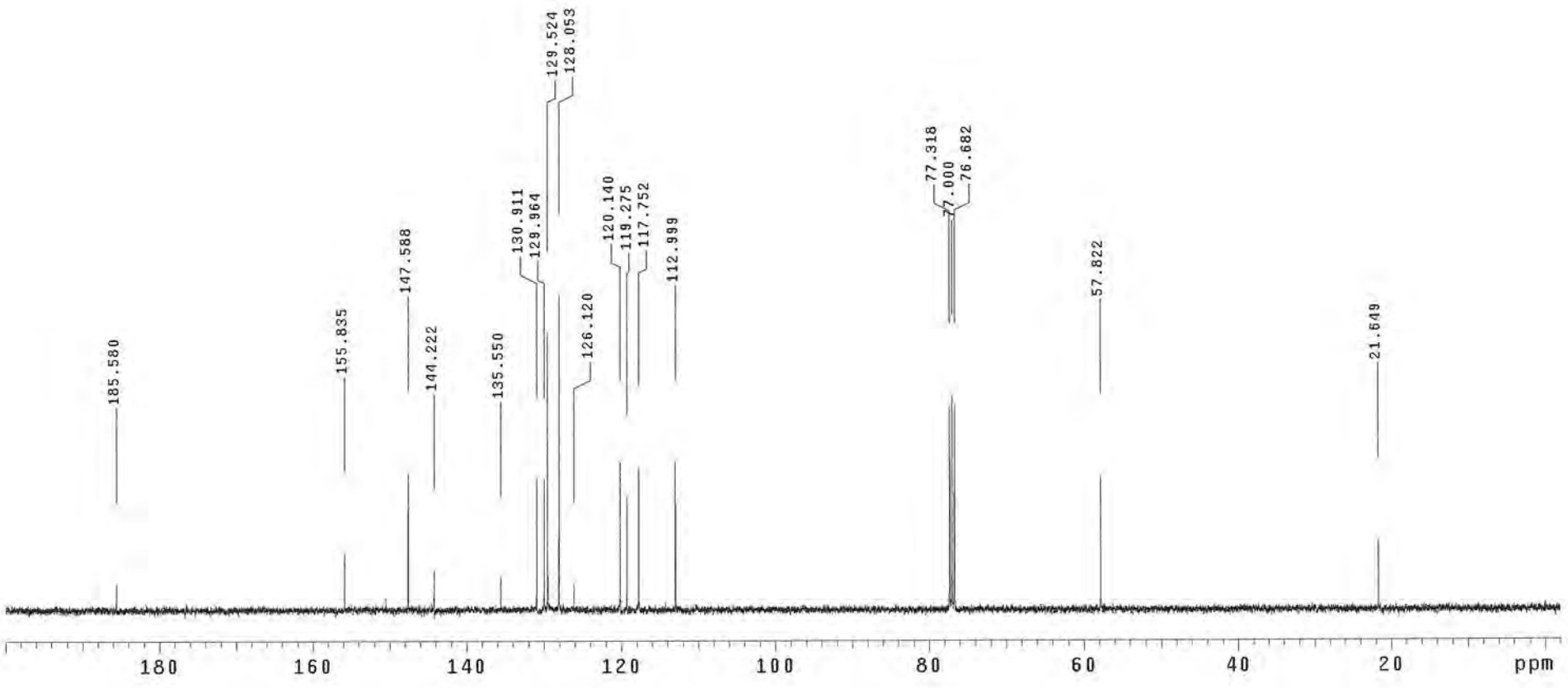
1n



SIVA-RP3-31

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 15 2013
Solvent: CDCl3
Ambient temperature
Total 6784 repetitions

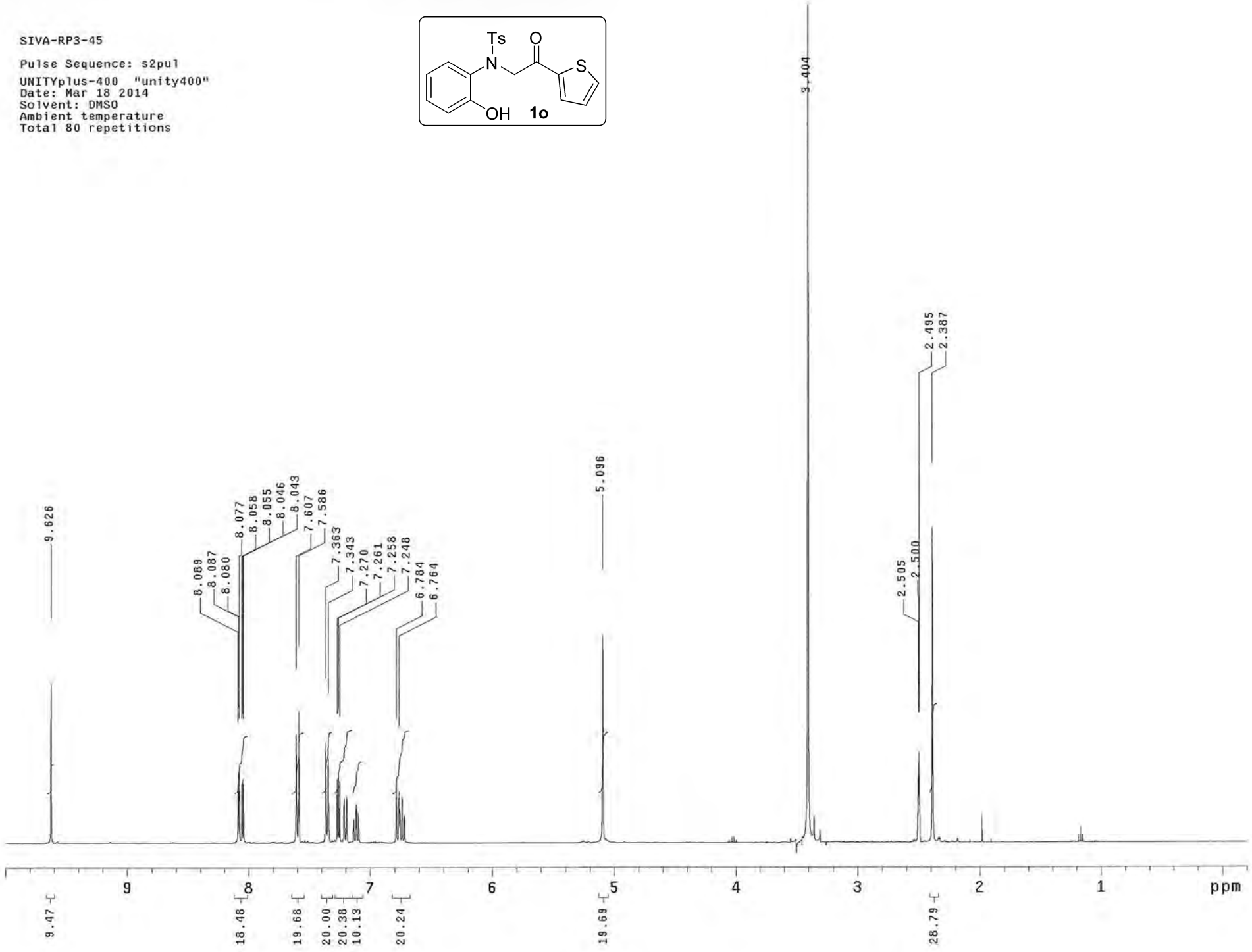


SIVA-RP3-45

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Mar 18 2014
Solvent: DMSO
Ambient temperature
Total 80 repetitions

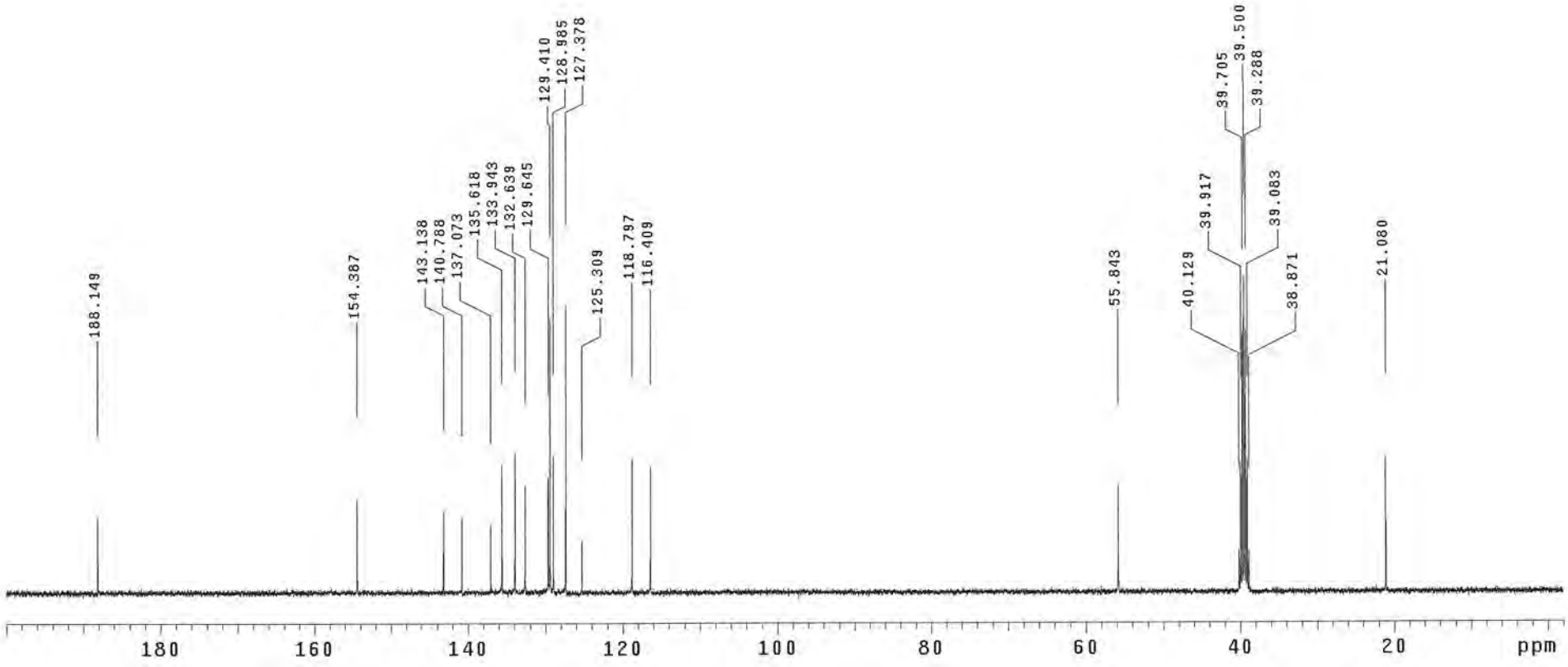
1o



SIVA-RP3-45

Pulse Sequence: s2pul

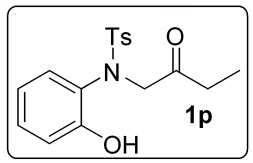
UNITYplus-400 "unity400"
Date: Mar 18 2014
Solvent: DMSO
Ambient temperature
Total 4816 repetitions

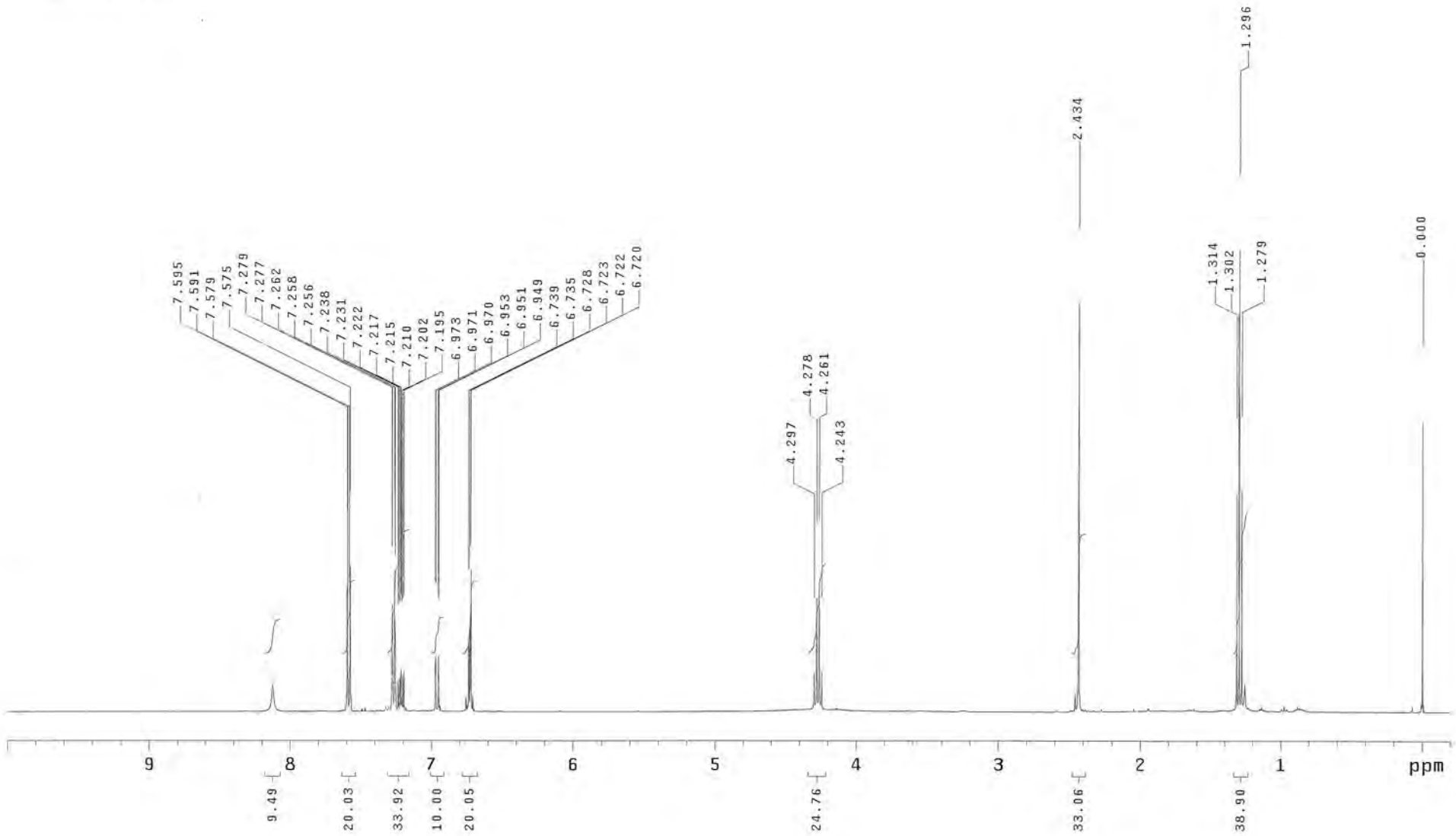


SIVA-RP3-073

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
64 repetitions
OBSERVE H1, 400.3978956 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec



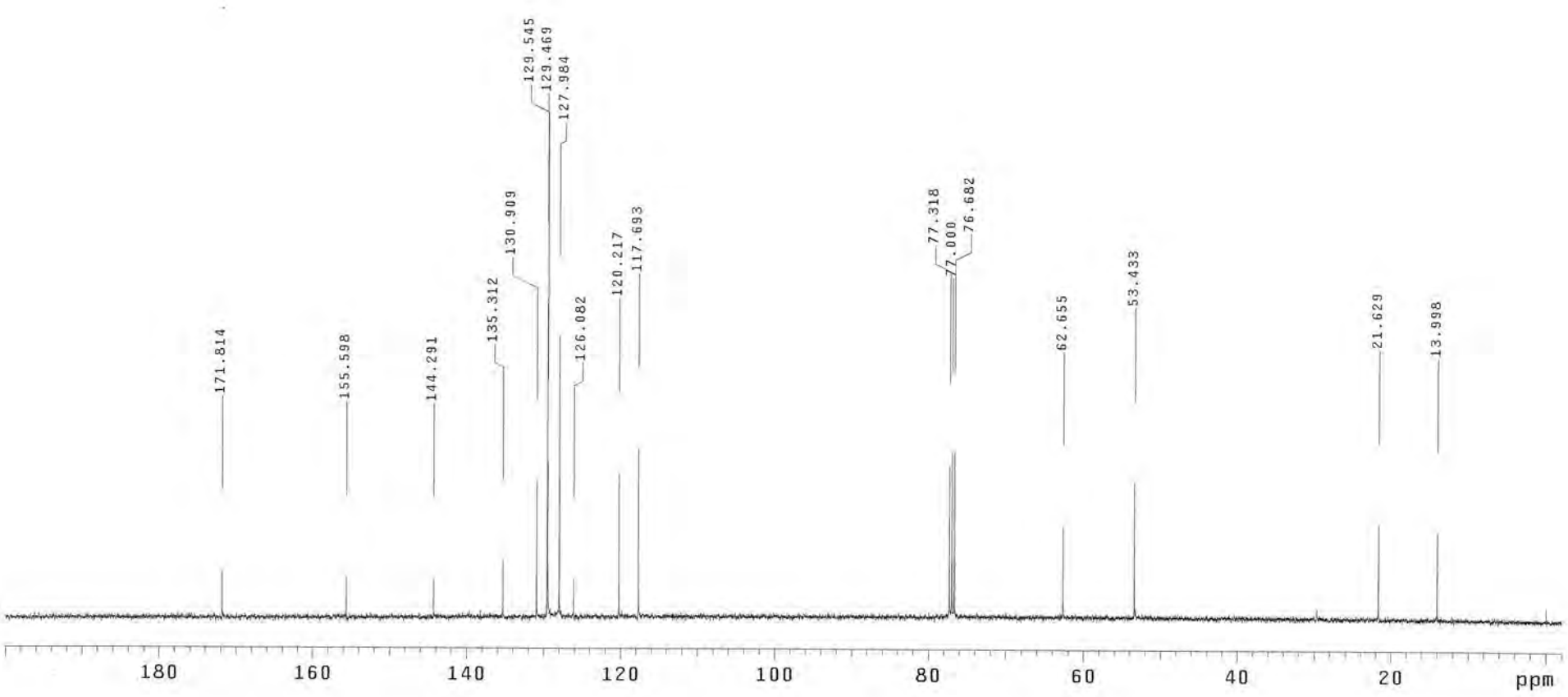


SIVA-RP3-073

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
2432 repetitions
OBSERVE C13, 100.6801323 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec

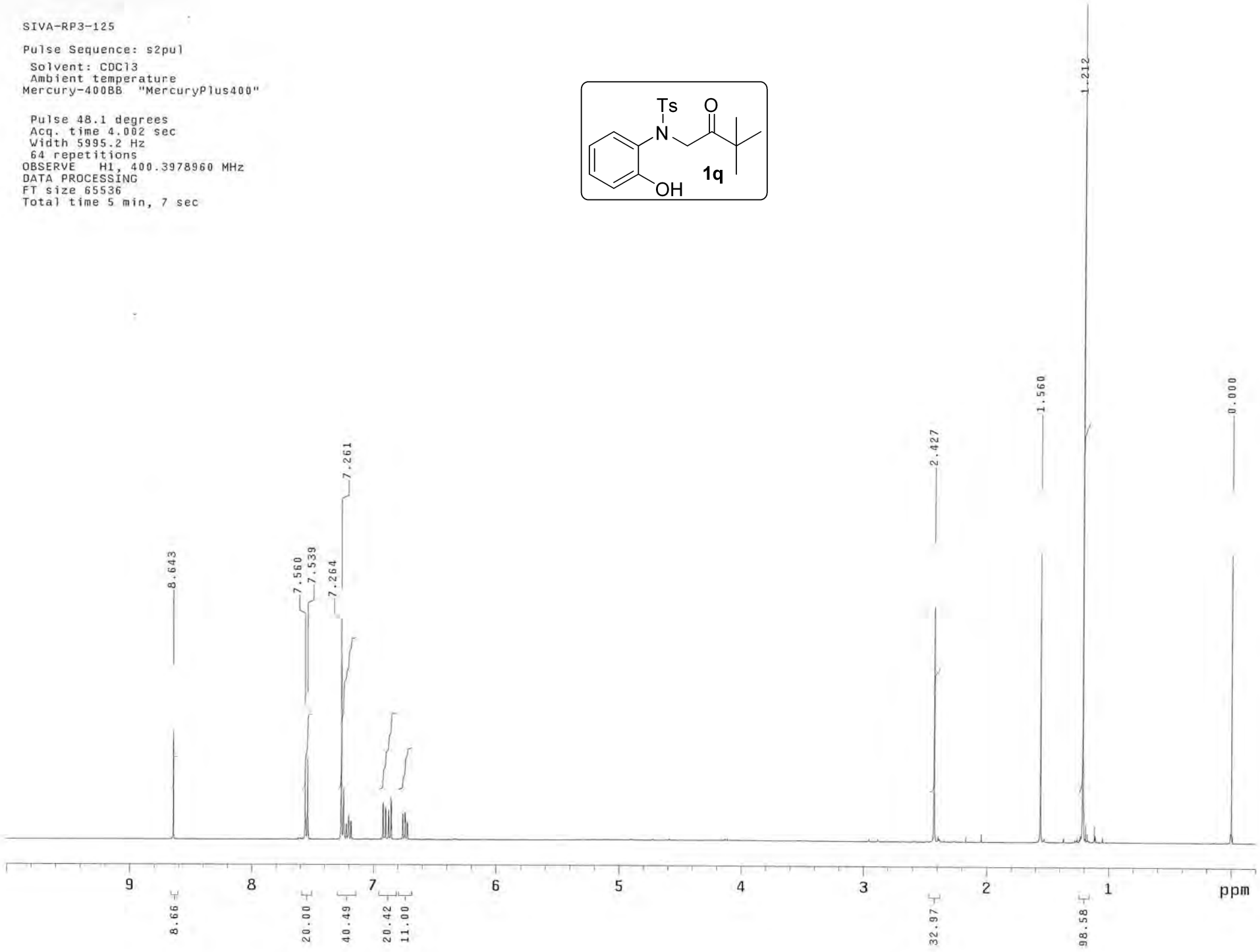


SIVA-RP3-125

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
64 repetitions
OBSERVE H1, 400.3978960 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec

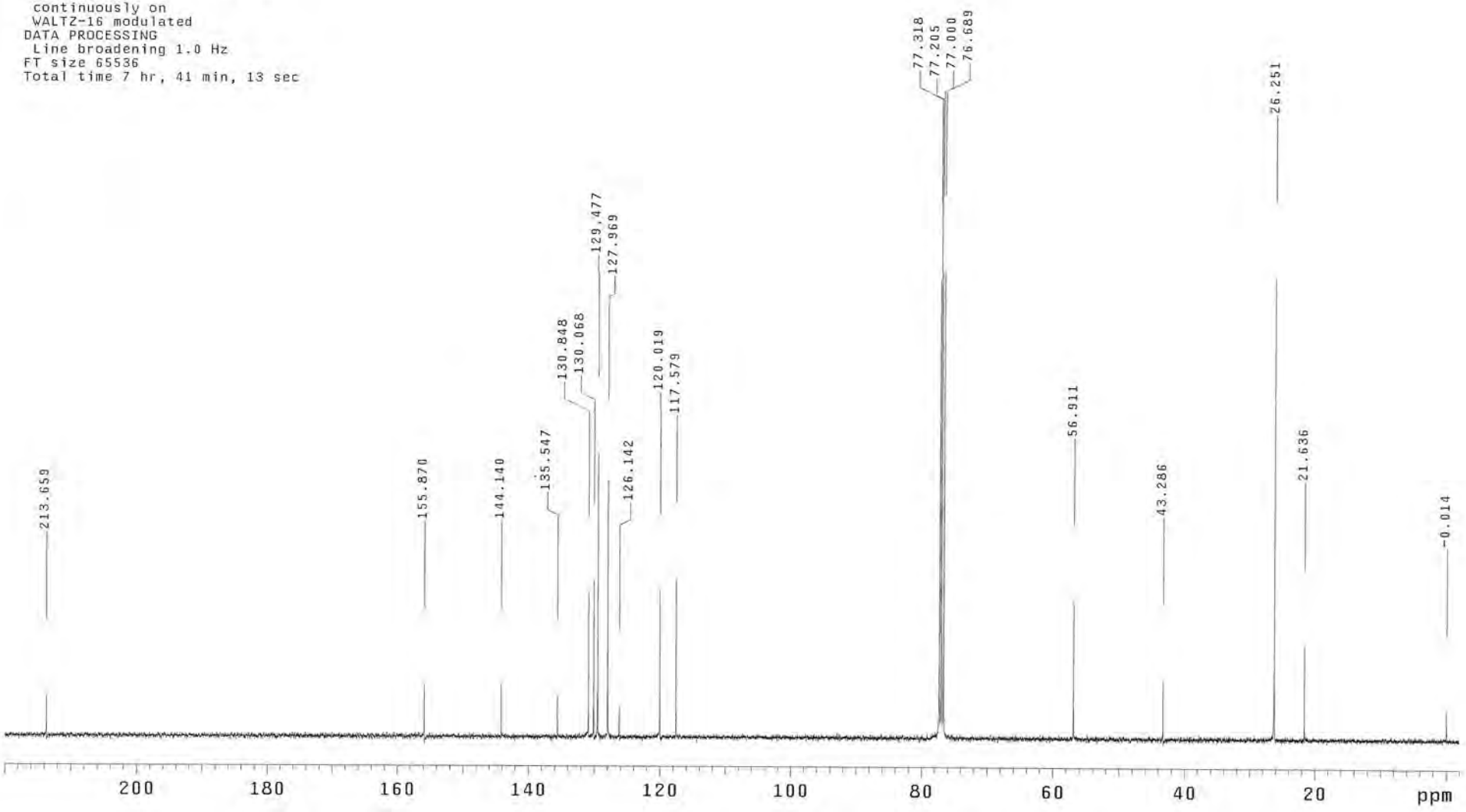
1q



SIVA-RP3-125

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

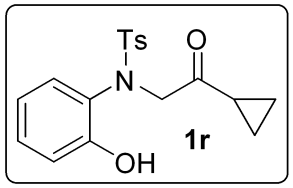
Relax. delay 0.500 sec
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
16000 repetitions
OBSERVE C13, 100.6801308 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 41 min, 13 sec



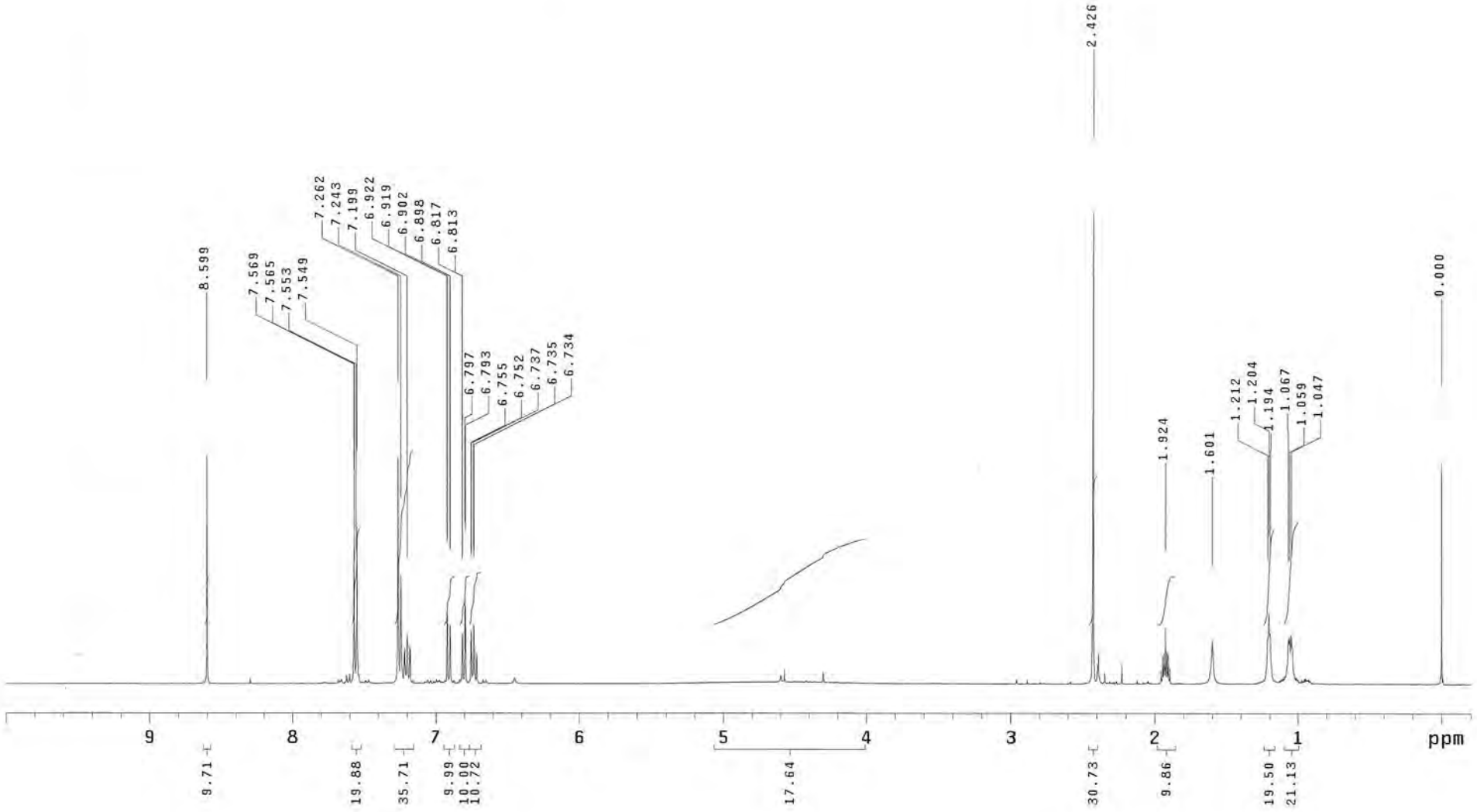
SIVA-RP4-157

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 48 repetitions



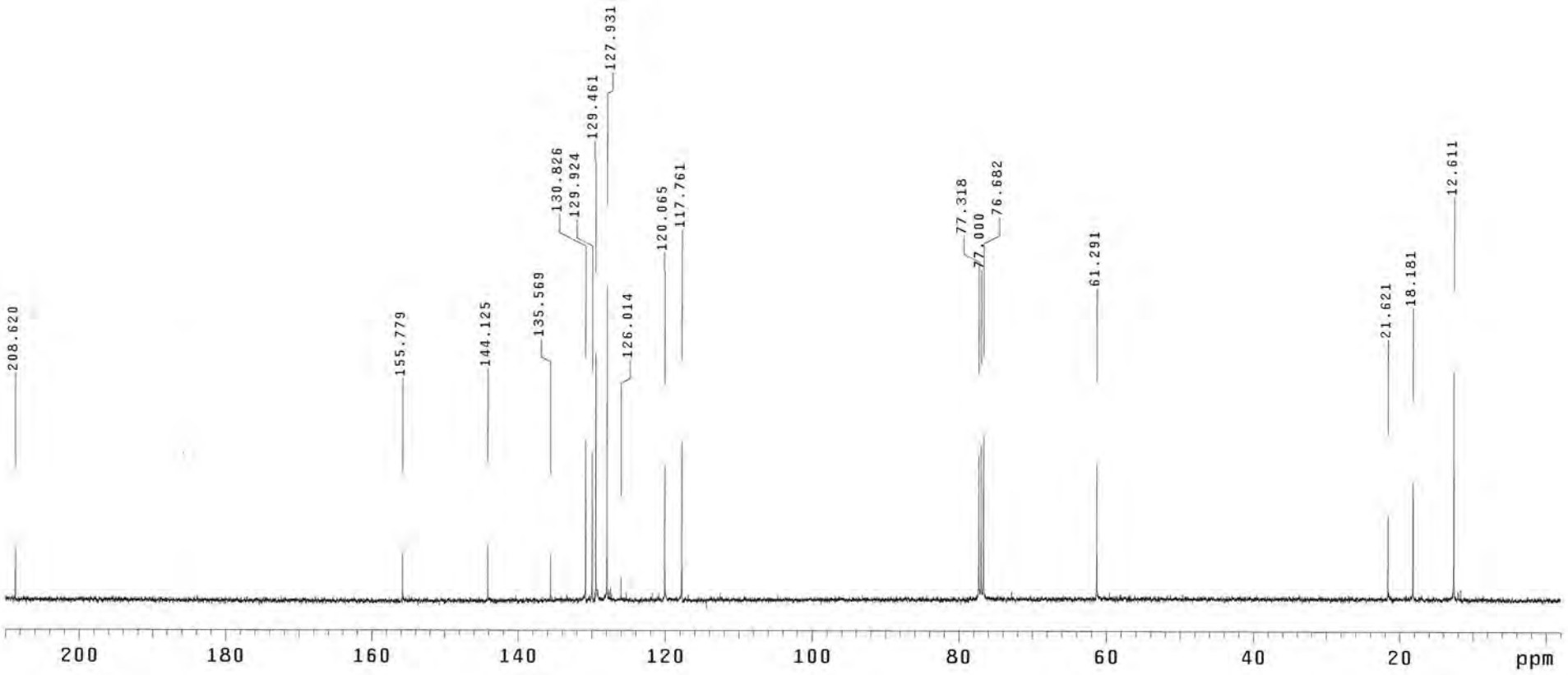
1r



SIVA-RP4-157

Pulse Sequence: s2pul

Mercury-400BB "MercuryPlus400"
Date: Mar 3 2014
Solvent: CDCl3
Ambient temperature
Total 5504 repetitions

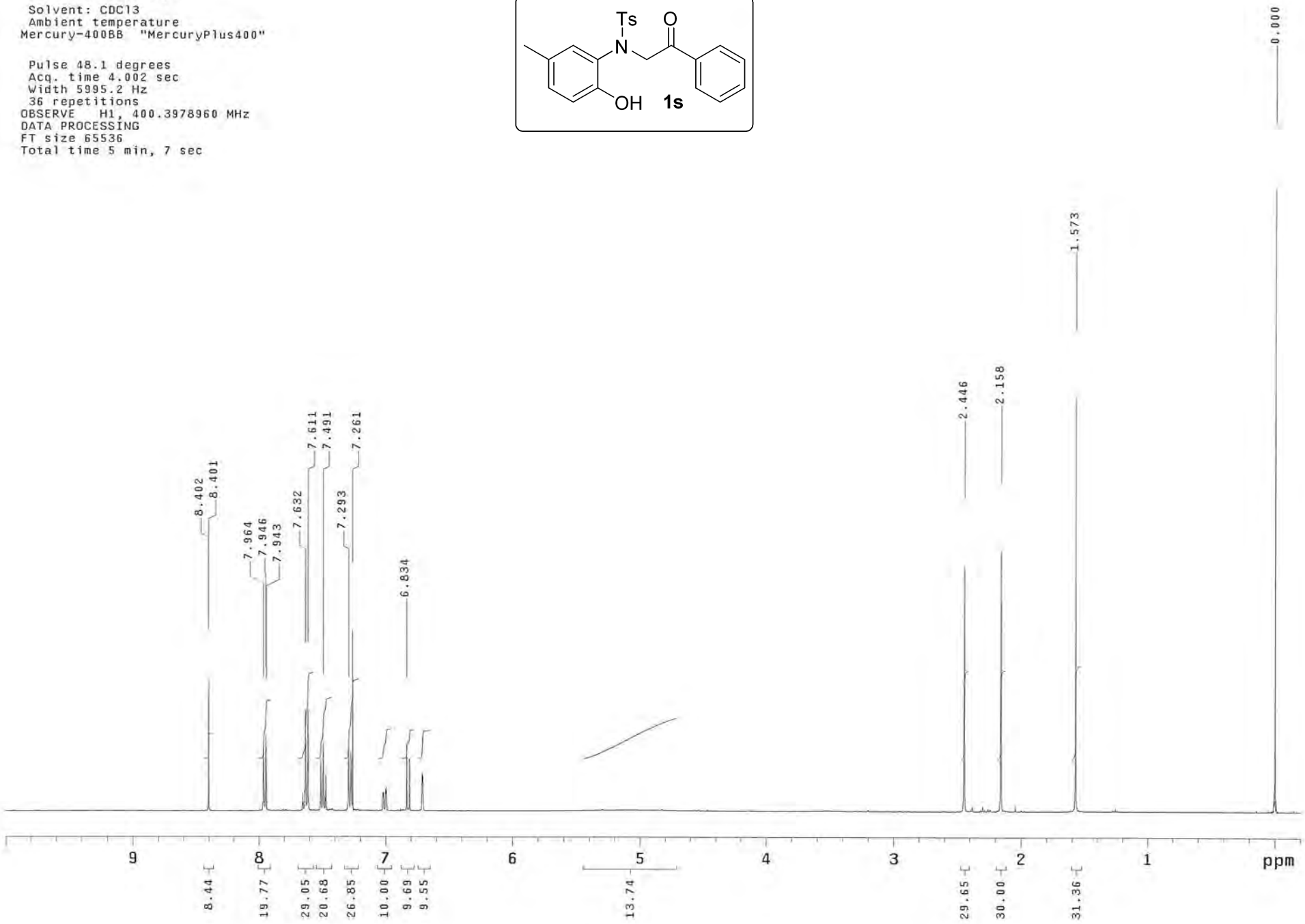


SIVA-RP3-56

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
36 repetitions
OBSERVE H1, 400.3978960 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec

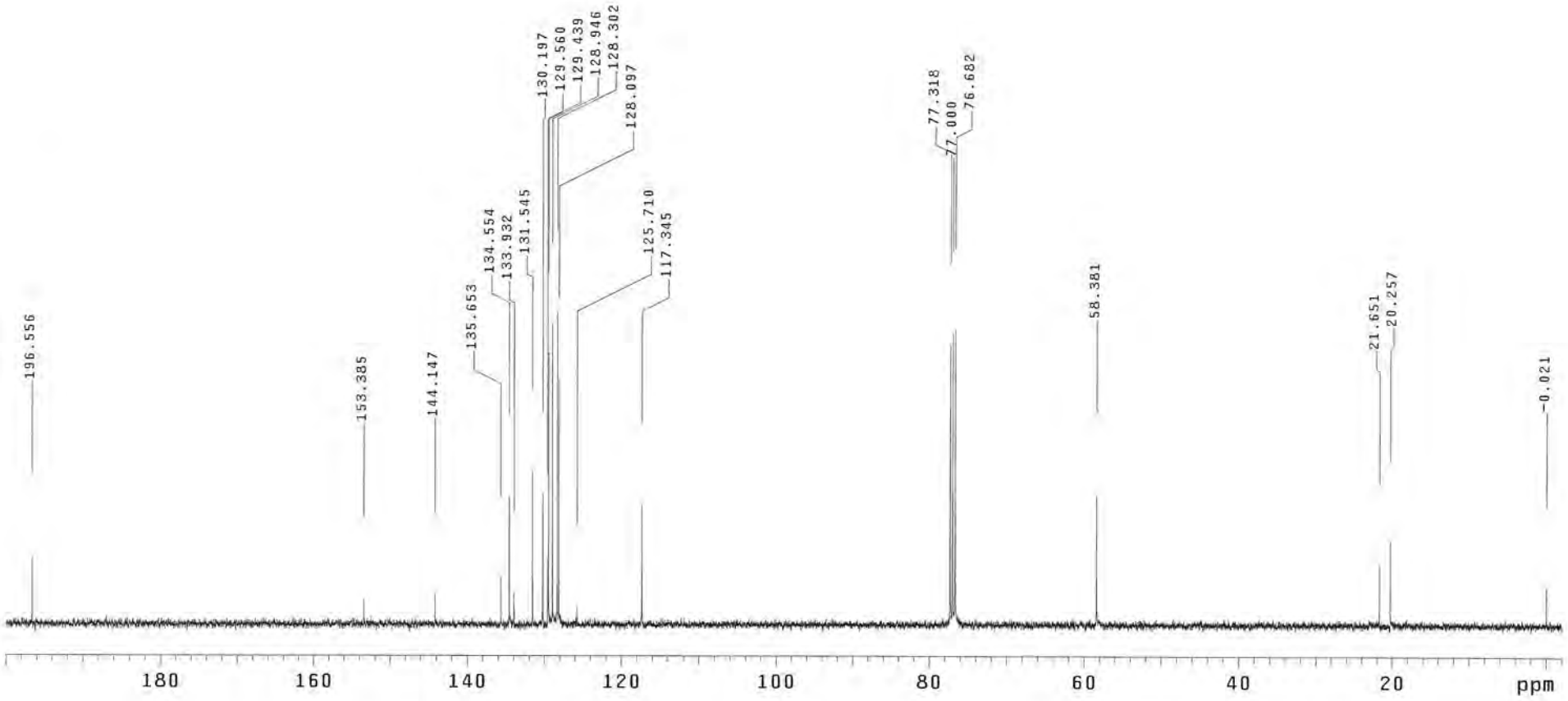
1s



SIVA-RP3-56

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
5024 repetitions
OBSERVE C13, 100.6801315 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec

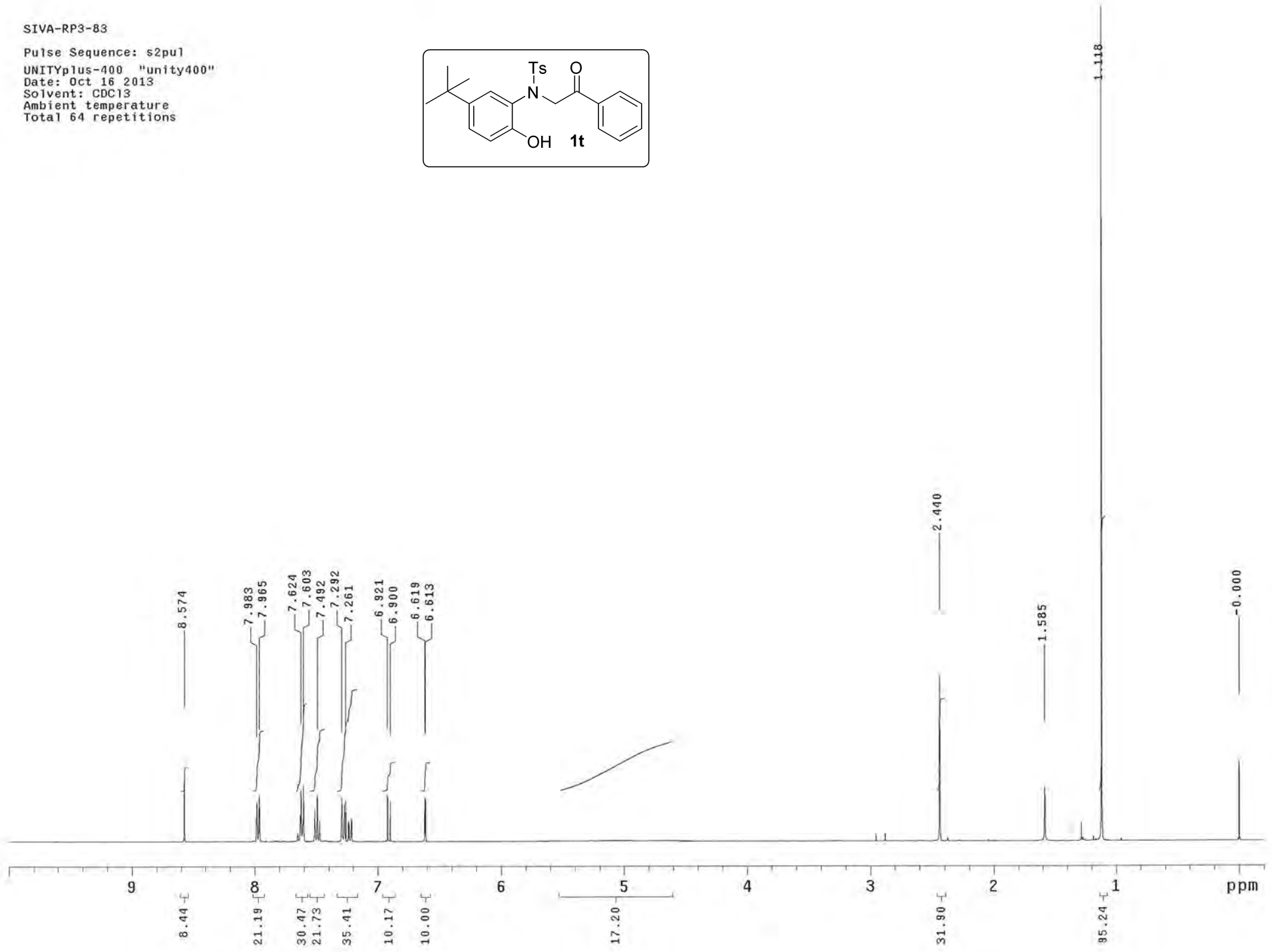


SIVA-RP3-83

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 16 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions

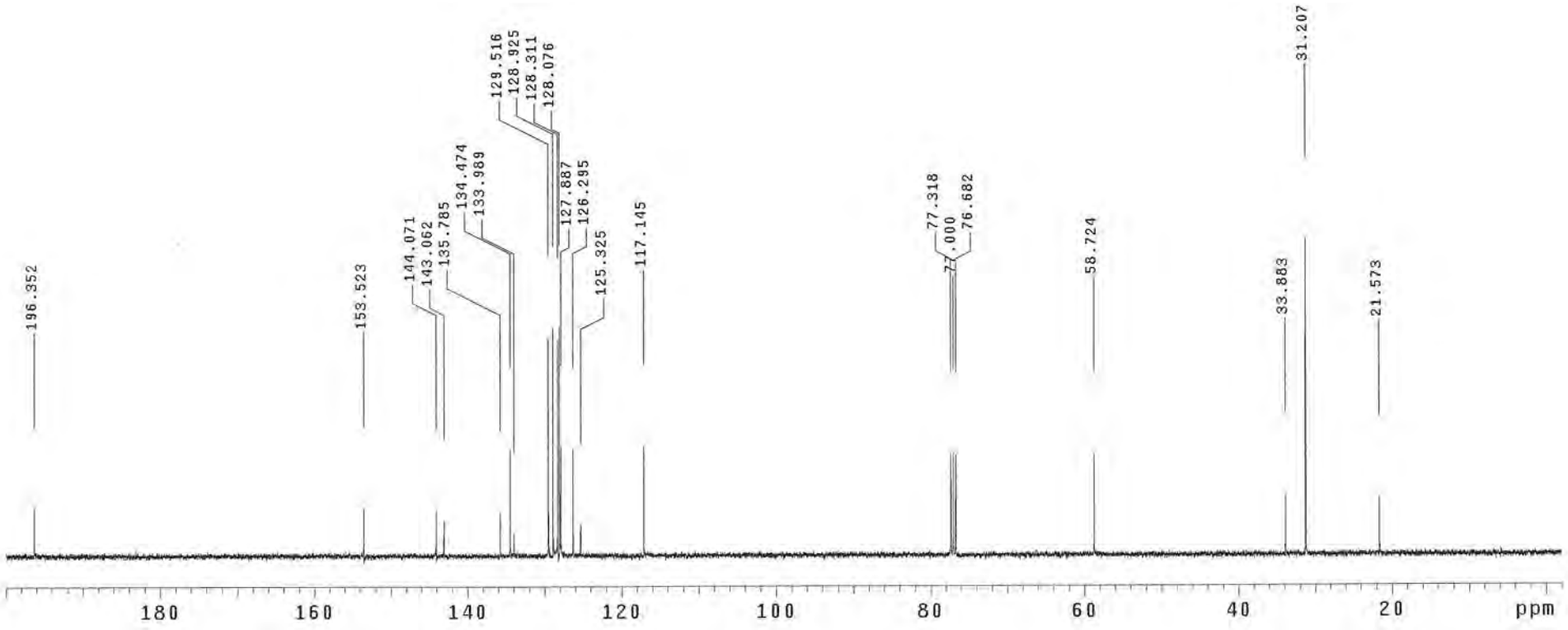
1t



SIVA-RP3-83

Pulse Sequence: s2pul

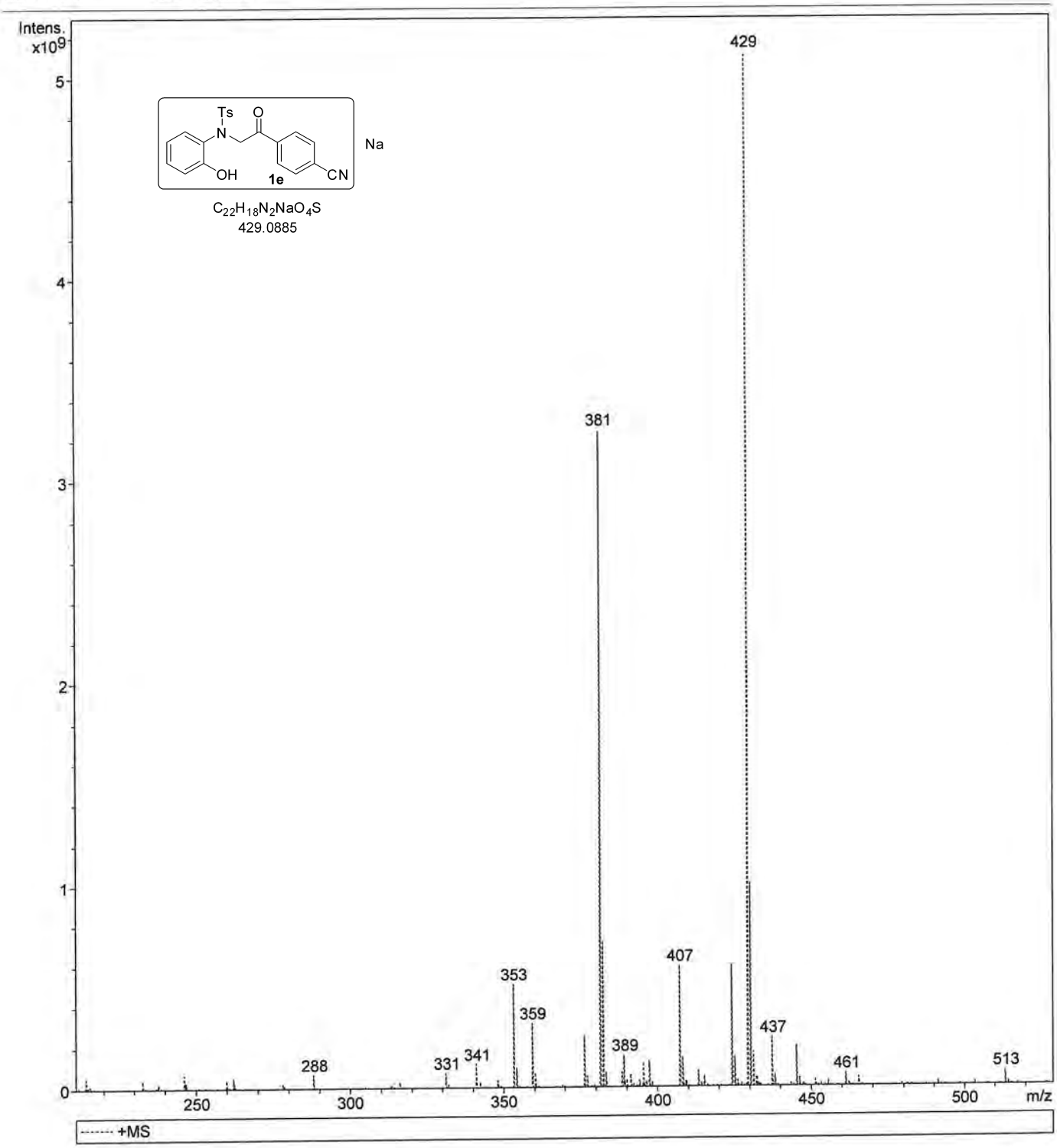
UNITYplus-400 "unity400"
Date: Oct 16 2013
Solvent: CDCl3
Ambient temperature
Total 3680 repetitions



FT-MS

Analysis Info

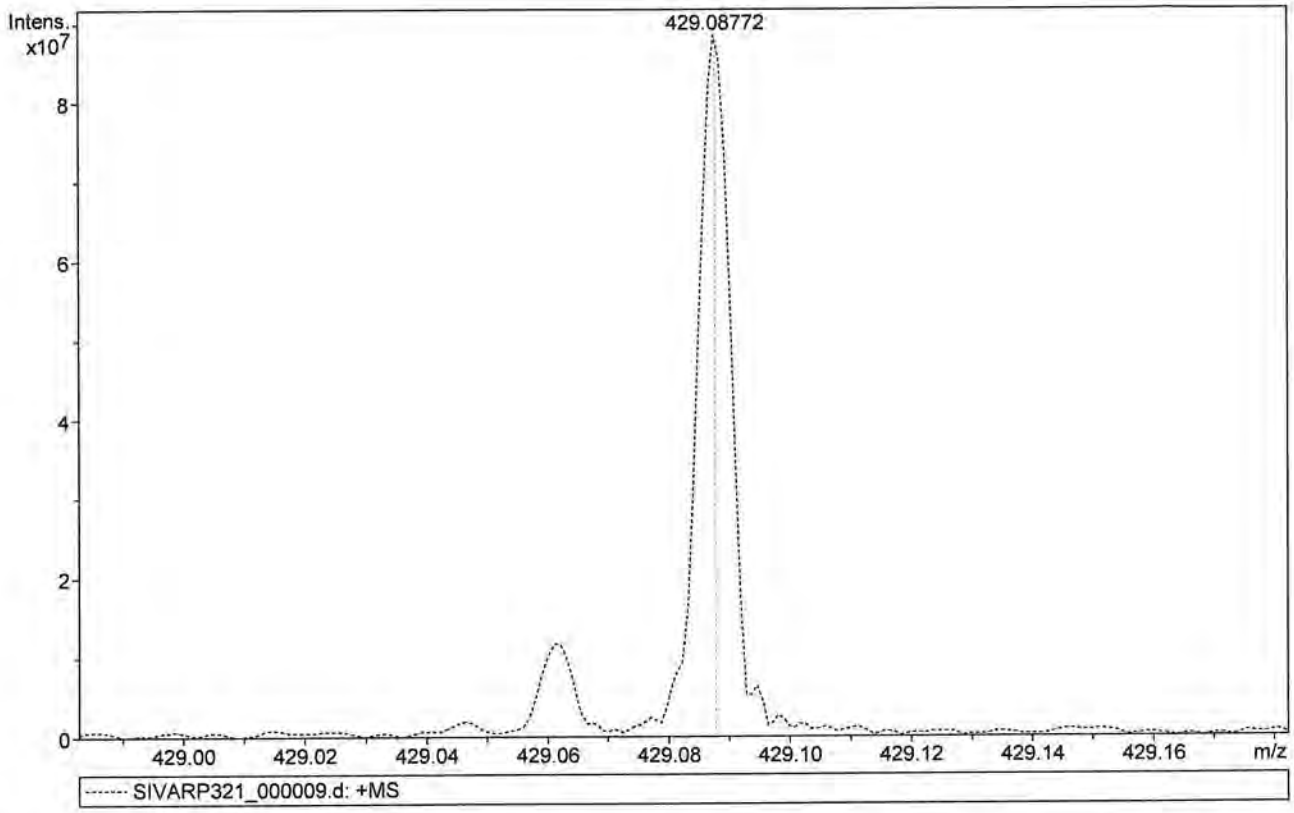
Analysis Name	D:\Data\a4\SIVARP321_000007.d	12/25/2013 3:25:28 PM	
Method	broadband first signal		
Sample Name	SIVA-RP3-21	Instrument: FT-MS solariX	
Comment	ESI Positive		



Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\a4\SIVARP321_000009.d	12/25/2013 3:23:40 PM
Method	broadband first signal	
Sample Name	SIVA-RP3-21	Instrument: FT-MS solariX
Comment	ESI Positive	

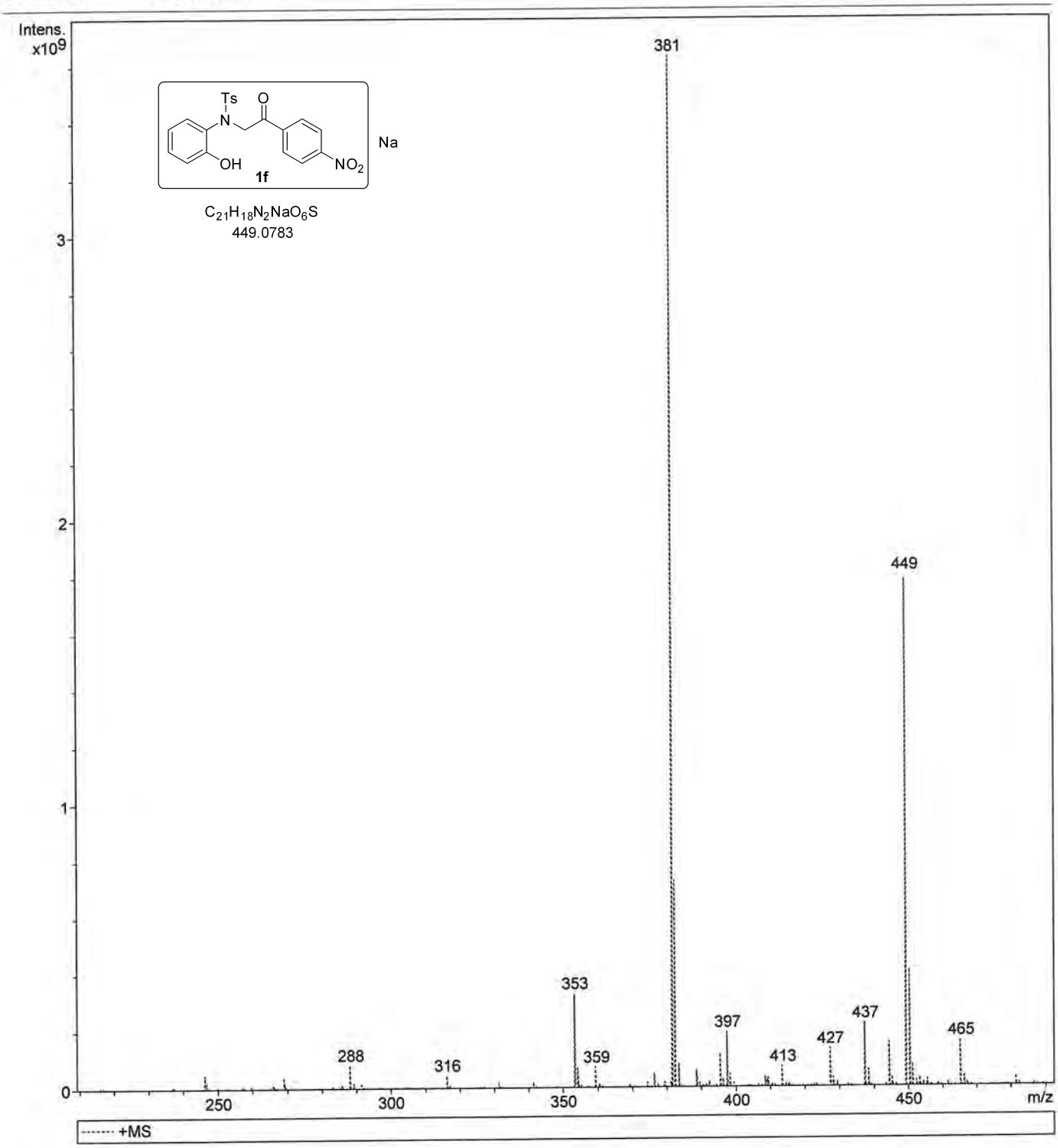


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
429.08772	1	C 22 H 18 N 2 Na O 4 S	100.00	429.08795	0.23	0.54	32.4	14.5	even	ok

FT-MS

Analysis Info

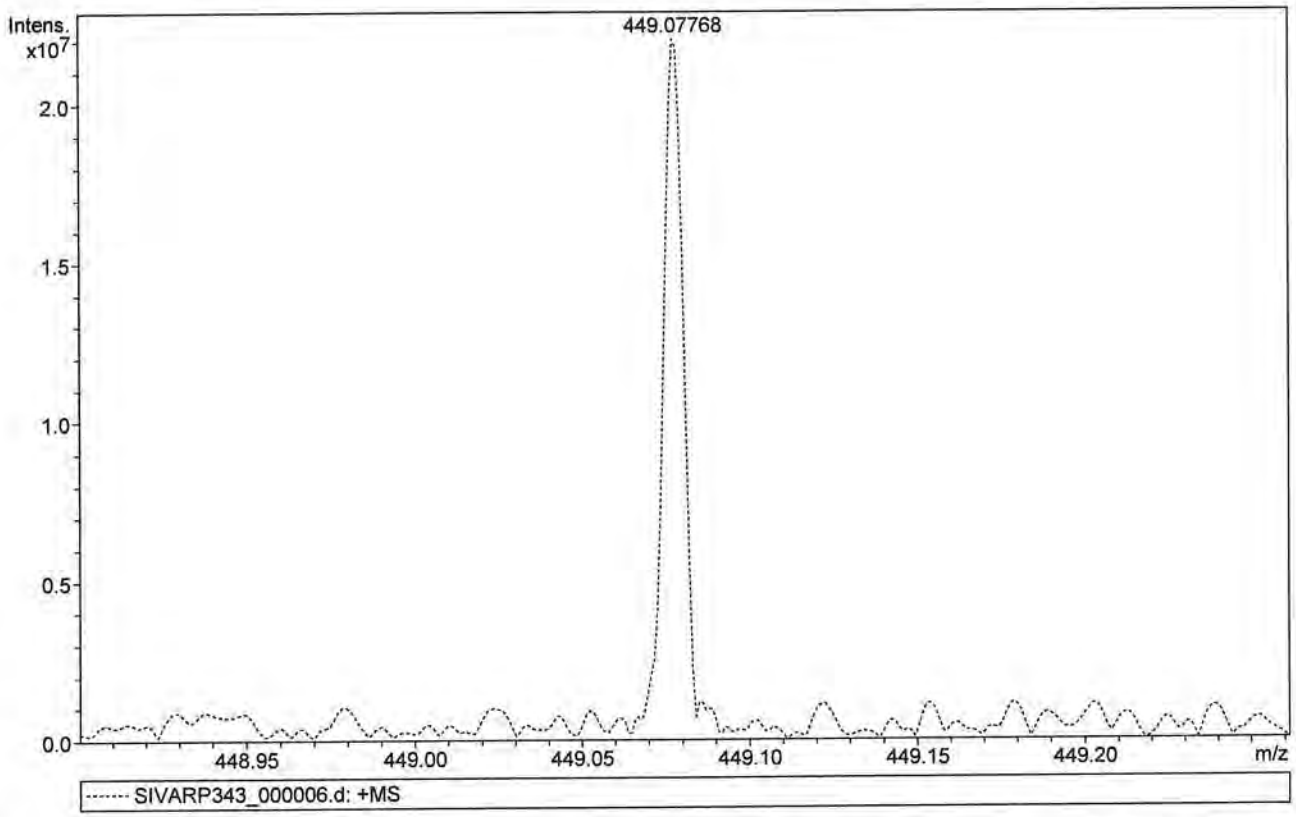
Analysis Name	D:\Data\a4\SIVARP343_000001.d	12/25/2013 4:09:08 PM	
Method	broadband first signal		
Sample Name	SIVA-RP3-43	Instrument: FT-MS solariX	
Comment	ESI Positive		



Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\a4\SIVARP343_000006.d	12/25/2013 4:08:00 PM
Method	broadband first signal	
Sample Name	SIVA-RP3-43	Instrument: FT-MS solariX
Comment	ESI Positive	

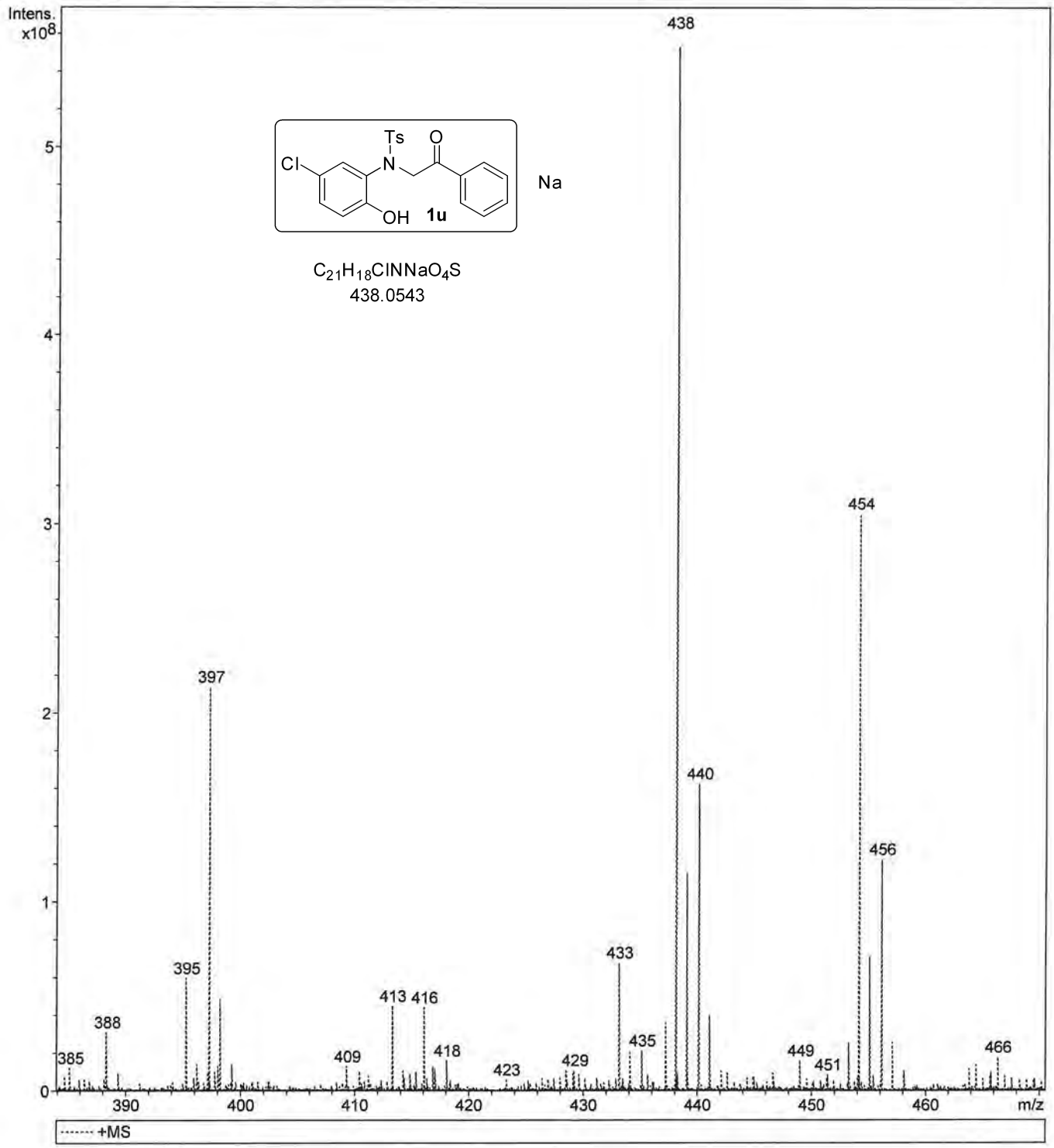


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
449.07768	1	C 21 H 18 N 2 Na O 6 S	100.00	449.07778	0.10	0.22	43.5	13.5	even	ok

FT-MS

Analysis Info

Analysis Name	D:\Data\a4\sivapr386S_000001.d	1/2/2014 3:30:38 PM
Method	broadband first signal	
Sample Name	SIVA-PR3-86S	Instrument: FT-MS solariX
Comment	ESI Positive	



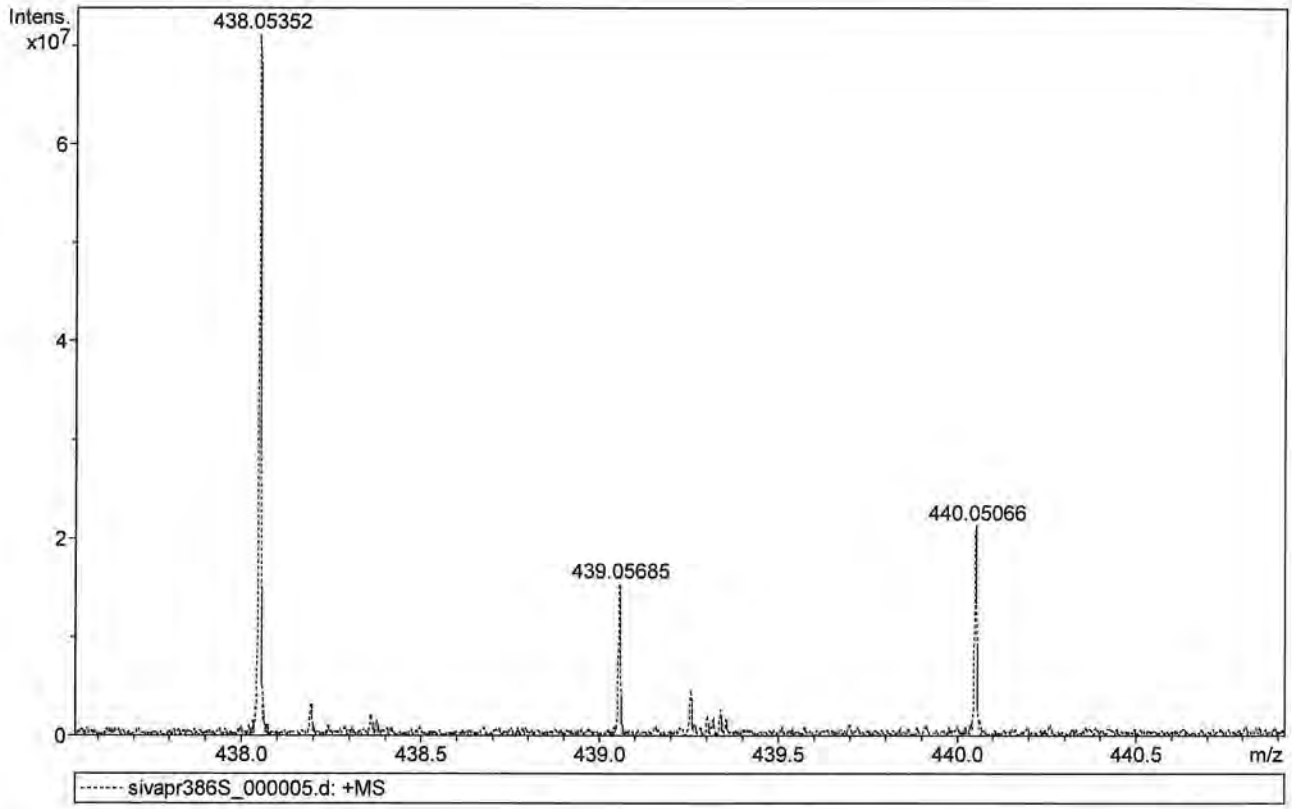
Na

$C_{21}H_{18}ClNNaO_4S$
438.0543

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\a4\sivapr386S_000005.d	1/2/2014 3:39:23 PM
Method	broadband first signal	
Sample Name	SIVA-PR3-86S	Instrument: FT-MS solariX
Comment	ESI Positive	



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSig ma	rdb	e⁻ Conf	N-R ule
438.05352	1	C 21 H 18 Cl N Na O 4 S	100.00	438.05373	0.21	0.48	48.2	12.5	even	ok

checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: sivapr357

```
Bond precision:  C-C = 0.0036 A          Wavelength=0.71073

Cell:            a=12.1035(4)    b=14.2703(6)       c=13.6727(6)
                 alpha=90        beta=97.263(2)     gamma=90
Temperature:     296 K
```

	Calculated	Reported
Volume	2342.61(16)	2342.61(16)
Space group	P 2/c	P 2/c
Hall group	-P 2yc	?
Moiety formula	C15 H11 N O2	C15 H11 N O2
Sum formula	C15 H11 N O2	C15 H11 N O2
Mr	237.25	237.25
Dx,g cm-3	1.345	1.345
Z	8	8
Mu (mm-1)	0.090	0.090
F000	992.0	992.0
F000’	992.46	
h,k,lmax	15,17,17	15,17,17
Nref	4818	4789
Tmin,Tmax	0.958,0.966	0.958,0.966
Tmin’	0.958	

```
Correction method= MULTI-SCAN

Data completeness= 0.994          Theta(max)= 26.400

R(reflections)= 0.0640( 3127)     wR2(reflections)= 0.2084( 4789)

S = 1.018                 Npar= Npar = 327
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

PLATON version of 05/02/2014; check.def file version of 05/02/2014

Datablock sivapr357 - ellipsoid plot