Supporting Information for:

A Phosphetane Catalyzes Deoxygenative Condensation of α-Keto Esters and Carboxylic Acids Via P^{III}/P^{V}=O Redox Cycling

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I. General Materials and Methods

All reagents were purchased from commercial vendors (Sigma-Aldrich, Alfa Aesar, Acros, TCI, or Oakwood Chemical) and used as received unless otherwise noted. 1,2-Dichloroethane was dried over 3 Å molecular sieves for 24 hours prior to use. All other solvents were purified under argon using a Glass Contour Solvent Purification System. ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded with Bruker DPX-300, CDPX-300, AV-360, and DRX-400 spectrometers. ^{1}H NMR chemical shifts are given in ppm with respect to solvent residual peak ($CDCl_3$, δ 7.26 ppm), $^{13}C\{^{1}H\}$ NMR shifts are given in ppm with respect to $CDCl_3$ (δ 77.2 ppm), and ^{31}P shift are given in ppm with respect to an external sample of 85% H_3PO_4 (δ 0.0 ppm). Coupling constants are reported as *J*-values in Hz. Column chromatography was performed using 230-400 mesh silica gel purchased from Silicycle as the stationary phase. High resolution EI and ESI mass spectra were obtained from the Mass Spectrometry Laboratory at the School of Chemical Sciences, University of Illinois at Urbana-Champaign.

II. Preparation of Phosphorus Compounds

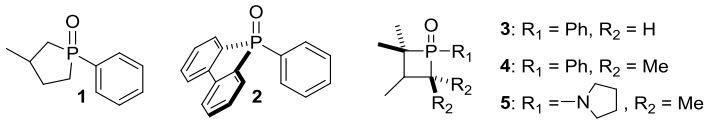


Figure S1. Phosphine *P*-oxides screened in the study.

3-Methyl-1-phenylphospholane 1-oxide (**1**): Synthesized via reduction of commercially available 3-methyl-1-phenyl-2-phospholene-1-oxide according to literature procedure.[1]

5-Phenylbenzo[b]phosphindole 5-oxide (**2**): Synthesized via oxidation of triphenylphosphine oxide according to literature procedure.[1b]

2,2,3-Trimethyl-1-phenylphosphetane 1-oxide (**3**) and *2,2,3,4,4-pentamethyl-1-phenylphosphetane 1-oxide* (**4**): Synthesized according to literature procedures.[2]

1 (a) O'Brien, C. J.; Tellez, J. L.; Nixon, Z. S.; Kang, L. J.; Carter, A. L.; Kunkel, S. R.; Przeworski, K. C.; Chass, G. A. *Angew. Chem. Int. Ed.* **2009**, *48*, 6836. (b) O'Brien, C. J.; Nixon, Z. S.; Holohan, A. J.; Kunkel, S. R.; Tellez, J. L.; Doonan, B. J.; Coyle, E. E.; Lavigne, F.; Kang, L. J.; Przeworski, K. C. *Chem. Eur. J.* **2013**, *19*, 15281.

2 Cremer, S. E.; Chorvat, R. J. *J. Org. Chem.* **1967**, *32*, 4066.

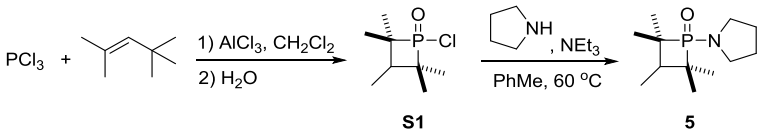


Figure S2. Synthesis of aminophosphetane *P*-oxide **5**.

1-Chloro-2,2,3,4,4-pentamethylphosphetane 1-oxide (**S1**): Synthesized according to literature procedure.[3] A round-bottom flask was charged with aluminum chloride (3.60 g, 27.0 mmol) and 15 mL anhydrous methylene chloride and cooled to 0 °C. Phosphorus trichloride (2.35 mL, 27.0 mmol) was then added via syringe at 0 °C, after which 2,4,4-trimethyl-2-pentene (4.20 mL, 27.0 mmol) was added slowly over 15 minutes. The aluminum chloride dissolved as the addition proceeded and a white precipitate formed. After addition was complete, the stirring was continued for an additional hour at 0 °C. Water (15 mL) was then added carefully at 0 °C to quench the reaction. The organic phase was separated. The aqueous layer was washed with an additional portion (15 mL) of methylene chloride. The combined organic phase was dried over anhydrous sodium sulfate. Solvent was removed in vacuo to give product **S1** as a white solid (4.3 g, 83 %, *dr > 10:1*), which could be used in the next step without further purification. Further recrystallization from hexanes gave **S1** as a single diastereomer for analysis. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 1.77 (q, 1H, J = 3.3 Hz), 1.42-1.29 (m, 12H), 0.91 (d, 3H, J = 7.1 Hz). ^{13}C NMR ($CDCl_3$, 90 MHz): δ 56.9 (d, J_{PC} = 56.5 Hz), 42.3, 25.9 (d, J_{PC} = 6.8 Hz), 18.2 (d, J_{PC} = 3.5 Hz), 7.3 (d, J_{PC} = 30.4 Hz). ^{31}P{^{1}H} NMR ($CDCl_3$, 145 MHz): δ 81.60 (major). MS (ESI) calcd for $C_8H_{17}OClP$ (M+H) 195.0706 found 195.0707.

2,2,3,4,4-Pentamethyl-1-(pyrrolidin-1-yl)phosphetane 1-oxide (**5**): To a stirred solution of phosphinic chloride **S1** (2.59 g, 13.3 mmol) in dry toluene (13 mL) was added sequentially triethylamine (2.22 mL, 16.0 mmol) and pyrrolidine (1.2 mL, 14.6 mmol). After the addition, the mixture was heated to 60 °C overnight. The reaction was then cooled to room temperature and the precipitate was removed by filtration. The solution was washed with dilute aqueous $NaHCO_3$ (~ 5% aq. soln.) and dried over anhydrous sodium sulfate. Solvent was evaporated in vacuo and the crude product was recrystallized from hexanes to yield white crystals of **5** as a single stereoisomer (2.47 g, 81 %). ^{1}H NMR ($CDCl_3$, 360 MHz): δ 3.25 (q, 4H, J = 5.4 Hz), 1.83 (s, 4H), 1.66 (d, 1H, J = 4.1 Hz), 1.23 (d, 6H, $^{3}J_{PH}$ = 17.4 Hz), 1.22 (d, 6H, $^{3}J_{PH}$ = 17.3 Hz), 0.84 (d, 3H, $^{4}J_{PH}$ = 7.1 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz):[4] δ 48.1 (d, J_{PC} = 72.8 Hz, P-**C**$(CH_3)_2$), 47.8 (d, J_{PC} = 1.4 Hz, P-C-**C**-CH_3), 43.3 (d, J_{PC} = 10.2 Hz, P-N-**C**H_2), 26.8 (d, J_{PC} = 3.2 Hz, P-C-(**C**$H_3)_2$), 26.2 (d, $^{1}J_{PC}$ = 5.7 Hz, P-C-(**C**$H_3)_2$), 19.2 (d, J_{PC} = 2.8 Hz, P-N-C-**C**H_2), 7.0 (d, J_{PC} = 22.3 Hz, P-C-C-**C**H_3). ^{31}P{^{1}H} NMR ($CDCl_3$, 145 MHz): δ 58.79 (major). MS (ESI) calcd for $C_{12}H_{25}NOP$ (M+H) 230.1674 found 230.1670.

3 (a) McBride Jr., J. J.; Jungermann, E.; Killheffer, J. V.; Clutter, R. J. *J. Org. Chem.* **1962**, *27*, 1833. (b) Coleman, D.; Edwards, P. G.; Kariuki, B. M.; Newman, P. D. *Dalton Trans.* **2010**, *39*, 3842.

4 Carbon atoms were assigned based on the following references: (a) Gary, G. A; Cremer, S. E. *Tetrahedron Lett.* **1971**, *12*, 3061. (b) Gary, G. A.; Cremer, S. E. *J. Org. Chem.* **1972**, *37*, 3458. (c) Gary, G. A.; Cremer, S. E.; Marsi, K. L. *J. Am. Chem. Soc.* **1976**, *98*, 2109.

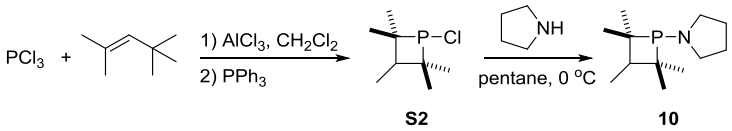


Figure S3. Synthesis of aminophosphetane **10**.

1-Chloro-2,2,3,4,4-pentamethylphosphetane (**S2**): Synthesized according to literature procedure.[5] To a suspension of 7.30 g (54.8 mmol) of anhydrous aluminum chloride in dry methylene chloride was added 5.0 mL (50 mmol) of PCl_3. The mixture was cooled to 0 °C and 7.8 mL (50 mmol) of 2,4,4-trimethyl-2-pentene was added. After stirring for 2 h at rt, a solution of triphenylphosphine (13.l g, 50 mmol) in methylene chloride was added at 0°C. The mixture was stirred at room temperature for 0.5 h and then diluted with pentane to give a biphasic mixture. The upper layer was transferred to another flask and solvent was removed. The residual air-sensitive oil was purified via reduced pressure kugelrohr distillation (50 °C @ ca. 5 mmHg). Phosphetane **S2** was obtained (7.6 g, 85% yield) as a 5:1 mixture of two diastereomers. ^{1}H NMR ($CDCl_3$, 360 MHz, mixture of two diastereomers): δ 2.81 (q, 1H, *J* = 7.1 Hz), 2.21-2.15 (m, 0.4H), 1.29-1.15 (m, 20H), 0.90 (d, 2.3H, *J* = 7.2 Hz), 0.78 (d, 3H, *J* = 7.2 Hz). ^{31}P{^{1}H} NMR ($CDCl_3$, 145 MHz): δ 169.3 (major), 149.1 (minor).

1-(2,2,3,4,4-Pentamethylphosphetan-1-yl)pyrrolidine (**10**): Synthesized according to literature procedure.[6] Pyrrolidine (2.46 mL, 30.0 mmol) was added dropwise to a 0 °C solution of **S2** (2.68 g, 15.0 mmol) in anhydrous pentane (30 mL) under nitrogen. The solution was then warmed to room temperature and stirred for additional 2 h. After filtration under nitrogen, the solvent was removed in vacuo giving product **10** as colorless liquid, which was further purified via reduced pressure kugelrohr distillation. Aminophosphetane **10** was obtained (2.27 g, 71 % yield) as a 10:3 mixture of two isomers. Characterization data was consistent with the literature report. ^{1}H NMR ($CDCl_3$, 360 MHz, mixture of two diastereomers): δ 3.22-3.14 (m, 4H), 1.66-1.60 (m, 5H), 1.23-1.02 (m, 13H), 0.78-0.68 (m, 4H). ^{31}P{^{1}H} NMR ($CDCl_3$, 145 MHz): δ 110.5 (minor), 84.1 (major).

5 Marinetti, A.; Ricard, L. *Tetrahedron* **1993**, *49*, 10291.
6 Oram, R. K.; Trippett, S. *J. Chem. Soc., Perkin Trans. 1* **1973**, 1300.

III. Evaluation of Stoichiometric Reactivity ($P^{III} \rightarrow P^{V}=O$ and $P^{V}=O \rightarrow P^{III}$)

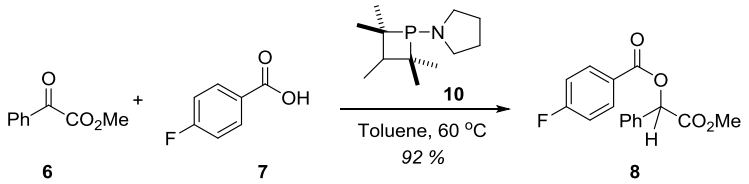


Figure S4. Stoichiometric reductive condensation of methyl benzoylformate and 4-fluorobenzoic acid.

*Stoichiometric reductive condensation of **6** and **7** by phosphorus(III) reagent **10**:* 4-Fluorobenzoic acid (100 mg, 0.71 mmol) was dissolved in anhydrous toluene (2.4 mL) in an oven-dried 20 mL vial under nitrogen. Methyl benzoylformate (0.1 mL, 0.68 mmol) was then added to the solution. A solution of phosphorus(III) reagent **10** (160 mg, 0.75 mmol) in toluene (1 mL) was then added in one portion. The reaction mixture was heated to 60 °C for 5 h. Crude ^{31}P NMR analysis showed complete conversion from phosphorus(III) (mixture of isomers, 110.5 an 84.1 ppm) to phosphorus(V) oxide (mixture of isomers, 58.0 and 56.3 ppm). After cooling to room temperature the reaction mixture was washed with saturated sodium bicarbonate solution and concentrated in vacuo. The product **8** was isolated by column chromatograph as white solid (180 mg, 92 %).

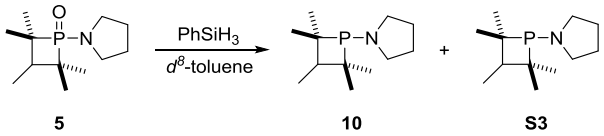


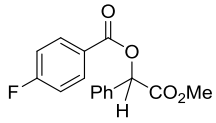
Figure S5. Reduction of aminophosphetane *P*-oxide **5** with phenylsilane.

*Reduction of aminophosphetane P-oxide **5** with phenylsilane.* To a solution of aminophosphetane(V) P-oxide **5** (74 mg, 0.32 mmol) and 1,3,5-trimethoxybenzene (internal standard, 54 mg, 0.32 mmol) in d^8-toluene (1.5 mL) was added phenylsilane (0.03 mL, 0.24 mmol) in one portion. The solution was transferred to a J-Young NMR tube and the reaction was monitored via ^{31}P NMR. Formation of aminophosphetane(III) **10** (5.7:1 *dr*; *major*: 110.5 ppm; *minor*: 84.1 ppm) was observed immediately after addition of phenylsilane, and peaks corresponding to secondary phosphetane(III) **S3** (1.1:1 *dr*; *major*: 16.3 ppm; *minor*: 13.7 ppm) were also observed. After 3 h, consumption of **5** was complete, ca. 67% of **10** and 33% of **S3** were observed based on ^{1}H NMR. With greater equivalencies of phenylsilane, the reaction continues exclusively to **S3**.

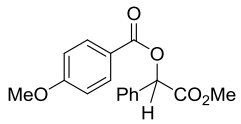
IV. General Procedure for Catalytic Reductive Condensation Reactions

General procedure: Carboxylic acid (1.2 equiv), catalyst **5** (15 or 20 mol%), solvent (toluene or 1,2-dichloroethane, 0.2 M), α-keto ester substrate (1.0 equiv), and phenylsilane (1.2 equiv) were added to a 20 mL vial sequentially. The vial was then sealed and heated to 80 ^{o}C with monitoring by TLC until completion (typically 8 – 20 h). After cooling to room temperature, a saturated aqueous sodium bicarbonate solution was added. The resulting biphasic mixture was separated and the aqueous layer was washed with additional methylene chloride. The combined organic layers were dried over anhydrous sodium sulfate and then concentrated in vacuo to a crude residue. The product was then isolated via column chromatography (silica gel, hexanes: ethyl acetate = 10:1).

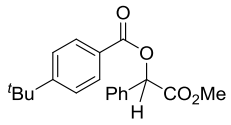
V. Characterization Data for Reductive Condensation Products



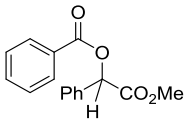
2-Methoxy-2-oxo-1-phenylethyl 4-fluorobenzoate (**8**): ^{1}H NMR ($CDCl_3$, 360 MHz): δ 8.17-8.13 (m, 2H), 7.59-7.56 (m, 2H), 7.47-7.42 (m, 3H), 7.15-7.10 (m, 2H), 6.16 (s, 1H), 3.75 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.3, 166.2 (d, $^{1}J_{CF}$ = 253.1 Hz), 165.0, 140.0, 132.7 (d, $^{3}J_{CF}$ = 9.6 Hz), 129.5, 129.0, 127.8, 125.6 (d, $^{4}J_{CF}$ = 2.9 Hz), 115.8 (d, $^{2}J_{CF}$ = 21.8 Hz), 75.1, 52.8. MS (ESI) calcd for $C_{16}H_{13}FO_4Na$ (M+Na) 311.0696 found 311.0696.



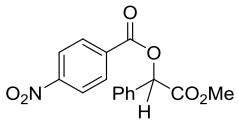
2-Methoxy-2-oxo-1-phenylethyl 4-methoxybenzoate (**11**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.11-8.08 (dd, 2H, *J* = 7.0 Hz, *J* = 1.9 Hz), 7.60-7.57 (m, 2H), 7.46-7.40 (m, 3H), 6.95-6.92 (dd, 2H, *J* = 7.1 Hz, *J* = 1.8 Hz), 6.15 (s, 1H), 3.85 (s, 3H), 3.75 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.6, 165.7, 163.9, 134.3, 132.2, 129.3, 128.9, 127.7, 121.7, 113.8, 74.7, 55.5, 52.7. MS (ESI) calcd for $C_{17}H_{16}O_5Na$ (M+Na) 323.0895 found 323.0896.



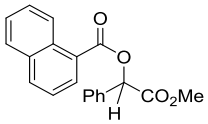
2-Methoxy-2-oxo-1-phenylethyl 4-(tert-butyl)benzoate (**12**): ^{1}H NMR ($CDCl_3$, 360 MHz): δ 8.08 (d, 2H, *J* = 8.7 Hz), 7.61-7.59 (m, 2H), 7.48-7.40 (m, 5H), 6.18 (s, 1H), 3.76 (s, 3H), 1.35 (s, 9H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.5, 166.0, 157.3, 134.2, 130.0, 129.3, 128.9, 127.7, 126.6, 125.6, 74.8, 52.7, 35.3, 31.2. MS (ESI) calcd for $C_{20}H_{22}O_4Na$ (M+Na) 349.1416 found 349.1414.



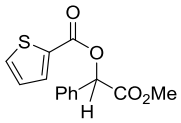
2-Methoxy-2-oxo-1-phenylethylbenzoate (**13**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.16-8.13 (m, 2H), 7.62-7.59 (m, 3H), 7.49-7.43 (m, 5H), 6.19 (s, 1H), 3.76 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.4, 166.0, 134.0, 133.6, 130.1, 129.4, 129.3, 129.0, 128.6, 127.8, 75.0, 52.8. MS (ESI) calcd for $C_{16}H_{15}O_4$ (M+H) 271.0970 found 271.0970.



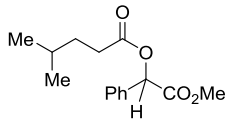
2-Methoxy-2-oxo-1-phenylethyl 4-nitrobenzoate (**14**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.33, (s, 4H), 7.59-7.56 (m, 2H), 7.49-7.44 (m, 3H), 6.20 (s, 1H), 3.78 (s. 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.9, 164.1, 151.0, 134.7, 133.4, 131.2, 129.8, 129.2, 127.9, 123.7, 75.6, 53.0. MS (ESI) calcd for $C_{16}H_{13}NO_6Na$ (M+Na) 338.0641 found 338.0641.



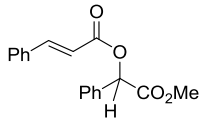
2-Methoxy-2-oxo-1-phenylethyl 1-naphthoate (**15**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.99 (d, 1H, *J* = 8.6 Hz), 8.37 (d, 1H, *J* = 7.3 Hz), 8.06 (d, 1H, *J* = 8.2 Hz), 7.90 (d, 1H, *J* = 8.0 Hz), 7.65-7.43 (m, 8H), 6.30 (s, 1H), 3.81 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.6, 166.9, 134.1, 134.0, 133.9, 131.6, 131.0, 129.4, 129.0, 128.7, 128.1, 127.8, 126.4, 126.1, 125.8, 124.6, 75.1, 52.9. MS (ESI) calcd for $C_{20}H_{17}O_4$ (M+H) 321.1127 found 321.1129.



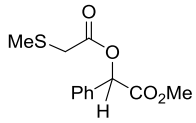
2-Methoxy-2-oxo-1-phenylethyl thiophene-2-carboxylate (**16**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.91 (t, 1H, *J* = 1.4 Hz), 7.61-7.56 (m, 3H), 7.43-7.42 (m, 3H), 7.12 (t, 1H, *J* = 3.3 Hz), 6.15 (s, 1H), 3.75 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.2, 161.5, 134.5, 133.8, 133.4, 132.6, 129.4, 128.9, 128.0, 127.7, 74.9, 52.8. MS (ESI) calcd for $C_{14}H_{13}SO_4$ (M+H) 277.0535 found 277.0531.



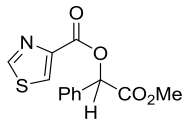
2-Methoxy-2-oxo-1-phenylethyl 4-methylpentanoate (**17**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.49-7.36 (m, 5H), 5.94 (s, 1H), 3.71 (s, 3H), 2.56-2.38 (m, 2H), 1.64-1.55 (m, 3H), 0.93-0.90 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 173.4, 169.5, 134.0, 129.3, 128.9, 127.7, 74.3, 52.6, 33.6, 32.1, 27.7, 22.3. MS (ESI) calcd for $C_{15}H_{20}O_4Na$ (M+Na) 287.1259 found 287.1262.



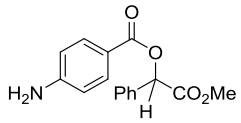
2-Methoxy-2-oxo-1-phenylethyl cinnamate (**18**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.80 (d, 1H, *J* = 16.0 Hz), 7.56-7.53 (m, 4H), 7.45-7.39 (m, 6H), 6.58 (d, 1H, *J* = 16.0 Hz), 6.09 (s, 1H), 3.76 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.5, 166.3, 146.5, 134.3, 134.0, 130.7, 129.4, 129.0, 129.0, 128.4, 127.8, 117.0, 74.6, 52.8. MS (ESI) calcd for $C_{18}H_{17}O_4$ (M+H) 297.1127 found 297.1125.



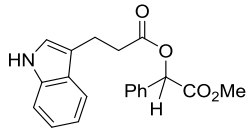
Methyl 2-(2-(methylthio)acetoxy)-2-phenylacetate (**19**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.49-7.45 (m, 2H), 7.41-7.38 (m, 3H), 5.97 (s, 1H), 3.73 (s, 3H), 3.33 (s, 2H), 2.25 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.8, 169.1, 133.5, 129.5, 129.0, 127.8, 75.1, 52.8, 35.5, 16.4. MS (ESI) calcd for $C_{12}H_{15}SO_4$ (M+H) 255.0691 found 255.0689.



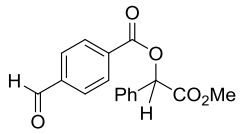
2-Methoxy-2-oxo-1-phenylethyl thiazole-4-carboxylate (**20**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.86 (d, 1H, *J* = 2.0 Hz), 8.35 (d, 1H, *J* = 2.1 Hz), 7.58-7.55 (m, 2H), 7.43-7.39 (m, 3H), 6.21 (s, 1H), 3.74 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.0, 160.4, 153.8, 147.0, 133.6, 129.5, 129.0, 128.6, 127.9, 75.1, 52.9. MS (ESI) calcd for $C_{13}H_{12}NO_4S$ (M+H) 278.0487 found 278.0478.



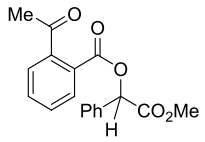
2-Methoxy-2-oxo-1-phenylethyl 4-aminobenzoate (**21**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.93 (d, 2H, *J* = 8.7 Hz), 7.59-7.56 (m, 2H), 7.43-7.41 (m, 3H), 6.63 (d, 2H, *J* = 8.7 Hz), 6.12 (s, 1H), 3.74 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.9, 166.0, 151.5, 134.5, 132.3, 129.2, 128.9, 127.7, 118.6, 113.9, 74.5, 52.7. MS (ESI) calcd for $C_{16}H_{16}NO_4$ (M+H) 286.1079 found 286.1080.



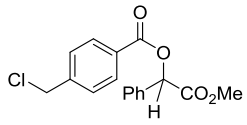
2-Methoxy-2-oxo-1-phenylethyl 3-(1H-indol-3-yl)propanoate (**22**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.01 (br, 1H), 7.57 (d, 1H, *J* = 7.8 Hz), 7.45-7.29 (m, 6H), 7.21-7.07 (m, 2H), 6.95 (d, 1H, *J* = 2.2 Hz), 5.95 (s, 1H), 3.69 (s, 3H), 3.14 (t, 2H, *J* = 7.6 Hz), 2.89-2.83 (m, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 172.9, 169.6, 136.3, 133.8, 129.3, 128.9, 127.7, 127.2, 122.1, 121.6, 119.4, 118.7, 114.6, 111.3, 74.5, 52.7, 34.7, 20.5. MS (ESI) calcd for $C_{20}H_{20}NO_4$ (M+H) 338.1392 found 338.1380.



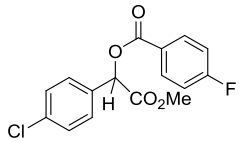
2-Methoxy-2-oxo-1-phenylethyl 4-formylbenzoate (**23**): ^{1}H NMR ($CDCl_3$, 360 MHz): δ 10.11 (s, 1H), 8.28 (d, 2H, *J* = 8.2 Hz), 7.98-7.96 (m, 2H), 7.59-7.56 (m, 2H), 7.46-7.43 (m, 3H), 6.18 (s, 1H), 3.76 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 191.7, 169.1 165.0, 139.6, 134.3, 133.6, 130.7, 129.7, 127.8, 75.4, 53.0. MS (ESI) calcd for $C_{17}H_{14}O_5Na$ (M+Na) 321.0739 found 321.0738.



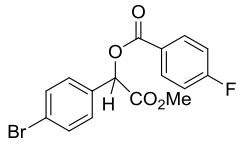
2-Methoxy-2-oxo-1-phenylethyl 2-acetylbenzoate (**24**): ^{1}H NMR ($CDCl_3$, 360 MHz): δ 8.01-7.99 (m, 1H), 7.61-7.57 (m, 1H), 7.54-7.50 (m, 3H), 7.42-7.40 (m, 4H), 6.14 (s, 1H), 3.76 (s, 3H), 2.50 (s, 3H). ^{13}C NMR ($CDCl_3$, 90 MHz): δ 203.1, 169.1, 166.2, 143.3, 133.5, 132.7, 130.3, 130.2, 129.5, 129.0, 127.9, 127.8, 126.7, 75.6, 52.9, 30.3. MS (ESI) calcd for $C_{18}H_{17}O_5$ (M+H) 313.1076 found 313.1073.



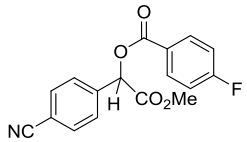
2-Methoxy-2-oxo-1-phenylethyl 4-(chloromethyl)benzoate (**25**): ^{1}H NMR ($CDCl_3$, 360 MHz): δ 8.12 (d, 2H, *J* = 8.3 Hz), 7.59-7.57 (m, 2H), 7.49-7.43 (m, 5H), 6.17 (s, 1H), 4.62 (s, 2H), 3.76 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.3, 165.5, 143.0, 133.9, 130.5, 129.5, 129.3, 129.0, 128.7, 127.8, 75.1, 52.9, 45.4. MS (ESI) calcd for $C_{17}H_{16}ClO_4$ (M+H) 319.0737 found 319.0726.



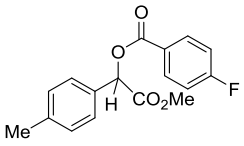
1-(4-Chlorophenyl)-2-methoxy-2-oxoethyl 4-fluorobenzoate (**26**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.15-8.11 (m, 2H), 7.52-7.50 (m, 2H), 7.43-7.39 (m, 3H), 7.14 (t, 2H, *J* = 8.7 Hz), 6.12 (s, 1H), 3.76 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.3, 166.6 (d, $^{1}J_{CF}$ = 253.6 Hz), 165.1, 135.8, 133.0 (d, $^{3}J_{CF}$ = 9.5 Hz), 132.7, 129.6, 129.4, 125.7 (d, $^{4}J_{CF}$ = 2.9 Hz), 116.2 (d, $^{2}J_{CF}$ = 22.0 Hz), 74.6, 53.3. MS (ESI) calcd for $C_{16}H_{12}ClFO_4Na$ (M+Na) 345.0306 found 345.0306.



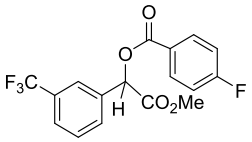
1-(4-Bromophenyl)-2-methoxy-2-oxoethyl 4-fluorobenzoate (**27**): ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.14-8.10 (m, 2H), 7.56 (d, 2H, *J* = 8.4 Hz), 7.44 (d, 2H, *J* = 8.4 Hz), 7.15-7.11 (m, 2H), 6.10 (s, 1H), 3.75 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.9, 166.3 (d, $^{1}J_{CF}$ = 253.6 Hz), 164.9, 133.0, 132.7 (d, $^{3}J_{CF}$ = 9.5 Hz), 132.3, 129.4, 125.4 (d, $^{4}J_{CF}$ = 2.9 Hz), 123.8, 115.9 (d, $^{2}J_{CF}$ = 21.9 Hz), 74.4, 53.0. MS (ESI) calcd for $C_{16}H_{12}BrFO_4Na$ (M+Na) 388.9801 found 388.9804.



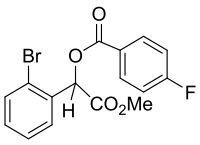
1-(4-Cyanophenyl)-2-methoxy-2-oxoethyl 4-fluorobenzoate (**28**): ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.16-8.12 (m, 2H), 7.73 (d, 2H, *J* = 8.2 Hz), 7.70 (d, 2H, *J* = 8.3 Hz), 7.18-7.13 (m, 2H), 6.21 (s, 1H), 3.77 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.3, 166.4 (d, $^{1}J_{CF}$ = 254.0 Hz), 164.6, 138.9, 132.8 (d, $^{3}J_{CF}$ = 8.8 Hz), 128.3, 125.1 (d, $^{4}J_{CF}$ = 3.0 Hz), 118.3, 116.0 (d, $^{2}J_{CF}$ = 21.9 Hz), 113.5, 74.1, 53.2. MS (ESI) calcd for $C_{17}H_{13}FNO_4$ (M+H) 314.0829 found 314.0835.



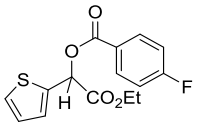
2-Methoxy-2-oxo-1-(p-tolyl)ethyl 4-fluorobenzoate (**29**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.13 (q, 2H, *J* = 4.8 Hz), 7.45 (d, 2H, *J* = 8.0 Hz), 7.24 (d, 2H, *J* = 8.7 Hz), 7.12 (t, 2H, *J* = 8.7 Hz), 6.11 (s, 1H), 3.75 (s, 3H), 2.38 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.5, 166.2 (d, $^{1}J_{CF}$ = 254.0 Hz), 165.1, 139.6, 132.7 (d, $^{3}J_{CF}$ = 9.4 Hz), 131.0, 129.7, 127.8, 125.6 (d, $^{4}J_{CF}$ = 2.8 Hz), 115.8 (d, $^{2}J_{CF}$ = 22.0 Hz), 75.0, 52.8, 21.4. MS (ESI) calcd for $C_{17}H_{15}FO_4Na$ (M+Na) 325.0852 found 325.0851.



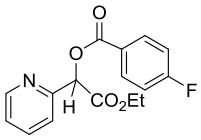
2-Methoxy-2-oxo-1-(3-(trifluoromethyl)phenyl)ethyl 4-fluorobenzoate (**30**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.07-8.02 (m, 2H), 7.74 (s, 1H), 7.68 (d, 1H, *J* = 7.5 Hz), 7.59 (d, 1H, *J* = 7.6 Hz), 7.49 (d, 1H, *J* = 7.7 Hz), 7.08-7.02 (m, 2H), 6.12 (s, 1H), 3.68 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.7, 166.3 (d, $^{1}J_{CF}$ = 253.9 Hz), 164.8, 135.0, 132.8 (d, $^{3}J_{CF}$ = 9.5 Hz), 131.5 (d, J_{CF} = 32.6 Hz), 131.1, 129.6, 126.4 (q, J_{CF} = 3.6 Hz), 125.2, (d, $^{4}J_{CF}$ = 2.9 Hz), 124.5 (q, J_{CF} = 3.7 Hz), 123.9 (d, J_{CF} = 271.0 Hz), 116.0 ($^{2}J_{CF}$ = 21.9 Hz), 74.3, 53.1. MS (ESI) calcd for $C_{17}H_{12}F_4O_4Na$ (M+Na) 379.0569 found 379.0568.



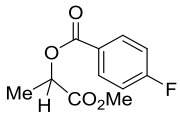
1-(2-Bromophenyl)-2-methoxy-2-oxoethyl 4-fluorobenzoate (**31**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.13 (q, 2H, *J* = 4.8 Hz), 7.45 (d, 2H, *J* = 8.0 Hz), 7.24 (d, 2H, *J* = 8.7 Hz), 7.12 (t, 2H, *J* = 8.7 Hz), 6.11 (s, 1H), 3.75 (s, 3H), 2.38 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.9, 166.3 (d, $^{1}J_{CF}$ = 253.4 Hz), 164.8, 134.0, 133.6, 132.8 (d, $^{3}J_{CF}$ = 9.3 Hz), 131.0, 129.9, 128.1, 125.4 (d, $^{4}J_{CF}$ = 2.9 Hz), 124.4, 115.8 (d, $^{2}J_{CF}$ = 22.1 Hz), 74.0, 53.0. MS (ESI) calcd for $C_{16}H_{12}BrFO_4Na$ (M+Na) 388.9801 found 388.9800.



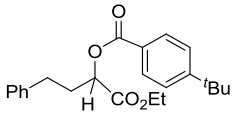
2-Ethoxy-2-oxo-1-(thiophen-2-yl)ethyl 4-fluorobenzoate (**32**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.13 (t, 2H, *J* = 6.9 Hz), 7.40 (d, 1H, *J* = 5.0 Hz), 7.12 (t, 2H, *J* = 8.5 Hz), 7.05 (d, 1H, *J* = 3.9 Hz), 6.39 (s, 1H), 4.32-4.22 (m, 2H), 1.27 (t, 3H, *J* = 7.1 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.0, 166.3 (d, $^{1}J_{CF}$ = 253.2 Hz), 164.8, 135.6, 132.8 (d, $^{3}J_{CF}$ = 9.4 Hz), 128.0, 127.3, 127.2, 125.4 (d, $^{4}J_{CF}$ = 2.9 Hz), 115.8 (d, $^{2}J_{CF}$ = 21.9 Hz), 71.0, 62.3, 14.2. MS (ESI) calcd for $C_{15}H_{13}SFO_4Na$ (M+Na) 331.0416 found 331.0418.



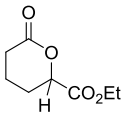
2-Ethoxy-2-oxo-1-(pyridin-2-yl)ethyl 4-fluorobenzoate (**33**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.66 (d, 1H, *J* = 4.8 Hz), 8.17-8.12 (m, 2H), 7.79-7.78 (m, 1H), 7.58 (d, 1H, *J* = 7.9 Hz), 7.35-7.33 (m, 1H), 7.15-7.09 (m, 2H), 6.30 (s, 1H), 4.32-4.25 (m, 2H), 1.25 (t, 3H, *J* = 7.1 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 168.0, 166.2 (d, $^{1}J_{CF}$ = 253.2 Hz), 164.8, 153.9, 150.0, 137.3, 132.8 (d, $^{3}J_{CF}$ = 9.4 Hz), 125.5, 124.1, 122.9, 115.8 (d, $^{2}J_{CF}$ = 22.0 Hz), 76.2, 62.2, 14.2. MS (ESI) calcd for $C_{16}H_{15}FNO_4$ (M+H) 304.0985 found 304.0981.



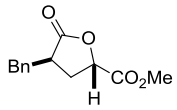
1-Methoxy-1-oxopropan-2-yl 4-fluorobenzoate (**34**): ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.13-8.08 (m, 2H), 7.16-7.09 (m, 2H), 5.35-5.28 (m, 1H), 3.77 (s, 3H), 1.63 (t, 3H, *J* = 6.9 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 171.3, 166.1 (d, $^{1}J_{CF}$ = 252.8 Hz), 165.1, 132.6 (d, $^{3}J_{CF}$ = 9.3 Hz), 125.8 (d, $^{4}J_{CF}$ = 2.9 Hz), 115.7 (d, $^{2}J_{CF}$ = 22.0 Hz), 69.3, 52.6, 17.2. MS (ESI) calcd for $C_{11}H_{11}FO_4Na$ (M+Na) 249.0539 found 249.0530.



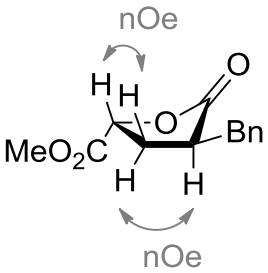
1-Ethoxy-1-oxo-4-phenylbutan-2-yl 4-(tert-butyl)benzoate (**35**): ^{1}H NMR ($CDCl_3$, 360 MHz): δ 8.02 (d, 2H, *J* = 8.5 Hz), 7.49 (d, 2H, *J* = 8.5 Hz), 7.30 (t, 2H, 7.4 Hz), 7.22 (t, 3H, 3.8 Hz), 5.22 (t, 1H, *J* = 6.4 Hz), 4.22 (q, 2H, *J* = 7.1 Hz), 2.85 (dt, 2H, *J* = 8.0 Hz, *J* = 2.0 Hz), 2.31 (dq, 2H, *J* = 7.5 Hz, *J* = 2.8 Hz), 1.36 (s, 9H), 1.27 (t, 3H, *J* = 7.1 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 170.3, 166.1, 157.2, 140.7, 129.9, 128.7, 128.6, 126.8, 126.4, 125.6, 72.1, 61.5, 35.3, 33.1, 31.6, 31.2, 14.3. MS (ESI) calcd for $C_{23}H_{29}O_4$ (M+H) 369.2066 found 369.2063.



Ethyl 6-oxotetrahydro-2H-pyran-2-carboxylate (**36**): ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.93 (t, 1H, *J* = 5.2 Hz), 4.31-4.20 (m, 2H), 2.66-2.50 (m, 2H), 1.29 (t, 3H, *J* = 7.1 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.8, 169.3, 76.7, 62.1, 29.6, 25.0, 17.6, 14.2. MS (ESI) calcd for $C_8H_{13}O_4$ (M+H) 173.0814 found 173.0815.



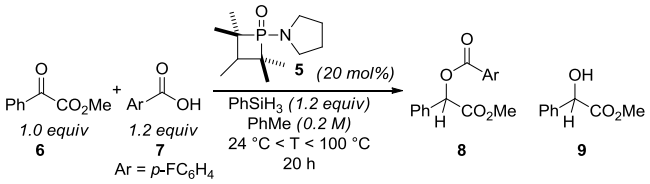
Methyl 4-benzyl-5-oxotetrahydrofuran-2-carboxylate (**37**): Major isomer ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.33-7.17 (m, 5H), 4.79 (t, 1H, *J* = 7.5 Hz), 3.78 (s, 3H), 3.30 (dd, 1H, *J* = 4.1 Hz, *J* = 13.9 Hz), 2.99-2.90 (m, 1H), 2.74 (dd, 1H, *J* = 10.2 Hz, *J* = 13.9 Hz), 2.60-2.52 (m, 1H), 2.07-1.99 (m, 1H). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 176.7, 169.9, 138.1, 128.9, 128.9, 127.0, 74.3, 52.9, 41.5, 36.4, 31.6. Minor isomer ^{1}H NMR ($CDCl_3$, 360 MHz): δ 7.34-7.18 (m, 5H), 4.79, (dd, 1H, *J* = 2.8 Hz, *J* = 8.5 Hz), 3.77 (s, 3H), 3.25 (dd, 1H, *J* = 4.0 Hz, *J* = 13.8 Hz), 3.04-2.96 (m, 1H), 2.77 (dd, 1H, *J* = 9.4 Hz, *J* = 13.8 Hz), 2.37-2.24 (m, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 177.5, 170.5, 137.9, 129.0, 129.0, 127.1, 74.0, 52.9, 39.5, 36.3, 31.7. MS (ESI): *submitted*. The relative stereochemistry of the major isomer was assigned as *trans* on the basis of a NOESY experiment.



VI. Temperature Dependence Study.

4-Fluorobenzoic acid (47 mg, 0.34 mmol), catalyst **5** (13 mg, 20 mol%), anhydrous toluene(1.4 mL, 0.2 M), methyl benzoylformate (0.04 ml, 0.28 mmol) and phenylsilane (0.04 mL, 0.34 mmol) were added to an oven-dried 20 mL vial sequentially. The vial was then sealed and placed at designated temperatures (100 ^{o}C, 75 ^{o}C, 50 ^{o}C, and room temperature ~ 24 ^{o}C) until completed (reaction was monitored by TLC). Saturated aqueous sodium bicarbonate was then added to the reaction mixture. Layers were separated and the aqueous layer was washed with methylene chloride. The combined organic layer was dried over anhydrous sodium sulfate and was concentrated *in vacuo*. Products **8** and **9** were then isolated via column chromatograph (silica gel, hexanes: ethyl acetate = 10:1).

Table 4 (from text). Dependence of product selectivity on temperature.



Entry	T (°C)	Yield[a] (%)	
		8	**9**
1	100	84	7
2	75	73	22
3	50	48	43
4	24	18	74

[a] Yields are for isolated products.

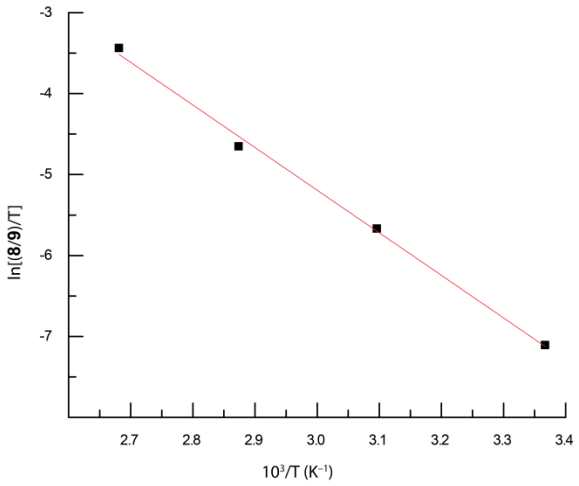
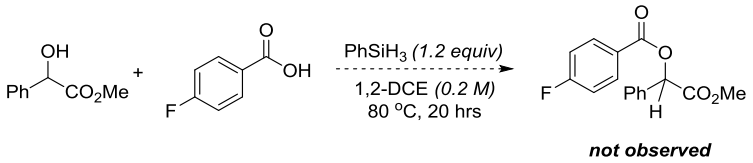
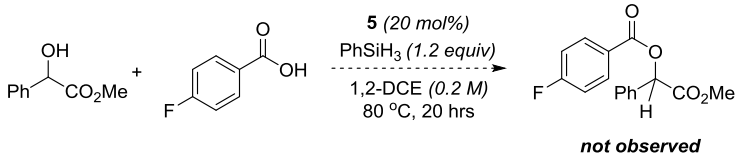


Figure 2 (from text). Correlation between product selectivity and temperature over the range 24 °C < *T* < 100 °C (see Table 4). Linear fit: $\ln[(\mathbf{8}/\mathbf{9})/T] = -5.26T^{-1} + 10.59$, $R^2 = 0.995$.

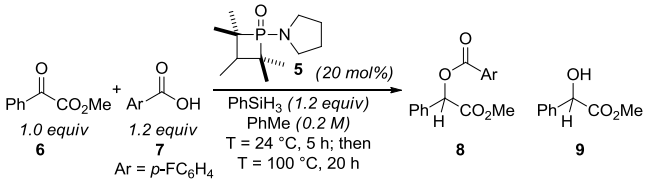
VII. Mechanistic Control Experiments



*Control experiment I - Attempted reaction with methyl mandelate, omitting **5**:* 4-Fluorobenzoic acid (68 mg, 0.49 mmol)%) and methyl mandelate (67 mg, 0.40 mmol) were dissolved in dry 1,2-dichloroethane in an oven-dried 20 mL vial. Phenylsilane (0.06 mL, 0.49 mmol) was added to the mixture in one portion. The vial was then sealed and heated to 80 °C for 20 hours. After cooling to room temperature, the crude reaction mixture was analyzed by TLC and ^{1}H NMR. No condensation product was observed.



*Control experiment II – Attempted reaction with methyl mandelate, including **5**:* 4-Fluorobenzoic acid (68 mg, 0.49 mmol)%), aminophosphetane *P*-oxide **5** (19 mg, 20 mmol%) and methyl mandelate (67 mg, 0.40 mmol) were dissolved in dry 1,2-dichloroethane in an oven-dried 20 mL vial. Phenylsilane (0.06 mL, 0.49 mmol) was added to the mixture in one portion. The vial was then sealed and heated to 80 °C for 20 hours. After cooling to room temperature, the crude reaction mixture was analyzed by TLC and ^{1}H NMR. No condensation product was observed.



Control experiment III – Temperature swing: 4-Fluorobenzoic acid (108 mg, 0.77 mmol), catalyst **5** (32 mg, 20 mol%), anhydrous toluene (3.5 mL, 0.2 M), methyl benzoylformate (0.10 ml, 0.70 mmol) and phenylsilane (0.1 mL, 0.84 mmol) were added to an oven-dried 20 mL vial sequentially. The vial was then sealed and stirred at room temperature until methyl benzoylformate was completely consumed (ca. 5 hours as monitored by TLC). The reaction mixture was separated into two portions. **Portion A**: Saturated aqueous sodium bicarbonate was added to reaction mixture. Layers were separated and the aqueous layer was washed with methylene chloride. The combined organic layer was dried over anhydrous sodium sulfate and was concentrated in vacuo. Products **8** (16 mg, 10 %) and **9** (48 mg, 41 %) were then isolated via column chromatograph (silica gel, hexanes: ethyl acetate = 10:1). **Portion B**: The vial was sealed and heated to 100 °C overnight. After cooling to room temperature, the reaction was worked up in the same procedure as portion A, giving product **8** (14 mg, 9 %) and **9** (41 mg, 35 %).

VIII. Crystallographic Details.

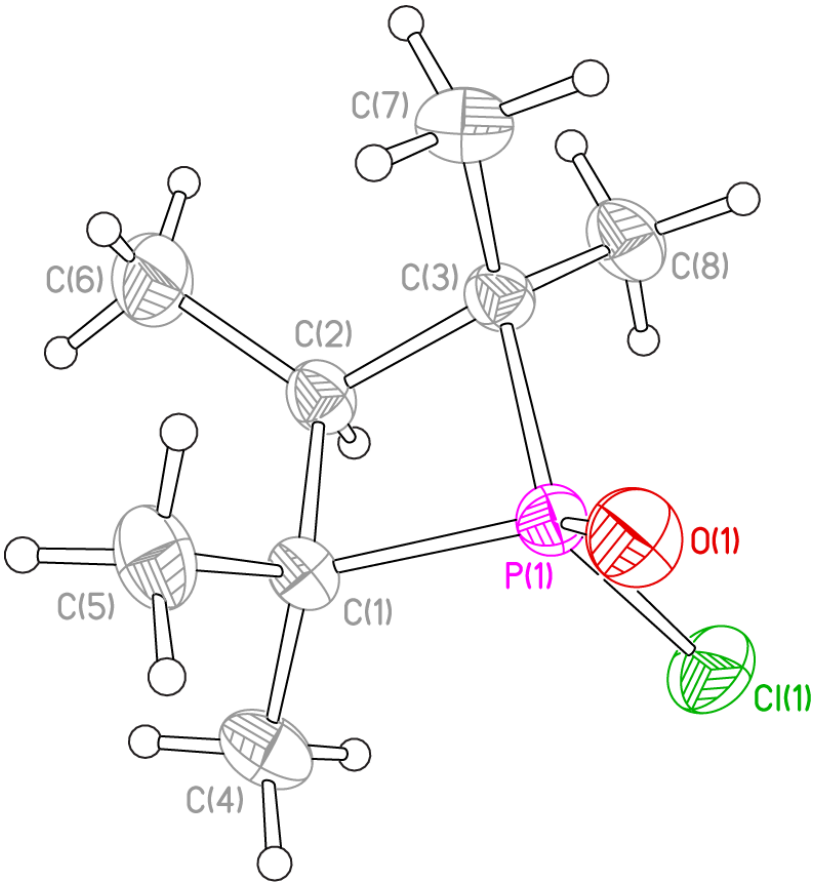


Figure S6. Thermal ellipsoid plot for **S1** depicted at the 50% probability level.

A colorless block shaped crystal of **S1** (C8 H16 Cl O P) with approximate dimensions 0.11 x 0.13 x 0.28 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 203(2) K, cooled by Rigaku-MSC X-Stream 2000, on a Bruker SMART APEX CCD area detector system equipped with a graphite monochromator and a MoKα fine-focus sealed tube (λ = 0.71073Å) operated at 1600 watts power (50 kV, 32 mA). The detector was placed at a distance of 5.8 cm from the crystal.

A total of 1850 frames were collected with a scan width of 0.3º in ω and an exposure time of 10 seconds/frame. The total data collection time was about 8 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame integration algorithm. The integration of the data using a Monoclinic unit cell yielded a total of 9388 reflections to a maximum θ angle of 28.29° (0.90 Å resolution), of which 2534 were independent, completeness = 99.2%, R_{int} = 0.0217, R_{sig} = 0.0223 and 2271 were greater than 2σ(I). The final cell constants: a = 7.7635(11)Å, b = 6.8938(9)Å, c = 19.576(3)Å, α = 90°, β = 101.058(2)°, γ = 90°, volume = 1028.3(2)$Å^3$, are based upon the refinement of the XYZ-centroids of 4175 reflections above 20σ(I) with 2.680° <θ <28.112°. Analysis of the data showed

negligible decay during data collection. Data were corrected for absorption effects using the multiscan technique (SADABS). The ratio of minimum to maximum apparent transmission was 0.8128.

The structure was solved and refined using the Bruker SHELXTL (Version 6.1) Software Package, using the space group P2(1)/n, with Z = 4 for the formula unit, C8 H16 Cl O P. The hydrogen atoms were placed geometrically and rode their parent atoms. The final anisotropic full-matrix least-squares refinement on F^2 with 106 variables converged at R1 = 3.46%, for the observed data and wR2 = 9.80% for all data. The goodness-of-fit was 0.913. The largest peak on the final difference map was 0.405 $e^-/Å^3$ and the largest hole was -0.365 $e^-/Å^3$. Based on the final model, the calculated density of the crystal is 1.257 g/cm^3 and F(000) amounts to 416 electrons.

Table S1. Sample and crystal data for **S1.**

Identification code	S1	
Empirical formula	C8 H16 Cl O P	
Formula weight	194.63	
Temperature	203(2) K	
Wavelength	0.71073 Å	
Crystal size	0.28 x 0.13 x 0.11 mm	
Crystal habit	colorless block	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 7.7635(11) Å	α= 90°
	b = 6.8938(9) Å	β= 101.058(2)°
	c = 19.576(3) Å	γ = 90°
Volume	1028.3(2) $Å^3$	
Z	4	
Density (calculated)	1.257 g/cm^3	
Absorption coefficient	0.476 mm^{-1}	
F(000)	416	

Table S2. Atomic coordinates and equivalent isotropic atomic displacement parameters ($Å^2$) for **S1.** U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C1	0.96836(18)	0.5754(2)	0.15989(7)	0.0267(3)
C2	0.88463(18)	0.7841(2)	0.14958(8)	0.0285(3)
C3	0.74557(18)	0.7532(2)	0.08040(7)	0.0259(3)
C4	0.9924(2)	0.5013(3)	0.23448(8)	0.0400(4)
C5	1.1382(2)	0.5479(3)	0.13247(9)	0.0400(4)
C6	1.0107(3)	0.9536(3)	0.15054(12)	0.0542(5)
C7	0.8064(2)	0.8146(3)	0.01367(8)	0.0381(4)
C8	0.5662(2)	0.8397(2)	0.08210(9)	0.0361(4)
Cl1	0.59571(5)	0.40270(7)	0.15012(2)	0.04555(14)
O1	0.79580(16)	0.35311(17)	0.03999(6)	0.0388(3)
P1	0.78055(5)	0.49317(5)	0.095097(19)	0.02506(12)

Table S3. Bond lengths (Å) for **S1.**

C1-C4	1.525(2)	C1-C5	1.528(2)
C1-C2	1.576(2)	C1-P1	1.8294(14)
C2-C6	1.522(2)	C2-C3	1.5762(19)
C2-H2	0.9900	C3-C8	1.521(2)
C3-C7	1.531(2)	C3-P1	1.8276(15)
C4-H4A	0.9700	C4-H4B	0.9700
C4-H4C	0.9700	C5-H5A	0.9700
C5-H5B	0.9700	C5-H5C	0.9700
C6-H6A	0.9700	C6-H6B	0.9700
C6-H6C	0.9700	C7-H7A	0.9700
C7-H7B	0.9700	C7-H7C	0.9700
C8-H8A	0.9700	C8-H8B	0.9700
C8-H8C	0.9700	Cl1-P1	2.0498(6)
O1-P1	1.4694(11)		

Table S4. Bond angles (°) for **S1.**

C4-C1-C5	109.85(13)	C4-C1-C2	113.60(12)
C5-C1-C2	115.53(13)	C4-C1-P1	119.68(12)
C5-C1-P1	110.17(11)	C2-C1-P1	86.55(8)
C6-C2-C3	116.55(13)	C6-C2-C1	116.81(13)
C3-C2-C1	100.63(10)	C6-C2-H2	107.4
C3-C2-H2	107.4	C1-C2-H2	107.4
C8-C3-C7	110.10(13)	C8-C3-C2	114.04(12)
C7-C3-C2	115.04(12)	C8-C3-P1	119.34(11)
C7-C3-P1	110.16(10)	C2-C3-P1	86.60(8)
C1-C4-H4A	109.5	C1-C4-H4B	109.5
H4A-C4-H4B	109.5	C1-C4-H4C	109.5
H4A-C4-H4C	109.5	H4B-C4-H4C	109.5
C1-C5-H5A	109.5	C1-C5-H5B	109.5
H5A-C5-H5B	109.5	C1-C5-H5C	109.5
H5A-C5-H5C	109.5	H5B-C5-H5C	109.5
C2-C6-H6A	109.5	C2-C6-H6B	109.5
H6A-C6-H6B	109.5	C2-C6-H6C	109.5
H6A-C6-H6C	109.5	H6B-C6-H6C	109.5
C3-C7-H7A	109.5	C3-C7-H7B	109.5
H7A-C7-H7B	109.5	C3-C7-H7C	109.5
H7A-C7-H7C	109.5	H7B-C7-H7C	109.5
C3-C8-H8A	109.5	C3-C8-H8B	109.5
H8A-C8-H8B	109.5	C3-C8-H8C	109.5
H8A-C8-H8C	109.5	H8B-C8-H8C	109.5
O1-P1-C3	124.02(7)	O1-P1-C1	123.15(7)
C3-P1-C1	83.10(6)	O1-P1-Cl1	110.42(6)
C3-P1-Cl1	106.46(5)	C1-P1-Cl1	105.97(5)

Table S5. Torsion angles (°) for **S1.**

C4-C1-C2-C6	97.36(18)	C5-C1-C2-C6	-30.91(19)
P1-C1-C2-C6	-141.65(14)	C4-C1-C2-C3	-135.45(13)
C5-C1-C2-C3	96.28(14)	P1-C1-C2-C3	-14.46(10)
C6-C2-C3-C8	-97.44(18)	C1-C2-C3-C8	135.20(13)
C6-C2-C3-C7	31.1(2)	C1-C2-C3-C7	-96.21(14)
C6-C2-C3-P1	141.83(14)	C1-C2-C3-P1	14.47(10)
C8-C3-P1-O1	106.28(13)	C7-C3-P1-O1	-22.50(14)
C2-C3-P1-O1	-137.96(9)	C8-C3-P1-C1	-128.07(12)
C7-C3-P1-C1	103.16(11)	C2-C3-P1-C1	-12.30(9)
C8-C3-P1-Cl1	-23.40(12)	C7-C3-P1-Cl1	-152.17(9)
C2-C3-P1-Cl1	92.37(8)	C4-C1-P1-O1	-105.95(13)
C5-C1-P1-O1	22.78(15)	C2-C1-P1-O1	138.75(9)
C4-C1-P1-C3	127.60(13)	C5-C1-P1-C3	-103.66(12)
C2-C1-P1-C3	12.31(9)	C4-C1-P1-Cl1	22.40(13)
C5-C1-P1-Cl1	151.14(11)	C2-C1-P1-Cl1	-92.89(8)

Table S6. Anisotropic atomic displacement parameters ($Å^2$) for **S1.** The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2a^{*2} U_{11} + ... + 2hka^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0248(6)	0.0288(7)	0.0257(7)	-0.0004(5)	0.0025(5)	0.0033(5)
C2	0.0288(7)	0.0268(7)	0.0281(7)	-0.0039(5)	0.0004(5)	0.0018(5)
C3	0.0272(6)	0.0234(6)	0.0258(6)	-0.0006(5)	0.0020(5)	0.0024(5)
C4	0.0454(9)	0.0452(10)	0.0270(8)	0.0030(6)	0.0005(7)	0.0094(7)
C5	0.0258(7)	0.0519(10)	0.0426(9)	-0.0035(8)	0.0072(6)	0.0060(7)
C6	0.0509(11)	0.0344(9)	0.0674(13)	-0.0003(9)	-0.0135(10)	-0.0107(8)
C7	0.0453(9)	0.0371(9)	0.0328(8)	0.0072(6)	0.0096(7)	0.0027(7)
C8	0.0315(7)	0.0360(8)	0.0386(8)	-0.0026(7)	0.0014(6)	0.0103(6)
Cl1	0.0363(2)	0.0473(3)	0.0563(3)	0.01039(19)	0.01688(19)	-0.00771(17)
O1	0.0459(6)	0.0310(6)	0.0375(6)	-0.0099(5)	0.0032(5)	0.0027(5)
P1	0.02503(19)	0.0232(2)	0.0266(2)	-0.00074(13)	0.00406(14)	0.00034(13)

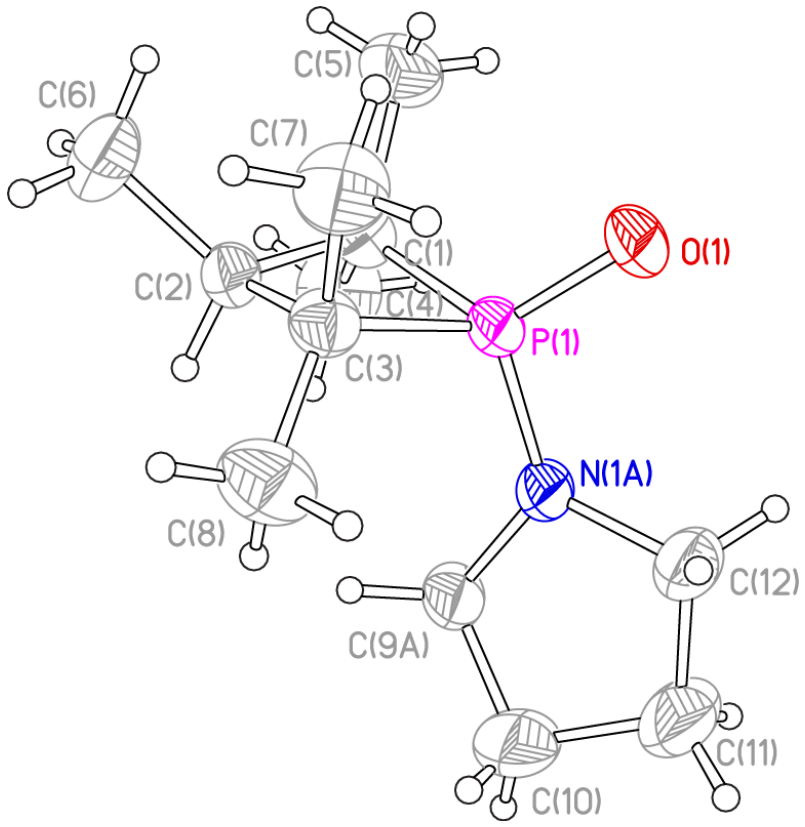


Figure S7. Thermal ellipsoid plot for **5** depicted at the 50% probability level. Only the major component of the disordered pyrrolidine moiety is shown for clarity.

A colorless plate shaped crystal of **5** (C12 H24 N O P) with approximate dimensions 0.12 x 0.16 x 0.19 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 203(2) K, cooled by Rigaku-MSC X-Stream 2000, on a Bruker SMART APEX CCD area detector system equipped with a graphite monochromator and a MoKα fine-focus sealed tube (λ = 0.71073Å) operated at 1600 watts power (50 kV, 32 mA). The detector was placed at a distance of 5.8 cm from the crystal.

A total of 1850 frames were collected with a scan width of 0.3º in ω and an exposure time of 10 seconds/frame. The total data collection time was about 8 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame integration algorithm. The integration of the data using a Triclinic unit cell yielded a total of 6358 reflections to a maximum θ angle of 28.28° (0.90 Å resolution), of which 3200 were independent, completeness = 96.8%, R_{int} = 0.0156, R_{sig} = 0.0216 and 2912 were greater than 2σ(I). The final cell constants: a = 6.0522(11) Å, b = 9.8966(18) Å, c = 11.401(2) Å, α = 94.862(4)°, β = 93.274(4)°, γ = 100.155(4)°, volume = 668.0(2) $Å^3$, are based upon the refinement of the XYZ-centroids of 4077 reflections above 20σ(I) with 2.629° <θ <28.230°. Analysis of the data showed negligible decay during data collection. Data were corrected for absorption effects using the multiscan technique (SADABS). The ratio of minimum to maximum apparent transmission was 0.8551.

The structure was solved and refined using the Bruker SHELXTL (Version 6.1) Software Package, using the space group P-1, with Z = 2 for the formula unit, C12 H24 N O P. The hydrogen atoms were placed geometrically and rode on their parent atoms. The final anisotropic full-matrix least-squares refinement on F^2 with 160 variables converged at R1 = 5.07%, for the observed data and wR2 = 14.21% for all data. The goodness-of-fit was 1.045. The largest peak on the final difference map was 0.637 $e^-/Å^3$ and the largest hole was -0.518 $e^-/Å^3$. Based on the final model, the calculated density of the crystal is 1.140 g/cm^3 and F(000) amounts to 252 electrons.

Table S7. Sample and crystal data for **5.**

Identification code	**5**	
Empirical formula	C12 H24 N O P	
Formula weight	229.29	
Temperature	203(2) K	
Wavelength	0.71073 Å	
Crystal size	0.19 x 0.16 x 0.12 mm	
Crystal habit	colorless plate	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 6.0522(11) Å	α= 94.862(4)°
	b = 9.8966(18) Å	β= 93.274(4)°
	c = 11.401(2) Å	γ = 100.155(4)°
Volume	668.0(2) $Å^3$	
Z	2	
Density (calculated)	1.140 g/cm^3	
Absorption coefficient	0.184 mm^{-1}	
F(000)	252	

Table S8. Atomic coordinates and equivalent isotropic atomic displacement parameters ($Å^2$) for **5.** U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C1	0.2932(3)	0.75732(19)	0.38965(15)	0.0389(4)
C2	0.1202(3)	0.66678(17)	0.29416(15)	0.0347(3)
C3	0.2510(3)	0.69207(17)	0.18129(15)	0.0365(3)
C4	0.1822(4)	0.8408(3)	0.4804(2)	0.0635(6)
C5	0.4513(4)	0.6786(3)	0.4547(2)	0.0626(6)
C6	0.0357(4)	0.5182(2)	0.3165(2)	0.0598(6)
C7	0.3820(4)	0.5794(2)	0.1411(2)	0.0559(5)
C8	0.1039(4)	0.7245(3)	0.07825(19)	0.0603(6)
C10	0.1776(4)	1.1454(3)	0.1800(3)	0.0805(9)
C11	0.4205(4)	1.2048(3)	0.1723(3)	0.0768(8)
C12	0.5410(3)	1.0867(2)	0.1904(2)	0.0538(5)
C9A	0.1736(4)	1.0423(2)	0.2681(3)	0.0430(8)
N1A	0.3861(4)	0.9919(2)	0.2562(3)	0.0357(5)
C9B	0.1506(17)	1.0160(12)	0.1780(14)	0.042(4)
N1B	0.3724(19)	0.9751(12)	0.1984(17)	0.035(3)
O1	0.6838(2)	0.83922(14)	0.26461(15)	0.0531(4)
P1	0.43901(6)	0.83639(4)	0.26899(4)	0.03522(15)

Table S9. Bond lengths (Å) for **5.**

C1-C4	1.528(3)	C1-C5	1.534(3)
C1-C2	1.568(2)	C1-P1	1.8335(18)
C2-C6	1.517(3)	C2-C3	1.564(2)
C2-H2	0.9900	C3-C8	1.523(3)
C3-C7	1.533(2)	C3-P1	1.8411(18)
C4-H4A	0.9700	C4-H4B	0.9700
C4-H4C	0.9700	C5-H5A	0.9700
C5-H5B	0.9700	C5-H5C	0.9700
C6-H6A	0.9700	C6-H6B	0.9700
C6-H6C	0.9700	C7-H7A	0.9700
C7-H7B	0.9700	C7-H7C	0.9700
C8-H8A	0.9700	C8-H8B	0.9700
C8-H8C	0.9700	C10-C9B	1.260(12)
C10-C9A	1.489(3)	C10-C11	1.494(4)
C10-H10A	0.9800	C10-H10B	0.9800
C11-C12	1.505(3)	C11-H11A	0.9800
C11-H11B	0.9800	C12-N1B	1.378(12)
C12-N1A	1.487(3)	C12-H12A	0.9800
C12-H12B	0.9800	C9A-N1A	1.469(3)
C9A-H9A	0.9800	C9A-H9B	0.9800
N1A-P1	1.643(2)	C9B-N1B	1.480(14)
C9B-H9B1	0.9800	C9B-H9B2	0.9800
N1B-P1	1.744(12)	O1-P1	1.4803(13)

Table S10. Bond angles (°) for **5.**

C4-C1-C5	108.97(18)	C4-C1-C2	113.18(16)
C5-C1-C2	114.98(17)	C4-C1-P1	121.98(15)
C5-C1-P1	108.62(14)	C2-C1-P1	88.07(10)
C6-C2-C3	117.26(16)	C6-C2-C1	117.30(16)
C3-C2-C1	99.79(12)	C6-C2-H2	107.2
C3-C2-H2	107.2	C1-C2-H2	107.2
C8-C3-C7	110.42(17)	C8-C3-C2	112.98(15)
C7-C3-C2	115.32(15)	C8-C3-P1	118.24(14)
C7-C3-P1	110.53(13)	C2-C3-P1	87.91(10)
C1-C4-H4A	109.5	C1-C4-H4B	109.5
H4A-C4-H4B	109.5	C1-C4-H4C	109.5
H4A-C4-H4C	109.5	H4B-C4-H4C	109.5
C1-C5-H5A	109.5	C1-C5-H5B	109.5
H5A-C5-H5B	109.5	C1-C5-H5C	109.5
H5A-C5-H5C	109.5	H5B-C5-H5C	109.5
C2-C6-H6A	109.5	C2-C6-H6B	109.5
H6A-C6-H6B	109.5	C2-C6-H6C	109.5
H6A-C6-H6C	109.5	H6B-C6-H6C	109.5
C3-C7-H7A	109.5	C3-C7-H7B	109.5
H7A-C7-H7B	109.5	C3-C7-H7C	109.5
H7A-C7-H7C	109.5	H7B-C7-H7C	109.5
C3-C8-H8A	109.5	C3-C8-H8B	109.5
H8A-C8-H8B	109.5	C3-C8-H8C	109.5
H8A-C8-H8C	109.5	H8B-C8-H8C	109.5
C9B-C10-C9A	43.2(7)	C9B-C10-C11	110.2(5)
C9A-C10-C11	105.7(2)	C9B-C10-H10A	136.6
C9A-C10-H10A	110.6	C11-C10-H10A	110.6
C9B-C10-H10B	69.1	C9A-C10-H10B	110.6
C11-C10-H10B	110.6	H10A-C10-H10B	108.7
C10-C11-C12	104.44(19)	C10-C11-H11A	110.9
C12-C11-H11A	110.9	C10-C11-H11B	110.9
C12-C11-H11B	110.9	H11A-C11-H11B	108.9
N1B-C12-N1A	26.4(7)	N1B-C12-C11	104.9(5)
N1A-C12-C11	104.65(18)	N1B-C12-H12A	86.7
N1A-C12-H12A	110.8	C11-C12-H12A	110.8
N1B-C12-H12B	131.7	N1A-C12-H12B	110.8
C11-C12-H12B	110.8	H12A-C12-H12B	108.9
N1A-C9A-C10	103.96(19)	N1A-C9A-H9A	111.0
C10-C9A-H9A	111.0	N1A-C9A-H9B	111.0
C10-C9A-H9B	111.0	H9A-C9A-H9B	109.0
C9A-N1A-C12	110.08(19)	C9A-N1A-P1	128.98(17)
C12-N1A-P1	118.22(17)	C10-C9B-N1B	109.2(9)
C10-C9B-H9B1	109.8	N1B-C9B-H9B1	109.8
C10-C9B-H9B2	109.8	N1B-C9B-H9B2	109.8
H9B1-C9B-H9B2	108.3	C12-N1B-C9B	109.5(9)
C12-N1B-P1	118.3(8)	C9B-N1B-P1	129.0(9)
O1-P1-N1A	109.35(10)	O1-P1-N1B	108.1(4)
N1A-P1-N1B	22.3(5)	O1-P1-C1	118.97(8)
N1A-P1-C1	111.38(11)	N1B-P1-C1	126.2(5)
O1-P1-C3	116.58(8)	N1A-P1-C3	117.05(12)
N1B-P1-C3	100.4(5)	C1-P1-C3	81.37(8)

Table S11. Torsion angles (°) for **5.**

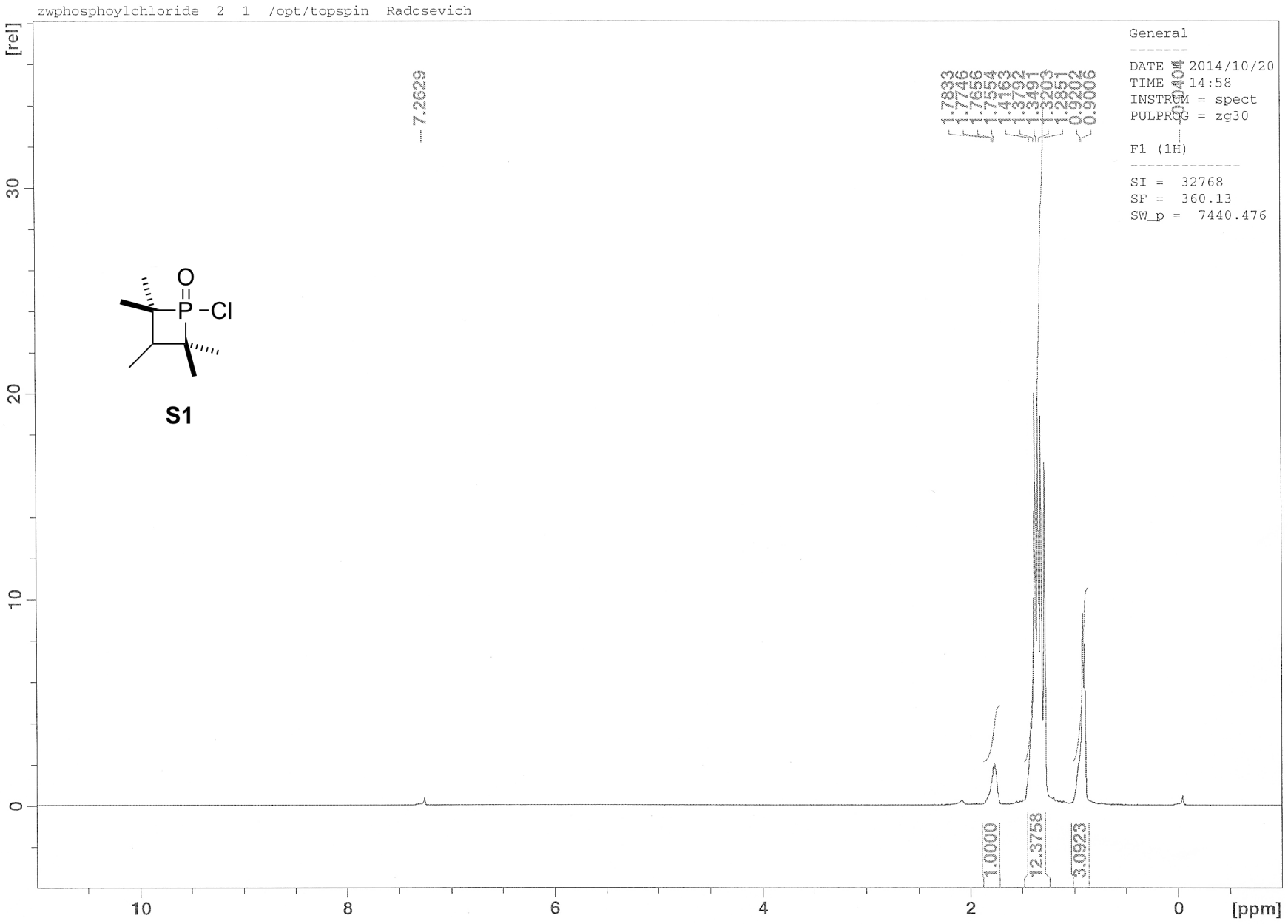
C4-C1-C2-C6	-94.2(2)	C5-C1-C2-C6	31.9(2)
P1-C1-C2-C6	141.63(16)	C4-C1-C2-C3	138.07(17)
C5-C1-C2-C3	-95.80(18)	P1-C1-C2-C3	13.88(11)
C6-C2-C3-C8	98.5(2)	C1-C2-C3-C8	-133.74(17)
C6-C2-C3-C7	-29.8(2)	C1-C2-C3-C7	97.96(18)
C6-C2-C3-P1	-141.59(16)	C1-C2-C3-P1	-13.82(11)
C9B-C10-C11-C12	-11.4(9)	C9A-C10-C11-C12	33.9(3)
C10-C11-C12-N1B	3.7(9)	C10-C11-C12-N1A	-23.6(3)
C9B-C10-C9A-N1A	72.6(7)	C11-C10-C9A-N1A	-30.4(3)
C10-C9A-N1A-C12	15.6(3)	C10-C9A-N1A-P1	-145.1(3)
N1B-C12-N1A-C9A	-89.0(12)	C11-C12-N1A-C9A	5.1(3)
N1B-C12-N1A-P1	74.0(11)	C11-C12-N1A-P1	168.1(2)
C9A-C10-C9B-N1B	-77.7(10)	C11-C10-C9B-N1B	13.9(13)
N1A-C12-N1B-C9B	96.8(16)	C11-C12-N1B-C9B	3.8(13)
N1A-C12-N1B-P1	-65.0(12)	C11-C12-N1B-P1	-158.0(9)
C10-C9B-N1B-C12	-11.6(16)	C10-C9B-N1B-P1	147.7(14)
C9A-N1A-P1-O1	-172.9(2)	C12-N1A-P1-O1	27.8(3)
C9A-N1A-P1-N1B	96.5(12)	C12-N1A-P1-N1B	-62.8(11)
C9A-N1A-P1-C1	-39.4(3)	C12-N1A-P1-C1	161.3(2)
C9A-N1A-P1-C3	51.7(3)	C12-N1A-P1-C3	-107.6(2)
C12-N1B-P1-O1	-23.2(14)	C9B-N1B-P1-O1	179.1(13)
C12-N1B-P1-N1A	73.9(14)	C9B-N1B-P1-N1A	-83.9(18)
C12-N1B-P1-C1	127.4(9)	C9B-N1B-P1-C1	-30.4(18)
C12-N1B-P1-C3	-145.8(11)	C9B-N1B-P1-C3	56.5(15)
C4-C1-P1-O1	116.30(17)	C5-C1-P1-O1	-11.63(18)
C2-C1-P1-O1	-127.40(11)	C4-C1-P1-N1A	-12.3(2)
C5-C1-P1-N1A	-140.20(18)	C2-C1-P1-N1A	104.04(15)
C4-C1-P1-N1B	-31.4(7)	C5-C1-P1-N1B	-159.3(7)
C2-C1-P1-N1B	84.9(7)	C4-C1-P1-C3	-128.02(17)
C5-C1-P1-C3	104.04(15)	C2-C1-P1-C3	-11.72(10)
C8-C3-P1-O1	-115.02(15)	C7-C3-P1-O1	13.59(16)
C2-C3-P1-O1	129.90(10)	C8-C3-P1-N1A	17.16(19)
C7-C3-P1-N1A	145.77(16)	C2-C3-P1-N1A	-97.92(14)
C8-C3-P1-N1B	1.4(5)	C7-C3-P1-N1B	130.0(5)
C2-C3-P1-N1B	-113.7(5)	C8-C3-P1-C1	126.82(16)
C7-C3-P1-C1	-104.57(14)	C2-C3-P1-C1	11.75(10)

Table S12. Anisotropic atomic displacement parameters ($Å^2$) for **5.** The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2a^{*2} U_{11} + ... + 2hka^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0349(8)	0.0426(9)	0.0391(8)	0.0034(7)	0.0008(6)	0.0075(7)
C2	0.0285(7)	0.0337(8)	0.0421(8)	0.0046(6)	0.0039(6)	0.0047(6)
C3	0.0358(8)	0.0373(8)	0.0378(8)	0.0027(6)	0.0049(6)	0.0105(6)
C4	0.0714(15)	0.0683(14)	0.0486(11)	-0.0097(10)	0.0135(10)	0.0113(12)
C5	0.0531(12)	0.0795(16)	0.0574(12)	0.0268(11)	-0.0087(10)	0.0129(11)
C6	0.0593(13)	0.0430(11)	0.0740(14)	0.0145(10)	0.0098(11)	-0.0049(9)
C7	0.0598(12)	0.0479(11)	0.0629(12)	-0.0067(9)	0.0153(10)	0.0204(9)
C8	0.0619(13)	0.0783(16)	0.0428(10)	0.0082(10)	-0.0031(9)	0.0194(11)
C10	0.0541(14)	0.0693(16)	0.133(3)	0.0522(17)	0.0149(15)	0.0304(12)
C11	0.0622(15)	0.0544(13)	0.127(2)	0.0449(15)	0.0294(15)	0.0212(11)
C12	0.0434(10)	0.0478(11)	0.0772(14)	0.0267(10)	0.0207(9)	0.0120(8)
C9A	0.0304(10)	0.0342(10)	0.0671(19)	0.0068(10)	0.0086(10)	0.0111(8)
N1A	0.0265(9)	0.0323(10)	0.0509(14)	0.0100(10)	0.0091(10)	0.0078(7)
C9B	0.020(5)	0.043(6)	0.067(10)	0.019(6)	-0.005(4)	0.009(4)
N1B	0.024(4)	0.027(5)	0.056(8)	0.011(6)	0.002(5)	0.008(3)
O1	0.0243(6)	0.0475(8)	0.0918(11)	0.0173(7)	0.0090(6)	0.0115(5)
P1	0.0229(2)	0.0310(2)	0.0540(3)	0.00850(17)	0.00531(16)	0.00824(15)

Table S13. Site occupancy factors that deviate from unity for **5.**

Atom	sof	Atom	sof	Atom	sof
C9A	0.843(8)	H9A	0.843(8)	H9B	0.843(8)
C9B	0.157(8)	H9B1	0.157(8)	H9B2	0.157(8)
N1A	0.843(8)				
N1B	0.157(8)				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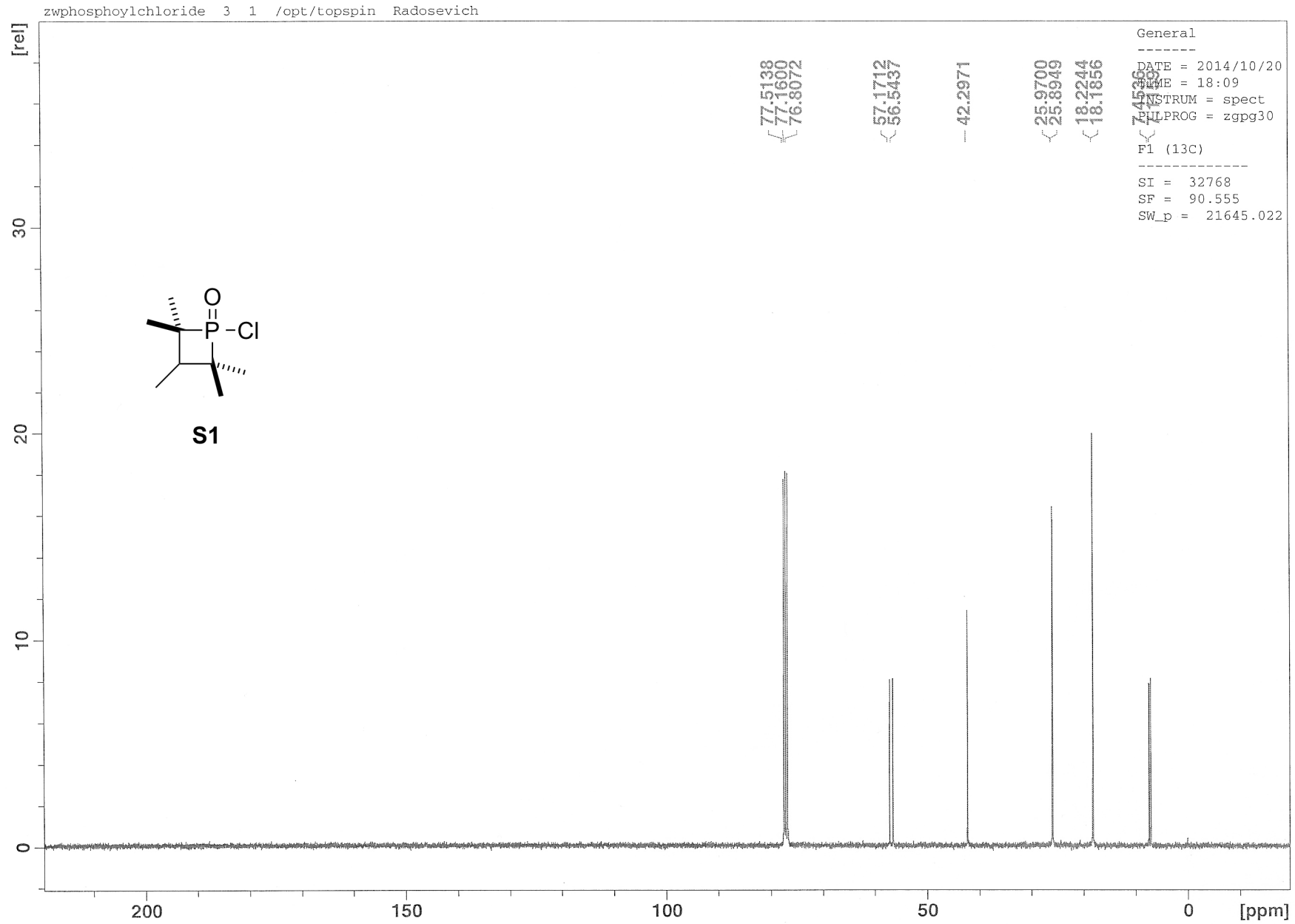
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25.8949
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18.1856
7.45366
7.44599
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P
Cl
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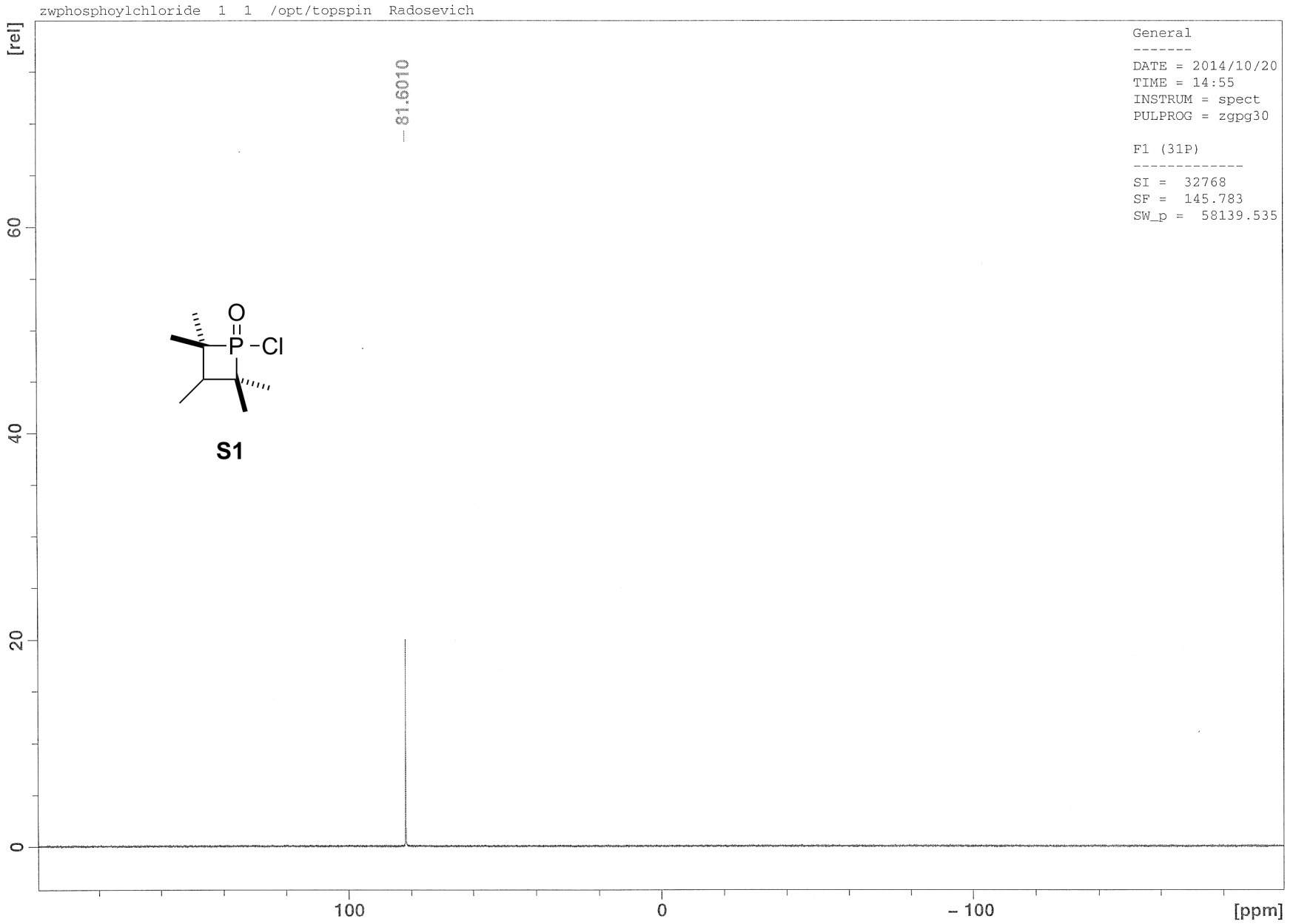
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Cl
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60
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– 100
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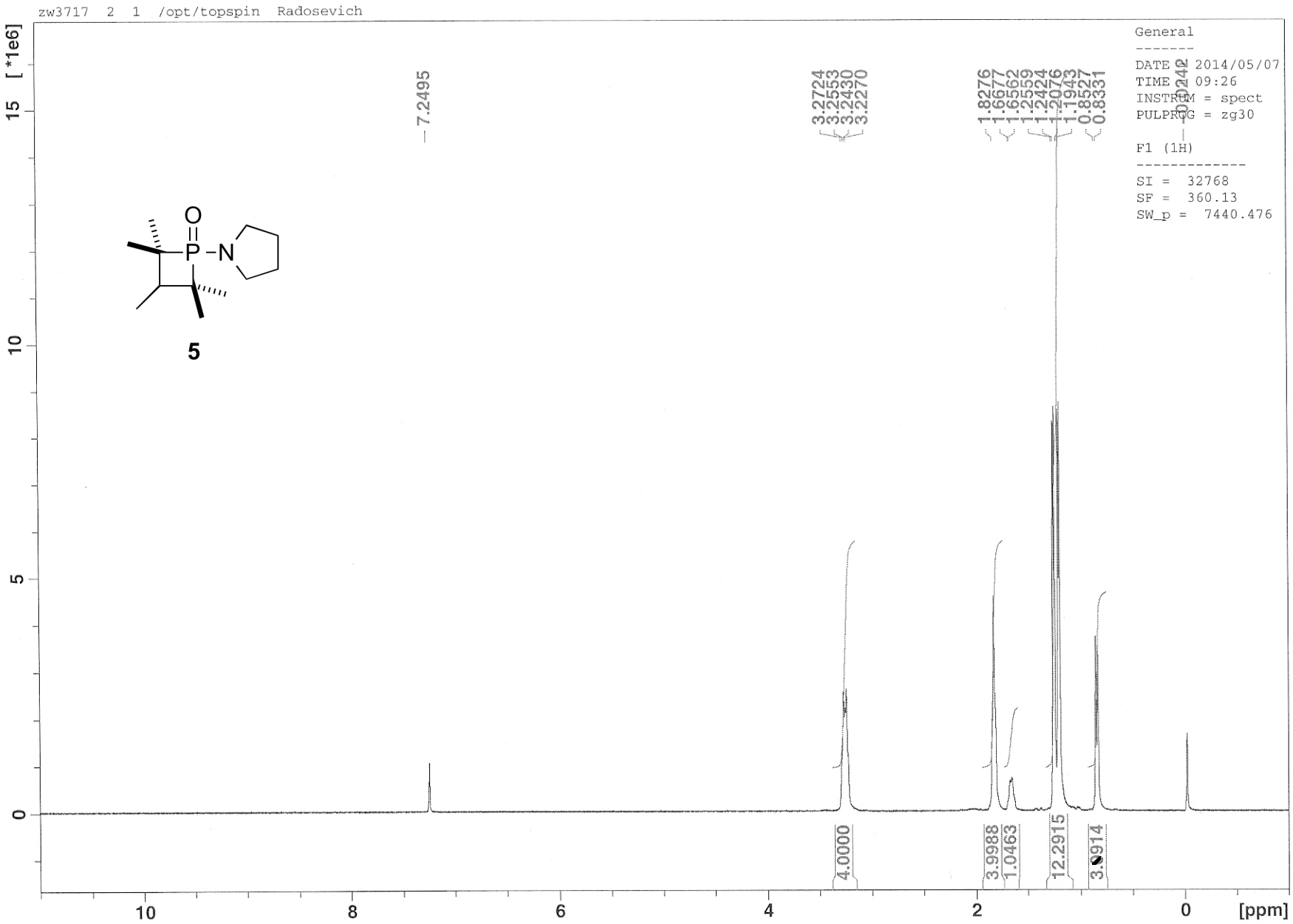
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3.2553
3.2430
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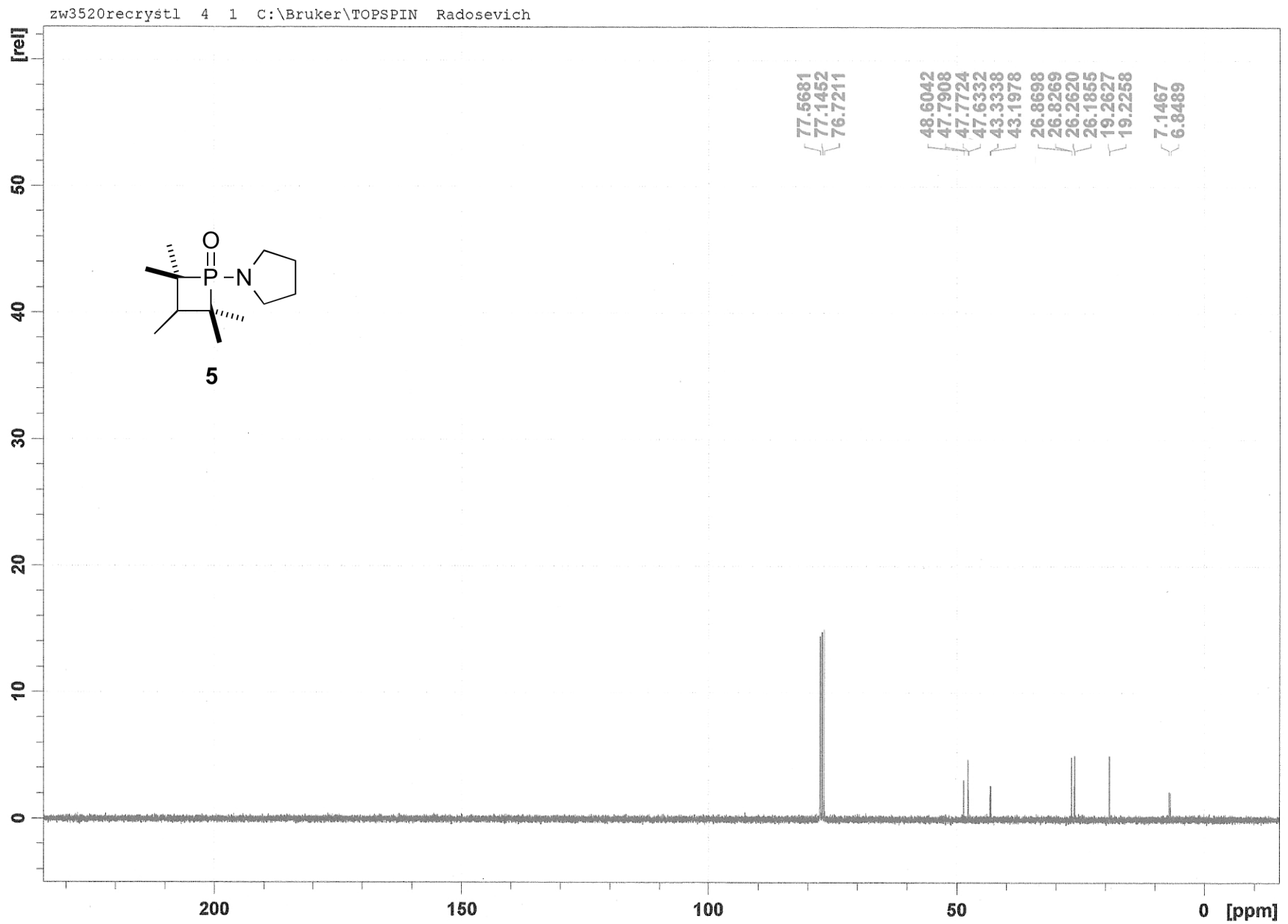
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N
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40
50
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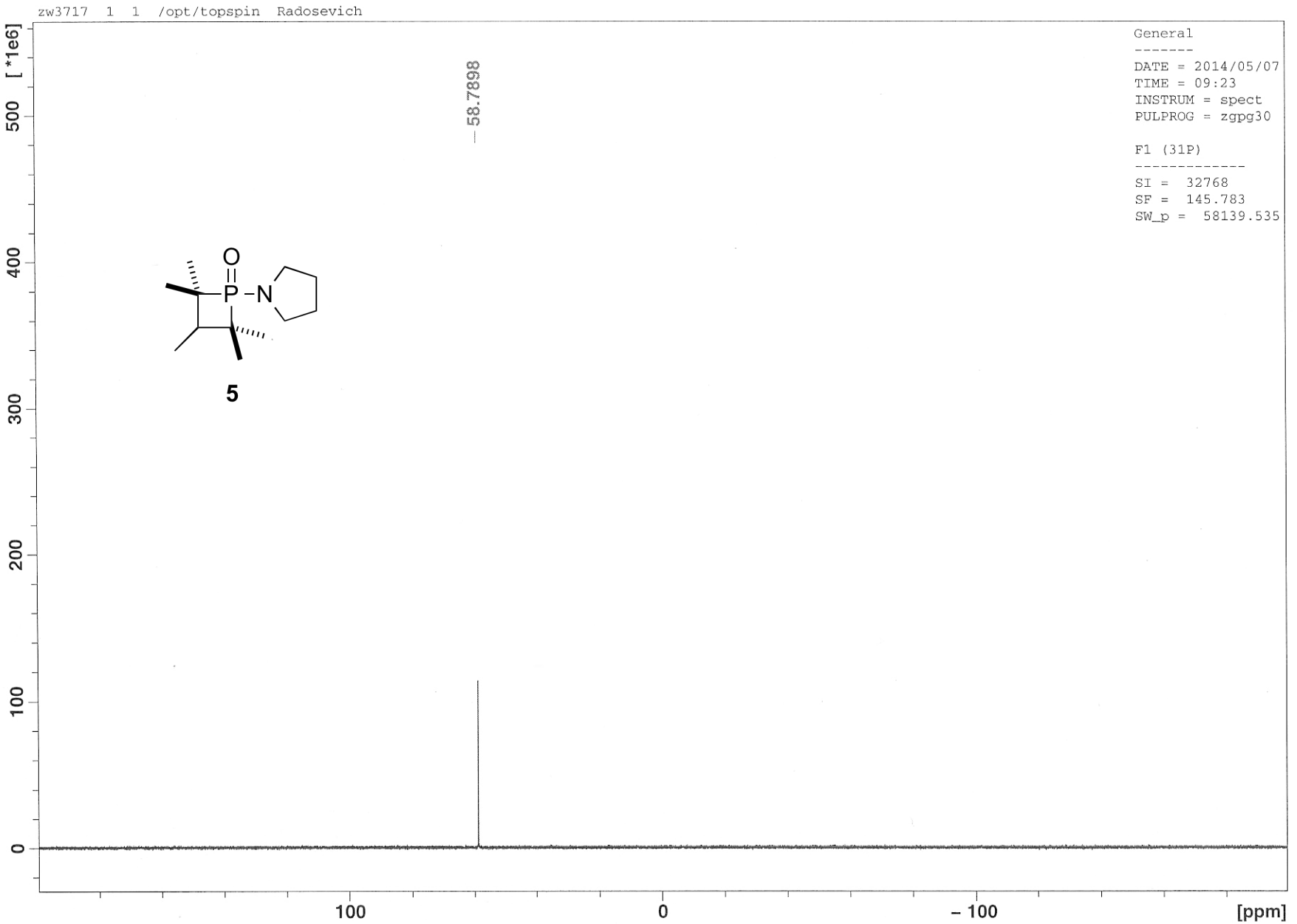
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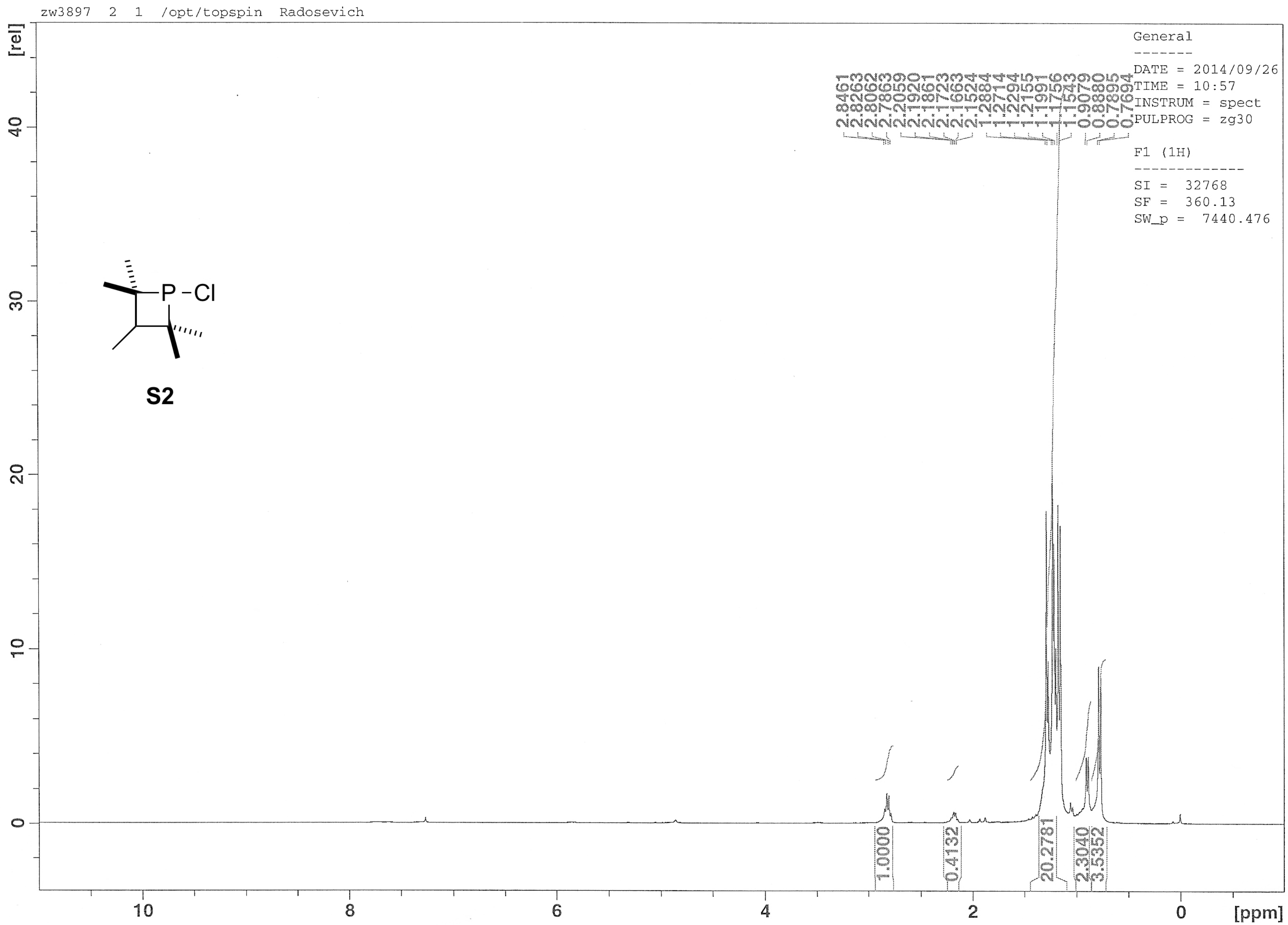
[ppm]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2.1524
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1.2294
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1.1991
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1.1543
0.9079
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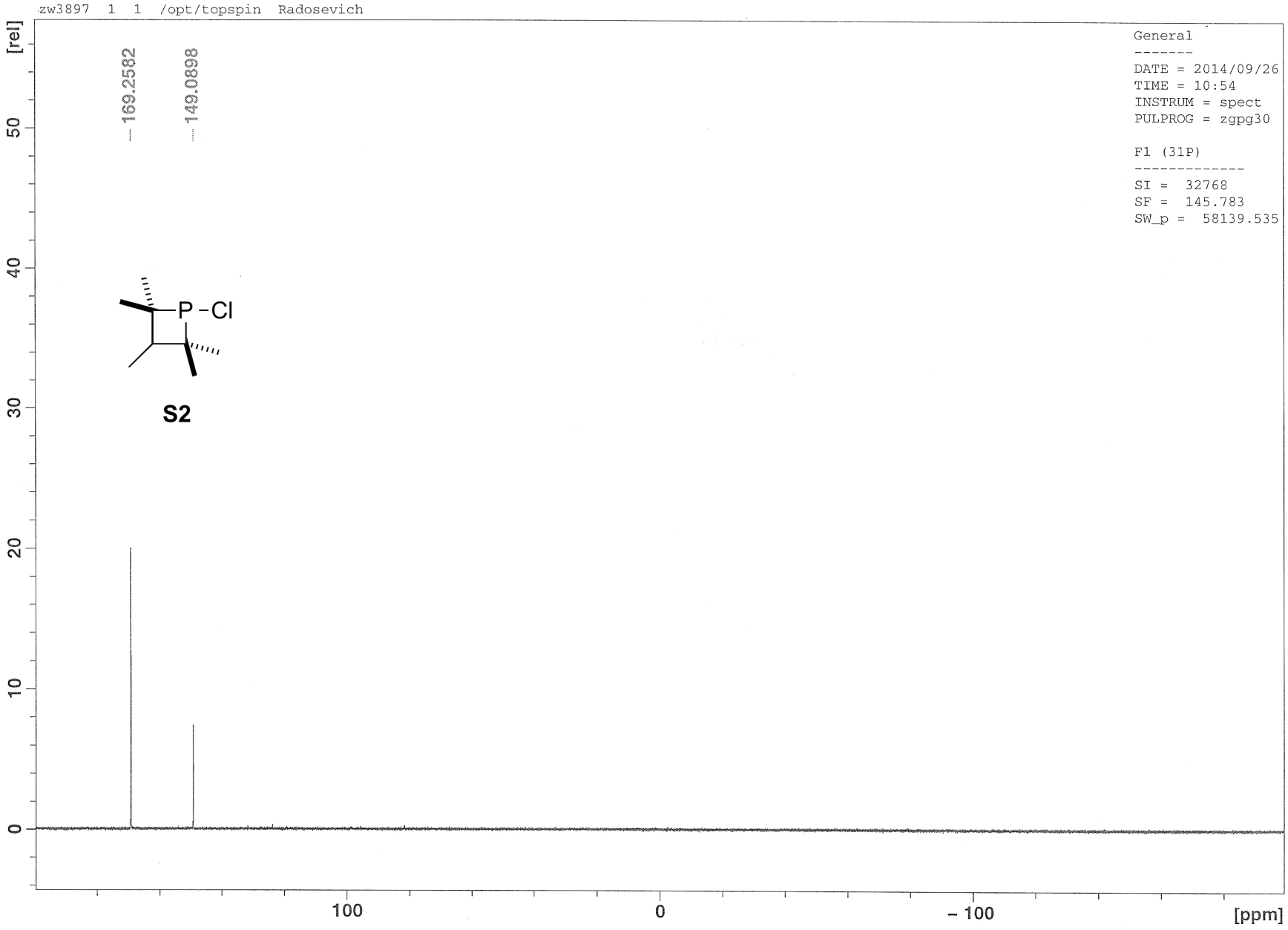
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SW_p = 58139.535
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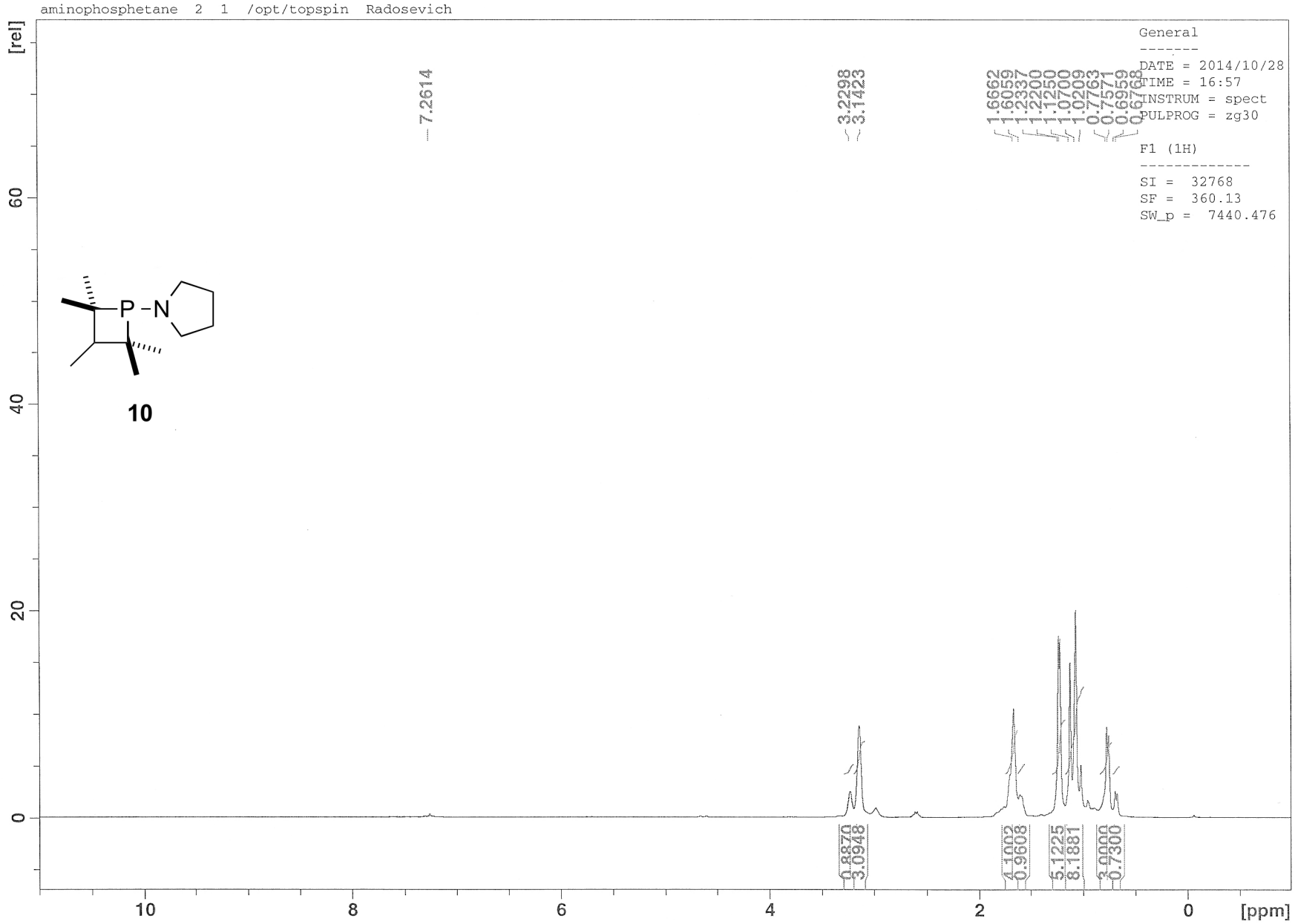
30
40
50
100
0
- 100
[ppm]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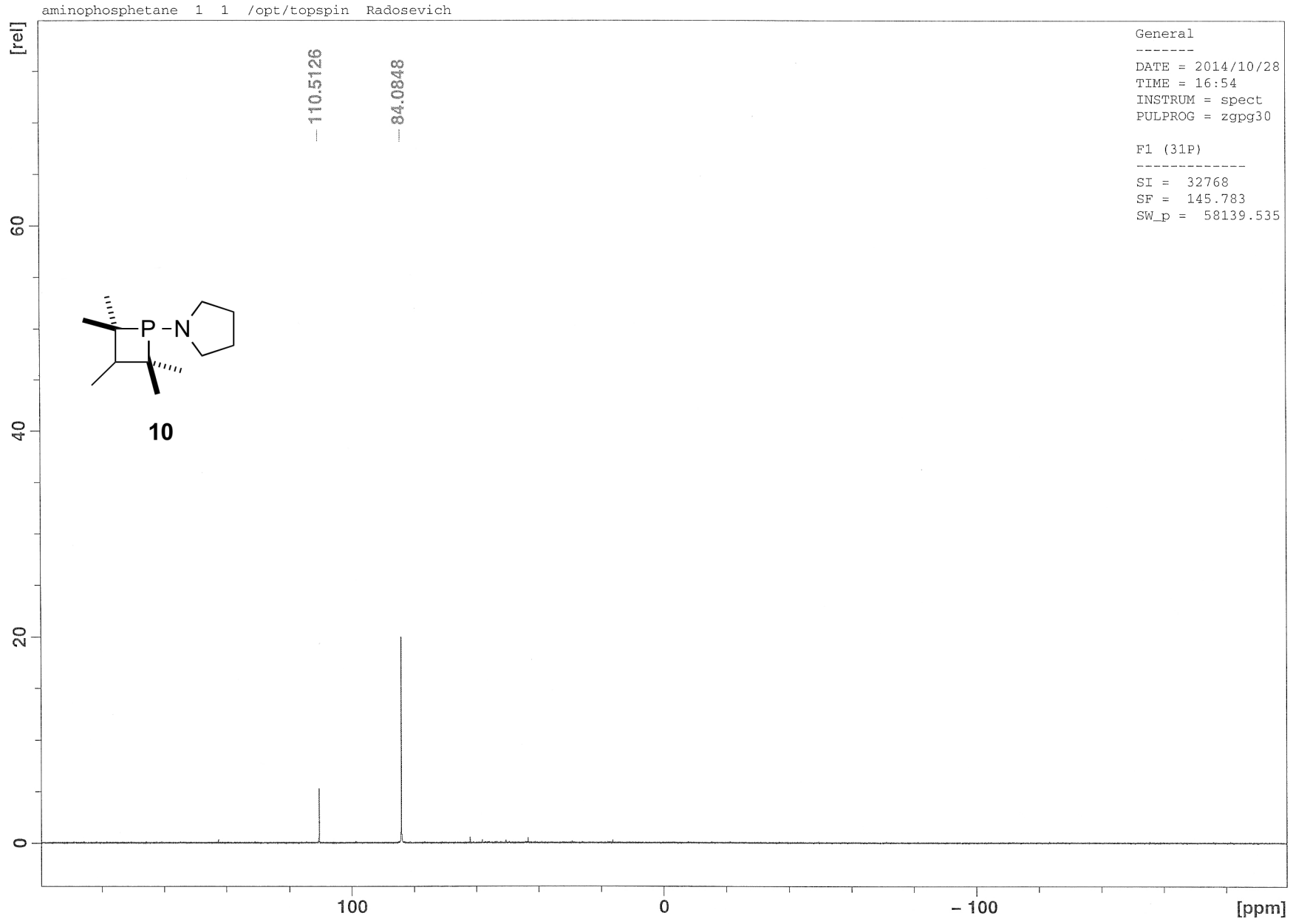
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20
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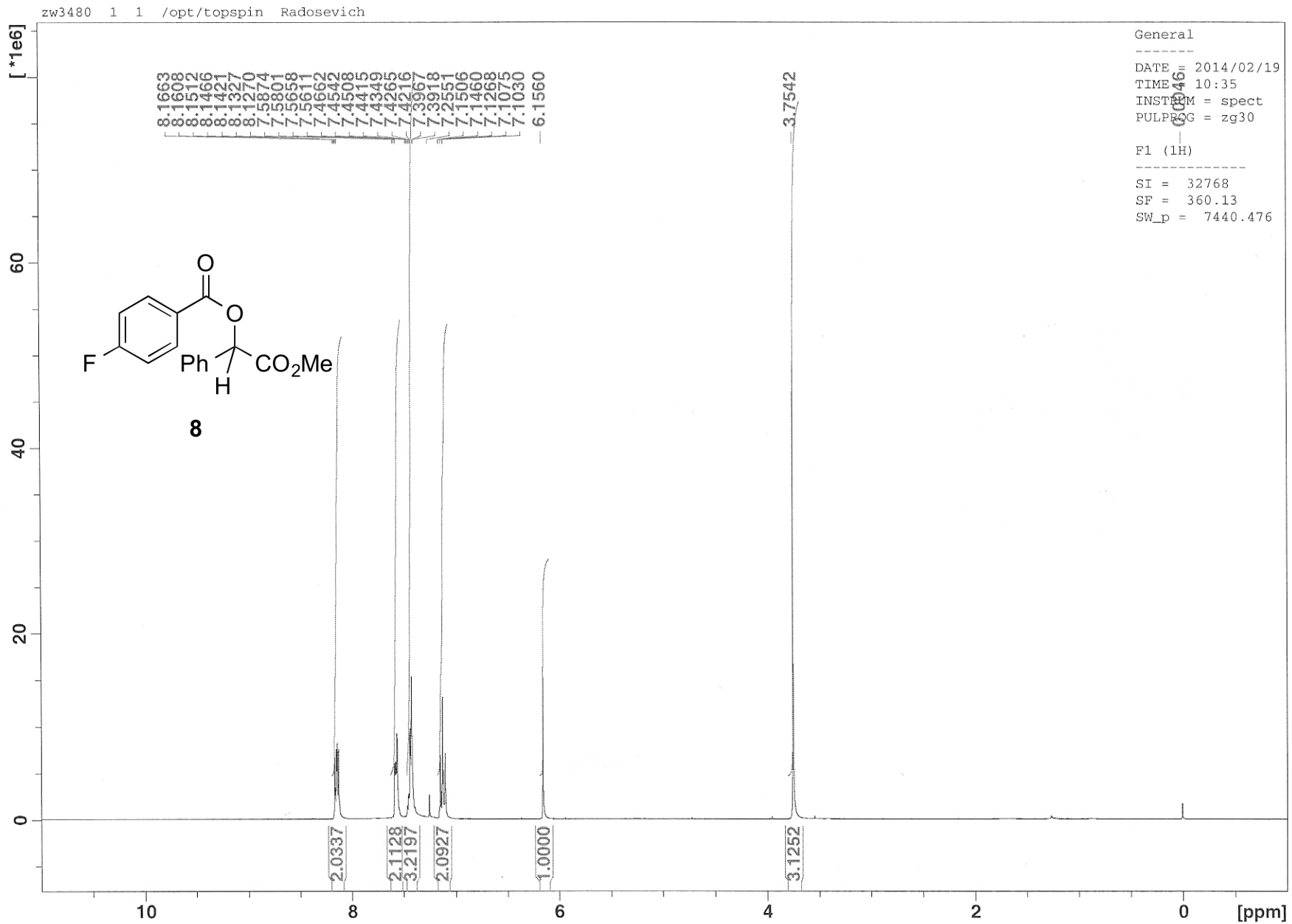
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7.5611
7.5542
7.4662
7.4508
7.4415
7.4349
7.4265
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7.1460
7.1268
7.1075
7.1030
6.1560
3.7542
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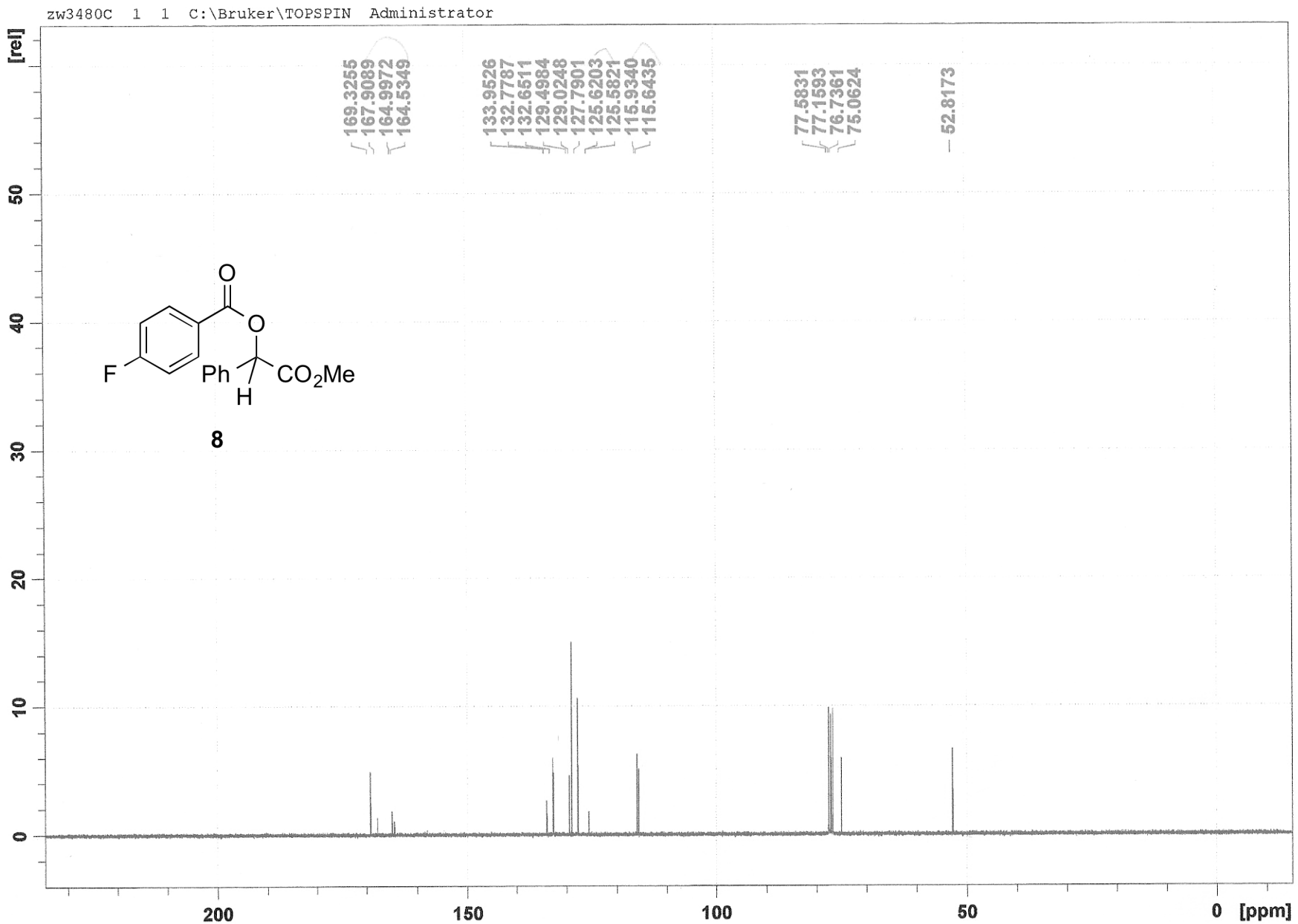
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0
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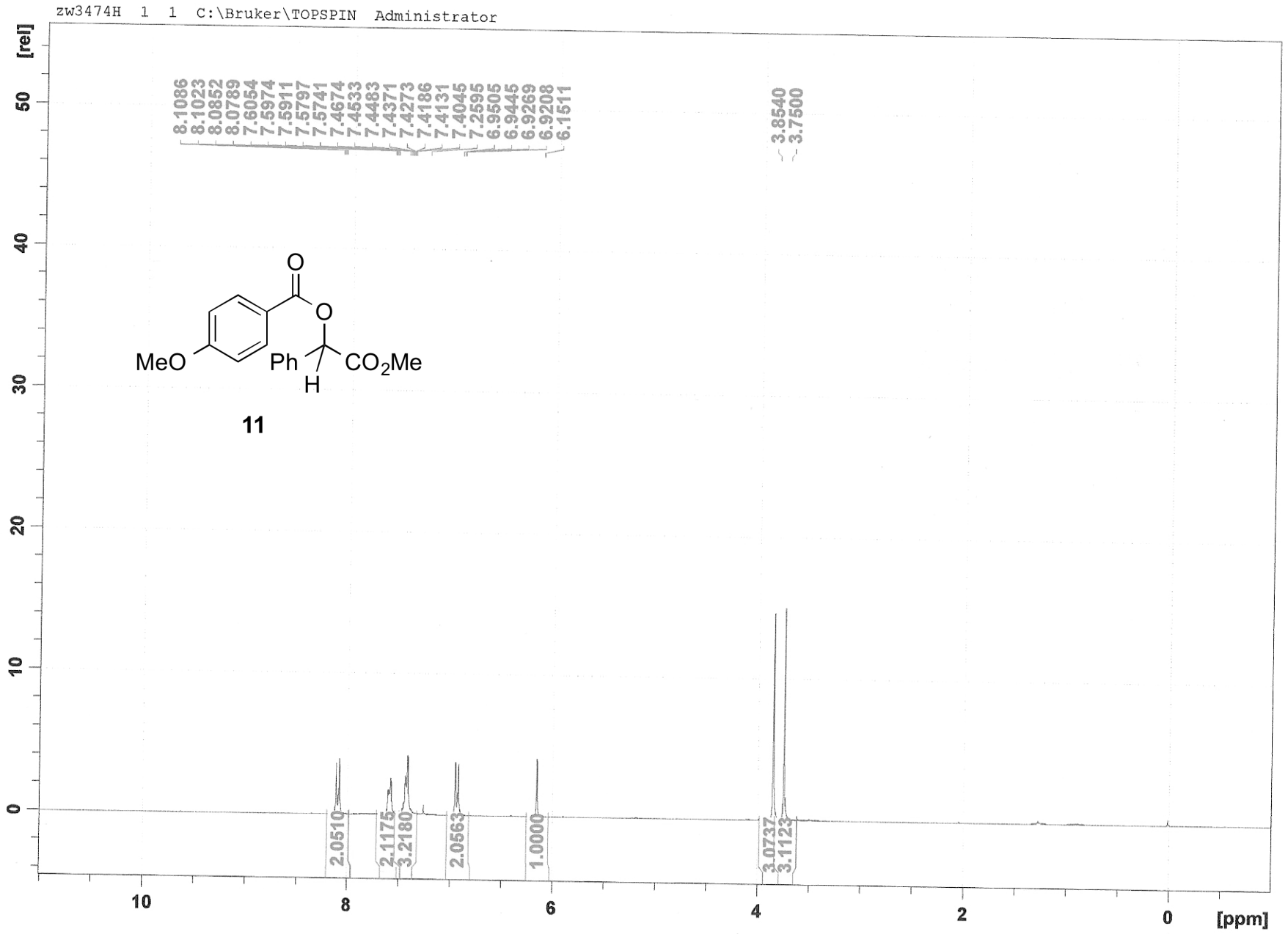
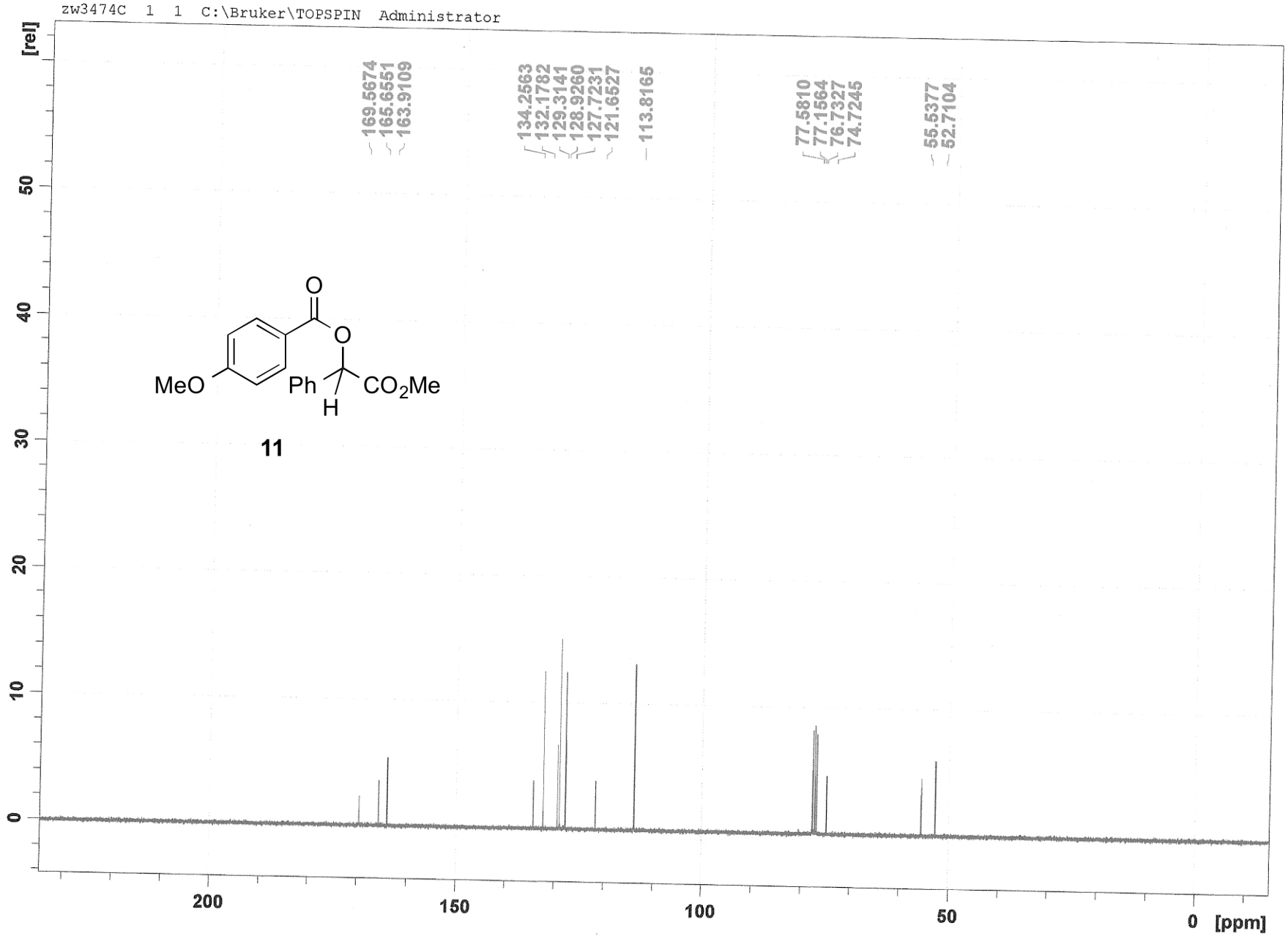
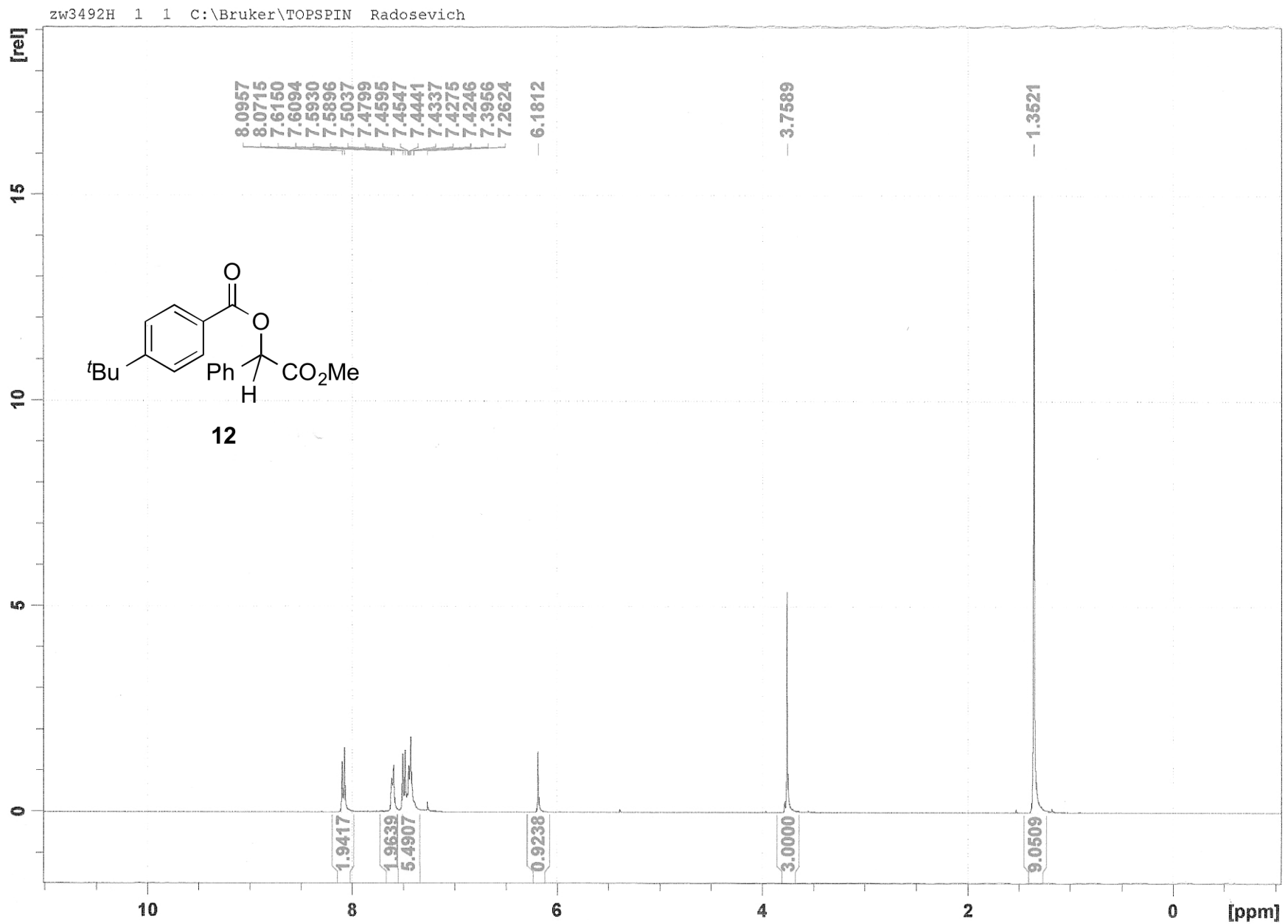
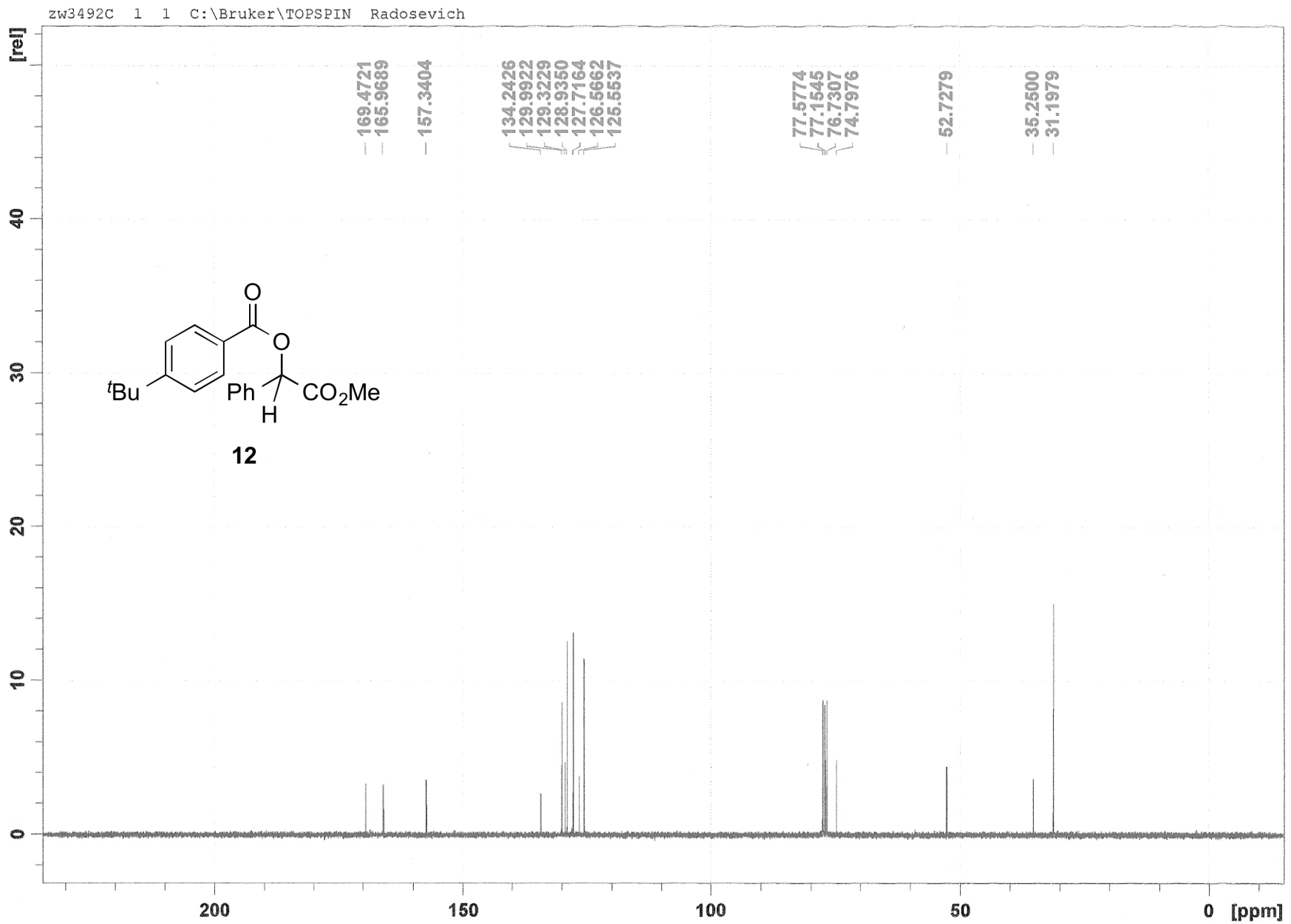
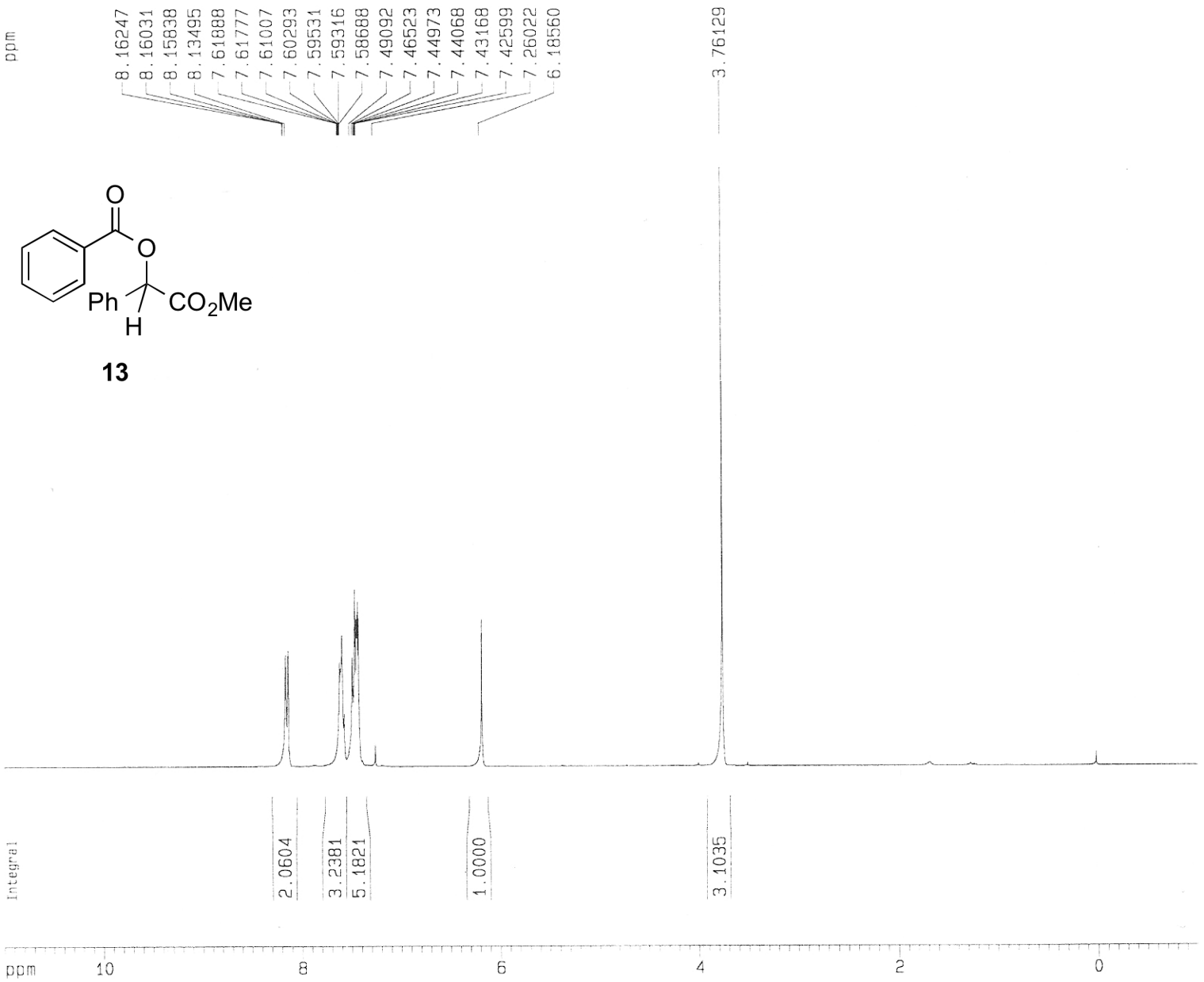

zw3474H 1 1 C:\Bruker\TOPSPIN Administrator
[rel]
8.1086
8.1023
8.0852
8.0789
7.6054
7.5974
7.5911
7.5797
7.5741
7.4674
7.4533
7.4483
7.4371
7.4273
7.4186
7.4131
7.4045
7.2595
6.9505
6.9445
6.9269
6.9208
6.1511
3.8540
3.7500
O
O
MeO
Ph
CO2Me
H
11
2.0510
2.1175
3.2180
2.0563
1.0000
3.0737
3.1123
[ppm]


zw3474C 1 1 C:\Bruker\TOPSPIN Administrator
[rel]
169.5674
165.6551
163.9109
134.2563
132.1782
129.3141
128.9260
127.7231
121.6527
113.8165
77.5810
77.1564
76.7327
74.7245
55.5377
52.7104
MeO
Ph
H
CO2Me
11
0
10
20
30
40
50
200
150
100
50
0
[ppm]

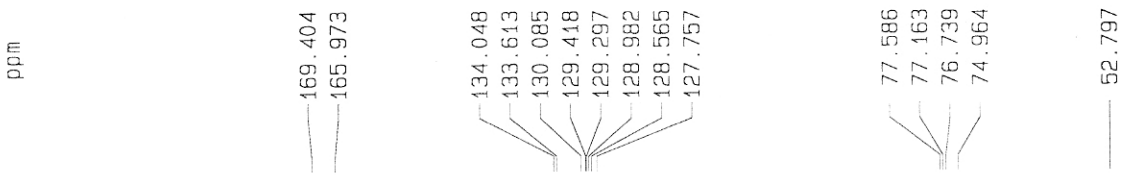

zw3492H 1 1 C:\Bruker\TOPSPIN Radosevich
[rel]
8.0957
8.0715
7.6150
7.6094
7.5930
7.5896
7.5037
7.4799
7.4595
7.4547
7.4441
7.4337
7.4275
7.4246
7.3956
7.2624
6.1812
3.7589
1.3521
1.9417
1.9639
5.4907
0.9238
3.0000
9.0509
[ppm]

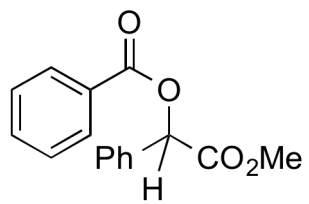
12


zw3492C 1 1 C:\Bruker\TOPSPIN Radosevich
[rel]
169.4721
165.9689
157.3404
134.2426
129.9922
129.3229
128.9350
127.7164
126.5662
125.5537
77.5774
77.1545
76.7307
74.7976
52.7279
35.2500
31.1979
tBu
Ph
CO2Me
H
O
O
12
0
10
20
30
40
200
150
100
50
0 [ppm]

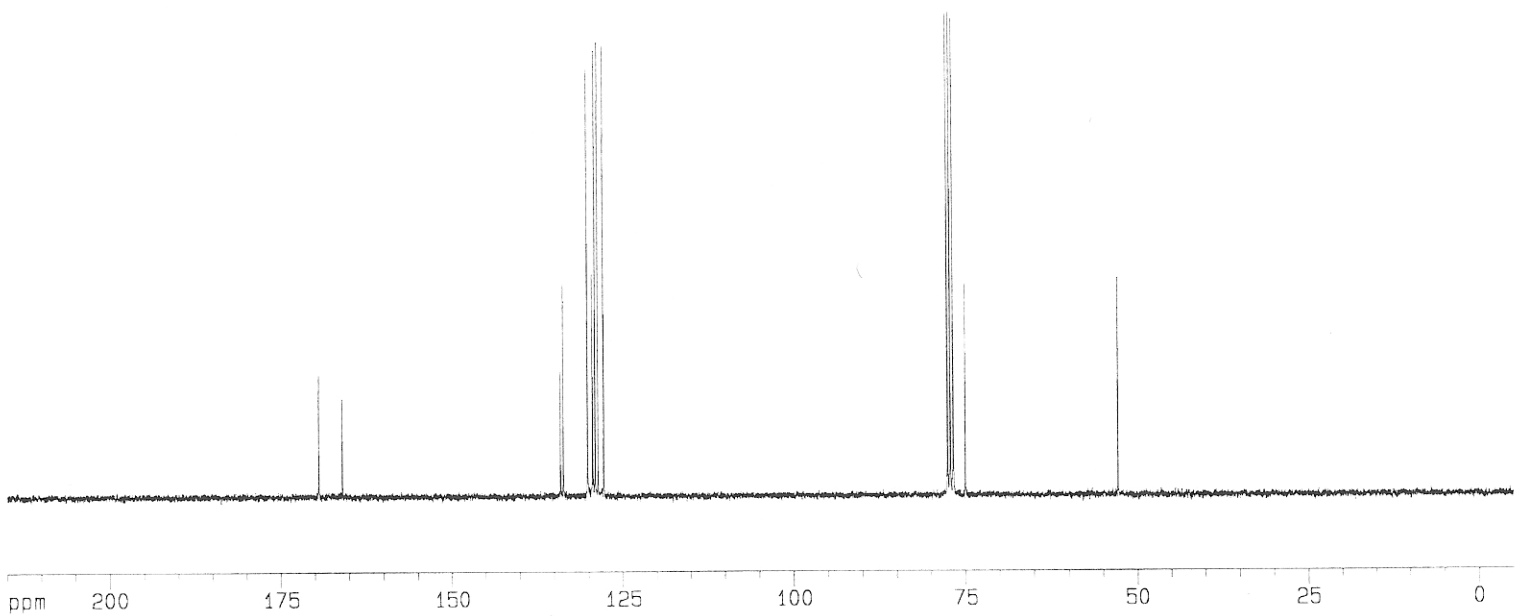



```
Current Data Parameters
NAME                  zw36808
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140421
Time                   16.38
INSTRUM                spect
PROBHD     5 mm Multinu
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    203.2
DW                 81.000 usec
DE                  6.00 usec
TE                  300.0 K
D1              1.00000000 sec

============ CHANNEL f1 =========
NUC1                      1H
P1                    10.10 usec
PL1                   -6.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF              300.1300057 MHz
WDW                          no
SSB                           0
LB                     0.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                    20.00 cm
F1P                  11.000 ppm
F1                  3301.43 Hz
F2P                  -1.000 ppm
F2                  -300.13 Hz
PPMCM                0.60000 ppm/cm
HZCM               180.07800 Hz/cm
```

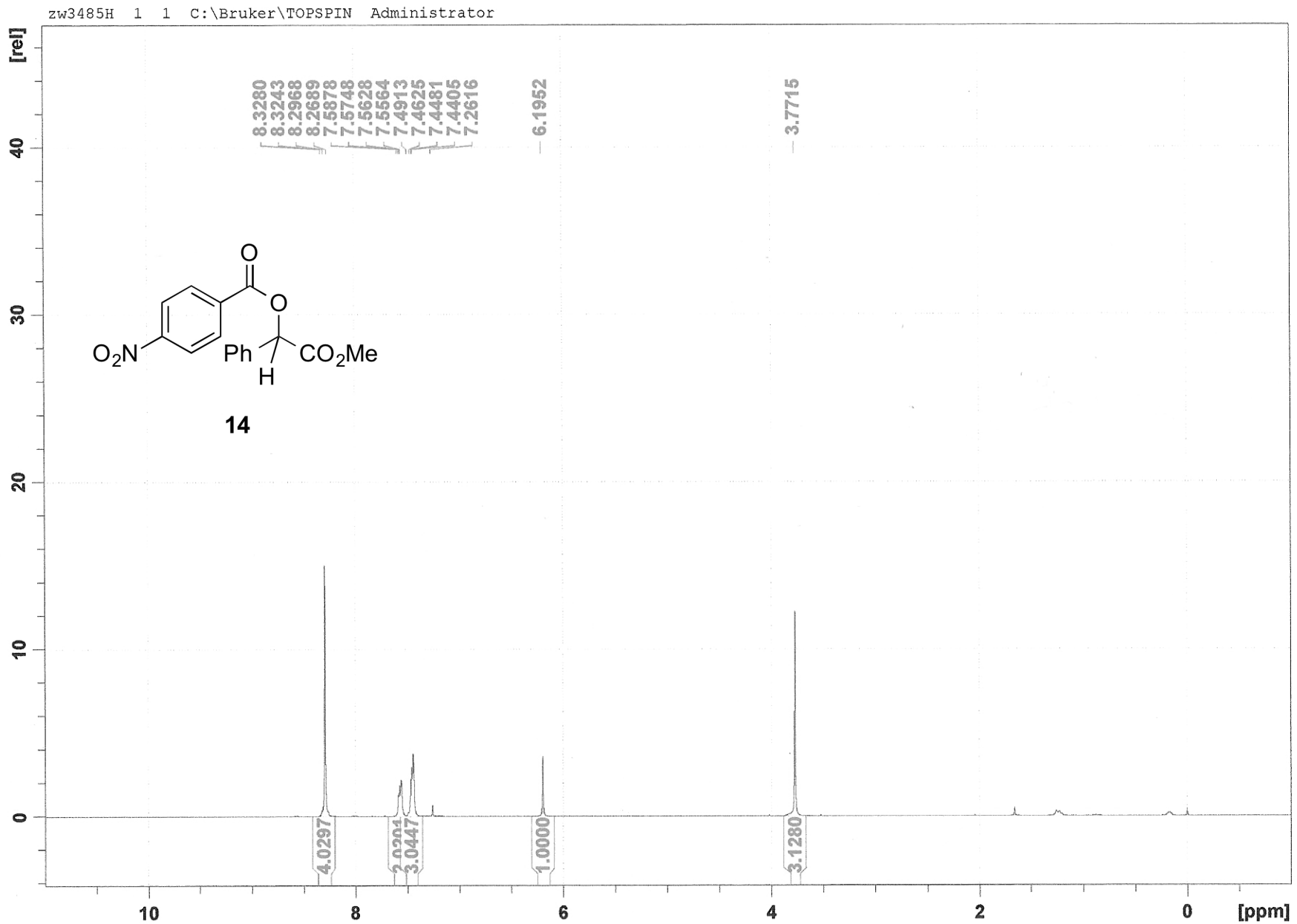


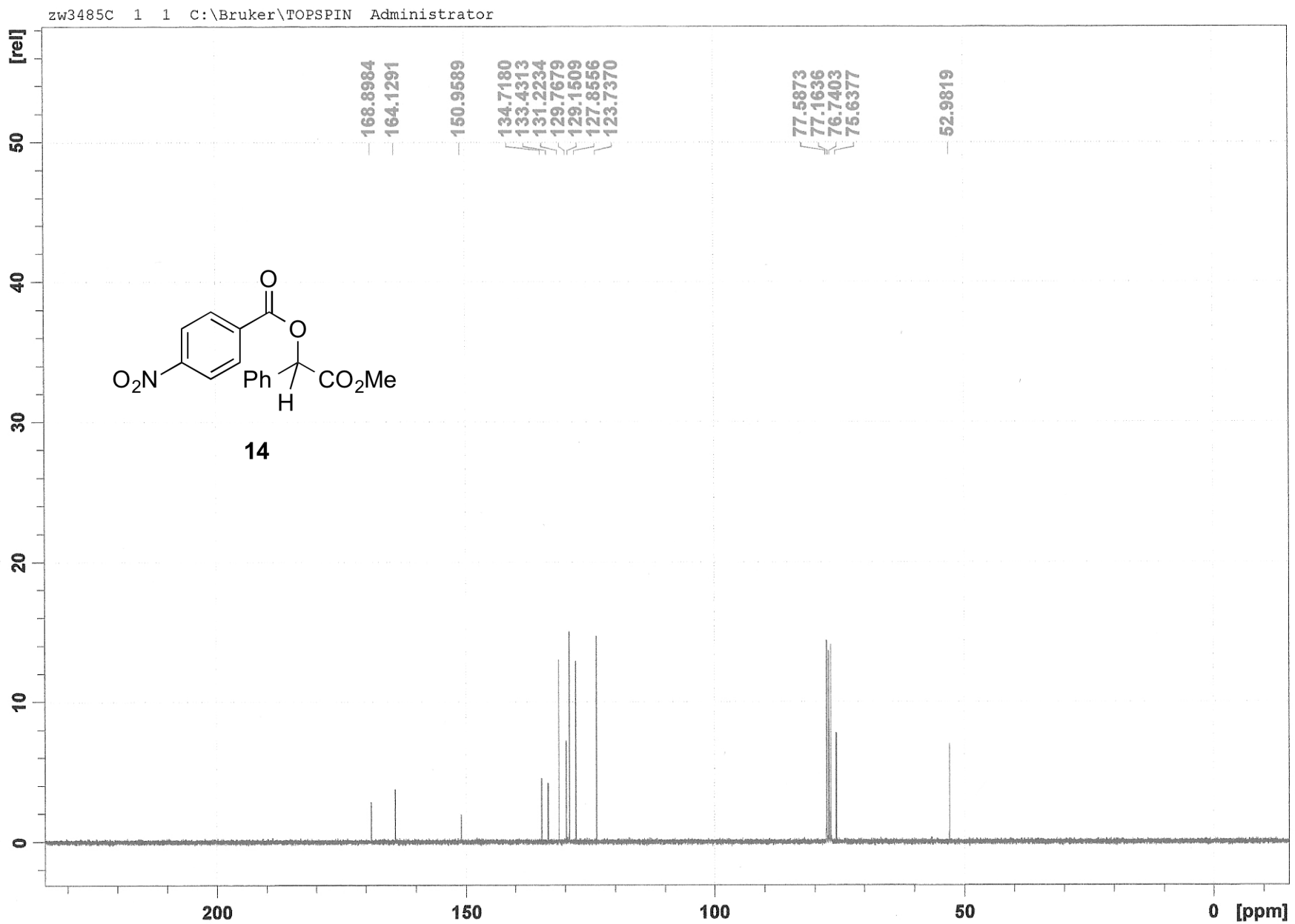


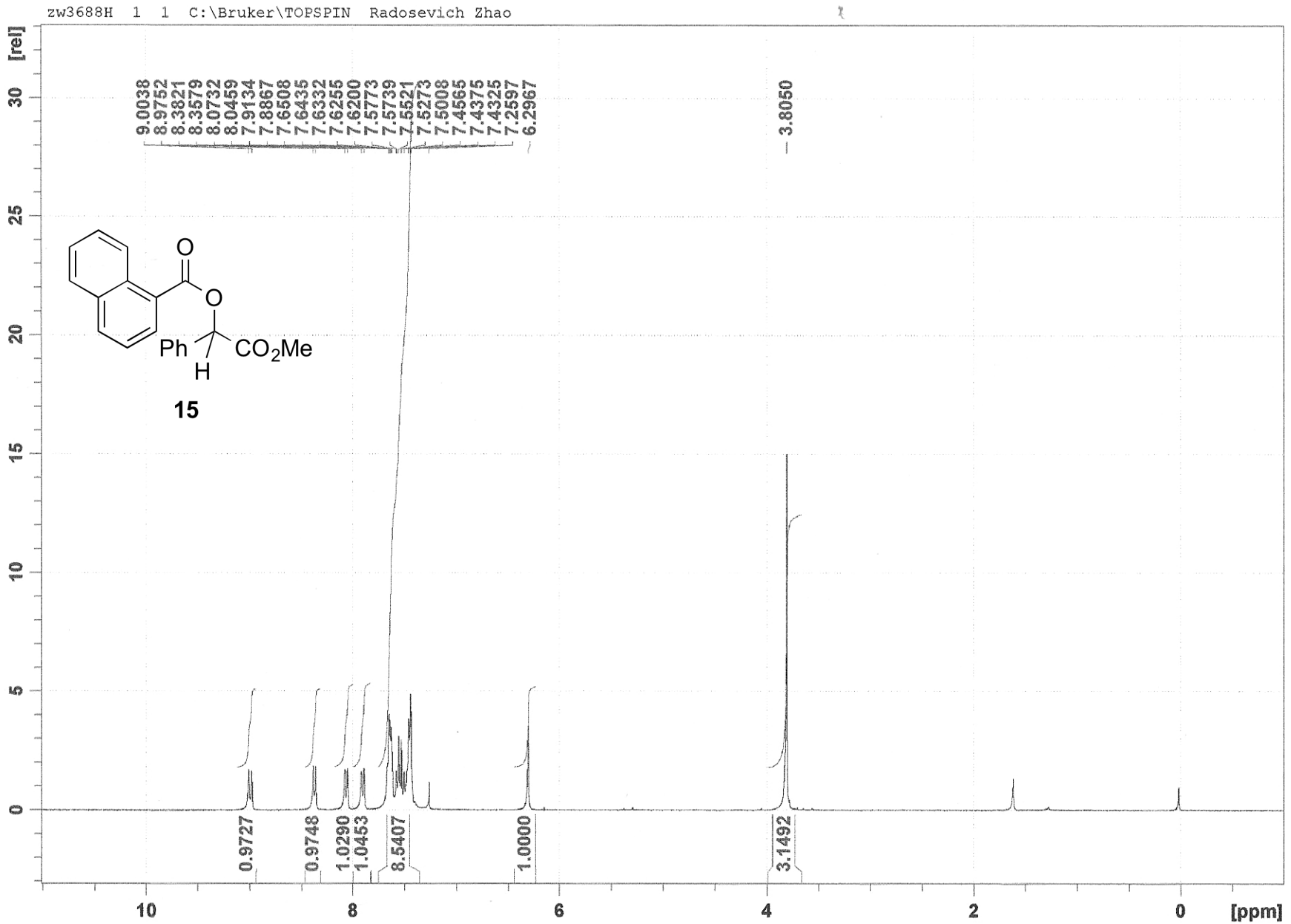
13

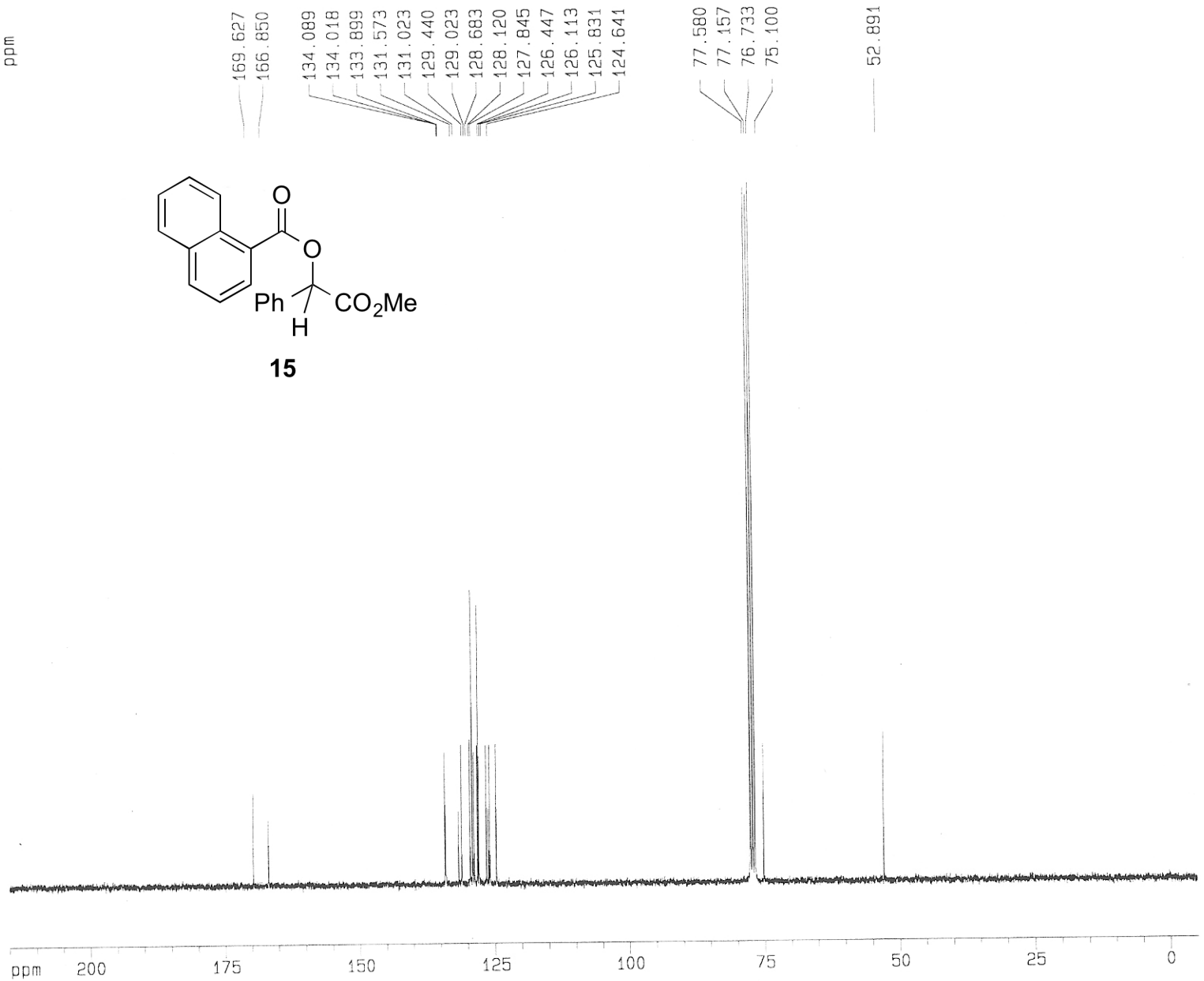



```
Current Data Parameters
NAME                  zw36808
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140421
Time                   17.46
INSTRUM                spect
PROBHD     5 mm Multinu
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1029
DS                        4
SWH               18832.393 Hz
FIDRES            0.287360 Hz
AG                1.7400308 sec
RG                     8192
DW                   26.550 usec
DE                    6.00 usec
TE                   300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 ============
NUC1                     13C
P1                      9.75 usec
PL1                     0.00 dB
SFO1             75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2              waltz16
NUC2                     1H
PCPD2                110.00 usec
PL2                     0.00 dB
PL12                   17.50 dB
PL13                   17.50 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                       32768
SF               75.4677434 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     215.000 ppm
F1                    16225.57 Hz
F2P                    -5.000 ppm
F2                    -377.34 Hz
PPMCM                 11.00000 ppm/cm
HZCM                830.14514 Hz/cm
```


zw3485H 1 1 C:\Bruker\TOPSPIN Administrator
[rel]
8.3280
8.3243
8.2968
8.2689
7.5878
7.5748
7.5628
7.5564
7.4913
7.4625
7.4481
7.4405
7.2616
6.1952
3.7715
O
O
O2N
Ph
CO2Me
H
14
4.0297
2.0201
3.0447
1.0000
3.1280
10
8
6
4
2
0
[ppm]

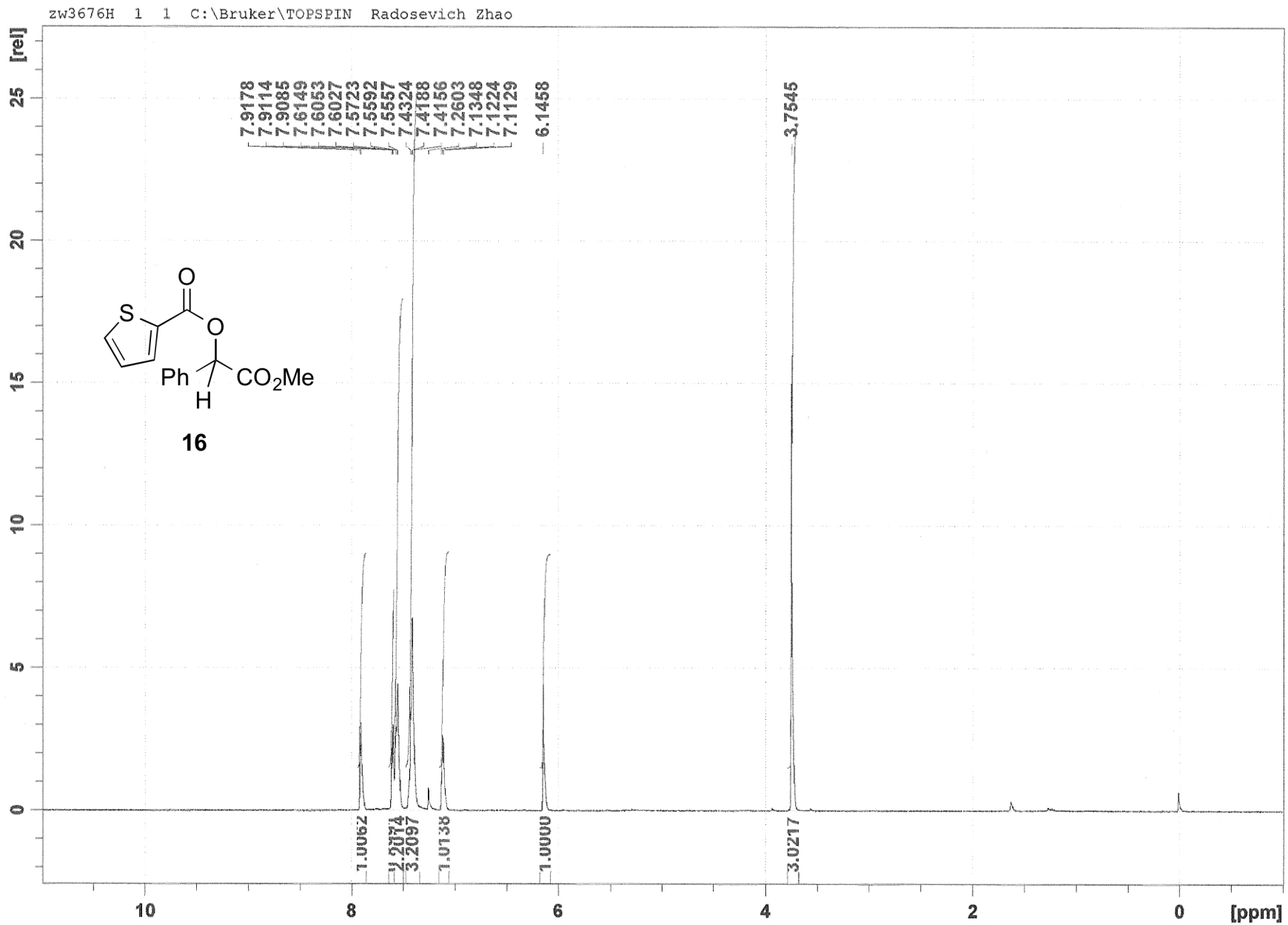

zw3485C 1 1 C:\Bruker\TOPSPIN Administrator
[rel]
168.8984
164.1291
150.9589
134.7180
133.4313
131.2234
129.7679
129.1509
127.8556
123.7370
77.5873
77.1636
76.7403
75.6377
52.9819
O
O
O2N
Ph
H
CO2Me
14
0
10
20
30
40
50
200
150
100
50
0 [ppm]

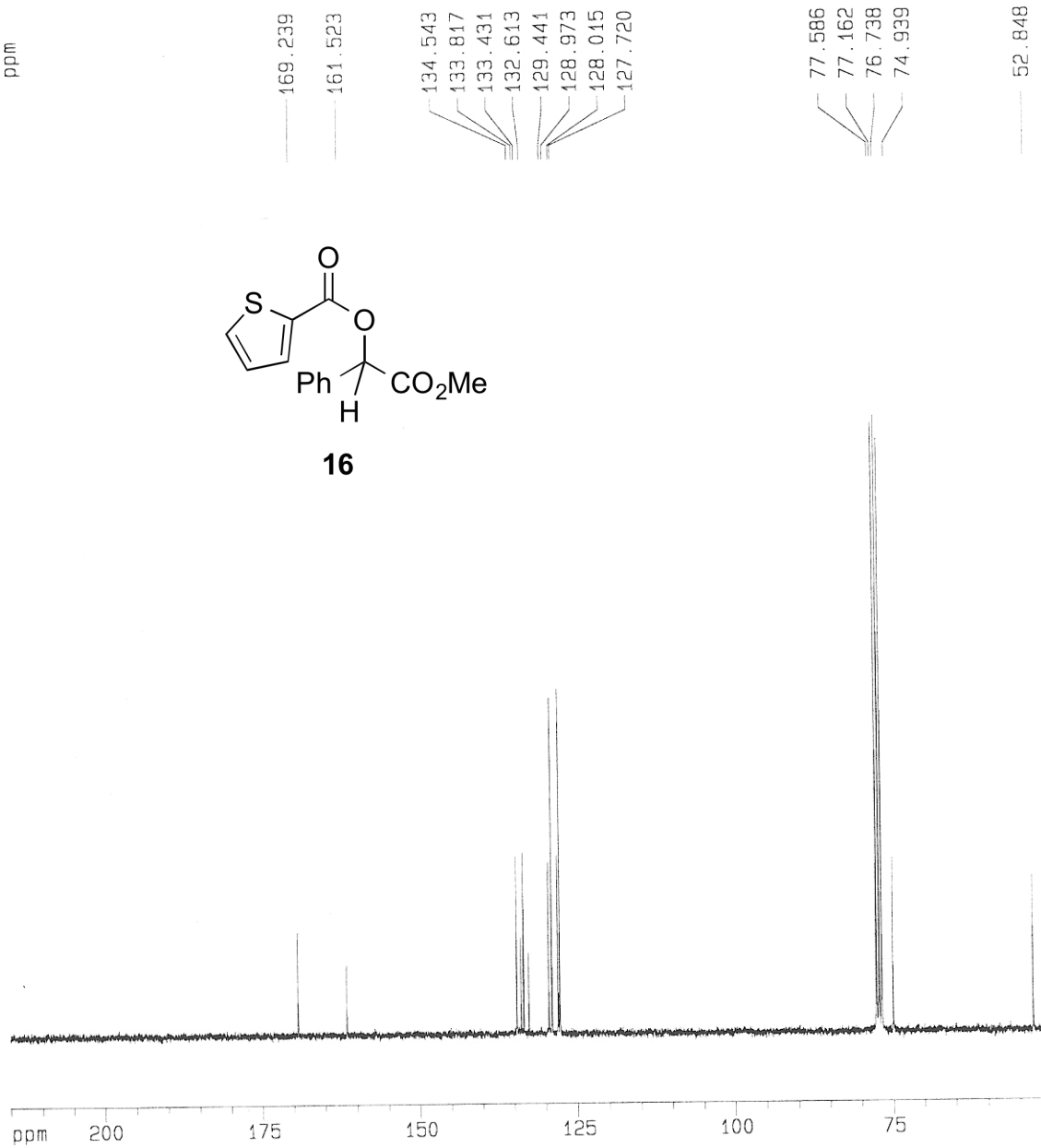

zw3688H 1 1 C:\Bruker\TOPSPIN Radosevich Zhao
[rel]
9.0038
8.9752
8.3821
8.3579
8.0732
8.0459
7.9134
7.8867
7.6508
7.6435
7.6332
7.6255
7.6200
7.5773
7.5739
7.5521
7.5273
7.5008
7.4565
7.4375
7.4325
7.2597
6.2967
3.8050
O
O
Ph
H
CO2Me
15
0.9727
0.9748
1.0290
1.0453
8.5407
1.0000
3.1492
[ppm]



15

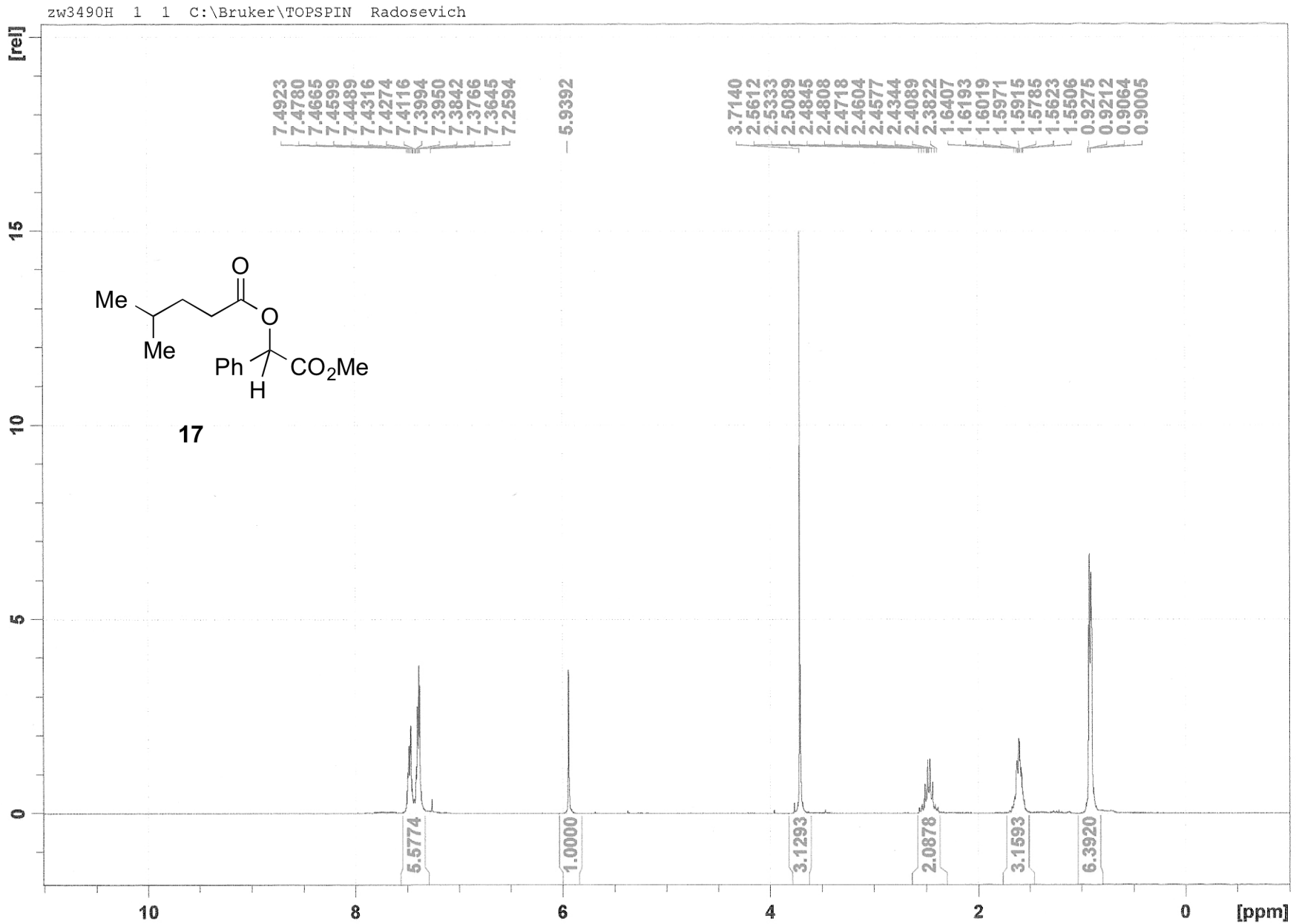
```
Current Data Parameters
NAME                  zw3688C
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140422
Time                  20.25
INSTRUM               spect
PROBHD     5 mm Multinu
PULPROG               zgpg30
TD                     65536
SOLVENT               CDCl3
NS                      2000
DS                         4
SWH               18832.393 Hz
FIDRES             0.287360 Hz
AQ                 1.7400308 sec
RG                   11585.2
DW                    26.550 usec
DE                     6.00 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 ============
NUC1                      13C
P1                       9.75 usec
PL1                      0.00 dB
SFO1               75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  110.00 usec
PL2                      0.00 dB
PL12                    17.50 dB
PL13                    17.50 dB
SFO2              300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                 75.4677422 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     215.000 ppm
F1                    16225.57 Hz
F2P                      -5.000 ppm
F2                     -377.34 Hz
PPMCM                 11.00000 ppm/cm
HZCM                830.14514 Hz/cm
```

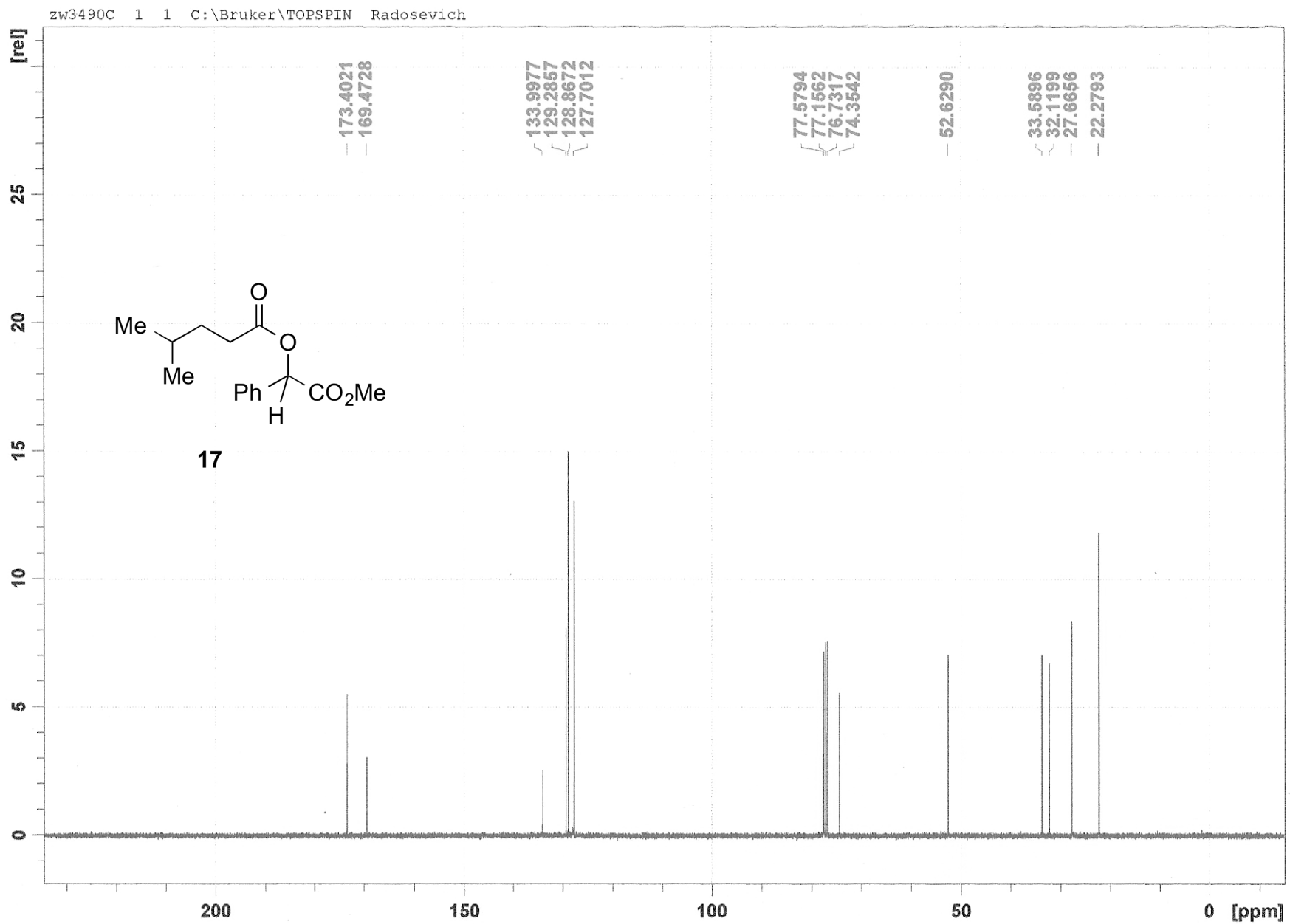

zw3676H 1 1 C:\Bruker\TOPSPIN Radosevich Zhao
[rel]
25
20
15
10
5
0
7.9178
7.9114
7.9085
7.6149
7.6053
7.6027
7.5723
7.5592
7.5557
7.4324
7.4188
7.4156
7.2603
7.1348
7.1224
7.1129
6.1458
3.7545
1.0062
2.2074
3.2097
1.0138
1.0000
3.0217
10
8
6
4
2
0
[ppm]
O
S
O
Ph
H
CO2Me
16

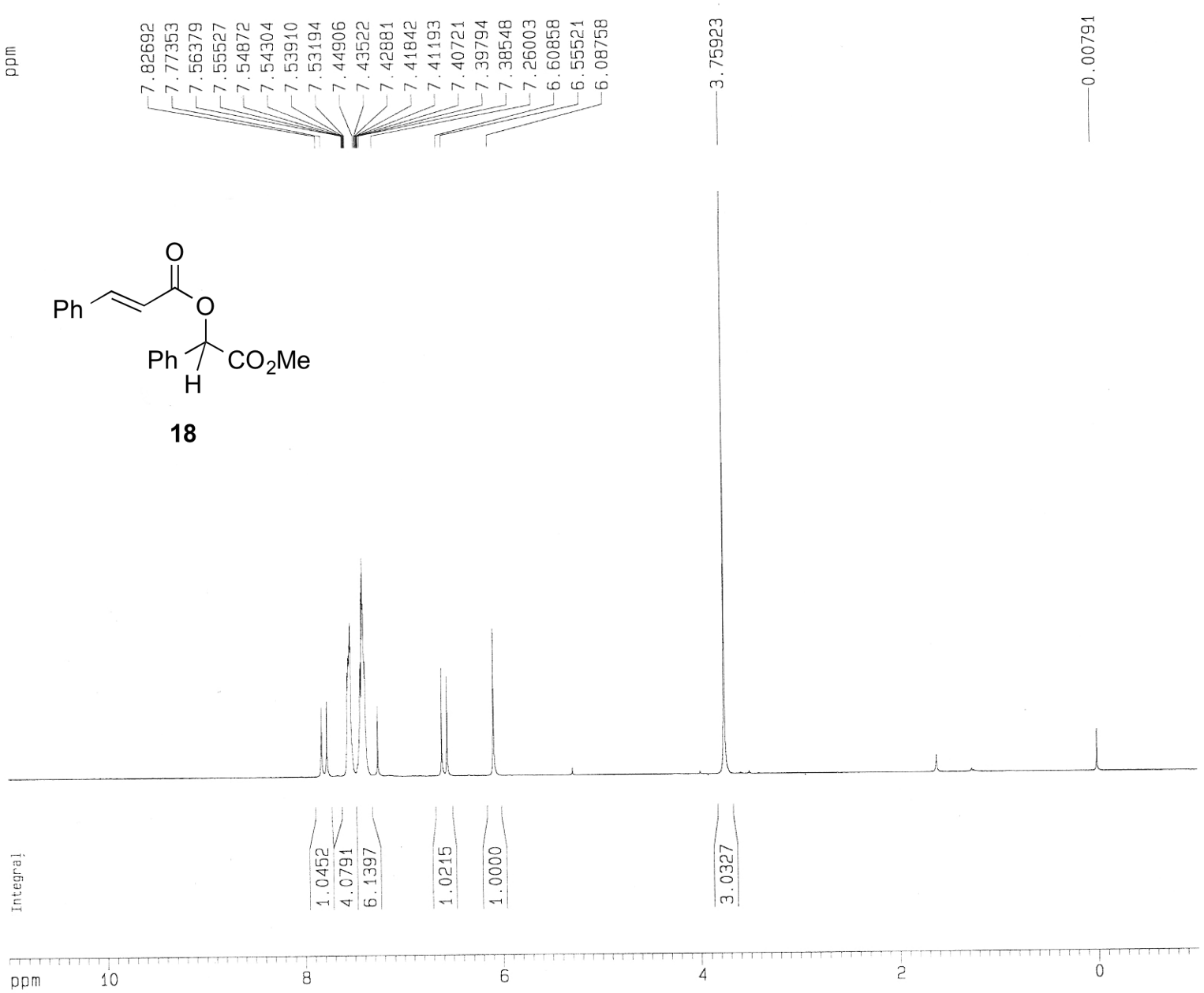


16

```
Current Data Parameters
NAME                 zw3676C
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20140418
Time                 16.58
INSTRUM              spect
PROBHD     5 mm Multinu
PULPROG              zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1418
DS                       4
SWH              18832.393 Hz
FIDRES            0.287360 Hz
AQ                1.7400308 sec
RG                  11585.2
DW                  26.550 usec
DE                   6.00 usec
TE                  300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 ===========
NUC1                   13C
P1                   9.75 usec
PL1                  0.00 dB
SFO1            75.4760200 MHz

============ CHANNEL f2 ===========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              110.00 usec
PL2                  0.00 dB
PL12                17.50 dB
PL13                17.50 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF              75.4677422 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                215.000 ppm
F1                16225.57 Hz
F2P                 -5.000 ppm
F2                 -377.34 Hz
PPMCM             11.00000 ppm/cm
HZCM             830.14514 Hz/cm
```


zw3490H 1 1 C:\Bruker\TOPSPIN Radosevich
[rel]
7.4923
7.4780
7.4665
7.4599
7.4489
7.4316
7.4274
7.4116
7.3994
7.3950
7.3842
7.3766
7.3645
7.2594
5.9392
3.7140
2.5612
2.5333
2.5089
2.4845
2.4808
2.4718
2.4604
2.4577
2.4344
2.4089
2.3822
1.6407
1.6193
1.6019
1.5971
1.5915
1.5785
1.5623
1.5506
0.9275
0.9212
0.9064
0.9005
Me
Me
O
O
Ph
H
CO2Me
17
5.5774
1.0000
3.1293
2.0878
3.1593
6.3920
[ppm]


zw3490C 1 1 C:\Bruker\TOPSPIN Radosevich
[rel]
173.4021
169.4728
133.9977
129.2857
128.8672
127.7012
77.5794
77.1562
76.7317
74.3542
52.6290
33.5896
32.1199
27.6656
22.2793
Me
Me
O
O
Ph
H
CO2Me
17
0
5
10
15
20
25
200
150
100
50
0 [ppm]



Current Data Parameters

NAME	zw3695H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20140423
Time	18.59
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	287.4
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

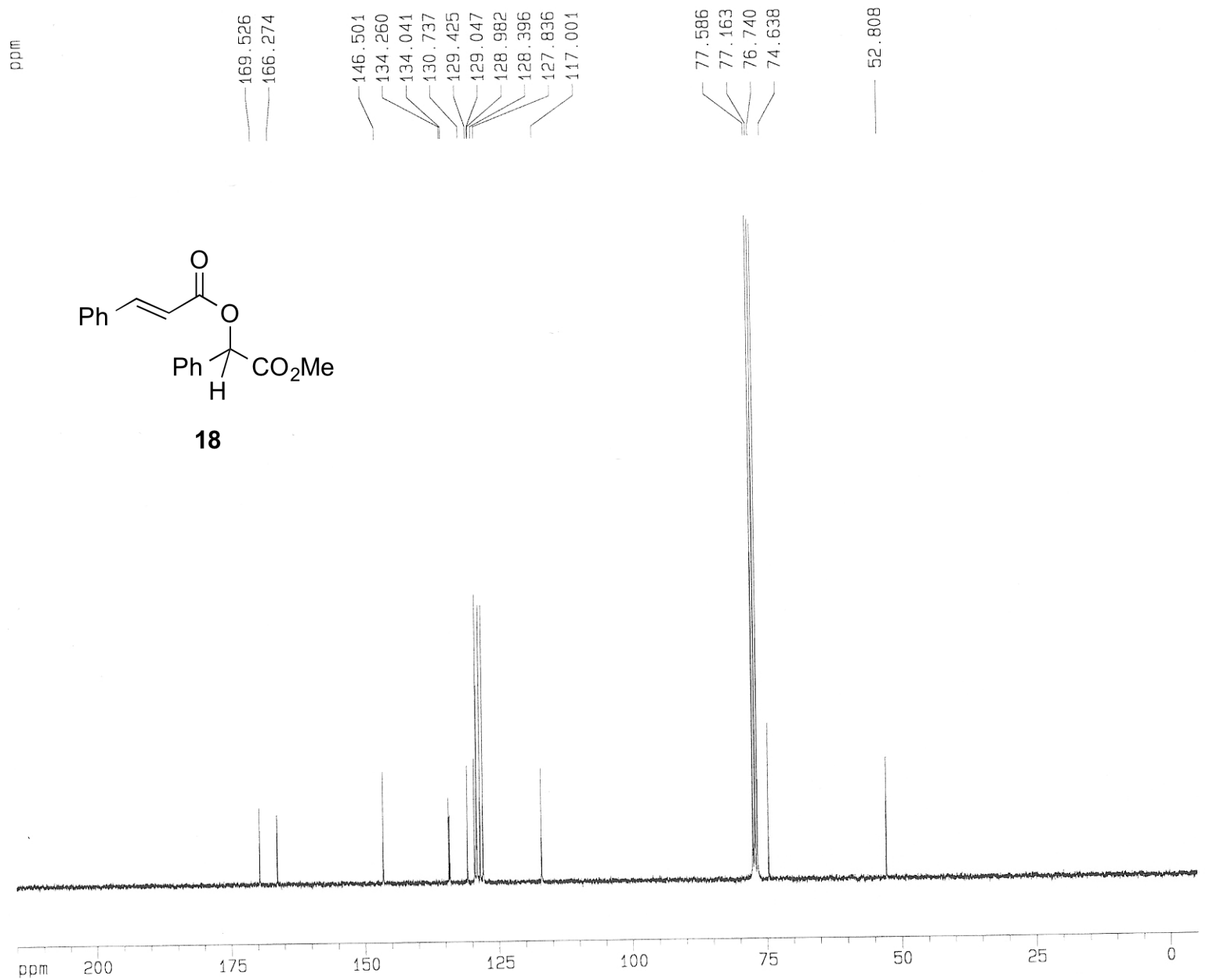
NUC1	1H
P1	10.10 usec
PL1	-6.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300062 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3301.43 Hz
F2P	-1.000 ppm
F2	-300.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	180.07800 Hz/cm



Current Data Parameters

NAME	zw3695C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20140424
Time	2.25
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4000
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	16384
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	9.75 usec
PL1	0.00 dB
SFO1	75.4760200 MHz

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

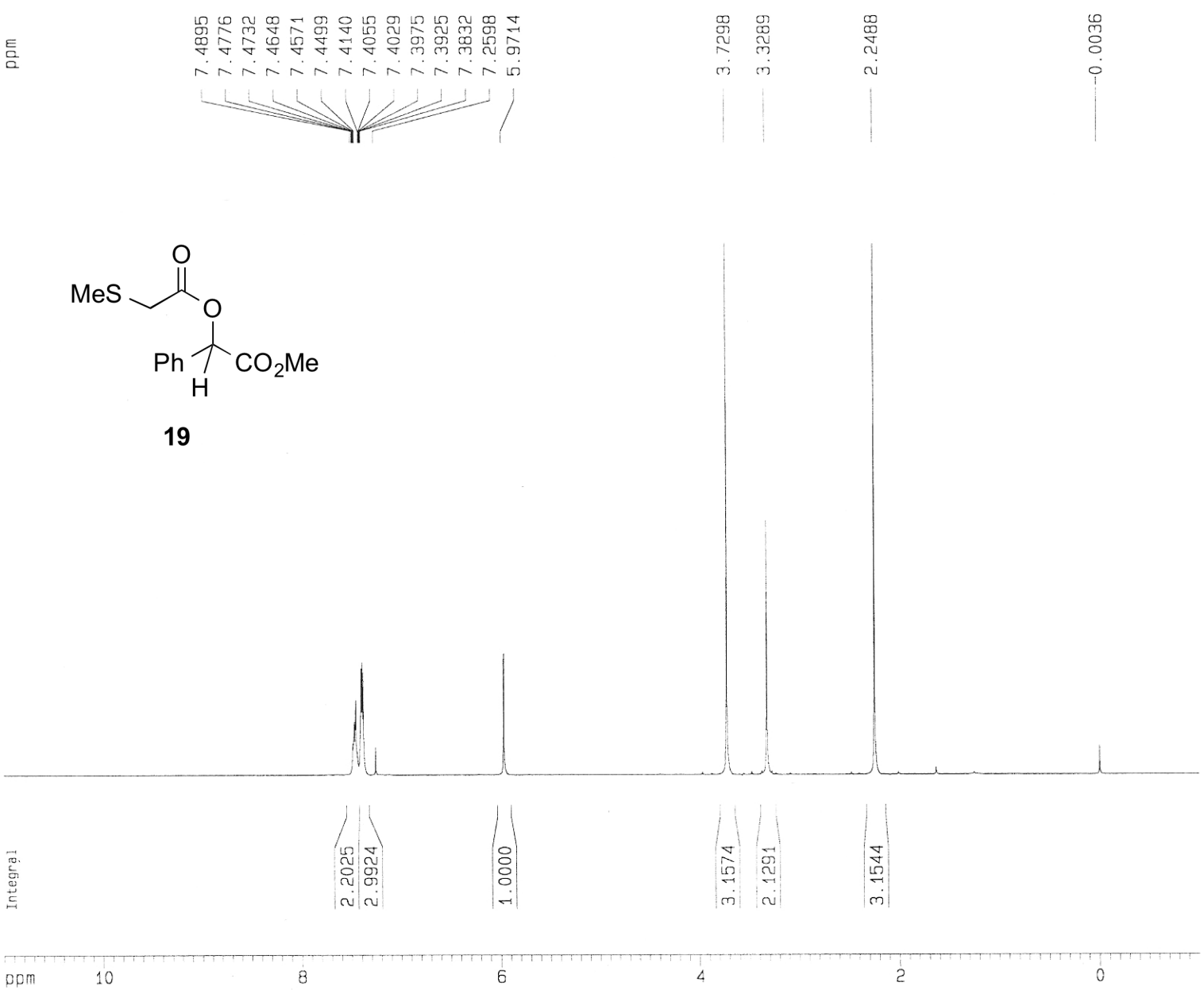
CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

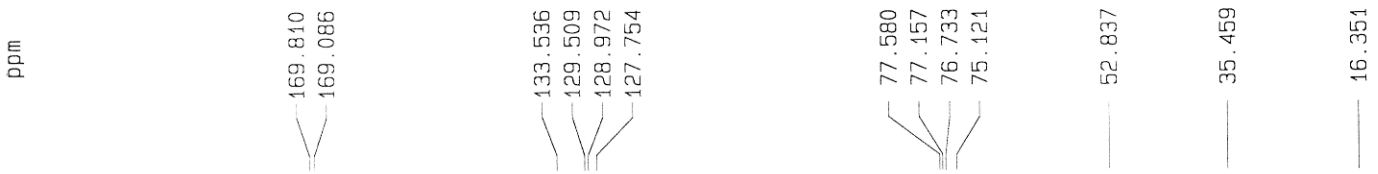
SI	32768
SF	75.4677417 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

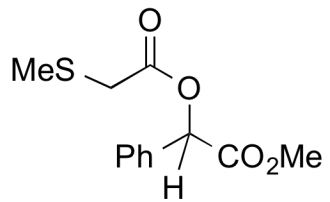
1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16225.57 Hz
F2P	-5.000 ppm
F2	-377.34 Hz
PPMCM	11.00000 ppm/c
HZCM	830.14514 Hz/cm

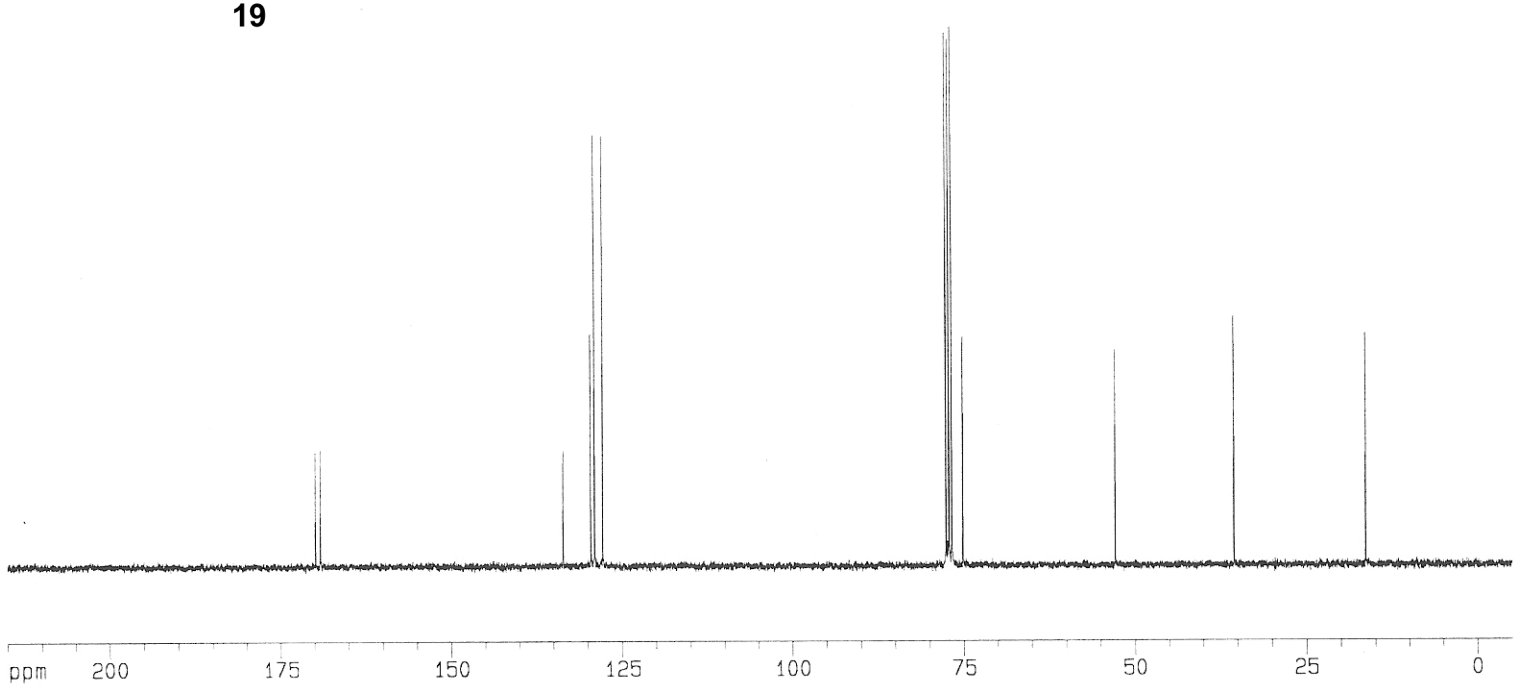



```
Current Data Parameters
NAME                zw3703B_H
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_                20140501
Time                    13.24
INSTRUM                 spect
PROBHD      5 mm Multinu
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                6172.839 Hz
FIDRES             0.094190 Hz
AQ                 5.3084660 sec
RG                     287.4
DW                  81.000 usec
DE                   6.00 usec
TE                   300.0 K
D1                1.00000000 sec

============ CHANNEL f1 ========
NUC1                       1H
P1                      10.10 usec
PL1                     -6.00 dB
SFO1             300.1318534 MHz

F2 - Processing parameters
SI                         32768
SF               300.1300064 MHz
WDW                           no
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
F1P                        11.000 ppm
F1                       3301.43 Hz
F2P                        -1.000 ppm
F2                       -300.13 Hz
PPMCM                   0.60000 ppm/cm
HZCM                  180.07800 Hz/cm
```

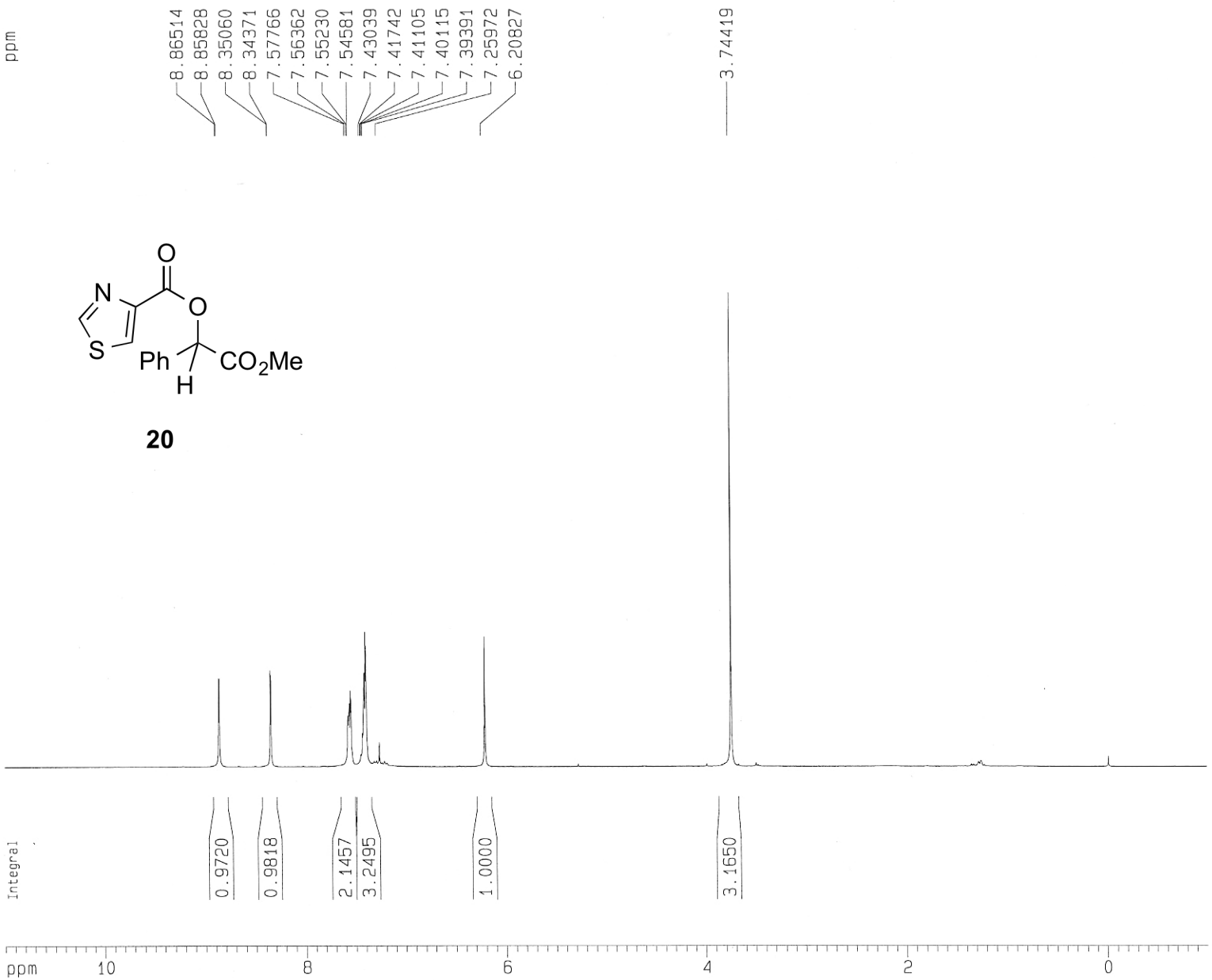




19

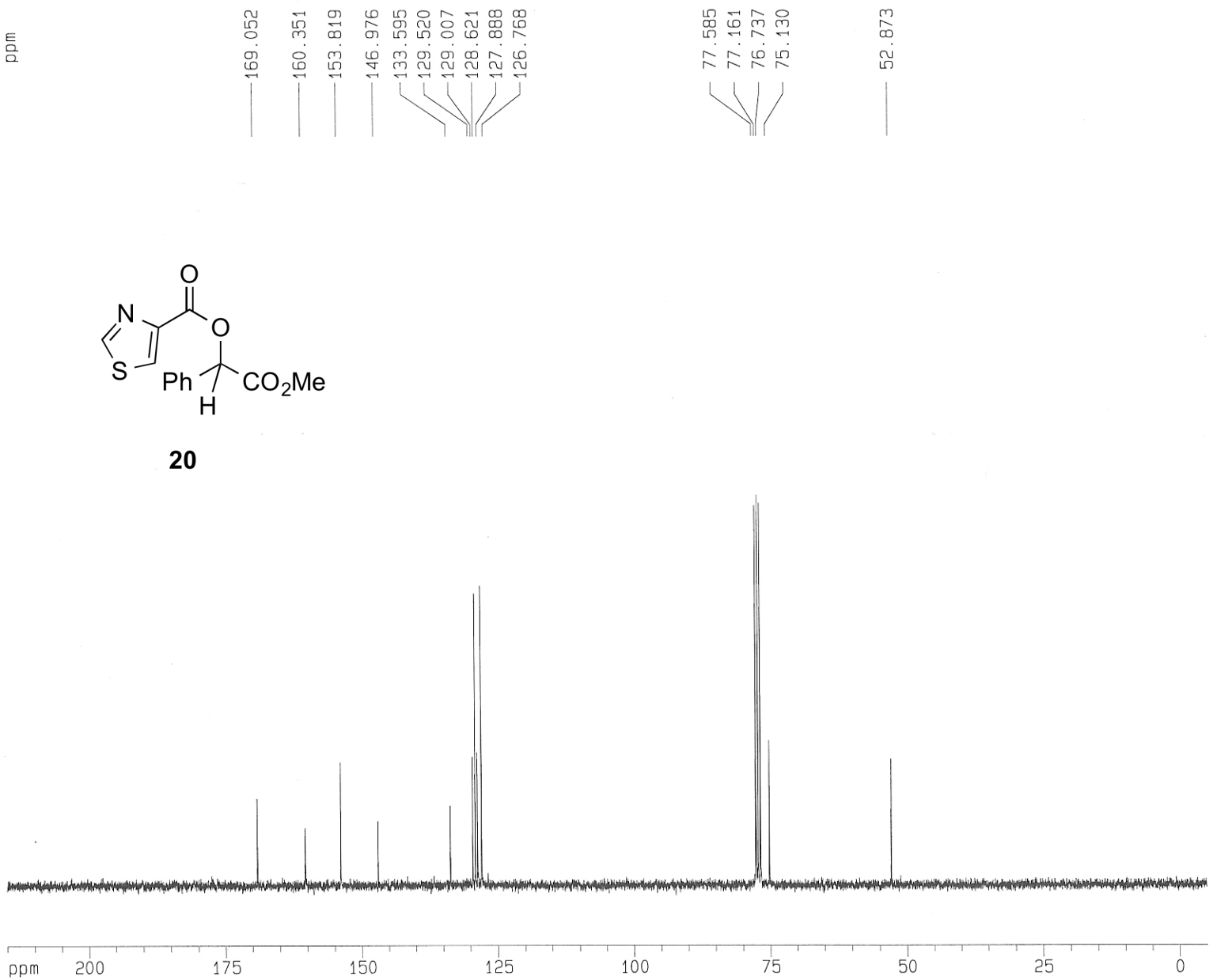



```
Current Data Parameters
NAME            zw37038_C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140501
Time               15.13
INSTRUM            spect
PROBHD     5 mm Multinu
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1700
DS                    4
SWH           18832.393 Hz
FIDRES         0.287360 Hz
AG             1.7400308 sec
RG                16384
DW               26.550 usec
DE                6.00 usec
TE               300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                9.75 usec
PL1               0.00 dB
SFO1         75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                1H
PCPD2           110.00 usec
PL2               0.00 dB
PL12              17.50 dB
PL13              17.50 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF           75.4677417 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             16225.57 Hz
F2P               -5.000 ppm
F2              -377.34 Hz
PPMCM          11.00000 ppm/cm
HZCM          830.14514 Hz/cm
```



20

```
Current Data Parameters
NAME                  zw3677B
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140418
Time                  15.46
INSTRUM               spect
PROBHD     5 mm QNP 1H/1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                   228.1
DW                  81.000 usec
DE                   6.00 usec
TE                  300.0 K
D1                1.00000000 sec

========== CHANNEL f1 ========
NUC1                    1H
P1                   12.10 usec
PL1                   0.00 dB
SFO1           299.8718518 MHz

F2 - Processing parameters
SI                      32768
SF             299.8700099 MHz
WDW                        no
SSB                         0
LB                    0.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                 11.000 ppm
F1                3298.57 Hz
F2P                -1.000 ppm
F2                -299.87 Hz
PPMCM             0.60000 ppm/cm
HZCM            179.92200 Hz/cm
```



20

Current Data Parameters

NAME	zw3677B
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20140418
Time	16.30
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	651
DS	4
SWH	18796.992 Hz
FIDRES	0.286819 Hz
AQ	1.7433076 sec
RG	16384
DW	26.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

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

NUC1	13C
P1	5.25 usec
PL1	-6.00 dB
SFO1	75.4106357 MHz

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

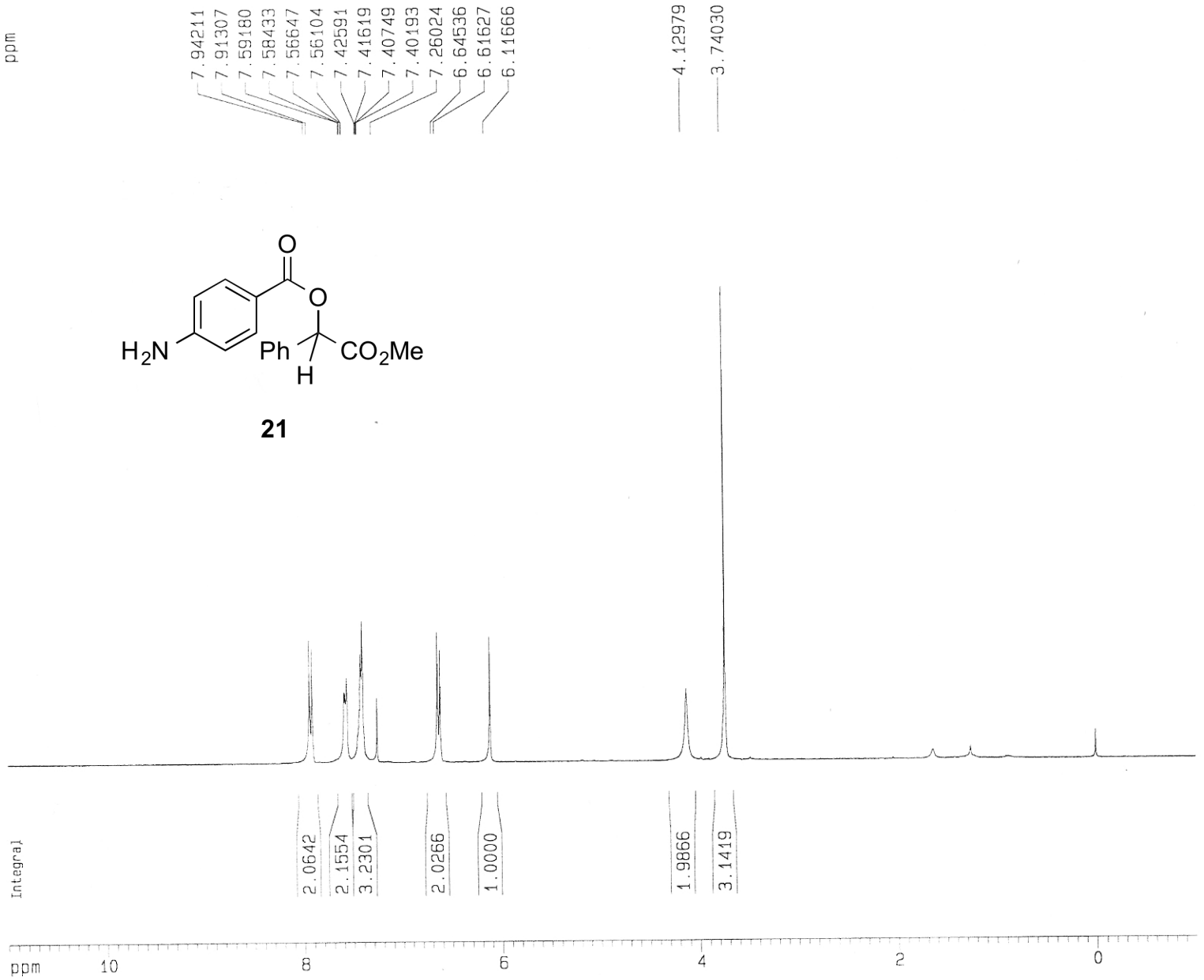
CPDPRG2	waltz16
NUC2	1H
PCPD2	115.00 usec
PL2	0.00 dB
PL12	19.70 dB
PL13	19.70 dB
SFO2	299.8711995 MHz

F2 - Processing parameters

SI	32768
SF	75.4023683 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

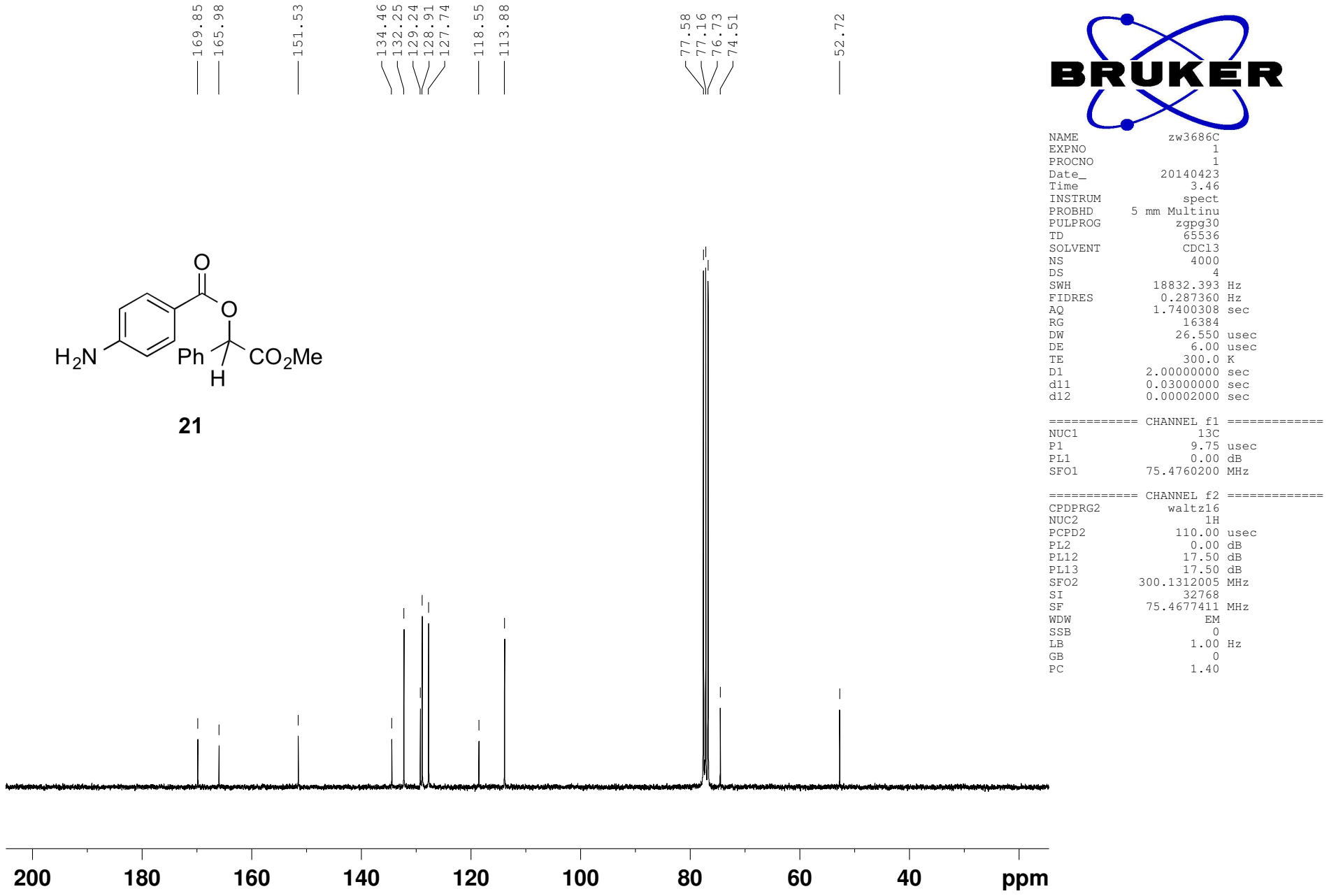
1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16211.51 Hz
F2P	-5.000 ppm
F2	-377.01 Hz
PPMCM	11.00000 ppm/cm
HZCM	829.42603 Hz/cm



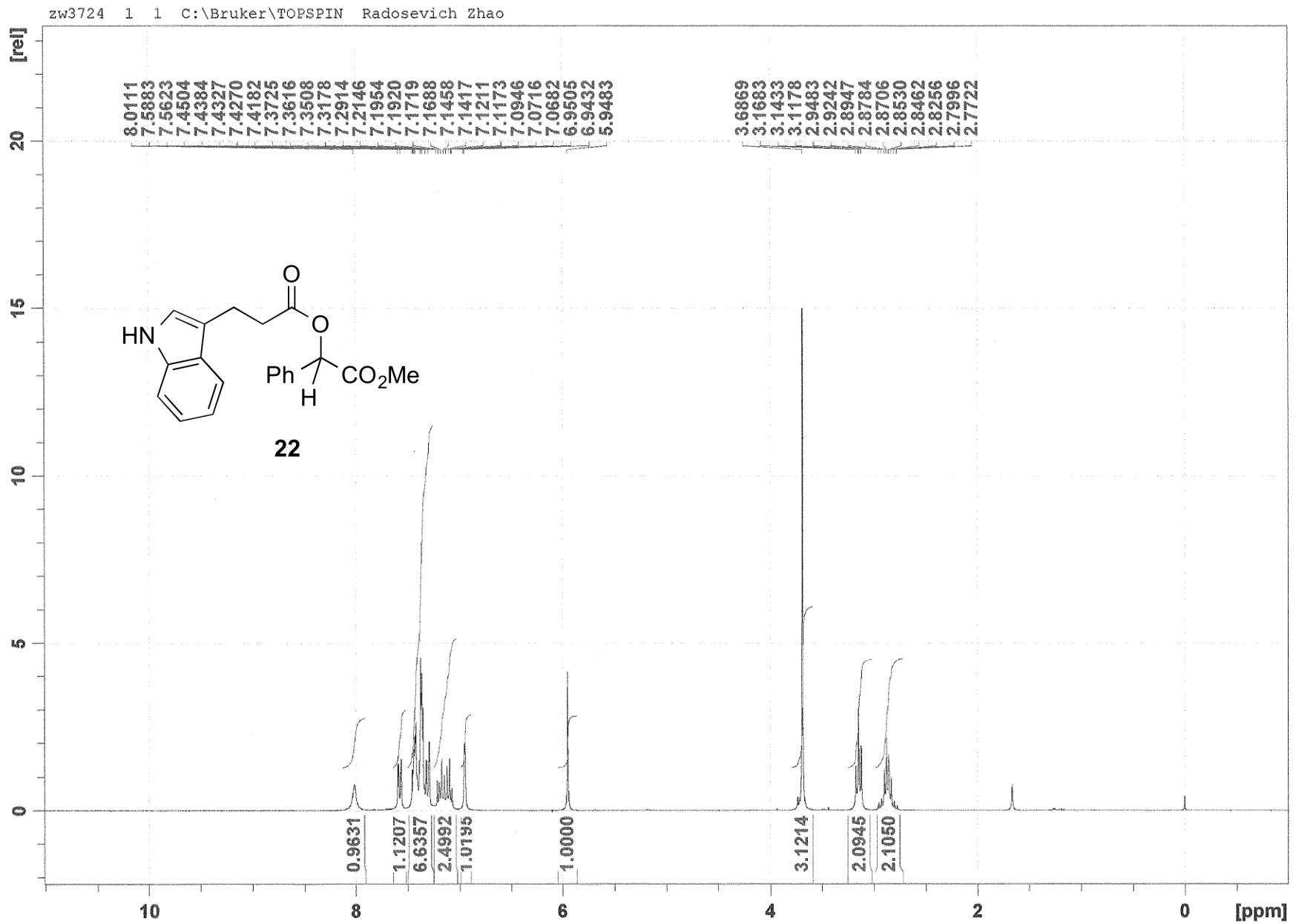
21

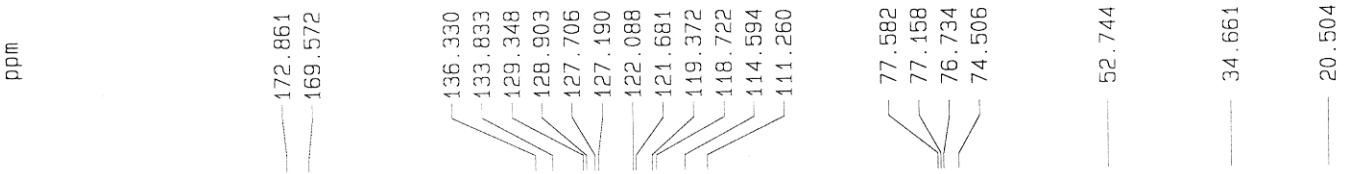
```
Current Data Parameters
NAME                  zw3686H
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140422
Time                  23.34
INSTRUM               spect
PROBHD      5 mm Multinu
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                     362
DW                 81.000 usec
DE                  6.00 usec
TE                 300.0 K
D1                1.00000000 sec

============ CHANNEL f1 =========
NUC1                    1H
P1                  10.10 usec
PL1                 -6.00 dB
SF01           300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300058 MHz
WDW                     no
SSB                      0
LB                  0.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                  20.00 cm
F1P                11.000 ppm
F1                3301.43 Hz
F2P                -1.000 ppm
F2                -300.13 Hz
PPMCM             0.60000 ppm/cm
HZCM            180.07800 Hz/cm
```

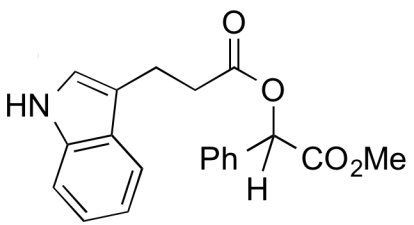


BRUKER

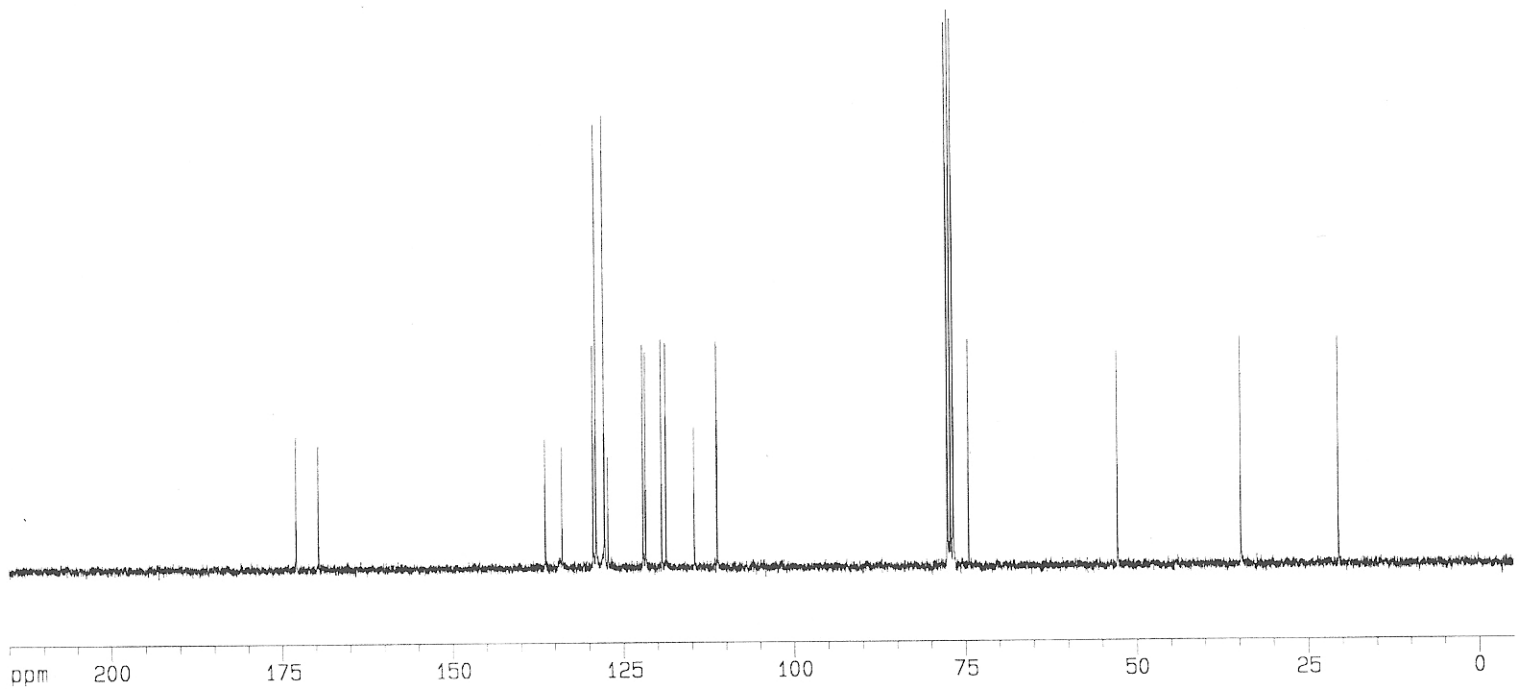
NAME	zw3686C
EXPNO	1
PROCNO	1
Date_	20140423
Time	3.46
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4000
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	16384
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec
========== CHANNEL f1 ==========	
NUC1	13C
P1	9.75 usec
PL1	0.00 dB
SFO1	75.4760200 MHz
========== CHANNEL f2 ==========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677411 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


zw3724 1 1 C:\Bruker\TOPSPIN Radosevich Zhao
[rel]
8.0111
7.5883
7.5623
7.4504
7.4384
7.4327
7.4270
7.4182
7.3725
7.3616
7.3508
7.3178
7.2914
7.2146
7.1954
7.1920
7.1719
7.1688
7.1458
7.1417
7.1211
7.1173
7.0946
7.0716
7.0682
6.9505
6.9432
5.9483
3.6869
3.1683
3.1433
3.1178
2.9483
2.9242
2.8947
2.8784
2.8706
2.8530
2.8462
2.8256
2.7996
2.7722
HN
O
O
Ph
H
CO2Me
22
0.9631
1.1207
6.6357
2.4992
1.0195
1.0000
3.1214
2.0945
2.1050
[ppm]

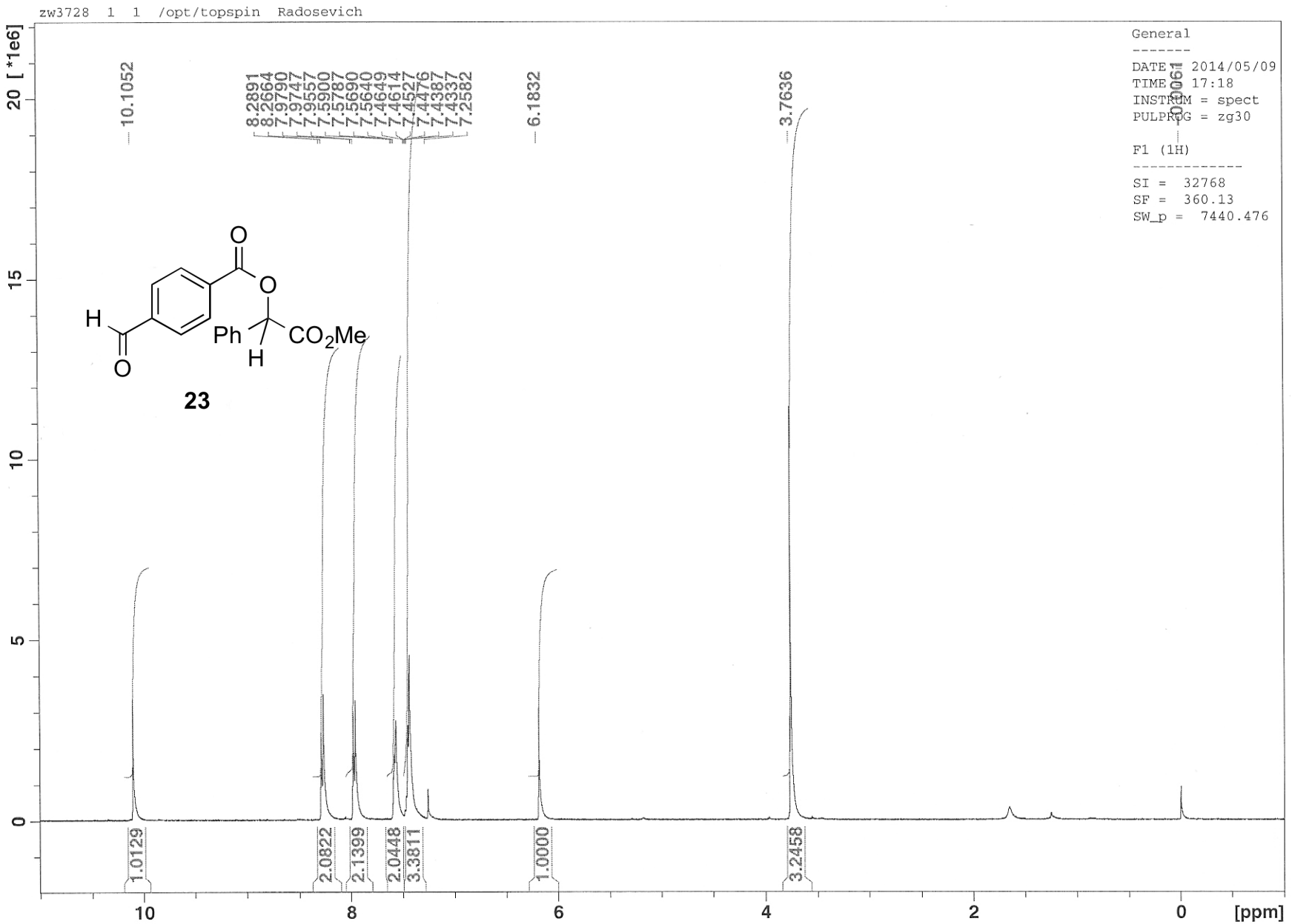




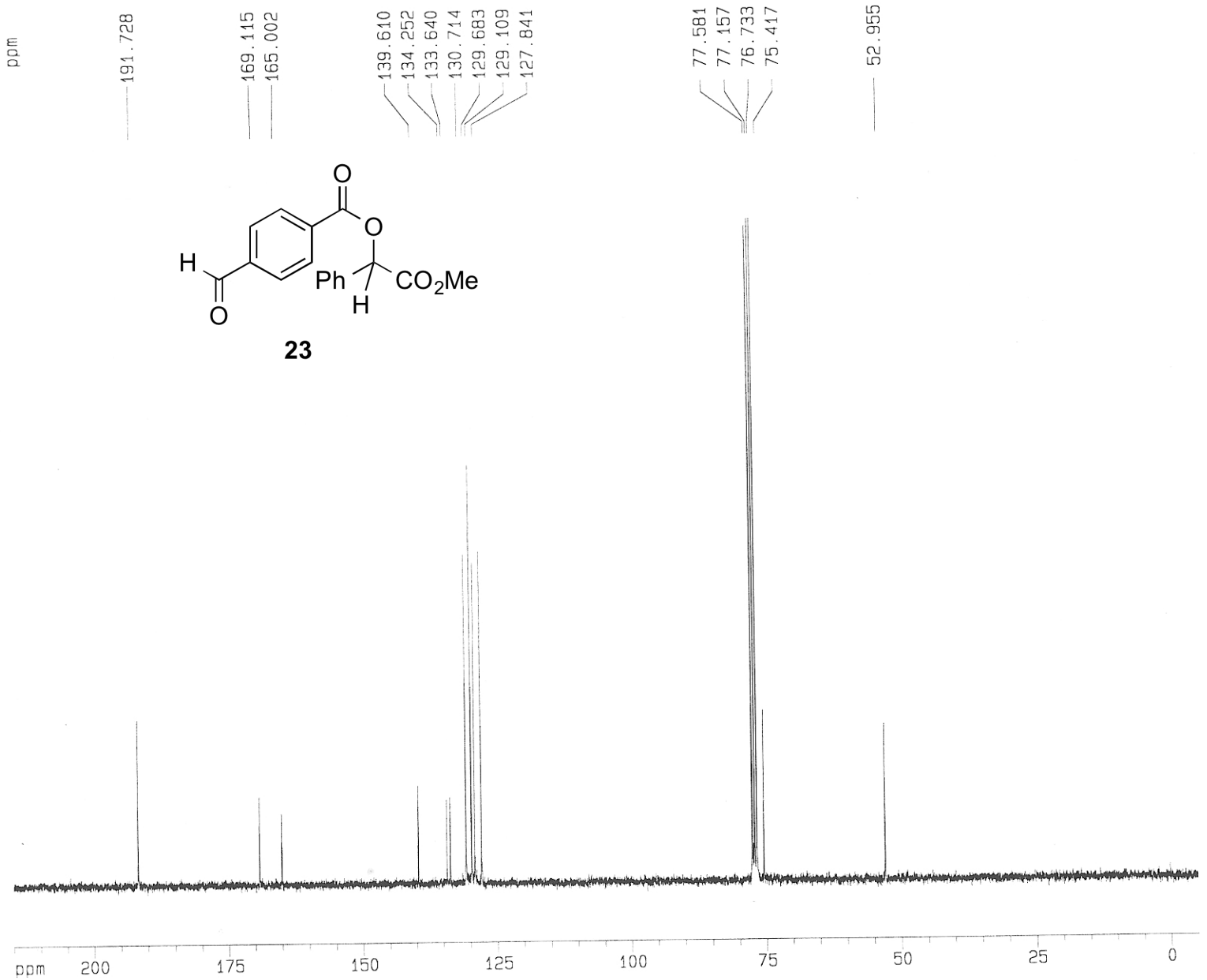
22




```
Current Data Parameters
NAME                  zw3724
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_              20140509
Time                  11.19
INSTRUM               spect
PROBHD     5 mm Multinu
PULPROG               zgpg30
TD                     65536
SOLVENT               CDCl3
NS                      1500
DS                         4
SWH               18832.393 Hz
FIDRES             0.287360 Hz
AQ                 1.7400308 sec
RG                     16384
DW                    26.550 usec
DE                     6.00 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 ============
NUC1                     13C
P1                      9.75 usec
PL1                     0.00 dB
SFO1              75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2              waltz16
NUC2                     1H
PCPD2                110.00 usec
PL2                     0.00 dB
PL12                   17.50 dB
PL13                   17.50 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                       32768
SF                75.4677463 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     215.000 ppm
F1                    16225.57 Hz
F2P                    -5.000 ppm
F2                    -377.34 Hz
PPMCM                 11.00000 ppm/cm
HZCM                830.14514 Hz/cm
```

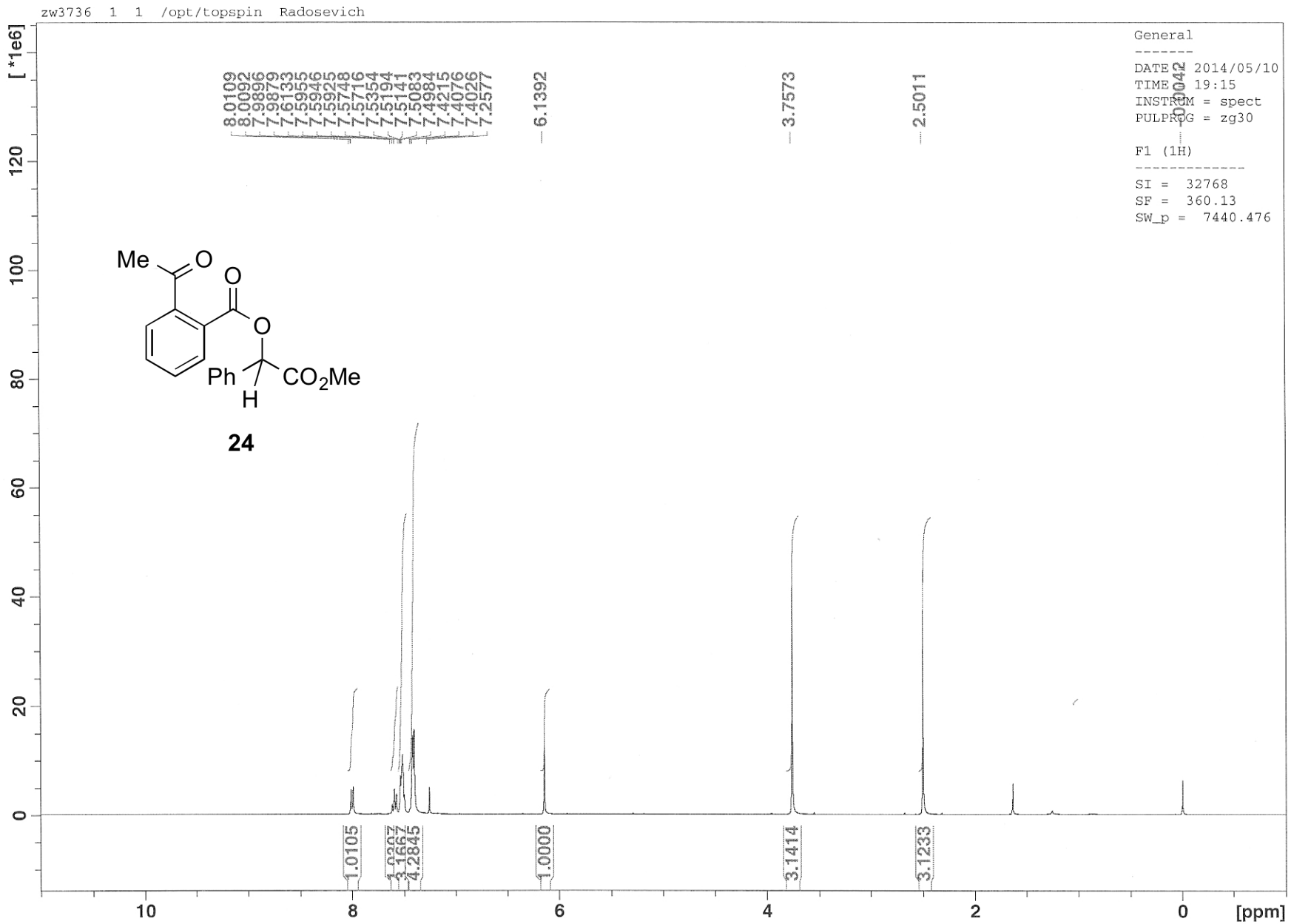

zw3728 1 1 /opt/topspin Radosevich
General
DATE = 2014/05/09
TIME = 17:18
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
10.1052
8.2891
8.2664
7.9790
7.9747
7.9557
7.5900
7.5787
7.5690
7.5640
7.4649
7.4614
7.4527
7.4476
7.4387
7.4337
7.2582
6.1832
3.7636
1.0129
2.0822
2.1399
2.0448
3.3811
1.0000
3.2458
20 [*1e6]
15
10
5
0
10
8
6
4
2
0
[ppm]

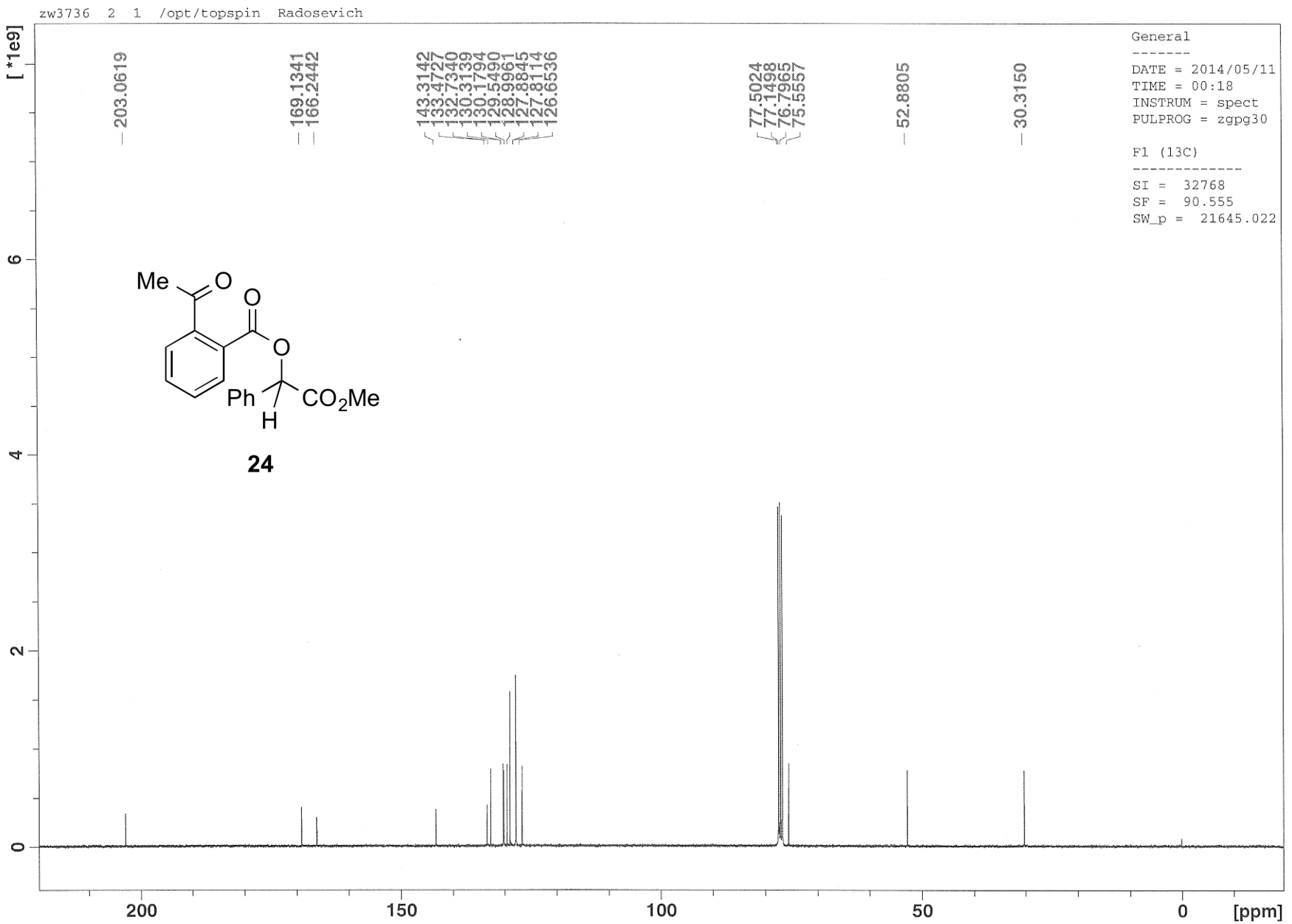
23

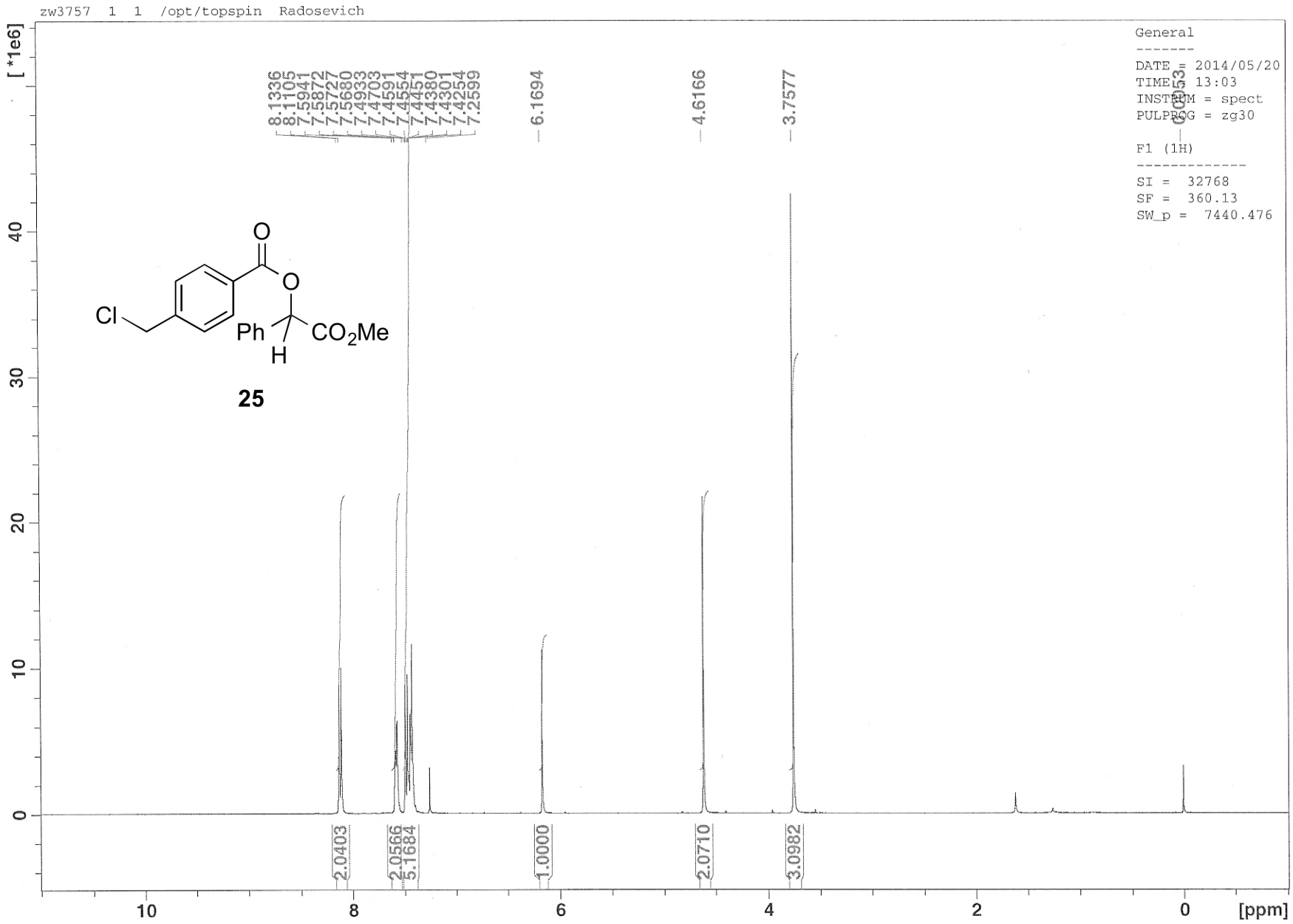


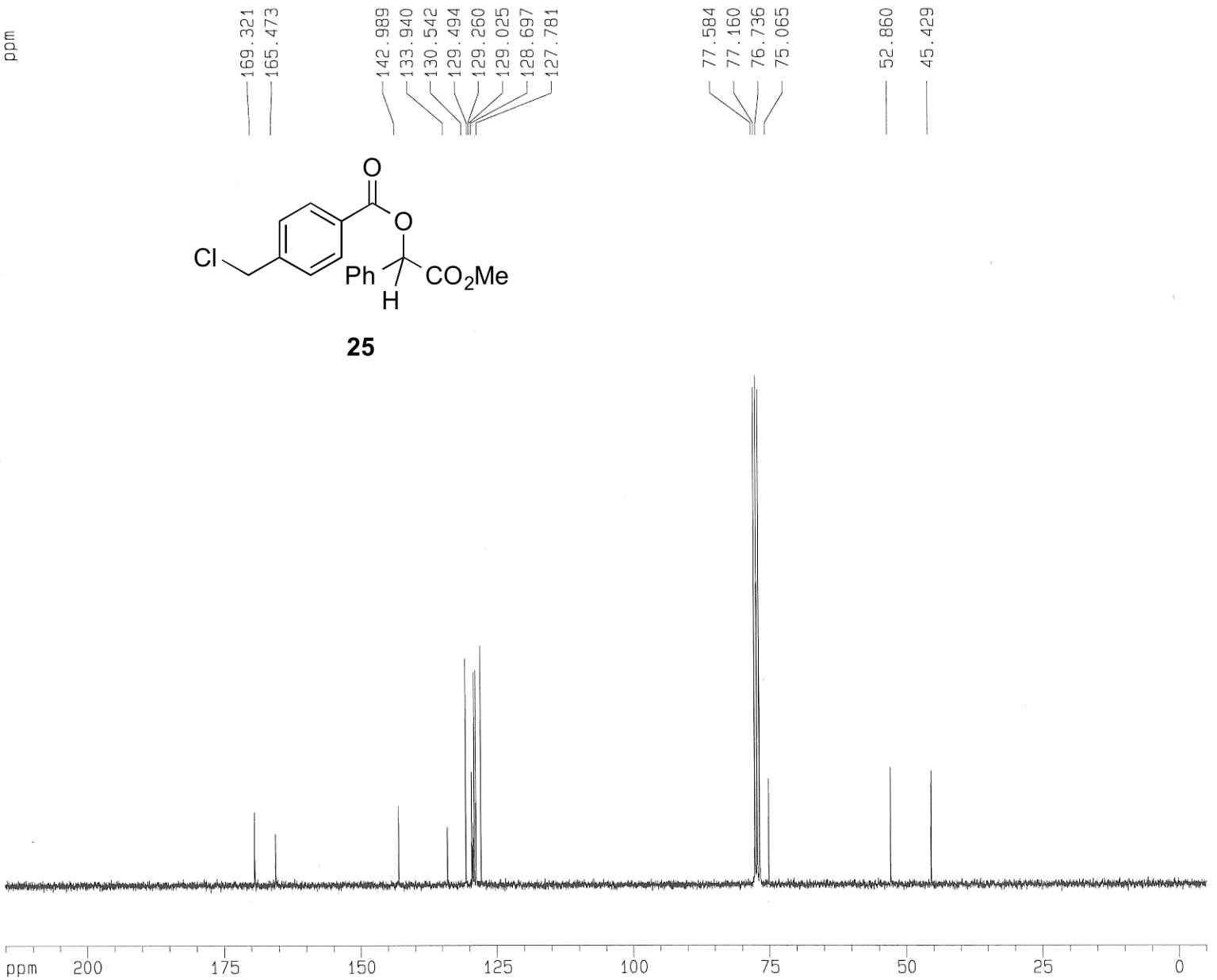
23

```
Current Data Parameters
NAME                  zw3728C
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140509
Time                 18.19
INSTRUM              spect
PROBHD     5 mm Multinu
PULPROG              zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    2694
DS                       4
SWH              18832.393 Hz
FIDRES            0.287360 Hz
AQ                1.7400308 sec
RG                  11585.2
DW                  26.550 usec
DE                   6.00 usec
TE                  300.0 K
D1               2.00000000 sec
d11              0.03000000 sec
d12              0.00002000 sec

============ CHANNEL f1 ============
NUC1                   13C
P1                   9.75 usec
PL1                  0.00 dB
SFO1            75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2             waltz16
NUC2                   1H
PCPD2              110.00 usec
PL2                  0.00 dB
PL12                17.50 dB
PL13                17.50 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF              75.4677405 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
F1P                215.000 ppm
F1               16225.57 Hz
F2P                -5.000 ppm
F2                -377.34 Hz
PPMCM             11.00000 ppm/cm
HZCM             830.14514 Hz/cm
```


zw3736 1 1 /opt/topspin Radosevich
General
DATE = 2014/05/10
TIME = 19:15
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
8.0109
8.0092
7.9896
7.9879
7.6133
7.5955
7.5946
7.5925
7.5748
7.5716
7.5354
7.5194
7.5141
7.5083
7.4984
7.4215
7.4076
7.4026
7.2577
6.1392
3.7573
2.5011
1.0042
Me
O
O
O
Ph
H
CO2Me
24
1.0105
1.0207
3.1667
4.2845
1.0000
3.1414
3.1233
[*1e6]
[ppm]

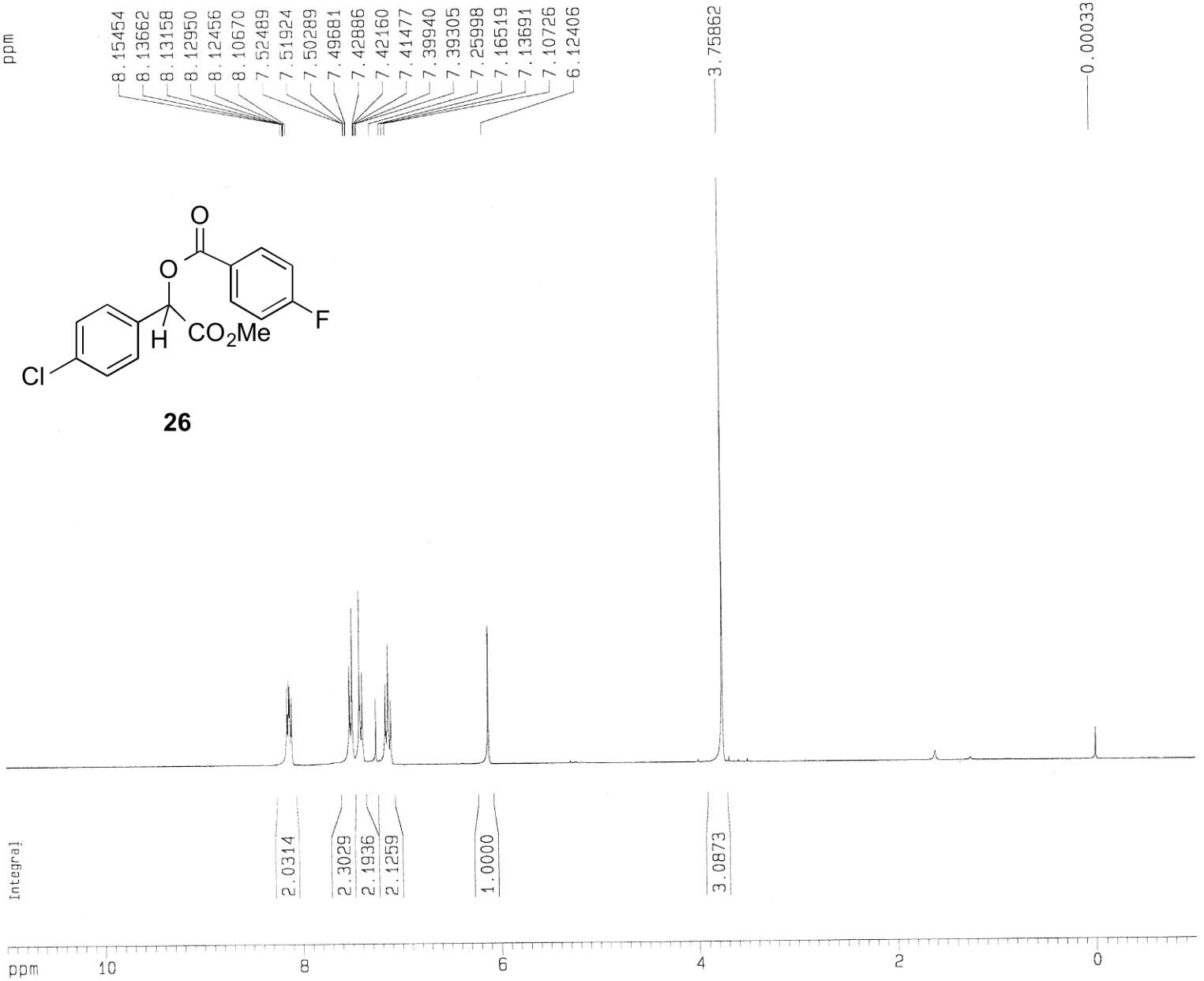

zw3736 2 1 /opt/topspin Radosevich
[*1e9]
203.0619
169.1341
166.2442
143.3142
133.4727
132.7340
130.3139
130.1794
129.5490
128.9961
127.8845
127.8114
126.6536
77.5024
77.1498
76.7965
75.5557
52.8805
30.3150
General
DATE = 2014/05/11
TIME = 00:18
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 90.555
SW_p = 21645.022
Me
O
O
O
Ph
H
CO2Me
24
0
2
4
6
200
150
100
50
0
[ppm]


zw3757 1 1 /opt/topspin Radosevich
[*1e6]
8.1336
8.1105
7.5941
7.5872
7.5727
7.5680
7.4933
7.4703
7.4591
7.4554
7.4451
7.4380
7.4301
7.4254
7.2599
6.1694
4.6166
3.7577
0.0053
General
DATE = 2014/05/20
TIME = 13:03
INSTRM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
2.0403
2.0566
5.1684
1.0000
2.0710
3.0982
[ppm]
Cl
O
O
Ph
H
CO2Me
25



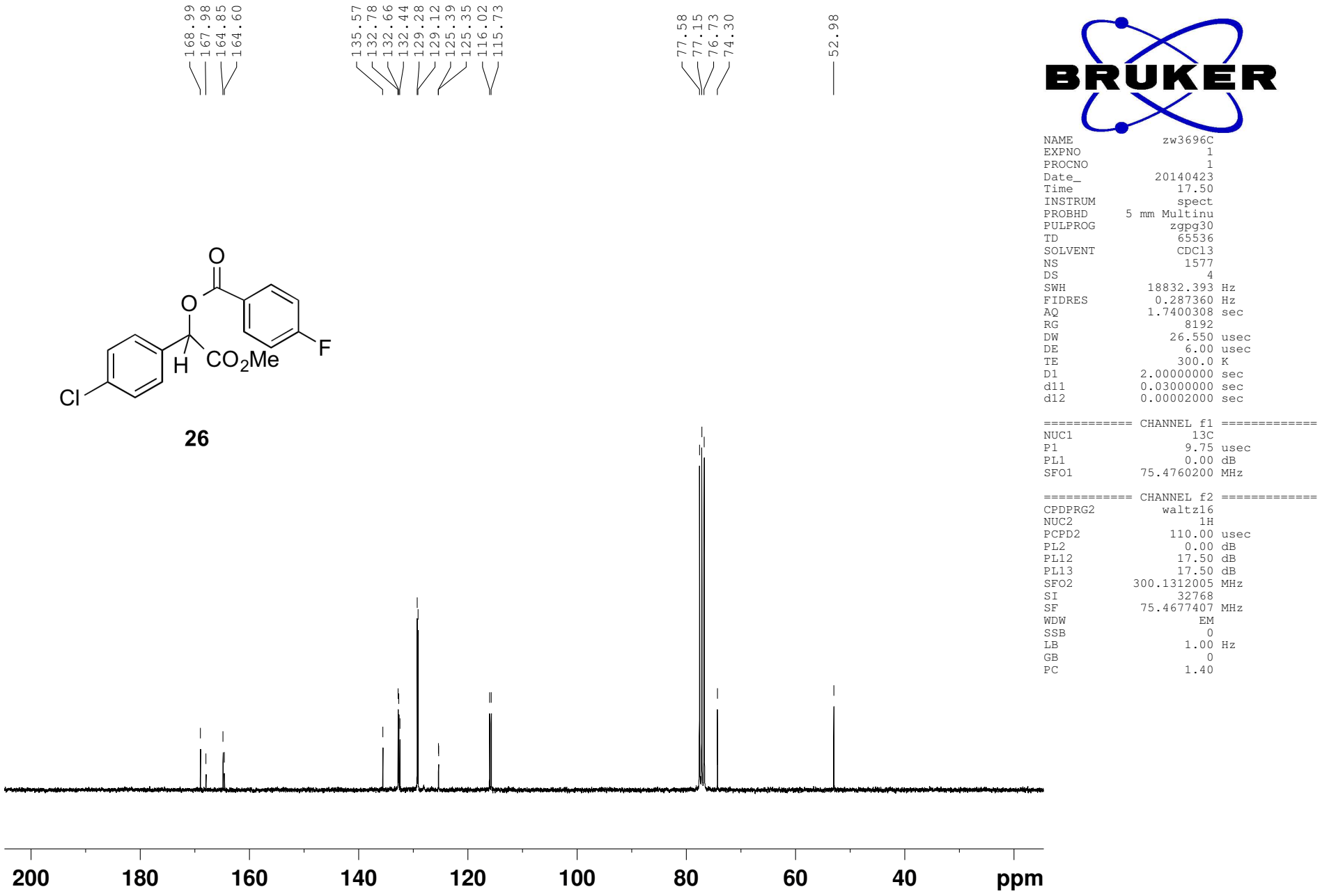
25

```
Current Data Parameters
NAME                  zw3757C
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140520
Time                  13.34
INSTRUM               spect
PROBHD     5 mm GNP 1H/1
PULPROG               zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1700
DS                        4
SWH              18796.992 Hz
FIDRES           0.286819 Hz
AQ               1.7433076 sec
RG                 18390.4
DW                  26.600 usec
DE                   6.00 usec
TE                  300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
D12              0.00002000 sec

============ CHANNEL f1 ============
NUC1                  13C
P1                   5.25 usec
PL1                 -6.00 dB
SFO1           75.4106357 MHz

============ CHANNEL f2 ============
CPDPRG2           waltz16
NUC2                  1H
PCPD2              115.00 usec
PL2                  0.00 dB
PL12                19.70 dB
PL13                19.70 dB
SFO2          299.8711995 MHz

F2 - Processing parameters
SI                    32768
SF             75.4023654 MHz
WDW                   EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                215.000 ppm
F1               16211.51 Hz
F2P                 -5.000 ppm
F2                -377.01 Hz
PPMCM           11.00000 ppm/cm
HZCM           829.42603 Hz/cm
```



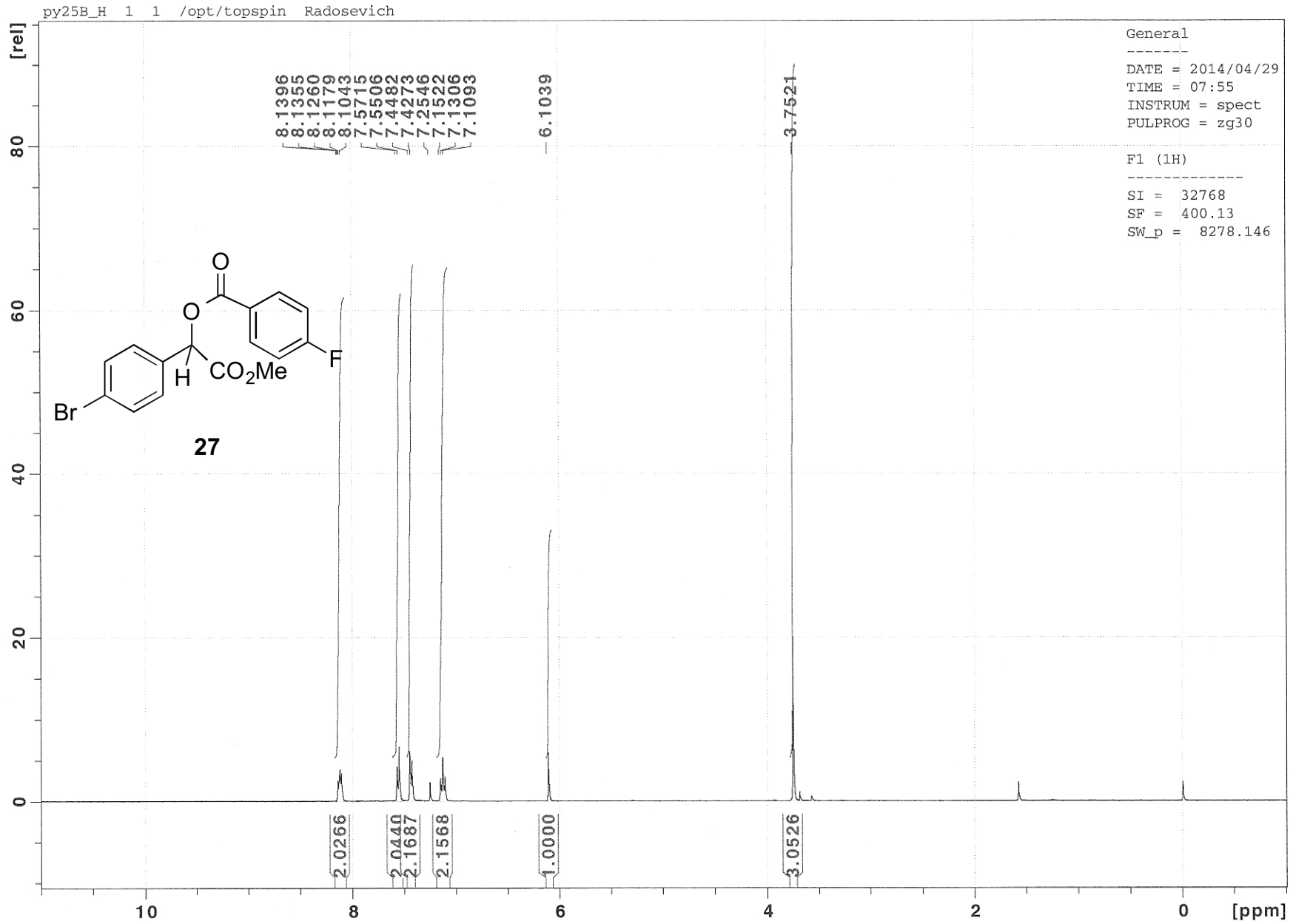
26

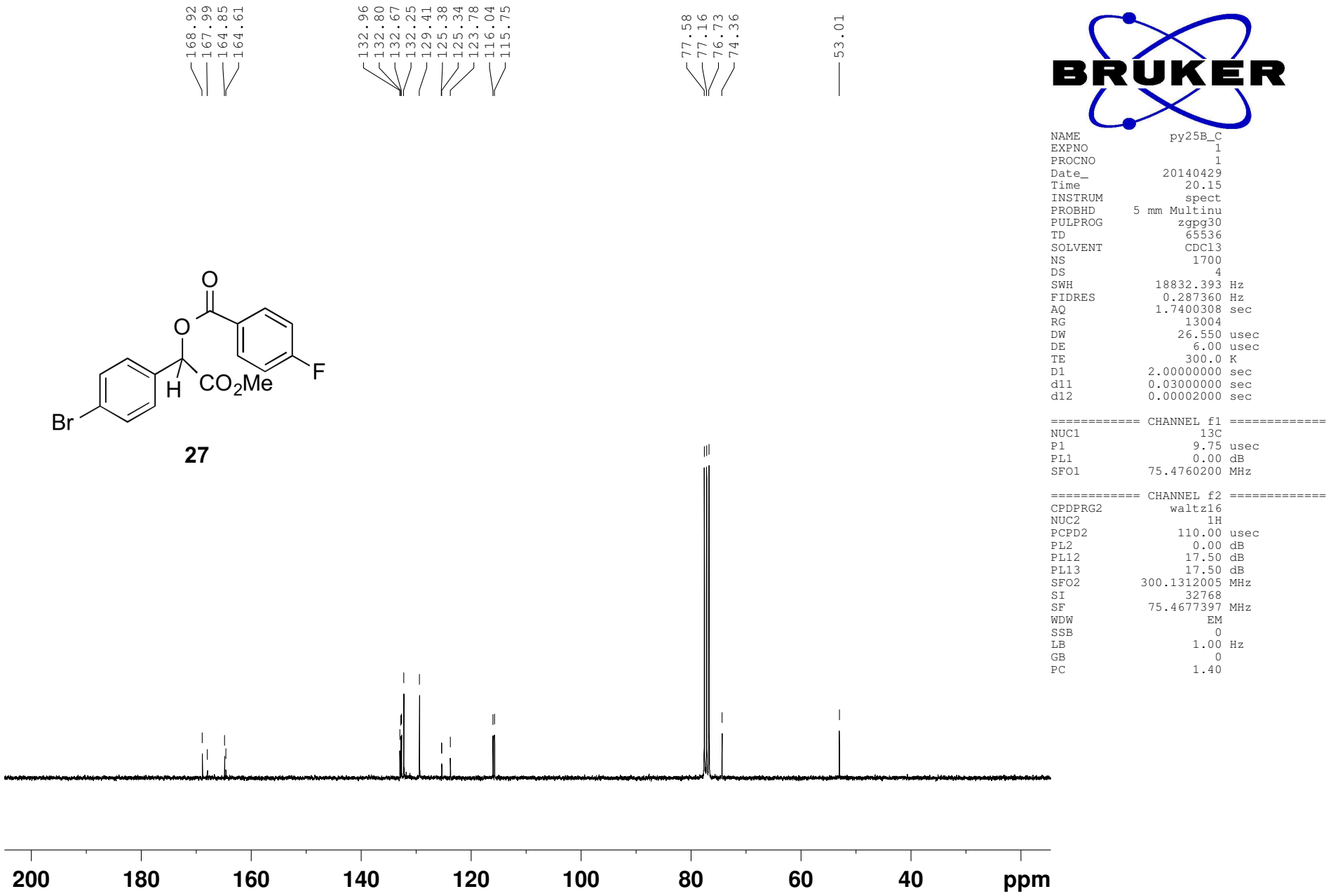
```
Current Data Parameters
NAME                  zw3696H
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140423
Time                  16.09
INSTRUM               spect
PROBHD      5 mm Multinu
PULPROG               zg30
TD                    65536
SOLVENT               CDCl3
NS                       13
DS                        2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    322.5
DW                 81.000 usec
DE                  6.00 usec
TE                  300.0 K
D1              1.00000000 sec

============ CHANNEL f1 ========
NUC1                      1H
P1                   10.10 usec
PL1                  -6.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF              300.1300060 MHz
WDW                       no
SSB                        0
LB                    0.00 Hz
GB                         0
PC                     1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                 11.000 ppm
F1                 3301.43 Hz
F2P                 -1.000 ppm
F2                -300.13 Hz
PPMCM              0.60000 ppm/cm
HZCM             180.07800 Hz/cm
```



BRUKER

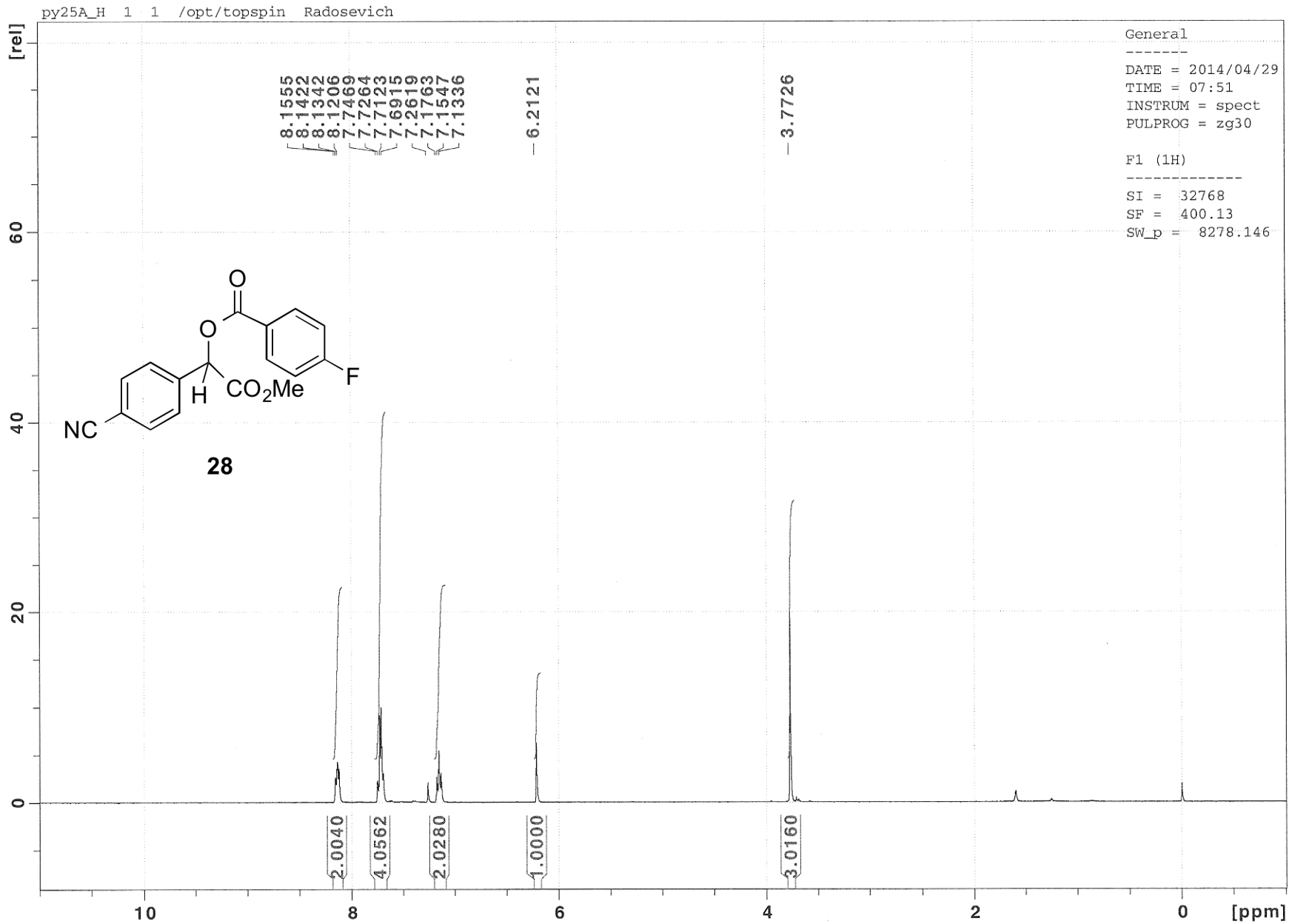
NAME	zw3696C
EXPNO	1
PROCNO	1
Date_	20140423
Time	17.50
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1577
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	8192
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec
========== CHANNEL f1 ==========	
NUC1	13C
P1	9.75 usec
PL1	0.00 dB
SFO1	75.4760200 MHz
========== CHANNEL f2 ==========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677407 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

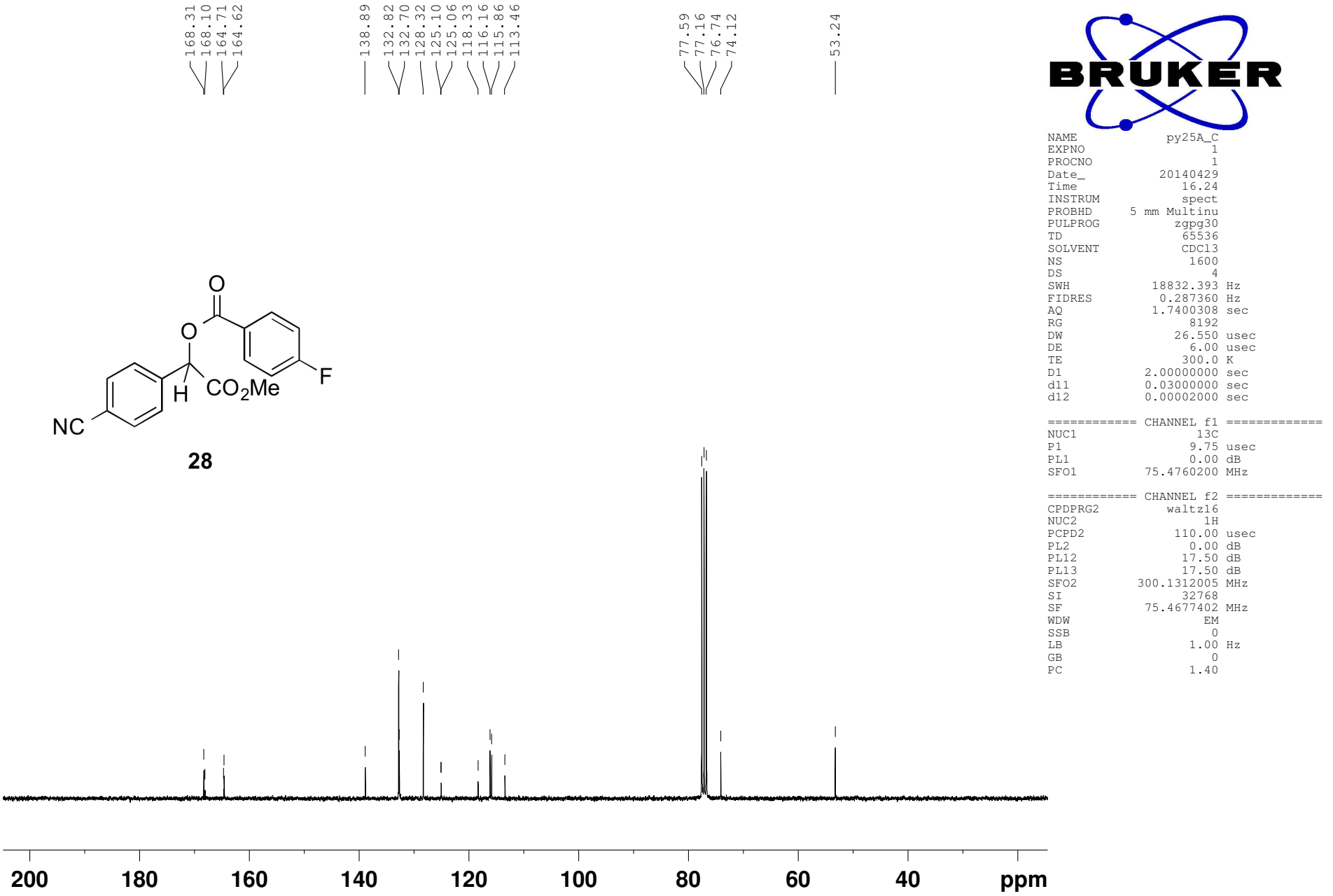

py25B_H 1 1 /opt/topspin Radosevich
[rel]
8.1396
8.1355
8.1260
8.1179
8.1043
7.5715
7.5506
7.4482
7.4273
7.2546
7.1522
7.1306
7.1093
6.1039
3.7521
General
DATE = 2014/04/29
TIME = 07:55
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
O
O
H
CO2Me
F
Br
27
2.0266
2.0440
2.1687
2.1568
1.0000
3.0526
[ppm]



BRUKER

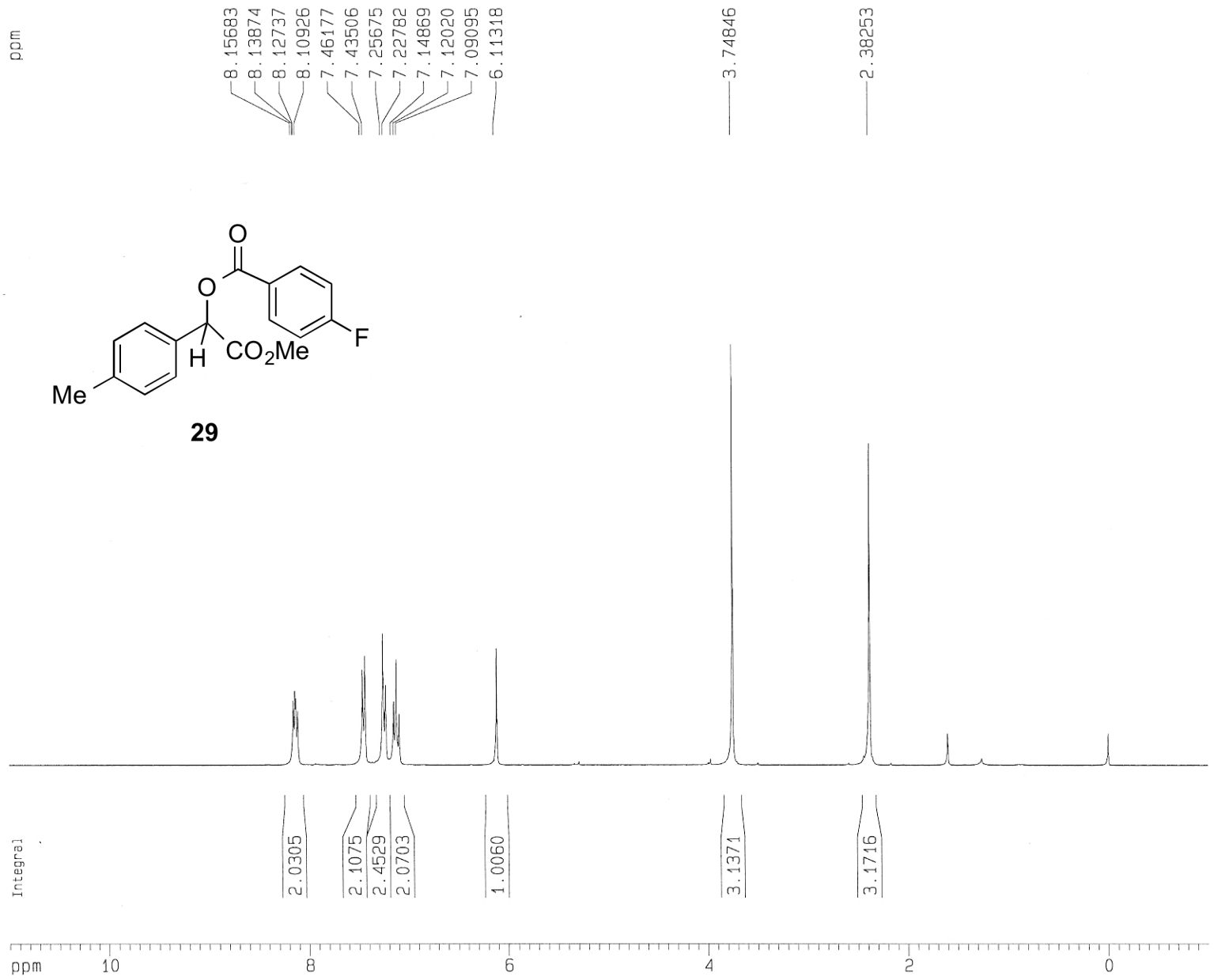
NAME	py25B_C
EXPNO	1
PROCNO	1
Date_	20140429
Time	20.15
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1700
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	13004
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec
========== CHANNEL f1 ==========	
NUC1	13C
P1	9.75 usec
PL1	0.00 dB
SFO1	75.4760200 MHz
========== CHANNEL f2 ==========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677397 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


py25A_H 1 1 /opt/topspin Radosevich
[rel]
8.1555
8.1422
8.1342
8.1206
7.7469
7.7264
7.7123
7.6915
7.2619
7.1763
7.1547
7.1336
6.2121
3.7726
General
DATE = 2014/04/29
TIME = 07:51
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
O
O
H
CO2Me
F
NC
28
2.0040
4.0562
2.0280
1.0000
3.0160
[ppm]



BRUKER

NAME	py25A_C	
EXPNO	1	
PROCNO	1	
Date_	20140429	
Time	16.24	
INSTRUM	spect	
PROBHD	5 mm Multinu	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1600	
DS	4	
SWH	18832.393	Hz
FIDRES	0.287360	Hz
AQ	1.7400308	sec
RG	8192	
DW	26.550	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
d11	0.03000000	sec
d12	0.00002000	sec
========== CHANNEL f1 ==========		
NUC1	13C	
P1	9.75	usec
PL1	0.00	dB
SFO1	75.4760200	MHz
========== CHANNEL f2 ==========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	110.00	usec
PL2	0.00	dB
PL12	17.50	dB
PL13	17.50	dB
SFO2	300.1312005	MHz
SI	32768	
SF	75.4677402	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	



Current Data Parameters

NAME	zw3713H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20140502
Time	16.02
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	362
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

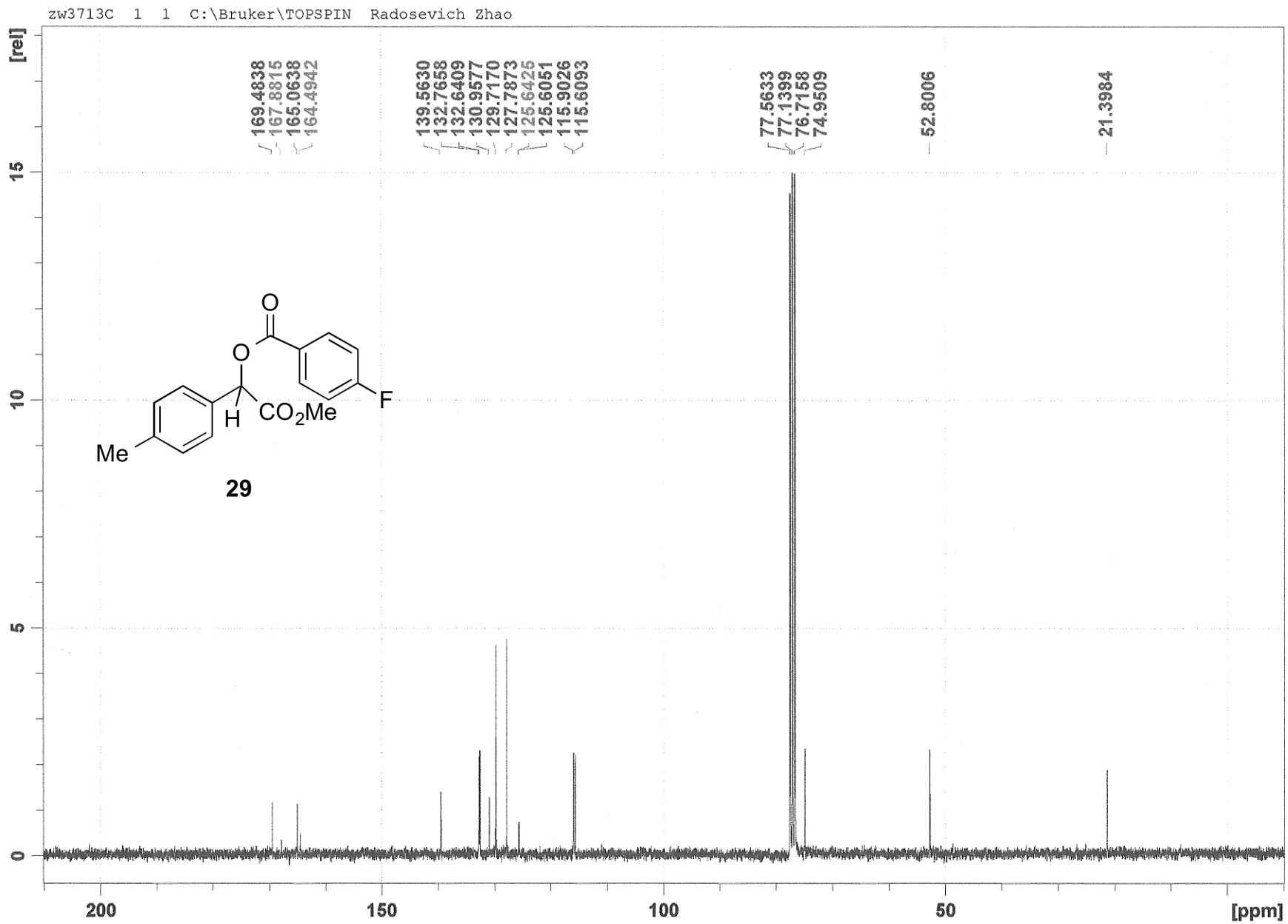
NUC1	1H
P1	12.10 usec
PL1	0.00 dB
SFO1	299.8718518 MHz

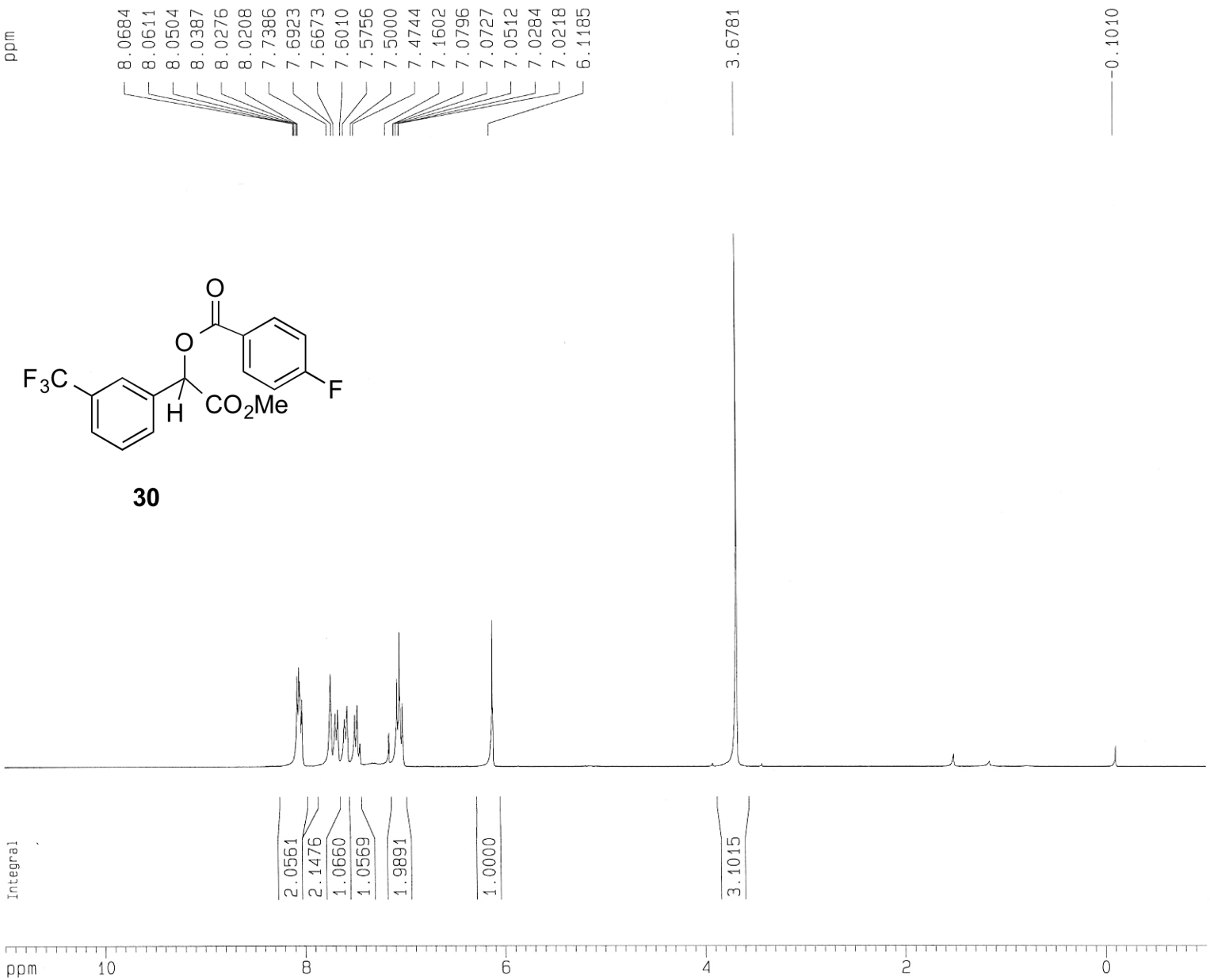
F2 - Processing parameters

SI	32768
SF	299.8700104 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

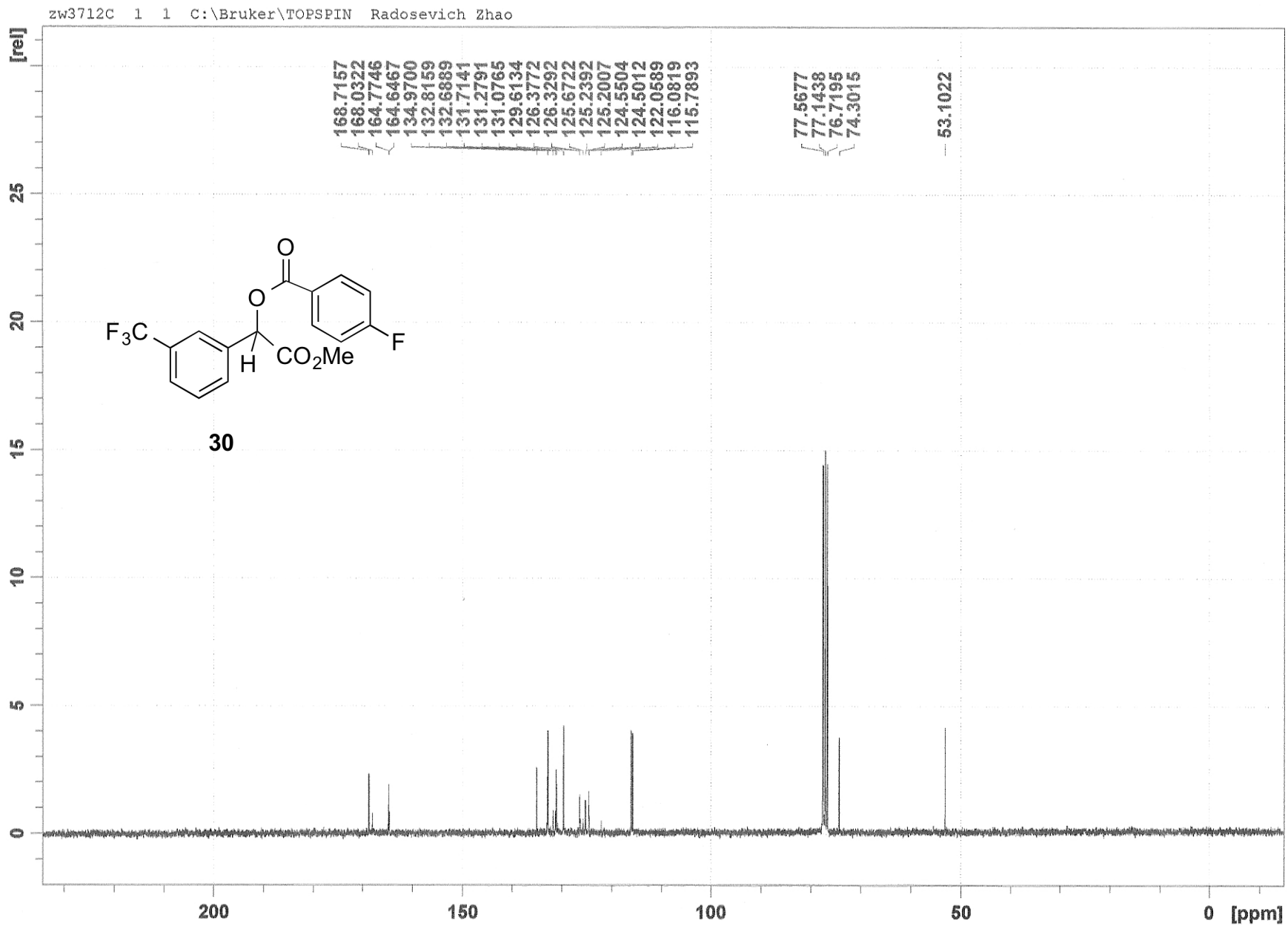
CX	20.00 cm
F1P	11.000 ppm
F1	3298.57 Hz
F2P	-1.000 ppm
F2	-299.87 Hz
PPMCM	0.60000 ppm/cm
HZCM	179.92201 Hz/cm

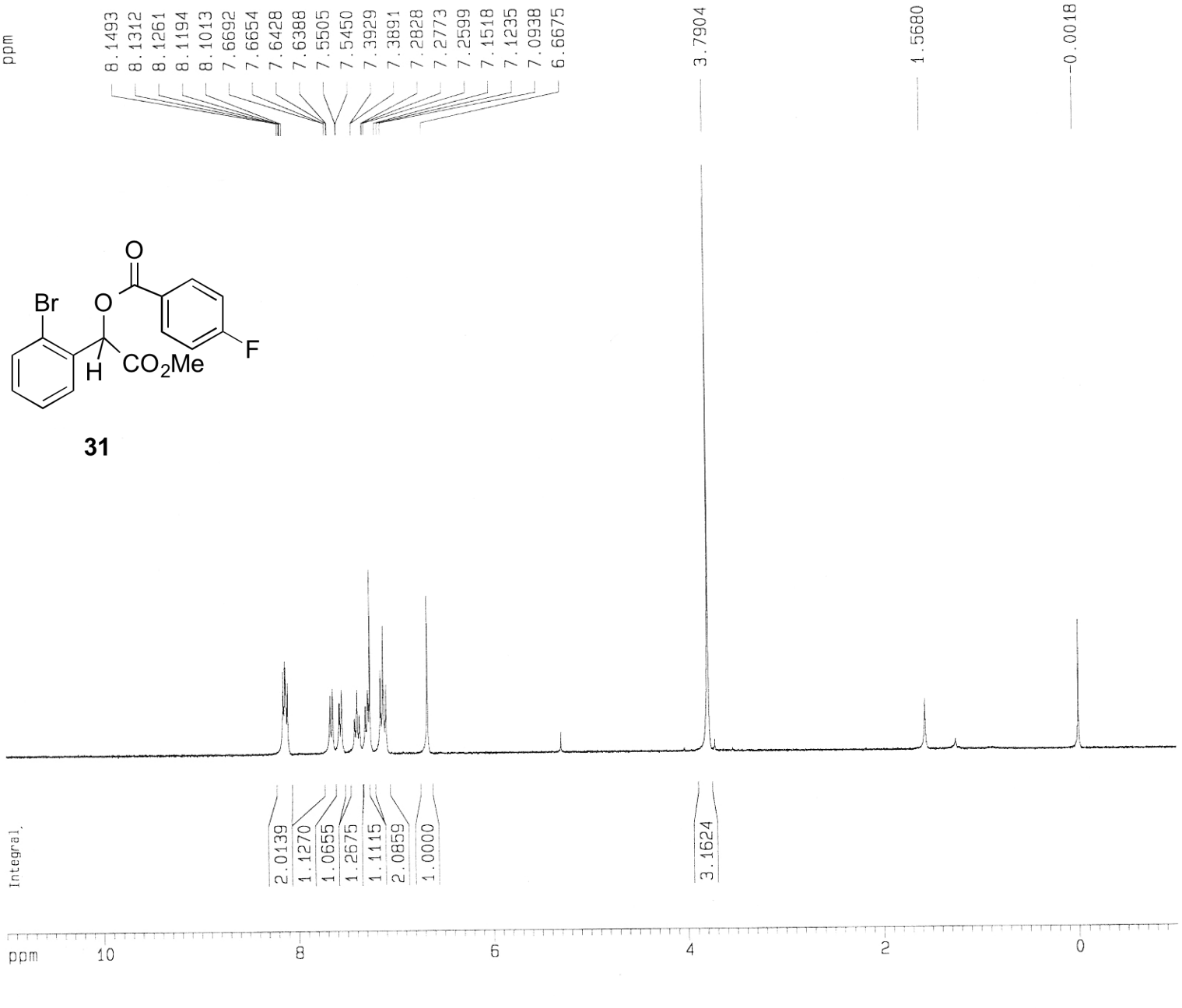

zw3713C 1 1 C:\Bruker\TOPSPIN Radosevich Zhao
[rel]
169.4838
167.8815
165.0638
164.4942
139.5630
132.7658
132.6409
130.9577
129.7170
127.7873
125.6425
125.6051
115.9026
115.6093
77.5633
77.1399
76.7158
74.9509
52.8006
21.3984
O
O
H
CO2Me
F
Me
29
200
150
100
50
[ppm]



30

```
Current Data Parameters
NAME                  zw3712H
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140502
Time                  14.11
INSTRUM               spect
PROBHD     5 mm QNP 1H/1
PULPROG               zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                      256
DW                 81.000 usec
DE                  6.00 usec
TE                 300.0 K
D1                1.00000000 sec

============ CHANNEL f1 ========
NUC1                      1H
P1                  12.10 usec
PL1                  0.00 dB
SFO1           299.8718518 MHz

F2 - Processing parameters
SI                        32768
SF             299.8700393 MHz
WDW                       no
SSB                        0
LB                    0.00 Hz
GB                         0
PC                     1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                 11.000 ppm
F1                3298.57 Hz
F2P                -1.000 ppm
F2                -299.87 Hz
PPMCM             0.60000 ppm/cm
HZCM            179.92201 Hz/cm
```


zw3712C 1 1 C:\Bruker\TOPSPIN Radosevich Zhao
[rel]
168.7157
168.0322
164.7746
164.6467
134.9700
132.8159
132.6889
131.7141
131.2791
131.0765
129.6134
126.3772
126.3292
125.6722
125.2392
125.2007
124.5504
124.5012
122.0589
116.0819
115.7893
77.5677
77.1438
76.7195
74.3015
53.1022
O
O
F3C
H
CO2Me
F
30
0
5
10
15
20
25
200
150
100
50
0
[ppm]



31

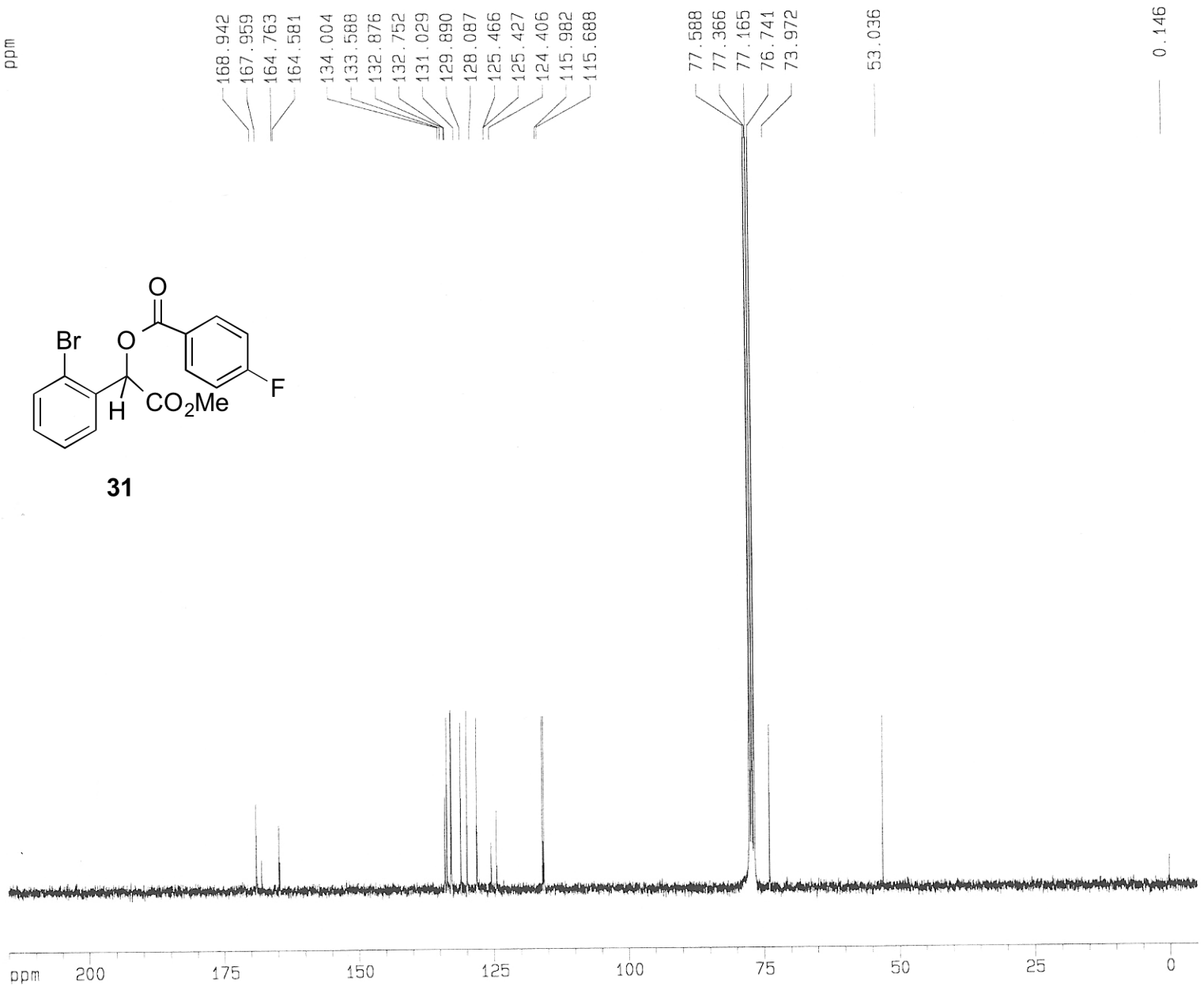
Current Data Parameters
NAME ~~zw3714H~~ zw3715
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140502
Time 17.12
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 512
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -6.00 dB
SF01 300.1318534 MHz

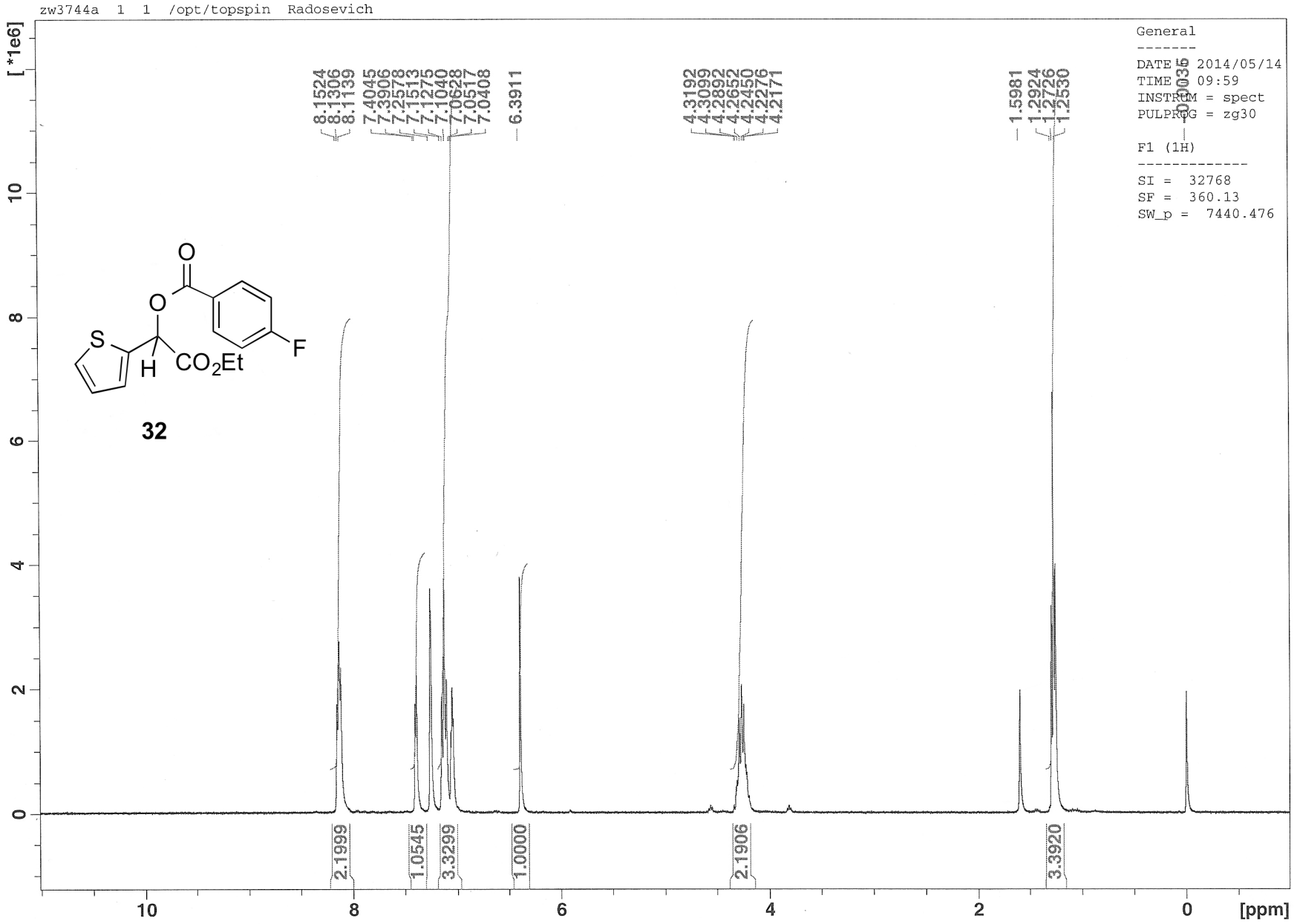
F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

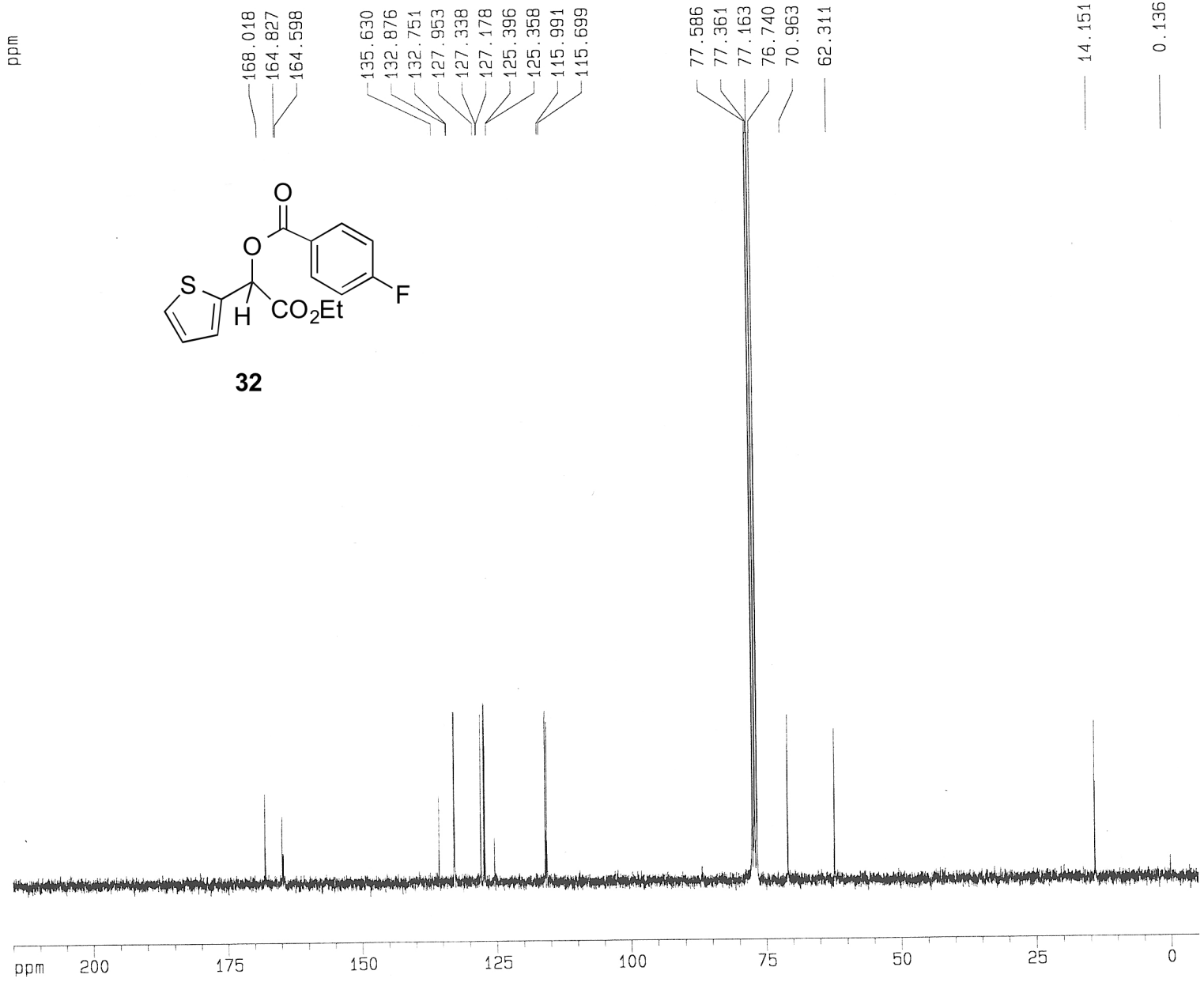
1D NMR plot parameters
CX 20.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.07800 Hz/cm



31

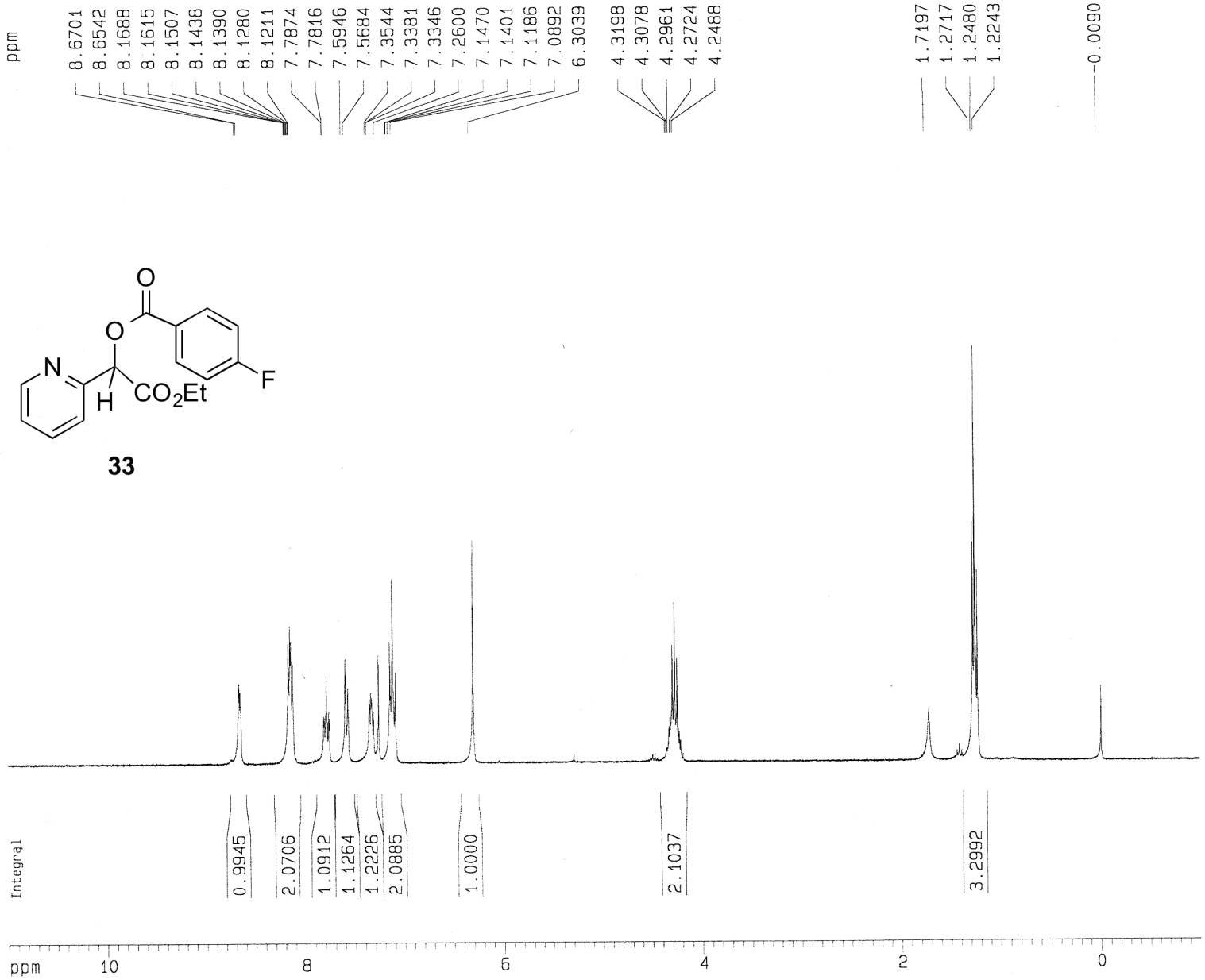
```
Current Data Parameters
NAME                  ~~zw37146~~ zw375
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140503
Time                       3.40
INSTRUM                   spect
PROBHD          5 mm Multinu
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                       10000
DS                           4
SWH                18832.393 Hz
FIDRES              0.287360 Hz
AQ                  1.7400308 sec
RG                       16384
DW                     26.550 usec
DE                      6.00 usec
TE                     300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 ============
NUC1                      13C
P1                      9.75 usec
PL1                     0.00 dB
SFO1               75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 110.00 usec
PL2                     0.00 dB
PL12                   17.50 dB
PL13                   17.50 dB
SFO2              300.1312005 MHz

F2 - Processing parameters
SI                       32768
SF                 75.4677388 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
F1P                  215.000 ppm
F1                 16225.56 Hz
F2P                   -5.000 ppm
F2                  -377.34 Hz
PPMCM                11.00000 ppm/cm
HZCM                830.14508 Hz/cm
```


zw3744a 1 1 /opt/topspin Radosevich
[*1e6]
8.1524
8.1306
8.1139
7.4045
7.3906
7.2573
7.1513
7.1275
7.1040
7.0628
7.0517
7.0408
6.3911
4.3192
4.3099
4.2892
4.2652
4.2450
4.2276
4.2171
1.5981
1.2924
1.2726
1.2530
0.0035
General
DATE = 2014/05/14
TIME = 09:59
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
O
O
S
H
CO2Et
F
32
2.1999
1.0545
3.3299
1.0000
2.1906
3.3920
[ppm]



32

```
Current Data Parameters
NAME                  zw3744aC
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20140514
Time                     13.17
INSTRUM                  spect
PROBHD       5 mm Multinu
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1600
DS                           4
SWH                  18832.393 Hz
FIDRES                0.287360 Hz
AG                    1.7400308 sec
RG                       16384
DW                      26.550 usec
DE                       6.00 usec
TE                      300.0 K
D1                    2.00000000 sec
d11                   0.03000000 sec
d12                   0.00002000 sec

============ CHANNEL f1 ============
NUC1                       13C
P1                        9.75 usec
PL1                       0.00 dB
SFO1                75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   110.00 usec
PL2                       0.00 dB
PL12                     17.50 dB
PL13                     17.50 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4677394 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                     215.000 ppm
F1                    16225.57 Hz
F2P                      -5.000 ppm
F2                     -377.34 Hz
PPMCM                 11.00000 ppm/cm
HZCM                830.14514 Hz/cm
```



Current Data Parameters

NAME	zw3726H
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20140514
Time	13.24
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	406.4
DW	81.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

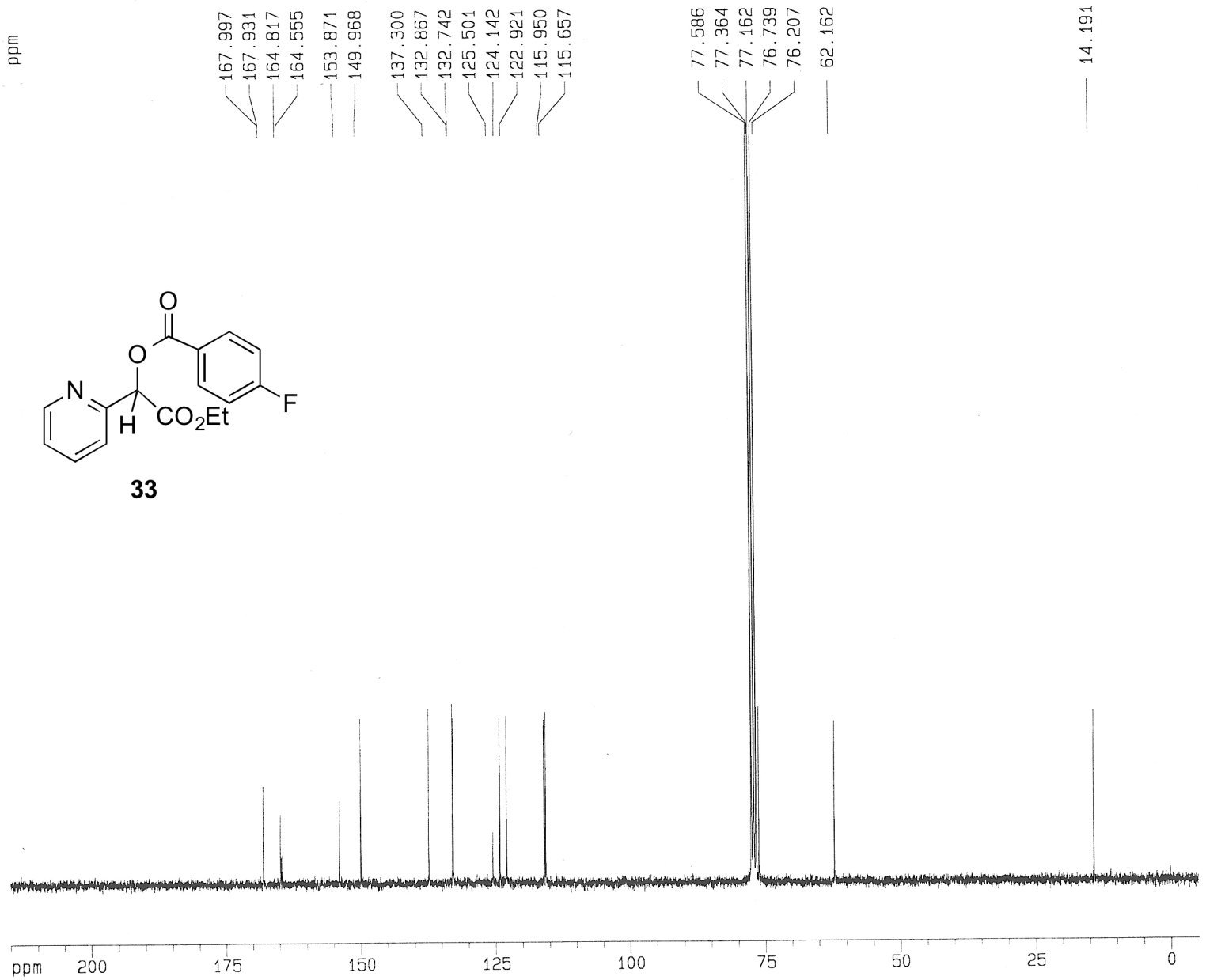
NUC1	1H
P1	10.10 usec
PL1	-6.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300058 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	3301.43 Hz
F2P	-1.000 ppm
F2	-300.13 Hz
PPMCM	0.60000 ppm/cm
HZCM	180.07800 Hz/cm



33

Current Data Parameters

NAME	zw3726C
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20140514
Time	22.29
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3200
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	8192
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	9.75 usec
PL1	0.00 dB
SFO1	75.4760200 MHz

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

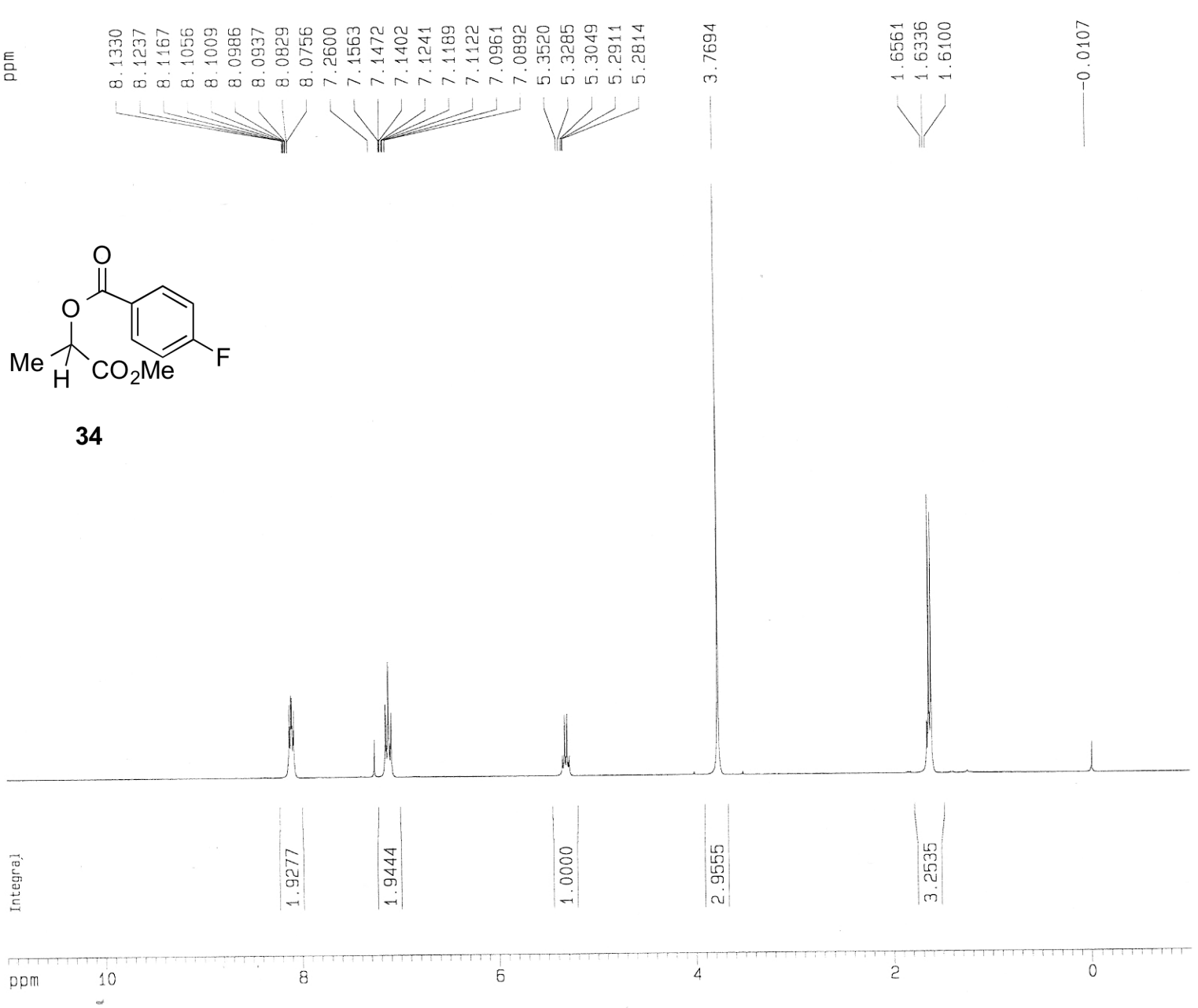
CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677399 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

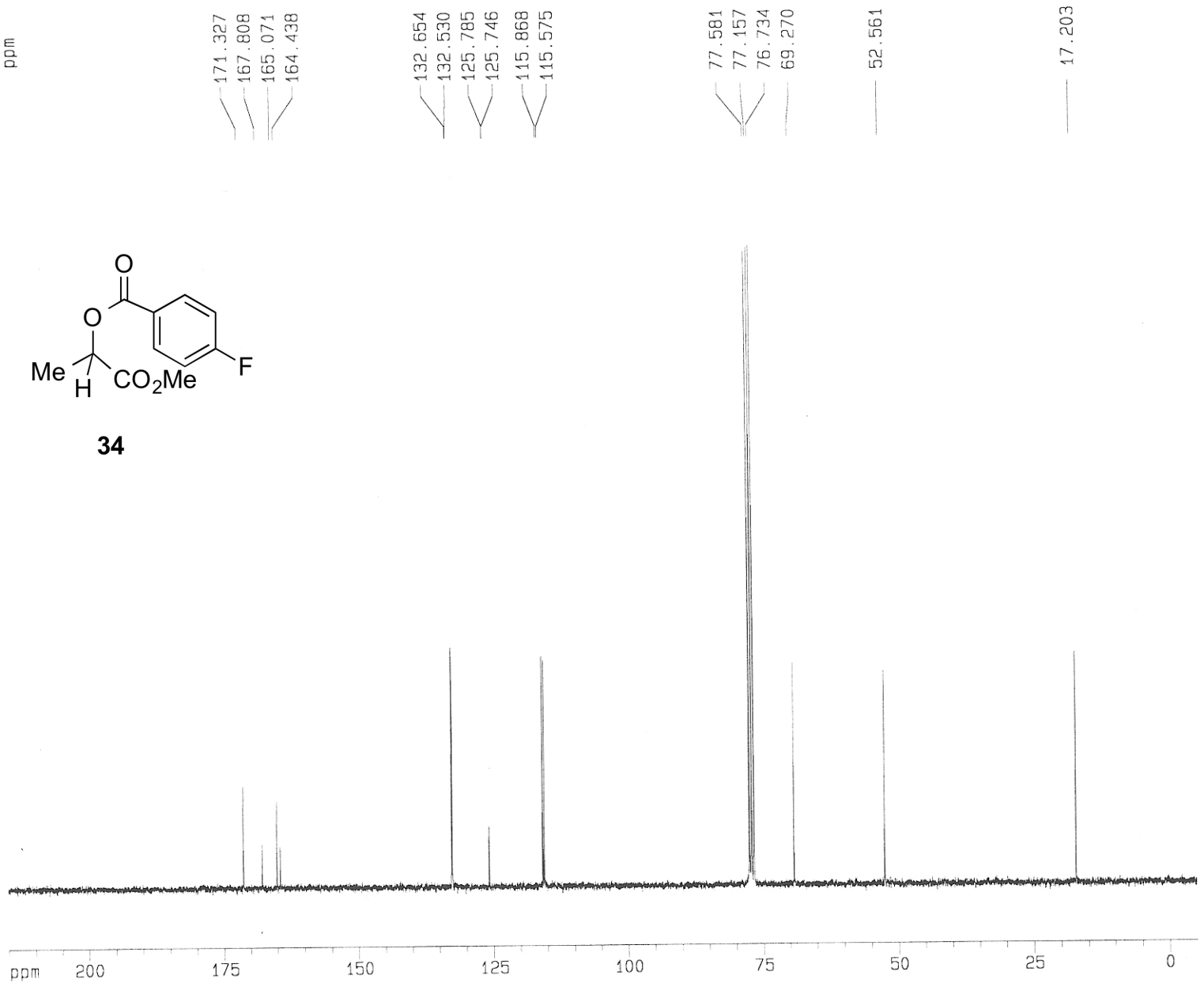
1D NMR plot parameters

CX	20.00 cm
F1P	215.000 ppm
F1	16225.57 Hz
F2P	-5.000 ppm
F2	-377.34 Hz
PPMCM	11.00000 ppm/cm
HZCM	830.14514 Hz/cm



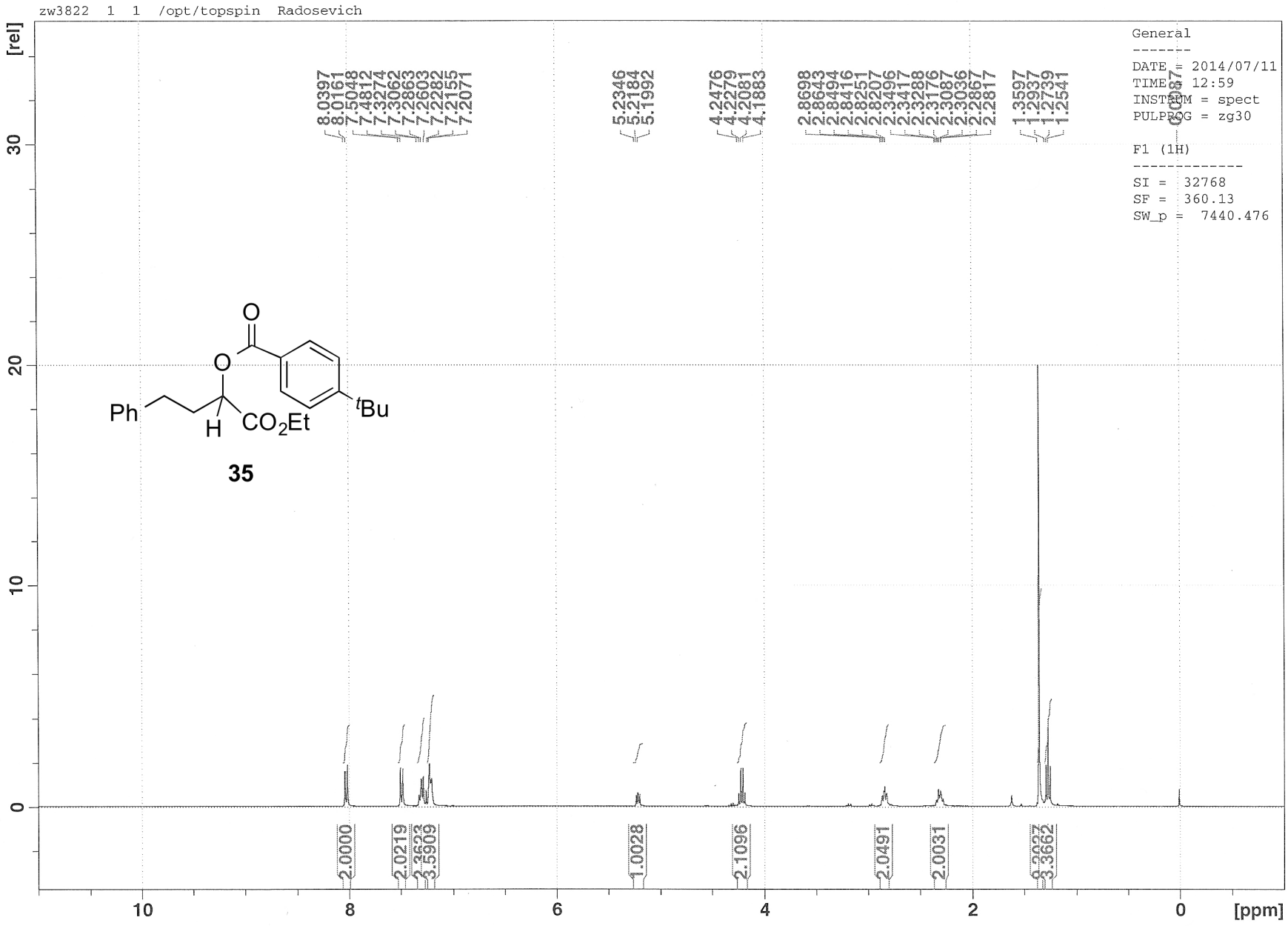
34

```
Current Data Parameters
NAME                  zw3740
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20140512
Time                  15.36
INSTRUM               spect
PROBHD      5 mm Multinu
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                   322.5
DW                  81.000 usec
DE                   6.00 usec
TE                  300.0 K
D1               1.00000000 sec

============ CHANNEL f1 ========
NUC1                    1H
P1                   10.10 usec
PL1                  -6.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300060 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                  11.000 ppm
F1                  3301.43 Hz
F2P                  -1.000 ppm
F2                  -300.13 Hz
PPMCM              0.60000 ppm/cm
HZCM             180.07800 Hz/cm
```



34

```
Current Data Parameters
NAME                  zw3740
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20140512
Time                  15.39
INSTRUM               spect
PROBHD     5 mm Multinu
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1800
DS                        4
SWH               18832.393 Hz
FIDRES             0.287360 Hz
AG                 1.7400308 sec
RG                    16384
DW                   26.550 usec
DE                    6.00 usec
TE                   300.0 K
D1               2.00000000 sec
d11              0.03000000 sec
d12              0.00002000 sec

============ CHANNEL f1 ============
NUC1                    13C
P1                    9.75 usec
PL1                   0.00 dB
SFO1             75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2             waltz16
NUC2                    1H
PCPD2                110.00 usec
PL2                   0.00 dB
PL12                 17.50 dB
PL13                 17.50 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                     32768
SF               75.4677405 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                215.000 ppm
F1                16225.57 Hz
F2P                 -5.000 ppm
F2                 -377.34 Hz
PPMCM             11.00000 ppm/cm
HZCM             830.14514 Hz/cm
```


zw3822 1 1 /opt/topspin Radosevich
[rel]
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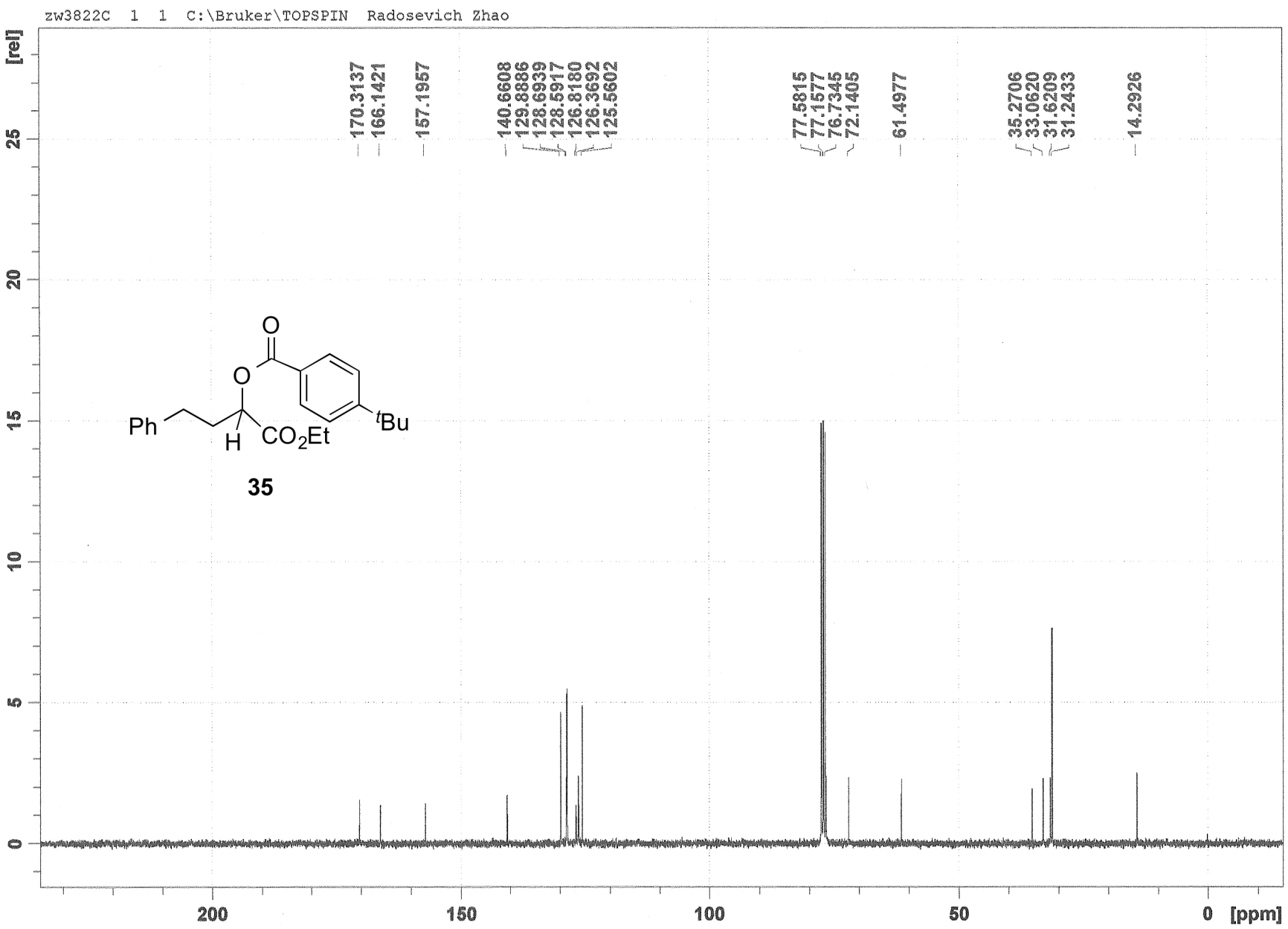
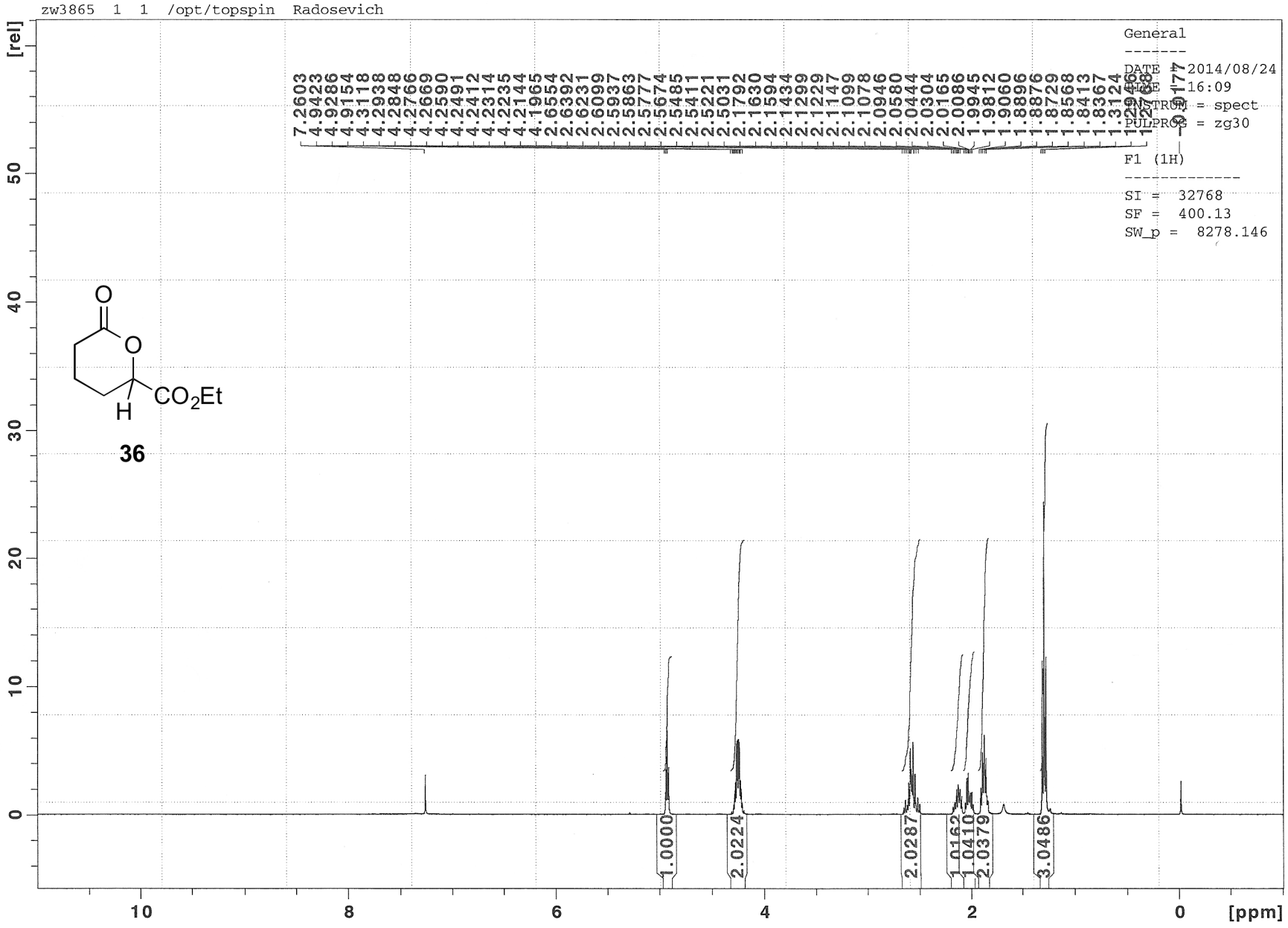
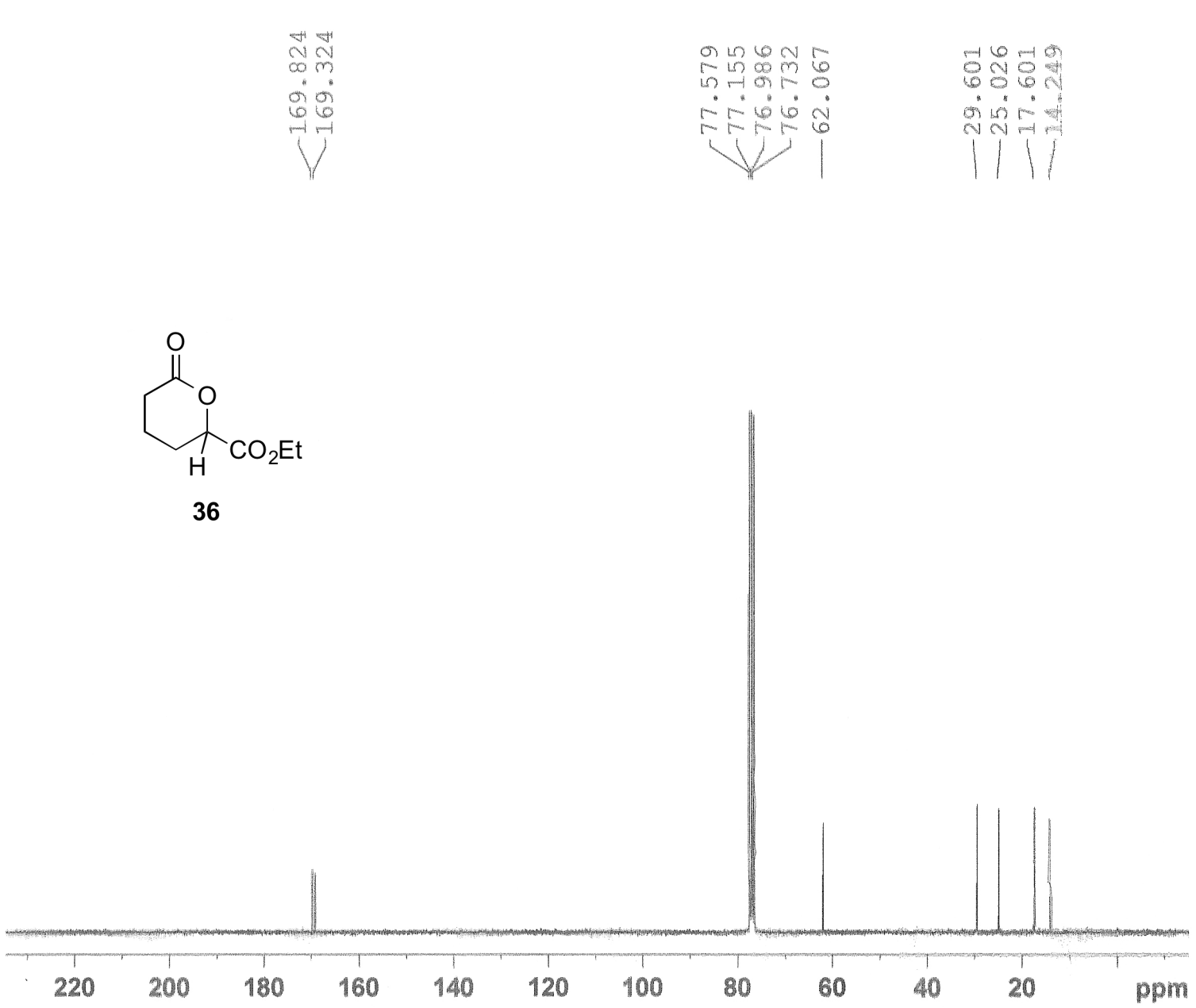
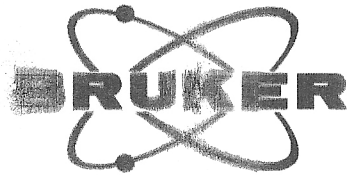
20
10
0
8.0397
8.0161
7.5048
7.4812
7.3274
7.3066
7.2863
7.2603
7.2282
7.2155
7.2071
5.2346
5.2184
5.1992
4.2476
4.2279
4.2081
4.1883
2.8698
2.8643
2.8494
2.8416
2.8251
2.8207
2.3496
2.3417
2.3288
2.3176
2.3087
2.3036
2.2867
2.2817
1.3597
1.2937
1.2739
1.2541
General
DATE = 2014/07/11
TIME = 12:59
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
O
O
Ph
H
CO2Et
tBu
35
2.0000
2.0219
2.2623
3.5909
1.0028
2.1096
2.0491
2.0031
9.3027
3.3662
10
8
6
4
2
0
[ppm]


zw3822C 1 1 C:\Bruker\TOPSPIN Radosevich Zhao
[rel]
25
20
15
10
5
0
170.3137
166.1421
157.1957
140.6608
129.8886
128.6939
128.5917
126.8180
126.3692
125.5602
77.5815
77.1577
76.7345
72.1405
61.4977
35.2706
33.0620
31.6209
31.2433
14.2926
O
O
Ph
H
CO2Et
tBu
35
200
150
100
50
0
[ppm]

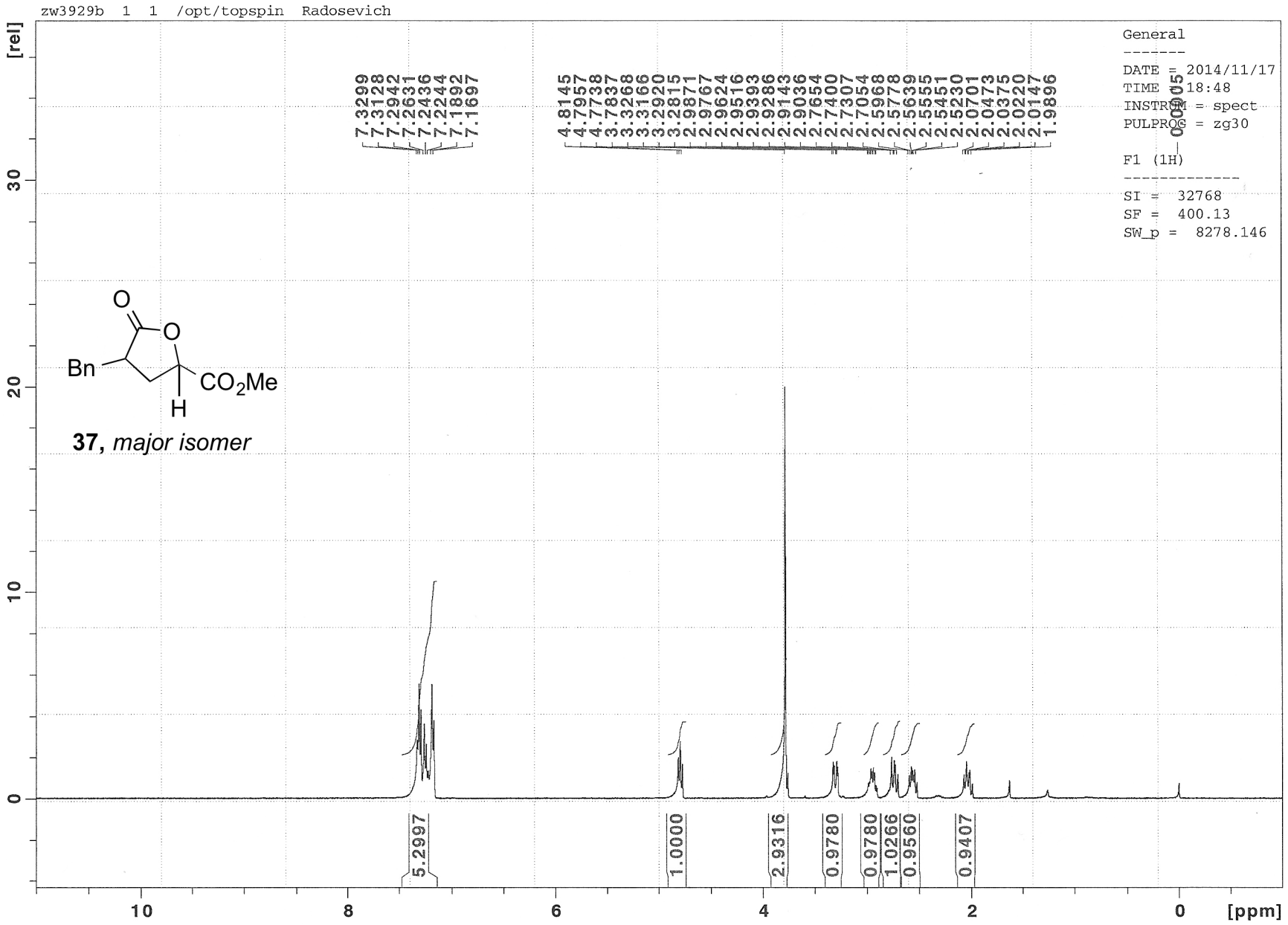

zw3865 1 1 /opt/topspin Radosevich
[rel]
[ppm]
General
DATE 2014/08/24
TIME 16:09
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
1.0000
2.0224
2.0287
1.0162
1.0410
2.0379
3.0486
O
O
H
CO2Et

36

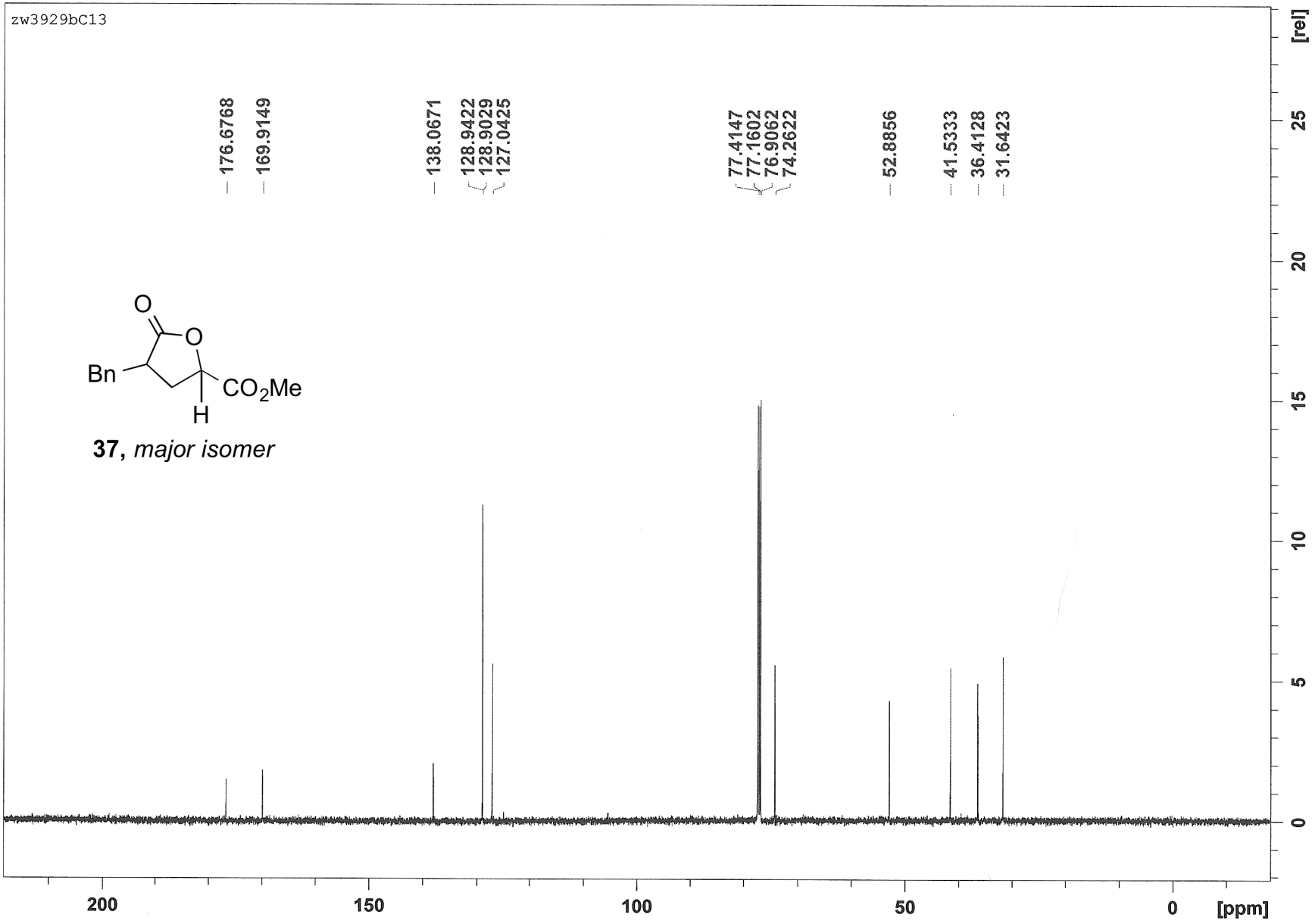




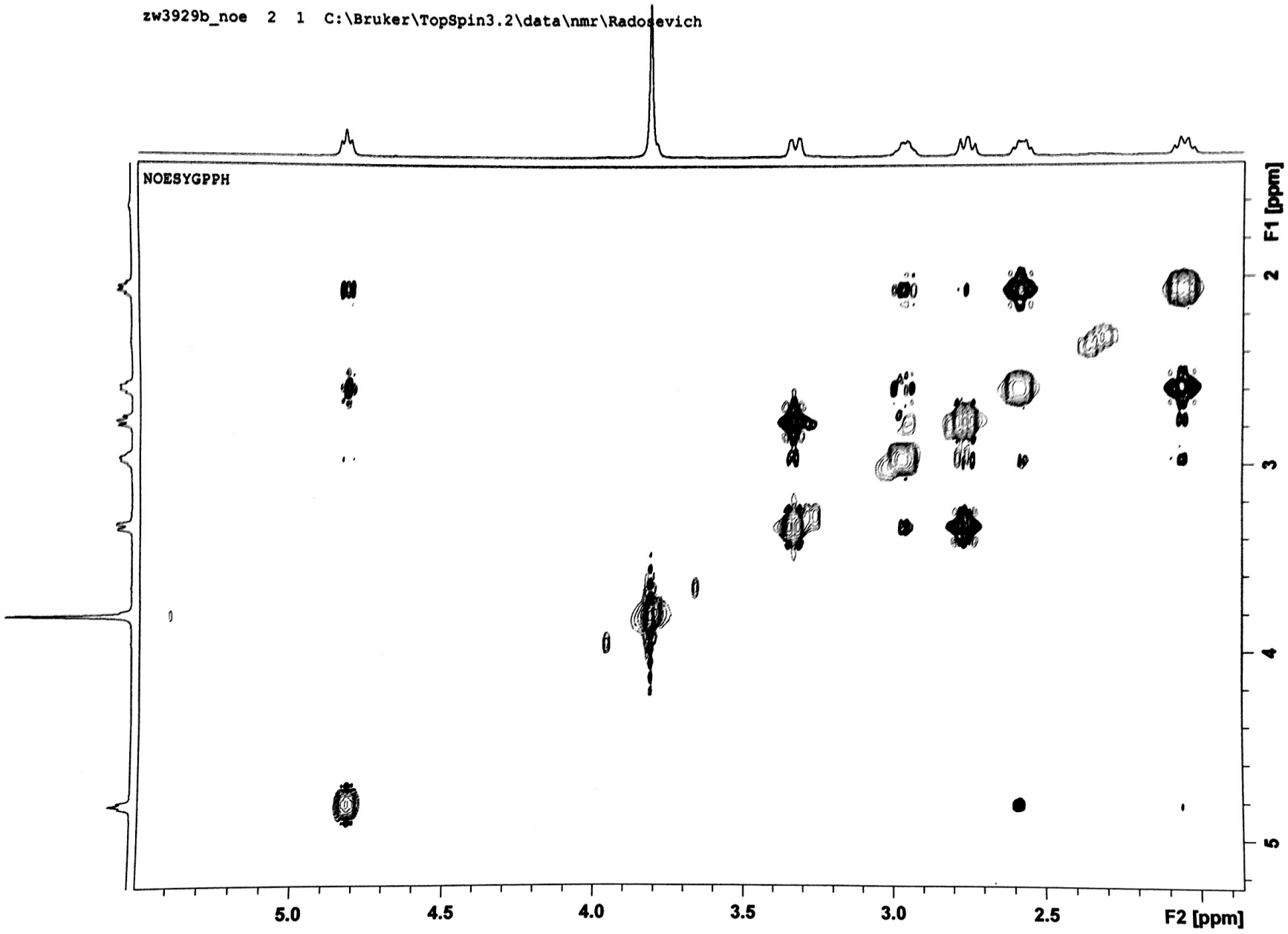
NAME	zw3865c	
EXPNO	1	
PROCNO	1	
Date_	20140824	
Time	17.03	
INSTRUM	qbeat	
PROBHD	5 mm Multinu	
PULPROG	zgdbg30	
TD	65536	
SOLVENT	CDCl3	
NS	5000	
DS	4	
SWH	18832.393	Hz
FIDRES	0.287360	Hz
AQ	1.7400308	sec
RG	8192	
DW	26.550	usec
DE	6.00	usec
TE	300.0	K
D1	2.00000000	sec
D11	0.03000000	sec
D12	0.00002000	sec
==========	CHANNEL f1	==========
NUC1	13C	
P1	9.75	usec
PL1	0.00	dB
SFO1	75.4760200	MHz
==========	CHANNEL f2	==========
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	110.00	usec
PL2	0.00	dB
PL12	17.50	dB
PL13	17.50	dB
SFO2	300.1312005	MHz
SI	32768	
SF	75.4677408	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

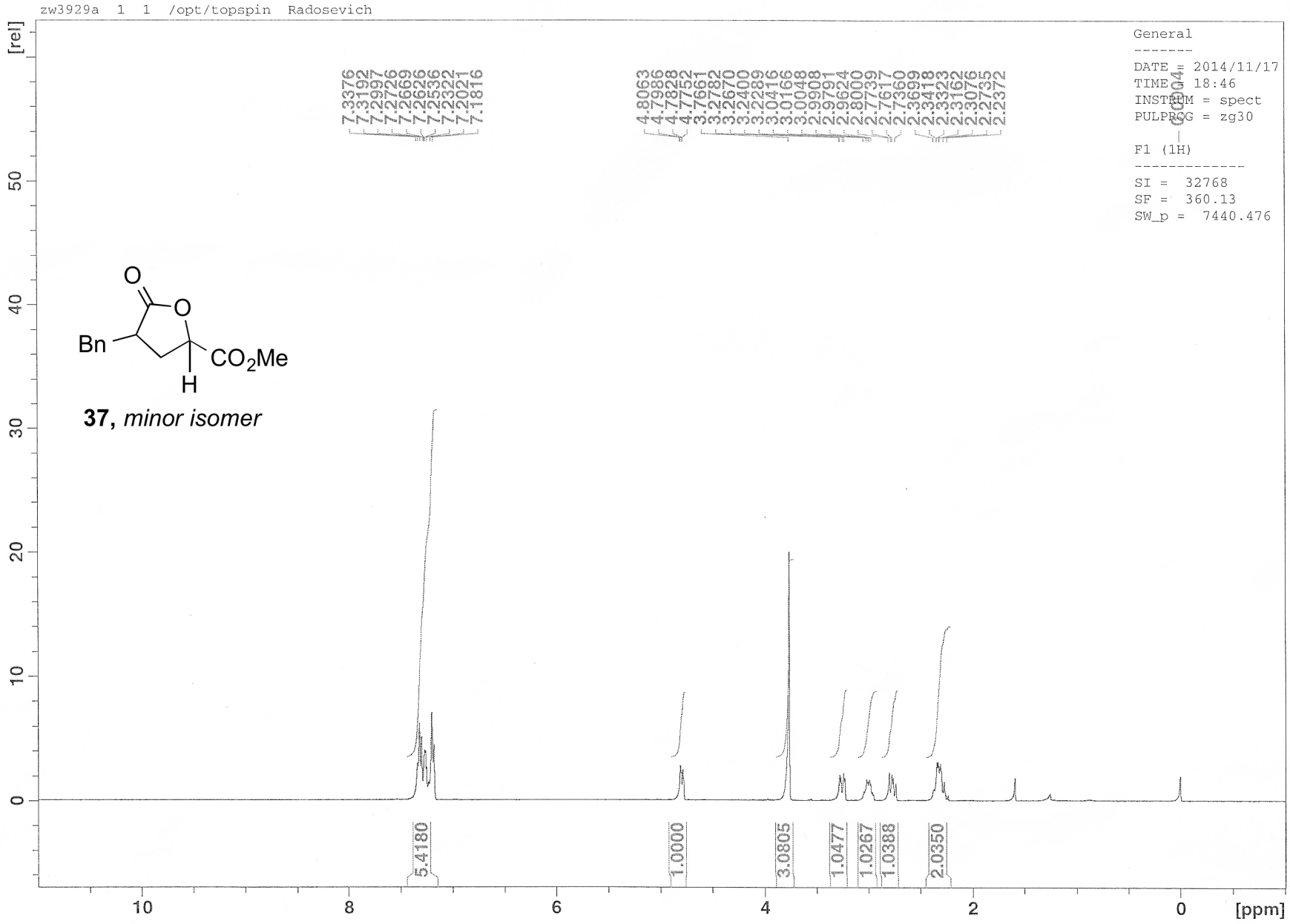

zw3929b 1 1 /opt/topspin Radosevich
[rel]
7.3299
7.3128
7.2941
7.2631
7.2436
7.2244
7.1892
7.1697
4.8145
4.7957
4.7738
3.7837
3.3268
3.3166
3.2920
3.2815
2.9871
2.9767
2.9624
2.9516
2.9393
2.9286
2.9143
2.9036
2.7654
2.7400
2.7307
2.7054
2.5968
2.5778
2.5639
2.5555
2.5451
2.5230
2.0701
2.0473
2.0375
2.0220
2.0147
1.9896
General
DATE = 2014/11/17
TIME = 18:48
INSTRM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
O
O
Bn
CO2Me
H
37, major isomer
5.2997
1.0000
2.9316
0.9780
0.9780
1.0266
0.9560
0.9407
[ppm]

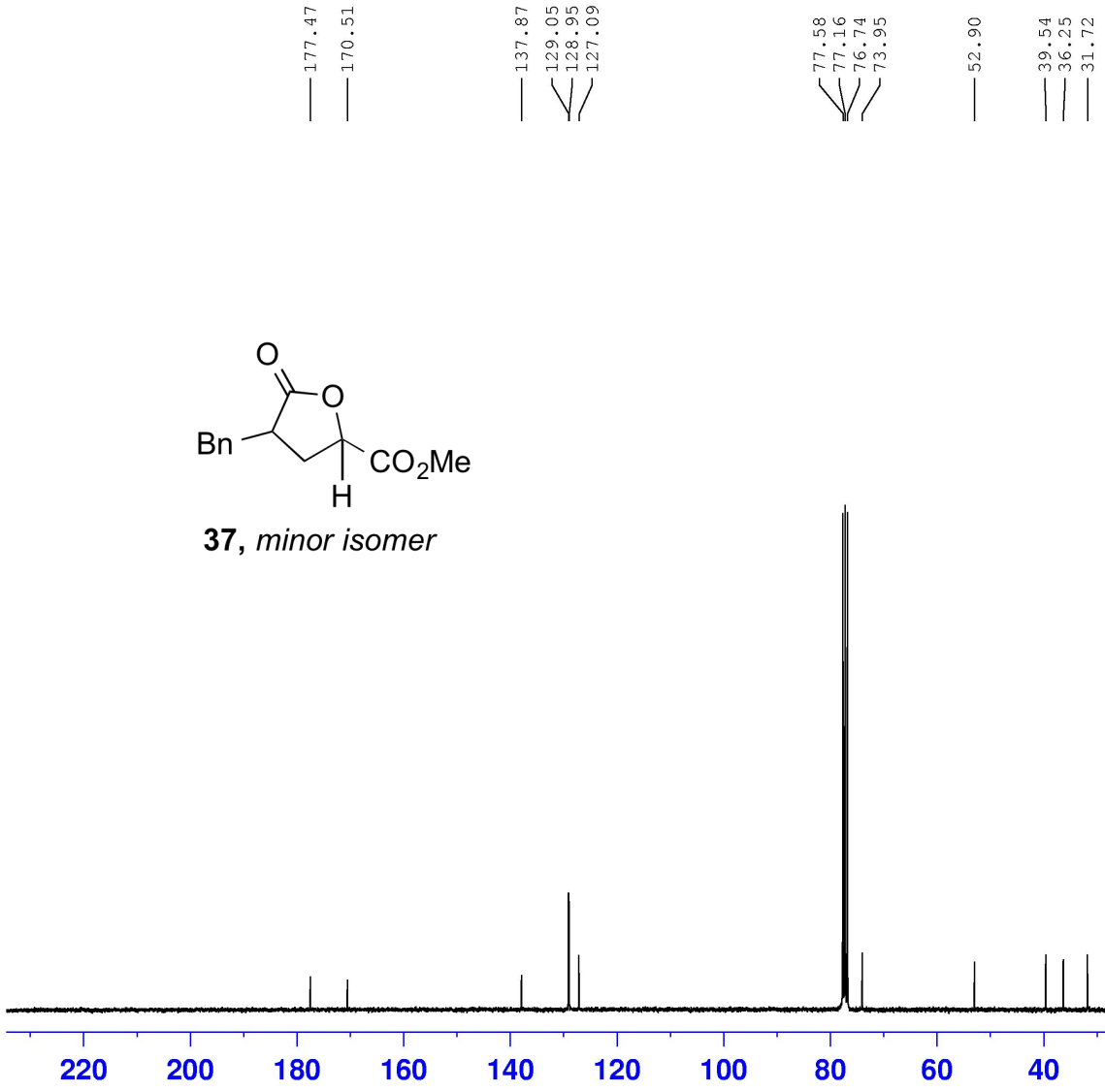
zw3929bC13 1 1 C:\Bruker\TopSpin3.2\data\nmr\Radosevich



37, *major isomer*


zw3929b_noe 2 1 C:\Bruker\TopSpin3.2\data\nmr\Radosevich
NOESYGPPH
F1 [ppm]
2
3
4
5
5.0
4.5
4.0
3.5
3.0
2.5
F2 [ppm]


zw3929a 1 1 /opt/topspin Radosevich
[rel]
7.3376
7.3192
7.2997
7.2726
7.2669
7.2626
7.2536
7.2322
7.2021
7.1816
4.8063
4.7986
4.7828
4.7752
4.7661
3.2782
3.2670
3.2400
3.2289
3.0416
3.0166
3.0048
2.9908
2.9791
2.9624
2.8000
2.7739
2.7617
2.7360
2.3699
2.3418
2.3323
2.3162
2.3076
2.2735
2.2372
General
DATE = 2014/11/17
TIME = 18:46
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476
O
O
Bn
CO2Me
H
37, minor isomer
5.4180
1.0000
3.0805
1.0477
1.0267
1.0388
2.0350
[ppm]



37, *minor isomer*

NAME	zw3929a
EXPNO	2
PROCNO	1
Date_	20141118
Time	6.19
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	10000
DS	4
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	16384
DW	26.550 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec
==========	CHANNEL f1 ==========
NUC1	13C
P1	9.75 usec
PL1	0.00 dB
SFO1	75.4760200 MHz
==========	CHANNEL f2 ==========
CPDPRG2	waltz16
NUC2	1H
PCPD2	110.00 usec
PL2	0.00 dB
PL12	17.50 dB
PL13	17.50 dB
SFO2	300.1312005 MHz
SI	32768
SF	75.4677395 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40