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Supporting Information

69451 Weinheim, Germany

Alternative Pathways for Heck Intermediates: Palladium-Catalyzed Oxyarylation of Homoallylic Alcohols**

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anie_201101857_sm_miscellaneous_information.pdf

1. General Procedures

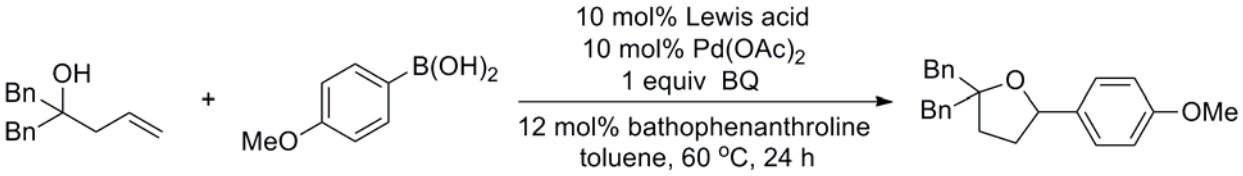
All reactions were maintained under an argon atmosphere unless otherwise stated. Anhydrous solvents (THF, DME, benzene) were freshly distilled from sodium benzophenone ketyl or from CaH_2 (CH_2Cl_2, toluene) under argon. Commercially available reagents were used without further purification. Flash chromatography (FC) was performed using E. Merck silica gel 60 (240–400 mesh). Thin layer chromatography (TLC) was performed using pre-coated plates purchased from E. Merck (silica gel 60 PF254, 0.25 mm). NMR spectra were recorded in $CDCl_3$, unless otherwise stated, on spectrometers at operating frequencies of 300/400/500 MHz (^{1}H) or 75/100/125 MHz (^{13}C) as indicated in the individual spectrum. Chemical shifts (δ) are given in ppm relative to residual solvent (usually chloroform δ = 7.26 for ^{1}H NMR or δ = 77.3 for proton decoupled ^{13}C NMR) and coupling constants (*J*) in Hz. Multiplicity is tabulated as s for singlet, d for doublet, t for triplet, q for quadruplet, and m for multiplet. Low resolution LC/MS spectra were obtained with an Agilent 1200 series API-LC/MSD spectrometer. High resolution mass spectral analyses were kindly provided by Professor Kasem Nithipatikom at the Medical College of Wisconsin Mass Spectroscopy Facility or at the Mass Spectrometry & Proteomic Facility, University of Notre Dame.

Compound **1b** and **1c** were purchased from Sigma-Aldrich. Compound **1e**, **1f**, *Z*-**1g**, *E*-**1g** and **1h** were purchased from Alfa Aesar. Compound **1j** was purchased from Nu-Chek Prep, Inc. Compound **1a**, **1d** and **4** were prepared according to the reported procedure.[1]

2. Survey of Reaction Conditions for the Oxyarylation of Olefins

[1] Schmidt, B. *J. Org. Chem.* **2004**, *69*, 7672-7687.

a. Screening of Lewis acids

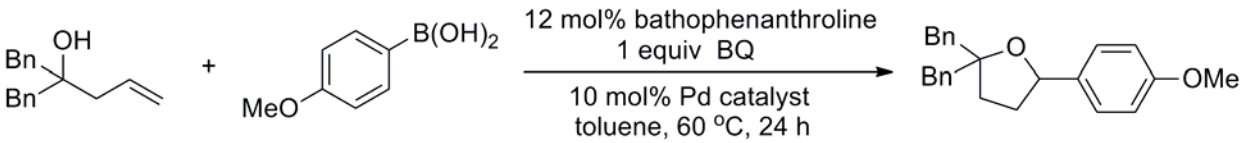


0.1 mmol 0.12 mmol

additive	none	$AuCl_3$	AuCl	Ag(TFA)	Ag(OTf)	$AgSbF_6$	Cp_2ZrCl_2
yield	< 10%	< 10%	< 10%	< 10%	0%	0%	0%

additive	$Cu(OAc)_2$	$CuCl_2$	$NiCl_2$	$Zn(OTf)_2$	$Yb(OTf)_3$	$RuCl_3$	$InCl_3$
yield	20%	0%	25%	20%	27%	32%	30%

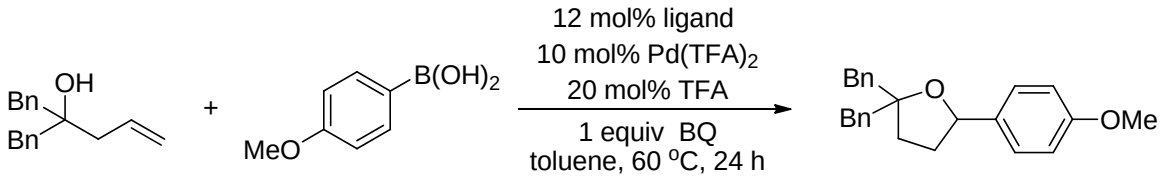
b. Screening of catalysts



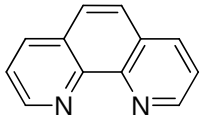
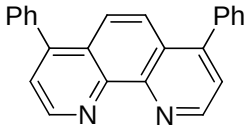
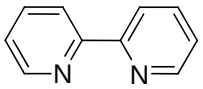
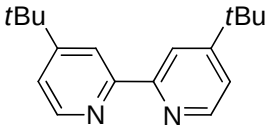
0.1 mmol 0.12 mmol

catalyst	10 mol% $Pd(OAc)_2$	10 mol% $Pd(OAc)_2$ 20 mol% TFA	10 mol% $Pd(OAc)_2$ 50 mol% TFA
yield	15%	< 10%	< 10%
catalyst	10 mol% $Pd(TFA)_2$	10 mol% $Pd(TFA)_2$ 20 mol% TFA	10 mol% $Pd(TFA)_2$ 50 mol% TFA
yield	< 10%	46%	40%

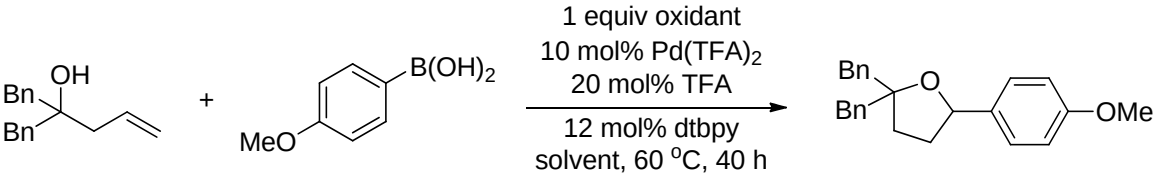
c. Screening of ligands



0.1 mmol 0.12 mmol

ligand				
yield	< 10%	46%	< 10%	50%

d. Screening of solvents and oxidants

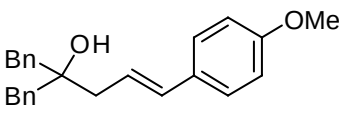


0.1 mmol 0.12 mmol

solvent	toluene	THF	CH_3CN	DMSO	DCE	DCE	DCE
oxidant	BQ	BQ	BQ	BQ	BQ	O_2	Air
yield	65%	< 10%	46%	15%	75%	< 10%	< 10%

3. Representative Procedure: Oxidative Heck Reaction

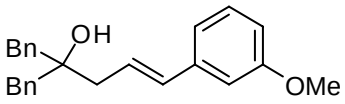
Compound **3a**



Homoallylic alcohol **1a** (25.2 mg, 0.1 mmol), boronic acid **2a** (18.2 mg, 0.12 mmol), benzoquinone (11 mg, 0.1 mmol), $Pd(TFA)_2$ (3.3 mg, 0.01 mmol) and bathophenanthroline (4.0 mg,

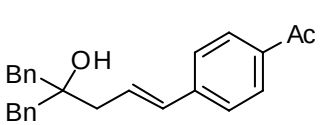
0.012 mmol) were loaded into a dry vial which was subjected to evacuation/flushing with dry argon three times. Anhydrous toluene (0.8 mL) was syringed into the mixture which was then stirred at 60 °C for 20 h or until the starting material had been consumed as determined by TLC. Upon cooling to room temperature, all volatiles were evaporated and the residue was purified by preparative TLC to give **3a** as yellow oil. *E*:*Z* = 13:1. *E*-isomer: ^{1}H NMR (500 MHz) δ 2.30 (d, *J* =7.0 Hz, 2H), 2.87 (s, 4H), 3.83 (s, 3H), 6.15 (ddd, *J* = 7.5, 7.5, 15.5 Hz, 1H), 6.37 (d, *J* = 16.0 Hz, 1H), 6.88 (d, *J* = 9.0 Hz, 2H), 7.19-7.35 (m, 12H); ^{13}C NMR (125 MHz) δ 42.6, 46.1, 55.56, 55.59, 74.5, 114.2, 123.4, 126.8, 127.5, 128.5, 130.4, 131.1, 133.6, 137.5, 159.2. FT-IR (CH_2Cl_2) 3559, 3028, 2919, 2836, 2361, 2343, 1607, 1511, 1495, 1454, 1298, 1248, 1175, 1113, 1086, 1033, 970, 888, 834, 753, 725, 702 cm^{-1}. HRMS calcd for $C_{25}H_{27}O_2$ $[M+1]^+$ 359.2006, found 359.2000.

Compound **3b**



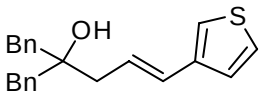
E:*Z* = 20:1. *E*-isomer: ^{1}H NMR (500 MHz) δ 2.33 (d, *J* = 7.0 Hz, 2H), 2.88 (s, 4H), 3.84 (s, 3H), 6.29 (ddd, *J* = 7.0, 7.0, 14.5 Hz, 1H), 6.40 (d, *J* = 16.0 Hz, 1H), 6.81 (dd, *J* = 2.5, 8.0 Hz, 1H), 6.91 (s, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 7.23-7.36 (m, 11H); ^{13}C NMR (125 MHz) δ 42.6, 46.2, 55.5, 74.5, 111.8, 113.1, 119.1, 126.2, 126.8, 128.5, 129.8, 131.0, 134.0, 137.4, 139.1, 160.1. FT-IR (CH_2Cl_2) 3558, 3027, 2919, 2835, 1599, 1579, 1494, 1453, 1433, 1289, 1264, 1155, 1086, 1045, 971, 875, 753, 722, 702 cm^{-1}. HRMS calcd for $C_{25}H_{27}O_2$ $[M+1]^+$ 359.2006, found 359.1991.

Compound **3c**



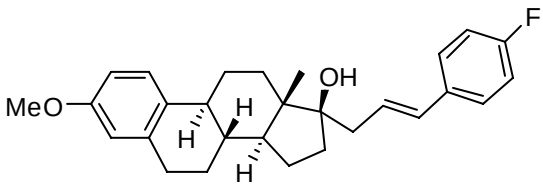
E:*Z* = 20:1. *E*-isomer: ^{1}H NMR (500 MHz) δ 2.37 (d, *J* = 5.5 Hz, 2H), 2.61 (s, 3H), 2.88 (s, 4H), 6.44-6.46 (m, 2H), 7.27-7.36 (m, 10H), 7.43 (d, *J* = 8.0 Hz, 2H), 7.92 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz) 26.8, 42.8, 46.3, 74.5, 126.4, 126.9, 128.6, 129.0, 129.4, 131.0, 133.0, 136.0, 137.3, 142.3, 197.8. FT-IR (CH_2Cl_2) 3558, 3028, 2919, 1678, 1602, 1494, 1410, 1358, 1268, 1181, 1086, 970, 754, 703 cm^{-1}. HRMS calcd for $C_{26}H_{27}O_2$ $[M+1]^+$ 371.2006, found 371.2012.

Compound **3d**



E:*Z* = 13:1. *E*-isomer: ^{1}H NMR (500 MHz) δ 2.30 (d, *J* = 7.5 Hz, 2H), 2.87 (s, 4H), 3.74 (s, 1H), 6.13 (ddd, *J* = 7.5, 7.5, 15.0 Hz, 1H), 6.44 (d, *J* = 16.0 Hz, 1H), 7.10 (s, 1H), 7.21-7.36 (m, 12H); ^{13}C NMR (125 MHz) 42.5, 46.2, 74.5, 121.5, 125.2, 125.7, 126.2, 126.8, 128.4, 128.5, 131.0, 137.5, 140.3. FT-IR (CH_2Cl_2) 3557, 3028, 2920, 1602, 1494, 1454, 1085, 1031, 967, 862, 788, 754, 728, 702 cm^{-1}. HRMS calcd for $C_{22}H_{23}OS$ $[M+1]^+$ 335.1464, found 335.1482.

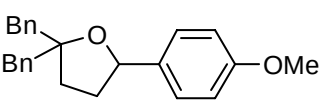
Compound **5**



E:*Z* = 20:1. *E*-isomer: ^{1}H NMR (500 MHz) δ 0.96 (s, 3H), 1.34-1.73 (m, 9H), 1.90-1.95 (m, 1H), 2.04-2.09 (m, 1H), 2.18-2.21 (m, 1H), 2.35-2.43 (m, 2H), 2.54 (dd, *J* = 6.5, 14.0 Hz, 1H), 2.87-2.91 (m, 2H), 3.80 (s, 3H), 6.35 (ddd, *J* = 7.5, 7.5, 15.0 Hz, 1H), 6.50 (d, *J* = 16.0 Hz, 1H), 6.66 (s, 1H), 6.73 (d, *J* = 8.5 Hz, 1H), 7.01 (dd, *J* = 8.5, 8.5 Hz, 2H), 7.23 (d, *J* = 8.5 Hz, 1H), 7.36 (dd, *J* = 5.5, 8.5 Hz, 2H); ^{13}C NMR (125 MHz) 14.6, 23.7, 26.6, 27.8, 30.1, 32.1, 35.4, 39.9, 41.2, 44.1, 46.9, 49.9, 55.5, 83.3, 111.7, 114.1, 115.6 (d, $J_{C\text{-}F}$ = 21.1 Hz), 126.5, 126.6, 127.8 (d, $J_{C\text{-}F}$ = 8.2 Hz), 132.8, 132.9, 133.9 (d, $J_{C\text{-}F}$ = 3.2 Hz), 138.2, 157.7, 162.3 (d, $J_{C\text{-}F}$ = 245 Hz). FT-IR (CH_2Cl_2) 3412, 2932, 2871, 1606, 1508, 1468, 1452, 1290, 1251, 1233, 1158, 1100, 1041, 972, 902, 846, 820, 786, 728 cm^{-1}. HRMS calcd for $C_{28}H_{33}FO_2Na$ $[M+Na]^+$ 443.2357, found 443.2344.

4. Representative Procedure: Oxyarylation of Olefins

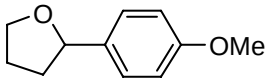
Compound **6a**



Homoallylic alcohol **1a** (25.2 mg, 0.1 mmol), boronic acid **2a** (18.2 mg, 0.12 mmol), benzoquinone (11 mg, 0.1 mmol), $Pd(TFA)_2$ (3.3 mg, 0.01 mmol) and dtbpy (3.2 mg, 0.012 mmol) were loaded into a dry vial which was subjected to evacuation/flushing with dry argon three times. Anhydrous dichloroethane (0.8 mL) followed by trifluoroacetic acid (1.5 μL, 0.02

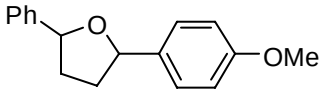
mmol) were syringed into the mixture which was then stirred at 60 °C for 40 h or until the starting material had been consumed as determined by TLC. Upon cooling to room temperature, all volatiles were evaporated and the residue was purified by preparative TLC to give **6a** as pale yellow oil. ^{1}H NMR (400 MHz) δ 1.37-1.43 (m, 1H), 1.85-1.90 (m, 1H), 1.90-2.01 (m, 2H), 2.86 (d, 2.93, *J* = 13.6 Hz, 1H), 2.92 (d, *J* = 13.6 Hz, 1H), 2.99 (d, *J* = 13.2 Hz, 1H), 3.01 (d, *J* = 13.6 Hz, 1H), 3.80 (s, 3H), 4.47 (dd, *J* = 5.2, 9.6 Hz, 1H), 6.84 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.23-7.34 (m, 10H); ^{13}C NMR (100 MHz) δ 33.5, 34.6, 46.1, 47.1, 55.5, 81.1, 85.7, 113.8, 126.4, 126.5, 128.0, 128.1, 128.2, 131.2, 131.3, 134.6, 138.3, 138.4, 159.2. FT-IR (CH_2Cl_2) 3060, 3027, 2936, 2835, 1613, 1513, 1494, 1454, 1302, 1246, 1173, 1082, 1035, 942, 827, 754, 736, 701 cm^{-1}. HRMS calcd for $C_{25}H_{27}O_2$ $[M+1]^+$ 359.2006, found 359.2016.

Compound **6b**[2]



^{1}H NMR (500 MHz) δ 1.69-1.75 (m, 1H), 1.91-1.97 (m, 2H), 2.18-2.22 (m, 1H), 3.73 (s, 3H), 3.83 (dd, *J* = 7.5, 14.5 Hz, 1H), 4.01 (dd, *J* = 7.0, 14.0 Hz, 1H), 4.76 (dd, *J* = 7.0, 7.5 Hz, 1H), 6.80 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 2H).

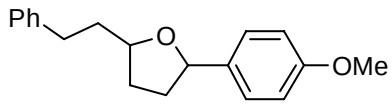
Compound **6c**



Mixture of two diastereomers (d.r. = 1.4:1). ^{1}H NMR (500 MHz) δ 1.97-2.05 (m, 4H), 2.40-2.52 (m, 4H), 3.83 (s, 3H), 3.84 (s, 3H), 5.02-5.07 (m, 2H), 5.21-5.29 (m, 2H), 6.91-6.94 (m, 4H), 7.27-7.32 (m, 2H), 7.35-7.47 (m, 12H); ^{13}C NMR (125 MHz) δ 34.4, 34.6 (two isomers), 35.4, 35.5 (two isomers), 55.5, 55.5 (two isomers), 81.2, 81.2 (two isomers), 81.3, 81.3 (two isomers), 114.2, 114.2 (two isomers), 125.7, 126.1 (two isomers), 127.0, 127.4 (two isomers), 127.2, 127.3 (two isomers), 128.4, 128.5 (two isomers), 135.4, 136.1 (two isomers), 143.4, 144.1 (two isomers), 159.3, 159.4 (two isomers). FT-IR (CH_2Cl_2) 2936, 2835, 1612, 1586, 1513, 1452, 1302, 1244, 1173, 1029, 937, 827, 753, 699 cm^{-1}. HRMS calcd for $C_{17}H_{18}O_2Na$ $[M+Na]^+$ 277.1199, found 277.1203.

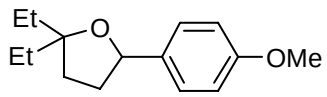
[2] Protti, S.; Dondi, D.; Fagnoni, M.; Albini, A. *Eur. J. Org. Chem.* **2008**, 2240–2247.

Compound **6d**



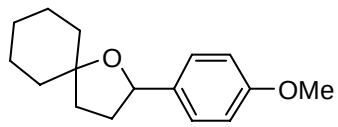
Mixture of two diastereomers (d.r. = 1.2:1). ^{1}H NMR (500 MHz) δ 1.65-1.72 (m, 2H), 1.78-1.92 (m, 4H), 1.98-2.16 (m, 4H), 2.22-2.35 (m, 2H), 2.68-2.85 (m, 4H), 3.80 (s, 6H), 4.00-4.04 (m, 1H), 4.18-4.22 (m, 1H), 4.83 (dd, *J* = 7.0, 7.0 Hz, 1H), 4.96 (dd, *J* = 7.0, 7.0 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 4H), 7.15-7.30 (m, 14H); ^{13}C NMR (125 MHz) δ 31.6, 32.7 (two isomers), 32.8, 32.8 (two isomers), 34.7, 35.7 (two isomers), 38.1, 38.2 (two isomers), 55.5, 55.6 (two isomers), 79.3, 79.4 (two isomers), 80.2, 80.9 (two isomers), 113.9, 114.0 (two isomers), 126.0, 126.0 (two isomers), 127.2, 127.4 (two isomers), 128.6, 128.6 (two isomers), 128.7, 128.7 (two isomers), 135.6, 136.0 (two isomers), 142.5, 142.5 (two isomers), 159.0, 159.1 (two isomers). FT-IR (CH_2Cl_2) 3061, 3026, 2934, 2861, 2835, 1613, 1585, 1513, 1454, 1302, 1246, 1173, 1035, 829, 747, 699 cm^{-1}. HRMS calcd for $C_{19}H_{23}O_2$ $[M+1]^+$ 283.1693, found 283.1696.

Compound **6e**



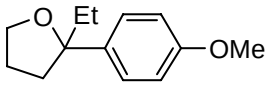
^{1}H NMR (500 MHz) δ 0.94 (t, *J* = 7.5 Hz, 3H), 0.98 (t, *J* = 7.5 Hz, 3H), 1.57-1.70 (m, 4H), 1.78-1.88 (m, 3H), 2.20-2.23 (m, 1H), 3.80 (s, 3H), 4.86 (dd, *J* = 5.5, 9.0 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (100 MHz) δ 8.8, 9.0, 31.2, 31.6, 35.1, 36.0, 55.5, 80.5, 86.2, 113.9, 127.4, 135.6, 159.0. FT-IR (CH_2Cl_2) 2964, 1613, 1514, 1462, 1302, 1246, 1172, 1037, 949, 830 cm^{-1}. HRMS calcd for $C_{15}H_{22}O_2Na$ $[M+Na]^+$ 257.1512, found 257.1524.

Compound **6f**



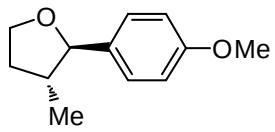
^{1}H NMR (500 MHz) δ 1.37-1.50 (m, 4H), 1.59-1.92 (m, 9H), 2.24-2.27 (m, 1H), 3.80 (s, 3H), 4.92 (dd, *J* = 6.0, 8.5 Hz, 1H), 6.87 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz) δ 24.0, 24.3, 26.0, 35.4, 36.9, 38.2, 38.6, 55.5, 79.8, 83.3, 113.9, 127.3, 136.1, 159.0. FT-IR (CH_2Cl_2) 2930, 2856, 1726, 1650, 1613, 1513, 1446, 1301, 1246, 1172, 1069, 1038, 952, 828 cm^{-1}. HRMS calcd for $C_{16}H_{22}O_2Na$ $[M+Na]^+$ 269.1512, found 269.1529.

Compound **6g**



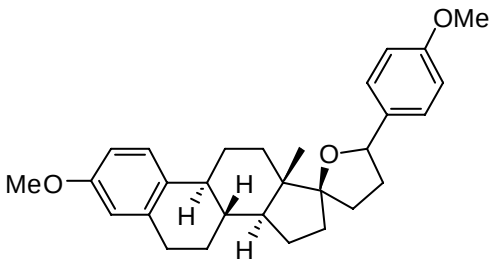
^{1}H NMR (500 MHz) δ 0.77 (t, *J* = 7.5 Hz, 3H), 1.76-1.84 (m, 3H), 1.92-2.05 (m, 2H), 2.13-2.18 (m, 1H), 3.81 (s, 3H), 3.86-3.90 (m, 1H), 3.96 (dd, *J* = 7.0, 14.5 Hz, 1H), 6.87 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz) δ 9.1, 25.8, 35.4, 37.9, 55.5, 67.6, 87.2, 113.5, 126.7, 138.8, 158.2. FT-IR (CH_2Cl_2) 2966, 2934, 2875, 2835, 1610, 1582, 1510, 1463, 1442, 1299, 1247, 1176, 1100, 1037, 911, 830 cm^{-1}. HRMS calcd for $C_{13}H_{19}O_2$ $[M+1]^+$ 207.1380, found 207.1380.

Compound **6h**



trans-Isomer. ^{1}H NMR (500 MHz) δ 1.06 (d, *J* = 7.0 Hz, 3H), 1.68-1.73 (m, 1H), 2.02-2.10 (m, 1H), 2.20-2.24 (m, 1H), 3.81 (s, 3H), 4.00-4.03 (m, 1H), 4.08 (dd, *J* = 8.0, 15.5 Hz, 1H), 4.23 (d, *J* = 8.5 Hz, 1H), 6.89 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz) δ 16.5, 35.2, 42.8, 55.5, 67.9, 87.9, 113.9, 127.7, 134.3, 159.3. FT-IR (CH_2Cl_2) 2958, 2871, 1613, 1514, 1462, 1377, 1302, 1246, 1172, 1035, 920, 828 cm^{-1}. HRMS calcd for $C_{12}H_{17}O_2$ $[M+1]^+$ 193.1223, found 193.1227.

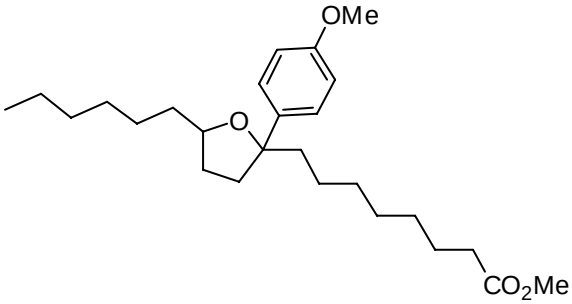
Compound **6i**



Mixture of two diastereomers (d.r. = 1.3:1). ^{1}H NMR (500 MHz) δ 1.00 (s, 3H), 1.05 (s, 3H), 1.34-1.96 (m, 24H), 2.17-2.38 (m, 10H), 2.87-2.92 (m, 4H), 3.81 (s, 6H), 3.82 (s, 3H), 3.83 (s, 3H), 4.88 (dd, *J* = 7.0, 7.0 Hz, 1H), 4.93 (dd, *J* = 7.0, 7.0 Hz, 1H), 6.67 (s, 2H), 6.75 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 6.90 (d, *J* = 8.5 Hz, 2H), 7.26 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.8, 14.9 (two isomers), 23.0, 23.1 (two isomers), 26.6, 26.8 (two isomers), 27.6, 27.6 (two isomers), 30.2, 30.2 (two isomers), 32.4, 33.2 (two isomers), 34.3, 35.9 (two isomers), 35.6, 35.6 (two isomers), 37.6, 37.7 (two isomers), 39.7, 39.8 (two isomers), 44.1, 44.2 (two isomers), 45.7, 45.8 (two isomers), 50.1, 50.1 (two isomers), 55.5, 55.5 (two isomers), 55.5, 55.6 (two isomers), 79.7, 80.2 (two isomers), 94.2, 94.6 (two isomers), 111.7,

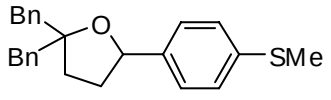
111.8 (two isomers), 113.9, 113.9 (two isomers), 114.0, 114.0 (two isomers), 126.6, 126.7 (two isomers), 127.1, 127.3 (two isomers), 132.9, 133.0 (two isomers), 136.2, 136.8 (two isomers), 138.3, 138.3 (two isomers), 157.7, 157.7 (two isomers), 159.0, 159.0 (two isomers). FT-IR (CH2Cl2, cm^{-1}): 2933, 2869, 2835, 1611, 1576, 1513, 1500, 1464, 1377, 1301, 1280, 1246, 1171, 1072, 1040, 872, 830, 782 cm^{-1}. HRMS calcd for $C_{29}H_{36}O_3Na$ $[M+Na]^+$ 455.2557, found: 455.2567.

Compound **6j**



Mixture of two diastereomers (d.r. = 1.3:1). ^1H NMR (500 MHz) δ 0.87-1.10 (m, 6H), 1.20-1.40 (m, 30H), 1.40-1.49 (m, 4H), 1.52-1.58 (m, 6H), 1.63-1.75 (m, 6H), 1.82-1.86 (m, 1H), 1.96-2.06 (m, 3H), 2.10-2.20 (m, 2H), 2.72 (dd, *J* = 7.5, 7.5 Hz, 4H), 3.66 (s, 6H), 3.81 (s, 6H), 3.87-3.90 (m, 1H), 4.01-4.07 (m, 1H), 6.85 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H), 7.31 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR (125 MHz) δ 14.4, 14.4 (two isomers), 22.9, 22.9 (two isomers), 24.6, 24.6 (two isomers), 25.2, 25.2 (two isomers), 26.4, 26.6 (two isomers), 29.3, 29.3 (two isomers), 29.4, 29.4 (two isomers), 29.7, 29.7 (two isomers), 30.0, 30.1 (two isomers), 31.5, 31.7 (two isomers), 32.1, 32.1 (two isomers), 34.3, 34.3 (two isomers), 36.4, 36.6 (two isomers), 37.8, 39.1 (two isomers), 43.1, 44.0 (two isomers), 51.7, 51.7 (two isomers), 55.4, 55.4 (two isomers), 78.7, 80.0 (two isomers), 86.4, 86.5 (two isomers), 113.2, 113.5 (two isomers), 126.6, 126.7 (two isomers), 140.1, 140.5 (two isomers), 158.0, 158.1 (two isomers), 174.6, 174.6 (two isomers). FT-IR (CH_2Cl_2) 2931, 2856, 1741, 1611, 1582, 1510, 1462, 1440, 1299, 1246, 1174, 1101, 1038, 831 cm^{-1}. HRMS calcd for $C_{26}H_{42}O_4Na$ $[M+Na]^+$ 441.2975, found 441.3011.

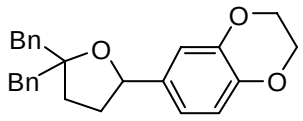
Compound **6k**



^1H NMR (500 MHz) δ 1.28-1.43 (m, 1H), 1.90-2.02 (m, 3H), 2.49 (s, 3H), 2.86 (d, *J* = 13.5 Hz, 1H), 2.93 (d, *J* = 13.5 Hz, 1H), 2.99 (d, *J* = 13.5 Hz, 1H), 3.01 (d, *J* = 13.5 Hz, 1H), 4.49 (dd, *J* = 5.5, 9.5 Hz, 1H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.5 Hz, 2H), 7.24-7.32 (m, 10H); ^{13}C NMR

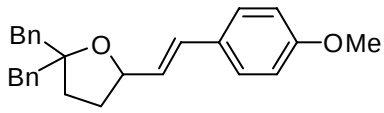
(125 MHz) δ 16.4, 33.4, 34.7, 46.1, 47.1, 80.9, 86.0, 126.47, 126.54, 126.9, 127.2, 128.1, 128.2, 131.2, 131.3, 137.3, 138.2, 138.3, 139.7. FT-IR (CH_2Cl) 3025, 2918, 1725, 1600, 1494, 1453, 1081, 1041, 942, 812, 754, 701 cm^{-1}. HRMS calcd for $C_{25}H_{27}OS$ $[M+1]^+$ 375.1777, found 375.1779.

Compound **6l**



^{1}H NMR (500 MHz) δ 1.28-1.43 (m, 1H), 1.84-1.88 (m, 1H), 1.90-2.01 (m, 2H), 2.85 (d, *J* = 13.5 Hz, 1H), 2.93 (d, *J* = 13.5 Hz, 1H), 3.00 (d, *J* = 14.0 Hz, 1H), 3.01 (d, *J* = 13.5 Hz, 1H), 4.26 (s, 4H), 4.40 (dd, *J* = 5.0, 8.0 Hz, 1H), 6.70 (dd, *J* = 2.0, 8.0 Hz, 1H), 6.80 (s, 1H), 6.81 (dd, *J* = 2.0, 8.0 Hz, 1H), 7.24-7.33 (m, 10H); ^{13}C NMR (125 MHz) δ 33.4, 34.6, 46.0, 47.2, 64.6, 64.6, 81.0, 85.7, 115.7, 117.1, 119.8, 126.4, 126.5, 128.2, 128.2, 131.3, 131.3, 135.8, 138.2, 138.3, 143.0, 143.5. FT-IR (CH_2Cl_2) 3061, 3027, 2973, 2934, 2874, 1592, 1509, 1495, 1454, 1434, 1309, 1287, 1258, 1069, 1051, 931, 904, 887, 814, 754, 702 cm^{-1}. HRMS calcd for $C_{26}H_{27}O_3$ $[M+1]^+$ 387.1955, found 387.1938.

Compound **6m**

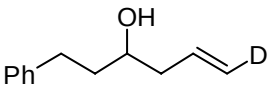


^{1}H NMR (500 MHz) δ 0.96-1.01 (m, 1H), 1.48-1.55 (m, 1H), 1.78-1.83 (m, 2H), 2.73 (d, *J* = 13.5 Hz, 1H), 2.74 (d, *J* = 14.0 Hz, 1H), 2.87 (d, *J* = 13.5 Hz, 1H), 2.91 (d, *J* = 14.0 Hz, 1H), 3.74 (s, 3H), 4.05 (dd, *J* = 8.0, 14.0 Hz, 1H), 5.71 (dd, *J* = 7.5, 15.5 Hz, 1H), 6.30 (d, *J* = 16.0 Hz, 1H), 6.77 (d, *J* = 8.5 Hz, 2H), 7.16-7.24 (m, 12H); ^{13}C NMR (125 MHz) δ 33.17, 33.20, 46.9, 47.3, 55.5, 81.1, 86.0, 114.1, 126.4, 126.5, 127.9, 128.0, 128.2, 128.7, 129.9, 130.6, 131.1, 131.3, 138.3, 138.4, 159.3. FT-IR (CH_2Cl_2) 2921, 2850, 1606, 1512, 1301, 1250, 1175, 1083, 1035, 967, 806, 755, 731, 702 cm^{-1}. HRMS calcd for $C_{27}H_{29}O_2$ $[M+1]^+$ 385.2162, found 385.2151.

5. Deuterium Experiments

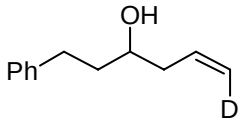
Compounds **7a** and **7b** were prepared according to reported procedures.[3]

Compound **7a**



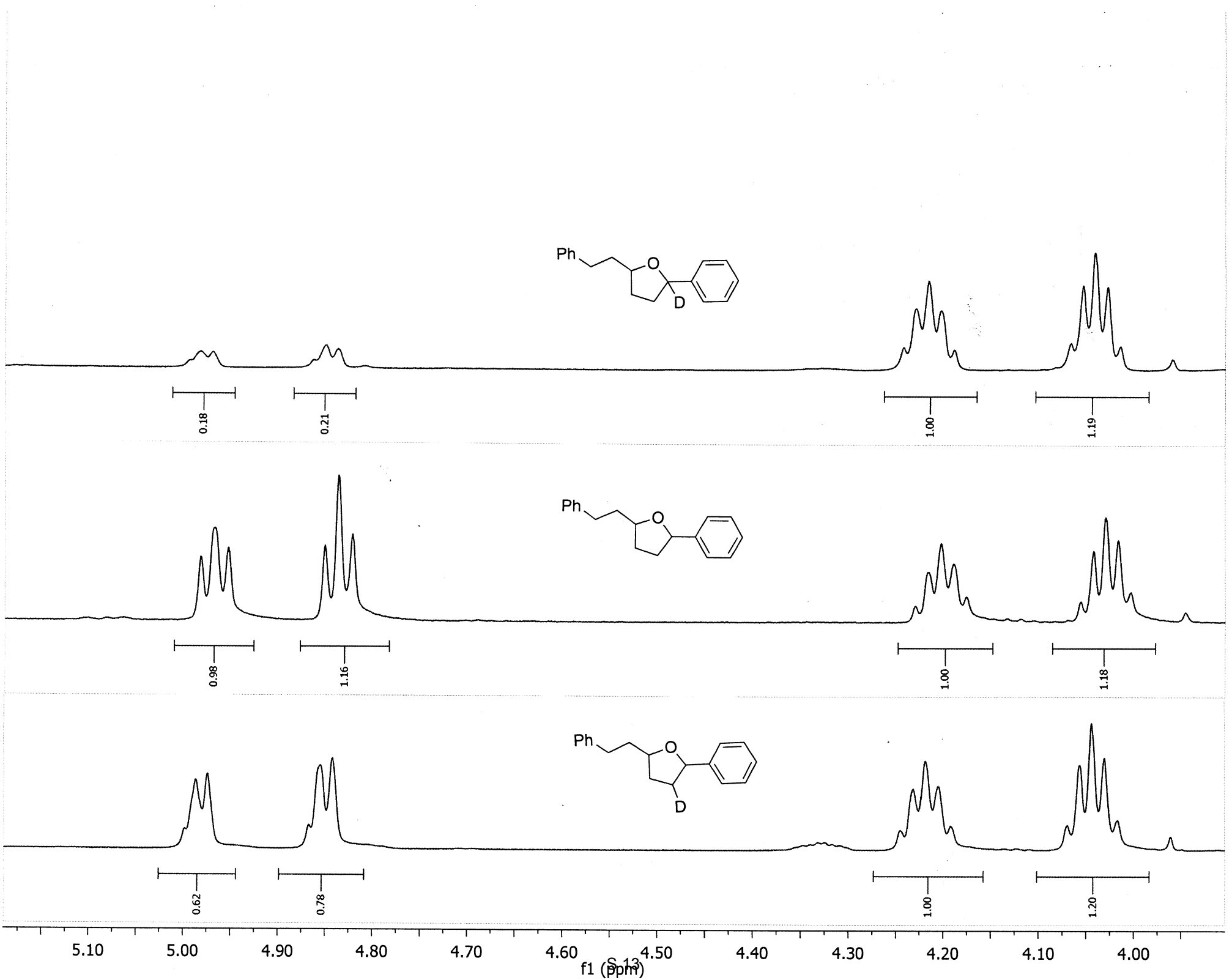
Deuterium incorporation was determined by ^{1}H NMR to be 86%. ^{1}H NMR (500 MHz) δ 1.65 (br s, 1H), 1.78-1.84 (m, 2H), 2.16-2.24 (m, 1H), 2.31-2.37 (m, 1H), 2.67-2.74 (m, 1H), 2.80-2.86 (m, 1H), 3.66-3.74 (m, 1H), 5.15 (d, *J* = 17.0 Hz, 1 H), 5.83 (dt, *J* = 17.0, 7.5 Hz, 1H), 7.18-7.26 (m, 3H), 7.30 (t, *J* = 7.5 Hz, 2 H).

Compound **7b**



Deuterium incorporation was determined by ^{1}H NMR to be 86%. ^{1}H NMR (500 MHz) δ 1.65 (br s, 1H), 1.78-1.84 (m, 2H), 2.17-2.23 (m, 1H), 2.32-2.38 (m, 1H), 2.66-2.75 (m, 1H), 2.78-2.87 (m, 1H), 3.67-3.73 (m, 1H), 5.15 (d, *J* = 10.0 Hz, 1 H), 5.79-5.88 (m, 1H), 7.18-7.25 (m, 3H), 7.32 (t, *J* = 7.5 Hz, 2 H).

[3] K. S. Lee, J. M. Ready, *Angew. Chem. Int. Ed*. **2011**, *50*, 2111.


Ph
O
D
0.18
0.21
1.00
1.19
Ph
O
0.98
1.16
1.00
1.18
Ph
O
D
0.62
0.78
1.00
1.20
5.10
5.00
4.90
4.80
4.70
4.60
4.50
4.40
4.30
4.20
4.10
4.00
f1 (ppm)

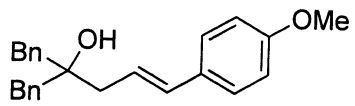
zc2-172

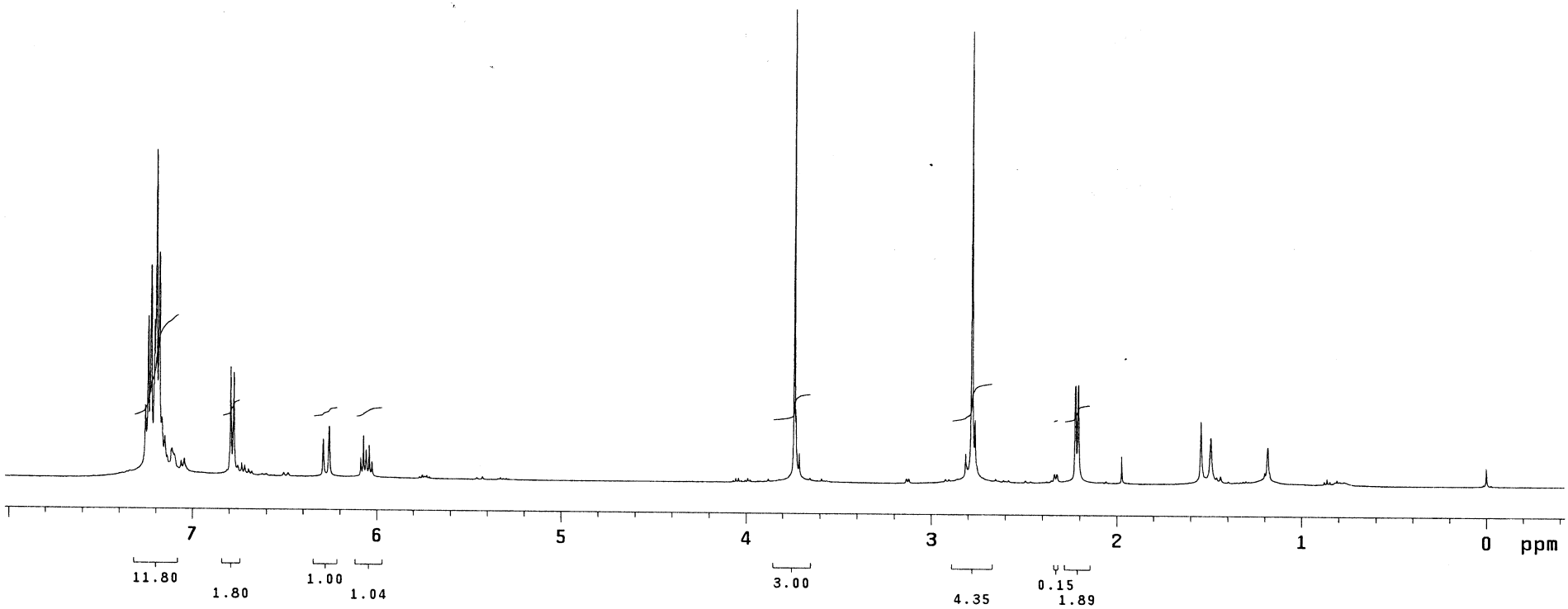
File: zc2-172

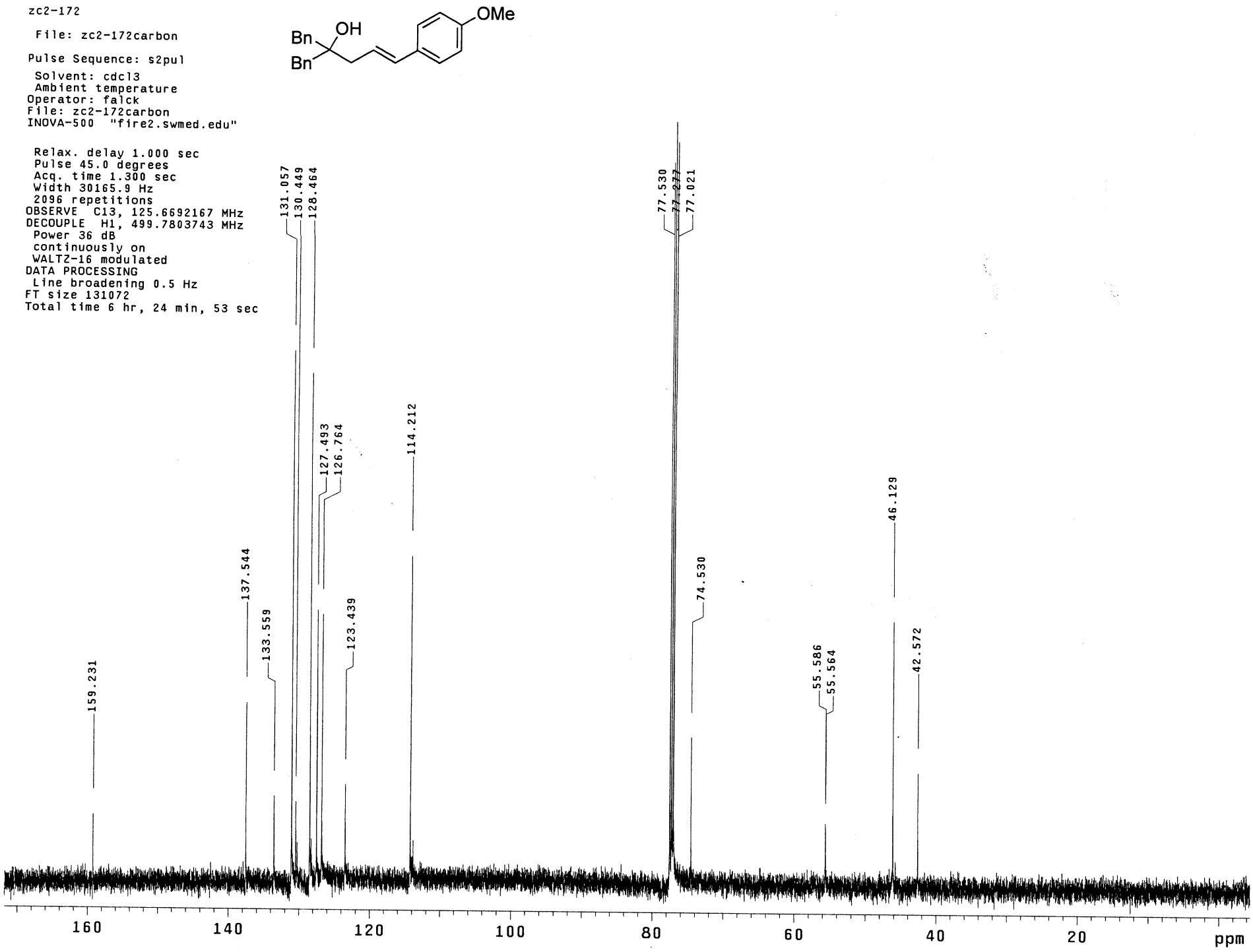
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-172
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7779208 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec





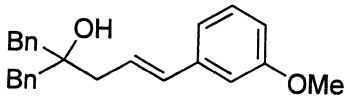

zc2-172
File: zc2-172carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-172carbon
INOVA-500 "fire2.swmed.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
2096 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 min, 53 sec
Bn
OH
Bn
OMe
159.231
137.544
133.559
131.057
130.449
128.464
127.493
126.764
123.439
114.212
77.530
77.277
77.021
74.530
55.586
55.564
46.129
42.572
160
140
120
100
80
60
40
20
ppm

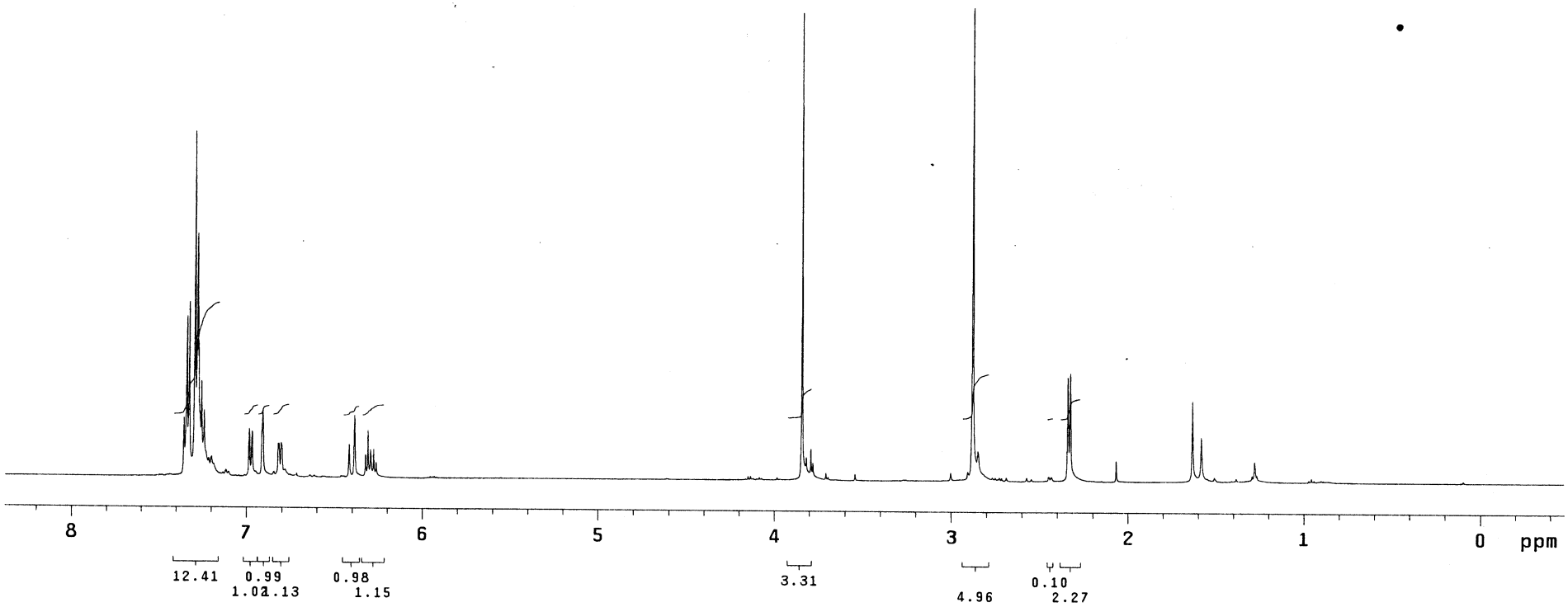
zc2-174

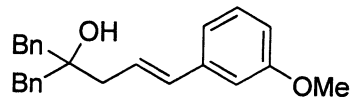
File: zc2-174

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-174
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec







zc2-174

File: Carbon

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

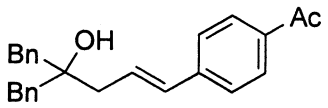
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
432 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 min, 53 sec

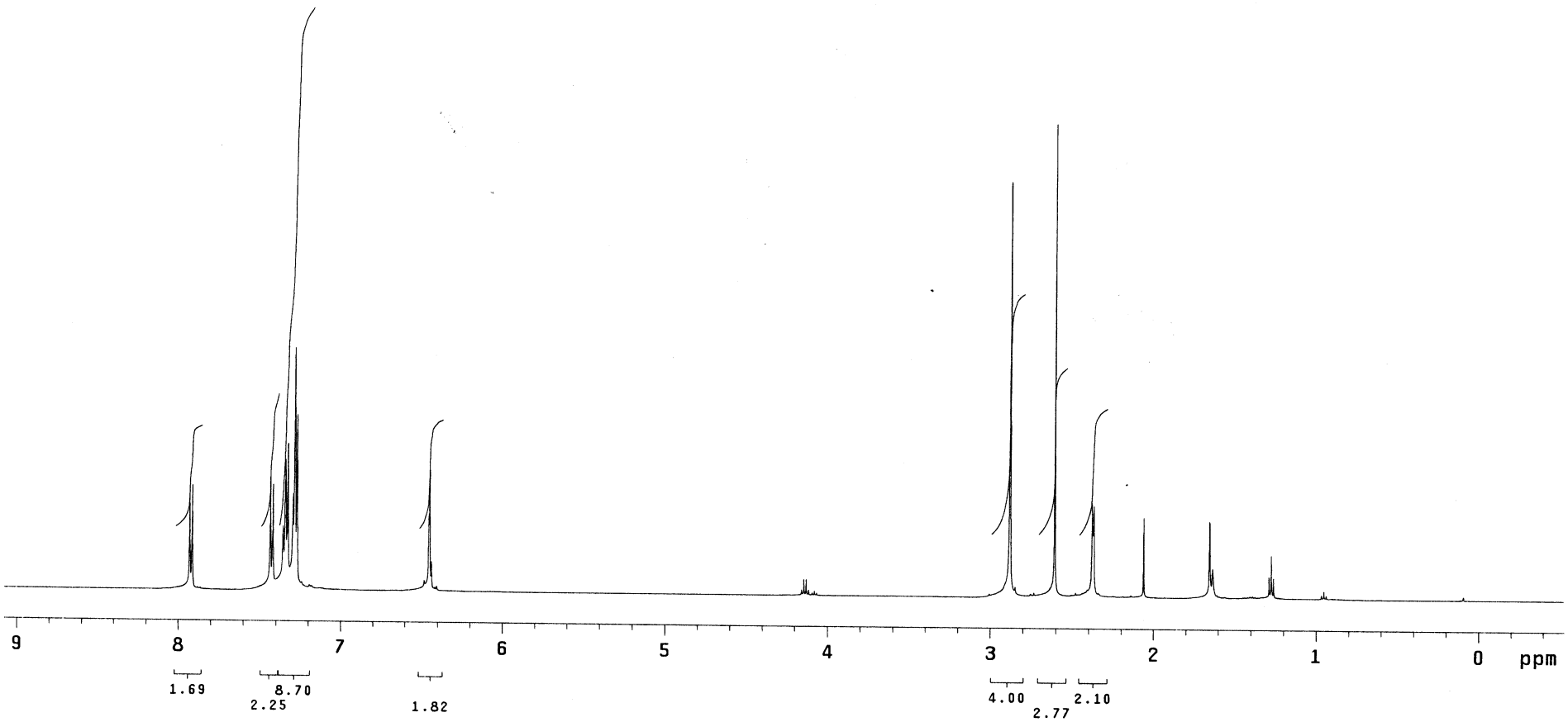
zc2-179

File: zc2-179

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-179
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



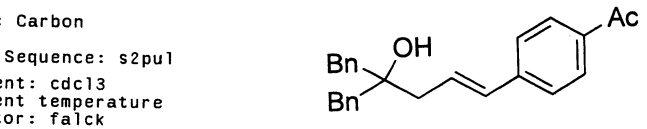


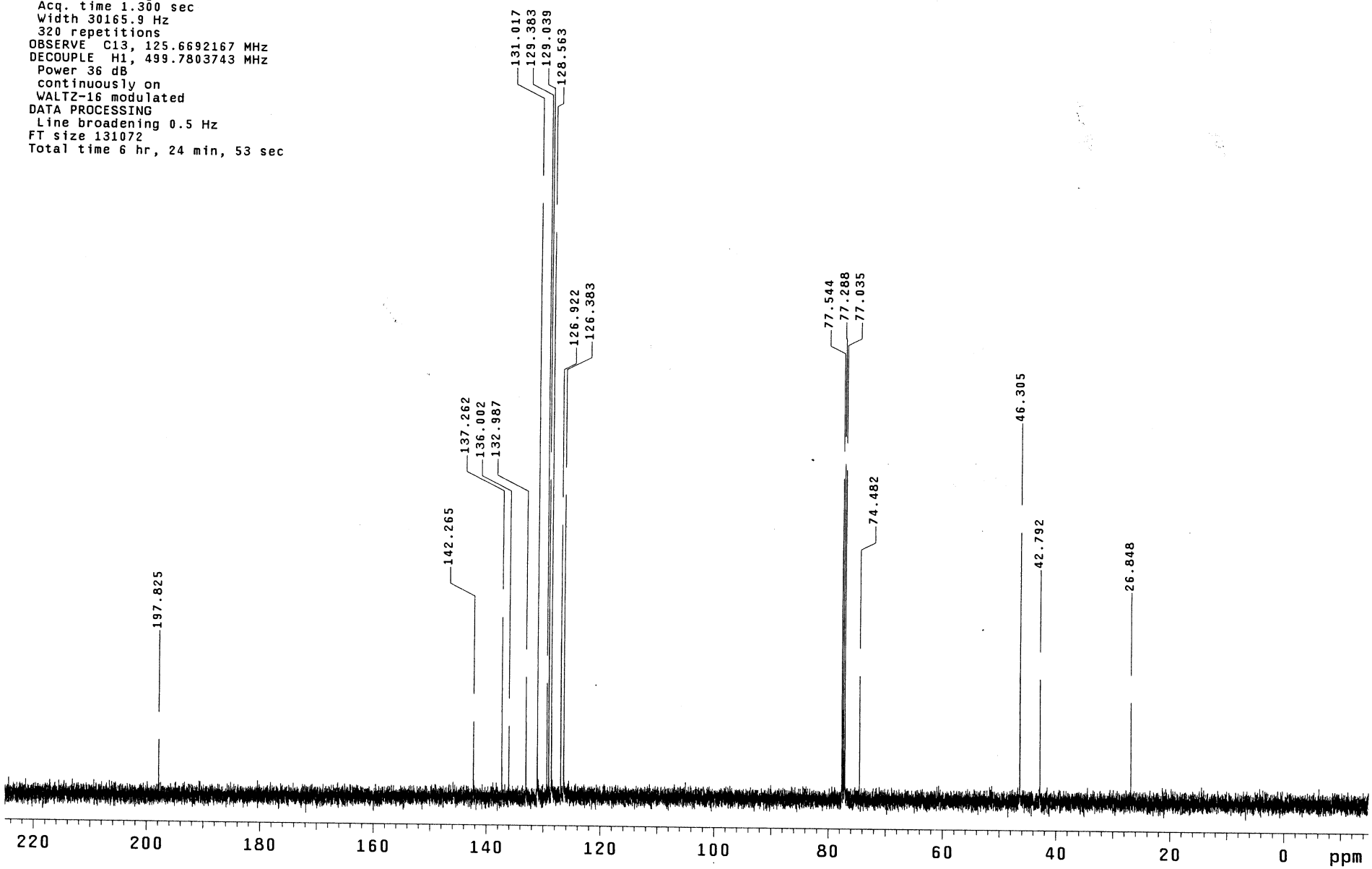
zc2-179

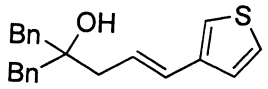
File: Carbon

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
320 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 min, 53 sec





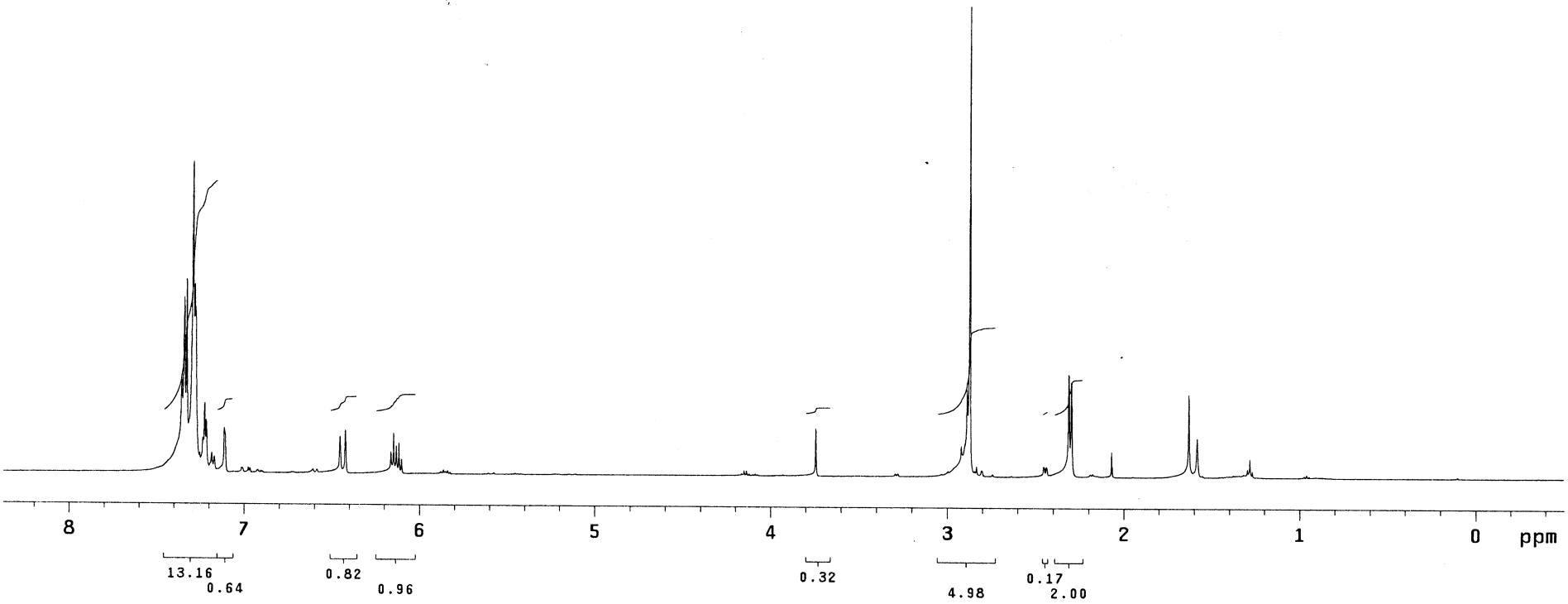


zc2-178

File: zc2-178

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-178
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

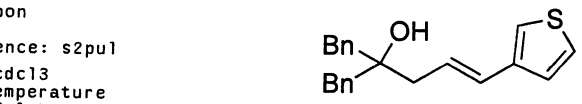


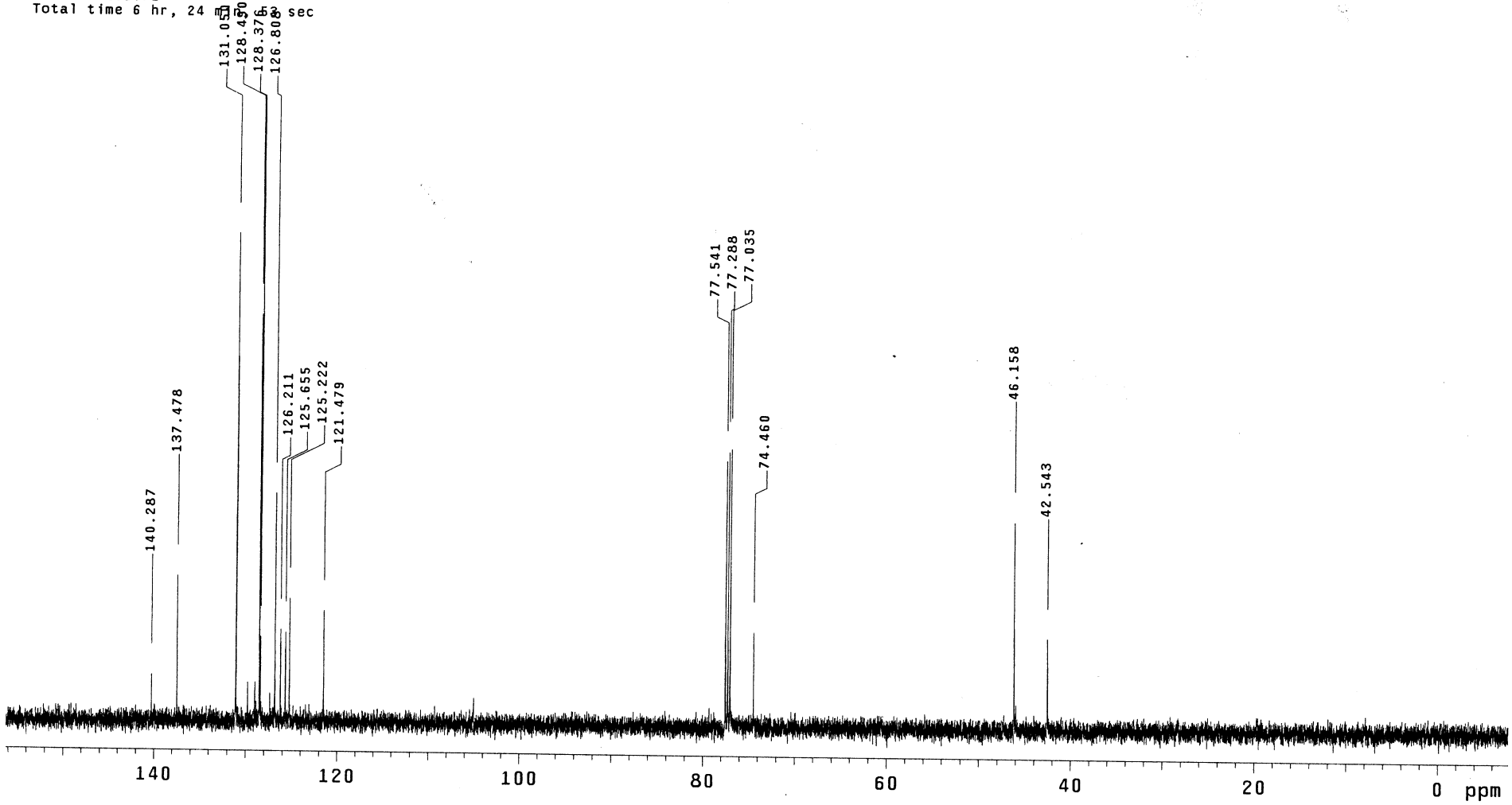
zc2-178

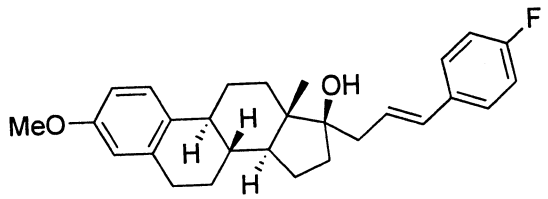
File: Carbon

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
208 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 m[illegible] sec





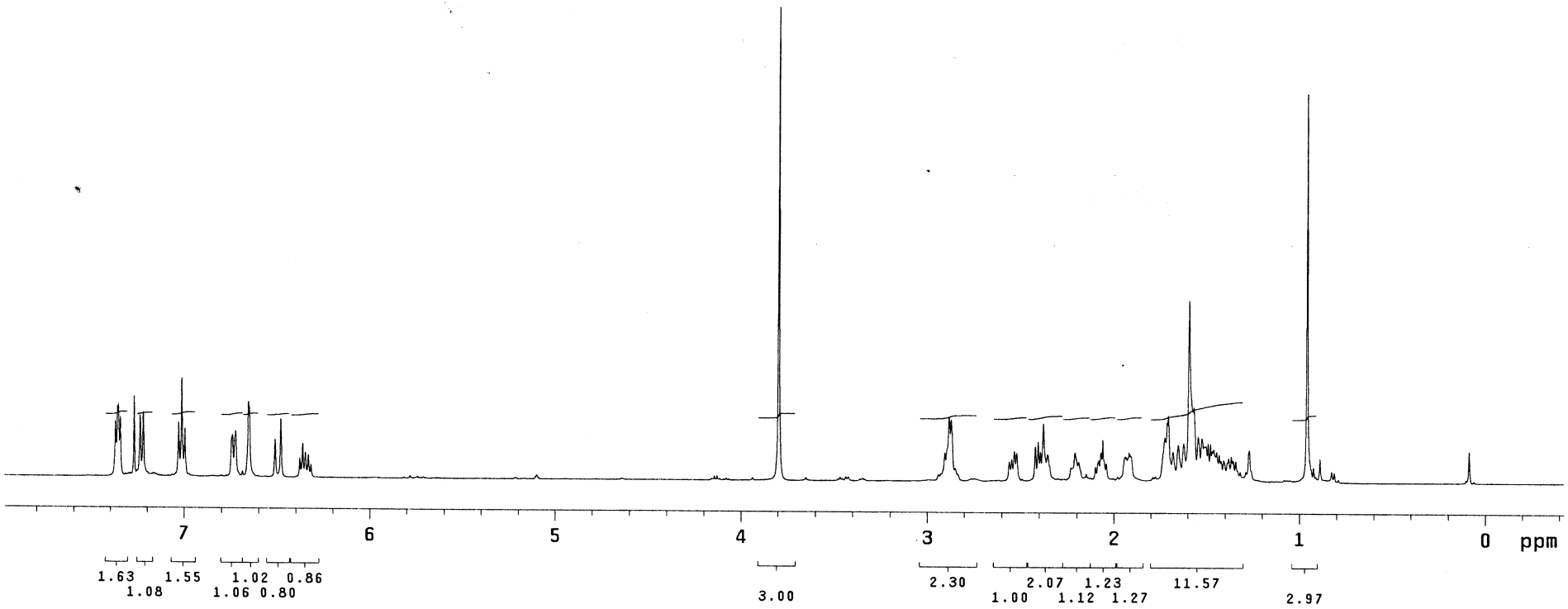


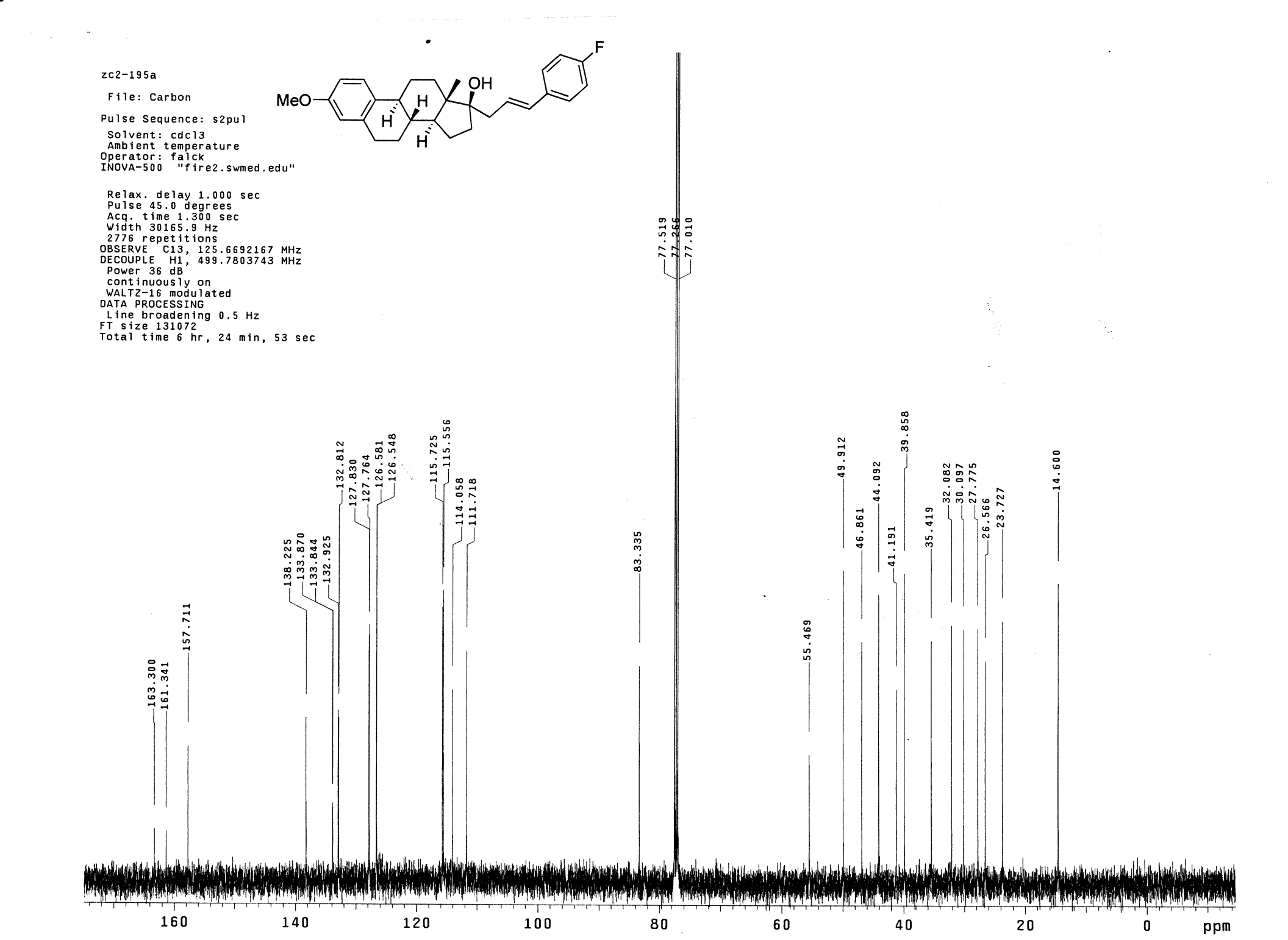
zc2-195a

File: Proton

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec




zc2-195a
File: Carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
2776 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 min, 53 sec
MeO
OH
F
H
H
H
163.300
161.341
157.711
138.225
133.870
133.844
132.925
132.812
127.830
127.764
126.581
126.548
115.725
115.556
114.058
111.718
83.335
77.519
77.266
77.010
55.469
49.912
46.861
44.092
41.191
39.858
35.419
32.082
30.097
27.775
26.566
23.727
14.600
160
140
120
100
80
60
40
20
0
ppm

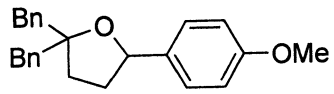
zc2-111

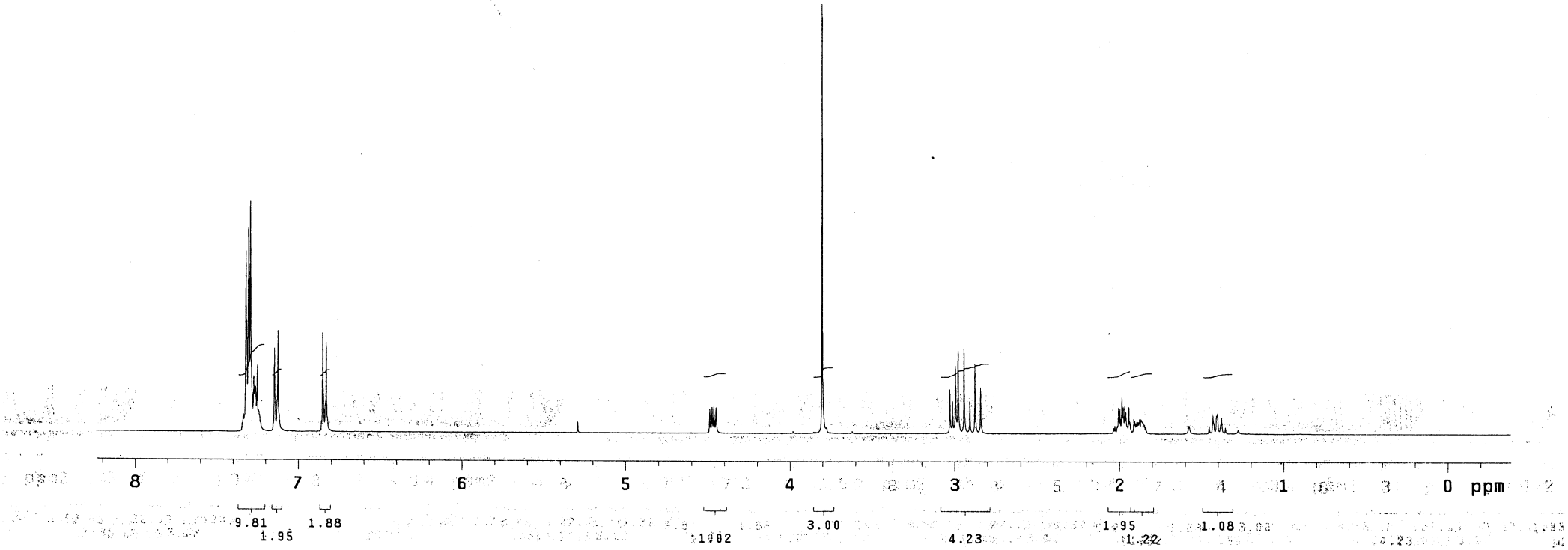
File: Proton

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 19.0 C / 292.1 K
Operator: falck
INOVA-400 "pele400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6396.4 Hz
8 repetitions
OBSERVE H1, 399.7793055 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



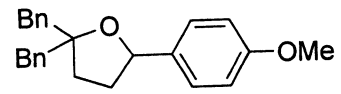


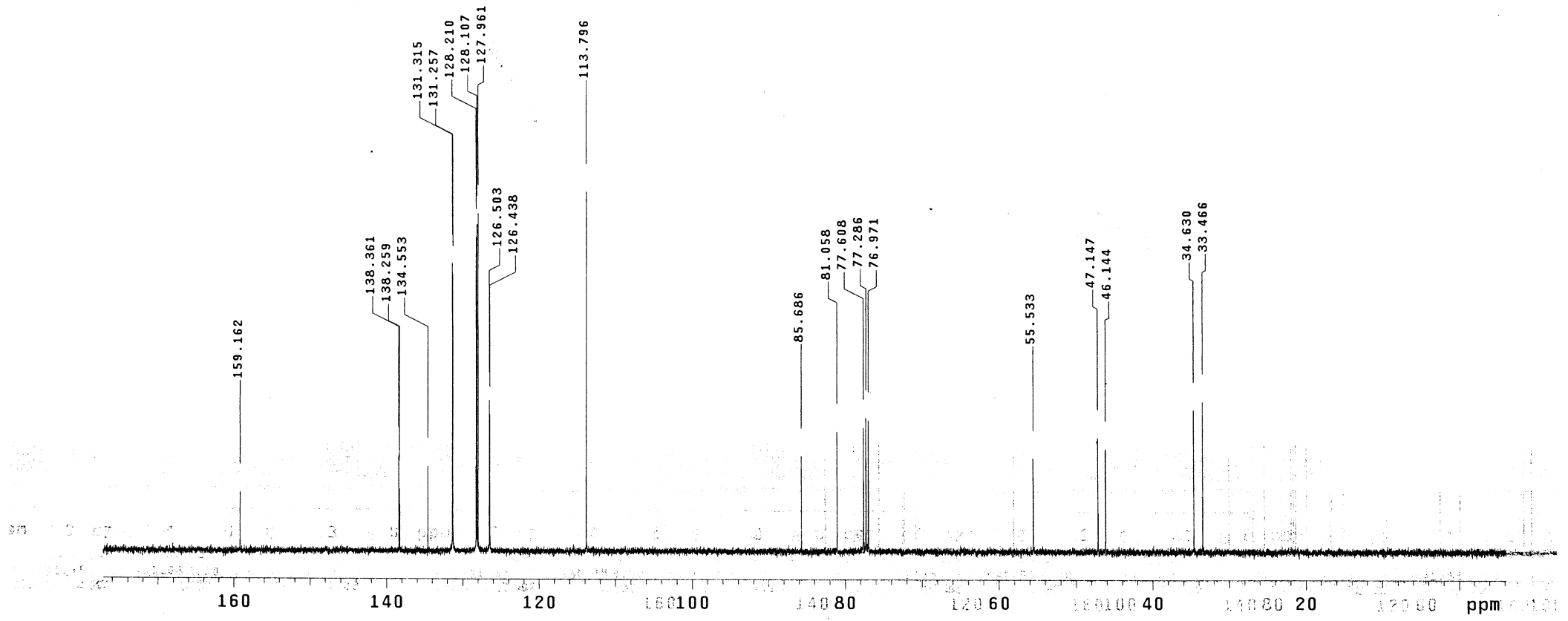
zc2-111

File: Carbon

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 19.0 C / 292.1 K
Operator: falck
INOVA-400 "pele400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24125.5 Hz
176 repetitions
OBSERVE C13, 100.5245623 MHz
DECOUPLE H1, 399.7813047 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 29 sec



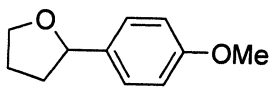


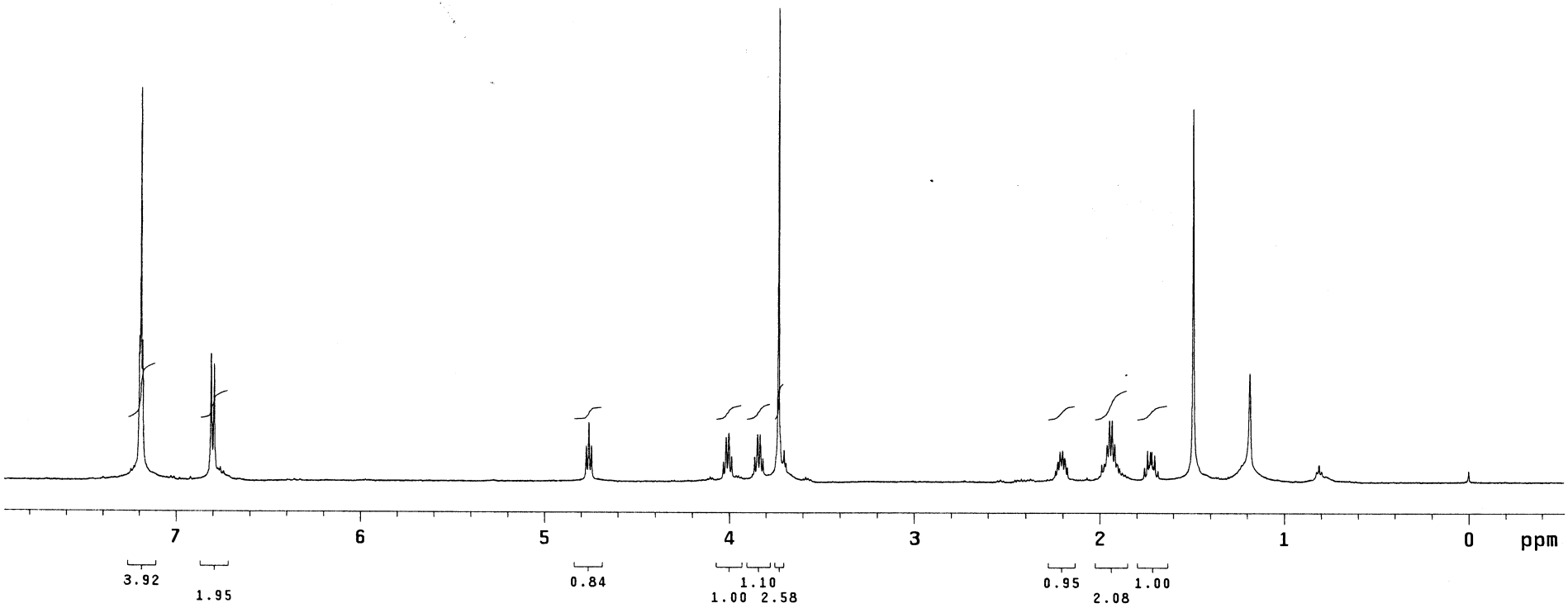
zc2-90b

File: zc2-90b

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-90b
INOVA-500 "fire2.swmed.edu"

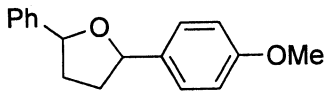
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7779159 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec





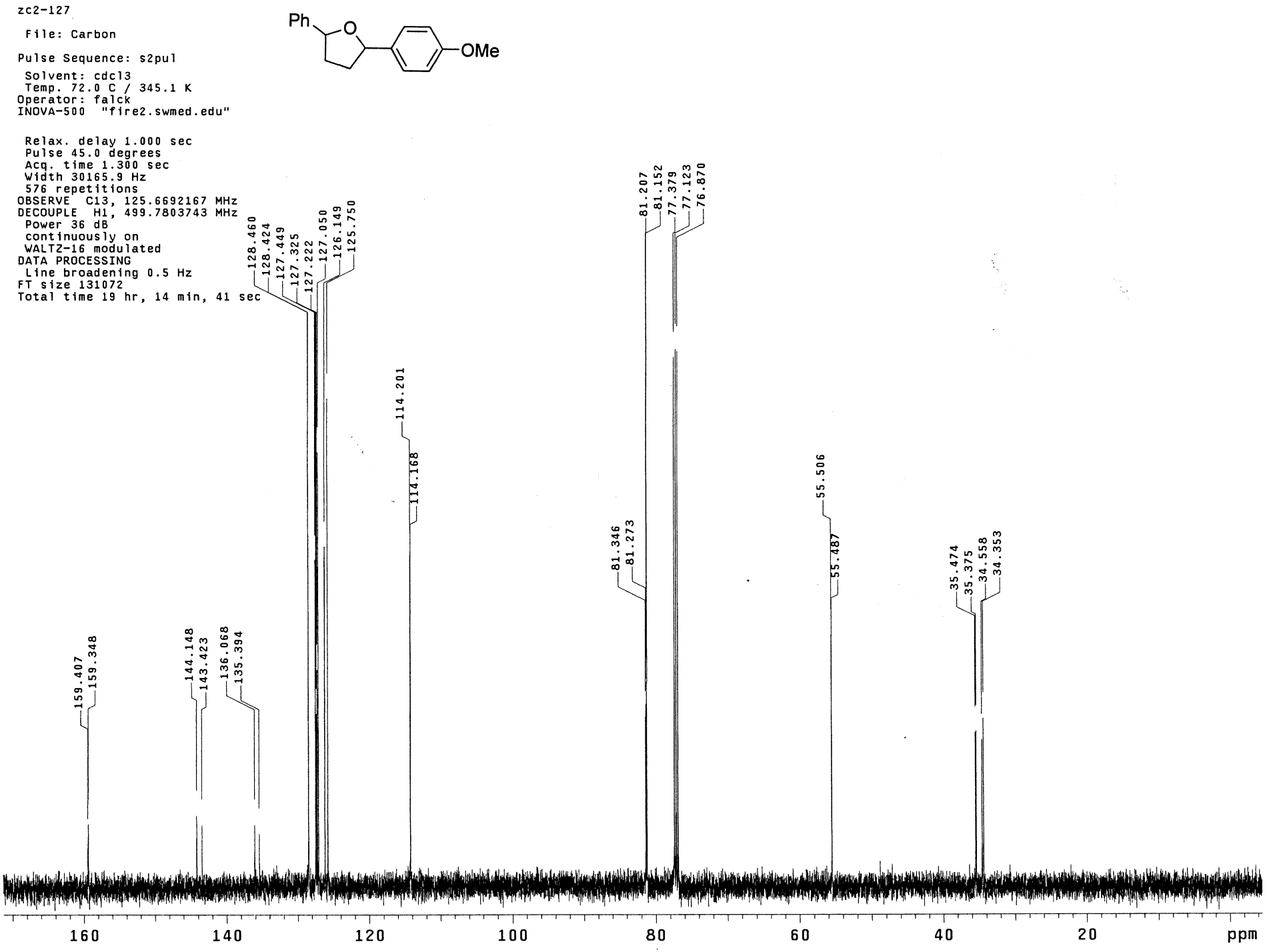
zc2-121

File: Proton



Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec

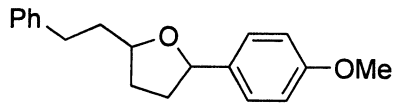

zc2-127
File: Carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 72.0 C / 345.1 K
Operator: falck
INOVA-500 "fire2.swmed.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
576 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 19 hr, 14 min, 41 sec
Ph
O
OMe
159.407
159.348
144.148
143.423
136.068
135.394
128.460
128.424
127.449
127.325
127.222
127.050
126.149
125.750
114.201
114.168
81.346
81.273
81.207
81.152
77.379
77.123
76.870
55.506
55.487
35.474
35.375
34.558
34.353
160
140
120
100
80
60
40
20
ppm

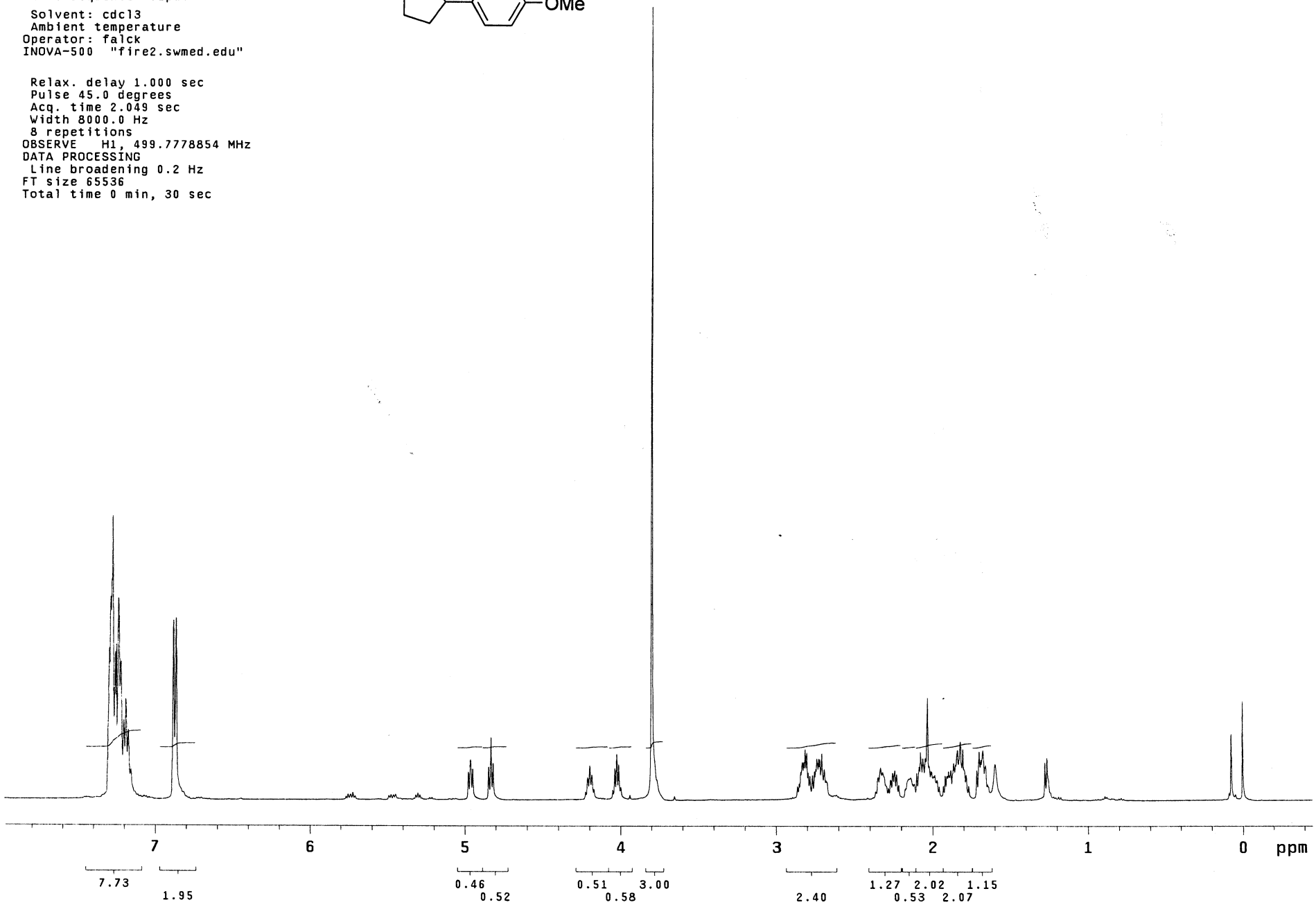
zc1-289

File: Proton

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

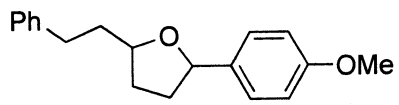
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778854 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec





zc1-289

File: Carbon



Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

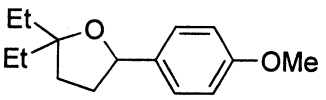
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
512 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 1 hr, 16 min, 58 sec

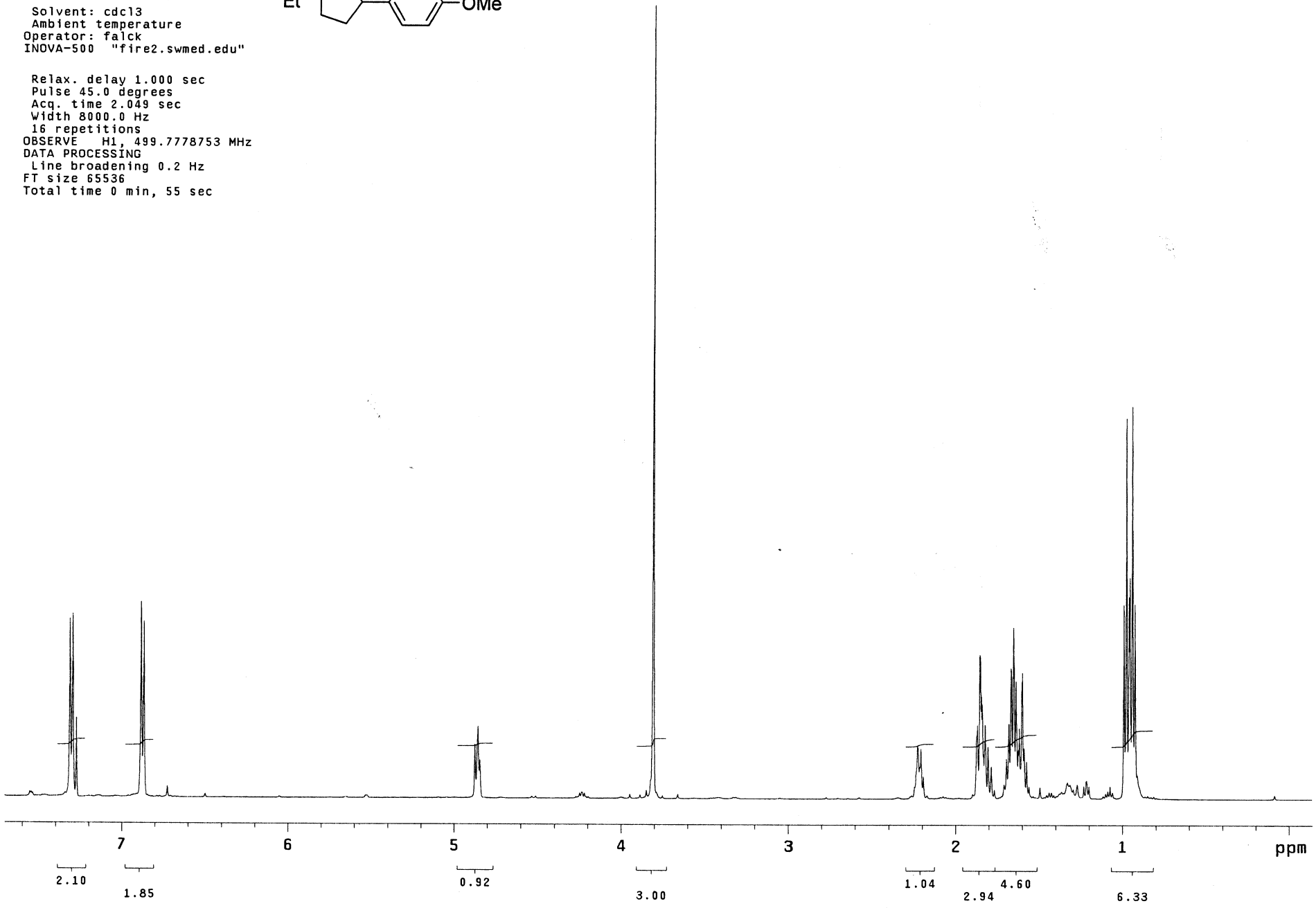
zc2-118

File: Proton

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

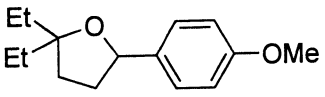
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec





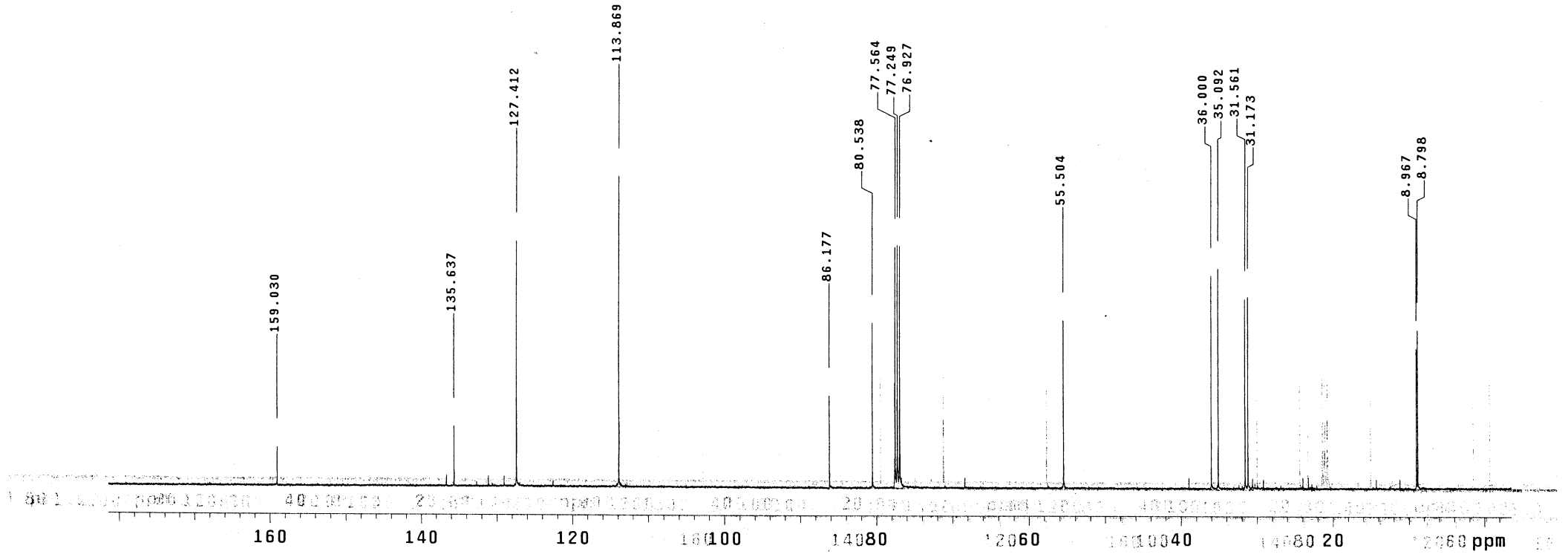
zc2-118

File: Carbon



Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-400 "pele400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24125.5 Hz
18480 repetitions
OBSERVE C13, 100.5245623 MHz
DECOUPLE H1, 399.7813047 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 13 hr, 8 min, 16 sec

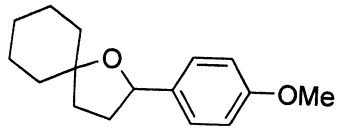


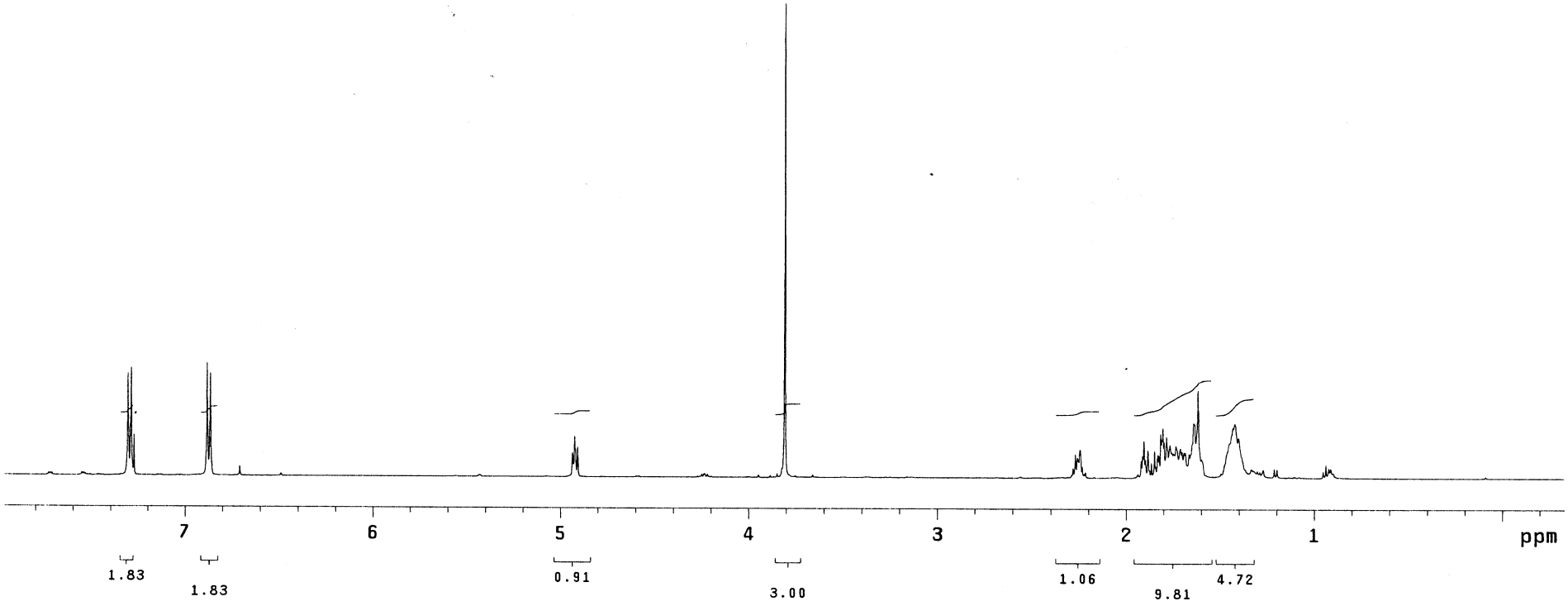
zc2-120

File: Proton

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec





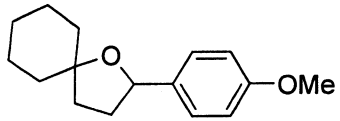
zc2-120

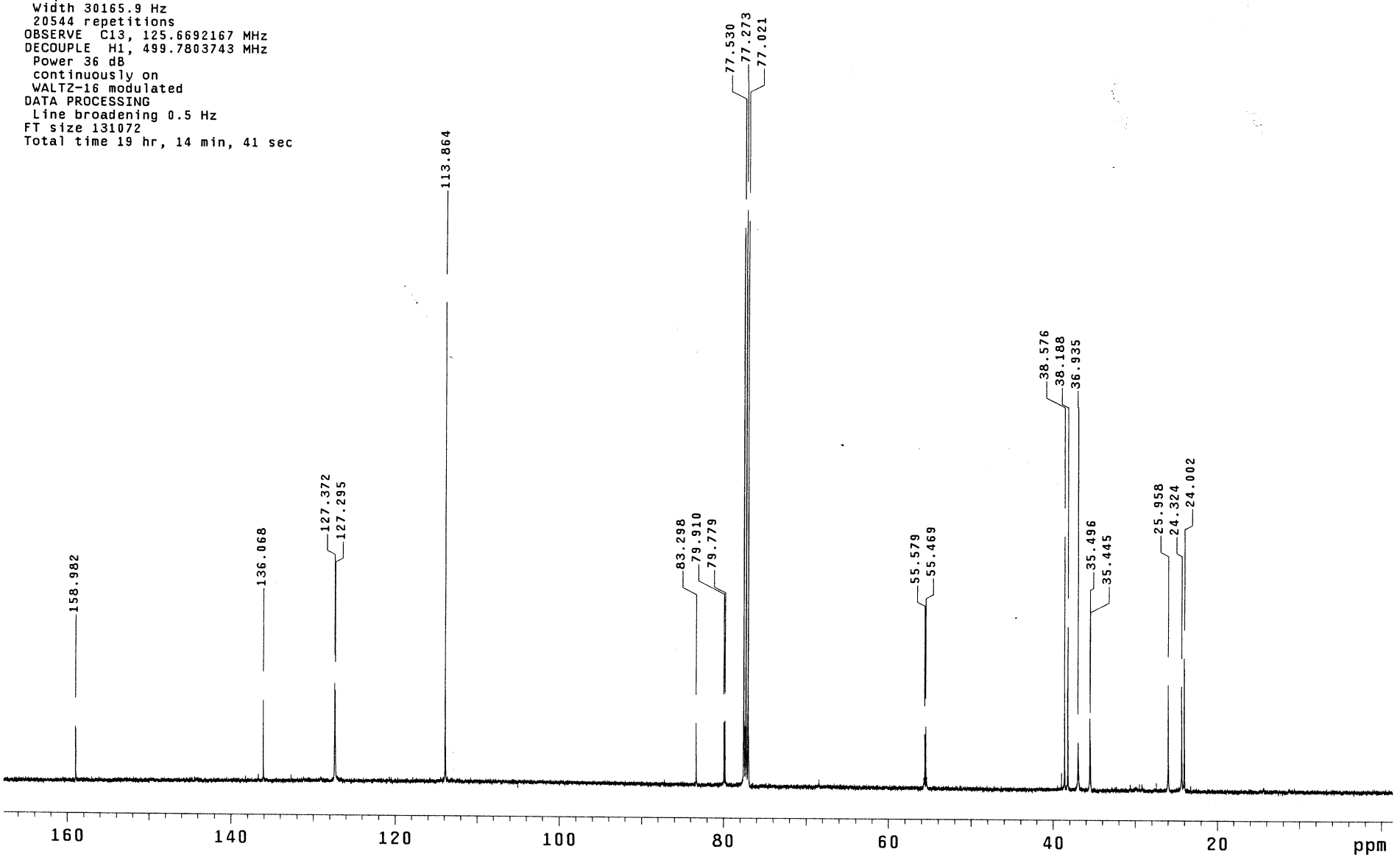
File: zc2-120carbon

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-120carbon
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
20544 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 19 hr, 14 min, 41 sec



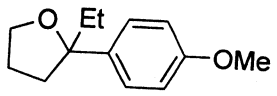


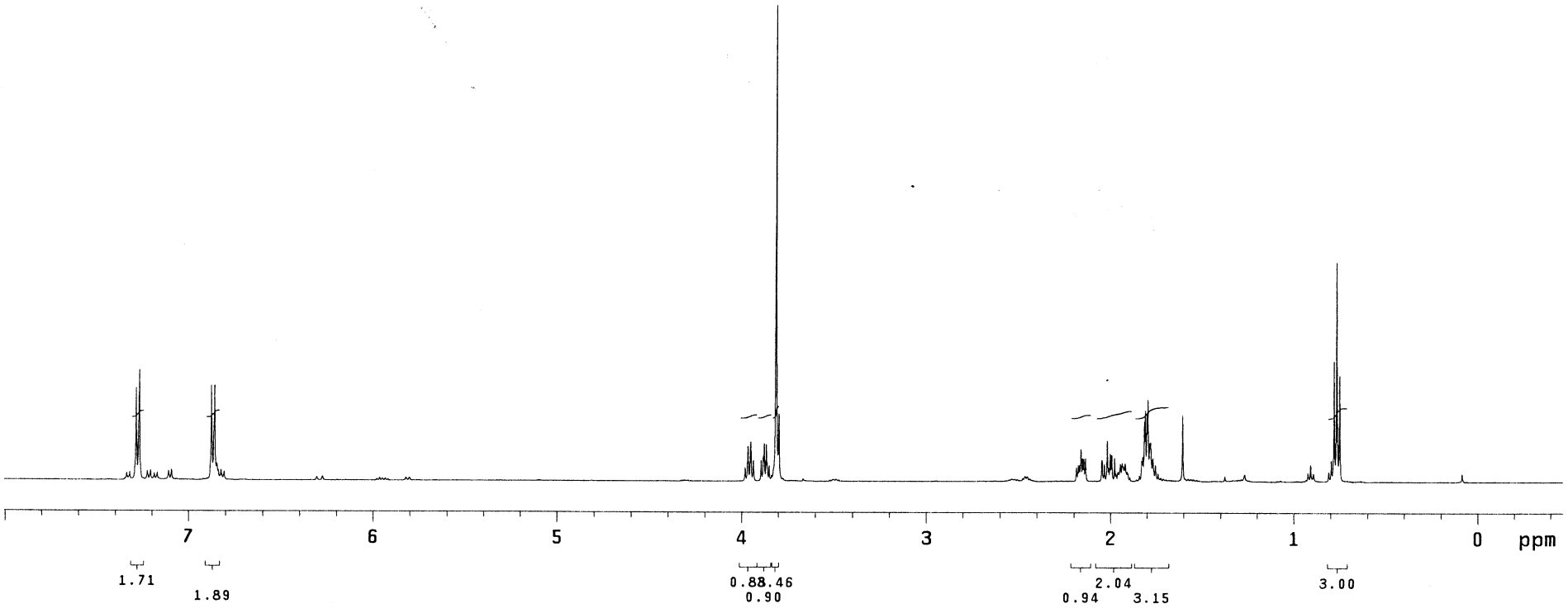
zc2-133

File: Proton

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

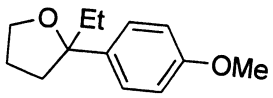




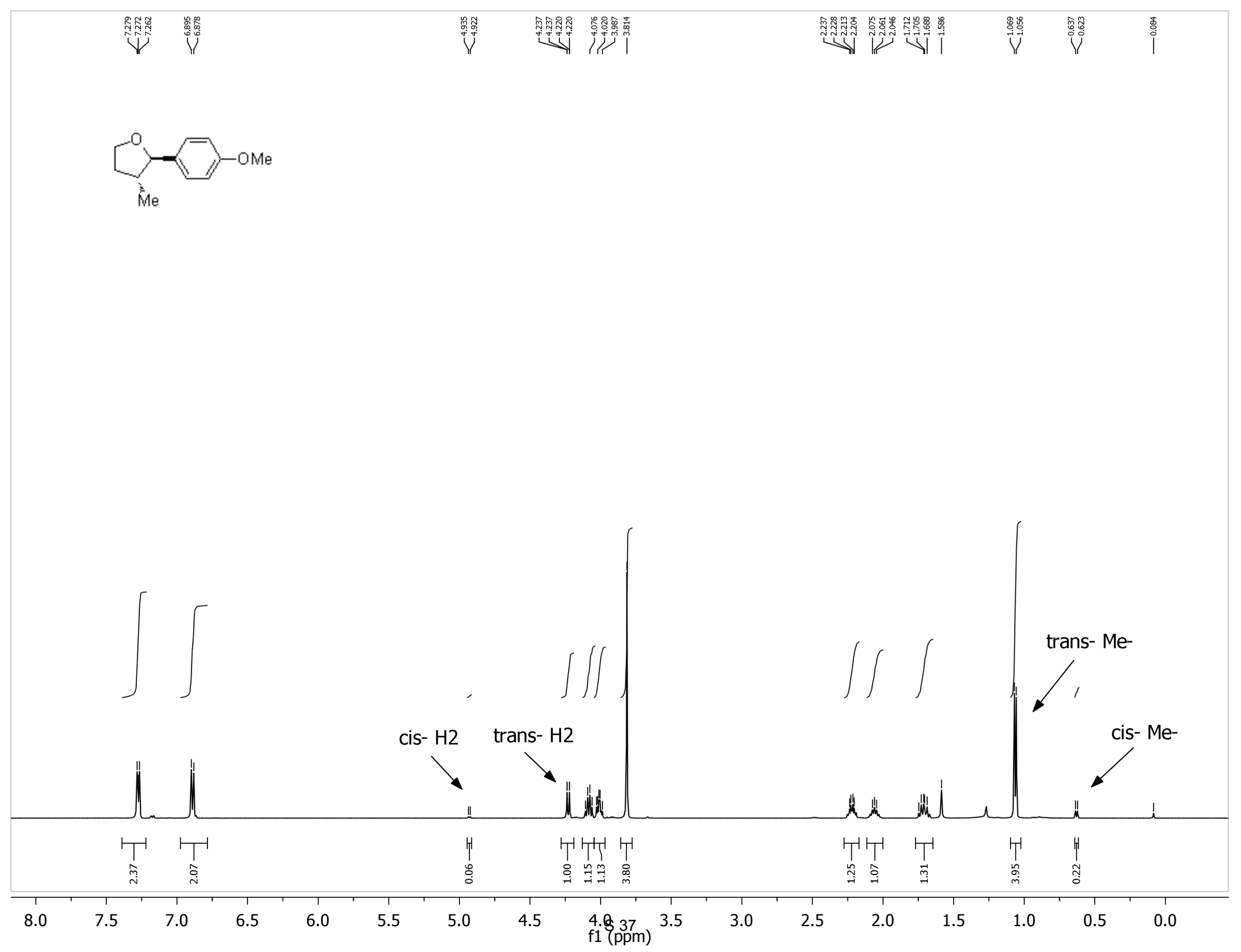
zc2-133

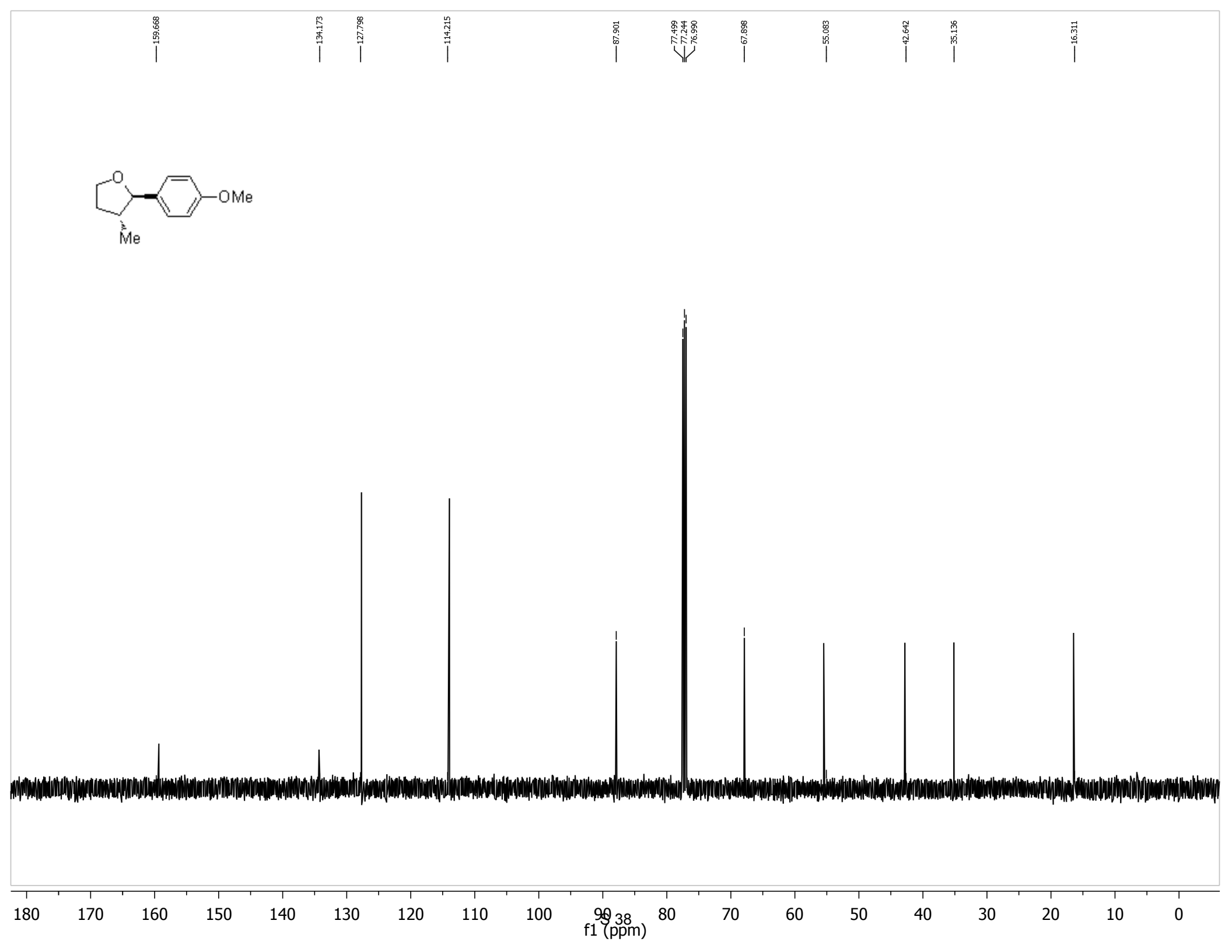
File: Carbon

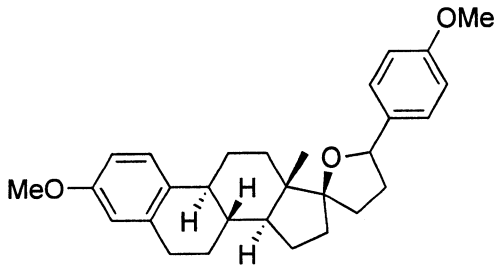
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"



Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
360 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 min, 53 sec


7.279
7.272
7.262
6.895
6.878
4.935
4.922
4.237
4.237
4.220
4.220
4.076
4.020
3.987
3.814
2.237
2.228
2.213
2.204
2.075
2.061
2.046
1.712
1.705
1.688
1.586
1.069
1.056
0.637
0.623
0.084
O
OMe
Me
cis- H2
trans- H2
trans- Me-
cis- Me-
2.37
2.07
0.06
1.00
1.15
1.13
3.80
1.25
1.07
1.31
3.95
0.22
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


159.668
134.173
127.798
114.215
87.901
77.499
77.244
76.990
67.898
55.083
42.642
35.136
16.311
O
OMe
Me
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

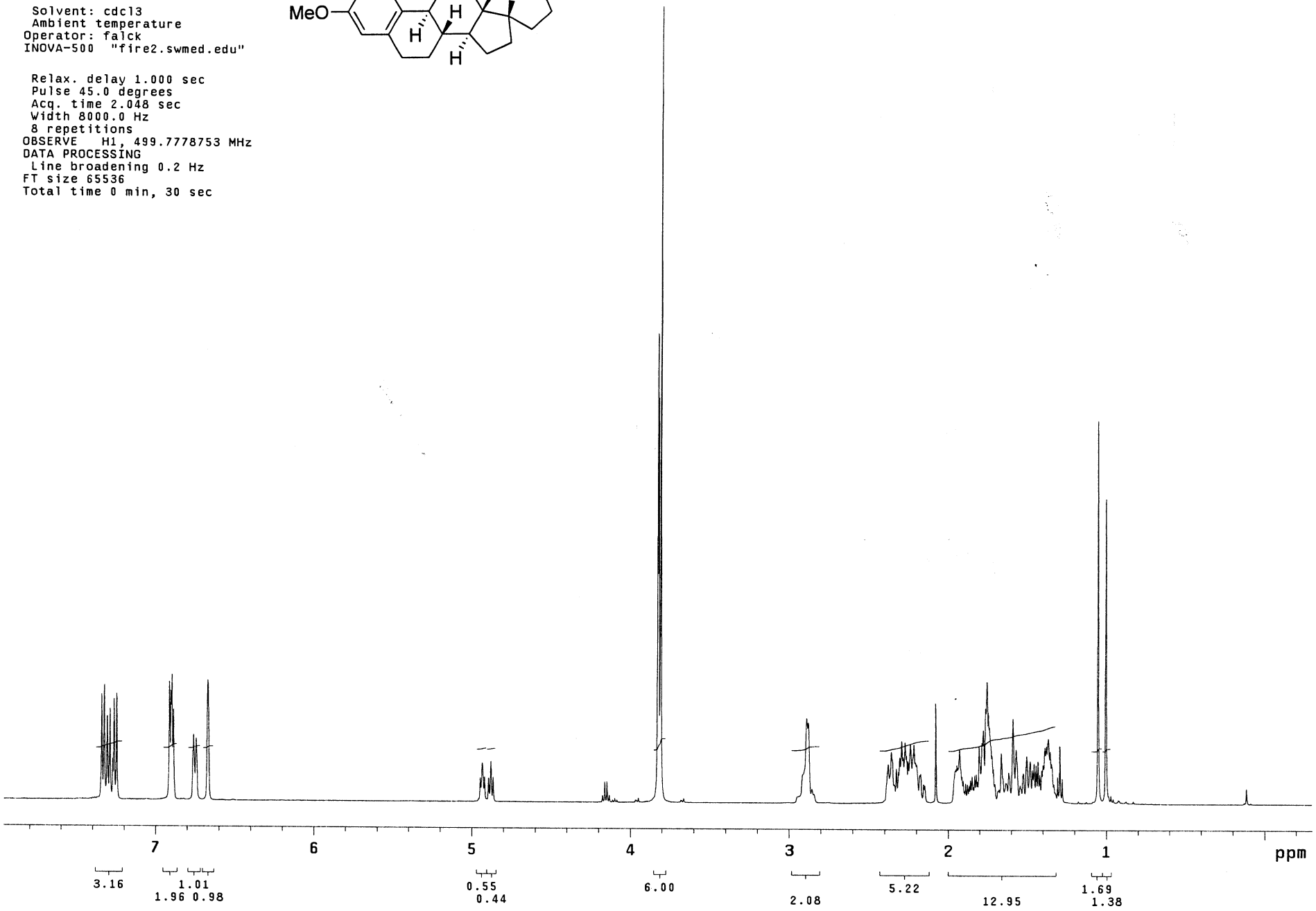


zc2-128

File: Proton

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

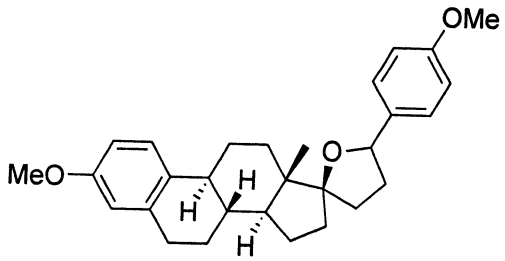


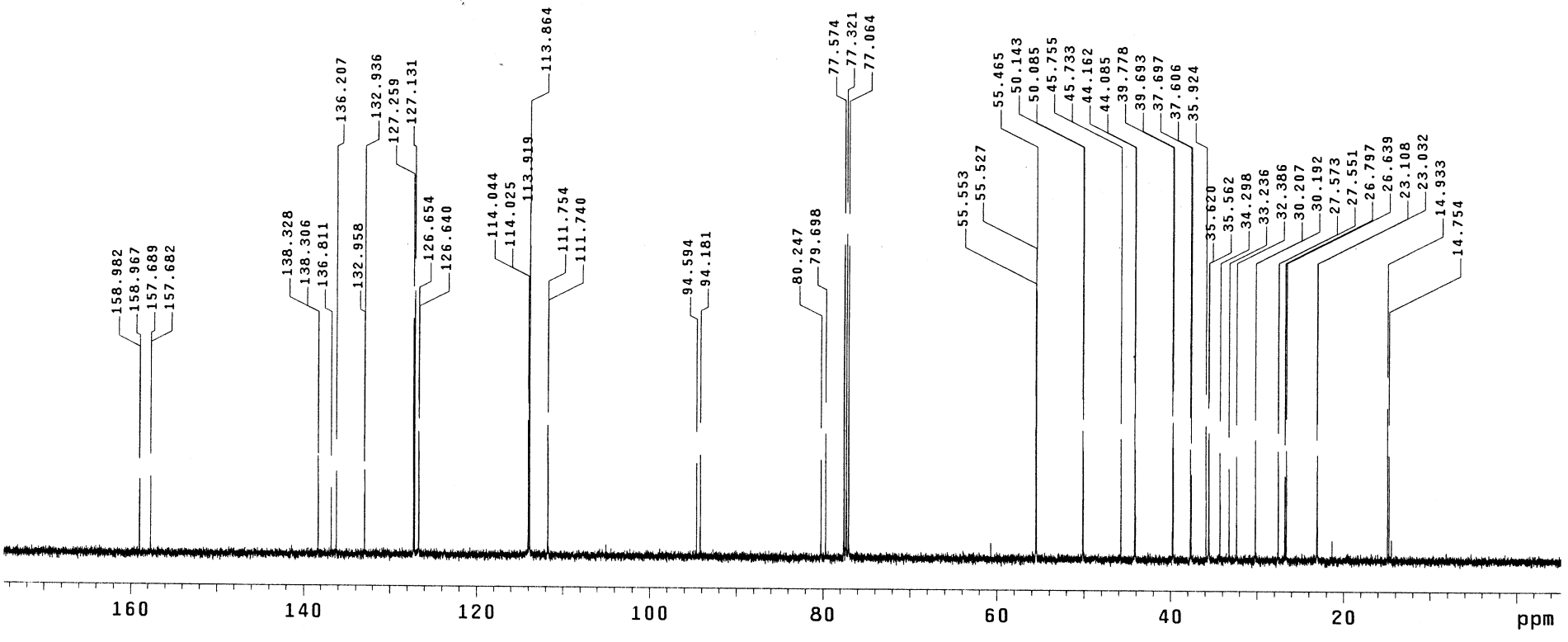
zc2-128

File: Carbon

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
776 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 19 hr, 14 min, 41 sec



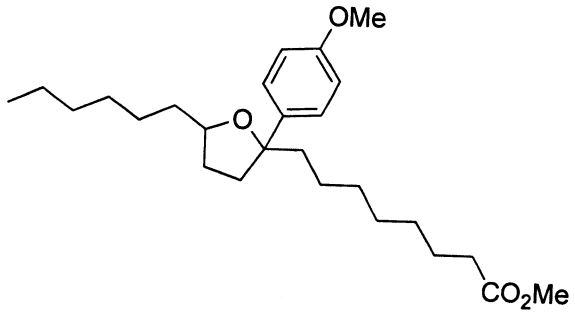


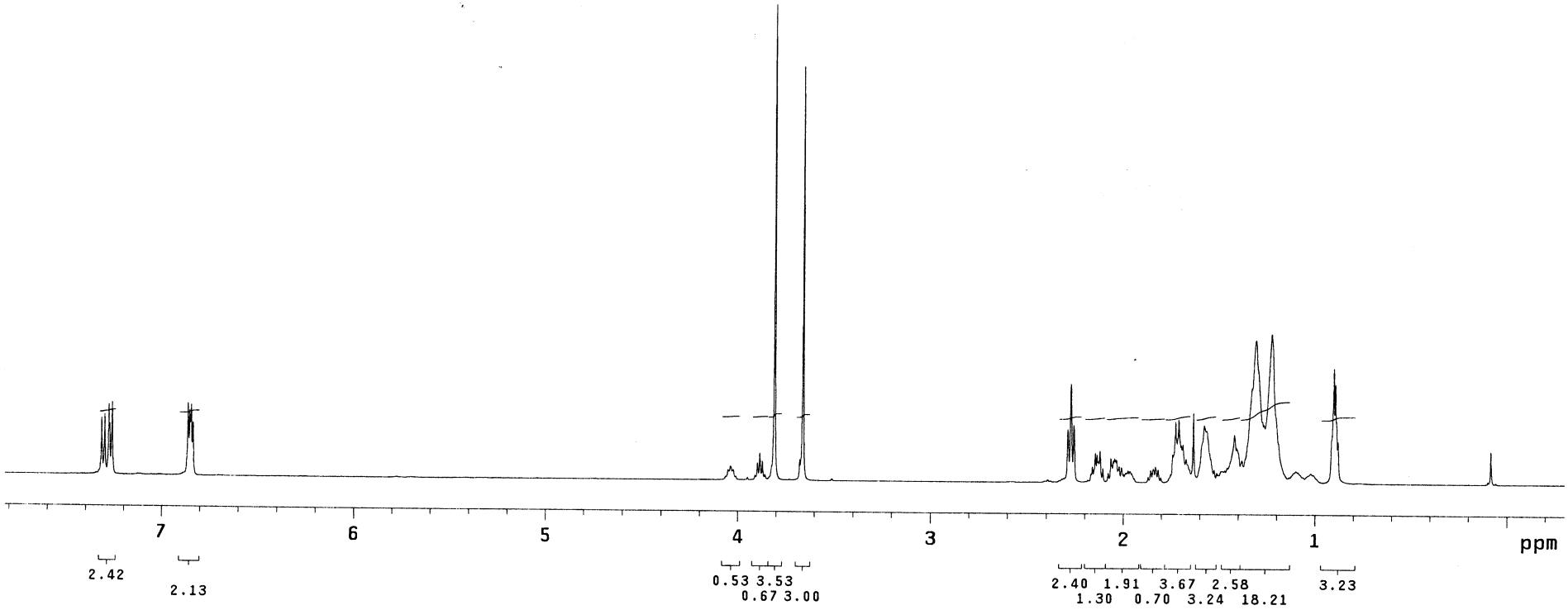
zc2-136a

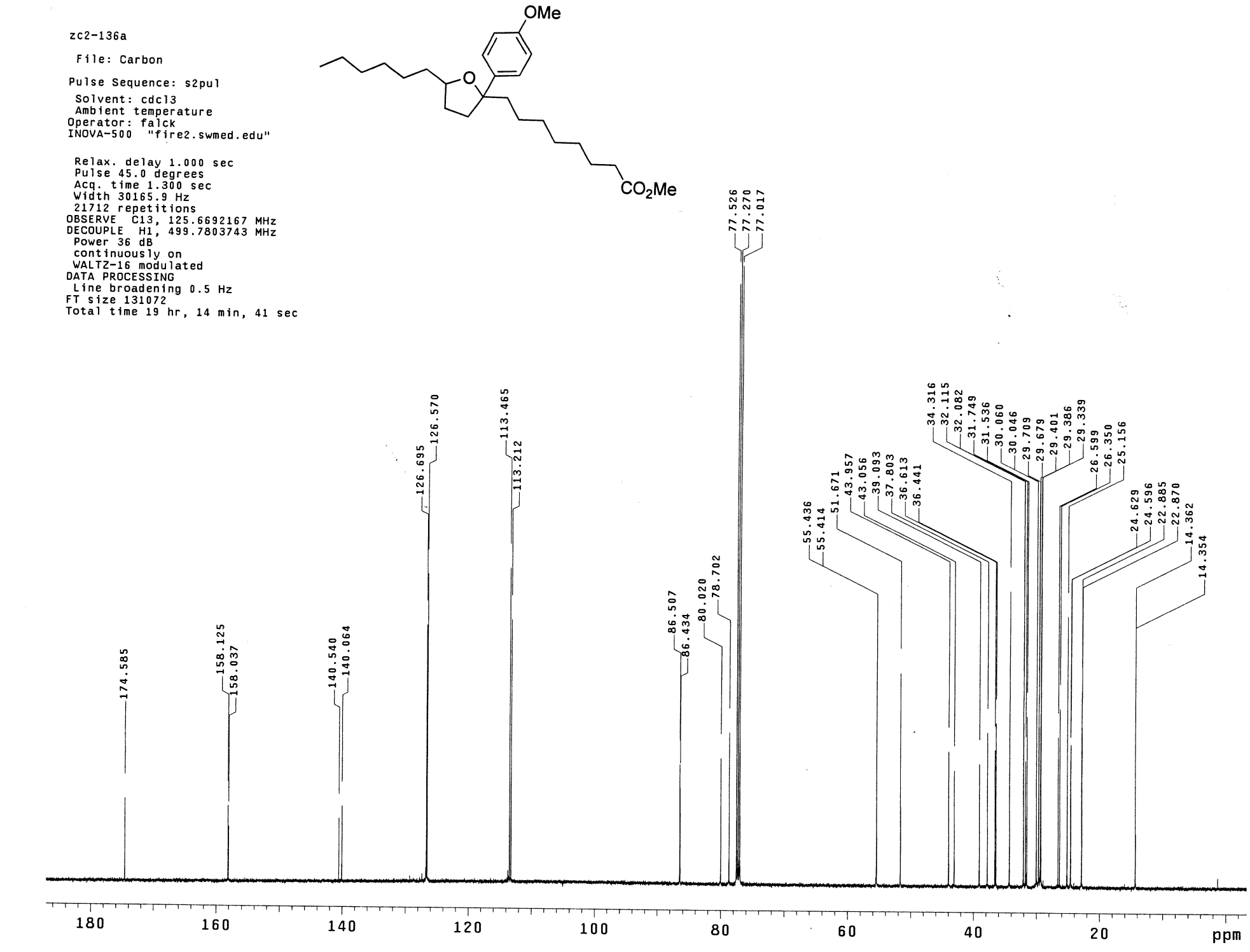
File: Proton

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

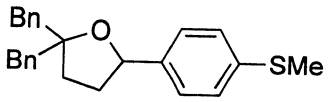





zc2-136a
File: Carbon
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
21712 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 19 hr, 14 min, 41 sec
OMe
O
CO2Me
174.585
158.125
158.037
140.540
140.064
126.695
126.570
113.465
113.212
86.507
86.434
80.020
78.702
77.526
77.270
77.017
55.436
55.414
51.671
43.957
43.056
39.093
37.803
36.613
36.441
34.316
32.115
32.082
31.749
31.536
30.060
30.046
29.709
29.679
29.401
29.386
29.339
26.599
26.350
25.156
24.629
24.596
22.885
22.870
14.362
14.354
180
160
140
120
100
80
60
40
20
ppm

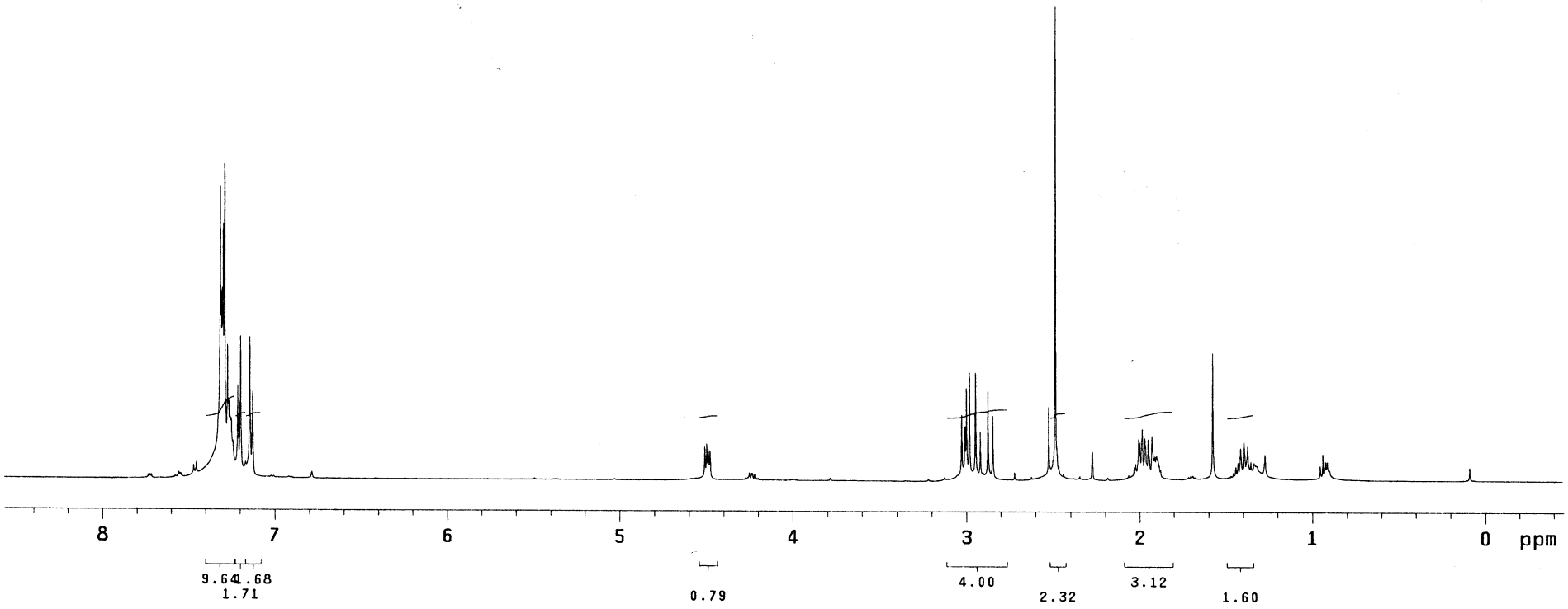
zc2-165

File: Proton



Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

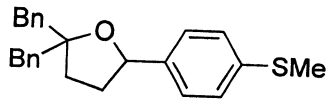


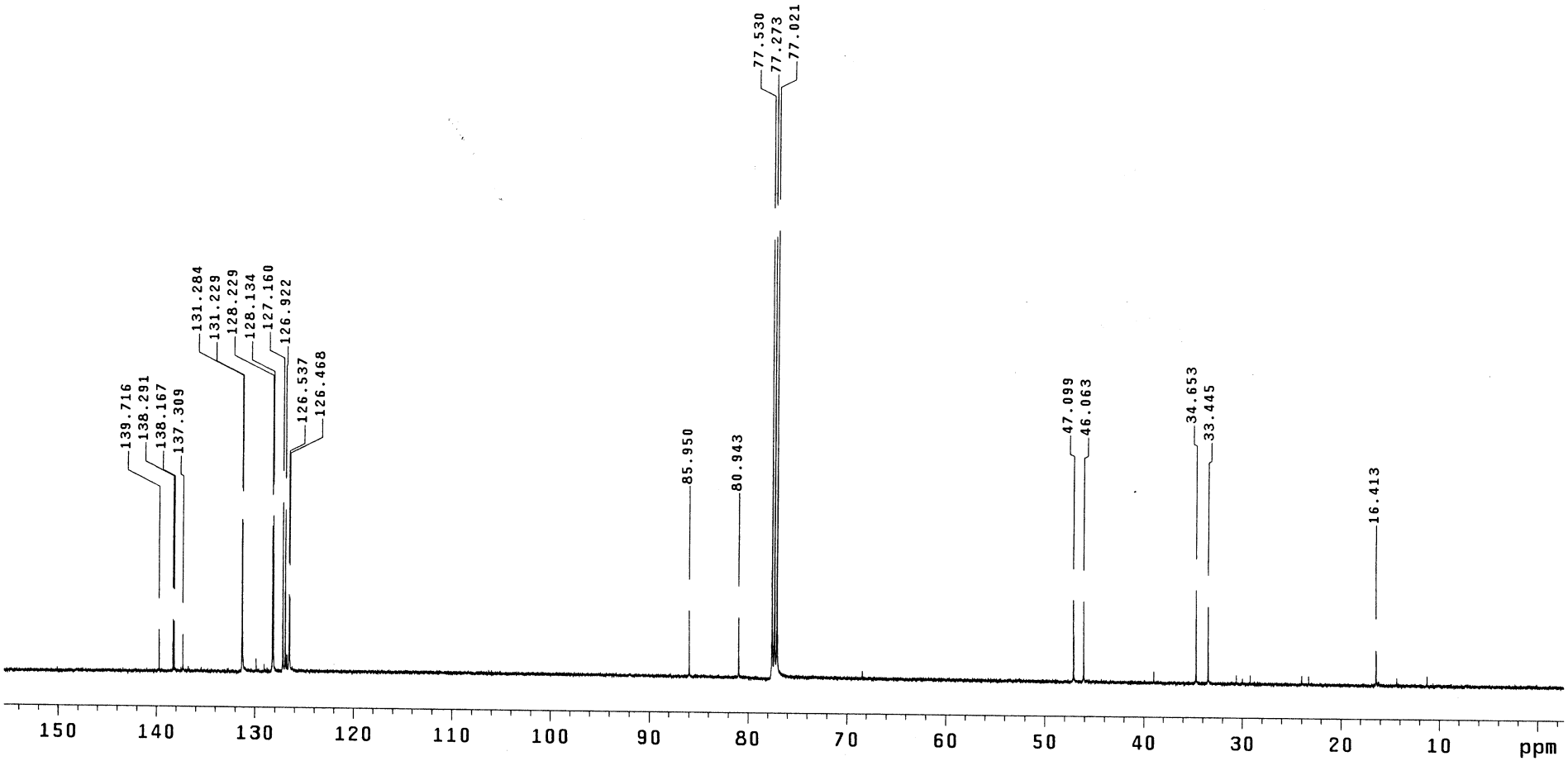
zc2-165

File: Carbon

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
21664 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 32 hr, 4 min, 29 sec



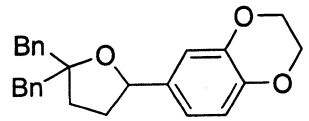


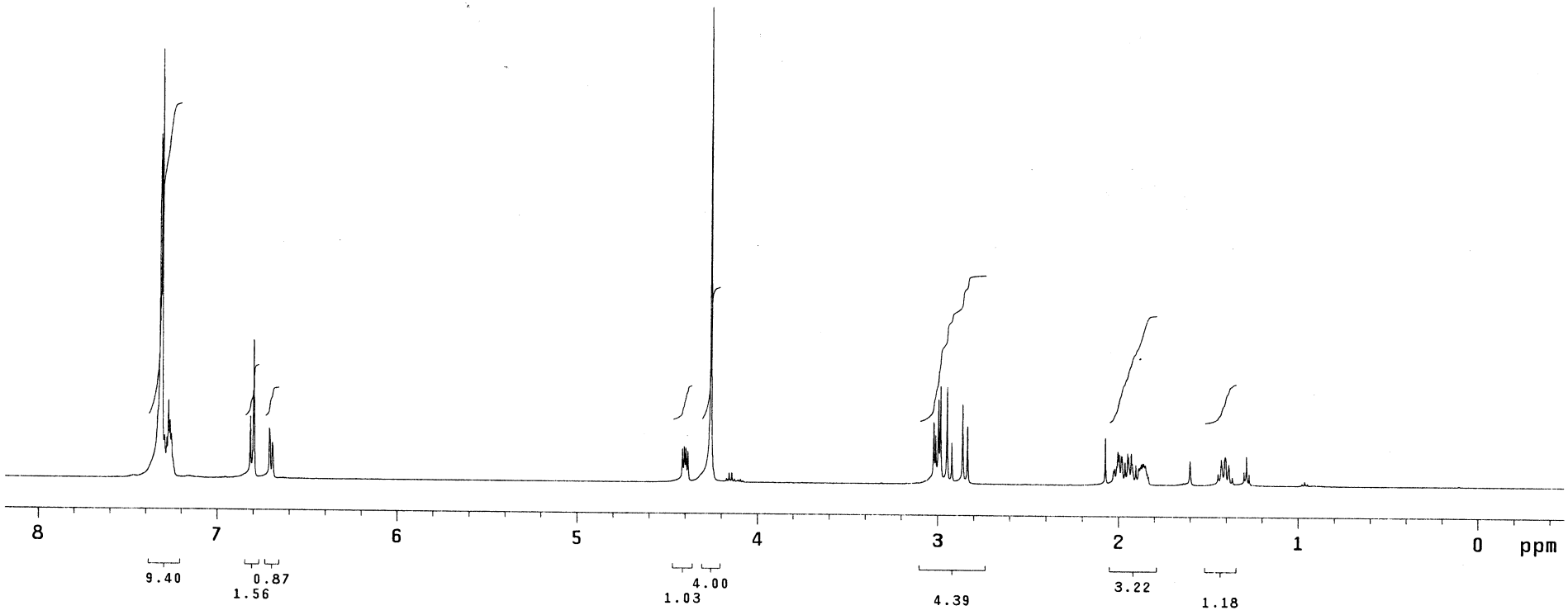
zc2-166

File: zc2-166

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-166
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7778753 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec



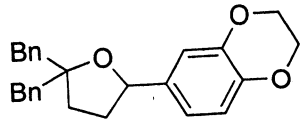


zc2-166

File: Carbon

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"



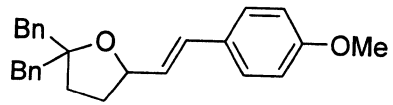
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
408 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 6 hr, 24 min, 53 sec

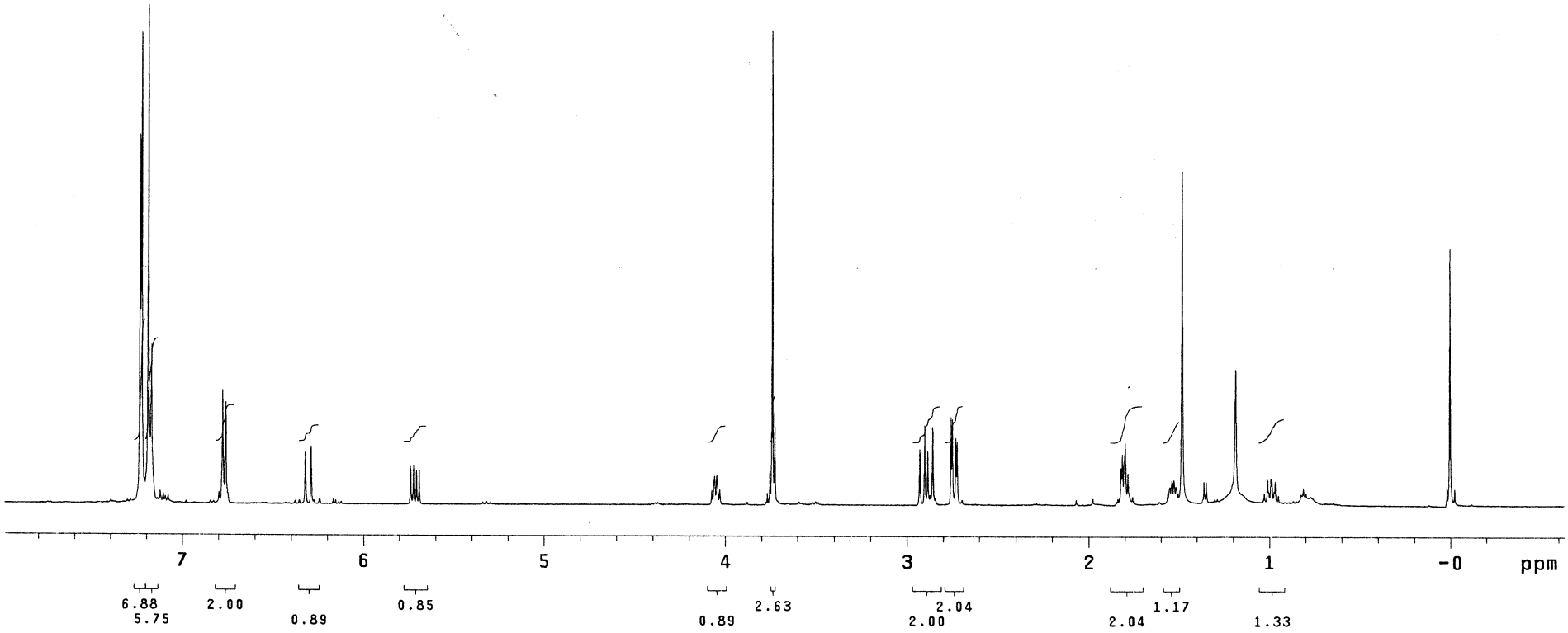
zc2-198

File: zc2-198

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
File: zc2-198
INOVA-500 "fire2.swmed.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7779166 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec



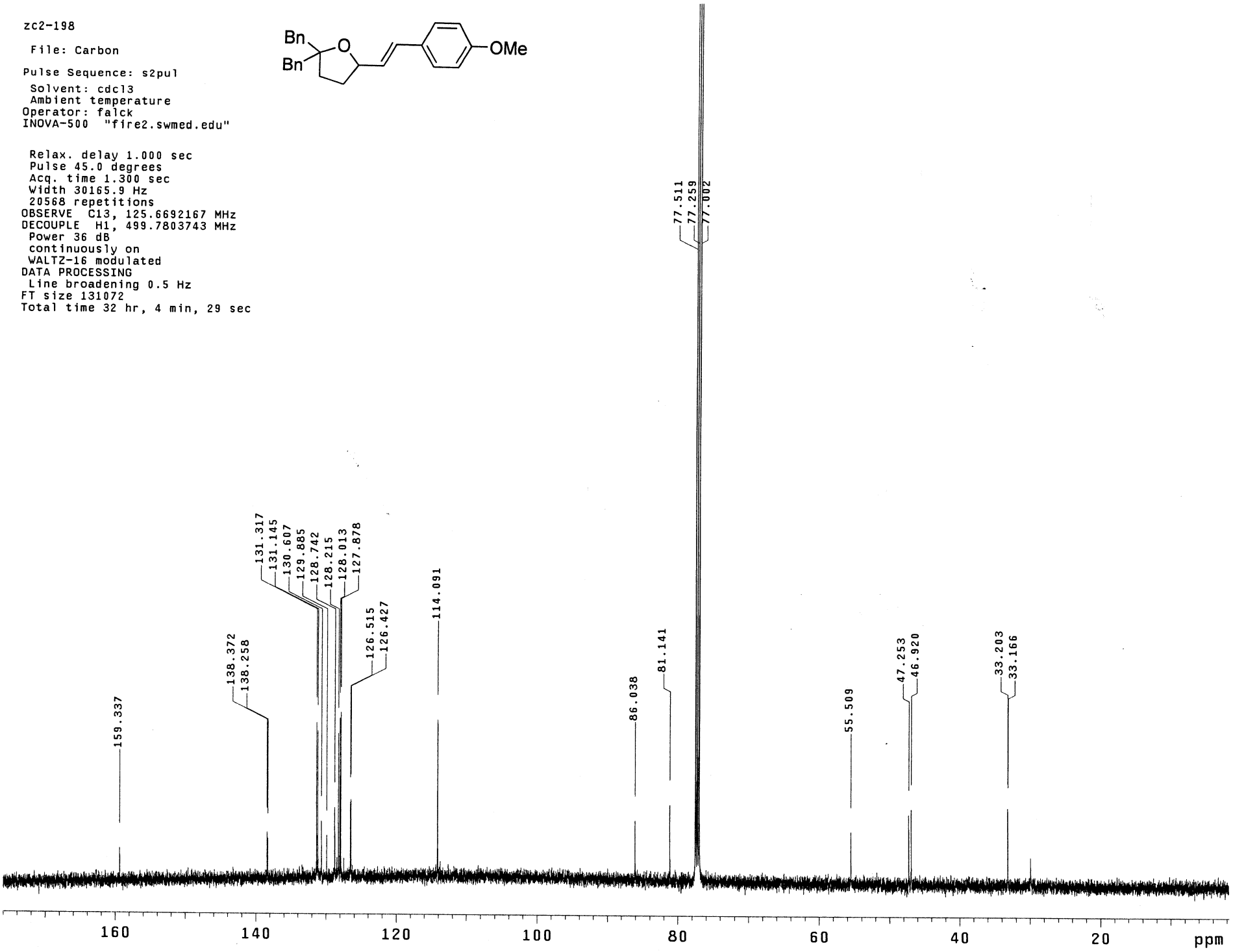


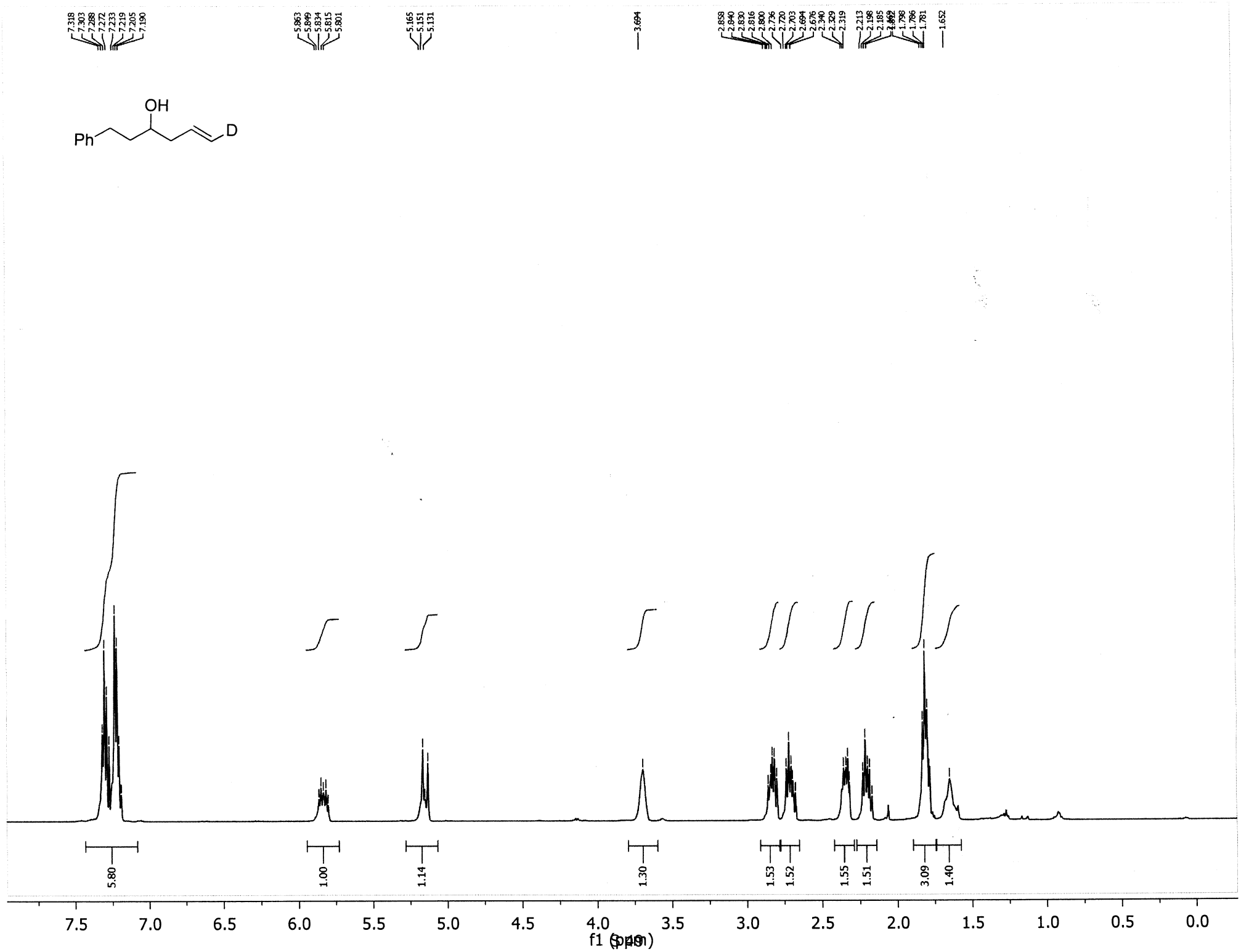
zc2-198

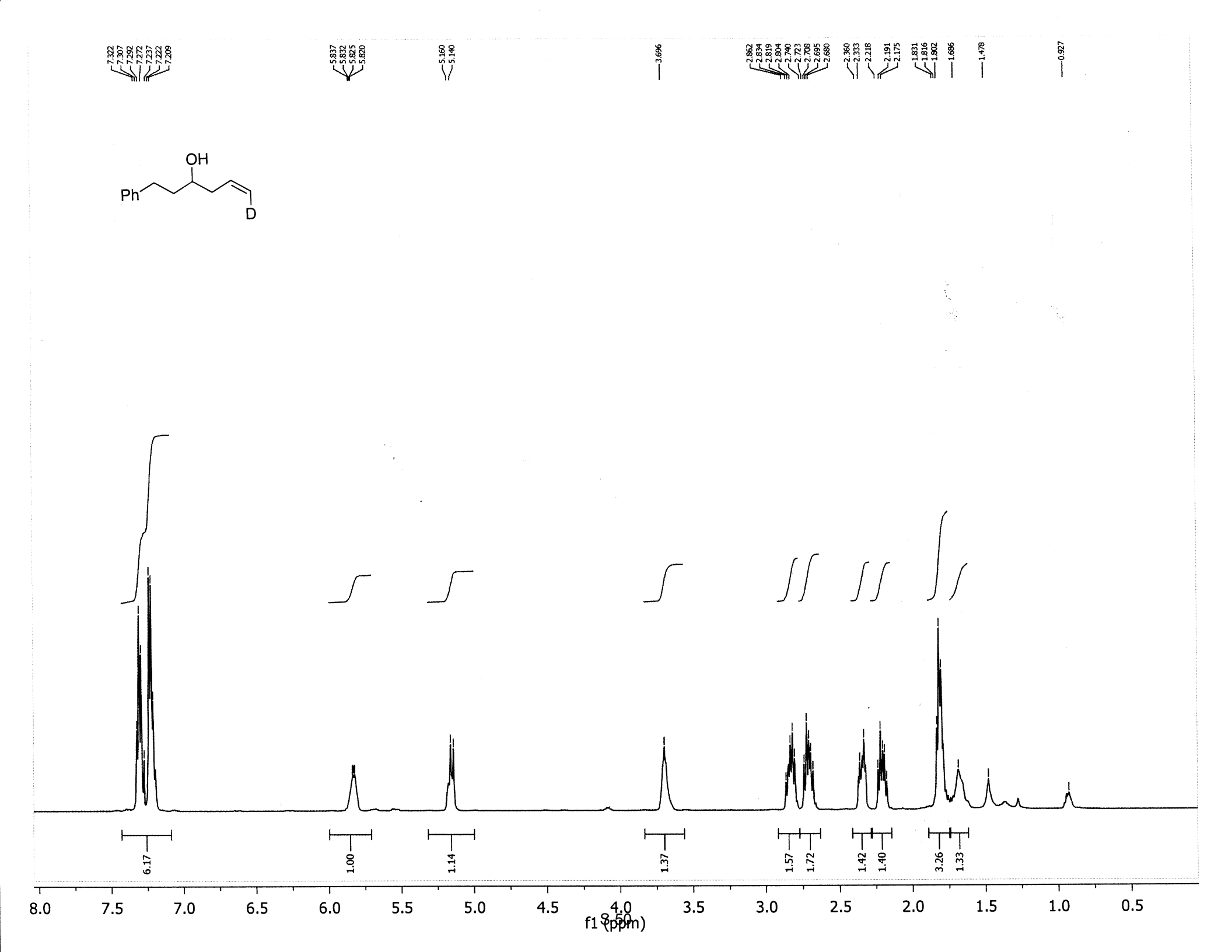
File: Carbon

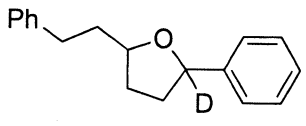
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: falck
INOVA-500 "fire2.swmed.edu"

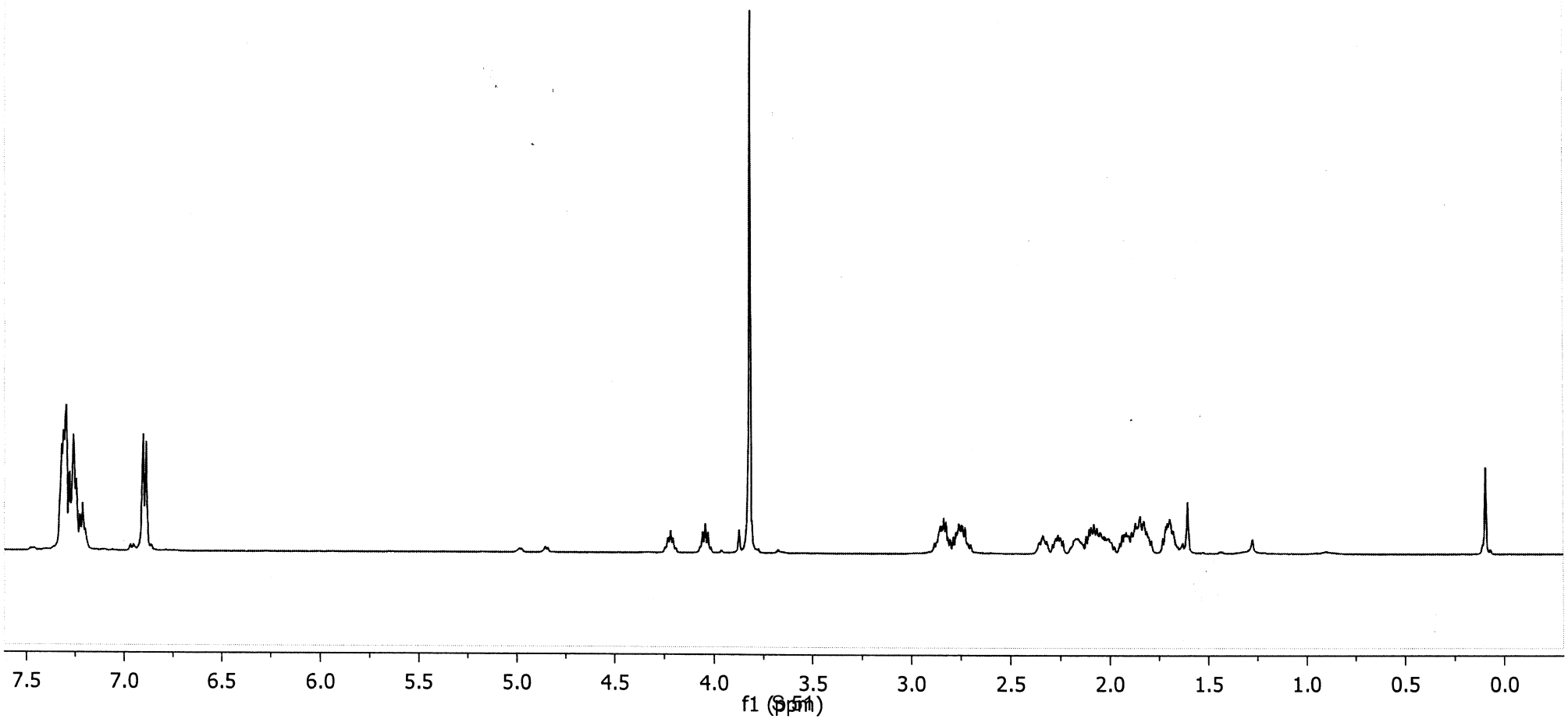
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
20568 repetitions
OBSERVE C13, 125.6692167 MHz
DECOUPLE H1, 499.7803743 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 32 hr, 4 min, 29 sec

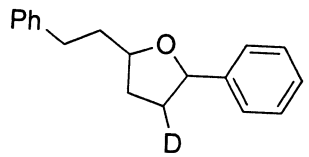


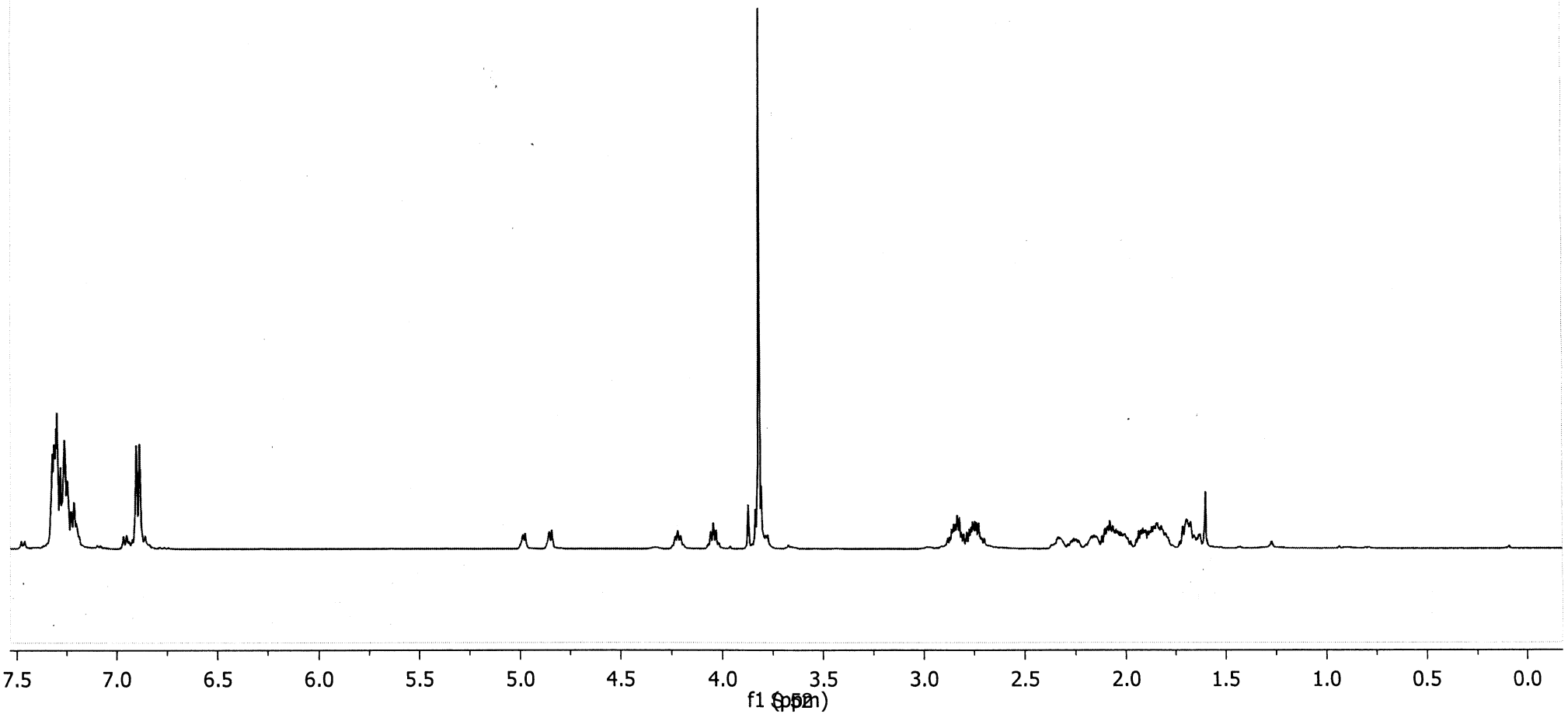

7.318
7.303
7.288
7.272
7.233
7.219
7.205
7.190
5.863
5.849
5.834
5.815
5.801
5.165
5.151
5.131
3.694
2.858
2.840
2.830
2.816
2.800
2.736
2.720
2.703
2.694
2.676
2.340
2.329
2.319
2.213
2.198
2.185
1.798
1.786
1.781
1.652
OH
Ph
D
5.80
1.00
1.14
1.30
1.53
1.52
1.55
1.51
3.09
1.40
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


7.322
7.307
7.292
7.272
7.237
7.222
7.209
5.837
5.832
5.825
5.820
5.160
5.140
3.696
2.862
2.834
2.819
2.804
2.740
2.723
2.708
2.695
2.680
2.360
2.333
2.218
2.191
2.175
1.831
1.816
1.802
1.686
1.478
0.927
OH
Ph
D
6.17
1.00
1.14
1.37
1.57
1.72
1.42
1.40
3.26
1.33
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


Ph
O
D


7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Ph
O
D


7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)