Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Chiral isoxazolidine-mediated stereoselctive umpolung α-phenylation of methyl ketones

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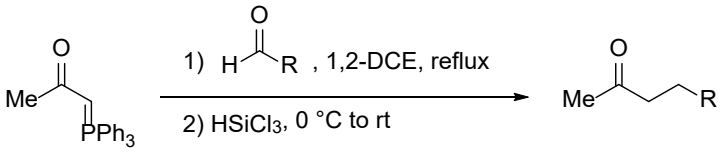
I. General Information

All reactions were carried out under an argon atmosphere with dry solvents under anhydrous conditions, unless otherwise noted. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. Flash column chromatography were performed using Silicycle silica gel (SiliaFlash® F60, 40-63 μm). Preparative thin-layer chromatography (preparative TLC) separations were carried out on 0.25 or 0.50 mm E. Merck silica gel plates (60 F_{254}). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian Mercury 300 MHz, a Varian VNS AS 500 MHz or a Varian VNS AS 600 MHz operating at 300 MHz/75 MHz, 500 MHz/125 MHz, or 600 MHz/150 MHz for ^{1}H and ^{13}C acquisitions, respectively. Chemical shifts are reported in ppm with the solvent resonance or TMS as the internal standard. Multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), qn (quintet), m (multiplet) and br (broad). Infrared (IR) spectra were recorded on a Perkin-Elmer SpectrumOne A spectrometer using NaCl plates. High-resolution mass spectra (HRMS) were obtained by ESI method on Thermo Fisher Scientific Exactive Instrument. Melting points (uncorrected) were determined on BÜCHI M-565 apparatus. HPLC analyses were carried out on a SHIMADZU LC-20AT pump and a SPD-20A UV/Vis detector or JASCO PU-4180 RHPLC pump and UV-4075 UV/Vis detector. Optical rotations were measured on a JASCO DIP-370 degital polarimeter. 4-Phenyl-2-butanone (**1a**), 4-(4-methoxyphenyl)-2-butanone (**1c**), 4-(1,3-benzodioxol-5-yl)-2-butanone (**1i**), 1-(4-methylphenyl) -2-propanone (**1o**), 5-hexen-2-one (**1p**), 5-oxo-hexanenitrile (**1q**), nabumethone, and pentoxifylline were purchased from Aldrich or TCI (Tokyo Chemincal Industry Co., Ltd.). Triphenylaluminium (1.0 M in *n*dibutyl ether) was purchased from Aldrich. (+)-Benzopyranoisoxazolidine **3** was prepared by the reported procedure.[1] The racemic materials **2a-2s** were prepared using isoxazolidine[2] or *rac*-benzopyranoisoxazolidine (*rac*-**3**) in the same procedure as their corresponding chiral compounds.

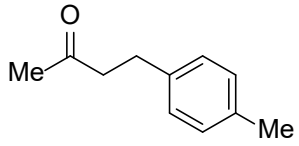
II. Experimental Section

General procedure for the preparation of 4-aryl-2-butanone (1)[3]



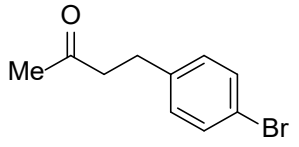
To a solution of (acetylmethylene)triphenylphosphorane (3.18 g, 10 mmol) in 1,2-dichloroethane (20 mL) was added aldehydes (10 mmol) at room temperature under an argon atmosphere. After being stirred at reflux for 3-7 h, the reaction mixture was cooled to 0 °C. Then trichlorosilane (2.0 mL, 20 mmol) was added at 0 °C in one portion. After being stirred at room temperature for 4 h, the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (*n*hexane/AcOEt = 10/1 to 1/1) to give 4-aryl-2-butanone **1**.

4-(4-Methylphenyl)-2-butanone (1b)[4]



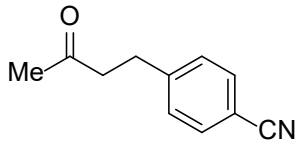
75% yield. a colorless oil; IR (neat) ν_{max} 1717 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.06 (m, 4H), 2.83 (br t, *J* = 7.5 Hz, 2H), 2.70 (br t, *J* = 7.5 Hz, 2H), 2.29 (s, 3H), 2.10 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.9, 137.8, 135.4, 129.0, 128.0, 45.2, 29.9, 29.2, 20.9; HRMS (ESI) calcd for $C_{11}H_{14}ONa$ [M+Na^+] 185.0937, found 185.0934.

4-(4-Bromophenyl)-2-butanone (1d)[5]



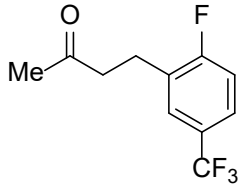
89% yield. white solid; mp 39-40 °C; IR ($CHCl_3$) ν_{max} 1716 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.33 (br d, *J* = 8.0 Hz, 2H), 7.00 (br d, *J* = 8.0 Hz, 2H), 2.78 (br t, *J* = 7.5 Hz, 2H), 2.68 (br t, *J* = 7.5 Hz, 2H), 2.07 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.3, 139.9, 131.4, 130.0, 119.7, 44.7, 30.0, 28.9; HRMS (ESI) calcd for $C_{10}H_{11}O^{78}BrNa$ [M+Na^+] 248.9885, found 248.9885.

4-(3-Oxobutyl)benzonitrile (1e)[6]



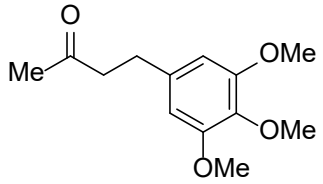
95% yield. white solid; mp 55-56 °C; IR ($CHCl_3$) ν_{max} 2230, 1717 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.57 (br d, *J* = 8.5 Hz, 2H), 7.30 (br d, *J* = 8.5 Hz, 2H), 2.96 (br t, *J* = 7.5 Hz, 2H), 2.79 (br t, *J* = 7.5 Hz, 2H), 2.16 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 206.6, 146.6, 132.0, 129.0, 118.7, 109.6, 43.9, 29.8, 29.3; HRMS (ESI) calcd for $C_{11}H_{11}ONNa$ [M+Na^+] 196.0733, found 196.0734.

4-[2-Fluoro-(5-trifluoromethyl)phenyl]-2-butanone (1g)



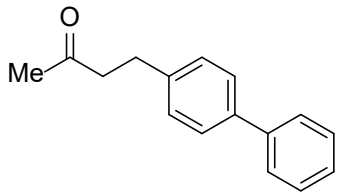
46% yield. a colorless oil; IR (neat) ν_{max} 1721 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.52-7.44 (m, 2H), 7.11 (br t, *J* = 9.0 Hz, 1H), 2.96 (br t, *J* = 7.5 Hz, 2H), 2.80 (br t, *J* = 7.5 Hz, 2H), 2.17 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 206.8, 162.9 (d, *J* = 249.0 Hz), 128.9 (d, *J* = 21.0 Hz), 128.2 (qd, *J* = 7.0, 3.0 Hz), 126.7 (qd, *J* = 32.0, 3.5 Hz), 125.5 (qd, *J* = 7.0, 3.0 Hz), 123.8 (q, *J* = 270.0 Hz), 115.8 (d, *J* = 23.0 Hz), 43.1, 29.9, 23.2; HRMS (ESI) calcd for $C_{11}H_{10}OF_4Na$ [M+Na^+] 257.0560, found 257.0560.

4-(3,4,5-Trimethoxyphenyl)-2-butanone (1h)[7]



47% yield. a colorless oil; IR (neat) ν_{max} 1717 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 6.41 (s, 2H), 3.85 (s, 6H), 3.82 (s, 3H), 2.84 (br td, *J* = 7.0, 3.0 Hz, 2H), 2.75 (br td, *J* = 7.0, 3.0 Hz, 2H), 2.16 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.7, 153.0, 136.7, 136.1, 105.1, 60.6, 55.9 (2C), 45.1, 29.9; HRMS (ESI) calcd for $C_{13}H_{18}O_4Na$ [M+Na^+] 261.1097, found 261.1096.

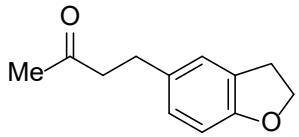
4-[1,1'-Biphenyl]-4-yl-2-butanone (1j)



80% yield. white solid; mp 77-78 °C; IR ($CHCl_3$) ν_{max} 1716 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.57 (br d, *J* = 7.5 Hz, 2H), 7.51 (br d, *J* = 7.5 Hz, 2H), 7.43 (br t, *J* = 7.5 Hz, 2H), 7.32 (br t, *J* = 7.0 Hz, 1H), 7.26 (br d, *J* = 7.5 Hz, 2H), 2.94 (br t, *J* = 7.5 Hz, 2H), 2.80 (br t, *J* = 7.5 Hz, 2H), 2.16 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.8, 140.8, 140.0, 139.0, 128.7, 127.1, 127.0, 126.9, 45.0, 30.0, 29.2; HRMS (ESI) calcd for $C_{16}H_{16}ONa$ [M+Na^+] 247.1093, found 247.1093.

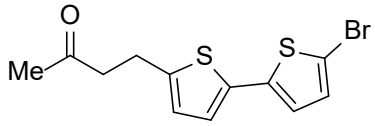
One of aromatic carbons overlapped with other aromatic carbons in ^{13}C NMR spectrum.

4-(2,3-Dihydro-5-benzofuranyl)-2-butanone (1l)



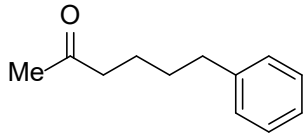
55% yield. white solid; mp 54-55 °C; IR ($CHCl_3$) ν_{max} 1715 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.01 (br s, 1H), 6.90 (br d, *J* = 8.0 Hz, 1H), 6.69 (br d, *J* = 8.0 Hz, 1H), 4.53 (t, *J* = 8.5 Hz, 2H), 3.16 (t, *J* = 8.5 Hz, 2H), 2.82 (br t, *J* = 7.5 Hz, 2H), 2.71 (br t, *J* = 7.5 Hz, 2H), 2.13 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 208.1, 158.3, 132.8, 127.5, 127.0, 124.7, 108.9, 71.0, 45.6, 30.0, 29.6, 29.1; HRMS (ESI) calcd for $C_{12}H_{14}O_2Na$ [M+Na^+] 213.0886, found 213.0886.

4-[5'-Bromo-(2,2'-bithiophene)-5-yl]-2-butanone (1m)



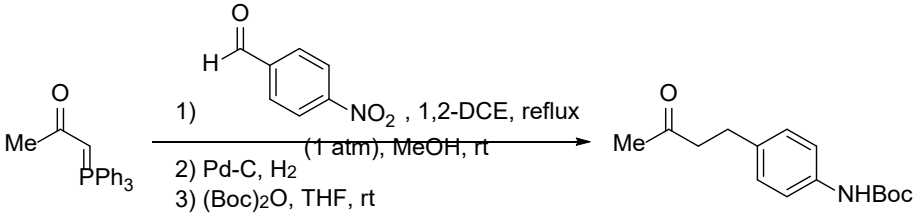
91% yield. white solid; mp 80-81°C (decomposed); IR ($CHCl_3$) ν_{max} 1719 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 6.93 (dd, *J* = 3.5, 1.0 Hz, 1H), 6.89 (d, *J* = 3.5 Hz, 1H), 6.82 (d, *J* = 3.5 Hz, 1H), 6.68 (br dd, *J* = 3.5, 1.0 Hz, 1H), 3.06 (br t, *J* = 7.5 Hz, 2H), 2.81 (br t, *J* = 7.5 Hz, 2H), 2.17 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 206.9, 143.5, 139.0, 134.3, 130.5, 125.4, 123.7, 123.2, 110.3, 44.8, 30.0, 23.9; HRMS (ESI) calcd for $C_{12}H_{11}O^{79}BrS_2Na$ [M+Na^+] 336.9327, found 336.9327.

6-Phenyl-2-hexanone (1n)[8]



94% yield. a colorless oil; IR (neat) ν_{max} 1715 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.26-7.21 (m, 2H), 7.15-7.12 (m, 3H), 2.58 (br t, *J* = 7.5 Hz, 2H), 2.38 (br t, *J* = 7.5 Hz, 2H), 2.05 (s, 3H), 1.59-1.56 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 208.7, 141.9, 128.13, 128.06, 125.5, 43.2, 35.5, 30.7, 29.6, 23.2; HRMS (APCI) calcd for $C_{12}H_{17}O$ [M+H^+] 177.1274, found 177.1274.

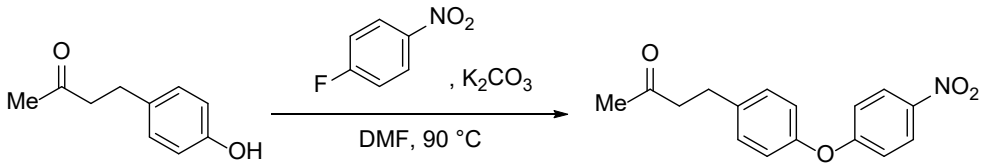
***N*-[4-(3-Oxobutyl)phenyl]carbamic acid 1,1-dimethylethyl ester (1f)**



To a solution of (acetylmethylene)triphenylphosphorane (3.18 g, 10 mmol) in 1,2-dichloroethane (20 mL) was added 4-nitrobenzaldehyde (1.51 g, 10 mmol) at room temperature under an argon atmosphere. After being stirred at reflux for 7 h, the reaction mixture was concentrated under

reduced pressure. The crude product was dissolved in Et_2O (100 mL), then mixture was cooled to 0 °C. A pale yellow precipitate was removed by filtration. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography (*n*hexane/AcOEt = 5/1 to 1/1) to give α,β-unsaturated methyl ketone. To a suspension of α,β-unsaturated methyl ketone (1.74 g, 9.1 mmol) in MeOH (30 mL) and AcOEt (10 mL) was added Pd-C (10% wt, 100 mg) at room temperature under a hydrogen atmosphere (1 atm). After being stirred at the same temperature for overnight, the resulting mixture was filtrated through a silica gel pad and washed with AcOEt. The filtrate was concentrated under reduced pressure. The crude product was used to next reaction without further purification. To a solution of 4-(4-aminophenyl)-2-butanone (1.28 g, 7.3 mmol) in THF (4.5 mL) was added Boc_2O (2.0 g, 9.2 mmol) at room temperature. After being at the same temperature for 7 h, the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (*n*hexane/AcOEt = 10/1 to 5/1) to give 4-aryl-2-butanone **1f** (1.38 g, 48%, 3 steps) as white solid. mp 95-96 °C; IR ($CHCl_3$) ν_{max} 3385, 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.28-7.25 (m, 2H), 7.09 (d, *J* = 8.5 Hz, 2H), 6.49 (br s, 1H), 2.84 (t, *J* = 7.5 Hz, 2H), 2.72 (t, *J* = 7.5 Hz, 2H), 2.12 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 208.1, 152.8, 136.4, 135.4, 128.6, 118.7, 80.1, 45.1, 30.0, 28.9, 28.2; HRMS (ESI) calcd for $C_{15}H_{21}O_3NNa$ [M+Na^+] 286.1414, found 286.1408.

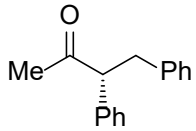
4-[4-(4-Nitrophenoxy)phenyl]-2-butanone (1k)



To a solution of 4-(4-hydroxyphenyl)-2-butanone (1.64 g, 10 mmol) in DMF (20 mL) was added K_2CO_3 (2.76 g, 20 mmol) and 1-fluoro-4-nitrobenzene (1.06 mL, 10 mmol) at room temperature under an argon atmosphere. After being stirred at 90 °C for 7 h, the reaction mixture was diluted with Et_2O. The resulting mixture was washed with H_2O (4 times) and saturated NaCl. The organic phase was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash column chromatography (*n*hexane/AcOEt = 5/1 to 1/1) to give 4-aryl-2-butanone **1k** (1.85 g, 65%) as yellow solid. mp 58-59 °C; IR ($CHCl_3$) ν_{max} 1715, 1515, 1344 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 8.18 (br d, *J* = 8.5 Hz, 2H), 7.25 (br d, *J* = 8.5 Hz, 2H), 7.02-6.98 (m, 4H), 2.93 (t, *J* = 7.0 Hz, 2H), 2.80 (t, *J* = 7.0 Hz, 2H), 2.18 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.4, 163.4, 152.7, 142.3, 138.2, 130.0, 125.7, 120.4, 116.7, 44.8, 29.9, 28.8; HRMS (ESI) calcd for $C_{16}H_{15}O_4NNa$ [M+Na^+] 308.0893, found 308.0892.

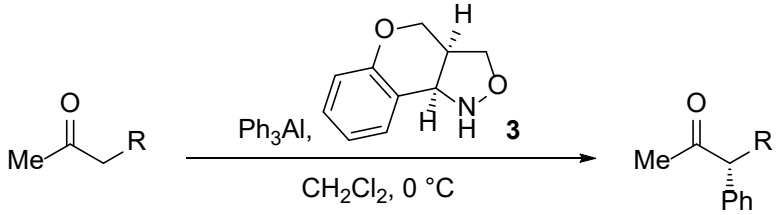
Asymmetric nucleophilic α-phenylation of ketone 1a (Table 1, entry 2)

(3*R*)-3,4-Diphenyl-2-butanone (2a)[9]



To a solution of (+)-benzopyranoisoxazolidine **3** (26.6 mg, 0.15 mmol) in CH_2Cl_2 (0.75 mL) were added 4-phenyl-2-butanone (**1a**) (11.1 mg, 0.075 mmol) and triphenylaluminium (1.0 M in *n*dibutyl ether, 0.23 mL, 0.23 mmol) dropwise at 0 °C under an argon atmosphere. After being stirred at the same temperature for 2 h, the reaction mixture was quenched with an aqueous Rochelle's salt (1.3 M). The resulting suspension was extracted with $CHCl_3$. The organic phase was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by preparative TLC (*n*hexane/AcOEt = 5/1) to give α-phenylated ketone **2a** (11.1 mg, 66%, 94% ee) as a colorless oil. $[\alpha]_D^{26}$ −221.0 (*c* 0.56, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 16.2 min (minor), 17.6 min (major)]. IR (neat) ν_{max} 1714 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.34-7.26 (m, 2H), 7.23-7.15 (m, 6H), 7.04 (br d, *J* = 7.0 Hz, 2H), 3.92 (br t, *J* = 7.5 Hz, 1H), 3.43 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.90 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.3, 139.5, 138.3, 128.8, 128.7, 128.2, 128.1, 127.2, 125.9, 61.5, 38.4, 29.5; HRMS (ESI) calcd for $C_{16}H_{16}ONa$ [$M+Na^+$] 247.1093, found 247.1095.

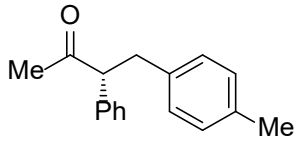
General procedure for asymmetric nucleophilic α-phenylation of ketones 1b-q (Table 2)



1b-q **2b-q**

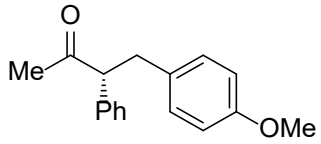
To a solution of (+)-benzopyranoisoxazolidine **3** (26.6 mg, 0.15 mmol) in CH_2Cl_2 (0.75 mL) were added ketone **1b-q** (0.075 mmol) and triphenylaluminium (1.0 M in *n*dibutyl ether, 0.23 mL, 0.23 mmol) dropwise at 0 °C under an argon atmosphere. After being stirred at the same temperature for 2-3 h, the reaction mixture was quenched with an aqueous Rochelle's salt (1.3 M). The resulting suspension was extracted with $CHCl_3$. The organic phase was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by preparative TLC (*n*hexane/AcOEt = 5/1) to give α-phenylated ketone **2b-q**.

(3*R*)-4-(4-Methylphenyl)-3-phenyl-2-butanone (2b)



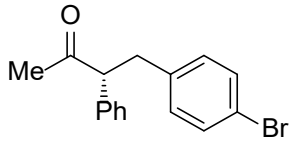
61% yield, 90% ee. a colorless oil. $[\alpha]_D^{25}$ −307 (*c* 0.30, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 26.1 min (minor), 29.9 min (major)]. IR (neat) ν_{max} 1715 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.33-7.25 (m, 3H), 7.20-7.17 (m, 2H), 7.00 (br d, *J* = 8.0 Hz, 2H), 6.93 (br d, *J* = 8.0 Hz, 2H), 3.90 (br t, *J* = 7.5 Hz, 1H), 3.38 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.86 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.27 (s, 3H), 2.01 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.5, 138.5, 136.5, 135.4, 128.83, 128.77, 128.72, 128.2, 127.2, 61.6, 38.0, 29.6, 21.1; HRMS (ESI) calcd for $C_{17}H_{18}ONa$ [M+Na^+] 261.1250, found 261.1250.

(3*R*)-4-(4-Methoxylphenyl)-3-phenyl-2-butanone (2c)



61% yield, 94% ee. white solid. mp 55-56 °C; $[\alpha]_D^{25}$ −262 (*c* 0.39, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IA (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 1.0 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 10.7 min (major), 12.2 min (minor)]. IR ($CHCl_3$) ν_{max} 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.34-7.25 (m, 3H), 7.19-7.16 (m, 2H), 6.96 (br d, *J* = 8.5 Hz, 2H), 6.74 (br d, *J* = 8.5 Hz, 2H), 3.88 (br t, *J* = 7.5 Hz, 1H), 3.75 (s, 3H), 3.36 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.84 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.9, 157.9, 138.5, 131.7, 129.9, 128.9, 128.3, 127.3, 113.6, 61.8, 55.2, 37.5, 29.6; HRMS (ESI) calcd for $C_{17}H_{18}O_2Na$ [M+Na^+] 277.1199, found 277.1199.

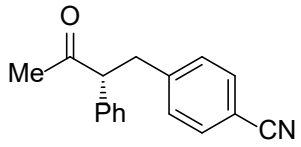
(3*R*)-4-(4-Bromophenyl)-3-phenyl-2-butanone (2d)



50% yield, 90% ee. white solid. mp 74-75 °C; $[\alpha]_D^{25}$ −218 (*c* 0.33, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C,

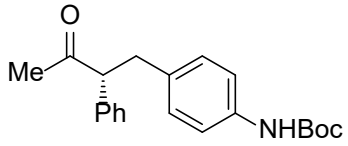
retention times: 18.2 min (minor), 19.2 min (major)]. IR ($CHCl_3$) ν_{max} 1713 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.34-7.28 (m, 5H), 7.16-7.13 (m, 2H), 6.89 (br d, *J* = 8.5 Hz, 2H), 3.85 (br t, *J* = 7.5 Hz, 1H), 3.36 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.84 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.0, 138.5, 137.9, 131.2, 130.7, 128.9, 128.2, 127.4, 119.9, 61.4, 37.8, 29.5; HRMS (ESI) calcd for $C_{16}H_{15}O^{79}BrNa$ [M+Na^+] 325.0199, found 325.0196.

(2*R*)-4-(3-Oxo-2-phenylbutyl)benzonitrile (2e)



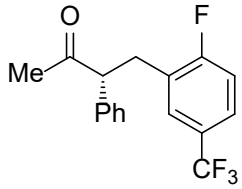
67% yield, 89% ee. white solid. mp 110-111 °C; $[\alpha]_D^{25}$ −317 (*c* 0.55, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IB (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 95/5, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 21.5 min (minor), 24.7 min (major)]. IR ($CHCl_3$) ν_{max} 2230, 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.45 (br d, *J* = 8.0 Hz, 2H), 7.30-7.24 (m, 3H), 7.11-7.08 (m, 4H), 3.85 (br t, *J* = 7.5 Hz, 1H), 3.44 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.94 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 206.4, 145.3, 137.5, 131.9, 129.7, 129.0, 128.2, 127.6, 118.8, 110.0, 61.0, 38.4, 29.3; HRMS (ESI) calcd for $C_{17}H_{15}ONNa$ [M+Na^+] 272.1046, found 272.1046.

(2*R*)-*N*-[4-(3-Oxo-2-phenylbutyl)phenyl]carbamic acid 1,1-dimethylethyl ester (2f)



58% yield, 91% ee. white solid. mp 102-103 °C; $[\alpha]_D^{25}$ −200 (*c* 0.74, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 80/20, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 15.3 min (minor), 16.8 min (major)]. IR ($CHCl_3$) ν_{max} 3440, 1714 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.33-7.26 (m, 3H), 7.19-7.15 (m, 4H), 6.95 (d, *J* = 8.5 Hz, 2H), 6.38 (br s, 1H), 3.87 (br t, *J* = 7.5 Hz, 1H), 3.36 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.84 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.01 (s, 3H), 1.50 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.8, 152.8, 138.3, 136.4, 134.2, 129.4, 128.8, 128.3, 127.3, 118.4, 80.3, 61.6, 37.6, 29.5, 28.3; HRMS (ESI) calcd for $C_{21}H_{25}O_3NNa$ [M+Na^+] 362.1727, found 362.1727.

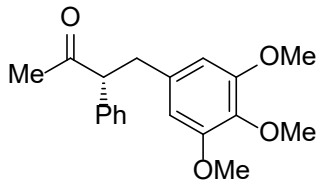
(3*R*)-4-[2-Fluoro-(5-trifluoromethyl)phenyl]-3-phenyl-2-butanone (2g)



61% yield, 95% ee. a colorless oil. $[\alpha]_D^{25}$ −222 (*c* 0.45, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IB (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 100/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 26.5 min (major), 29.9 min (minor)]. IR ($CHCl_3$) ν_{max} 1717 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.42-7.37 (m, 1H), 7.33-7.25 (m, 3H), 7.21-7.19 (m, 1H), 7.13-7.10 (m, 2H), 7.05 (t, *J* = 9.0 Hz, 1H), 3.94 (br t, *J* = 7.5 Hz, 1H), 3.44 (br dd, *J* = 13.5, 7.5 Hz, 1H), 2.97 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.05 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 206.8, 162.9 (d, *J* = 249.0 Hz), 137.6, 129.1 (m), 128.2, 127.7, 127.5 (d, *J* = 16.5 Hz), 126.3 (qd, *J* = 32.0, 3.5 Hz), 125.4 (qd, *J* = 7.0, 3.0 Hz), 123.7 (q, *J* = 270.0 Hz), 115.6 (d, *J* = 23.0 Hz), 59.4, 31.6, 29.2; HRMS (ESI) calcd for $C_{17}H_{14}OF_4Na$ [M+Na^+] 333.0873, found 333.0870.

One of aromatic carbons overlapped with other aromatic carbons in ^{13}C NMR spectrum.

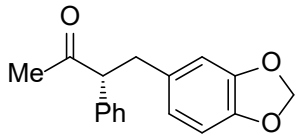
(3*R*)-4-(3,4,5-Trimethoxyphenyl)-3-phenyl-2-butanone (2h)



54% yield, 93% ee. white solid. mp 88-89 °C; $[\alpha]_D^{25}$ −182 (*c* 0.62, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IA (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 95/5, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 23.9 min (major), 26.9 min (minor)]. IR ($CHCl_3$) ν_{max} 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.33-7.21 (m, 3H), 7.16-7.14 (m, 2H), 6.17 (s, 2H), 3.85 (br t, *J* = 7.5 Hz, 1H), 3.78 (s, 3H), 3.71 (s, 6H), 3.33 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.84 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.05 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.3, 152.7, 138.4, 135.2, 128.8, 128.3, 127.3, 106.0, 61.6, 60.8, 56.0, 38.7, 29.6; HRMS (ESI) calcd for $C_{19}H_{22}O_4Na$ [M+Na^+] 337.1410, found 337.1408.

One of aromatic carbons overlapped with other aromatic carbons in ^{13}C NMR spectrum.

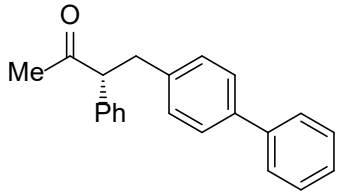
(3*R*)-4-(1,3-Benzodioxol-5-yl)-3-phenyl-2-butanone (2i)



58% yield, 93% ee. a colorless oil. $[\alpha]_D^{25}$ −225 (*c* 0.44, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IA (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.)

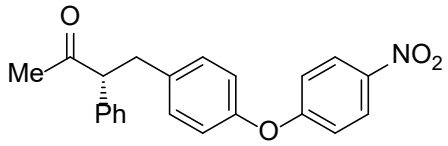
*n*hexane/*i*PrOH = 99/1, flow rate = 1.0 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 12.5 min (minor), 14.2 min (major)]. IR (neat) ν_{max} 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.32-7.20 (m, 3H), 7.16-7.14 (m, 2H), 6.62 (d, *J* = 7.5 Hz, 1H), 6.52 (d, *J* = 1.5 Hz, 1H), 6.47 (dd, *J* = 7.5, 1.5 Hz, 1H), 5.86 (s, 2H), 3.85 (br t, *J* = 7.5 Hz, 1H), 3.33 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.80 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.7, 147.4, 145.8, 138.3, 133.4, 128.9, 128.3, 127.4, 121.9, 109.4, 108.0, 100.7, 61.7, 38.0, 29.6; HRMS (ESI) calcd for $C_{17}H_{16}O_3Na$ [M+Na^+] 291.0992, found 291.0996.

(3*R*)-4-[1,1'-Biphenyl]-4-yl-3-phenyl-2-butanone (2j)



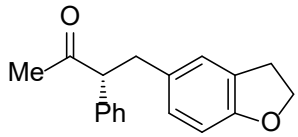
72% yield, 92% ee. white solid. mp 96-97 °C; $[\alpha]_D^{25}$ −259 (*c* 0.83, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK ID (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 13.4 min (minor), 15.0 min (major)]. IR ($CHCl_3$) ν_{max} 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.55 (br d, *J* = 7.5 Hz, 2H), 7.46-7.19 (m, 10H), 7.12 (d, *J* = 8.0 Hz, 2H), 3.96 (br t, *J* = 7.5 Hz, 1H), 3.47 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.94 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.05 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.3, 140.7, 138.8, 138.7, 138.3, 129.3, 128.8, 128.6, 128.2, 127.3, 126.9, 126.81, 126.80, 61.5, 38.0, 29.6; HRMS (ESI) calcd for $C_{22}H_{20}ONa$ [M+Na^+] 323.1406, found 323.1406.

(3*R*)-4-[4-(4-Nitrophenoxy)phenyl]-3-phenyl-2-butanone (2k)



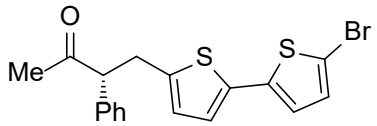
61% yield, 92% ee. pale yellow solid. mp 72-73 °C; $[\alpha]_D^{25}$ −226 (*c* 0.82, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IB (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 95/5, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 23.4 min (minor), 24.8 min (major)]. IR ($CHCl_3$) ν_{max} 1713, 1516, 1344 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 8.15 (br d, *J* = 9.0 Hz, 2H), 7.33-7.24 (m, 3H), 7.15 (br d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.94-6.88 (m, 4H), 3.90 (br t, *J* = 7.5 Hz, 1H), 3.43 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.92 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.05 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.1, 163.3, 152.7, 142.4, 138.0, 136.9, 130.7, 128.9, 128.2, 127.4, 125.7, 120.2, 116.8, 61.5, 37.6, 29.5; HRMS (ESI) calcd for $C_{22}H_{19}O_4NNa$ [M+Na^+] 384.1206, found 384.1208.

(3*R*)-4-(2,3-Dihydro-5-benzofuranyl)-3-phenyl-2-butanone (2l)



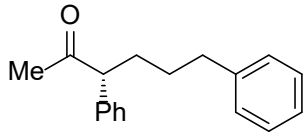
51% yield, 80% ee. a colorless oil. $[\alpha]_D^{25}$ −65 (*c* 0.17, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/THF = 90/10, flow rate = 0.3 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 21.4 min (minor), 22.4 min (major)]. IR (neat) ν_{max} 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.34-7.26 (m, 3H), 7.19 (br d, *J* = 7.5 Hz, 2H), 6.89 (br s, 1H), 6.78 (br d, *J* = 8.0 Hz, 1H), 6.62 (br d, *J* = 8.0 Hz, 1H), 4.51 (t, *J* = 8.5 Hz, 2H), 3.88 (br t, *J* = 7.5 Hz, 1H), 3.35 (dd, *J* = 13.5, 7.5 Hz, 1H), 3.12 (t, *J* = 8.5 Hz, 2H), 2.81 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 208.0, 158.4, 138.6, 131.6, 128.9, 128.5, 128.33, 128.31, 127.3, 125.5, 108.8, 71.1, 62.1, 62.0, 37.8, 29.7; HRMS (ESI) calcd for $C_{18}H_{18}O_2Na$ [M+Na^+] 289.1199, found 289.1202.

(3*R*)-4-[5'-Bromo-(2,2'-bithiophene)-5-yl]-3-phenyl-2-butanone (2m)



57% yield, 93% ee. white solid. mp 98-99 °C; $[\alpha]_D^{25}$ −202 (*c* 0.50, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 24.1 min (minor), 29.1 min (major)]. IR ($CHCl_3$) ν_{max} 1713 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.36-7.20 (m, 5H), 6.92 (dd, *J* = 3.5, 1.0 Hz, 1H), 6.83 (br d, *J* = 3.5 Hz, 1H), 6.79 (br d, *J* = 3.5 Hz, 1H), 6.54 (br dd, *J* = 3.5, 1.0 Hz, 1H), 3.93 (br t, *J* = 7.5 Hz, 1H), 3.59 (dd, *J* = 13.5, 7.5 Hz, 1H), 3.07 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.07 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 206.7, 141.8, 139.0, 137.6, 134.5, 130.4, 129.0, 128.2, 127.6, 126.3, 123.5, 123.1, 110.3, 61.4, 32.7, 29.4; HRMS (ESI) calcd for $C_{18}H_{15}O^{79}BrS_2Na$ [M+Na^+] 412.9640, found 412.9647.

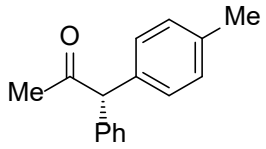
(3*R*)-3,6-diphenyl-2-hexanone (2n)



61% yield, 80% ee. a colorless oil. $[\alpha]_D^{25}$ −122 (*c* 0.45, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 19.4 min (minor), 23.2 min (major)]. IR (neat) ν_{max} 1712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.35-7.11 (m, 10H), 3.60 (br t, *J* = 7.5 Hz, 1H), 2.68-2.51 (m, 2H), 2.14-2.03 (m, 1H), 2.03, (s, 3H), 1.81-1.69 (m, 1H), 1.62-1.43 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 208.4, 142.1, 138.8, 128.9,

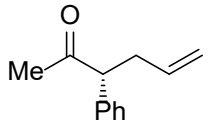
128.34, 128.26, 128.22, 127.2, 125.7, 59.6, 35.8, 31.4, 29.2, 29.0; HRMS (ESI) calcd for $C_{18}H_{20}ONa$ [M+Na$^+$] 275.1406, found 275.1405.

1-(4-Methylphenyl)-1-pheny-2-propanone (2o)[10]



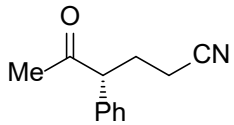
51% yield, 5% ee. a colorless oil. The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IA (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 20.1 min (major), 21.8 min (minor)]. IR (neat) ν_{max} 1714 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.36-7.19 (m, 6H), 7.16-7.09 (m, 3H), 5.08 (s, 1H), 2.32 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 206.7, 138.5, 136.9, 135.2, 129.4, 128.9, 128.8, 128.7, 127.1, 64.7, 30.0, 21.0; HRMS (ESI) calcd for $C_{16}H_{16}ONa$ [M+Na$^+$] 247.1093, found 247.1092.

(3*R*)-3-Phenyl-5-hexen-2-one (2p)



54% yield, 85% ee. a colorless oil. $[\alpha]_D^{25}$ −214 (*c* 0.35, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 13.0 min (minor), 14.0 min (major)]. IR (neat) ν_{max} 1715 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.37-7.26 (m, 3H), 7.23-7.20 (m, 2H), 5.67 (ddt, *J* = 17.0, 10.0, 6.5 Hz, 1H), 4.99 (br d, *J* = 17.0 Hz, 1H), 4.95 (br d, *J* = 10.0 Hz, 1H), 3.70 (br t, *J* = 7.5 Hz, 1H), 2.85-2.75 (m, 1H), 2.48-2.38 (m, 1H), 2.07 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.5, 138.3, 135.7, 128.9, 128.3, 127.4, 116.6, 59.4, 36.1, 29.1; HRMS (ESI) calcd for $C_{12}H_{14}ONa$ [M+Na$^+$] 197.0937, found 197.0934.

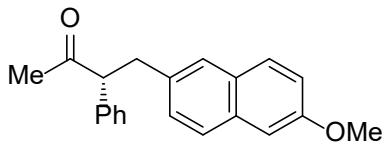
(4*R*)-5-Oxo-4-phenylhexanenitrile (2q)



61% yield, 83% ee. a colorless oil. $[\alpha]_D^{25}$ −293 (*c* 0.43, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IA (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 1.0 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 16.2 min (minor), 18.4 min (major)]. IR (neat) ν_{max} 2247, 1713 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.41-7.30 (m, 3H), 7.22-7.19 (m, 2H), 3.81 (br dd, *J* = 8.0, 6.5 Hz, 1H), 2.40-2.28 (m, 2H), 2.23-1.94 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 206.6, 136.8, 129.4, 128.2, 128.1, 119.1, 57.5, 29.1, 27.2,

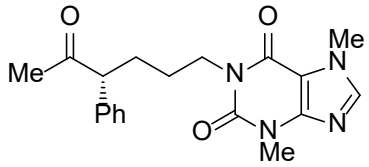
15.0; HRMS (ESI) calcd for $C_{12}H_{13}ONNa$ [M+Na$^+$] 210.0889, found 210.0890.

(3*R*)-4-(6-Methoxy-2-naphthalenyl)-3-phenyl-2-butanone (2r)



According to the general procedure for asymmetric nucleophilic α-phenylation of ketones **2b-q**, the reaction of nabumethone (17.1 mg, 0.075 mmol) with (+)-benzopyranoisoxazolidine **3** (26.6 mg, 0.15 mmol) and triphenylaluminium (1.0 M in *n*dibutyl ether, 0.23 mL, 0.23 mmol) gave α-phenylated nabumetone **2r** (15.3 mg, 67%, 89% ee) as white solid. mp 85-86 °C; $[\alpha]_D^{25}$ −195 (*c* 0.82, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/THF = 90/10, flow rate = 0.5 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 12.1 min (minor), 12.3 min (major)]. IR ($CHCl_3$) ν_{max} 1712 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.60 (br d, *J* = 7.0 Hz, 1H), 7.58 (br d, *J* = 7.0 Hz, 1H), 7.42 (br s, 1H), 7.32-7.10 (m, 7H), 7.07 (br s, 1H), 4.00 (br t, *J* = 7.5 Hz, 1H), 3.89 (s, 3H), 3.55 (dd, *J* = 13.5, 7.5 Hz, 1H), 3.01 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 207.8, 157.2, 138.5, 134.8, 133.0, 129.0, 128.9 (2C), 128.3, 128.0, 127.4, 127.2, 126.6, 118.6, 105.5, 61.6, 55.2, 38.3, 29.6; HRMS (ESI) calcd for $C_{21}H_{20}O_2Na$ [M+Na$^+$] 327.1356, found 327.1357.

(*R*)-3,7-Dihydro-3,7-dimethyl-1-(5-oxo-4-phenylhexyl)-1*H*-purine-2,6-dione (2s)

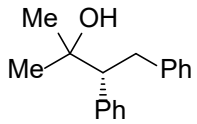


According to the general procedure for asymmetric nucleophilic α-phenylation of ketones **1b-q**, the reaction of pentoxifylline (20.9 mg, 0.075 mmol) with (+)-benzopyranoisoxazolidine **3** (26.6 mg, 0.15 mmol) and triphenylaluminium (1.0 M in *n*dibutyl ether, 0.23 mL, 0.23 mmol) gave α-phenylated pentoxifylline **2s** (13.3 mg, 50%, 75% ee) as white solid. mp 168-169 °C; $[\alpha]_D^{25}$ −147 (*c* 0.58, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IC (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 50/50, flow rate = 2.0 mL/min, λ = 215 nm, temperature: 25 °C, retention times: 15.0 min (minor), 19.4 min (major)]. IR ($CHCl_3$) ν_{max} 1706, 1658 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.50 (s, 1H), 7.35-7.20 (m, 5H), 4.00 (br t, *J* = 7.5 Hz, 2H), 3.97 (s, 3H), 3.71 (br t *J* = 7.5 Hz, 1H), 3.55 (s, 3H), 2.14-2.05 (m, 1H), 2.06 (s, 3H), 1.84-1.68 (m, 1H), 1.65-1.14 (m, 2H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 208.2, 155.2, 151.3, 148.7, 141.4, 138.7, 128.9, 128.3, 127.2, 107.5, 59.1, 40.8, 33.5, 29.6, 29.1, 28.8, 25.8; HRMS (ESI) calcd for $C_{19}H_{22}O_3N_4Na$ [M+Na$^+$] 377.1584, found 377.1585.

Asymmetric nucleophilic α-phenylation of ketone 1a on 3.0 mmol scale (Scheme 4)

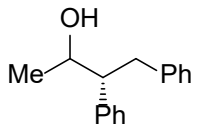
According to the experimental procedure for asymmetric α-phenylation of ketone **1a** (Table 1, entry 2), the reaction of 4-phenyl-2-butanone (**1a**) (444.6 mg, 3.0 mmol) and (+)-benzopyranoisoxazolidine **3** (1.06 g, 6.0 mmol) in CH_2Cl_2 (60 mL) with triphenylaluminium (1.0 M in *n*dibutyl ether, 9.0 mL, 9.0 mmol) at 0 °C for 2 h gave α-phenylated ketone **2a** (376.8 mg, 56%, 94% ee) as a colorless oil.

(3*R*)-3,4-diphenyl-2-methyl-2-butanol (4)



To a solution of **2a** (11.2 mg, 0.050 mmol) in THF (0.20 mL) was added MeMgBr (3.0 M in Et_2O, 36.7 μL, 0.11 mmol) dropwise at 0 °C under an argon atmosphere. After being stirred at the same temperature for 1 h, the reaction mixture was quenched with saturated NH_4Cl. The resulting suspension was extracted with $CHCl_3$. The organic phase was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by preparative TLC (*n*hexane/AcOEt = 3/1) to give *tert*-alcohol **4** (8.8 mg, 73%, 94% ee) as white solid. mp 68-69 °C; $[\alpha]_D^{25}$ −96 (*c* 0.38, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IA (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 254 nm, temperature: 25 °C, retention times: 36.6 min (major), 44.8 min (minor)]. IR ($CHCl_3$) ν_{max} 3448 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.23-7.01 (m, 8H), 6.95 (br d, *J* = 7.5 Hz, 2H), 3.28 (dd, *J* = 13.0, 3.0 Hz, 1H), 3.02 (br t, *J* = 13.0 Hz, 1H), 2.90 (dd, *J* = 13.0, 3.0 Hz, 1H), 1.41 (br s, 1H), 1.27 (s, 3H), 1.25 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 141.0, 140.6, 129.6, 128.7, 127.9 (2C), 126.5, 125.4, 73.0, 59.3, 36.2, 28.4, 28.0; HRMS (ESI) calcd for $C_{17}H_{20}ONa$ $[M+Na^+]$ 263.1406, found 263.1407.

(3*R*)-3,4-Diphenyl-2-butanol (5)[11]



To a solution of **2a** (10.8 mg, 0.044 mmol) in MeOH (0.50 mL) was added $NaBH_4$ (3.3 mg, 0.088 mmol) at 0 °C under an argon atmosphere. After being stirred at the same temperature for 30 min, the reaction mixture was quenched with saturated NH_4Cl. The resulting mixture was extracted with $CHCl_3$. The organic phase was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by preparative TLC (*n*hexane/Et_2O = 1/2) to give alcohol **5** (7.4 mg, 90%, 94% ee) as a colorless oil. The diastereomeric ratio (dr = 9:1) was calculated by ^1H NMR analysis of

crude product and the enantiomeric excess was calculated by HPLC analysis. The relative stereochemistry of the major diastereomer has not been established. The minor diastereomer could not be isolated.

(major diastereomer) $[\alpha]_D^{25}$ −61 (*c* 0.33, $CHCl_3$); The enantiomeric purity was determined by HPLC analysis [CHIRALPAK IB (0.46 cm x 25 cm, from Daicel Chemical Ind. Ltd.) *n*hexane/*i*PrOH = 99/1, flow rate = 0.5 mL/min, λ = 254 nm, temperature: 25 °C, retention times: 22.4 min (major), 27.1 min (minor)]. IR (neat) ν_{max} 3428 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.29-7.03 (m, 10H), 3.98 (m, 1H), 3.16 (dd, *J* = 13.0, 6.5 Hz, 1H), 2.90 (dd, *J* = 13.0, 8.5 Hz, 1H), 2.85-2.78 (m, 1H), 1.29 (br s, 1H), 1.17 (d, *J* = 6.0 H, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 140.4, 140.2, 129.00, 128.98, 128.2, 128.0, 126.7, 125.7, 69.5, 55.3, 38.6, 21.7; HRMS (ESI) calcd for $C_{16}H_{18}ONa$ [$M+Na^+$] 249.1250, found 249.1250.

III. References

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STANDARD 1H OBSERVE

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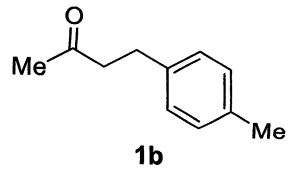

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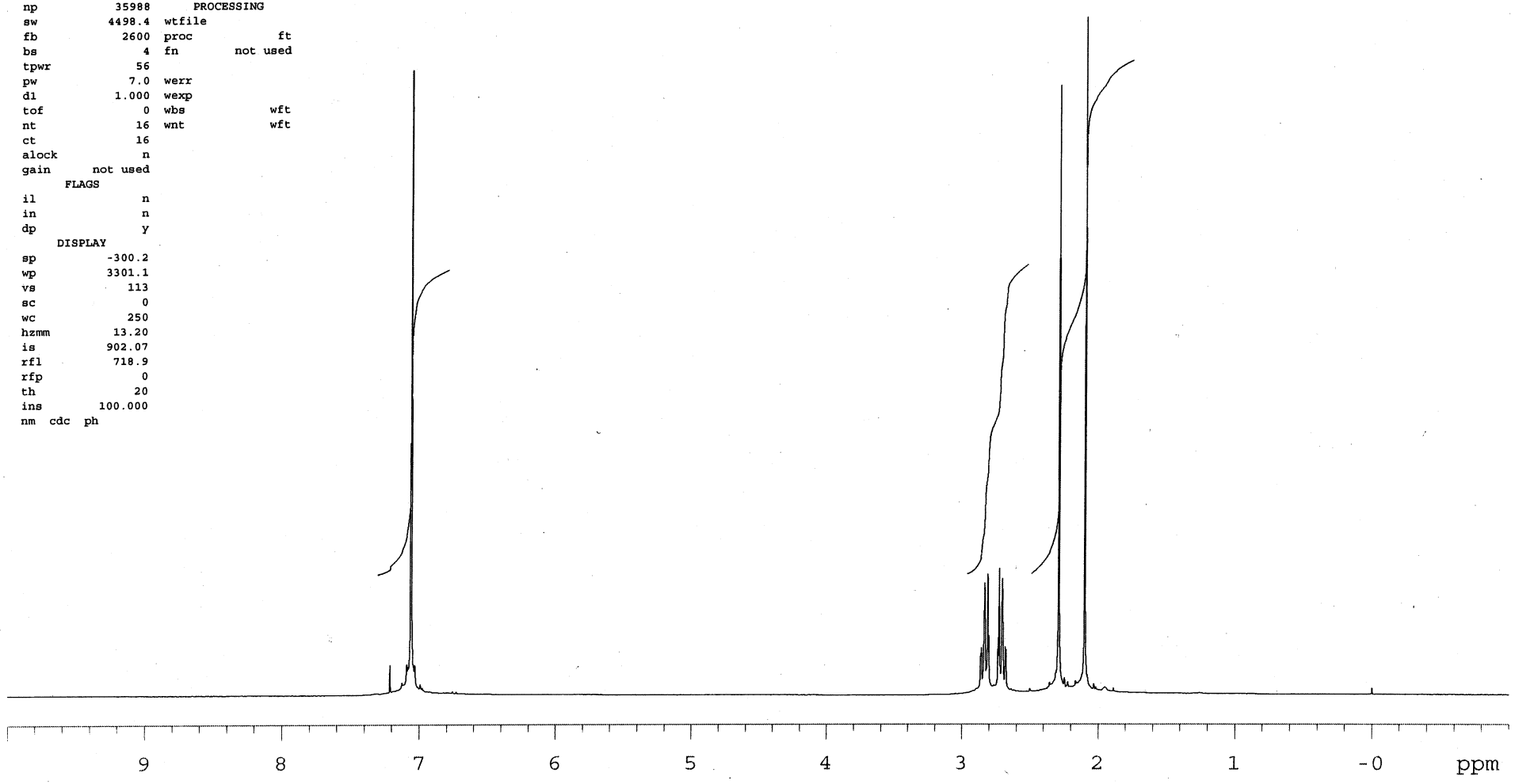

TN A-37 13C

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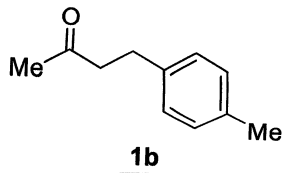

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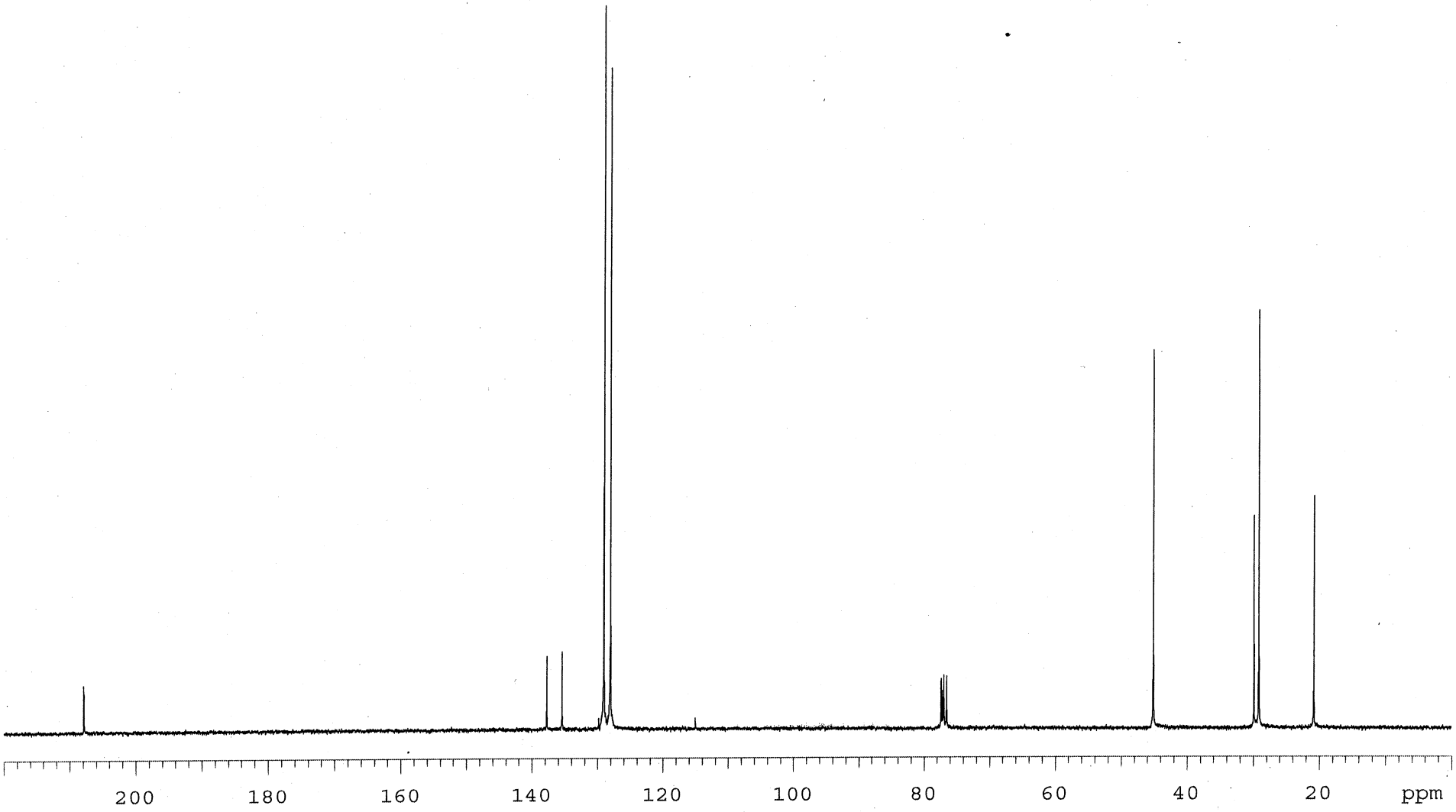

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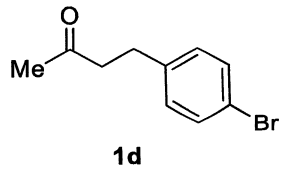

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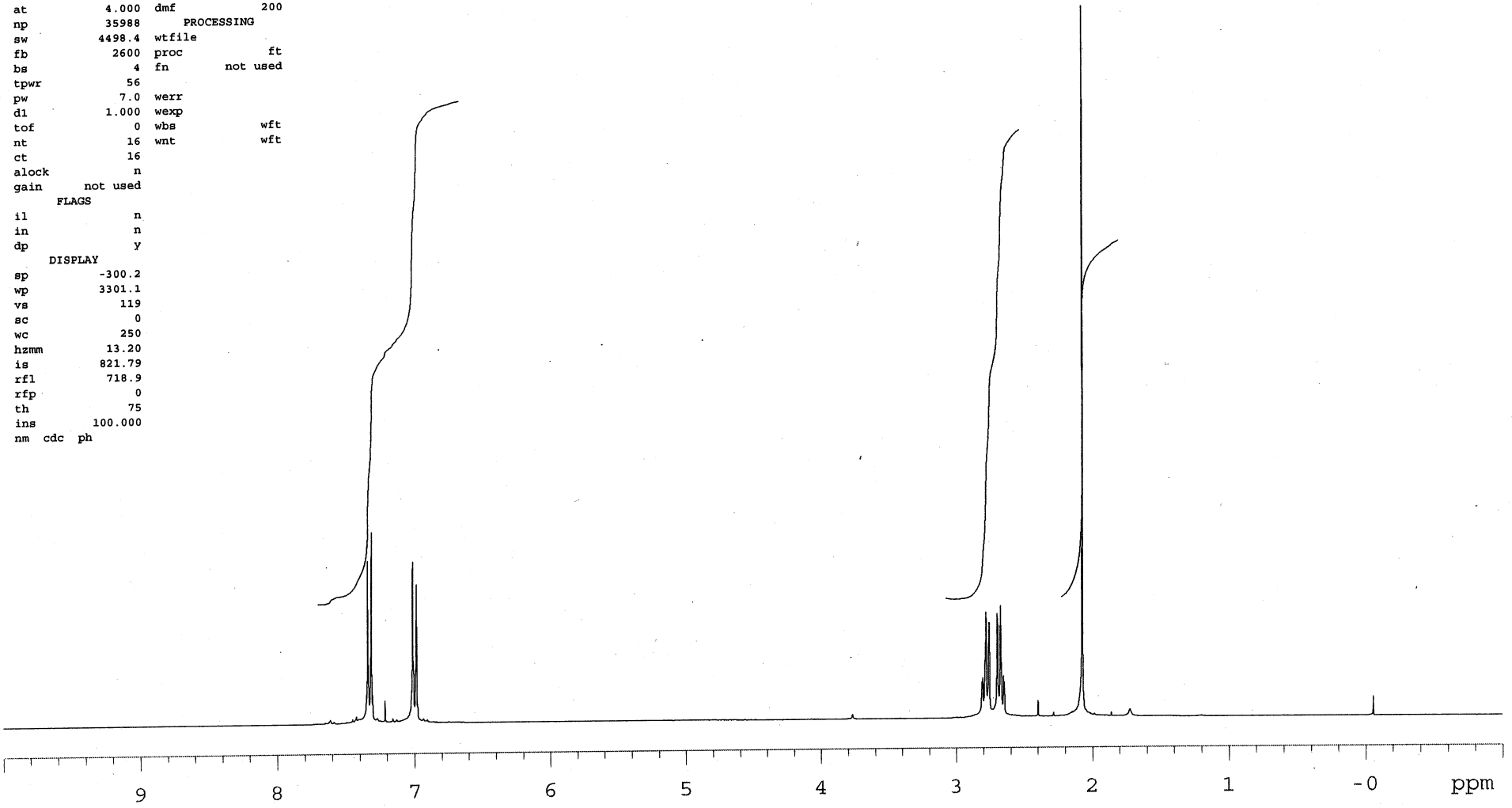

KO A-17 FF2 13C

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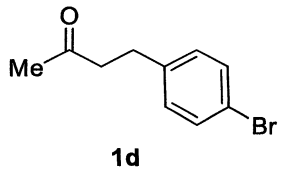

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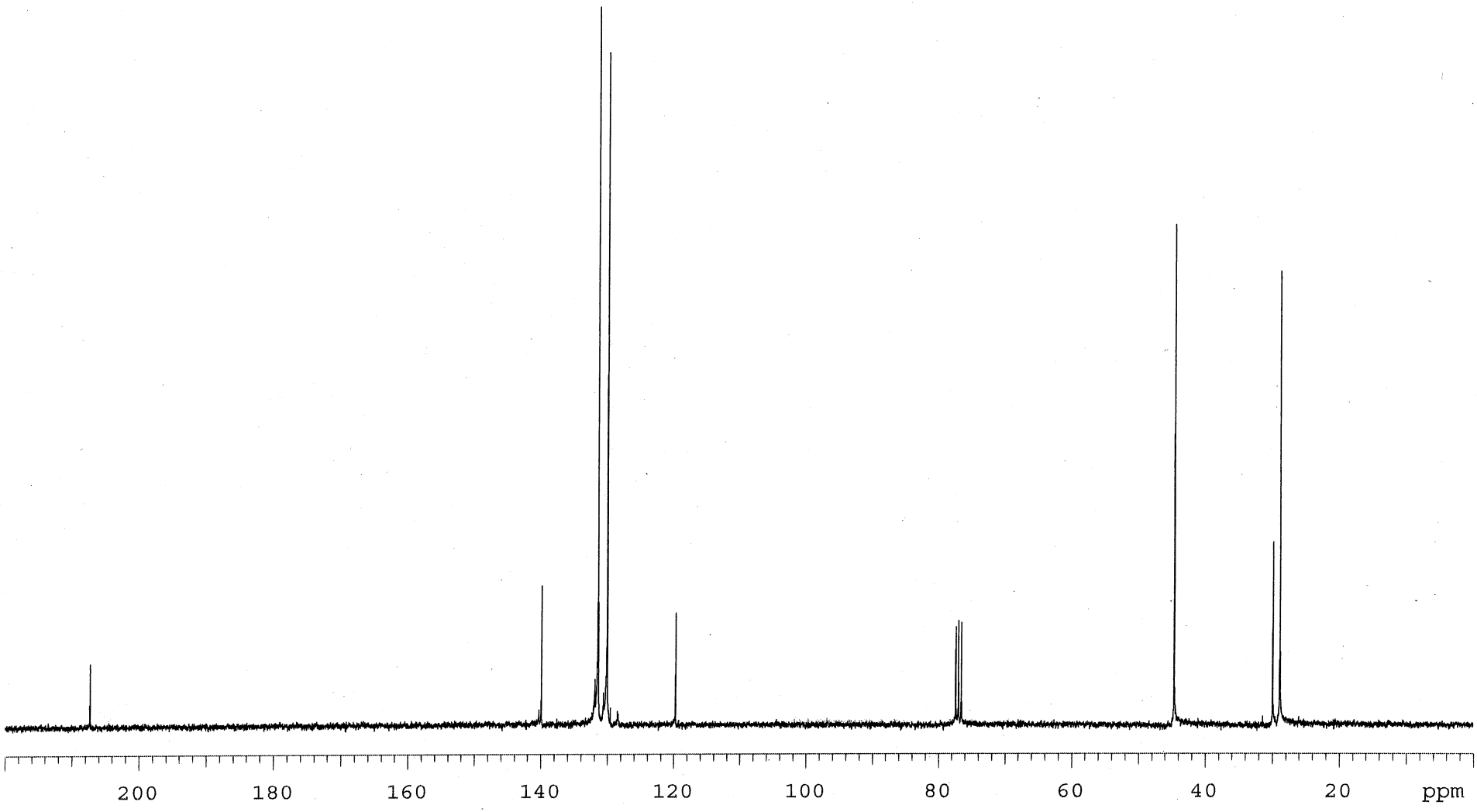

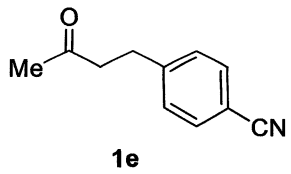

1e

MF D-52 FF1

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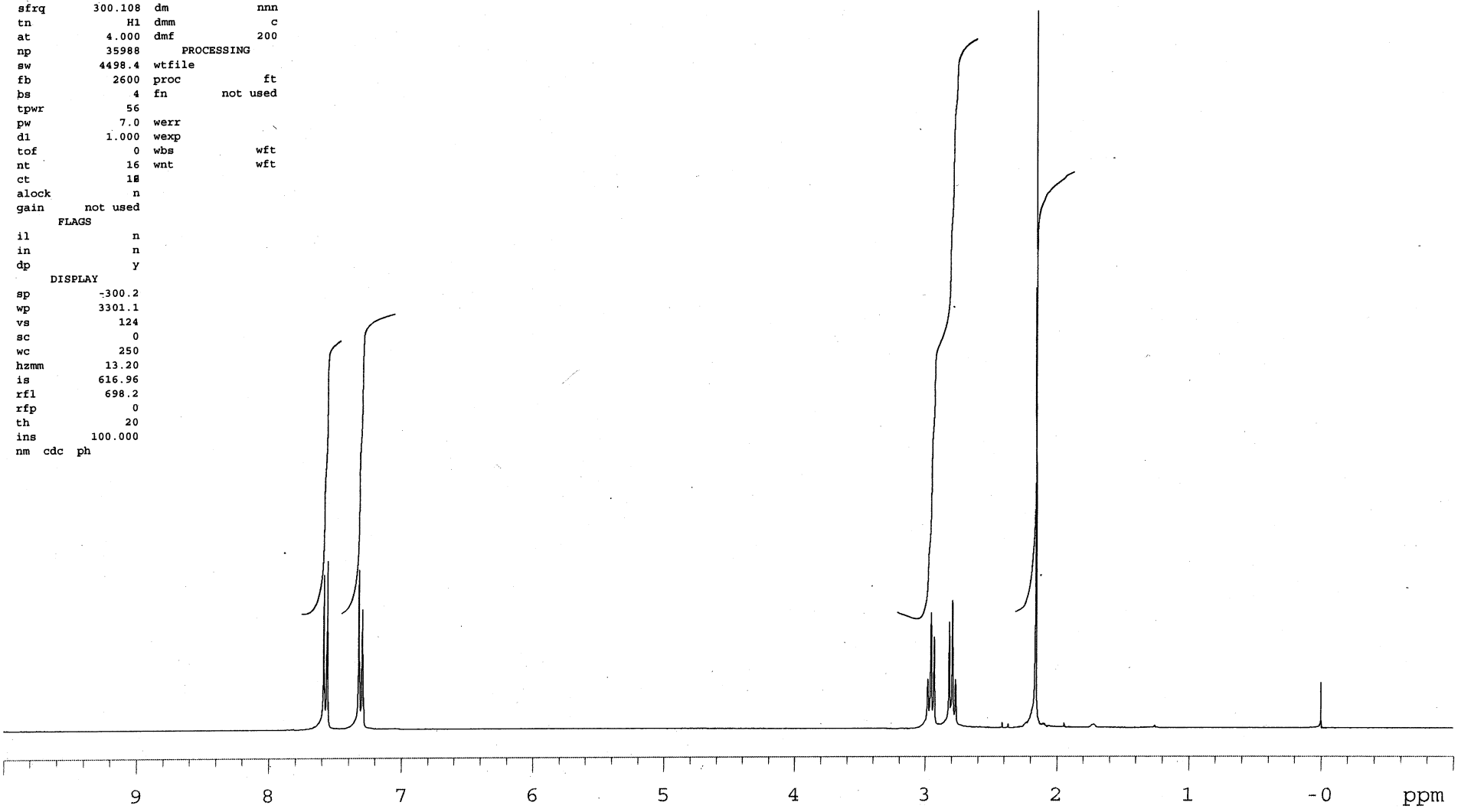

MF D-52 FF1 13C

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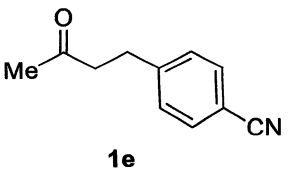

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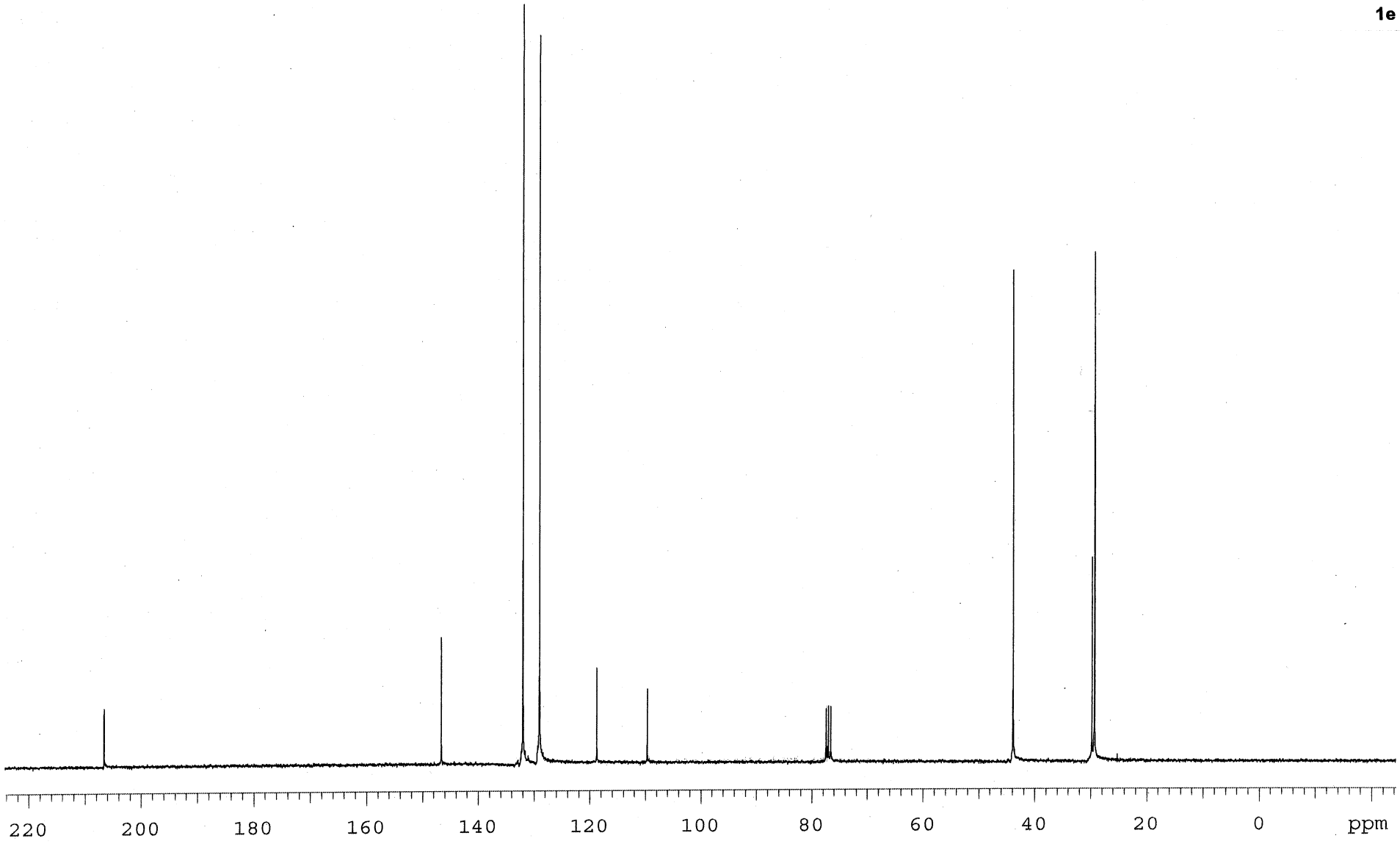

MM A-33

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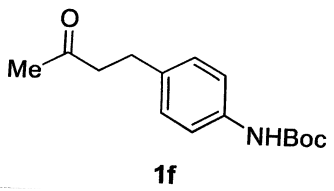

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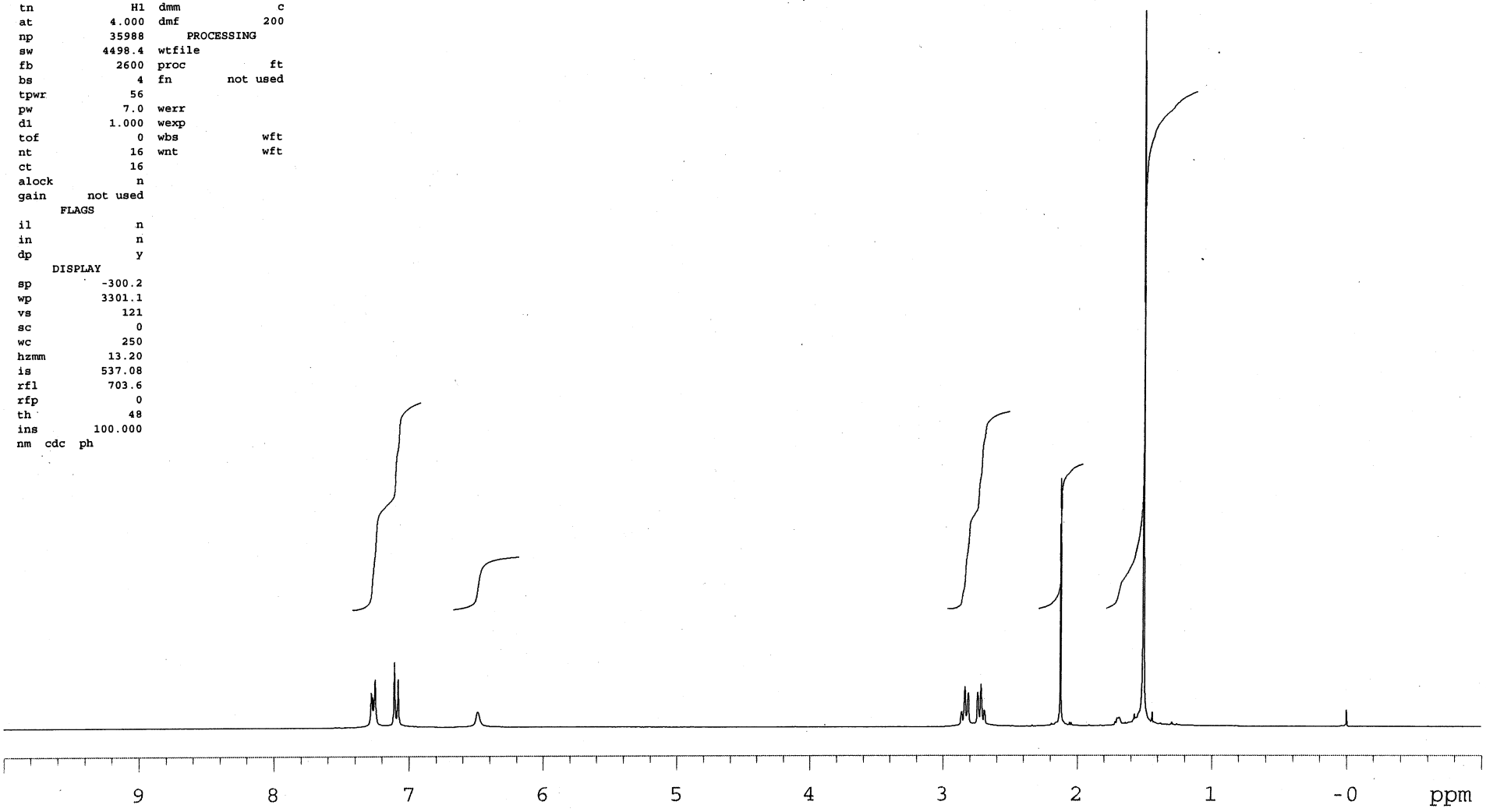

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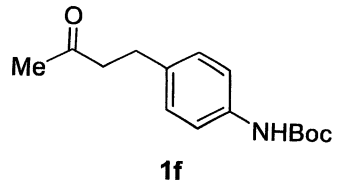

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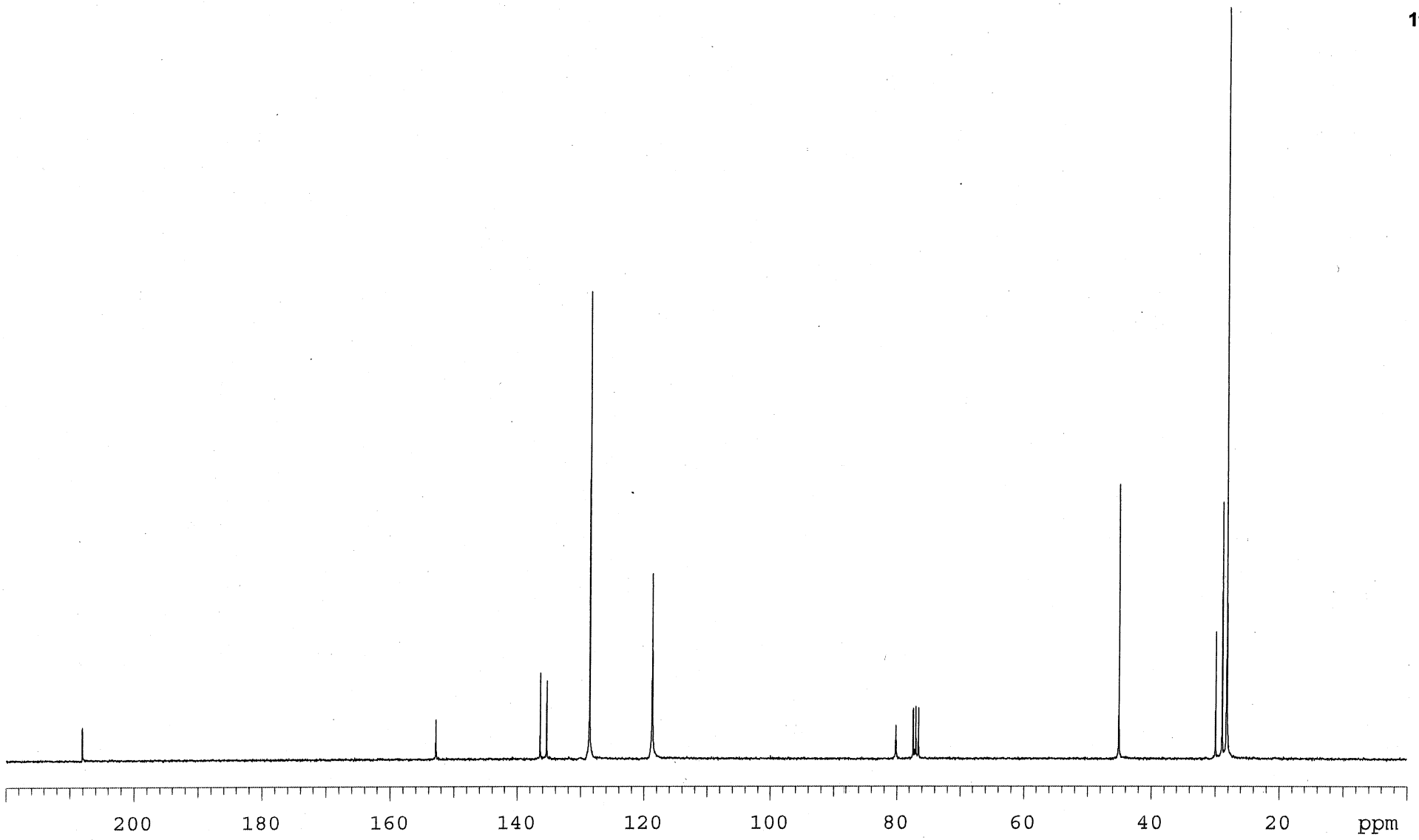
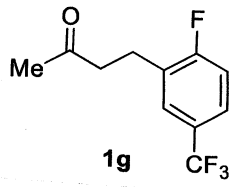
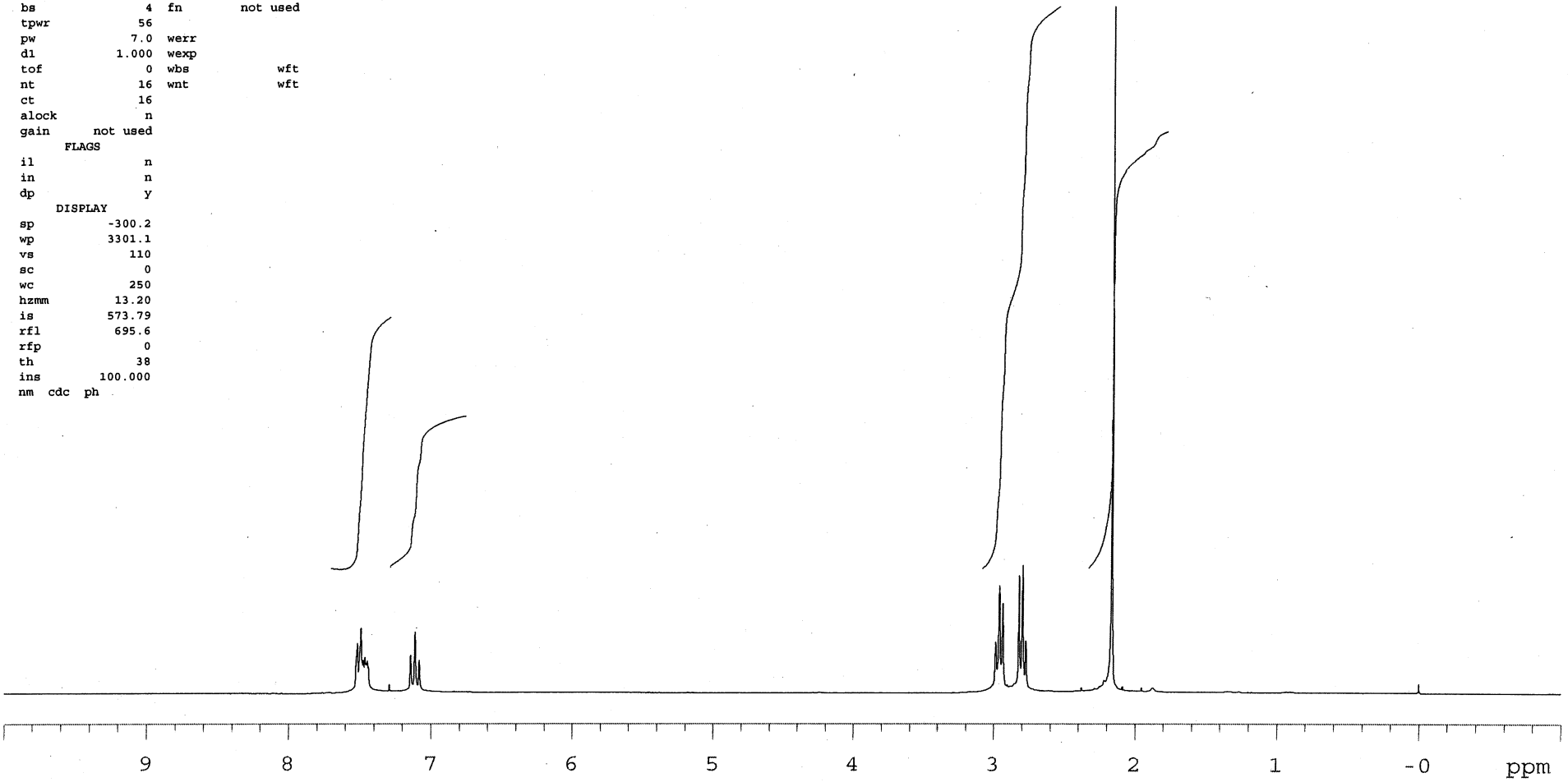


MF E-13 FF1

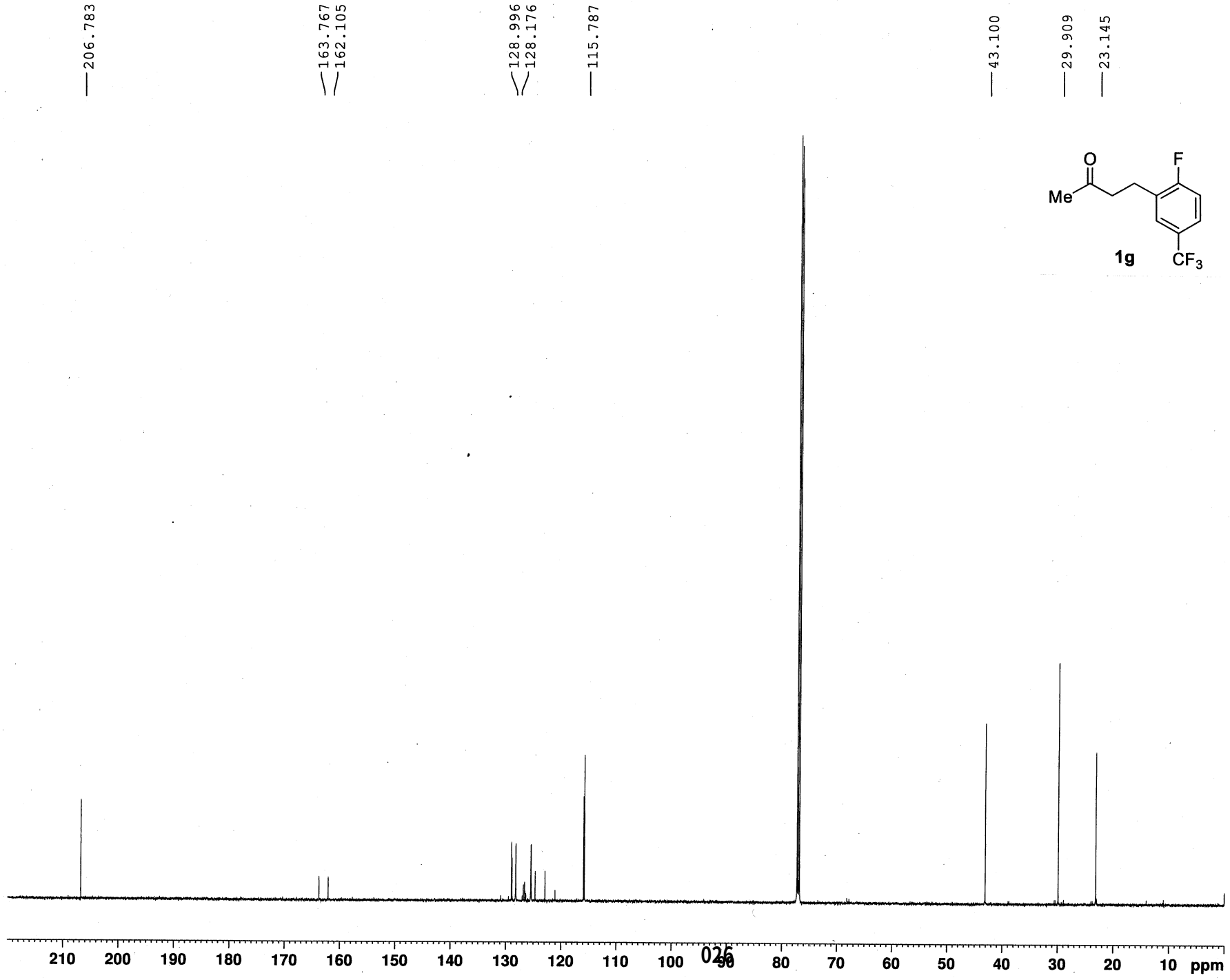
exp2 std1h

SAMPLE		DEC. & VT	
date	May 16 2017	dfrq	300.108
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.108	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	110		
sc	0		
wc	250		
hzmm	13.20		
is	573.79		
rfl	695.6		
rfp	0		
th	38		
ins	100.000		
nm cdc ph			





KPNN-5614 C-13

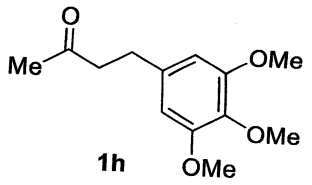



```
Current Data Parameters
NAME                  KPNN-5614
EXPNO                        20
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20170803
Time                     11.39 h
INSTRUM                  spect
PROBHD     Z114607_0202 (
PULPROG                  zgpg30
TD                        65536
SOLVENT                  CDCl3
NS                        1601
DS                           4
SWH                  36231.883 Hz
FIDRES                1.105709 Hz
AQ                    0.9043968 sec
RG                       194.13
DW                       13.800 usec
DE                        6.50 usec
TE                       296.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         300
SFO1           151.0184889 MHz
NUC1                       13C
P1                       12.00 usec
PLW1           80.00000000 W
SFO2           600.5336032 MHz
NUC2                       1H
CPDPRG[2                waltz65
PCPD2                    80.00 usec
PLW2           24.00600052 W
PLW12           0.37509000 W
PLW13           0.16671000 W

F2 - Processing parameters
SI                        32768
SF             151.0033860 MHz
WDW                         EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```

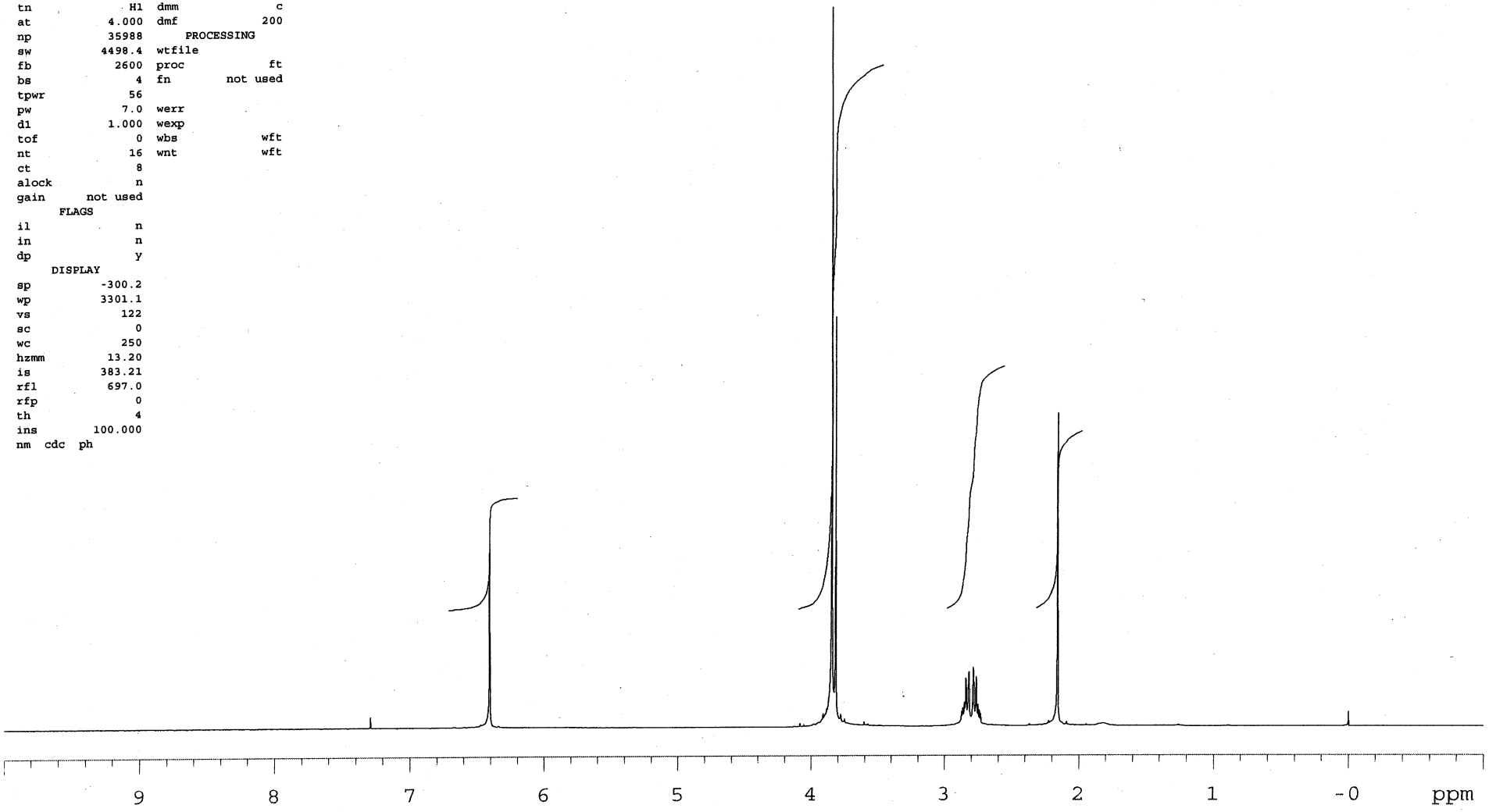
MF D-45 FF2

exp1 std1h

SAMPLE		DEC. & VT	
date	May 29 2017	dfrq	300.108
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.108	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	122		
sc	0		
wc	250		
hzmm	13.20		
is	383.21		
rfl	697.0		
rfp	0		
th	4		
ins	100.000		
nm cdc ph			

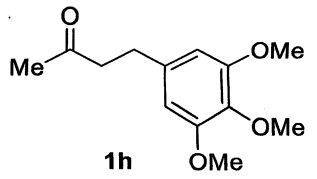


1h

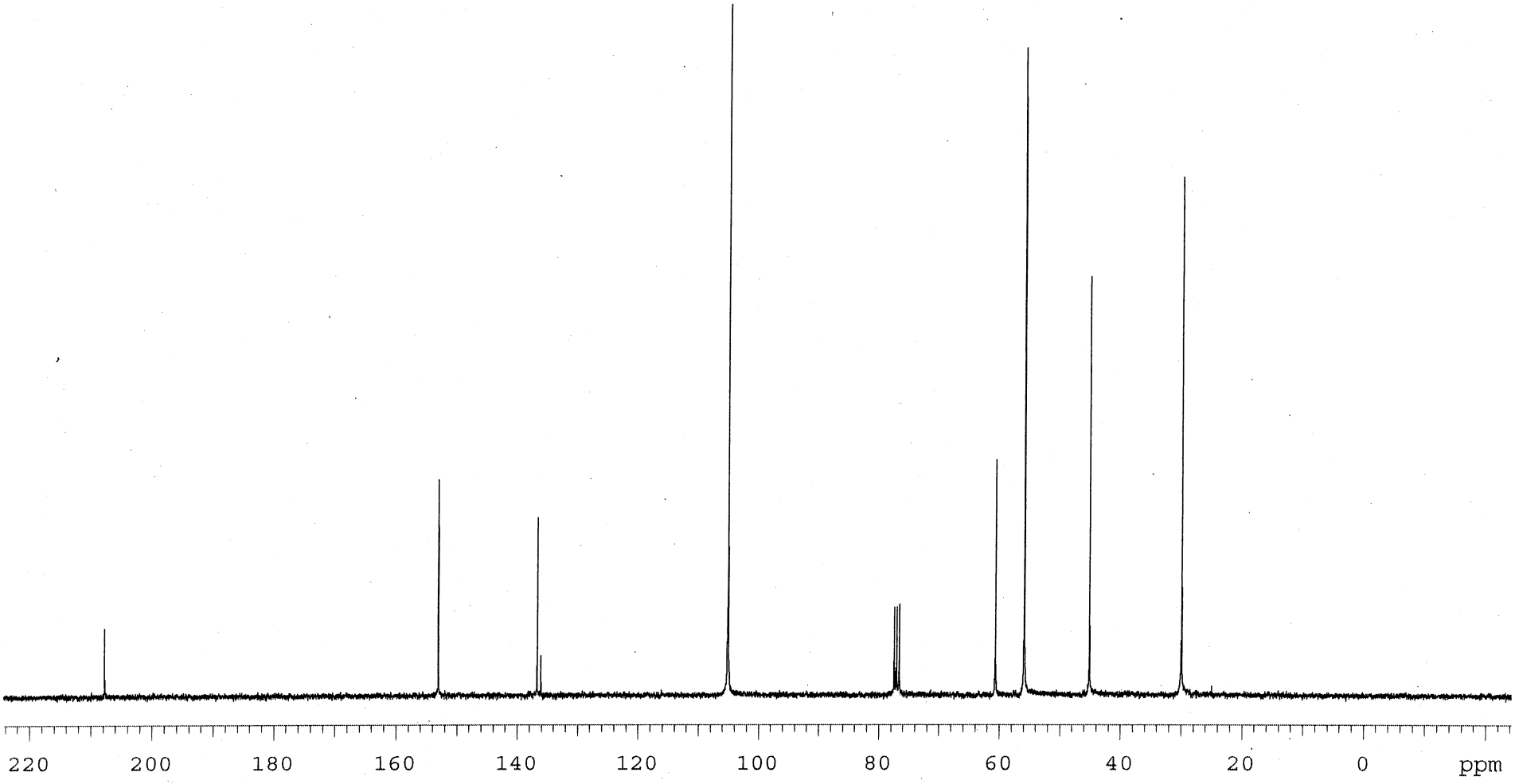


MF D-45 FF2 13C

Pulse Sequence: s2pul



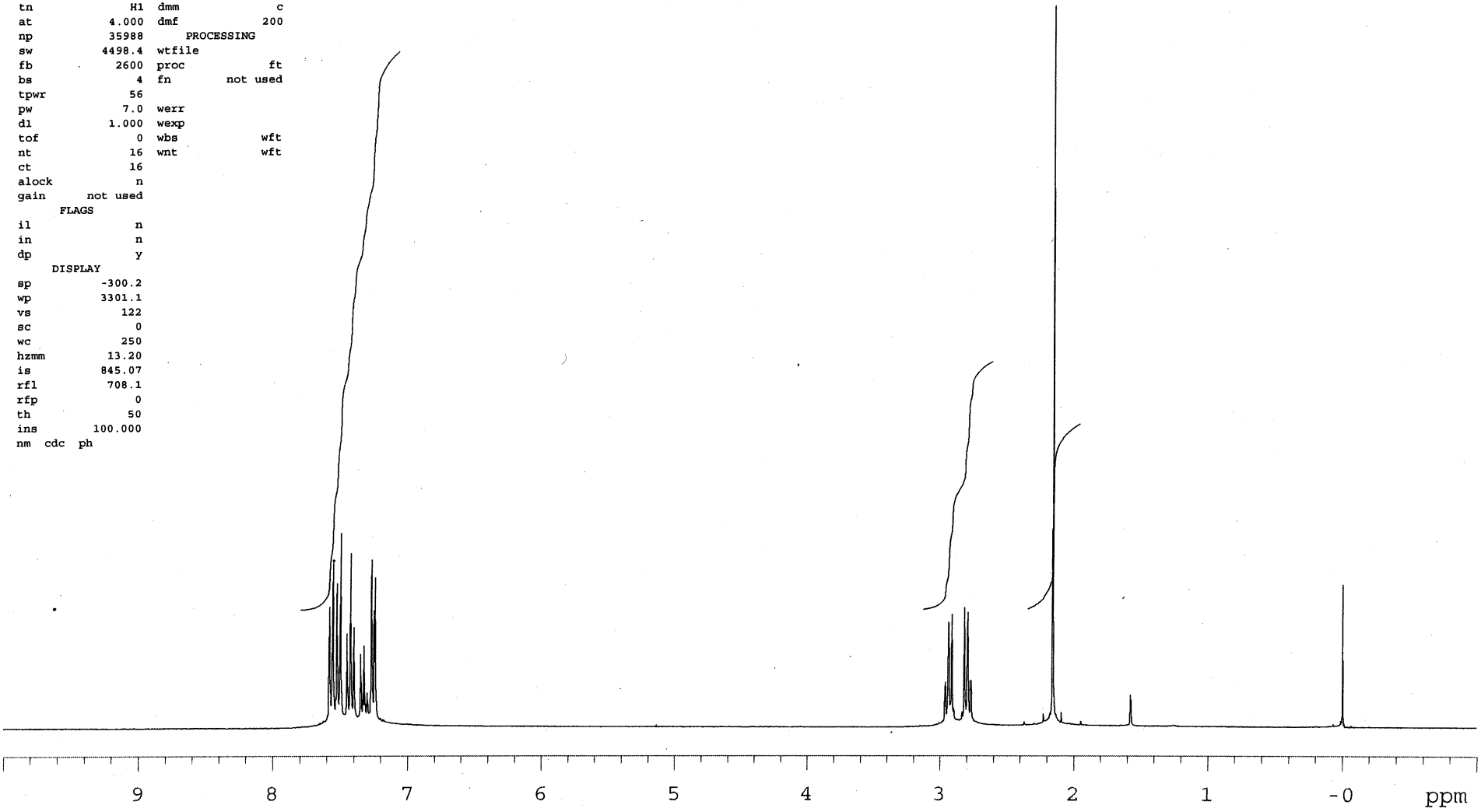
1h

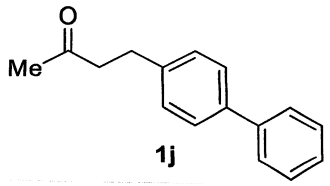


MF A-34

exp2 stdlh

SAMPLE		DEC. & VT	
date	May 13 2017	dfrq	300.108
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.108	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	122		
sc	0		
wc	250		
hzmm	13.20		
is	845.07		
rfl	708.1		
rfp	0		
th	50		
ins	100.000		
nm cdc ph			

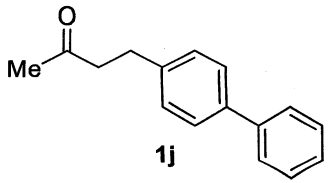




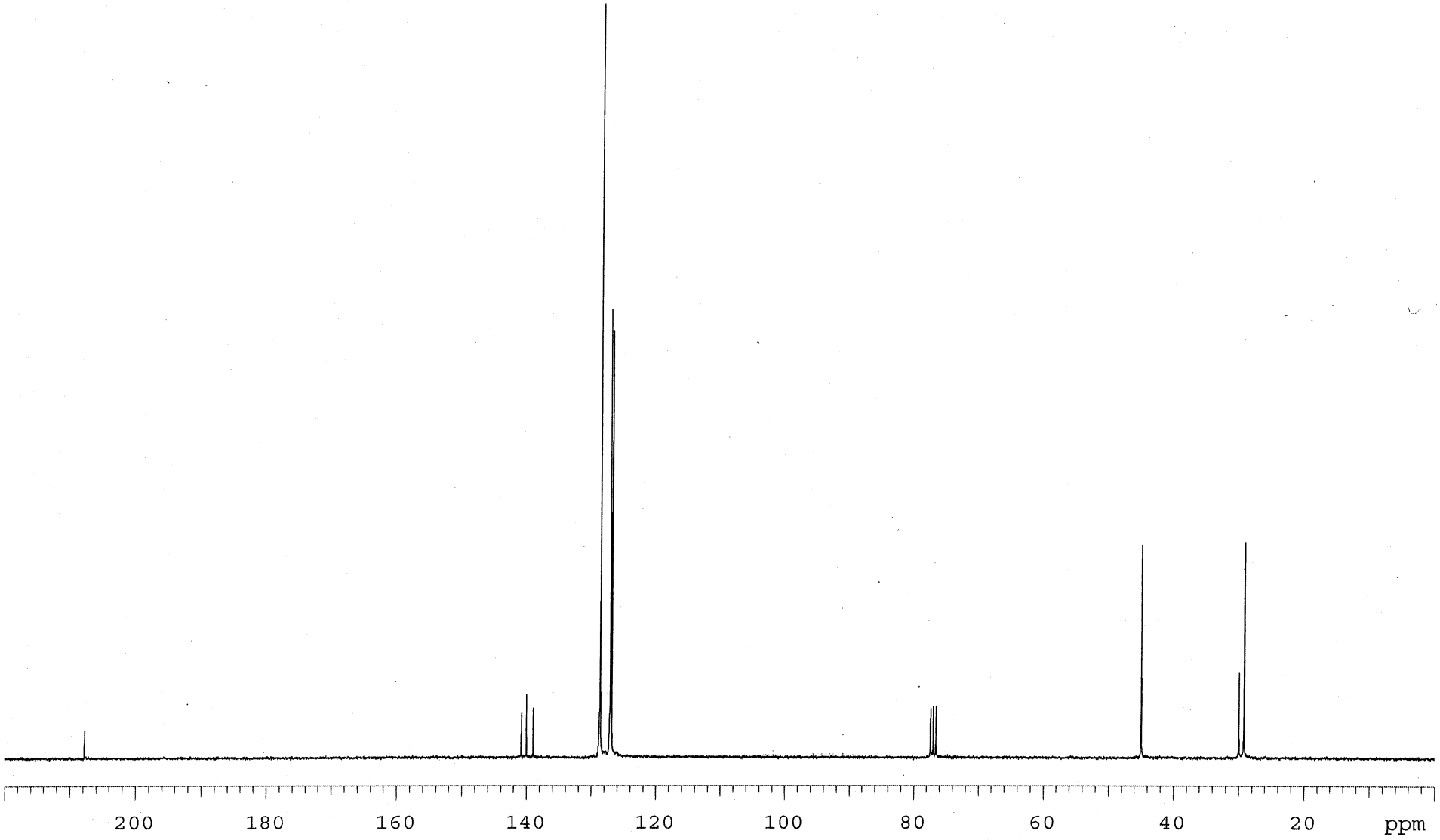
1j

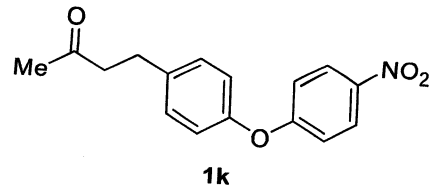
MF A-34 13C

Pulse Sequence: s2pul



1j



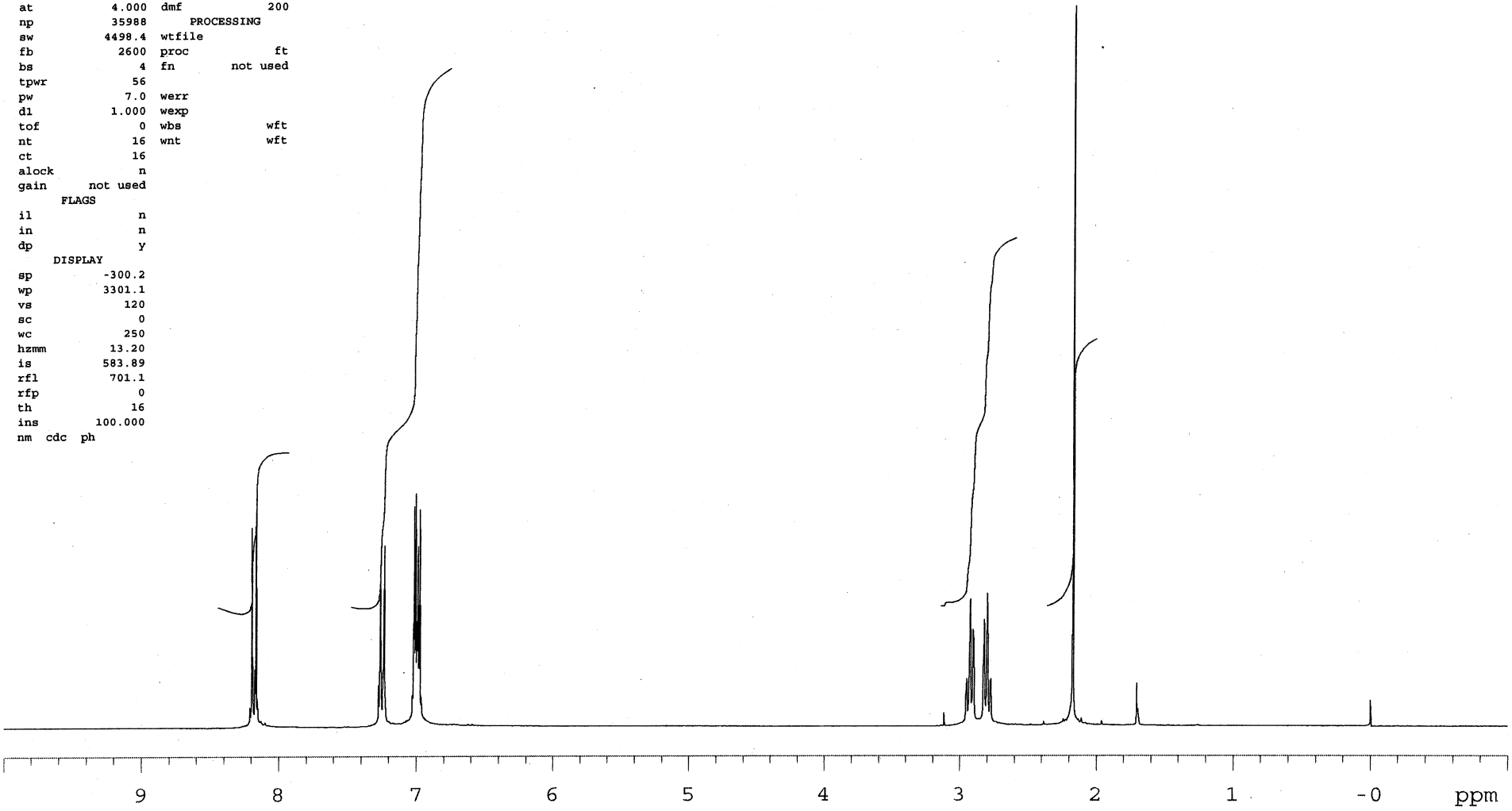


1k

MF D-47 FF1

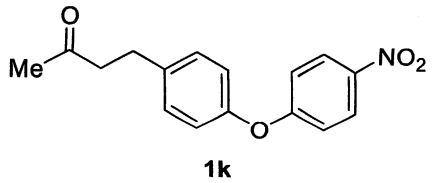
exp2 stdlh

SAMPLE		DEC. & VT	
date	May 16 2017	dfrq	300.108
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.108	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	120		
sc	0		
wc	250		
hzmm	13.20		
is	583.89		
rfl	701.1		
rfp	0		
th	16		
ins	100.000		
nm cdc ph			

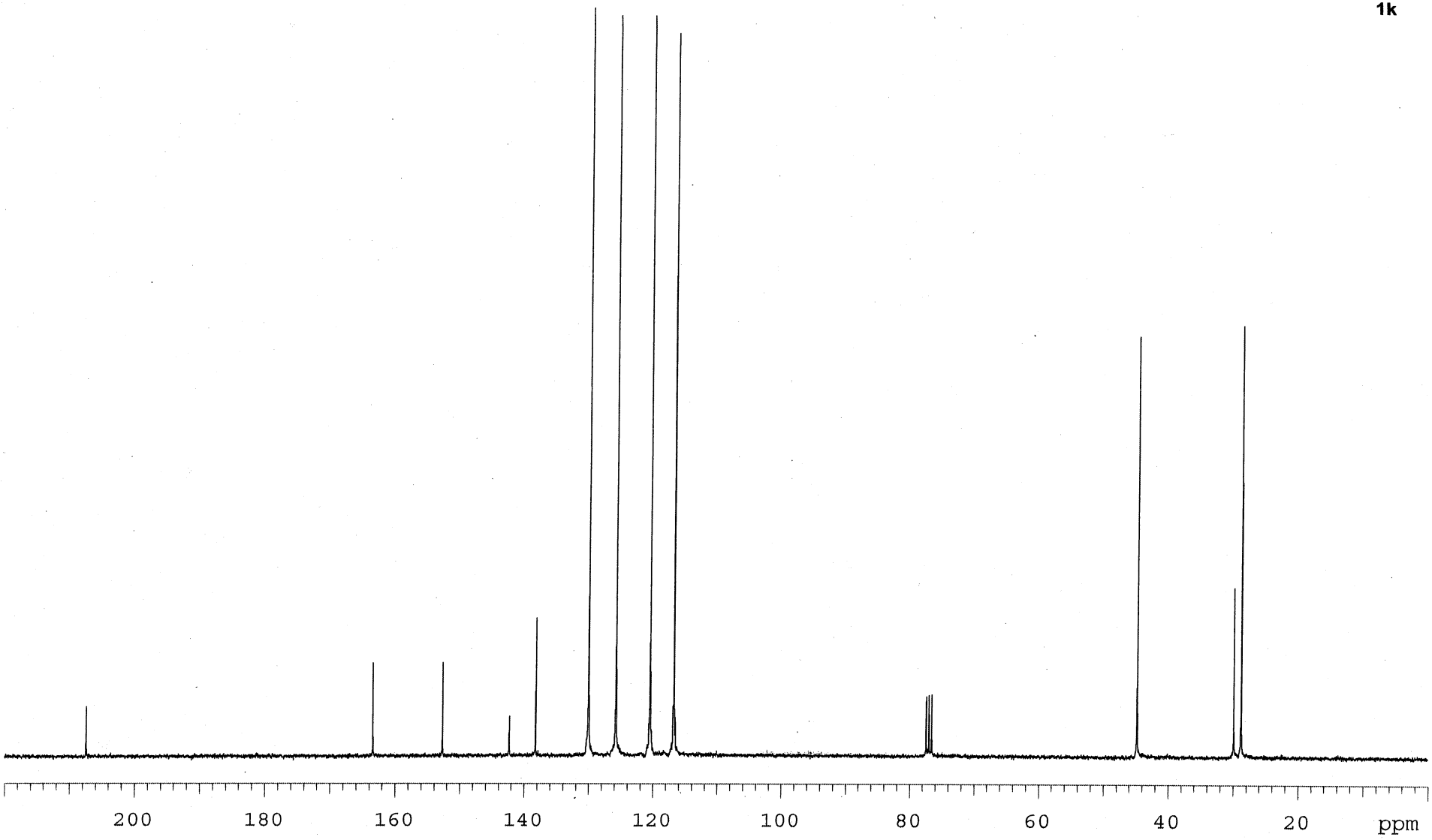


MF D-47 FF1 13C

Pulse Sequence: s2pul



1k

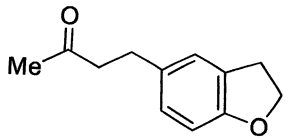


TK A-14

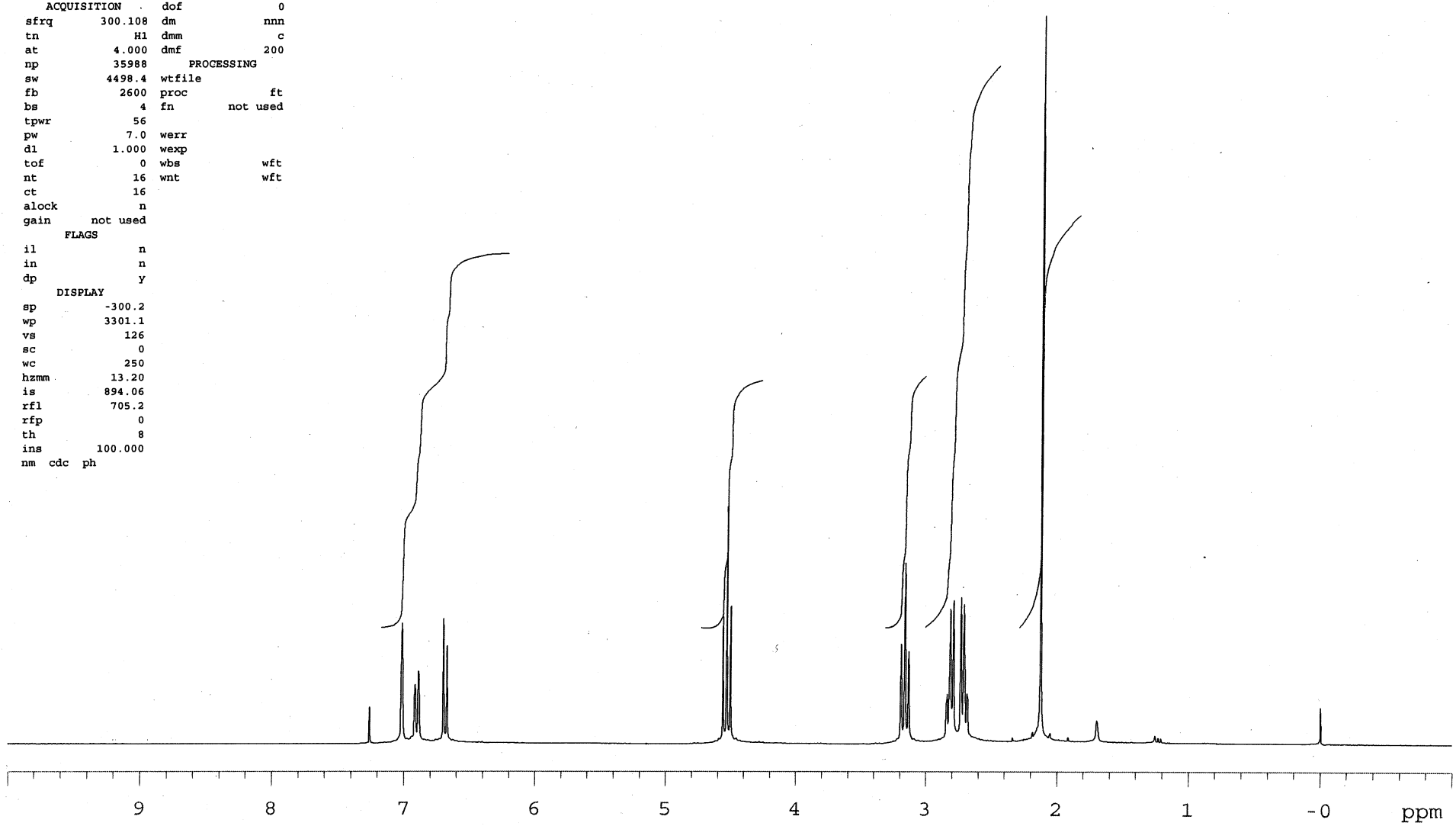
exp2 std1h

SAMPLE		DEC. & VT	
date	May 16 2017	dfrq	300.108
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.108	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	126		
sc	0		
wc	250		
hzmm	13.20		
is	894.06		
rfl	705.2		
rfp	0		
th	8		
ins	100.000		

nm cdc ph

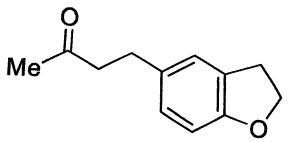


1l

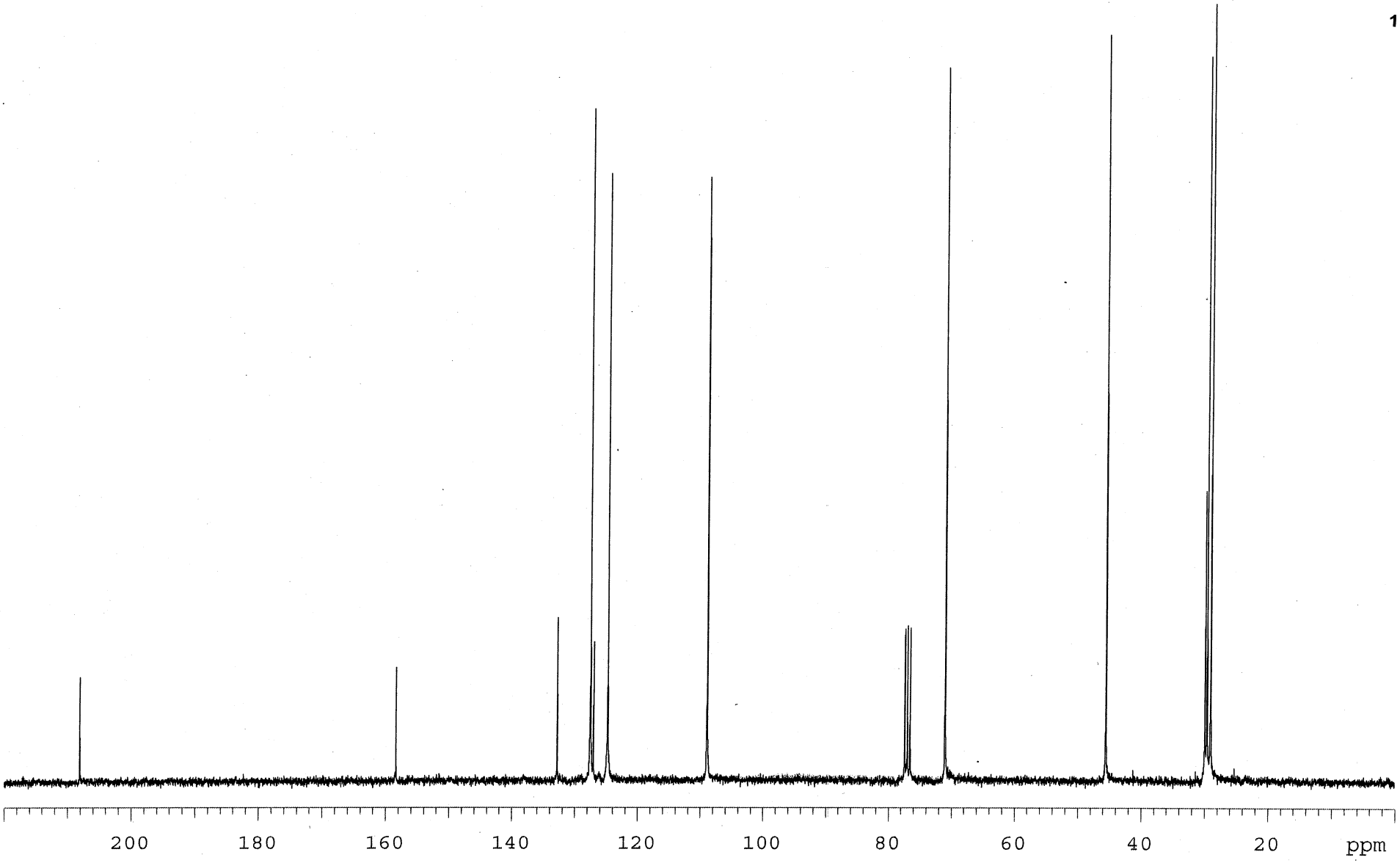


TK A-14 13C

Pulse Sequence: s2pul



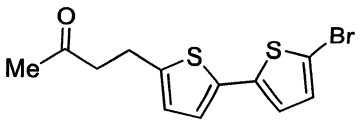
1l



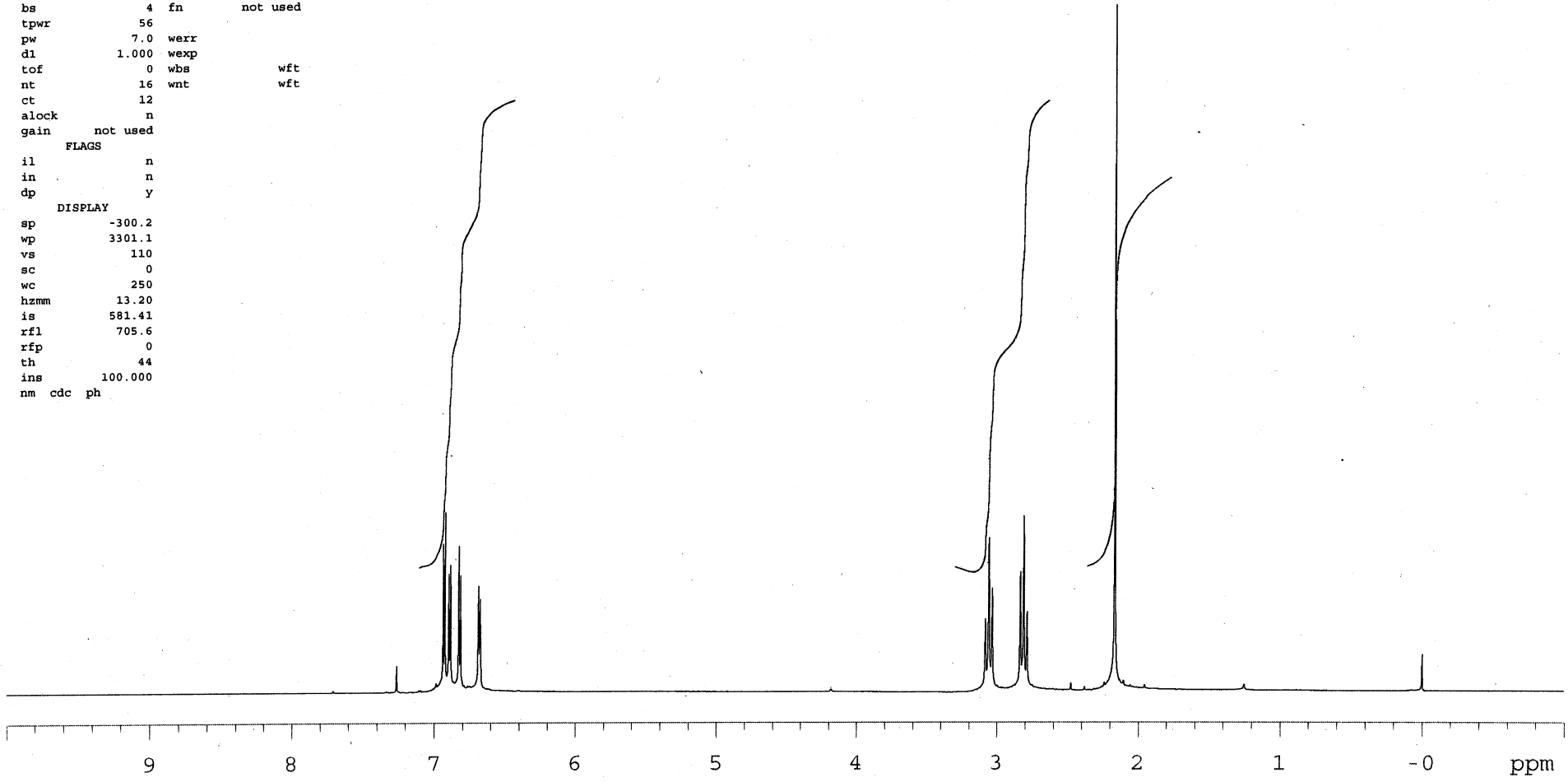
MF E-16 PTLC1

exp1 stdlh

SAMPLE		DEC. & VT	
date	May 29 2017	dfrq	300.108
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.108	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	12		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	110		
sc	0		
wc	250		
hzmm	13.20		
is	581.41		
rfl	705.6		
rfp	0		
th	44		
ins	100.000		
nm cdc ph			

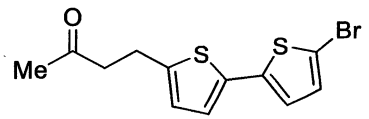


1m

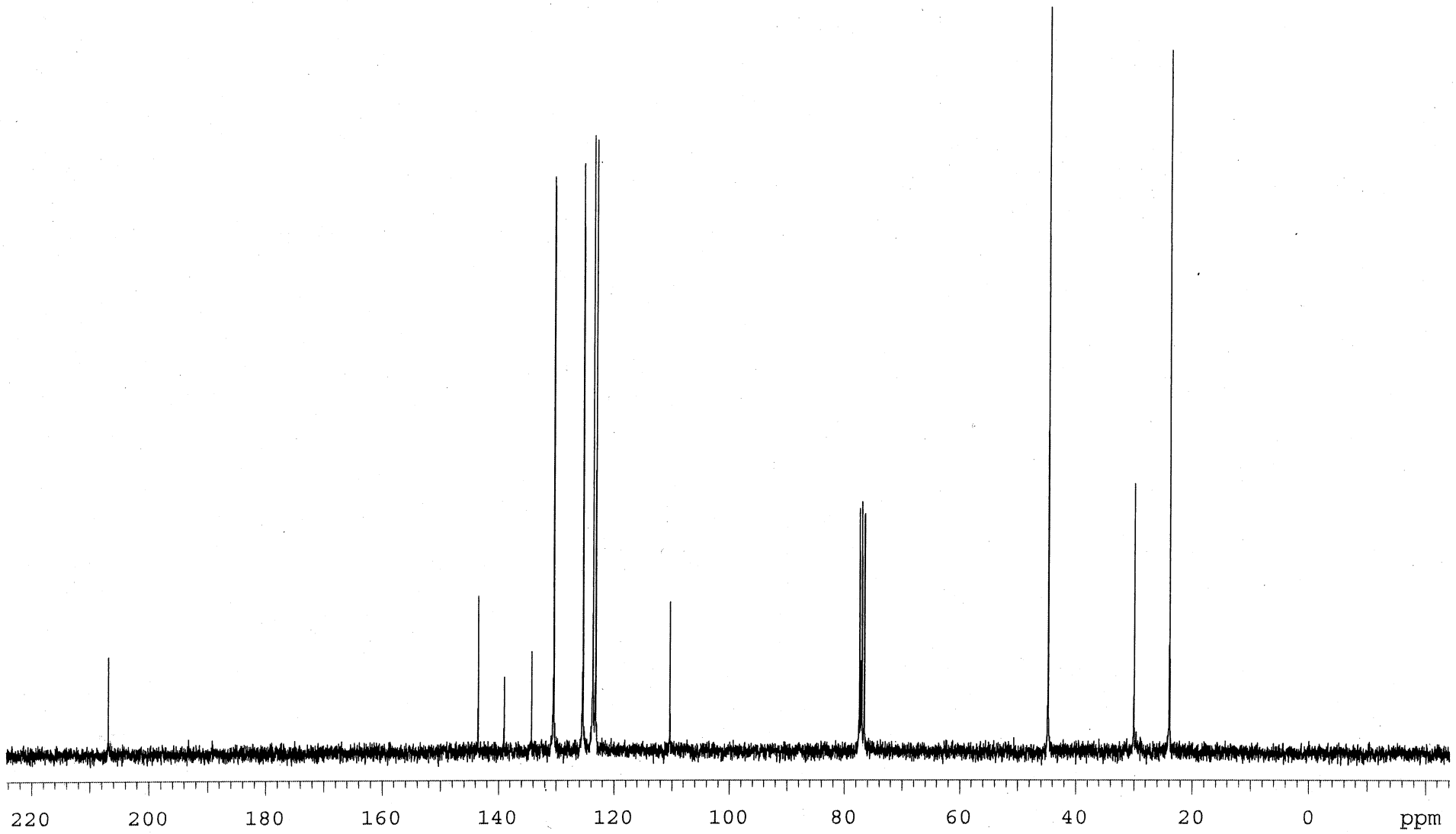


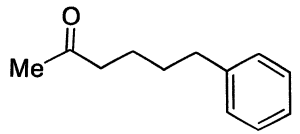
MF F-16 PTLC1 13C

Pulse Sequence: s2pul



1m



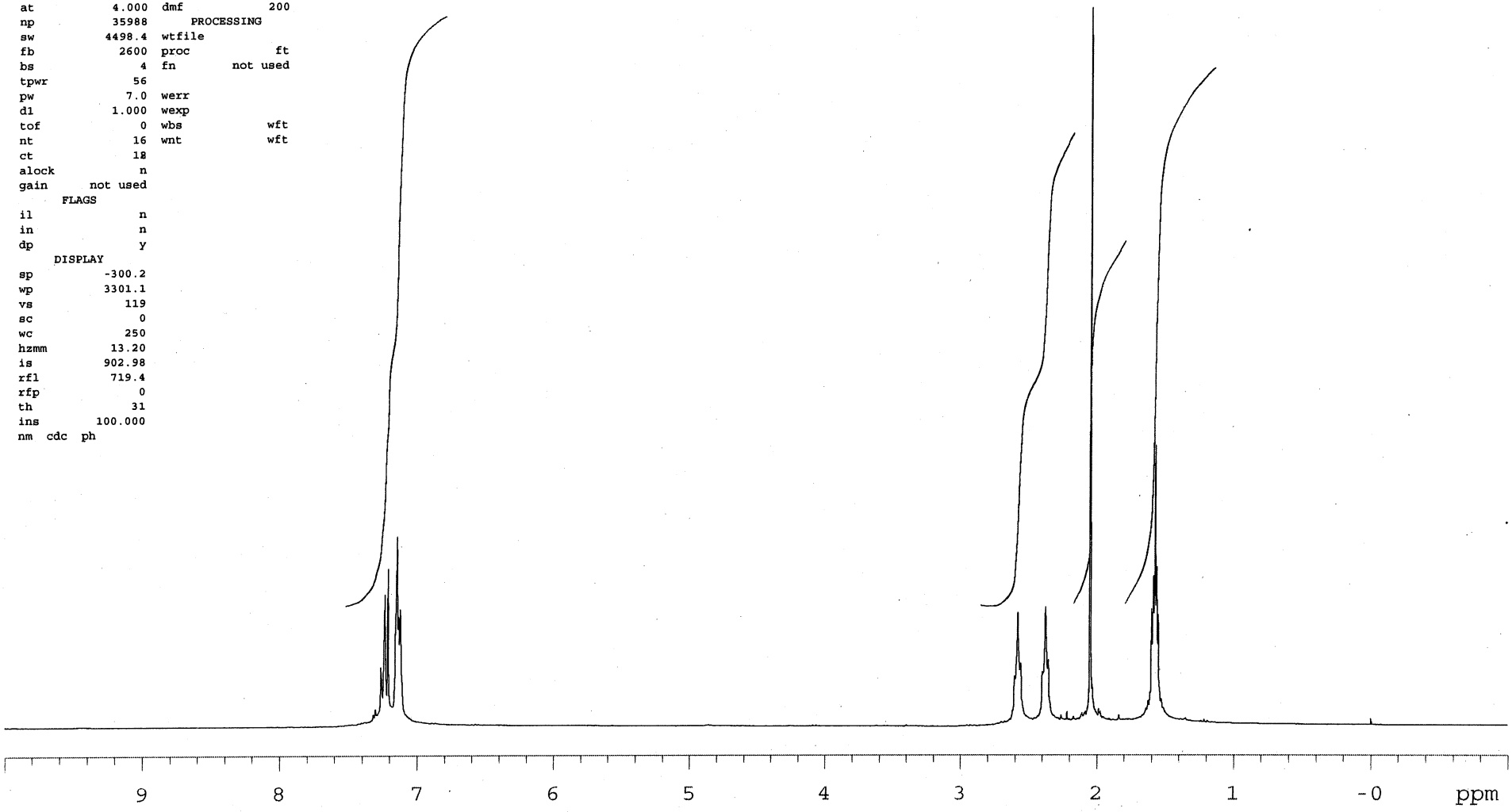


1n

MF A-49

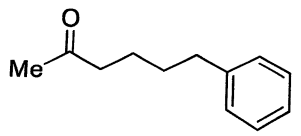
exp2 stdlh

SAMPLE		DEC. & VT	
date	May 16 2017	dfrq	300.108
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.108	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	18		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	119		
sc	0		
wc	250		
hzmm	13.20		
is	902.98		
rfl	719.4		
rfp	0		
th	31		
ins	100.000		
nm cdc ph			

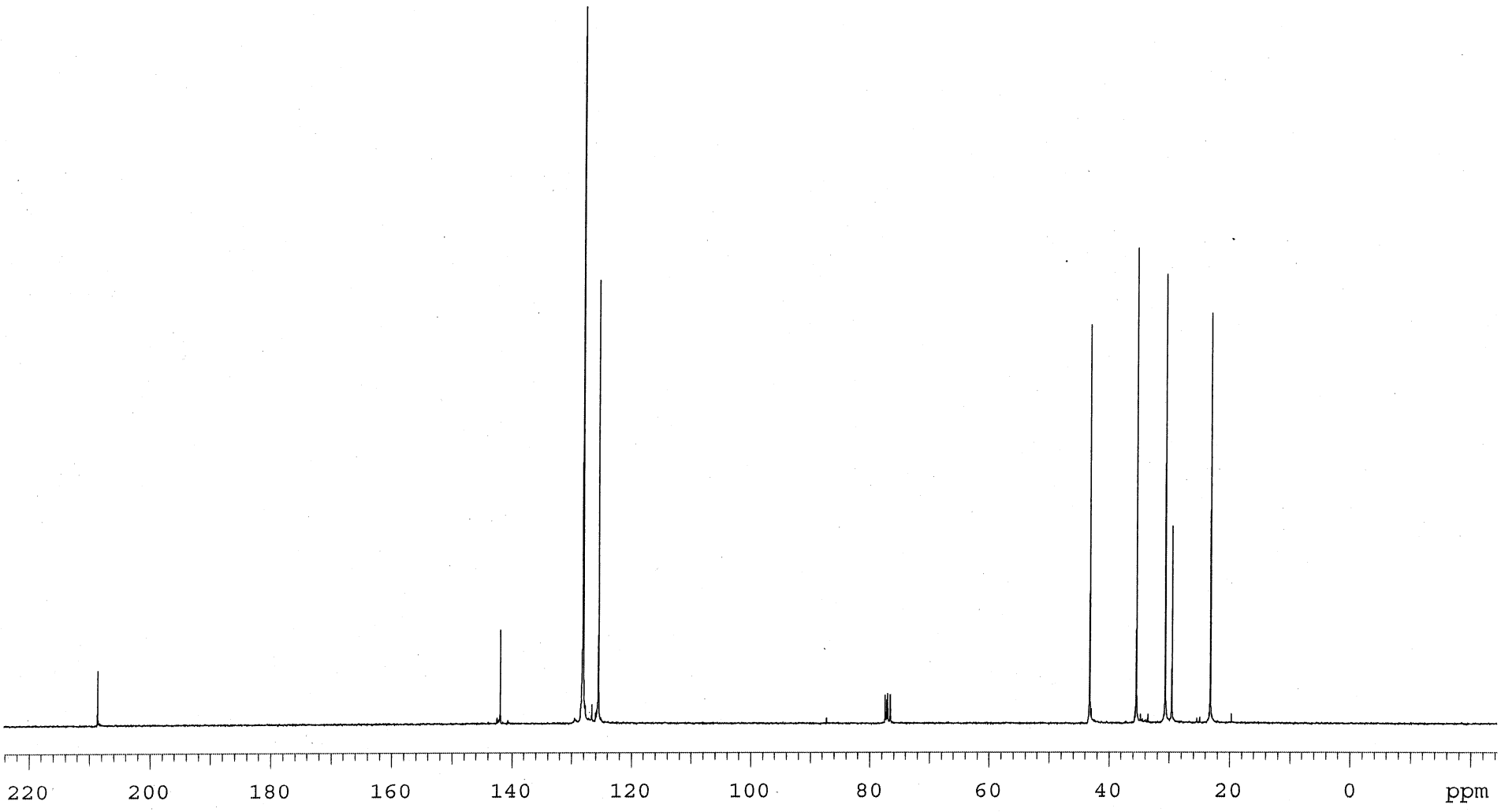


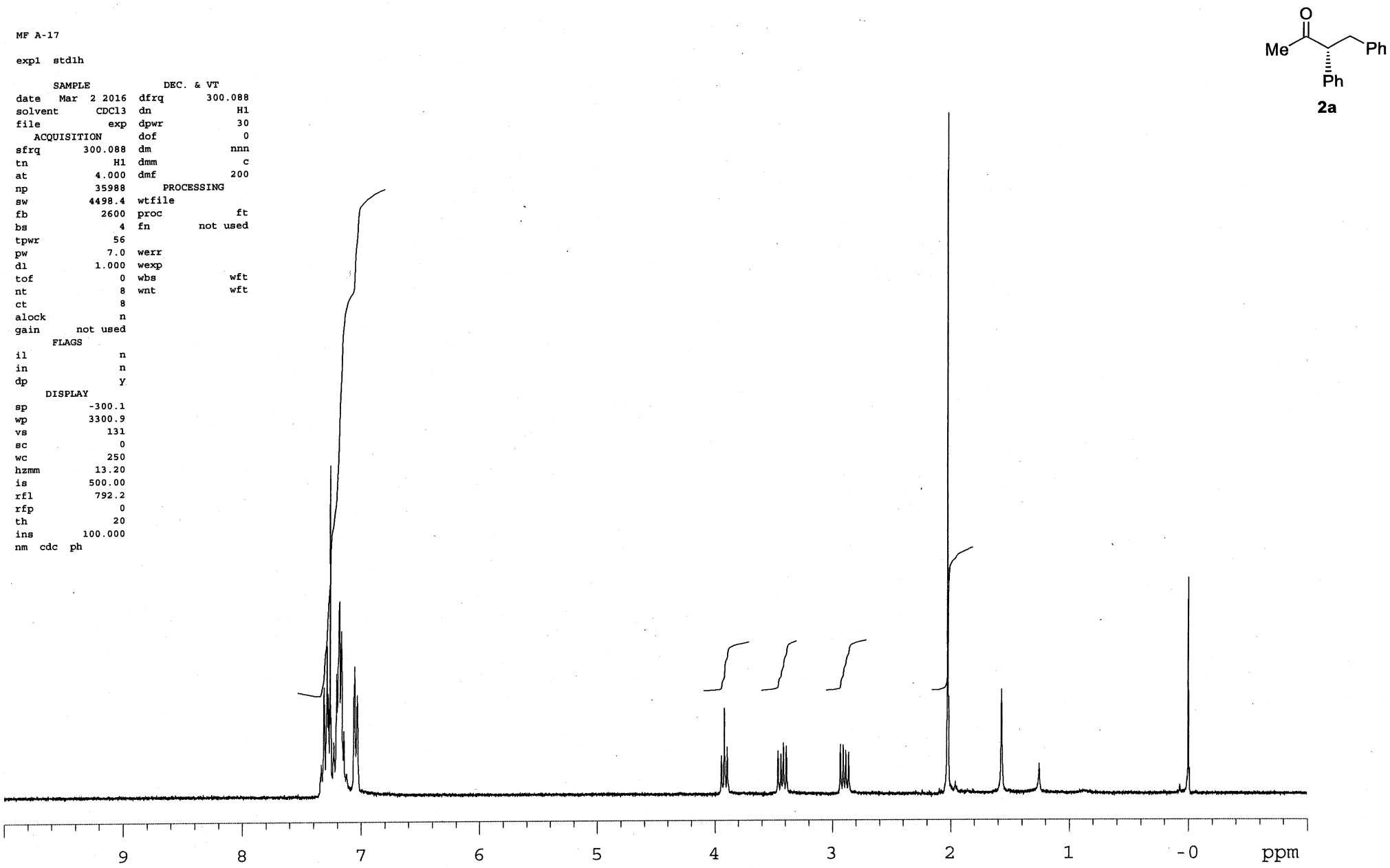
MF A-49 13C

Pulse Sequence: s2pul



1n




MF A-17
exp1 std1h
SAMPLE
date Mar 2 2016
solvent CDCl3
file exp
ACQUISITION
sfrq 300.088
tn H1
at 4.000
np 35988
sw 4498.4
fb 2600
bs 4
tpwr 56
pw 7.0
d1 1.000
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -300.1
wp 3300.9
vs 131
sc 0
wc 250
hzmm 13.20
is 500.00
rfl 792.2
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.088
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs wft
wnt wft
9
8
7
6
5
4
3
2
1
-0
ppm

O
Me
Ph
Ph

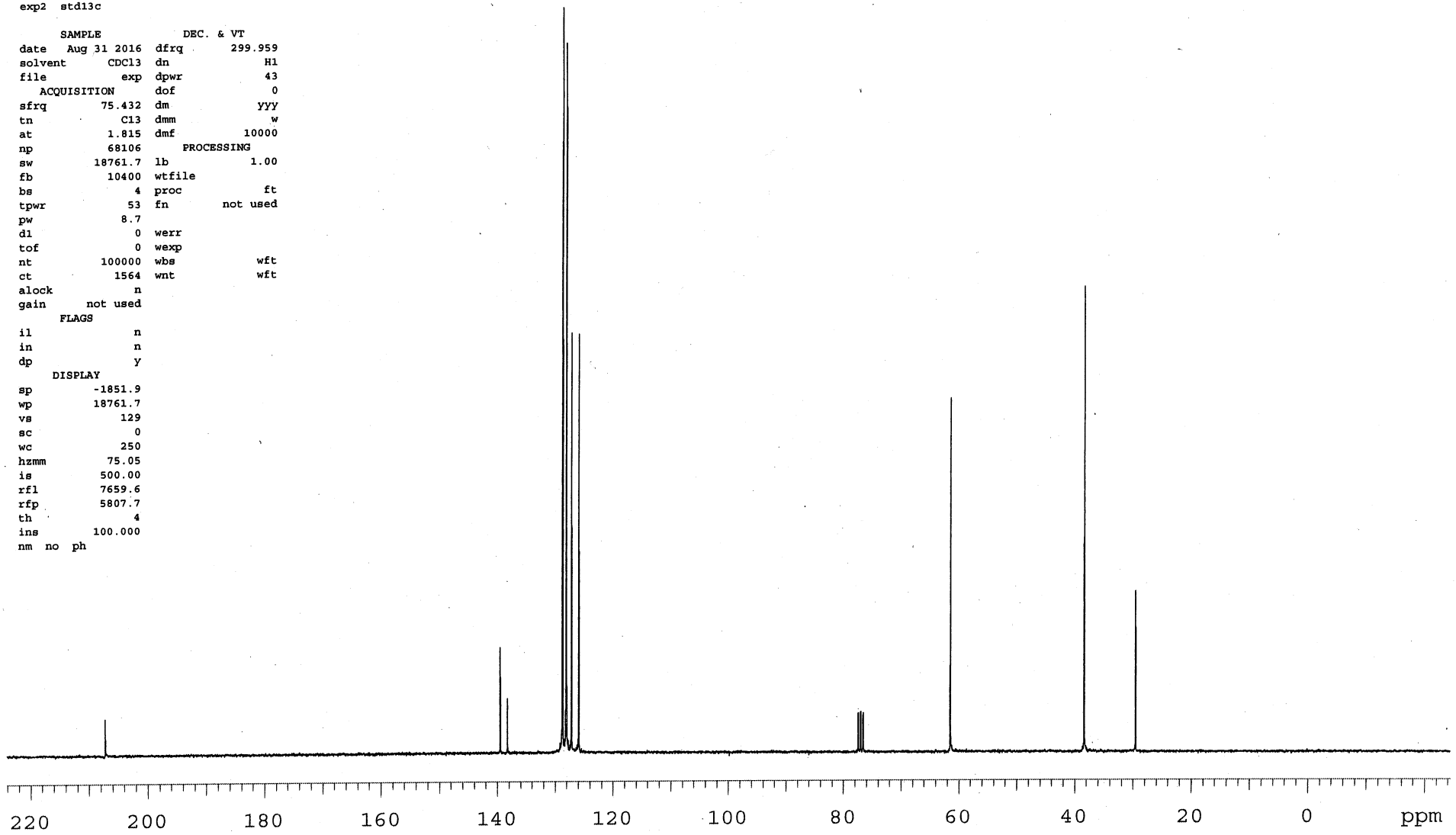
2a

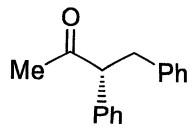
MF C-21 FF1 13C

exp2 std13c

SAMPLE		DEC. & VT	
date	Aug 31 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.432	dm	yyy
tn	C13	dmm	w
at	1.815	dmf	10000
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
dl	0	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	1564	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1851.9		
wp	18761.7		
vs	129		
sc	0		
wc	250		
hzmm	75.05		
is	500.00		
rfl	7659.6		
rfp	5807.7		
th	4		
ins	100.000		

nm no ph





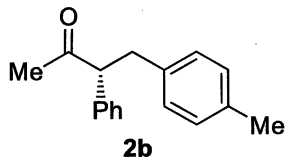
2a

2b 1H

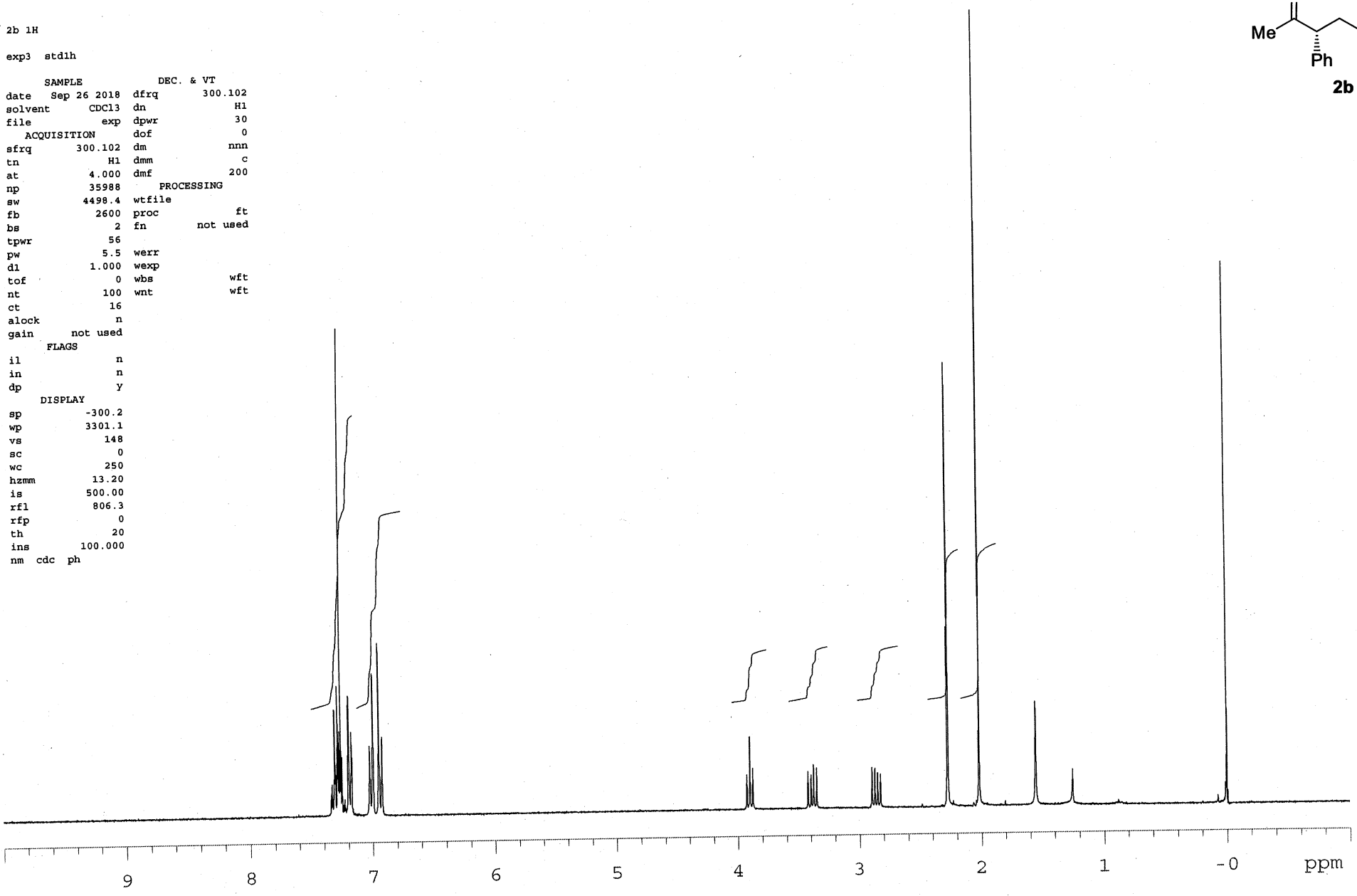
exp3 std1h

SAMPLE		DEC. & VT	
date	Sep 26 2018	dfrq	300.102
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.102	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	2	fn	not used
tpwr	56		
pw	5.5	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	100	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	148		
sc	0		
wc	250		
hzmm	13.20		
is	500.00		
rfl	806.3		
rfp	0		
th	20		
ins	100.000		

nm cdc ph

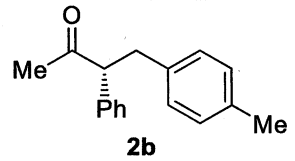


2b

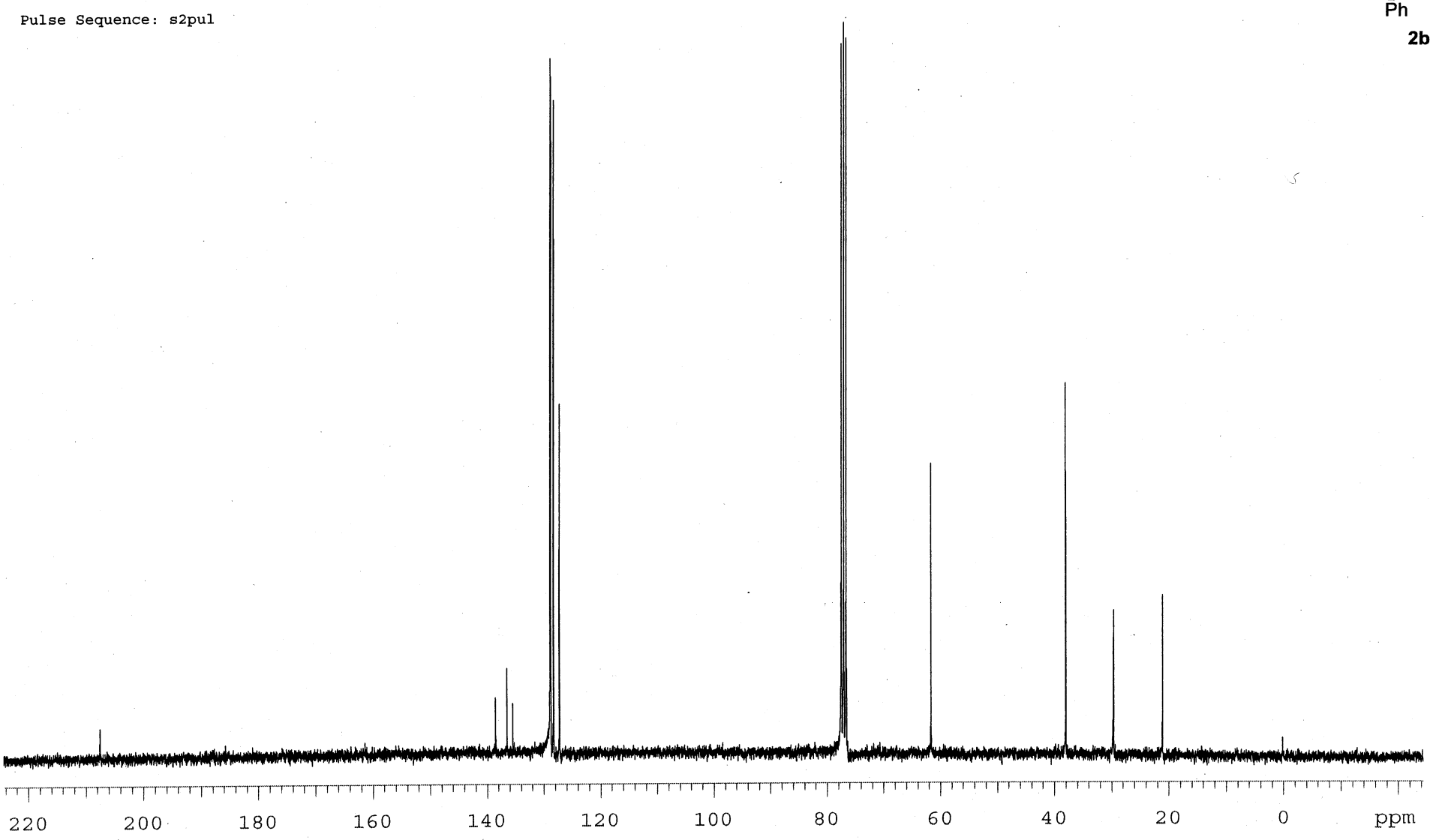


TN A-40 13C

Pulse Sequence: s2pul



2b

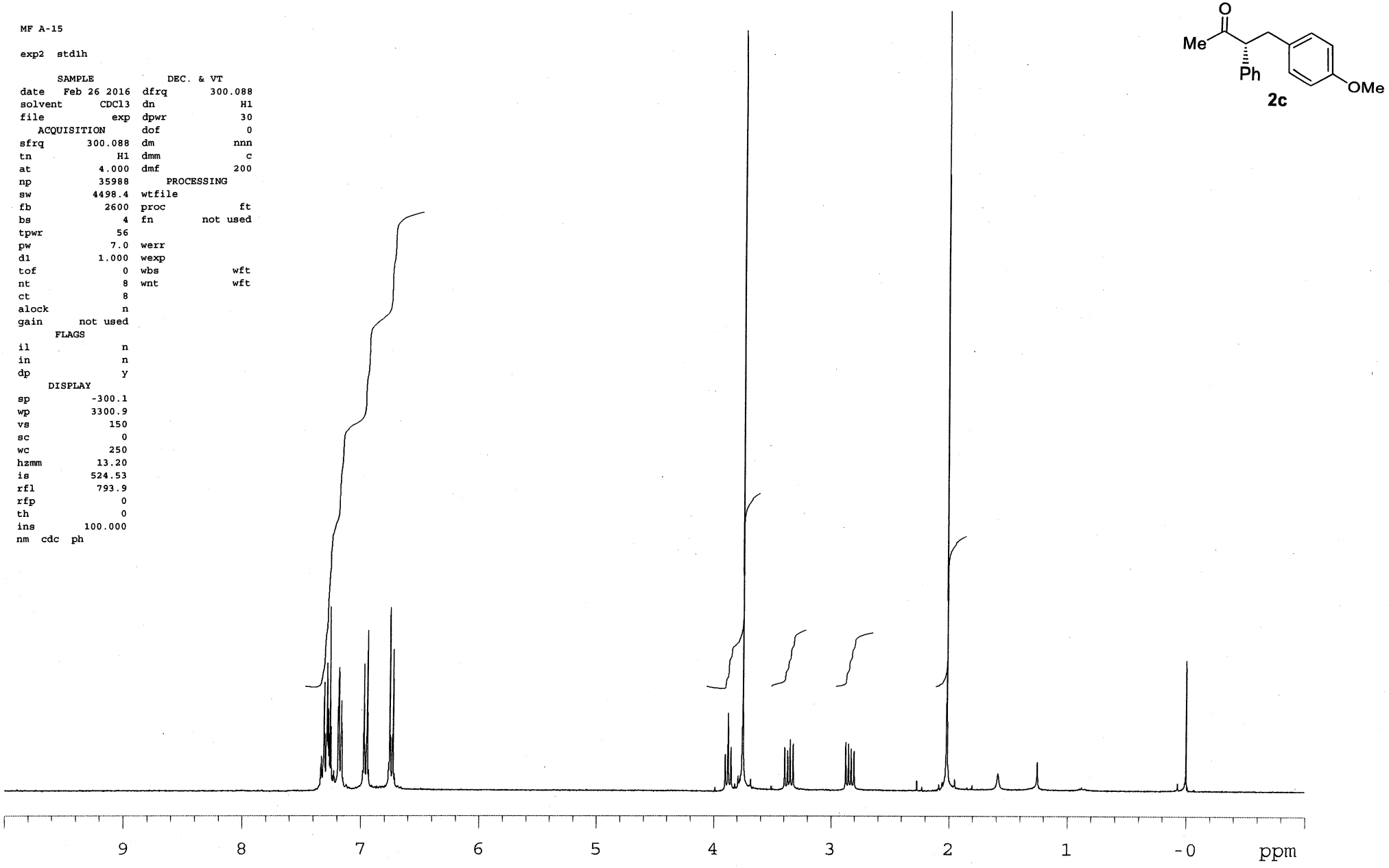


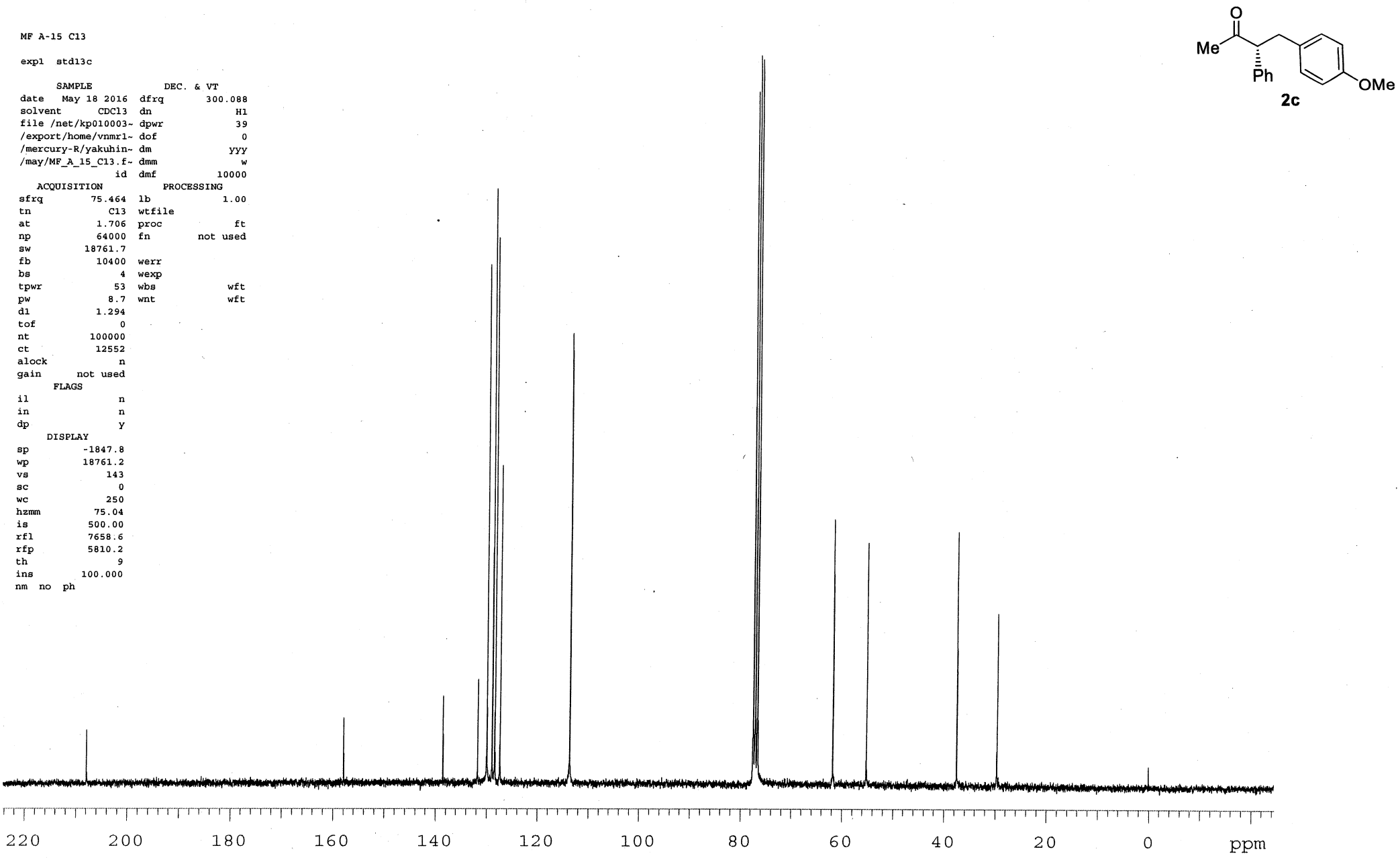
MF A-15

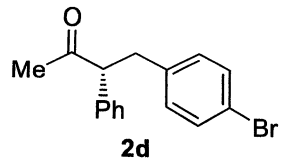
exp2 stdlh

SAMPLE		DEC. & VT	
date	Feb 26 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	150		
sc	0		
wc	250		
hzmm	13.20		
is	524.53		
rfl	793.9		
rfp	0		
th	0		
ins	100.000		

nm cdc ph




MF A-15 C13
exp1 std13c
SAMPLE
date May 18 2016
solvent CDCl3
file /net/kp010003~
/export/home/vnmr1~
/mercury-R/yakuhin~
/may/MF_A_15_C13.f~
id
ACQUISITION
sfrq 75.464
tn C13
at 1.706
np 64000
sw 18761.7
fb 10400
bs 4
tpwr 53
pw 8.7
d1 1.294
tof 0
nt 100000
ct 12552
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1847.8
wp 18761.2
vs 143
sc 0
wc 250
hzmm 75.04
is 500.00
rfl 7658.6
rfp 5810.2
th 9
ins 100.000
nm no ph
DEC. & VT
dfrq 300.088
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 10000
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs wft
wnt wft
Me
O
Ph
OMe
2c
220
200
180
160
140
120
100
80
60
40
20
0
ppm



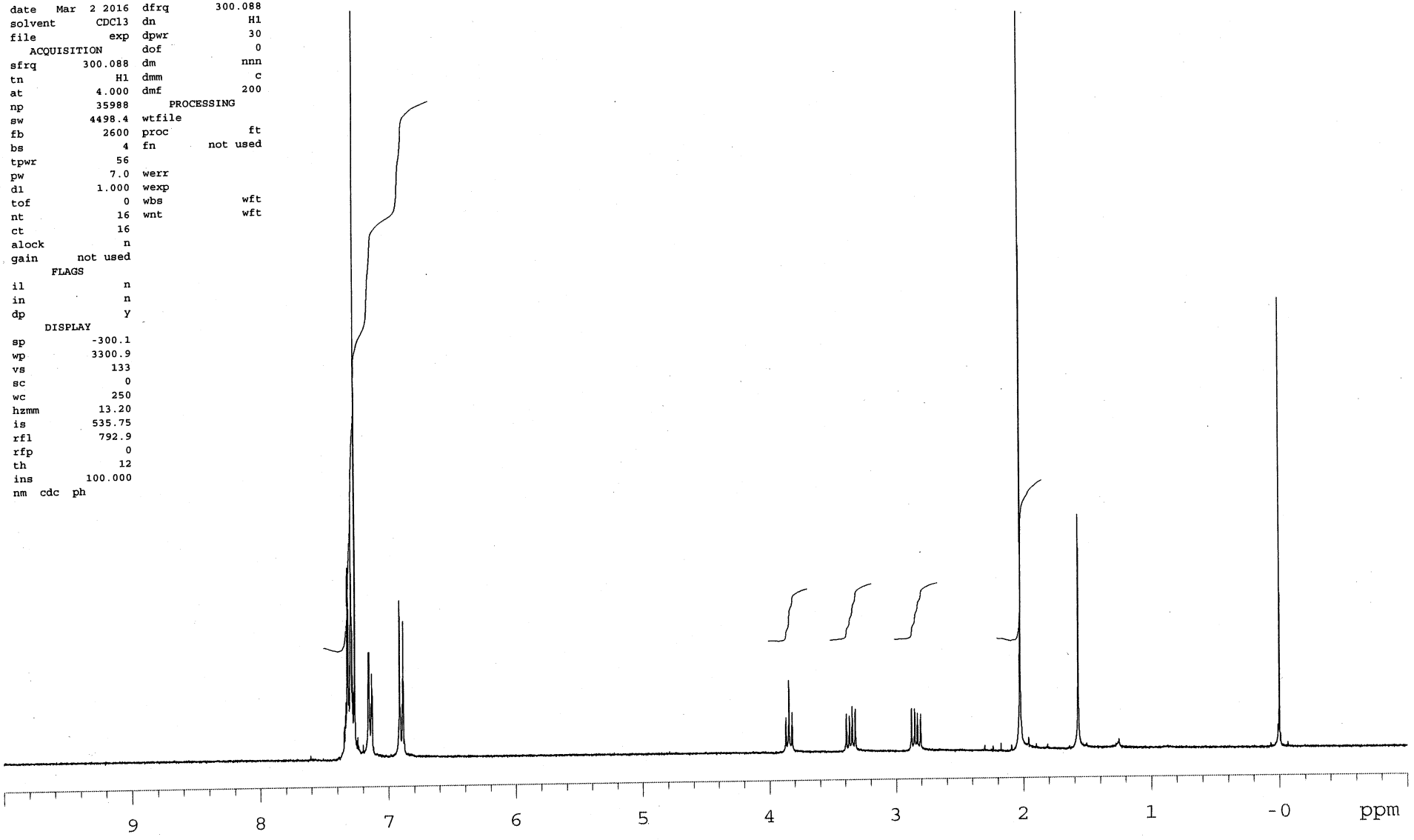
2d

KO A-18 PTLC1

exp1 std1h

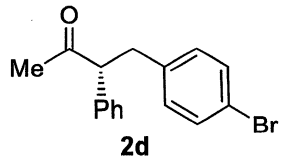
SAMPLE		DEC. & VT	
date	Mar 2 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	133		
sc	0		
wc	250		
hzmm	13.20		
is	535.75		
rfl	792.9		
rfp	0		
th	12		
ins	100.000		

nm cdc ph

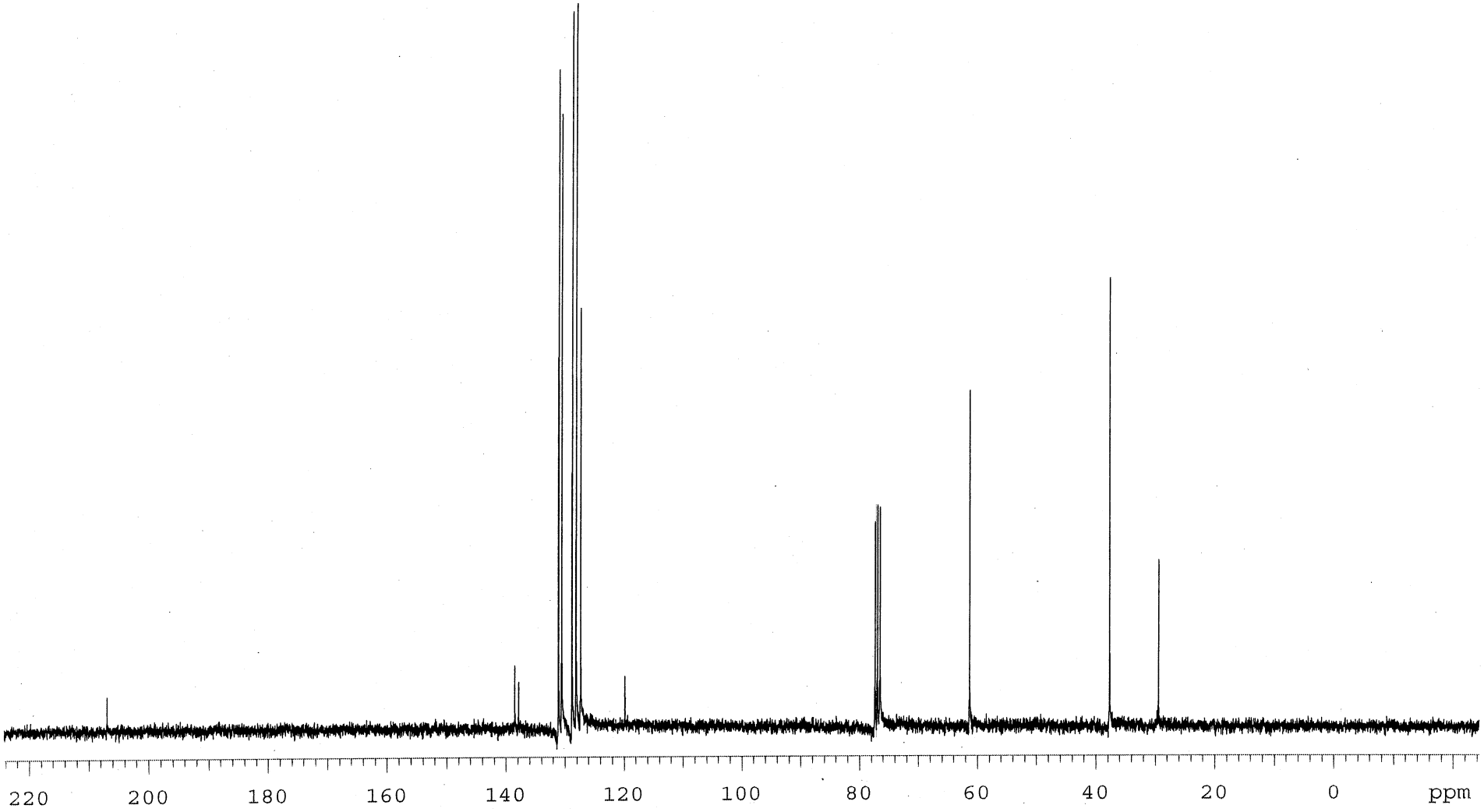


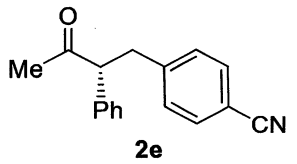
KO-A-18 PTLC1

Pulse Sequence: s2pul



2d



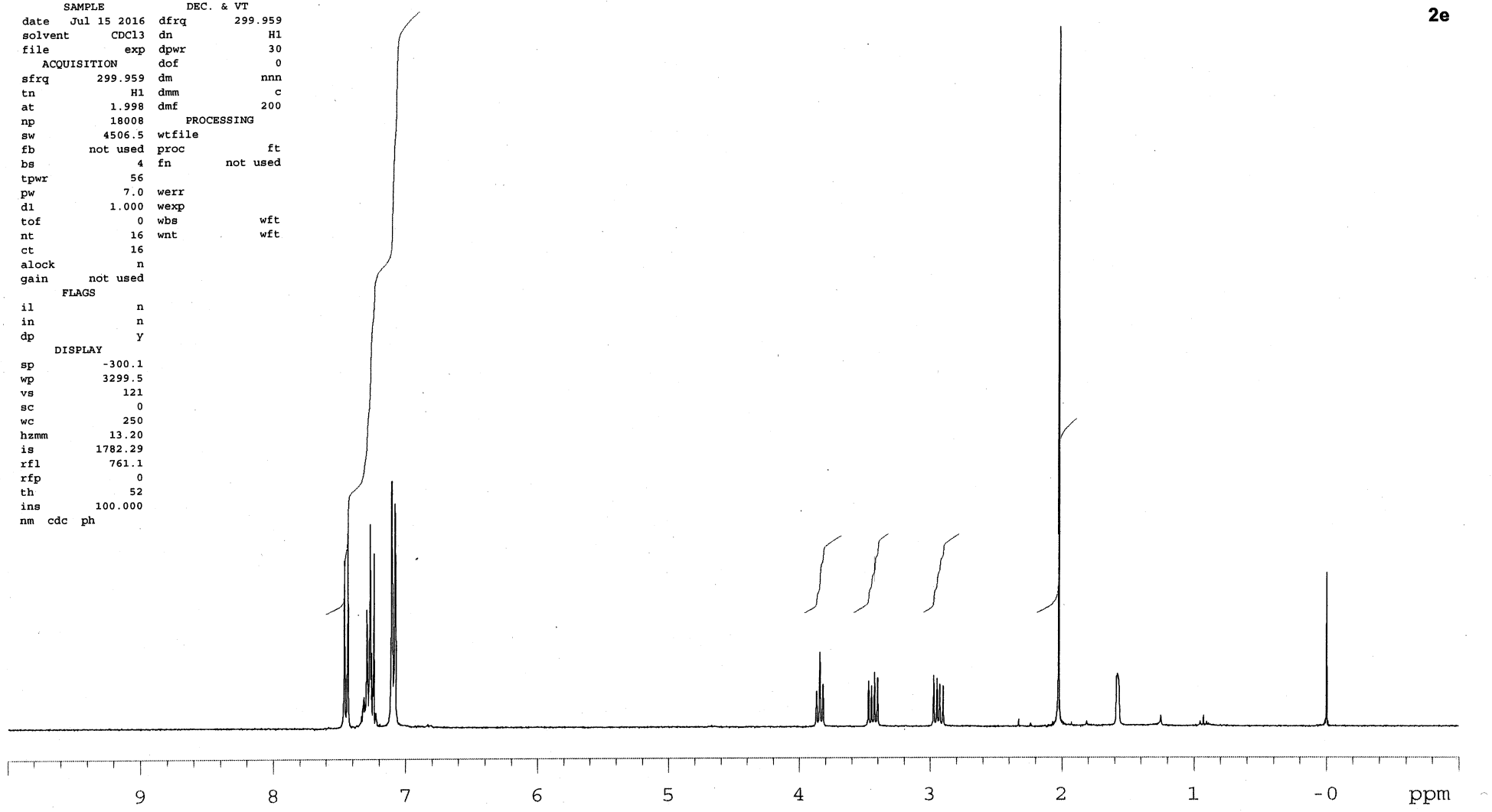


2e

MF D-59 PTLC2

exp1 std1h

SAMPLE		DEC. & VT	
date	Jul 15 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.959	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3299.5		
vs	121		
sc	0		
wc	250		
hzmm	13.20		
is	1782.29		
rfl	761.1		
rfp	0		
th	52		
ins	100.000		
nm cdc ph			

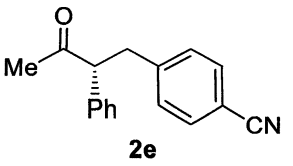


MF-D-59 PTLC2 13C

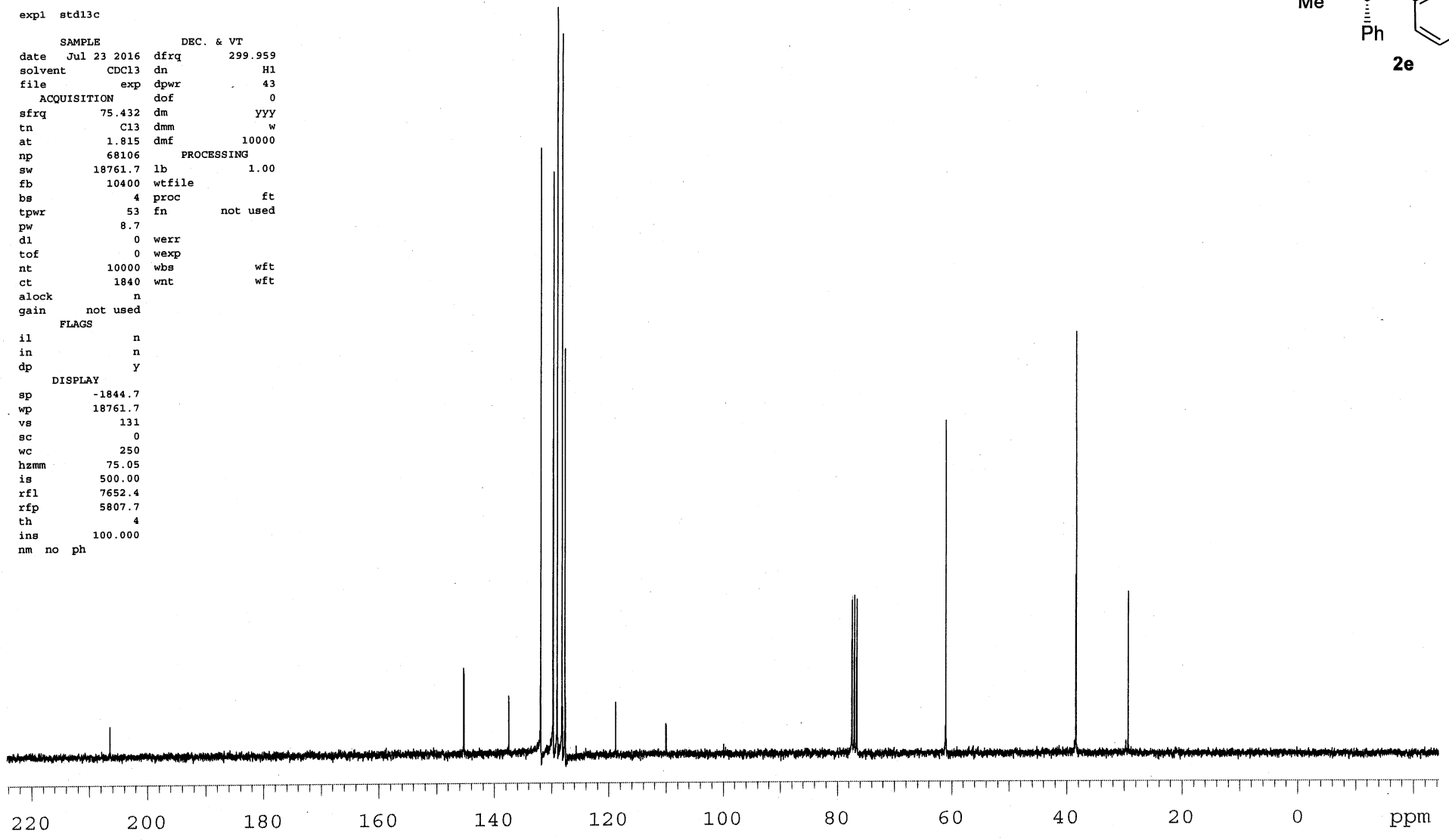
exp1 std13c

SAMPLE		DEC. & VT	
date	Jul 23 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.432	dm	yyy
tn	C13	dmm	w
at	1.815	dmf	10000
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	0	werr	
tof	0	wexp	
nt	10000	wbs	wft
ct	1840	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1844.7		
wp	18761.7		
vs	131		
sc	0		
wc	250		
hzmm	75.05		
is	500.00		
rfl	7652.4		
rfp	5807.7		
th	4		
ins	100.000		

nm no ph



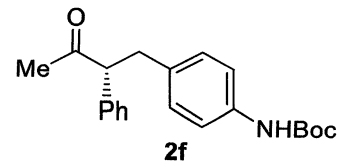
2e



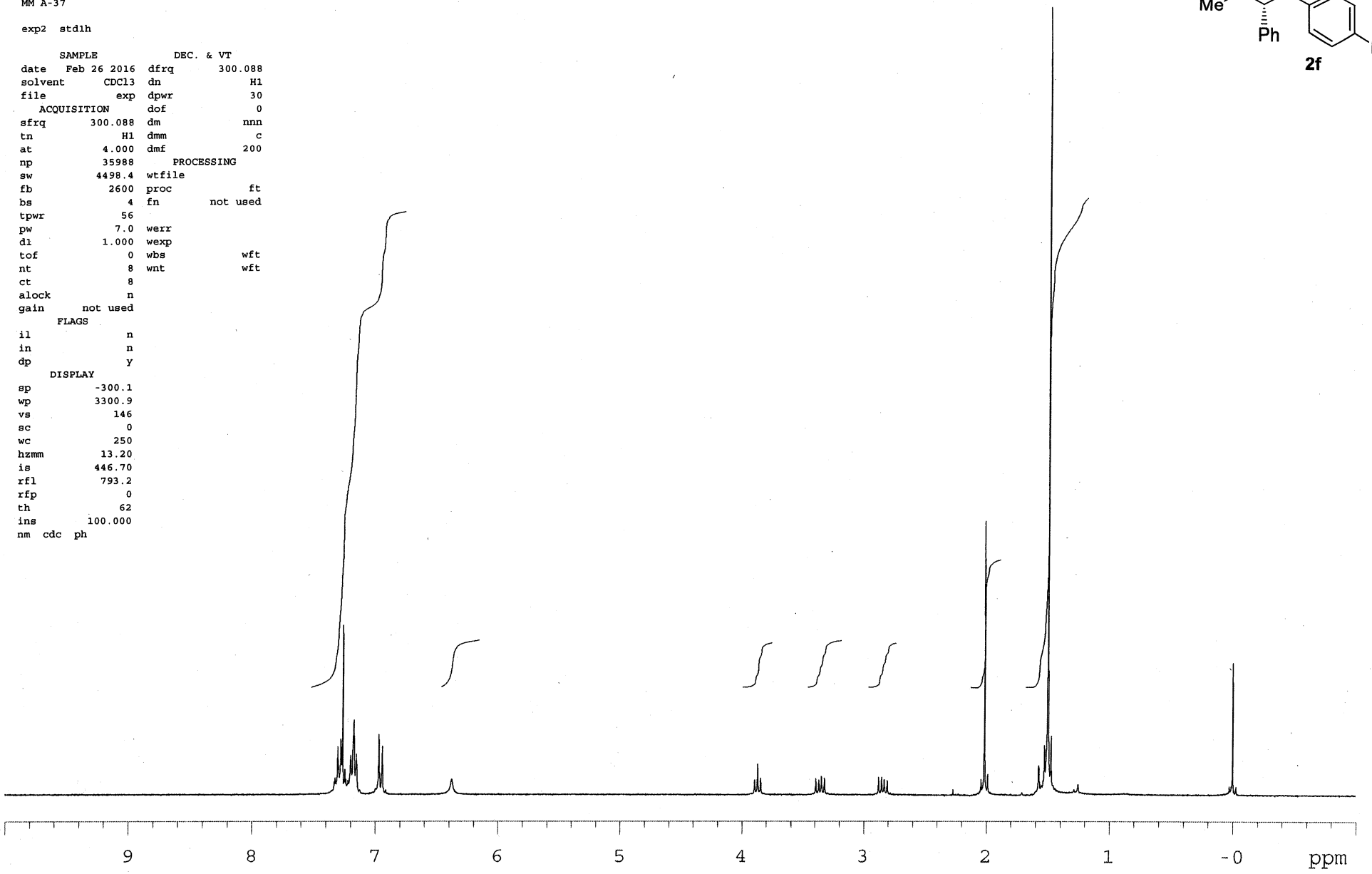
MM A-37

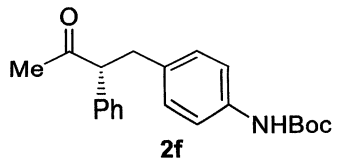
exp2 stdlh

SAMPLE		DEC. & VT	
date	Feb 26 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	146		
sc	0		
wc	250		
hzmm	13.20		
is	446.70		
rfl	793.2		
rfp	0		
th	62		
ins	100.000		
nm cdc ph			



2f





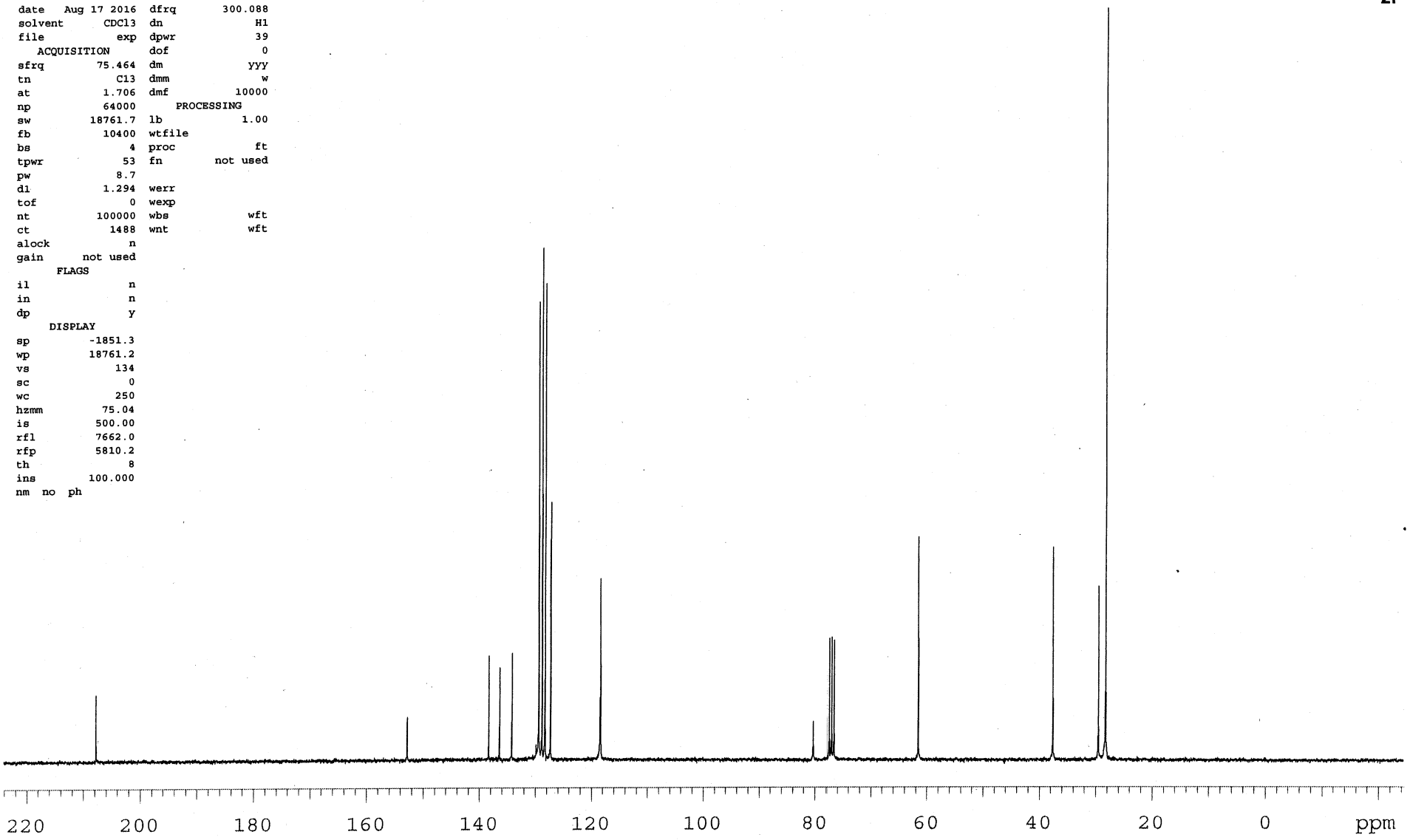
2f

MM A-37 13C

exp2 std13c

SAMPLE		DEC. & VT	
date	Aug 17 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	75.464	dm	yyy
tn	C13	dmm	w
at	1.706	dmf	10000
np	64000	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	1.294	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	1488	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1851.3		
wp	18761.2		
vs	134		
sc	0		
wc	250		
hzmm	75.04		
is	500.00		
rfl	7662.0		
rfp	5810.2		
th	8		
ins	100.000		

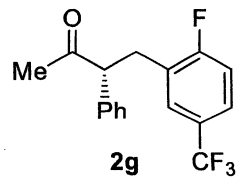
nm no ph



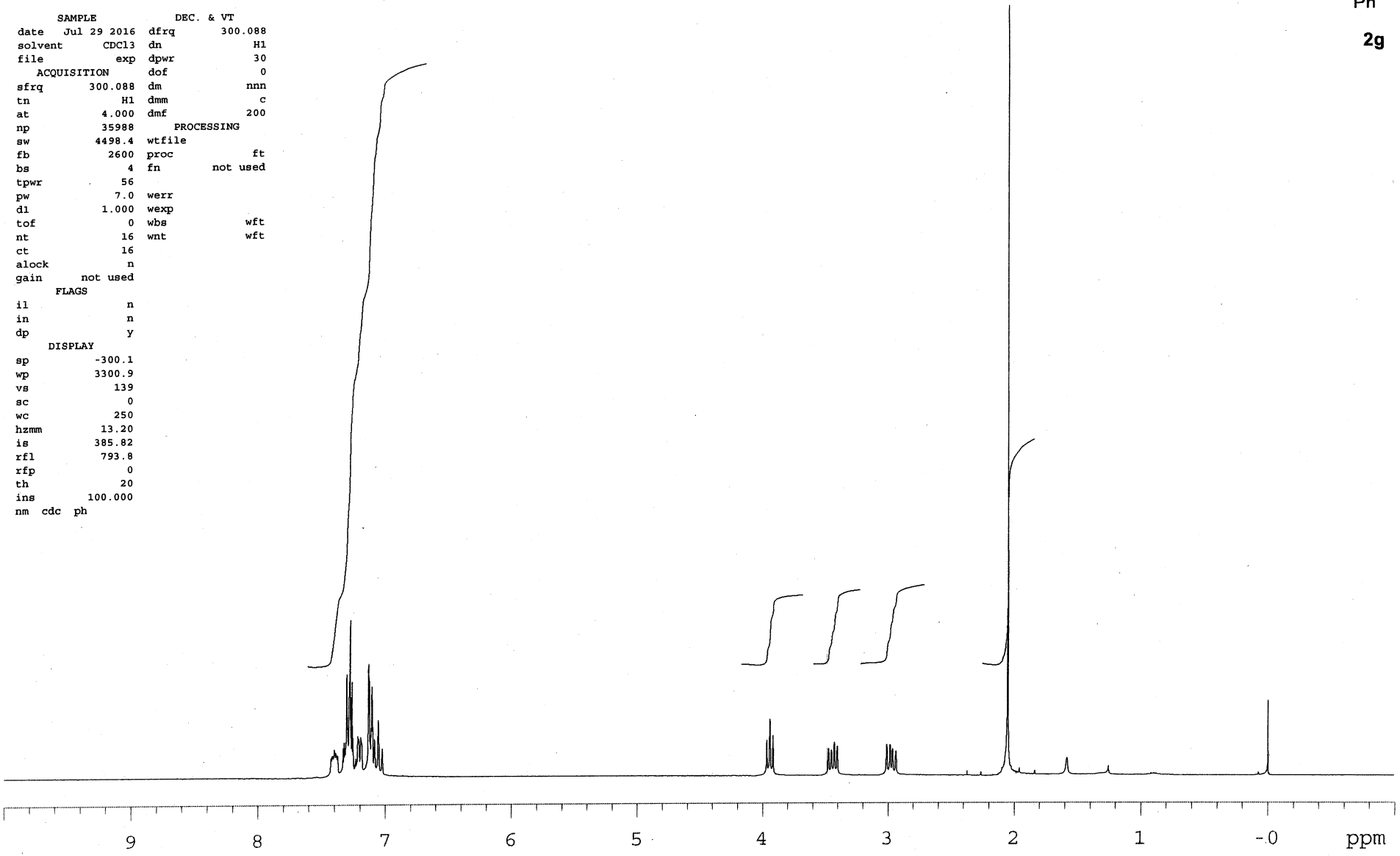
MF E-15 PTLC1
19

exp2 std1h

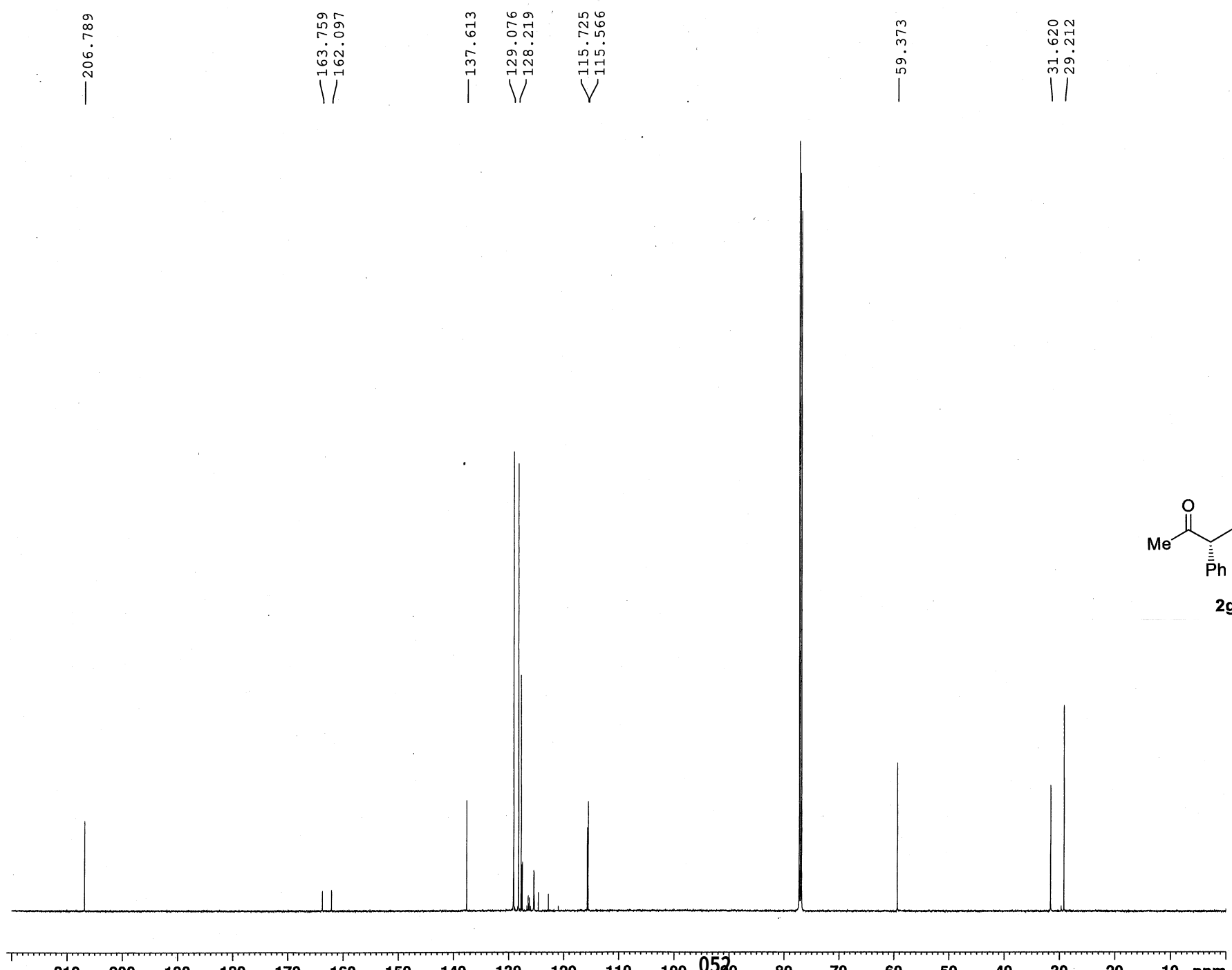
SAMPLE		DEC. & VT	
date	Jul 29 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	139		
sc	0		
wc	250		
hzmm	13.20		
is	385.82		
rfl	793.8		
rfp	0		
th	20		
ins	100.000		
nm	cdc ph		



2g

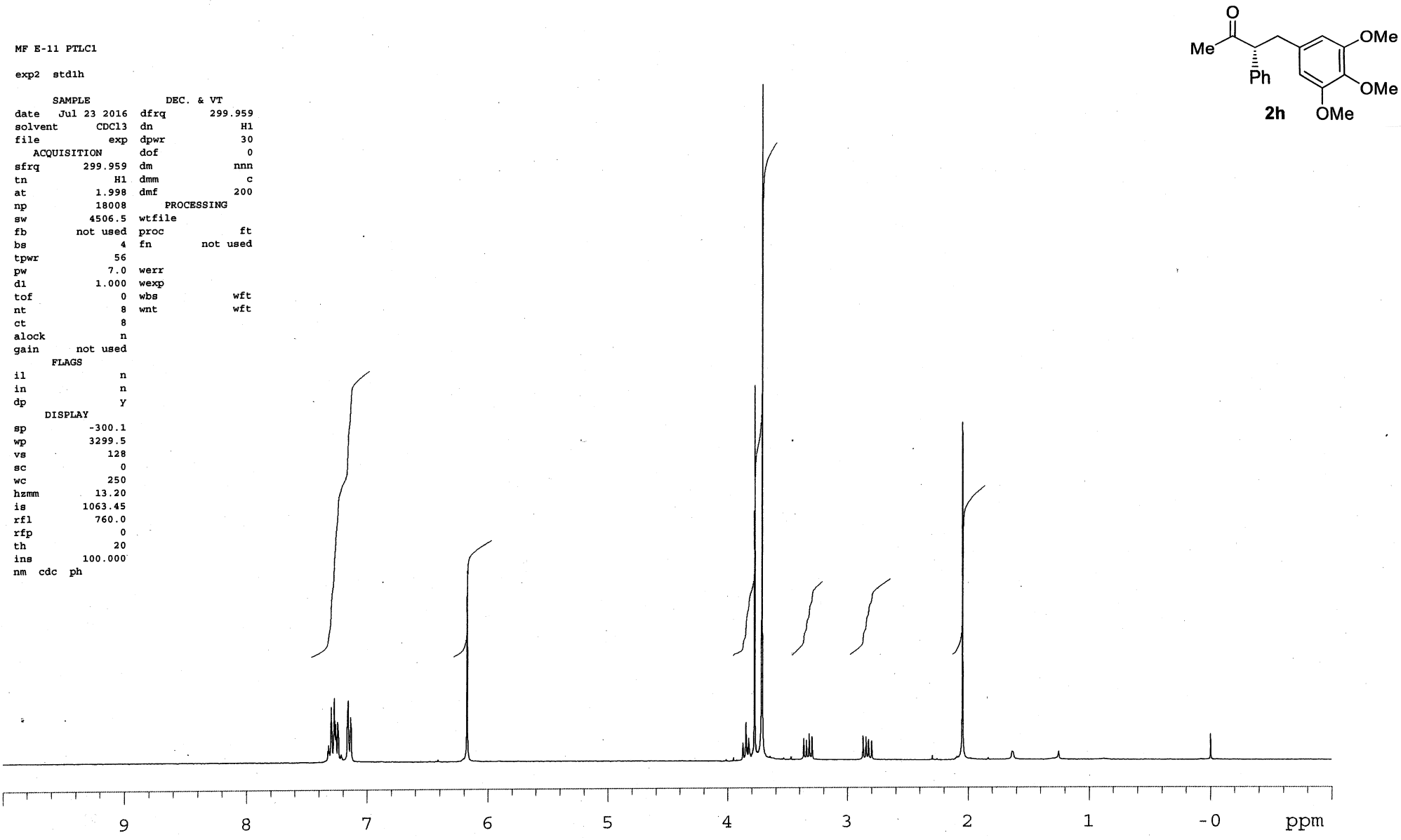


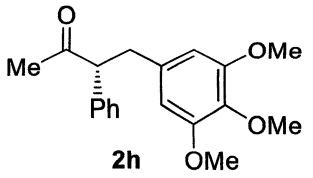
KPNN-5613 C-13




```
Current Data Parameters
NAME                  KPNN-5613
EXPNO                        20
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20170803
Time                      14.22 h
INSTRUM                   spect
PROBHD    Z114607_0202 (
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        3208
DS                           4
SWH                 36231.883 Hz
FIDRES               1.105709 Hz
AQ                   0.9043968 sec
RG                      194.13
DW                      13.800 usec
DE                       6.50 usec
TE                      296.4 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         300
SFO1               151.0184889 MHz
NUC1                        13C
P1                      12.00 usec
PLW1                80.00000000 W
SFO2                600.5336032 MHz
NUC2                        1H
CPDPRG[2                waltz65
PCPD2                   80.00 usec
PLW2                24.00600052 W
PLW12                0.37509000 W
PLW13                0.16671000 W

F2 - Processing parameters
SI                        32768
SF                 151.0033883 MHz
WDW                          EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```


2g


MF E-11 PTLC1
exp2 std1h
SAMPLE
date Jul 23 2016
solvent CDCl3
file exp
ACQUISITION
sfrq 299.959
tn H1
at 1.998
np 18008
sw 4506.5
fb not used
bs 4
tpwr 56
pw 7.0
d1 1.000
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -300.1
wp 3299.5
vs 128
sc 0
wc 250
hzmm 13.20
is 1063.45
rfl 760.0
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 299.959
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs wft
wnt wft
O
Me
Ph
OMe
OMe
OMe
2h
9
8
7
6
5
4
3
2
1
-0
ppm



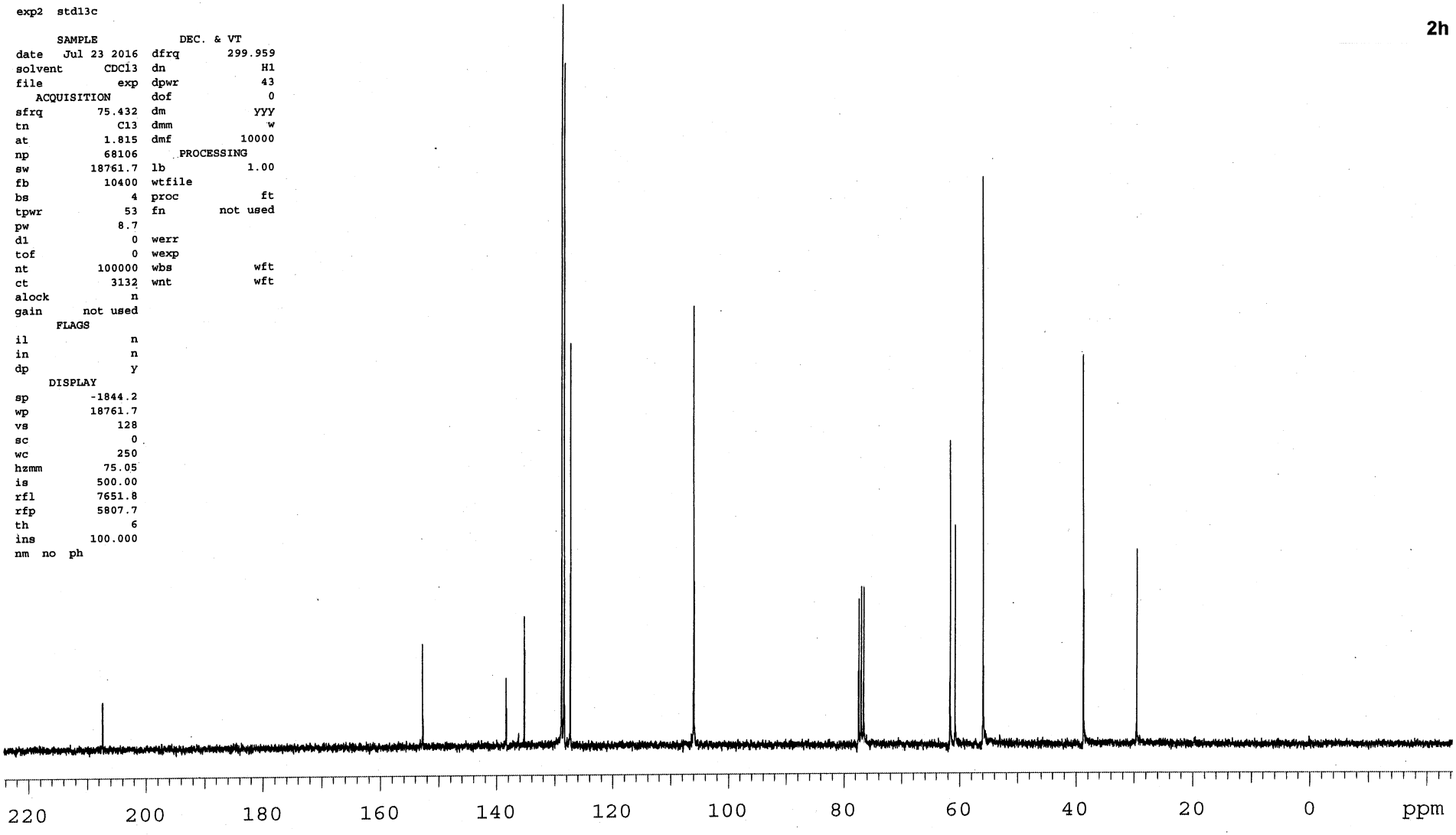
2h

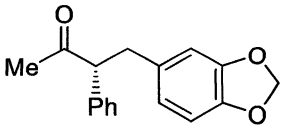
13C OBSERVE

exp2 std13c

SAMPLE		DEC. & VT	
date	Jul 23 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.432	dm	yyy
tn	C13	dmm	w
at	1.815	dmf	10000
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	0	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	3132	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1844.2		
wp	18761.7		
vs	128		
sc	0		
wc	250		
hzmm	75.05		
is	500.00		
rfl	7651.8		
rfp	5807.7		
th	6		
ins	100.000		

nm no ph



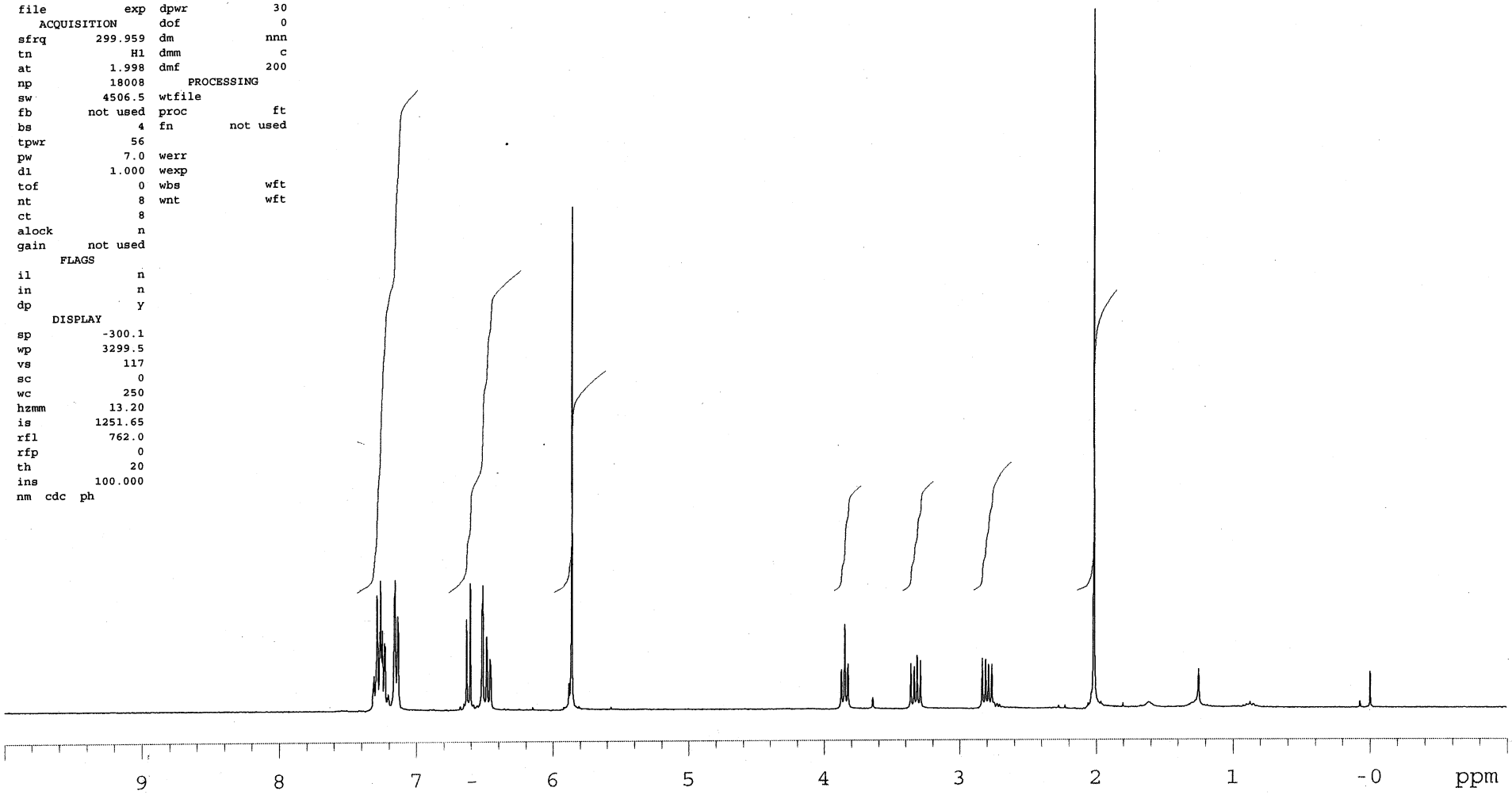


2i

MF E-58 PTLC1

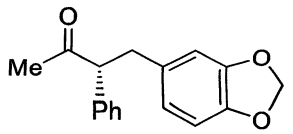
exp2 std1h

SAMPLE		DEC. & VT	
date	Dec 14 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.959	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3299.5		
vs	117		
sc	0		
wc	250		
hzmm	13.20		
is	1251.65		
rfl	762.0		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			

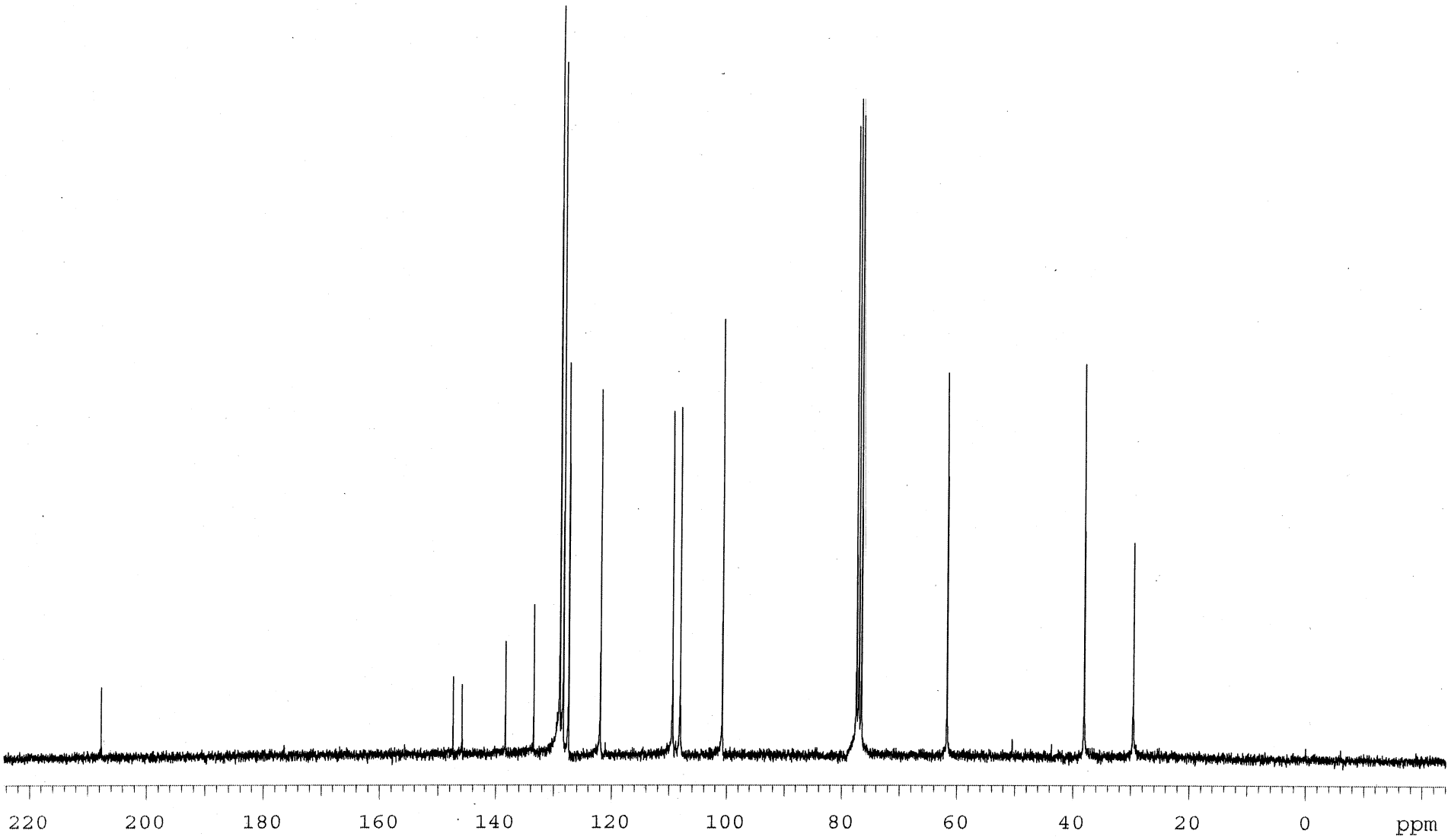


MF E-58 PTLC1

Pulse Sequence: s2pul



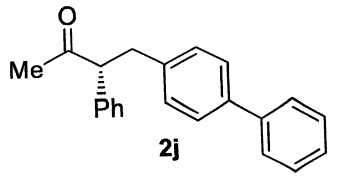
2i



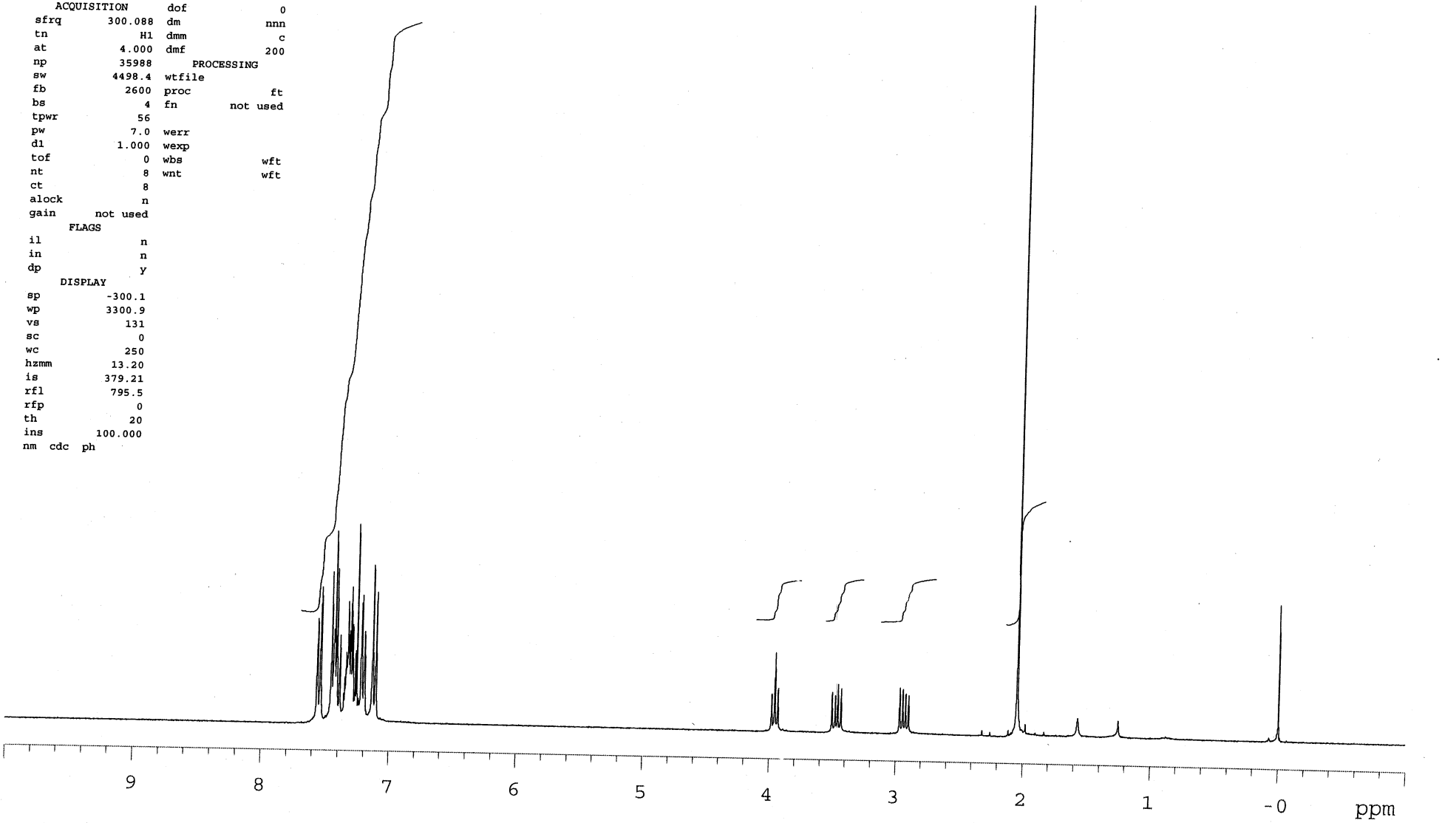
MF A-36 PTLC1

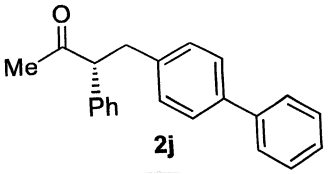
exp2 std1h

SAMPLE		DEC. & VT	
date	Feb 26 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	131		
sc	0		
wc	250		
hzmm	13.20		
is	379.21		
rfl	795.5		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			



2j





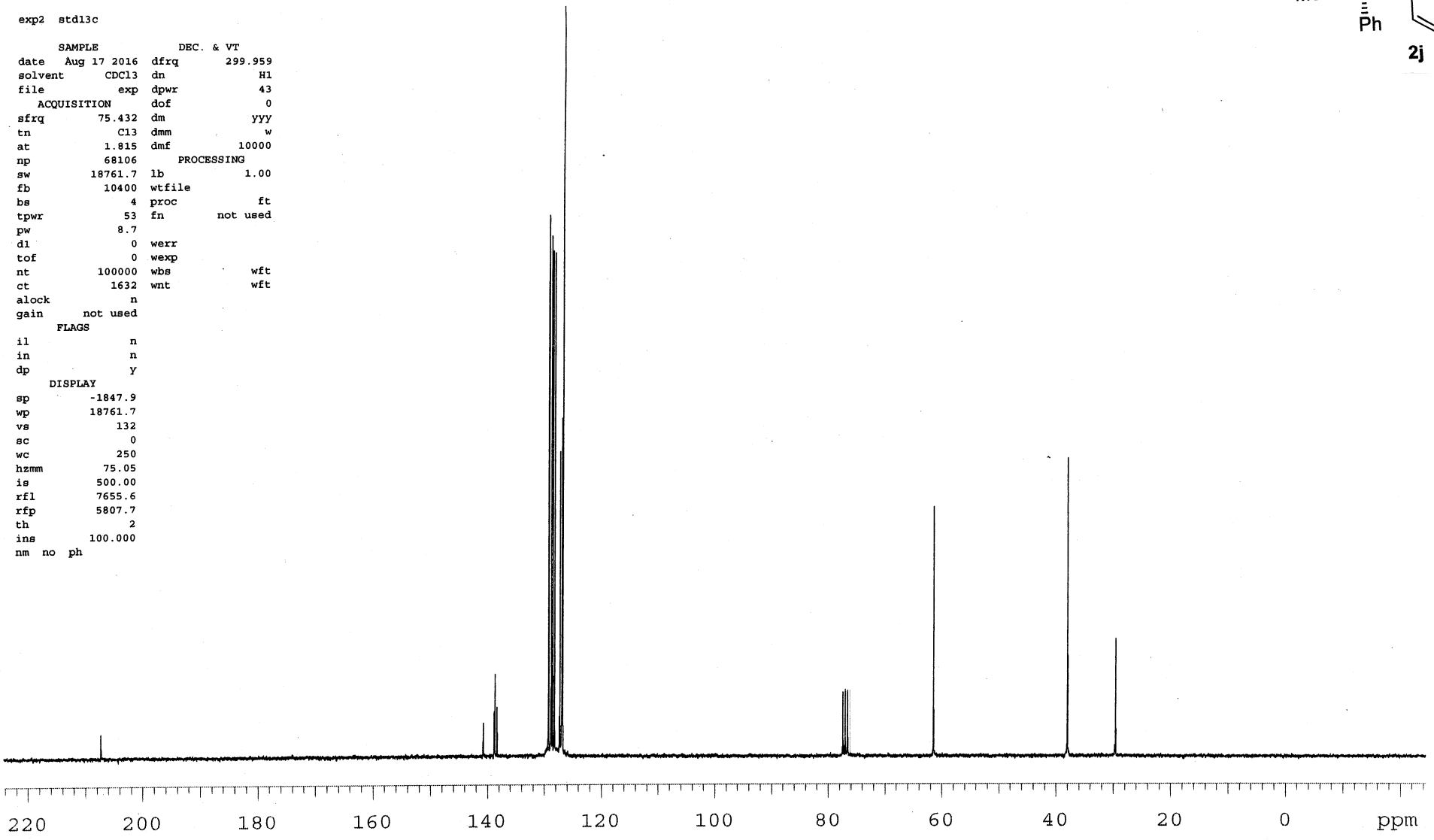
2j

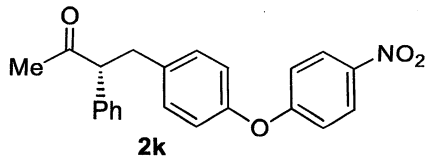
MF A-36 13C

exp2 std13c

SAMPLE		DEC. & VT	
date	Aug 17 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.432	dm	yyy
tn	C13	dmm	w
at	1.815	dmf	10000
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	0	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	1632	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1847.9		
wp	18761.7		
vs	132		
sc	0		
wc	250		
hzmm	75.05		
is	500.00		
rfl	7655.6		
rfp	5807.7		
th	2		
ins	100.000		

nm no ph



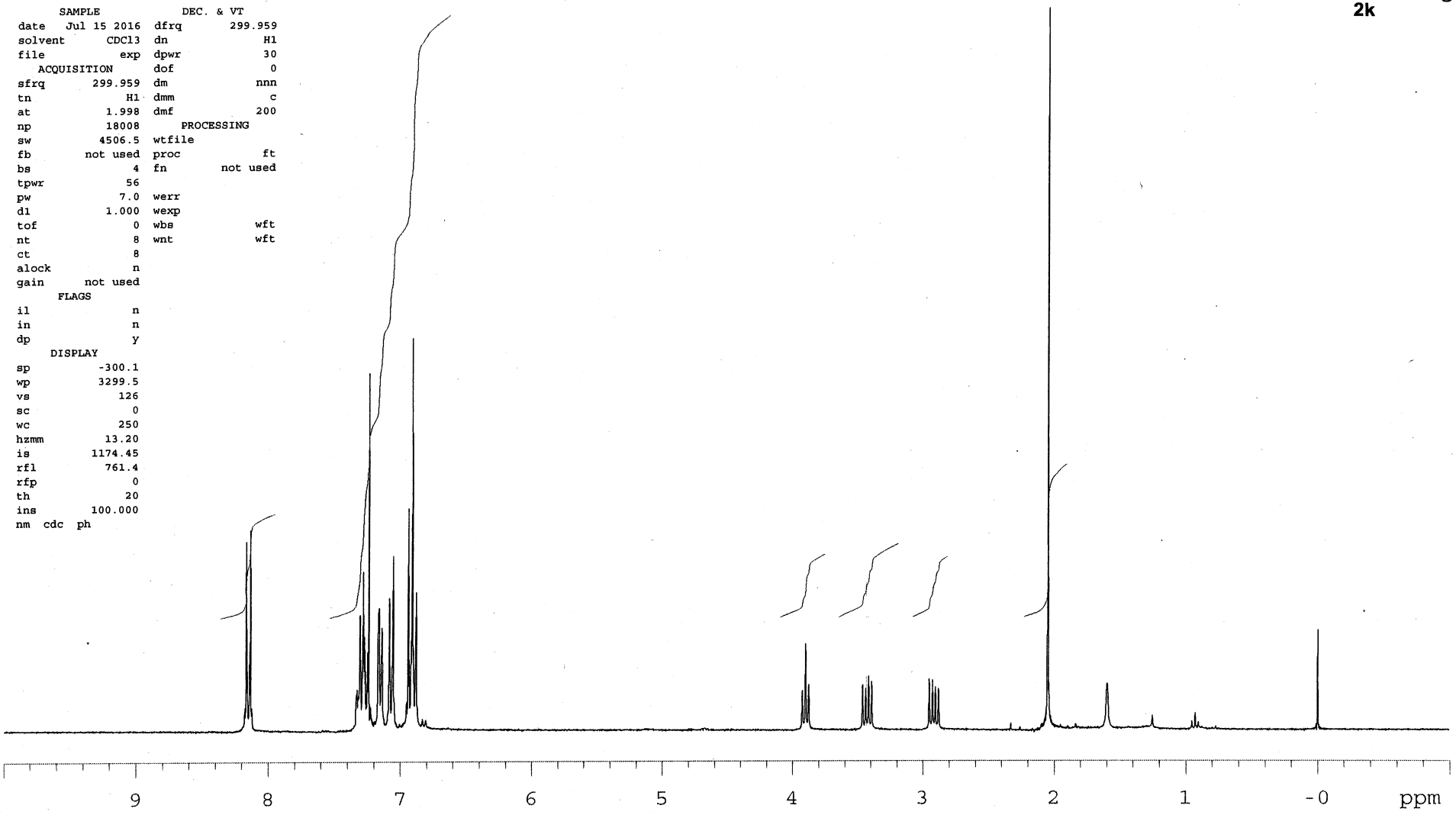


2k

MF E-9 PTLC1

exp2 std1h

SAMPLE		DEC. & VT	
date	Jul 15 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.959	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3299.5		
vs	126		
sc	0		
wc	250		
hzmm	13.20		
is	1174.45		
rfl	761.4		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			

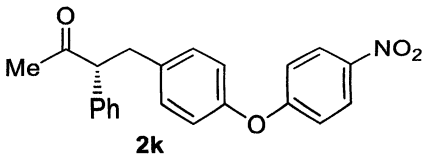


MF E-9 PTLC1 13C

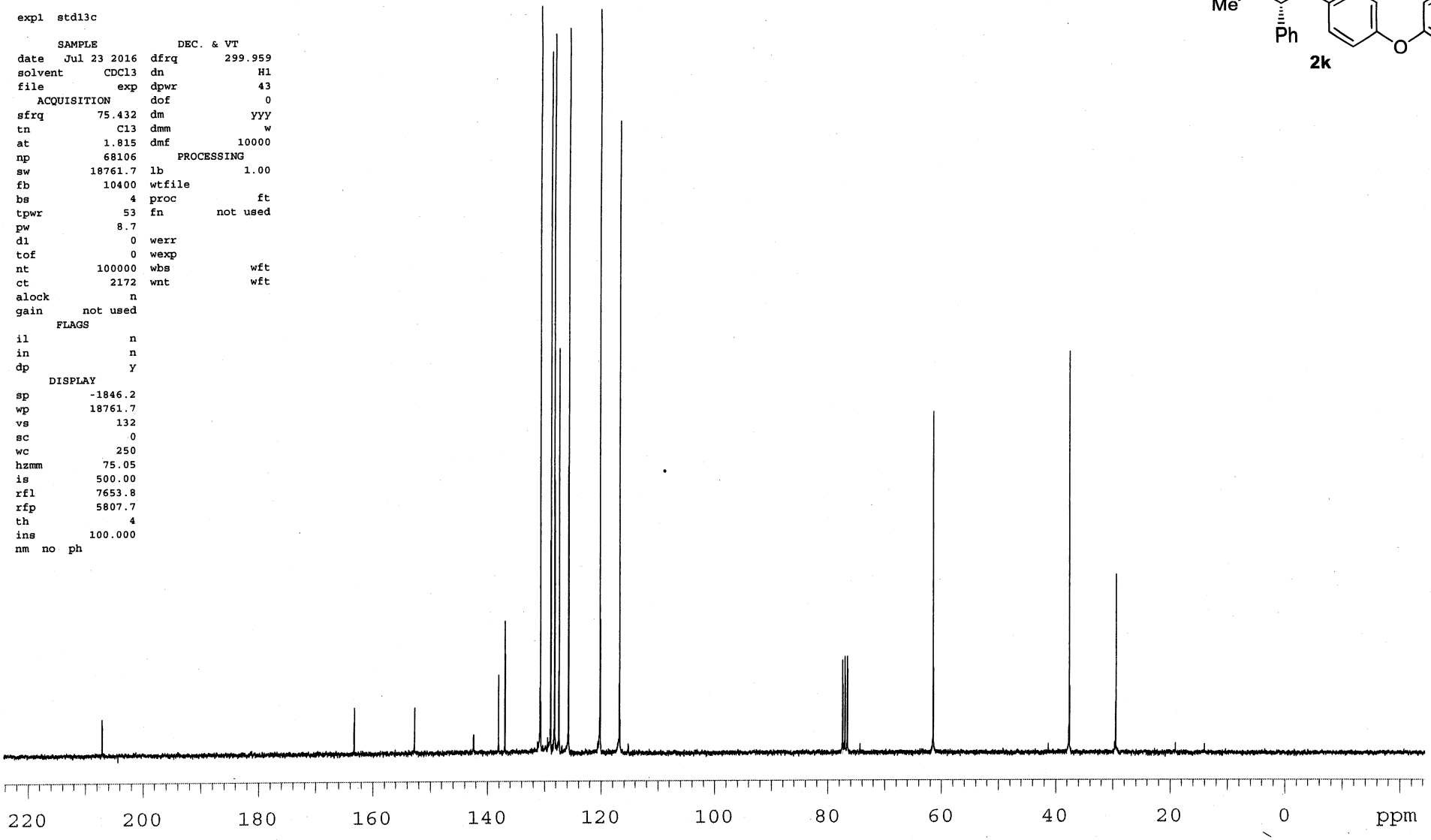
exp1 std13c

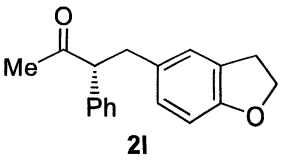
SAMPLE		DEC. & VT	
date	Jul 23 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.432	dm	yyy
tn	C13	dmm	w
at	1.815	dmf	10000
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	0	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	2172	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1846.2		
wp	18761.7		
vs	132		
sc	0		
wc	250		
hzmm	75.05		
is	500.00		
rfl	7653.8		
rfp	5807.7		
th	4		
ins	100.000		

nm no ph



2k





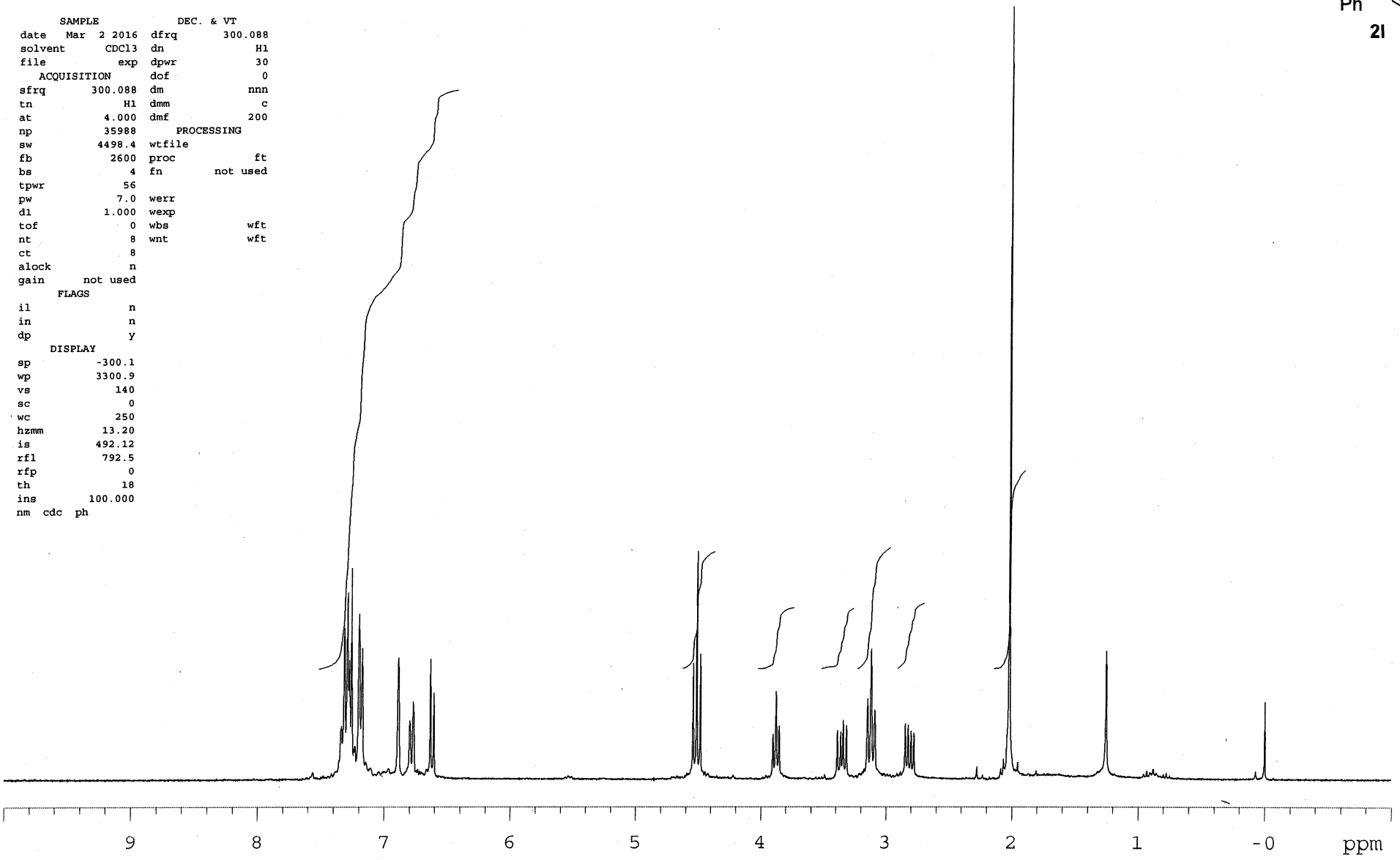
2l

TK A-16 PTLC1

exp1 stdlh

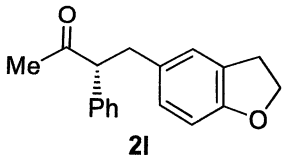
SAMPLE		DEC. & VT	
date	Mar 2 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	140		
sc	0		
wc	250		
hzmm	13.20		
is	492.12		
rfl	792.5		
rfp	0		
th	18		
ins	100.000		

nm cdc ph

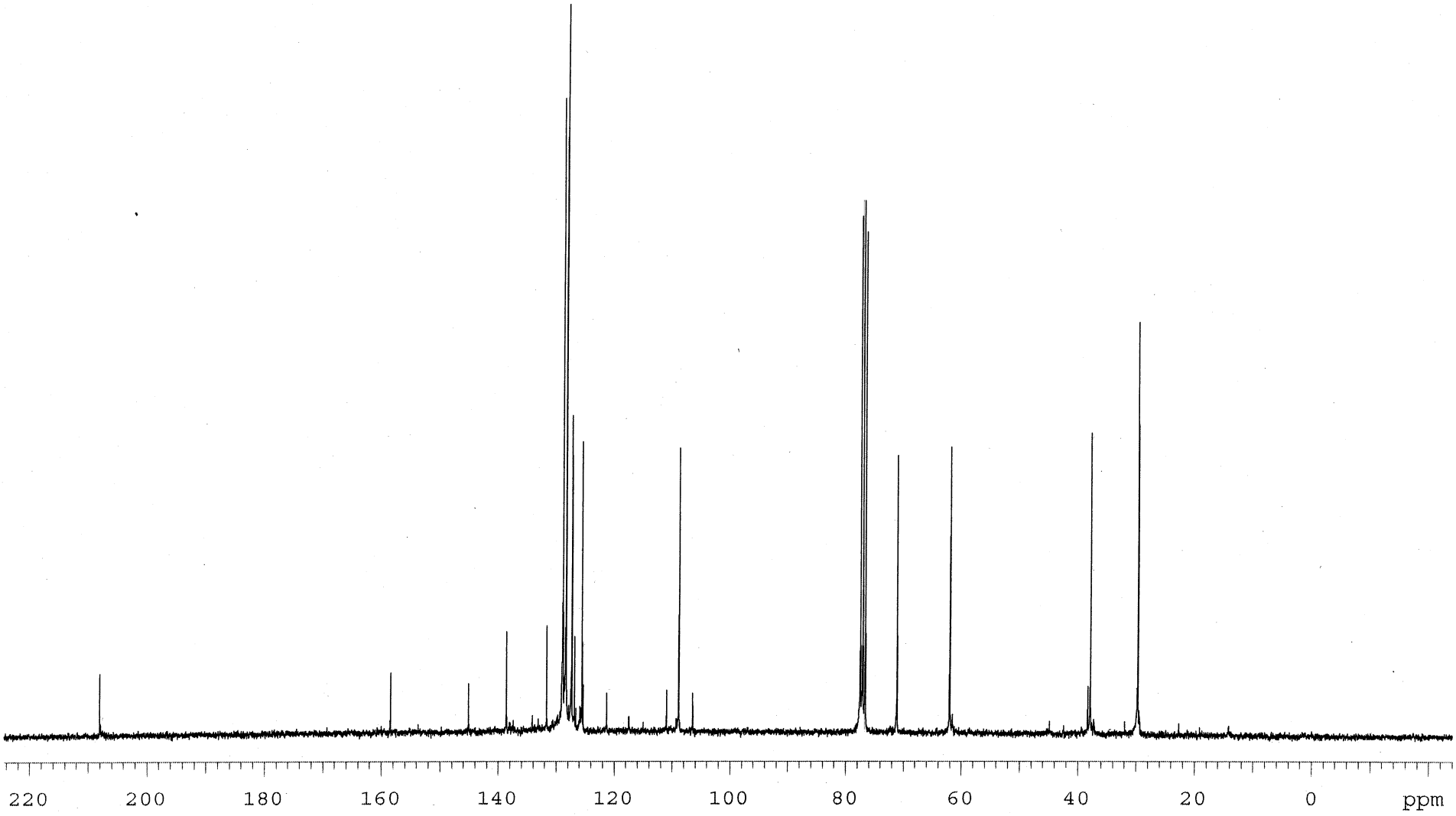


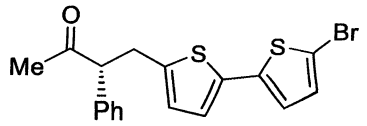
TK A-16 PTLC1 13C

Pulse Sequence: s2pul



2l



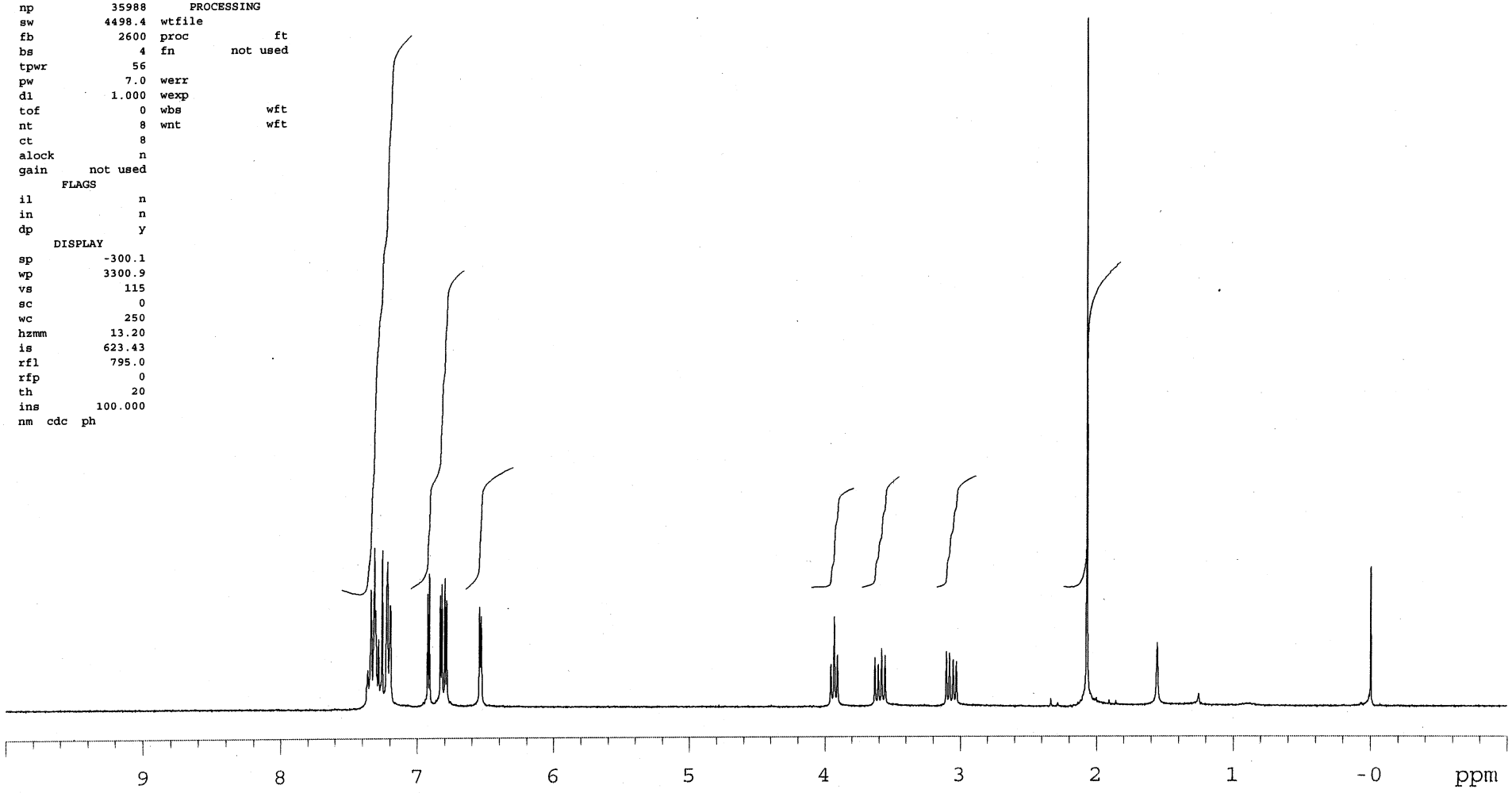


2m

MF-E-21 PTLC1

exp1 stdlh

SAMPLE		DEC. & VT	
date	Jul 29 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	115		
sc	0		
wc	250		
hzmm	13.20		
is	623.43		
rfl	795.0		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			

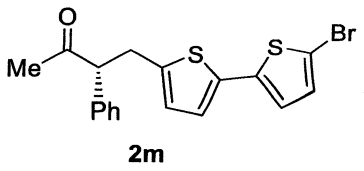


ME E-21 PTLC1 13C

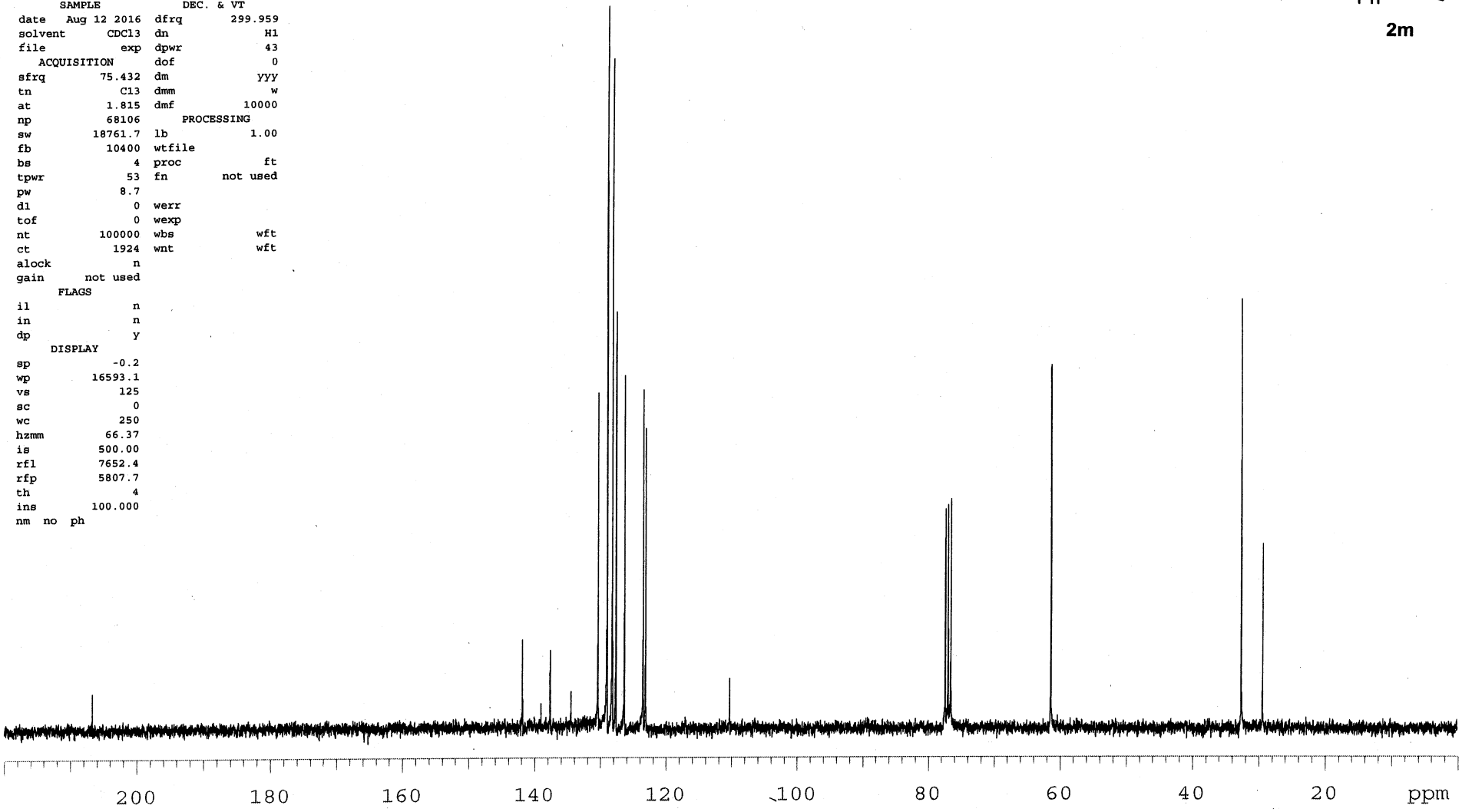
exp1 std13c

SAMPLE		DEC. & VT	
date	Aug 12 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.432	dm	yyy
tn	C13	dmm	w
at	1.815	dmf	10000
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	0	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	1924	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-0.2		
wp	16593.1		
vs	125		
sc	0		
wc	250		
hzmm	66.37		
is	500.00		
rfl	7652.4		
rfp	5807.7		
th	4		
ins	100.000		

nm no ph



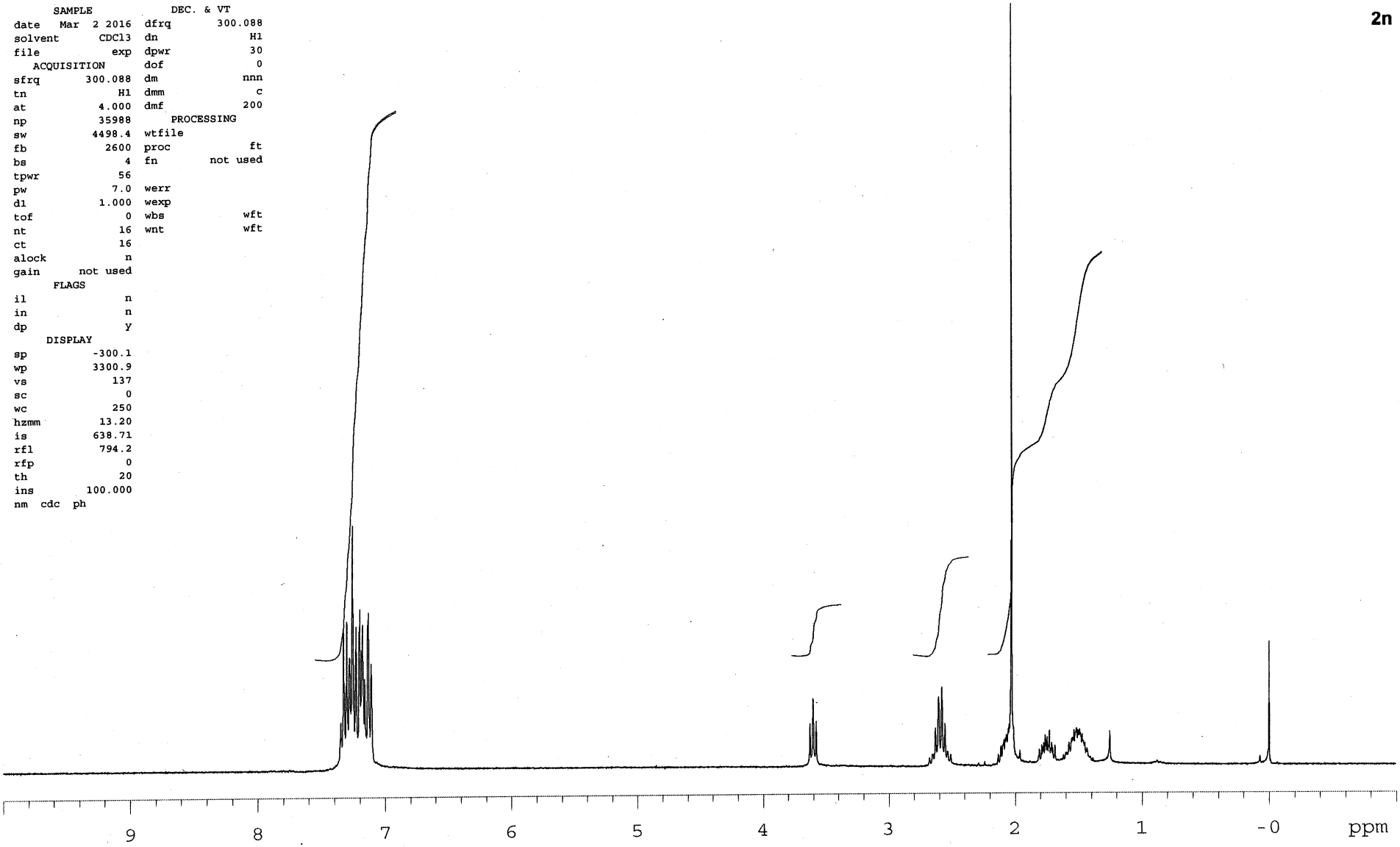
2m

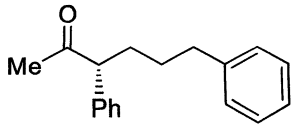


MF B-33 PTLC1

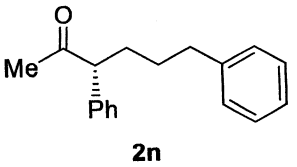
exp1 std1h

SAMPLE		DEC. & VT	
date	Mar 2 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	137		
sc	0		
wc	250		
hzmm	13.20		
is	638.71		
rfl	794.2		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			





2n



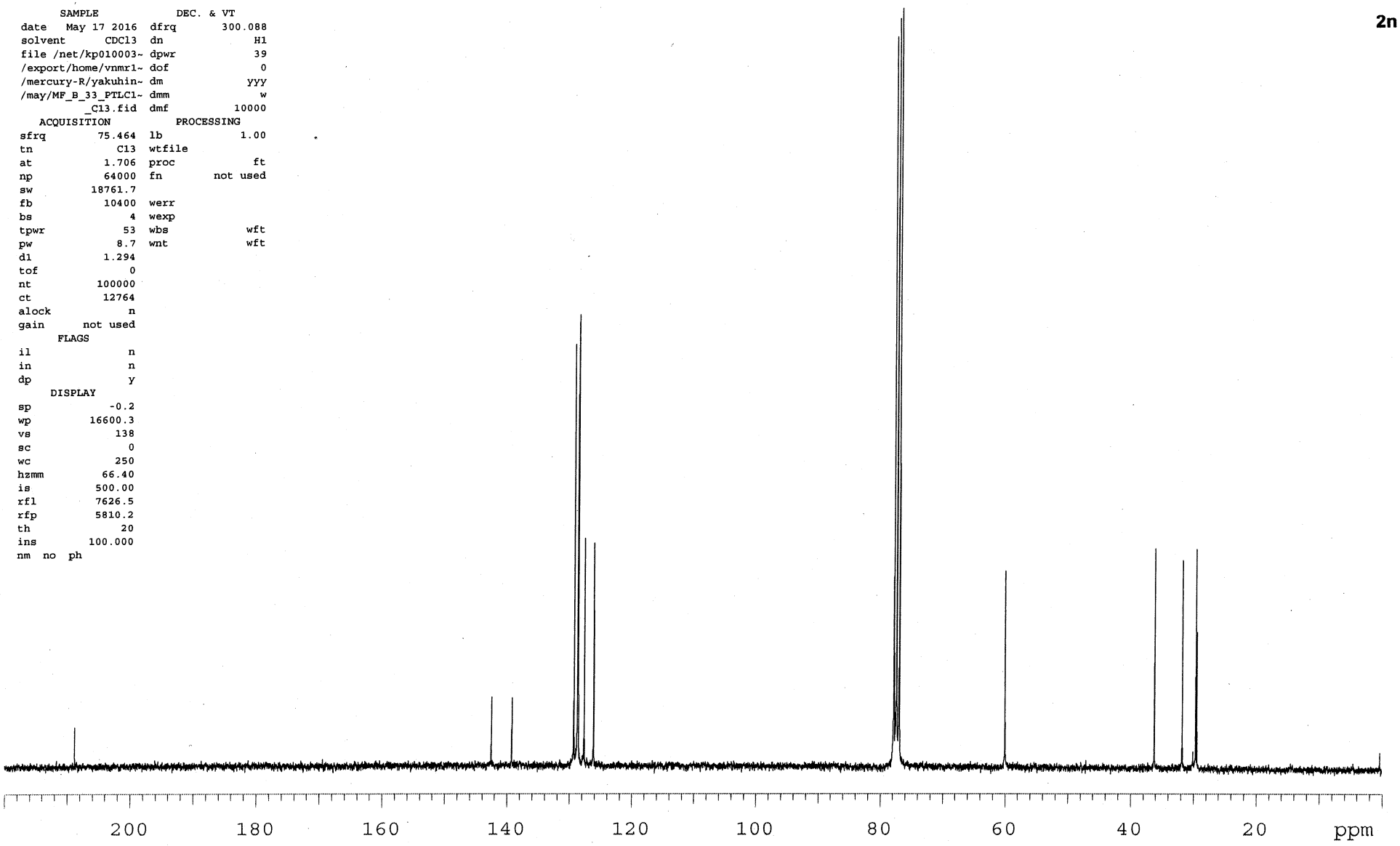
2n

MF B-33 PTLC1 C13

exp1 std13c

SAMPLE		DEC. & VT	
date	May 17 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	/net/kp010003~	dpwr	39
	/export/home/vnmr1~	dof	0
	/mercury-R/yakuhin~	dm	yyy
	/may/MF_B_33_PTLC1~	dmm	w
	_C13.fid	dmf	10000
ACQUISITION		PROCESSING	
sfrq	75.464	lb	1.00
tn	C13	wtfile	
at	1.706	proc	ft
np	64000	fn	not used
sw	18761.7		
fb	10400	werr	
bs	4	wexp	
tpwr	53	wbs	wft
pw	8.7	wnt	wft
d1	1.294		
tof	0		
nt	100000		
ct	12764		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-0.2		
wp	16600.3		
vs	138		
sc	0		
wc	250		
hzmm	66.40		
is	500.00		
rfl	7626.5		
rfp	5810.2		
th	20		
ins	100.000		

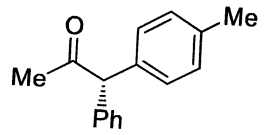
nm no ph



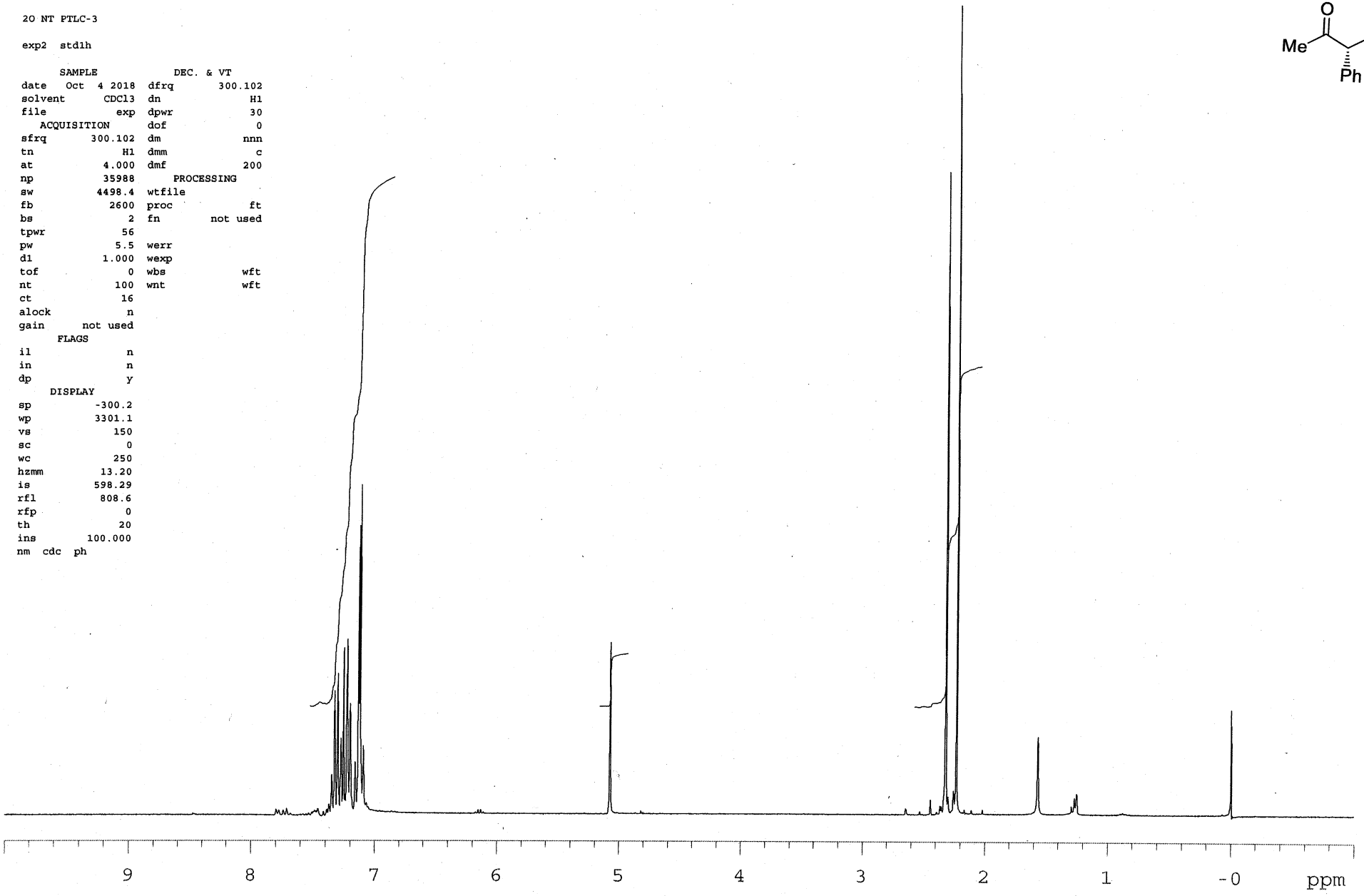
20 NT PTLC-3

exp2 std1h

SAMPLE		DEC. & VT	
date	Oct 4 2018	dfrq	300.102
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.102	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	2	fn	not used
tpwr	56		
pw	5.5	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	100	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	150		
sc	0		
wc	250		
hzmm	13.20		
is	598.29		
rfl	808.6		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			



2o

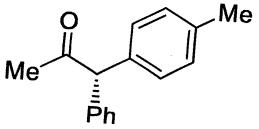


2o NT PTLC-3 13C

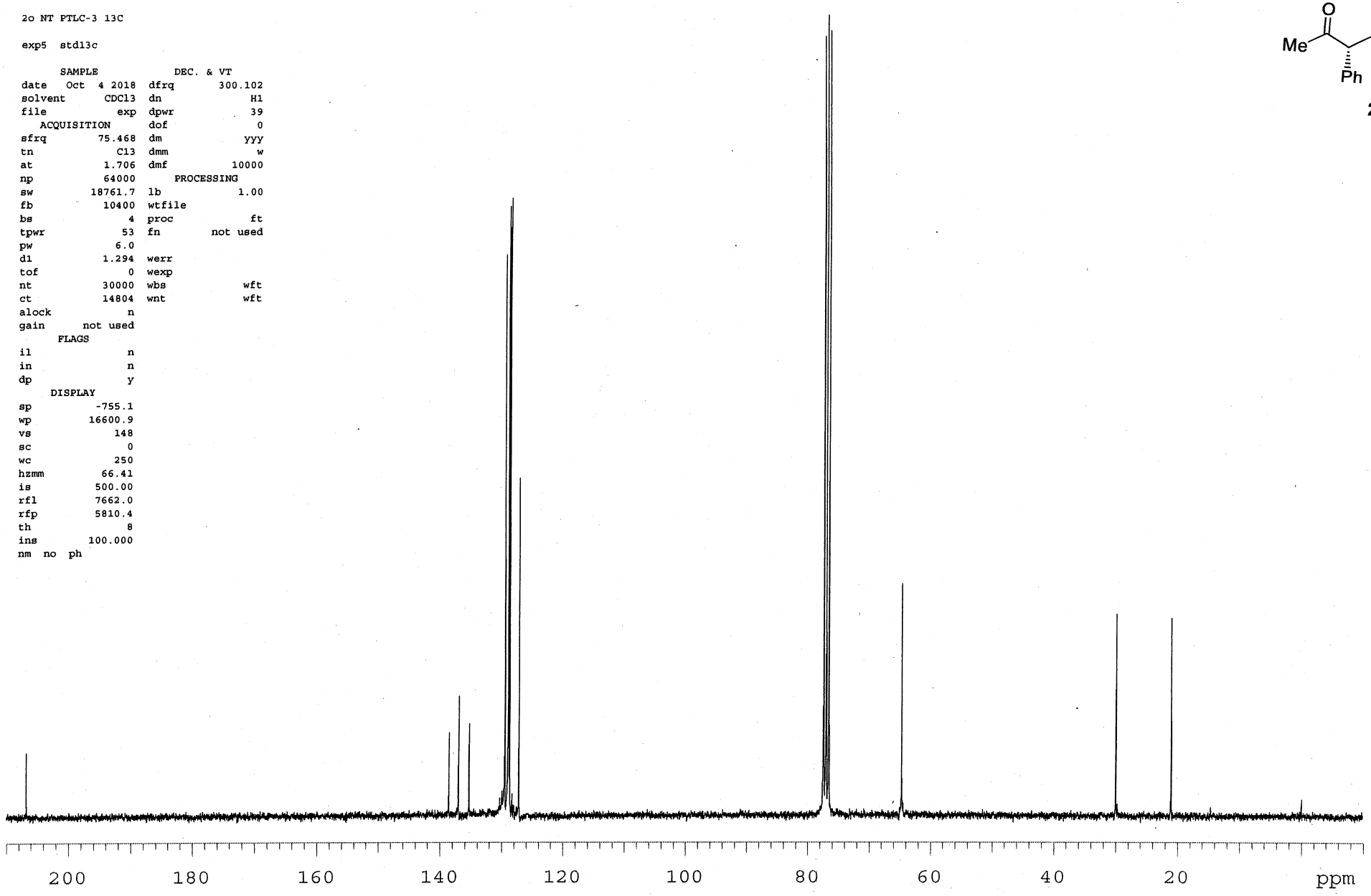
exp5 std13c

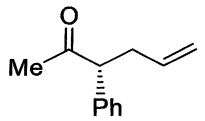
SAMPLE		DEC. & VT	
date	Oct 4 2018	dfrq	300.102
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	75.468	dm	yyy
tn	C13	dmm	w
at	1.706	dmf	10000
np	64000	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	6.0		
d1	1.294	werr	
tof	0	wexp	
nt	30000	wbs	wft
ct	14804	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-755.1		
wp	16600.9		
vs	148		
sc	0		
wc	250		
hzmm	66.41		
is	500.00		
rfl	7662.0		
rfp	5810.4		
th	8		
ins	100.000		

nm no ph



2o



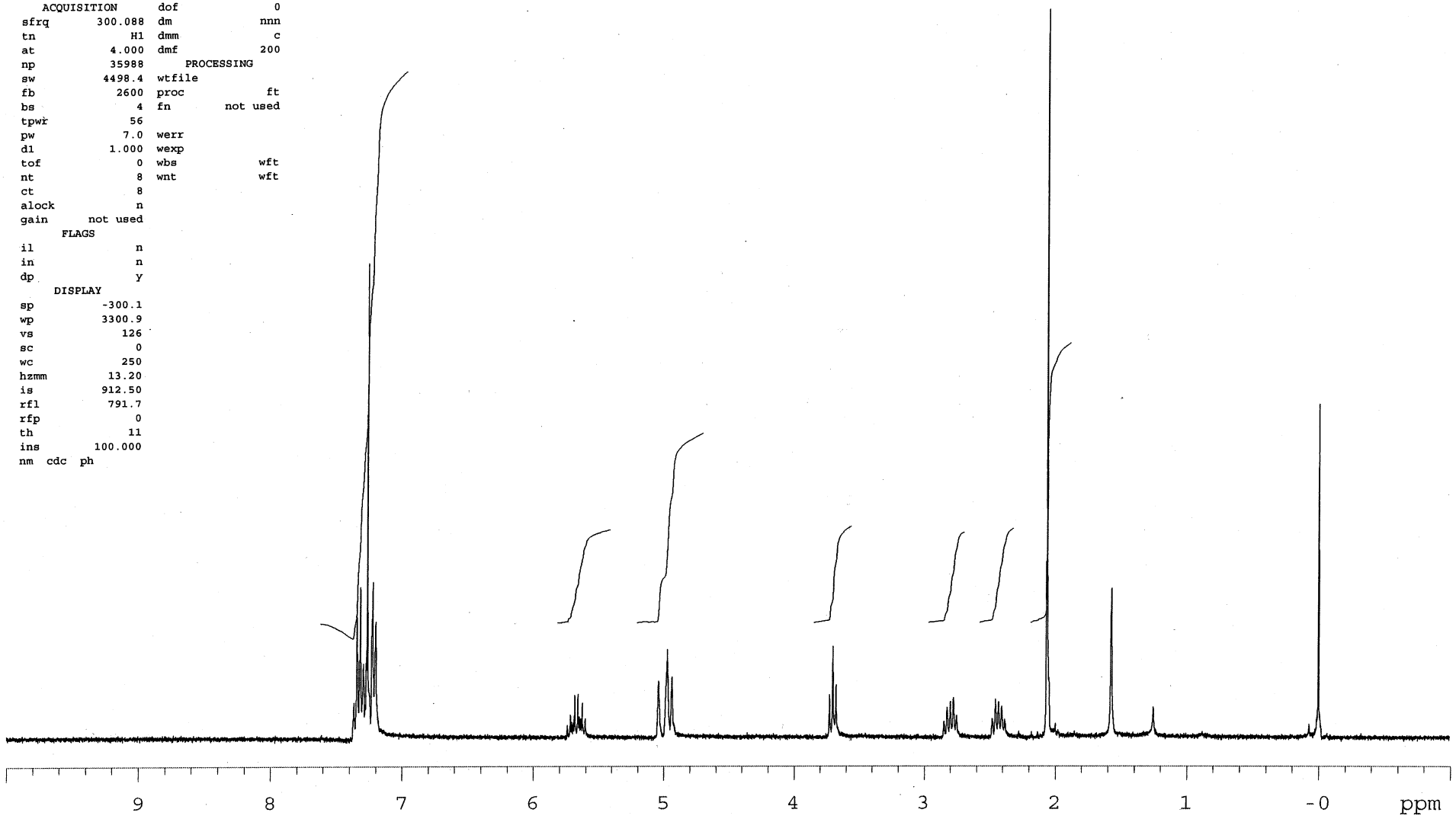


2p

MF A-33

exp2 std1h

SAMPLE		DEC. & VT	
date	Mar 1 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	126		
sc	0		
wc	250		
hzmm	13.20		
is	912.50		
rfl	791.7		
rfp	0		
th	11		
ins	100.000		
nm cdc ph			

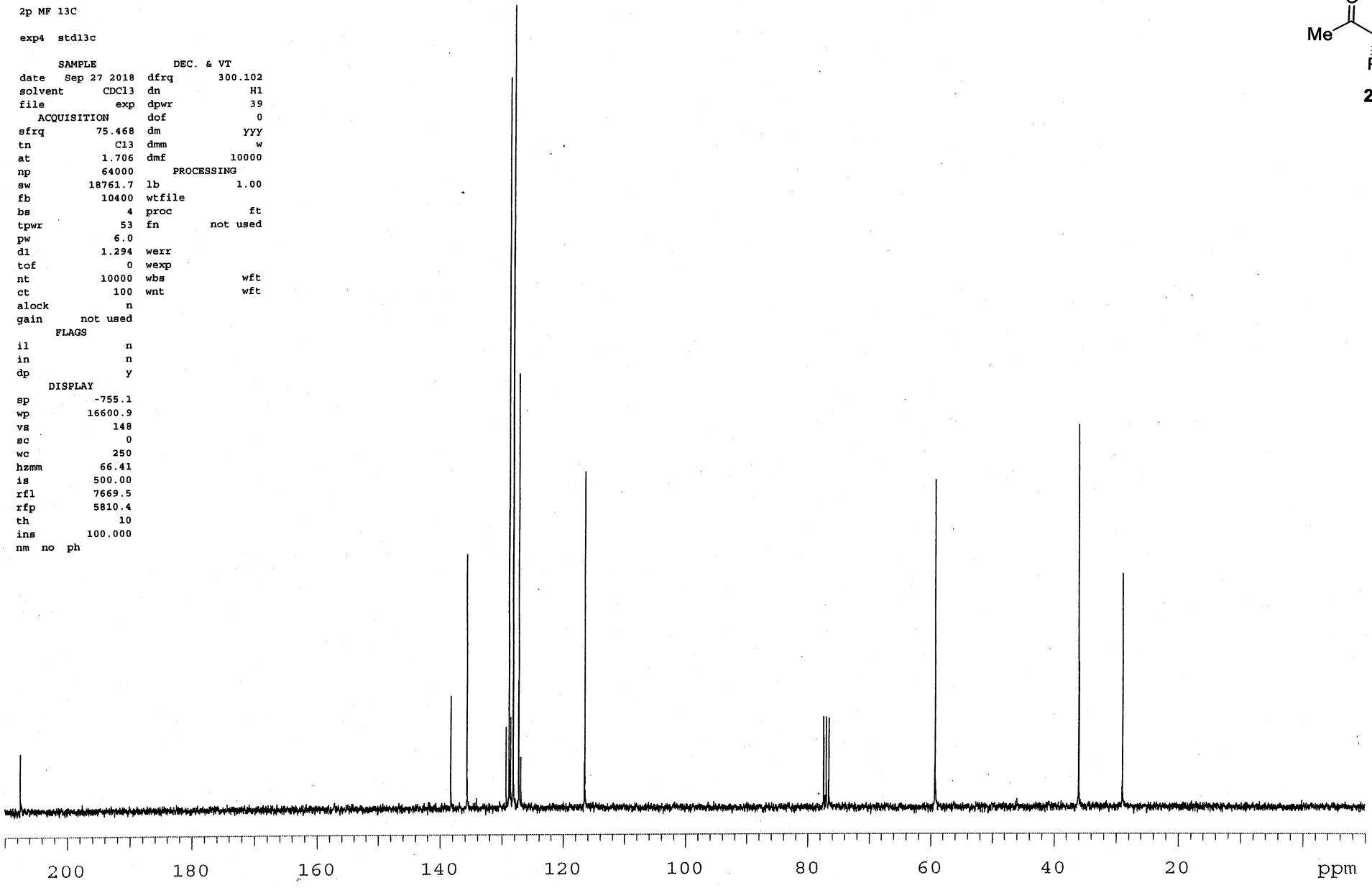


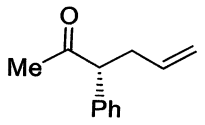
2p MF 13C

exp4 std13c

SAMPLE		DEC. & VT	
date	Sep 27 2018	dfrq	300.102
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	75.468	dm	yyy
tn	C13	dmm	w
at	1.706	dmf	10000
np	64000	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	6.0		
d1	1.294	werr	
tof	0	wexp	
nt	10000	wbs	wft
ct	100	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-755.1		
wp	16600.9		
vs	148		
sc	0		
wc	250		
hzmm	66.41		
is	500.00		
rfl	7669.5		
rfp	5810.4		
th	10		
ins	100.000		

nm no ph



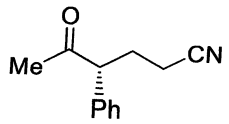


2p

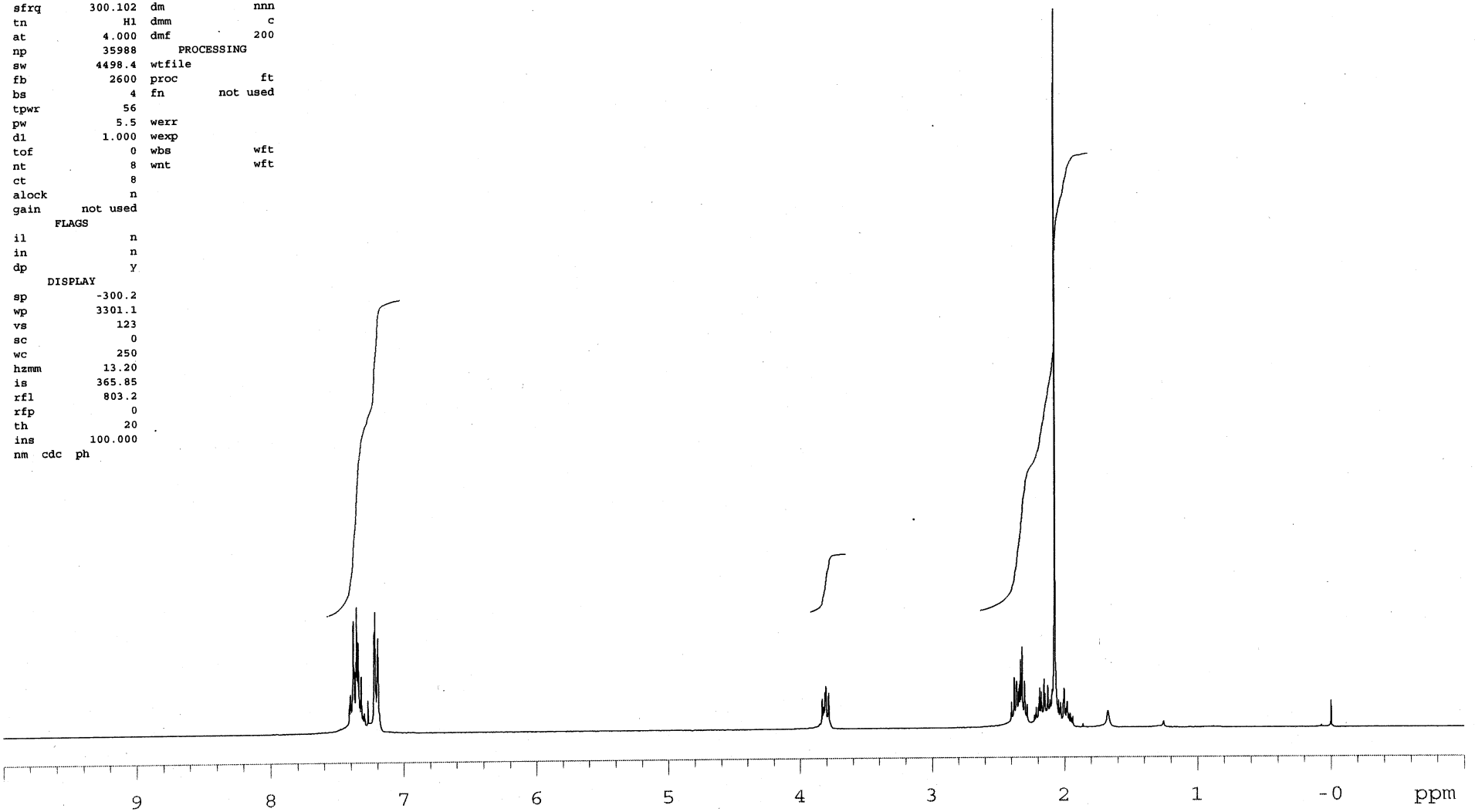
MF F-10 PTLC2

exp2 stdlh

SAMPLE		DEC. & VT	
date	Jul 24 2017	dfrq	300.102
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.102	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	5.5	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.2		
wp	3301.1		
vs	123		
sc	0		
wc	250		
hzmm	13.20		
is	365.85		
rfl	803.2		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			

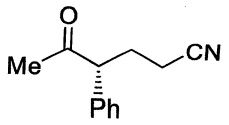


2q

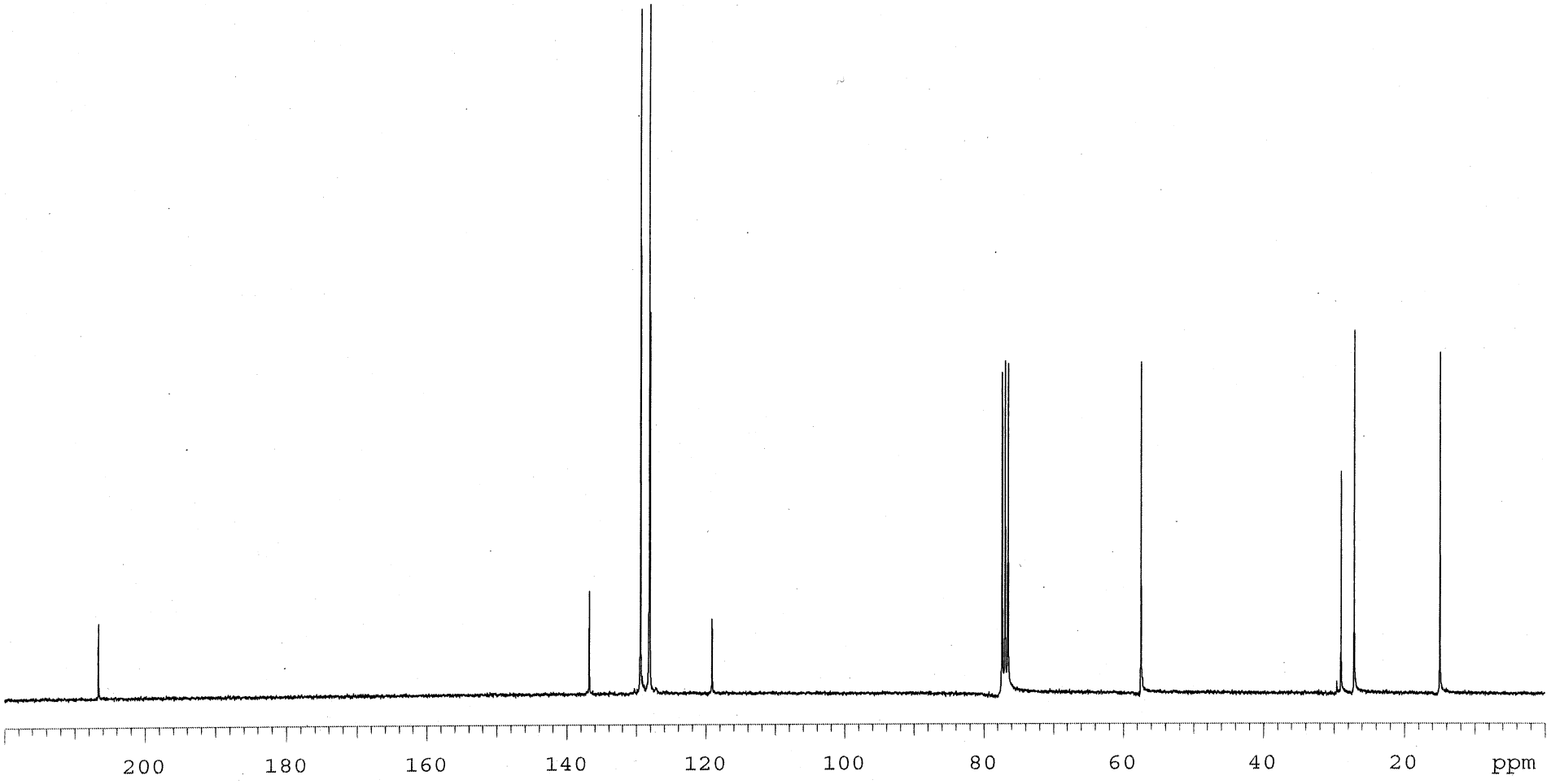


MF F-10 PTLC2

Pulse Sequence: s2pul



2q

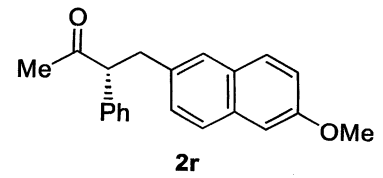


AT A-47 PTLC1

exp2 std1h

SAMPLE		DEC. & VT	
date	Mar 1 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	197		
sc	0		
wc	250		
hzmm	13.20		
is	412.30		
rfl	795.7		
rfp	0		
th	8		
ins	100.000		

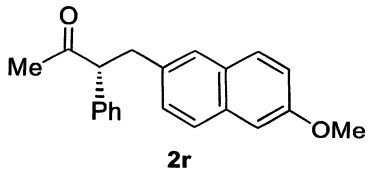
nm cdc ph



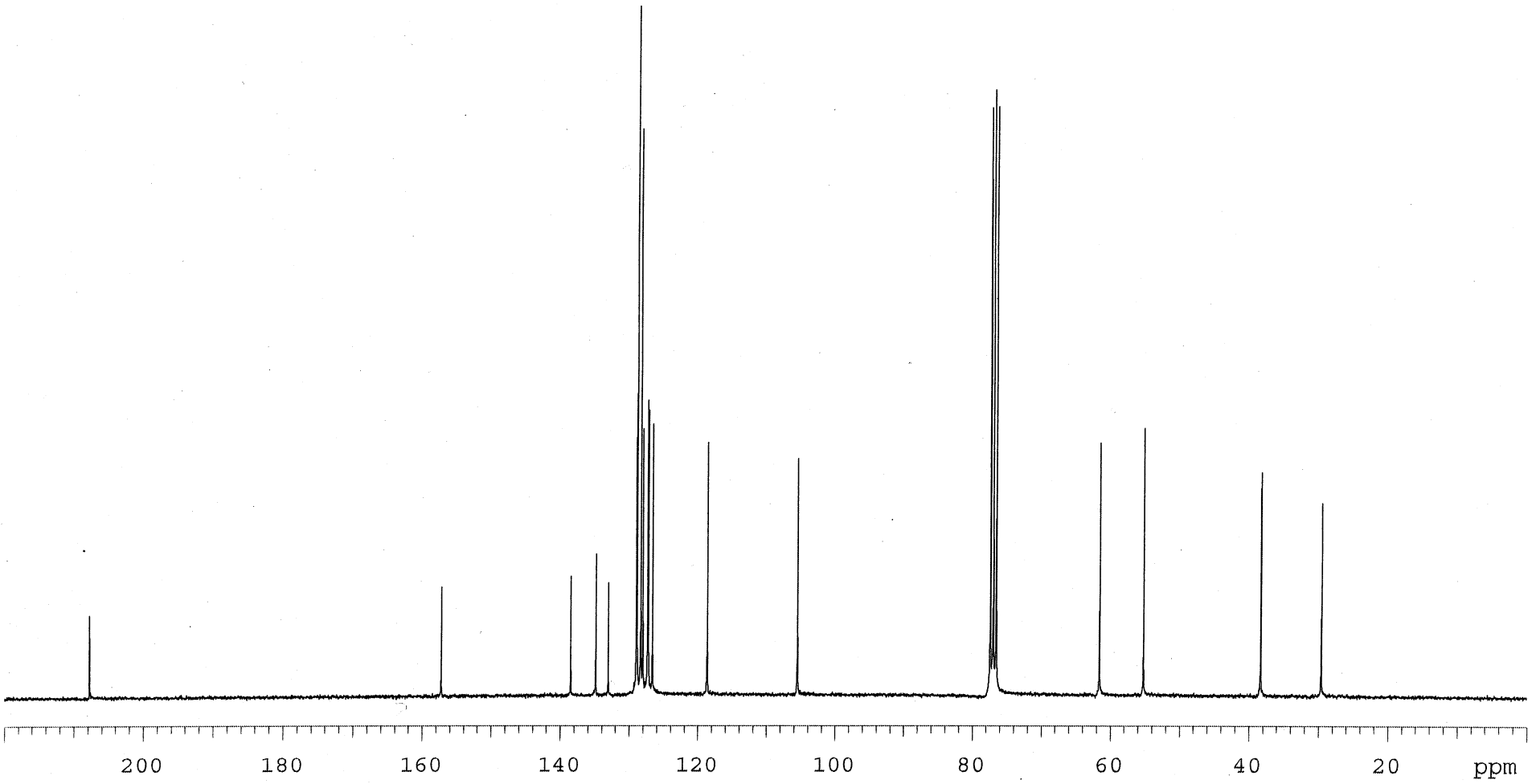
2r

AT A-47 PTLC1

Pulse Sequence: s2pul



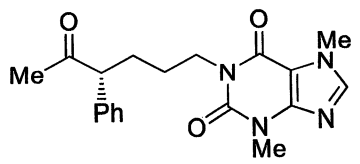
2r



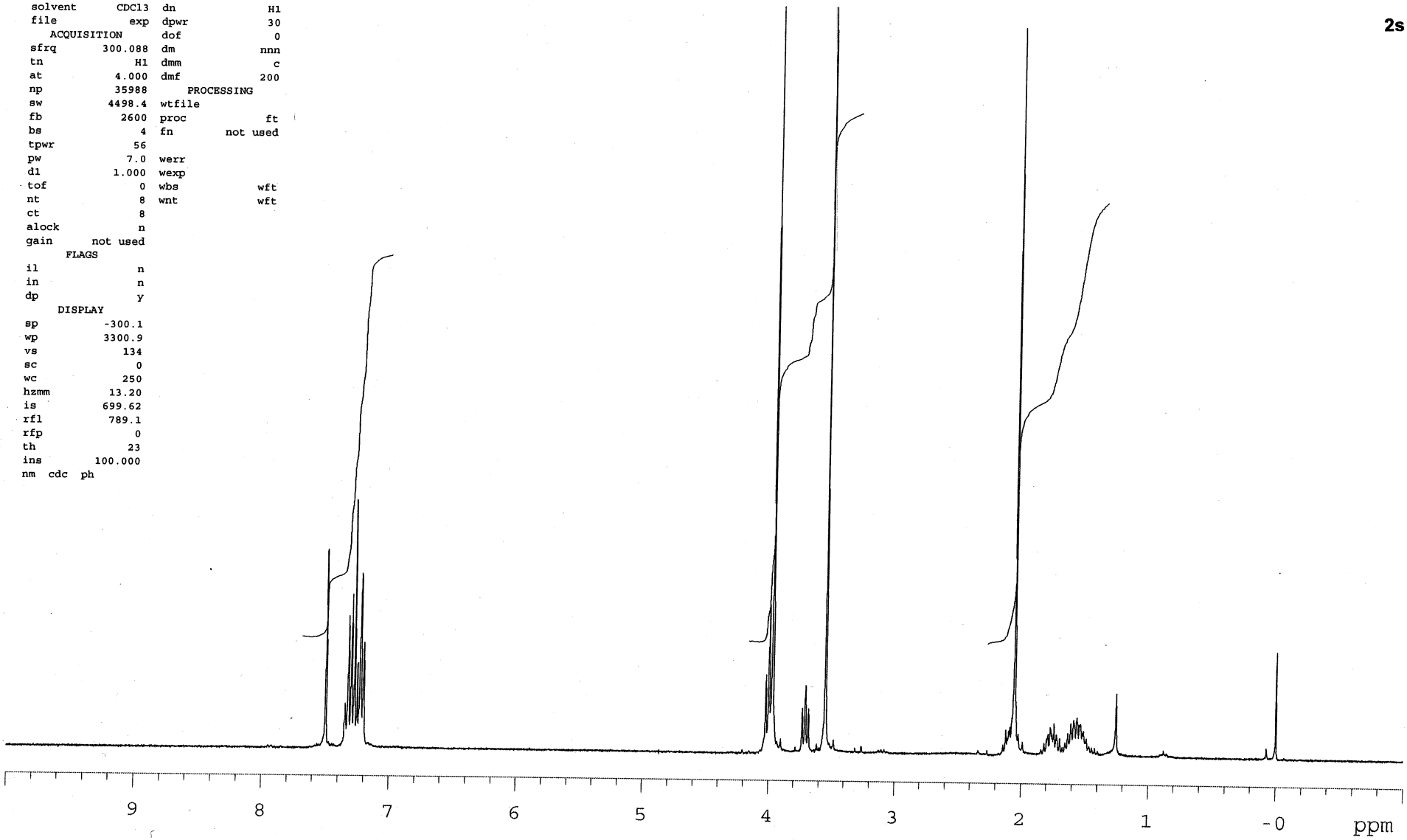
TK A-12 PTLC2

expl stdlh

SAMPLE		DEC. & VT	
date	Mar 2 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.088	dm	nnn
tn	H1	dmm	c
at	4.000	dmf	200
np	35988	PROCESSING	
sw	4498.4	wtfile	
fb	2600	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3300.9		
vs	134		
sc	0		
wc	250		
hzmm	13.20		
is	699.62		
rfl	789.1		
rfp	0		
th	23		
ins	100.000		
nm cdc ph			



2s

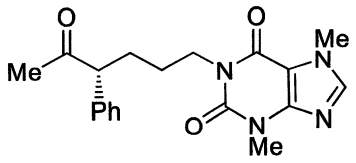


TK-A-12 PTLC2 13C

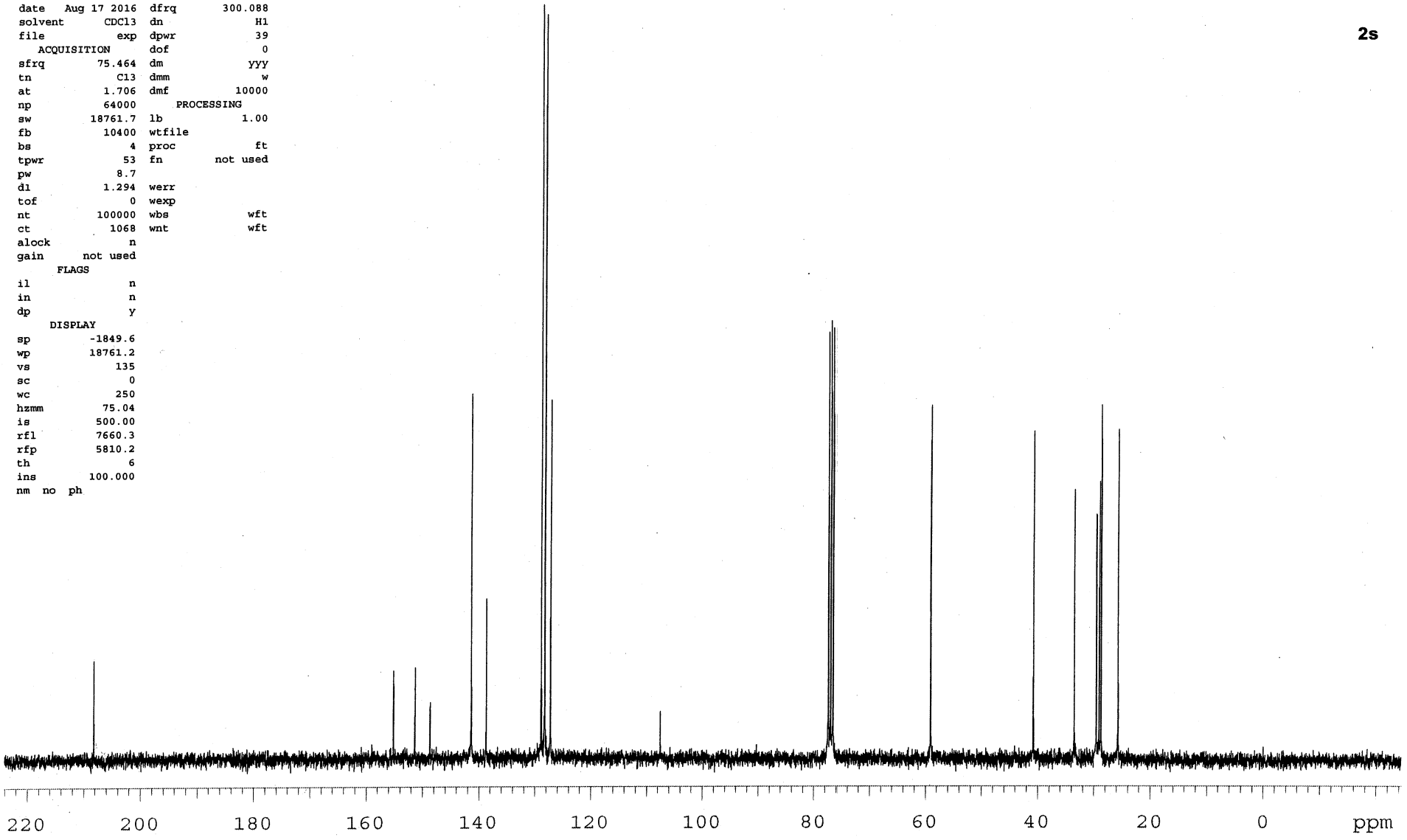
expl std13c

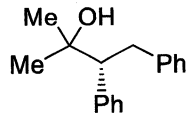
SAMPLE		DEC. & VT	
date	Aug 17 2016	dfrq	300.088
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	75.464	dm	yyy
tn	C13	dmm	w
at	1.706	dmf	10000
np	64000	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	1.294	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	1068	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1849.6		
wp	18761.2		
vs	135		
sc	0		
wc	250		
hzmm	75.04		
is	500.00		
rfl	7660.3		
rfp	5810.2		
th	6		
ins	100.000		

nm no ph



2s



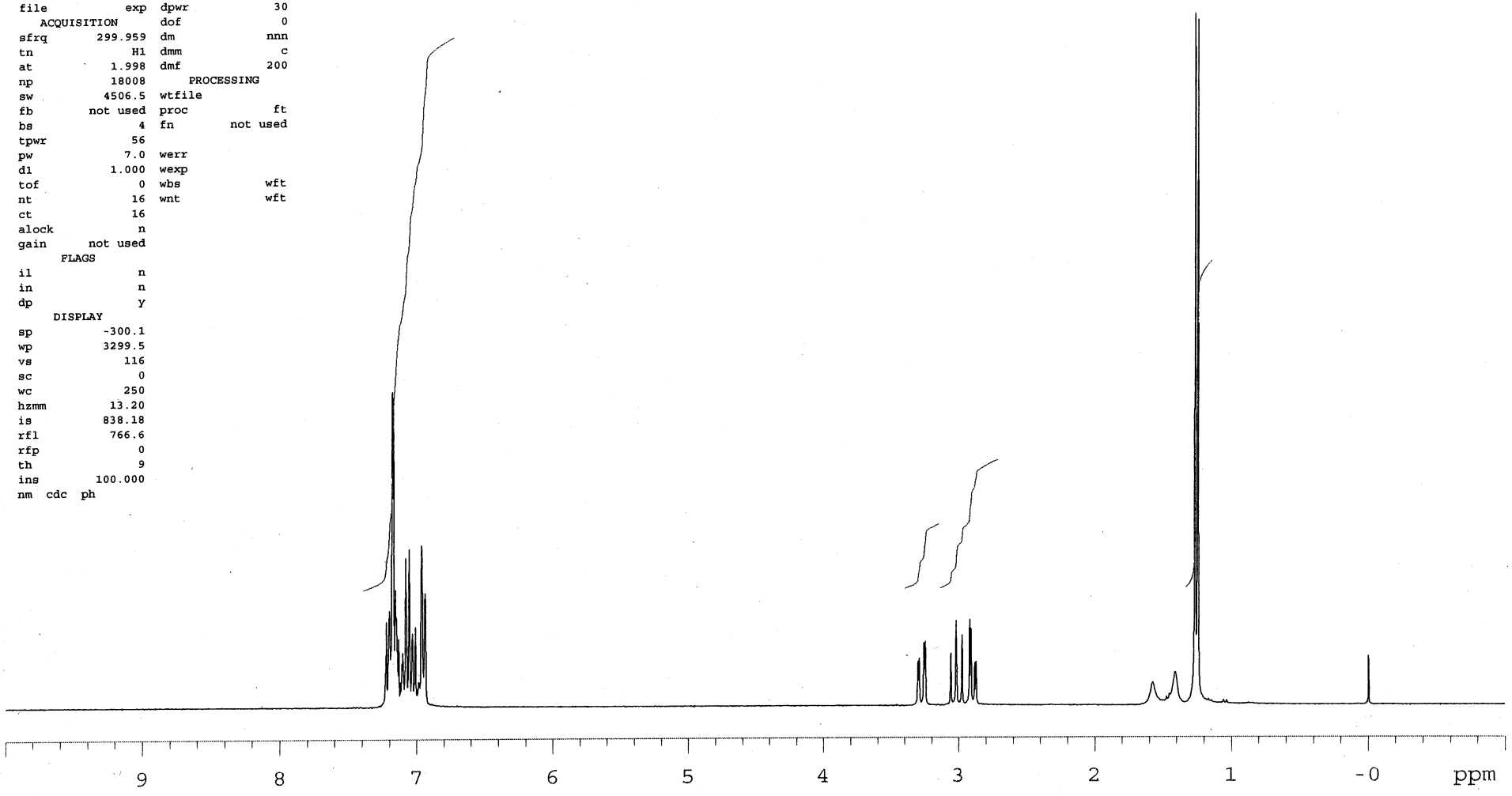


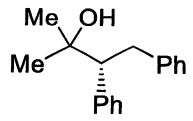
4

MF D-14 PTLC1

exp2 std1h

SAMPLE		DEC. & VT	
date	Jul 15 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	299.959	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	18008	PROCESSING	
sw	4506.5	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	56		
pw	7.0	werr	
d1	1.000	wexp	
tof	0	wbs	wft
nt	16	wnt	wft
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-300.1		
wp	3299.5		
vs	116		
sc	0		
wc	250		
hzmm	13.20		
is	838.18		
rfl	766.6		
rfp	0		
th	9		
ins	100.000		
nm cdc ph			





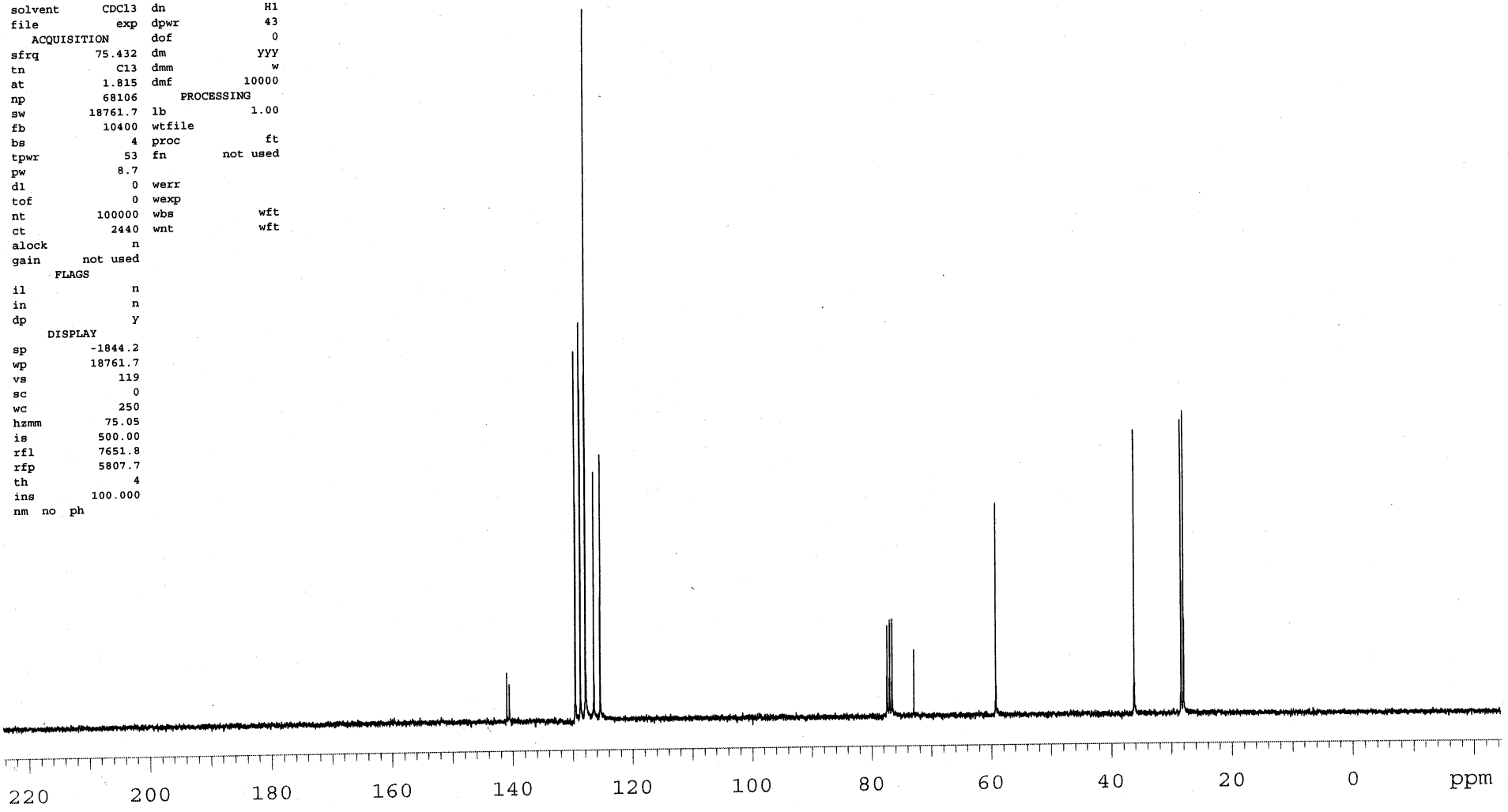
4

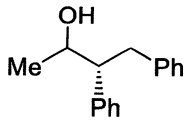
MF D-14 PTLC1 13C

exp2 std13c

SAMPLE		DEC. & VT	
date	Jul 25 2016	dfrq	299.959
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	75.432	dm	YYY
tn	C13	dmm	w
at	1.815	dmf	10000
np	68106	PROCESSING	
sw	18761.7	lb	1.00
fb	10400	wtfile	
bs	4	proc	ft
tpwr	53	fn	not used
pw	8.7		
d1	0	werr	
tof	0	wexp	
nt	100000	wbs	wft
ct	2440	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1844.2		
wp	18761.7		
vs	119		
sc	0		
wc	250		
hzmm	75.05		
is	500.00		
rfl	7651.8		
rfp	5807.7		
th	4		
ins	100.000		

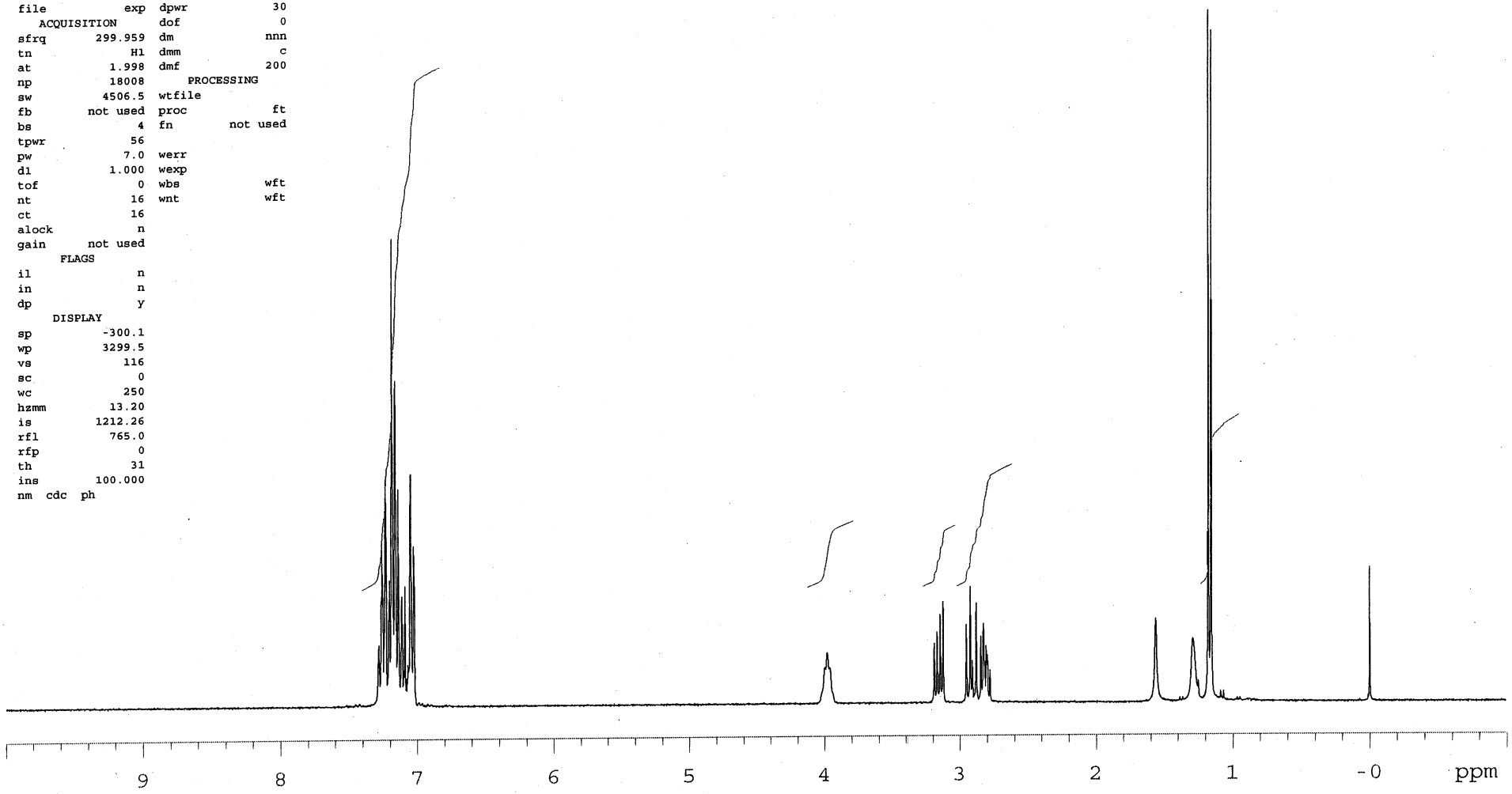
nm no ph





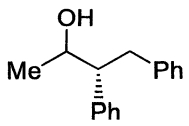
5 (major)

```
MF D-8 PTLC1

exp2  std1h

      SAMPLE
date    Jul 15 2016
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        299.959
tn               H1
at            1.998
np            18008
sw           4506.5
fb        not used
bs                4
tpwr             56
pw              7.0
d1            1.000
tof               0
nt               16
ct               16
alock             n
gain      not used
     FLAGS
il                n
in                n
dp                y
    DISPLAY
sp           -300.1
wp           3299.5
vs              116
sc                0
wc              250
hzmm          13.20
is          1212.26
rfl           765.0
rfp               0
th               31
ins         100.000
nm  cdc  ph

      DEC. & VT
dfrq        299.959
dn               H1
dpwr             30
dof               0
dm              nnn
dmm               c
dmf             200
    PROCESSING
wtfile
proc             ft
fn        not used

werr
wexp
wbs             wft
wnt             wft
```

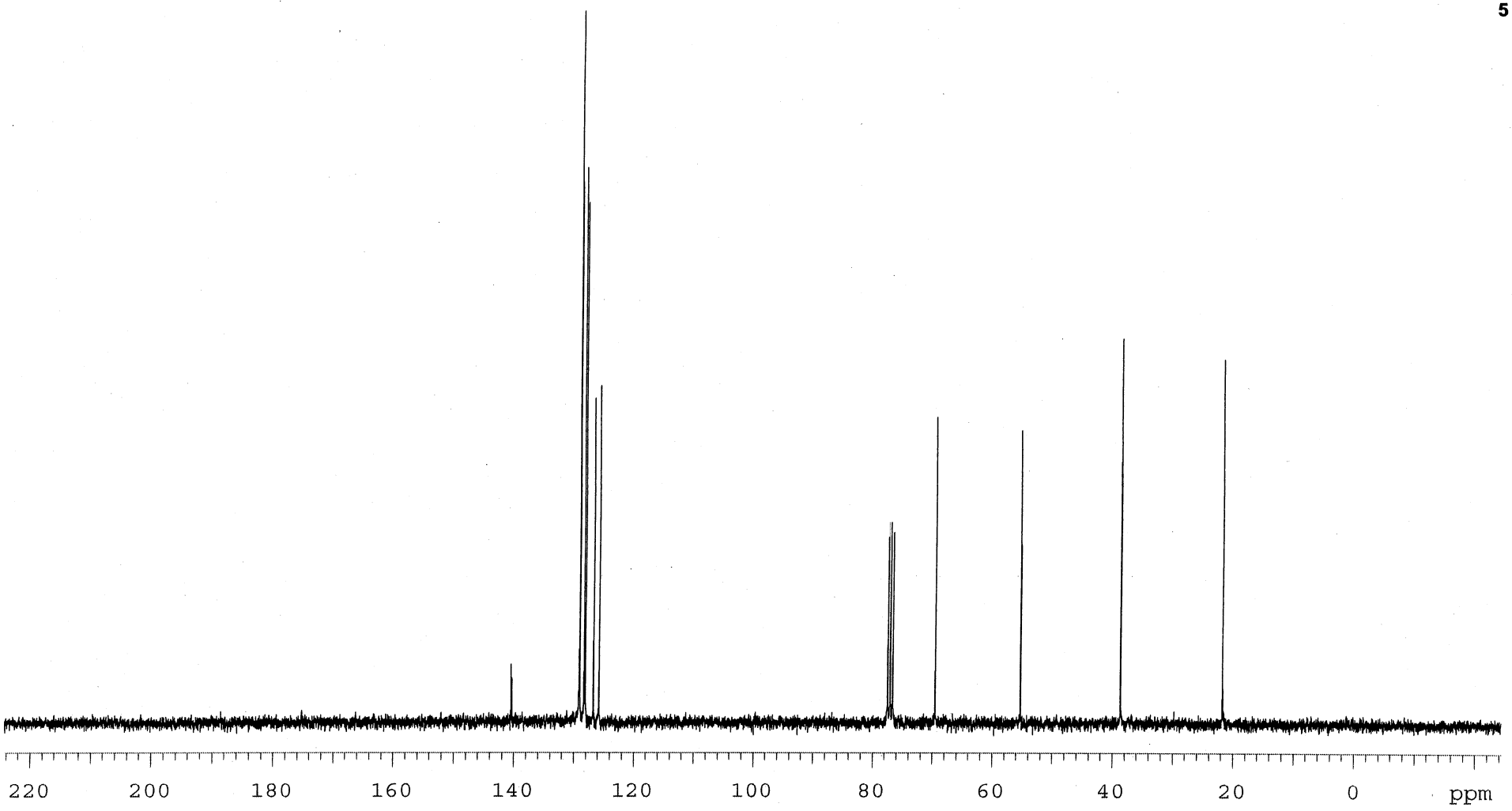


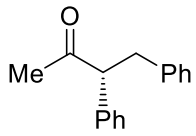
MF D-8 PTLC1 major 13C

Pulse Sequence: s2pul

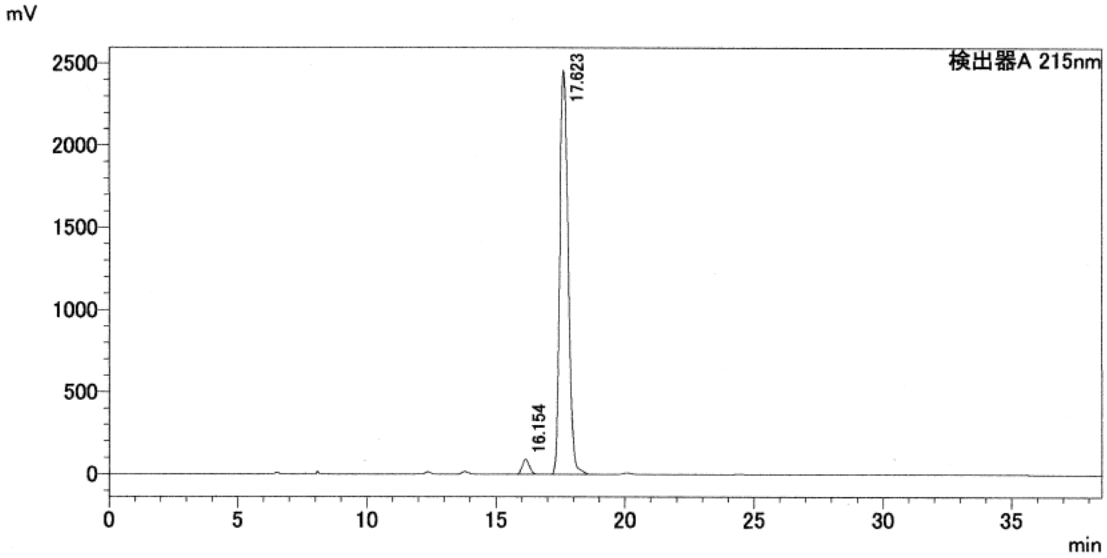


5 (major)



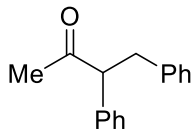


2a

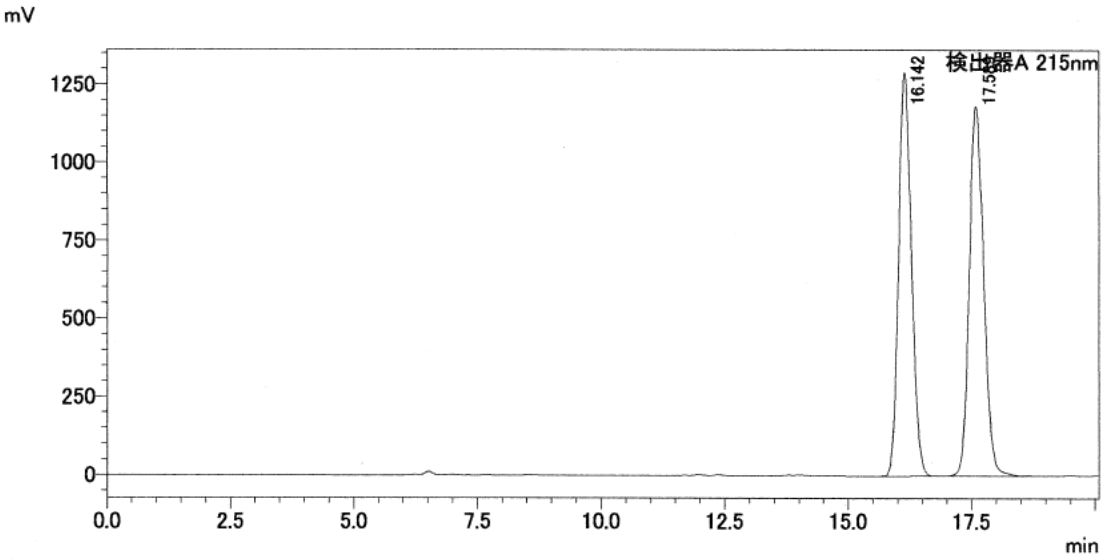


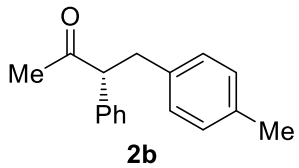
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	16.154	1780230	92318	3.110			
2	17.623	55469793	2458425	96.890			
合計		57250023	2550743				

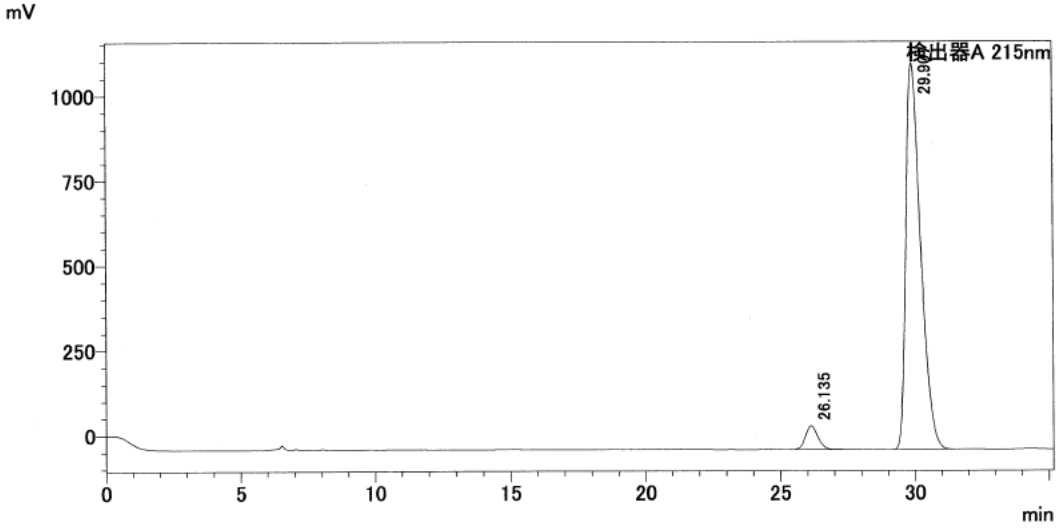


rac-**2a**



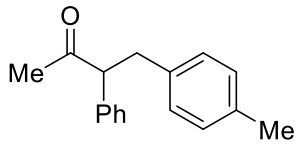


2b

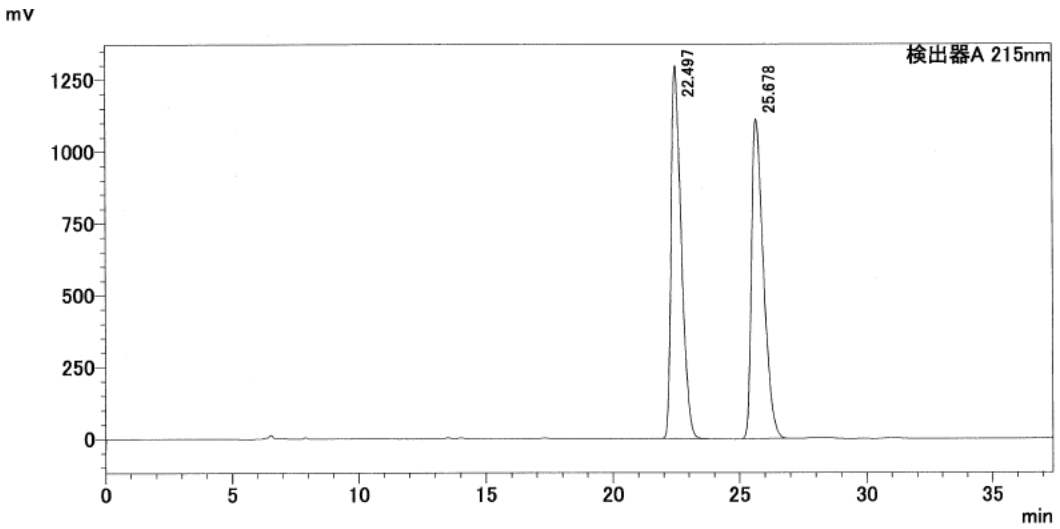


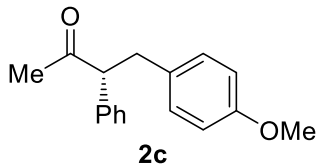
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	26.135	2202748	69045	4.789			
2	29.907	43795973	1136605	95.211			
合計		45998721	1205650				

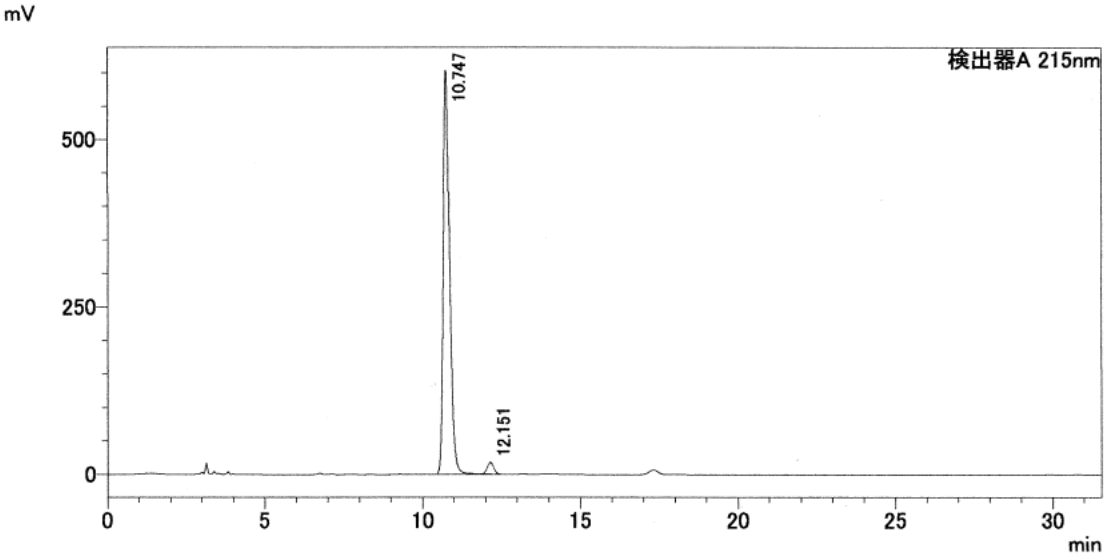


rac-**2b**



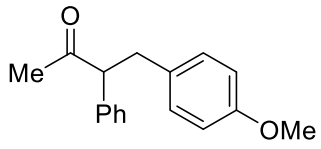


2c

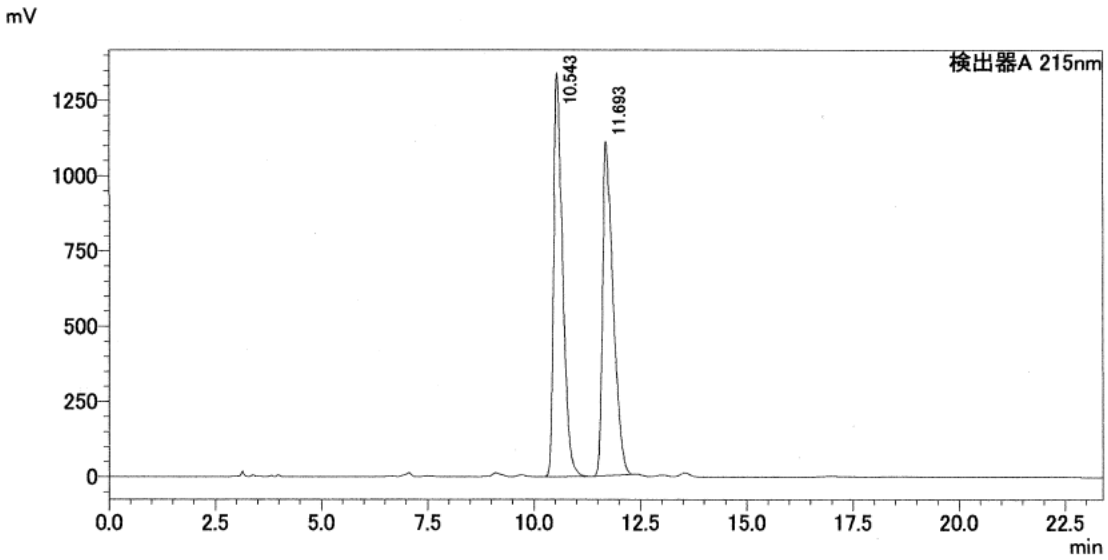


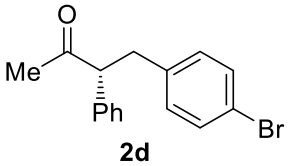
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	10.747	8498327	603610	97.174		S	
2	12.151	247170	17345	2.826		T	
合計		8745497	620955				

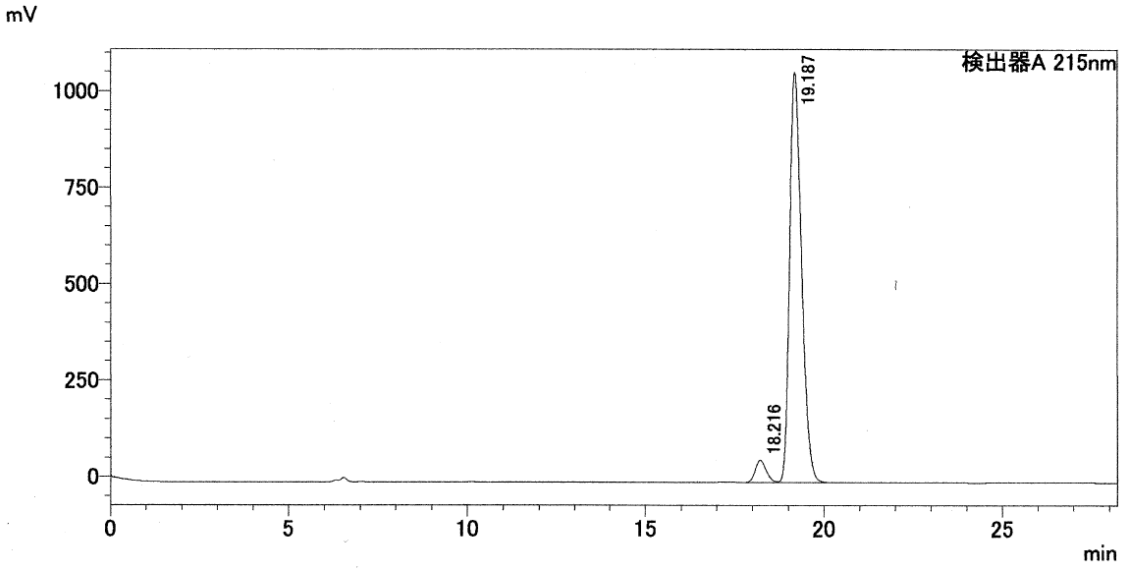


rac-**2c**



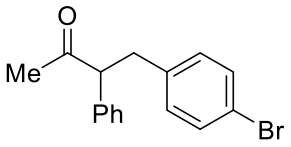


2d

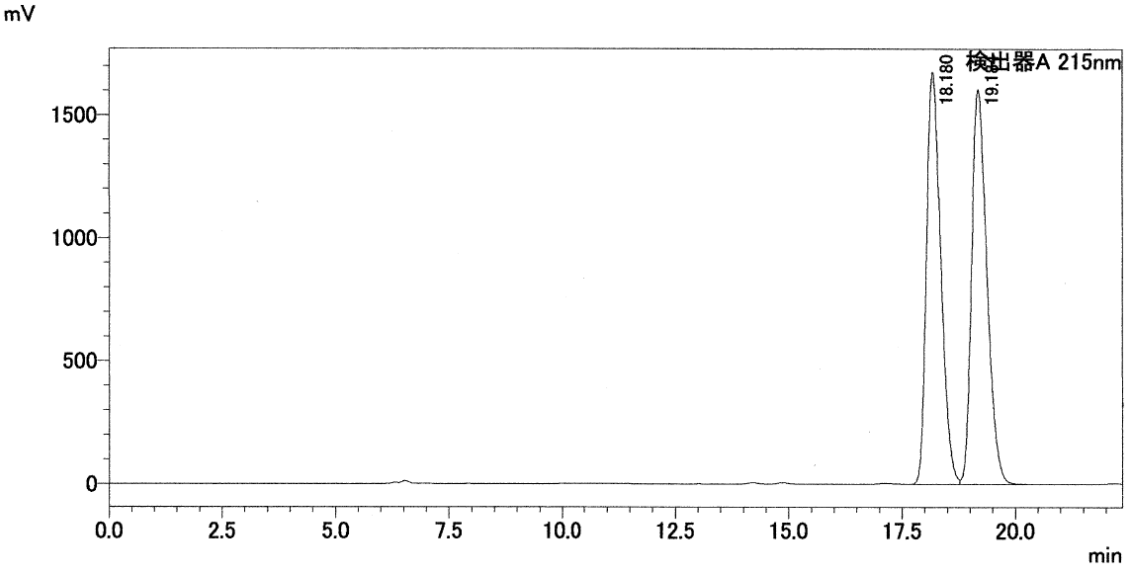


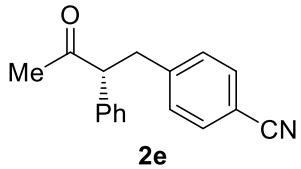
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	18.216	1248264	57927	4.786			
2	19.187	24834901	1063180	95.214		V	
合計		26083166	1121107				

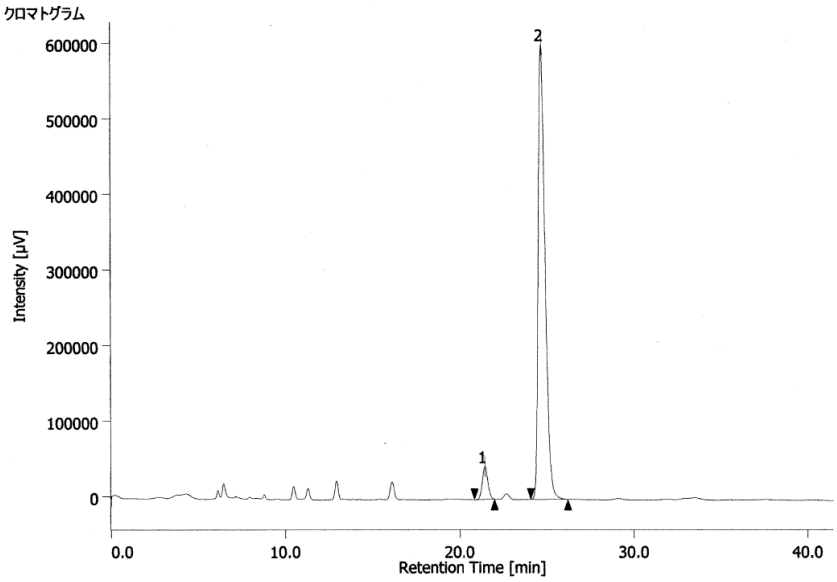


rac-**2d**



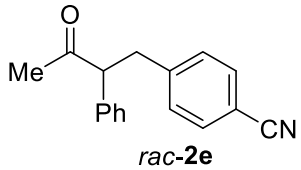


2e

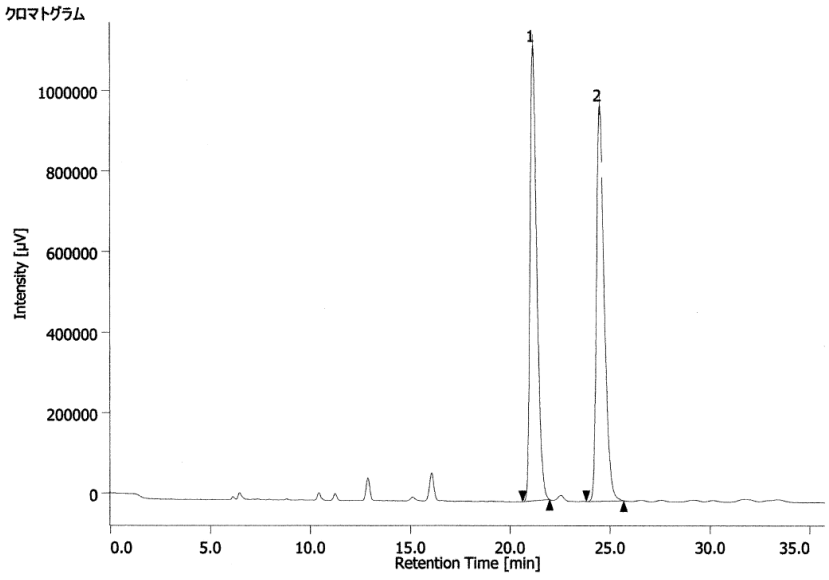


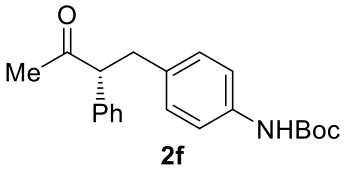
ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	21.458	958253	43450	5.595	6.735
2	1	24.700	16169003	601680	94.405	93.265

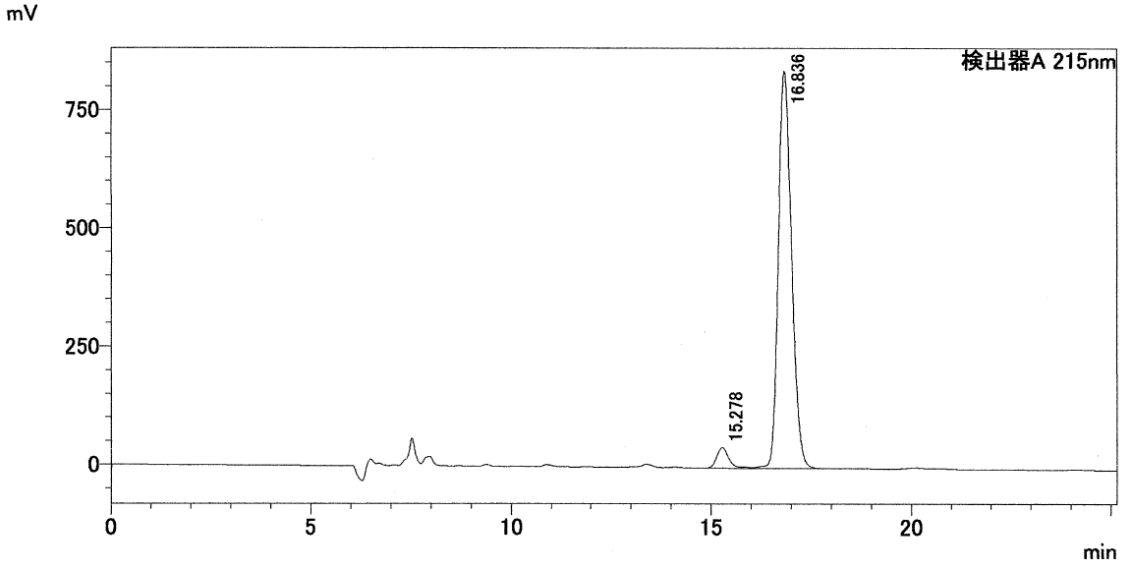


rac-**2e**



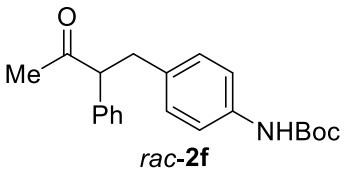


2f

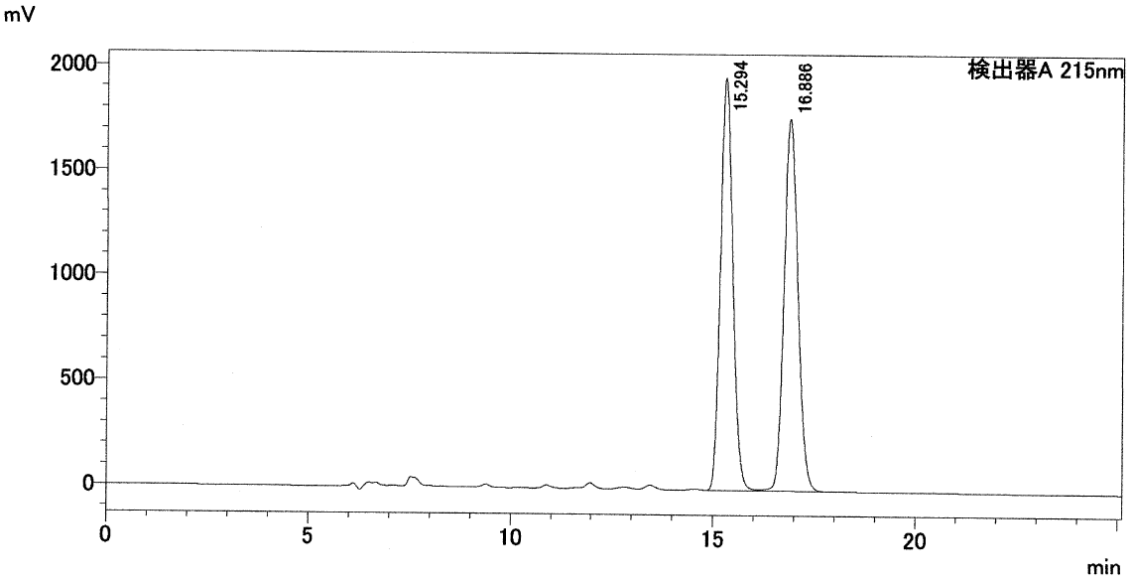


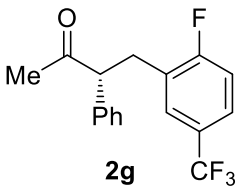
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	15.278	919960	43460	4.541			
2	16.836	19340456	839970	95.459		V	
合計		20260415	883430				

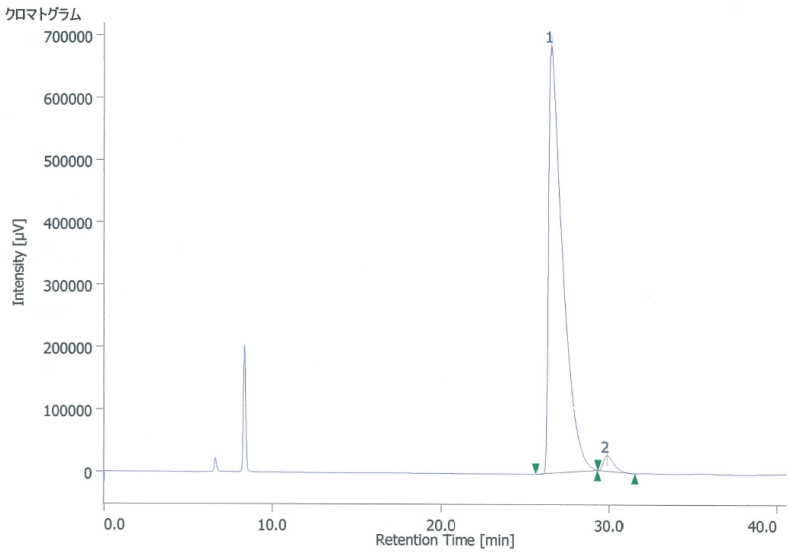


rac-**2f**



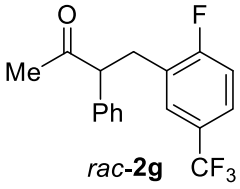


2g

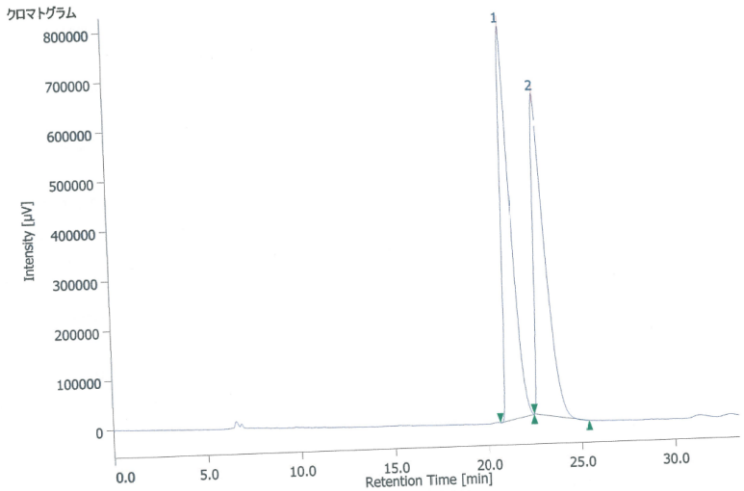


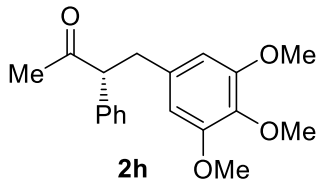
ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	26.533	39317243	685294	97.417	96.512
2	1	29.867	1042561	24764	2.583	3.488

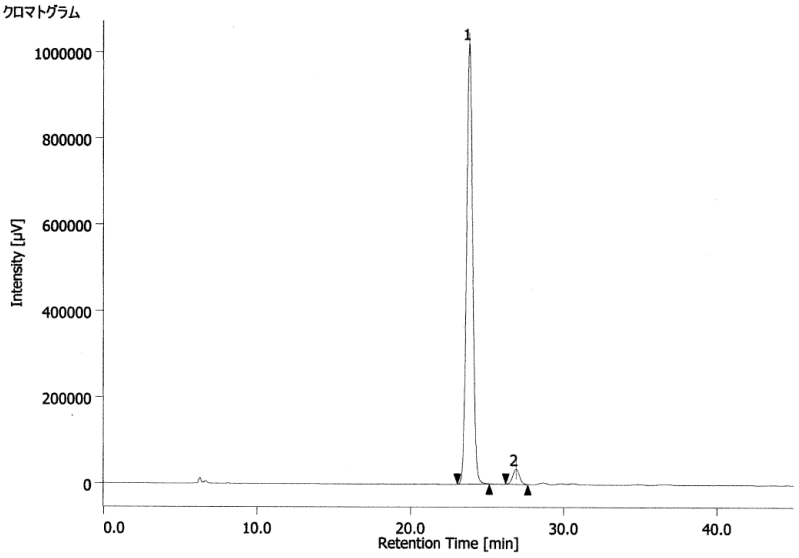


rac-**2g**



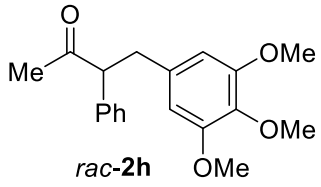


2h

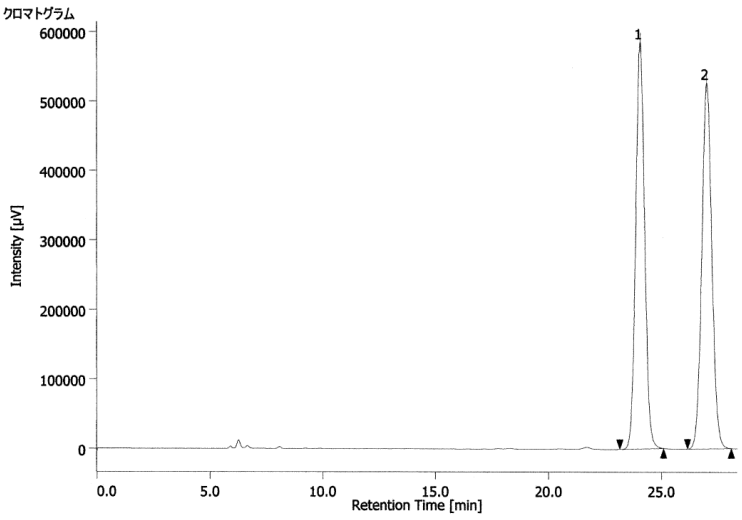


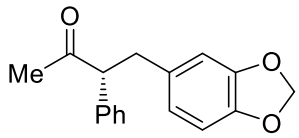
ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	23.858	28004449	1022506	96.301	96.683
2	1	26.892	1075787	35077	3.699	3.317

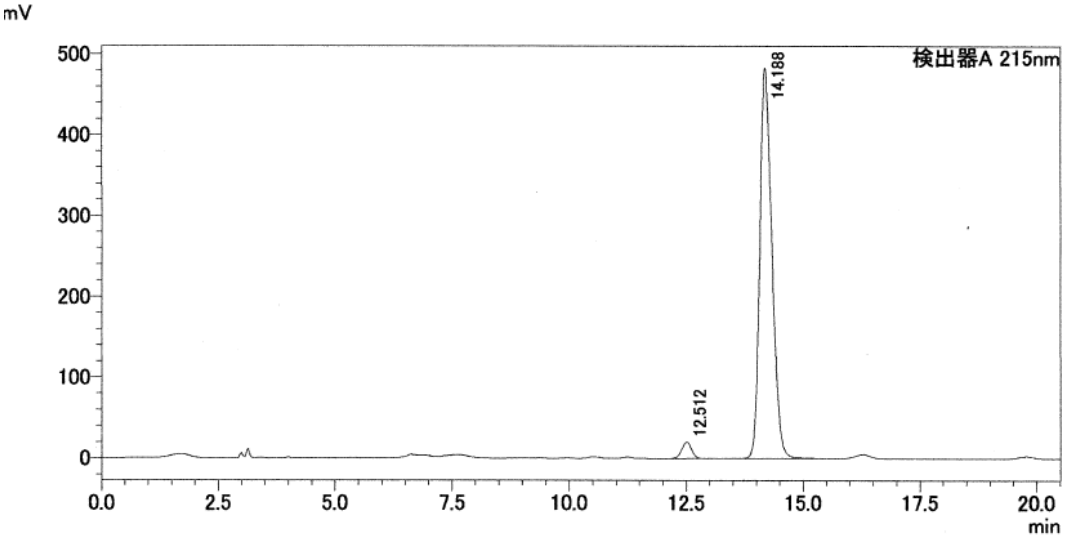


rac-**2h**



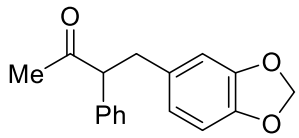


2i

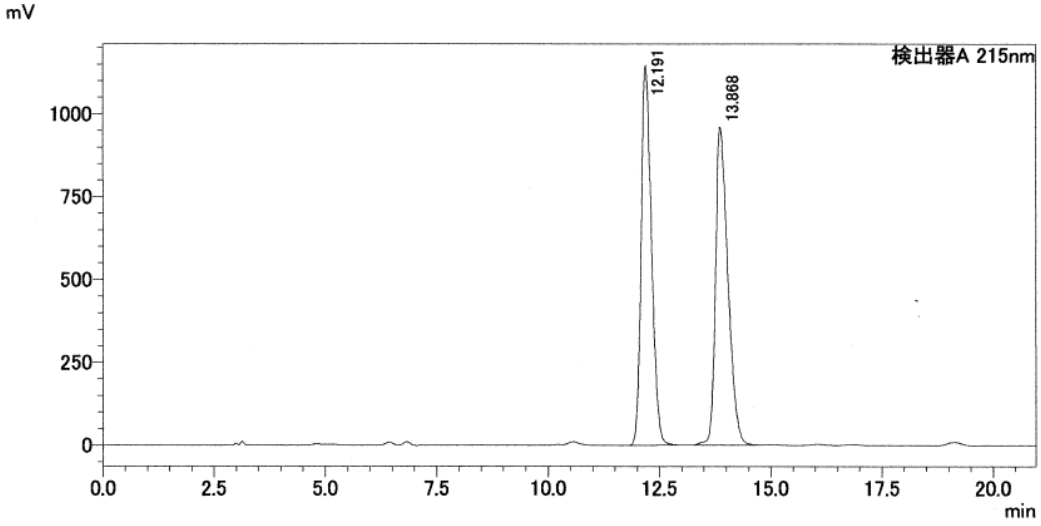


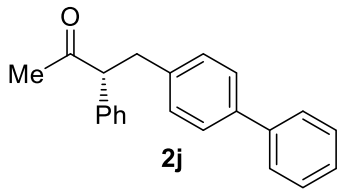
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	12.512	303433	20343	3.382			
2	14.188	8667260	482779	96.618			
合計		8970693	503122				

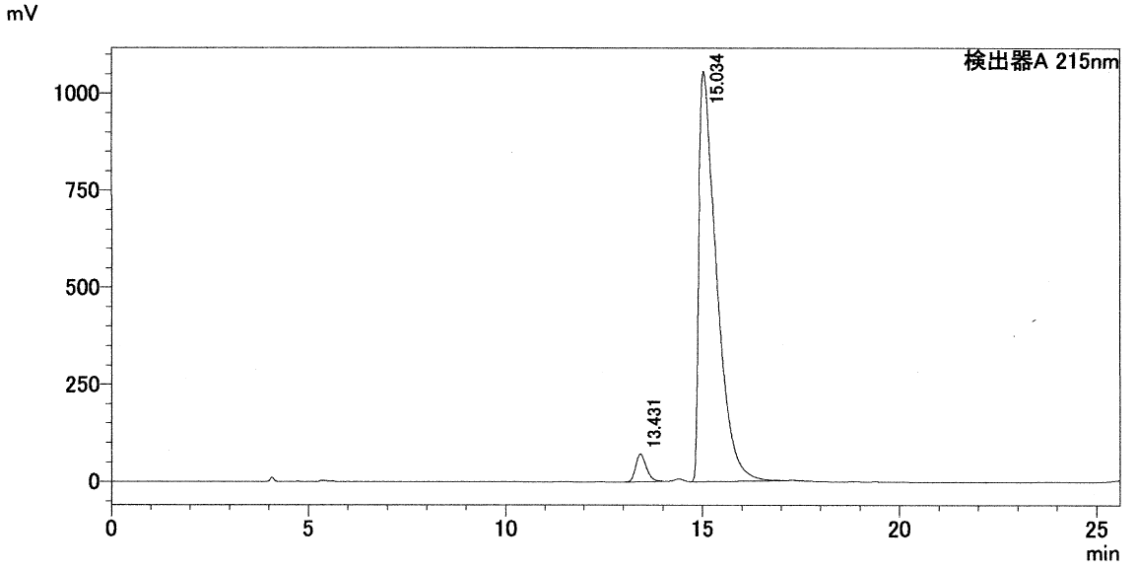


rac-**2i**



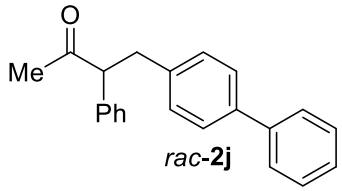


2j

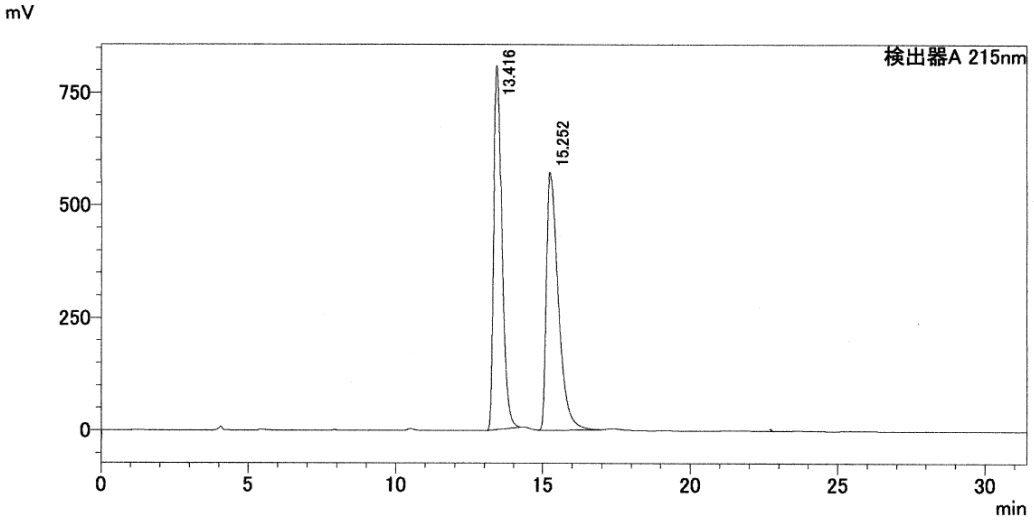


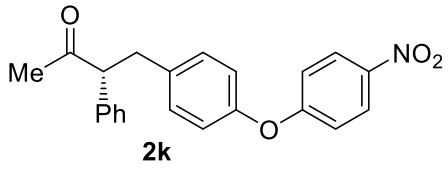
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	13.431	1312220	70644	3.837			
2	15.034	32884389	1056371	96.163			
合計		34196609	1127015				

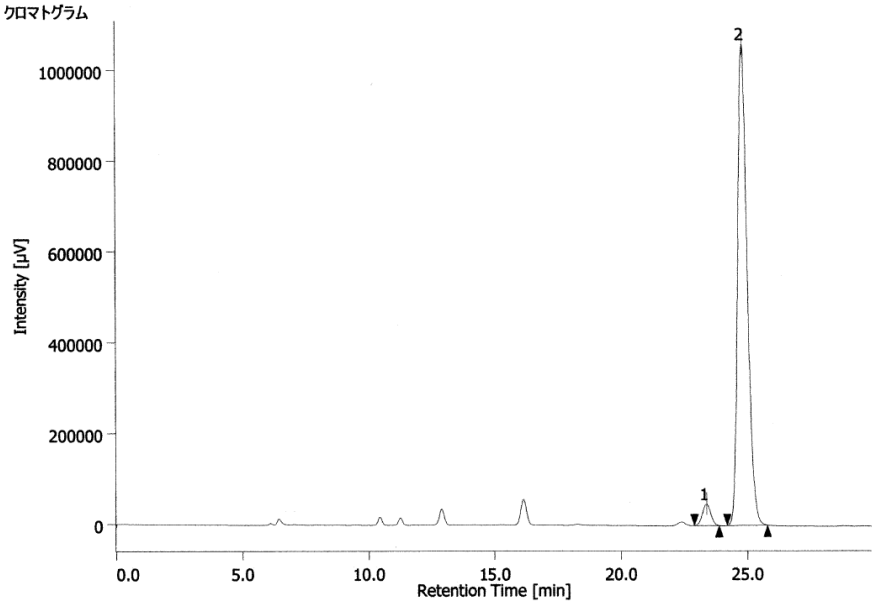


rac-**2j**



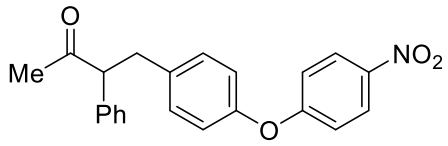


2k

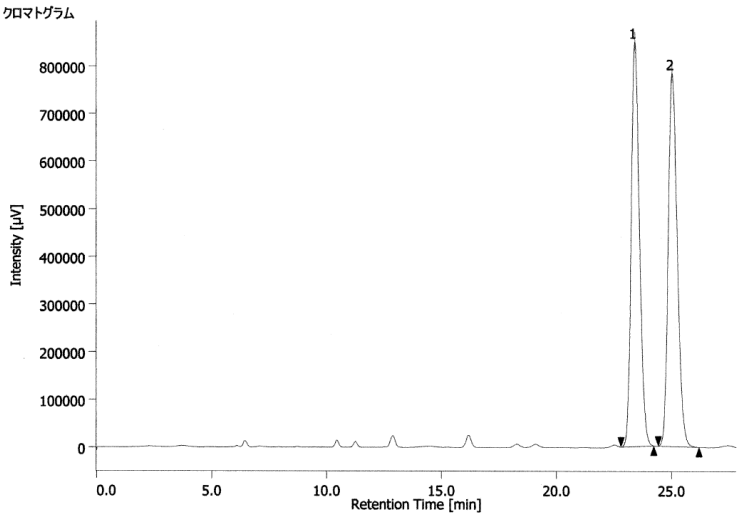


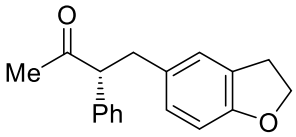
ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	23.358	1097756	47774	3.797	4.314
2	1	24.792	27814514	1059776	96.203	95.686

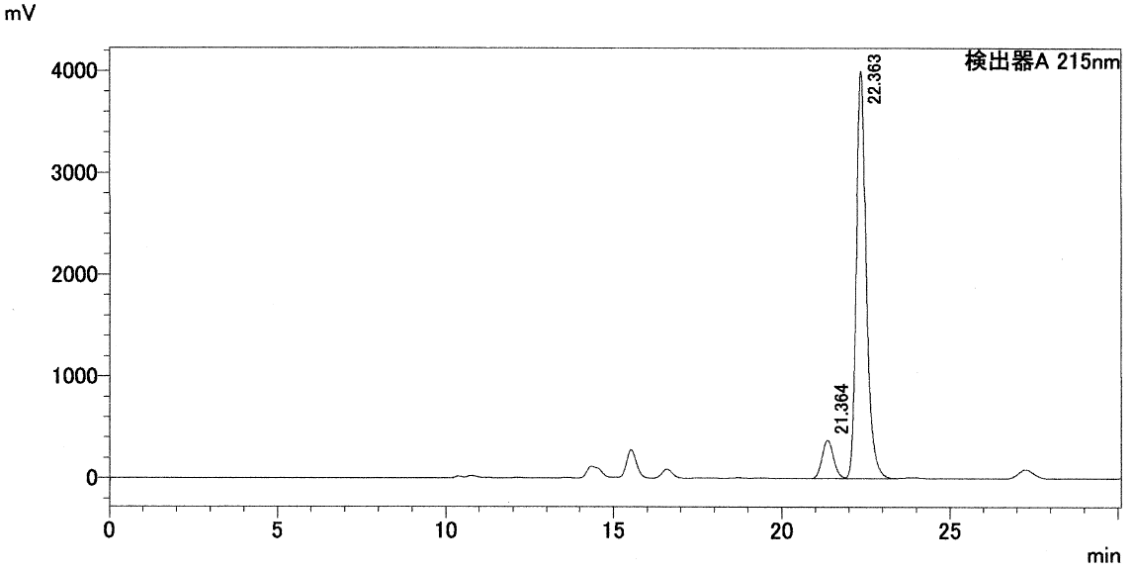


rac-**2k**



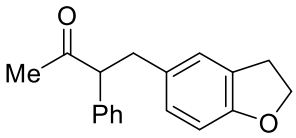


2l

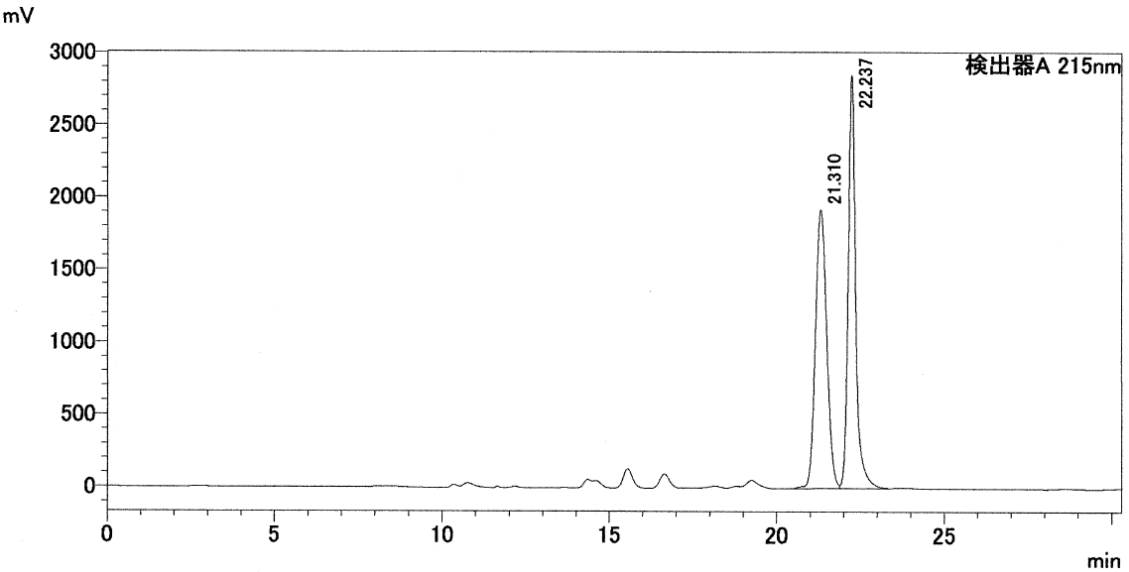


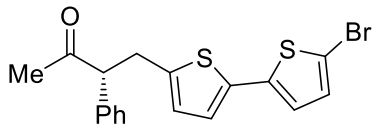
検出器A 215nm

ﾋﾟｰｸ#	保持時間	面積	高さ	濃度	単位	ﾏｰｸ	化合物名
1	21.364	9022241	376133	9.935			
2	22.363	81789261	3997662	90.065		V	
合計		90811502	4373795				

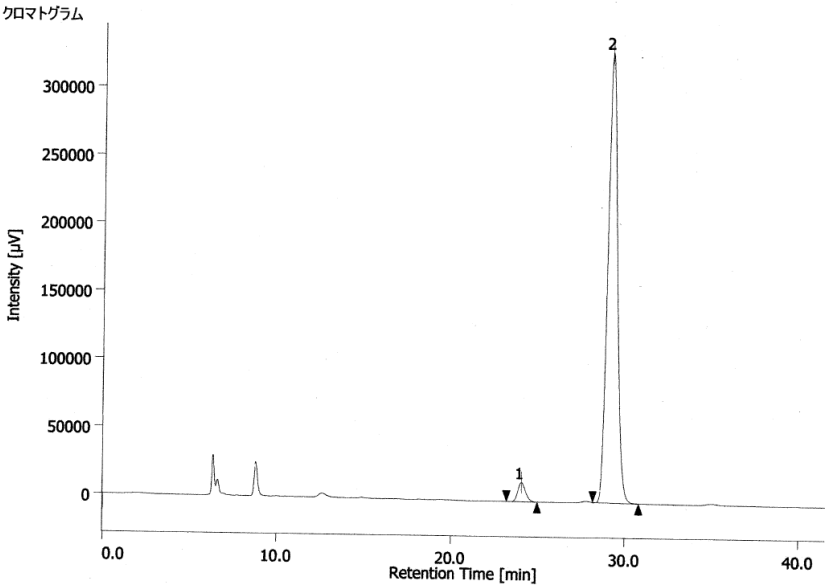


rac-**2l**



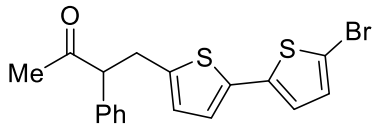


2m

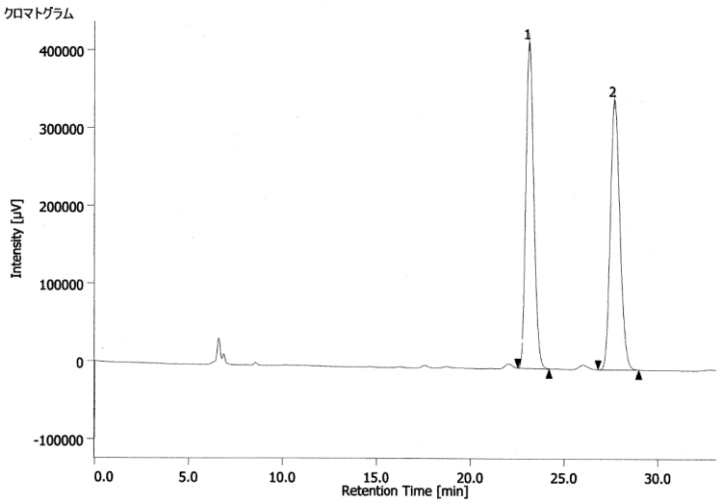


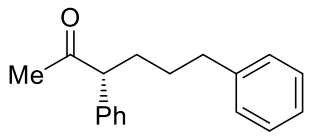
ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	24.067	429255	14083	3.282	4.081
2	1	29.133	12649864	331023	96.718	95.919

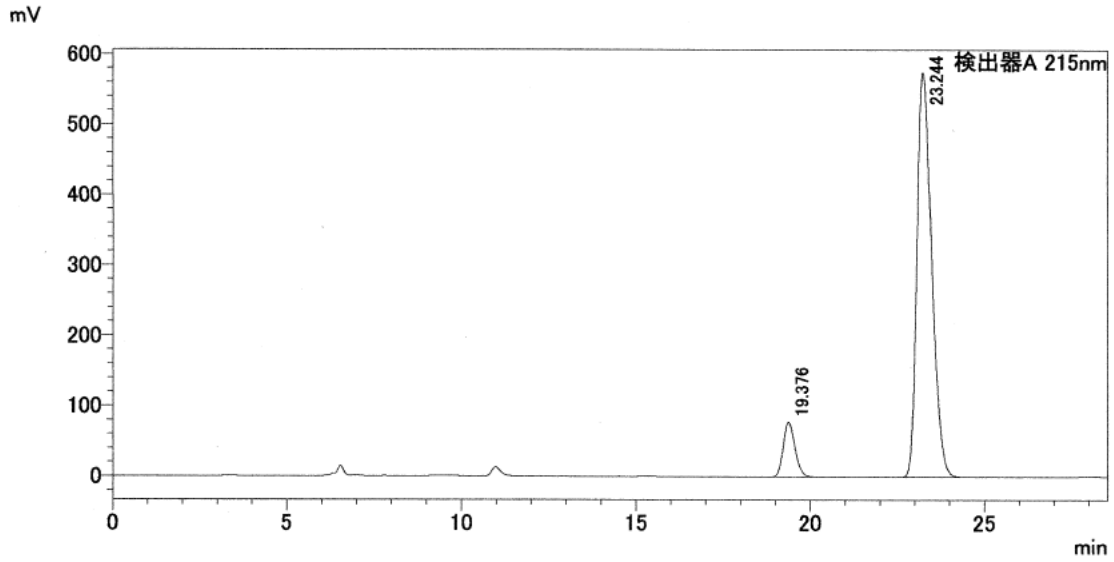


rac-**2m**



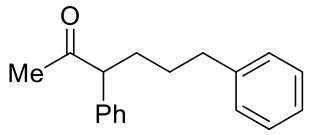


2n

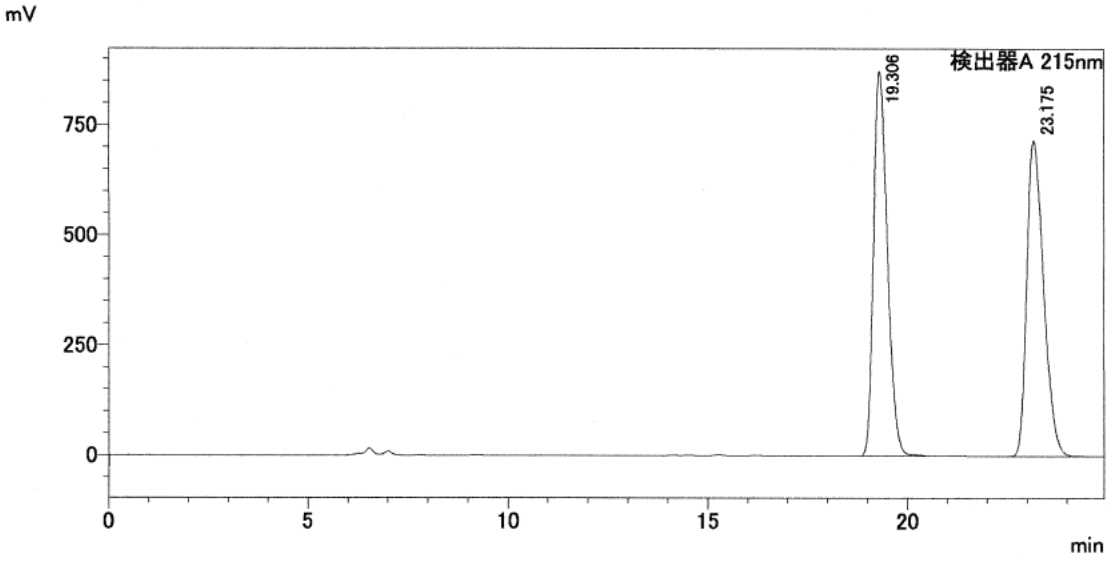


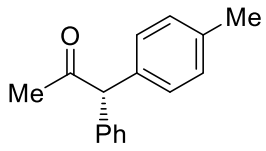
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	19.376	1838038	77569	9.809			
2	23.244	16900772	574708	90.191			
合計		18738810	652277				

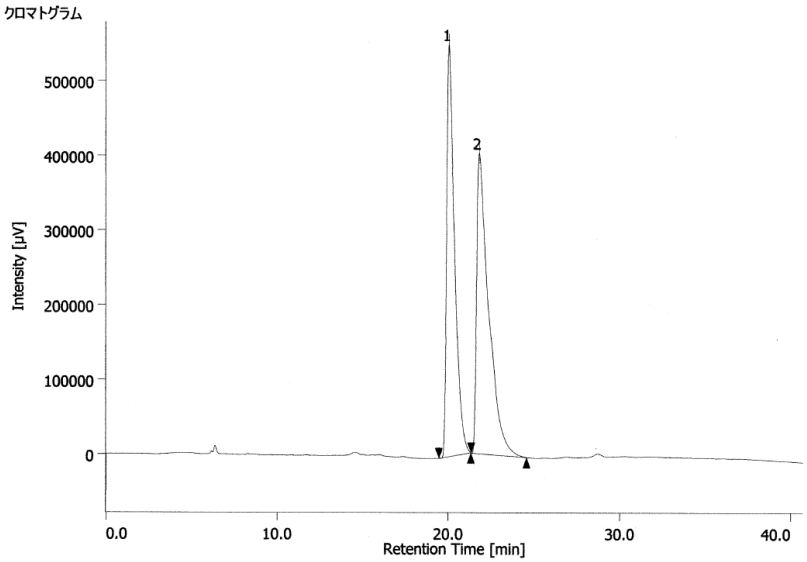


rac-**2n**



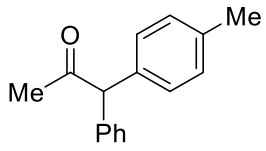


2o

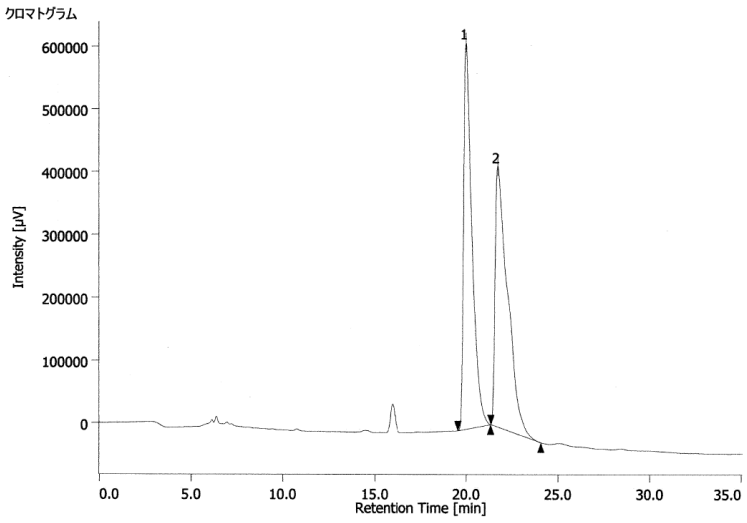


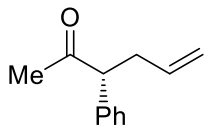
ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	20.050	17567995	552155	47.319	57.808
2	1	21.800	19558944	402996	52.681	42.192

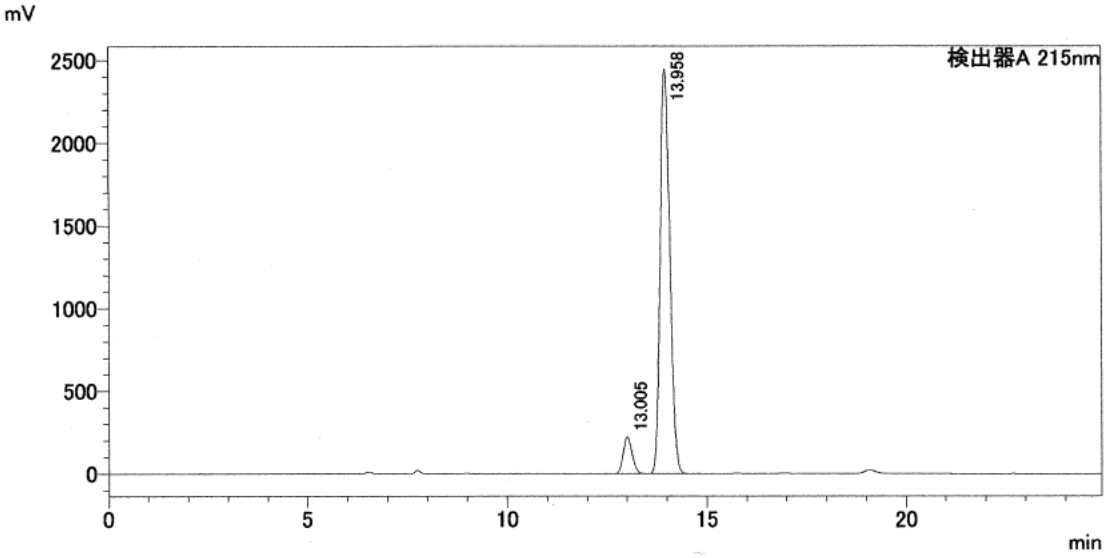


rac-**2o**



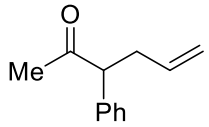


2p

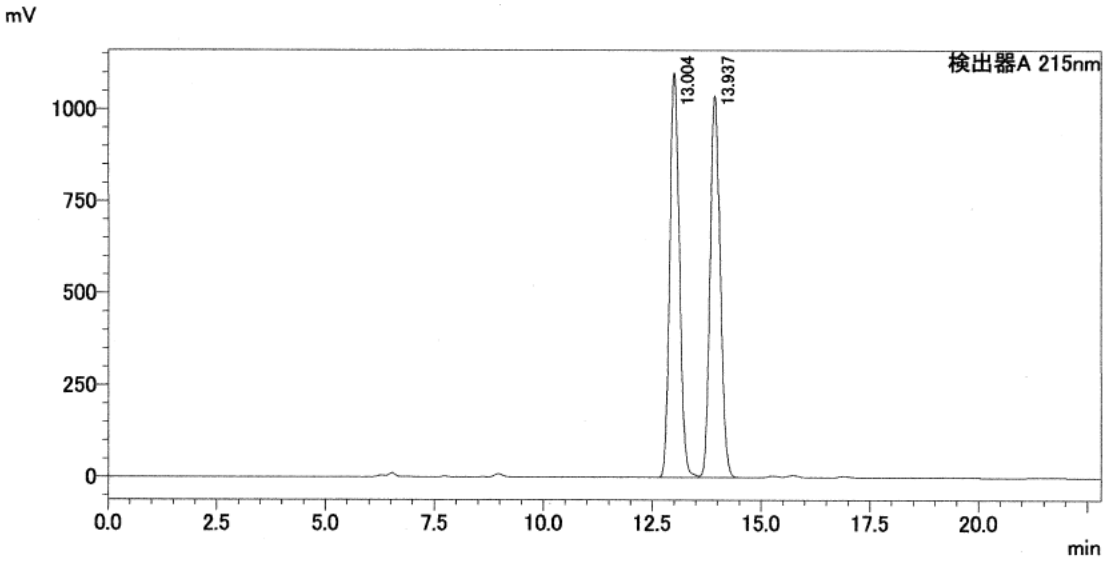


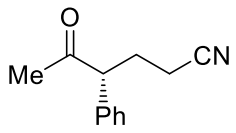
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	13.005	3402608	222128	7.536			
2	13.958	41748293	2446955	92.464		V	
合計		45150901	2669083				

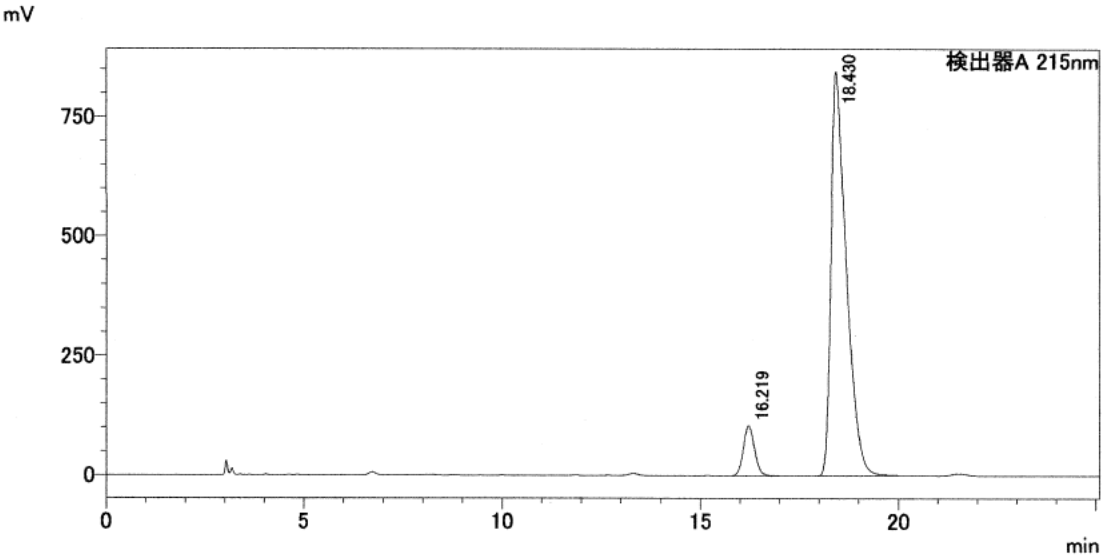


rac-**2p**



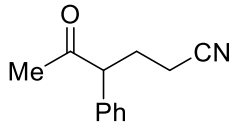


2q

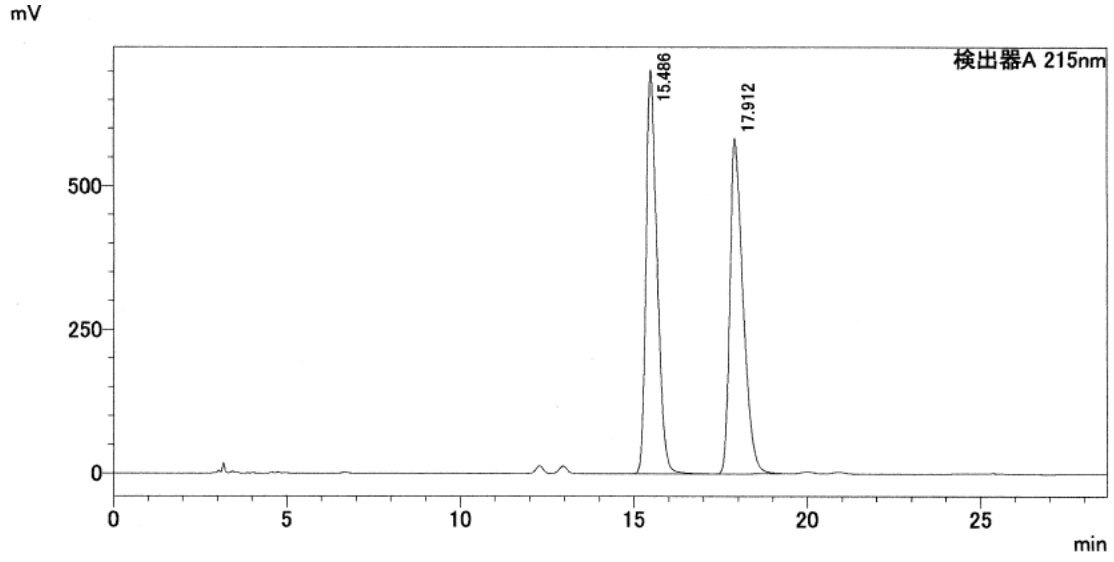


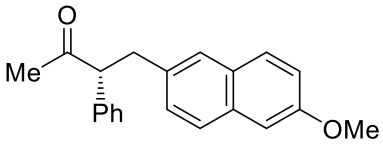
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	16.219	2070363	104688	8.316			
2	18.430	22826527	844659	91.684			
合計		24896890	949347				

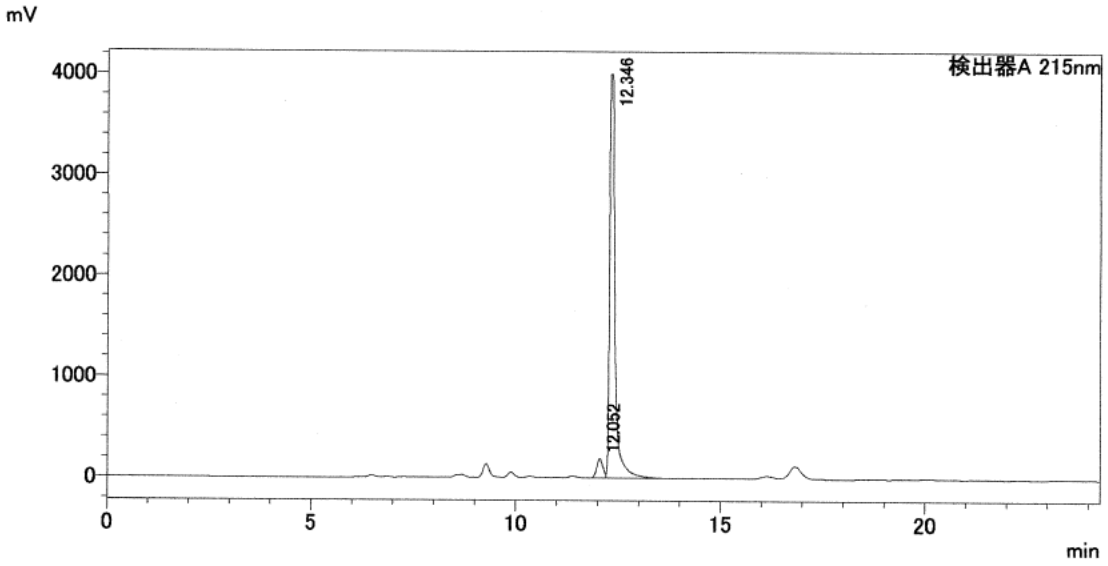


rac-**2q**



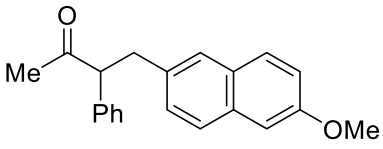


2r

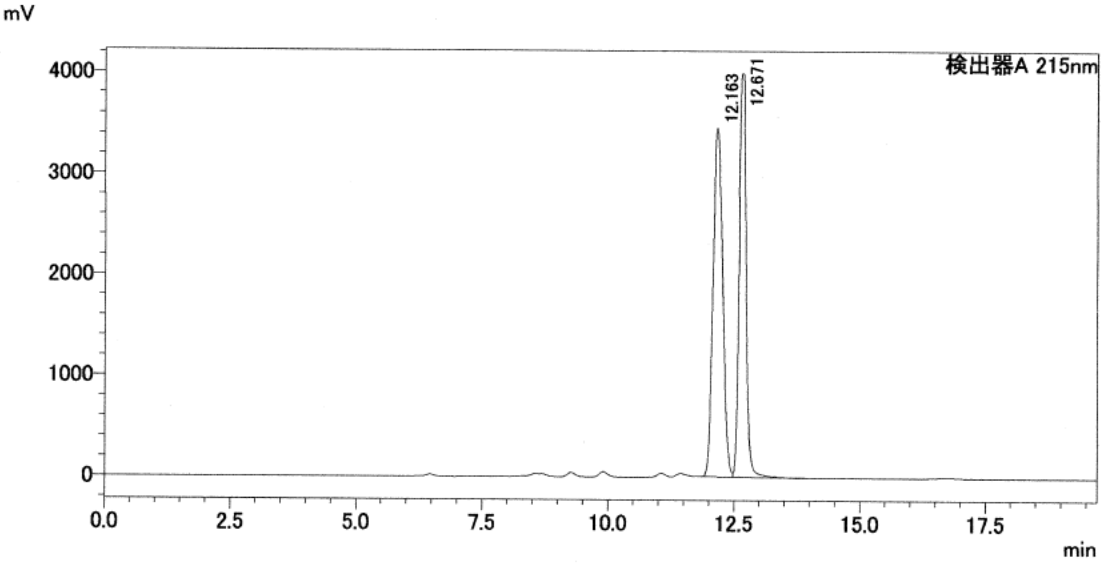


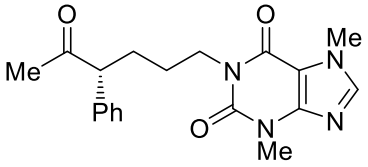
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	12.052	2088119	194969	5.421			
2	12.346	36434207	3999171	94.579		V	
合計		38522326	4194140				

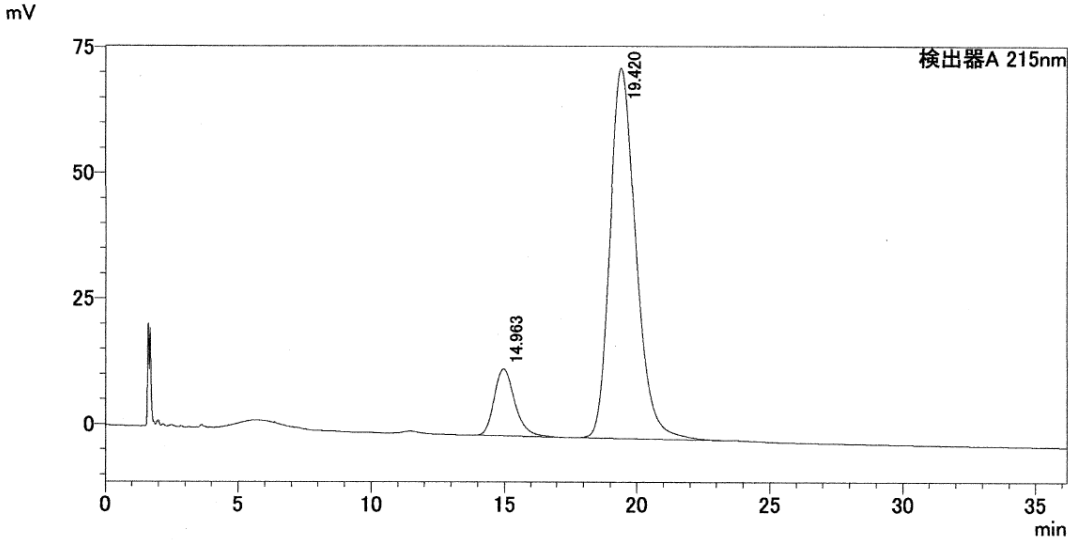


rac-**2r**



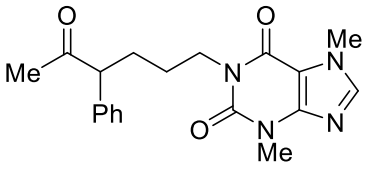


2s

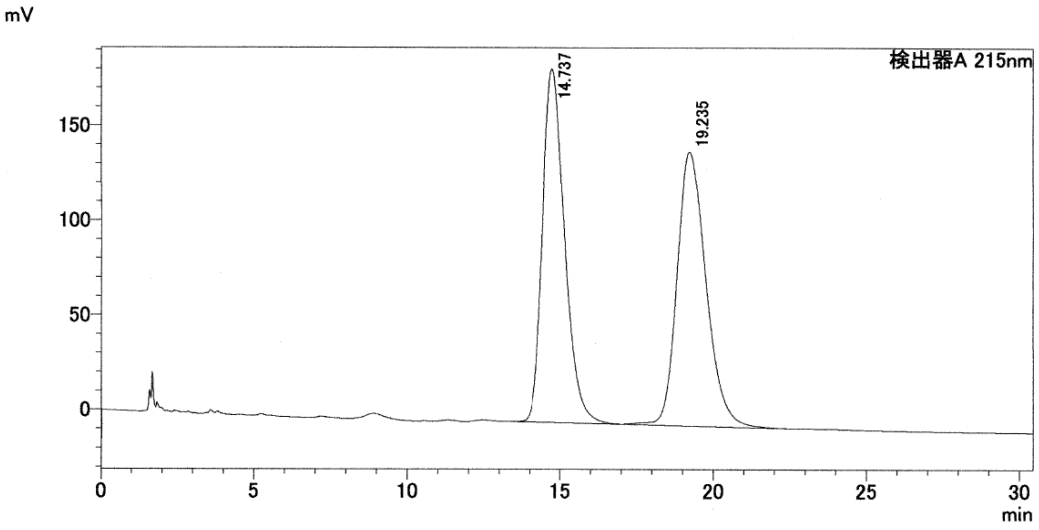


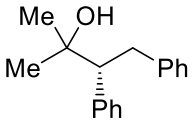
検出器A 215nm

ピーク#	保持時間	面積	高さ	濃度	単位	マーク	化合物名
1	14.963	724244	13312	12.635			
2	19.420	5007997	73609	87.365			
合計		5732241	86921				

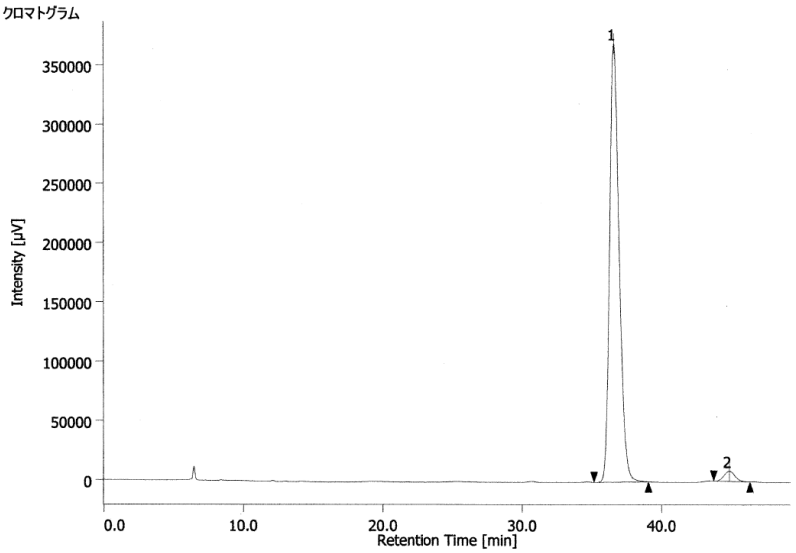


rac-**2s**



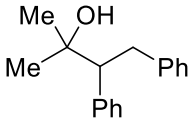


4

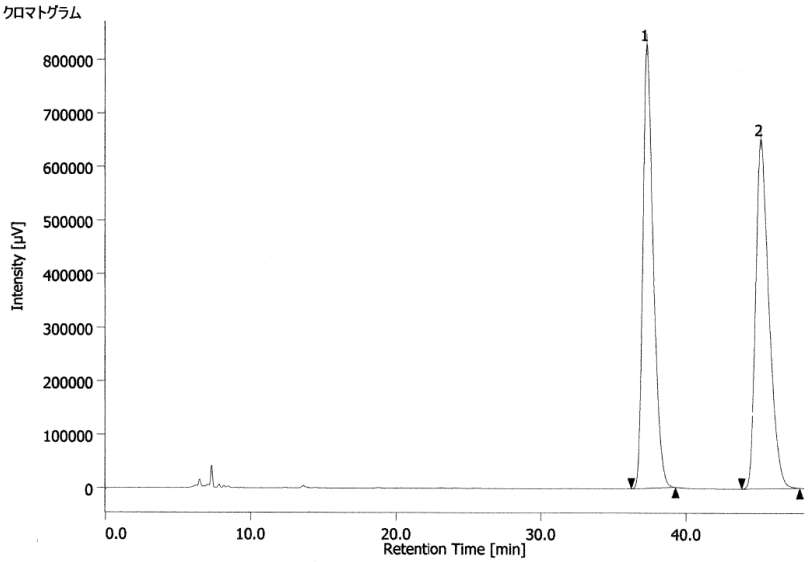


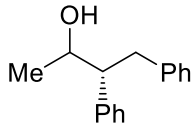
ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	36.575	17417317	369939	97.380	97.716
2	1	44.833	468621	8647	2.620	2.284

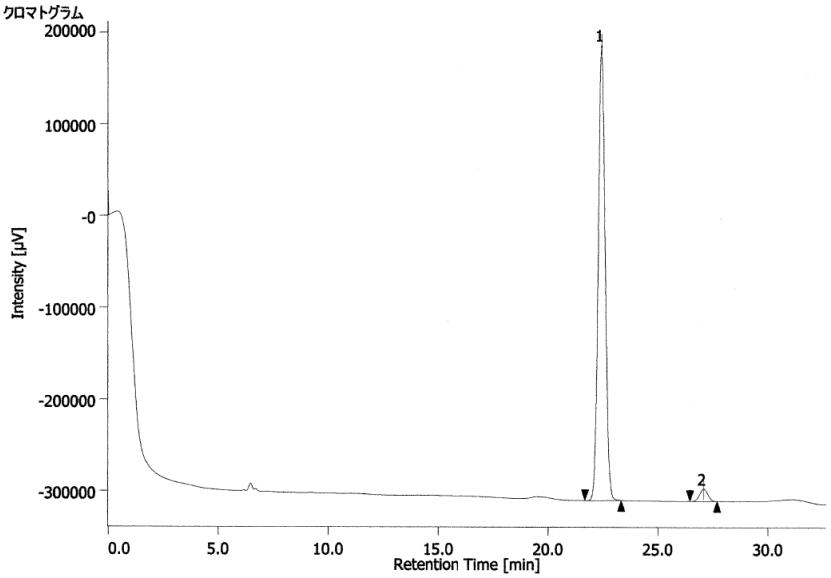


rac-**4**



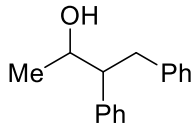


5 (major)



ピーク情報

ピークNo.	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%
1	1	22.425	11082620	496291	96.843	97.332
2	1	27.067	361249	13603	3.157	2.668



rac-**5** (major)