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Supplementary Information

For

Vanadyl Species-Catalyzed, Complementary β-Oxidative Carbonylations of Styrene Derivatives with Aldehydes

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Supplementary Information

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General. ^{1}H-NMR and ^{13}C-NMR were recorded Jeol JVM-EX400 (400 MHz ^{1}H, 100 MHz ^{13}C) or Varian UNITY INOVA-500 (400 MHz ^{1}H, 100 MHz ^{13}C) spectrometers in deuterochloroform with chloroform as an internal reference unless otherwise stated. Chemical shifts are reported in ppm (δ), coupling constants, *J*, are reported in Hz. Electrospray ionization (ESI) mass spectra were recorded with data reported in the form m/e (intensity relative to base = 100%). Analytical TLC was performed on Merck silica gel plates with F-254 indicator. Visualization was accomplished with UV light or with phosphomolybdic acid (PMA) and KMnO4 staining agents. Column (flash) chromatography was performed by using 40-63 μm silica gel. Toluene was dried over Na with benzophenone-ketyl intermediate under nitrogen atmosphere and distilled before use. CH_3CN was dried over CaH_2 and distilled before use. $VO(acac)_2$, $VO(SO_4)$, $Fe(acac)_2$ were purchased from Aldrich Chemicals. Oxovanadium (IV) dibenzoylmethanate ($VO(dbm)_2$); oxovanadium (IV) dipivaloylmethanate ($VO(tmhd)_2$); oxovanadium (IV) di-1,1,1,5,5,5-hexafluoroacetylacetonate ($VO(hfacac)_2$) were prepared according to the literature reports.[1]

General procedure for perparation of vanadyl catalyst ($VOCl_2$-$(H_2O)_x$ or $VO(OTf)_2$)[2]

In a flame-dried, 50-mL, two-necked, round-bottomed flask was placed 2.5 mmol vanadyl sulfate-$VOSO_4$-$5H_2O$ followed by addition of anhydrous MeOH (2.5 mL). To the above solution, a solution of 2.5 mmol (1 equiv) of $BaCl_2$ (or $Ba(OTf)_2$) in MeOH (2.5 mL) was slowly added at ambient temperature. After having been stirred

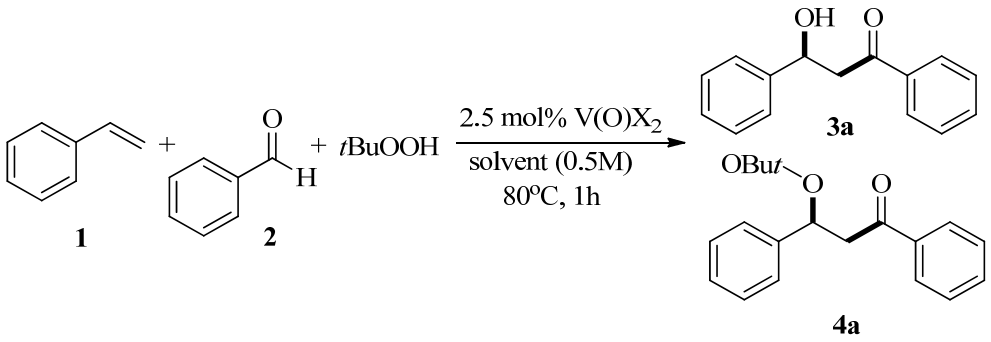
[1] a) S. Ideda, A. Yamamoto, S. Kurita, K. Takahashi, T. Watanabe, *Inorg. Chem.* **1966**, *5*, 611; b)

Johnson, D. A.; Waugh, A. B. *Polyhedron* **1983**, *2*, p. 1323

for 30 min, the reaction mixture became turbid with copious amount of barium sulfate precipitation. The mixture was filtered through a short plug of Celite.The filtrate was evaporated to give faint blue solid which was further dried at 120 ^{o}C for 4 hours invacuo. It can be stored at ambient temperature for several weeks in dry cabinet and can be used directly. The VOX_2 series of compounds (brand name as Clip-all® series, US patent # 6,541,659 B1, 2003) is now available directly from the institution (e-mail: ctchen@mx.nthu.edu.tw).

[2] a) C.-T. Chen, S. Bettigeri, S.-S. Weng, V. D. Pawar, Y.-H. Lin, C.-Y. Liu, W.-Z. Lee, *J. Org. Chem.* **2007**, *72*, 8175; b) N. B. Barhate, C.-T. Chen, *Org. Lett.* **2002**, *4*, 2529; g) S.-W. Hon, C.-H. Li, J.-H. Kuo, N. B. Barhate, Y,-H. Liu, Y. Wang, C.-T. Chen, *Org. Lett.* **2001**, *3*, 869

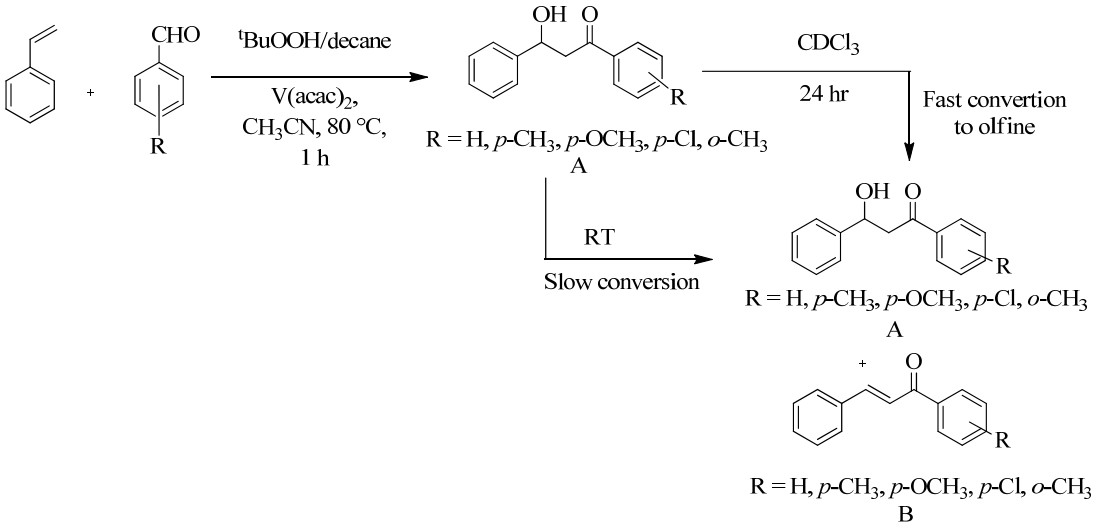
Catalyst screening for β-hydroxylation-carbonylationation or β-peroxidation-carbonylationation of styrene.: Table S1[a].

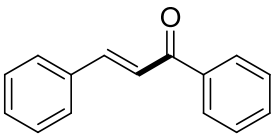
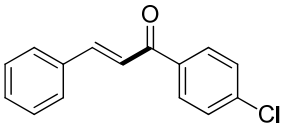
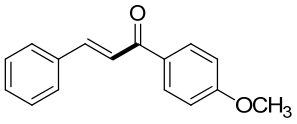
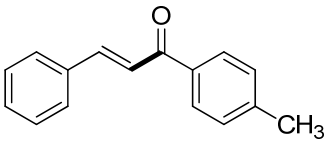
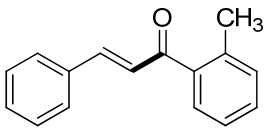


Entry	Catalyst	Solvent	Yielde of **3a** (%)[b]	Yield of **4a** (%)[b]
1.	MoO_2Cl_2	CH_3CN	trace	13
2.	$HfO(Cl)_2$	CH_3CN	trace	-[c]
3.	$TiO(OTf)_2$	CH_3CN	-[c]	-[c]
4.	CrO_2Cl_2	CH_3CN	-[c]	18
5.	$Fe(acac)_2$	CH_3CN	trace	16
6.	$VO(Cl)_2$	Toluene	6	53
7.	$VO(Cl)_2$	THF	trace	13
8.	$VO(Cl)_2$	$ClCH_2CH_2Cl$	<5	42

[a] Reaction condition: **1** (1 mmol), **2** (5 mmol), oxometallic species (0.025 mmol), *t*-BuOOH (3 mmol, 5 M in decane), acetonitrile (2 mL), 80 °C, under N_2. [b] Yields of isolated products are given. [c] No desired product was found.

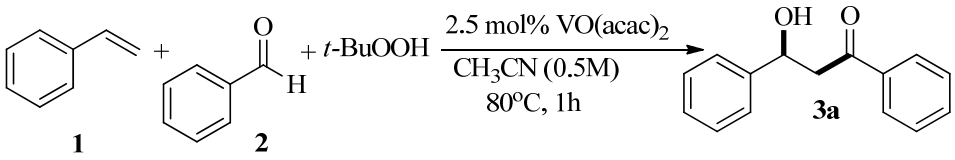
Dehydration of β-hydroxy carbonyl compounds: Table S2.



Entry	Conv. (%)[a]	α,β-unsaturated krtone (B)
1	29	
2	none	
3	34	
4	30	
5	31	

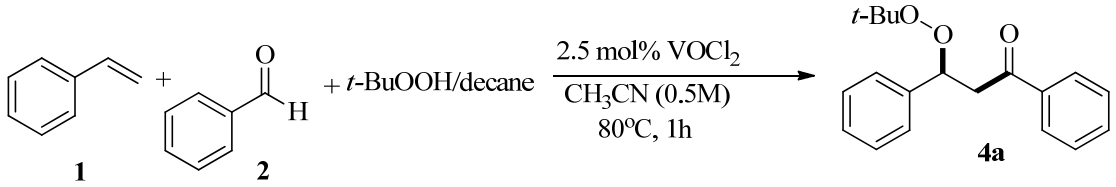
[a] Determined by ^{1}H NMR analysis.

Representative procedure for β-hydroxylation-carbonylation (procedure I):



To a mixture of benzaldehyde (510 μL, 5.0 mmol) and $VO(acac)_2$ (6.6 mg, 0.025 mmol, 2.5 mol%) in CH_3CN (1 mL), styrene (115 μL, 1 mmol) was added via syringe under nitrogen at room temperature. The reaction flask was transfer to a preheated oil spot (80 ^oC) and the 70% aqueous *tert*-Butyl hydrogenperoxide (600 μL,3 mmol) in CH_3CN (1mL) was added dropwise to the reaction mixture over 30 min via addition funnel under nitrogen at 80 ^oC and stirred for additional 30 min at 80 ^oC. The solvent and excess amount of benzaldehyde was removed in vacuum, and the crude product was purified using flash column chromatography on silica gel with ethyl acetate/hexane (1:10) as eluent to afford the β-hydroxyketone **3a** (190 mg, 84% yield).

Representative procedure for β-peroxidation-carbonylation (procedure II)

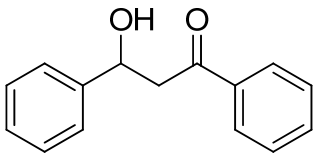


To a mixture of benzaldehyde (510 μL, 5.0 mmol) and $VOCl_2$ (3.4 mg, 0.025 mmol, 2.5 mol%) in anhydrous CH_3CN (1 mL), styrene (115μL, 1 mmol) was added via syringe under nitrogen at room temperature. The reaction flask was transfer to a

preheated oil spot (80 ^{o}C). *tert*-Butyl hydrogenperoxide (600 μL, 1.5 mmol, 5-6 M in Decane) was diluted with anhydrous CH_3CN (1 mL) and added dropwise to the reaction mixture over 30 min via addition funnel under nitrogen at 80 ^{o}C and stirred for additional 30 min at 80 ^{o}C. The solvent and excess amount of benzaldehyde was removed in vacuum, and the crude product was purified using flash column chromatography on silica gel with ethyl acetate/hexane (1:50) as eluent to afford the β-peroxyketones **4a** (238 mg, 80% yield).

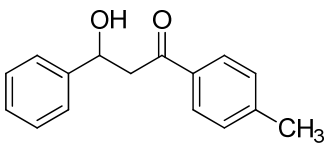
Analytical data for β-hydroxyketones 3a-v

3-Hydroxy-1, 3-diphenyl-propan-1-one (3a)[3]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.15). The title compound was obtained as a colorless oil. ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.96 (d, *J*=7.5Hz, 2H), 7.60 (t, *J*=7.5Hz, 1H), 7.49-7.45 (m, 3H), 7.39 (t, *J*=7.5Hz, 2H), 7.31 (d, *J*=7.5Hz, 1H), 5.36 (dd, *J*=7.5, 4Hz, 1H), 3.67 (br, s, 1H), 3.43-3.35 (m, 2H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 200.1, 142.9, 136.5, 133.6, 128.6, 128.5, 128.1, 127.6, 125.7, 70.0, 47.3; M.W. ($C_{15}H_{14}O_2$) 226.2; MS (EI, 20 eV): 226 (M^+, 52), 209 (M-H_2O, 6), 119 (100), 107 (52).

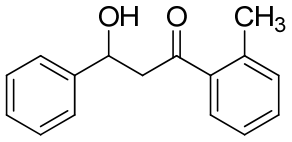
3-Hydroxy-3-phenyl-3-*p*-tolyl-propan-1-one (3b)[3]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.16). The title compound was obtained as a colorless oil. ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.85 (d, *J*=8.0Hz, 2H), 7.46-7.30 (m, 5H), 7.25 (t, *J*=8Hz, 2H), 5.34 (t, *J*=6.0 Hz, 1H), 3.77 (br, s, 1H), 3.34 (d, *J*=6.0, 2H), 2.42

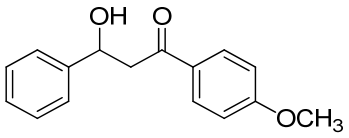
(s, 3H). ^{13}C NMR ($CDCl_3$, 100MHz): δ 199.8, 144.5, 143.0, 134.1, 129.3, 128.5, 128.2, 127.5, 125.7, 70.0, 47.1, 21.6; M.W. ($C_{16}H_{16}O_2$) 240.3; MS (EI, 20 eV): 240 (M^+, 48), 223 (M-H_2O, 7), 133 (100), 107 (83).

3-Hydroxy-3-phenyl-3-*o*-tolyl-propan-1-one (3c)[3]



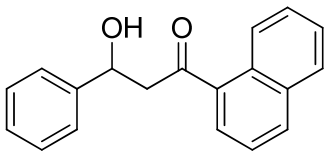
Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.16). The title compound was obtained as a colorless oil. ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.64 (d, *J*=7.6Hz, 2H), 7.42-7.25 (m, 7H), 5.32 (dd, *J*=8.4, 3.6 Hz, 1H), 3.84-3.26 (m, 2H), 2.54 (s, 3H). ^{13}C NMR ($CDCl_3$, 100MHz): δ 203.8, 143.0, 138.6, 137.1, 132.1, 131.8, 128.9, 128.5, 127.6, 125.7, 70.3, 49.9, 21.5; M.W. ($C_{16}H_{16}O_2$) 240.3; MS (EI, 20 eV): 240 (M^+, 30), 223 (M-H_2O, 11), 133 (100), 107 (67).

3-Hydroxy-1-(4-methoxy-phenyl)-3-phenyl-propan-1-one (3d)[3]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.13). The title compound was obtained as a colorless oil. ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.93 (d, *J*=9.2Hz, 2H), 7.45-7.27 (m, 5H), 6.92 (d, *J*=8.8 Hz, 2H), 5.32 (dd, *J*=7.2, 4.8 Hz, 1H), 3.86 (s, 3H), 3.32-3.30 (m, 2H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 198.7, 163.9, 143.0, 130.4, 129.6, 128.5, 127.5, 125.7, 113.8, 70.1, 55.4, 46.8.; M.W. ($C_{16}H_{16}O_3$) 256.3; MS (EI, 20 eV): 256 (M^+, 38), 239 (M-H_2O, 18), 149 (100), 107 (78).

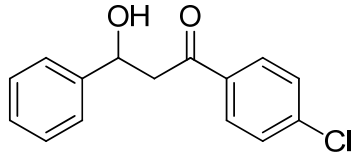
3-Hydroxy-1-naphthalen-2-yl-3-phenyl-propan-1-one (3e)[3]



Follow the representative procedure I, isolated by column

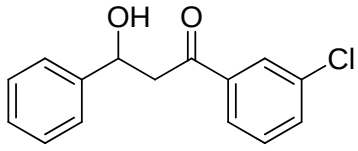
chromatography (EtOAc/hexanes = 1:20, R_f = 0.10); ^{1}H NMR ($CDCl_3$, 400MHz): δ 8.71 (d, *J*=8.4 Hz, 1H), 8.01 (d, *J*=8.0 Hz, 1H), 7.90-7.87 (m, 2H), 7.62 (t, *J*=6.8 Hz, 1H), 7.54 (t, *J*=7.2 Hz, 1H),7.49-7.29 (m, 7H), 5.43 (dd, *J*=9.2, 3.2 Hz, 1H), 3.66 (br, s, 1H), 3.52 (dd, *J*= 17.2, 8.8 Hz, 1H), 3.44 (dd, *J*= 17.6, 3.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 203.9, 142.9, 135.1, 133.9, 133.3, 130.0, 128.53, 128.45, 128.3, 128.2, 127.7, 126.5, 125.73, 125.68, 124.3, 70.5, 50.4; M.W. ($C_{19}H_{16}O_2$) 276.3; MS (EI, 20 eV): 276 (M^+, 62), 261 (M-H_2O, 13), 155 (100), 121 (72).

1-(*p*-Chlorophenyl)-3-hydroxy-3-phenylpropan-1-one (3f)[3]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.17); ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.88 (d, *J*=8.8Hz, 2H), 7.43-7.37 (m, 7H), 5.32 (d, *J*=8.4 Hz, 1H), 3.55 (br, s, 1H), 3.37 (dd, *J*= 17.6, 8.8 Hz, 1H), 3.29 (dd, *J*= 17.2, 3.2Hz, 1H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 198.7, 142.8, 140.1, 134.9, 129.5, 129.0, 128.6, 127.7, 125.7, 69.9, 47.4; M.W. ($C_{15}H_{13}ClO_2$) 260.7; MS (EI, 20 eV): 262 (M+2, 13), 260 (M^+, 42), 243 (M-H_2O, 7), 155 (32), 153 (100), 107 (92).

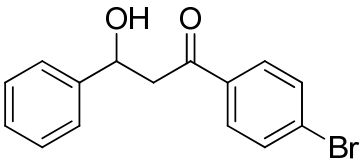
1-(*m*-Chlorophenyl)-3-hydroxy-3-phenylpropan-1-one (3g)[3]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.16) ; ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.92 (t, *J*=2 Hz, 1H), 7.81 (dt, *J*=8, 1.2 Hz, 1H), 7.92 (dq, *J*=8, 1.8 Hz, 1H),7.44-7.28 (m, 5H), 5.33 (dd, *J*=8.8, 3.2 Hz, 1H), 3.51 (br, s, 1H), 3.29 (dd, *J*= 17.6, 9.2 Hz, 1H), 3.29 (dd, *J*= 17.6, 3.2Hz, 1H).; ^{13}C NMR ($CDCl_3$, 100MHz): δ 198.6, 142.7, 138.0, 135.0, 133.4,

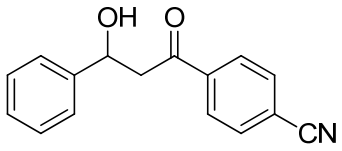
130.0, 128.6, 128.2, 127.7, 126.2, 125.7, 69.9, 47.5; M.W. ($C_{15}H_{13}ClO_2$) 260.7; MS (EI, 20 eV): 262 (M+2, 17), 260 (M^+, 50), 243 (M-H_2O, 6), 155 (29), 153 (100), 107 (87).

1-(*p*-Bromophenyl)-3-hydroxy-3-phenylpropan-1-one (3h)[3]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.17); ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.79 (d, *J*=8.8 Hz, 2H), 7.59 (d, *J*=8.8 Hz, 2H), 7.44-7.27 (m, 5H), 5.33 (dd, *J*=8.8, 3.2 Hz, 1H), 3.57 (br, s, 1H), 3.36 (dd, *J*= 17.6, 8.8 Hz, 1H), 3.27(dd, *J*= 17.6, 3.2Hz, 1H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 198.8, 142.8, 135.2, 131.9, 129.6, 128.8, 128.5, 127.7, 125.6, 69.9, 47.3; M.W. ($C_{15}H_{13}BrO_2$) 304.2; MS (EI, 20 eV): 306 (M+2, 54), 304 (M+ 2, 7), 287 (M-H_2O, 11), 200 (18), 198 (100), 107 (83).

1-(4-Cyanophenyl)-3-hydroxy-3-phenyl-1-propanone (3i)[4]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:5, R_f = 0.15); ^{1}H NMR ($CDCl_3$, 400MHz): δ 8.02 (d, *J*=8.4 Hz, 2H), 7.75 (d, *J*=8.4 Hz, 2H), 7.44-7.28 (m, 5H), 5.35 (dd, *J*=9.2, 2.8 Hz, 1H), 3.44 (dd, *J*= 17.6, 9.2 Hz, 1H), 3.29 (dd, *J*= 17.6, 2.8Hz, 1H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 198.4, 142.6, 139.5, 132.5, 128.6, 128.5, 128.3, 127.9, 125.6, 117.7, 116.6, 69.9, 47.8; M.W. ($C_{16}H_{13}NO_2$) 251.3; MS (EI, 20 eV): 251 (M^+, 76), 234 (M-H_2O, 20), 130 (100), 121 (92).

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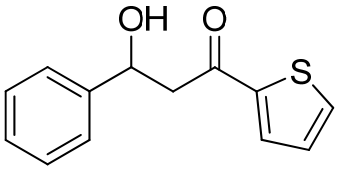
[3] a) H.-J. Xu, Y.-C. Liu, Y. Fu, Y.-D. Wu, *Org Lett.* **2006**, *8*, 3449; b) H.-J. Xu, X. Wan, Y.-Y.

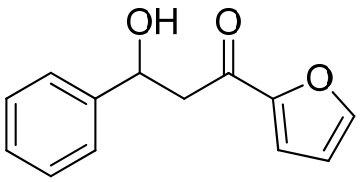
Shen, S. Xu,; Y.-S. Feng, *Org. Lett.* **2012**, *14*, 1210.

3-Hydroxy-3-phenyl-1-(thiophen-2-yl)propan-1-one (3j)[4]



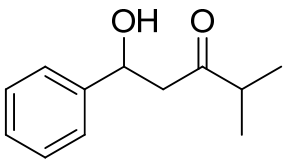
Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 3:7, R_f = 0.65); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (dd, *J* = 1.0, 3.8 Hz, 1H), 7.68 (dd, *J* = 1.2, 5.0 Hz, 1H), 7.45 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.33-7.29 (m, 1H), 7.13 (dd, *J* = 3.8, 5.0 Hz, 1H), 5.33 (dd, *J* = 4.8, 7.4 Hz, 1H), 3.33-3.30 (m, 2H); 2.64-2.46 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.7, 143.7, 142.7, 134.5, 132.7, 128.5, 128.2, 127.7, 125.7, 70.1, 47.9; HRMS(ESI) calcd for $C_{13}H_{12}O_2S$ (M^++Na): 255.0456; found: 255.0448.

1-(Furan-2-yl)-3-hydroxy-3-phenylpropan-1-one (3k)[4]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 3:7, R_f = 0.55).; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (dd, *J* = 0.8, 1.7 Hz, 1H), 7.43 (d, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.2 Hz, 2H), 7.31-7.27 (m, 1H), 7.23 (dd, *J* = 0.8, 3.6 Hz, 1H), 6.55 (dd, *J* = 1.7, 3.6 Hz, 1H), 5.31 (dd, *J* = 4.5, 7.9 Hz, 1H), 3.25 (d, *J* = 4.5 Hz, 1H), 3.24 (d, *J* = 1.2 Hz, 1H), 2.99-2.70 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.6, 152.3, 146.9, 142.7, 128.5, 127.7, 125.7, 118.0, 112.4, 70.0, 46.9; HRMS(ESI) calcd for $C_{13}H_{12}O_3$ (M^++Na): 239.0684; found: 239.0679.

1-Hydroxy-4-methyl-1-phenyl-3-pentanone (3l)[5]

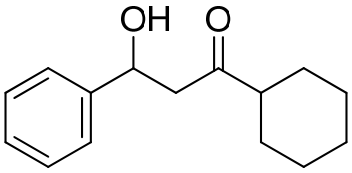


Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.15). The title compound was obtained as a colorless oil.

[4] E. Hasegawa, K. Ishiyama, T. Kato, T. Horaguchi, T. Shimizu, *J. Org. Chem.* **1992**, *57*, 5352.

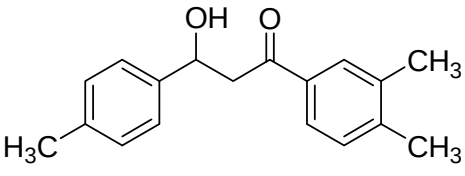
^{1}H NMR ($CDCl_3$, 400MHz): δ 7.39-7.25 (m, 5H), 5.15 (dd, *J*=8.8, 3.6 Hz, 1H), 3.53 (br, s, 1H), 2.89 (dd, *J*= 17.2, 8.4 Hz, 1H), 3.80 (dd, *J*= 17.2, 3.6 Hz, 1H), 5.15 (heptet, *J*=7.2 Hz, 1H), 2.36-2.21 (heptet, 1H) 1.86-1.64 (m, 5H), 1.37-1.16 (m, 5H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 215.2, 142.9, 128.5, 127.6, 125.6, 69.9, 48.7, 41.5, 17.8; M.W. ($C_{12}H_{16}O_2$) 192.2; MS (EI, 20 eV); 192 (M^+, 73), 177 (M-H_2O, 7), 107 (82), 85 (100).

1-Cyclohexyl-3-hydroxy-3-phenylpropan-1-one (3m)[6]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.14). The title compound was obtained as a colorless oil. ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.35-7.26 (m, 5H), 5.14 (dd, *J*=8, 4 Hz, 1H), 3.52 (br, s, 1H), 2.87 (dd, *J*= 17.6, 8.0 Hz, 1H), 3.80 (dd, *J*= 17.6, 4.0 Hz, 1H), 2.36-2.21 (m, 1H) 1.86-1.64 (m, 5H), 1.37-1.16 (m, 5H).; ^{13}C NMR ($CDCl_3$, 100MHz): δ 214.6, 142.9, 128.4, 128.2, 127.5, 125.5, 69.8, 51.3, 48.9, 28.08, 28.05, 25.7, 25.4; M.W. ($C_{15}H_{20}O_2$) 232.3; MS (EI, 20 eV): 232 (M^+, 38), 217 (M-H_2O, 40), 121 (78), 111 (100).

3-Hydroxy-1-(3, 4-dimethylphenyl)-3-p-tolylpropan-1-one (3n)[7]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.11). The title compound was obtained as a colorless oil.

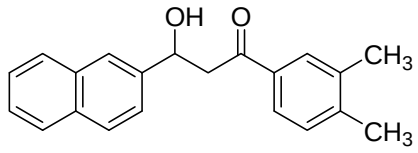
[5] a) Y. Kondo, H. Kudo, H. Naka, D. J. Eisler, F. Garcia, J. Haywood, M. McPartlin, J. V.

Morey, A. E. H. Wheatley, M. Uchiyama, *J. Am. Chem. Soc.* **2008** , 130, 16193; b) F. h Arikan, J. Li, D. Menche, S. Rudolph, *Org. Lett.* **2007**, *9*, 267.

[6] S. Kanemasa, Y. Norisue, H. Suga, O. Tsuge, *Bull. Chem. Soc. Jap.* **1988**, *61*, 3973.

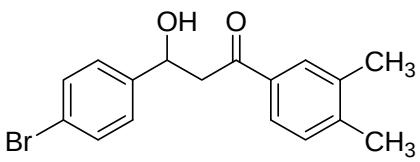
^{1}H NMR ($CDCl_3$, 400MHz): δ 7.72-7.67 (m, 2H), 7.34 (d, *J*=8.0 Hz, 2H), 7.22-7.18 (m, 3H), 5.30 (dd, *J*=6.8, 5.2Hz, 1H), 3.7 (br, s, 1H), 3.34-3.32 (m, 2H),2.36 (s, 3H), 2.32 (s, 3H), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 200.2, 143.3, 140.1, 137.2, 137.0, 134.5, 129.9, 129.2, 125.9, 125.7, 69.9, 47.2, 21.1, 20.0, 19.7; M.W. ($C_{18}H_{20}O_2$) 268.4; MS (EI, 20 eV): 268 (M^+, 54), 250 (M-H_2O, 26), 135 (100), 133 (36).

3-Hydroxy-1-(3,4-dimethylphenyl)-3-(naphthalen-3-yl)propan-1-one (3o)



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.12); ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.92-7.82 (m, 4H), 7.44-7.69 (m, 2H), 7.55 (dd, *J*=8.4, 1.6 Hz, 1H), 7.52-7.46 (m, 2H), 7.21 (d, *J*=8. 0 Hz, 1H), 5.51 (dd, *J*=6.8, 4.4, 1H), 3.90 (br, s, 1H), 3.45 (dd, *J*=17.6, 4.0 Hz, 1H), 3.40 (dd, *J*=17.6, 8.0 Hz, 1H), 2.32 (s, 3H), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 200.1, 143.3, 140.4, 137.0, 134.4, 133.3, 132.9, 129.9, 129.2, 128.3, 127.9, 127.6, 126.1, 125.9, 125.8, 124.4, 123.9, 70.2, 47.1, 20.0, 19.7; M.W. ($C_{21}H_{20}O_2$) 304.4; MS (EI, 20 eV); 304 (M^+, 77), 2869 (M-H_2O, 12), 156 (100), 133 (61); HRMS(ESI) calcd for $C_{21}H_{20}O_2$(M^++Na): 327.1361; found: 327.1356.

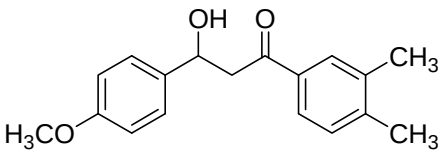
3-(4-Bomophenyl)-3-hydroxy-1-(3, 4-dimethylphenyl)propan-1-one (3p)[7]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.13); ^{1}H NMR ($CDCl_3$, 400MHz): δ

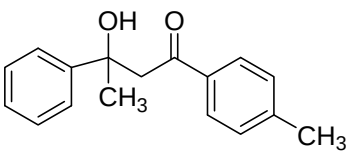
7.71-7.65 (m, 2H), 7.35 (dt, *J*=9.2, 2.4 Hz, 2H), 7.20 (dt, *J*=8.8, 2.0 Hz, 1H), 7.21 (d, *J*=7.6 Hz, 1H), 5.28 (dd, *J*=8.4, 3.2, 1H), 3.81 (br, s, 1H), 3.33 (dd, *J*=17.6, 3.6 Hz, 1H), 3.25 (dd, *J*=17.6, 8.4 Hz, 1H), 2.32 (s, 3H), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 199.9, 143.5, 142.1, 137.1, 134.3, 131.6, 129.9, 129.2, 127.5, 125.9, 121.3, 69.5, 47.0, 20.1, 19.7.; M.W. ($C_{17}H_{17}BrO_2$) 332.2; MS (EI, 20 eV); 334 (M+2, 70), 332 (M^+, 13), 314 (M-H_2O, 12), 198 (100), 133 (56).

3-Hydroxy-3-(4-methoxyphenyl)-1-(3, 4-dimethylphenyl)propan-1-one (3q)[7]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.1). The title compound was obtained as a colorless oil. ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.72-7.68 (m, 2H), 7.35 (d, *J*=8.0 Hz, 2H), 7.20 (d, *J*=7.6 Hz, 1H), 6.91 (d, *J*=8.0 Hz, 2H), 5.27 (t, *J*=7.2 1H), 3.81 (s, 3H), 3.68 (br, s, 1H), 2.31 (s, 3H), 2.30 (s, 3H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 200.2, 159.0, 143.3, 137.0, 135.2, 134.5, 129.9, 129.2, 127.0, 125.9, 113.9, 69.7, 55.3, 47.1, 29.7, 20.0, 19.7; M.W. ($C_{18}H_{20}O_3$) 284.4; MS (EI, 20 eV): 284 (M^+, 72), 266 (M-H_2O, 7), 151 (100), 133 (63).

3-Hydroxy-3-phenyl-1-p-tolylbutan-1-one (3r)



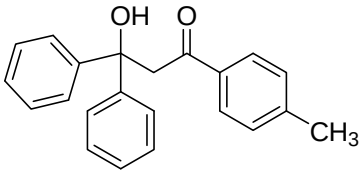
Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:40, R_f =0.13). The title compound was obtained as a pale yellow solid, Mp: 55-56 ^{o}C (ether/hexane); ^{1}H NMR ($CDCl_3$, 400MHz): δ 7.81 (d, *J*=8.4 Hz, 2H), 7.50 (d, *J*=8.4 Hz, 2H), 7.34-7.19 (m, 5H), 5.01 (s, 1H), 3.77 (d, *J*=17.2, 1H), 3.32 (d, *J*=17.2 Hz, 1H), 2.41 (s, 3H), 1.63 (s, 3H); ^{13}C NMR ($CDCl_3$,

100MHz): δ 201.0, 147.6, 144.6, 134.4, 129.3, 128.2, 128.15, 126.6, 124.3, 73.5,

[7] K.-M. Qiu, R. Yan, M. Xing, H.-H. Wang, H.-E. Cui, H.-L. Zhu, H.-B. Gong, *Bioorg.& Med. Chem.* **2012**, *20*, 6648

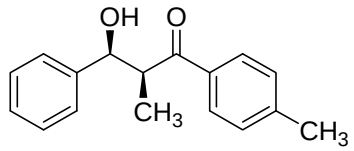
48.4, 30.9, 21.6; M.W. ($C_{17}H_{18}O_2$) 254.3; MS (EI, 20 eV): 255 (M+1, 2), 239 (M-H_2O, 25), 134(100), 119 (57); HRMS(ESI) calcd for C17$H_{18}O_2$(M^++Na): 277.1205; found: 277.1196.

3-Hydroxy-3, 3-diphenyl-1-p-tolylpropan-1-one (3s)[8]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:40, R_f =0.15). The title compound was obtained as a pale yellow solid, Mp: 122-124 ^oC (ether/hexane); ^1H NMR ($CDCl_3$, 400MHz): δ 7.82 (d, *J*=8.4 Hz, 2H), 7.42-7.34 (m, 4H), 7.28-7.14 (m, 8H), 5.56 (s, 1H), 3.87 (s, 2H), 2.39 (s, 3H); ^{13}C NMR ($CDCl_3$, 100MHz): δ 200.6, 146.5, 129.7, 129.4, 128.8, 128.5, 128.19, 128.18, 128.0, 126.9, 125.7, 77.4, 48.0, 21.6; M.W. ($C_{22}H_{20}O_2$) 316.4 ; MS (EI, 20 eV): 316 (M^+, 4), 298 (M-H_2O, 42), 197 (100), 119 (60).

***syn*-3-Hydroxy-2-methyl-3-phenyl-1-p-tolylpropan-1-one (3t)**[9]



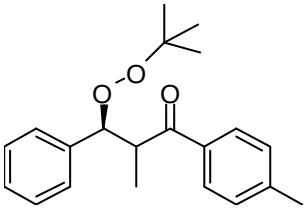
Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f =0.11). The title compound was obtained as a colorless oil. ^1H NMR ($CDCl_3$, 400MHz): δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.43-7.25 (m, 7H), 5.56 (dd, *J* = 8.0, 2.8 Hz, 1H), 3.81 (quintet, *J* = 7.2 Hz, 1H), 3.08 (d, *J*=4.0 Hz, 1H), 2.41 (s, 3H), 1.07 (d, *J*=7.2 Hz, 3H);

[8] M. Kamata, M. Kato, Y. Nishikata, *Chem. Commun.* **1996**, *21*, 2407.

[9] a) H.-J. Xu, X. Wan, S. Xu, Y.-Y. Shen, Y.-S. Feng, *Org. Lett.* **2012**, *14*, 1210; b) Y. Mei, D. J. Averill, M. J. Allen, *J. Org. Chem.* **2012**, *77*, 5624; c) H.-J. Xu, Y.-C. Liu, Y. Fu, Y.-D. Wu, *Org. Lett.* **2006**, *8*, 3449.

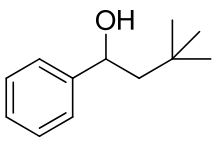
^{13}C NMR ($CDCl_3$, 100MHz): δ 204.6, 144.2, 142.2, 134.2, 129.7, 129.3, 129.1, 129.07, 128.6, 128.5, 128.4, 127.8, 127.0, 126.7, 76.8, 47.8,29.7, 21.6, 15.8; M.W. ($C_{17}H_{18}O_2$) 254.3 ; MS (EI, 20 eV): 254(M^+, 43), 237 (M-H_2O, 11), 135 (100), 119 (78).

syn-3-_t_-Butylperoxy-2-methyl-3-phenyl-1-_p_-tolylpropan-1-one (3t')



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f =0.25). The title compound was obtained as a colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.83 (d, *J* = 8.2 Hz, 1H), 7.65 (d, *J* = 8.2 Hz, 2H), 7.38-7.34 (m, 3H), 7.32-7.28 (m, 3H), 7.22-7.18 (m, 4H), 7.14 (d, *J* = 8.0 Hz, 3H), 5.21 (d, *J* = 8.2 Hz, 1H), 5.16 (d, *J* = 9.6 Hz, 1H), 3.89 (dq, *J* = 6.9, 13.8 Hz, 1H), 3.78 (dq, *J* = 6.9, 13.8 Hz, 1H), 2.41 (s, 3H), 2.33 (s, 3H), 1.36 (d, *J* = 6.9 Hz, 3H), 1.15 (s, 9H), 0.98 (s, 9H), 0.85 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.3, 201.0, 143.6, 143.5, 140.1, 139.3, 135.1, 134.1, 129.1, 129.1, 128.5, 128.3, 128.2, 128.0, 127.9, 127.6, 127.5, 127.3, 88.4, 86.4, 80.8, 80.4, 46.2, 44.5, 26.4, 26.2, 21.6, 21.5, 14.7, 14.6; HRMS (ESI) calcd for $C_{21}H_{26}O_3$ (M^++Na): 349.1780; found: 349.1779.

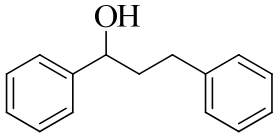
3,3-Dimethyl-1-phenylbutan-1-ol (3u)[10]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.3). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.34

(m, 4H), 7.28 – 7.25 (m, 1H), 4.82 (dd, J = 3.6, 8.4 Hz, 1H), 1.82 (s, br, OH), 1.77 (dd, J = 8.4, 14.5 Hz, 1H), 1.61 (dd, J = 3.6, 14.5 Hz, 1H), 1.00 (s, 9H); ^{13}C NMR: (100 MHz, $CDCl_3$) δ 146.4, 128.4, 127.2, 125.7, 72.4, 52.8, 30.4, 30.1; M.W. ($C_{12}H_{18}O$) 178.1 ; MS (EI, 70 eV): 178 (M^+, 1), 145 (2), 107 (100), 105 (18), 79 (69), 77 (29).

1,3-diphenylpropan-1-ol (3v)[11]



Follow the representative procedure I, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.2). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.33 (m, 4H), 7.32-7.26 (m, 3H), 7.21-7.17 (m, 3H), 4.70 (dd, J = 5.4, 7.8 Hz, 1H), 2.79-2.63 (m, 2H), 2.19-1.99 (m, 2H), 1.77 (s, br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ(100 MHz, $CDCl_3$) δ 114.5, 141.7, 128.5, 128.4, 128.3, 127.6, 125.9, 125.8, 73.8, 40.4, 32.0; HRMS(ESI) calcd for $C_{15}H_{16}O$ (M^++Na): 235.1099; found: 235.1090.

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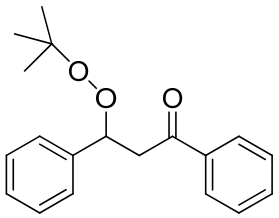
[10] R. Scholz, G. Hellmann, S. Rohs, D. Özdemir, G. Raabe, C. Vermeeren, H. Gais, *Eur. J. Org. Chem.* **2010**, 4588.

[11] a) J. Yang, X. Liu, D.-L. Meng, H.-Y. Chen, Z.-H. Zong, T.-T. Feng, K. Sun, *Adv. Synth. Catal.* **2012**, *354*, 328; b) P. Satyanarayana, G. M. Reddy, H. Maheswaran, M. L. Kantam, *Adv. Synth. Catal.* **2013**, *355*, 1859.

Analytical data for β-peroxyketones 4a-t, and 5

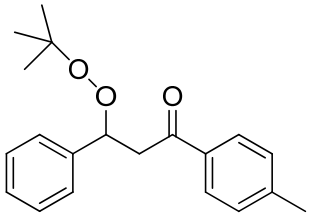
3-(*tert*-Butylperoxy)-1,3-diphenylpropan-1-one (4a)[12]



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.3). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J = 7.1 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.47-7.42 (m, 4H), 7.35 (t, J = 7.1 Hz, 2H), 7.31-7.28 (m, 1H), 5.60 (t, J = 6.7 Hz,

1H), 3.78 (dd, *J* = 7.1, 16.3 Hz, 1H), 3.22 (dd, *J* = 5.9, 16.2 Hz, 1H), 1.17 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.4, 139.9, 137.1, 133.0, 128.5, 128.3, 128.2, 128.1, 127.0, 82.3, 80.7, 43.9, 26.3; HRMS(ESI) calcd for $C_{19}H_{22}O_3$(M^++Na): 321.1467; found: 321.1463.

3-(*tert*-Butylperoxy)-3-phenyl-1-(p-tolyl)propan-1-one (4b)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 7.4 Hz, 2H), 7.37 (t, *J* = 6.9 Hz, 2H), 7.33-7.31 (m, 1H), 7.28-7.26 (m,2H), 5.62 (t, *J* = 6.6 Hz, 1H), 3.77 (dd, *J* = 7.1, 16.2 Hz, 1H), 3.21 (dd, *J* = 6.1, 16.2 Hz, 1H), 2.44 (s, 3H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.0, 143.8, 140.0, 134.6, 129.1, 128.3, 128.0, 127.0, 82.3, 80.7, 43.8, 26.3, 21.6; HRMS(ESI) calcd for $C_{20}H_{24}O_3$(M^++Na): 335.1623; found: 335.1617.

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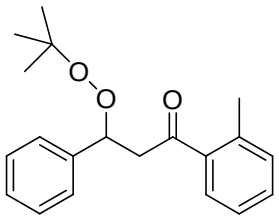
[12] W. Liu, Y. Li, K. Liu, Z. Li, J. *Am. Chem. Soc.* **2011**, *133*, 10756.

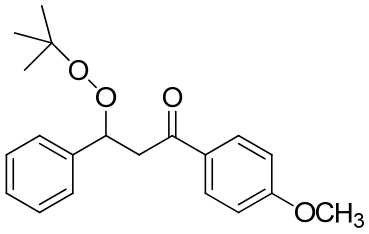
3-(*tert*-Butylperoxy)-3-phenyl-1-(o-tolyl)propan-1-one (4c)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 7.8 Hz, 1H), 7.40 (d, *J* = 6.8 Hz, 2H), 7.38-7.32 (m, 3H), 7.31-7.7.28 (m, 1H), 7.27-7.21 (m, 2H), 5.56 (dd, *J* = 6.0, 7.3 Hz, 1H), 3.67 (dd, *J* = 7.4, 16.2 Hz, 1H), 3.16 (dd, *J* = 6.0, 16.2 Hz, 1H), 2.41 (s, 3H),

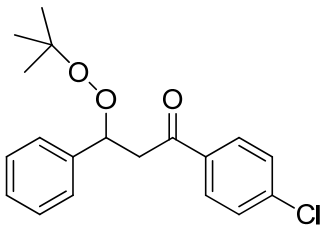
1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.2, 139.9, 138.1, 138.0, 131.8, 131.2, 128.5, 128.3, 128.0, 127.1, 125.5, 82.2, 80.7, 46.9, 26.3, 21.0; HRMS(ESI) calcd for $C_{20}H_{24}O_3$(M^++Na): 335.1623; found: 335.1618.

3-(*tert*-Butylperoxy)-1-(4-methoxyphenyl)-3-phenylpropan-1-one (4d)



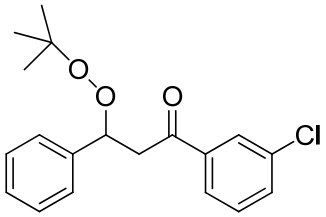
Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.9 Hz, 2H), 7.43 (d, *J* = 8.5 Hz, 2H), 7.36-7.32 (m, 2H), 7.31-7.28 (m, 1H), 6.92 (d, *J* = 8.9 Hz, 2H), 5.58 (t, *J* = 6.5 Hz, 1H), 3.87 (s, 3H), 3.72 (dd, *J* = 7.1, 16.0 Hz, 1H), 3.16 (dd, *J* = 6.0, 16.0 Hz, 1H), 1.17 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.8, 163.4, 140.1, 130.5, 130.2, 128.3, 127.9, 127.0, 113.6, 82.4, 80.7, 55.4, 43.57, 26.3; HRMS(ESI) calcd for $C_{20}H_{24}O_4$ (M^++Na): 351.1573; found: 351.1567.

3-(*tert*-Butylperoxy)-1-(4-chlorophenyl)-3-phenylpropan-1-one (4e)



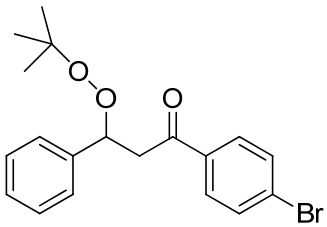
Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.6 Hz, 2H), 7.43-7.7.41 (m, 4H), 7.36 (t, *J* = 6.9 Hz, 2H), 7.32-7.30 (m, 1H), 5.57 (dd, *J* = 6.0, 7.0 Hz, 1H), 3.75 (dd, *J* = 7.2, 16.0 Hz, 1H), 3.16 (dd, *J* = 5.8, 16.0 Hz, 1H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.3, 139.7, 139.5, 135.4, 129.6, 128.8, 128.4, 128.2, 127.0, 82.3, 80.8, 43.9, 26.3; HRMS(ESI) calcd for $C_{19}H_{21}ClO_3$ (M^++Na): 355.1077; found: 355.1070.

3-(*tert*-Butylperoxy)-1-(3-chlorophenyl)-3-phenylpropan-1-one (4f)



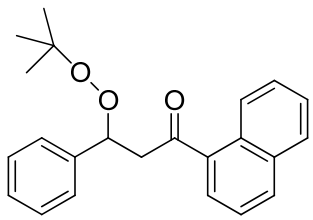
Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (t, *J* = 2.0 Hz, 1H), 7.83 (d, *J* = 7.8 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.44-7.40 (m, 3H), 7.36 (dt, *J* = 1.4, 8.3 Hz, 2H), 7.33-7.28 (m, 1H), 5.57 (dd, *J* = 5.8, 7.1 Hz, 1H), 3.76 (dd, *J* = 7.2, 16.2 Hz, 1H), 3.17 (dd, *J* = 5.8, 16.2 Hz, 1H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.3, 139.6, 138.6, 134.8, 132.9, 129.8, 128.4, 128.2, 127.0, 126.3, 82.2, 80.8, 44.1, 26.3; HRMS(ESI) calcd for $C_{19}H_{21}ClO_3$ (M^++Na): 355.1077; found: 355.1073.

3-(*tert*-Butylperoxy)-1-(4-bromophenyl)-3-phenylpropan-1-one (4g)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 8.8 Hz, 2H), 7.59 (d, *J* = 8.8 Hz, 2H), 7.42 (dd, *J* = 1.7, 8.4 Hz, 2H), 7.38-7.34 (m, 2H), 7.32-7.28 (m, 1H), 5.57 (dd, *J* = 5.8, 7.2 Hz, 1H), 3.74 (dd, *J* = 7.2, 16.1 Hz, 1H), 3.16 (dd, *J* = 5.7, 16.1 Hz, 1H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.5, 139.6, 135.8, 131.8, 129.7, 128.4, 128.2, 128.2, 127.0, 82.3, 80.8, 43.9, 26.3; HRMS(ESI) calcd for $C_{19}H_{21}BrO_3$ (M^++Na): 399.0572; found: 399.0567.

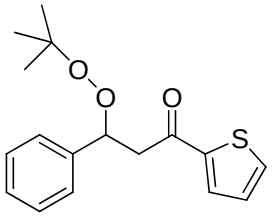
3-(*tert*-Butylperoxy)-1-(naphthalen-1-yl)-3-phenylpropan-1-one (4h)



Follow the representative procedure II, isolated by column

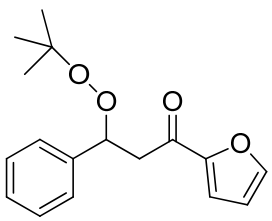
chromatography (EtOAc/hexanes = 1:10, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$); δ 8.51 (d, *J* = 8.0 Hz, 1H), 7.98 (d, *J* = 8.4 Hz, 1H), 7.88 (dt, *J* = 1.4, 7.3 Hz, 2H), 7.57-7.48 (m, 3H), 7.45-7.43 (m, 2H), 7.38-7.31 (m, 3H), 5.68 (dd, *J* = 5.5, 7.7 Hz, 1H), 3.84 (dd, *J* = 7.7, 16.0 Hz, 1H), 3.30 (dd, *J* = 5.5, 16.0 Hz, 1H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.2, 139.9, 136.0, 133.8, 132.7, 130.0, 128.4, 128.3, 128.1, 127.8 (x2), 127.1, 126.4, 125.8, 124.2, 82.4, 80.7, 47.6, 26.3; HRMS(ESI) calcd for $C_{23}H_{24}O_3$ (M^++Na): 371.1623; found: 371.1615.

3-(*tert*-Butylperoxy)-1-(thiophen-2-yl)-3-phenylpropan-1-one (4i)[12]



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (dd, *J* =0.4, 3.9 Hz, 1H), 7.63 (dd, *J* = 0.4, 4.9 Hz, 1H), 7.43 (d, *J* = 6.9 Hz, 2H), 7.36 (t, *J* = 6.9 Hz, 2H), 7.32-7.28 (m, 1H), 7.12 (dd, *J* = 4.0, 4.9 Hz, 1H), 5.57 (dd, *J* = 5.7, 7.4 Hz, 1H), 3.67 (dd, *J* = 7.5, 15.6 Hz, 1H), 3.14 (dd, *J* = 5.7, 15.6 Hz, 1H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.1, 144.5, 139.7, 133.8, 132.2, 128.4, 128.1, 128.0, 126.9, 82.2, 80.8, 44.9, 26.2; HRMS(ESI) calcd for $C_{17}H_{20}O_3S$ (M^++Na): 327.1031; found: 327.1023.

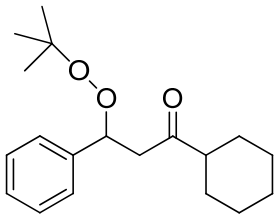
3-(*tert*-Butylperoxy)-1-(furan-2-yl)-3-phenylpropan-1-one (4j)[12]



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 1.2 Hz, 1H), 7.43-7.41 (m, 2H),

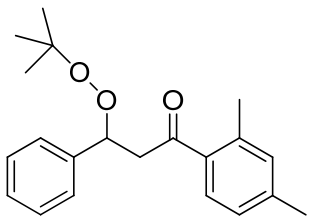
7.37-7.33 (m, 2H), 7.31-7.27 (m, 1H), 7.19 (d, *J* = 3.6 Hz, 1H), 6.52 (dd, *J* = 1.6, 3.6 Hz, 1H), 5.57 (dd, *J* = 5.4, 12.0 Hz, 1H), 3.60 (dd, *J* = 8.0, 15.6 Hz, 1H), 3.04 (dd, *J* = 5.4, 15.6 Hz, 1H), 1.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 186.2, 152.8, 146.4, 139.7, 128.4, 128.1, 127.0, 117.4, 112.2, 82.0, 80.8, 44.1, 26.2; HRMS(ESI) calcd for $C_{17}H_{20}O_4$ (M^++Na): 311.126; found: 311.1252.

3-(*tert*-Butylperoxy)-1-cyclohexyl-3-phenylpropan-1-one (4k)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:5, R_f = 0.6). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.31 (m, 4H), 7.30-7.28 (m,1H), 5.42 (dd, *J* = 5.5, 7.7 Hz, 1H), 3.15 (dd, *J* = 7.7, 16.2 Hz, 1H), 2.68 (dd, *J* = 5.5, 16.2 Hz, 1H), 2.34-2.30 (m, 1H), 1.86-1.58 (m, 10H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 210.9, 140.1, 128.3, 127.9, 126.9, 81.9, 80.7, 51.4, 46.0, 28.0, 28.0, 26.3, 25.8, 25.6, 25.5; HRMS(ESI) calcd for $C_{19}H_{28}O_3$ (M^++Na): 327.1936; found: 327.1930.

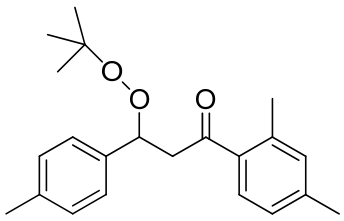
3-(*tert*-Butylperoxy)-1-(2,4-dimethylphenyl)-3-phenylpropan-1-one (4l)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.45). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 7.8 Hz, 1H), 7.41-7.38 (m, 2H), 7.36-7.32 (m, 2H), 7.30-7.29 (m, 1H), 7.05-7.03 (m, 2H), 5.55 (dd, *J* = 6.2, 7.2 Hz, 1H), 3.66 (dd, *J* = 7.4, 16.0 Hz, 1H), 3.14 (dd, *J* = 6.0, 16.0 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.4, 141.9, 140.0, 138.6,

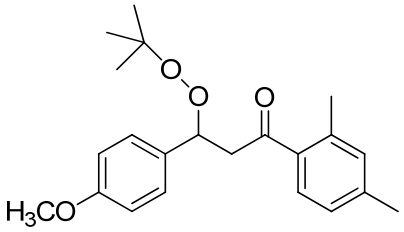
135.0, 132.7, 129.1, 128.3, 128.0, 127.1, 126.2, 82.3, 80.6, 46.6, 26.3, 21.3, 21.2; HRMS(ESI) calcd for $C_{21}H_{26}O_3$ (M^++Na): 349.178; found: 349.1774.

3-(*tert*-Butylperoxy)-1-(2,4-dimethylphenyl)-3-(p-tolyl)propan-1-one (4m)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.45). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 7.9 Hz, 1H), 7.29 (d, *J* = 7.8 Hz, 2H), 7.15 (d, *J* = 7.8 Hz, 2H), 7.06 (d, *J* = 7.9 Hz, 1H), 7.03 (s, 1H), 5.52 (t, *J* = 6.6 Hz, 1H), 3.67 (dd, *J* = 7.2, 16.0 Hz, 1H), 3.14 (dd, *J* = 6.1, 16.0 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H), 2.34 (s, 3H), 1.17 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.6, 141.8, 138.6, 137.7, 136.8, 135.0, 132.7, 129.2, 129.0, 127.1, 126.2, 82.2, 80.6, 46.5, 26.3, 21.3, 21.3, 21.1; HRMS(ESI) calcd for $C_{22}H_{28}O_3$ (M^++Na): 363.1936; found: 363.1930.

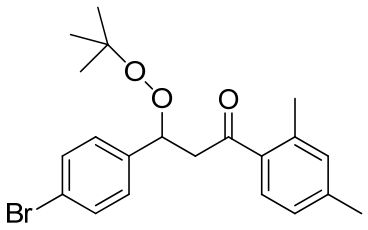
3-(*tert*-Butylperoxy)-1-(2,4-dimethylphenyl)-3-(4-methoxyphenyl)propan-1-one (4n)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.7 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 1H), 7.03 (s, 1H), 6.87 (d, *J* = 8.7 Hz, 2H), 5.50 (t, *J* = 6.7 Hz, 1H), 3.79 (s, 3H), 3.70 (dd, *J* = 7.0, 16.0 Hz, 1H), 3.17 (dd, *J* = 6.5, 16.0 Hz, 1H), 2.40 (s, 3H), 2.35 (s, 3H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.6, 159.3, 141.8, 138.6, 135.0, 132.7, 131.7, 129.1, 128.5,

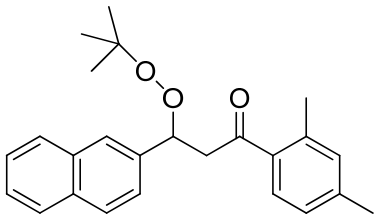
126.1, 113.6, 82.0, 80.5, 55.1, 46.3, 26.3, 21.3, 21.2; HRMS(ESI) calcd for $C_{22}H_{28}O_4$ (M^++Na): 379.1886; found: 379.1880.

3-(*tert*-Butylperoxy)-3-(4-bromophenyl)-1-(2,4-dimethylphenyl)propan-1-one (4o)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 8.0 Hz, 1H), 7.48-7.45 (m, 2H), 7.30-7.27 (m, 2H), 7.07-7.04 (m, 2H), 5.51 (t, *J* = 6.8 Hz, 1H), 3.60 (dd, *J* = 7.3, 16.2 Hz, 1H), 3.09 (dd, *J* = 6.1, 16.2 Hz, 1H), 2.42 (s, 3H), 2.35 (s, 3H), 1.17 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.8, 142.1, 139.3, 138.7, 134.7, 132.8, 131.4, 129.1, 128.8, 126.2, 121.8, 81.6, 80.7, 46.3, 26.3, 21.3; HRMS(ESI) calcd for $C_{21}H_{25}BrO_3$ (M^++Na): 427.0885; found: 427.0877.

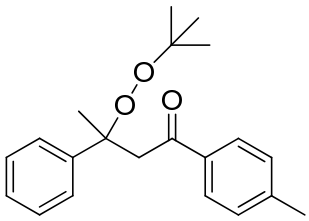
3-(*tert*-Butylperoxy)-1-(2,4-dimethylphenyl)-3-(naphthalen-2-yl)propan-1-one (4p)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.36). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.80 (m, 4H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.55 (dd, *J* = 1.5, 8.7 Hz, 1H), 7.50-7.44 (m, 2H), 7.07-7.03 (m, 2H), 5.73 (dd, *J* = 5.9, 7.4 Hz, 1H), 3.73 (dd, *J* = 7.5, 16.1 Hz, 1H), 3.22 (dd, *J* = 5.8, 16.1 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H), 1.20 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.3, 141.9, 138.7, 137.6, 135.0, 133.2, 133.1, 132.7, 129.2, 128.1, 128.0,

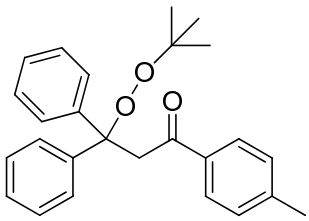
127.6, 126.2, 126.0, 125.9, 124.8, 82.4, 80.7, 46.6, 26.4, 21.3, 21.3; HRMS(ESI) calcd for $C_{25}H_{28}O_3$ (M^++Na): 399.1936; found: 399.1927.

3-(*tert*-Butylperoxy)-3-phenyl-1-(p-tolyl)butan-1-one (4q)



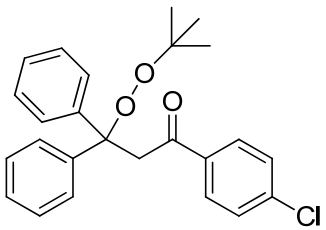
Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.49 (dd, *J* = 1.4, 8.7 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 2H), 7.26-7.23 (m, 1H), 7.22-7.20 (m, 2H), 3.63 (d, *J* = 14.5 Hz, 1H), 3.29 (d, *J* = 14.5 Hz, 1H), 2.39 (s, 3H), 1.84 (s, 3H), 1.10 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.4, 144.5, 143.4, 135.6, 128.9, 128.7, 127.9, 127.0, 125.7, 83.0, 79.2, 48.1, 26.5, 23.5, 21.6; HRMS(ESI) calcd for $C_{21}H_{26}O_3$ (M^++Na): 349.178; found: 349.1767.

3-(*tert*-Butylperoxy)-3,3-diphenyl-1-(p-tolyl)propan-1-one (4r)



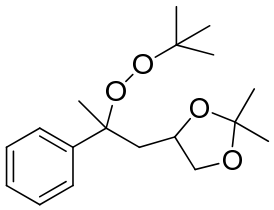
Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.6 Hz, 2H), 7.40-7.38 (m, 4H), 7.31-7.26 (m, 4H), 7.25-7.22 (m, 2H), 7.21-7.19 (m, 2H), 4.16 (s, 2H), 2.39 (s, 3H), 1.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.6, 143.7, 143.2, 135.8, 128.8, 128.5, 127.5, 127.3, 127.0, 86.2, 79.6, 44.6, 26.4, 21.5; HRMS(ESI) calcd for $C_{26}H_{28}O_3$ (M^++Na): 411.1936; found: 411.1930.

3-(*tert*-Butylperoxy)-1-(4-chlorophenyl)-3,3-diphenylpropan-1-one (4s)



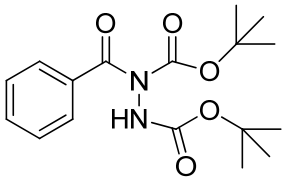
Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.4). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.6 Hz, 1H), 7.36-7.33 (m, 6H), 7.30-7.29 (m, 1H), 7.28-7.26 (m, 3H), 7.25-7.23 (m, 1H), 4.12 (s, 2H), 1.03 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.1, 143.4, 138.9, 136.7, 129.9, 128.4, 127.5, 127.2, 127.1, 86.2, 79.9, 44.8, 26.4; HRMS(ESI) calcd for $C_{25}H_{25}ClO_3$ (M^++Na): 431.139; found: 431.1383.

4-(2-(*tert*-Butylperoxy)-2-phenylpropyl)-2,2-dimethyl-1,3-dioxolane (4t)



Follow the representative procedure II, isolated by column chromatography (EtOAc/hexanes = 1:20, R_f = 0.5). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (t, *J* = 7.3 Hz, 4H), 7.34 (d, *J* = 7.3 Hz, 2H), 7.30 (d, *J* = 7.7 Hz, 2H), 7.24 - 7.21 (m, 2H), 4.32 – 4.26 (m, 1H), 3.97 (dd, *J* = 5.6, 8.2 Hz, 1H), 3.76 (dd, ; *J* = 5.7, 8.2 Hz, 1H), 3.74 – 3.68 (m, 1H), 3.57 (t, *J* = 8.1 Hz, 1H), 3.27 (t, *J* = 8.0 Hz, 1H), 2.32 - 2.28 (m, 2H), 2.16 (dd, *J* = 9.0, 14.0 Hz, 1H), 2.04 (dd, *J* = 7.9, 14.3 Hz, 1H), 1.67 (s, 3H), 1.66 (s, 3H), 1.36 (s, 3H), 1.35 (s, 3H), 1.33 (s, 3H), 1.27 (s, 9H), 1.24 (s, 9H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.5, 143.9, 128.0, 127.9, 126.8, 126.7, 125.4, 125.4, 107.7, 107.4, 82.5, 82.0, 79.2, 79.0, 72.9, 72.7, 70.4, 70.2, 44.8, 44.2, 26.8, 26.7, 26.7, 26.0, 25.8, 25.8, 24.3; HRMS(ESI) calcd for $C_{18}H_{28}O_4$(M^++Na): 331.1879; found: 331.1876.

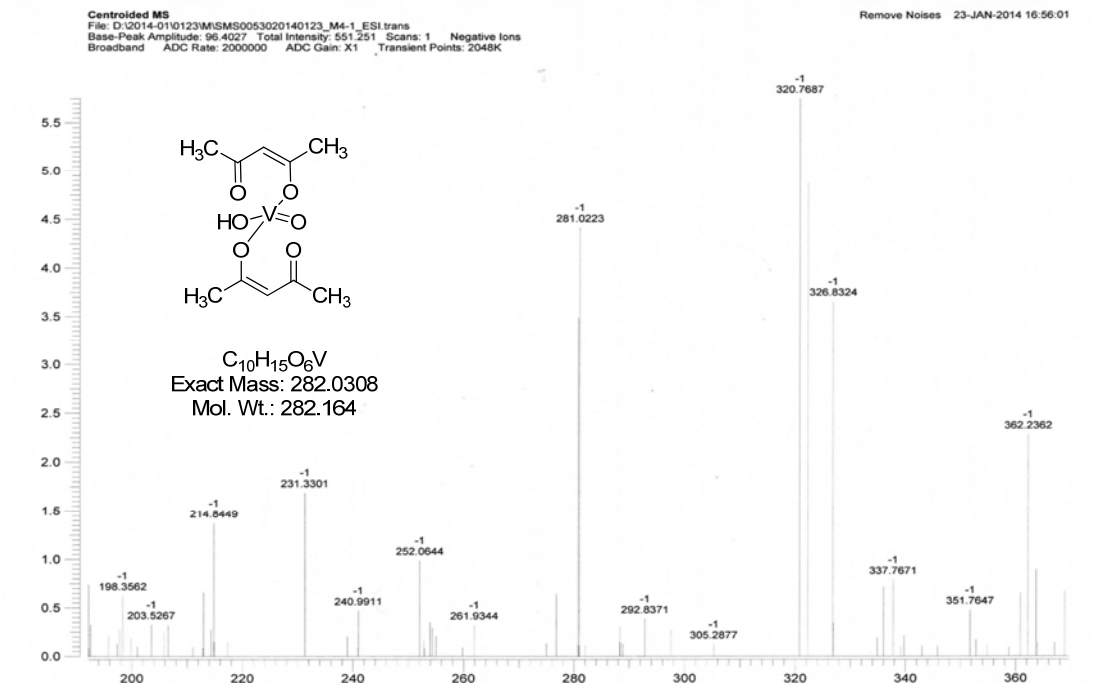
Di-*tert*-butyl 1-benzoylhydrazine-1,2-dicarboxylate (5)



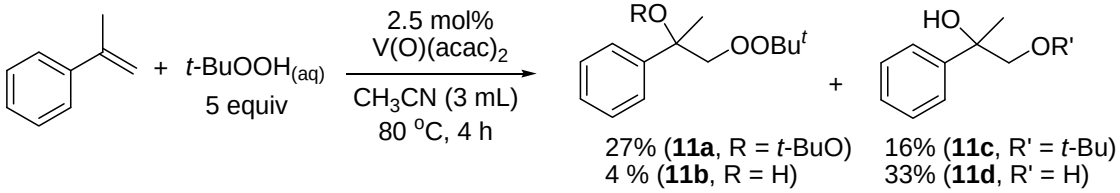
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 – 7.67 (m, 2H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.8 Hz, 2H), 6.79 (s, br, NH),

1.49 (s, 9H), 1.22 (s, br, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.5, 151.7, 131.7, 128.1, 128.0, 84.4, 82.2, 28.1, 27.3; HRMS(ESI) calcd for $C17H_{24}N_2O_5(M^+$+Na): 359.1577; found: 359.1583; R_f = 0.55 (EtOAc/hexanes = 3:7).

ESI mass spectrum of vanadyl hydroxo complex 8 ($VO(acac)_2$-OH)



Cross dioxygenation of α-methylstyrene catalyzed by $V(O)(acac)_2$:

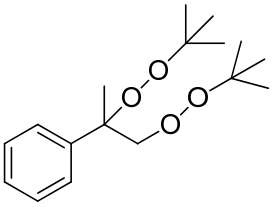


27% (**11a**, R = *t*-BuO)
4 % (**11b**, R = H)
16% (**11c**, R' = *t*-Bu)
33% (**11d**, R' = H)

To a test tube, $V(O)(acac)_2$ (7.2 mg, 2.5 mol%) was dissolved in 3 mL anhydrous CH_3CN and α-methylstyrene (0.13 mL, 1.01 mmol) was added under nitrogen atmosphere via syringe and stirred for 5 min at 80˚C. A solution of 70% aqueous *tert*-Butyl hydrogenperoxide (690 μL) was added to the reaction and stirred for 4 h at 80˚C. After complete consuming of the α-methylstyrene monitored by TLC, the

solvent and all violates were removed under reduced pressure. The residue was purified using flash chromatography on silica gel with EA/hexane 1:10.

(1,2-Bis(*tert*-butylperoxy)propan-2-yl)benzene (11a)[13]



Isolated by column chromatography (EtOAc/hexanes = 1:10, R_f = 0.6). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48-7.46 (m, 2H), 7.32 (t, *J* = 7.2 Hz, 2H), 7.26-7.25 (m, 1H), 4.26 (d, *J* = 9.6 Hz, 1H), 4.22 (d, *J* = 9.6 Hz, 1H), 1.66 (s, 3H), 1.25 (s, 9H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.0, 127.8, 127.1, 126.0, 82.9, 80.5, 79.4, 79.2, 26.6, 26.2, 21.8; HRMS(ESI) calcd for $C_{17}H_{28}O_4$(M^++Na): 319.1886; found: 319.1882.

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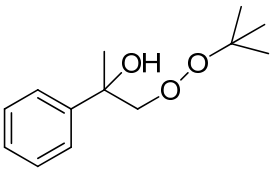
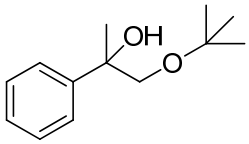
[13] F. Fontana, S. Araneo, F. Recupero, S. Banfi, S. Quici, F. Minisci, *J. Am. Chem. Soc.* **1995**, *117*, 226.

1-(*tert*-Butylperoxy)-2-phenylpropan-2-ol (11b)[14]



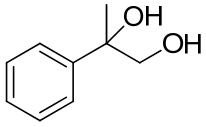
Isolated by column chromatography (EtOAc/hexanes = 1:10, R_f = 0.5). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45-7.43 (m, 2H), 7.35 (t, *J* = 7.2 Hz, 2H), 7.29-7.26 (m, 1H), 4.00 (d, *J* = 11.8 Hz, 1H), 3.89 (d, *J* = 11.8 Hz, 1H), 1.66 (s, 3H), 1.53 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.2, 128.1, 127.2, 125.8, 84.2, 79.8, 68.2, 26.5, 22.3; HRMS(ESI) calcd for $C_{13}H_{20}O_3$(M^++Na): 247.131; found: 247.1308.

1-(*tert*-Butoxy)-2-phenylpropan-2-ol (11c)



Isolated by column chromatography (EtOAc/hexanes = 1:10, R_f = 0.55). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.6 Hz, 2H), 7.34 (t, *J* = 8.6 Hz, 2H), 7.27 – 7.25 (m, 1H), 4.18 (d, *J* = 12.2 Hz, 1H), 4.08 (d, *J* = 12.2 Hz, 1H), 1.58 (s, 3H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 128.1, 126.9, 125.1, 82.9, 81.2, 75.1, 26.6, 26.2; HRMS(ESI) calcd for $C_{13}H_{20}O_2$(M^++Na): 231.1361; found: 231.1356.

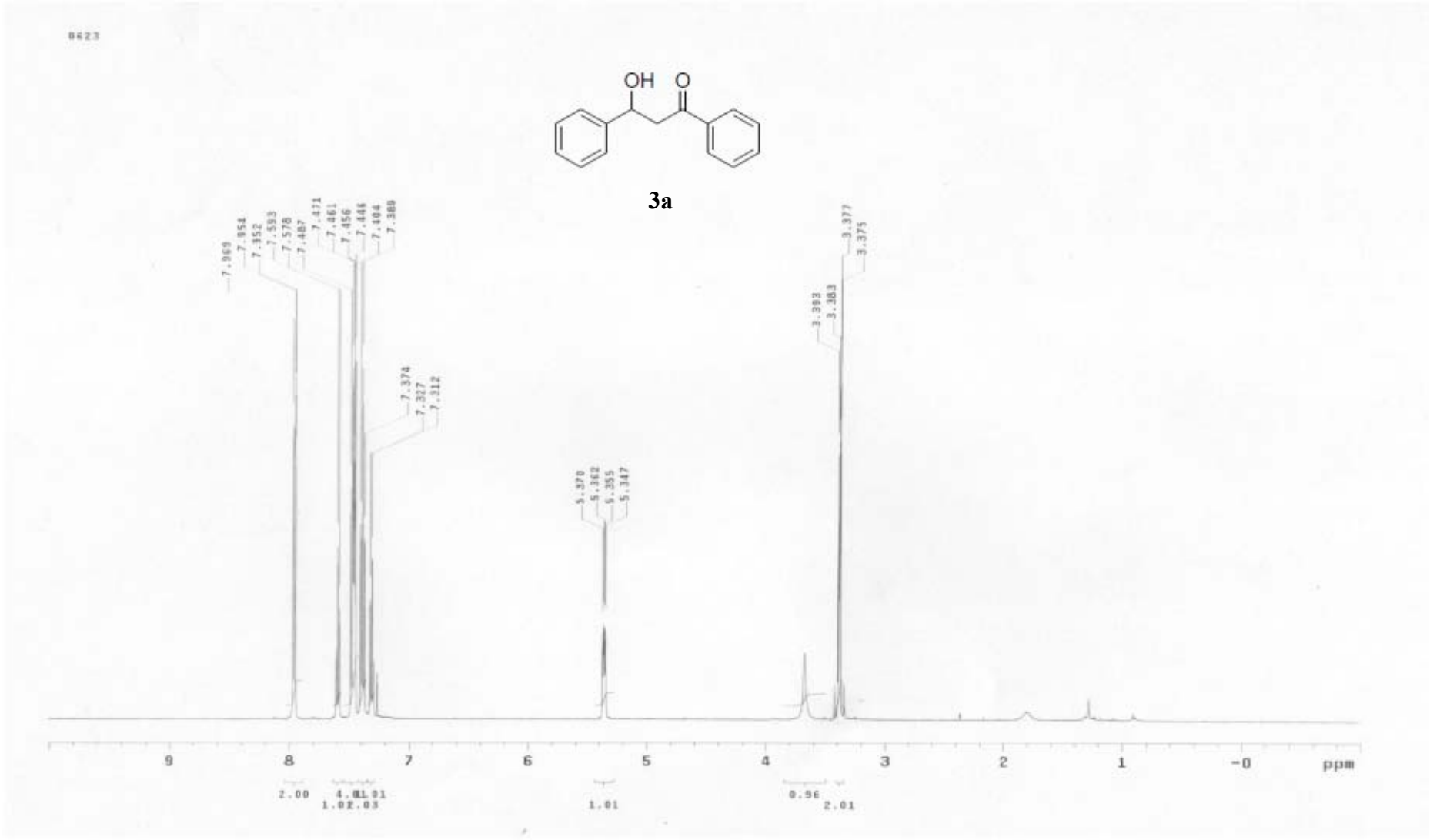
2-Phenylpropane-1, 2-diol (11d)[15]



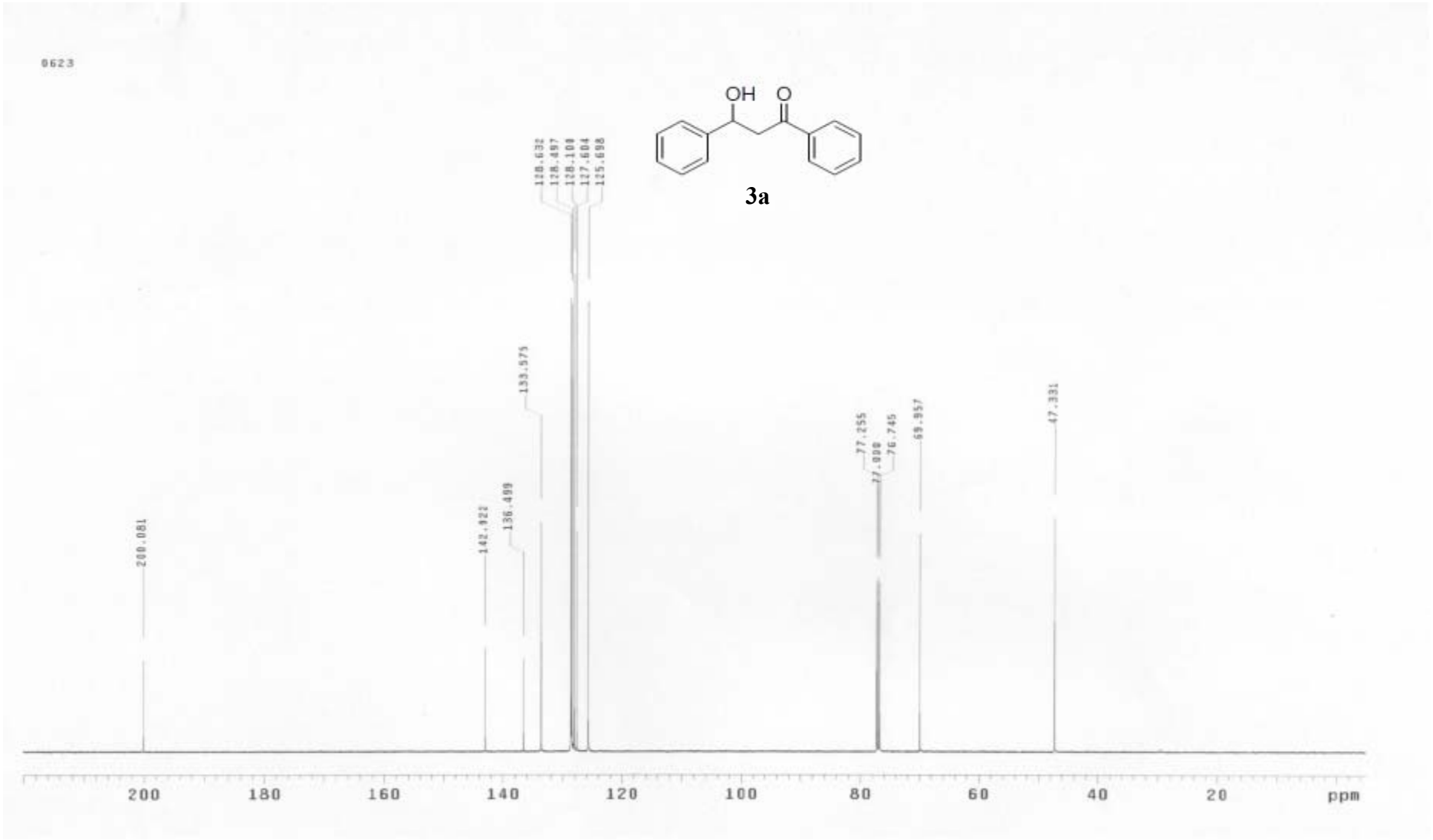
Isolated by column chromatography (EtOAc/hexanes = 1:10, R_f = 0.45). The title compound was obtained as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 7.8 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 2H), 7.27 (t, *J* = 7.5 Hz, 1H), 3.79 (d, *J* = 11.2 Hz, 1H), 3.63 (d, *J* = 11.2 Hz, 1H), 2.10 (s, br, 2H), 1.53 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.9, 128.4, 127.1, 125.0, 74.8, 71.0, 26.0; HRMS(ESI) calcd for $C_9H_{12}O_2$(M^++Na): 175.0735; found: 175.0724.

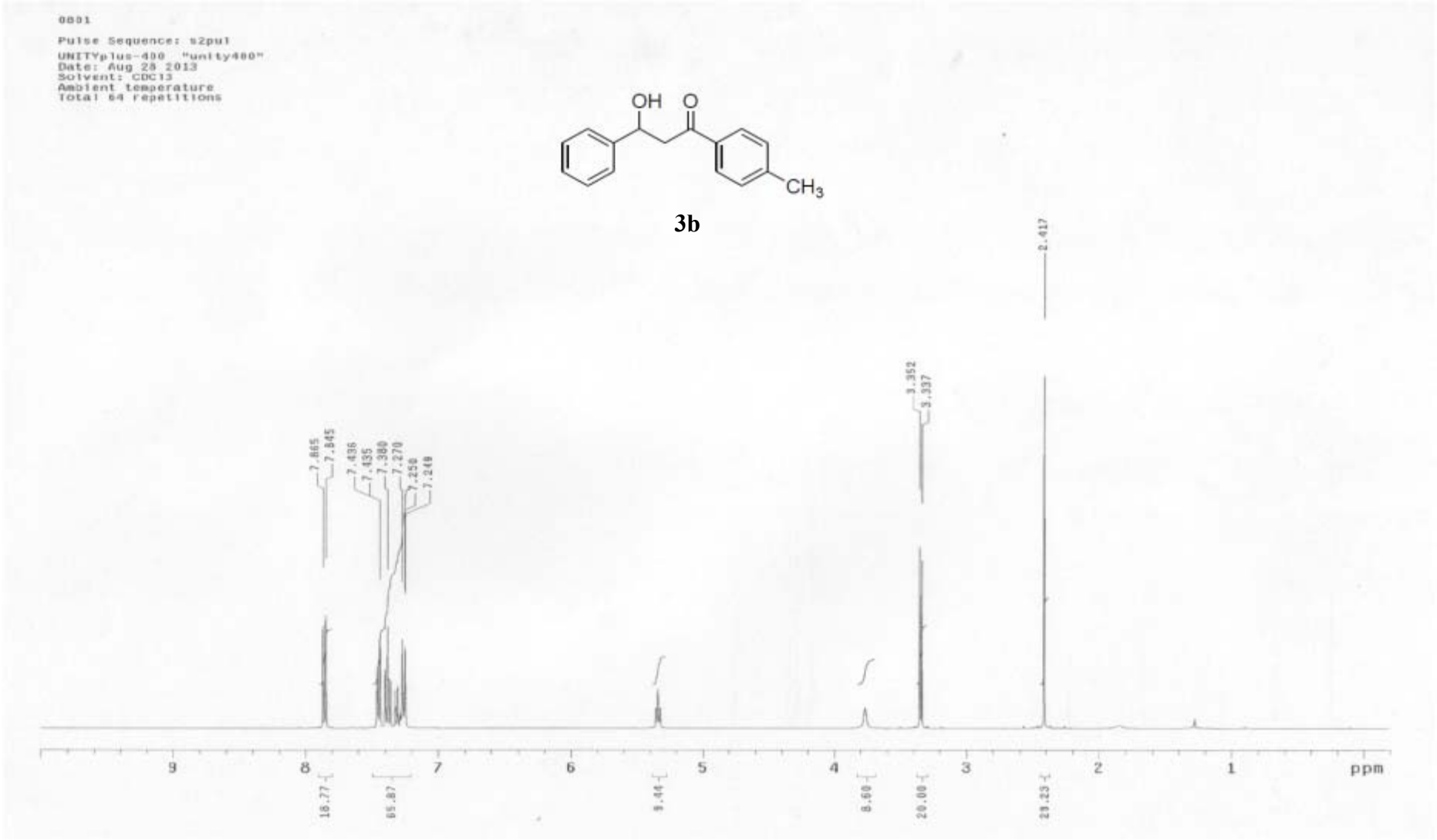
[14] S. Bhat, S. Chandrasekaran, *Tetrahedron Lett.* **1996**, *37*, 3581.

[15] a) Q. Yao, *Org. Lett.* **2002**, *4*, 2197-2199; b) Z. Wang, Y.-T. Cui, Z.-B. Xu, J. Qu, *J. Org. Chem.* **2008**, *73*, 2270

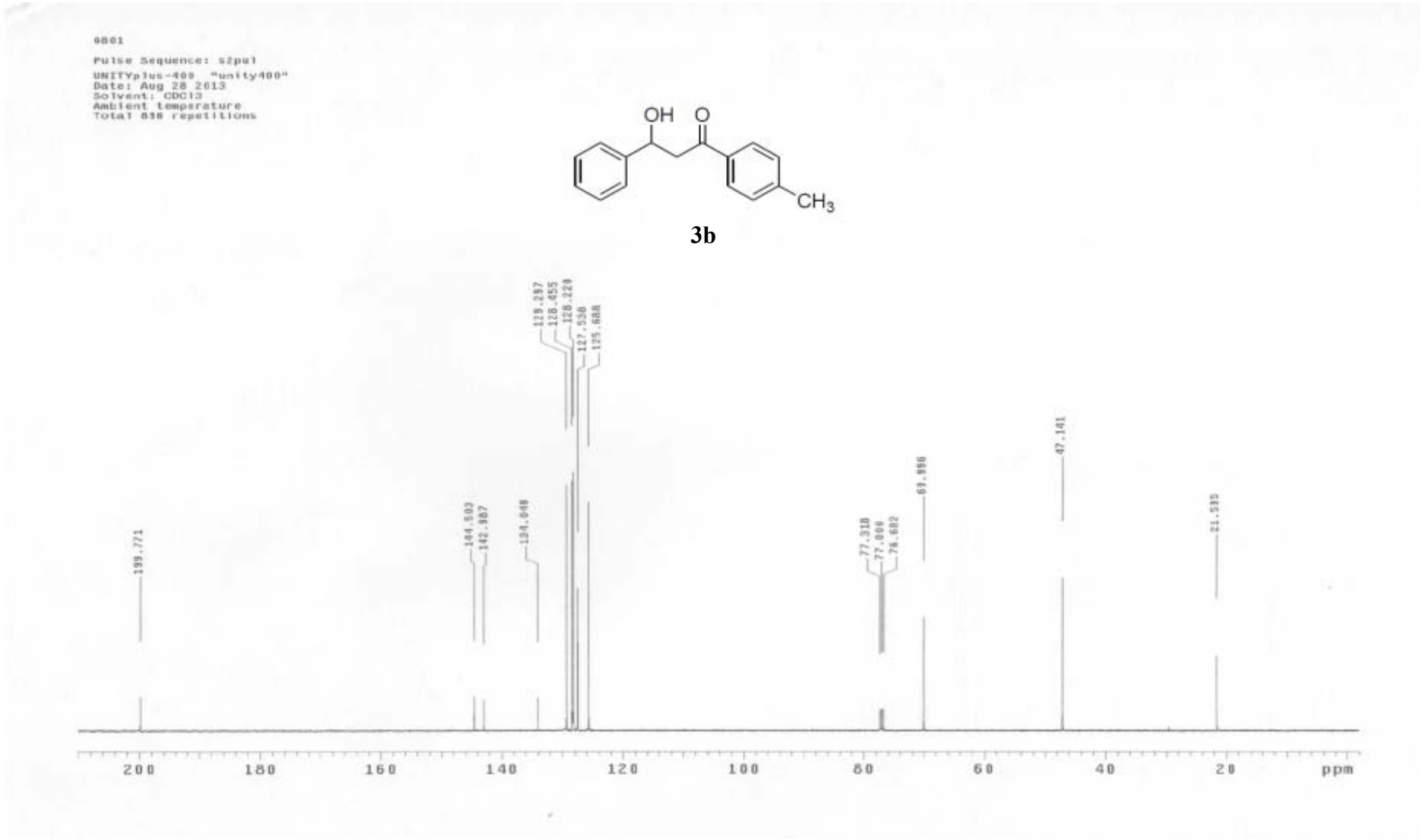

0623
OH
O
7.969
7.954
7.952
7.593
7.578
7.487
7.471
7.461
7.456
7.446
7.404
7.389
7.374
7.327
7.312
5.370
5.362
5.355
5.347
3.393
3.383
3.377
3.375
9
8
7
6
5
4
3
2
1
-0
ppm
2.00
1.01
0.96
2.01

3a


0623
OH
O
3a
200.081
142.922
136.499
133.575
77.255
77.000
76.745
69.957
47.331
200
180
160
140
120
100
80
60
40
20
ppm


0801
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions
OH
O
CH3
2.417
3.352
3.337
7.865
7.845
7.436
7.435
7.380
7.270
7.256
7.240
9
8
7
6
5
4
3
2
1
ppm
18.77
65.87
9.44
8.60
20.00
29.23

3b


0801
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 836 repetitions
OH
O
CH3
199.771
144.503
142.387
134.648
129.287
128.455
128.220
127.536
125.688
77.318
77.000
76.682
69.956
47.141
21.535
200
180
160
140
120
100
80
60
40
20
ppm

3b

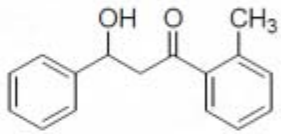

0802
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 48 repetitions
OH
O
CH3
2.540
3.340
3.319
3.315
3.306
7.653
7.634
7.448
7.422
7.421
7.394
7.392
7.374
7.370
7.354
7.298
7.272
7.271
7.251
9
8
7
6
5
4
3
2
1
ppm
9.87
48.38
30.78
10.00
21.95
20.03

3c

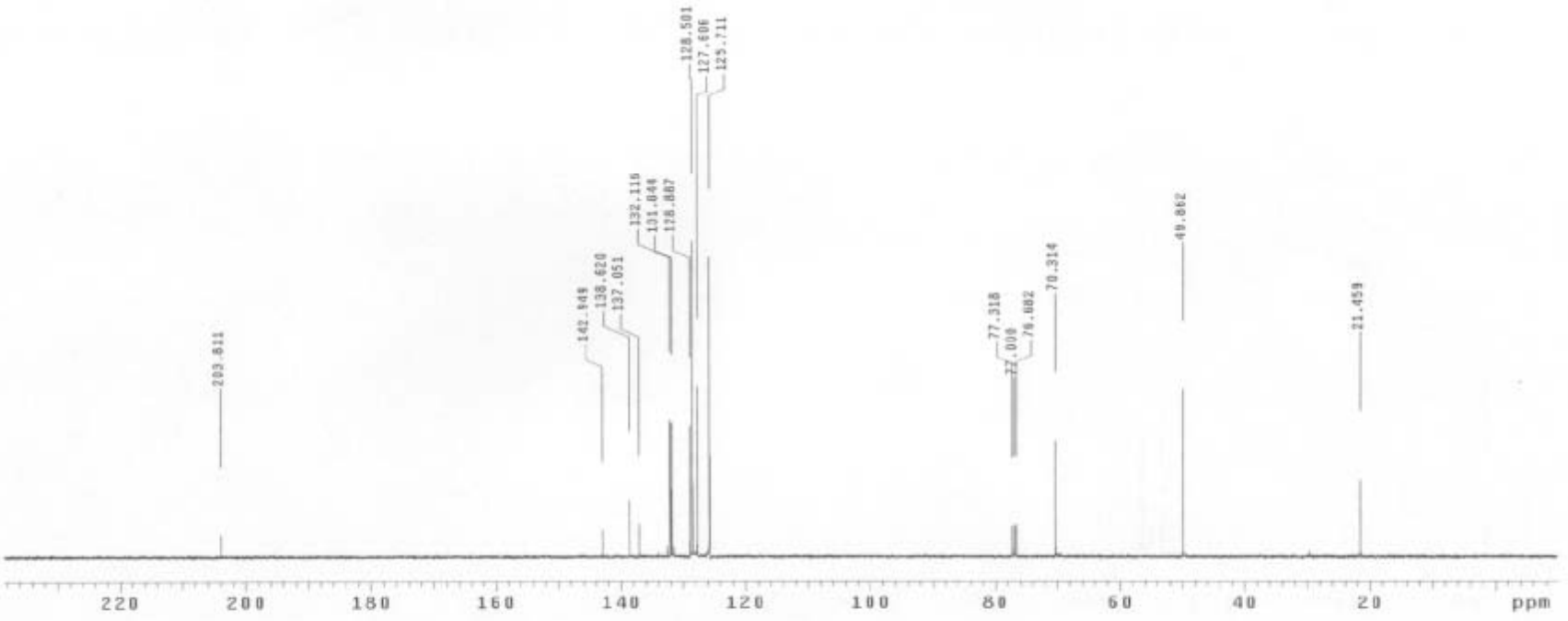
0802

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 896 repetitions



3c



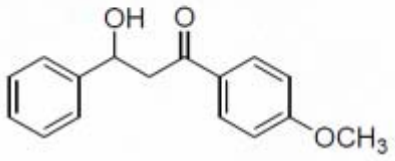

0804
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions
OH
O
OCH3
7.936
7.916
7.448
7.447
7.431
7.430
7.428
7.391
7.375
7.353
7.233
6.434
6.412
3.860
3.320
3.308
3.302
9
8
7
6
5
4
3
2
1
ppm
20.98
52.88
21.84
16.00
49.34
20.76

3d

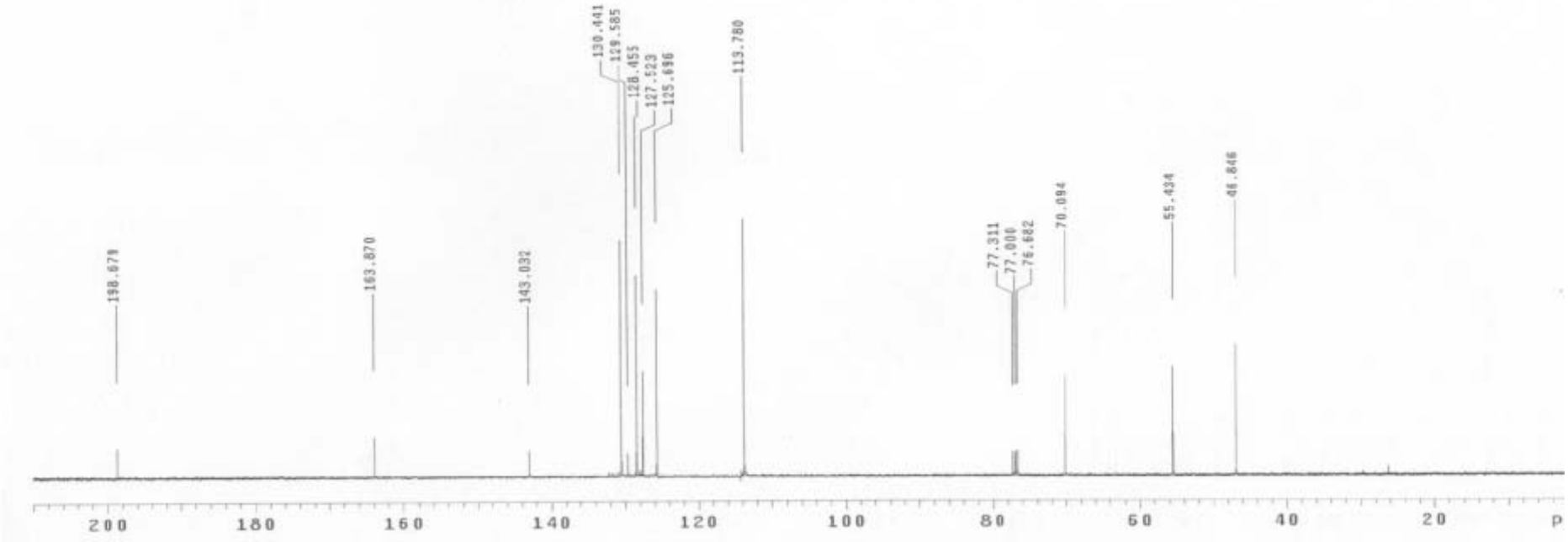
0804

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 448 repetitions



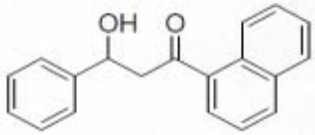
3d



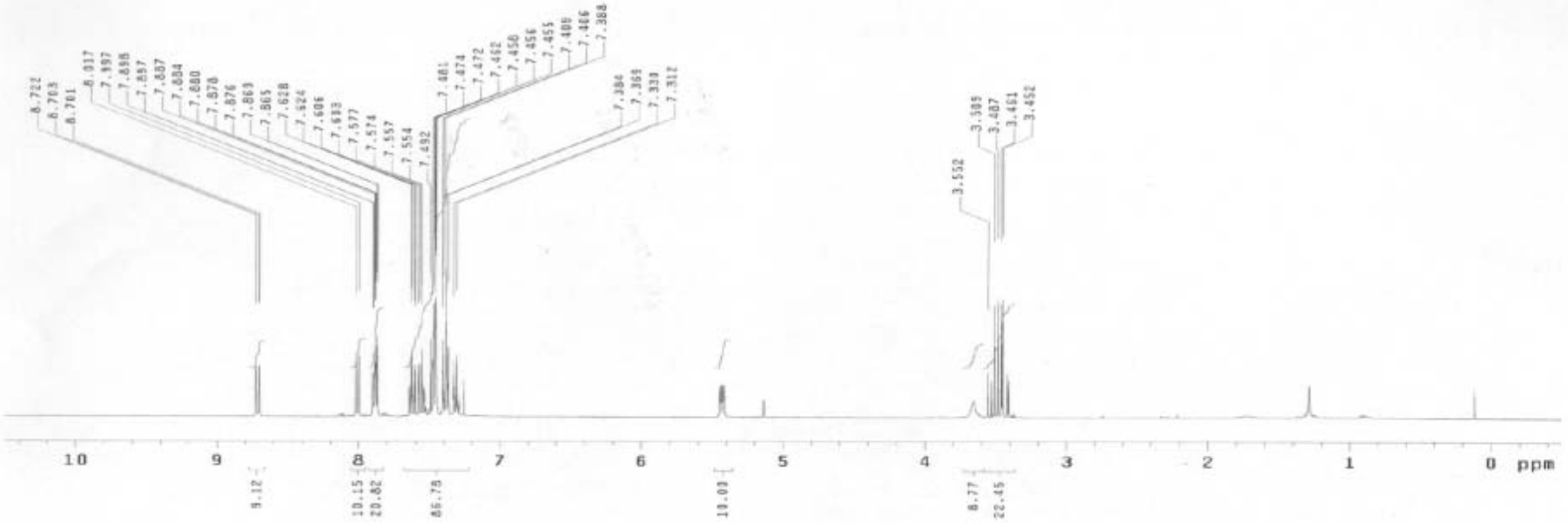
0907

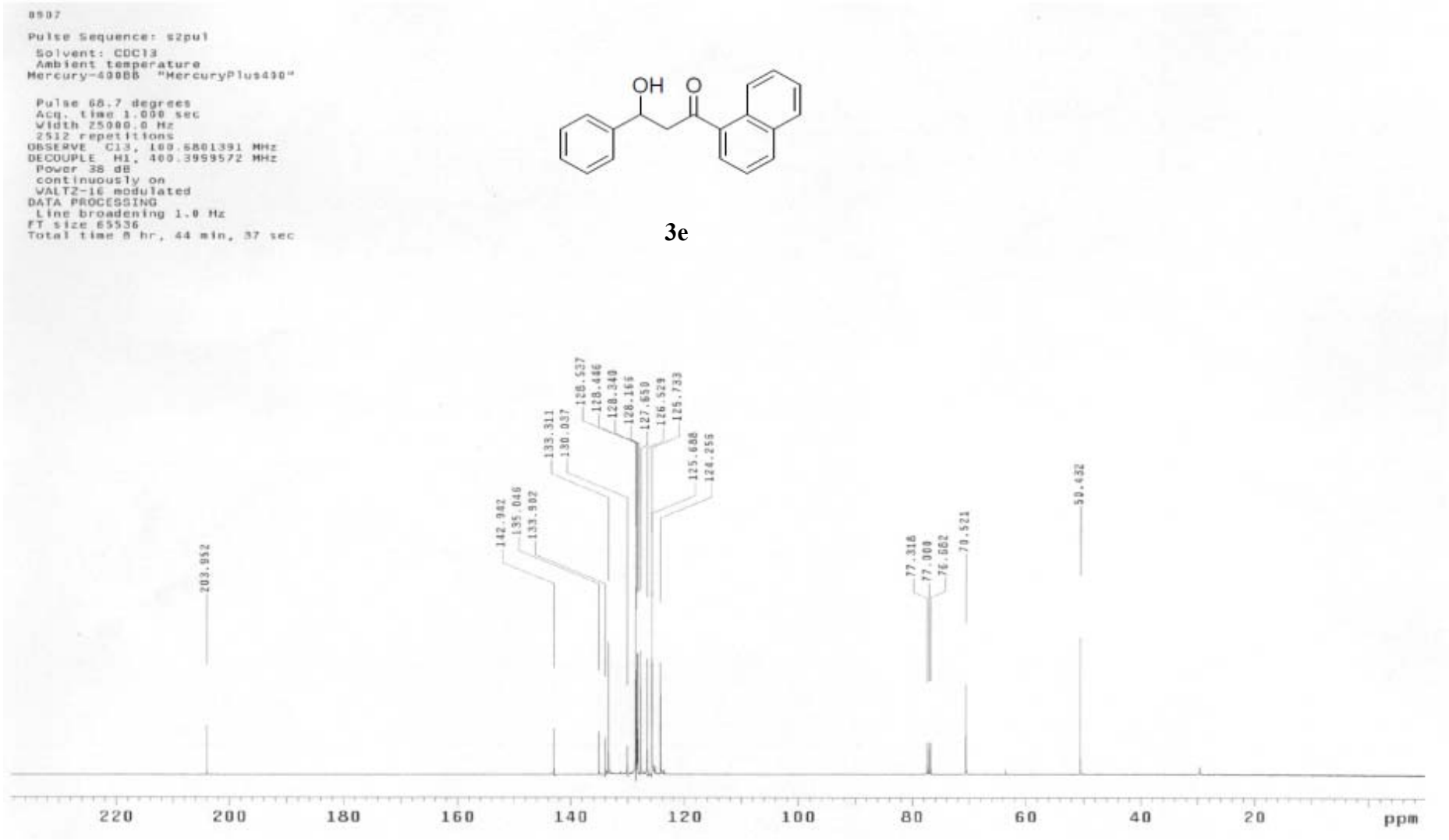
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 48.1 degrees
Acq. time 4.802 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978961 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 2 min, 33 sec

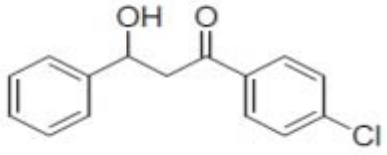


3e

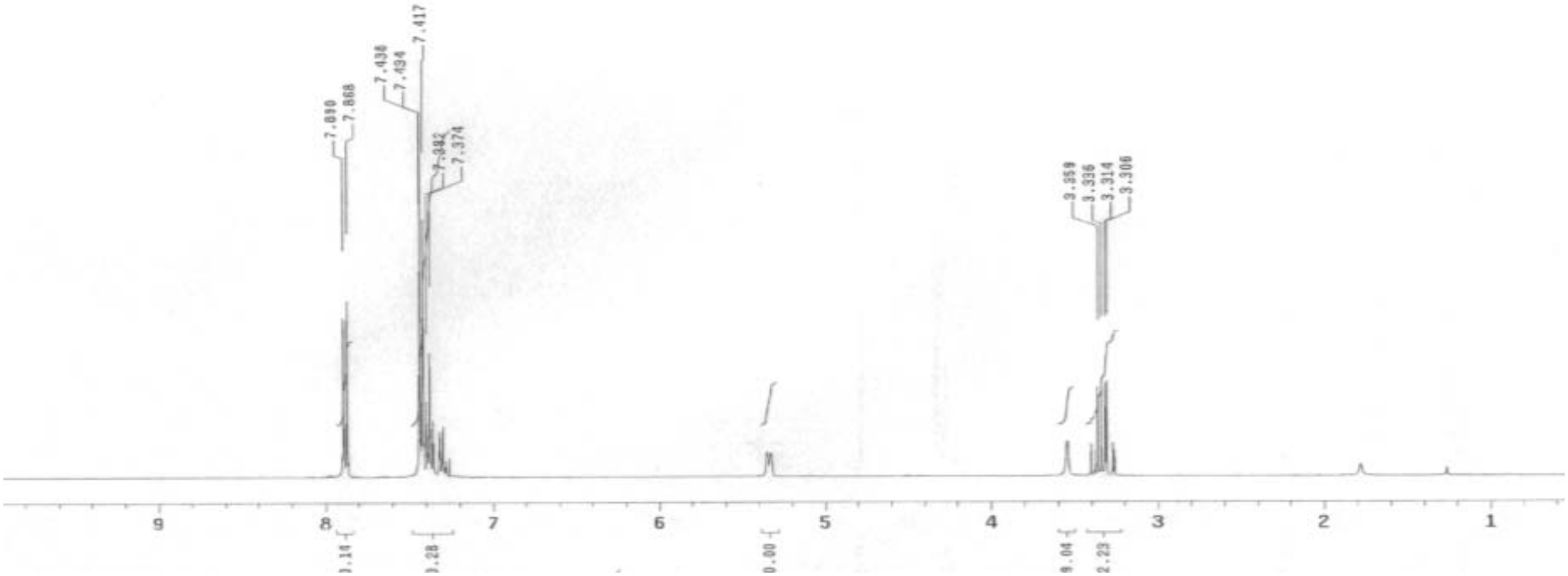


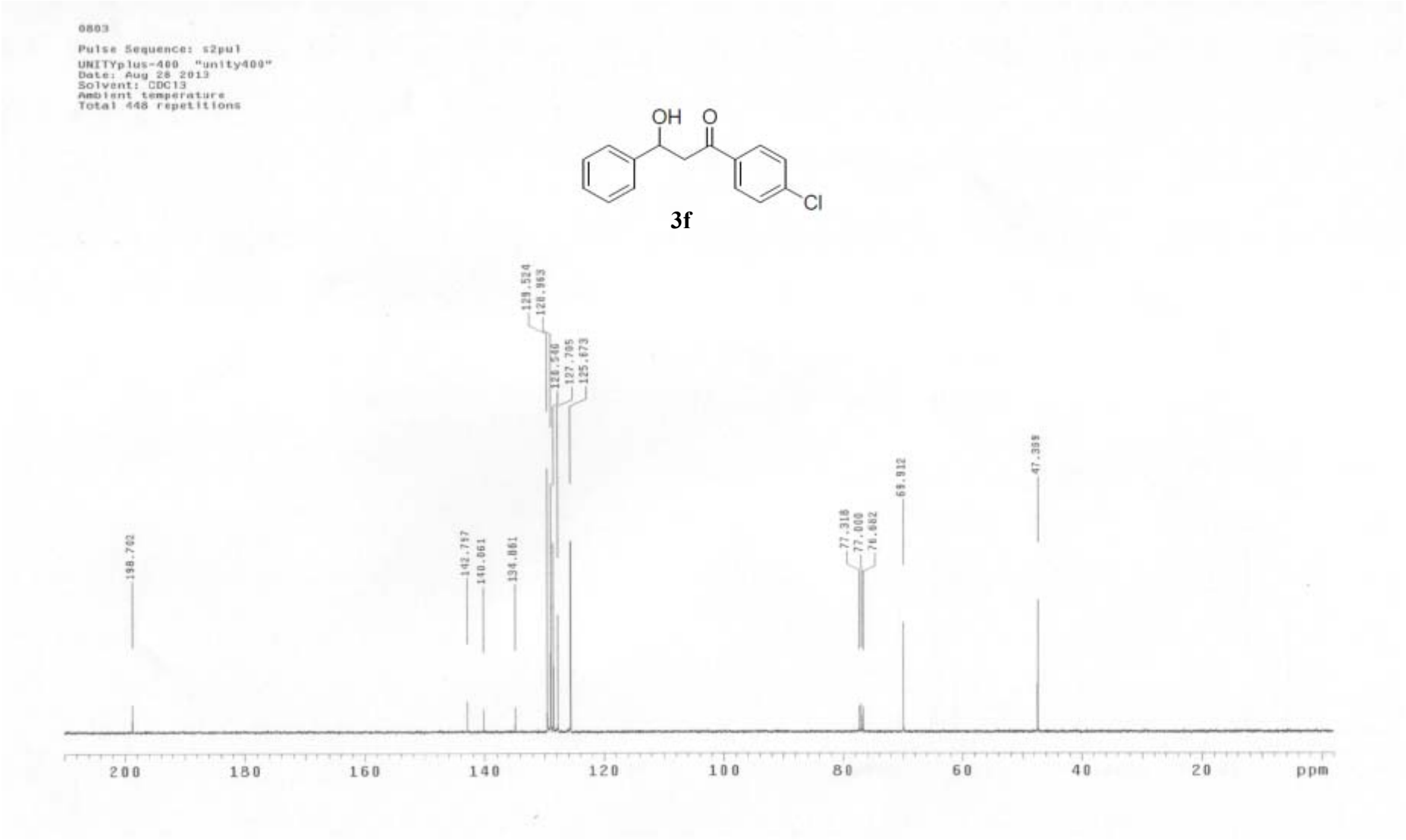

8507
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
2512 repetitions
OBSERVE C13, 100.5801391 MHz
DECOUPLE H1, 400.3959572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 44 min, 37 sec
OH
O
3e
203.952
142.982
135.046
133.302
133.311
130.037
128.537
128.446
128.340
128.165
127.650
126.529
125.733
125.688
124.255
77.316
77.000
76.682
70.521
50.432
220
200
180
160
140
120
100
80
60
40
20
ppm

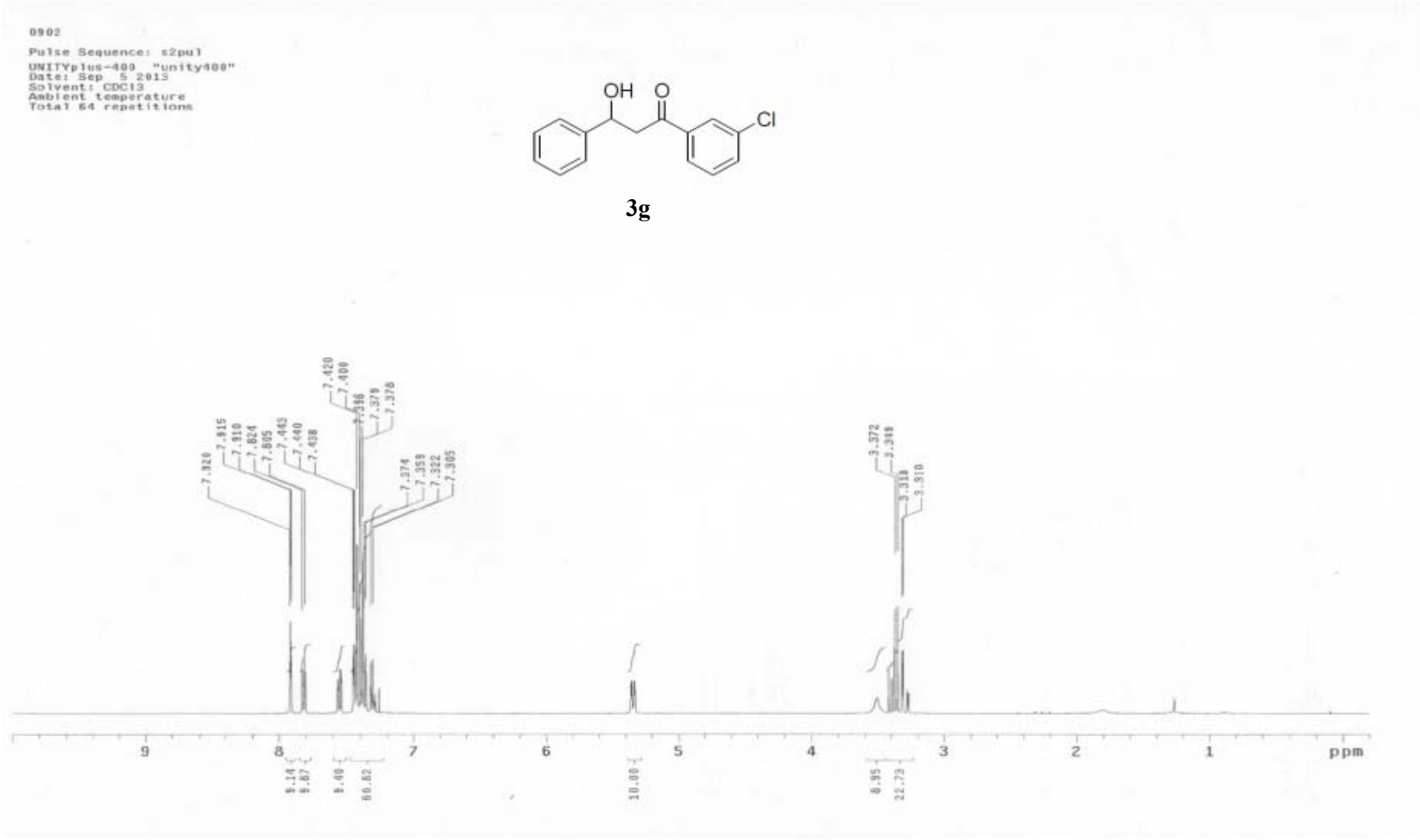
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions



3f




0803
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 448 repetitions
OH
O
Cl
3f
198.702
142.757
140.061
134.861
129.524
128.963
128.546
127.705
125.673
77.318
77.000
76.682
69.912
47.309
200
180
160
140
120
100
80
60
40
20
ppm

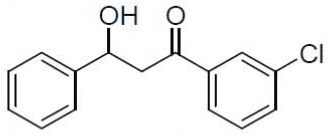

0902
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 64 repetitions
OH
O
Cl
7.420
7.400
7.396
7.379
7.378
7.374
7.359
7.322
7.305
7.920
7.915
7.810
7.824
7.805
7.443
7.440
7.438
3.372
3.349
3.318
3.310
9
8
7
6
5
4
3
2
1
ppm
9.14
9.87
8.40
60.82
10.00
8.95
22.73

3g

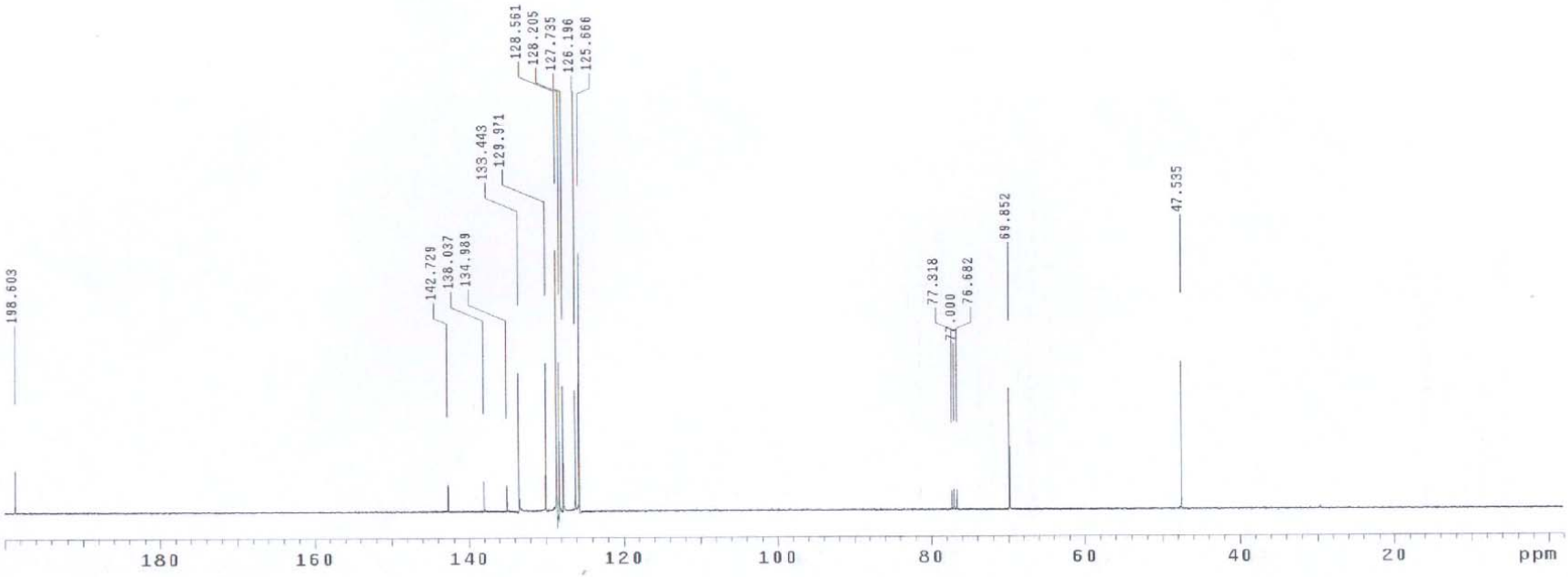
0902

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 1360 repetitions



3g



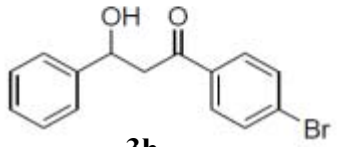

9826
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 32 repetitions
OH
O
Br
7.804
7.799
7.788
7.782
7.691
7.595
7.584
7.579
7.432
7.414
7.412
7.391
7.385
7.371
7.367
7.352
7.288
3.352
3.331
3.299
3.211
9
8
7
6
5
4
3
2
1
ppm
28.36
11.55
58.11
11.00
1.35
22.08

3h

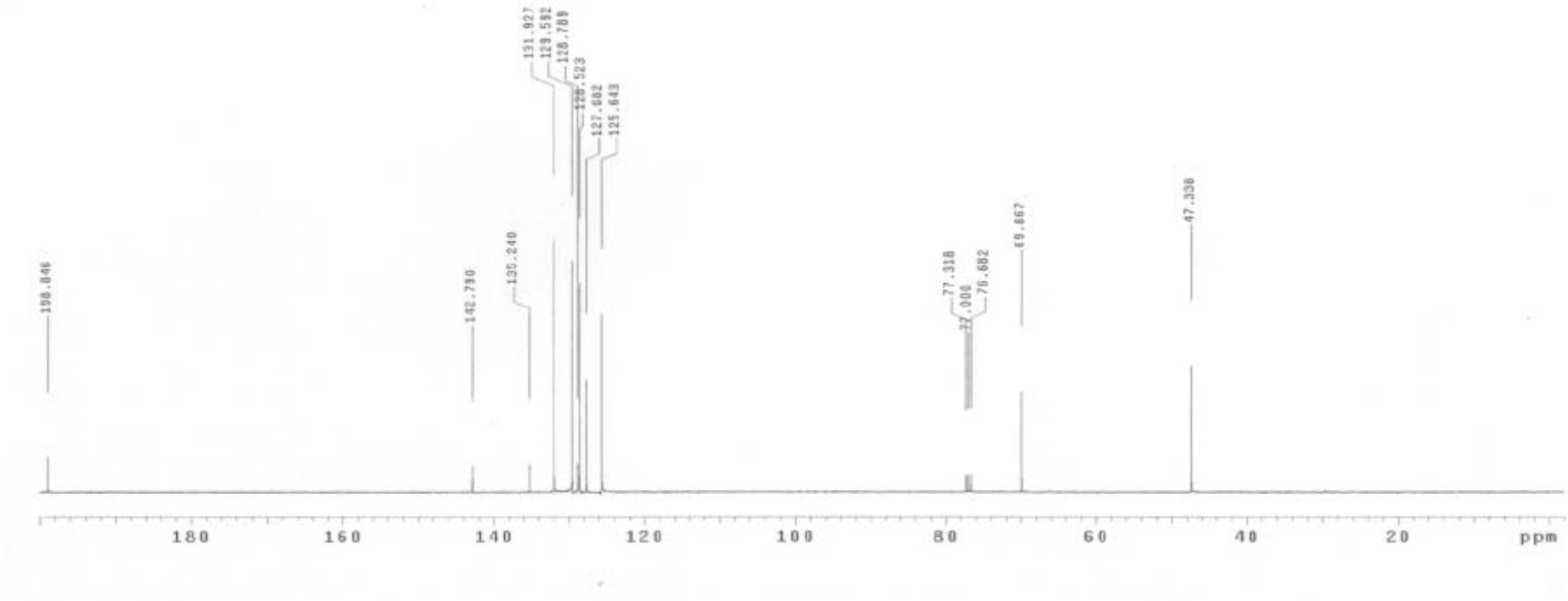
0826

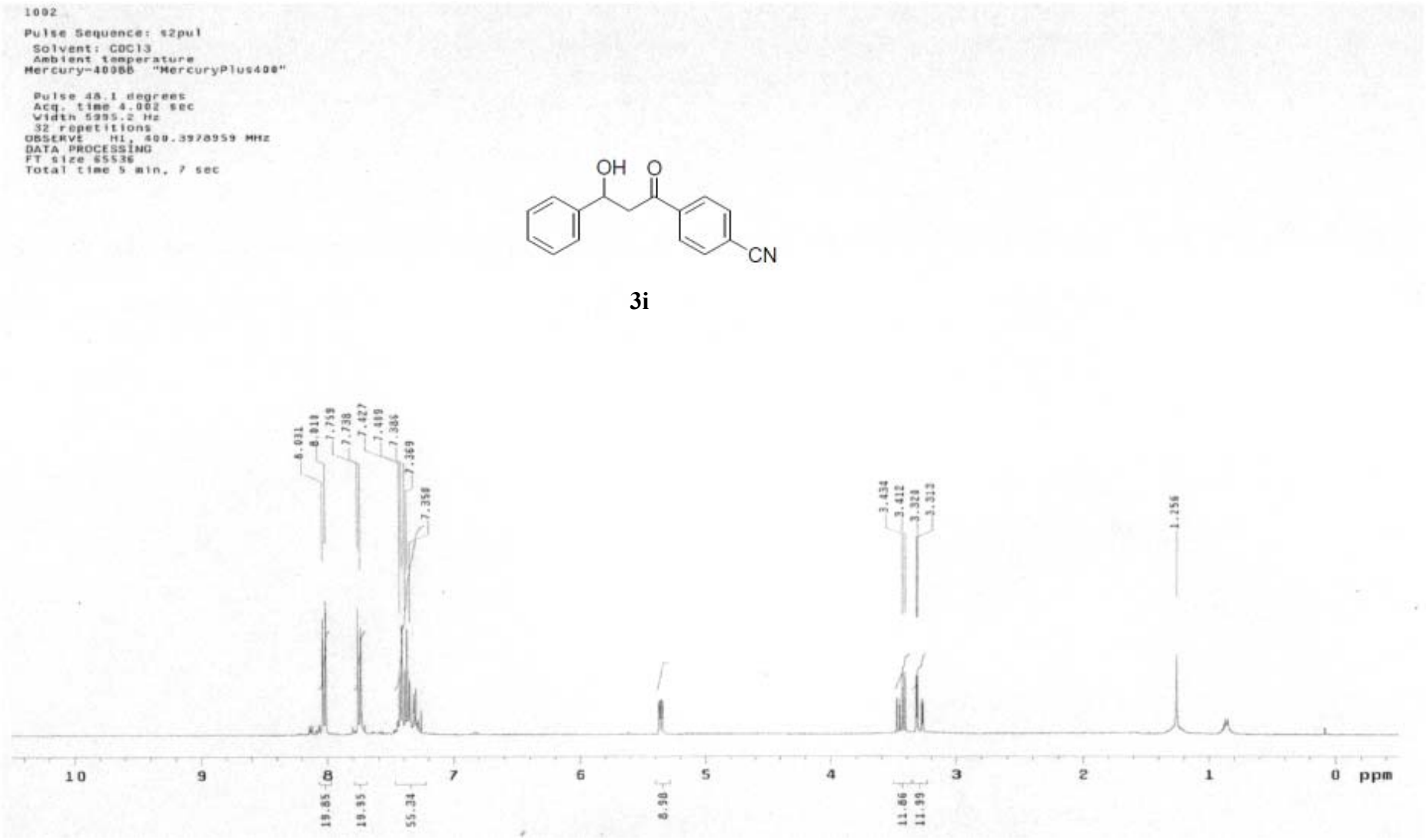
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 784 repetitions

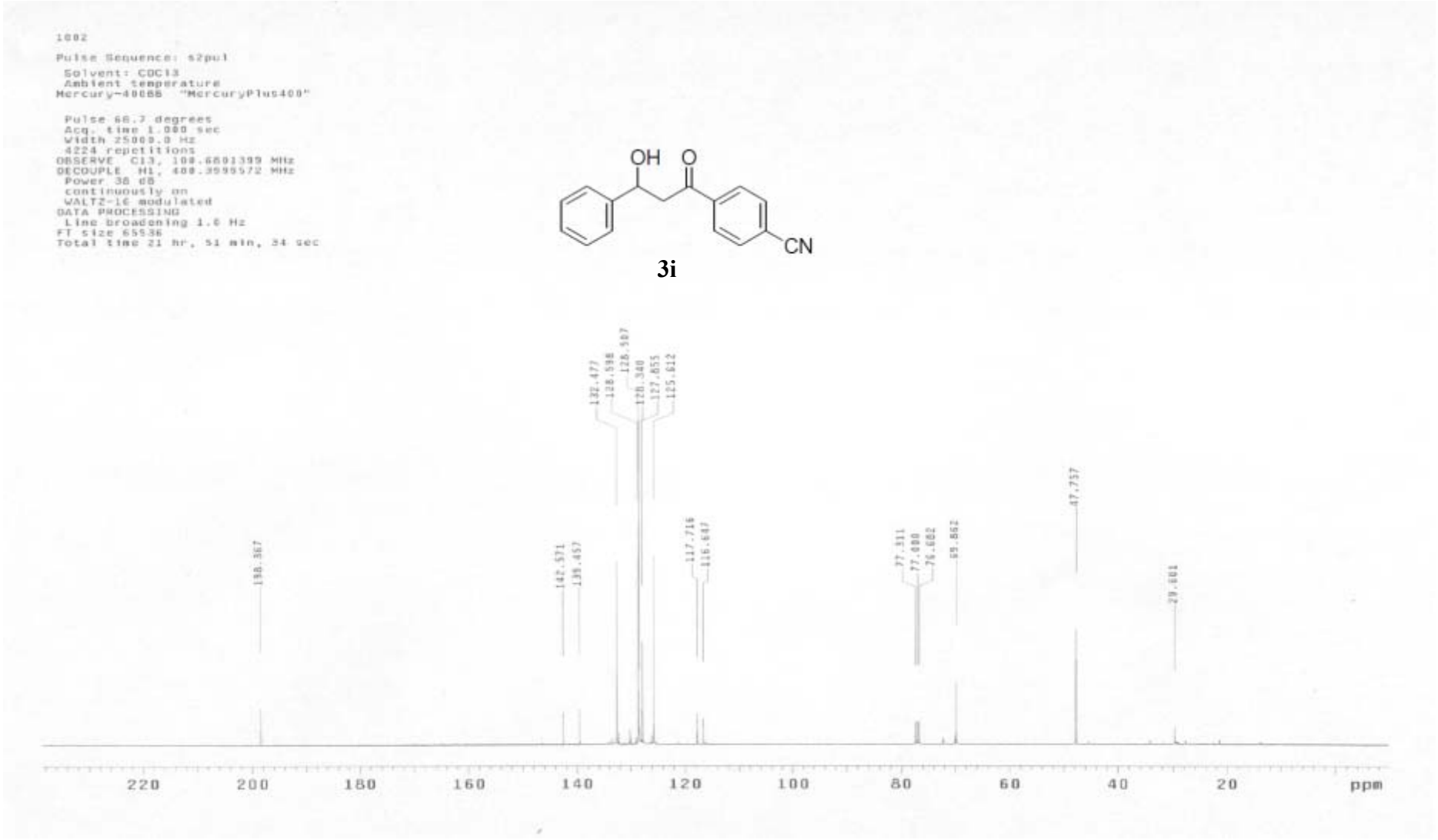


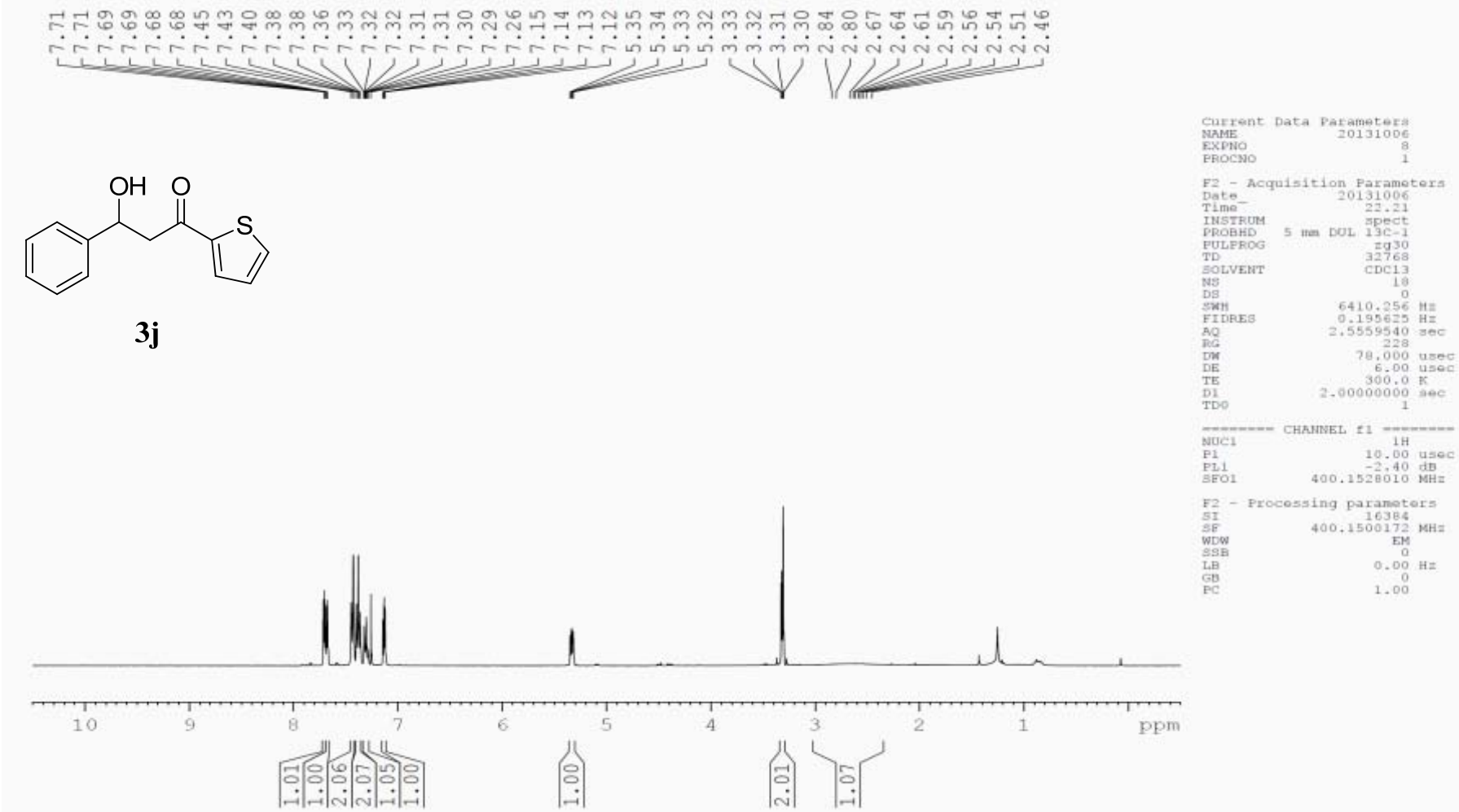
3h

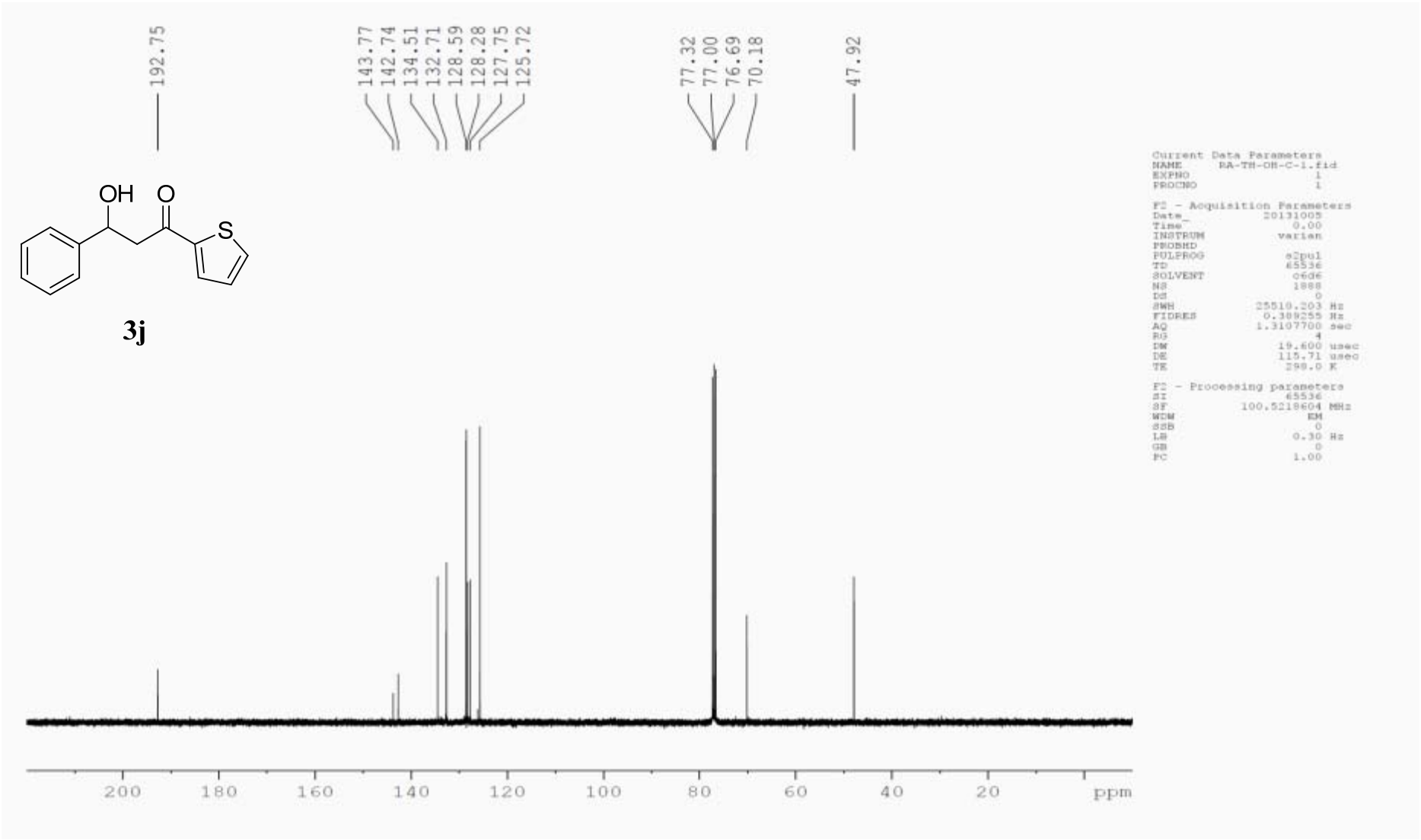


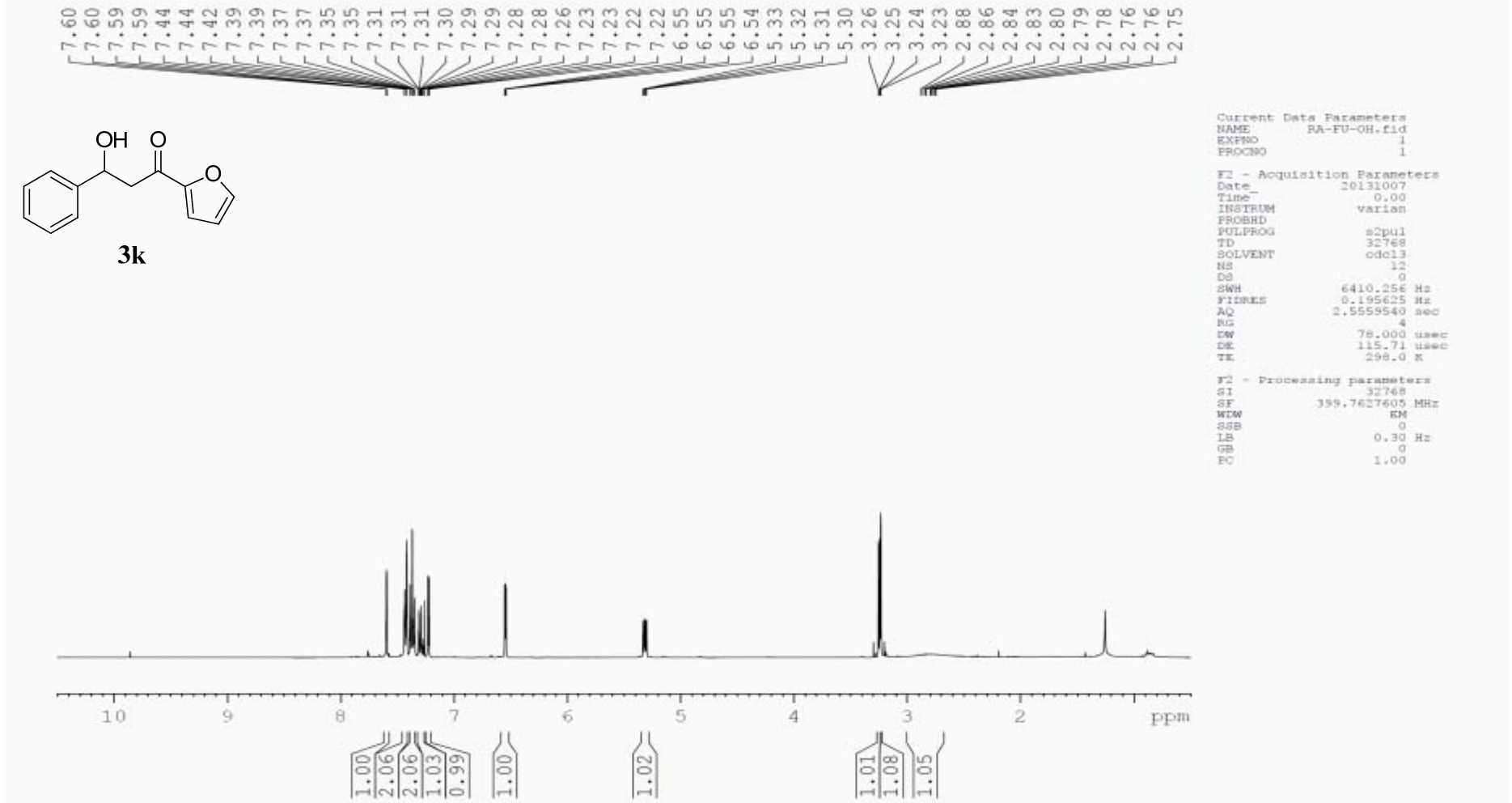

1092
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978959 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 7 sec
OH
O
CN
8.031
8.010
7.759
7.738
7.427
7.409
7.386
7.369
7.359
3.434
3.412
3.328
3.313
1.256
10
9
8
7
6
5
4
3
2
1
0
ppm
19.85
19.95
55.34
8.90
11.86
11.99

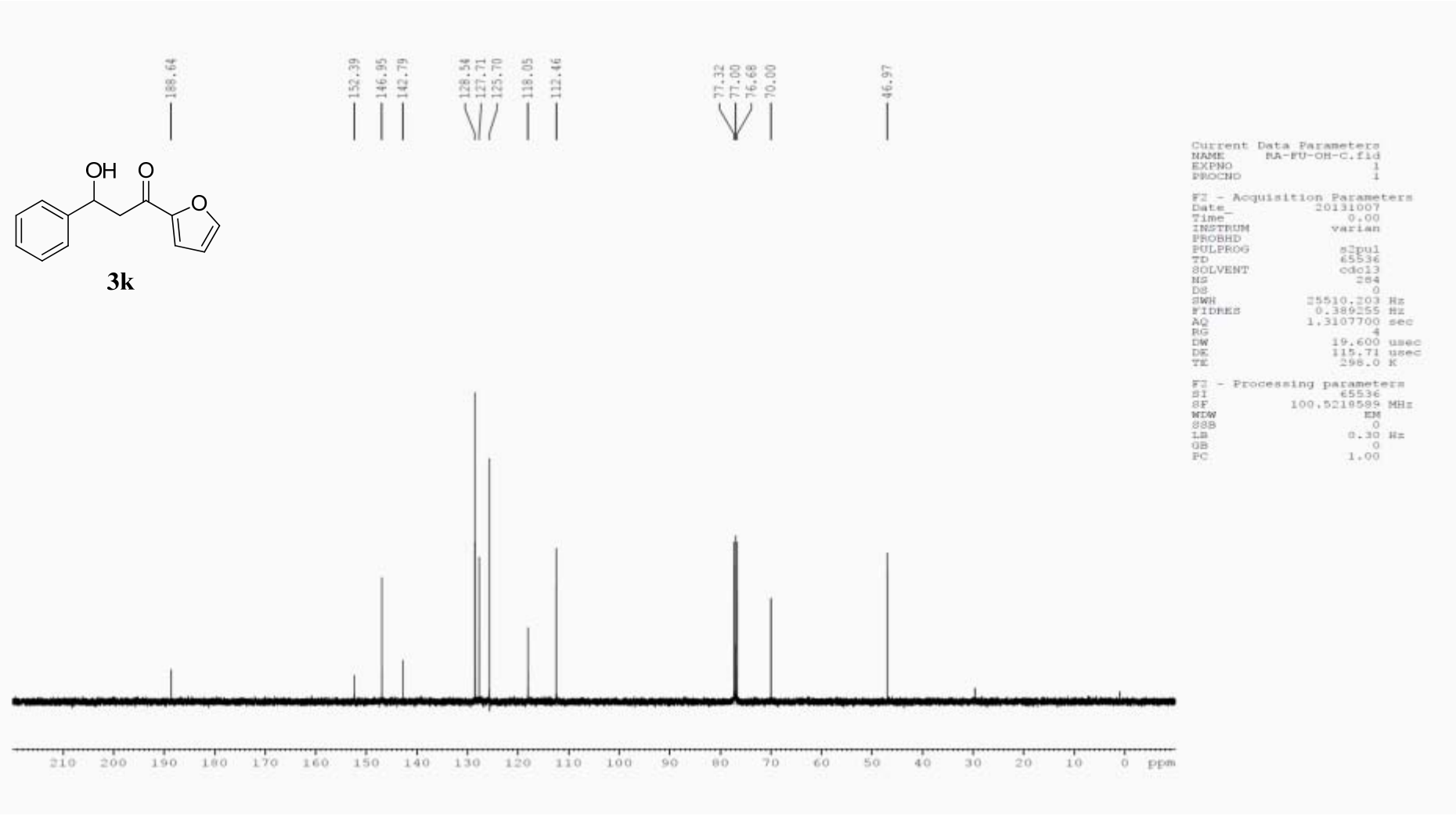
3i

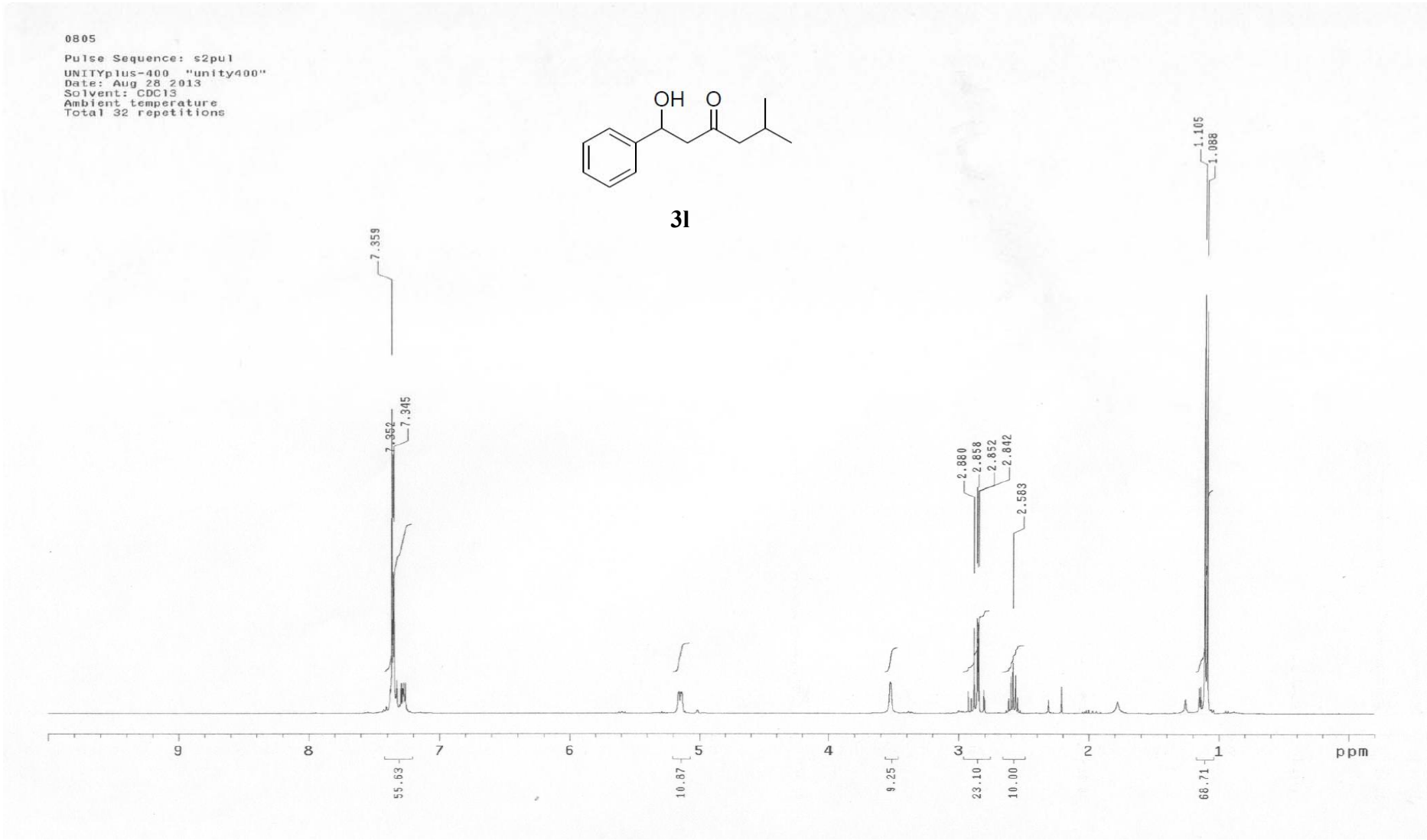

1002
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 66.7 degrees
Acq. time 1.000 sec
Width 25069.8 Hz
4224 repetitions
OBSERVE C13, 100.6801399 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.6 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec
OH
O
CN
3i
138.367
142.571
139.457
132.477
128.598
128.567
128.340
127.855
125.612
117.716
116.647
77.311
77.000
76.682
69.862
47.737
29.681
220
200
180
160
140
120
100
80
60
40
20
ppm


7.71
7.71
7.69
7.69
7.68
7.68
7.45
7.43
7.40
7.38
7.38
7.36
7.33
7.32
7.32
7.31
7.31
7.30
7.29
7.26
7.15
7.14
7.13
7.12
5.35
5.34
5.33
5.32
3.33
3.32
3.31
3.30
2.84
2.80
2.67
2.64
2.61
2.59
2.56
2.54
2.51
2.46
OH
O
S
3j
Current Data Parameters
NAME 20131006
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131006
Time 22.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 10
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 228
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.01
1.00
2.06
2.07
1.05
1.00
1.00
2.01
1.07

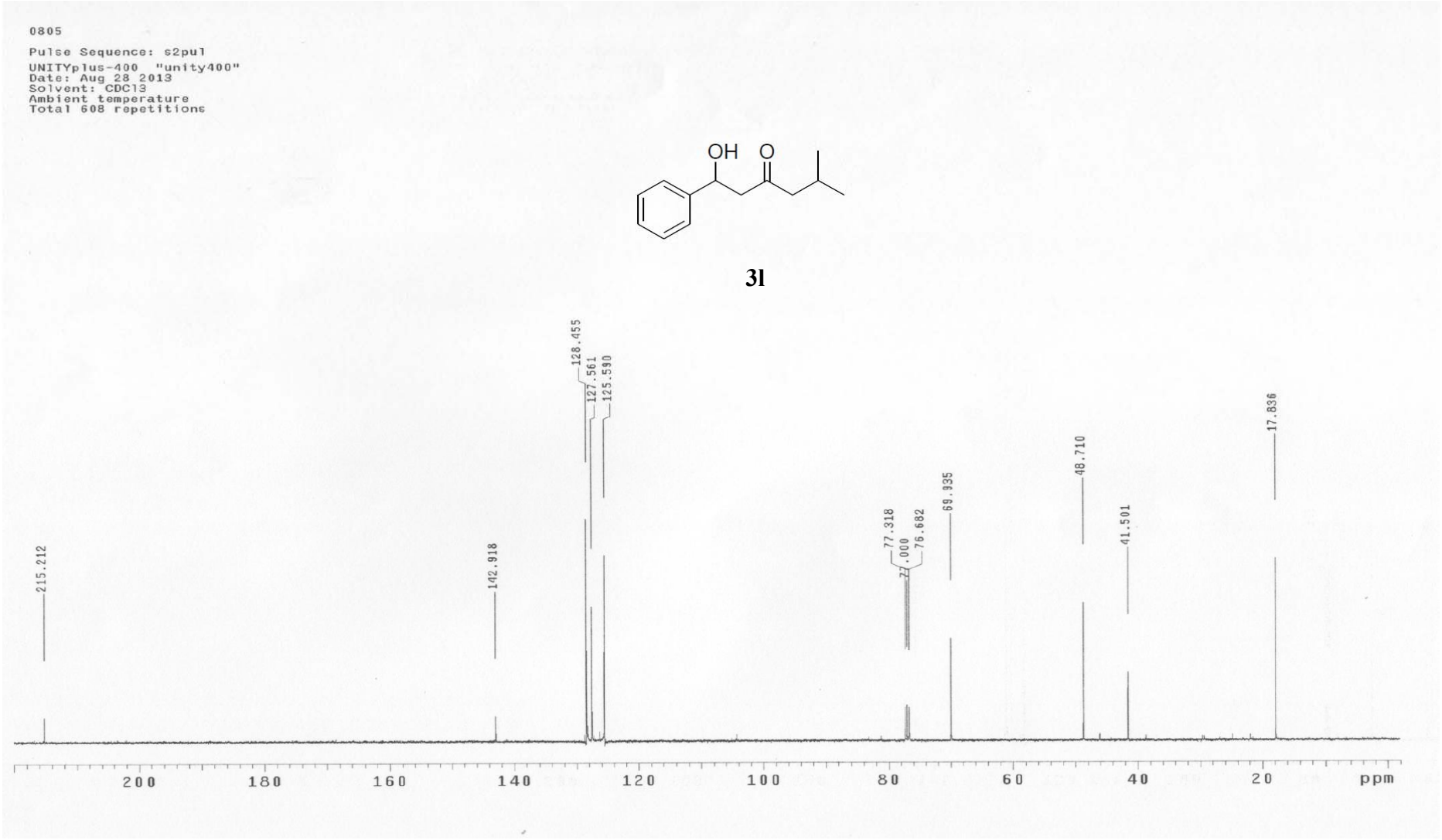

192.75
143.77
142.74
134.51
132.71
128.59
128.28
127.75
125.72
77.32
77.00
76.69
70.18
47.92
OH
O
S
3j
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME RA-TH-OH-C-1.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131005
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 1888
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218604 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


7.60 7.60 7.59 7.59 7.44 7.44 7.42 7.39 7.39 7.37 7.37 7.35 7.35 7.31 7.31 7.31 7.30 7.29 7.29 7.28 7.28 7.26 7.23 7.23 7.22 7.22 6.55 6.55 6.55 6.54 5.33 5.32 5.31 5.30 3.26 3.25 3.24 3.23 2.88 2.86 2.84 2.83 2.80 2.79 2.78 2.76 2.76 2.75
OH
O
O
3k
Current Data Parameters
NAME RA-FU-OH.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131007
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 12
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627605 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 ppm
1.00 2.06 2.06 1.03 0.99 1.00 1.02 1.01 1.08 1.05

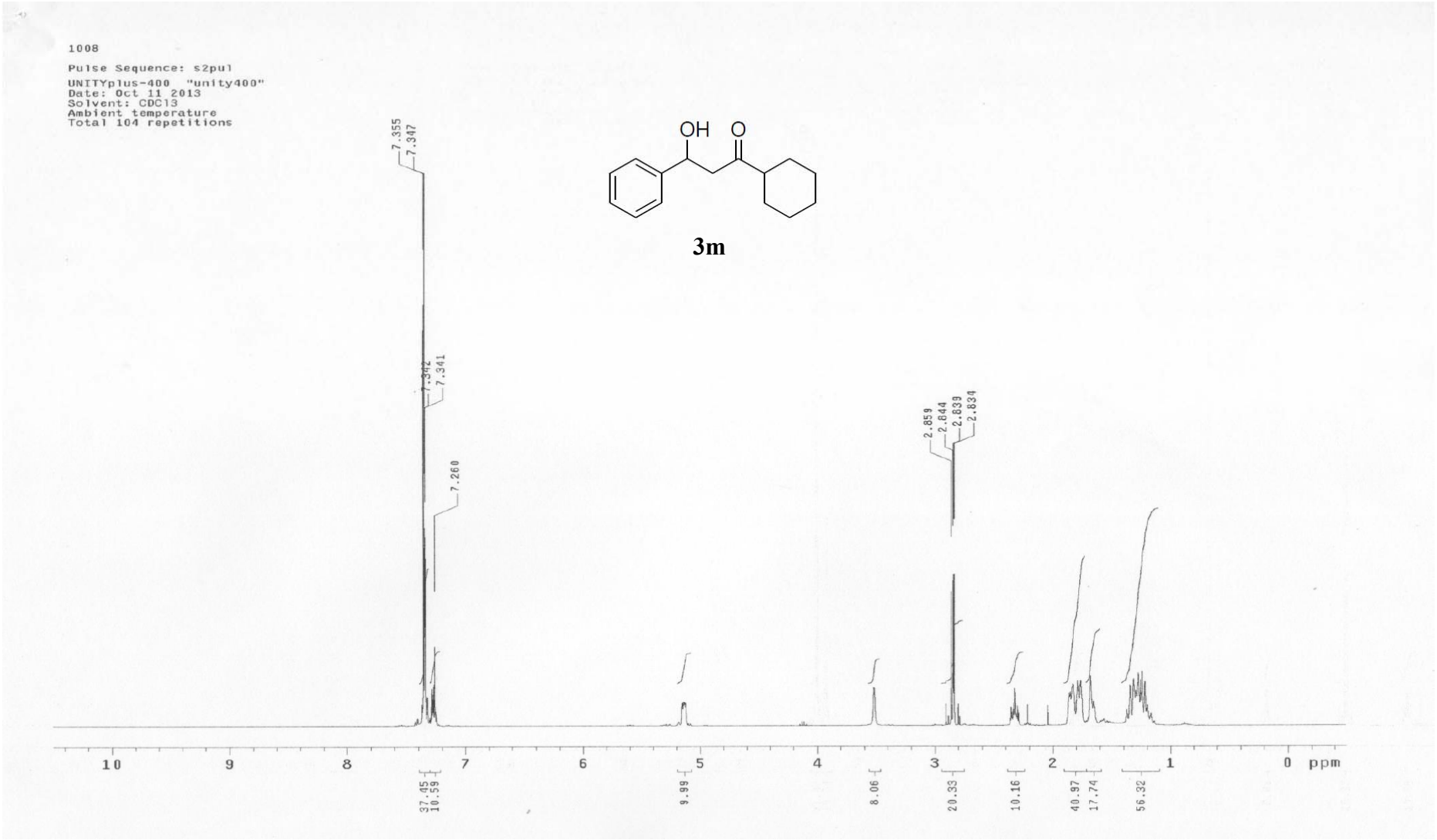

188.64
152.39
146.95
142.79
128.54
127.71
125.70
118.05
112.46
77.32
77.00
76.68
70.00
46.97
OH
O
O
3k
Current Data Parameters
NAME RA-FU-OH-C.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131007
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 284
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218589 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


0805
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 32 repetitions
OH
O
7.359
7.352
7.345
2.880
2.858
2.852
2.842
2.583
1.105
1.088
9
8
7
6
5
4
3
2
1
ppm
55.63
10.87
9.25
23.10
10.00
68.71

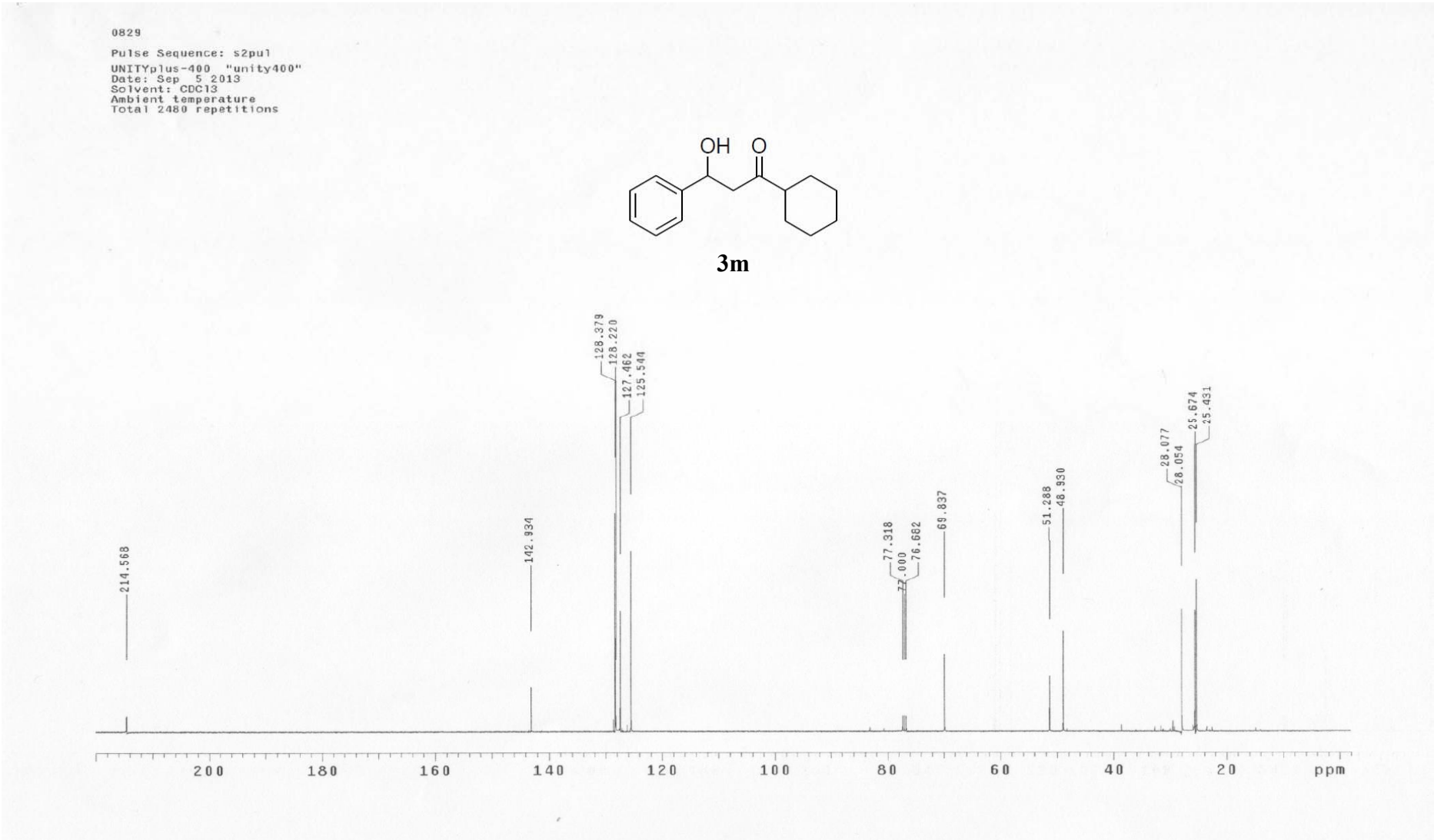
3l

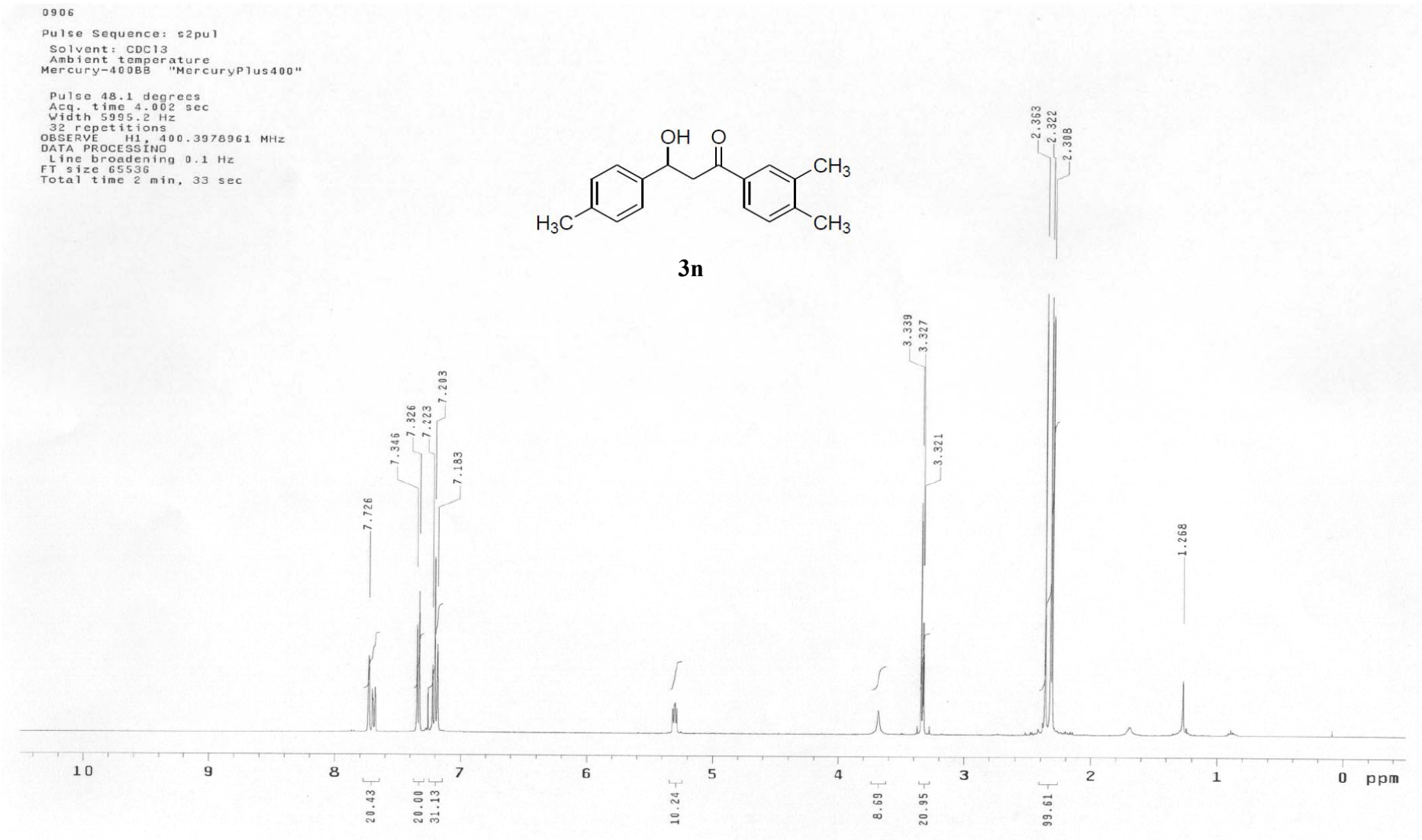

0805
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 28 2013
Solvent: CDCl3
Ambient temperature
Total 608 repetitions
OH
O
215.212
142.918
128.455
127.561
125.590
77.318
77.000
76.682
69.935
48.710
41.501
17.836
200
180
160
140
120
100
80
60
40
20
ppm

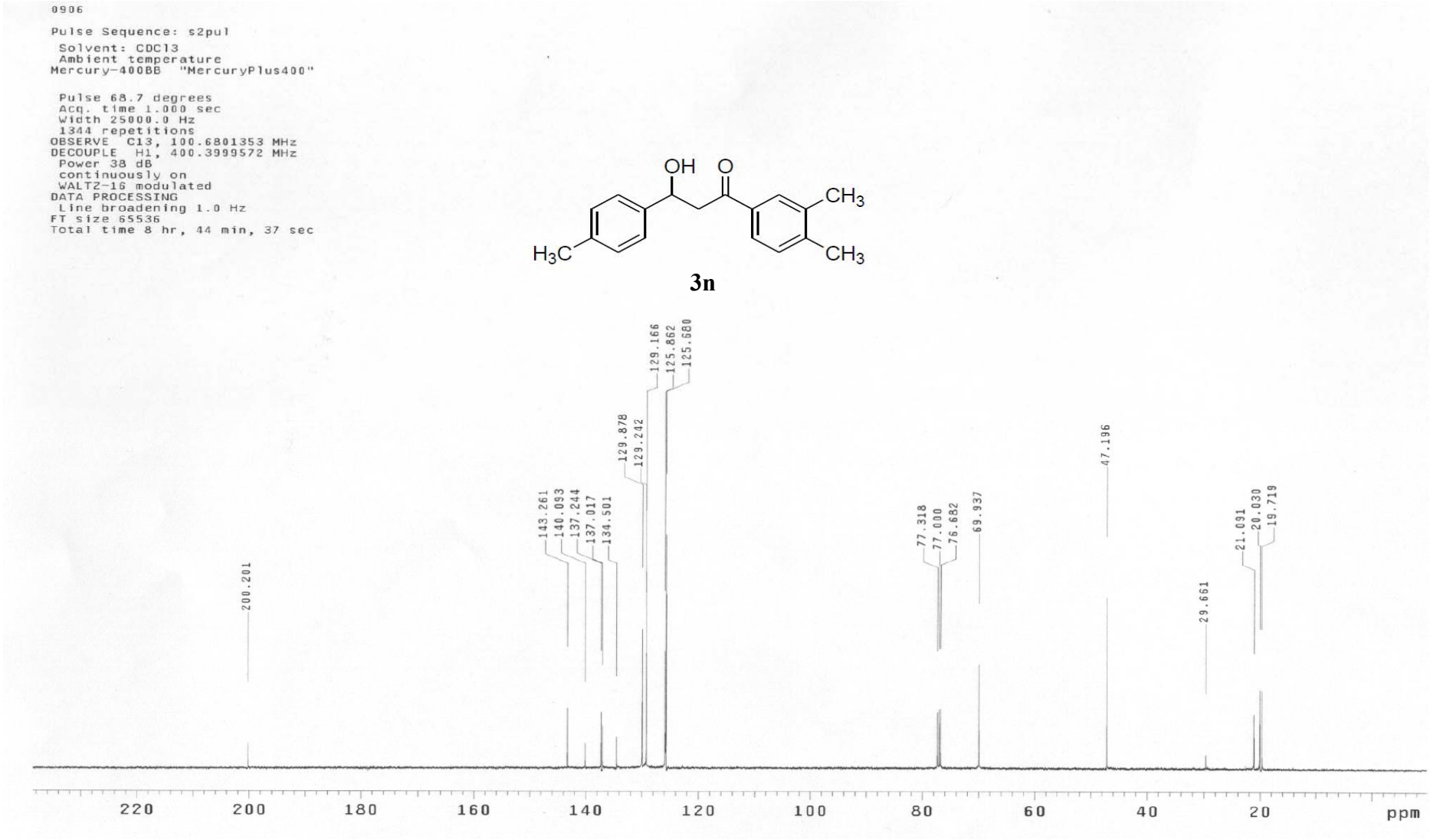
3l

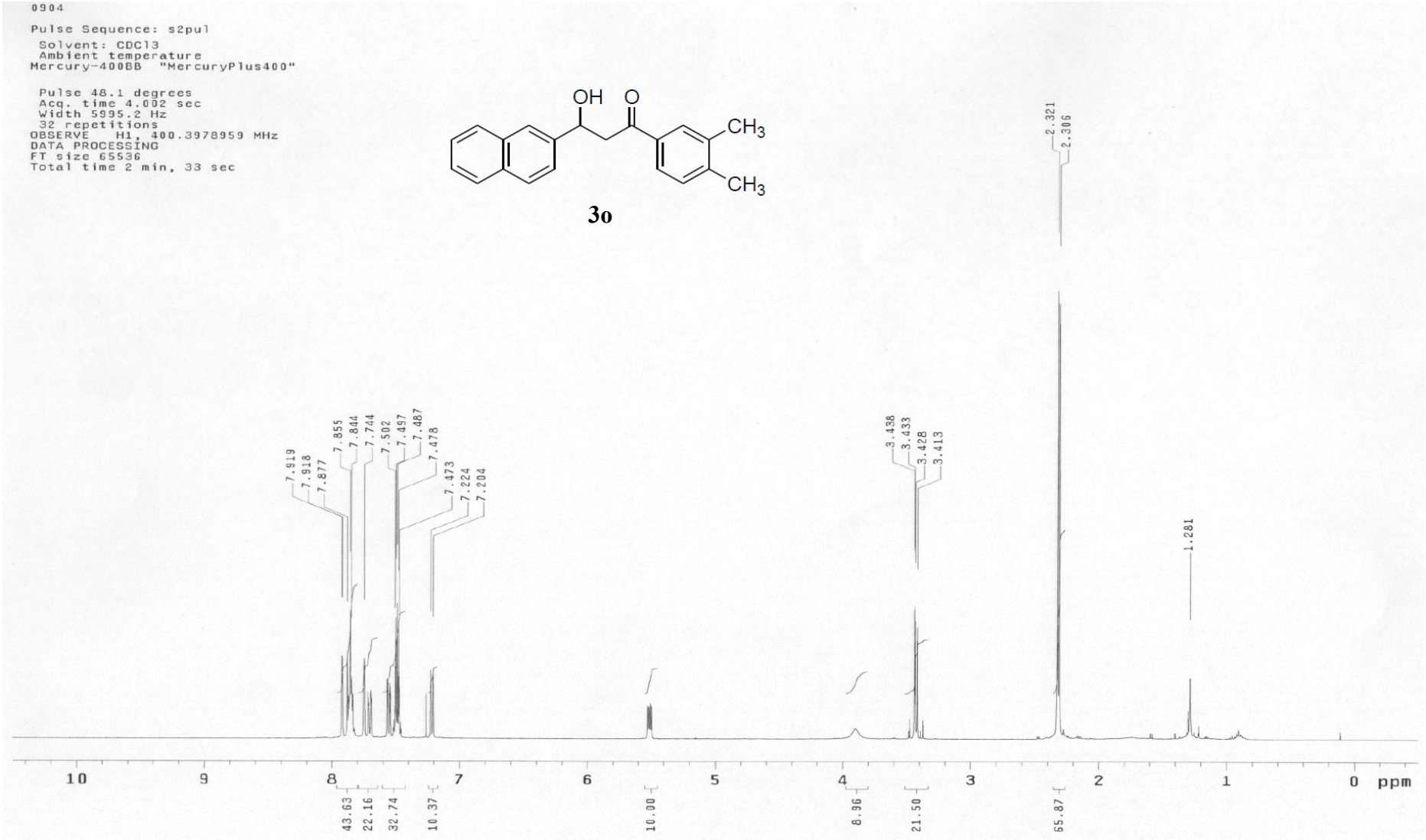

1008
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Oct 11 2013
Solvent: CDCl3
Ambient temperature
Total 104 repetitions
OH
O
7.355
7.347
7.342
7.341
7.260
2.859
2.844
2.839
2.834
10
9
8
7
6
5
4
3
2
1
0 ppm
37.45
10.59
9.99
8.06
20.33
10.16
40.97
17.74
56.32

3m


0829
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 2480 repetitions
OH
O
3m
214.568
142.934
128.379
128.220
127.462
125.544
77.318
77.000
76.682
69.837
51.288
48.930
28.077
28.054
25.674
25.431
200
180
160
140
120
100
80
60
40
20
ppm


0906
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978961 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 2 min, 33 sec
OH
O
CH3
H3C
CH3
3n
2.363
2.322
2.308
3.339
3.327
3.321
7.726
7.346
7.326
7.223
7.203
7.183
1.268
10
9
8
7
6
5
4
3
2
1
0 ppm
20.43
20.00
31.13
10.24
8.69
20.95
99.61


0906
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
1344 repetitions
OBSERVE C13, 100.6801353 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 44 min, 37 sec
OH
O
CH3
H3C
CH3
3n
200.201
143.261
140.053
137.244
137.017
134.501
129.878
129.242
129.166
125.862
125.680
77.318
77.000
76.682
69.937
47.196
29.661
21.091
20.030
19.719
220
200
180
160
140
120
100
80
60
40
20
ppm

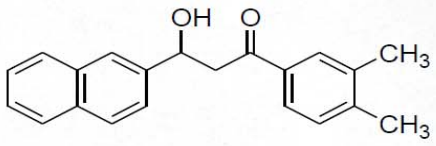

0904
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978959 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec
OH
O
CH3
CH3
7.919
7.918
7.877
7.855
7.844
7.744
7.502
7.497
7.487
7.478
7.473
7.224
7.204
3.438
3.433
3.428
3.413
2.321
2.306
1.281
10
9
8
7
6
5
4
3
2
1
0 ppm
43.63
22.16
32.74
10.37
10.00
8.96
21.50
65.87

3o

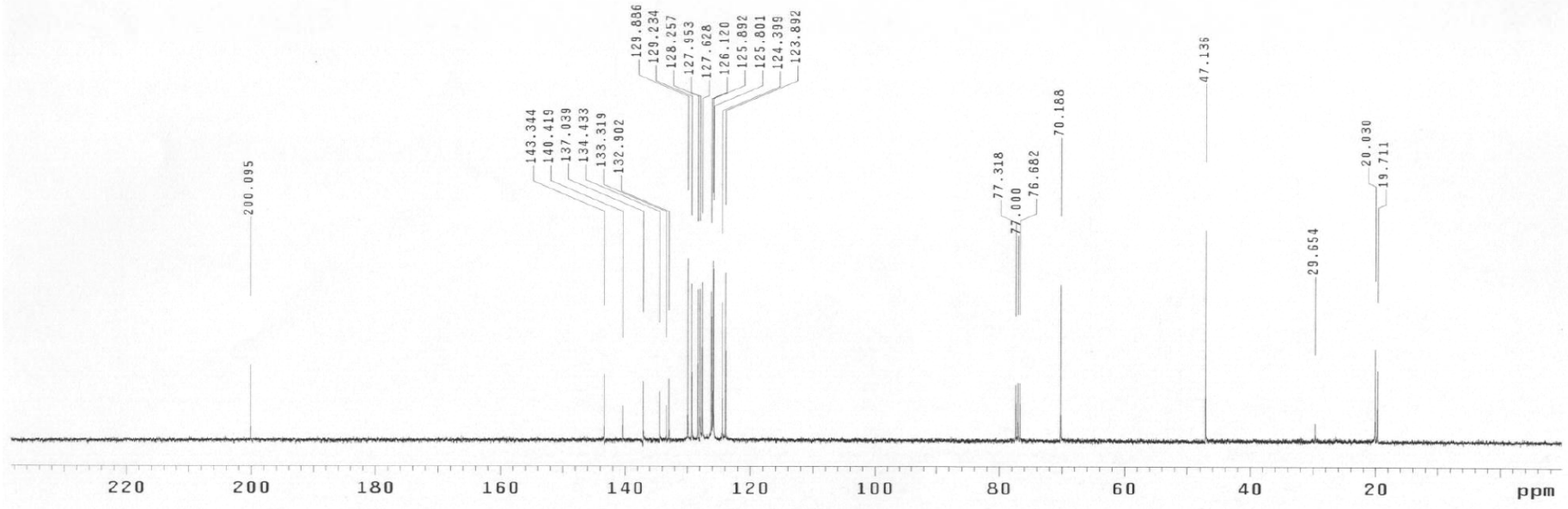
0904

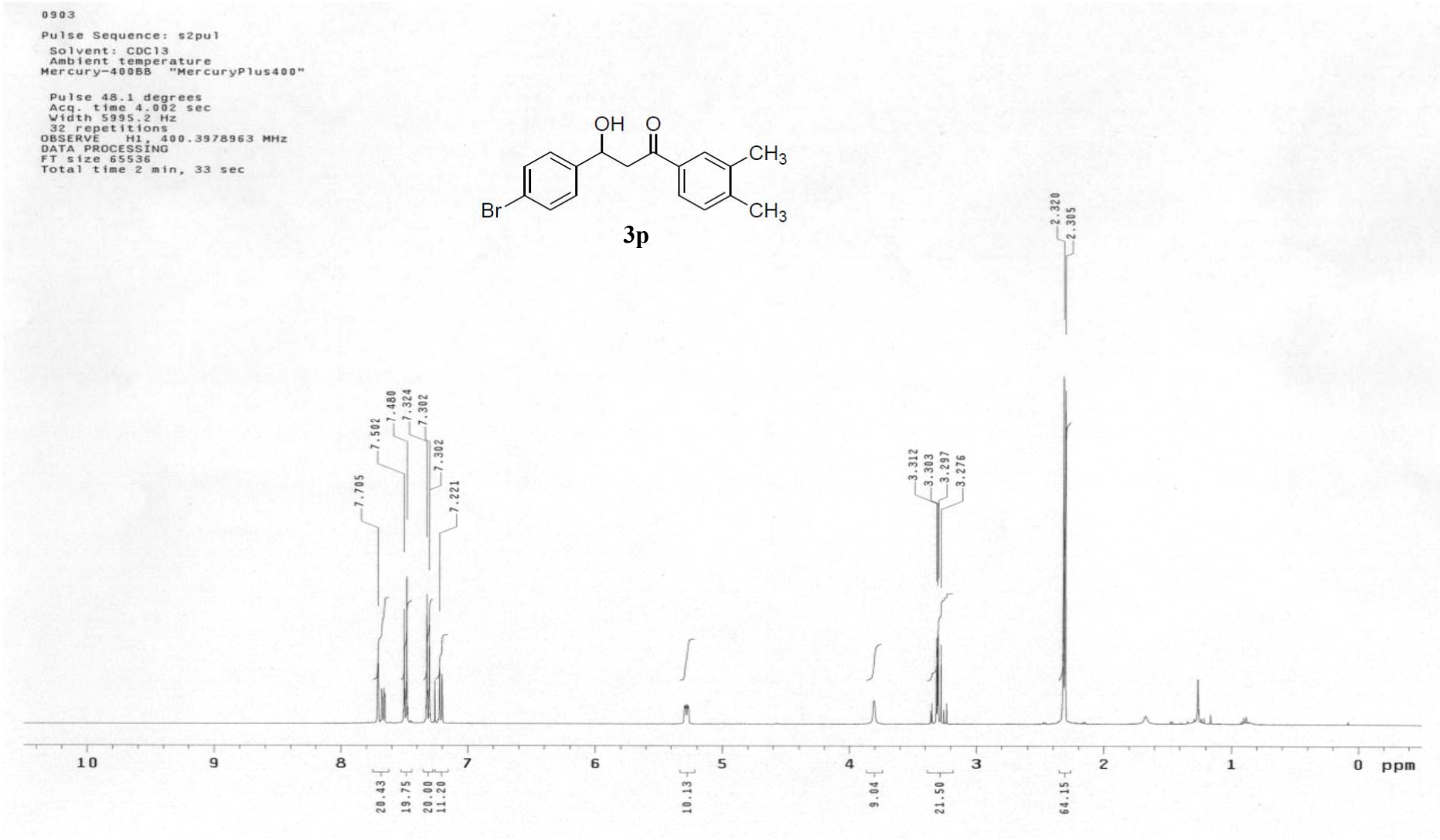
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

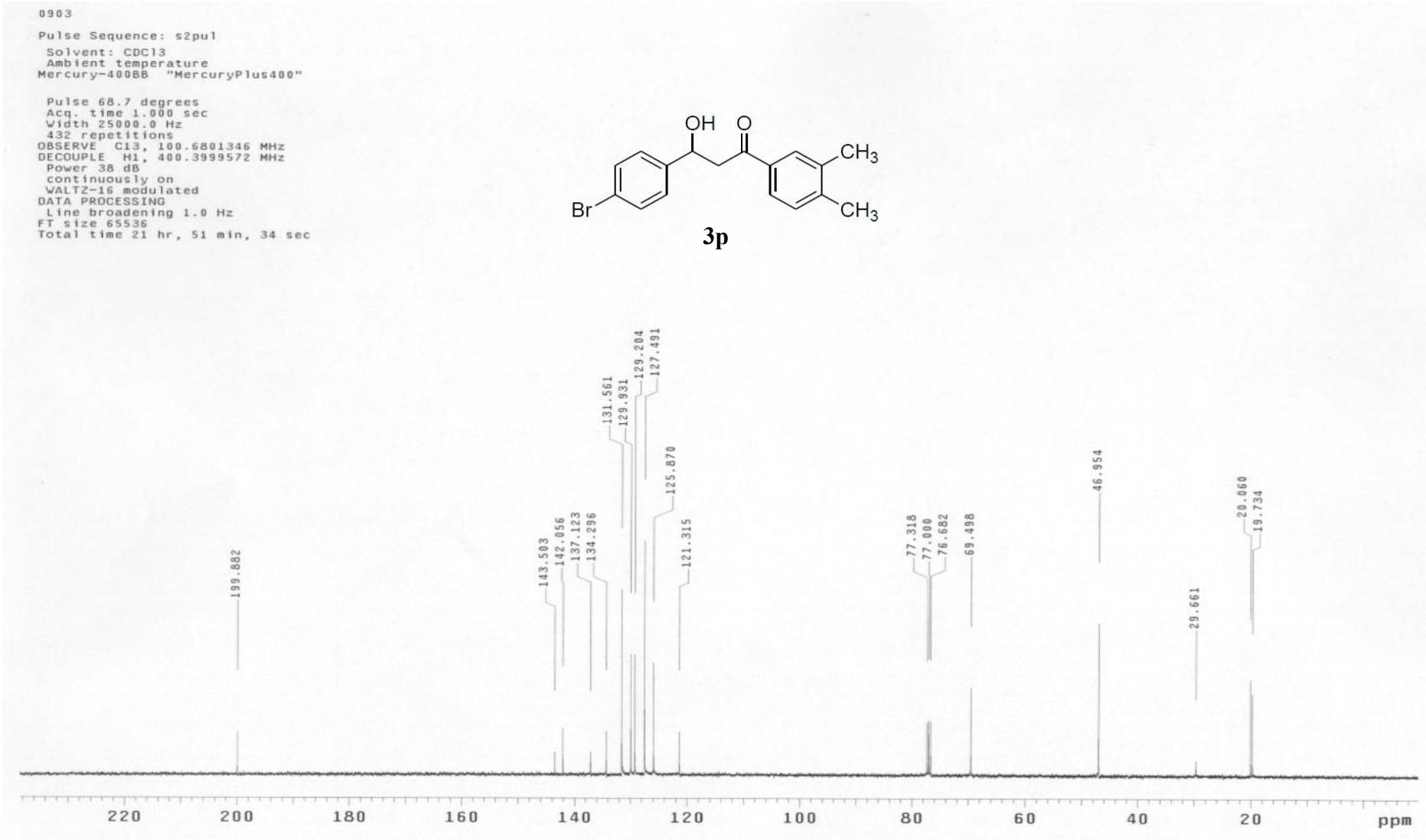
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
320 repetitions
OBSERVE C13, 100.6801376 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec

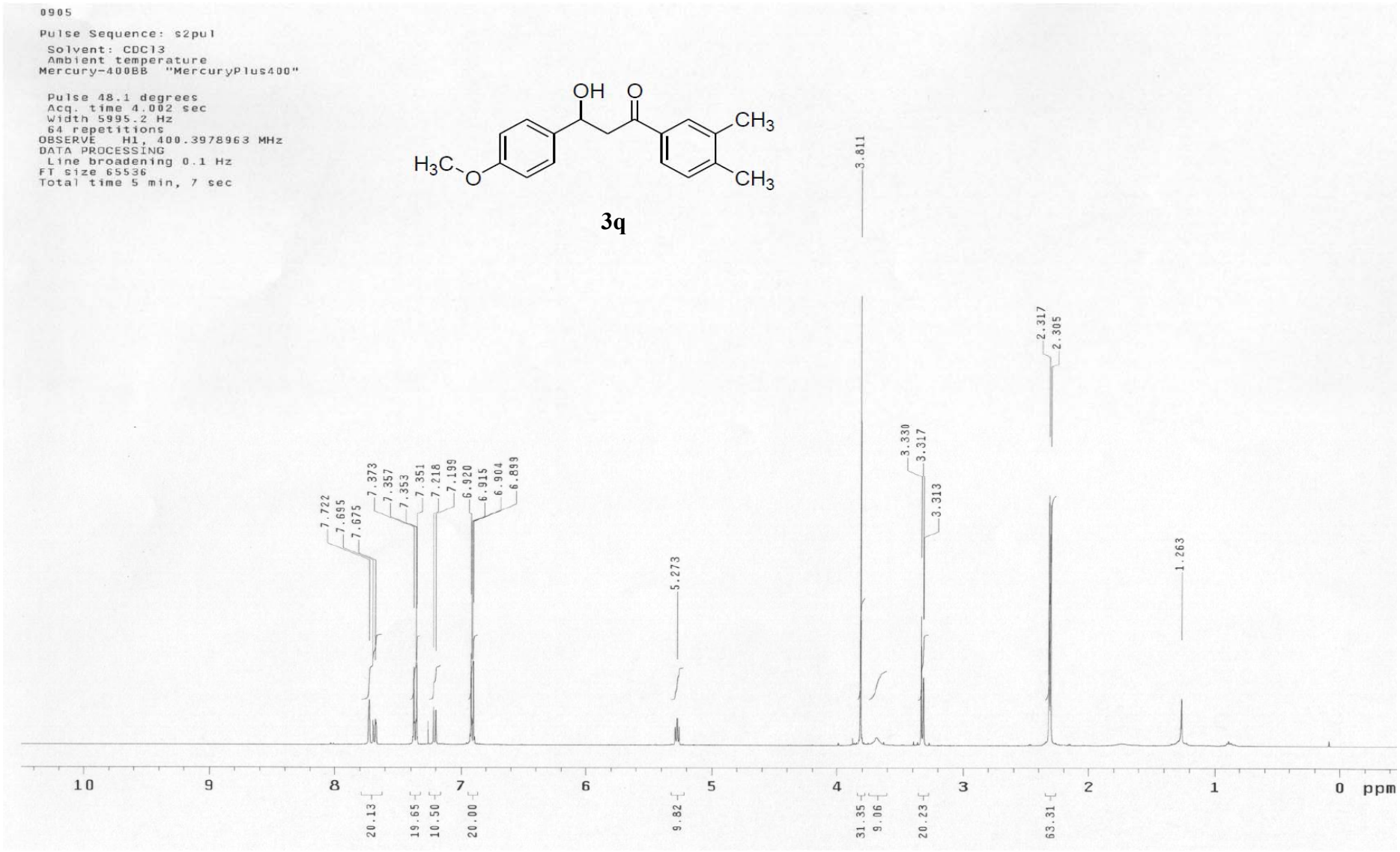


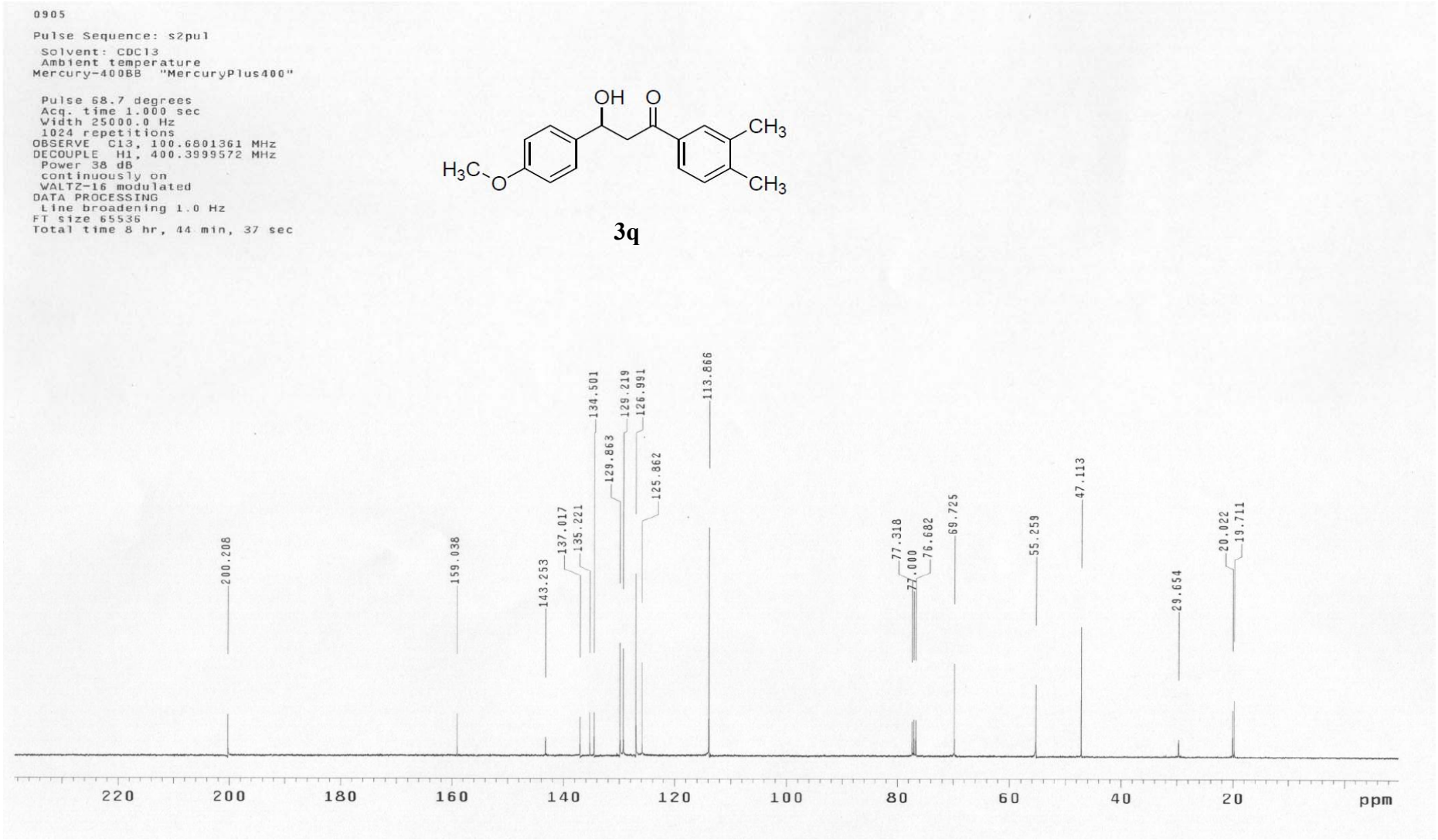
3o

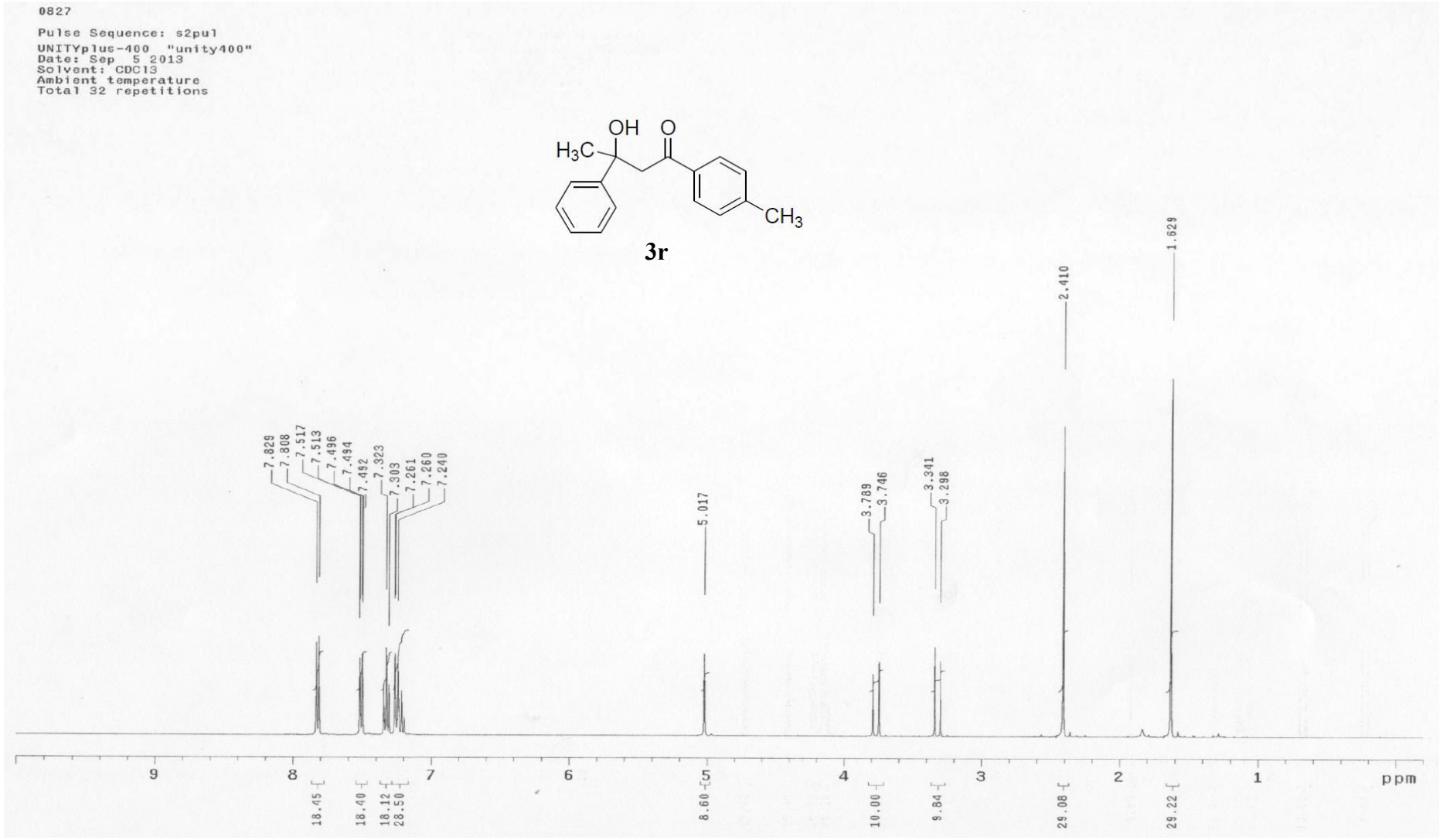


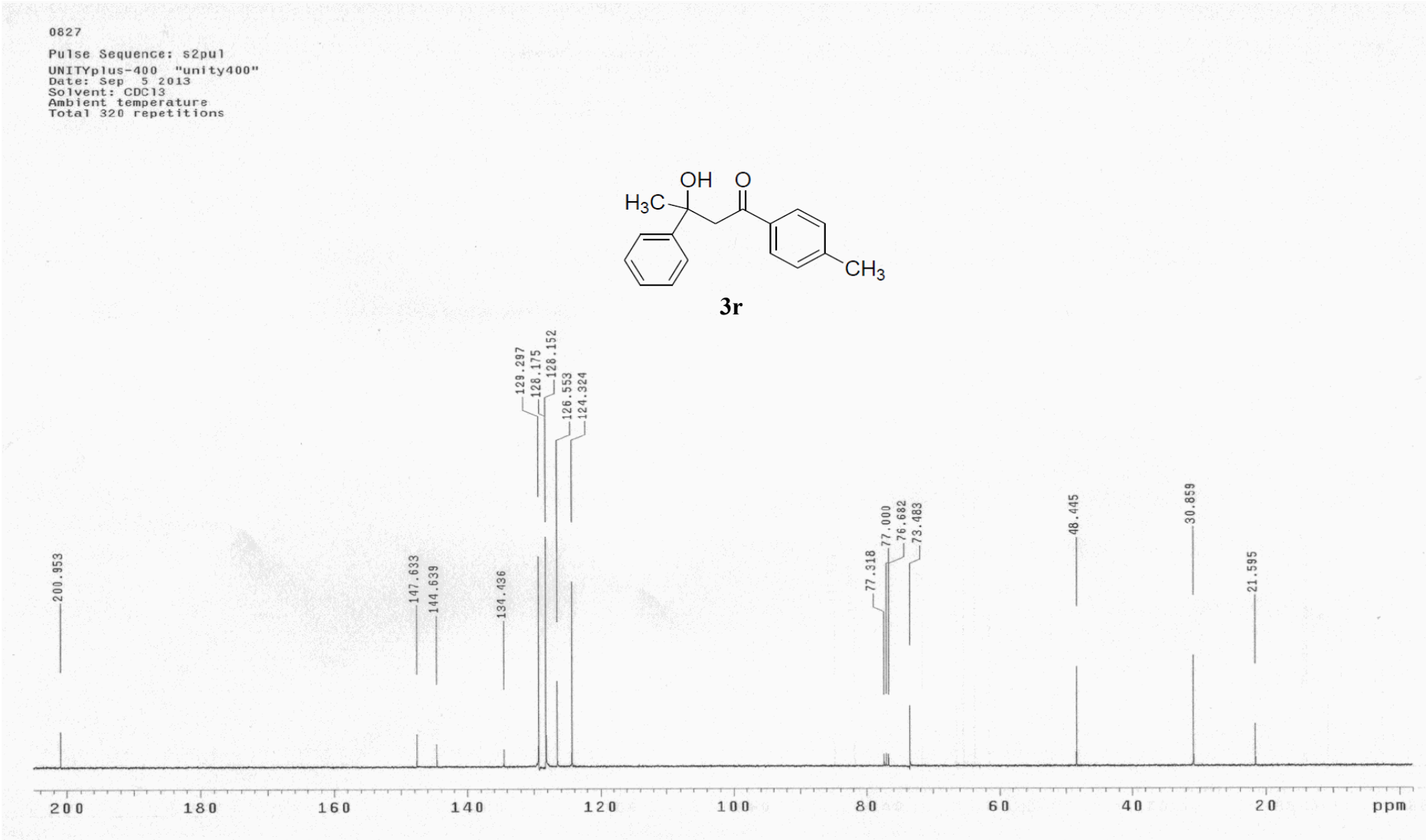

0903
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.3978963 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 33 sec
OH
O
CH3
CH3
Br
3p
7.705
7.502
7.480
7.324
7.302
7.302
7.221
3.312
3.303
3.297
3.276
2.320
2.305
10
9
8
7
6
5
4
3
2
1
0 ppm
20.43
19.75
20.00
11.20
10.13
9.04
21.50
64.15

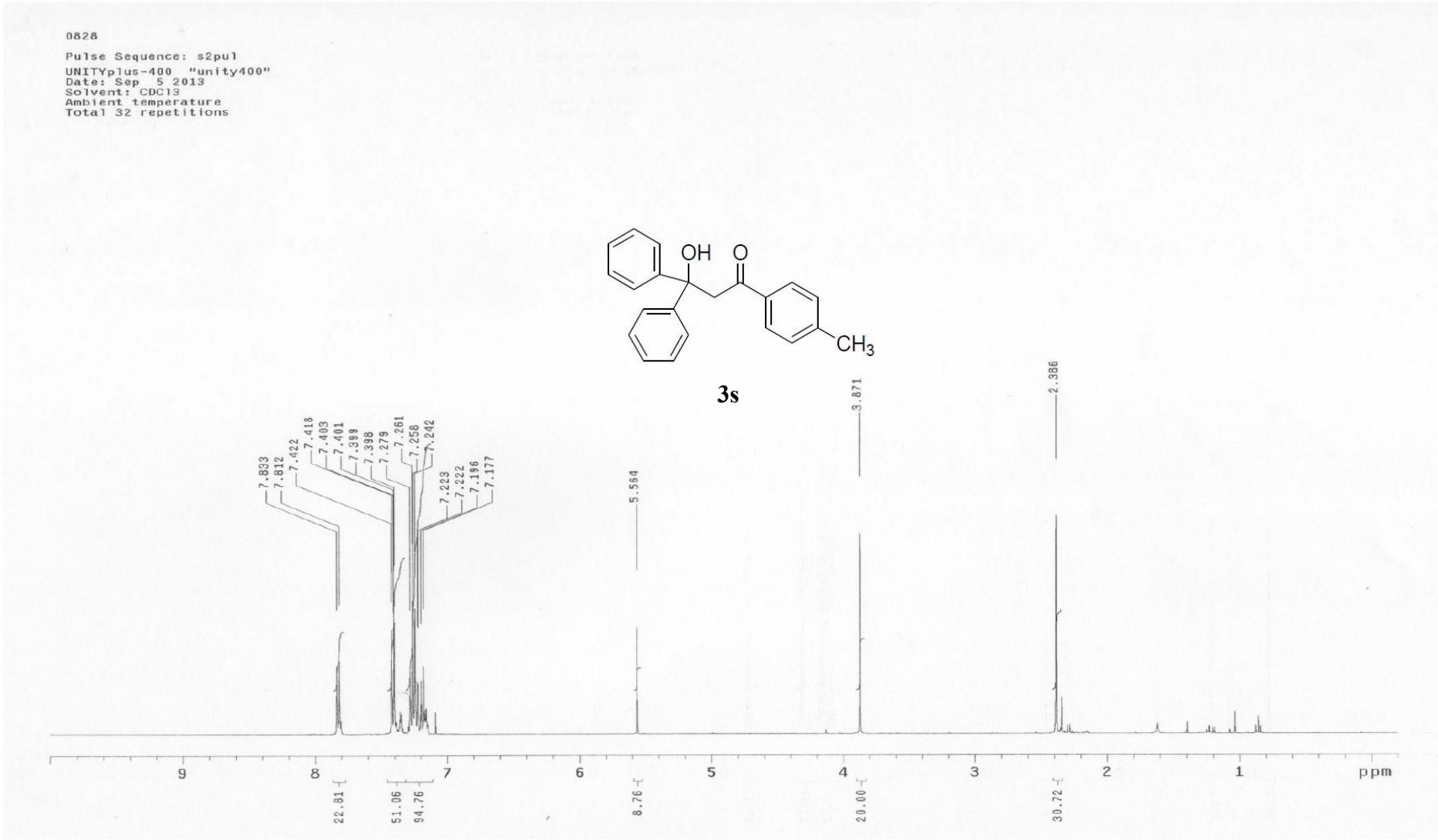

0903
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
432 repetitions
OBSERVE C13, 100.6801346 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21 hr, 51 min, 34 sec
OH
O
Br
CH3
CH3
3p
199.882
143.503
142.056
137.123
134.296
131.561
129.931
129.204
127.491
125.870
121.315
77.318
77.000
76.682
69.498
46.954
29.661
20.060
19.734
220
200
180
160
140
120
100
80
60
40
20
ppm

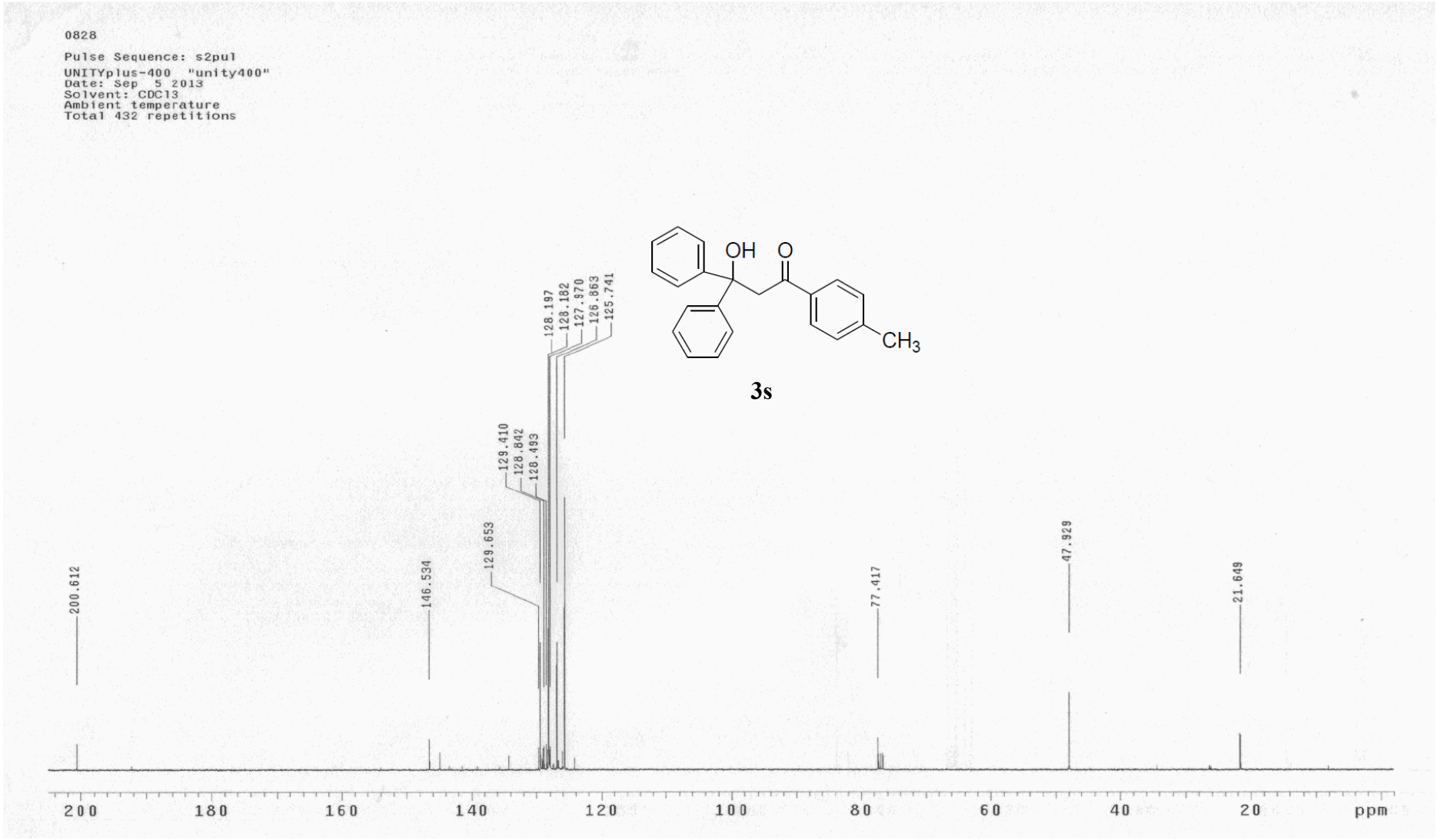

0905
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 48.1 degrees
Acq. time 4.002 sec
Width 5995.2 Hz
64 repetitions
OBSERVE H1, 400.3978963 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 5 min, 7 sec
OH
O
CH3
CH3
H3C
O
3q
7.722
7.695
7.675
7.373
7.357
7.353
7.351
7.218
7.199
6.920
6.915
6.904
6.899
5.273
3.811
3.330
3.317
3.313
2.317
2.305
1.263
10
9
8
7
6
5
4
3
2
1
0 ppm
20.13
19.65
10.50
20.00
9.82
31.35
9.06
20.23
63.31

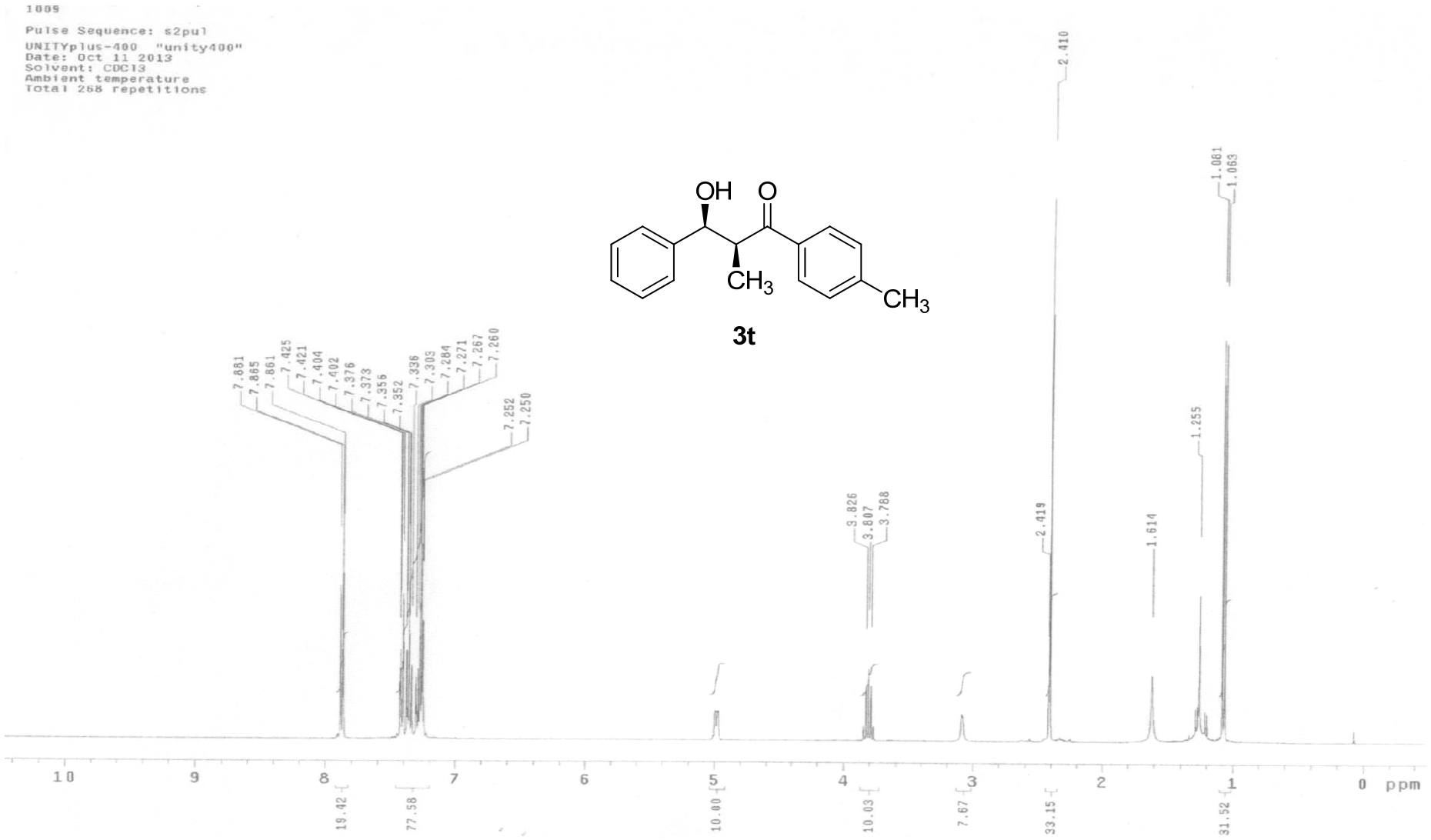

0905
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"
Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
1024 repetitions
OBSERVE C13, 100.6801361 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 44 min, 37 sec
OH
O
CH3
H3C
O
CH3
3q
200.208
159.038
143.253
137.017
135.221
134.501
129.863
129.219
126.991
125.862
113.866
77.318
77.000
76.682
69.725
55.259
47.113
29.654
20.022
19.711
220
200
180
160
140
120
100
80
60
40
20
ppm


0827
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 32 repetitions
OH
O
H3C
CH3
3r
7.829
7.808
7.517
7.513
7.496
7.494
7.492
7.323
7.303
7.261
7.260
7.240
5.017
3.789
3.746
3.341
3.298
2.410
1.629
9
8
7
6
5
4
3
2
1
ppm
18.45
18.40
18.12
28.50
8.60
10.00
9.84
29.08
29.22


0827
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 320 repetitions
OH
O
H3C
CH3
3r
200.953
147.633
144.639
134.436
129.297
128.175
128.152
126.553
124.324
77.318
77.000
76.682
73.483
48.445
30.859
21.595
200
180
160
140
120
100
80
60
40
20
ppm


0828
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 32 repetitions
OH
O
CH3
3s
7.833
7.812
7.422
7.418
7.403
7.401
7.399
7.398
7.279
7.261
7.258
7.242
7.223
7.222
7.196
7.177
5.564
3.871
2.386
9
8
7
6
5
4
3
2
1
ppm
22.81
51.06
94.76
8.76
20.00
30.72

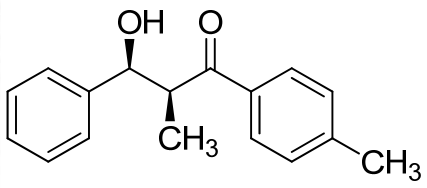

0828
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Sep 5 2013
Solvent: CDCl3
Ambient temperature
Total 432 repetitions
OH
O
CH3
3s
200.612
146.534
129.653
129.410
128.842
128.493
128.197
128.182
127.970
126.863
125.741
77.417
47.929
21.649
200
180
160
140
120
100
80
60
40
20
ppm


1009
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Oct 11 2013
Solvent: CDCl3
Ambient temperature
Total 268 repetitions
OH
O
CH3
CH3
3t
7.881
7.865
7.861
7.425
7.421
7.404
7.402
7.376
7.373
7.356
7.352
7.336
7.303
7.284
7.271
7.267
7.260
7.252
7.250
3.826
3.807
3.788
2.419
2.410
1.614
1.255
1.081
1.063
10
9
8
7
6
5
4
3
2
1
0
ppm
19.42
77.58
10.00
10.03
7.67
33.15
31.52

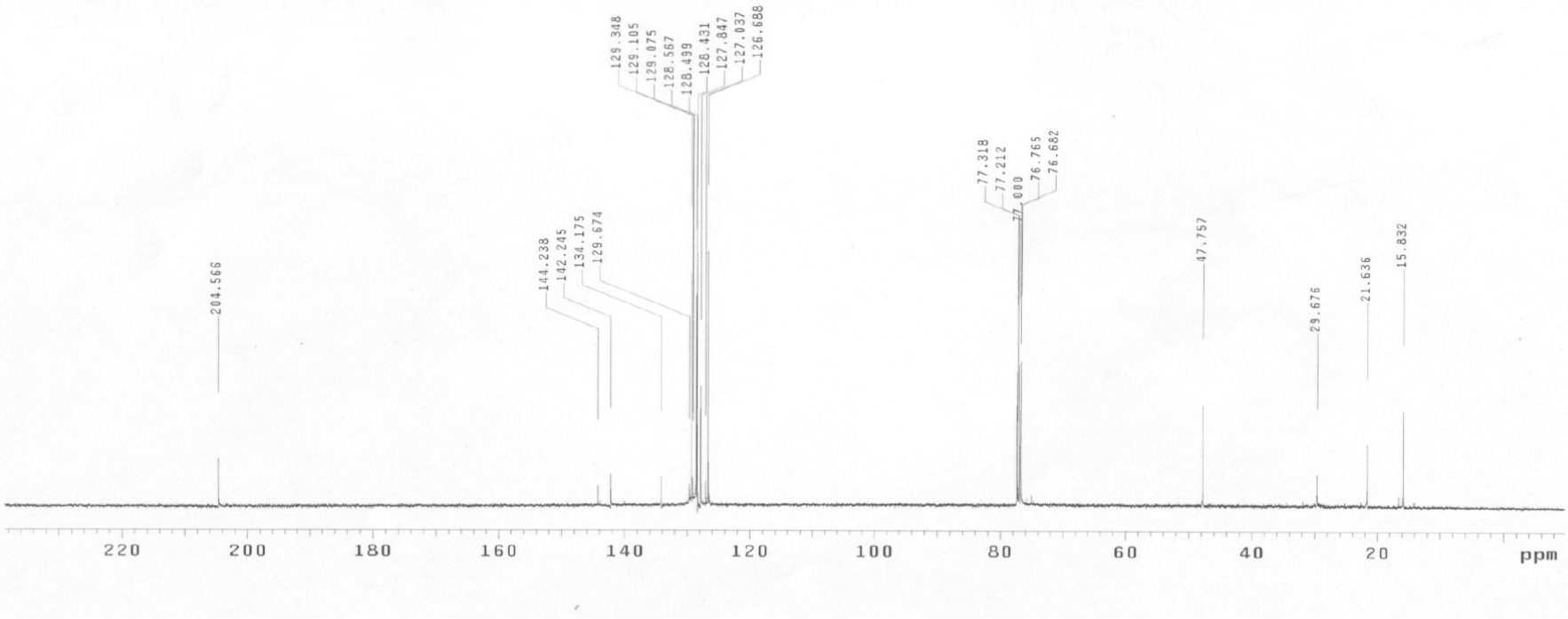
0921

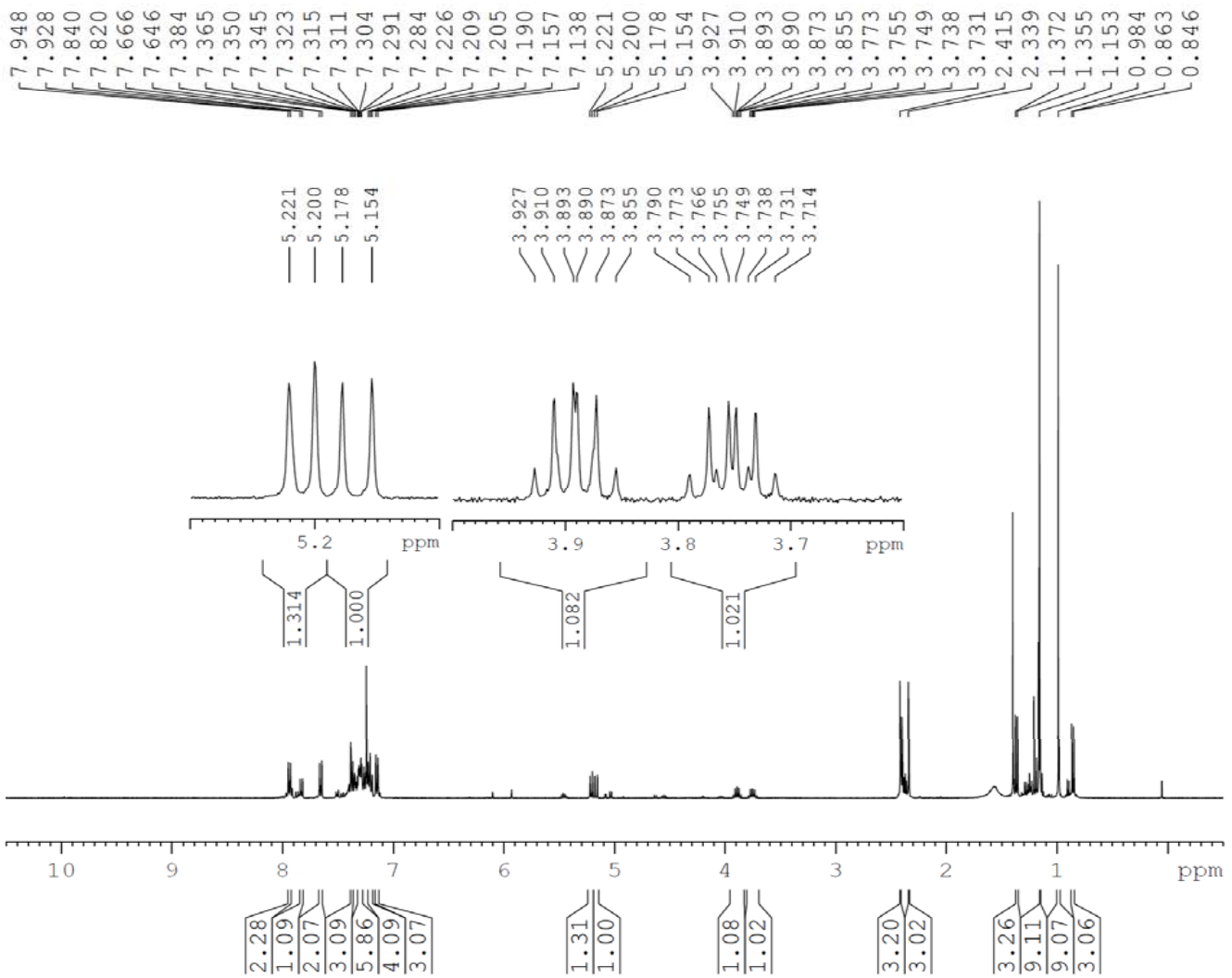
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "MercuryPlus400"

Pulse 68.7 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
3760 repetitions
OBSERVE C13, 100.6801323 MHz
DECOUPLE H1, 400.3999572 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 44 min, 37 sec

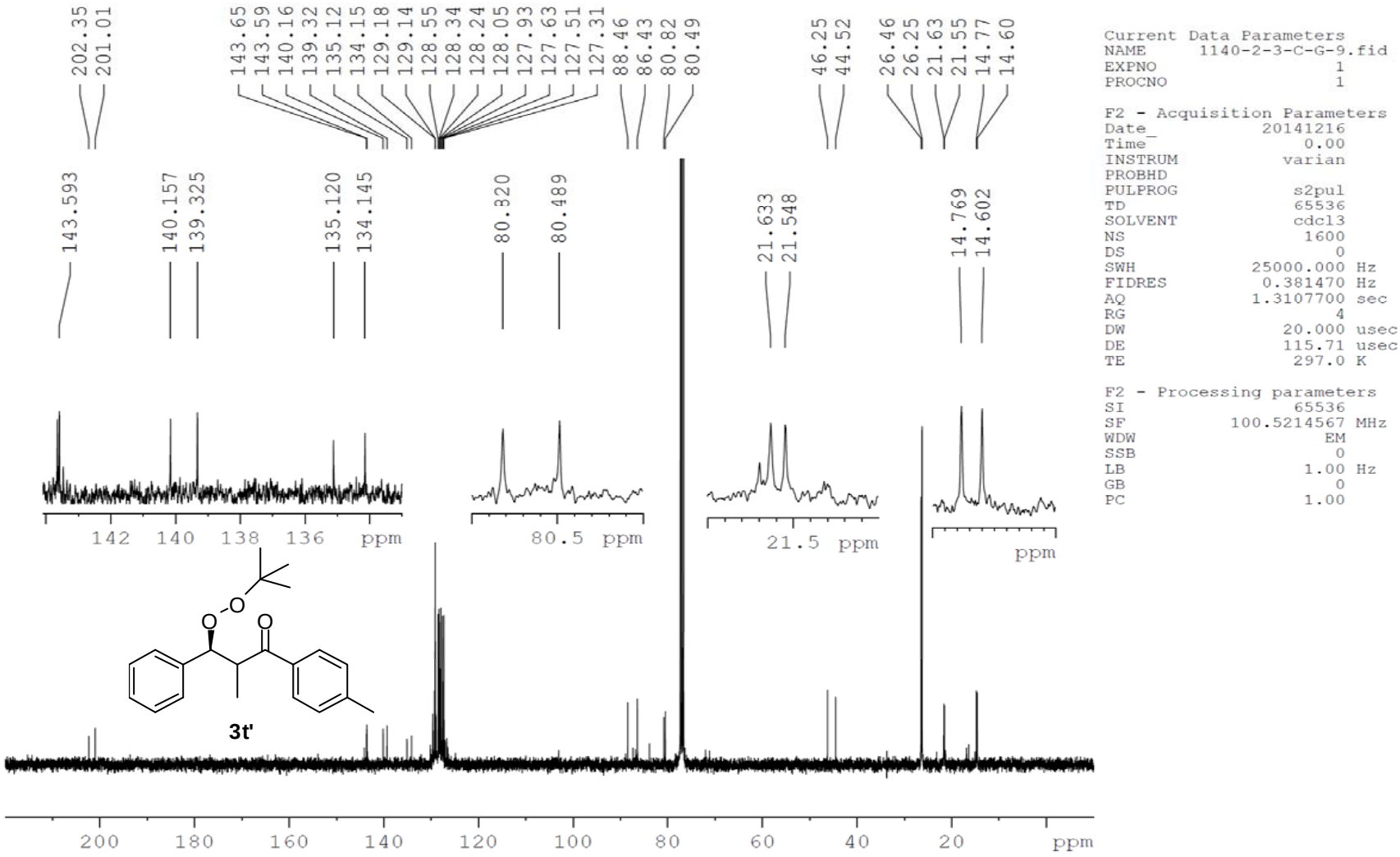


3t





NAME	20141215
EXPNO	2
PROCNO	1
Date_	20141215
Time	20.36
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	42
DS	0
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	4
DW	78.000 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
PL1	-2.40 dB
SFO1	400.1528010 MHz
SI	16384
SF	400.1500168 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



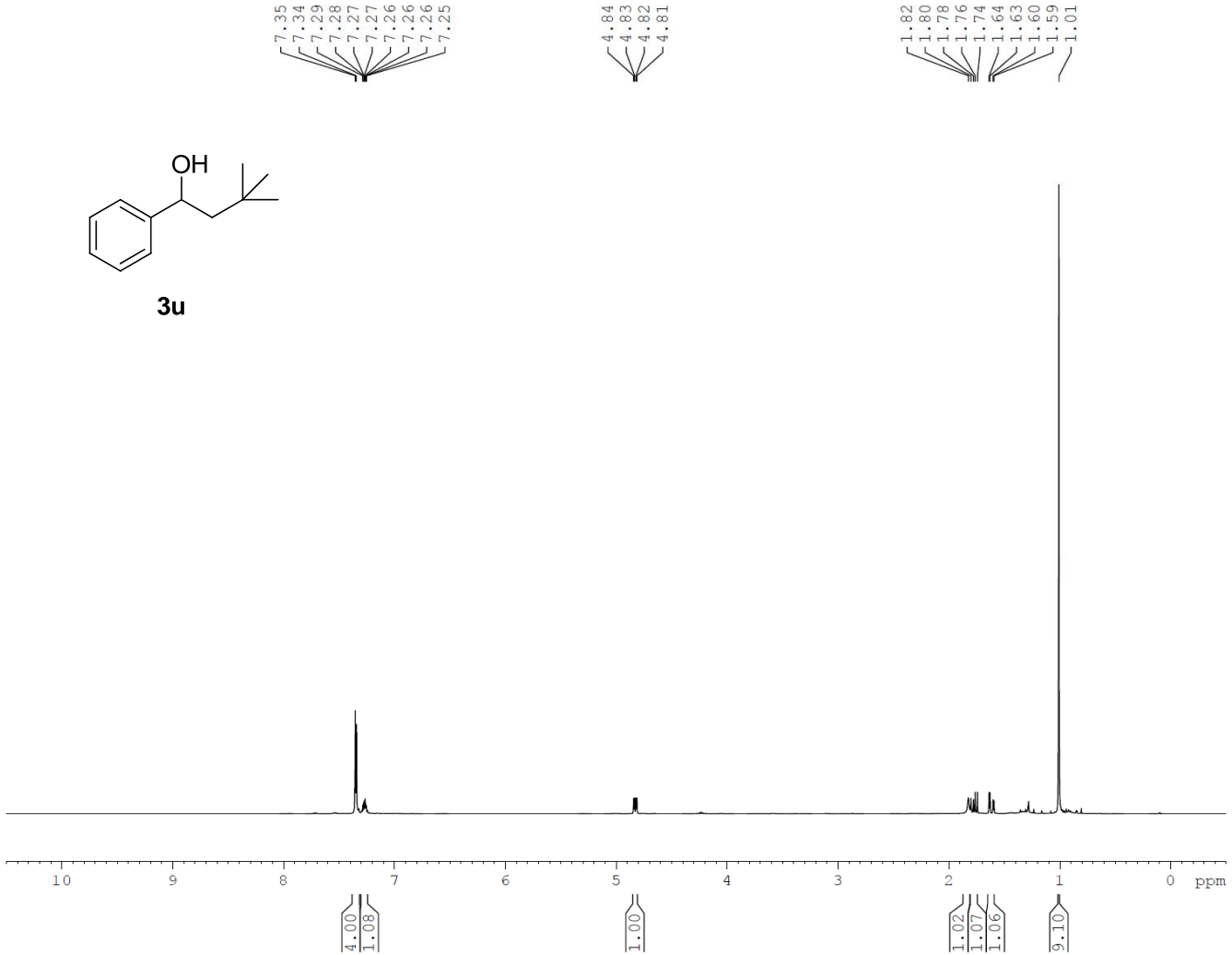
Current Data Parameters
NAME 1140-2-3-C-G-9.fid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

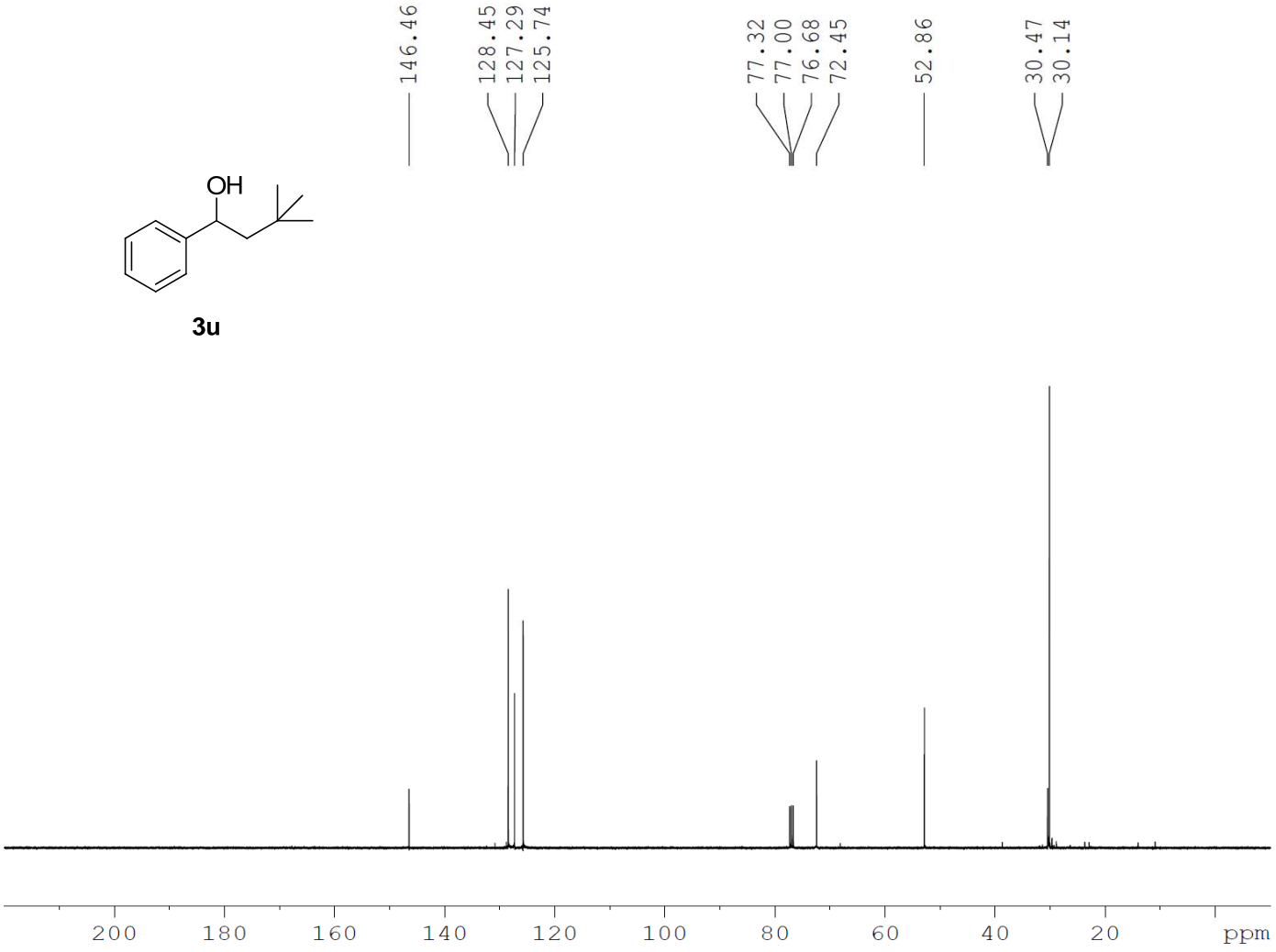
Date_	20141216
Time	0.00
INSTRUM	varian
PROBHD	
PULPROG	s2pul
TD	65536
SOLVENT	cdcl3
NS	1600
DS	0
SWH	25000.000 Hz
FIDRES	0.381470 Hz
AQ	1.3107700 sec
RG	4
DW	20.000 usec
DE	115.71 usec
TE	297.0 K

F2 - Processing parameters

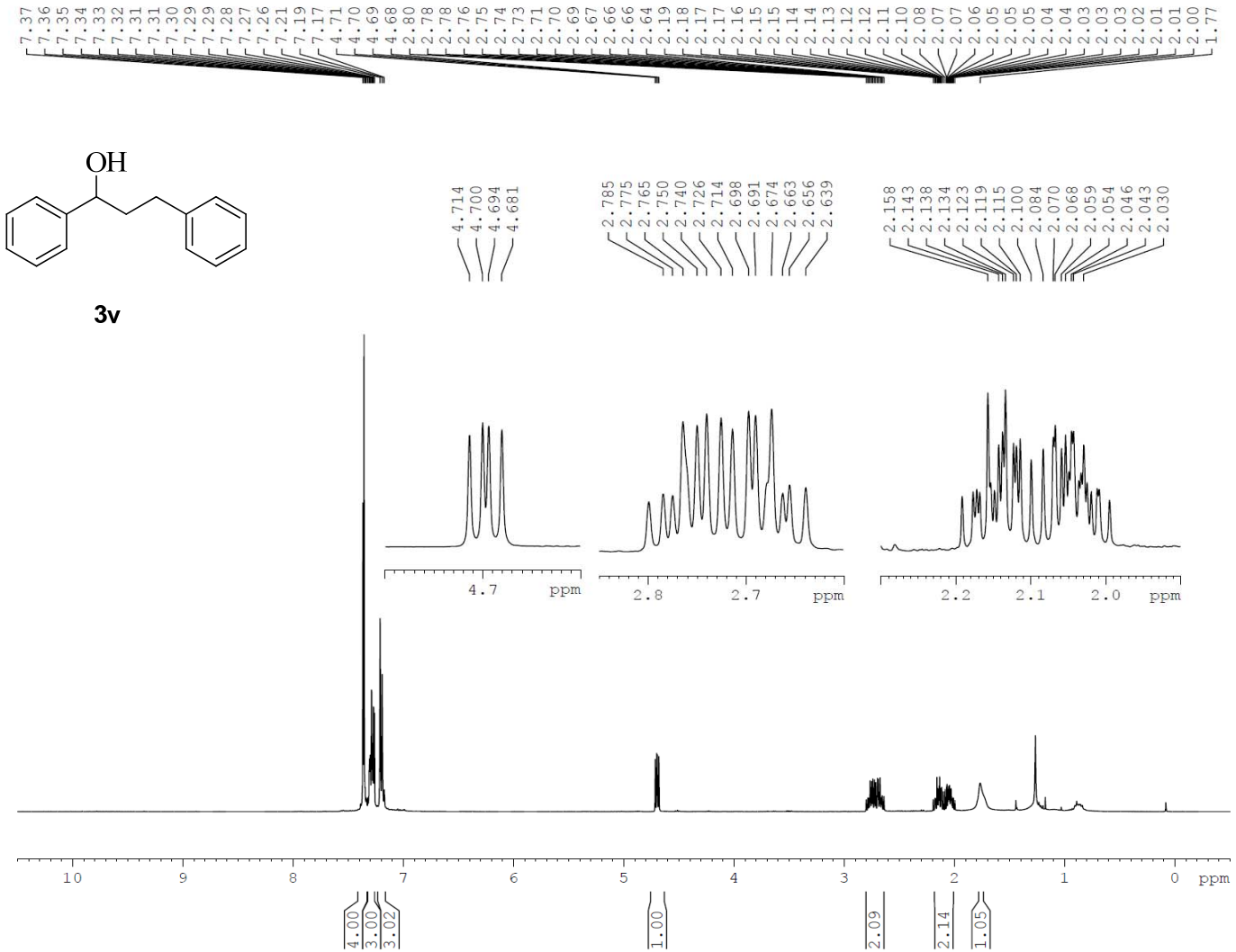
SI	65536
SF	100.5214567 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00




```
Current Data Parameters
NAME      1117-1-H-G.fid
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141002
Time                0.00
INSTRUM          varian
PROBHD
PULPROG           s2pul
TD                32768
SOLVENT           cdcl3
NS                   40
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                    4
DW               78.000 usec
DE               115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                32768
SF          399.7627610 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
```



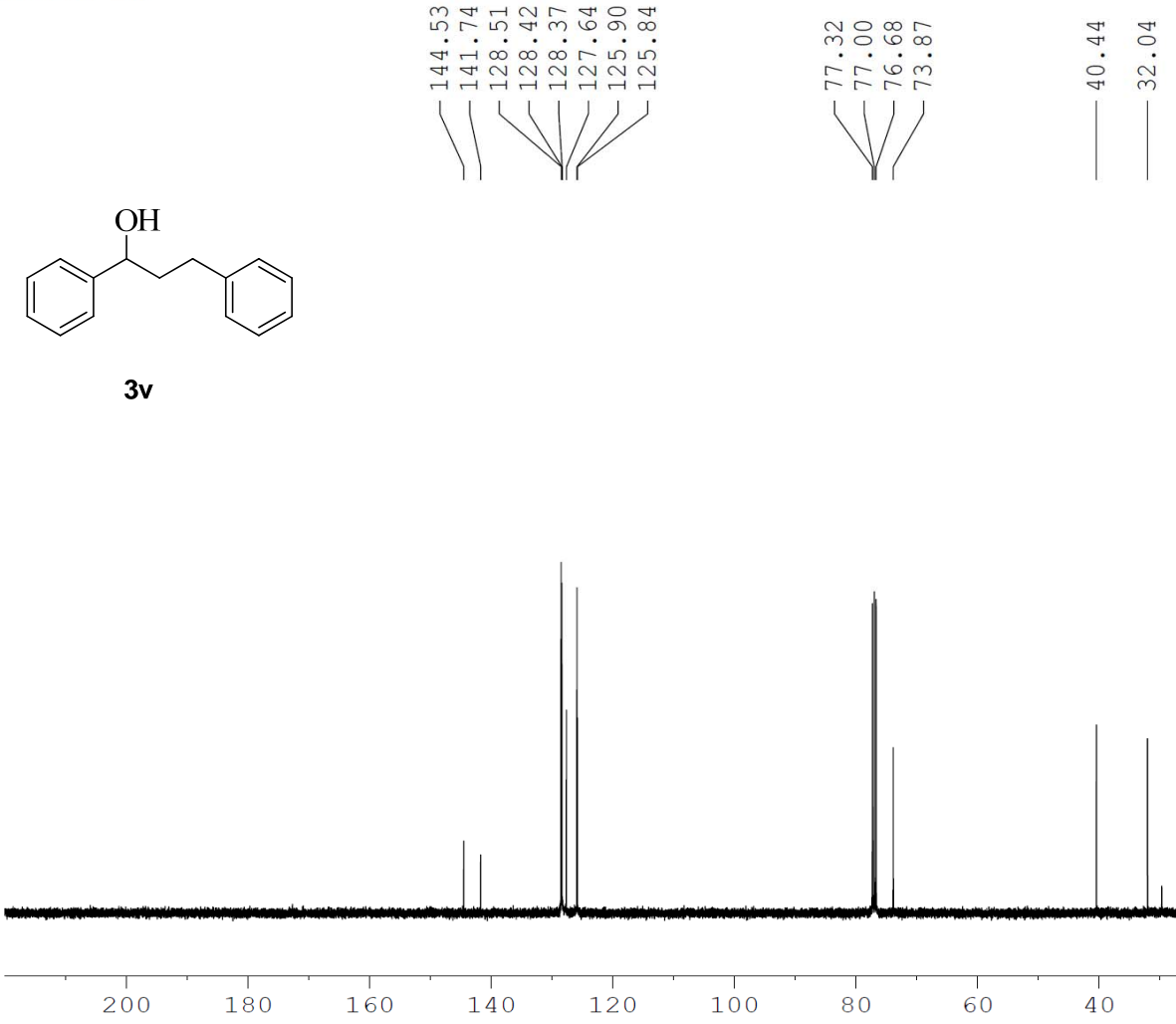

```
Current Data Parameters
NAME      1117-1-C-G-4.fid
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20141002
Time                   0.00
INSTRUM             varian
PROBHD
PULPROG              s2pul
TD                   65536
SOLVENT              cdcl3
NS                     328
DS                       0
SWH              25510.203 Hz
FIDRES            0.389255 Hz
AQ               1.3107700 sec
RG                       4
DW                  19.600 usec
DE                  115.71 usec
TE                   298.0 K

F2 - Processing parameters
SI                   65536
SF           100.5218605 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



3v

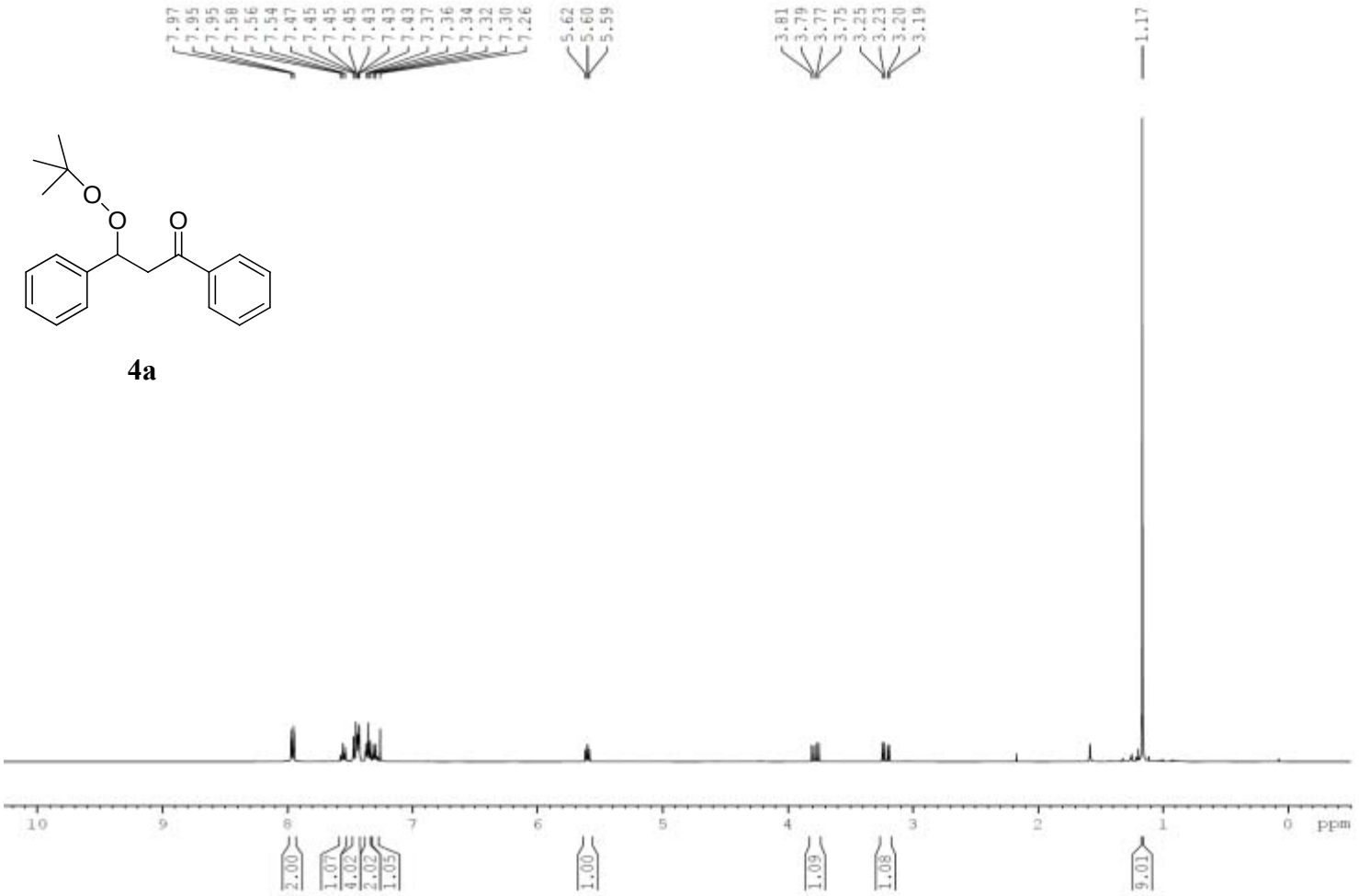
```
Current Data Parameters
NAME      ra-pen-oh-1.fid
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131004
Time                 0.00
INSTRUM           varian
PROBHD
PULPROG            s2pul
TD                 32768
SOLVENT            cdcl3
NS                    48
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                     4
DW                78.000 usec
DE                115.71 usec
TE                 298.0 K

F2 - Processing parameters
SI                 32768
SF          399.7627826 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

RA-PEN-OH



3v

```
Current Data Parameters
NAME      RA-PEN-OH-C.fid
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131004
Time                0.00
INSTRUM          varian
PROBHD
PULPROG           s2pul
TD                65536
SOLVENT           cdcl3
NS                  948
DS                    0
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.3107700 sec
RG                    4
DW               19.600 usec
DE               115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                65536
SF          100.5218604 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

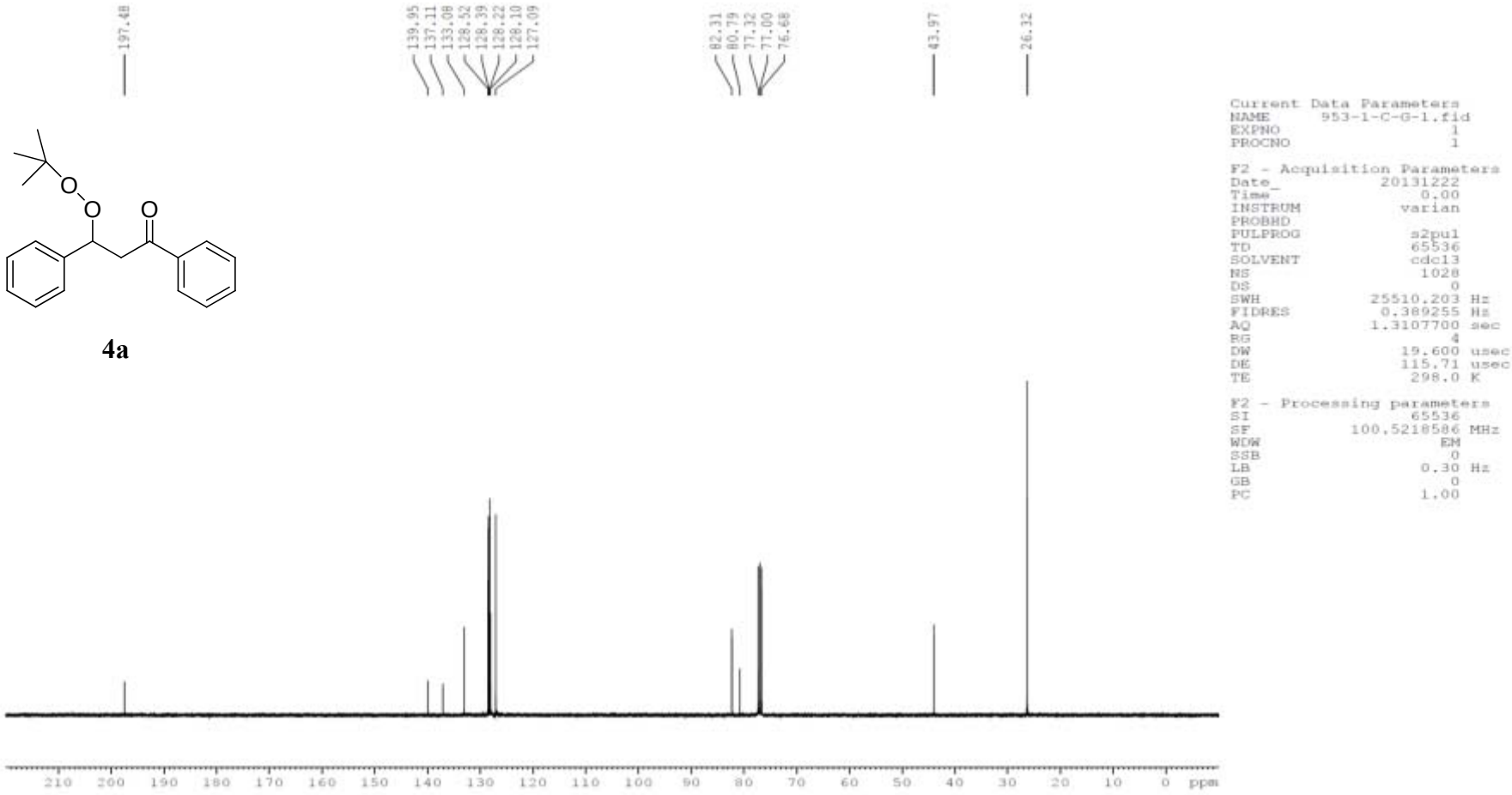


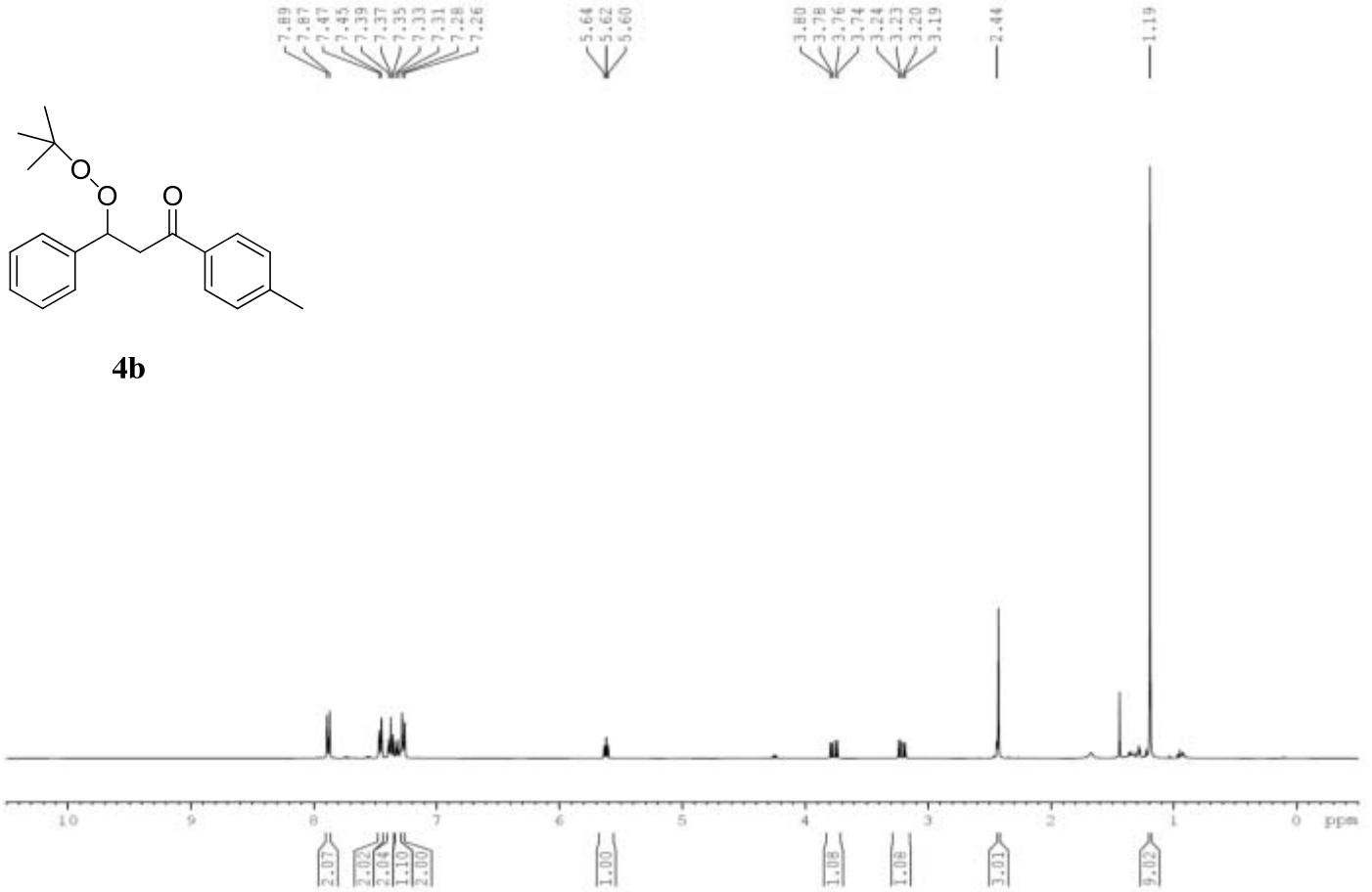
4a

Current Data Parameters
NAME RA-135-OP.fid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131222
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K

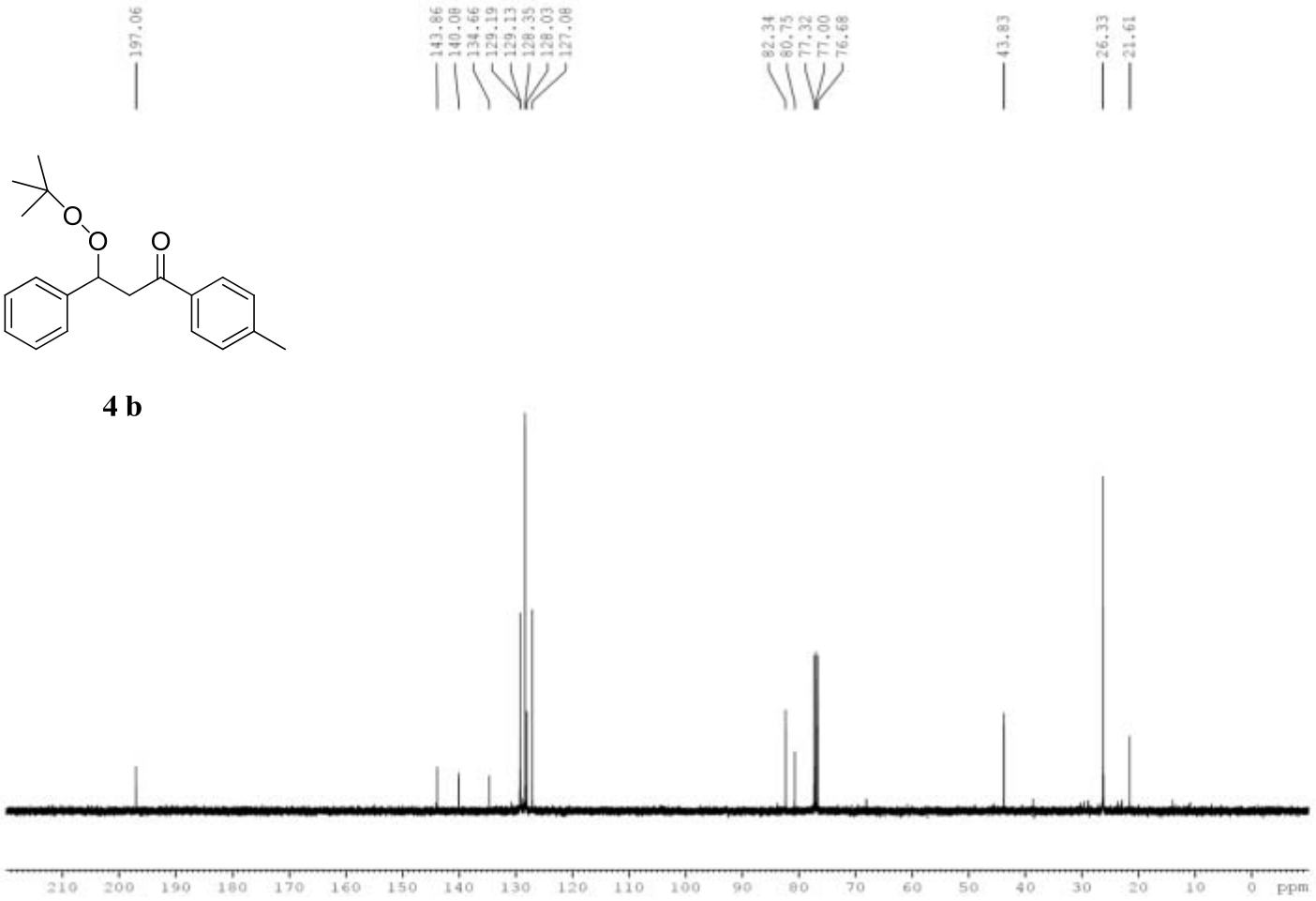
F2 - Processing parameters
SI 32768
SF 399.7627613 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


197.48
139.95
137.11
133.08
128.52
128.39
128.22
128.10
127.09
82.31
80.79
77.32
77.00
76.68
43.97
26.32
O
O
O
4a
Current Data Parameters
NAME 953-1-C-G-1.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131222
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 1028
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218586 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



4b

```
Current Data Parameters
NAME                  961-1-H.fid
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20130924
Time                         0.00
INSTRUM                   varian
PROBHD
PULPROG                    s2pul
TD                         32768
SOLVENT                    cdcl3
NS                            44
DS                             0
SWH                     6410.256 Hz
FIDRES                  0.195625 Hz
AQ                     2.5559540 sec
RG                             4
DW                        78.000 usec
DE                        115.71 usec
TE                        298.0 K

F2 - Processing parameters
SI                         32768
SF                399.7627679 MHz
WDW                          EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```

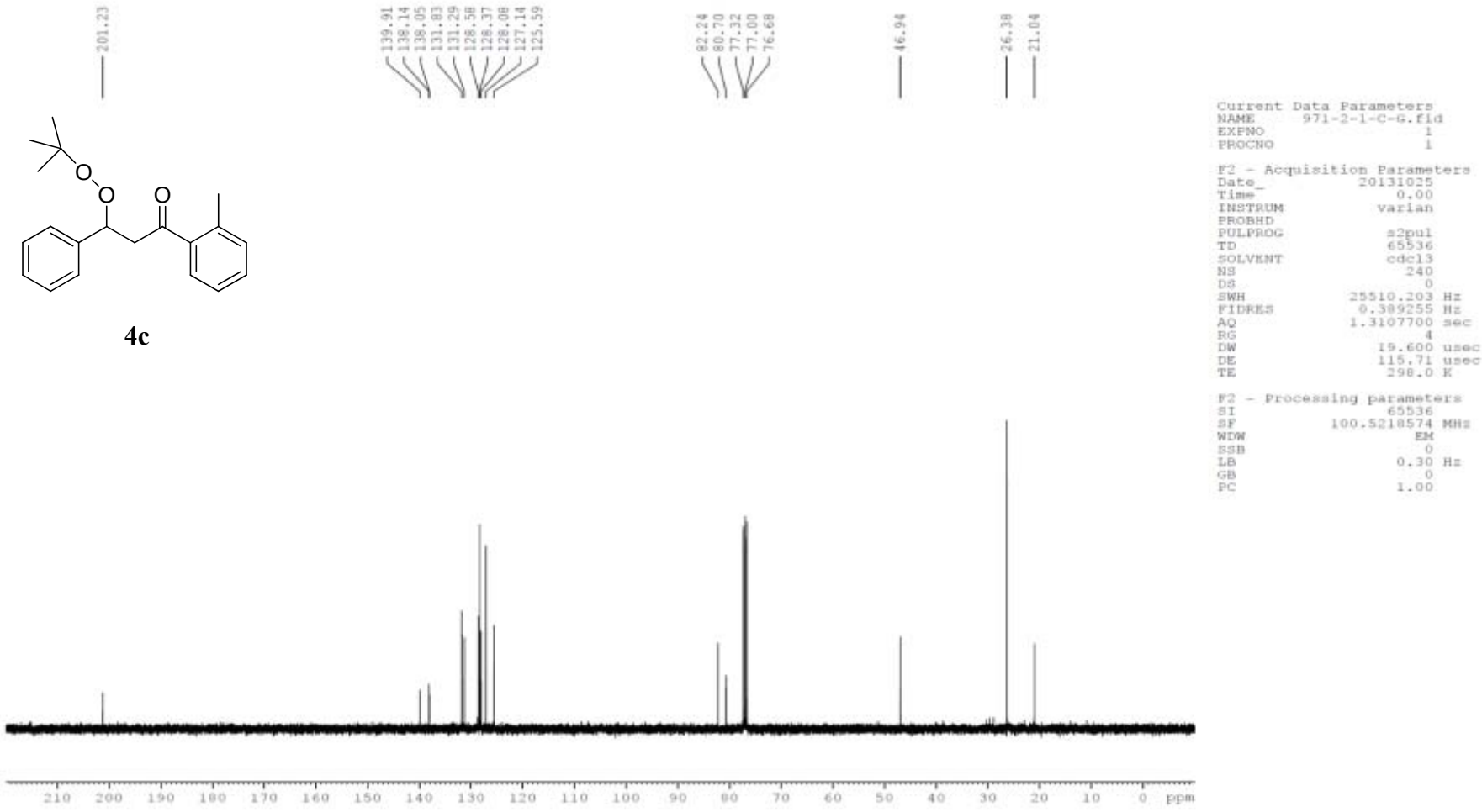


4 b

```
Current Data Parameters
NAME                  961-1-C.fid
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20130924
Time                         0.00
INSTRUM                    varian
PROBHD
PULPROG                     s2pul
TD                          65536
SOLVENT                     cdcl3
NS                            352
DS                              0
SWH                     25510.203 Hz
FIDRES                   0.389255 Hz
AQ                      1.3107700 sec
RG                              4
DW                         19.600 usec
DE                         115.71 usec
TE                          298.0 K

F2 - Processing parameters
SI                          65536
SF                    100.5218652 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```

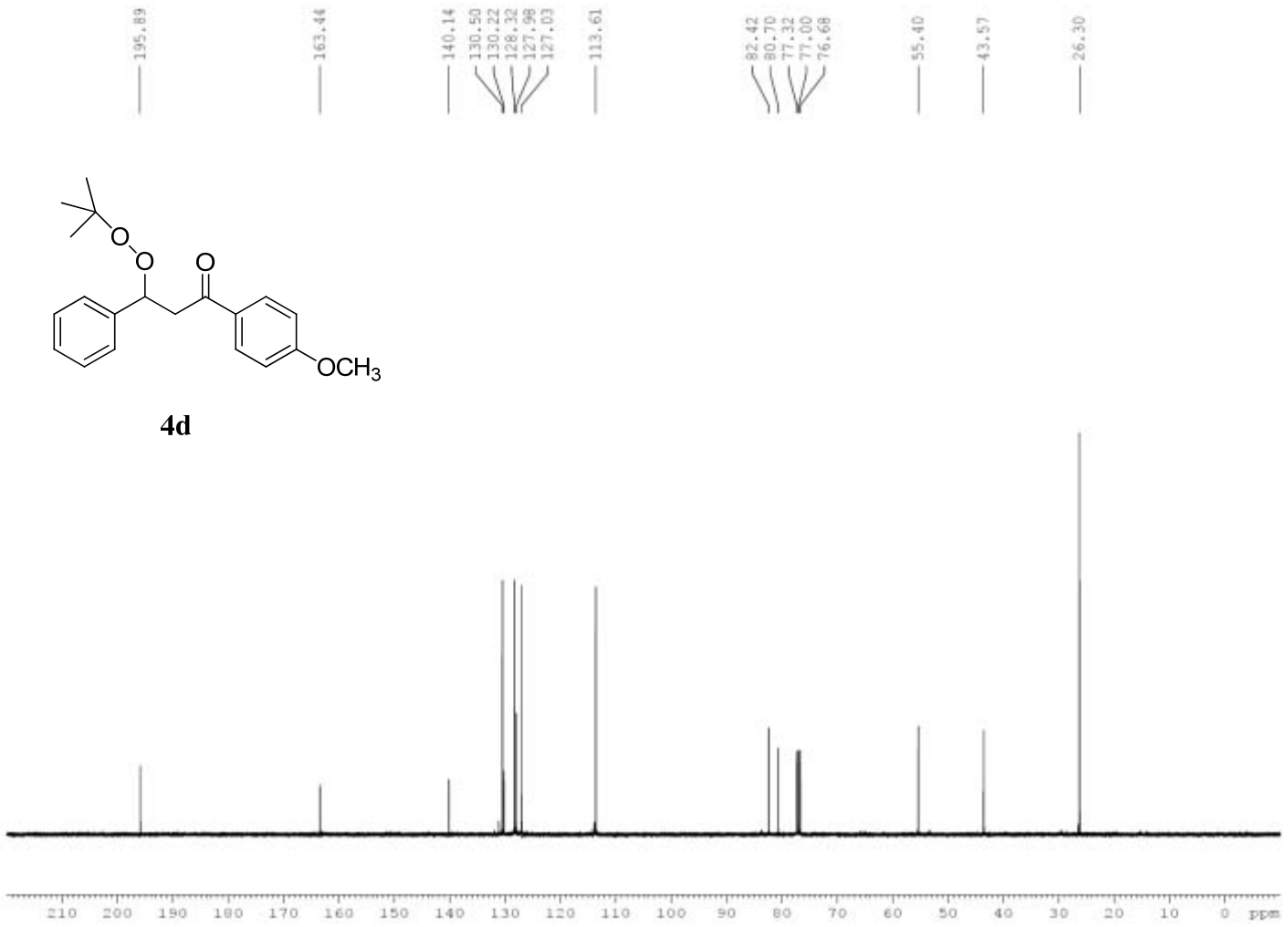
4c

```
Current Data Parameters
NAME      971-2-1-H-G.fid
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131025
Time                 0.00
INSTRUM           varian
PROBHD
PULPROG             s2pul
TD                  32768
SOLVENT             cdcl3
NS                     32
DS                      0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ              2.5559540 sec
RG                      4
DW                78.000 usec
DE                115.71 usec
TE                 298.0 K

F2 - Processing parameters
SI                  32768
SF          399.7627607 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```


201.23
139.91
138.14
138.05
131.83
131.29
128.58
128.37
128.08
127.14
125.59
82.24
80.70
77.32
77.00
76.68
46.94
26.38
21.04
O
O
O
4c
Current Data Parameters
NAME 971-2-1-C-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131025
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 240
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218574 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

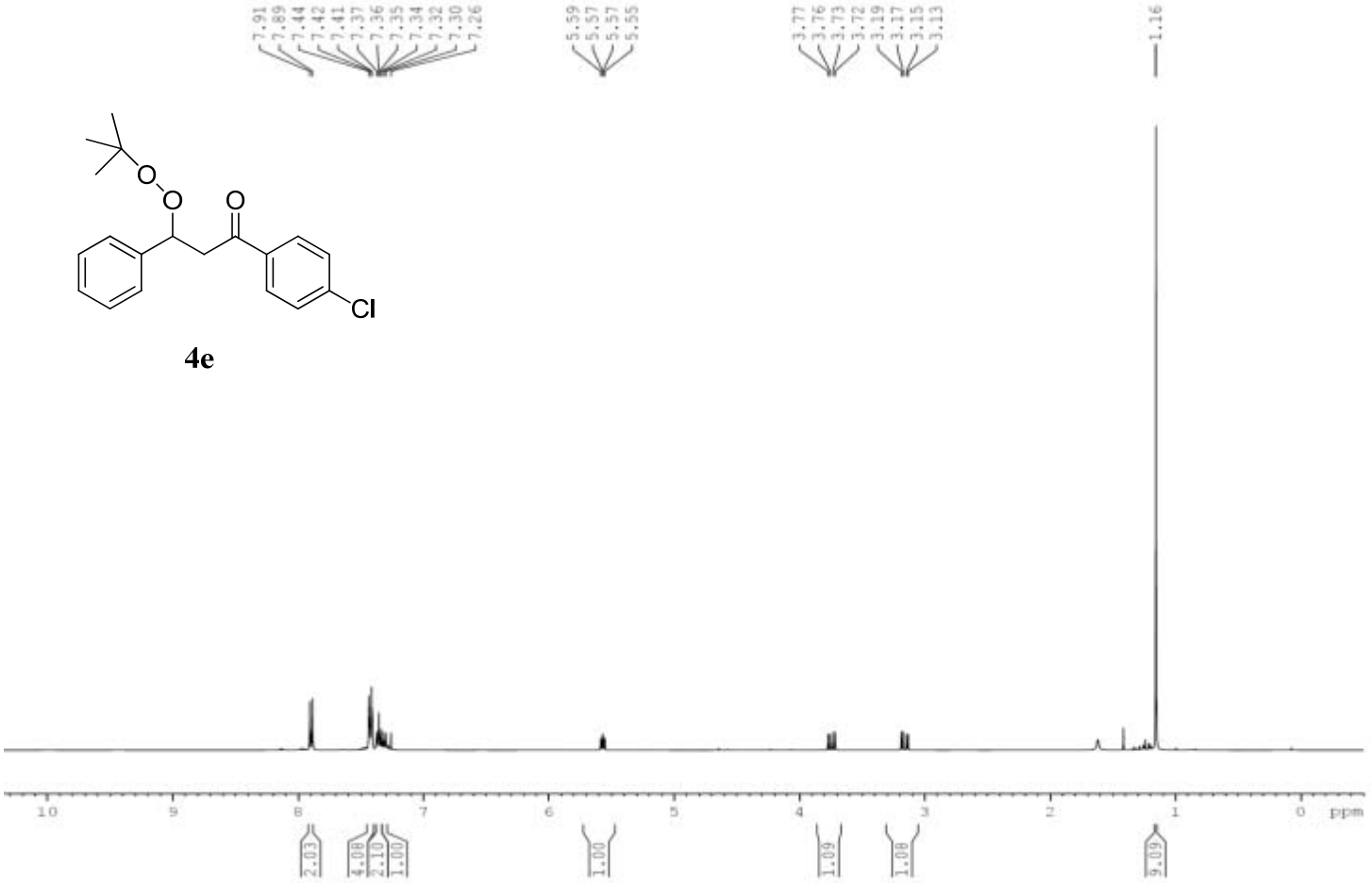
4d

```
Current Data Parameters
NAME      966-1-1-1-H.fid
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20131003
Time                0.00
INSTRUM          varian
PROBHD
PULPROG            s2pul
TD                 32768
SOLVENT            cdcl3
NS                    32
DS                     0
SWH             6402.049 Hz
FIDRES          0.195375 Hz
AQ              2.5559540 sec
RG                     4
DW                78.100 usec
DE                115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                 32768
SF          399.7627669 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



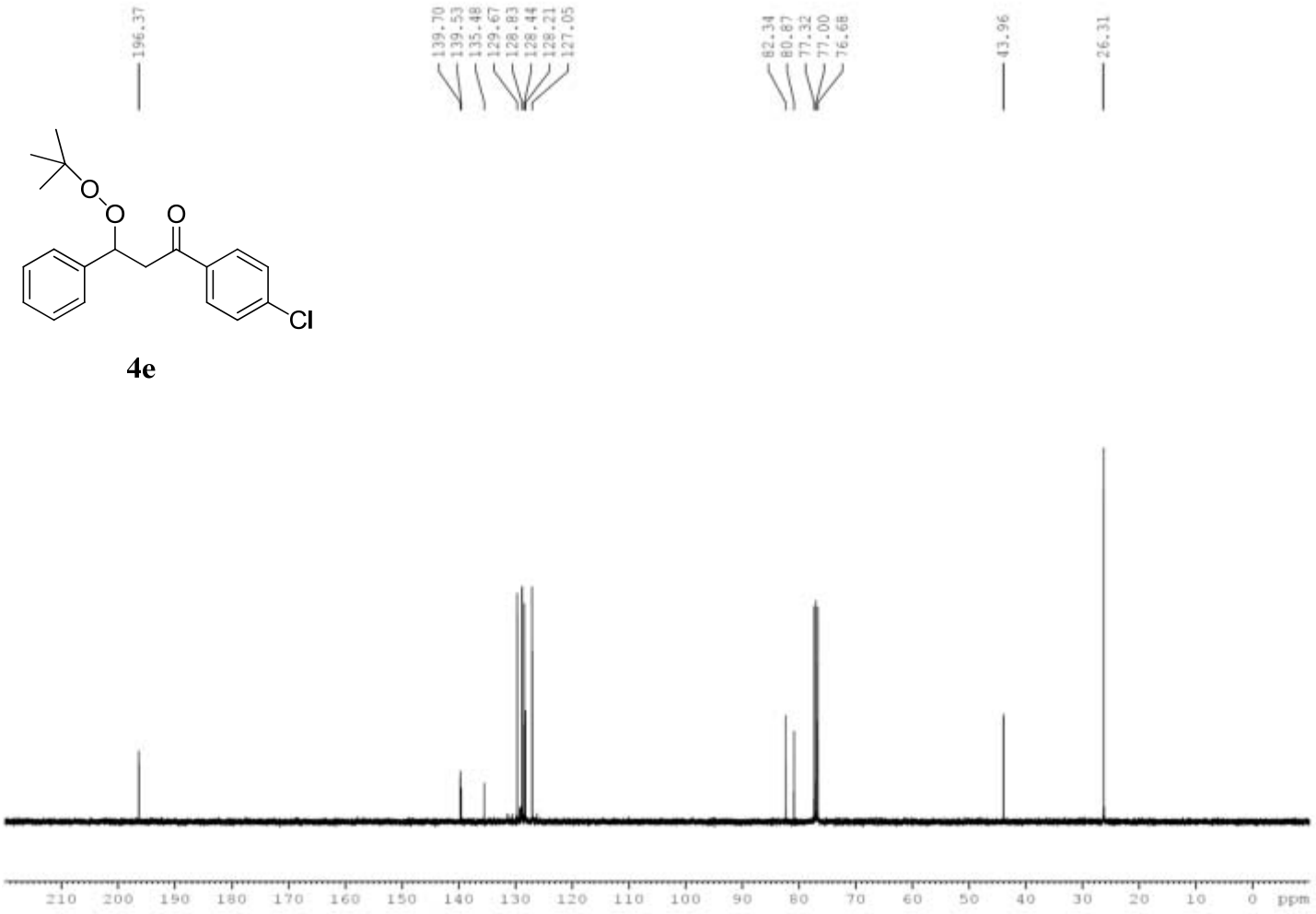
4d

```
Current Data Parameters
NAME      RA-202-OP-C-3.fid
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140107
Time                  0.00
INSTRUM             varian
PROBHD
PULPROG              s2pul
TD                   65536
SOLVENT              cdcl3
NS                     212
DS                       0
SWH              25510.203 Hz
FIDRES           0.389255 Hz
AQ               1.3107700 sec
RG                       4
DW                  19.600 usec
DE                  115.71 usec
TE                  298.0 K

F2 - Processing parameters
SI                   65536
SF           100.5218621 MHz
WDW                    EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

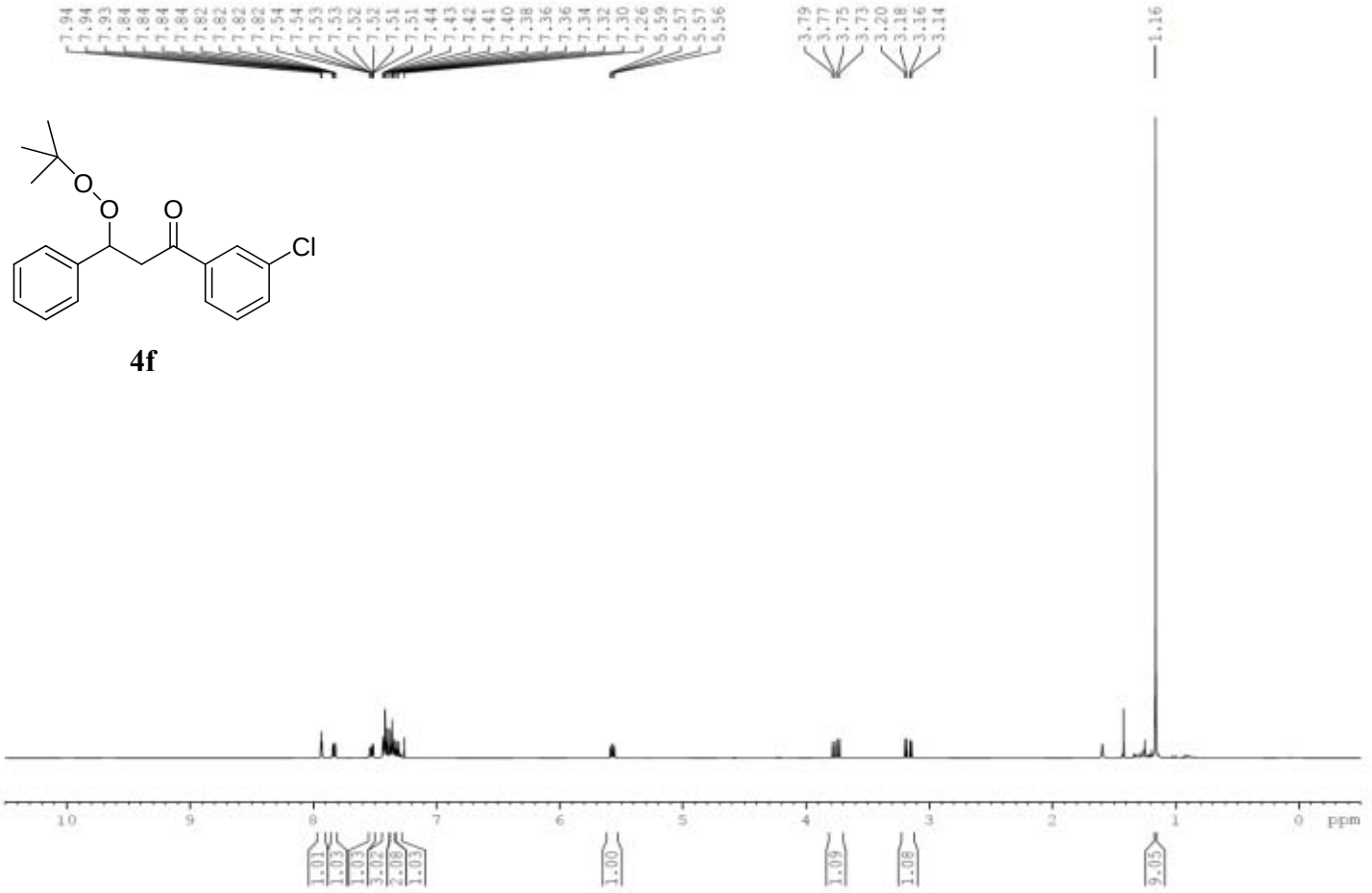


4e

```
Current Data Parameters
NAME       974-2-2-H.fid
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131014
Time                0.00
INSTRUM          varian
PROBHD
PULPROG           s2pul
TD                32768
SOLVENT           cdcl3
NS                   32
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                    4
DW               78.000 usec
DE               115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                32768
SF        399.7627604 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

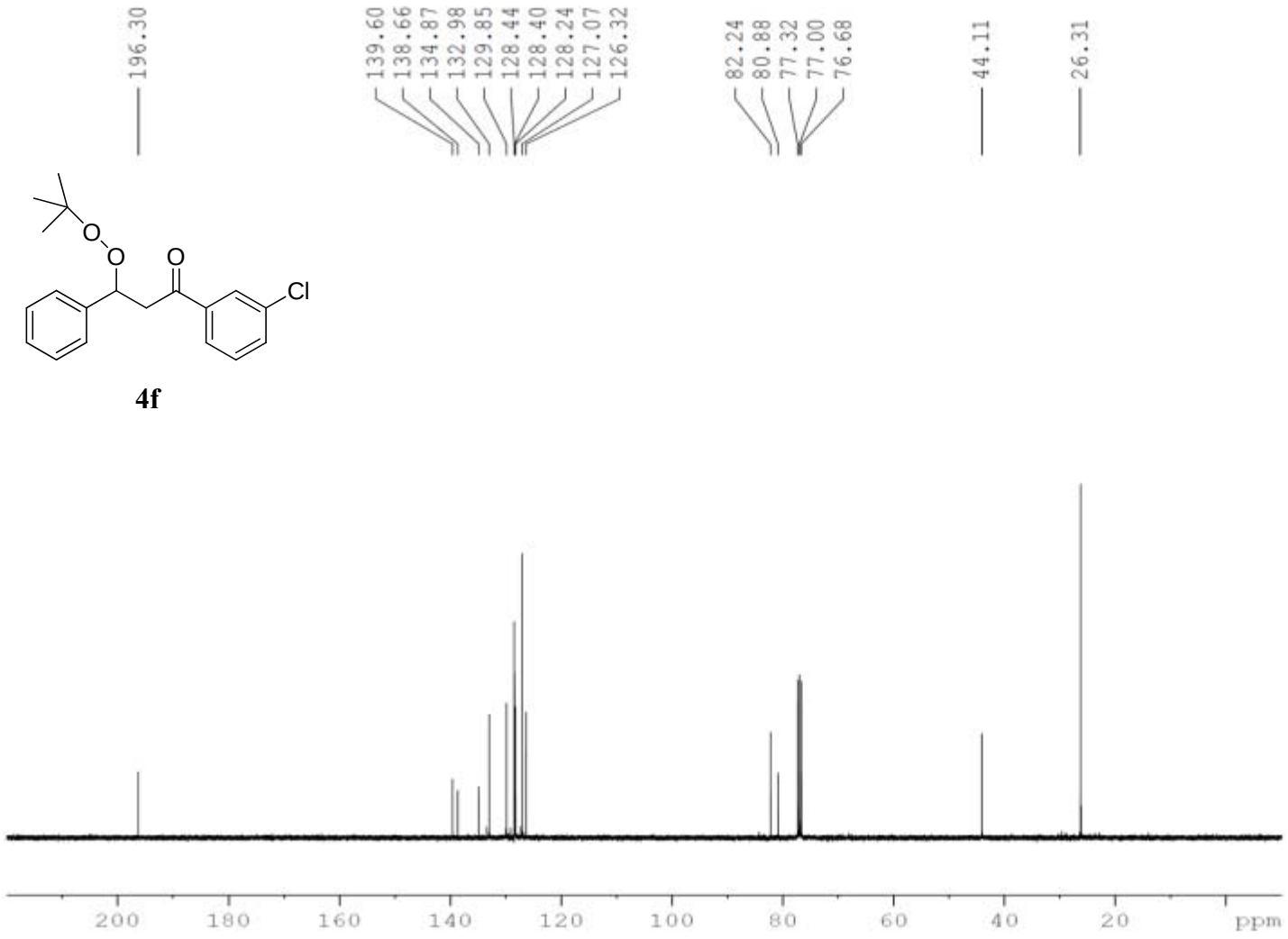



```
Current Data Parameters
NAME      974-2-2-c-1.fid
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131014
Time                 0.00
INSTRUM           varian
PROBHD
PULPROG            s2pul
TD                 65536
SOLVENT            cdcl3
NS                   840
DS                     0
SWH            25510.203 Hz
FIDRES         0.389255 Hz
AQ             1.3107700 sec
RG                     4
DW                19.600 usec
DE                115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                 65536
SF          100.5218574 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



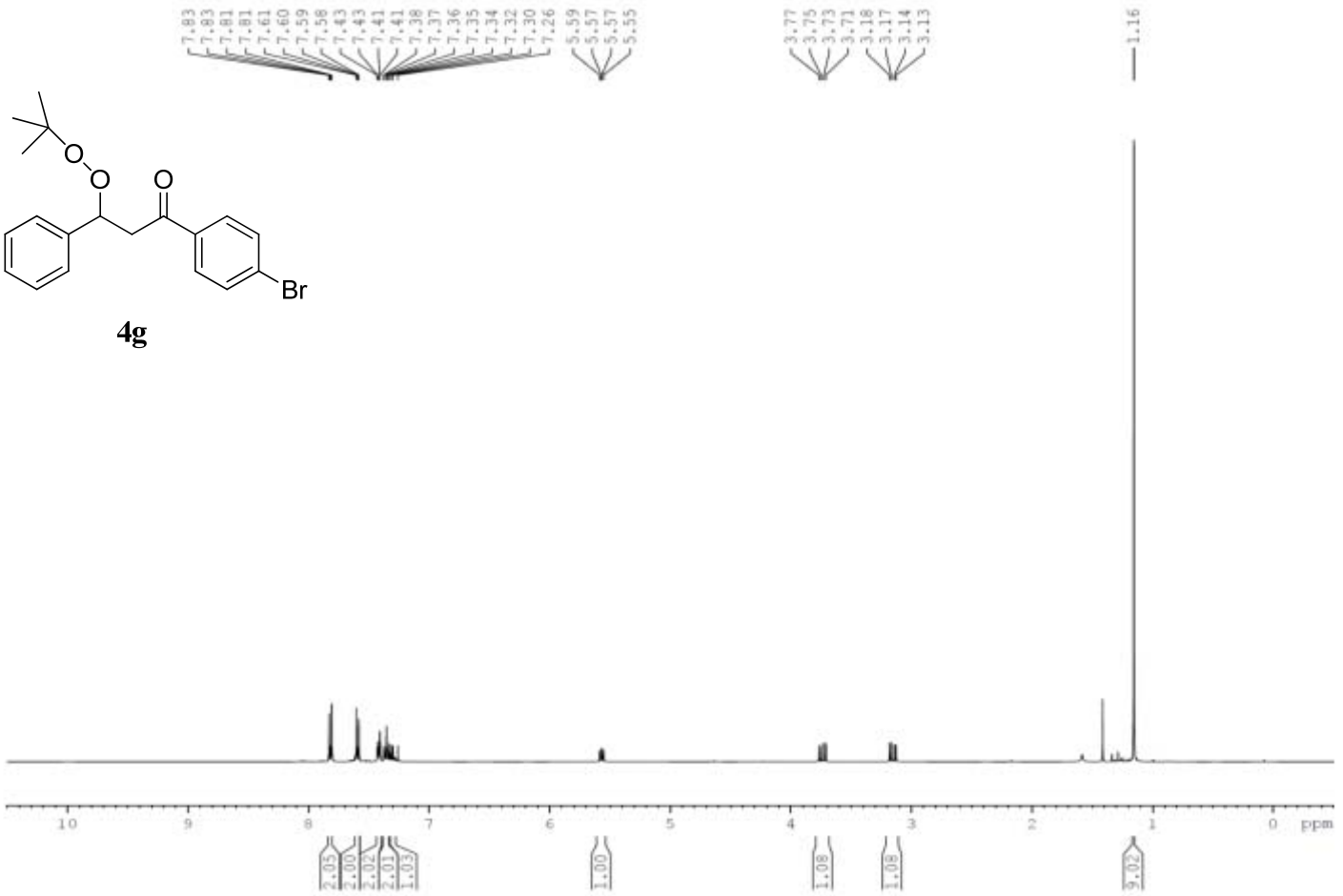
4f

```
Current Data Parameters
NAME        975-2-H-G.fid
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131026
Time                 0.00
INSTRUM           varian
PROBHD
PULPROG            s2pul
TD                 32768
SOLVENT            cdcl3
NS                    32
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ              2.5559540 sec
RG                     4
DW                78.000 usec
DE                115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                 32768
SF          399.7627607 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



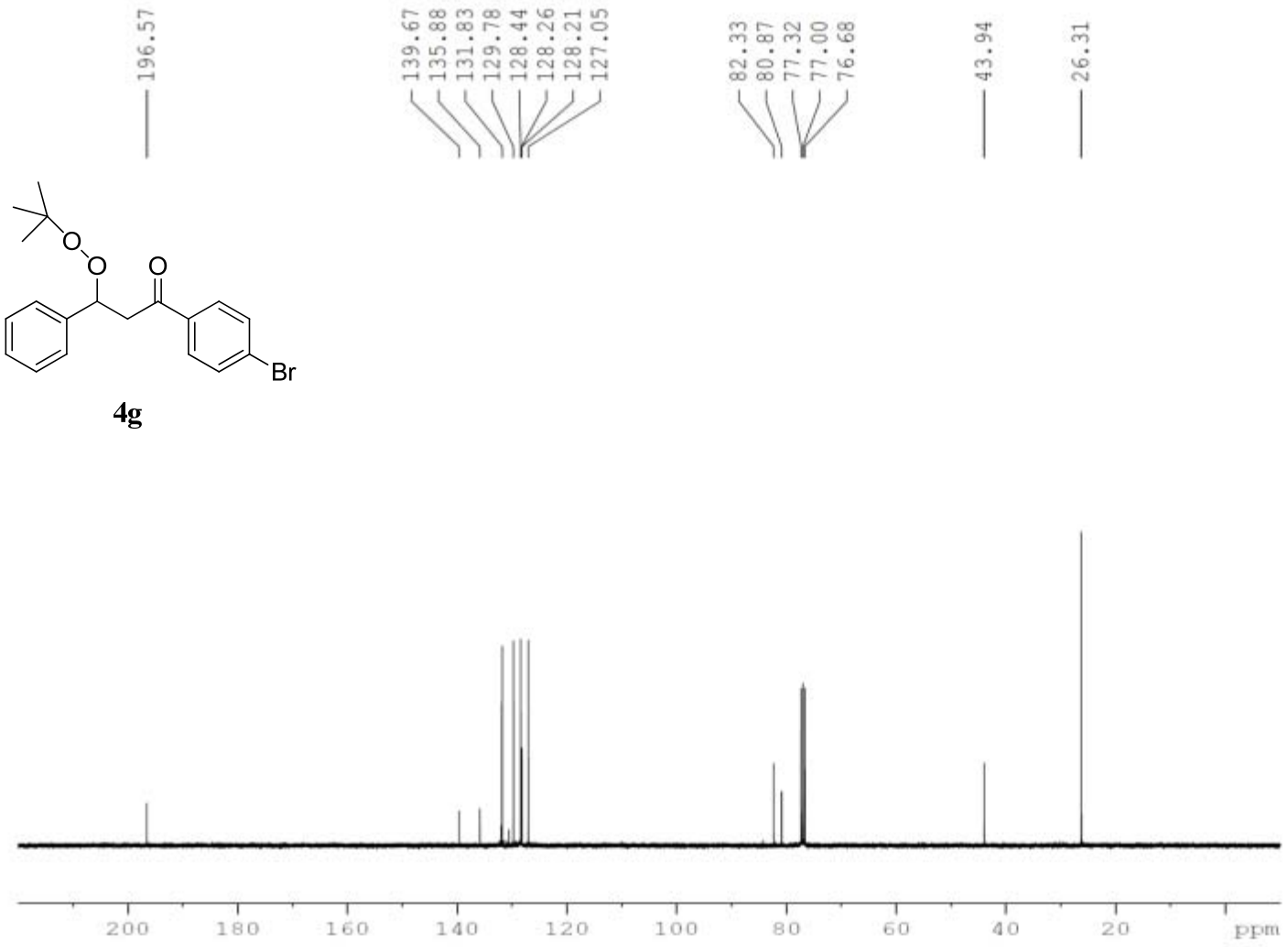
4f

```
Current Data Parameters
NAME      975-2-C-G-1.fid
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131026
Time                 0.00
INSTRUM           varian
PROBHD
PULPROG             s2pul
TD                  65536
SOLVENT             cdcl3
NS                    768
DS                      0
SWH          25510.203 Hz
FIDRES        0.389255 Hz
AQ           1.3107700 sec
RG                      4
DW              19.600 usec
DE              115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                  65536
SF         100.5218579 MHz
WDW                    EM
SSB                     0
LB               0.30 Hz
GB                      0
PC                   1.00
```



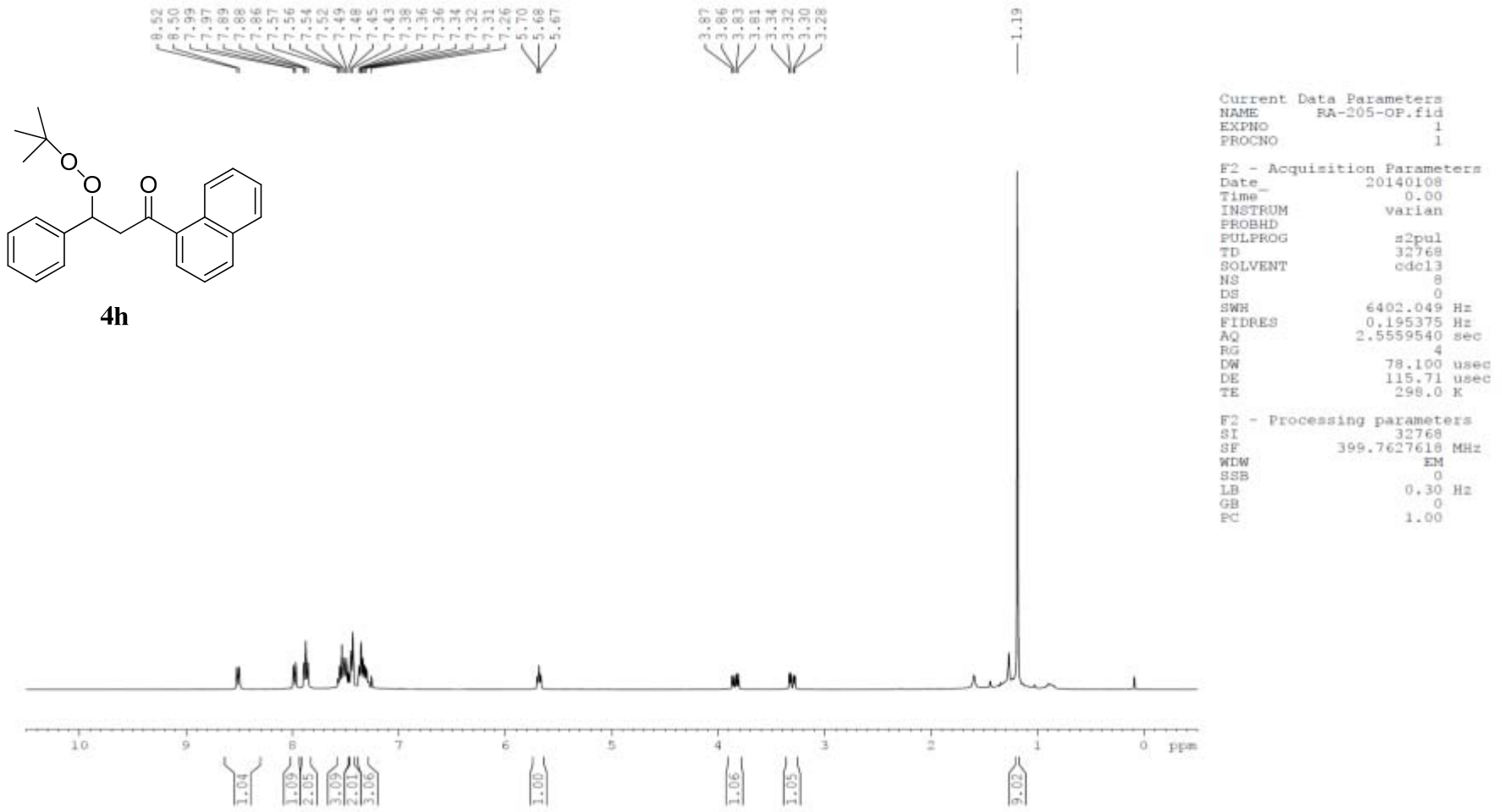
4g

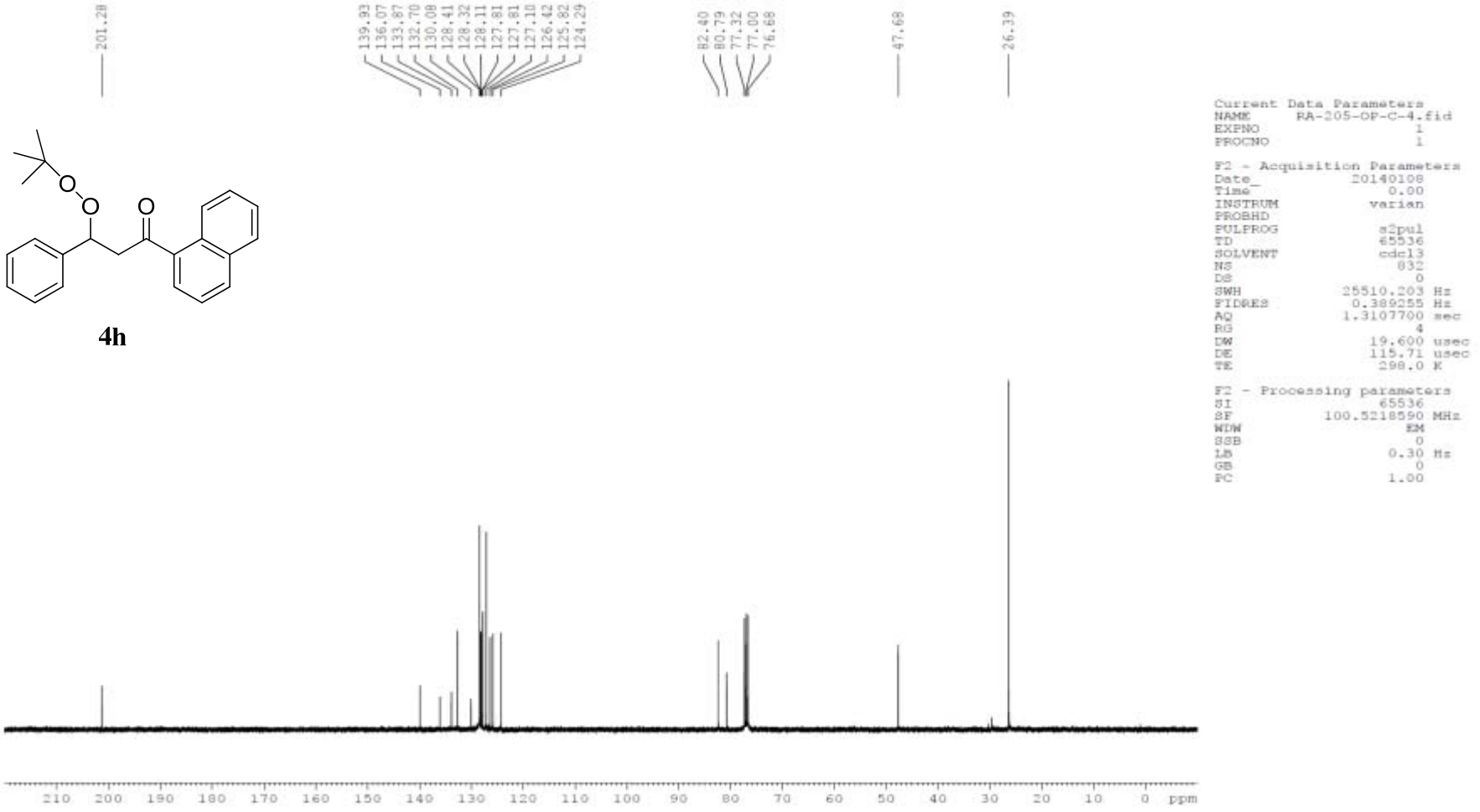
```
Current Data Parameters
NAME      976-2-2-1-H-G.fid
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20131025
Time                  0.00
INSTRUM            varian
PROBHD
PULPROG             s2pul
TD                  32768
SOLVENT             cdcl3
NS                     32
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ               2.5559540 sec
RG                      4
DW                 78.000 usec
DE                 115.71 usec
TE                  298.0 K

F2 - Processing parameters
SI                  32768
SF          399.7627605 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

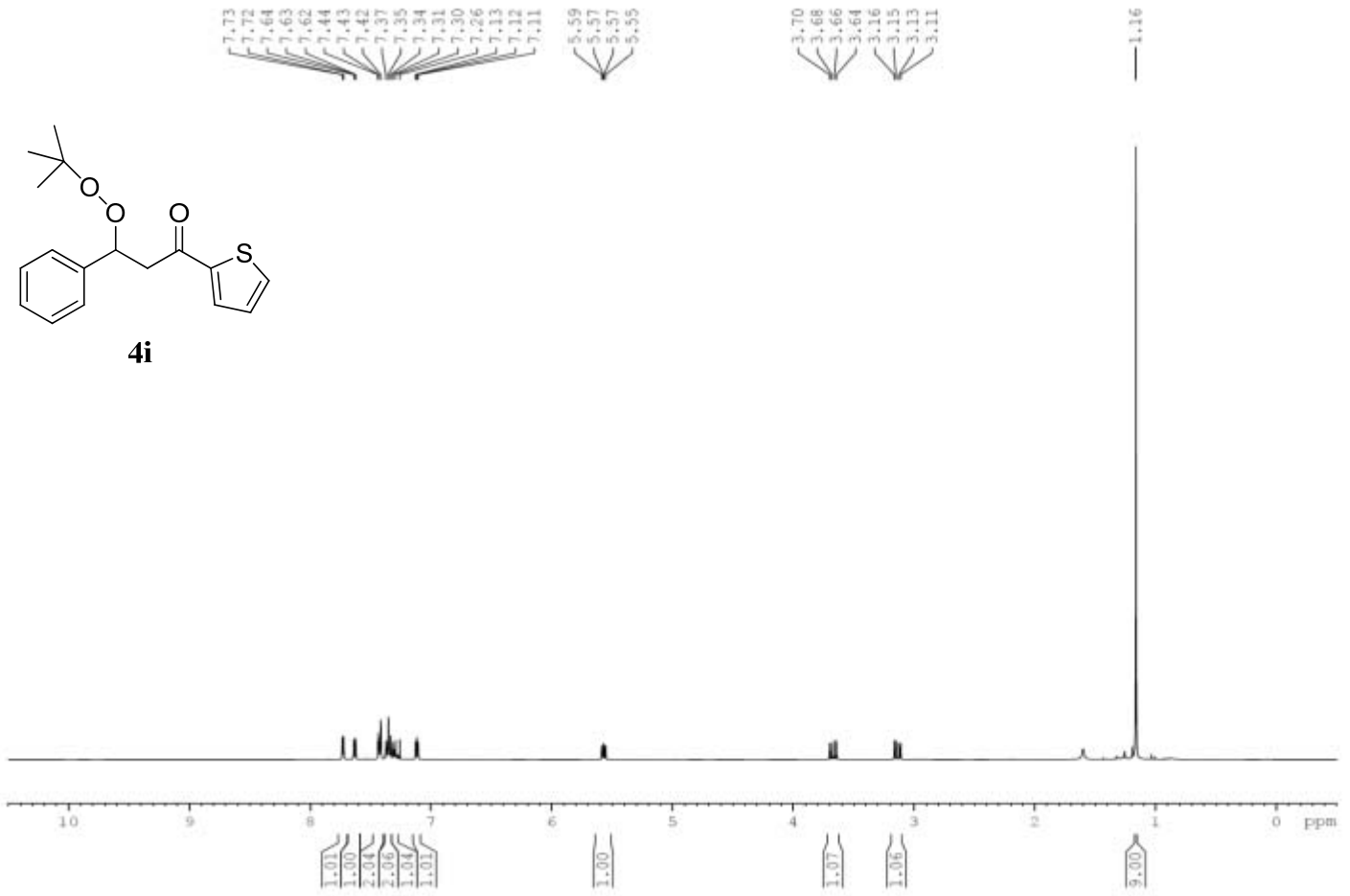


4g

```
Current Data Parameters
NAME       976-2-2-H-G-2.fid
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20131025
Time_                  0.00
INSTRUM              varian
PROBHD
PULPROG              s2pul
TD                   65536
SOLVENT              cdcl3
NS                     720
DS                       0
SWH              25510.203 Hz
FIDRES           0.389255 Hz
AQ               1.3107700 sec
RG                       4
DW                  19.600 usec
DE                  115.71 usec
TE                   298.0 K

F2 - Processing parameters
SI                   65536
SF           100.5218578 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

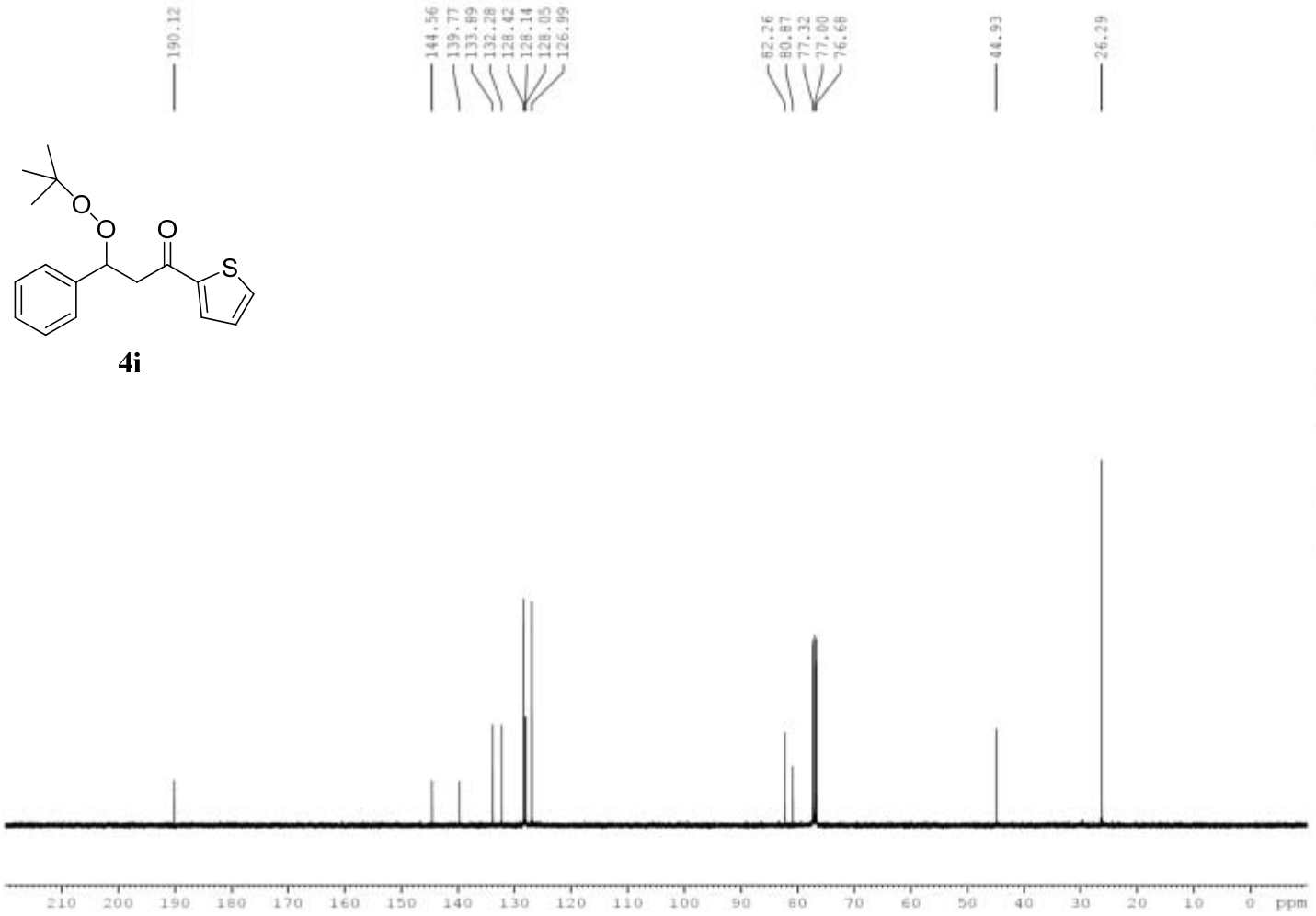

8.52
8.50
7.99
7.97
7.89
7.88
7.86
7.57
7.56
7.54
7.52
7.49
7.48
7.45
7.43
7.38
7.36
7.36
7.34
7.32
7.31
7.26
5.70
5.68
5.67
3.87
3.86
3.83
3.81
3.34
3.32
3.30
3.28
1.19
O
O
O
4h
Current Data Parameters
NAME RA-205-OP.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140108
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 8
DS 0
SWH 6402.049 Hz
FIDRES 0.195375 Hz
AQ 2.5559540 sec
RG 4
DW 78.100 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627618 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.04
1.09
2.05
3.09
2.01
3.06
1.00
1.06
1.05
9.02

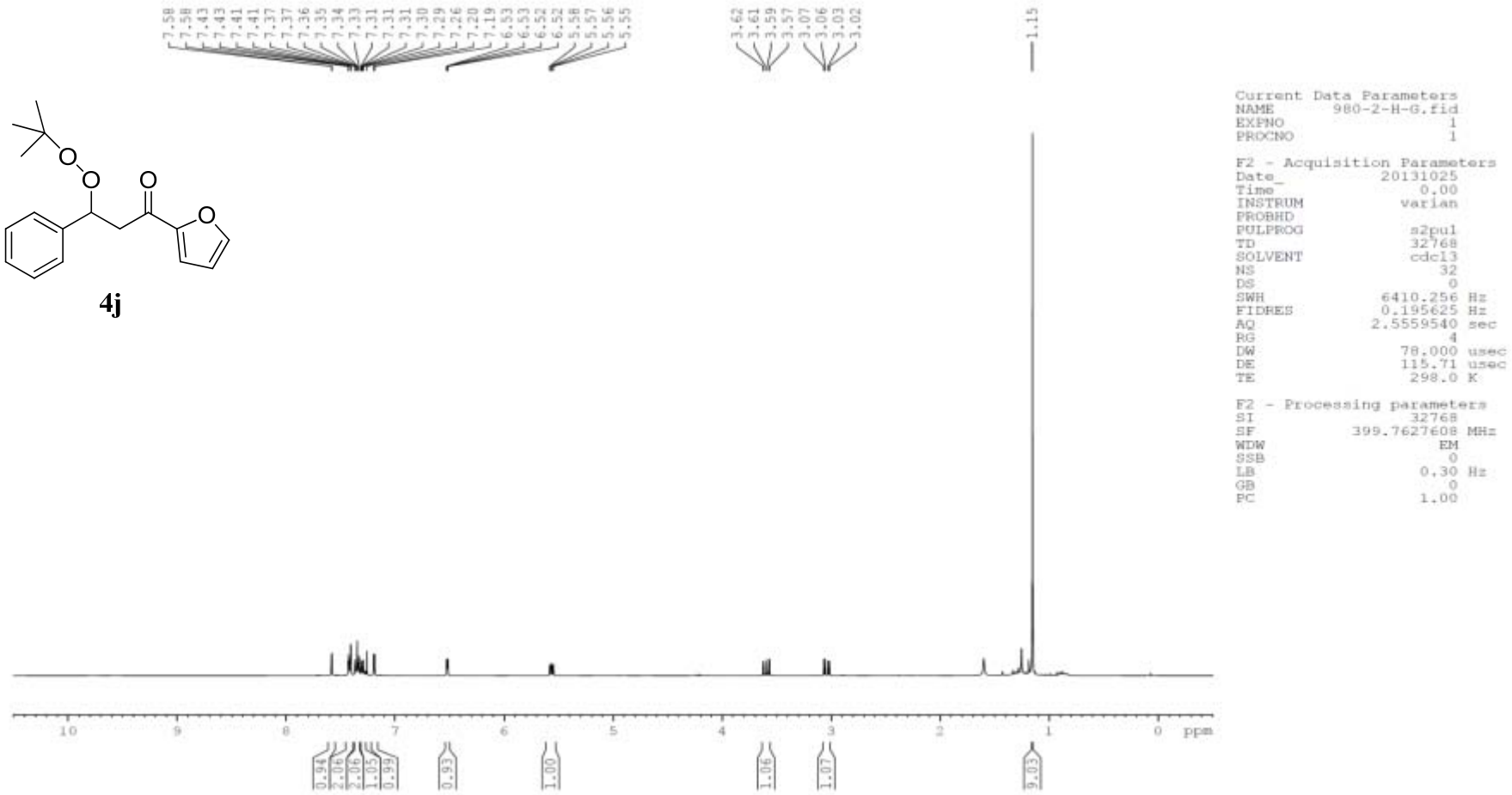

201.28
139.93
136.07
133.87
132.70
130.08
128.41
128.32
128.11
127.81
127.81
127.10
126.42
125.82
124.29
82.40
80.79
77.32
77.00
76.68
47.68
26.39
O
O
O
4h
Current Data Parameters
NAME RA-205-OP-C-4.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140108
Time_ 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 032
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218590 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

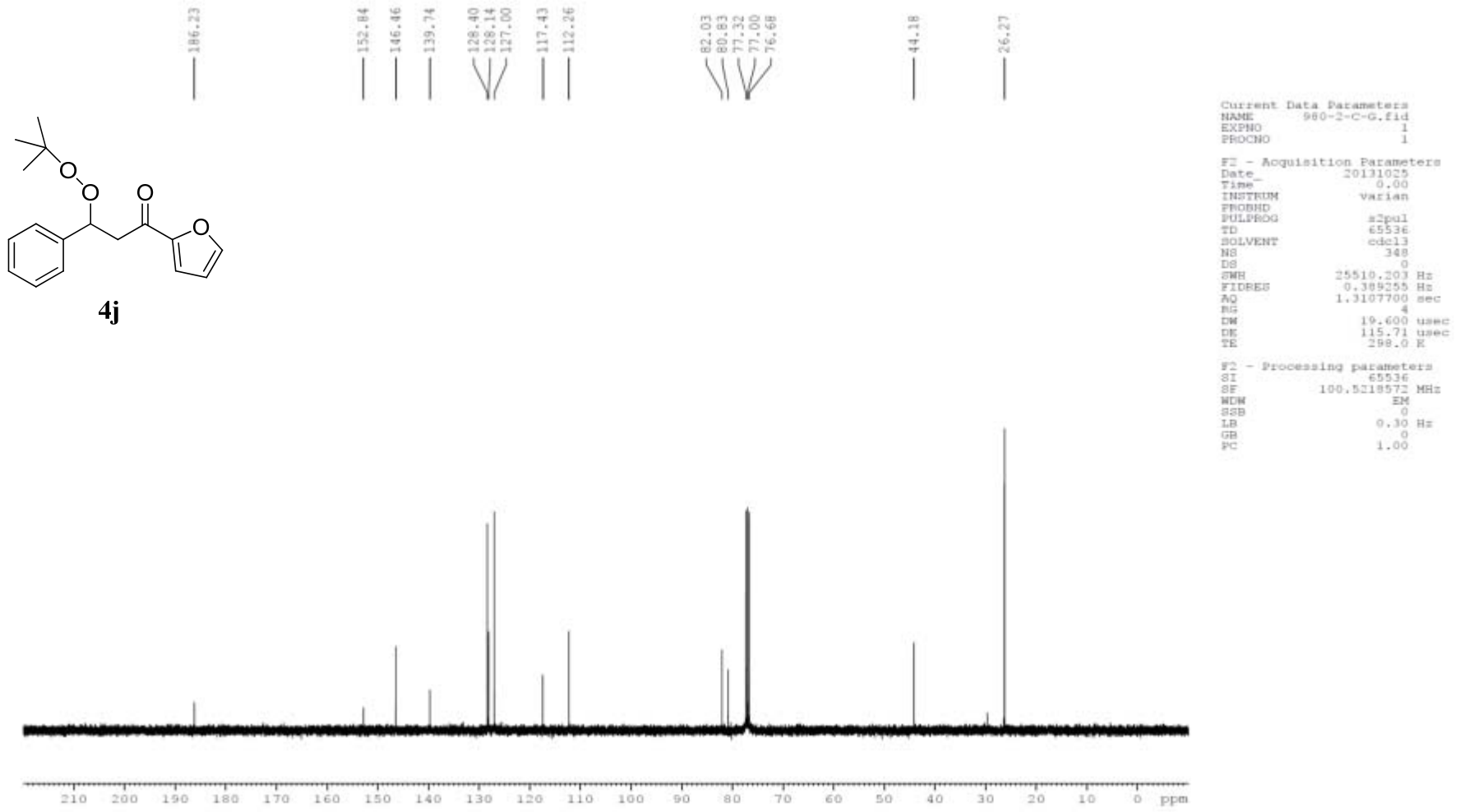


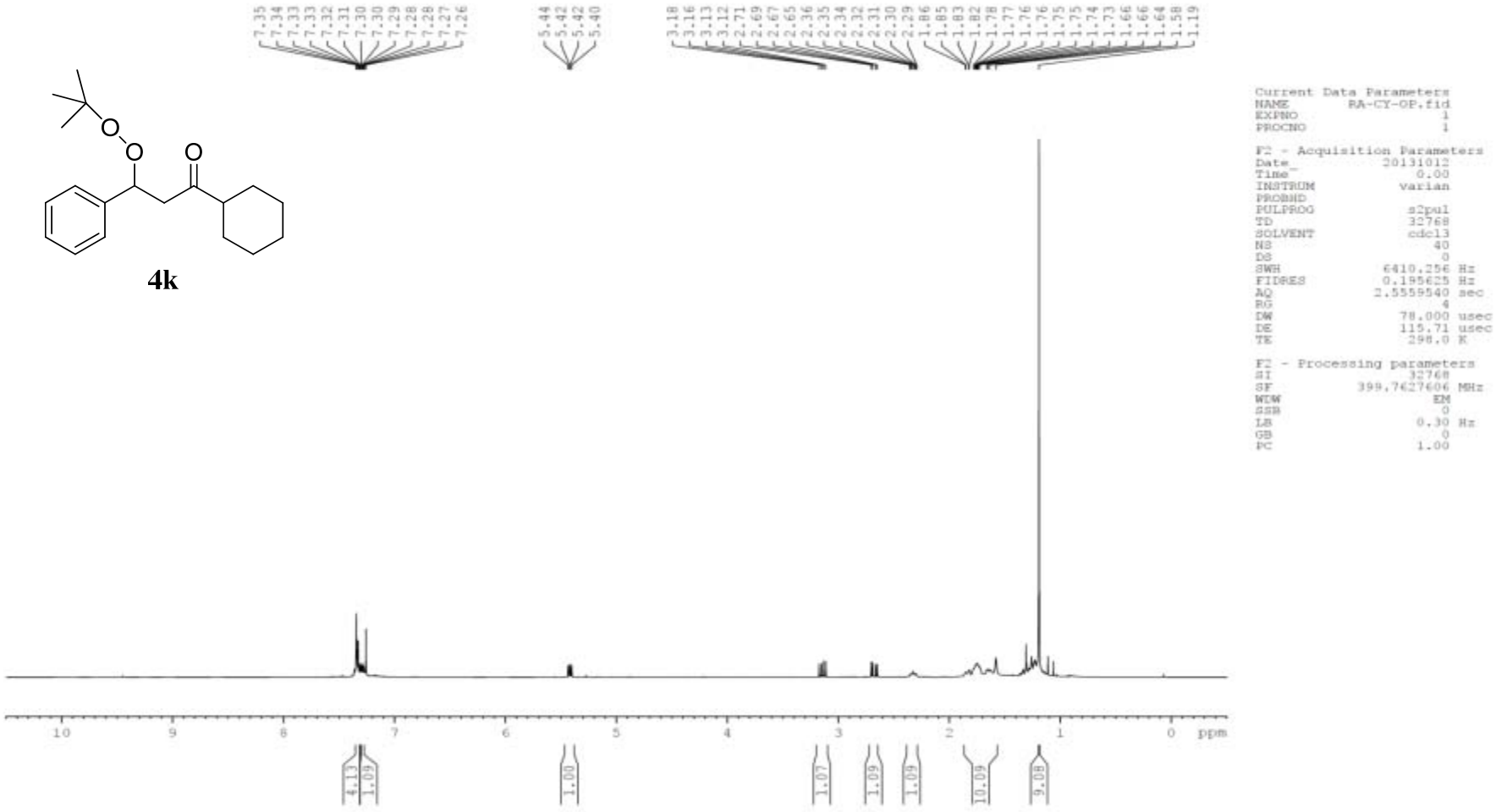
4i

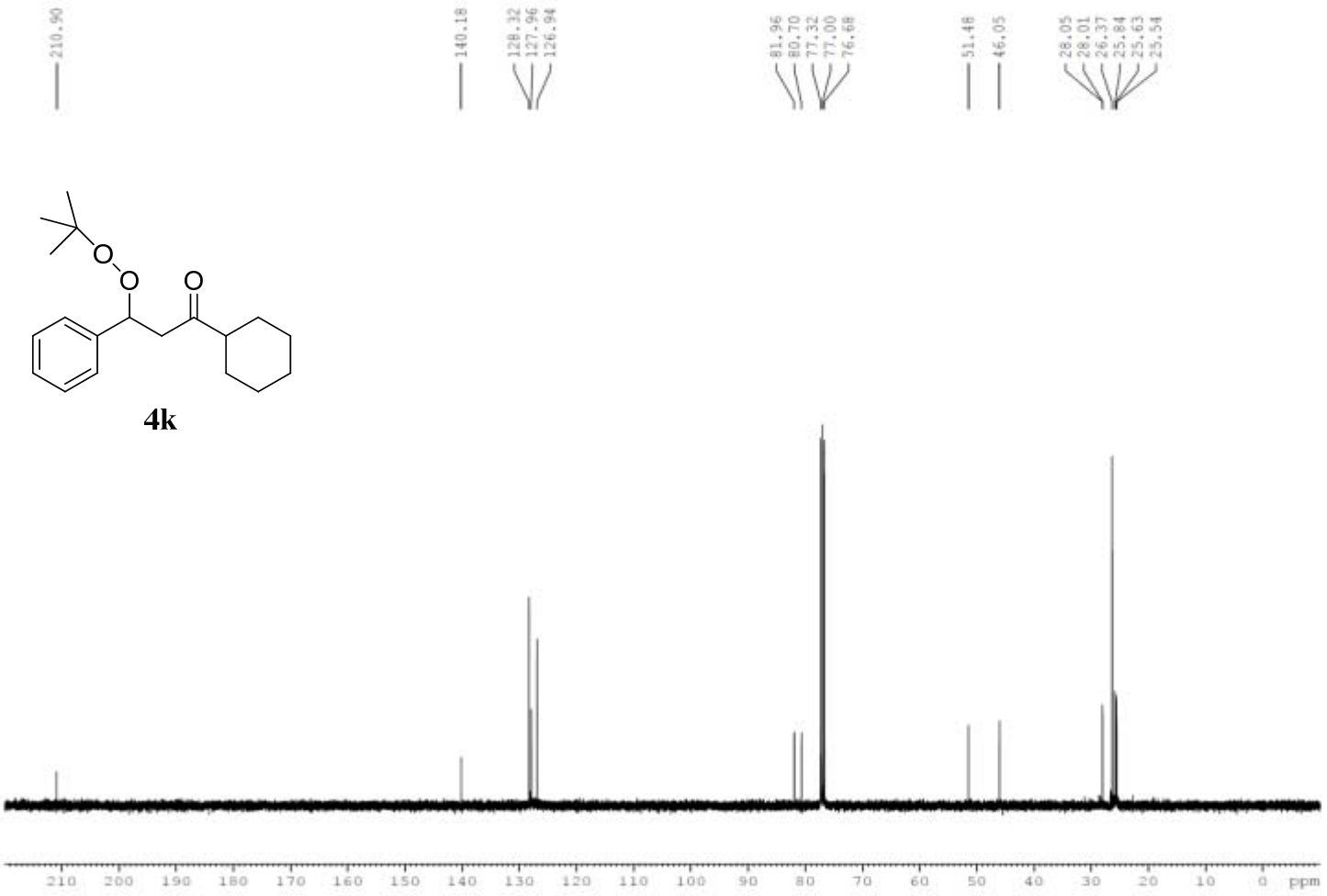
```
Current Data Parameters
NAME       979-2-1-H.fid
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131024
Time                0.00
INSTRUM          varian
PROBHD
PULPROG           s2pul
TD                32768
SOLVENT           cdcl3
NS                   32
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                    4
DW               78.000 usec
DE               115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                32768
SF        399.7627608 MHz
WDW                  EM
SSB                   0
LB             0.30 Hz
GB                    0
PC                 1.00
```

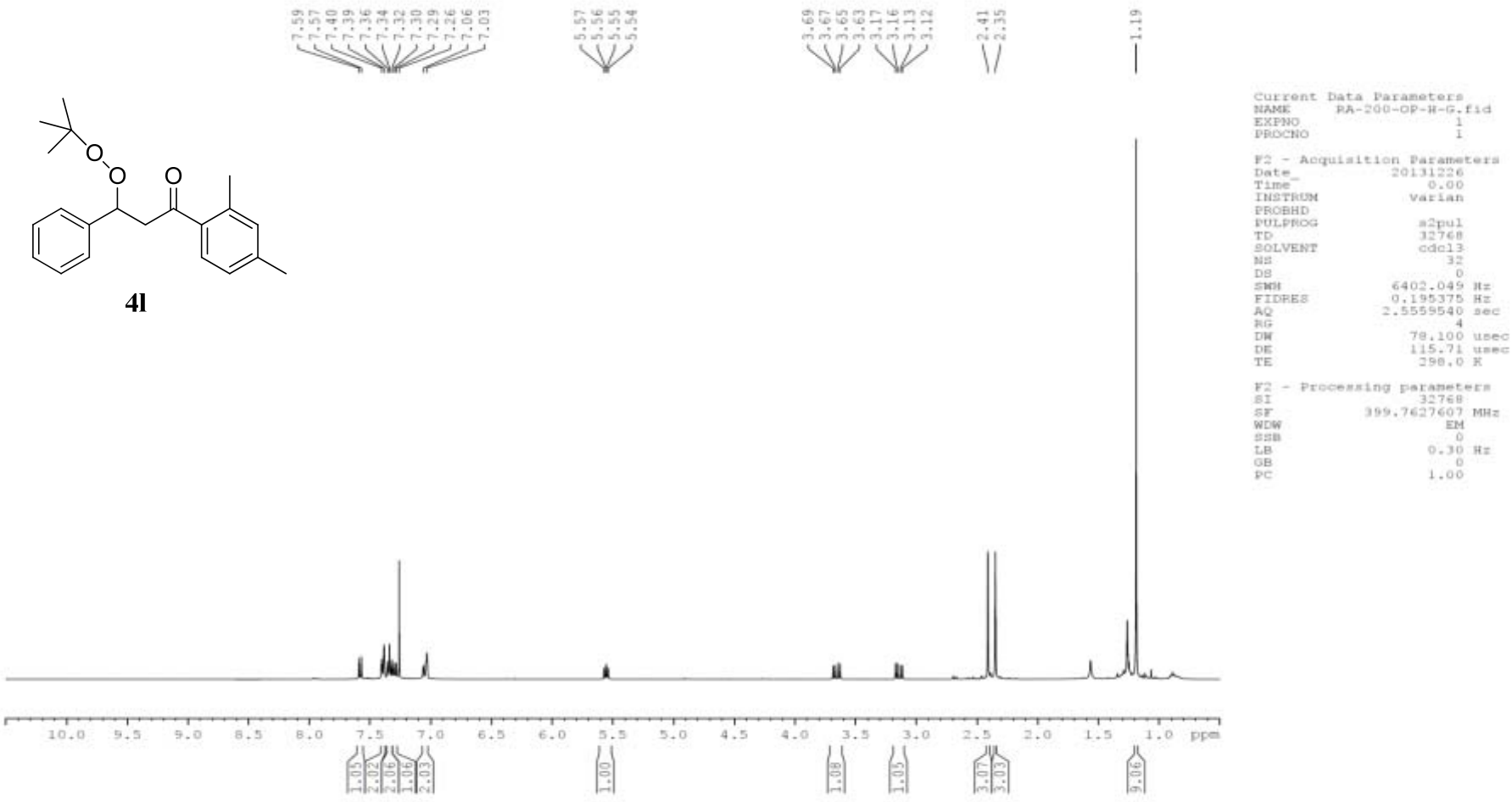

190.12
144.56
139.77
133.89
132.28
128.42
128.14
128.05
126.99
82.26
80.87
77.32
77.00
76.68
44.93
26.29
O
O
O
S
4i
Current Data Parameters
NAME 979-2-1-C-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131024
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 880
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218578 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

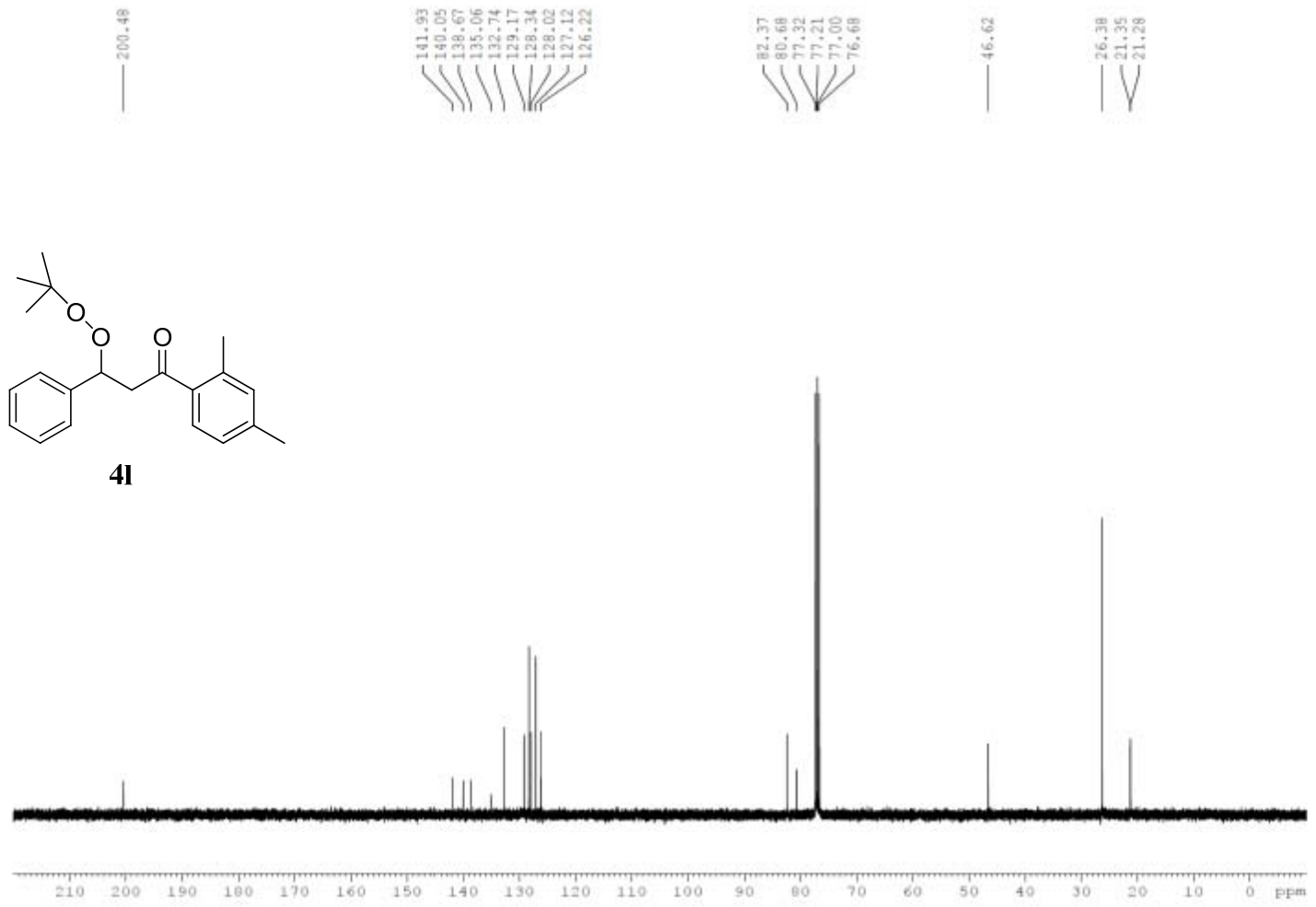

7.58
7.58
7.43
7.43
7.41
7.41
7.37
7.37
7.36
7.35
7.34
7.33
7.31
7.31
7.31
7.30
7.29
7.26
7.20
7.19
6.53
6.53
6.52
6.52
5.58
5.57
5.56
5.55
3.62
3.61
3.59
3.57
3.07
3.06
3.03
3.02
1.15
4j
Current Data Parameters
NAME 980-2-H-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131025
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627608 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
2.06
2.06
1.05
0.99
0.93
1.00
1.06
1.07
9.03


186.23
152.84
146.46
139.74
128.40
128.14
127.00
117.43
112.26
82.03
80.83
77.32
77.00
76.68
44.18
26.27
O
O
O
O
4j
Current Data Parameters
NAME 980-2-C-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131025
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 348
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218572 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

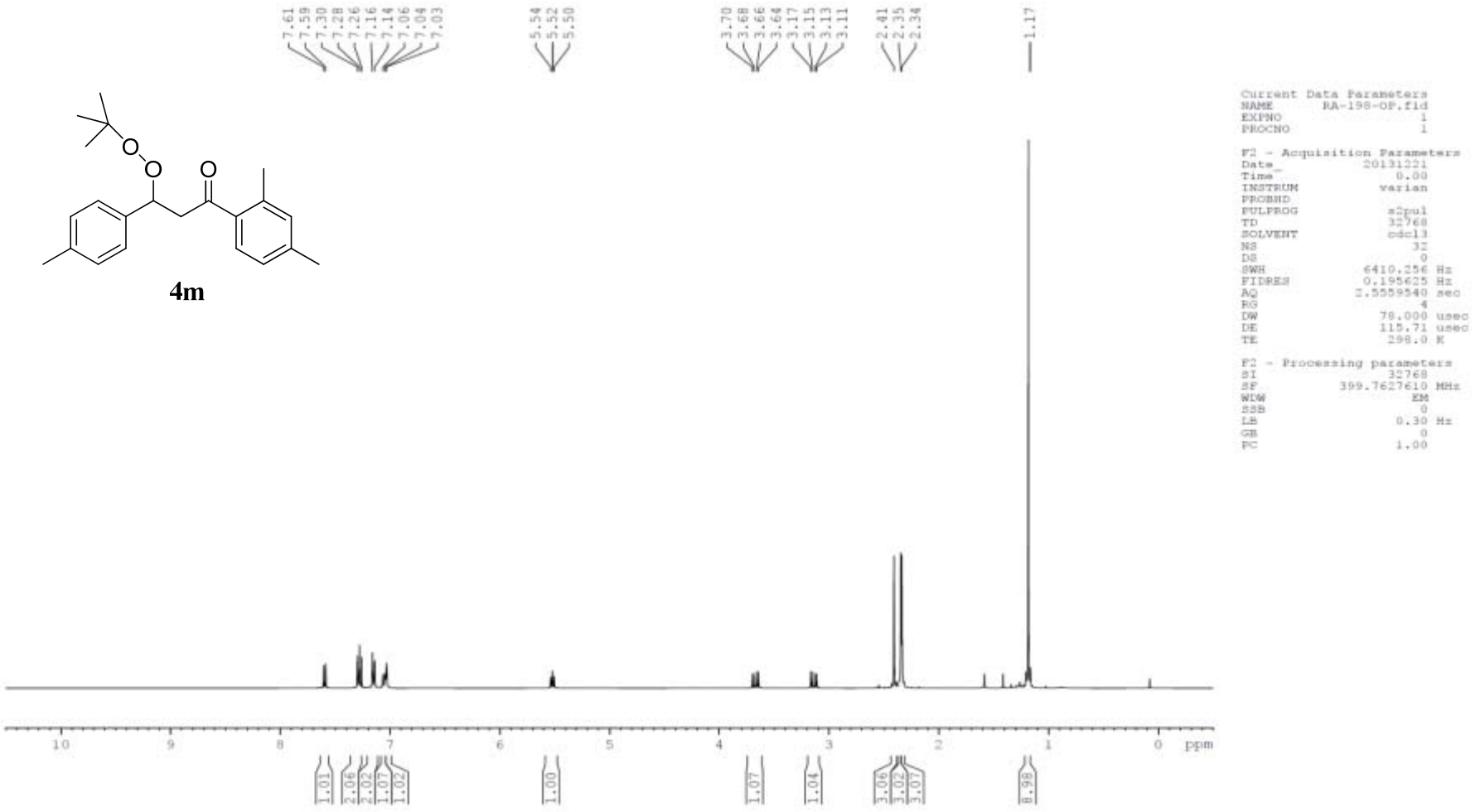

7.35
7.34
7.33
7.33
7.32
7.31
7.30
7.30
7.29
7.28
7.28
7.27
7.26
5.44
5.42
5.42
5.40
3.18
3.16
3.13
3.12
2.71
2.69
2.67
2.65
2.36
2.35
2.34
2.32
2.31
2.30
2.29
1.86
1.85
1.83
1.82
1.78
1.77
1.76
1.76
1.75
1.75
1.74
1.73
1.66
1.66
1.64
1.58
1.19
O
O
O
4k
Current Data Parameters
NAME RA-CY-OP.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131012
Time_ 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 40
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627606 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
4.13
1.09
1.00
1.07
1.09
1.09
10.09
9.08


210.90
140.18
128.32
127.96
126.94
81.96
80.70
77.32
77.00
76.68
51.48
46.05
28.05
28.01
26.37
25.84
25.63
25.54
4k
Current Data Parameters
NAME RA-CY-OP-C.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131012
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 912
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218566 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.59
7.57
7.40
7.39
7.36
7.34
7.32
7.30
7.29
7.26
7.06
7.03
5.57
5.56
5.55
5.54
3.69
3.67
3.65
3.63
3.17
3.16
3.13
3.12
2.41
2.35
1.19
O
O
O
4l
Current Data Parameters
NAME RA-200-OP-R-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131226
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 32
DS 0
SWH 6402.049 Hz
FIDRES 0.195375 Hz
AQ 2.5559540 sec
RG 4
DW 78.100 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627607 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.05
2.02
2.06
1.06
2.03
1.00
1.08
1.05
3.07
3.03
9.06

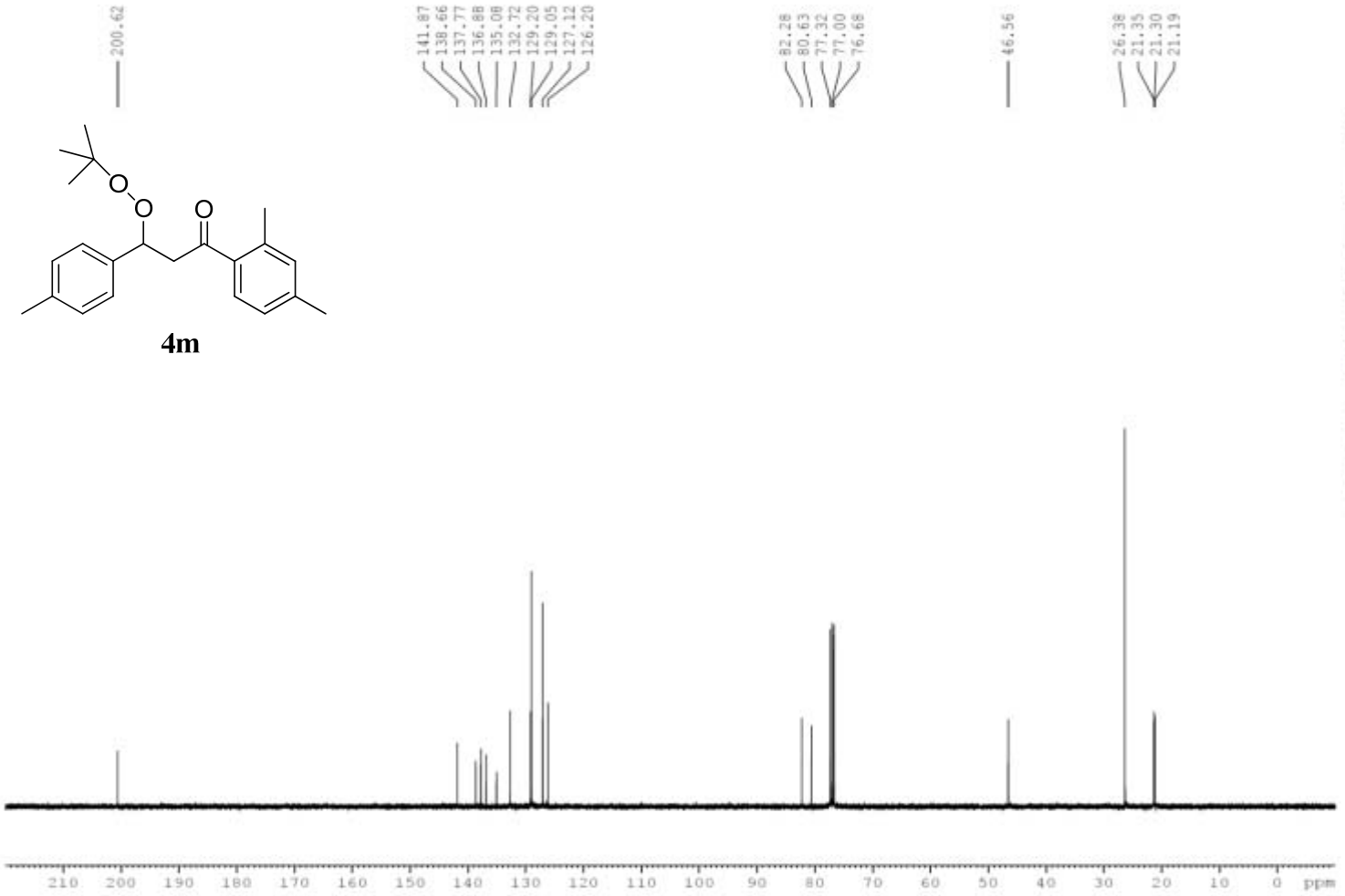


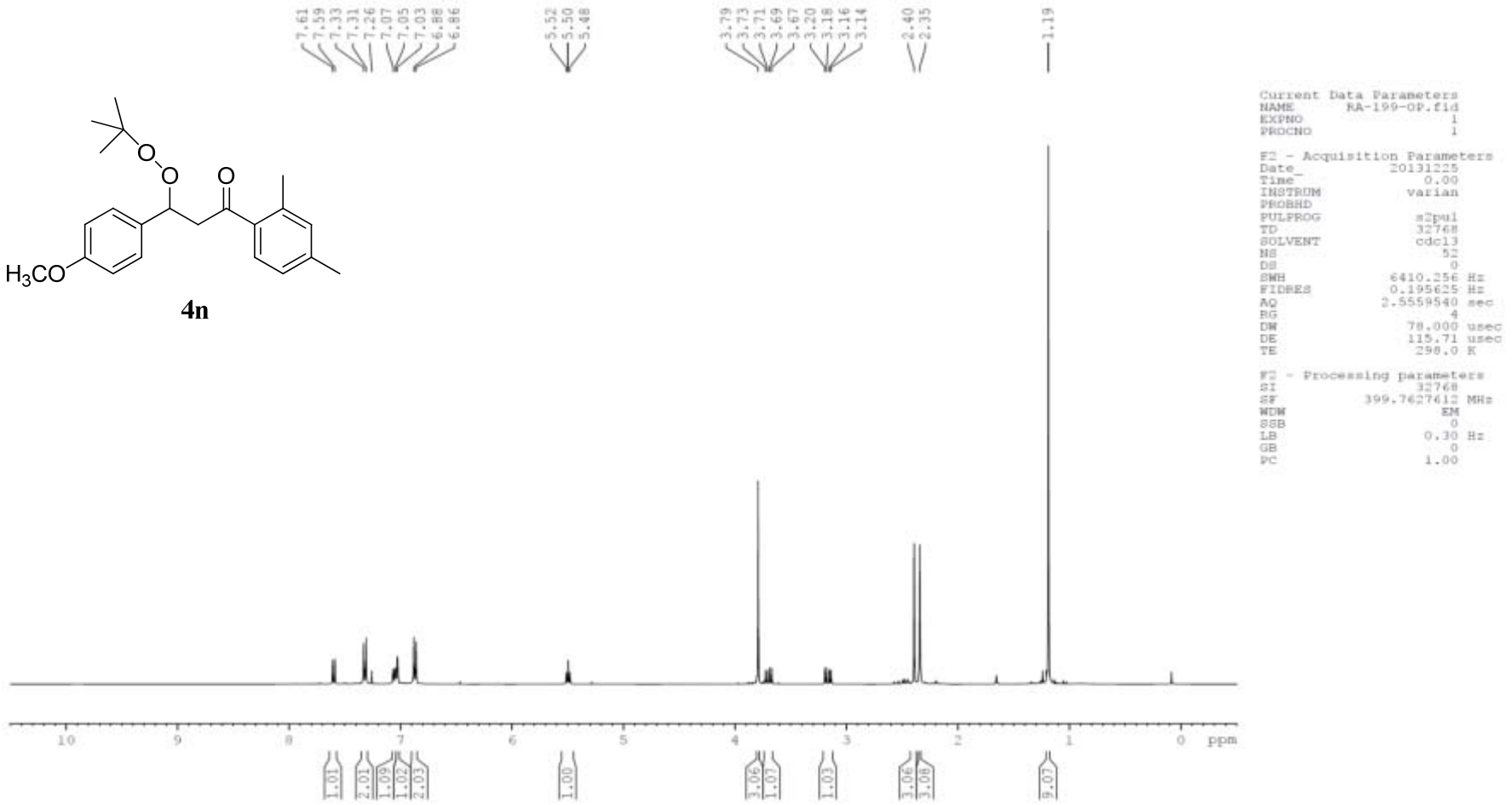

```
Current Data Parameters
NAME      RA-200-1-2-C.fid
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20131216
Time                  0.00
INSTRUM             varian
PROBHD
PULPROG              s2pul
TD                   65536
SOLVENT              cdcl3
NS                    1196
DS                       0
SWH              25510.203 Hz
FIDRES           0.389255 Hz
AQ               1.3107700 sec
RG                       4
DW                  19.600 usec
DE                  115.71 usec
TE                  298.0 K

F2 - Processing parameters
SI                   65536
SF           100.5218574 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

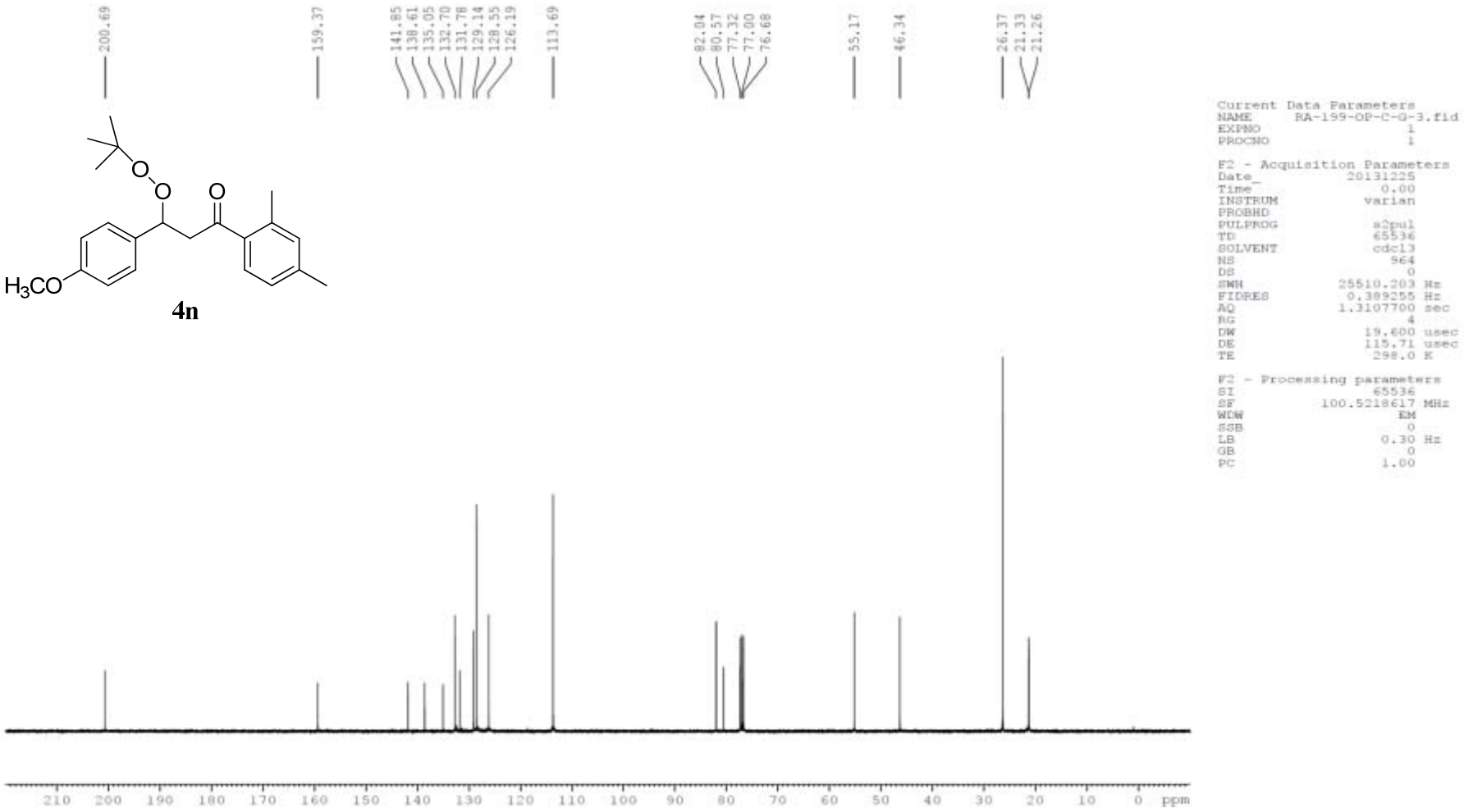


4m

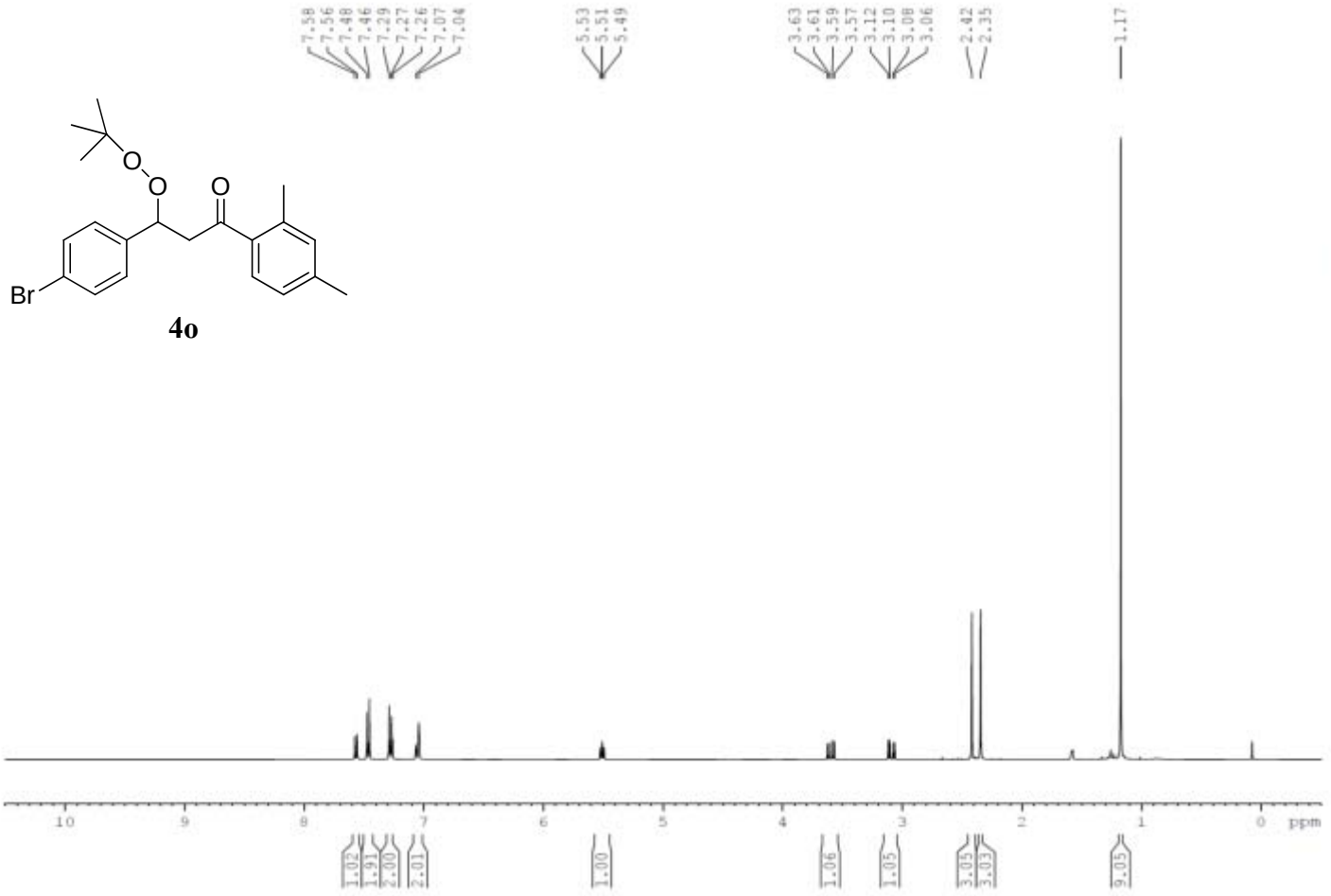
```
Current Data Parameters
NAME          RA-198-OP.fid
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20131221
Time               0.00
INSTRUM            varian
PROBHD
PULPROG            s2pul
TD                 32768
SOLVENT            cdcl3
NS                 32
DS                 0
SWH                6410.256 Hz
FIDRES             0.195625 Hz
AQ                 2.5559540 sec
RG                 4
DW                 78.000 usec
DE                 115.71 usec
TE                 298.0 K

F2 - Processing parameters
SI                 32768
SF                 399.7627610 MHz
WDW                EM
SSB                0
LB                 0.30 Hz
GB                 0
PC                 1.00
```


200.62
141.87
138.66
137.77
136.88
135.08
132.72
129.20
129.05
127.12
126.20
82.28
80.63
77.32
77.00
76.68
46.56
26.38
21.35
21.30
21.19
4m
Current Data Parameters
NAME RA-198-OP-C-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131221
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 724
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218586 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.61
7.59
7.33
7.31
7.26
7.07
7.05
7.03
6.88
6.86
5.52
5.50
5.48
3.79
3.73
3.71
3.69
3.67
3.20
3.18
3.16
3.14
2.40
2.35
1.19
H3CO
O
O
O
4n
Current Data Parameters
NAME RA-199-OP.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131225
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 52
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7827612 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.01
1.09
1.02
2.03
1.00
3.06
1.07
1.03
3.06
3.08
9.07

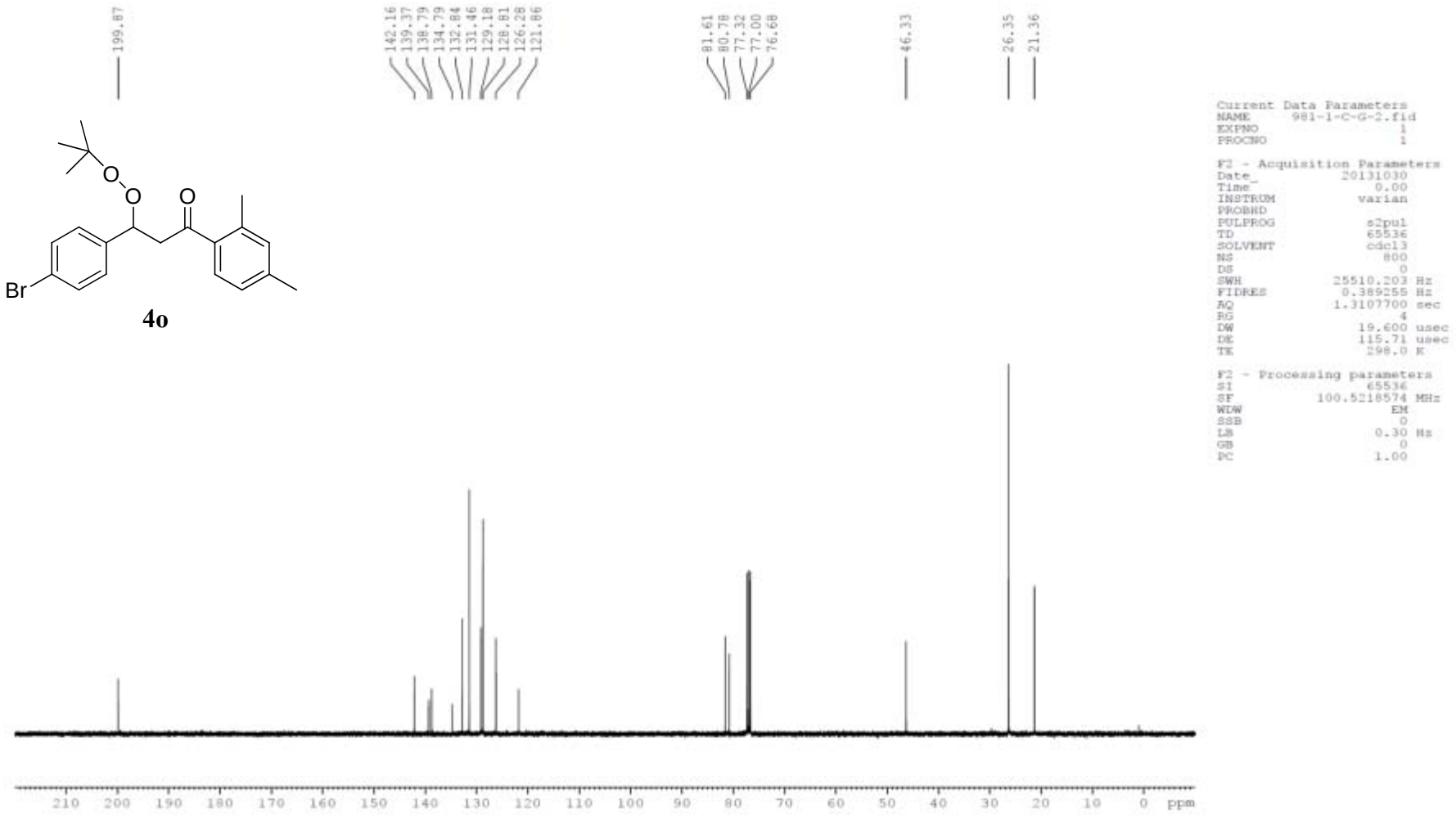



```
Current Data Parameters
NAME      RA-199-OP-C-G-3.fid
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20131225
Time                    0.00
INSTRUM                varian
PROBHD
PULPROG                s2pul
TD                     65536
SOLVENT                cdcl3
NS                       964
DS                         0
SWH                25510.203 Hz
FIDRES              0.389255 Hz
AQ                  1.3107700 sec
RG                         4
DW                    19.600 usec
DE                    115.71 usec
TE                     298.0 K

F2 - Processing parameters
SI                     65536
SF            100.5218617 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

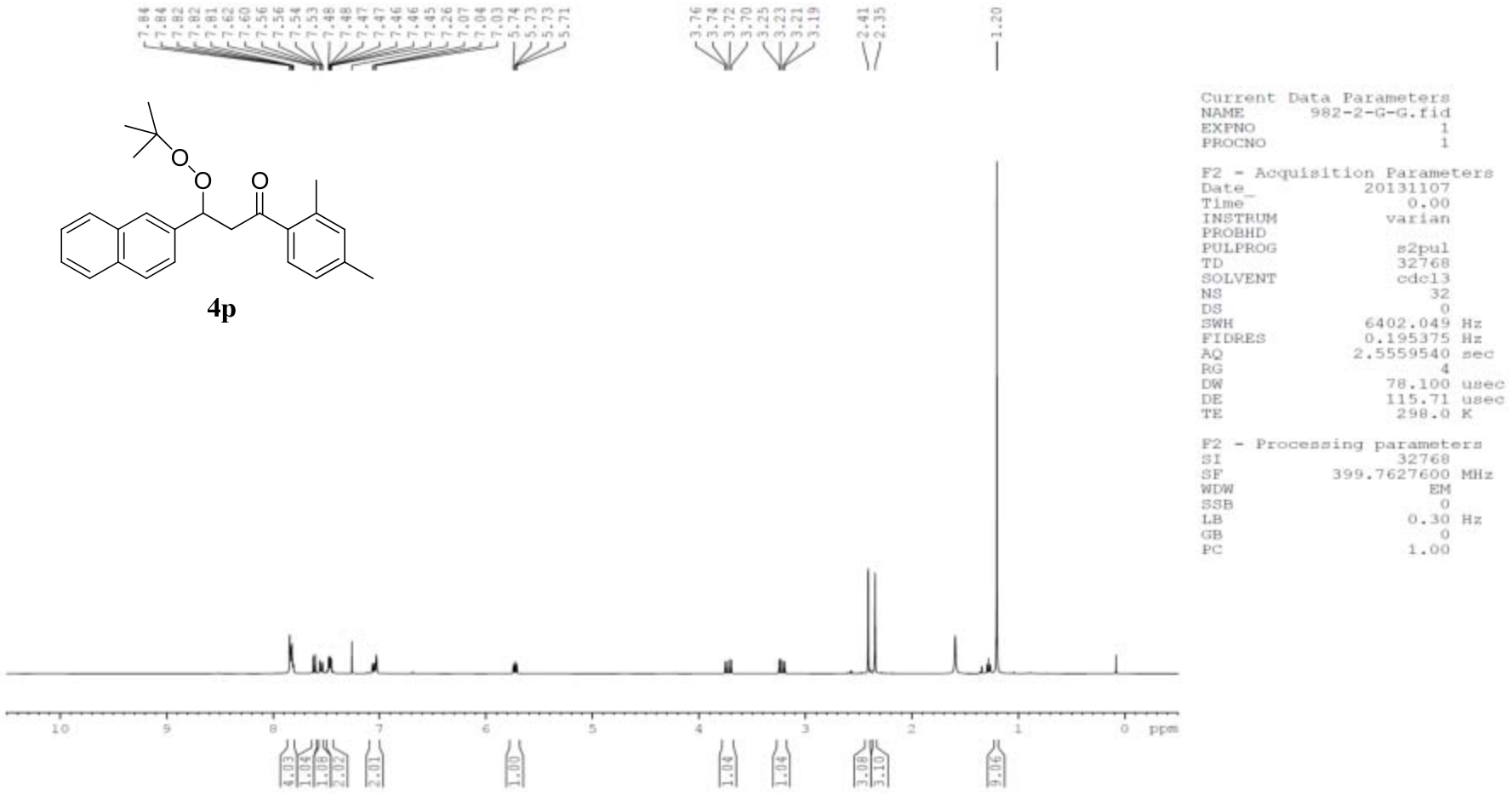


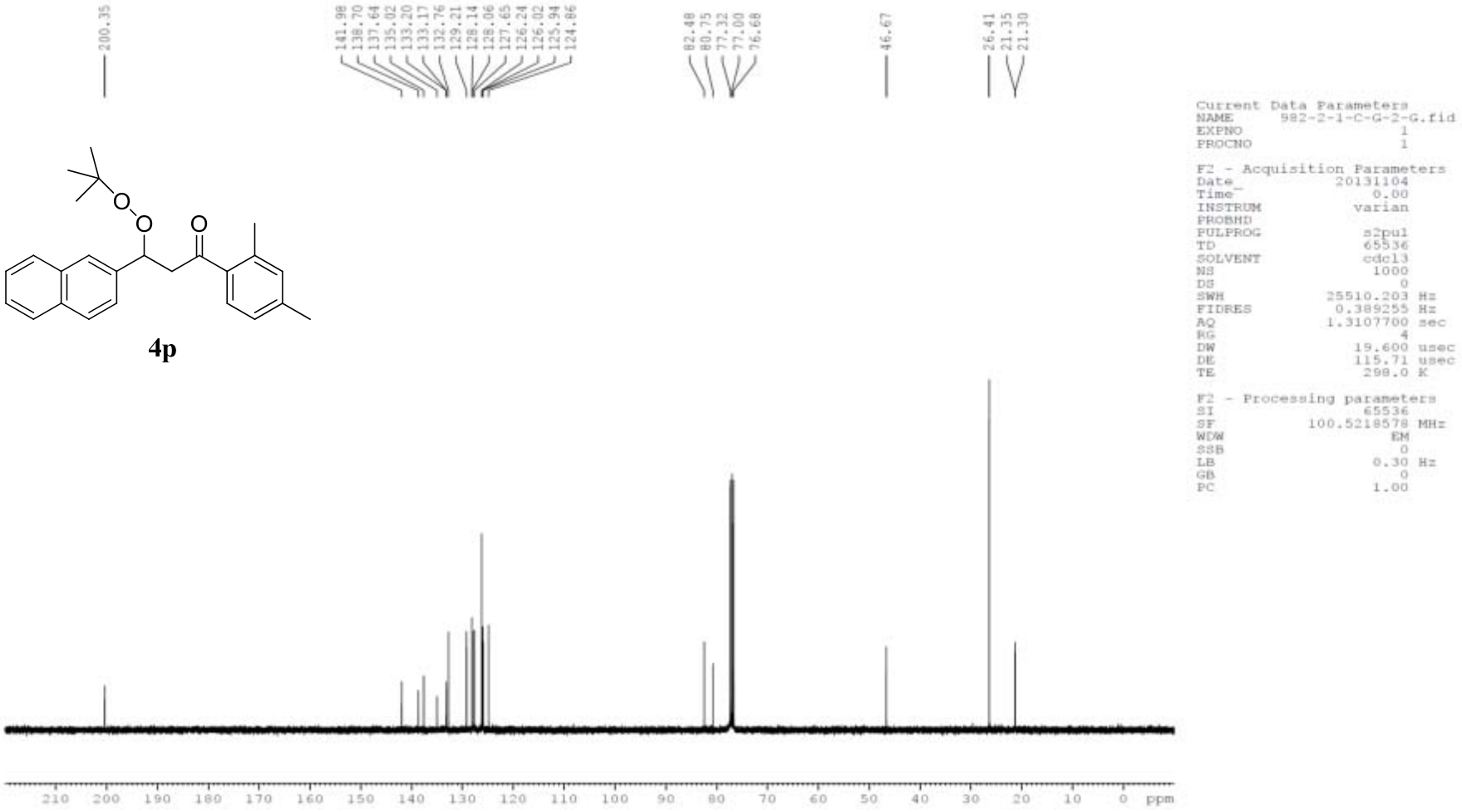
4o

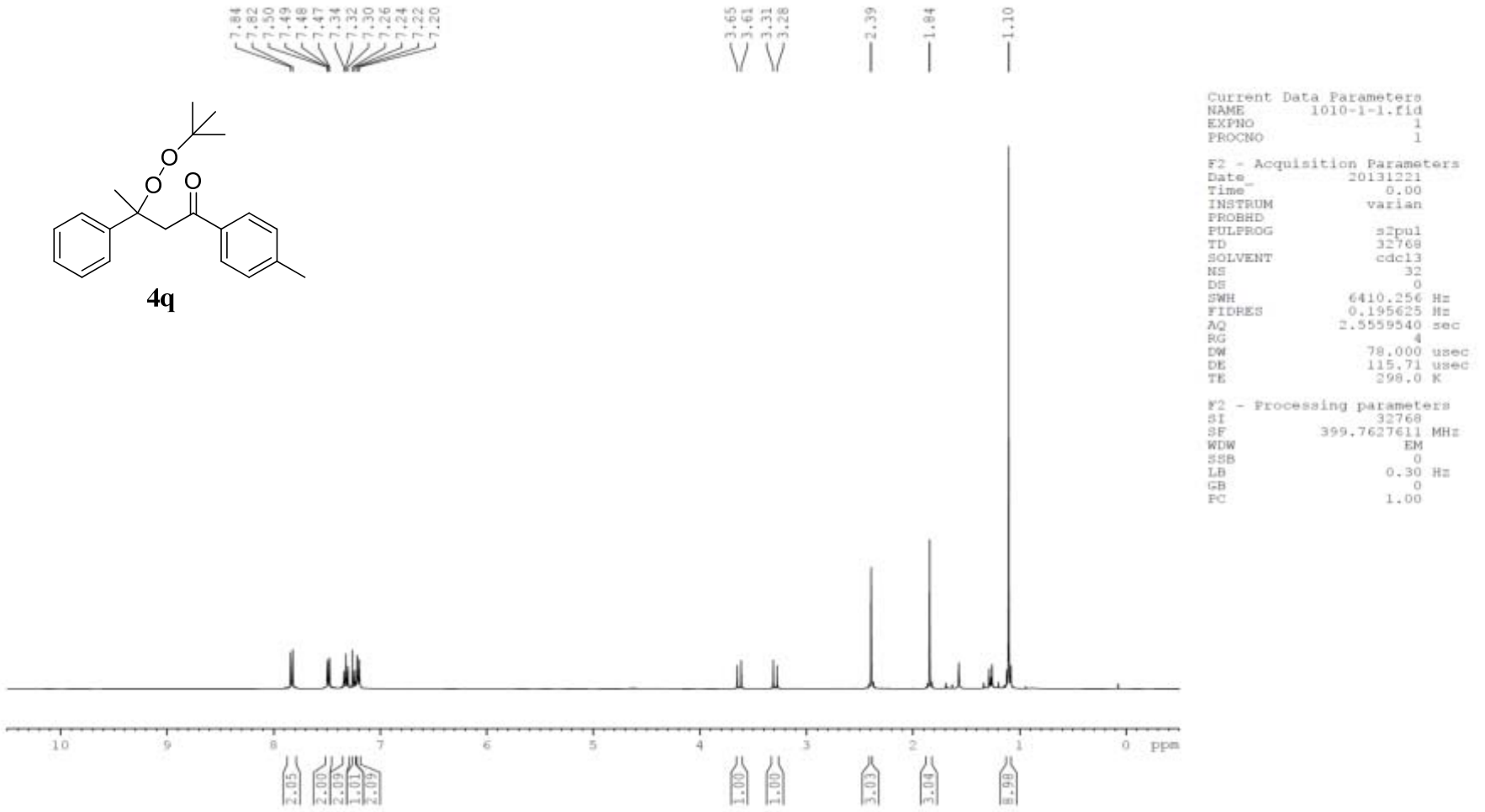
```
Current Data Parameters
NAME      981-1-H-G-1.fid
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131030
Time                 0.00
INSTRUM           varian
PROBHD
PULPROG             s2pul
TD                  32768
SOLVENT             cdcl3
NS                     32
DS                      0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ                2.5559540 sec
RG                      4
DW                 78.000 usec
DE                 115.71 usec
TE                 298.0 K

F2 - Processing parameters
SI                  32768
SF            399.7627605 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

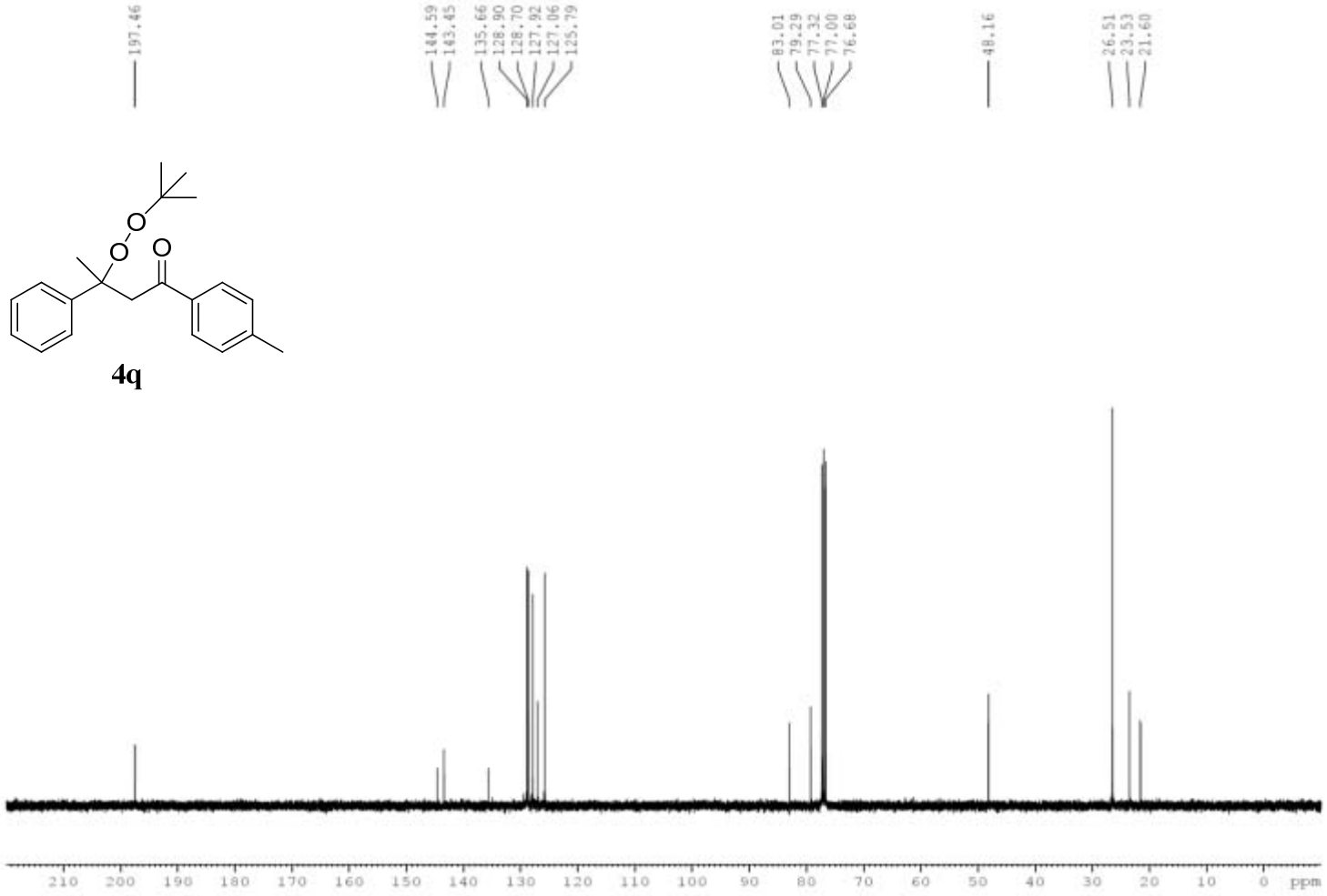

199.87
142.16
139.37
138.79
134.79
132.84
131.46
129.18
128.81
126.28
121.86
81.61
80.78
77.32
77.00
76.68
46.33
26.35
21.36
O
O
O
Br
Current Data Parameters
NAME 981-1-C-G-2.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131030
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 800
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218574 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

4o

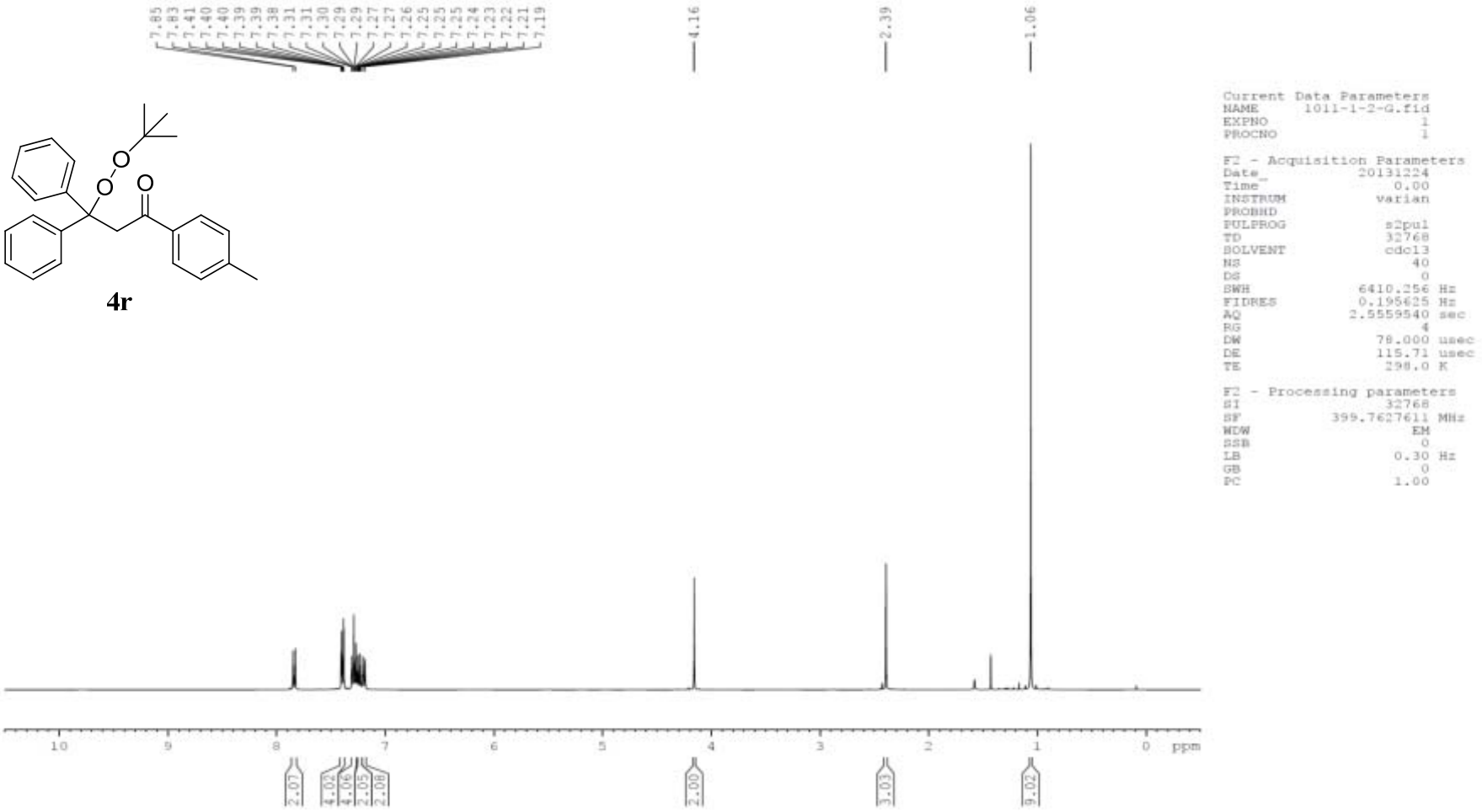

7.84
7.84
7.82
7.82
7.81
7.62
7.60
7.56
7.56
7.54
7.53
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.26
7.07
7.04
7.03
5.74
5.73
5.73
5.71
3.76
3.74
3.72
3.70
3.25
3.23
3.21
3.19
2.41
2.35
1.20
O
O
O
4p
Current Data Parameters
NAME 982-2-G-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131107
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 32
DS 0
SWH 6402.049 Hz
FIDRES 0.195375 Hz
AQ 2.5559540 sec
RG 4
DW 78.100 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627600 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
4.03
1.04
1.08
2.02
2.01
1.00
1.04
1.04
3.08
3.10
9.06

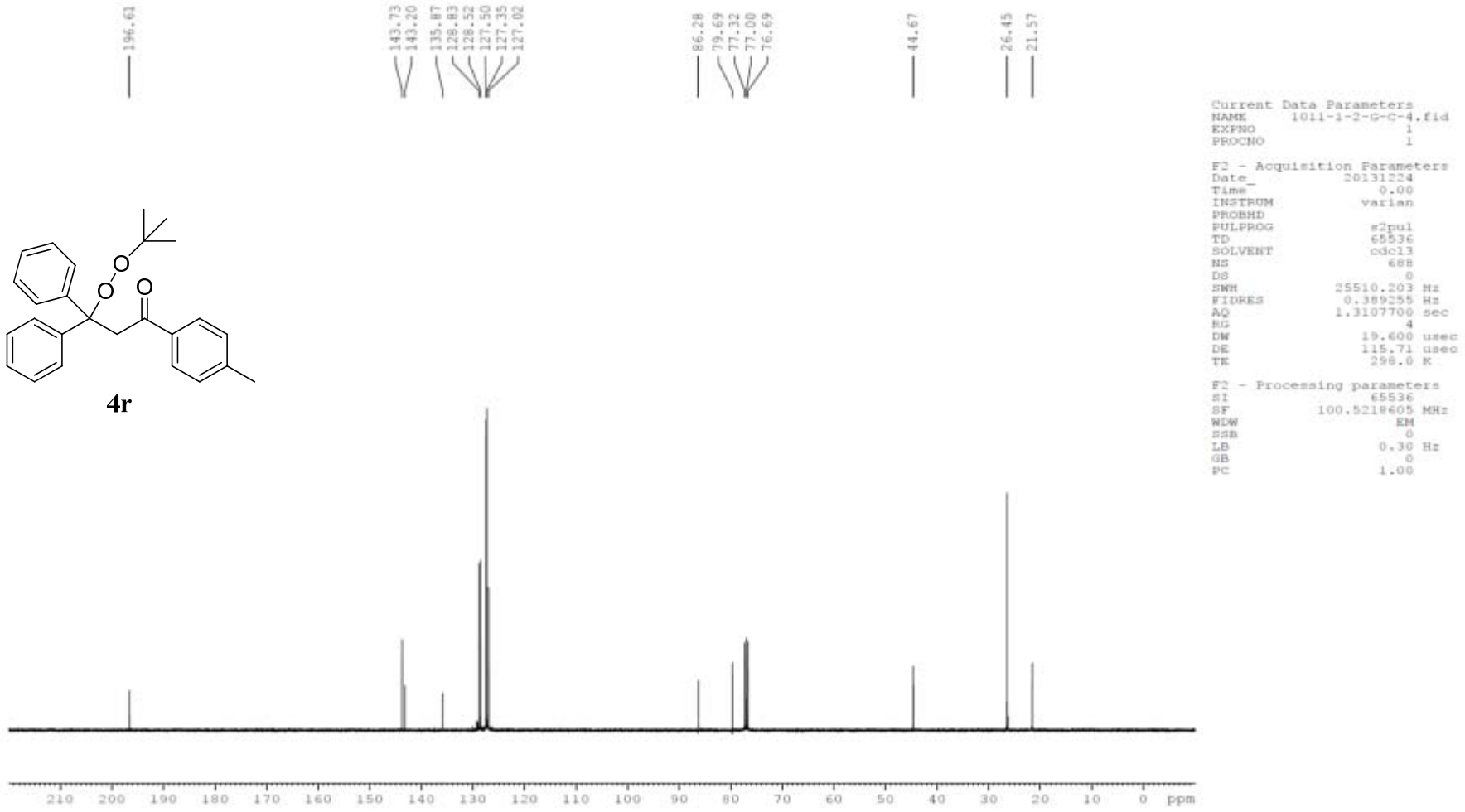

200.35
141.98
138.70
137.64
135.02
133.20
133.17
132.76
129.21
128.14
128.06
127.65
126.24
126.02
125.94
124.86
82.48
80.75
77.32
77.00
76.68
46.67
26.41
21.35
21.30
O
O
O
4p
Current Data Parameters
NAME 982-2-1-C-G-2-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131104
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 1000
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218578 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

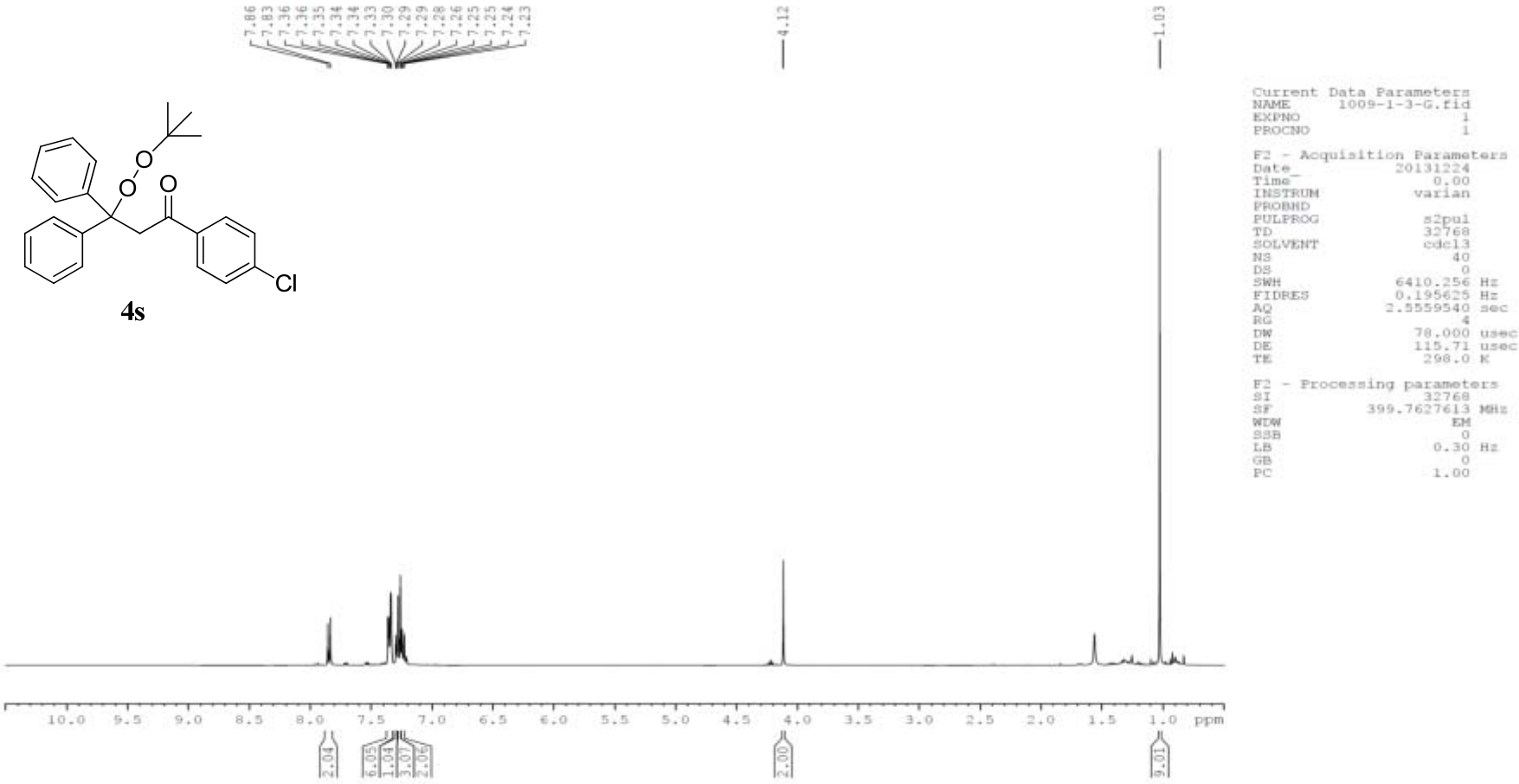

7.84
7.82
7.50
7.49
7.48
7.47
7.34
7.32
7.30
7.26
7.24
7.22
7.20
3.65
3.61
3.31
3.28
2.39
1.84
1.10
4q
Current Data Parameters
NAME 1010-1-1.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131221
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627611 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.05
2.00
2.09
1.01
2.09
1.00
1.00
3.03
3.04
8.98


197.46
144.59
143.45
135.66
128.90
128.70
127.92
127.06
125.79
83.01
79.29
77.32
77.00
76.68
48.16
26.51
23.53
21.60
Current Data Parameters
NAME 1010-1-1-C-G-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131221
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 640
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5216570 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

4q


7.85
7.83
7.41
7.40
7.40
7.39
7.39
7.38
7.31
7.31
7.30
7.29
7.29
7.27
7.27
7.26
7.25
7.25
7.24
7.23
7.22
7.21
7.19
4.16
2.39
1.06
O
O
O
4r
Current Data Parameters
NAME 1011-1-2-G.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131224
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 40
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 32768
SF 399.7627611 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.07
4.02
4.06
2.05
2.06
2.00
3.03
9.02


196.61
143.73
143.20
135.87
128.83
128.52
127.50
127.35
127.02
86.28
79.69
77.32
77.00
76.69
44.67
26.45
21.57
4r
Current Data Parameters
NAME 1011-1-2-G-C-4.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131224
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 688
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218605 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

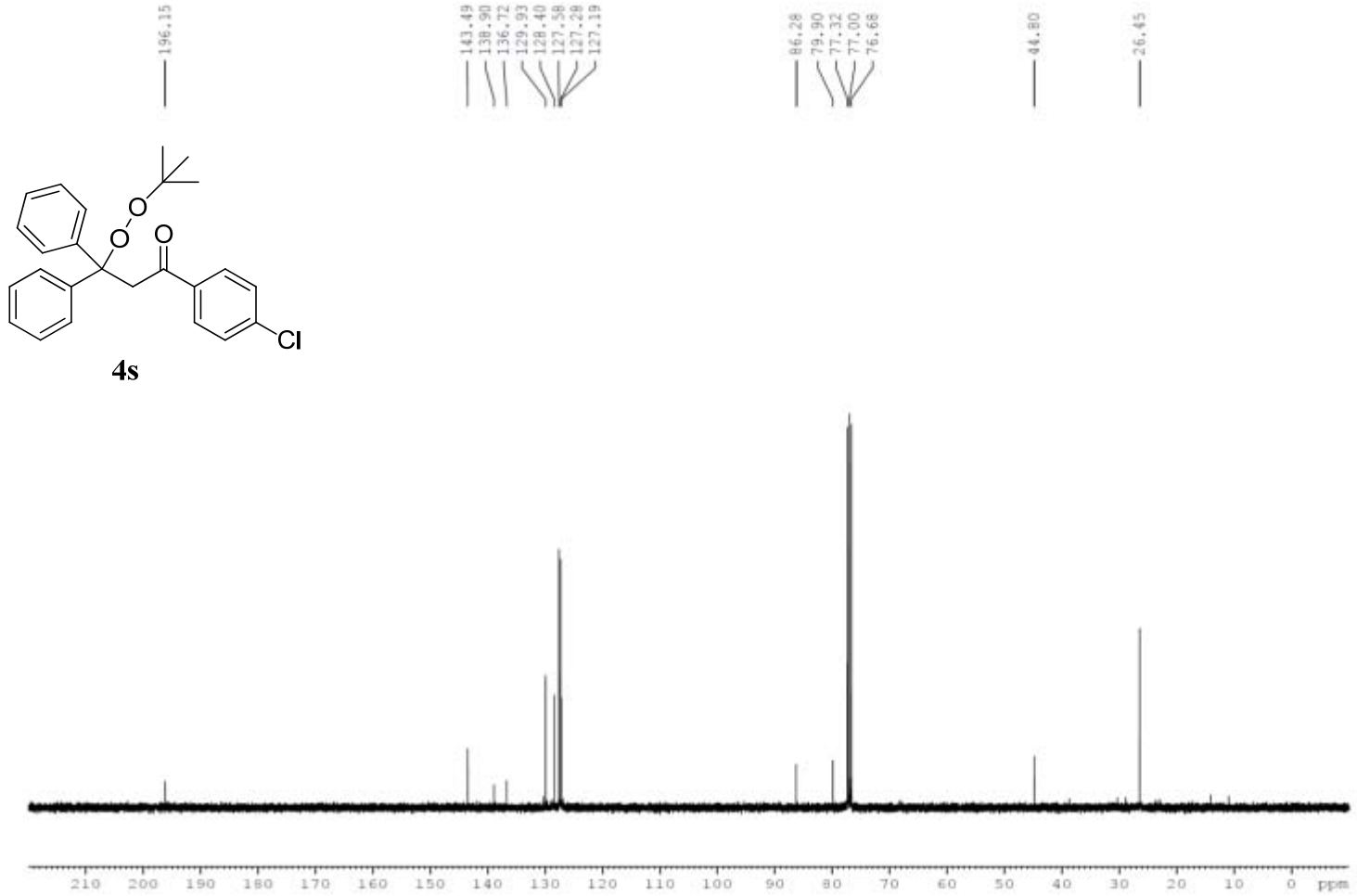


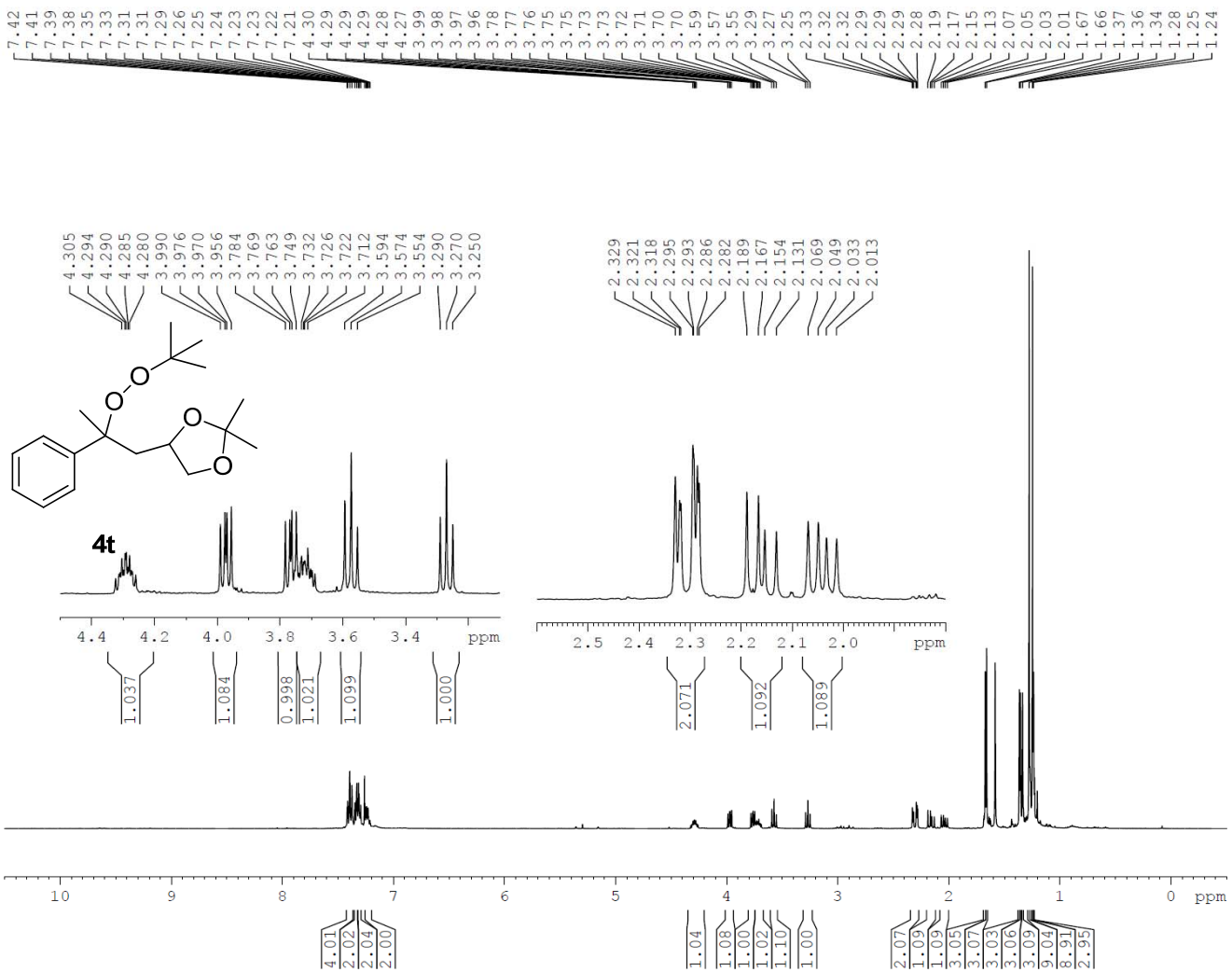
4s

Current Data Parameters
NAME 1009-1-3-G.fid
EXPNO 1
PROCNO 1

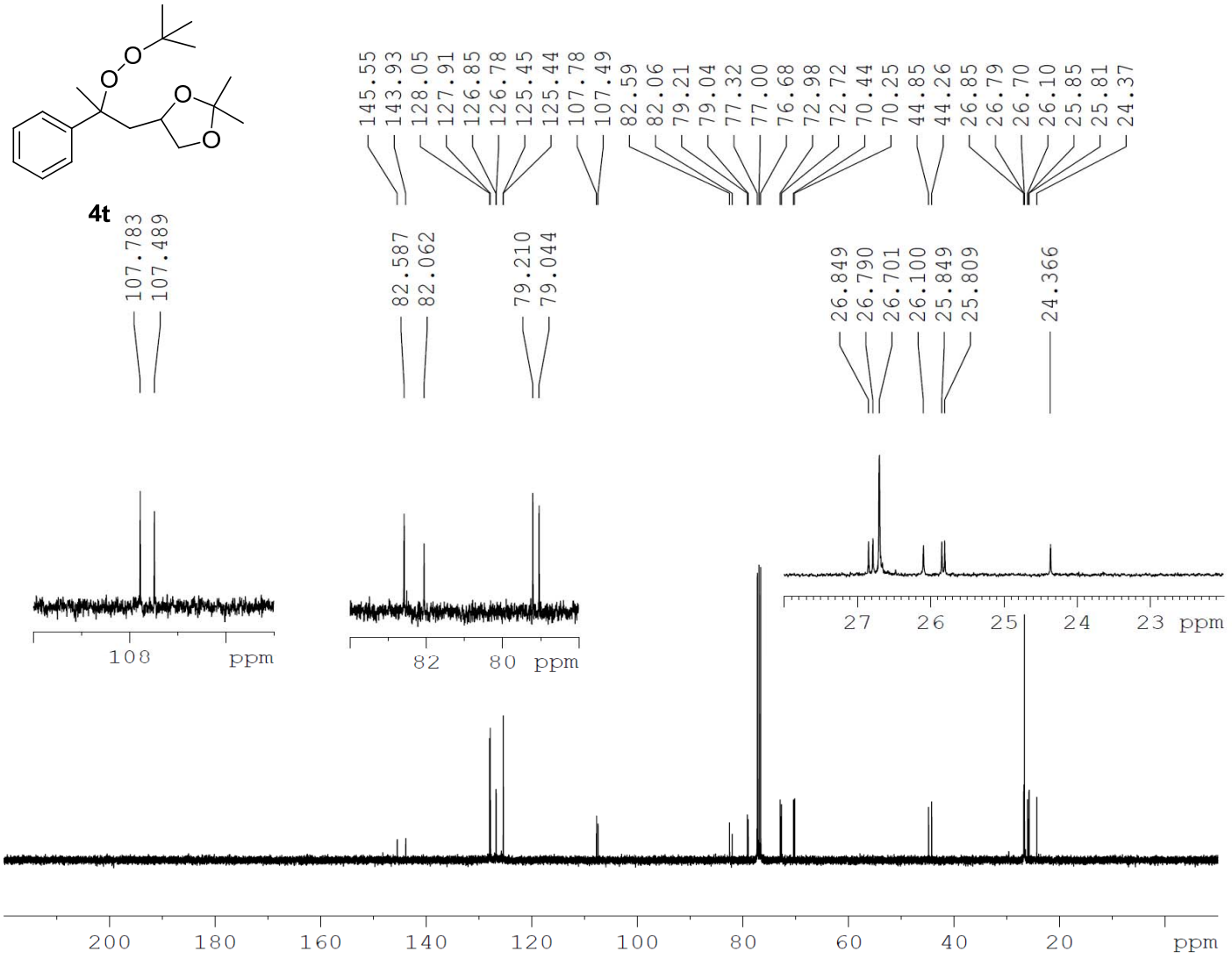
F2 - Acquisition Parameters
Date_ 20131224
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 40
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K

F2 - Processing parameters
SI 32768
SF 399.7627613 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

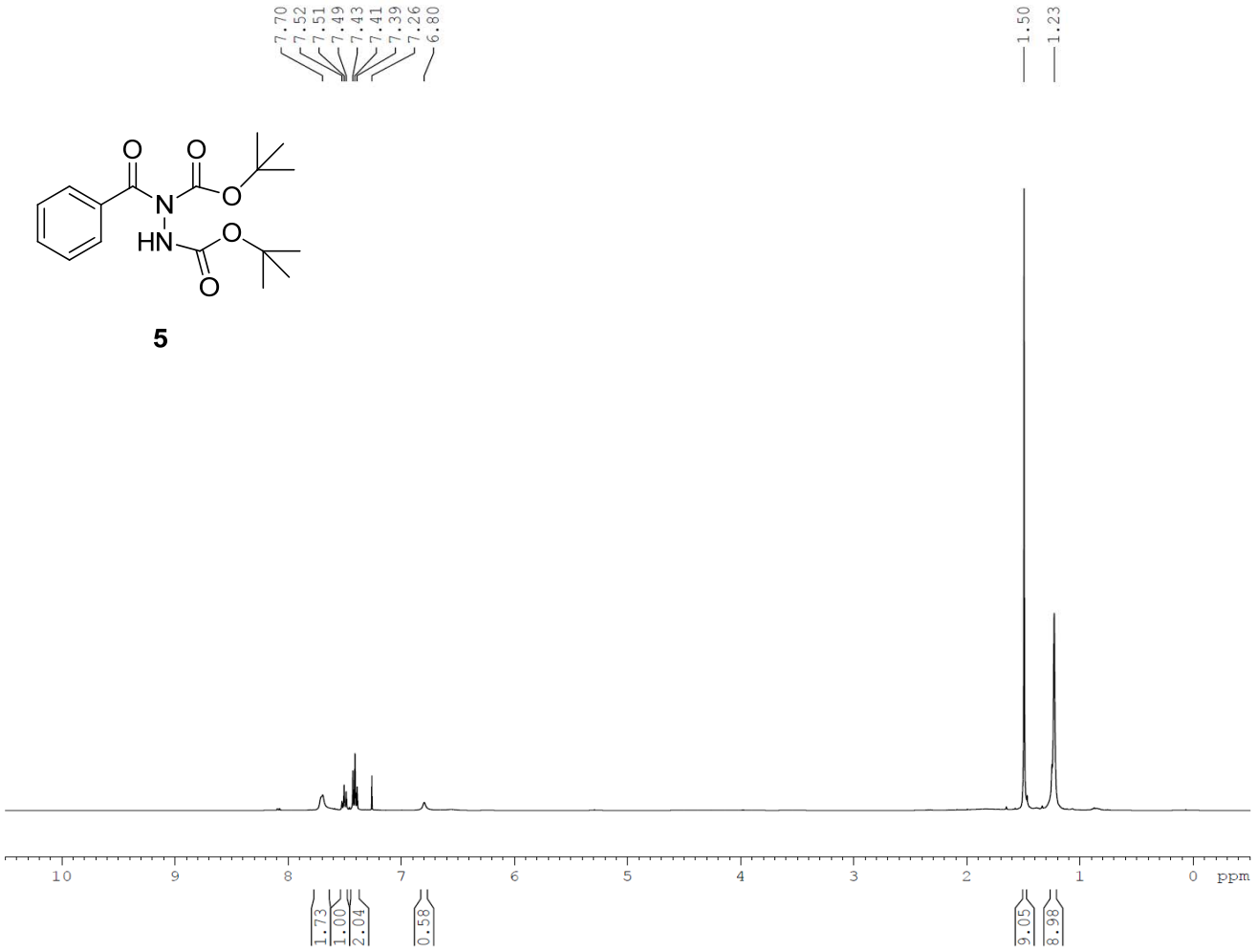

196.15
143.49
138.90
136.72
129.93
128.40
127.58
127.28
127.19
86.28
79.90
77.32
77.00
76.68
44.80
26.45
O
O
O
Cl
4s
Current Data Parameters
NAME 1009-1-3-C-3-0.fid
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131222
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 65536
SOLVENT cdcl3
NS 1240
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.3107700 sec
RG 4
DW 19.600 usec
DE 115.71 usec
TE 298.0 K
F2 - Processing parameters
SI 65536
SF 100.5218574 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm




```
Current Data Parameters
NAME        1093-2.fid
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20140714
Time              0.00
INSTRUM          varian
PROBHD
PULPROG           s2pul
TD                32768
SOLVENT           cdcl3
NS                   32
DS                    0
SWH            6402.049 Hz
FIDRES         0.195375 Hz
AQ             2.5559540 sec
RG                    4
DW               78.100 usec
DE               115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                32768
SF          399.7627603 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

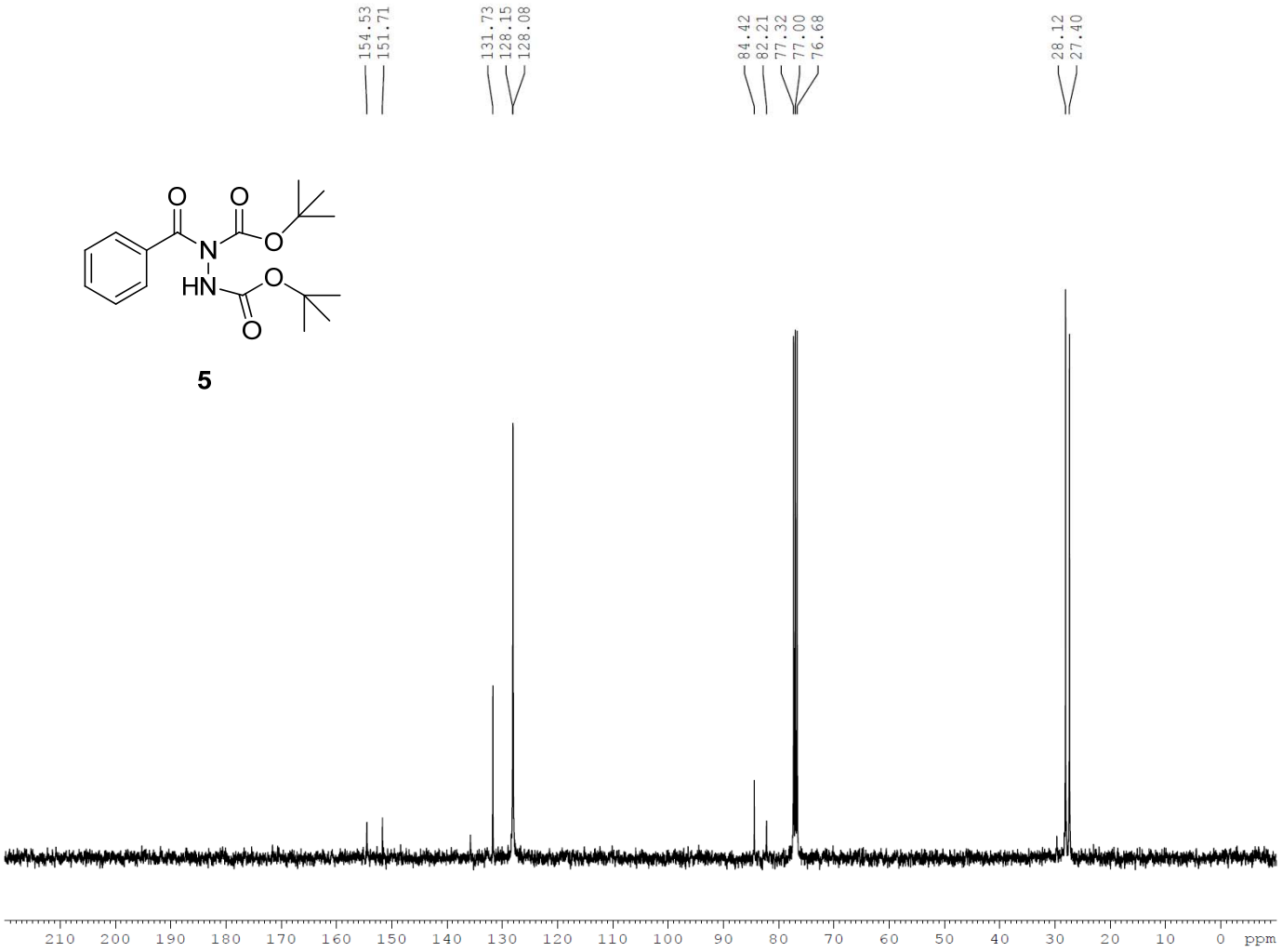



```
Current Data Parameters
NAME      1093-2-C-5.fid
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20140714
Time               0.00
INSTRUM          varian
PROBHD
PULPROG           s2pul
TD                65536
SOLVENT           cdcl3
NS                 1320
DS                    0
SWH           25510.203 Hz
FIDRES        0.389255 Hz
AQ            1.3107700 sec
RG                    4
DW               19.600 usec
DE               115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                65536
SF          100.5218558 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



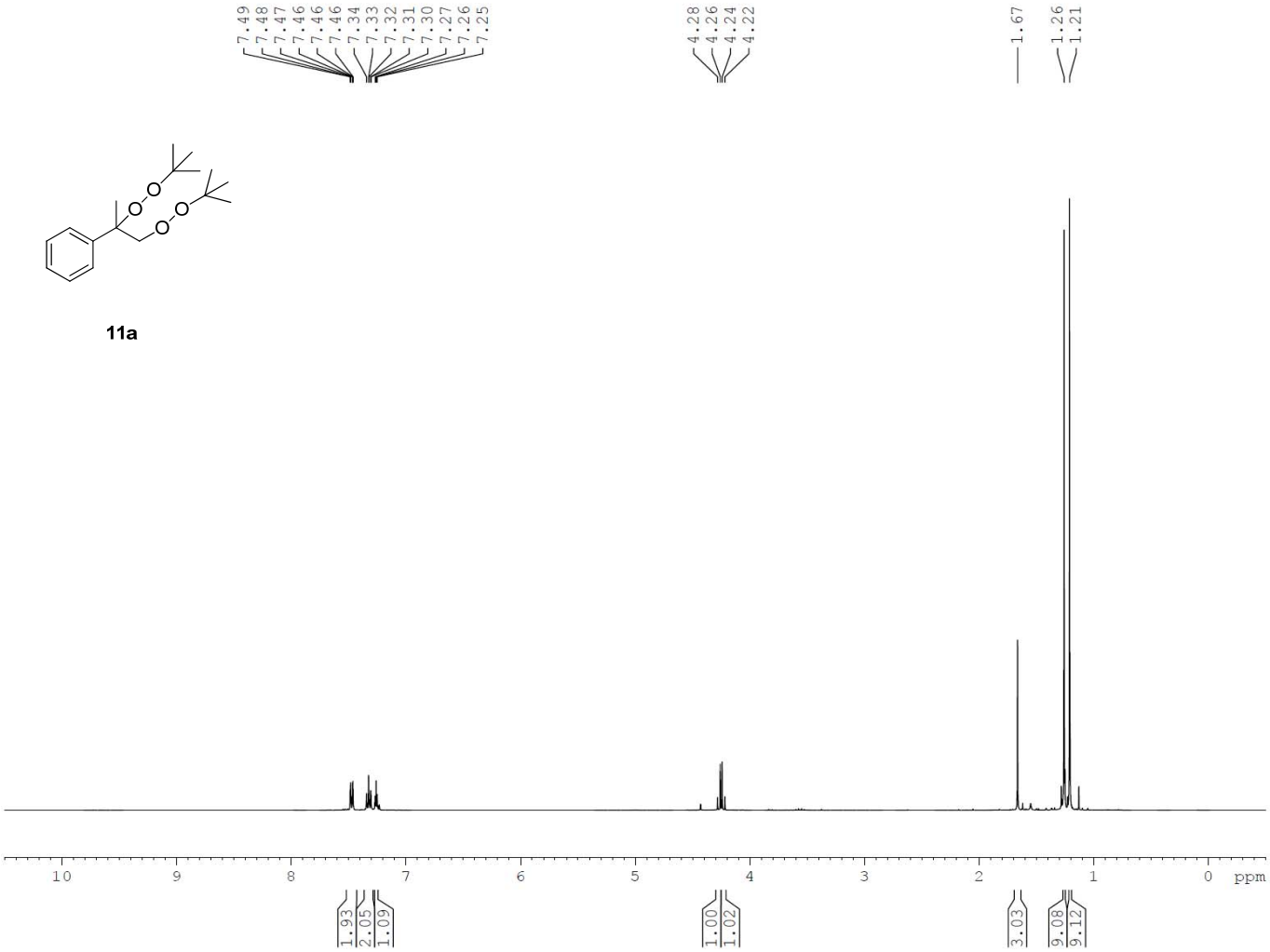
5

```
Current Data Parameters
NAME      1116-1-1-H-G.fid
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141001
Time                  0.00
INSTRUM            varian
PROBHD
PULPROG             s2pul
TD                  32768
SOLVENT             cdcl3
NS                     32
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ               2.5559540 sec
RG                      4
DW                 78.000 usec
DE                 115.71 usec
TE                  298.0 K

F2 - Processing parameters
SI                  32768
SF            399.7627610 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



5

```
Current Data Parameters
NAME      1116-1-1-H-G-C-4.fid
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20141001
Time                   0.00
INSTRUM             varian
PROBHD
PULPROG              s2pul
TD                   65536
SOLVENT              cdcl3
NS                     400
DS                       0
SWH              25510.203 Hz
FIDRES           0.389255 Hz
AQ               1.3107700 sec
RG                       4
DW                  19.600 usec
DE                  115.71 usec
TE                   298.0 K

F2 - Processing parameters
SI                   65536
SF           100.5218568 MHz
WDW                    EM
SSB                      0
LB                  3.00 Hz
GB                       0
PC                    1.00
```



Current Data Parameters

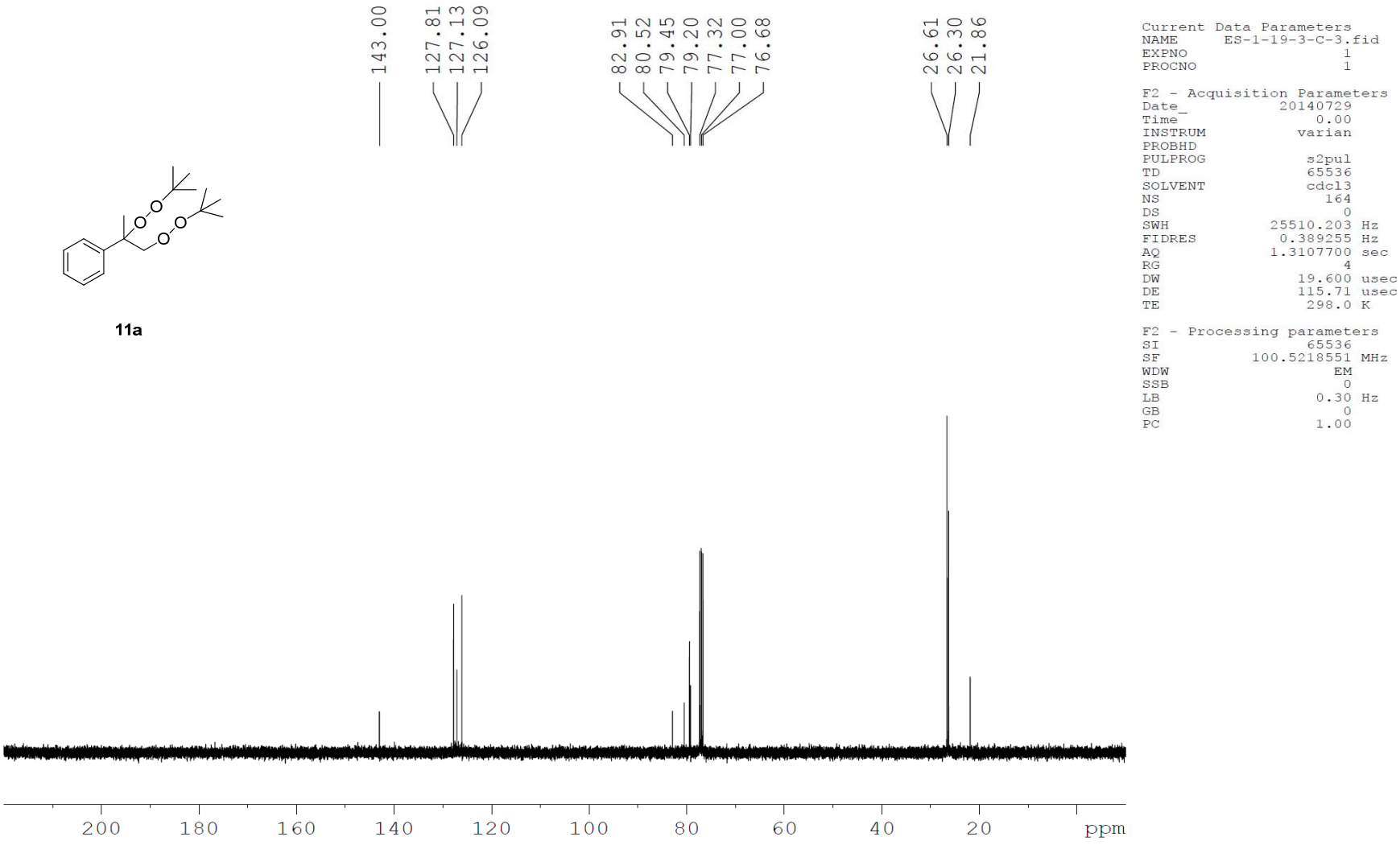
NAME	ES-1-19-3.fid
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20140729
Time	0.00
INSTRUM	varian
PROBHD	
PULPROG	s2pul
TD	32768
SOLVENT	cdcl3
NS	24
DS	0
SWH	6402.049 Hz
FIDRES	0.195375 Hz
AQ	2.5559540 sec
RG	4
DW	78.100 usec
DE	115.71 usec
TE	298.0 K

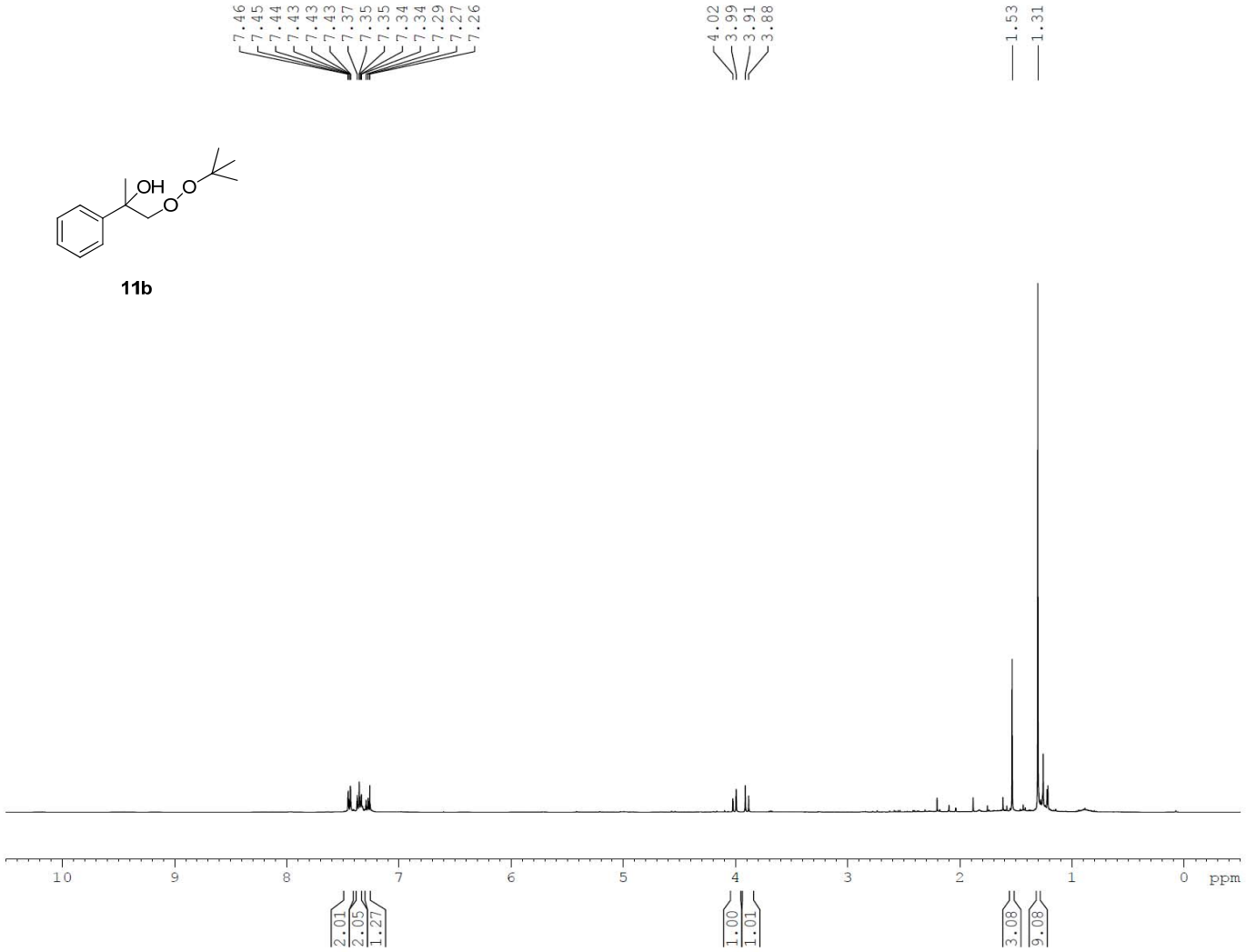
F2 - Processing parameters

SI	32768
SF	399.7627607 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

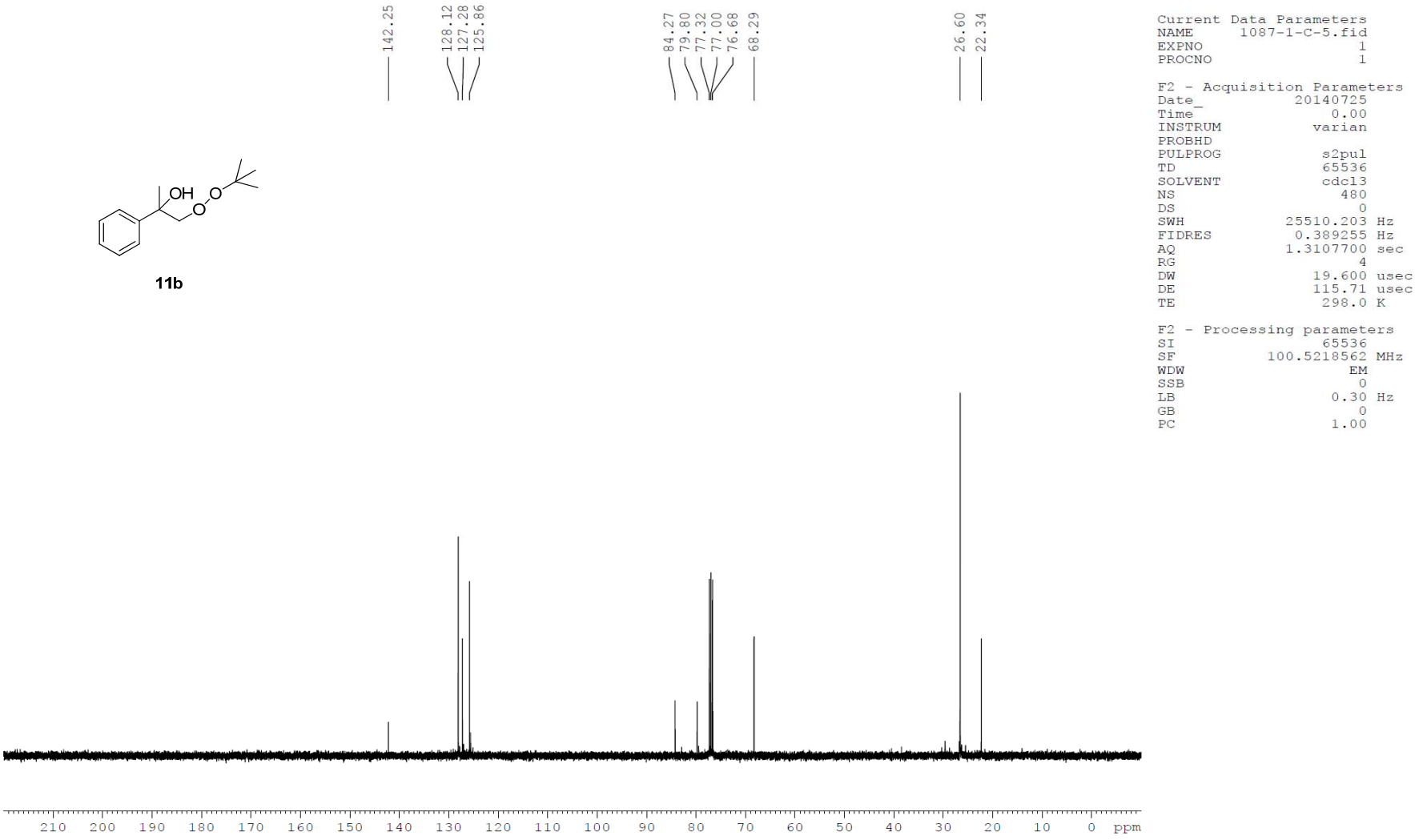


11a

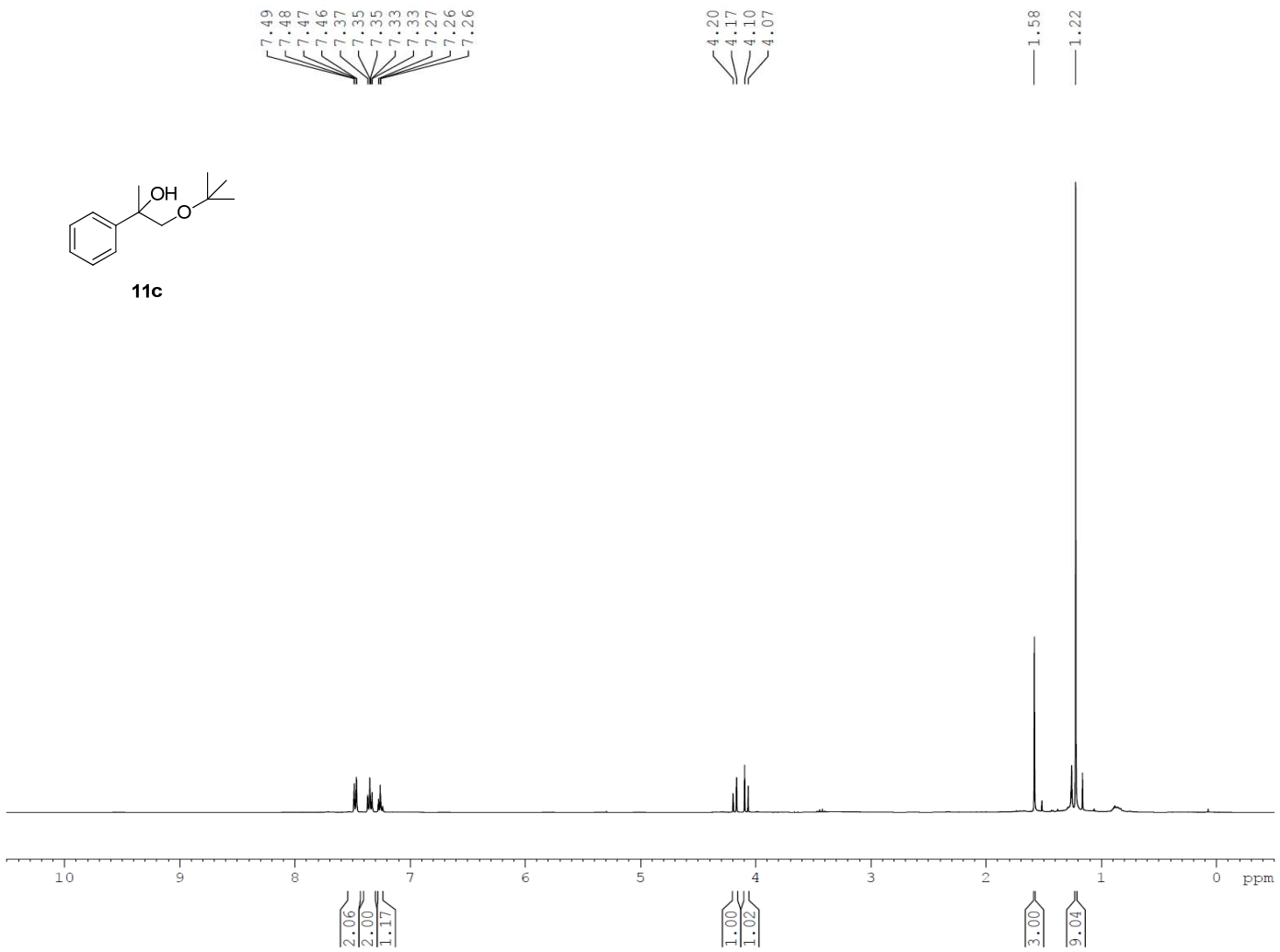
```
Current Data Parameters
NAME      ES-1-19-3-C-3.fid
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140729
Time                  0.00
INSTRUM            varian
PROBHD
PULPROG             s2pul
TD                  65536
SOLVENT             cdcl3
NS                    164
DS                      0
SWH             25510.203 Hz
FIDRES          0.389255 Hz
AQ              1.3107700 sec
RG                      4
DW                 19.600 usec
DE                 115.71 usec
TE                 298.0 K

F2 - Processing parameters
SI                  65536
SF            100.5218551 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                   1.00
```



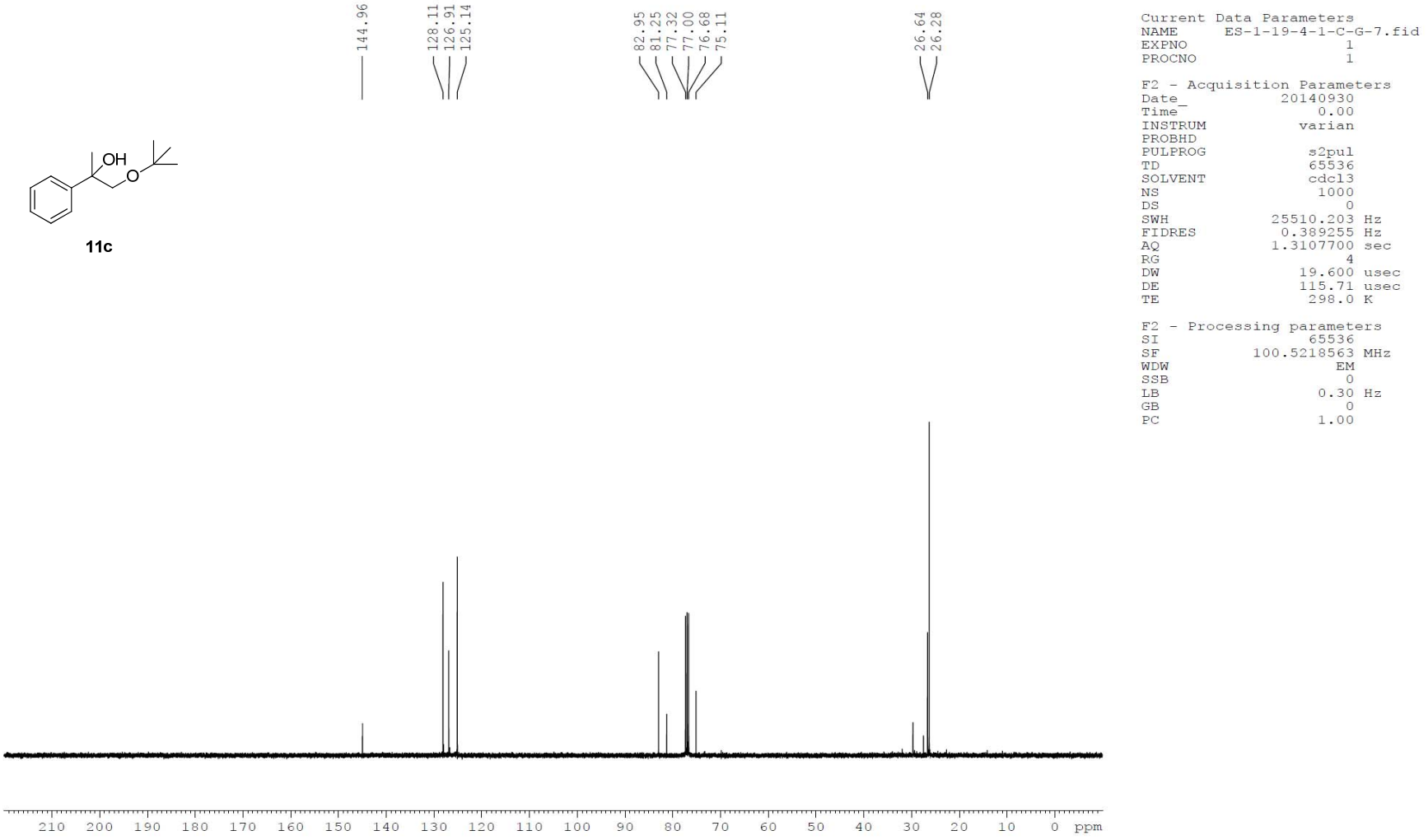

```
Current Data Parameters
NAME        1087-1-1.fid
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140725
Time                0.00
INSTRUM          varian
PROBHD
PULPROG            s2pul
TD                 32768
SOLVENT            cdcl3
NS                    32
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ            2.5559540 sec
RG                     4
DW                78.000 usec
DE                115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                 32768
SF          399.7627611 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



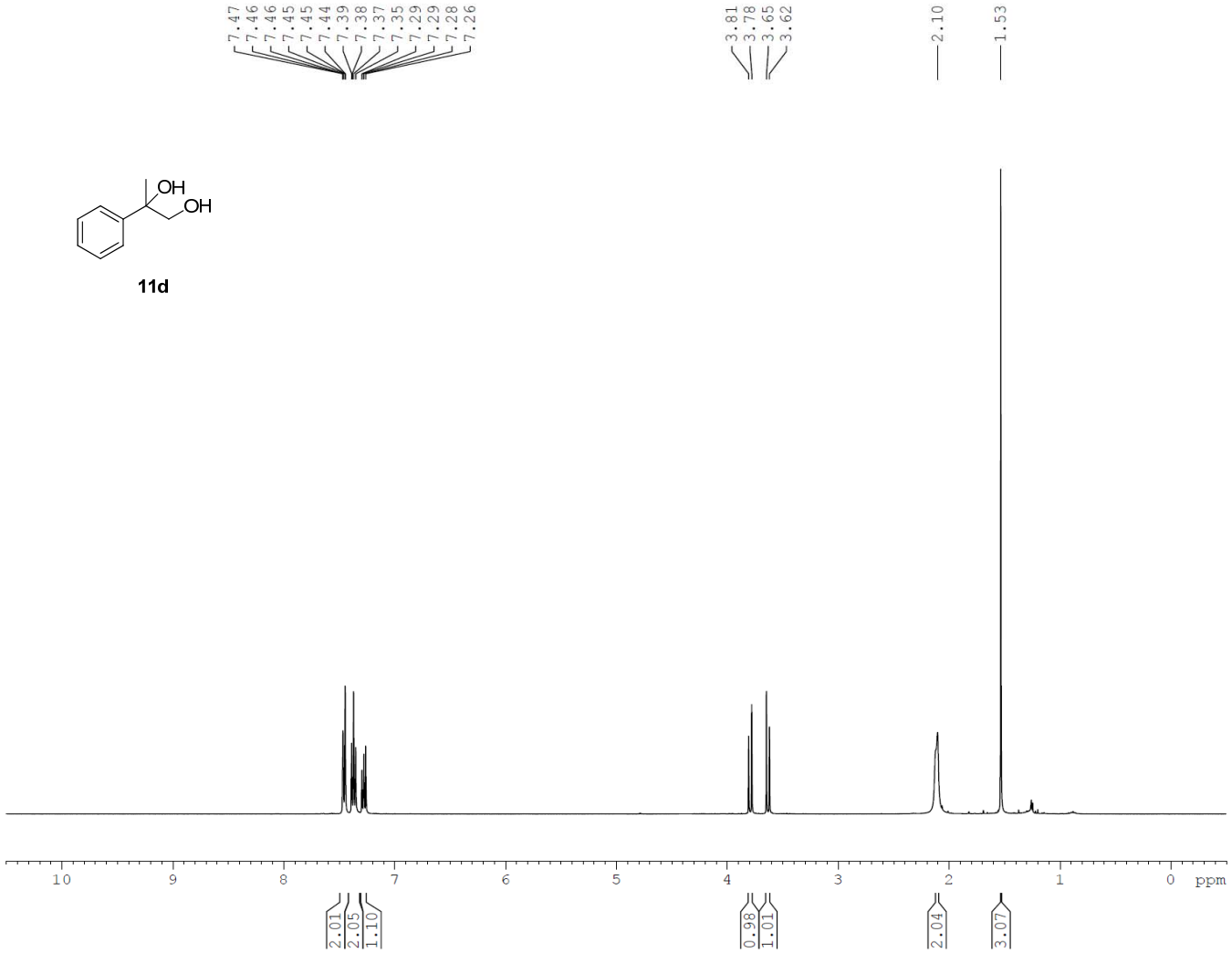

```
Current Data Parameters
NAME      1087-1-C-5.fid
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140725
Time                0.00
INSTRUM          varian
PROBHD
PULPROG            s2pul
TD                 65536
SOLVENT            cdcl3
NS                   480
DS                     0
SWH            25510.203 Hz
FIDRES         0.389255 Hz
AQ             1.3107700 sec
RG                     4
DW                19.600 usec
DE                115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                 65536
SF          100.5218562 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```




```
Current Data Parameters
NAME      ES-1-19-4-1-H-G.fid
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20140930
Time                   0.00
INSTRUM              varian
PROBHD
PULPROG              s2pul
TD                   32768
SOLVENT              cdcl3
NS                      48
DS                       0
SWH                6410.256 Hz
FIDRES            0.195625 Hz
AQ                2.5559540 sec
RG                       4
DW                  78.000 usec
DE                  115.71 usec
TE                   298.0 K

F2 - Processing parameters
SI                   32768
SF           399.7627611 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



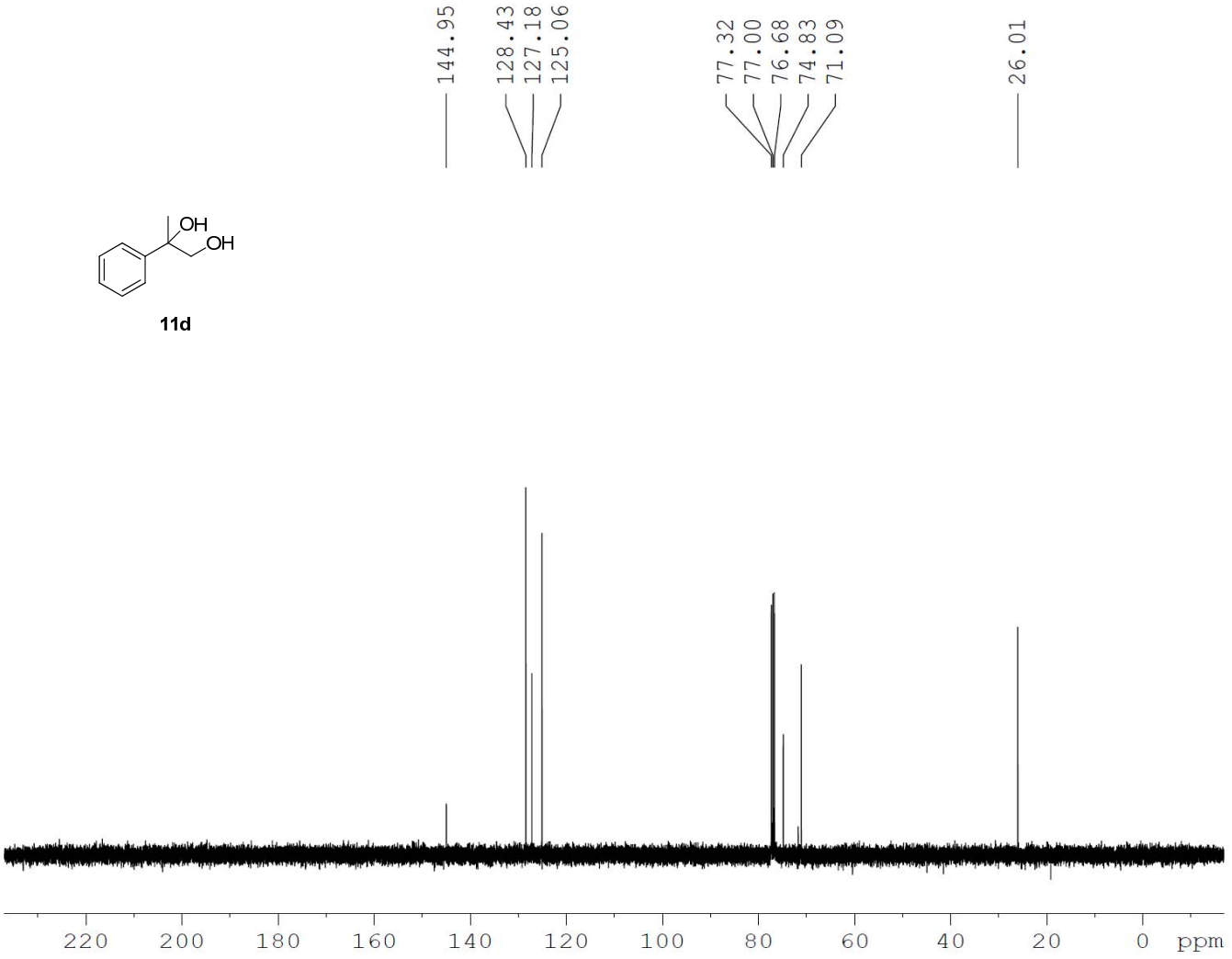

```
Current Data Parameters
NAME      ES-1-19-4-1-C-G-7.fid
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20140930
Time                   0.00
INSTRUM              varian
PROBHD
PULPROG               s2pul
TD                    65536
SOLVENT               cdcl3
NS                     1000
DS                        0
SWH               25510.203 Hz
FIDRES             0.389255 Hz
AQ                1.3107700 sec
RG                        4
DW                   19.600 usec
DE                   115.71 usec
TE                    298.0 K

F2 - Processing parameters
SI                    65536
SF            100.5218563 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



Current Data Parameters
NAME ES-1-19-5.fid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140729
Time 0.00
INSTRUM varian
PROBHD
PULPROG s2pul
TD 32768
SOLVENT cdcl3
NS 24
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 115.71 usec
TE 298.0 K

F2 - Processing parameters
SI 32768
SF 399.7627612 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME      ES-1-19-5-C-3.fid
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20140729
Time                0.00
INSTRUM          varian
PROBHD
PULPROG           s2pul
TD                65536
SOLVENT           cdcl3
NS                  132
DS                    0
SWH           25510.203 Hz
FIDRES         0.389255 Hz
AQ            1.3107700 sec
RG                    4
DW               19.600 usec
DE               115.71 usec
TE                298.0 K

F2 - Processing parameters
SI                65536
SF          100.5218566 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```