Supplementary information for the paper under the title:

A Useful Synthetic Equivalent of a Hydroxyacetone Enolate

Miljan Bigovic,[a] Veselin Maslak,[a] Zorana Tokic-Vujosevic,[b] Vladimir Divjakovic[c] and Radomir N. Saicic*,[a]

[a)] Faculty of Chemistry, University of Belgrade, Studentski trg 16, P. O. B. 51, 11158 Belgrade, Serbia, Fax: (+381) 11 32 82 537, E-mail: rsaicic@chem.bg.ac.rs
[b)] Faculty of Pharmacy, Vojvode Stepe 450, Belgrade
[c)] Faculty of Science, Trg Dositeja Obradovica 3, 21000 Novi Sad

Table of content

General experimental

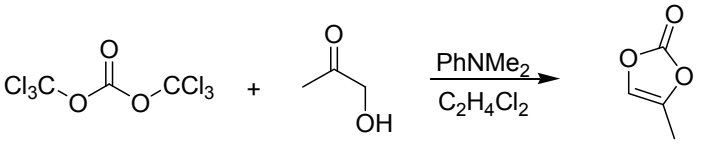
All chromatographic separations[1] were performed on Silica, 10-18, 60A, ICN Biomedicals. Standard techniques were used for the purification of reagents and solvents.[2] Indium was obtained from Aldrich (cat. No. 277959, 99.99% pure, with 1% Mg as anticaking agent). NMR spectra were recorded on a Varian Gemini 200, (^{1}H NMR at 200 MHz, ^{13}C NMR at 50 MHz, for samples in deuterated chloroform), and on Bruker Avance III 500 (^{1}H NMR at 500 MHz, ^{13}C NMR at 125 MHz). Chemical shifts are expressed in ppm (δ) using tetramethylsilane as internal standard, coupling constants (J) are in Hz. IR spectra were recorded on a Nicolet 6700 FT instrument, and are expressed in cm^{-1}. Mass spectra were obtained on Agilent technologies 6210 TOF LC/MS instrument (LC: series 1200). Microanalyses were performed at the Vario EL III instrument CHNOS Elementar Analyzer, Elementar

Analysensysteme GmbH, Hanau-Germany. Melting points were determined on a Kofler hot-stage apparatus and are uncorrected. 1-Tosyl-1H-indole-3-carbaldehyde[3] and enantiomerically pure 2,3,4,5-tetra-*O*-acetyl-D-arabinose[4] were prepared according to literature procedures.

X-ray crystal structure determination

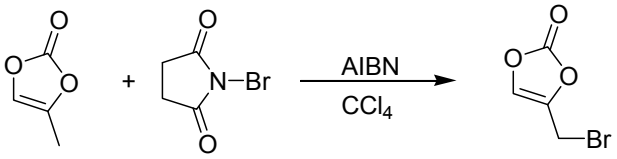
A single colorless crystal was selected and glued on glass fiber. Diffraction data were collected on an Oxford Diffraction KM4 four-circle goniometer equipped with Sapphire CCD detector. The crystal to detector distance was 45.0 mm and a graphite monochromated MoKα (λ = 0.71073 Å) X-radiation was employed in the measurements. The frame widths of 1° in ω, with 19 and 27 s were used to acquire each frame. More than a hemisphere of three-dimensional data was collected in all measurements. The data were reduced using the Oxford Diffraction program CrysAlis^{Pro}. A semiempirical absorption-correction based upon the intensities of equivalent reflections was applied, and the data were corrected for Lorentz, polarization, and background effects. Scattering curves for neutral atoms, together with anomalous-dispersion corrections, were taken from *International Tables for X-ray Crystallography*.[5] The structures were solved by direct methods,[6] and the figures were drawn using MERCURY.[7] Refinements were based on F^2 values and done by full-matrix least-squares[8] with all non-H atoms anisotropic. The positions of all non H-atoms were located by direct methods. The positions of hydrogen atoms were found from the inspection of the difference Fourier maps. The final refinement included atomic positional and displacement parameters for all non-H atoms. The non-H atoms were refined anisotropically. However, at the final stage of the refinement, H atoms belonging to molecules were positioned geometrically (O−H = 0.82 and C−H = 0.93-0.97 Å) and refined using a riding model with fixed isotropic displacement parameters.

4-Methyl-1,3-dioxol-2-one



This compound was obtained in two steps, from hydroxyacetone, according to the modified literature procedure described for the preparation of the 4,5-dimethyl derivative.[9] Triphosgene (3.0 g; 11 mmol) was added to a cold (0 °C) solution of hydroxyacetone (2.1 g; 28 mmol) in dichloroethane (20 mL), followed by a dropwise addition of *N,N*-dimethylaniline (3.7 g; 4 mL; 30 mmol), while maintaining temperature below 8 °C.The reaction mixture was stirred for 15 min at 0 °C, then two more hours at rt. The reaction mixture was cooled to 5 °C, washed with cold 3 M aqueous hydrochloric acid (40 mL), water (30 mL) and brine (30 mL), dried over anh. $MgSO_4$, filtered and concentrated under reduced pressure to the half of the initial volume. The residue was heated to reflux for three hours. The solvent was completely removed at rotavap, and the remaining oil was heated in a stream of argon to 170 °C, for two and half hours, when considerable darkening occurs. The crude mixture was distilled under reduced pressure, to give 1.4 g (52%) of 4-methyl-1,3-dioxol-2-one, bp 130-140 °C/30 mmHg, as a light-yellow oil. Although the compound has been mentioned in the literature,[10] no spectral data were given: IR_{film}: 3169, 2934, 1828, 1801, 1124, 1071. ^{1}H NMR: 6.84 (q, *J*=1.6, H); 2.13 (d, *J*=1.6, 3H). ^{13}C NMR: 153.4 (C); 141.1 (C); 126.0 (CH); 9.7 (CH_3). Anal. calcd. for $C_4H_4O_3$: C 48.00; H 4.00; found: C 47.93; H 4.16.

4-(Bromomethyl)-1,3-dioxol-2-one 1



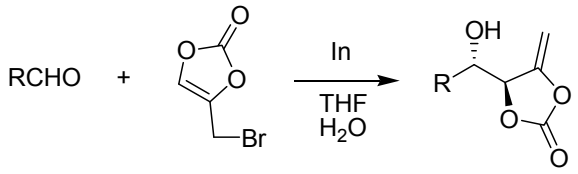
This compound was obtained according to the literature procedure.[11] A mixture of 4-methyl-1,3-dioxol-2-one (1 g; 10 mmol), *N*-bromosuccinimide (2.3 g; 13 mmol), azo-bis-isobutyronitrile (AIBN; 10 mg) and carbon tetrachloride (40 mL) was heated to reflux for 1.5 h. The reaction mixture was concentrated to the half of the initial volume, filtered, concentrated at rotavap and distilled under reduced pressure, to give 1.3 g (72%) of 4-(bromomethyl)-1,3-dioxol-2-one **1**, as a light-yellow oil, bp 100-110 °C/1 mmHg). No ^{13}C NMR data are provided in the literature, and the literature ^{1}H NMR was

recorded in CCl_4: IR_{film}: 3169, 2934, 1828, 1801, 1124, 1071. ^1H NMR ($CDCl_3$): 7.12 (t, *J*=1.2, H), 4.19 (d, *J*=1.2, 2H) ^{13}C NMR: 153.4 (C); 141.1 (C); 126.0 (CH); 9.7 (CH_3). Anal. calcd. for $C_4H_3BrO_3$: C 26.82; H 1.68; Found: C 26.74; H 1.81.

Due to a ban on the use of carbon tetrachloride, the possibility of using other solvents was investigated. The reaction could be performed in an analogous way in 1,2-dichloroethane: under these conditions 4-(bromomethyl)-1,3-dioxol-2-one **1** was obtained in 49% yield.

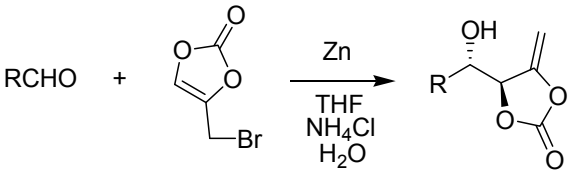
General procedure for the allylation of carbonyl compounds with 4-(bromomethyl)-1,3-dioxol-2-one 1

Indium-promoted allylation



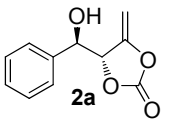
Aldehyde (0.19 mmol) was added to a mixture of **1** (50 mg; 0.28 mmol), indium (32.1 mg; 0.28 mmol), THF (0.5 mL) and water (1 mL), and the reaction mixture was stirred at rt. The reaction was monitored by TLC (eluent: 20% acetone in petroleum-ether) and it was usually complete after 15 min. The reaction mixture was diluted with dichloromethane (5 mL) and water (5 mL), the aqueous layer was extracted with dichloromethane (2 x 5 mL), combined organic extracts were dried over anh. $MgSO_4$, filtered, concentrated under reduced pressure and the crude product was purified by dry-flash chromatography.

Zinc-promoted allylation



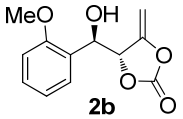
Aldehyde (0.09 mmol) was added to a mixture of **1** (50 mg; 0.28 mmol), zinc (23 mg; 0.36 mmol), THF (0.2 mL) and saturated aqueous solution of NH_4Cl (0.8 mL), and the reaction mixture was stirred at rt. The reaction was monitored by TLC (eluent: 20% acetone in petroleum-ether) and it was usually complete after 30 min. Work-up as previously described provided the crude product, which was purified by dry-flash chromatography.

4-(Hydroxy(phenyl)methyl)-5-methylene-1,3-dioxolan-2-one 2a



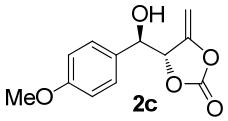
According to the general procedure for the indium-mediated allylation, starting from benzaldehyde (19.7 mg, 19 µL, 0.19 mmol); after purification by column chromatography (eluent: 20% acetone in hexanes), 37.7 mg (96%) of the title compound **2a** was obtained, as a mixture of diastereoisomers in a ratio *anti*:*syn*=12:1. Recrystallization from 5% EtOAc in hexanes afforded white crystals of pure ***2a-anti***, mp 70-71 °C. FT-IR (KBr): 3477, 3064, 2891, 1832, 1690, 1341, 1280, 1147, 1062, 766, 708. ^1H NMR δ: 7.43-7.31 (m, 5H), 5.28 (ddd, J_1=4.1, J_2=2.3, J_3=1.7, 1H), 5.14 (bt, *J*=4.1, 1H), 4.78 (dd, J_1=3.9, J_2=2.3, 1H), 3.87 (dd, J_1=3.9, J_2=1.7, 1H), 2.78 (d, *J*=4.1, 1H). ^{13}C NMR δ: 152.3 (C), 148.6 (C), 136.1 (C), 128.7 (CH), 128.6 (CH), 126.4 (CH), 89.7 (CH_2), 82.3 (CH), 73.4 (CH). HRMS (ESI): calcd. for [$C_{11}H_{10}O_4$ + NH_4^+]: 224.0923, found for $[M+NH_4]^+$: 224.0911. Anal. calcd. for $C_{11}H_{10}O_4$: C 64.08, H 4.85; found: C 63.81, H 4.73.

4-(Hydroxy(2-methoxyphenyl)methyl)-5-methylene-1,3-dioxolan-2-one 2b



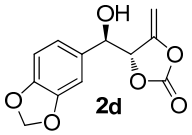
According to the general procedure for the indium-mediated allylation, starting from 2-methoxybenzaldehyde (38 mg, 0.28 mmol); purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 38 mg (57%) of **2b-*anti***, followed by 29 mg (39%) of **2b-*syn*** (Combined yield: 63.4 mg; 96%). Physical data for **2b-*anti***: recrystallized from 5% EtOAc in hexanes, white solid, mp 106-108 °C. FT-IR (KBr): 3487, 3011, 2969, 1809, 1690, 1492, 1346, 1248, 1158, 1054, 756. ^{1}H NMR δ: 7.48-7.46 (m, 1H), 7.35-7.32 (m, 1H), 7.01 (td, J_1=7.9, J_2=0.7, 1H), 6.90 (dd, J_1=7.9, J_2=1.3, 1H), 5.42 (ddd, J_1=3.3, J_2=2.2, J_3=1.5, 1H), 5.38 (dd, J_1=5.7, J_2=3.3, 1H), 4.76 (dd, J_1=3.7 J_2=2.2, 1H), 3.87 (s, 3H), 3.68 (dd, J_1=3.7, J_2=1.5, 1H), 3.02 (d, J=5.7, 1H). ^{13}C NMR δ: 155.7 (C), 152.7 (C), 149.0 (C), 129.6 (CH), 128.0 (CH), 124.1 (C), 120.7 (CH), 110.1 (CH), 89.3 (CH_2), 80.5 (CH), 69.5 (CH), 55.4 (CH_3). HRMS (ESI): calcd. for [$C_{12}H_{12}O_5$ + NH_4^+]: 254.1028, found for [M+NH_4]$^+$: 254.1026. Anal. calcd. for $C_{12}H_{12}O_5$: C 61.02, H 5.08; found: C 60.68; H 5.14. Physical data for **2b-*syn***: recrystallized from 5% EtOAc in hexanes, white solid, mp 80 °C. FT-IR (KBr): 3462, 3072, 2970, 1807, 1684, 1603, 1493, 1463, 1359, 1236, 1164, 1084, 1050, 848, 763. ^{1}H NMR δ: 7.44 (dd, J_1=7.8, J_2=1.5, 1H), 7.36-7.32 (m, 1H), 7.02 (td, J_1=7.8, J_2=0.7, 1H), 6.93-6.91 (m, 1H), 5.36 (ddd, J_1=3.7, J_2=2.0, J_3=1.7, 1H), 5.17 (dd, J_1=6.0, J_2= 3.7, 1H), 4.90 (dd, J_1=3.9, J_2=2.0, 1H), 4.25 (dd, J_1=3.9, J_2=1.7, 1H), 3.87 (s, 3H), 2.82 (d, J=6.0, 1H). ^{13}C NMR δ: 156.1 (C), 152.1 (C), 151.0 (C), 129.8 (CH), 128.0 (CH), 125.3 (C), 121.0 (CH), 110.5 (CH), 87.7 (CH_2), 81.5 (CH), 71.1 (CH), 55.5 (CH_3). HRMS (ESI): calcd. for $C_{12}H_{12}O_5$: 236.0685, found for [M]$^+$: 236.0677. Anal. calcd. for $C_{12}H_{12}O_5$: C 61.02, H 5.08; found: C 60.77, H 5.26.

4-(Hydroxy(4-methoxyphenyl)methyl)-5-methylene-1,3-dioxolan-2-one 2c



According to the general procedure for the indium-mediated allylation, starting from 4-methoxybenzaldehyde (25.8 mg, 23 µl, 0.19 mmol); purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 36.6 mg (82 %) of the title compound **2c**, as a mixture of diastereoisomers in a ratio *anti*:*syn*=8:1 (the ratio of diastereoisomers did not change after crystallization from 5% EtOAc in hexanes). White crystals, m.p. 60-2 °C. Spectral data for **2c**: FT-IR (KBr): 3478, 3013, 2960, 1828, 1690, 1514, 1250, 1145, 1055, 849, 764. ^{1}H NMR δ: 7.29 (d, J=9.0, 2H), 6.90 (d, J=9.0, 2H), 5.25 (ddd, J_1=4.0, J_2=2.2, J_3=1.8, 1H), 5.03 (bt, J_1=4.0, 1H), 4.81 (dd, J_1=3.4, J_2=2.2, 1H), 3.95 (dd, J_1=3.4, J_2=1.8, 1H), 3.80 (s, 3H), 3.26 (d, J=4.0, 1H). ^{13}C NMR δ: 159.8 (C), 148.8 (C), 128.5 (C), 128.2 (C), 127.8 (CH), 113.9 (CH), 89.5 (CH_2), 82.3 (CH), 73.2 (CH), 55.2 (CH_3). HRMS (ESI): calcd. for [$C_{12}H_{12}O_5$ + Na^+]: 259.0582, found for [M+Na]$^+$: 259.0577.

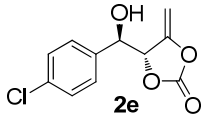
4-(Benzo[*d*][1,3]dioxol-5-yl(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one 2d



According to the general procedure for the indium-mediated allylation, starting from piperonal (28 mg, 0.19 mmol); purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 45.2 mg (95%) of the title compound **2d**, as a mixture of diastereoisomers in a ratio *anti*:*syn*=6:1. White crystals (recrystallized from 10% EtOAc in petroleum-ether), m.p. 103-4 °C. Spectral data for **2d**: FT-IR (KBr): 3455, 3020, 2909, 1825, 1695, 1499, 1338, 1246, 1160, 1062, 1036, 931, 865, 742. ^{1}H NMR δ: 6.89-6.82 (m, 3H), 5.99 (bs, 2H), 5.21 (ddd, J_1=4.2, J_2=2.3, J_3=1.9, 1H), 5.00 (bt, J=4.2, 1H), 4.87 (dd, J_1=3.8, J_2=2.3, 1H), 4.05 (dd, J_1=3.8, J_2=1.9, 1H), 2.51 (bd, J=4.2, 1H). ^{13}C NMR δ: 152.0 (C), 148.8 (C), 148.0 (C), 148.0 (C), 130.1 (C), 120.1 (CH), 108.4 (CH), 106.9 (CH), 101.4

(CH_2), 89.8 (CH_2), 82.0 (CH), 73.6 (CH). HRMS (ESI): calcd. for [$C_{12}H_{10}O_6$ + Na^+]: 273.0375, found for [M+Na]$^+$: 273.0356. Anal. calcd. for $C_{12}H_{10}O_6$: C 57.60, H 4.00; found: C 57.29, H 4.25.

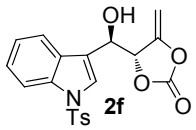
4-((4-Chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one 2e



2e

According to the general procedure for the indium-mediated allylation, starting from 4-chloro-benzaldehyde (33 mg, 0.23 mmol); purification by column chromatography (eluent: 30% acetone in petroleum-ether) afforded 52.8 mg (96%) of the title compound **2e**, as a mixture of diastereoisomers in a ratio *anti*:*syn*=7:1. Monocrystal of pure **2e-*anti***, suitable for X-ray crystallographic analysis, was obtained by crystallization from 5% EtOAc in hexanes. Physical data for **2e-*anti***: white, crystalline compound, mp 88-90 °C. FT-IR (KBr): 3847, 3075, 2983, 1816, 1691, 1342, 1154, 1062, 871, 744. ^{1}H NMR δ: 7.39 (d, *J*=8.5, 2H), 7.34 (d, *J*=8.5, 2H), 5.24 (ddd, J_1=4.4, J_2=2.0, J_3=1.8, 1H), 5.11 (bt, *J*=4.4, 1H), 4.84 (dd, J_1=4.0, J_2=2.0, 1H), 3.91 (dd, J_1=4.0, J_2=1.8, 1H), 2.96 (d, *J*=4.4, 1H). ^{13}C NMR δ: 152.2 (C), 148.4 (C), 134.7 (C), 134.6 (C), 128.8 (CH), 127.9 (CH), 89.9 (CH_2), 82.0 (CH), 72.9 (CH). HRMS (ESI): calcd. for [$C_{11}H_9O_4Cl$ + Na^+]: 263.0087, found for [M+Na]$^+$: 263.0083. Anal. calcd. for $C_{11}H_9O_4Cl$: C 54.88, H 3.74; found: C 54.67, H 3.79.

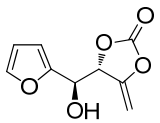
4-(Hydroxy(1-tosyl-1*H*-indol-3-yl)methyl)-5-methylene-1,3-dioxolan-2-one 2f



2f

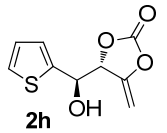
According to the general procedure for the indium-mediated allylation, starting from 1-tosyl-1*H*-indole-3-carbaldehyde (23 mg, 77 μmol); purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 23 mg (78%) of the title compound **2f**. Physical data for **2f**: pale-yellow oil, FT-IR (film): 3483, 3058, 2927, 1833, 1690, 1447, 1370, 1274, 1173, 1126, 1065, 744, 674, 574. ^{1}H NMR δ: 8.03 (d, *J*=8.4, 1H), 7.78 (d, *J*=7.8, 2H), 7.86 (s, 1H), 7.53 (d, *J*=7.8, 1H), 7.41-7.22 (m, 4H), 5.35 (bs, 2H), 4.78-4.76 (m, 1H), 3.79-3.76 (m, 1H), 2.77 (bs, 1H), 2.35 (s, 3H). ^{13}C NMR δ: 152.2 (C), 148.3 (C), 145.4 (C), 135.2 (C), 134.7 (C), 130.0 (C), 127.7 (C), 126.9 (CH), 125.4 (CH), 125.2 (CH), 123.7 (CH), 119.4 (CH), 118.3 (CH), 114.0 (CH), 89.8 (CH_2), 80.6 (CH), 68.4 (CH), 21.5 (CH_3). HRMS (ESI): calcd. for [$C_{20}H_{17}NO_6S$ + Na^+]: 422.0674, found for [M+Na]$^+$: 422.0683.

4-(Furan-2-yl(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one 2g



According to the general procedure for the indium-mediated allylation, starting from furane-2-carbaldehyde (18.2 mg, 0.19 mmol); purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 21 mg (56%) of **2g-*anti***, followed by 10 mg (27%) of **2g-*syn*** (combined yield: 83%). Physical data for **2g-*anti***: colorless oil. FT-IR (film): 3460, 2925, 1827, 1692, 1345, 1271, 1144, 1061, 859, 749. ^{1}H NMR δ: 7.45 (dd, J_1=1.9, J_2=0.9, 1H), 6.46-6.45 (m, J_1=3.3, J_2=0.9, 1H), 6.42 (dd, J_1=3.3, J_2=1.9, 1H), 5.45 (ddd, J_1=3.7, J_2=2.2, J_3=2.0, 1H), 5.08 (ddd, J_1=6.8, J_2=3.7, J_3=0.7, 1H), 4.90 (dd, J_1=3.9, J_2=2.2, 1H), 4.10 (dd, J_1=3.9, J_2=2.0, 1H), 2.57 (d, *J*=6.8, 1H). ^{13}C NMR δ: 151.8 (C), 149.8 (C), 148.5 (C), 143.0 (CH), 110.8 (CH), 109.1 (CH), 89.5 (CH_2), 80.2 (CH), 68.9 (CH). HRMS (ESI): calcd. for [$C_9H_8O_5$ + CH_3COO^-]: 255.0505, found for [M+CH_3COO]$^-$: 255.0518. Physical data for **2g-*syn***: colorless oil. FT-IR (film): 3461, 2926, 1823, 1692, 1352, 1278, 1148, 1072, 860, 747. ^{1}H NMR δ: 7.45 (dd, J_1=1.8, J_2=1.0, 1H), 6.48 (dt, J_1=2.6, J_2=1.0, 1H), 6.42 (dd, J_1=2.6, J_2=1.8, 1H), 5.42 (ddd, J_1=4.9, J_2=2.3, J_3=2.0, 1H), 4.93 (dd, J_1=4.2, J_2=2.3, 1H), 4.91 (t, *J*=4.9, 1H), 4.22 (dd, J_1=4.2, J_2=2.0, 1H), 2.56 (d, *J*=4.9, 1H). ^{13}C NMR δ: 151.6 (C), 149.9 (C), 149.6 (C), 143.2 (CH), 110.8 (CH), 109.6 (CH), 88.8 (CH_2), 80.1 (CH), 69.1 (CH). HRMS (ESI): calcd. for [$C_9H_8O_5$ + $HCOO^-$]: 241.0348, found for [M+$HCOO$]$^-$: 241.0358.

4-(Hydroxy(thiophen-2-yl)methyl)-5-methylene-1,3-dioxolan-2-one 2h

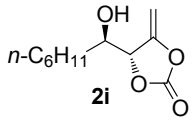


2h

According to the general procedure for the indium-mediated allylation, starting from thiophene-2-carbaldehyde (21 mg, 0.19 mmol); purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 36.5 mg (91%) of **2h**, as a mixture of diastereoisomers in a ratio *anti*:*syn*=6.4:1.

Physical data for **2h**: pale-yellow oil, FT-IR (film): 3462, 3111, 3023, 1828, 1691, 1346, 1273, 1145, 1057, 856, 709. ^{1}H NMR δ: 7.34 (dd, J_1=1.8, J_2=1.6, 1H), 7.09-7.02 (m, 2H), 5.36-5.27 (m, 2H), 4.88 (dd, J_1=3.6, J_2=1.9, 1H), 4.12 (dd, J_1=3.6, J_2=1.6, 1H), 3.29 (d, J=5.0, 1H). ^{13}C NMR δ: 152.1 (C), 148.5 (C), 139.6 (C), 127.3 (CH), 126.1 (CH), 125.4 (CH), 89.9 (CH_2), 81.9 (CH), 70.8 (CH). HRMS (ESI): calcd. for [$C_9H_8O_4S$ + Na^+]: 235.0041, found for $[M+Na]^+$: 235.0027.

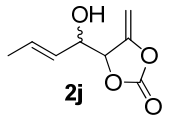
4-(1-Hydroxyheptyl)-5-methylene-1,3-dioxolan-2-one 2i



2i

According to the general procedure for the indium-mediated allylation, starting from heptanal (16 mg, 0.14 mmol); purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 26.4 mg (88%) of **2i**, as an unseparable mixture of diastereoisomers in a ratio *anti*:*syn*=6:1. Physical data for **2i**: colorless oil, FT-IR (film): 3479, 2930, 2856, 1831, 1690, 1463, 1348, 1157, 1069. ^{1}H NMR δ: 5.04 (ddd, J_1=3.8, J_2=2.2, J_3=1.9, 1H), 4.97 (dd, J_1=3.8, J_2=2.2, 1H), 4.49 (dd, J_1=3.8, J_2=1.9, 1H), 3.84 (s, 1H), 2.42 (s, 1H), 1.58-1.54 (m, 2H), 1.36-1.30 (m, 8H), 0.89 (t, J=6.8, 3H). ^{13}C NMR δ: 152.3 (C), 149.9 (C), 89.0 (CH_2), 82.0 (CH), 72.0 (CH), 31.6 (CH_2), 31.0 (CH_2), 29.0 (CH_2), 25.3 (CH_2), 22.5 (CH_2), 14.0 (CH_3). HRMS (ESI): calcd. for [$C_{11}H_{18}O_4$ + NH_4^+]: 232.1549, found for $[M+NH_4]^+$: 232.1531.

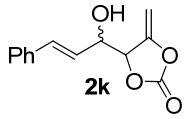
(*E*)-4-(1-Hydroxybut-2-en-1-yl)-5-methylene-1,3-dioxolan-2-one 2j



2j

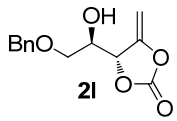
According to the general procedure for the indium-mediated allylation, starting from crotonaldehyde (13 mg, 0.19 mmol), purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 26.4 mg (82%) of **2j**, as an unseparable, equimolar mixture of diastereoisomers. Physical data for **2j**: colorless oil, FT-IR (film): 3464, 3025, 2920, 1825, 1690, 1448, 1348, 1276, 1146, 1067, 971. ^{1}H NMR δ: 5.96-5.88 (m, 2H, *anti, syn*), 5.55 (ddq, J_1=15.0, J_2=3.0, J_3=1.5, 1H, *anti*), 5.49 (ddq, J_1=15.5, J_2=3.5, J_3=1.8, 1H, *syn*), 5.09 (ddd, J_1=5.5, J_2=3.0, J_3=1.5, 1H, *syn*), 5.07 (ddd, J_1=4.3, J_2=2.5, J_3=1.3, 1H, *anti*), 4.95 (dd, J_1=2.3, J_2=1.5, 1H, *anti*), 4.94 (dd, J_1=2.3, J_2=1.3, 1H, *syn*), 4.52 (dd, J_1=3.0, J_2=2.3, 1H, *anti*), 4.47 (dd, J_1=2.5, J_2=2.3, 1H, *syn*), 4.39 (bs, 1H, *syn*), 4.28 (bdd, J_1=3.0, J_2=4.3, 1H, *anti*), 2.45 (bs, 1H, *syn*), 2.30 (bs, 1H, *anti*), 1.78-1.76 (m, 6H, *anti, syn*). ^{13}C NMR δ: ***2j-anti*****:** 152.1 (C), 150.2 (C), 132.9 (CH), 126.3 (CH), 88.4 (CH_2), 81.2 (CH), 73.4 (CH), 17.8 (CH_3); ***2j-syn***: 152.2 (C), 149.4 (C), 131.9 (CH), 125.4 (CH), 89.0 (CH_2), 81.7 (CH), 72.8 (CH), 17.8 (CH_3). HRMS (ESI): calcd. for [$C_8H_{10}O_4$ + NH_4^+]: 188.0923, found for $[M+NH_4]^+$: 188.0914.

(*E*)-4-(1-hydroxy-3-phenylallyl)-5-methylene-1,3-dioxolan-2-one 2k



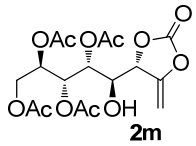
According to the general procedure for the indium-mediated allylation, starting from cinnamaldehyde (25 mg, 0.19 mmol), purification by column chromatography (eluent: 30% acetone in petroleum-ether) afforded 40 mg (91%) of **2k**, as an unseparable mixture of diastereoisomers in a ratio *anti*:*syn*=1.4:1. Physical data for **2k**: colorless oil, FT-IR (film): 3466, 3027, 1828, 1690, 1344, 1274, 1147, 1066, 973, 859, 754. ^{1}H NMR δ: 7.40-7.28 (m, 10H), 6.79 (dd, J_1=16.0, J_2=1.3, 1H, *anti*), 6.77 (d, J=16.0, 1H, *syn*), 6.24 (dd, J_1=16.0, J_2=7.5, 1H, *syn*), 6.15 (dd, J_1=16.0, J_2=5.5, 1H, *anti*), 5.18-5.15 (m, 2H, *anti, syn*), 4.98 (dd, J_1=3.8, J_2=2.3, 1H, *syn*), 4.95 (dd, J_1=4.0, J_2=2.0, 1H, *anti*), 4.66-4.64 (m, 1H, *anti*), 4.55 (dd, J_1=3.8, J_2=1.8, 1H, *syn*), 4.50 (ddd, J_1=7.5, J_2=3.8, J_3=1.3, 1H, *syn*), 4.48 (dd, J_1=4.0, J_2=2.0, 1H, *anti*), 2.63 (bs, 1H), 2.45 (bs, 1H). ^{13}C NMR δ: ***2k-anti***: 152.1 (C), 149.1 (C), 135.5 (CH), 128.7 (CH), 128.6 (CH), 128.5 (C), 126.7 (CH), 123.2 (CH), 89.3 (CH_2), 81.5 (CH), 72.7 (CH); ***2k-syn***: 152.0 (C), 150.0 (C), 128.7 (CH), 128.6 (C), 128.5 (CH), 126.8 (CH), 134.5 (CH), 123.9 (CH), 88.7 (CH_2), 81.3 (CH), 73.6 (CH). HRMS (ESI): calcd. for [$C_{13}H_{12}O_4$ + NH_4^+]: 250.1079, found for [M+NH_4]$^+$: 250.1070.

4-(2-(Benzyloxy)-1-hydroxyethyl)-5-methylene-1,3-dioxolan-2-one 2l



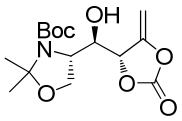
According to the general procedure for the indium-mediated allylation, starting from benzyloxyacetaldehyde (10 mg, 67 μmol), purification by column chromatography (eluent: 20% acetone in petroleum-ether) afforded 12.2 mg (76%) of **2l**, as an unseparable mixture of *syn* and *anti* diastereoisomers in a ratio *anti*:*syn*=3:1, as determined by HPLC analysis. Physical data for **2l**: pale-yellow oil, FT-IR (film): 3457, 3064, 3030, 2922, 2870, 1832, 1690, 1454, 1328, 1272, 1147, 1061, 858, 747, 700. ^{1}H NMR δ: 7.46-7.32 (m, 5H), 5.22 (dd, J_1=2.6, J_2=4.8, 1H, *syn*), 5.17 (ddd, J_1=1.7, J_2=3.9, J_3 =5.6, 1H, *anti*), 4.94-4.92 (m, 2H, *anti, syn*), 4.57-4.50 (m, 3H), 4.43 (dd, J_1=1.7 Hz, J_2=3.9 Hz, 1H, *anti*), 3.99-3-97 (m, 2H, *anti, syn*), 3.68-3.62 (m, 2H), 2.67 (bs, 1H, *anti*), 2.47 (bs, 1H, *syn*). ^{13}C NMR δ: 152.0 (C), 150.7 (C), 150.0 (C), 137.0 (C), 128.6 (CH), 128.2 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 128.0 (CH), 89.4 (CH_2, *anti*), 87.7 (CH_2, *syn*), 79.0 (CH, *syn*), 78.6 (CH, *anti*), 73.7 (CH_2), 71.2 (CH, *syn*), 70.4 (CH, *anti*), 69.2 (CH_2, *syn*), 68.8 (CH_2, *anti*). HRMS (ESI): calcd. for [$C_{13}H_{14}O_5$ + Na^+]: 273.0739, found for [M+Na]$^+$: 273.0738.

(2*R*,3*R*,4*R*,5*S*)-5-hydroxy-5-((*S*)-5-methylene-2-oxo-1,3-dioxolan-4-yl)pentane-1,2,3,4-tetrayl tetraacetate 2m

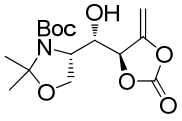


According to the general procedure for the indium-mediated allylation, starting from peracetyl arabinose (63.7 mg; 0.2 mmol); purification by dry-flash chromatography afforded 50 mg (60%) of the title compound **2m**. Colorless crystals, mp 102-5 °C (from petroleum ether/ethyl acetate). α_D +11 (c 0.2, $CHCl_3$). FT-IR (KBr): 3469, 2975, 1834, 1745, 1690, 1372, 1216, 1148, 1055. ^{1}H NMR δ: 5.42 (dd, J_1=1.2, J_2=10, 1H), 5.26 (dd, J_1=1.2, J_2=10, 1H), 5.08 (t, J=2.8, 1H), 5.00-5.04 (m, 1H), 4.90-4.98 (m, 2H), 4.26 (d, J=2.8, 2H), 4.02 (m, 1H), 3.68-3.79 (m, 1H), 2,23 (s, 3H), 2.14 (s, 3H), 2.08 (s, 3H), 2.06 (s, 3H). ^{13}C NMR δ: 172.3 (C), 170.5 (C), 170.2 (C), 169.8 (C), 152.1 (C), 148.6 (C), 90.1 (CH_2), 78.8 (CH), 68.7 (CH), 68.6 (CH), 67.9 (CH), 67.3 (CH), 61.4 (CH_2), 20.7 (CH_3), 20.7 (CH_3), 20.6 (CH_3), 20.5 (CH_3). Anal. calcd. for $C_{17}H_{22}O_{12}$: C 48.81, H 5.30; found: C 48.58, H 5.27.

(*S*)-Tert-butyl 4-((*R*)-hydroxy((*R*)-5-methylene-2-oxo-1,3-dioxolan-4-yl)methyl)-2,2-dimethyloxazolidine-3-carboxylate 2n (*SRR*), and (*S*)-Tert-butyl 4-((*S*)-hydroxy((*S*)-5-methylene-2-oxo-1,3-dioxolan-4-yl)methyl)-2,2-dimethyloxazolidine-3-carboxylate 2n (*SSS*)



2n (*SRR*)

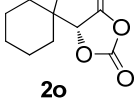


2n (*SSS*)

According to the general procedure for the indium-mediated allylation, starting from 42 mg (0.18 mmol) of the Garner aldehyde (*(4S)-tert*-butyl-4-formyl-2,2-dimethyloxazolidine-3-carboxylate); purification by dry-flash chromatography (SiO_2, eluent: 30% acetone in petroleum-ether) afforded 49 mg (81%) of the title product as an equimolar mixture of diastereoisomers **2n (*SRR*)** and **2n (*SSS*)**. Crystallization from 20% acetone in hexanes afforded white crystals which were also a 1:1 diastereoisomeric mixture. The isomers could be separated by rapid flash chromatography (SiO_2, gradient elution: chloroform/MeOH from 99/1 to 97/3), where **2n (*SRR*)** is a less polar and **2n (*SSS*)** is the more polar isomer. Both isomers were submitted to X-ray crystallographic analysis, the results of which are graphically represented on pages S61 and S63.

Physical data for **2n (*SRR*)**: White, rhombohedral crystals, mp 180-182 °C. FT-IR (KBr): 2924, 2853, 1827, 1689, 1653, 1392, 1372, 1147, 1058, 863, 767. ^{1}H NMR (d_6-DMSO, 340 K) δ: 5.61 (d, J=4.5, 1H), 5.32 (ddd, J_1=2.4, J_2=2.2, J_3=1.5, 1H), 4.90 (dd, J_1=3.5, J_2=2.2, 1H), 4.70 (dd, J_1=3.5, J_2=2.4, 1H), 4.06 (br. t, J=6.5, 1H), 4.03 (dd, J_1=9.5, J_2=1.5, 1H), 3.96-3.91 (m, 2H), 1.54 (s, 3H), 1.42 (s, 3H), 1.41 (s, 9 H). ^{13}C NMR (d_6-DMSO, 340 K) δ: 151.8 (C), 150.1 (C), 93.8 (C), 89.0 (CH_2), 79.8 (CH), 70.3 (CH), 63.5 (CH_2), 57.9 (CH), 28.0 (CH_3), 26.3 (CH_3), 23.4 (CH_3). HRMS (ESI): calcd. for [$C_{15}H_{23}NO_7$ + Na^+]: 352.1372, found for [M+Na]$^+$: 352.1351. $[\alpha]_D^{20}$ -37 (c 1.0, $CHCl_3$). Physical data for **2n *(SSS)***: White, rod-like crystals, mp 114-116 °C. FT-IR (KBr): 3467, 2980, 2936, 1835, 1690, 1392, 1373, 1149, 1059, 862, 768. ^{1}H NMR (d_6-DMSO, 343 K) δ: 5.95 (s, 1H), 5.21 (ddd, J_1=5.5, J_2=3.5, J_3=2.0, 1H), 4.95-4.89 (m, 2H), 4.09 (dd, J_1=8.9, J_2=1.4, 1H), 3.95 (ddd, J_1=6.5, J_2=1.4, J_3=1.0, 1H), 3.87 (dd, J_1=8.9, J_2=6.5, 1H), 3.76 (bs, 1H), 1.50 (s, 3H), 1.46 (s, 3H), 1.44 (s, 9H). ^{13}C NMR (d_6-DMSO, 343 K) δ: 152.0 (C), 151.5 (C), 150.0 (C), 93.2 (C), 88.7 (CH_2), 80.1 (C), 79.8 (CH), 70.6 (CH), 62.9 (CH_2), 56.6 (CH), 27.7 (CH_3), 26.8 (CH_3), 23.8 (CH_3).HRMS (ESI): calcd. for [$C_{15}H_{23}NO_7$ + Na^+]: 352.1372, found for [M+Na]$^+$: 352.1357. Anal. calcd. for $C_{15}H_{23}NO_7$: C 54.71, H 6.99, N 4.25; found: C 54.39, H 6.73, N 4.04.

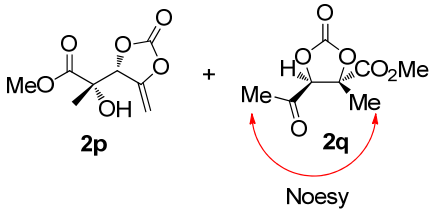
4-(1-Hydroxycyclohexyl)-5-methylene-1,3-dioxolan-2-one 2o



2o

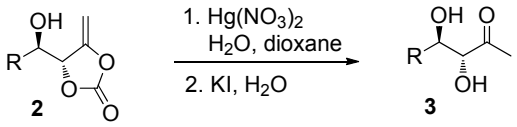
According to the general procedure for the indium-mediated allylation, starting from cyclohexanone (19.6 mg; 0.2 mmol); purification by dry-flash chromatography afforded 21 mg (52%) of the **2o**. Colorless crystals, mp 107-9 °C (from hexanes/ethyl acetate). FT-IR (KBr): 3483, 2985, 2936, 2863, 1798, 1686, 1345, 1159, 1049. ^{1}H NMR δ: 5.00 (dd, J_1=1.8, J_2=4.0, 1H), 4.84 (t, J=1.8, 1H), 4.53 (dd, J_1=1.6, J_2=4.0, 1H), 1.42-1.72 (m, 10H), 1.21-1.28 (m, 1H). ^{13}C NMR δ: 149.9 (C), 90.0 (C), 84.8 (CH_2), 72.4 (CH), 32.1 (CH_2), 31.3 (CH_2), 25.2 (CH_2), 20.8 (CH_2). Anal. calcd. for $C_{10}H_{14}O_4$: C 60.59, H 7.12; found: C 60.50, H 7.11.

***anti*-Methyl 2-hydroxy-2-(5-methylene-2-oxo-1,3-dioxolan-4-yl)propanoate 2p and *trans*-methyl 5-acetyl-4-methyl-2-oxo-1,3-dioxolane-4-carboxylate 2q**



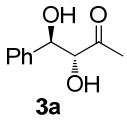
According to the general procedure for the indium-mediated allylation, starting from methyl pyruvate (18.2 mg; 0.18 mmol); purification by dry flash chromatography (gradient elution: from 20% to 30% acetone in petroleum-ether) afforded 19 mg (52%) of **2p**, followed by 3 mg (8%) of **2q**.
Physical data for **2p**: Colorless crystals, mp 93-5 °C (from hexanes/ethyl acetate). FT-IR (KBr): 3481, 2959, 1832, 1740, 1689, 1336, 1267 1141, 1059. ^{1}H NMR δ: 5.17 (m, 1H), 5.00 (dd, J_1=3.4, J_2=4.0, 1H), 4.58 (dd, J_1=1.8, J_2=4.0, 1H), 3.87 (s, 3H), 3.64 (s, 1H), 1.53 (s, 3H). ^{13}C NMR δ: 173.5 (C), 151.6 (C), 148.9 (C), 90.2 (CH_2), 81.4 (CH), 75.1 (C), 53.8 (CH_3), 21.3 (CH_3). HRMS (ESI): calcd. for [$C_8H_{10}O_6$ + Na^+]: 225.0369; found for $[M+Na]^+$: 225.0368. Physical data for **2q**: Colorless oil. FT-IR (film): 2962, 1827, 1741, 1692, 1445, 1273, 1225, 1113, 1078. ^{1}H NMR δ: 5.10 (s, 1H), 3.91 (s, 3H), 2.36 (s, 1H), 1.58 (s, 3H). ^{13}C NMR δ: 201.6 (C), 168.9 (C), 151.9 (C), 148.9 (C), 85.6 (C), 82.5 (CH), 54 (CH_3), 28.0 (CH_3), 18.5 (CH_3). HRMS (ESI) calcd. for $C_8H_{11}O_6$ $[M+H]^+$: 302.0550; found: 203.0549.

General procedure for the deprotection of enol carbonates 2 into α,β-dihydroxy ketones 3



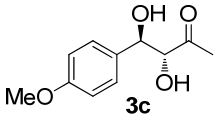
Mercury(II) nitrate (118 mg; 0.364 mmol) was added to a cold (0 °C) solution of compound **2** (0.121 mmol) in dioxane (2 mL) and water (0.6 mL). The reaction mixture was stirred for 5 min, when TLC (eluent: 50% EtOAc in petroleum-ether) indicated the disappearance of the starting material. Saturated aqueous solution of KI (10 mL) was added at 0 °C, the mixture was allowed to reach rt, and was stirred at rt for an additional 5 min. Standard work-up with diethyl ether, followed by purification by dry-flash chromatography, afforded the pure compound **3**.

***anti*-3,4-Dihydroxy-4-phenylbutan-2-one 3a**[12]



According to the general procedure for the deprotection of enol carbonates, starting from **2a** (25 mg, 0.12 mmol); after purification by column chromatography (eluent: 50% EtOAc in hexanes), 14.6 mg (67%) of the title compound **3a** was obtained, as a mixture of isomers in a ratio *anti*:*syn*=12:1 (the ratio of isomers did not change after crystallization from 5% EtOAc in hexanes). Physical data for **3a**: White crystals, m.p. 106-7 °C. FT-IR (KBr): 3417, 3032, 2916, 1712, 1357, 1231, 1101, 1055, 759, 704. ^{1}HNMR δ: 7.42-7.31 (m, 5H), 5.02-4.98 (m, 1H), 4.46 (bt, J_1 = 4.4, 1H), 3.74 (d, J = 4.4, 1H), 3.12 (d, J = 4.4, 1H), 1.95 (s, 3H). ^{13}C NMR δ: 208.2 (C), 138.9 (C), 128.6 (CH), 128.2 (CH), 126.2 (CH), 81.1 (CH), 74.9 (CH), 27.6 (CH_3). HRMS (ESI): calcd. for [$C_{10}H_{12}O_3$ + NH_4^+]: 198.1130, found for $[M+NH_4]^+$: 198.1124.

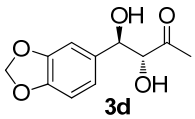
***anti*-3,4-Dihydroxy-4-(4-methoxyphenyl)butan-2-one 3c**[13]



3c

According to the general procedure for the deprotection of enol carbonates, starting from **2c** (43.5 mg, 0.18 mmol); after purification by column chromatography (eluent: 50% EtOAc in petroleum-ether), 25 mg (64%) of the title compound **3c** was obtained, as a mixture of diastereoisomers in a ratio: *anti*:*syn*=10:1. Physical data for **3c**: white crystals, mp 50-3 °C (recrystallized from 5% EtOAc in hexanes), FT-IR (KBr): 3429, 3004, 2913, 1712, 1514, 1357, 1249, 1178, 1031, 836. ^{1}H NMR δ: 7.32 (d, *J*=8.8, 2H), 6.90 (d, *J*=8.8, 2H), 4.93 (d, *J*=4.8, 1H), 4.43 (d, *J*=4.8, 1H), 3.81 (s, 3H), 3.67 (bd, *J*=4.8, 1H), 3.05 (bs, 1H), 2.00 (s, 3H). ^{13}C NMR δ: 208.4 (C), 159.4 (C), 131.0 (C), 127.5 (CH), 113.9 (CH), 80.9 (CH), 74.5 (CH), 55.2 (CH_3), 27.6 (CH_3). HRMS (ESI): calcd. for [$C_{11}H_{14}O_4$ + NH_4^+]: 228.1236, found for [M+NH_4]$^+$: 228.1225.

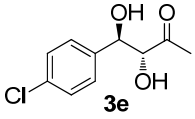
***anti*-4-(Benzo[*d*][1,3]dioxol-5-yl)-3,4-dihydroxybutan-2-one 3d**



3d

According to the general procedure for the deprotection of enol carbonates, starting from **2d** (72 mg, 0.29 mmol); after purification by column chromatography (eluent: 50% EtOAc in petroleum-ether), 31.6 mg (49%) of the title compound **3d** was obtained, as a mixture of diastereoisomers in a ratio: *anti*:*syn*=6.4:1. Physical data for **3d**: white crystals, mp 112-5 °C, FT-IR (KBr): 3426, 2902, 1713, 1490, 1444, 1358, 1247, 1037, 930. ^{1}H NMR δ: 6.92-6.77 (m, 3H), 5.97 (s, 2H), 4.88 (bt, *J*=3.9, 1H), 4.40 (t, *J*=4.9, 1H), 3.59 (d, *J*=4.9, 1H), 2.93 (d, *J*=3.9, 1H), 2.06 (s, 3H). ^{13}C NMR δ: 208.3 (C), 147.9 (C), 147.5 (C), 133.0 (C), 119.8 (CH), 108.2 (CH), 106.8 (CH), 101.2 (CH_2), 80.8 (CH), 74.7 (CH), 27.6 (CH_3). HRMS (ESI): calcd. for [$C_{11}H_{12}O_5$ + Na^+]: 247.0582, found for [M+Na]$^+$: 247.0573.

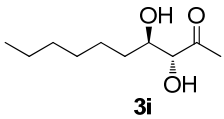
***anti*-3,4-Dihydroxy-4-(4-chlorophenyl)butan-2-one 3e**[14]



3e

According to the general procedure for the deprotection of enol carbonates, starting from **2e** (30 mg, 0.13 mmol); after purification by column chromatography (eluent: 50% EtOAc in petroleum-ether), 20.1 mg (75%) of the title compound **3e** was obtained, as unseparable mixture of diastereoisomers in a ratio: *anti*:*syn*=7.7:1. Physical data for **3e**: white crystals, mp 52-4 °C, FT-IR (KBr): 3416, 3032, 2919, 1712, 1358, 1231, 1102, 1056, 759, 705. ^{1}H NMR δ: 7.39-7.29 (m, 4H), 4.96 (bs, 1H), 4.42 (bt, *J*=4.8, 1H), 3.75 (d, *J*=4.8, 1H), 3.19 (bs, 1H), 1.98 (s, 3H). ^{13}C NMR δ: 207.9 (C), 137.6 (C), 134.0 (C), 128.7 (CH), 127.6 (CH), 80.8 (CH), 74.3 (CH), 27.7 (CH_3). HRMS (ESI): calcd. for [$C_{10}H_{11}O_3Cl$ + Na^+]: 237.0294, found for [M+Na]$^+$: 237.0290. Anal. calcd. for $C_{10}H_{11}O_3Cl$: C 55.94, H 5.13; found: C 55.98, H 5.08.

***anti*-3,4-Dihydroxydecan-2-one 3i**

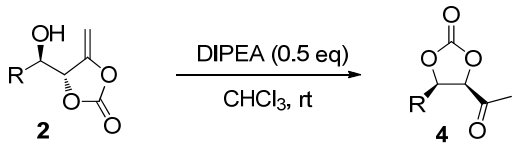


3i

According to the general procedure for the deprotection of enol carbonates, starting from **2i** (18 mg, 0.08 mmol); after purification by column chromatography (eluent: 50% EtOAc in petroleum-ether), 10 mg (67%) of the title compound **3i** was obtained. Physical data for **3i**: white crystals, mp 50-2 °C (from 5% EtOAc in hexanes), FT-IR (KBr): 3303, 3210, 2926, 2852, 1719, 1361, 1080, 1053. ^{1}H NMR δ: 4.28 (t, *J*=5.2, 1H), 3.88 (bs, 1H), 3.51 (d, *J*=5.2, 1H), 2.26 (s, 3H), 2.08 (d, *J*=7.5, 1H), 1.53-1.49 (m, 2H), 1.28-1.27 (m, 8H), 0.88 (t, *J*=7.0, 3H). ^{13}C NMR δ: 208.0 (C), 80.5 (CH), 72.7 (CH), 31.8(CH_2),

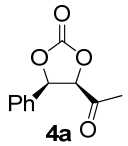
31.7 (CH_2), 29.1 (CH_2), 26.9 (CH_3), 25.6 (CH_2), 22.6 (CH_2), 14.0 (CH_3). HRMS (ESI): calcd. for [$C_{10}H_{20}O_3$ + Na^+]: 211.1310, found for $[M+Na]^+$: 211.1307.

General procedure for the rearrangement of enol carbonates 2 into *cis* cyclic carbonates 4



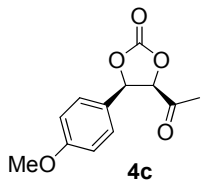
Diisopropylethylamine (6 mg; 8.0 µL; 46 µmol) was added to a solution of compound **2** (0.083 mmol) in chloroform (1.5 mL). Reaction mixture was stirred at rt, and the progress of the reaction was monitored by TLC (eluent: 40% EtOAc in petroleum-ether; the reactions are usually complete in 1-3 h). The reaction mixture was concentrated at rotavap and the crude product purified by dry-flash chromatography.

***cis*-4-Acetyl-5-phenyl-1,3-dioxolan-2-one 4a**



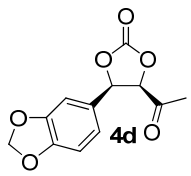
According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **4**, starting from **2a** (20 mg; 97 µmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 14.1 mg (71%) of the title compound **4a**. Physical data for **4a**: white crystals, mp 106-7 °C, FT-IR (KBr): 3429, 3047, 2980, 1809, 1721, 1339, 1173, 1074, 768. ^{1}H NMR δ: 7.42-7.40 (m, 3H); 7.26-7.25 (m, 2H); 5.92 (d, *J*=8.8, 1H); 5.23 (d, *J*=8.8, 1H); 1.77 (s, 3H). ^{13}C NMR δ: 153.7 (C), 148.5 (C), 131.9 (C), 130.0 (CH), 129.1 (CH), 126.1 (CH), 82.5 (CH), 79.4 (CH), 27.6 (CH_3). HRMS (ESI): calcd. for [$C_{11}H_{10}O_4$ + Na^+]: 229.0477, found for $[M+Na]^+$: 229.0477.

***cis*-4-Acetyl-5-(4-methoxyphenyl)-1,3-dioxolan-2-one 4c**



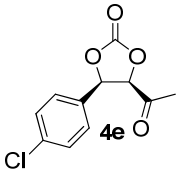
According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **4**, starting from **2c** (25 mg; 0.106 mmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 14 mg (56%) of the title compound **4c**. Physical data for **4c**: white crystals, mp 130-132 °C. IR (KBr): 2974, 2841, 1790, 1726, 1617, 1519, 1340, 1260, 1175, 1173, 835, 767. ^{1}H NMR δ: 7.18 (d, *J*=9.0, 2H); 6.91 (d, *J*=9.0, 2H); 5.88 (d, *J*=8.7, 1H); 5.21 (d, *J*=8.7, 1H); 3.81 (s, 3H), 1.80 (s, 3H). ^{13}C NMR δ: 201.9 (C), 160.7 (C), 153.7 (C), 127.6 (CH), 123.7 (C), 114.5 (CH), 82.6 (CH), 79.4 (CH), 55.3 (CH_3), 27.7 (CH_3). HRMS (ESI): calcd. for [$C_{12}H_{12}O_5$ + Na^+]: 259.0582, found for $[M+Na]^+$: 259.0571. Anal. calcd. for $C_{12}H_{12}O_5$: C 61.02, H 5.08; found: C 60.50, H 5.08.

***cis*-4-Acetyl-5-(benzo[*d*][1,3]dioxol-5-yl)-1,3-dioxolan-2-one 4d**



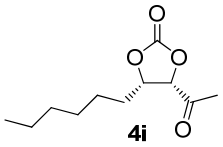
According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **4**, starting from **2d** (43 mg; 0.172 mmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 22.7 mg (53%) of the title compound **4d**. Physical data for **4d**: white crystals, mp 136-8 °C (5% EtOAc in hexanes), FT-IR (KBr): 3437, 2908, 1794, 1725, 1502, 1261, 1180, 1075, 812, 767. ^{1}H NMR δ: 6.82 (d, *J*=8.0, 1H); 6.74 (dd, J_1=8.0, J_2=2.0, 1H); 6.70 (d, *J*=2.0, 1H); 6.00 (s, 2H); 5.82 (d, *J*=8.8, 1H); 5.19 (d, *J*=8.8, 1H); 1.88 (s, 3H). ^{13}C NMR δ: 201.7 (C), 153.5 (C), 149.0 (C), 148.4 (C), 125.4 (C), 120.2 (CH), 108.7 (CH), 106.4 (CH), 101.7 (CH_2), 82.4 (CH), 79.4 (CH), 27.8 (CH_3). HRMS (ESI): calcd. for [$C_{12}H_{10}O_6$ + NH_4^+]: 268.0821, found for [M+NH_4]$^+$: 268.0817; Anal. calcd. for $C_{12}H_{10}O_6$: C 57.60, H 4.00; found: C 57.28, H 4.25.

cis-4-Acetyl-5-(4-chlorophenyl)-1,3-dioxolan-2-one 4e



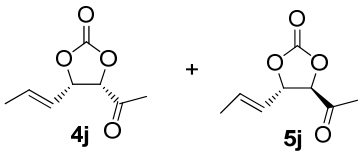
According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **4**, starting from **2e** (20 mg; 0.08 mmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 12.6 mg (63%) of the title compound **4e**. Physical data for **4e**: white crystals, mp 102-3 °C, FT-IR (KBr): 3438, 3004, 2922, 1793, 1341, 1179, 1075, 814. ^{1}H NMR δ: 7.40 (d, *J*=8.8, 2H); 7.20 (d, *J*=8.8, 2H); 5.89 (d, *J*=9.0, 1H); 5.21 (d, *J*=9.0, 1H); 1.85 (s, 3H). ^{13}C NMR δ: 201.8 (C), 153.3 (C), 136.2 (C), 130.5 (C), 129.4 (CH), 127.4 (CH), 82.3 (CH), 78.7 (CH), 27.8 (CH_3). HRMS (ESI): calcd. for [$C_{11}H_9O_4Cl$ + NH_4^+]: 258.0533, found for [M+NH_4]$^+$: 258.0532. Anal. calcd. for $C_{11}H_9O_4Cl$: C 54.88, H 3.74; found: C 54.60, H 3.90.

cis-4-Acetyl-5-hexyl-1,3-dioxolan-2-one 4i



According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **4**, starting from **2i** (17 mg, 0.08 mmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 10 mg (59%) of the title compound **4i**, as a colorless oil. Physical data for **4i**: FT-IR (KBr): 2956, 2930, 2859, 1812, 1727, 1463, 1363, 1166, 1080. ^{1}H NMR δ: 4.61 (q, *J*=6.2, 1H), 4.47 (d, *J*=6.2, 1H), 2.36 (s, 3H), 1.84-1.79 (m, 2H), 1.56-1.29 (m, 8H), 0.89 (t, *J*=6.8, 3H). ^{13}C NMR δ: 204.0 (C), 153.3 (C), 83.0 (CH), 79.1 (CH), 34.6 (CH_2), 31.4 (CH_2), 28.6 (CH_2), 26.5 (CH_3), 24.1 (CH_2), 22.4 (CH_2), 13.9 (CH_3). HRMS (ESI) calcd. for [$C_{11}H_{18}O_4$ + Na^+]: 237.1103; found for [M+Na]$^+$: 237.1090.

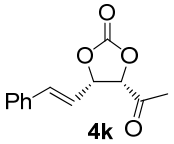
4-Acetyl-5-((*E*)-prop-1-en-1-yl)-1,3-dioxolan-2-one – mixture of _cis_ isomer 4j and _trans_ isomer 5j

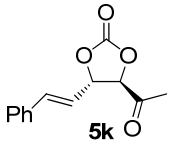


According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **5**, starting from **2j** (20 mg; 0.28 mmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 10 mg (50%) of **5j** (*trans*), followed by 8 mg (40%) of **4j** (*cis*). Physical data for **5j**: colorless oil, FT-IR (film): 2922, 1804, 1726, 1356, 1174, 1078. ^{1}H NMR δ: 6.06-5.98 (m, 1H), 5.58 (ddq, J_1=15.0, J_2=6.9, J_3=1.8, 1H), 5.01 (bt, *J*=6.9, 1H), 4.57 (d, *J*=6.9, 1H), 2.36 (s, 3H), 1.80 (ddd, J_1=7.0, J_2=1.8, J_3=0.5, 3H). ^{13}C NMR δ: 202.7 (C), 153.0 (C), 135.0 (CH), 124.9 (CH), 83.0 (CH), 79.3 (CH), 26.7 (CH_3), 17.8 (CH_3). HRMS (ESI): calcd. for [$C_8H_{10}O_4$ + NH_4]: 188.0923, found for [M+NH_4]$^+$: 188.0919. Physical data for **4j**: colorless oil, FT-IR (film): 2928, 1799, 1723, 1337, 1174, 1076. ^{1}H NMR δ: 6.06-5.99 (m, 1H), 5.34-5.26 (m, 2H), 4.98 (d, *J*=8.5, 1H), 2.25 (s, 3H), 1.78-

1.77 (m, 3H). ^{13}C NMR δ: 202.4 (C), 153.4 (C), 135.8 (CH), 121.2 (CH), 81.4 (CH), 78.9 (CH), 28.2 (CH_3), 17.8 (CH_3). HRMS (ESI): calcd. for [$C_8H_{10}O_4$+ NH_4^+]: 188.0923, found for [M+NH_4^+]$^+$: 188.0919.

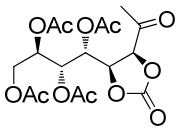
(*E*)-4-Acetyl-5-styryl-1,3-dioxolan-2-one - *cis* isomer 4k and *trans* isomer 5k





According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **4**, starting from **2k** (36 mg; 0.129 mmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 16.3 mg (45%) of **5k**, followed by 12.2 mg (34%) of **4k**. Physical data for **4k**: white crystals, mp 92-4 °C (from 5% EtOAc in hexanes), FT-IR (KBr): 3060, 3029, 1808, 1730, 1336, 1171, 1082, 1024, 978, 757, 695. ^{1}H NMR δ: 7.39-7.32 (m, 5H), 6.83 (dd, J_1=15.7, J_2=1.1, 1H), 5.96 (dd, J_1=15.7, J_2=7.3, 1H), 5.57-5.48 (m,1H), 5.10 (d, J=9.0, 1H), 2.27 (s, 3H). ^{13}C NMR δ: 202.5 (C), 153.3 (C), 137.0 (CH), 134.5 (C), 129.3 (CH), 128.8 (CH), 127.1 (CH), 118.3 (CH), 81.4 (CH), 78.6 (CH), 28.3 (CH_3). HRMS (ESI): calcd. for [$C_{13}H_{12}O_4$ + K^+]: 271.0373, found for [M+K]$^+$: 271.0367. Physical data for **5k**: white crystals, mp 115-6 °C (from 5% EtOAc in hexanes), FT-IR (KBr): 3028, 2923, 1810, 1729, 1358, 1170, 1090, 972, 758, 695. ^{1}H NMR δ: 7.45-7.33 (m, 5H), 6.83 (d, J=15.7, 1H), 6.20 (dd, J_1=15.7, J_2=7.4, 1H), 5.26 (t, J=6.2, 1H), 4.68 (d, J=6.2, 1H), 2.41 (s, 3H). ^{13}C NMR δ: 202.8 (C), 153.0 (C), 136.7 (CH), 134.6 (C), 129.3 (CH), 128.9 (CH), 127.1 (CH), 121.9 (CH), 83.0 (CH), 79.2 (CH), 26.8 (CH_3). HRMS (ESI): calcd. for [$C_{13}H_{12}O_4$ + K^+]: 271.0373, found for [M+K]$^+$: 271.0365. Anal. calcd. for $C_{13}H_{12}O_4$: C 67.24, H 5.17; found: C 66.93, H 5.32.

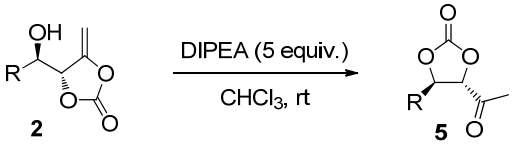
(1*S*,2*R*,3*R*)-1-((4*S*,5*S*)-5-acetyl-2-oxo-1,3-dioxolan-4-yl)butane-1,2,3,4-tetrayl tetraacetate 4m



4m

According to the general procedure for the rearrangement of enol carbonates **2** into *cis* cyclic carbonates **4**, starting from **2m** (10 mg; 24 μmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 7.5 mg (75%) of the title compound **4m**. Physical data for **4m**: yellow oil, FT-IR (film): 3358, 2924, 2853, 2363, 1821, 1746, 1371, 1209, 1134, 1161, 736, 602. ^{1}H NMR δ: 5.50-5.46 (m, 2H), 5.13-5.10 (m, 1H), 4.93 (d, J=4.6, 1H), 4.78 (t, J=4.6, 1H), 4.27 (dd, J_1=13.0, J_2=3.0, 1H), 4.08 (dd, J_1=13.0, J_2=4.8, 1H), 2.37 (s, 3H), 2.14 (s, 3H), 2.10 (s, 3H), 2.08 (s, 3H), 2.08 (s, 3H). ^{13}C NMR δ: 203.2 (C), 170.5 (C), 169.8 (C), 169.7 (C), 169.2 (C), 152.1 (C), 79.5 (CH), 76.4 (CH), 69.2 (CH), 68.2 (CH), 67.8 (CH), 61.3 (CH_2), 26.7 (CH_3), 20.8 (CH_3), 20.7 (CH_3), 20.6 (CH_3), 20.4 (CH_3). HRMS (ESI): calcd. for [$C_{17}H_{22}O_{12}$ + NH_4^+]: 436.1455, found for [M+NH_4]$^+$: 436.1449.

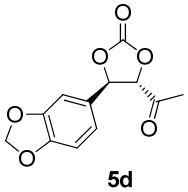
General procedure for the rearrangement of enol carbonates 2 into *trans* cyclic carbonates 5



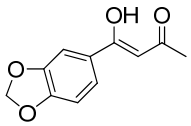
Diisopropylethylamine (103 mg; 140 μL; 0.8 mmol) was added to a solution of compound **2** (0.16 mmol) in chloroform (1.5 mL). Reaction mixture was stirred at rt, and the progress of the reaction was monitored by TLC (eluent: 40% EtOAc in petroleum-ether; the reactions are usually complete in 45

min). The reaction mixture was concentrated at rotavap and the crude product purified by dry-flash chromatography.

trans-4-Acetyl-5-(benzo[_d_][1,3]dioxol-5-yl)-1,3-dioxolan-2-one 5d and 4-(benzo[_d_][1,3]dioxol-5-yl)-4-hydroxybut-3-en-2-one 6d



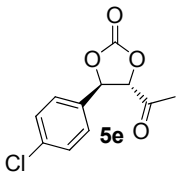
5d



6d

According to the general procedure for the rearrangement of enol carbonates **2** into *trans* cyclic carbonates **5**, starting from **2d** (40 mg; 0.16 mmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 20 mg (50%) of the title compound **5d**. When the reaction time was extended to several hours, in addition to **5d**, compound **6d** (a less polar spot on TLC) could also be isolated in 20% yield. This compound is described in the literature,[15] and the copies of its' ^{1}H and ^{13}C NMR spectra are on pages S112 and S113. Physical data for **5d**: white crystals, mp 102-5 °C, FT-IR (KBr): 3360, 2924, 1803, 1730, 1659, 1498, 1452, 1256, 1169, 1080, 1036, 767. ^{1}H NMR δ: 6.86-6.83 (m, 3H), 6.01 (s, 2H), 5.55 (d, *J*=6.3, 1H), 4.72 (d, *J*=6.3, 1H), 2.40 (s, 3H). ^{13}C NMR δ: 202.8 (C), 148.9 (CH), 148.7 (C), 129.2 (C), 120.0 (CH), 108.8 (CH), 105.9 (CH), 101.7 (CH_2), 84.8 (CH), 79.4(CH), 26.9 (CH_3); one carbon resonance, corresponding to the quaternary carbon atom from carbonate, was not observed under the recording conditions. HRMS (ESI): calcd. for [$C_{12}H_{10}O_6$ + NH_4^+]: 268.0821, found for [M+NH_4]$^+$: 268.0810. Anal. calcd. for $C_{12}H_{10}O_6$: C 57.60, H 4.00; found: C 57.91, H 3.88.

trans-4-Acetyl-5-(4-chlorophenyl)-1,3-dioxolan-2-one 5e



5e

According to the general procedure for the rearrangement of enol carbonates **2** into *trans* cyclic carbonates **5**, starting from **2e** (20 mg; 80 μmol); purification by column chromatography (eluent: 50% EtOAc in petroleum-ether) afforded 12 mg (60%) of the title compound **5e**. Physical data for **5e**: white crystals, mp 92-3 °C (from 5% EtOAc in hexanes), FT-IR (KBr): 3424, 2920, 1815, 1718, 1164, 1095, 761. ^{1}H NMR δ: 7.43 (d, *J*=8.3, 2H), 7.34 (d, *J*=8.3, 2H), 5.66 (d, *J*=6.5, 1H), 4.69 (d, *J*=6.5, 1H), 2.43 (s, 3H). ^{13}C NMR δ: 202.8 (C), 152.7 (C), 135.8 (C), 134.2 (C), 129.6 (CH), 126.9 (CH), 84.6 (CH), 78.5 (CH), 26.9 (CH_3). HRMS (ESI): calcd. for [$C_{11}H_9O_4Cl$ + Na^+]: 263.0087, found for [M+Na]$^+$: 263.0085. Anal. calcd. for $C_{11}H_9O_4Cl$: C 54.88, H 3.74; found: C 54.47, H 3.58.

---

[1] For description of the technique of dry-flash chromatography, see: a) Harwood, L. M. *Aldrichimica Acta* **1985**, *18*, 25; b) *Vogel's Textbook of Practical Organic Chemistry*, Longman Scientific&Technical, 5th edition, London, 1989, p. 220; c) An account which includes some improvements of the separation technique: Pedersen, D. S.; Rosenbohm, C. *Synthesis* **2001**, 2431.

[2] Perrin, D. D.; Armarego, W. L. F. *Purification of Laboratory Chemicals*, 3rd edition, Pergamon Press, **1988**.

[3] *Syn*thesis of 1-Tosyl-1*H*-indole-3-carbaldehyde: Guo, X.; Hu, W.; Cheng, S.; Wang, L.; Chang, J. *Synth. Commun.* **2006**, *36*, 781.

[4] Wolfrom, M. L.; Weisblat, D. I.; Zophy, W. H.; Waisbrot, S. W. *J. Am. Chem. Soc*. **1941**, *63*, 201.

[5] Ibers, J. A.; Hamilton, W. C. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham, 1974.

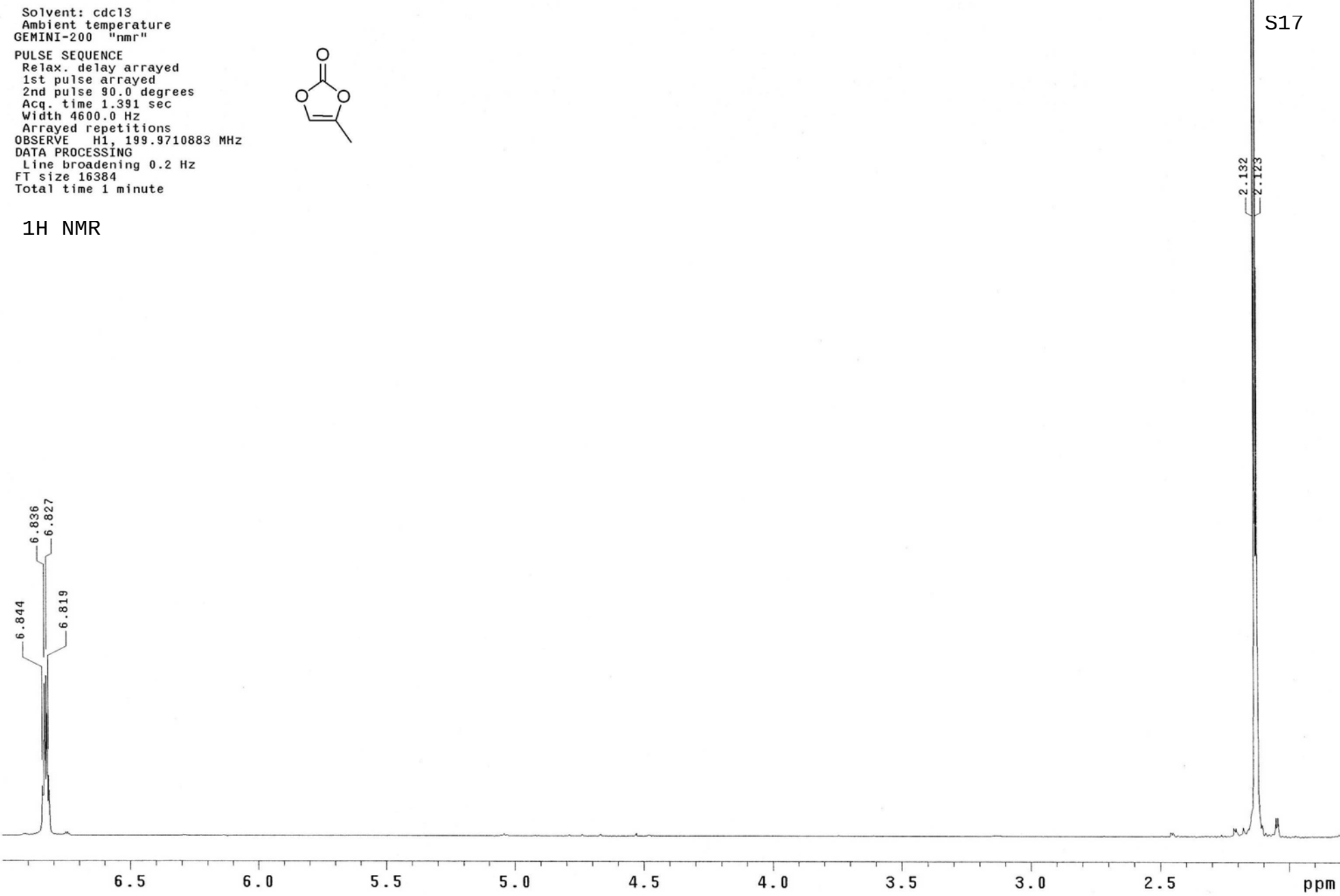
[6] SIR-92: Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A. *J. Appl. Cryst.* **1993**, *26*, 343.

[7] Mercury: visualization and analysis of crystal structures, Macrae, C. F.; Edgington, P. R.; McCabe, P.; Pidcock, E.; Shields, G. P.; Taylor, R.; Towler, M.; Van de Streek, J. *J. Appl. Cryst.* **2006**, *39*, 453.
[8] Sheldrick, M. SHELXL-97, Program for crystal structure refinement, University of Goettingen, Germany, 1997.
[9] Alexander, J. U. S. Patent 5 466 811, 1995; *Chem. Abstr*. **1995**, *124*, 176148x.
[10] Jung, M.; Blum, R.; Gaede, B.; Gisler, M. *Heterocycles*, **1989,** *28*, 93
[11] a) Sakamoto, F.; Ikeda, S.; Tsukamoto, G. *Chem. Pharm. Bull*. **1984**, *32*, 2241. b) For the explanation of the mechanism of the reaction sequence, see: Fischler, H.-M.; Heine, H.-G.; Hartmen, W. *Tetrahedron Lett*. **1972**, 1701.
[12] a) Markert, M.; Mulzer, M.; Schetter, B., Mahrwald, R. *J. Am. Chem. Soc.* **2007,** *129*, 7258; b) Rogozinska, M.; Mlynarski, J. *Tetrahedron Lett*. **2009**, *50*, 1639.
[13] In the literature, this compound has not been characterized as a pure *anti* isomer, but as a mixture of diastereoisomers; therefore its' spectral data are provided here. See: Aelvoet, K.; Batsanov, A. S.; Blatch, A. J.; Grosjean, C.; Patrick, L. G. F.; Smethurst, C. A.; Whiting, A. *Angew. Chem. Int. Ed.* **2008**, *47*, 768.
[14] Schetter, B.; Stosiek, C.; Ziemer, B.; Mahrwald, R. *Appl. Organometal. Chem.* **2007,** *21*, 139.
[15] a) M. Julia, M.; Jassonneix, C. B. *Bull. Soc. Chim. Fr*. **1975**, 751; b) Mors, W.; Gottliee, O. R.; Djerassi, C. *J. Am. Chem. Soc.* **1957**, *79*, 4507.

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

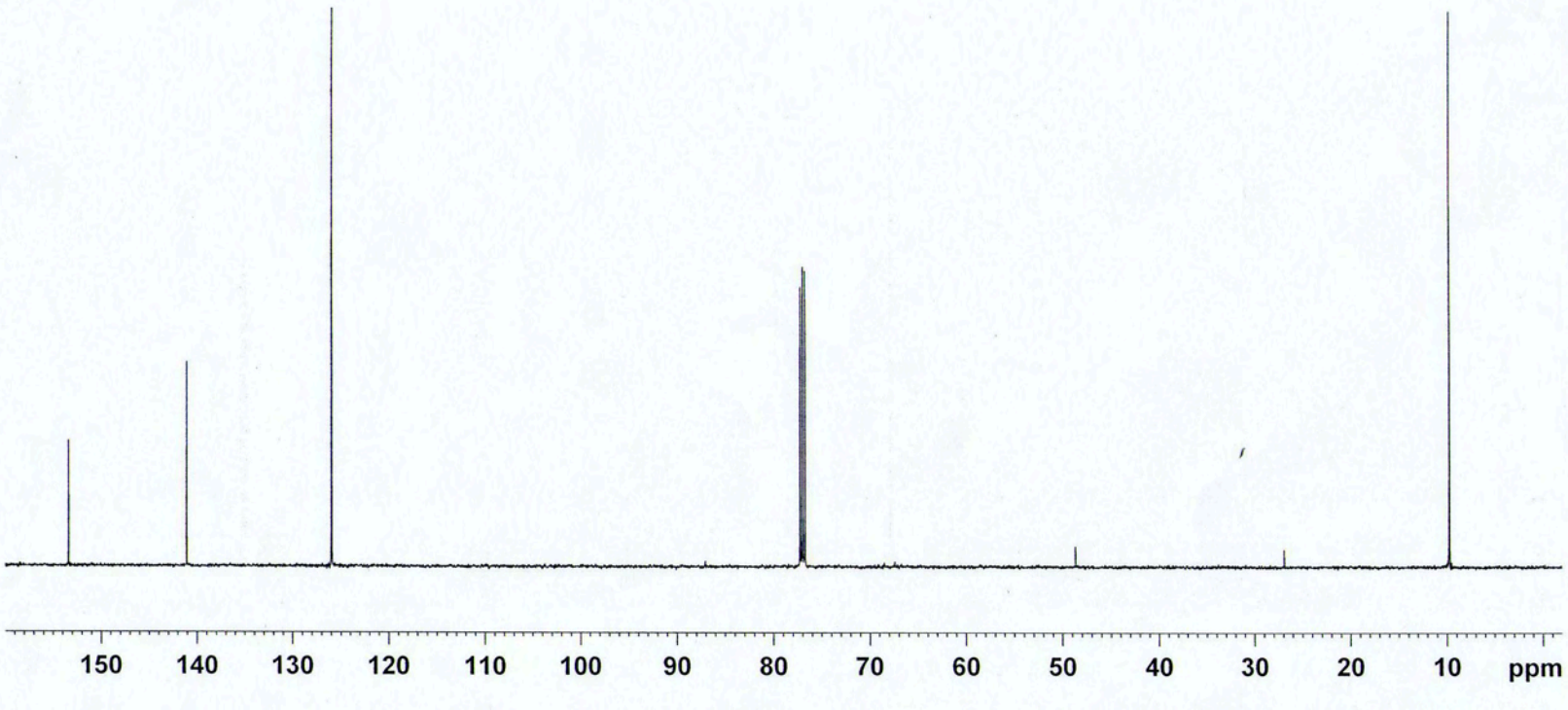
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710883 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute

1H NMR

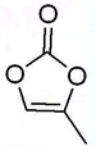


153.37
141.08
125.97
9.72

13C NMR



NAME	VZ-CO3
EXPNO	2
PROCNO	1
Date_	20100309
Time	11.53
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	204
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	812
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618940 MHz
SI	32768
SF	125.7904923 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



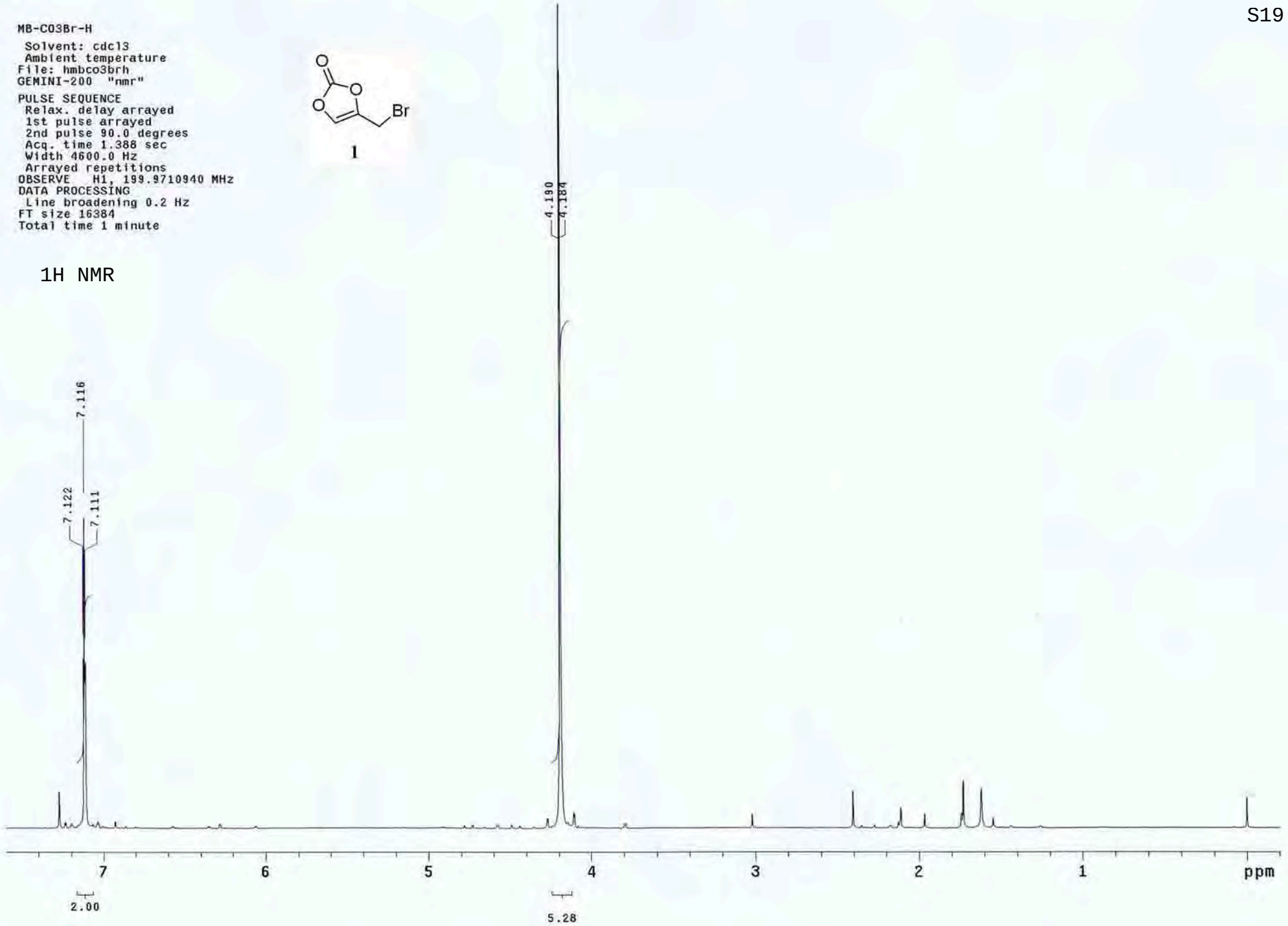
MB-CO3Br-H

Solvent: cdcl3
Ambient temperature
File: hmbco3brh
GEMINI-200 "nmr"

PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.388 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710940 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute

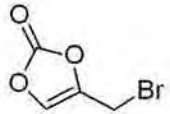
1

1H NMR



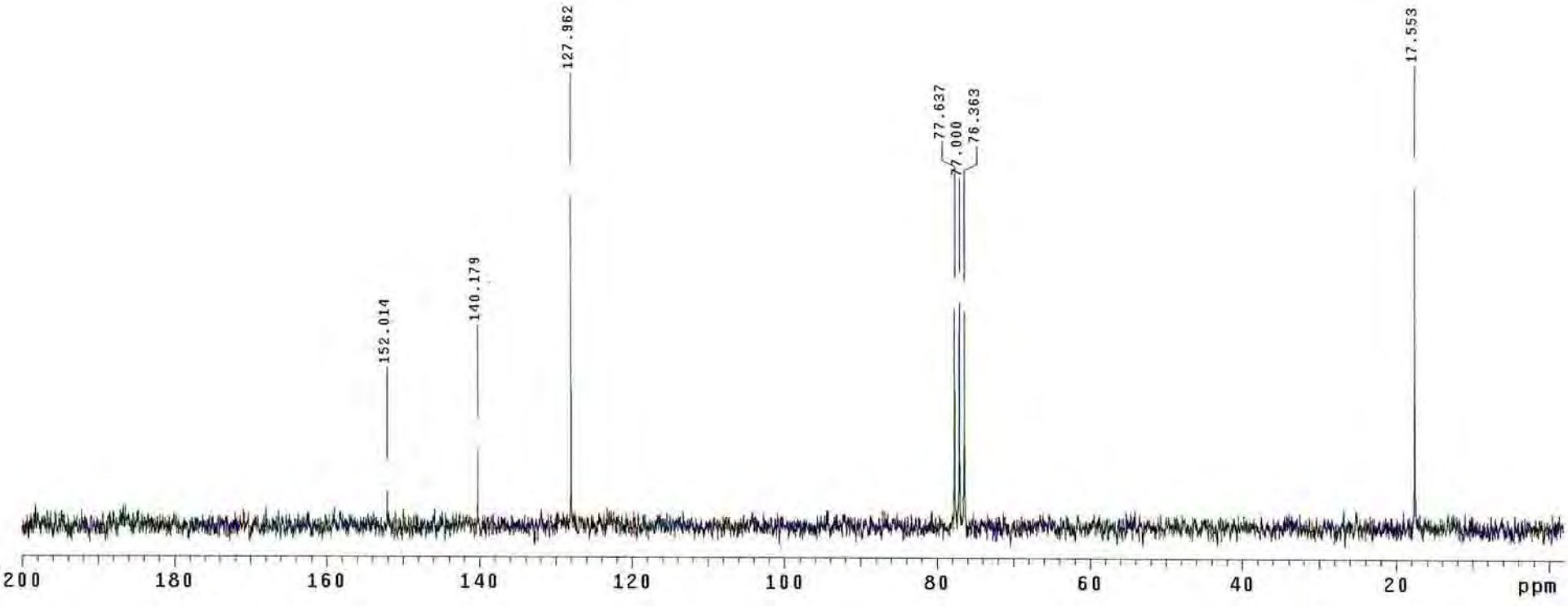
Ambient temperature/
GEMINI-200 "nmr"

PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827800 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 22 minutes

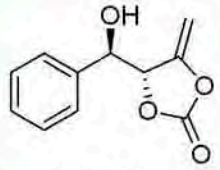


1

13C NMR

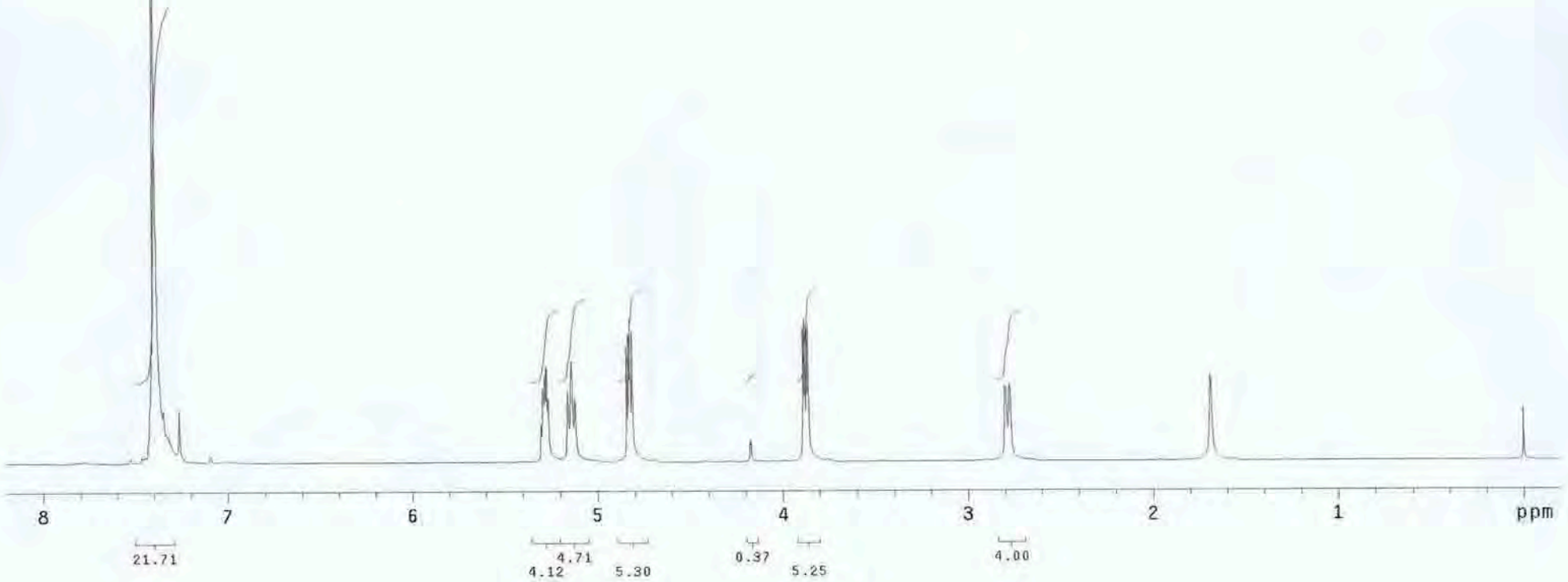


MB-68-D
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710962 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 20 minutes



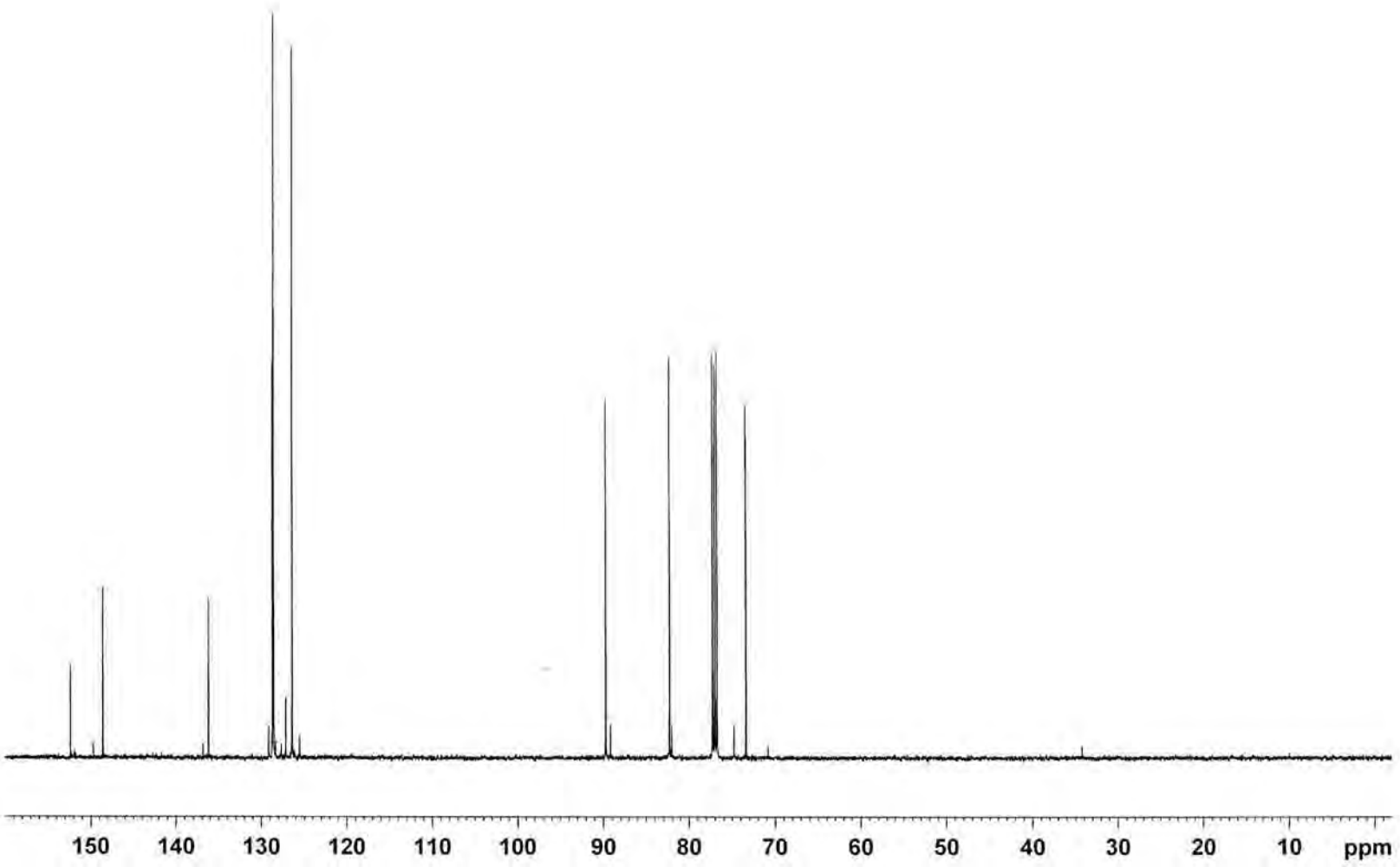
2a

1H NMR

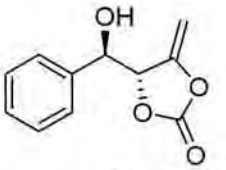


152.33
148.57
136.15
128.69
128.59
126.41
89.71
82.27
73.38

13C NMR

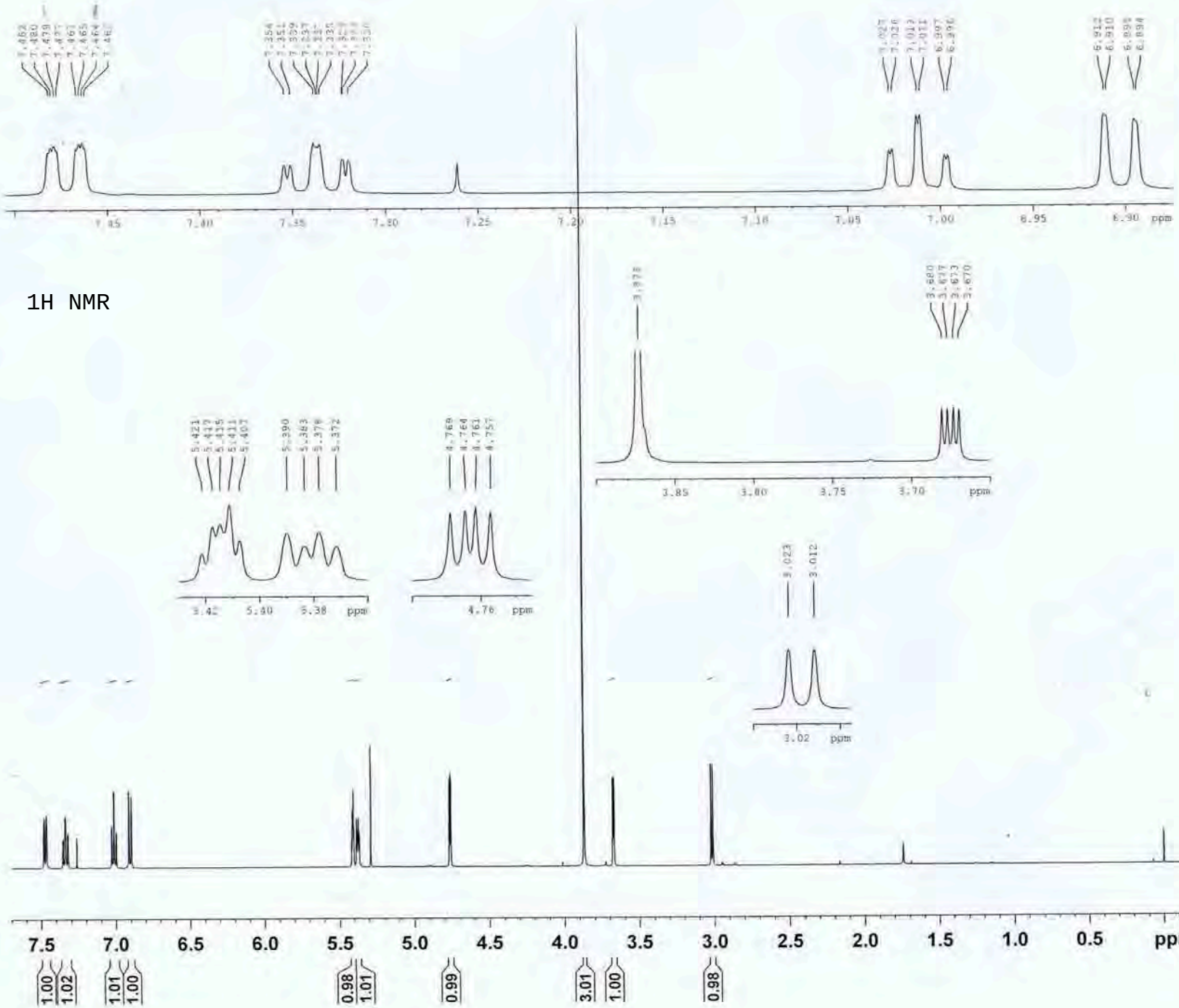


NAME	MB-68
EXPNO	2
PROCNO	1
Date_	20100322
Time	13.37
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	403
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 se
RG	1820
DW	16.800 us
DE	6.50 us
TE	298.0 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 us
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MH
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 us
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2617699 MH
SI	32768
SF	125.7904864 MH
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

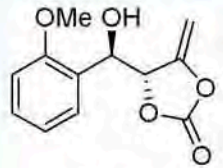


2a

1H NMR

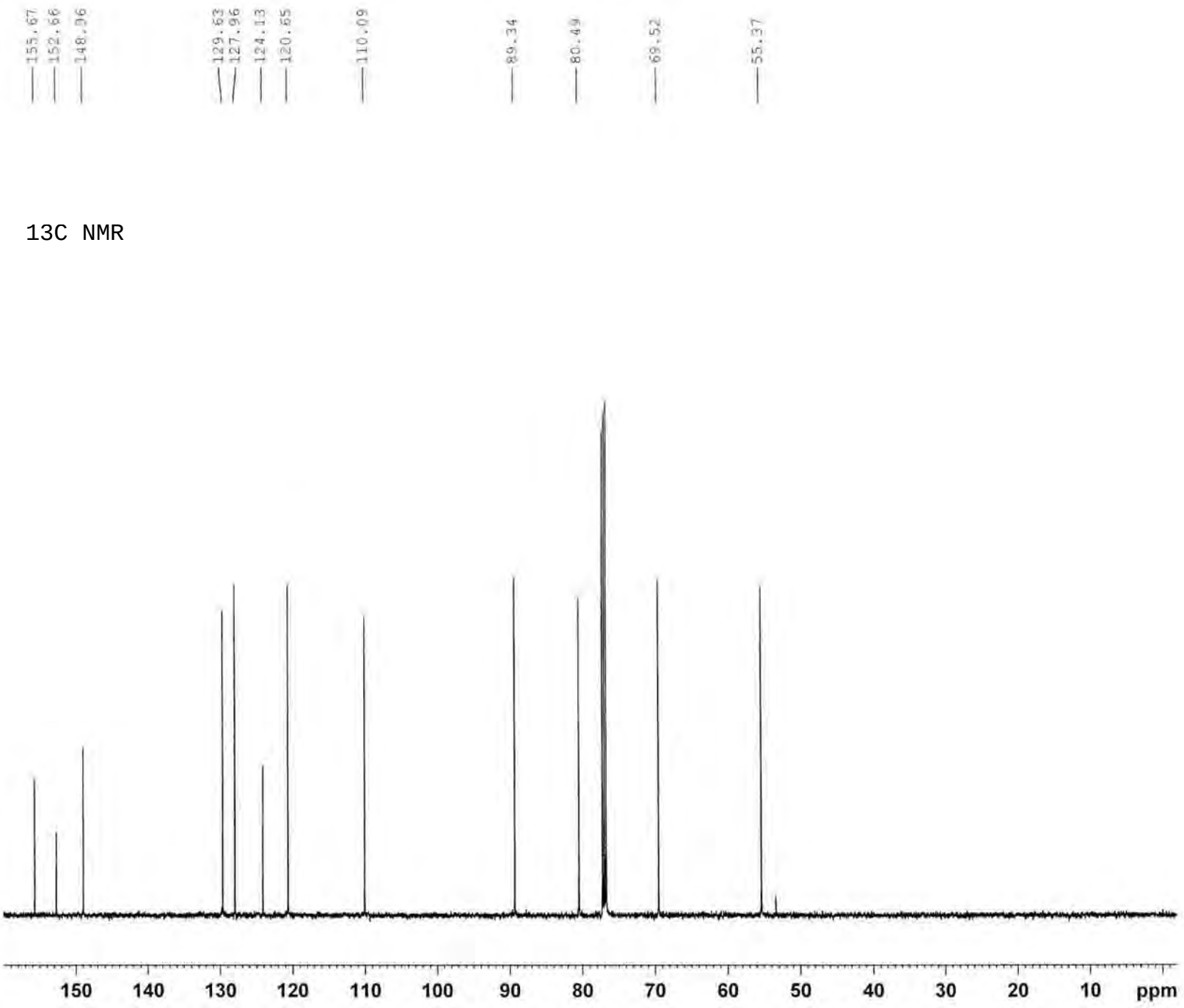


NAME	MB-66-F1
EXPNO	1
PROCNO	1
Date_	20100323
Time	10.57
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4652.605 Hz
FIDRES	0.141986 Hz
AQ	3.5215178 sec
RG	101
DW	107.467 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
CHANNEL f1	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2620986 MHz
SI	32768
SF	500.2600141 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

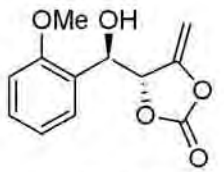


2b *anti*

13C NMR



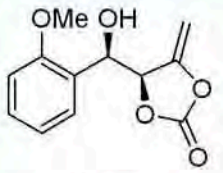
NAME	MB-66-F1
EXPNO	2
PROCNO	1
Date_	20100323
Time	11.1
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	405
DS	4
SWH	29761.904
FIDRES	0.908261
AQ	0.550552
RG	1030
DW	16.800
DE	6.50
TE	298.0
D1	2.00000000
D11	0.03000000
TD0	1
======== CHANNEL f1 ==========	
NUC1	13C
P1	11.50
PL1	3.00
PL1W	32.22848893
SFO1	125.8043140
======== CHANNEL f2 ==========	
CPDPRG2	waltz1
NUC2	1H
PCPD2	80.00
PL2	1.20
PL12	18.40
PL13	18.40
PL2W	20.76952171
PL12W	0.39575511
PL13W	0.39575511
SFO2	500.262098
SI	32768
SF	125.790484
WDW	EM
SSB	0
LB	1.50
GB	0
PC	1.40



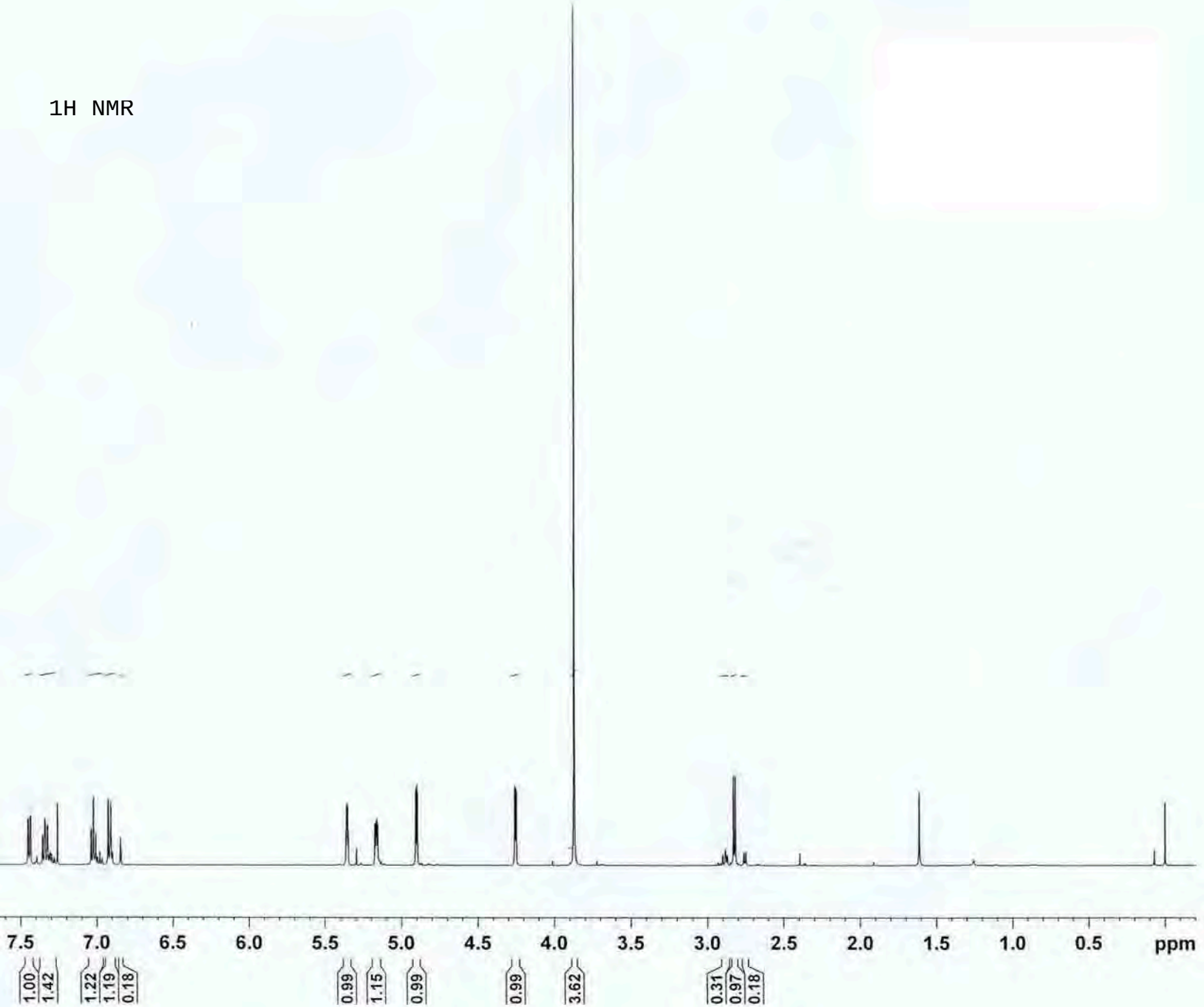
2b-*anti*

1H NMR

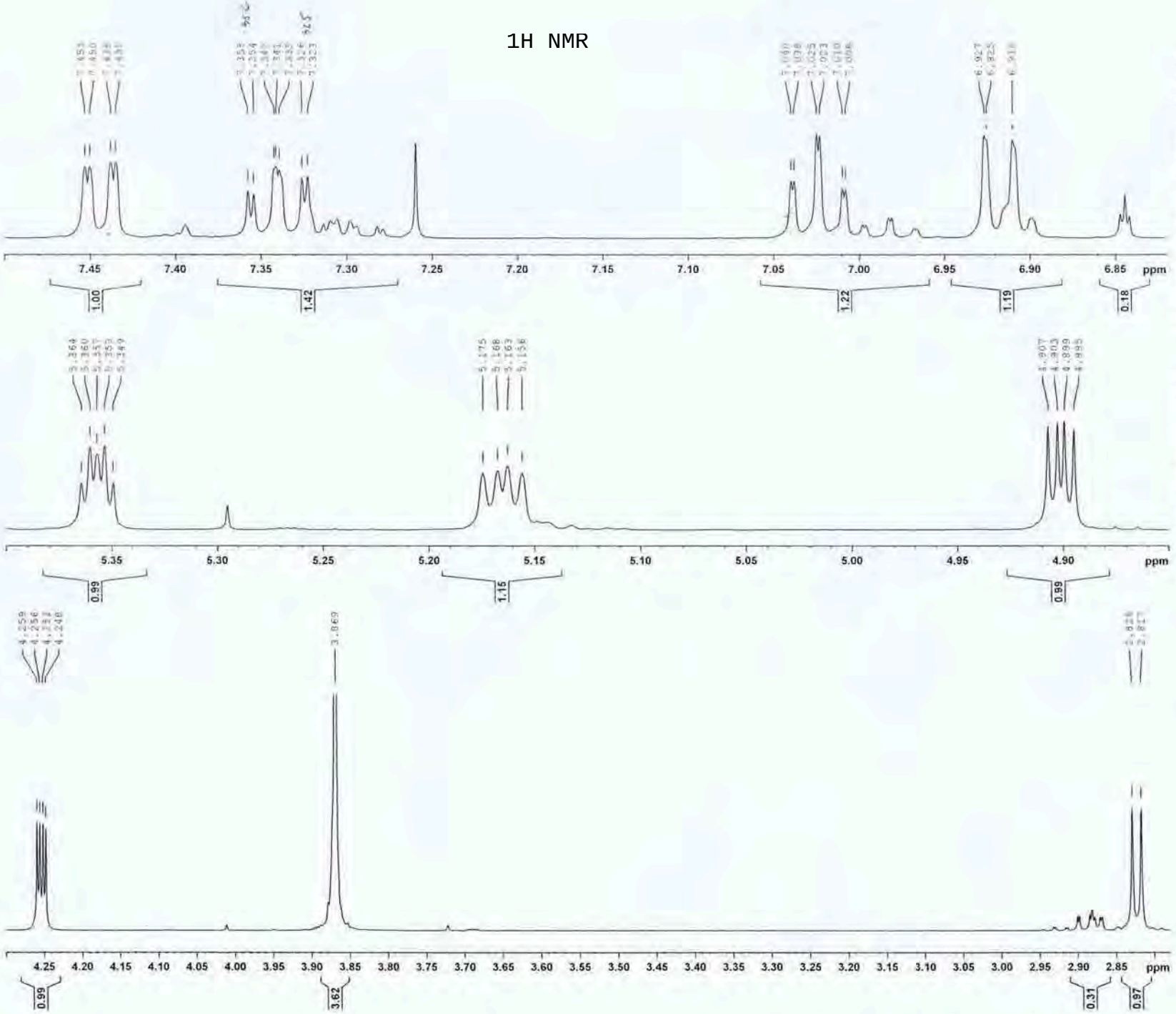
NAME	MB-66-P2
EXPNO	1
PROCNO	1
Date_	20100324
Time	14.30
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4480.287 Hz
FIDRES	0.136727 Hz
AQ	3.6569588 sec
RG	228
DW	111.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2617792 MHz
SI	32768
SF	500.2600146 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



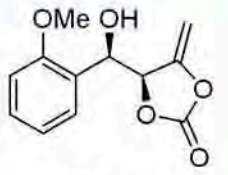
2b *syn*



1H NMR

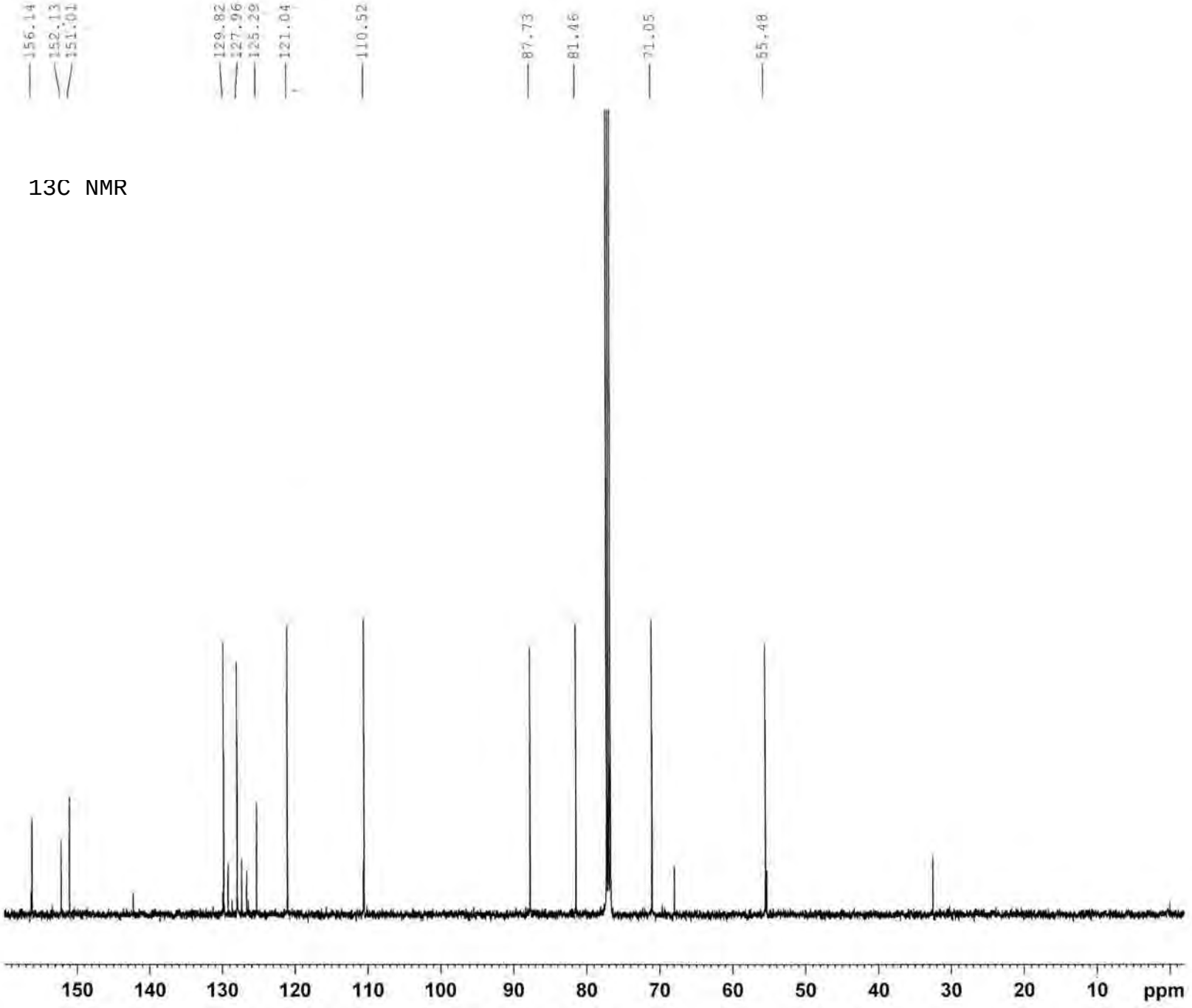


NAME	MB-66-F2
EXPNO	1
PROCNO	1
Date	20100324
Time	14.30
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4480.287 Hz
FIDRES	0.136727 Hz
AQ	3.6569598 sec
RG	228
DW	111.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
CHANNEL f1	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2617792 MHz
SI	32768
SF	500.2600146 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

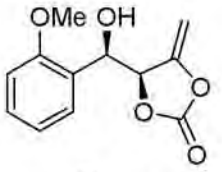


2b ***syn***

13C NMR

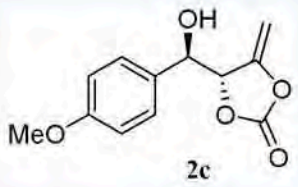


NAME	MB-66-F2
EXPNO	S27
PROCNO	
Date_	20100324
Time	14.38
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	810
DS	4
SWH	29761.904
FIDRES	0.908263
AQ	0.5505524
RG	1030
DW	16.800
DE	6.50
TE	298.0
D1	2.00000000
D11	0.03000000
TD0	1
======== CHANNEL f1 =========	
NUC1	13C
P1	11.50
PL1	3.04
PL1W	32.22848892
SFO1	125.8043140
======== CHANNEL f2 =========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00
PL2	1.20
PL12	18.40
PL13	18.40
PL2W	20.76952171
PL12W	0.39575511
PL13W	0.39575511
SFO2	500.2617794
SI	32768
SF	125.790482
WDW	EM
SSB	
LB	1.50
GB	
PC	1.40



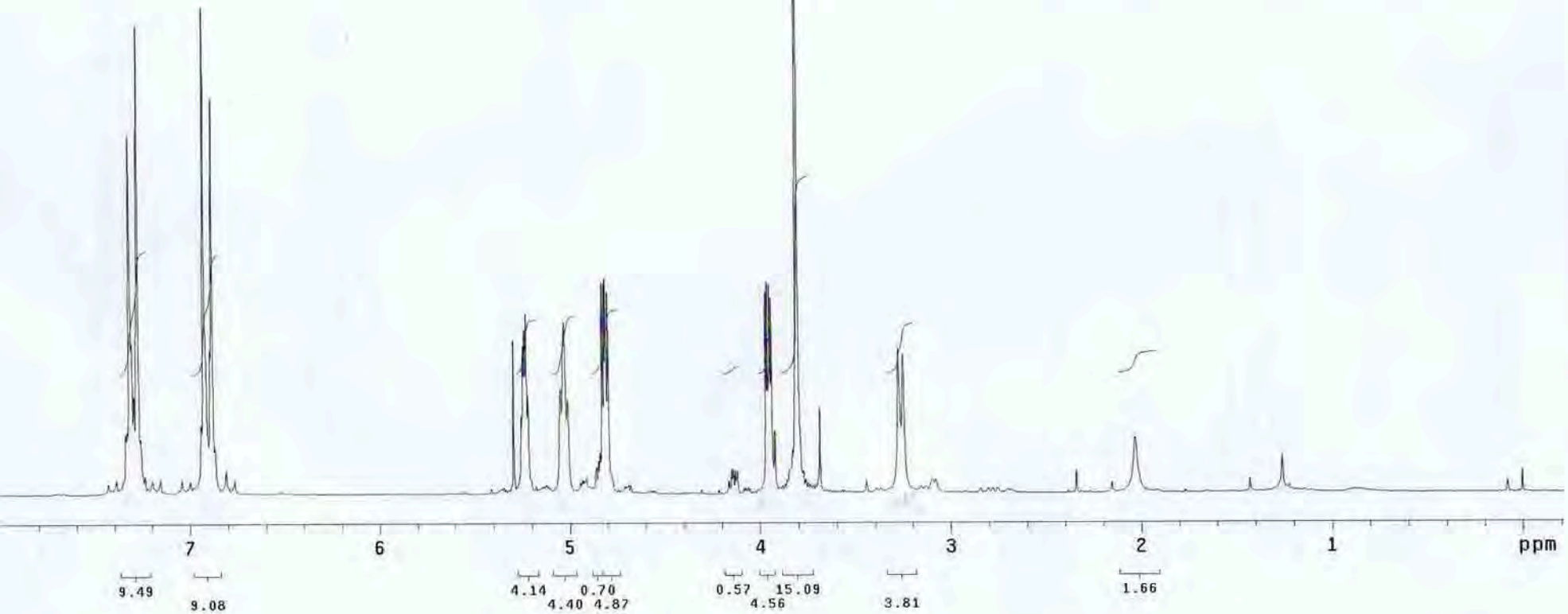
2b-syn

MB-71
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710945 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute



2c

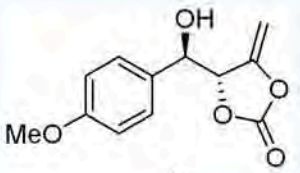
1H NMR



MB-71

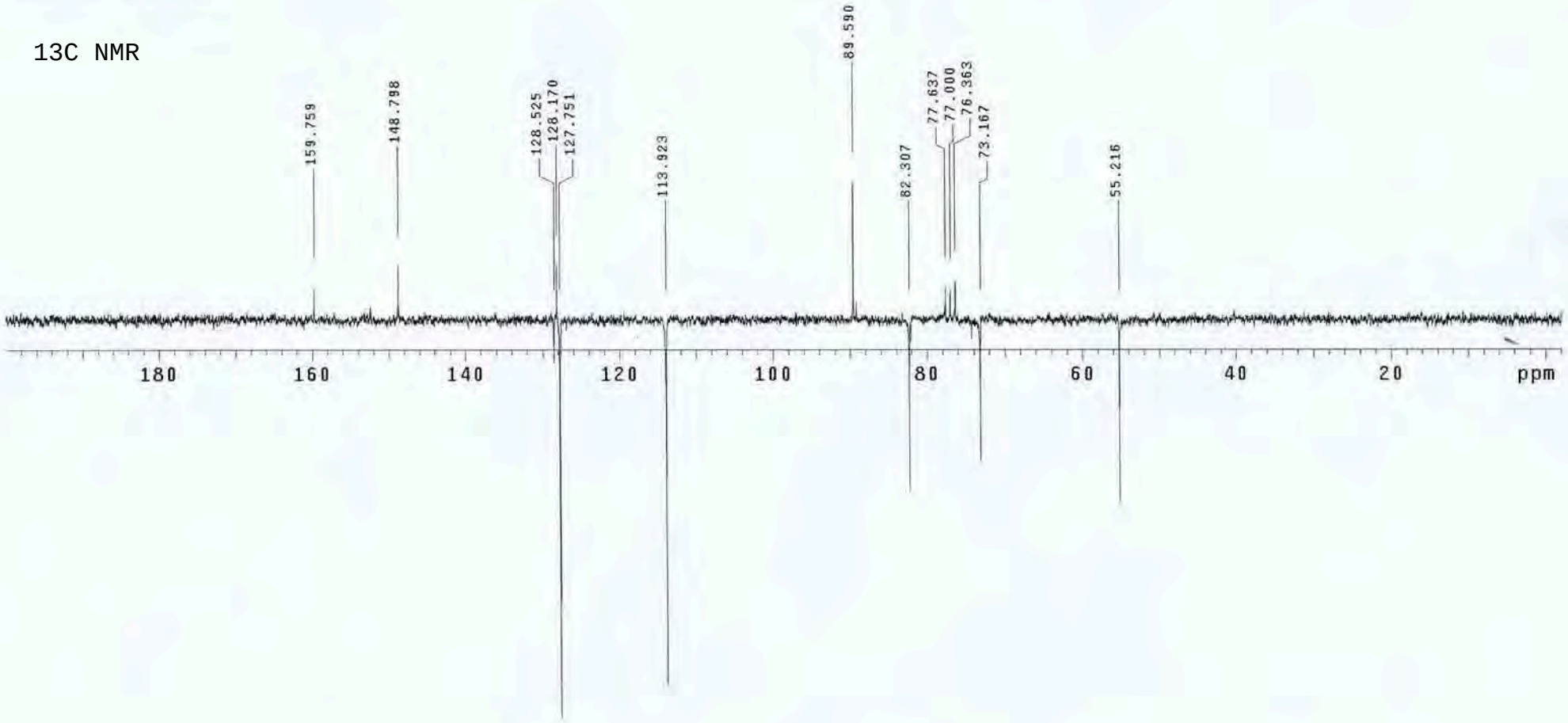
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827812 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 19 minutes



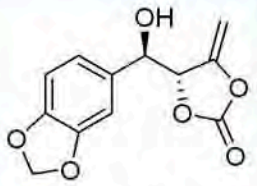
2c

13C NMR



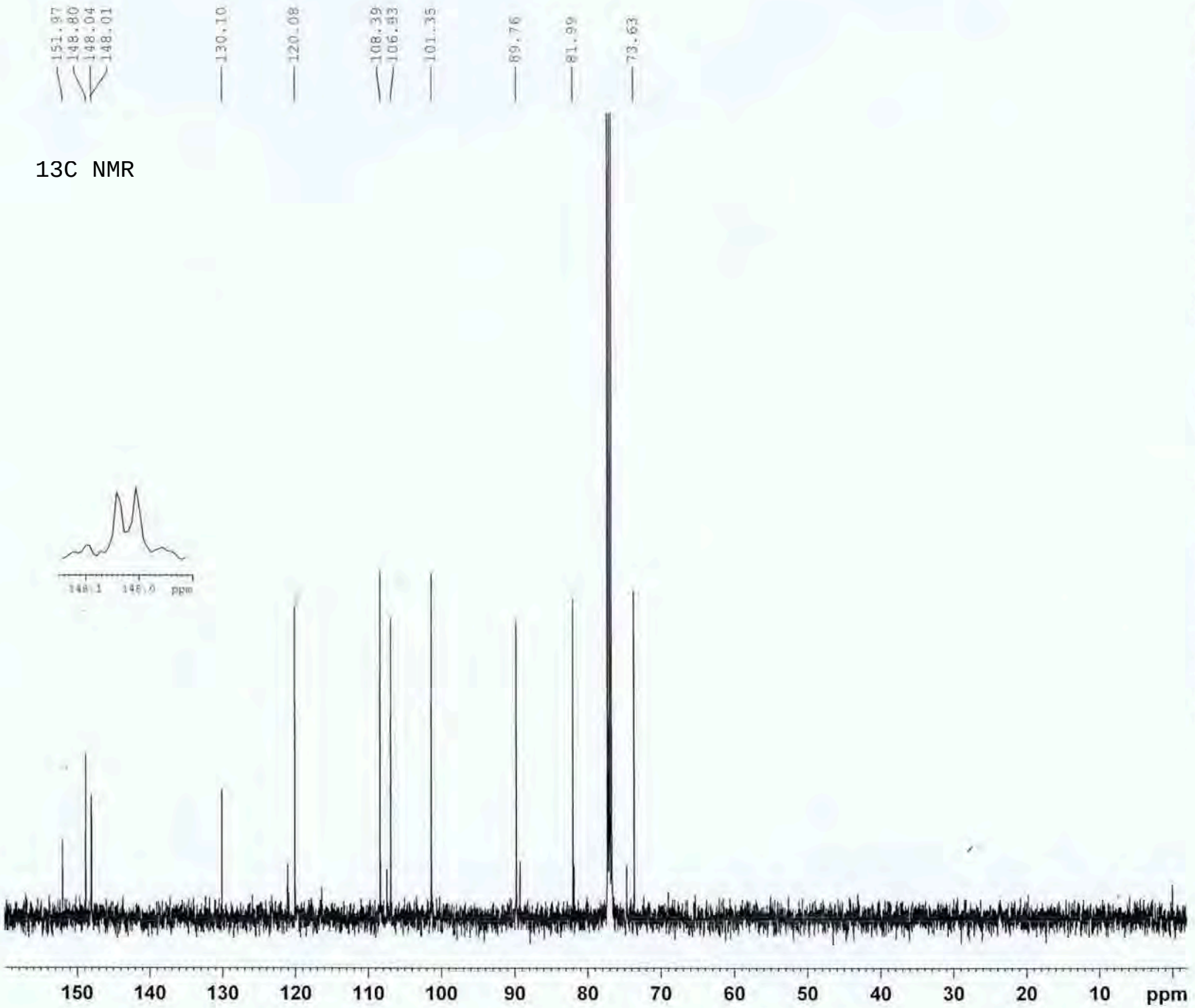
1H NMR

NAME	MB-69
EXPNO	1
PROCNO	1
Date	20100427
Time	12.36
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4464.286 Hz
FIDRES	0.136239 Hz
AQ	3.6700661 sec
RG	322
DW	112.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
CHANNEL f1	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2617863 MHz
SI	32768
SF	500.2600139 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

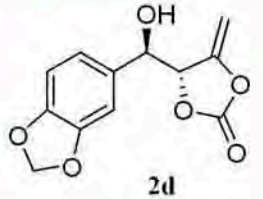


2 d

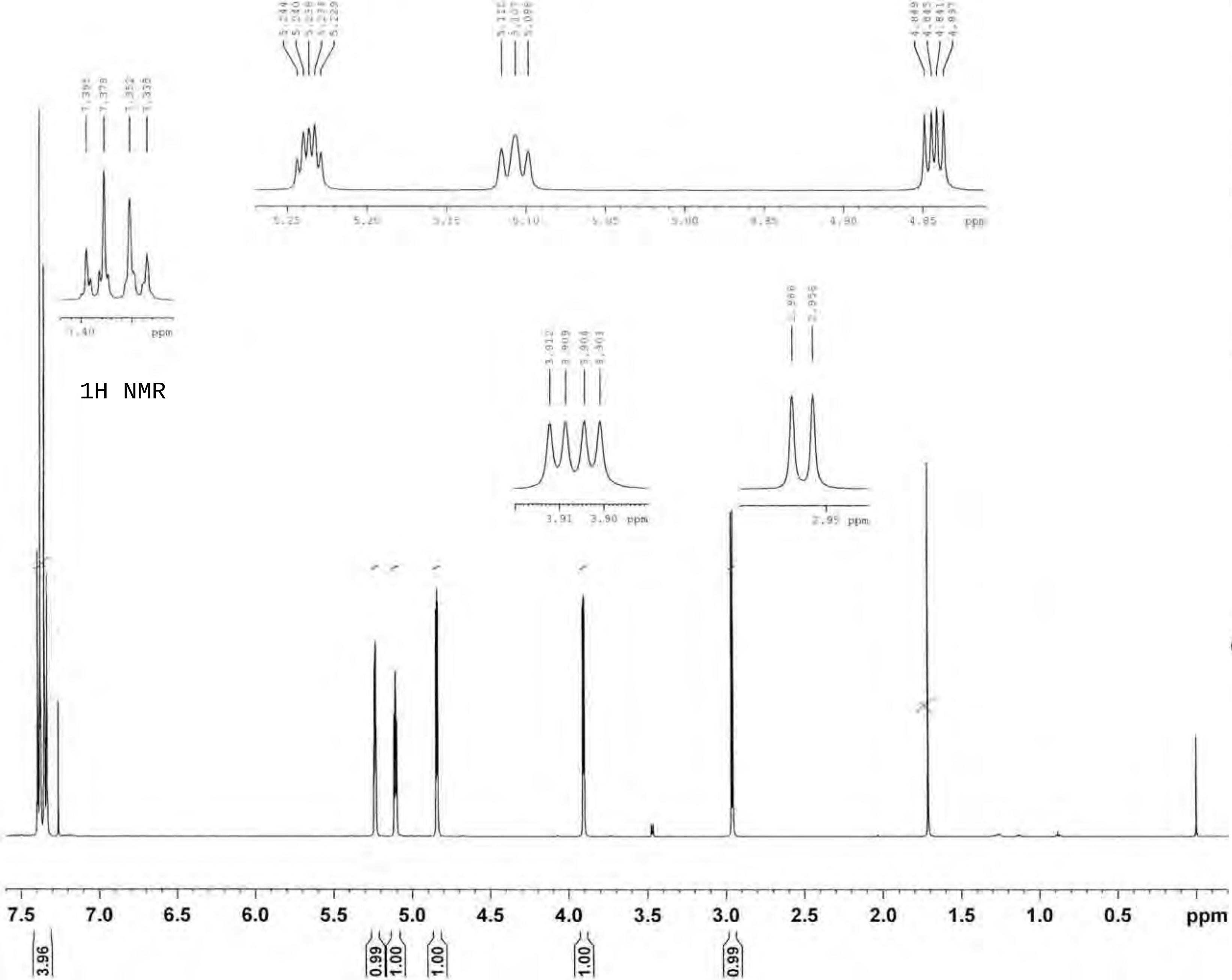
13C NMR



NAME	MB-69
EXPNO	2
PROCNO	1
Date_	20100427
Time	12.47
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	402
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1440
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2617864 MHz
SI	32768
SF	125.7904805 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

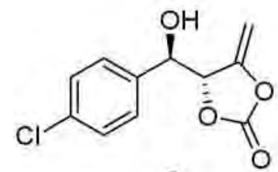


2d



PROCNO
Date_ 201006
Time 11.
INSTRUM spe
PROBHD 5 mm BBO BB-
PULPROG zg
TD 327
SOLVENT CDC
NS
DS
SWH 4485.6
FIDRES 0.1368
AQ 3.65258
RG
DW 111.
DE 6.
TE 298
D1 2.000000
TD0

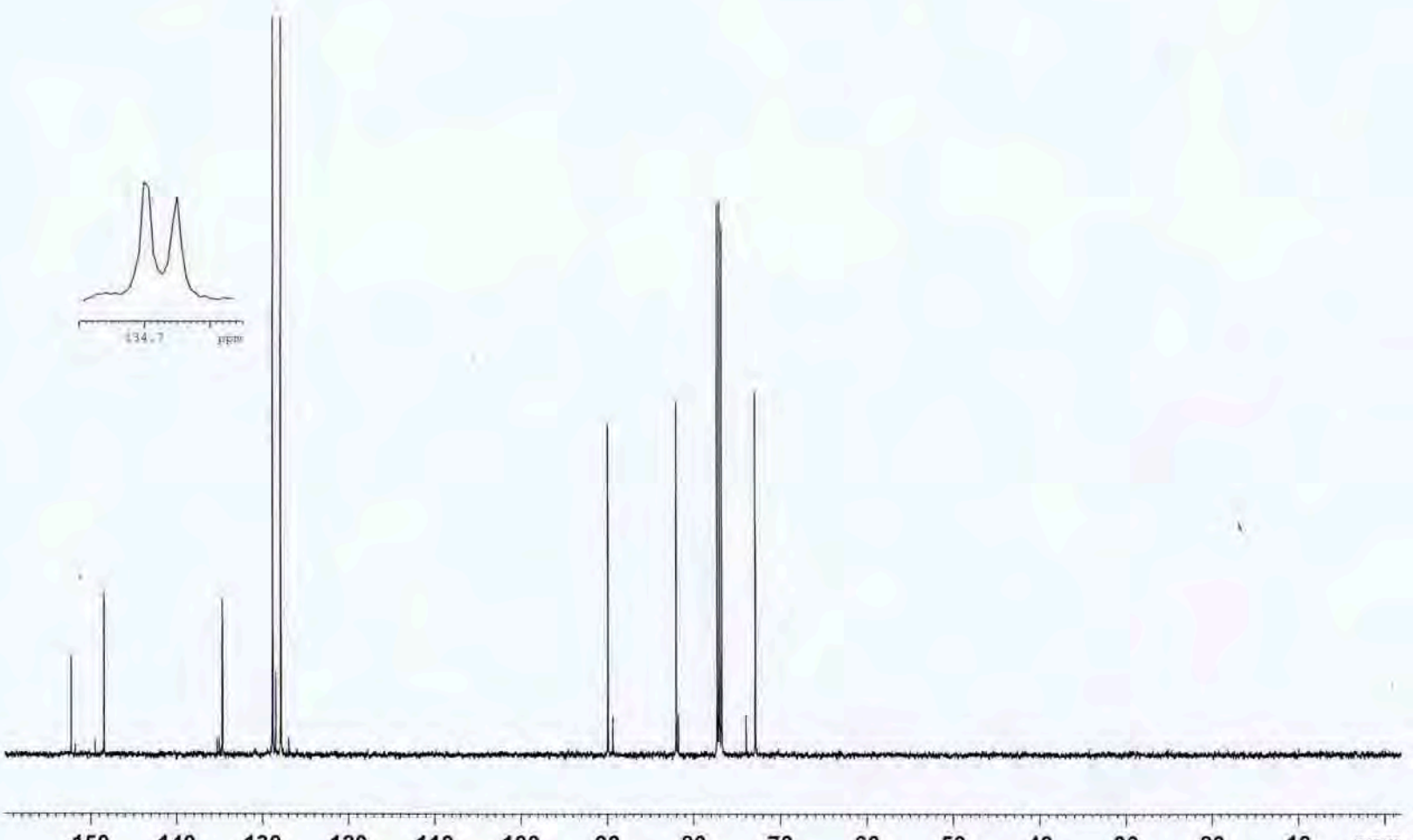
CHANNEL f1
NUC1
P1
PL1
PL1W 27.379562
SFO1 500.26190
SI 327
SF 500.26001
WDW
SSB
LB 0.
GB
PC 1.



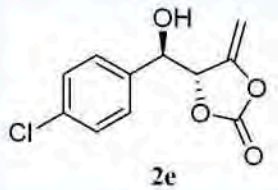
2e

152.22
148.42
134.70
134.65
128.82
127.88
89.92
82.00
72.86

13C NMR



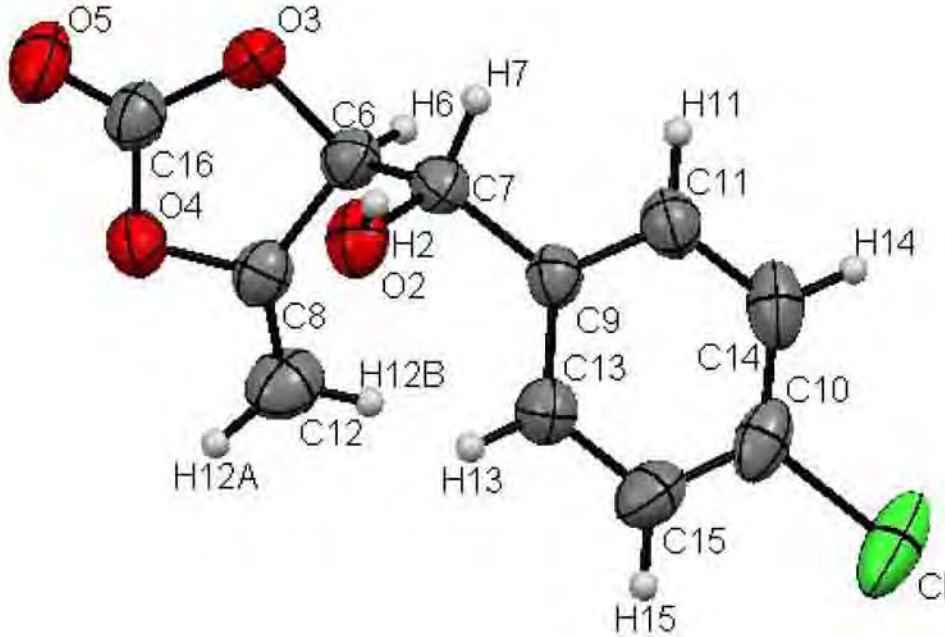
NAME	MB-82
EXPNO	2
PROCNO	1
Date_	20100520
Time	14.14
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	482
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	812
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2620336 MHz
SI	32768
SF	125.7904838 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

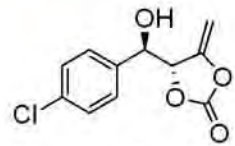


2e

V

ORTEP diagram for 2e



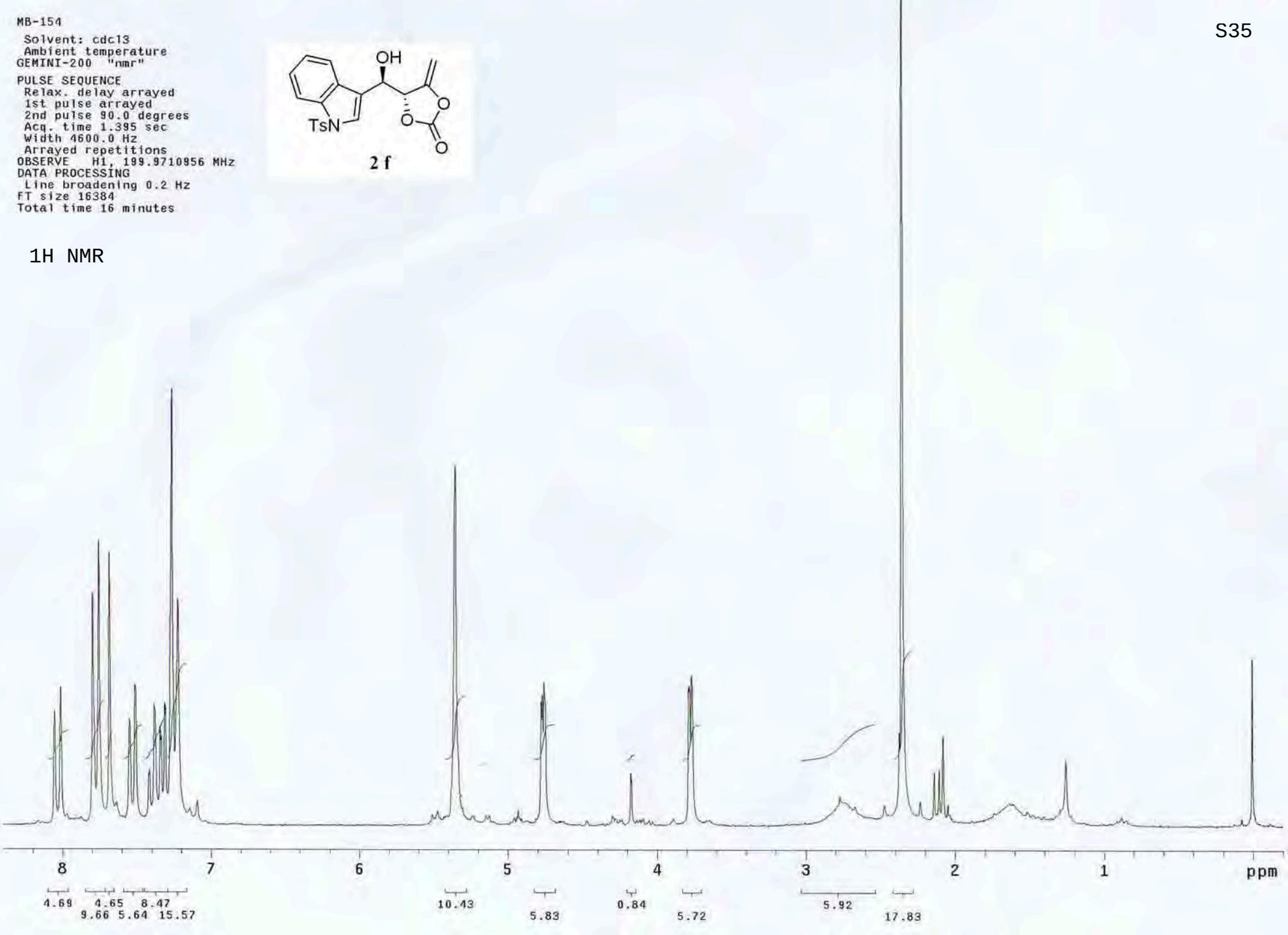


2e

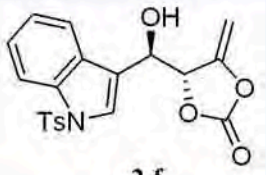
MB-154
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.395 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710956 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 16 minutes

2 f

1H NMR

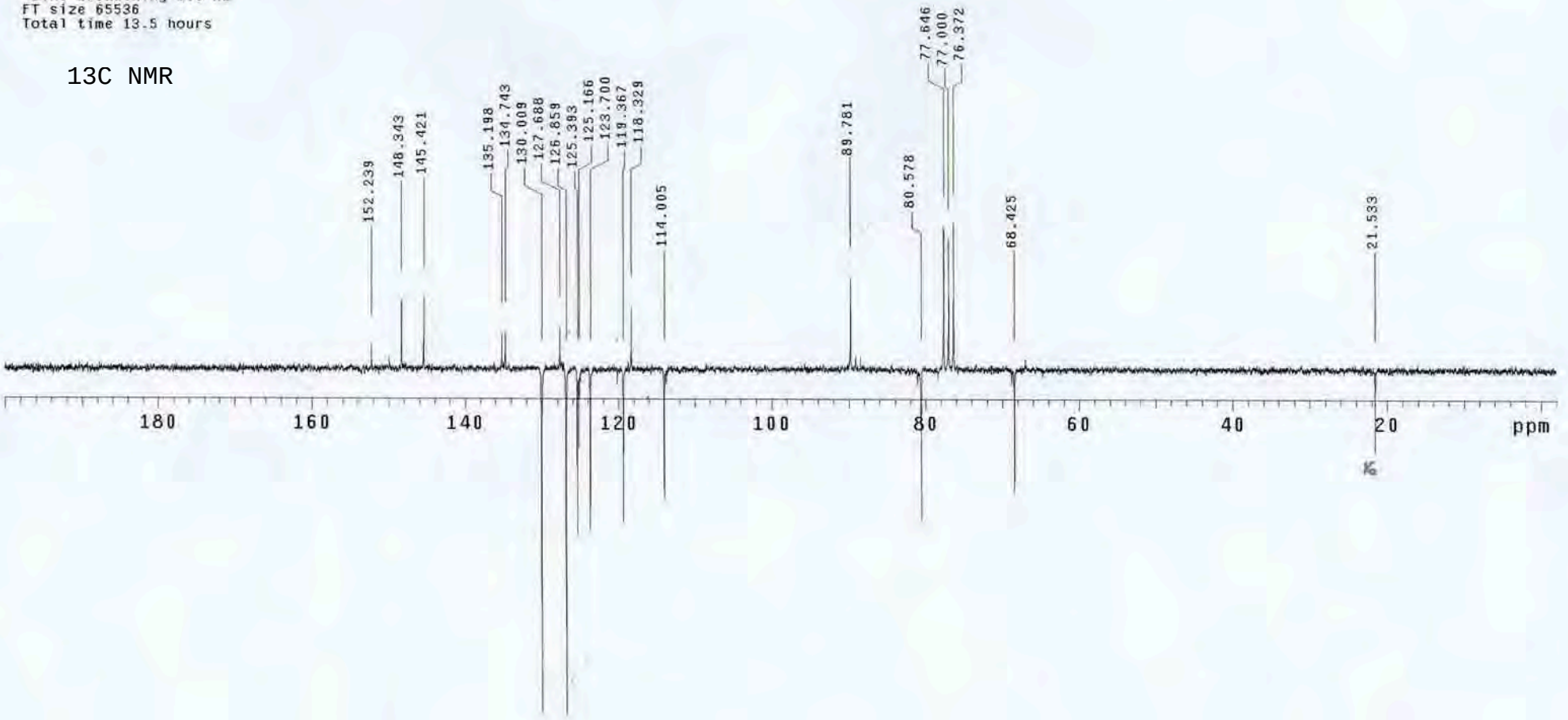


MB-154
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827794 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 13.5 hours



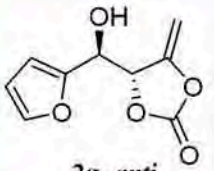
2 f

13C NMR

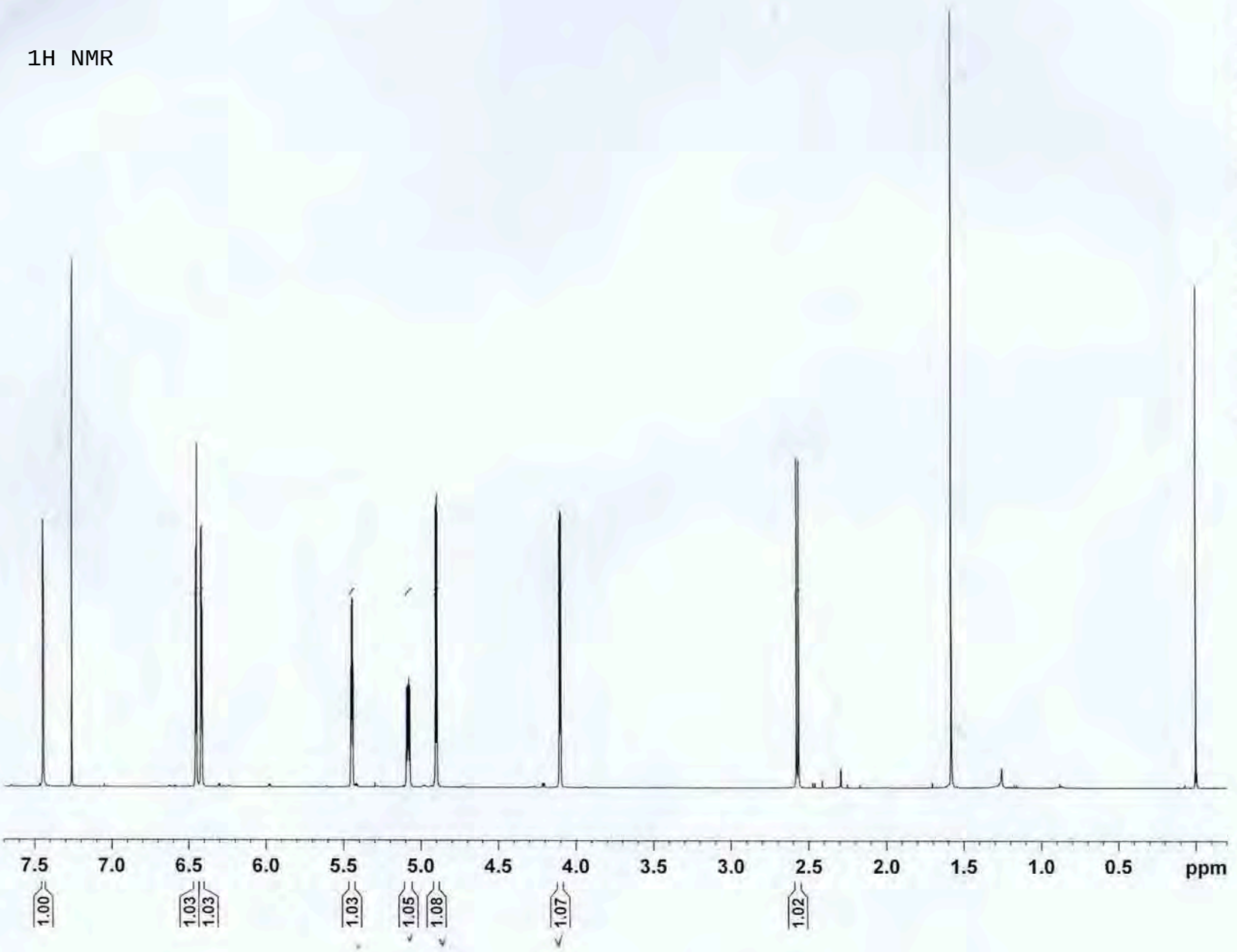


1H NMR

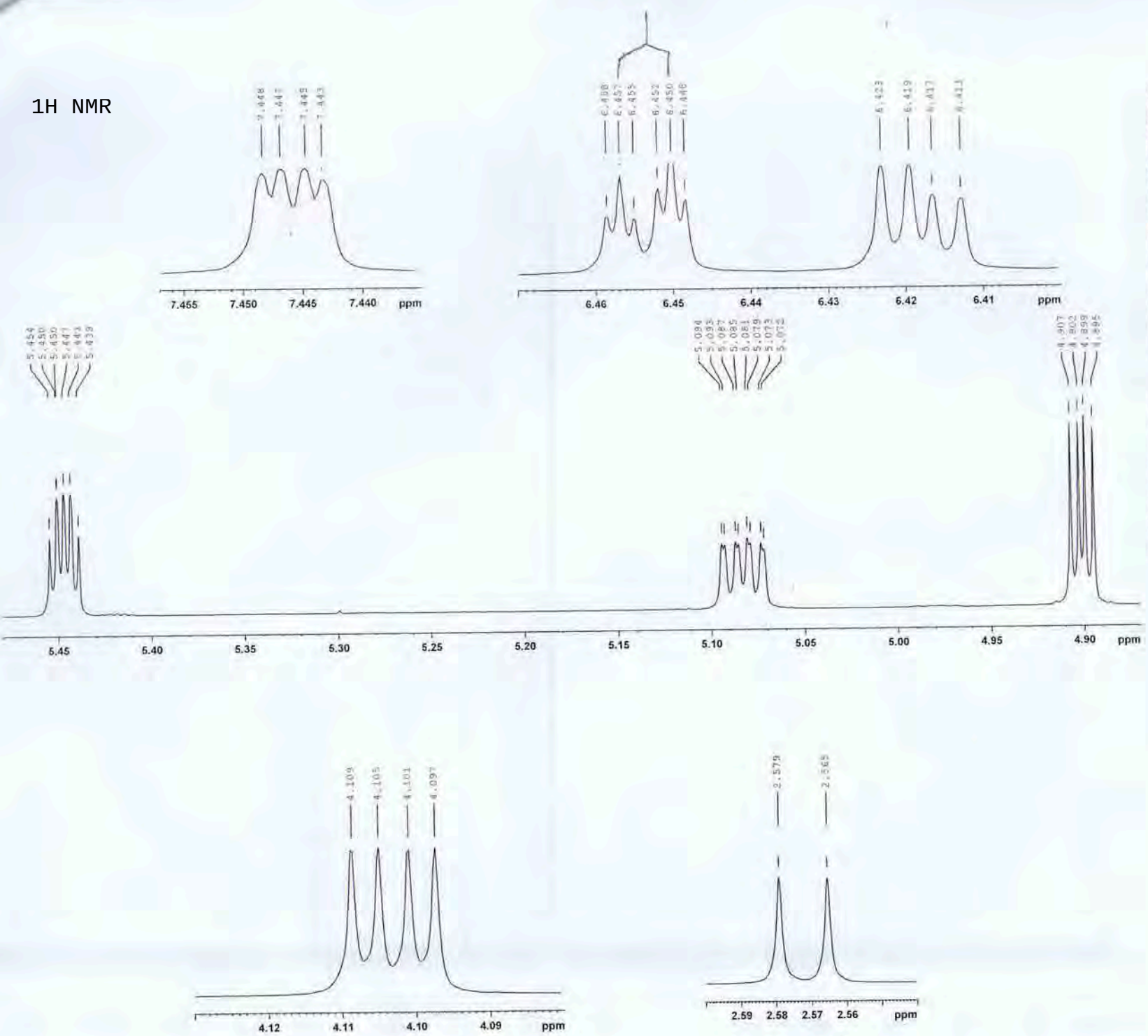
NAME	MB-72-D1
EXPNO	1
PROCNO	1
Date_	20100330
Time	13.40
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4496.403 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	287
DW	111.200 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618990 MHz
SI	32768
SF	500.2600139 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



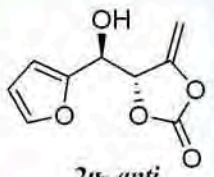
2g- *anti*



1H NMR

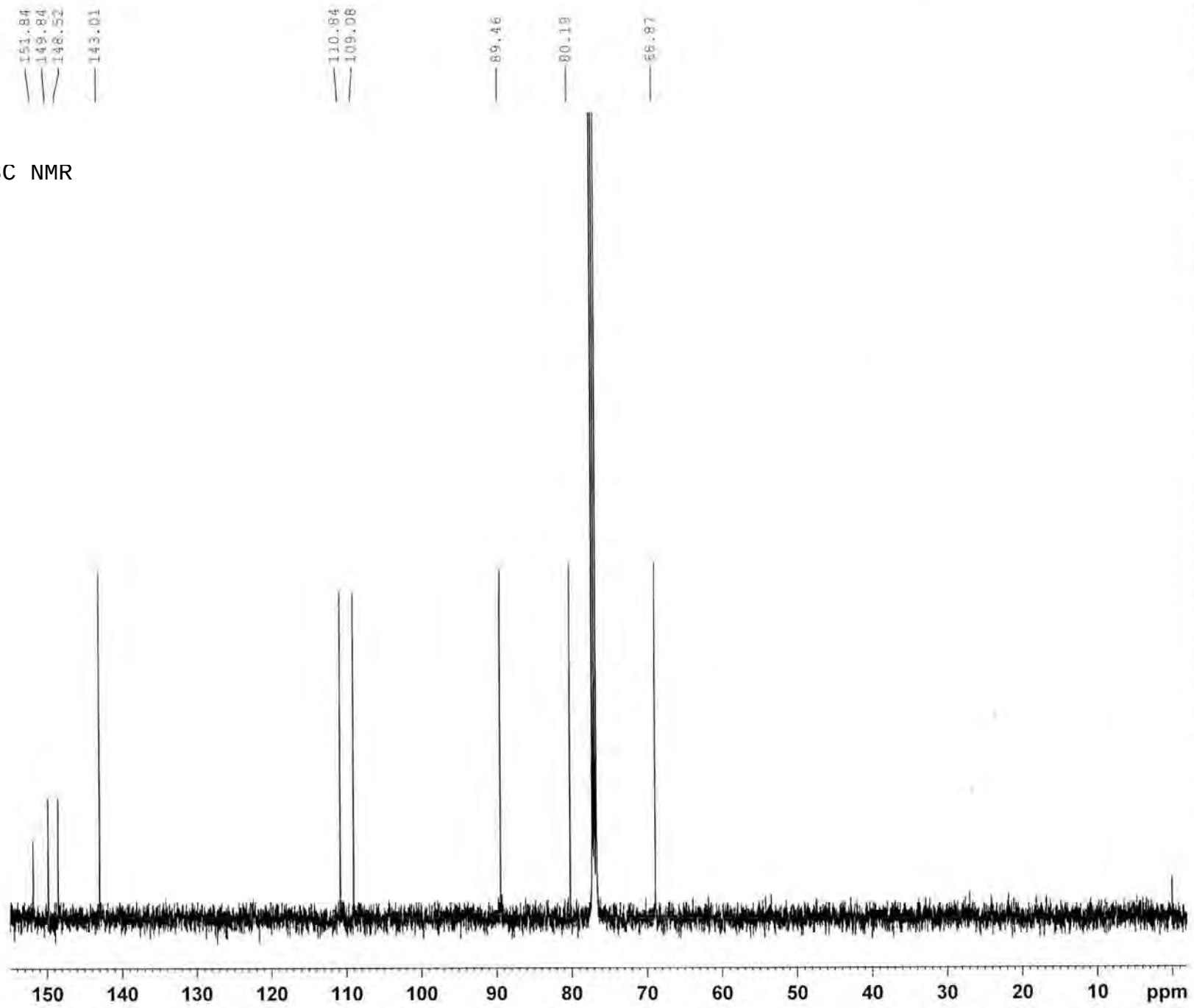



```
NAME            MB-72-DJ
EXPNO                  1
PROCNO                 1
Date_           20100330
Time               13.40
INSTRUM             spect
PROBHD  5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT             CDCl3
NS                    16
DS                     0
SWH          4496.403 Hz
FIDRES       0.137219 Hz
AQ           3.6438515 sec
RG                   287
DW           111.200 usec
DE             6.50 usec
TE              298.0 K
D1          2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1             9.35 usec
PL1              0.00 dB
PL1W     27.37956238 W
SFO1     500.2618990 MHz
SI                 32768
SF       500.2600139 MHz
WDW                   EM
SSB                    0
LB             0.20 Hz
GB                     0
PC                  1.00
```

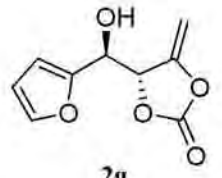


2g- *anti*

13C NMR



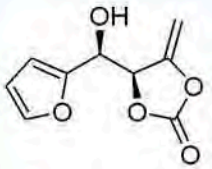
NAME	MB-72-D1
EXPNO	2
PROCNO	1
Date_	20100330
Time	14.05
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	811
DS	4
SWH	29761.904
FIDRES	0.908261
AQ	0.5505524
RG	1030
DW	16.800
DE	6.50
TE	298.0
D1	2.00000000
D11	0.03000000
TD0	1
======== CHANNEL f1 ==========	
NUC1	13C
P1	11.50
PL1	3.00
PL1W	32.22848892
SFO1	125.8043140
======== CHANNEL f2 ==========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00
PL2	1.20
PL12	18.40
PL13	18.40
PL2W	20.76952171
PL12W	0.39575511
PL13W	0.39575511
SFO2	500.2618990
SI	32768
SF	125.7904798
WDW	EM
SSB	0
LB	1.50
GB	0
PC	1.40



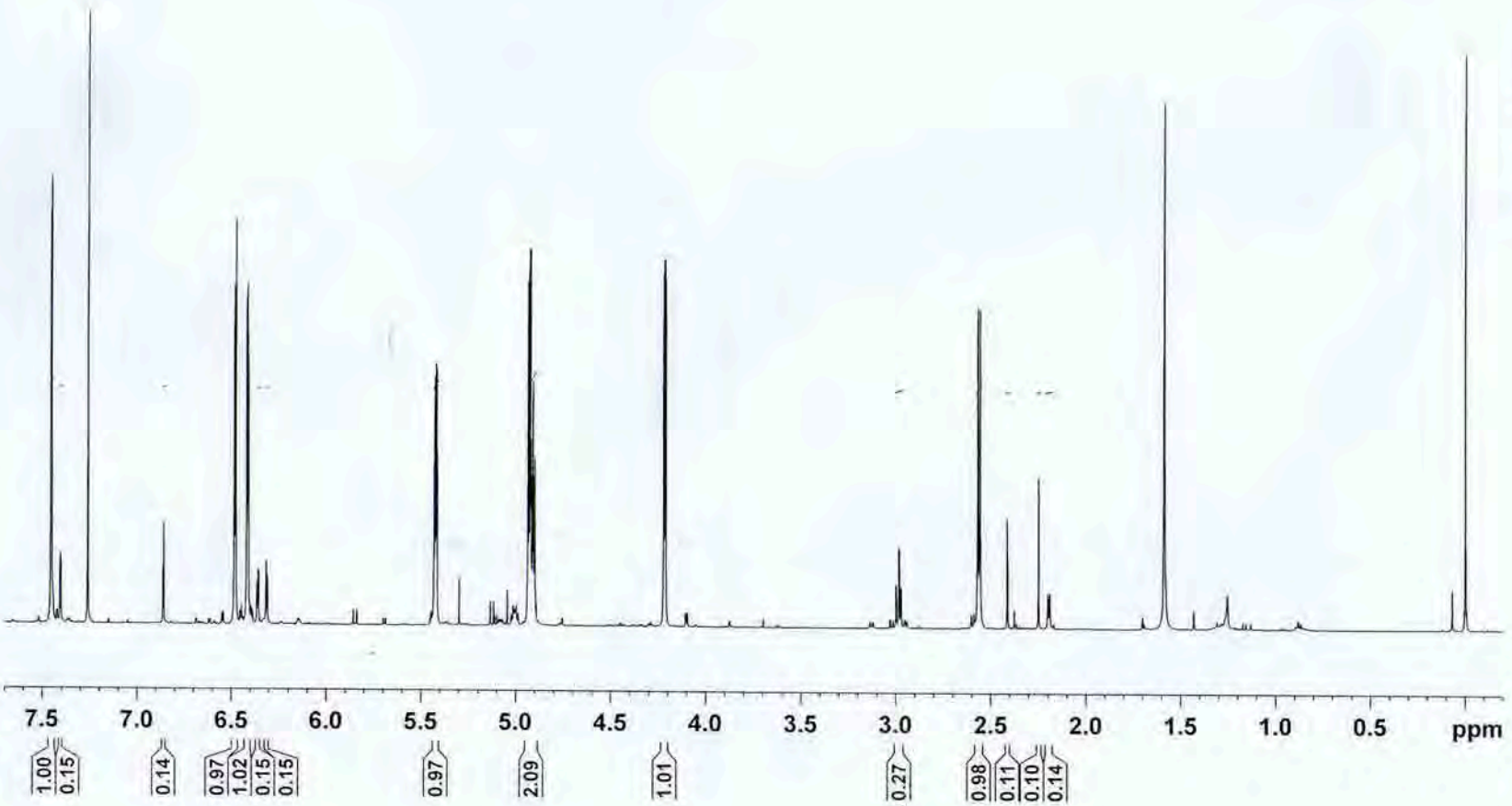
2g

1H NMR

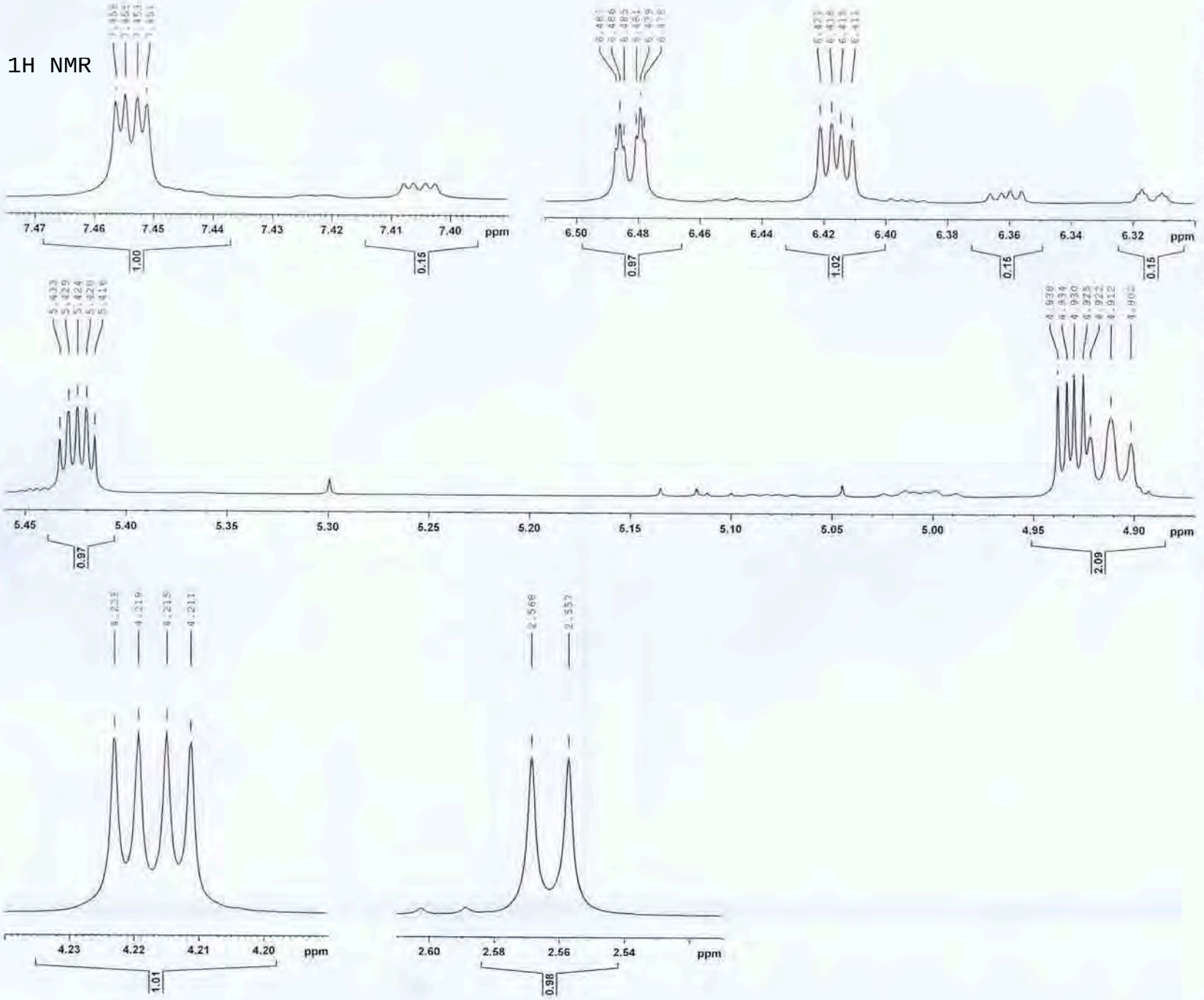
NAME	MB-72-D2
EXPNO	1
PROCNO	1
Date	20100330
Time	12.23
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4480.287 Hz
FIDRES	0.136727 Hz
AQ	3.6569588 sec
RG	287
DW	111.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618942 MHz
SI	32768
SF	500.2600137 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



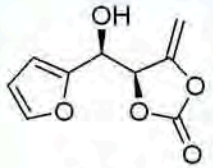
2 g- syn



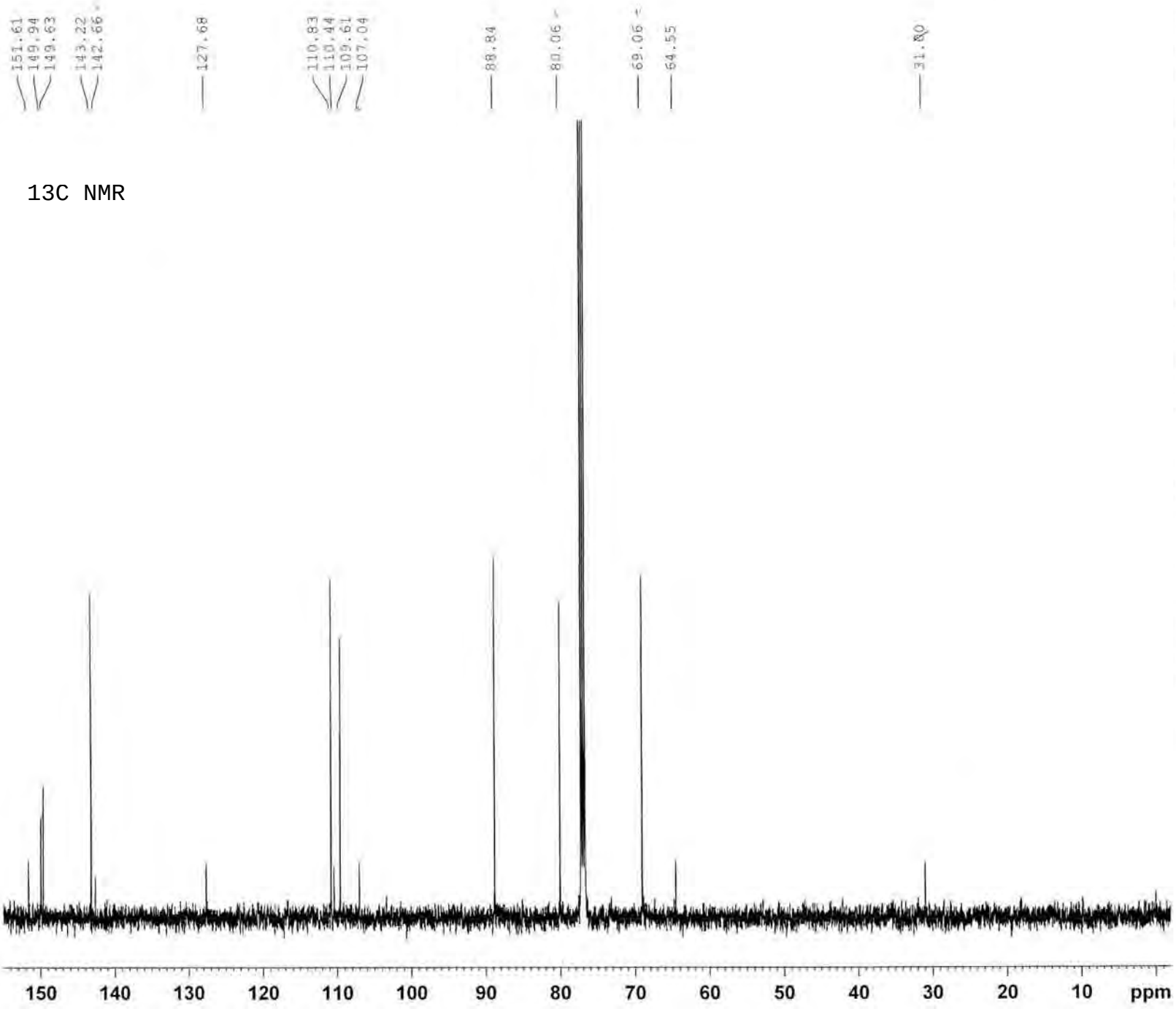
1H NMR



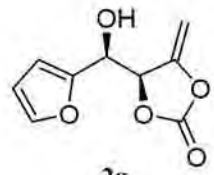
NAME	MB-72-D2
EXPNO	1
PROCNO	1
Date_	20100330
Time	12.23
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4480.287 Hz
FIDRES	0.136727 Hz
AQ	3.6569588 sec
RG	287
DW	111.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618942 MHz
SI	32768
SF	500.2600137 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



2 g- syn

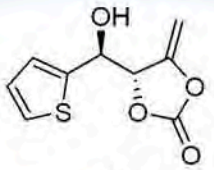


NAME	MB-72-D2
EXPNO	2
PROCNO	1
Date_	20100330
Time	12.34
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	802
DS	4
SWH	29761.904
FIDRES	0.908261
AQ	0.5505524
RG	912
DW	16.800
DE	6.50
TE	298.0
D1	2.00000000
D11	0.03000000
TD0	1
======== CHANNEL f1 =========	
NUC1	13C
P1	11.50
PL1	3.00
PL1W	32.22848892
SFO1	125.8043140
======== CHANNEL f2 =========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00
PL2	1.20
PL12	18.40
PL13	18.40
PL2W	20.76952171
PL12W	0.39575511
PL13W	0.39575511
SFO2	500.2618940
SI	32768
SF	125.7904802
WDW	EM
SSB	0
LB	1.50
GB	0
PC	1.40



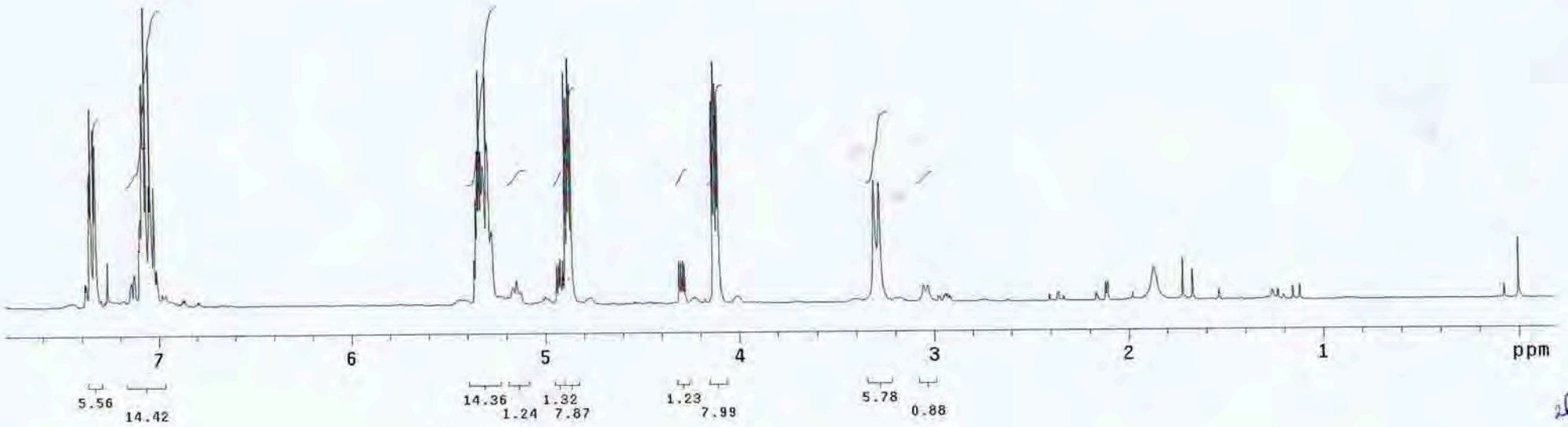
2g

MB-73
Solvent: cdcl3
Ambient temperature
File: hmb73
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710956 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 2 minutes

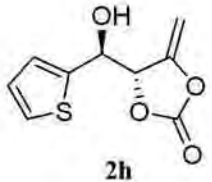


2 h

1H NMR

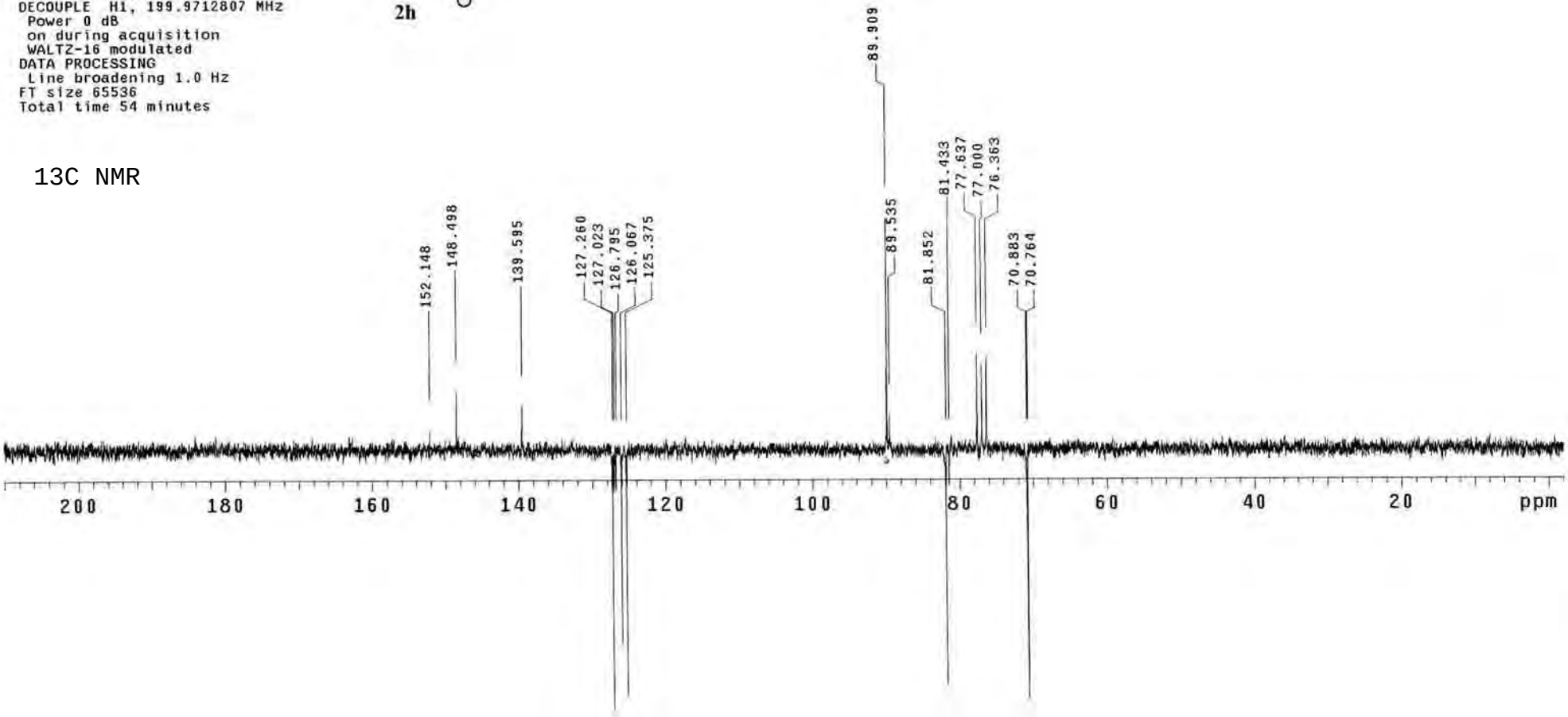


MB-73
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827798 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 54 minutes

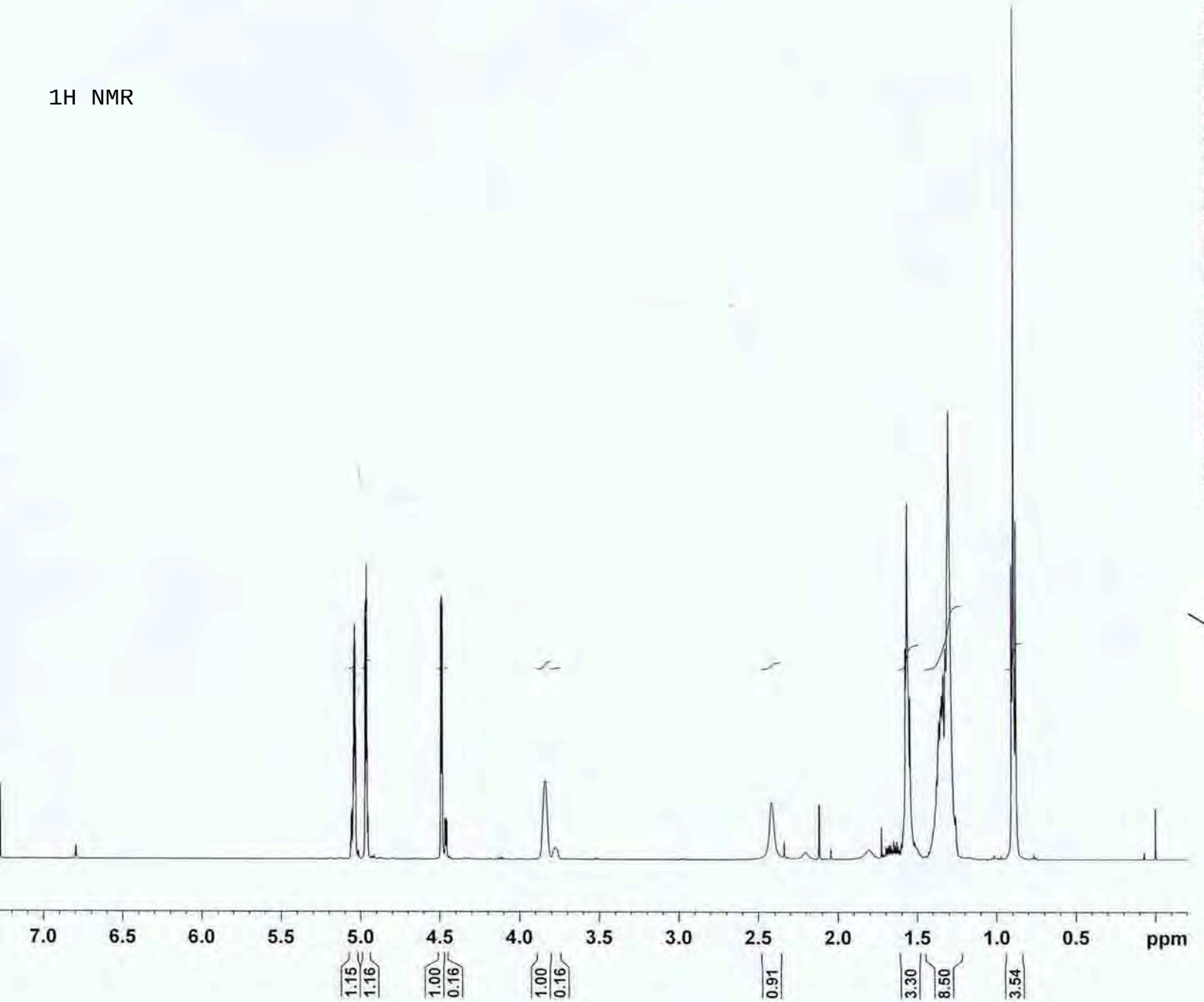


2h

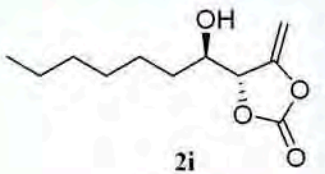
13C NMR



1H NMR

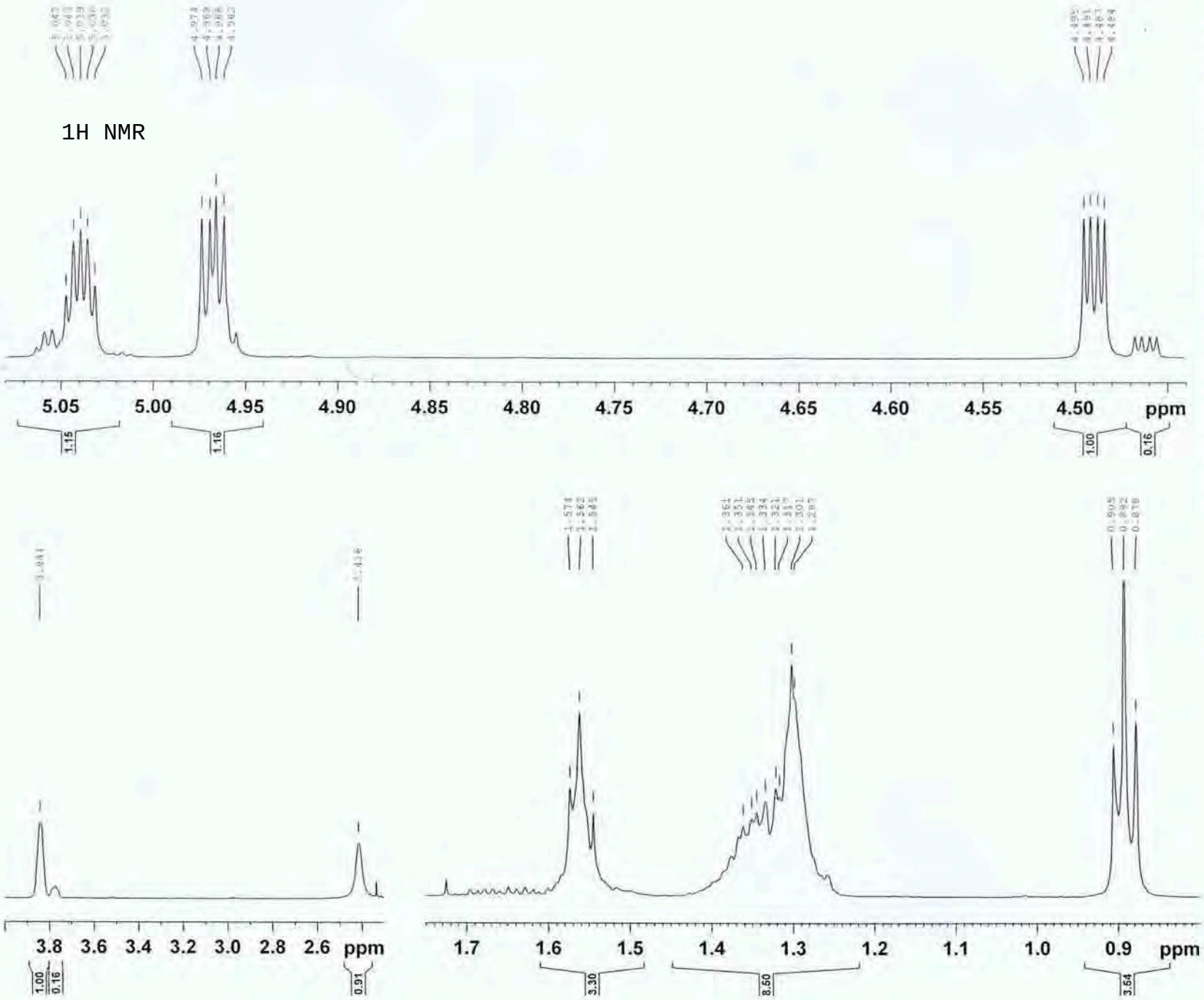


NAME	MB-63-1
EXPNO	1
PROCNO	1
Date_	20100825
Time	13.01
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4251.701 Hz
FIDRES	0.129752 Hz
AQ	3.8535669 sec
RG	114
DW	117.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2617803 MHz
SI	32768
SF	500.2600084 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

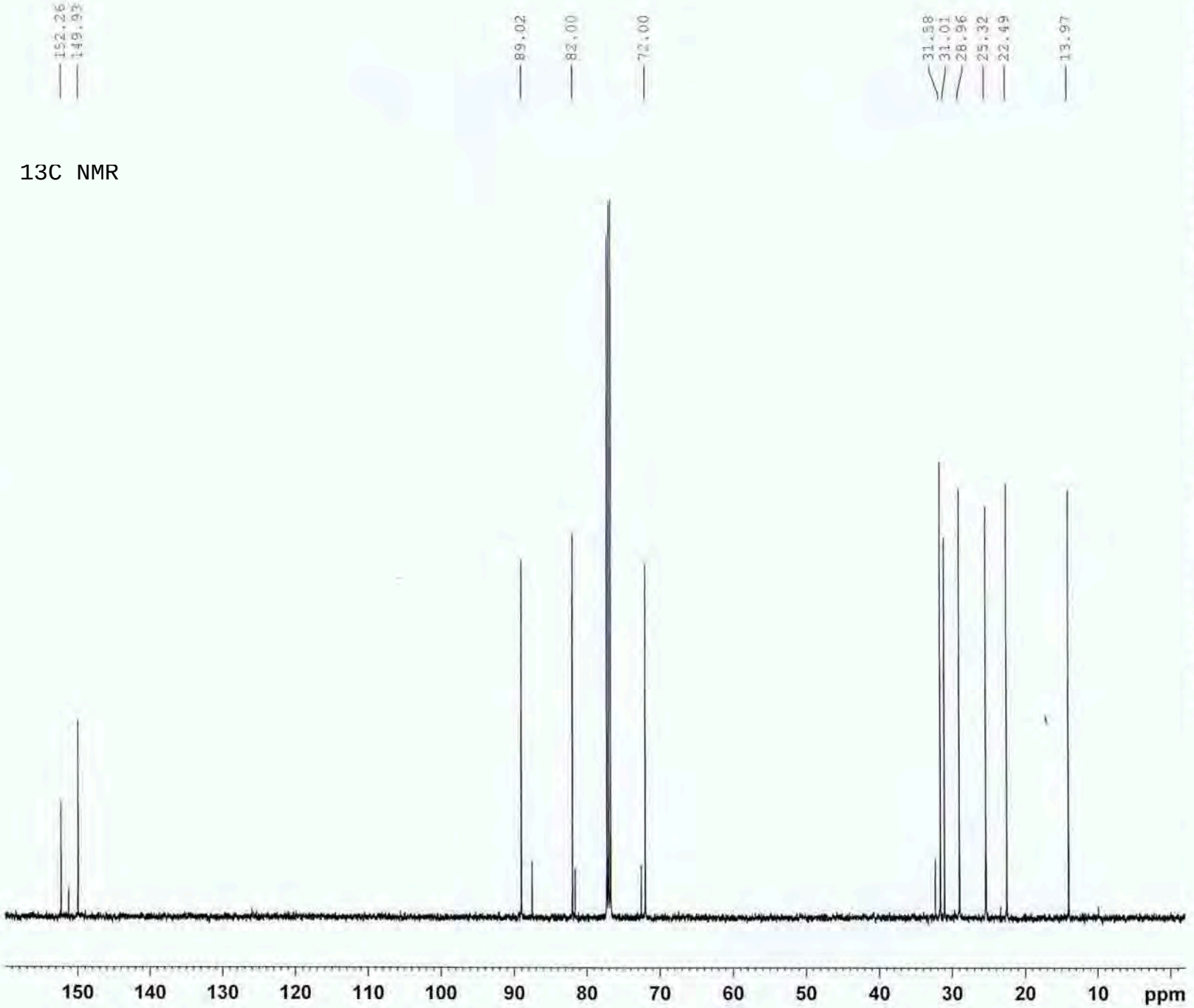


2i

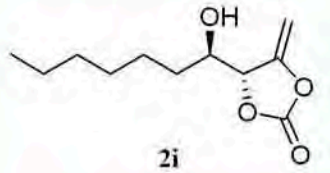
1H NMR


NAME MB-63-1
EXPNO 1
PROCNO 1
Date_ 20100825
Time 13.01
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4251.701 Hz
FIDRES 0.129752 Hz
AQ 3.8535669 sec
RG 114
DW 117.600 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 9.35 usec
PL1 0.00 dB
PL1W 27.37956238 W
SFO1 500.2617803 MHz
SI 32768
SF 500.2600084 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
5.05 5.00 4.95 4.90 4.85 4.80 4.75 4.70 4.65 4.60 4.55 4.50 ppm
1.15 1.16 1.00 0.16
3.8 3.6 3.4 3.2 3.0 2.8 2.6 ppm
1.00 0.16 0.91
1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 ppm
3.30 8.50 3.64
OH
O
O
O
2i

13C NMR



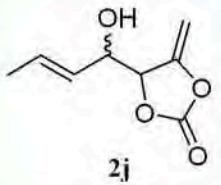
NAME	MB-63-1
EXPNO	2
PROCNO	1
Date_	20100825
Time	13.10
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	259
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	912
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
========	CHANNEL f2 ========
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2617804 MHz
SI	32768
SF	125.7904833 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



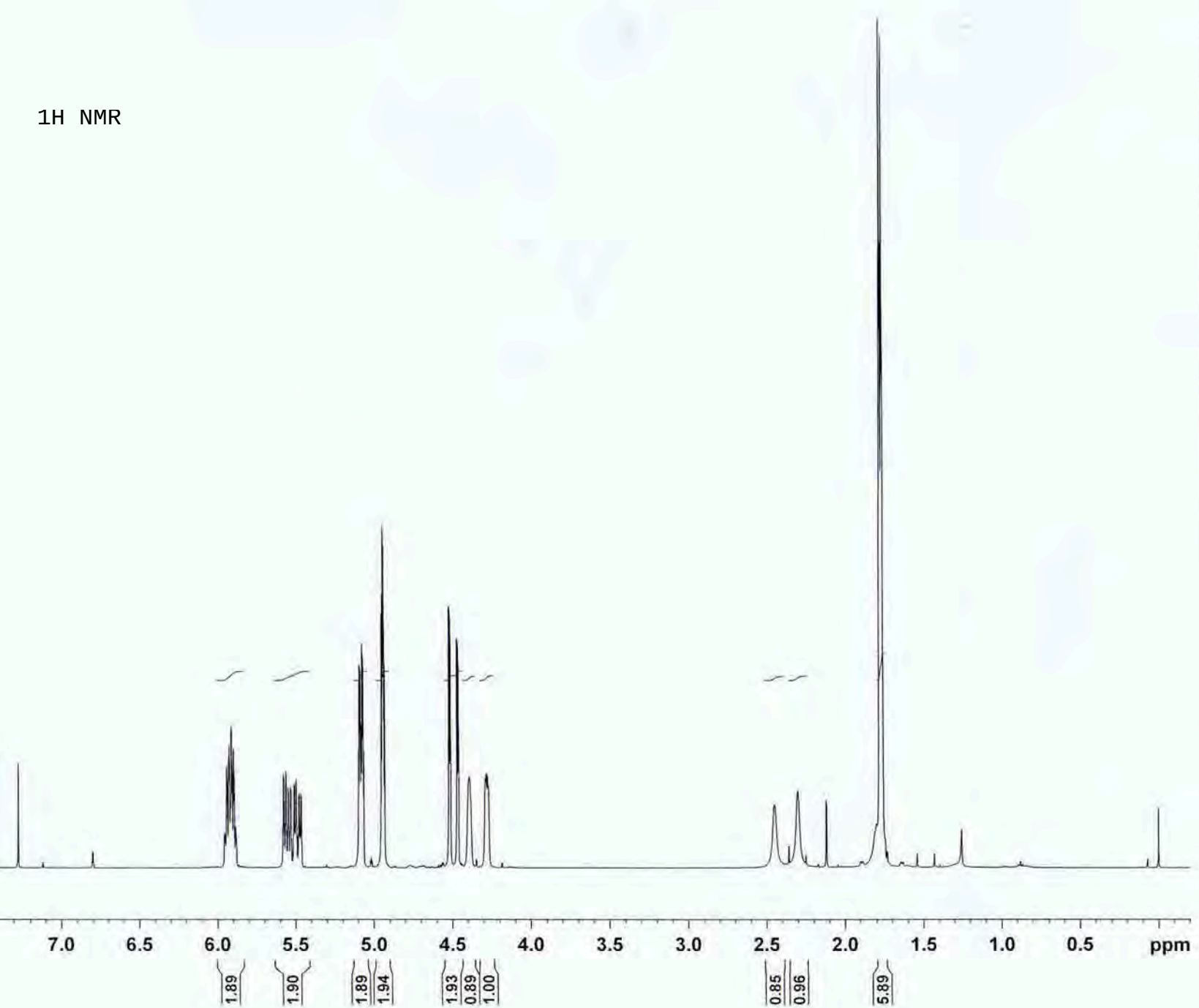
2i

1H NMR

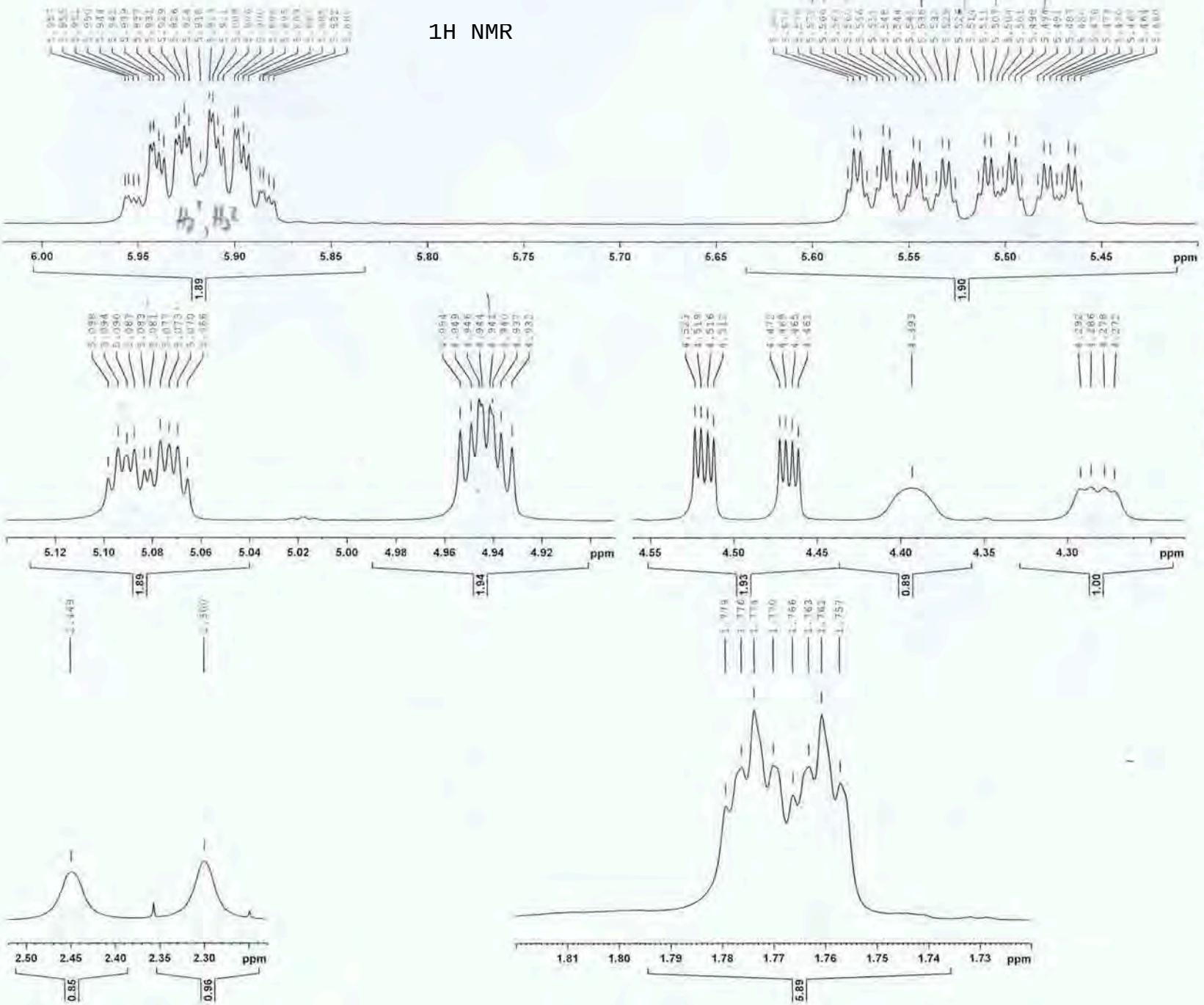
NAME	MB-64-1
EXPNO	1
PROCNO	1
Date_	20100903
Time	11.48
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4385.965 Hz
FIDRES	0.133849 Hz
AQ	3.7356019 sec
RG	114
DW	114.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618178 MHz
SI	32768
SF	500.2600079 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



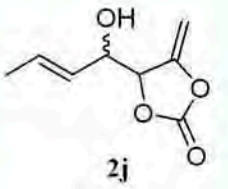
2j



1H NMR

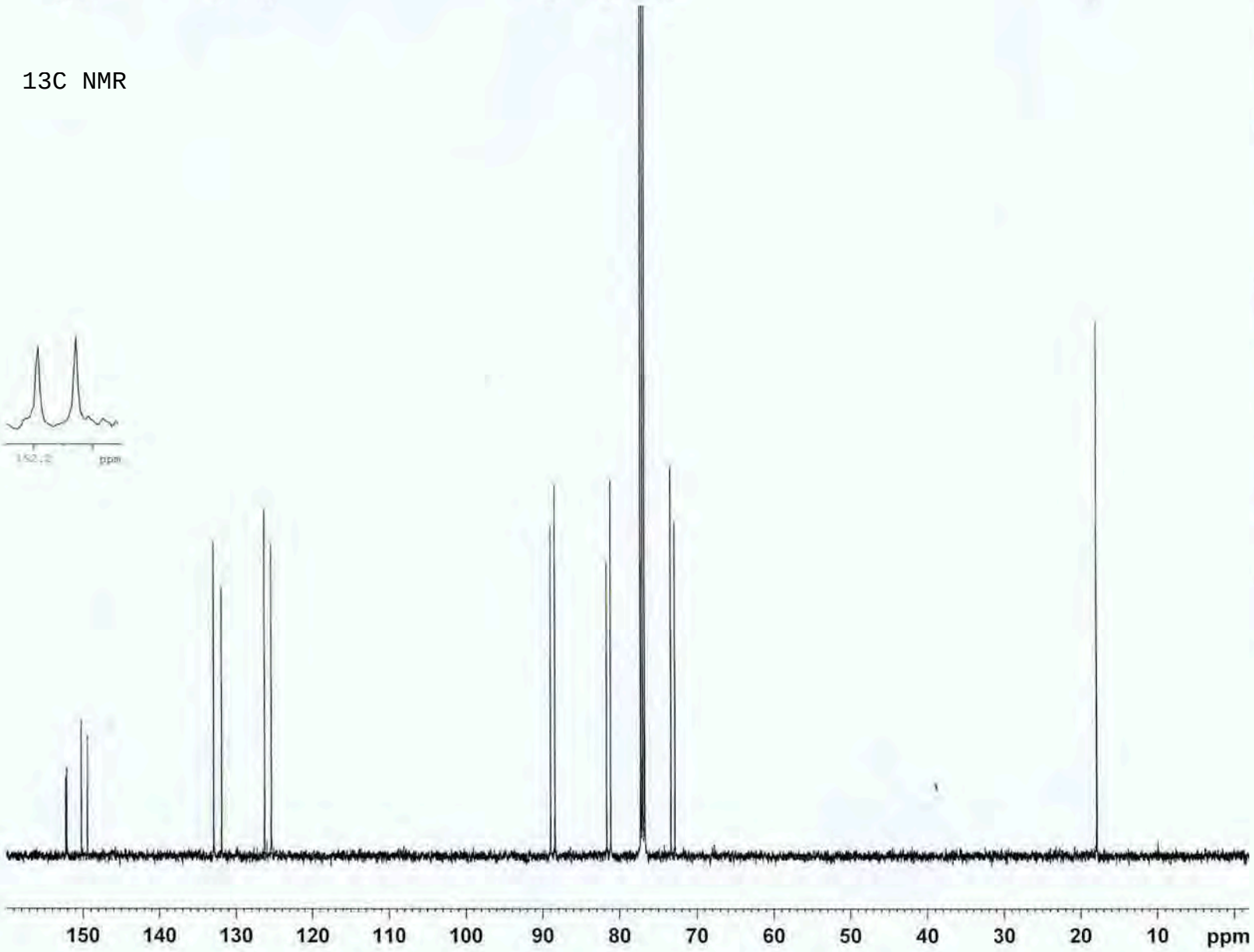


NAME	MB-64-1
EXPNO	1
PROCNO	1
Date_	20100903
Time	11.48
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4385.965 Hz
FIDRES	0.133849 Hz
AQ	3.7356019 sec
RG	114
DW	114.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
====== CHANNEL f1 ======	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618178 MHz
SI	32768
SF	500.2600079 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

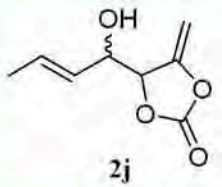


2j

13C NMR



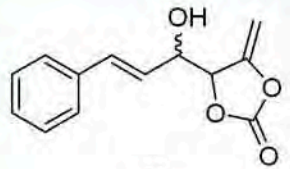
NAME	MB-64-1
EXPNO	2
PROCNO	1
Date_	20100903
Time	11.54
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	323
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	912
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618179 MHz
SI	32768
SF	125.7904842 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



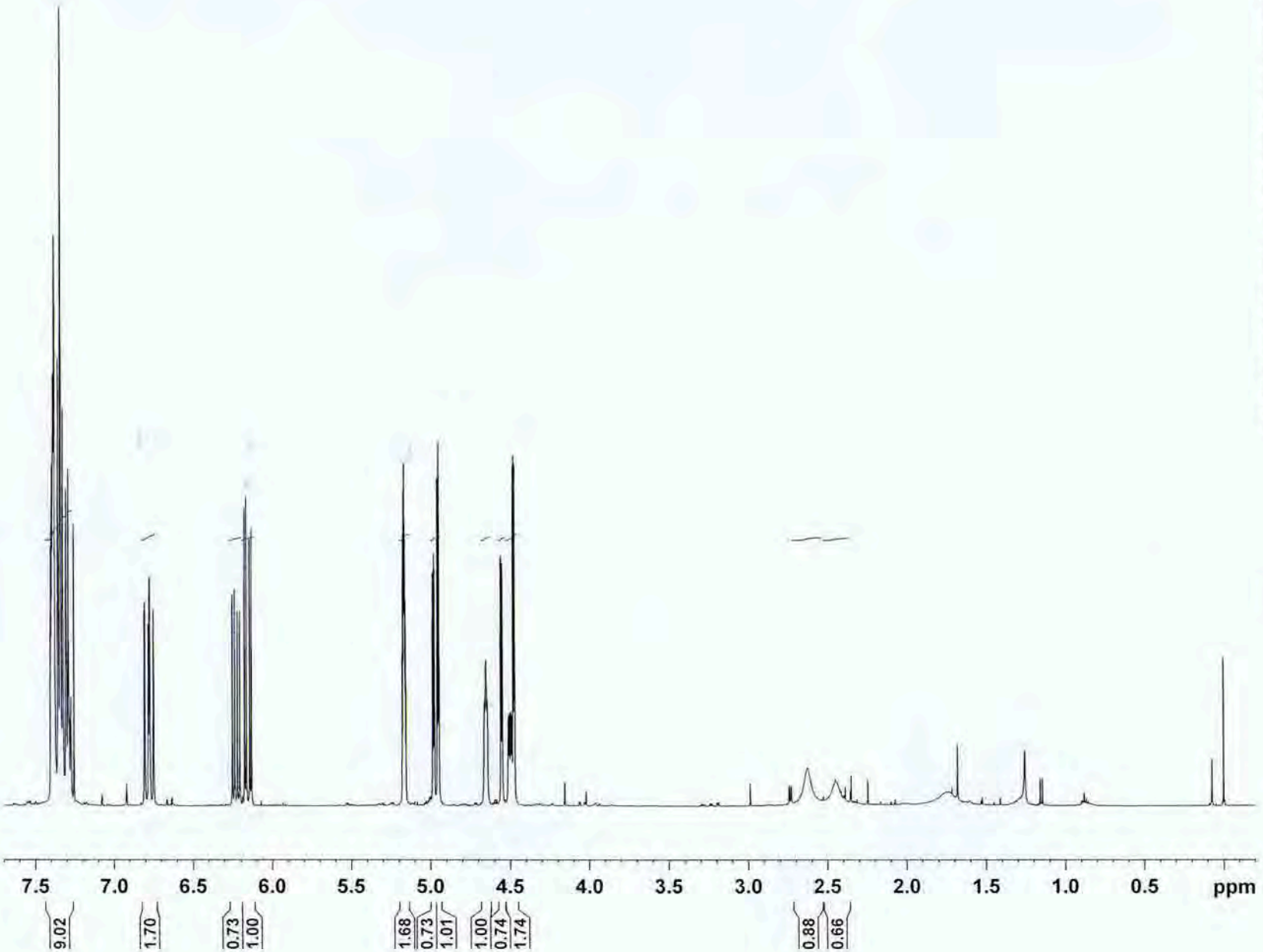
2j

1H NMR

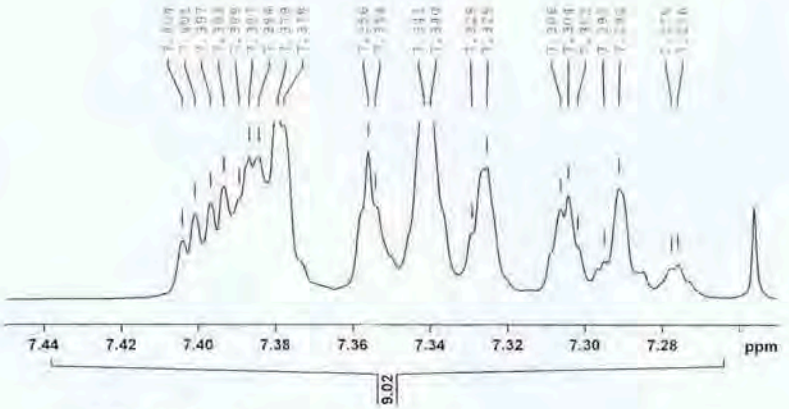
NAME	MB-62
EXPNO	1
PROCNO	1
Date_	20100903
Time	13.29
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4725.898 Hz
FIDRES	0.144223 Hz
AQ	3.4669044 sec
RG	114
DW	105.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
====== CHANNEL f1 ======	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618975 MHz
SI	32768
SF	500.2600165 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

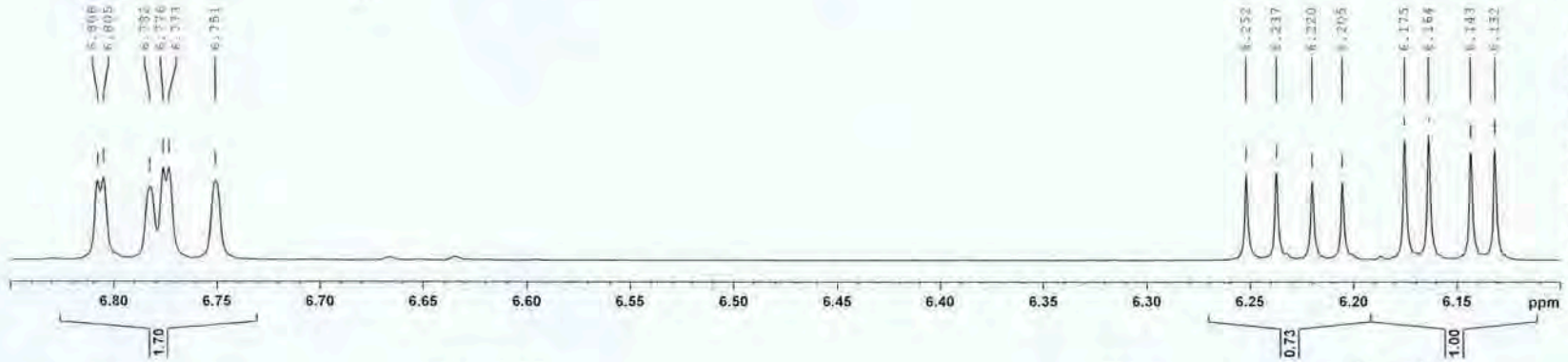


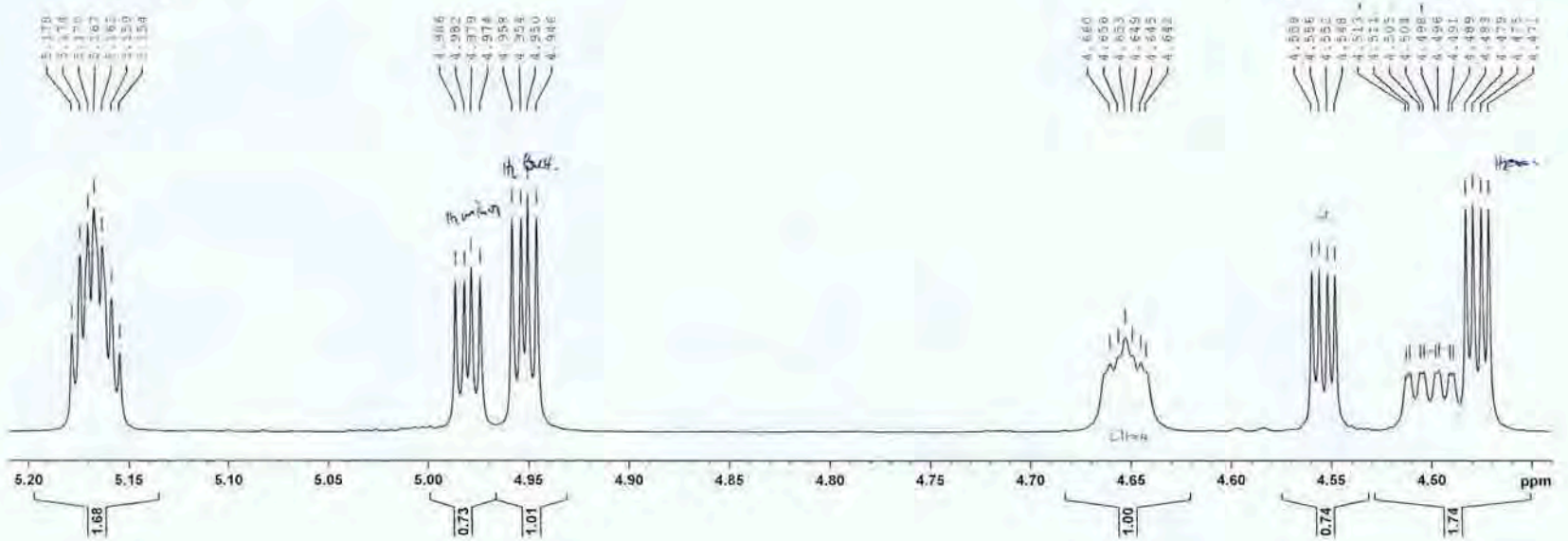
2k



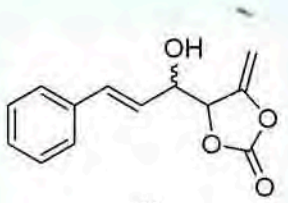
1H NMR



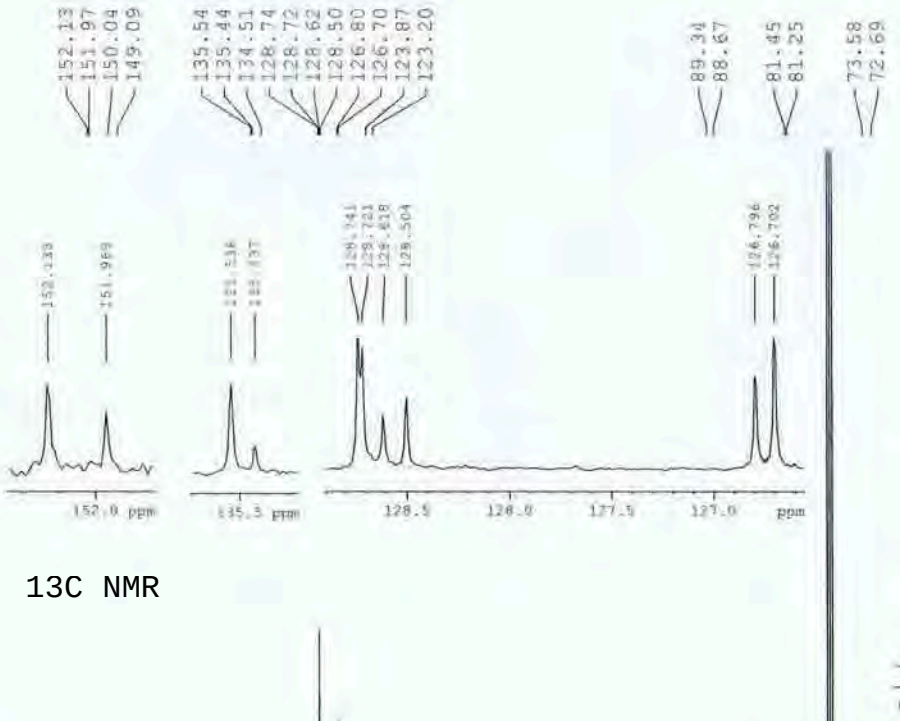




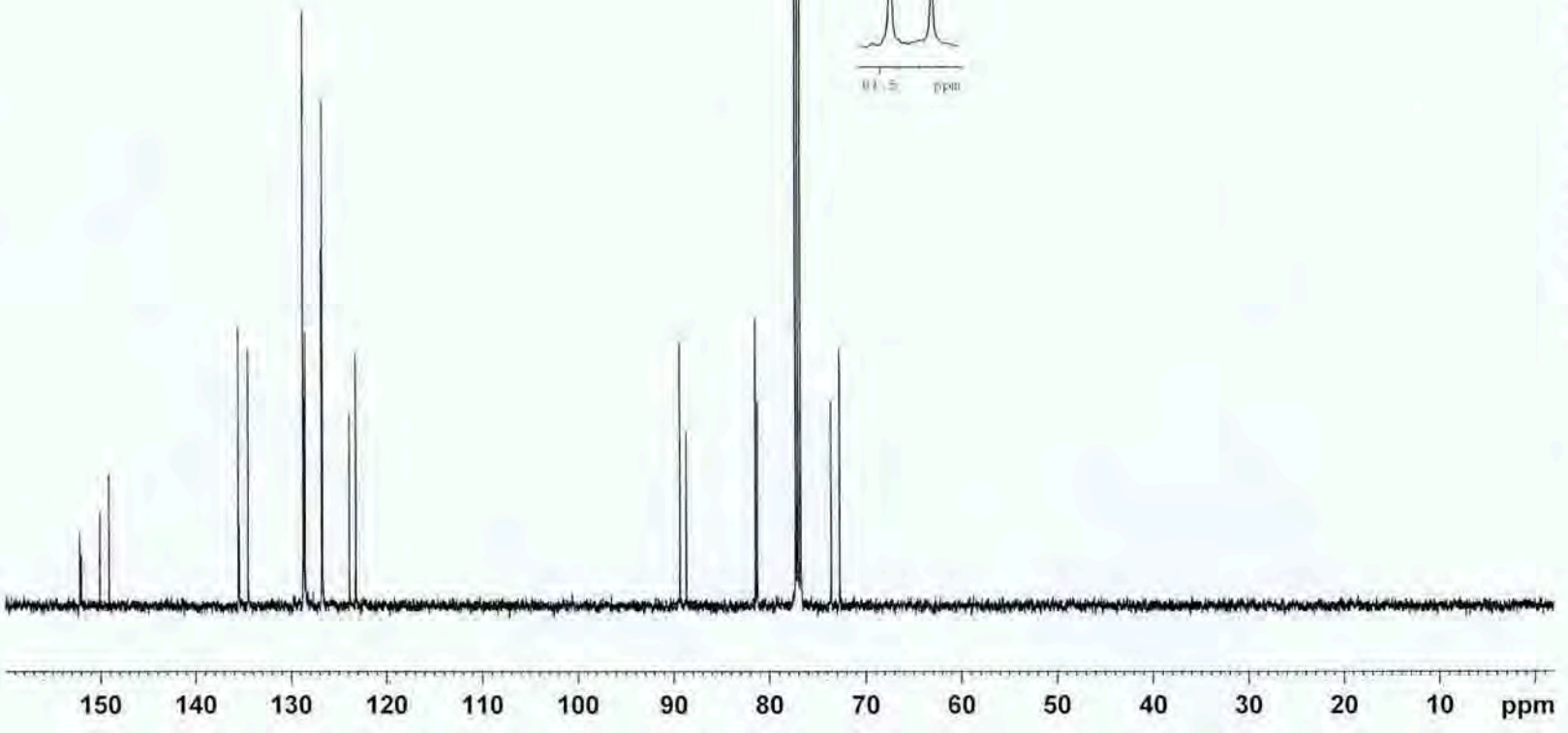

```
NAME            MB-62
EXPNO           1
PROCNO          1
Date_           20100903
Time            13.29
INSTRUM         spect
PROBHD     5 mm BBO BB-1H
PULPROG         zg30
TD              32768
SOLVENT         CDCl3
NS              16
DS              0
SWH             4725.898 Hz
FIDRES          0.144223 Hz
AQ              3.4669044 sec
RG              114
DW              105.800 usec
DE              6.50 usec
TE              298.0 K
D1              2.00000000 sec
TD0             1

======== CHANNEL f1 ========
NUC1            1H
P1              9.35 usec
PL1             0.00 dB
PL1W            27.37956238 W
SFO1            500.2618975 MHz
SI              32768
SF              500.2600165 MHz
WDW             EM
SSB             0
LB              0.20 Hz
GB              0
PC              1.00
```



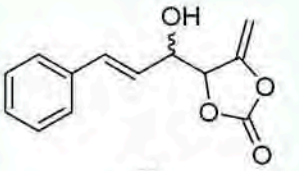
2k



13C NMR



NAME	MB-62
EXPNO	2
PROCNO	1
Date_	20100903
Time	13.37
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	332
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	812
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618975 MHz
SI	32768
SF	125.7904842 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

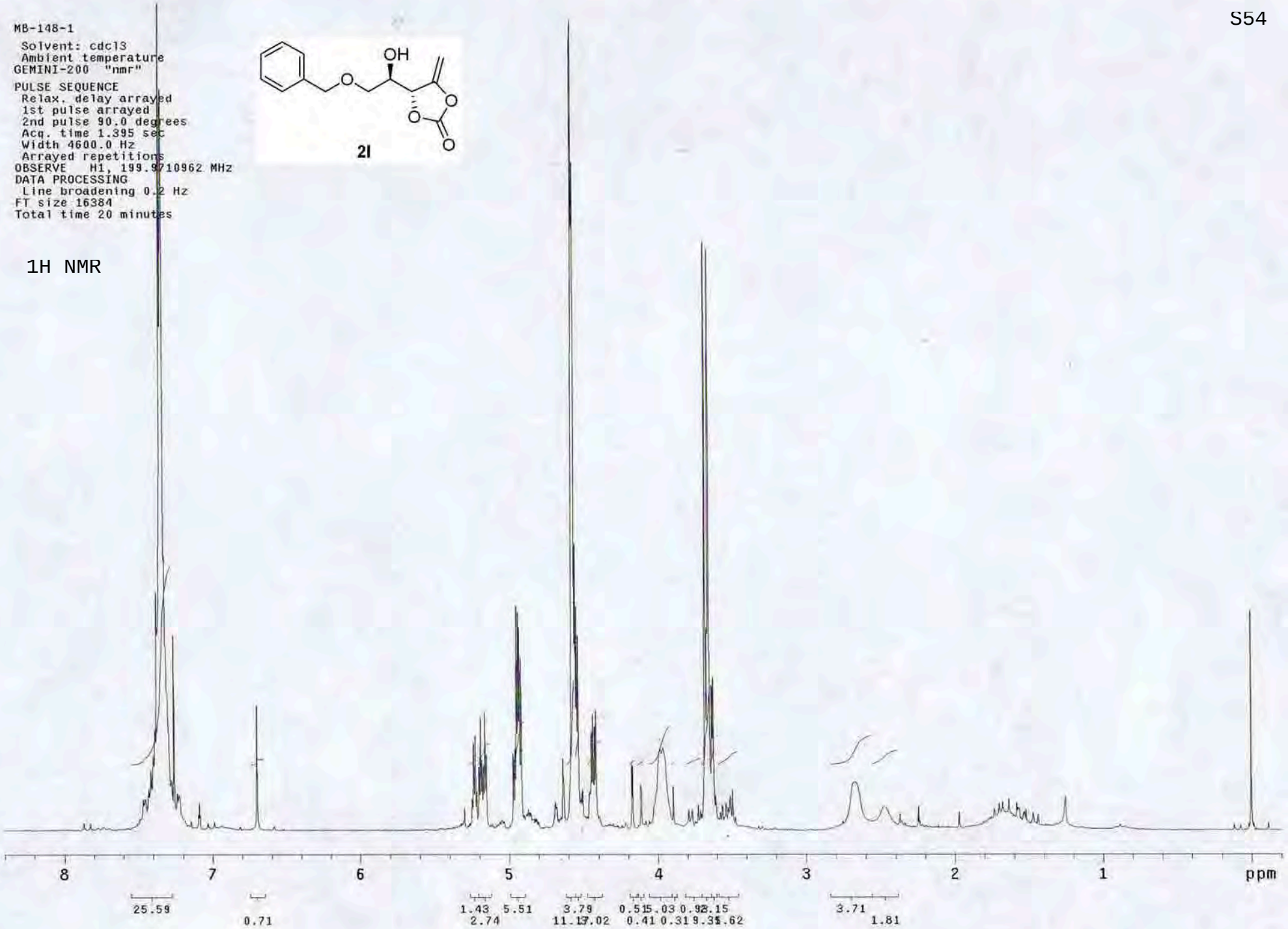


2k

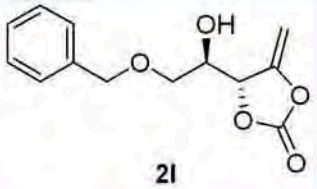
MB-148-1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.395 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710962 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 20 minutes

2l

1H NMR

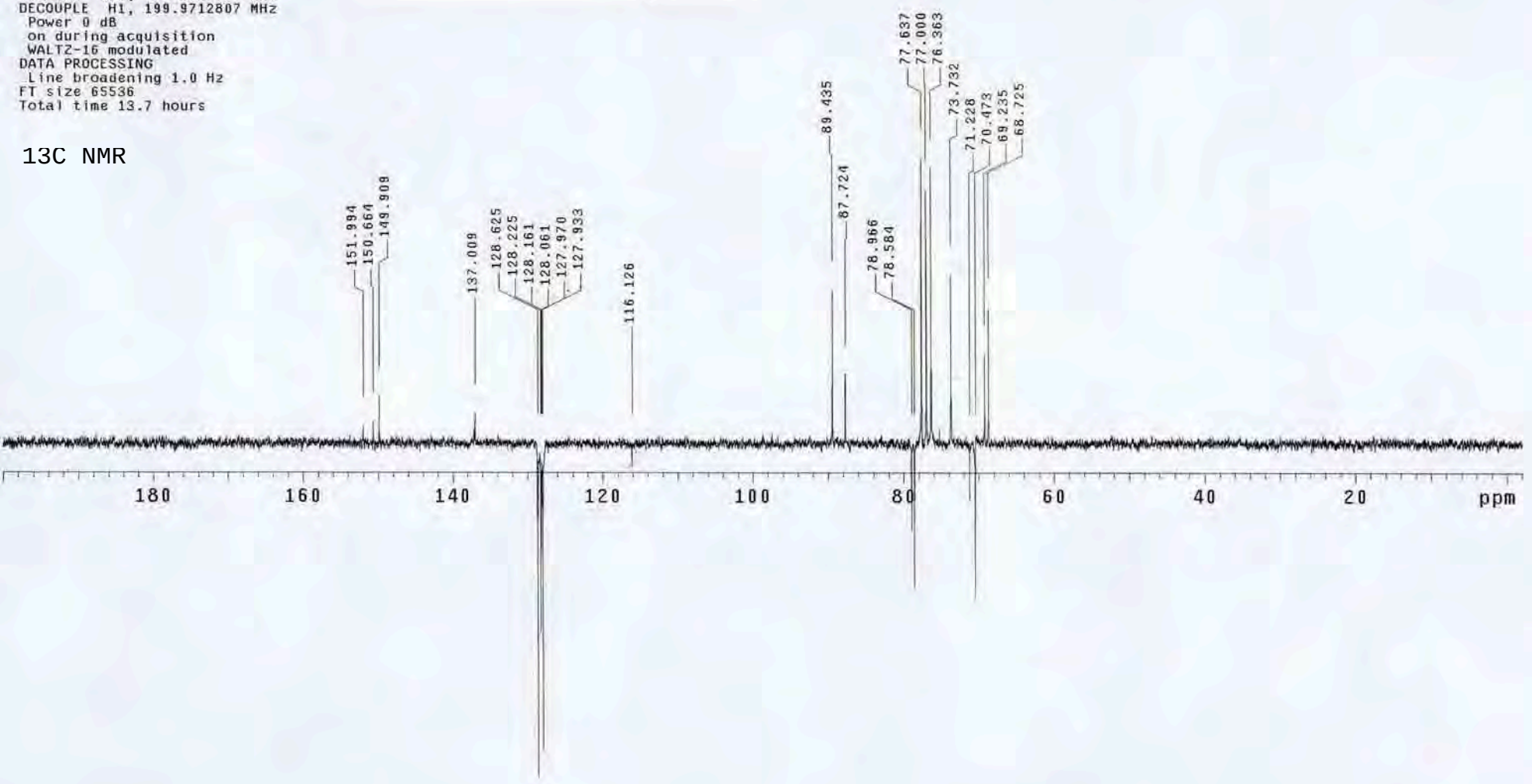


MB-148-1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827789 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 13.7 hours



2l

13C NMR



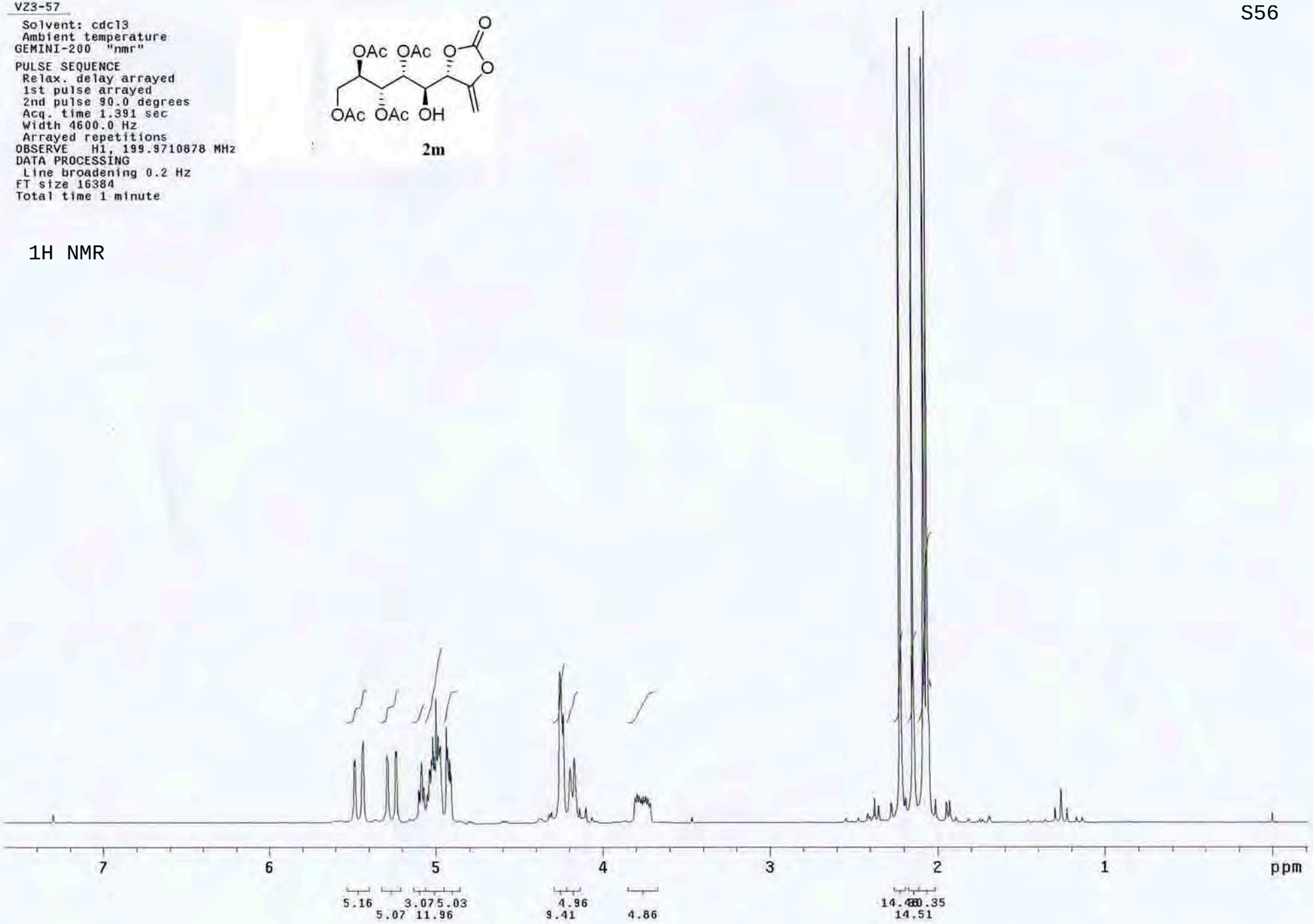
VZ3-57

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

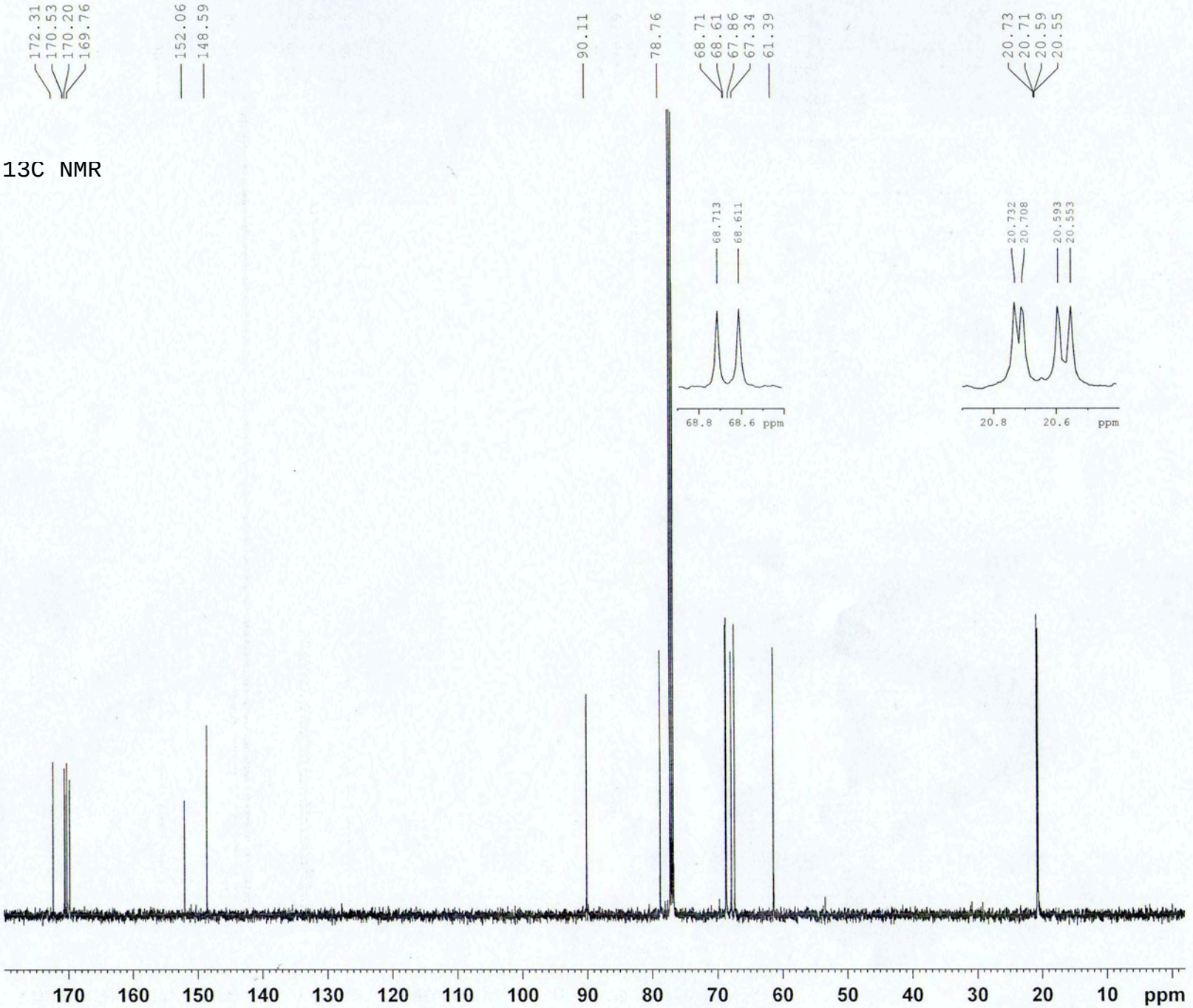
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710878 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute

2m

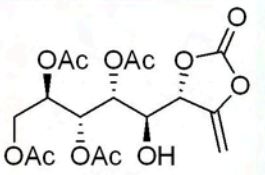
1H NMR



13C NMR

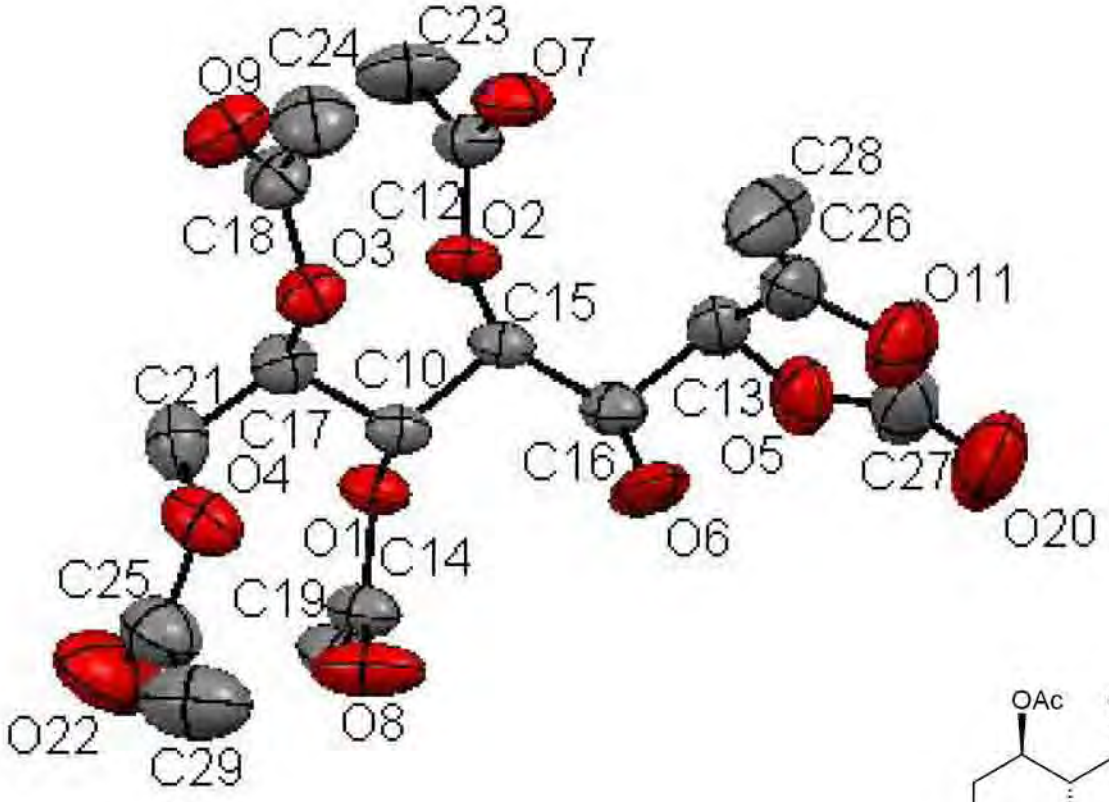


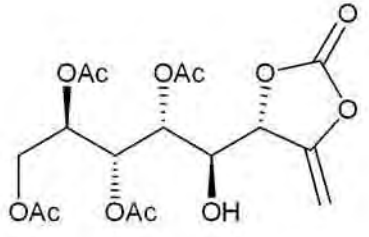
NAME	VZ3-57
EXPNO	2
PROCNO	1
Date_	20110801
Time	11.26
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	167
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618950 MHz
SI	32768
SF	125.7904840 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



2m

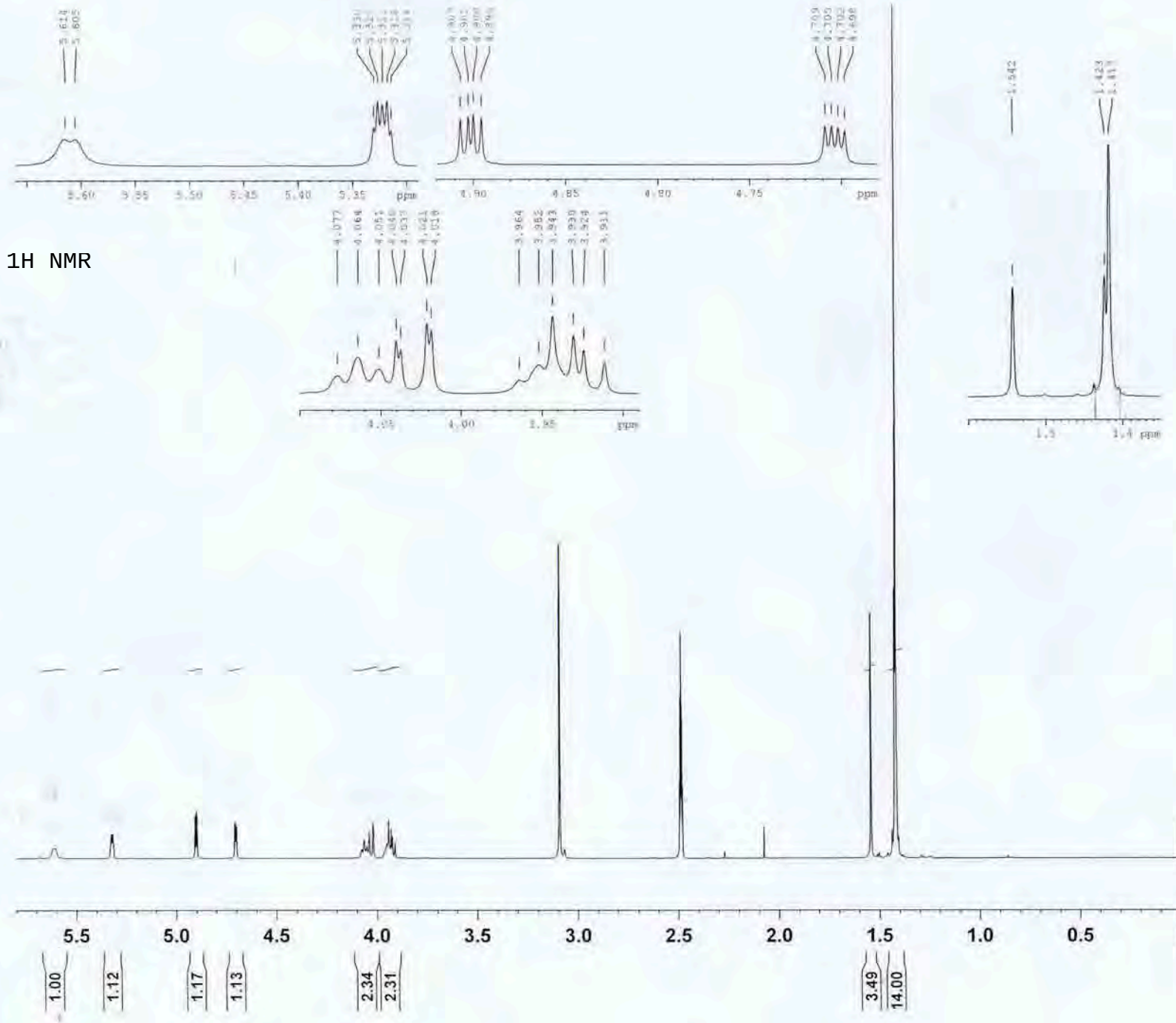
ORTEP diagram for 2m



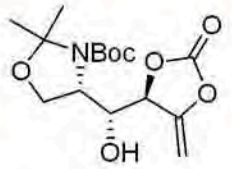


2m

1H NMR

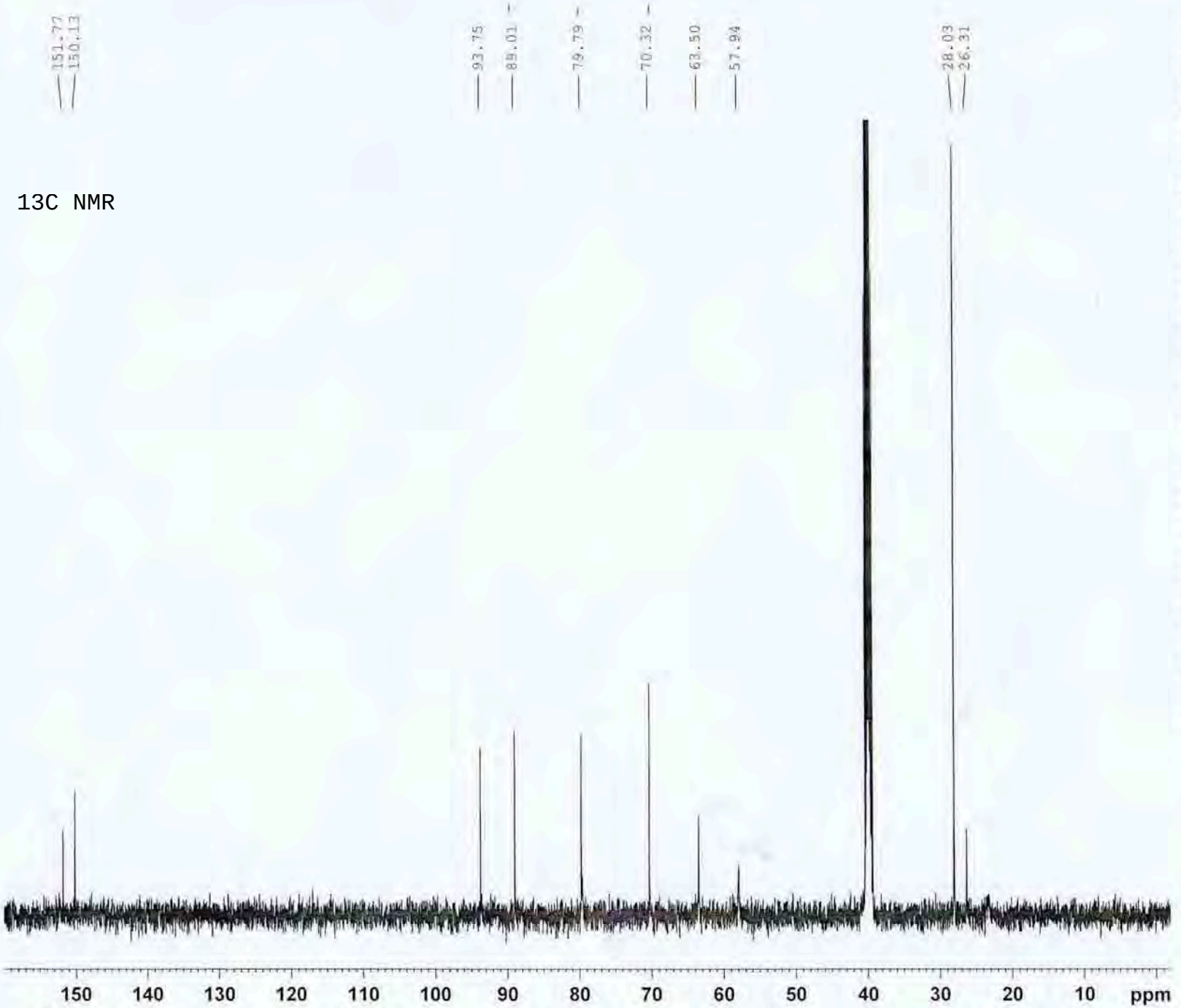



```
NAME                MB-162
EXPNO                  997
PROCNO                   1
Date_            20110426
Time                 12.45
INSTRUM               spect
PROBHD  5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT                DMSO
NS                       16
DS                        0
SWH              3605.769 Hz
FIDRES           0.110039 Hz
AQ               4.5438795 sec
RG                      203
DW               138.667 usec
DE                  6.50 usec
TE                 340.1 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  9.35 usec
PL1                 0.00 dB
PL1W        27.37956238 W
SFO1        500.2615068 MHz
SI                   32768
SF          500.2600132 MHz
WDW                    EM
SSB                     0
LB                 0.20 Hz
GB                      0
PC                   1.00
```

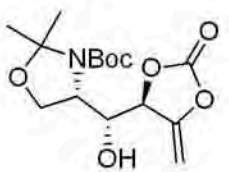


2n (*S,R,R*)

13C NMR

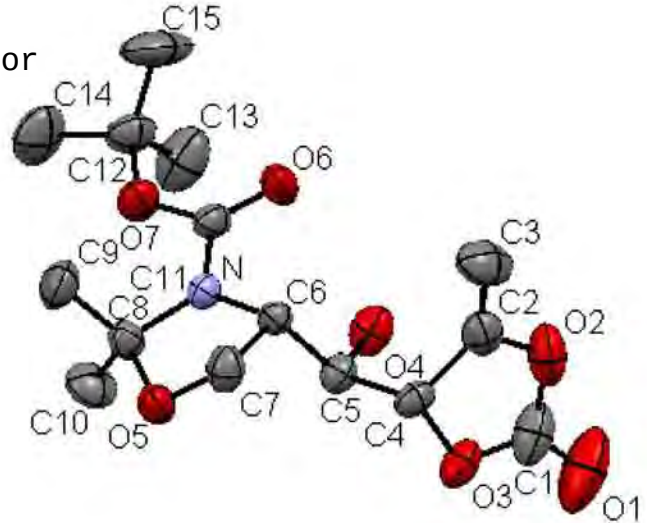


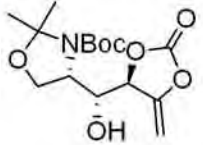
NAME	MB-162
EXPNO	2
PROCNO	1
Date_	20110426
Time	12.07
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	DMSO
NS	803
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	339.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2615068 MHz
SI	32768
SF	125.7905404 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



2n (*S*,*R*,*R*)

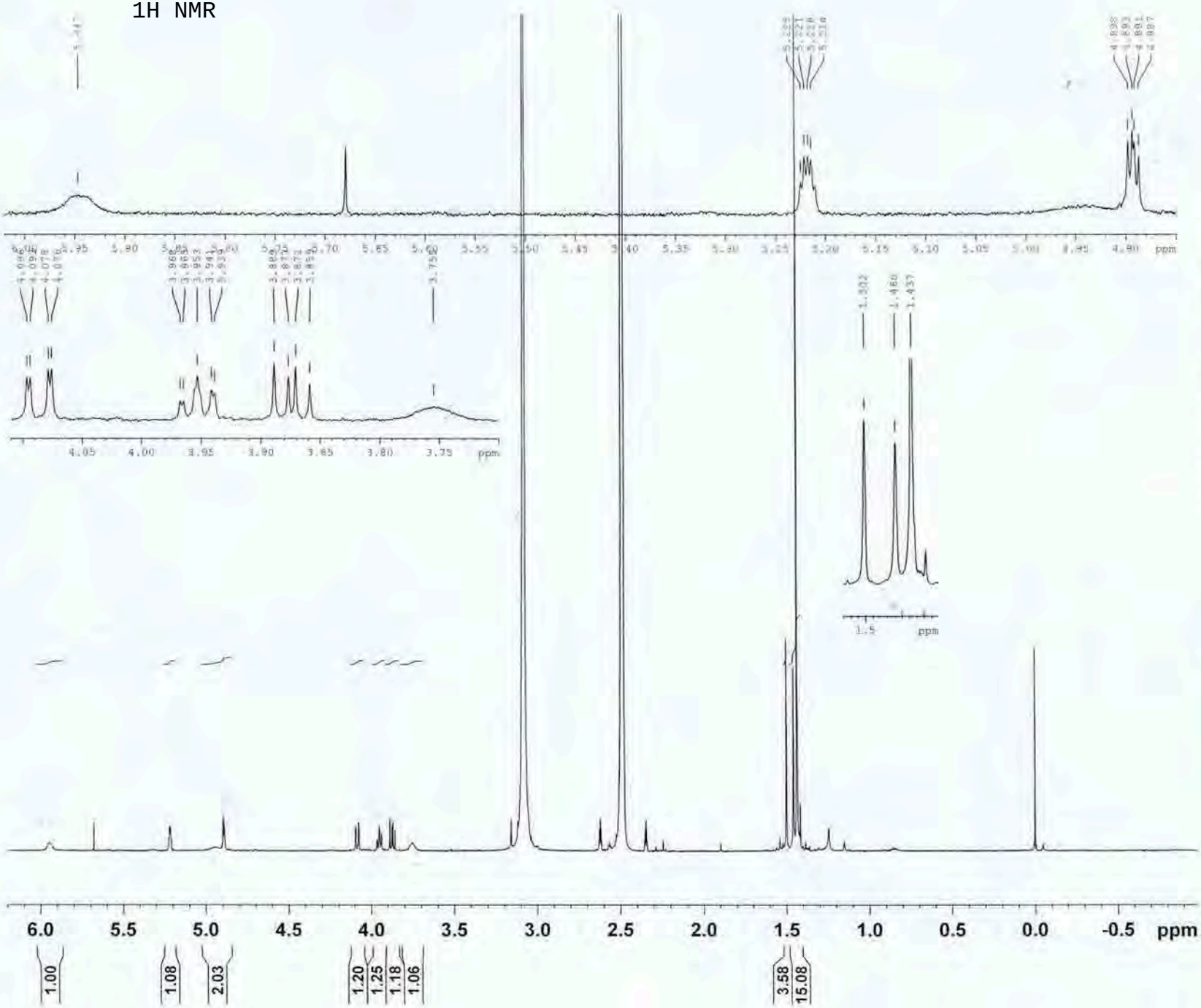
ORTEP diagram for
2n (*S,R,R*)



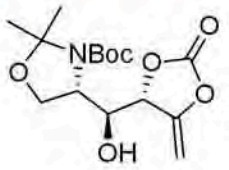


2n (*S.R.R*)

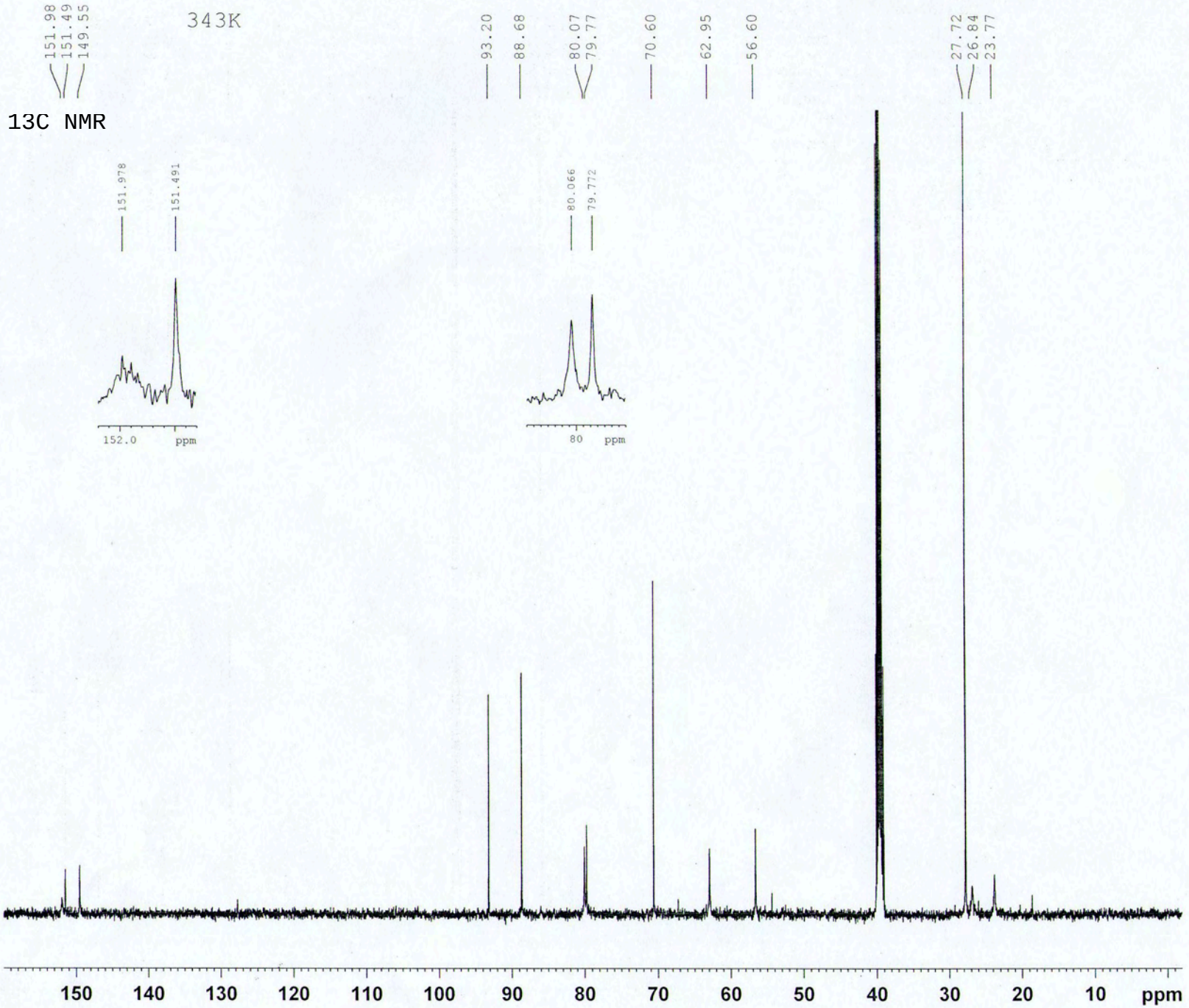
1H NMR



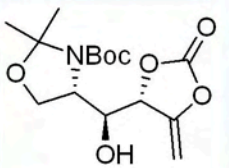
NAME	ME-02-162
EXPNO	2
PROCNO	1
Date_	20110614
Time	13.14
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	DMSO
NS	128
DS	0
SWH	4091.653 Hz
FIDRES	0.124867 Hz
AQ	4.0042996 sec
RG	322
DW	122.200 usec
DE	6.50 usec
TE	343.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2615703 MHz
SI	32768
SF	500.2600135 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



2n (*S*,*S*,*S*)

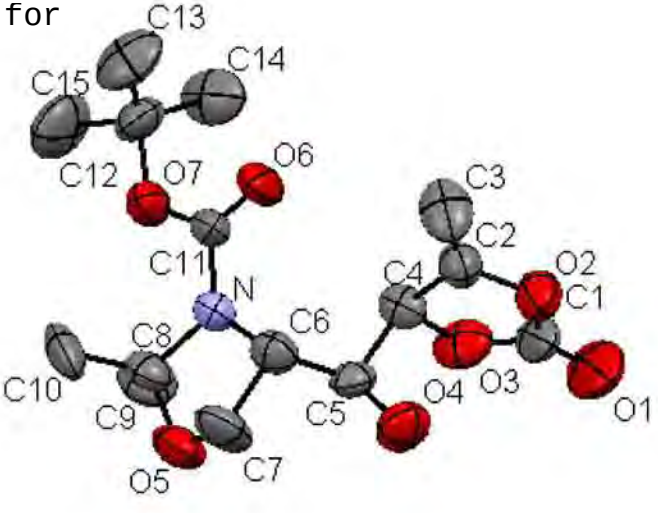


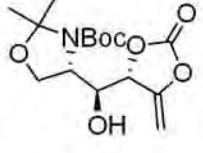
NAME	MB-162-d21
EXPNO	3
PROCNO	1
Date_	20110729
Time	11.51
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	DMSO
NS	1013
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	2050
DW	16.800 usec
DE	6.50 usec
TE	343.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2621251 MHz
SI	32768
SF	125.7905779 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



2n (*S*,*S*,*S*)

ORTEP diagram for
2n (*S,S,S*)



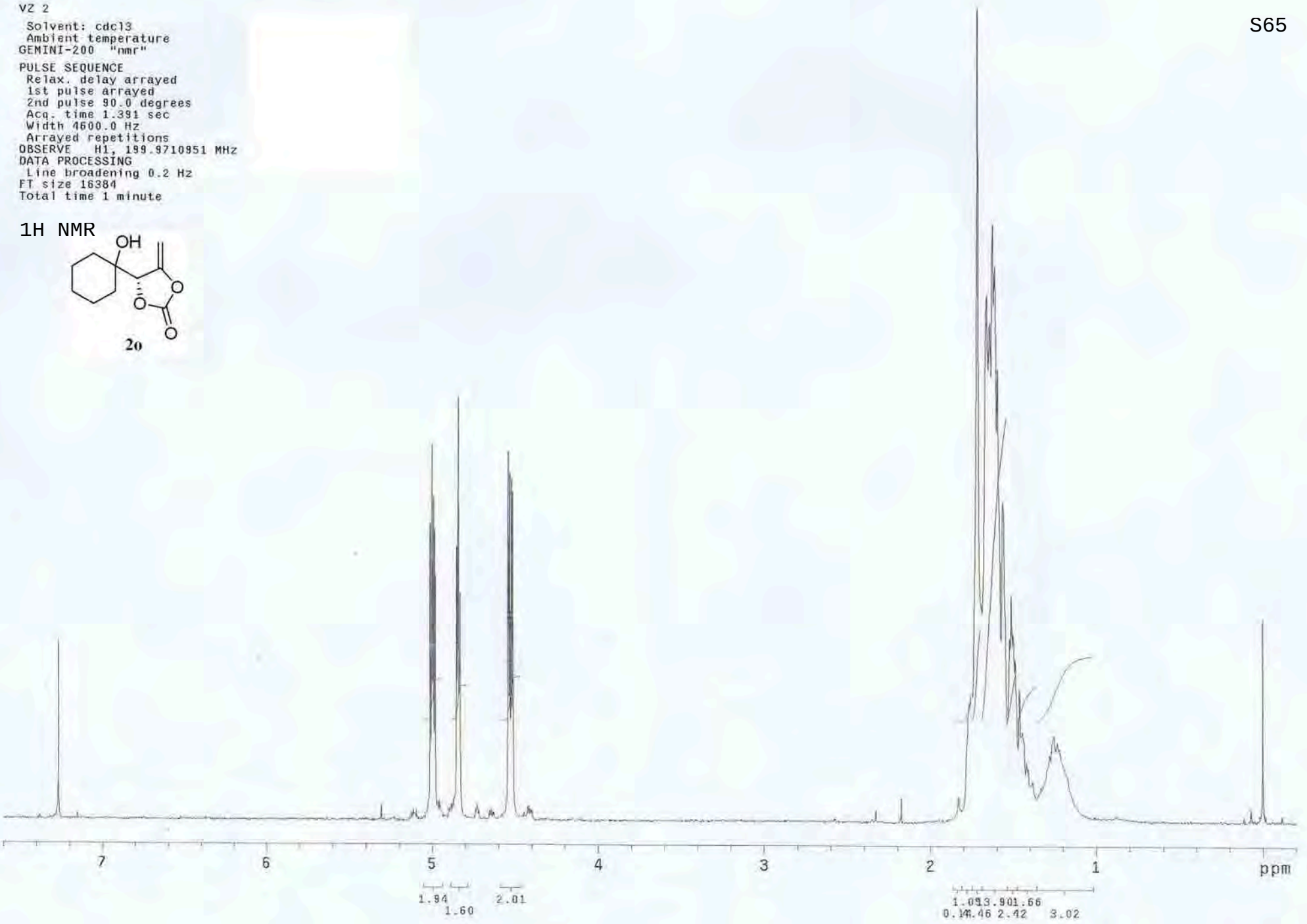


2n (*S.S.S*)

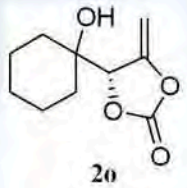
VZ 2
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710951 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute

1H NMR

2o

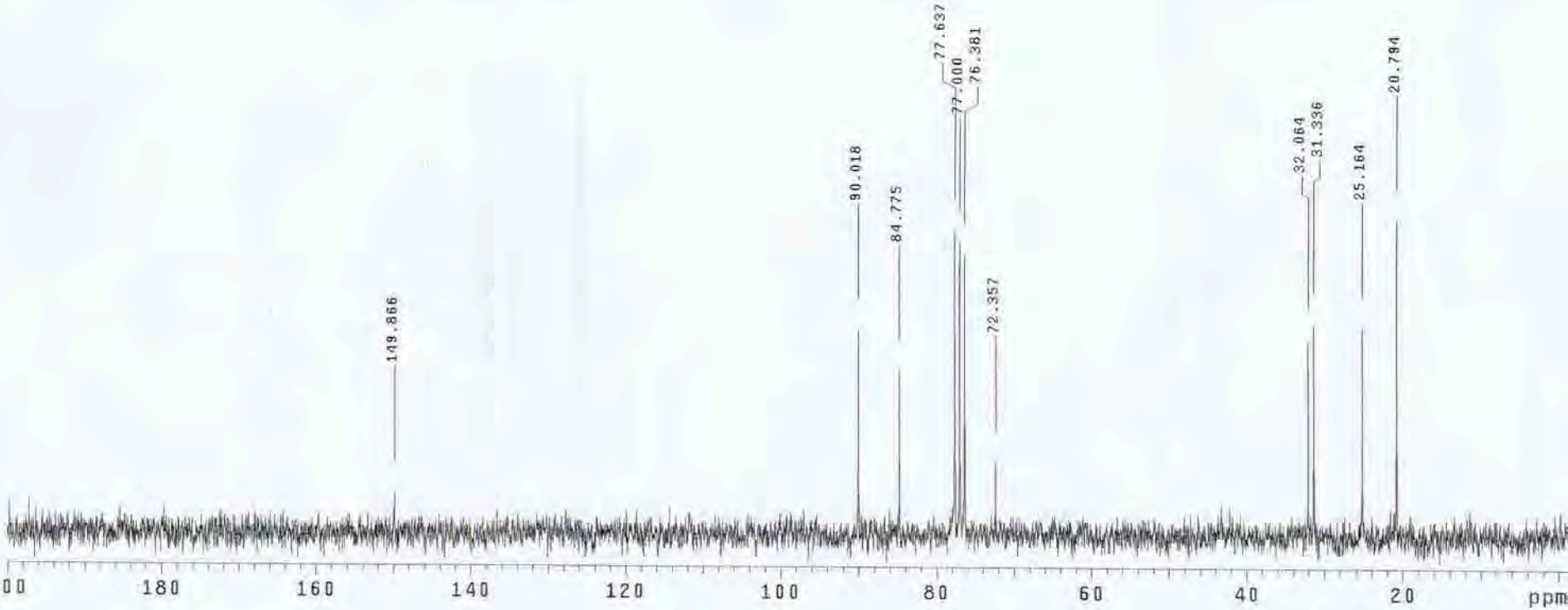


VZ 2
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827782 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 20 minutes



2o

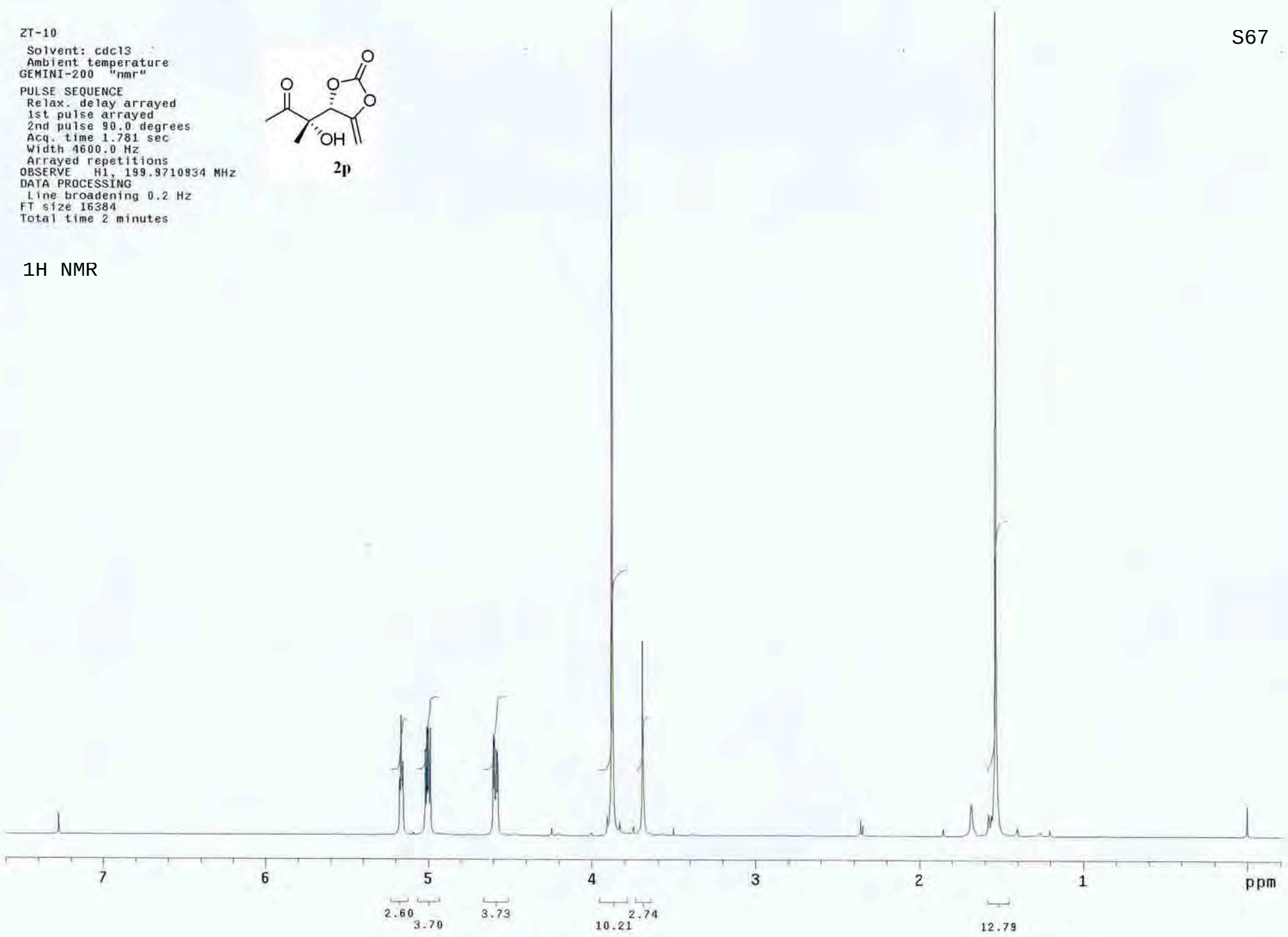
13C NMR



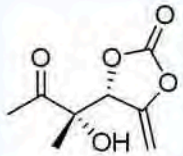
ZT-10
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.781 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710934 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 2 minutes

2p

1H NMR

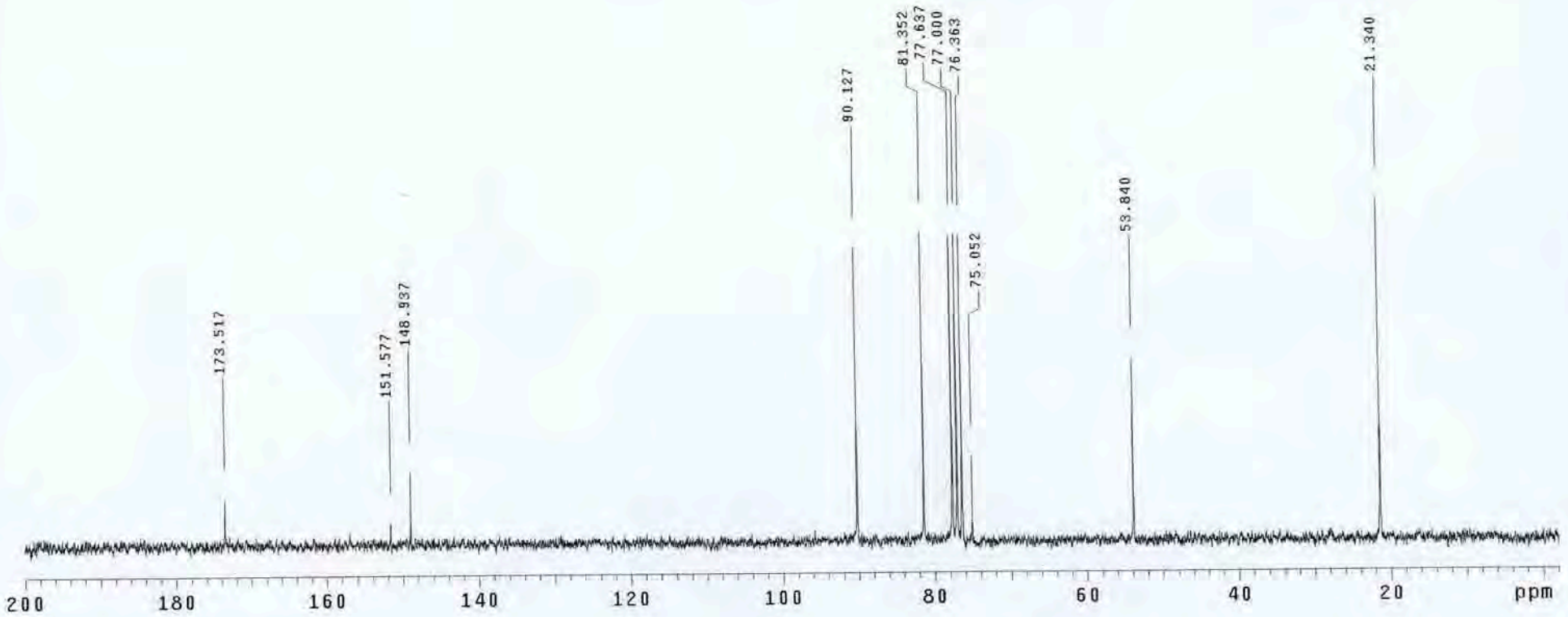


ZT-10a
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827782 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 2.5 hours



2p

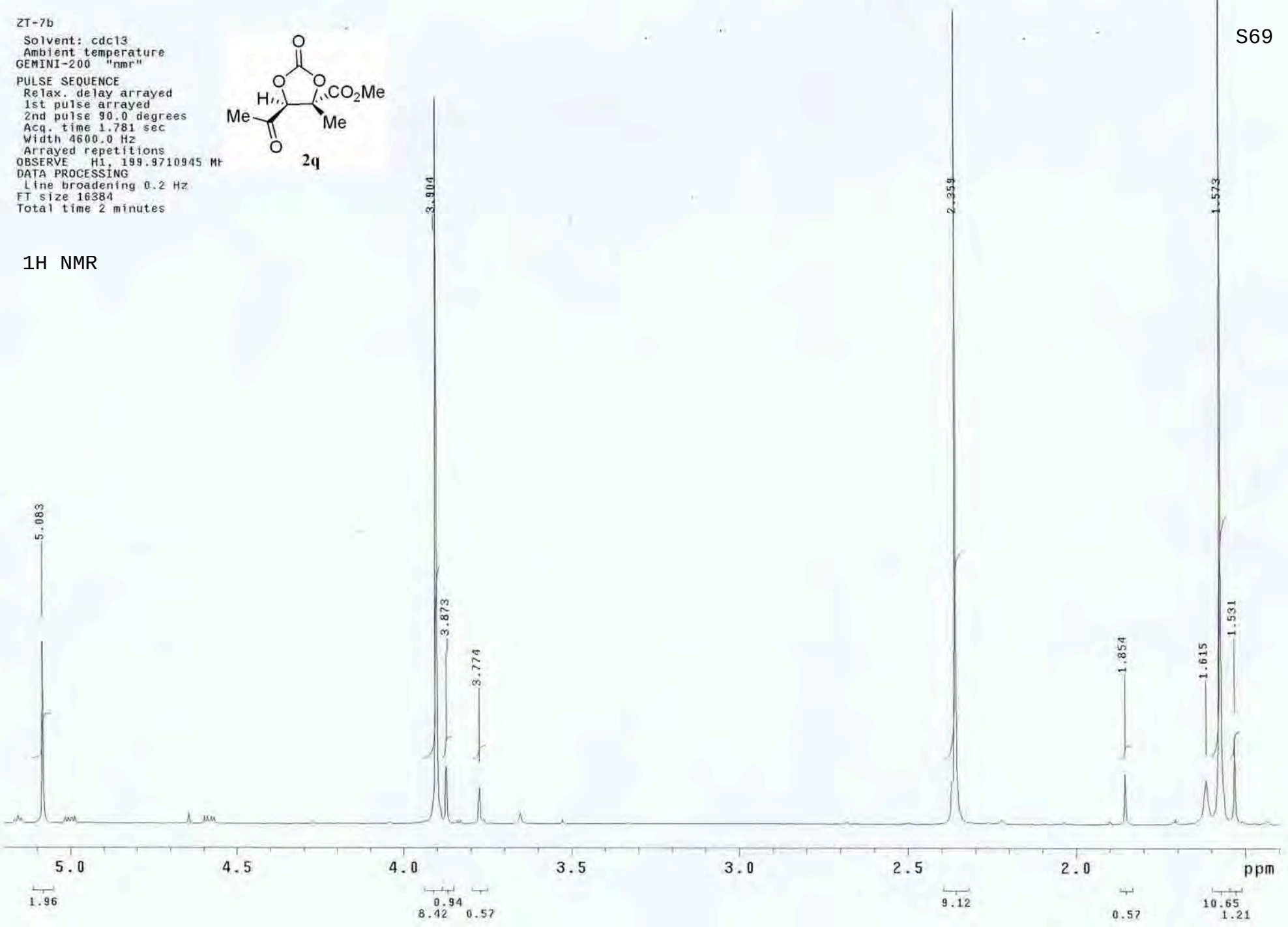
13C NMR



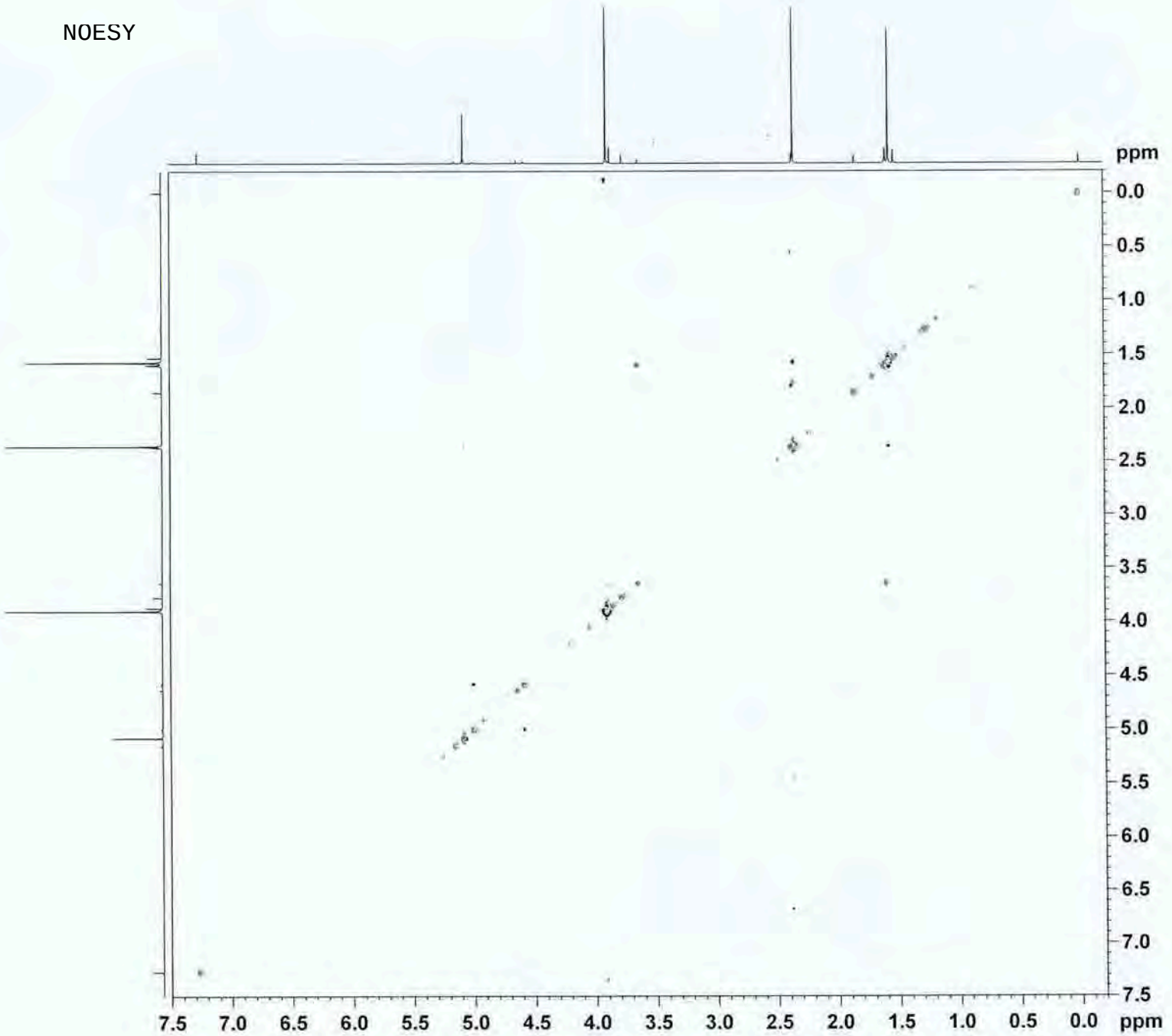
ZT-7b
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.781 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710945 MH
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 2 minutes

2q

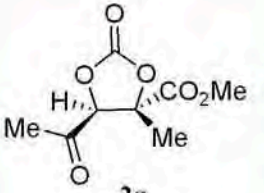
1H NMR



NOESY

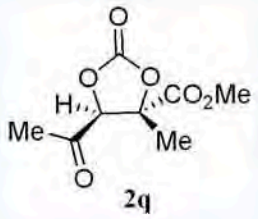


NAME	ET-7b
EXPNO	2
PROCNO	1
Date_	20100812
Time	12.02
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesyph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4325.259 Hz
FIDRES	4.223886 Hz
AQ	0.1184244 sec
RG	144
DW	115.600 usec
DE	6.50 usec
TE	298.0 K
D0	0.00010370 sec
D1	2.00000000 sec
D8	1.00000000 sec
IN0	0.00023120 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618139 MHz
ND0	1
TD	256
SFO1	500.2618 MHz
FIDRES	16.895561 Hz
SW	8.646 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600062 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600064 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0



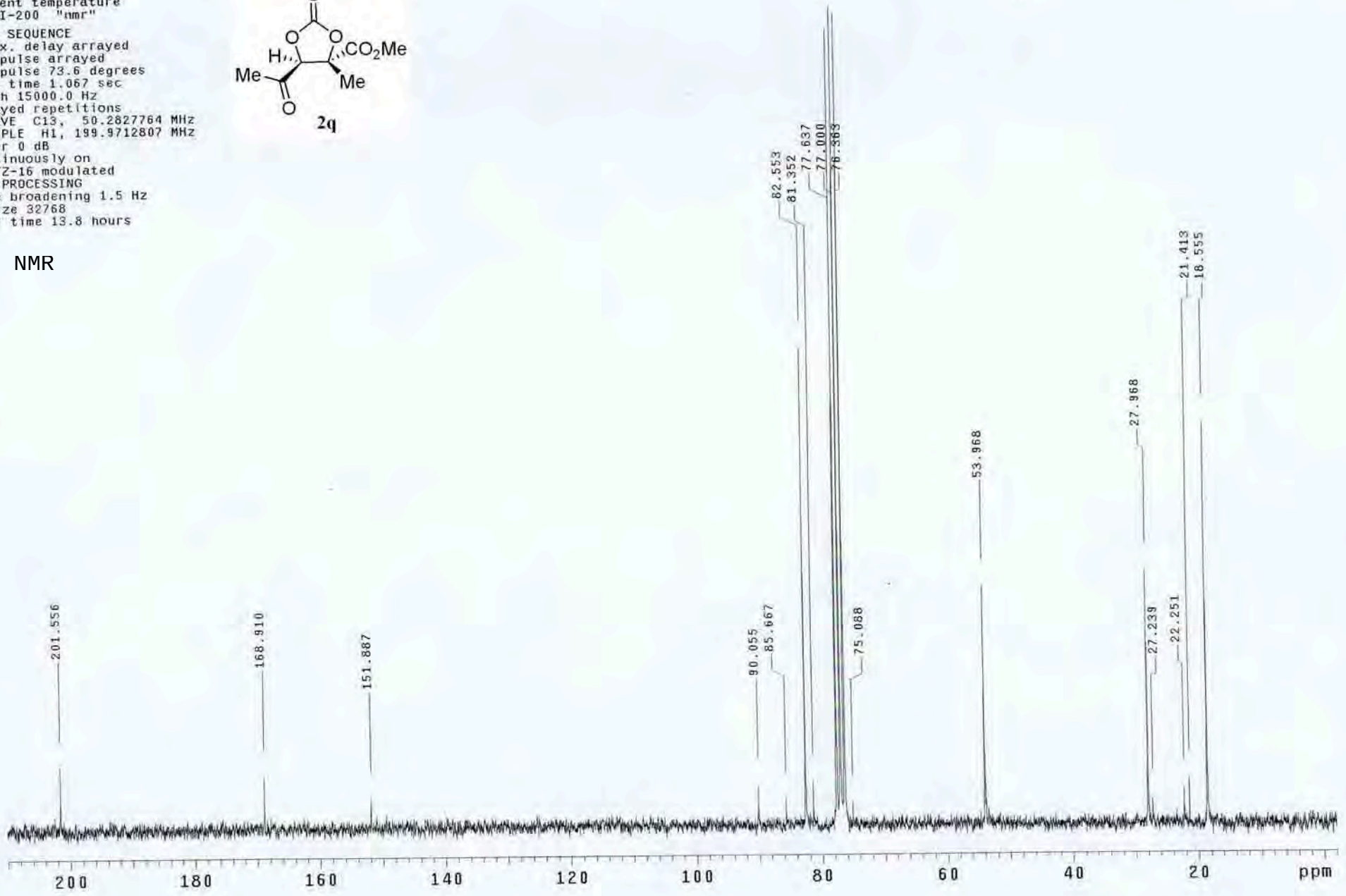
2q

ZT-7b
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827764 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 13.8 hours



2q

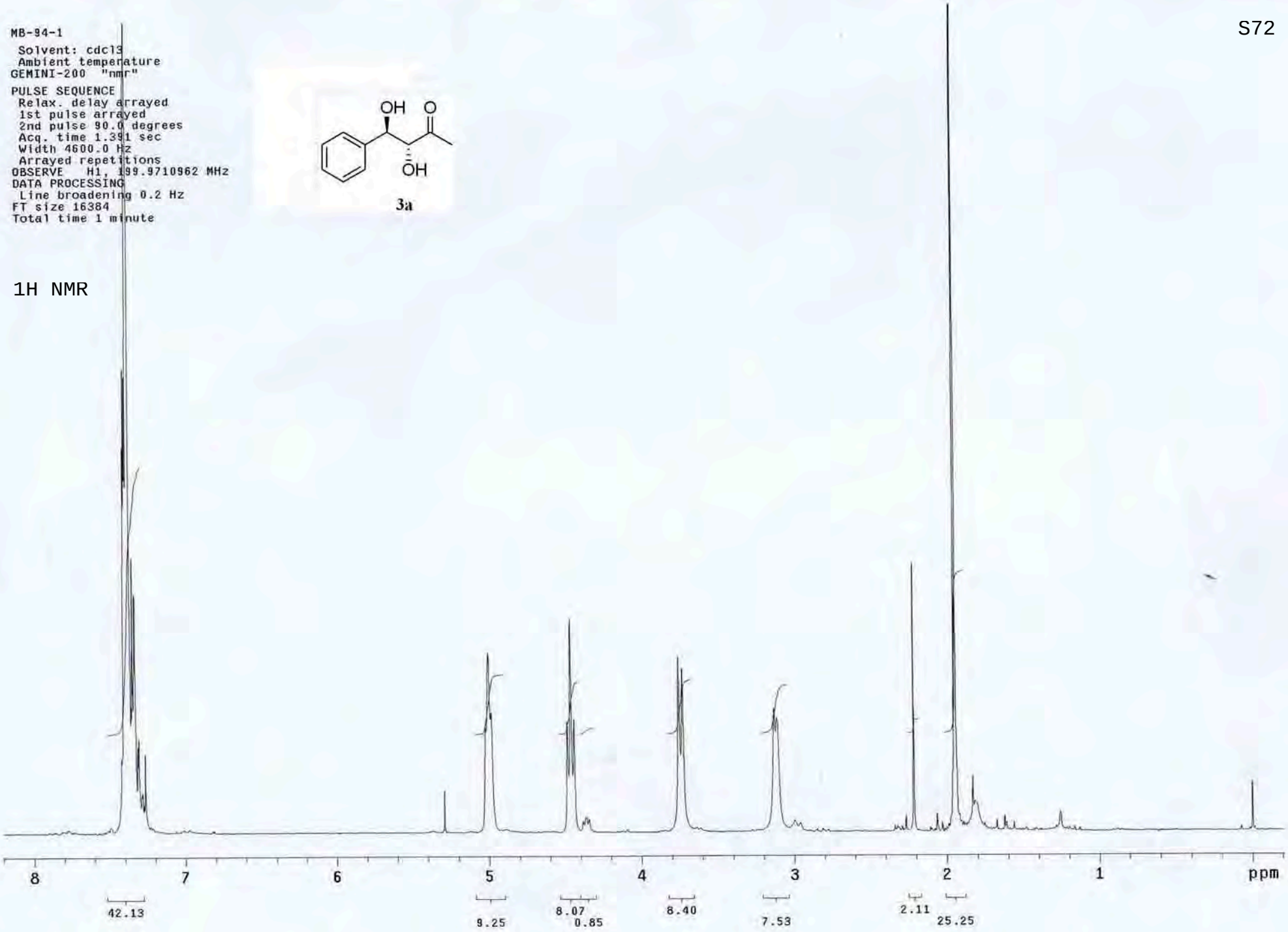
13C NMR



MB-94-1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710962 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute

3a

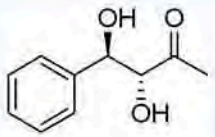
1H NMR



MB-94

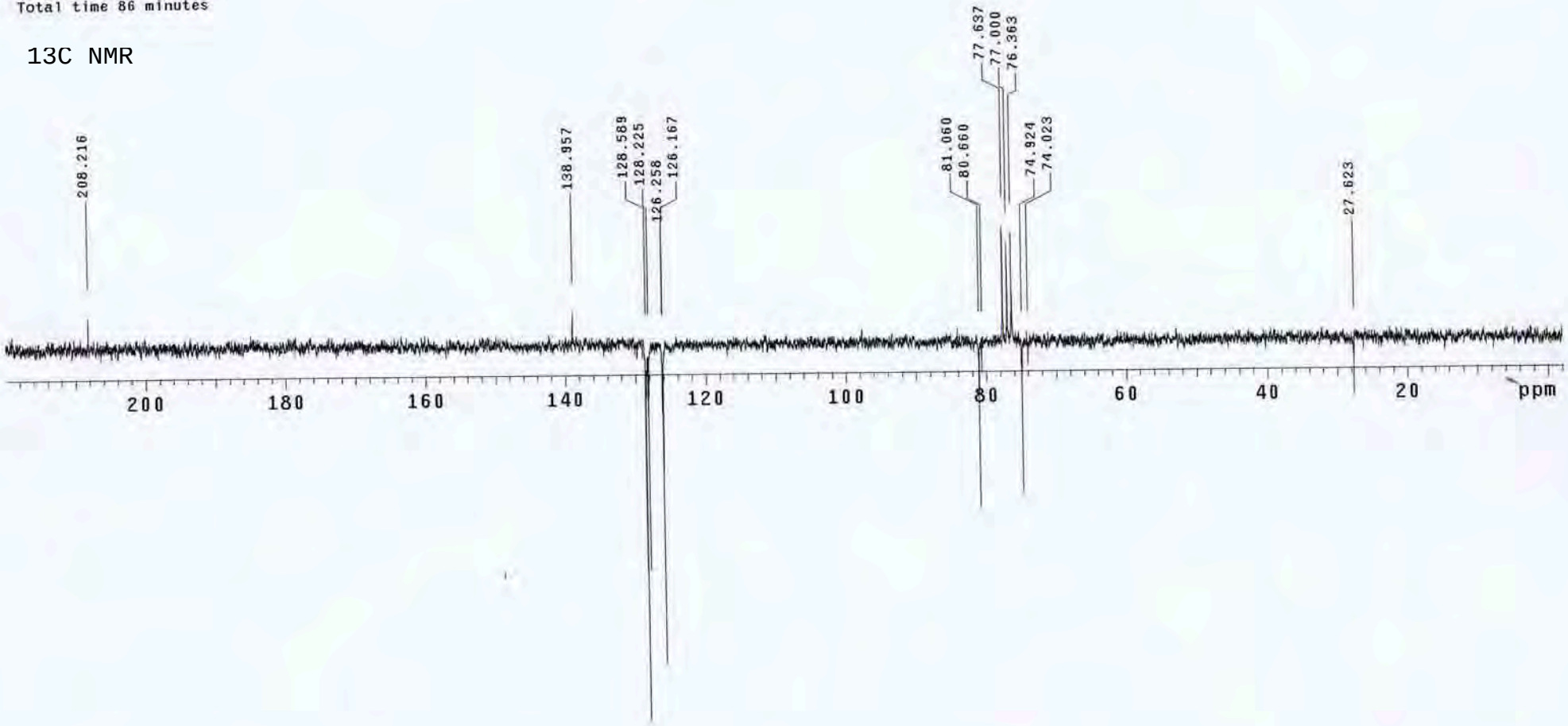
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827794 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 86 minutes



3a

13C NMR

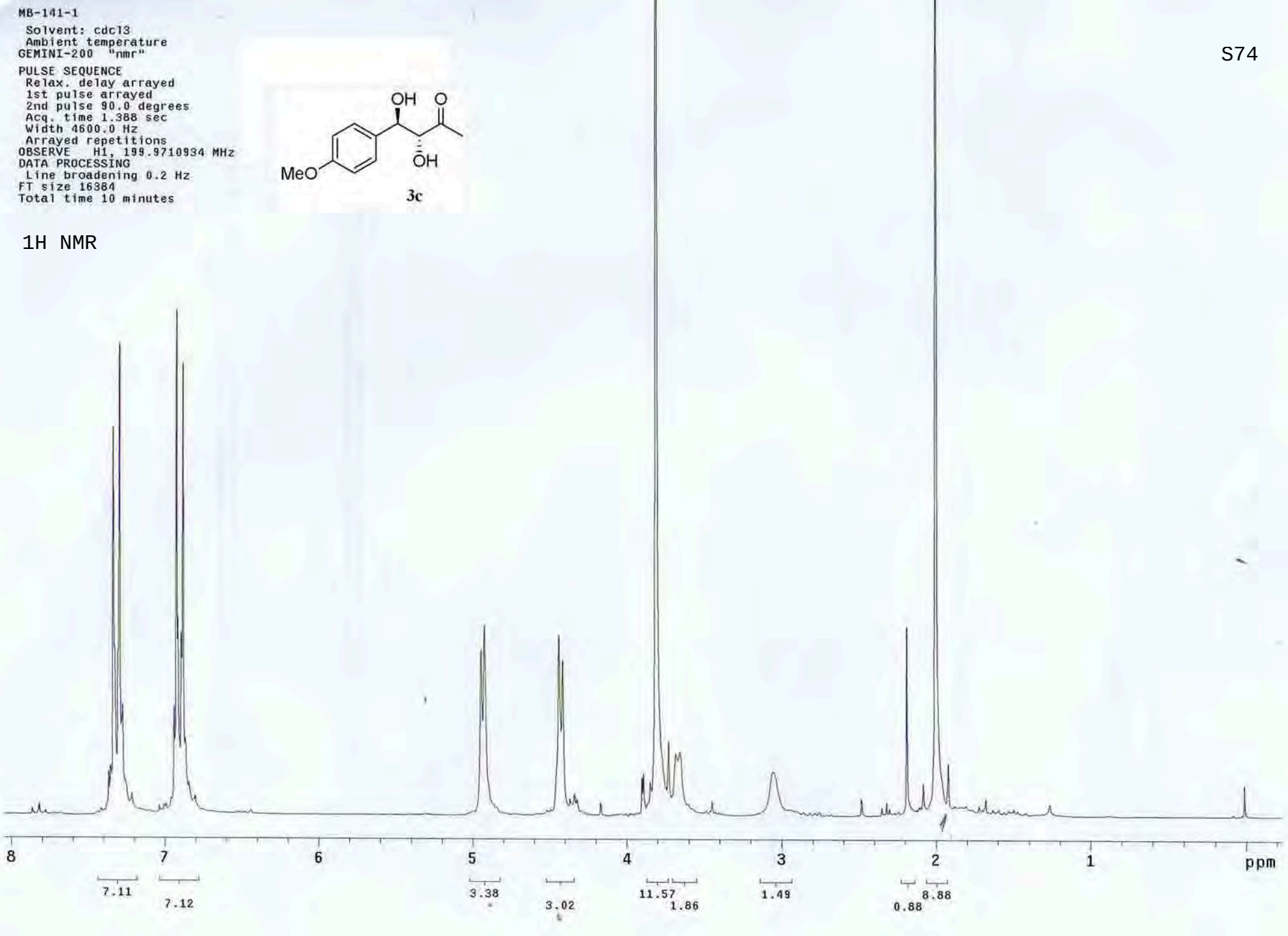


MB-141-1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.388 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710934 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 10 minutes

3c

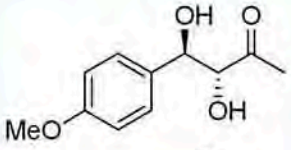
1H NMR



MB-141-1

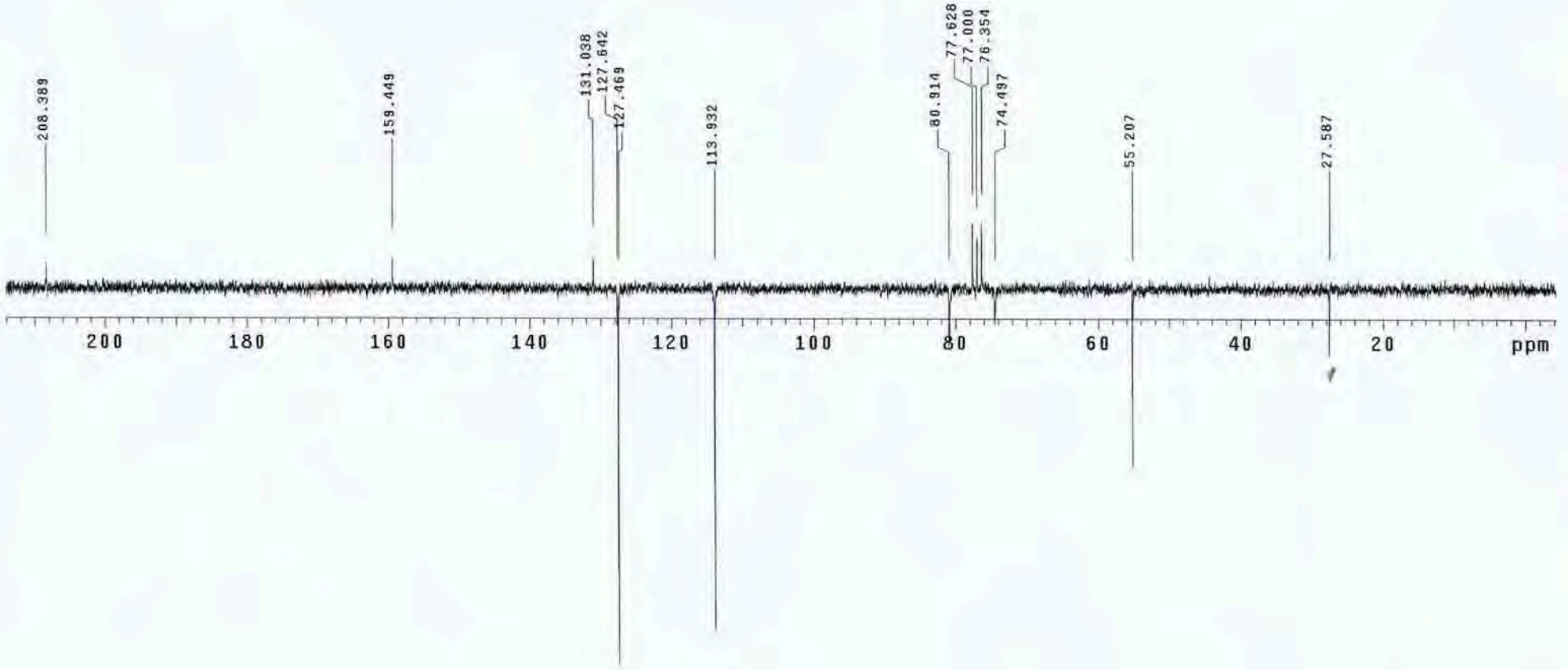
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827798 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 49 minutes



3c

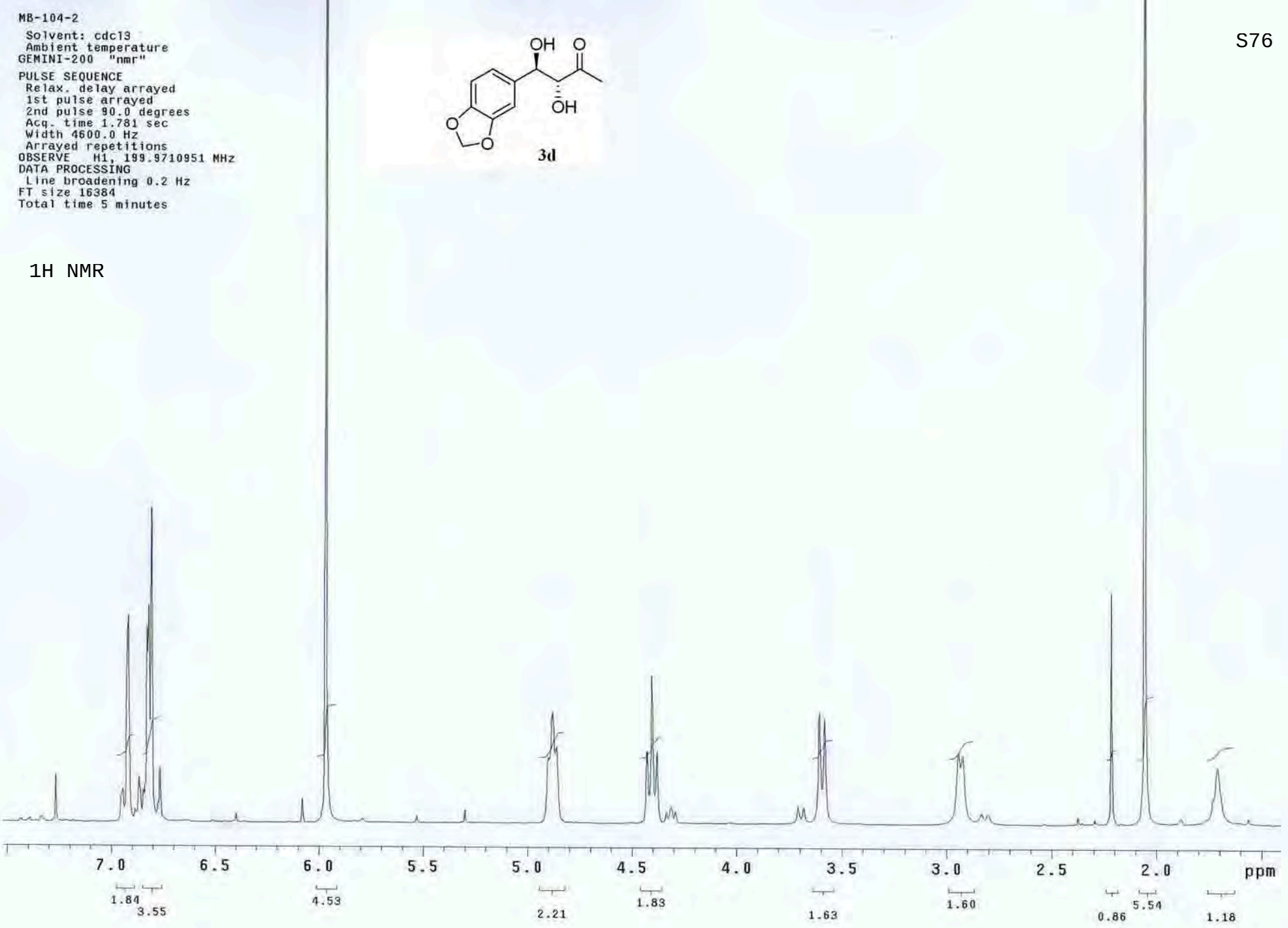
13C NMR



MB-104-2
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.781 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710951 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 5 minutes

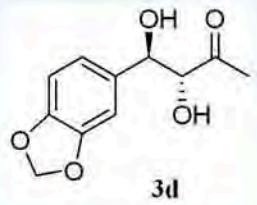
3d

1H NMR



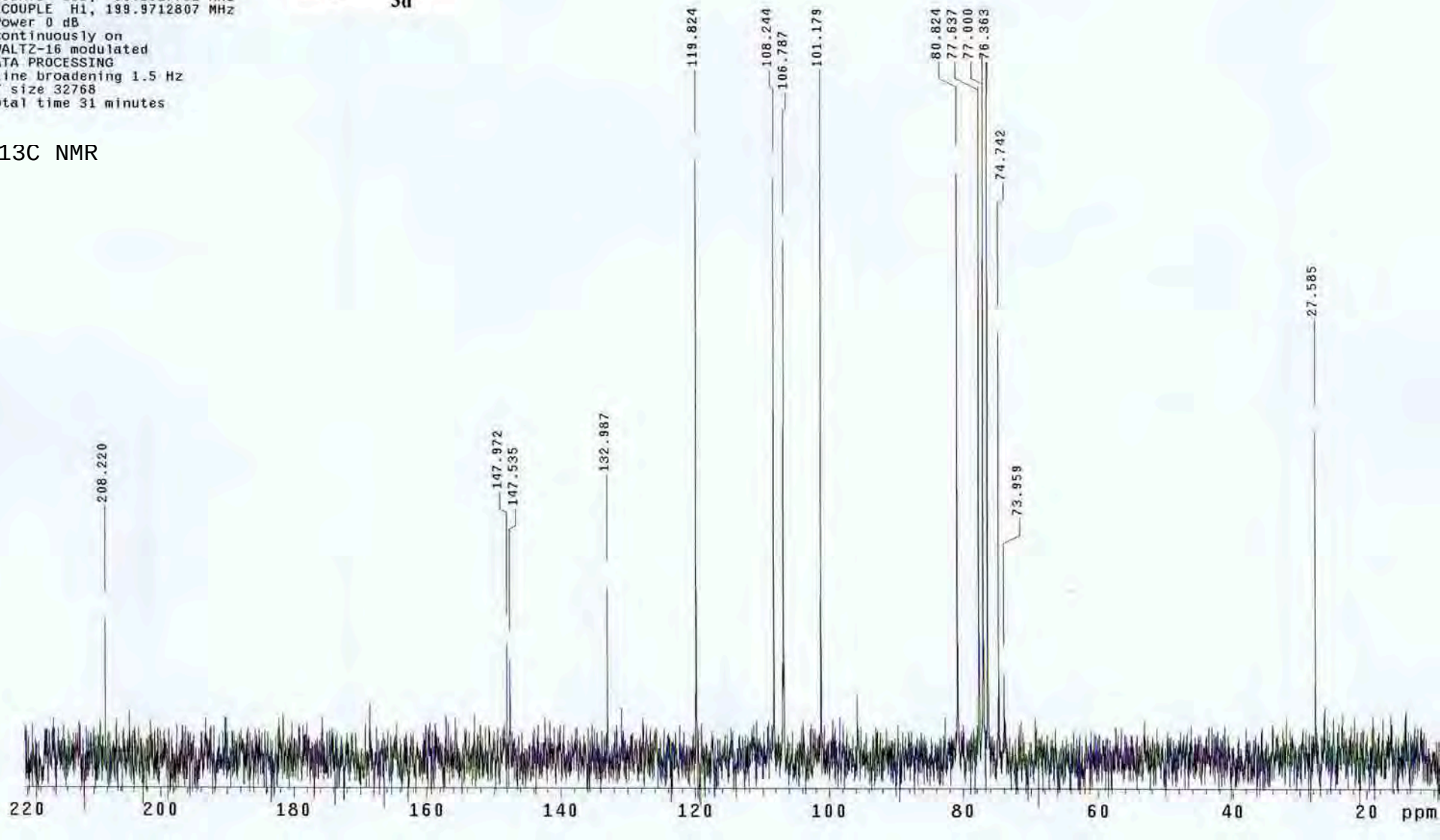
MB-104-2
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827782 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 31 minutes



3d

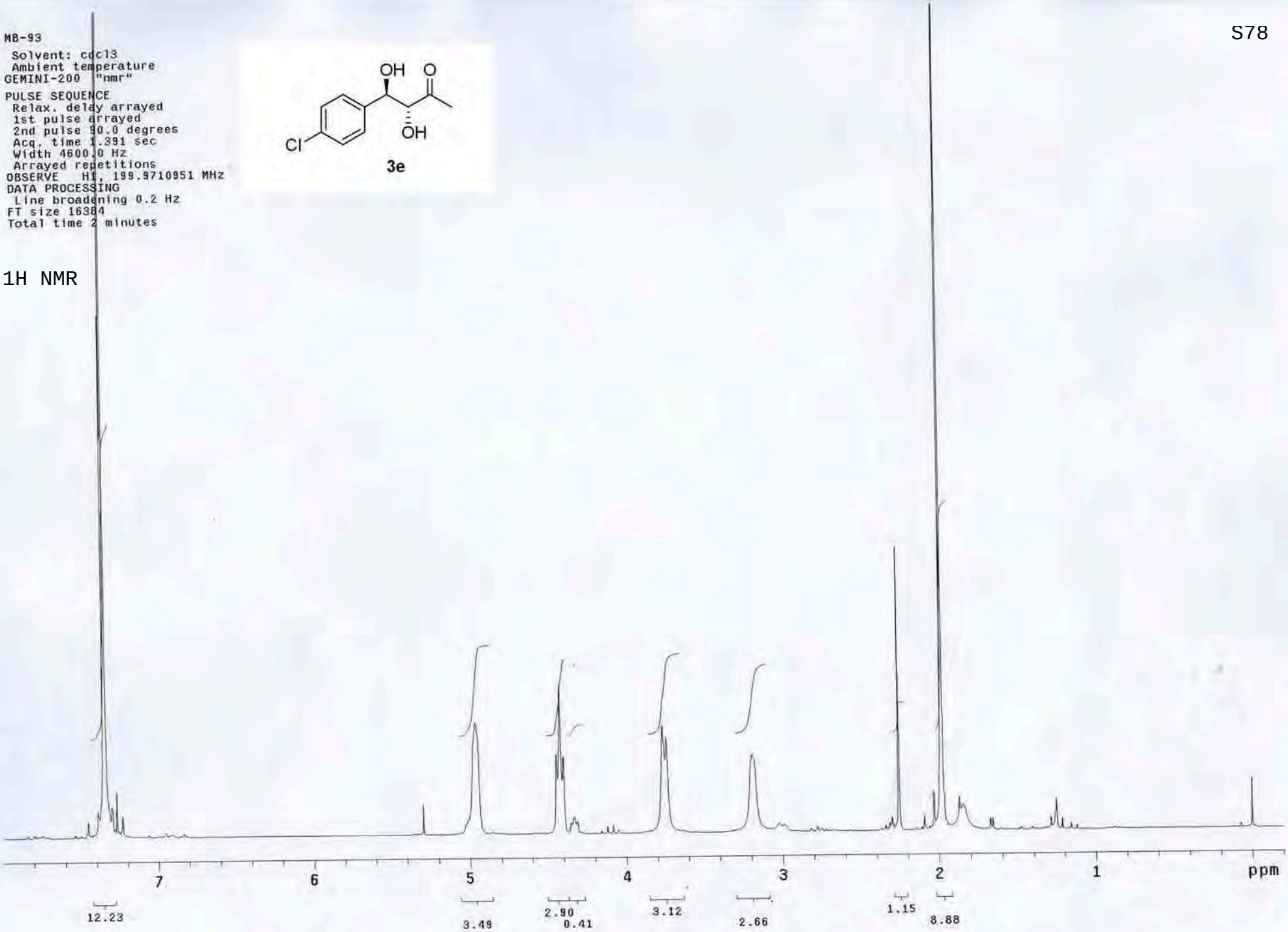
13C NMR



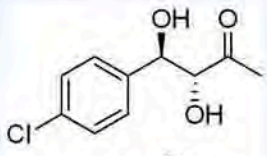
MB-93
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710951 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 2 minutes

3e

1H NMR

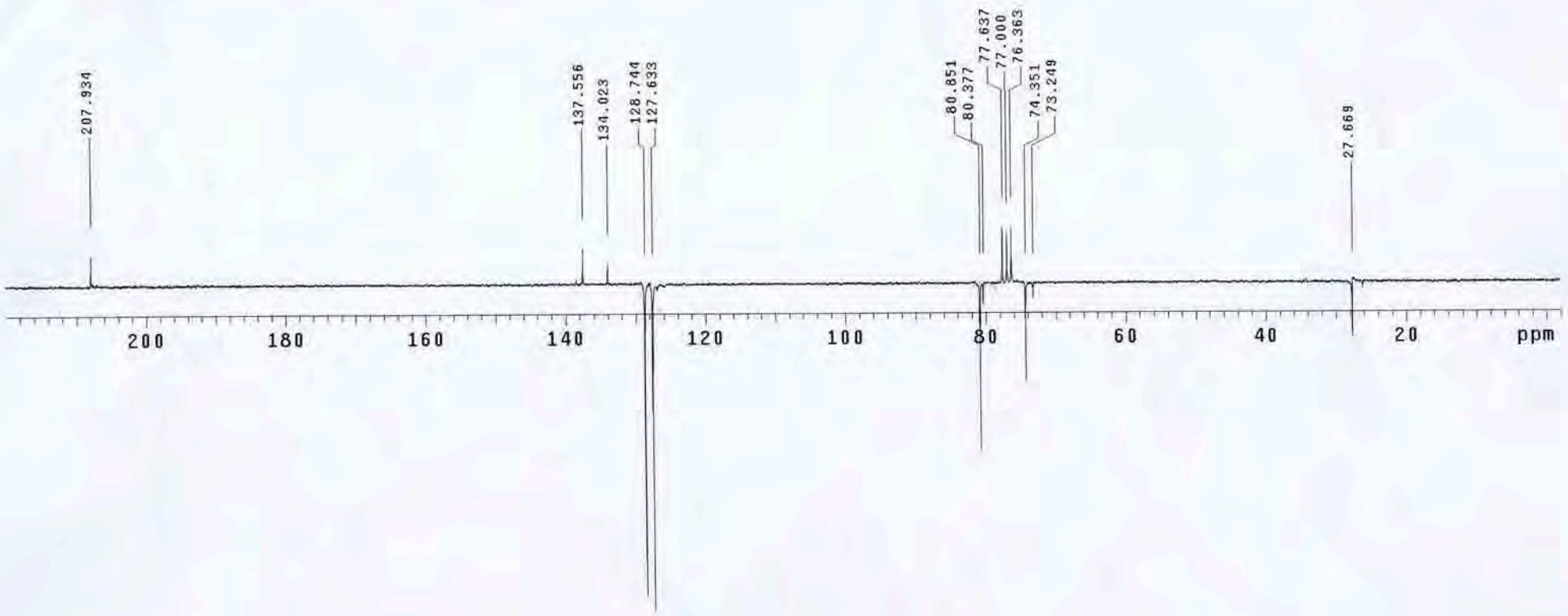


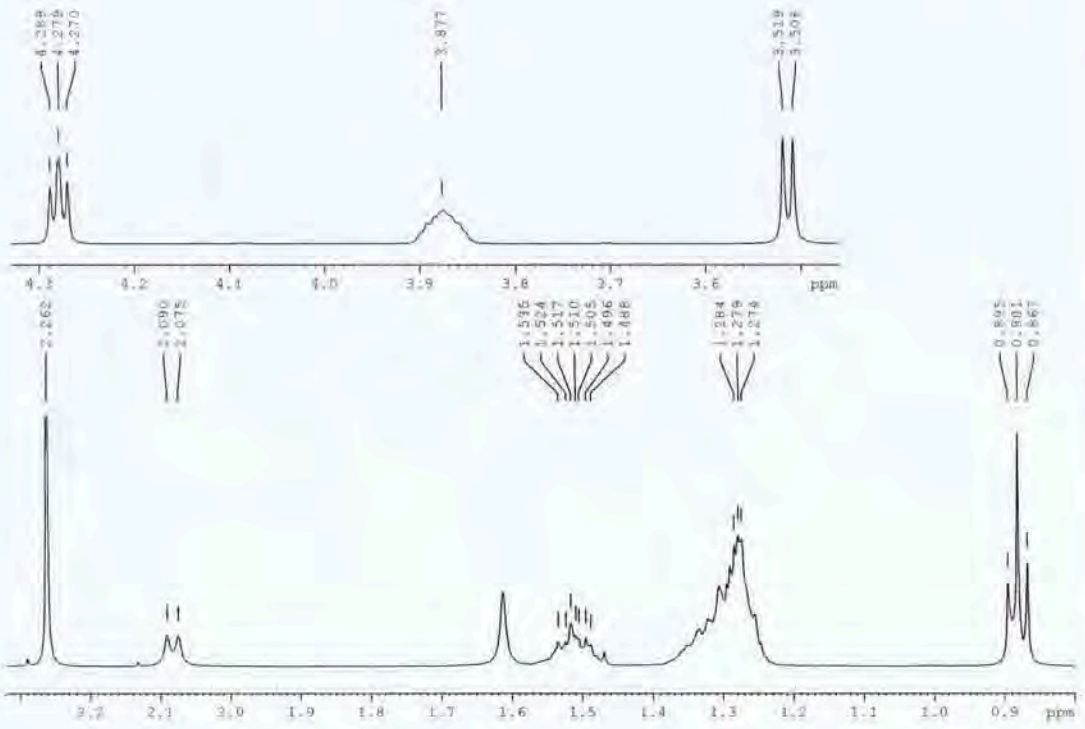
MB-93
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827794 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 14.0 hours



3e

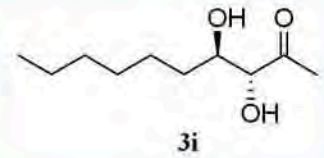
13C NMR



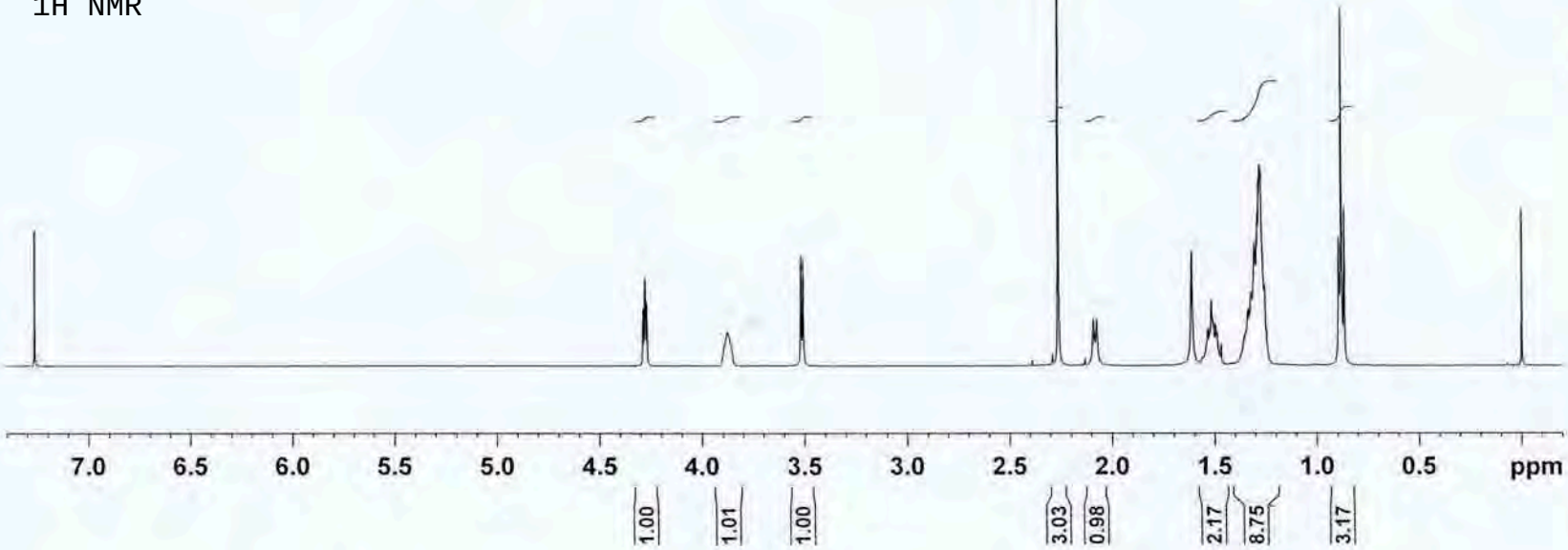


NAME	MB-103
EXPNO	1
PROCNO	1
Date_	20100802
Time	12.24
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4370.629 Hz
FIDRES	0.133381 Hz
AQ	3.7487092 sec
RG	256
DW	114.400 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618233 MHz
SI	32768
SF	500.2600126 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

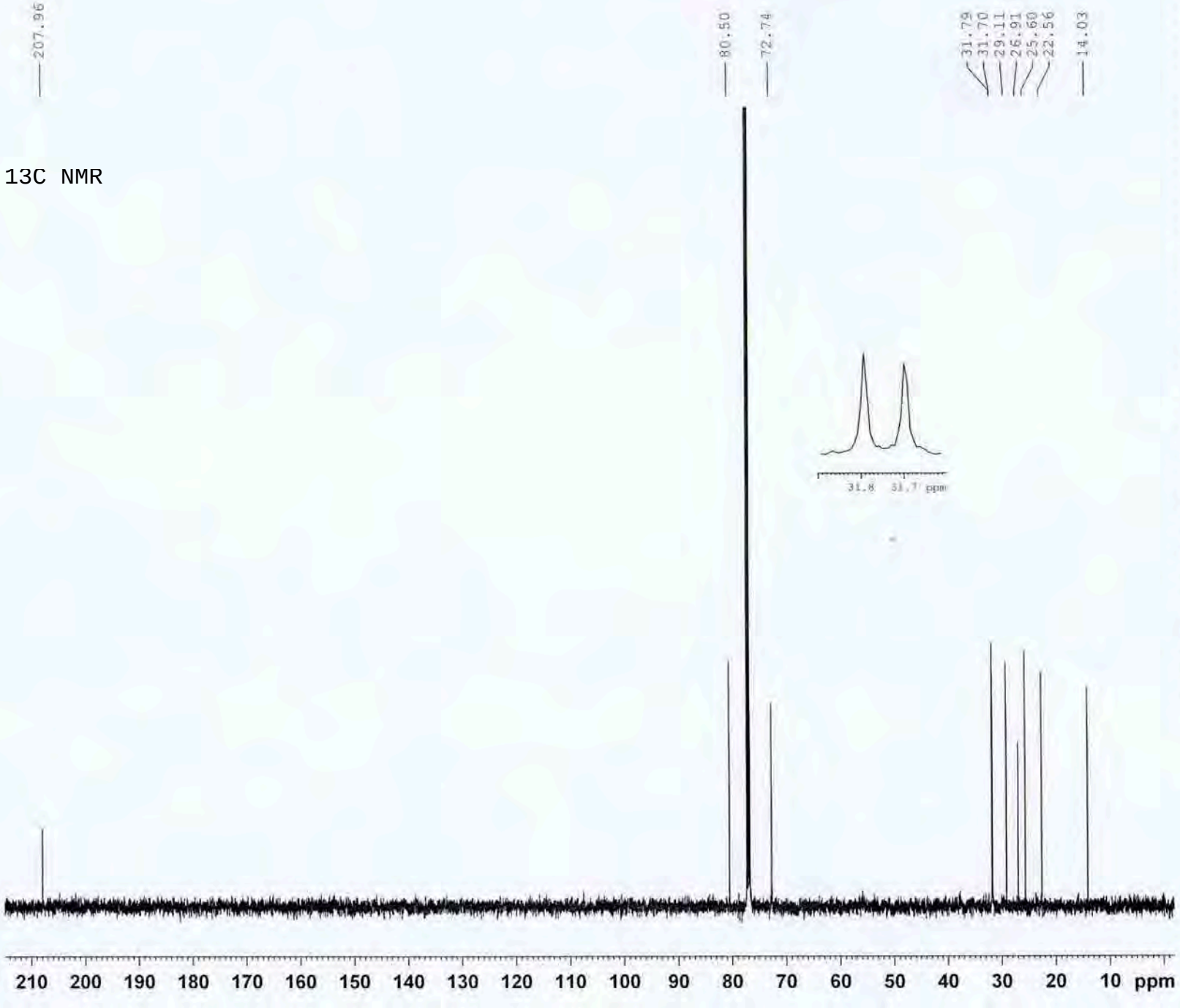
1H NMR



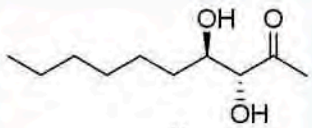
3i



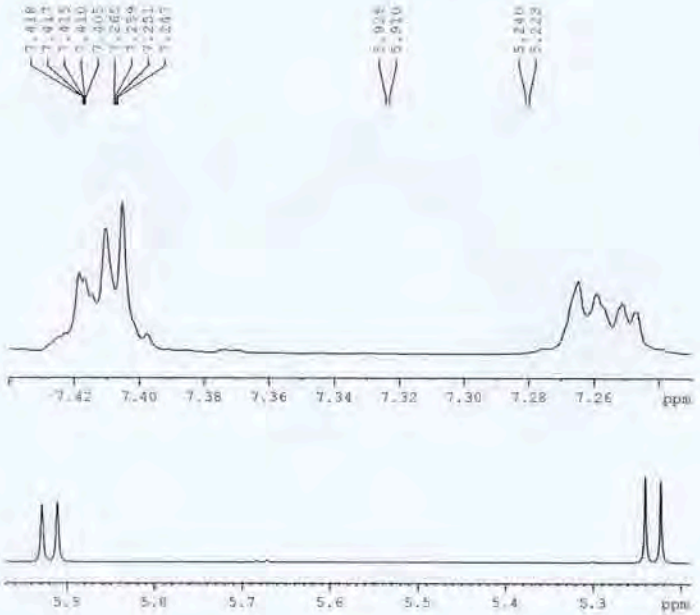
13C NMR



NAME	MB-103
EXPNO	2
PROCNO	1
Date_	20100802
Time	12.31
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	410
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618234 MHz
SI	32768
SF	125.7904796 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

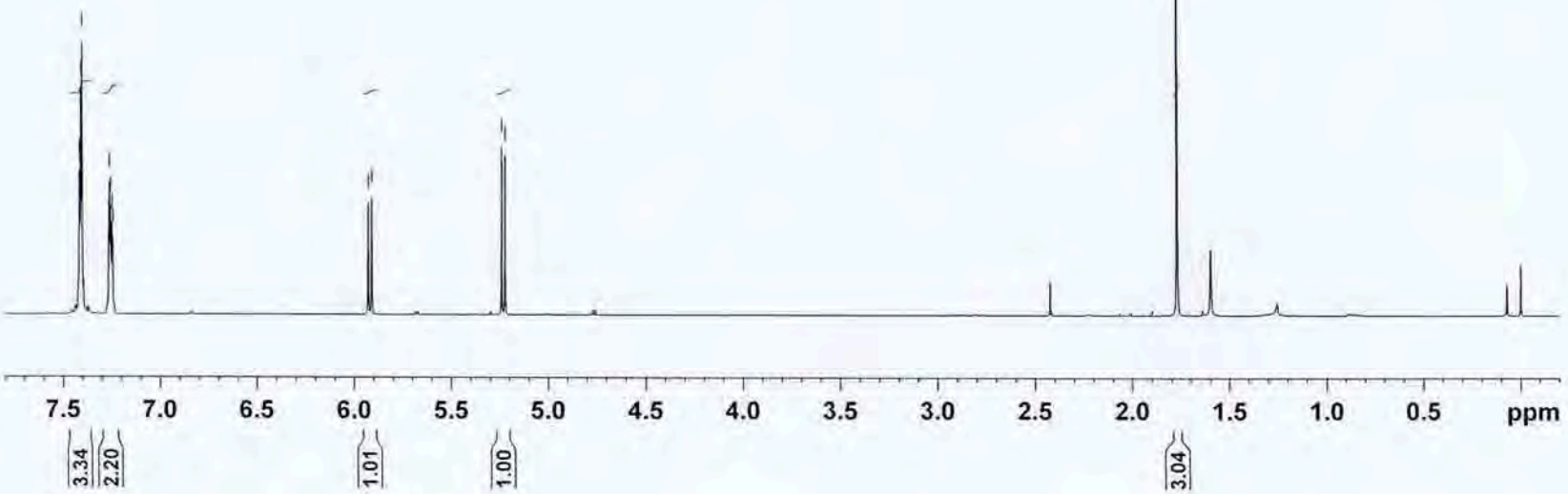


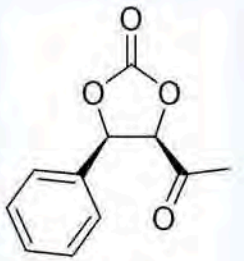
3i



NAME	MB-98
EXPNO	1
PROCNO	1
Date_	20100708
Time	11.16
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
OS	0
SWH	4432.624 Hz
FIDRES	0.135273 Hz
AQ	3.6962805 sec
RG	256
DW	112.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2619095 MHz
SI	32768
SF	500.2600122 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

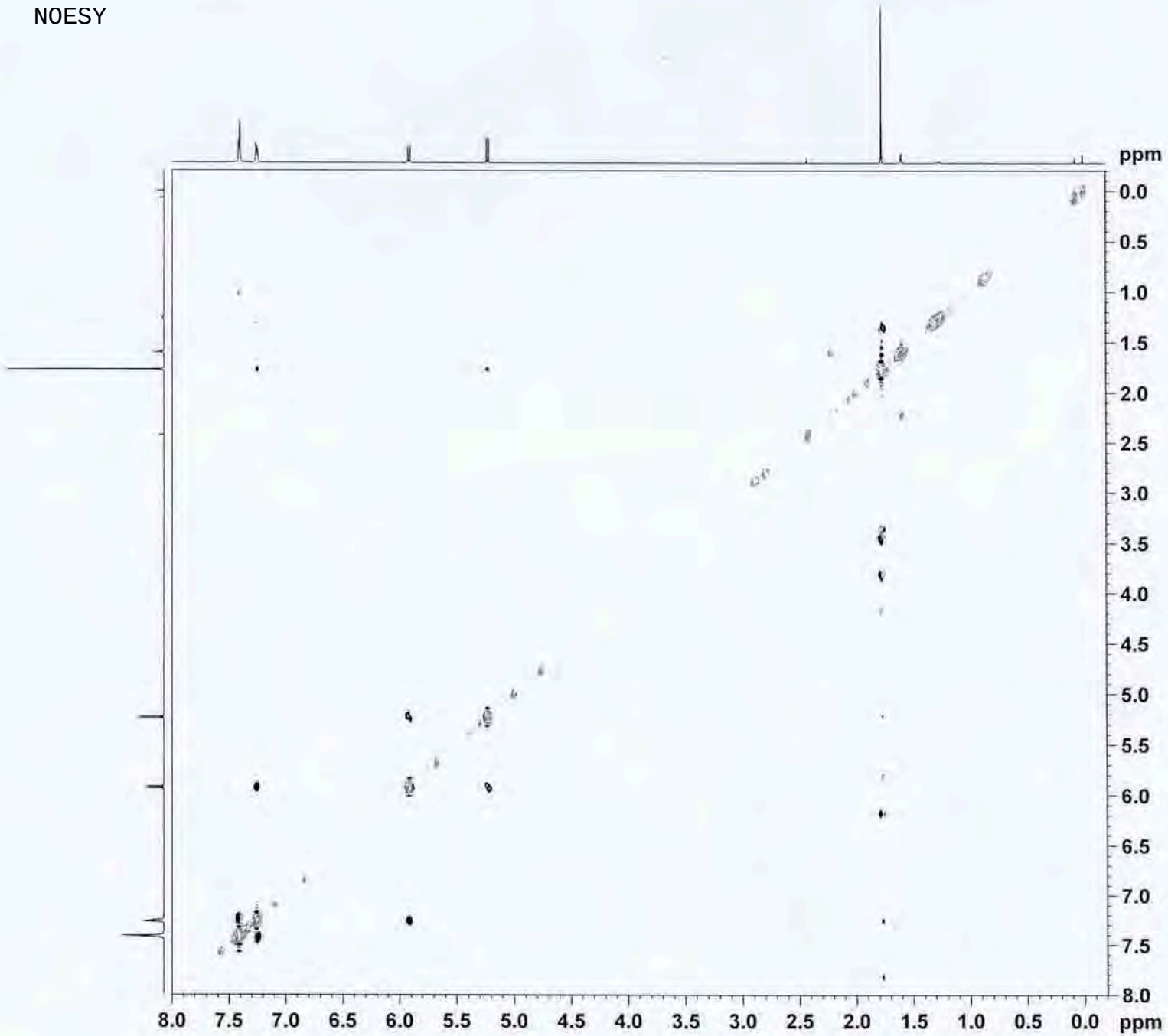
1H NMR



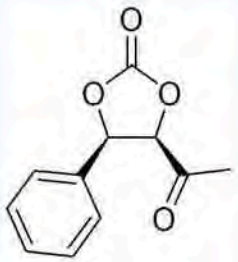


4a

NOESY



NAME	MB-98
EXPNO	2
PROCNO	1
Date_	20100708
Time	11.18
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesyph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4432.624 Hz
FIDRES	4.328734 Hz
AQ	0.1155572 sec
RG	144
DW	112.800 usec
DE	6.50 usec
TE	298.0 K
D0	0.00010090 sec
D1	2.00000000 sec
D8	1.00000000 sec
IN0	0.00022560 sec
========	CHANNEL f1 ========
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2619095 MHz
NDO	1
TD	128
SFO1	500.2619 MHz
FIDRES	34.629848 Hz
SW	8.861 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600092 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600087 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

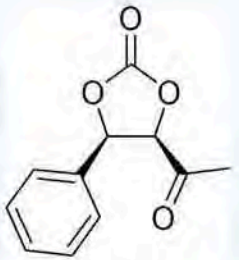


4a

MB-98

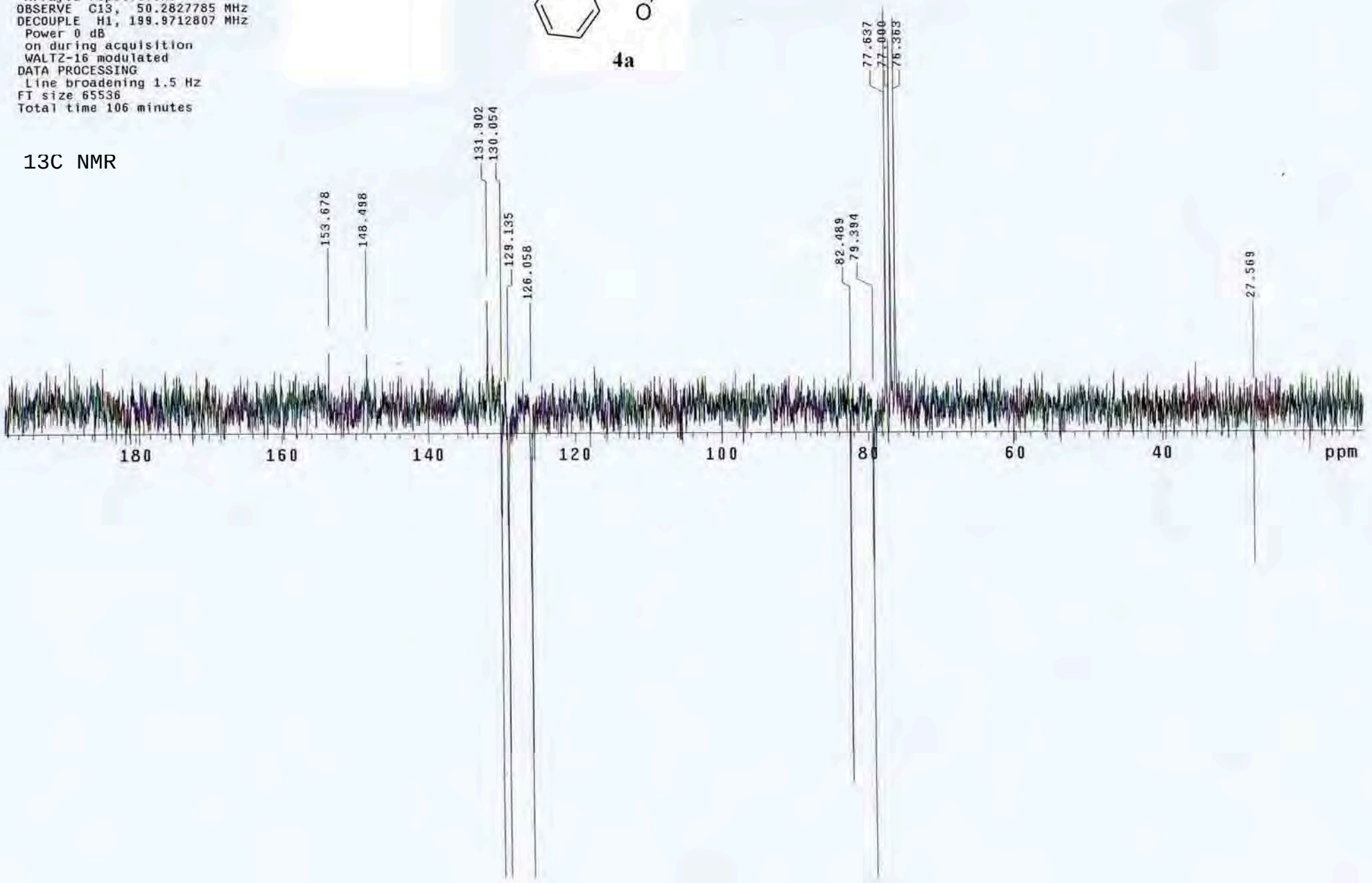
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827785 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 106 minutes



4a

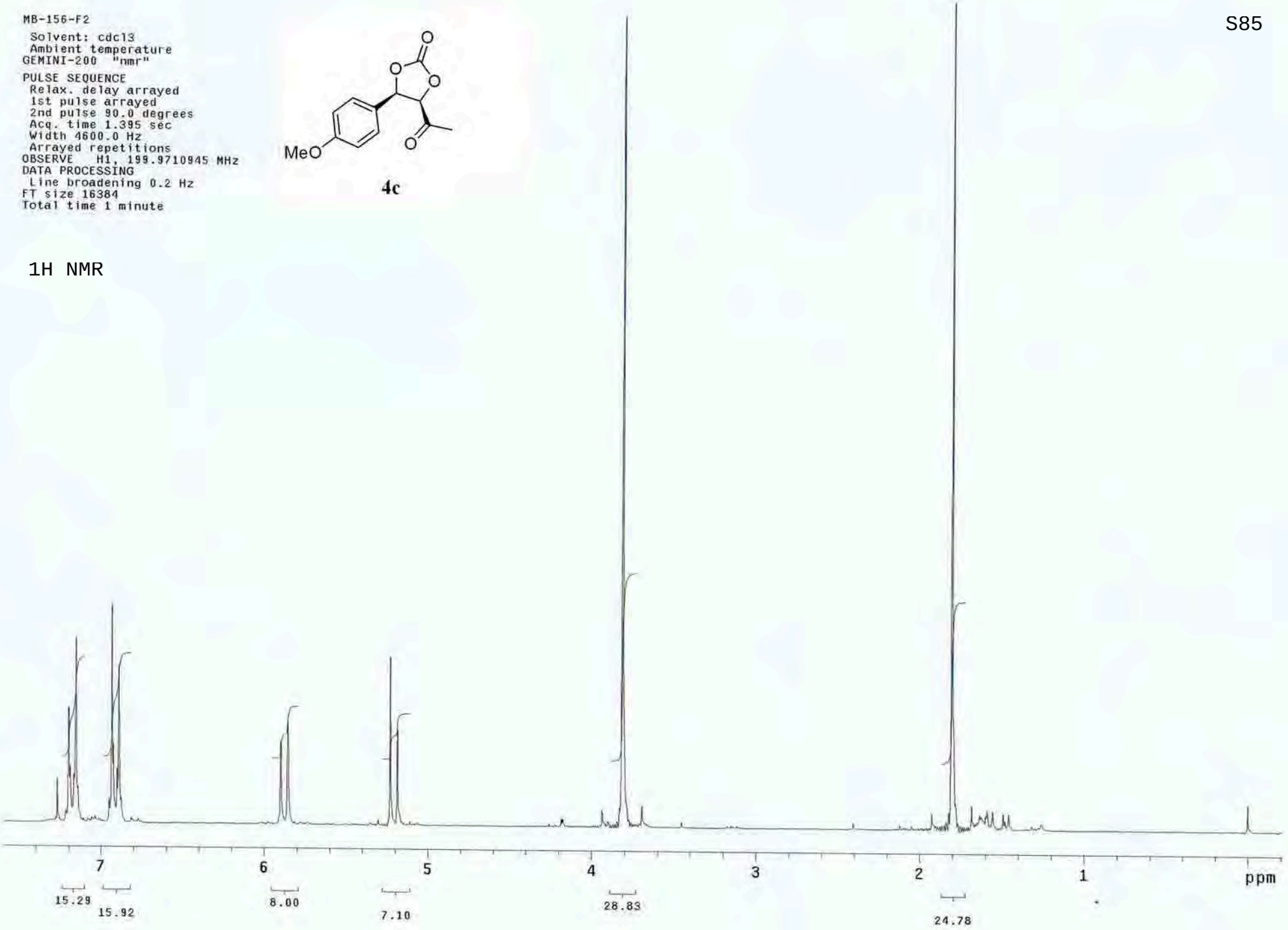
13C NMR



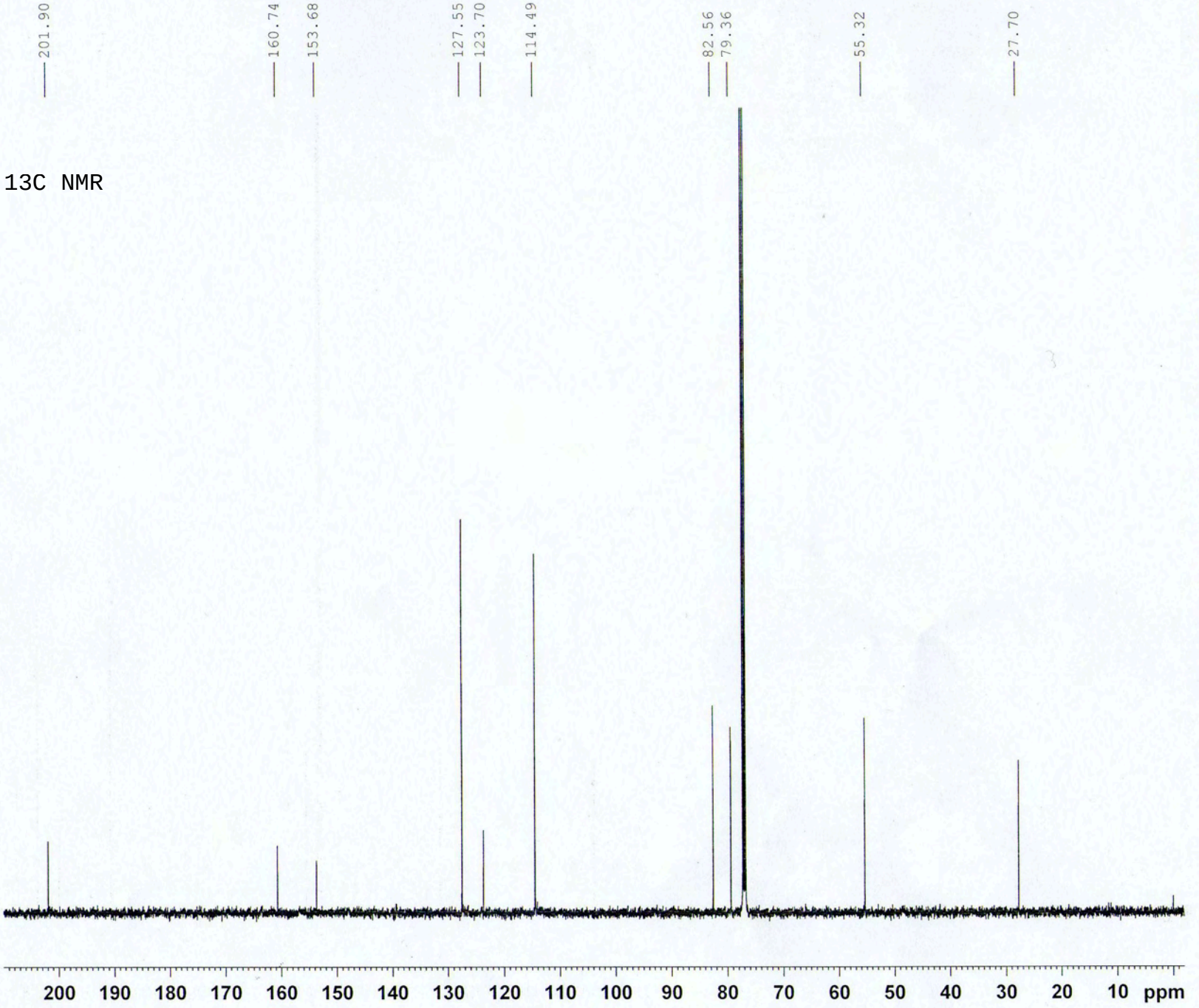
MB-156-F2
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.395 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710945 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute

4c

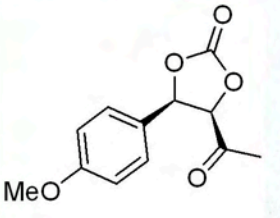
1H NMR



13C NMR

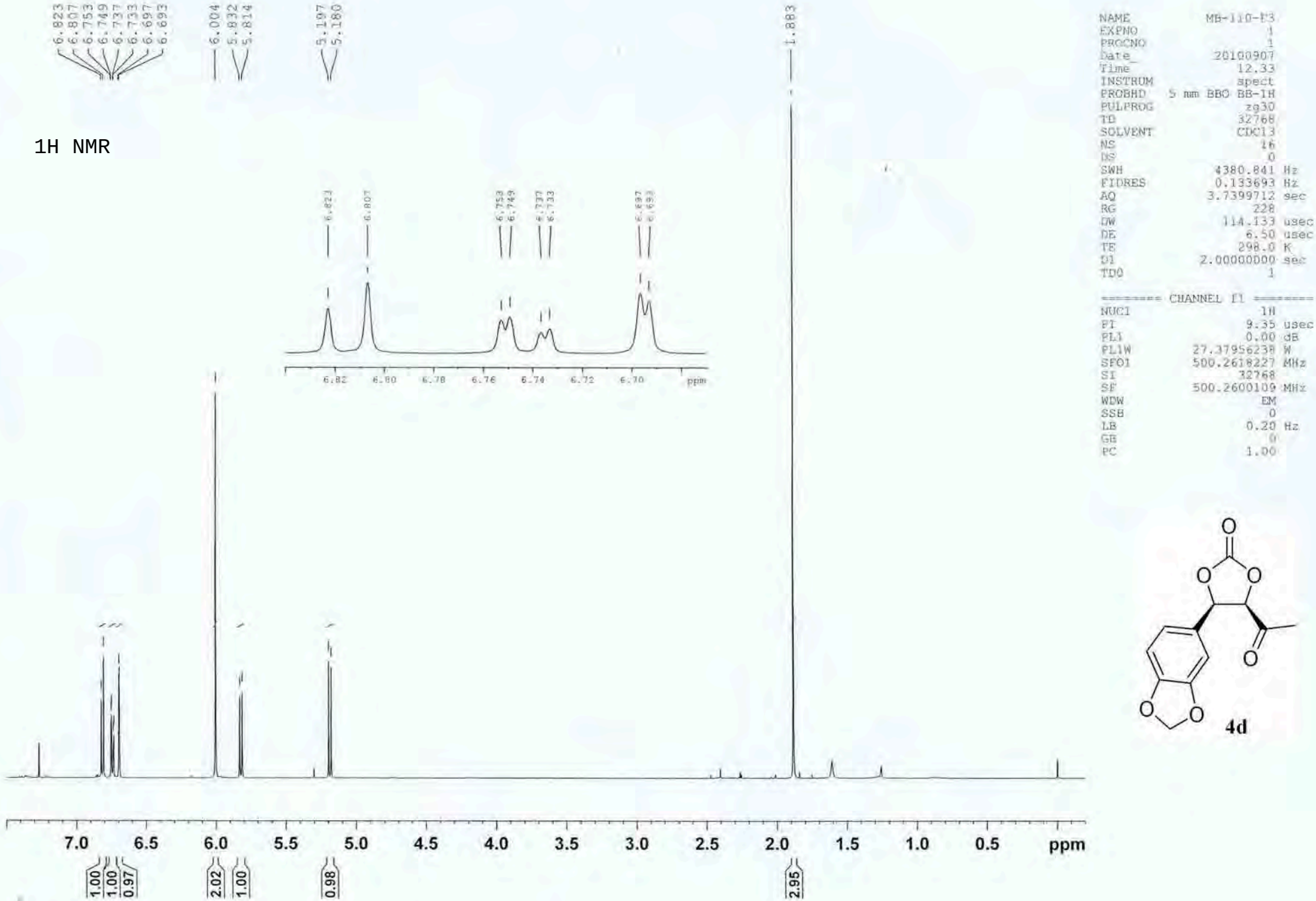


NAME	MB-156f2
EXPNO	2
PROCNO	1
Date_	20110801
Time	11.48
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	474
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2620045 MHz
SI	32768
SF	125.7904811 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



4c

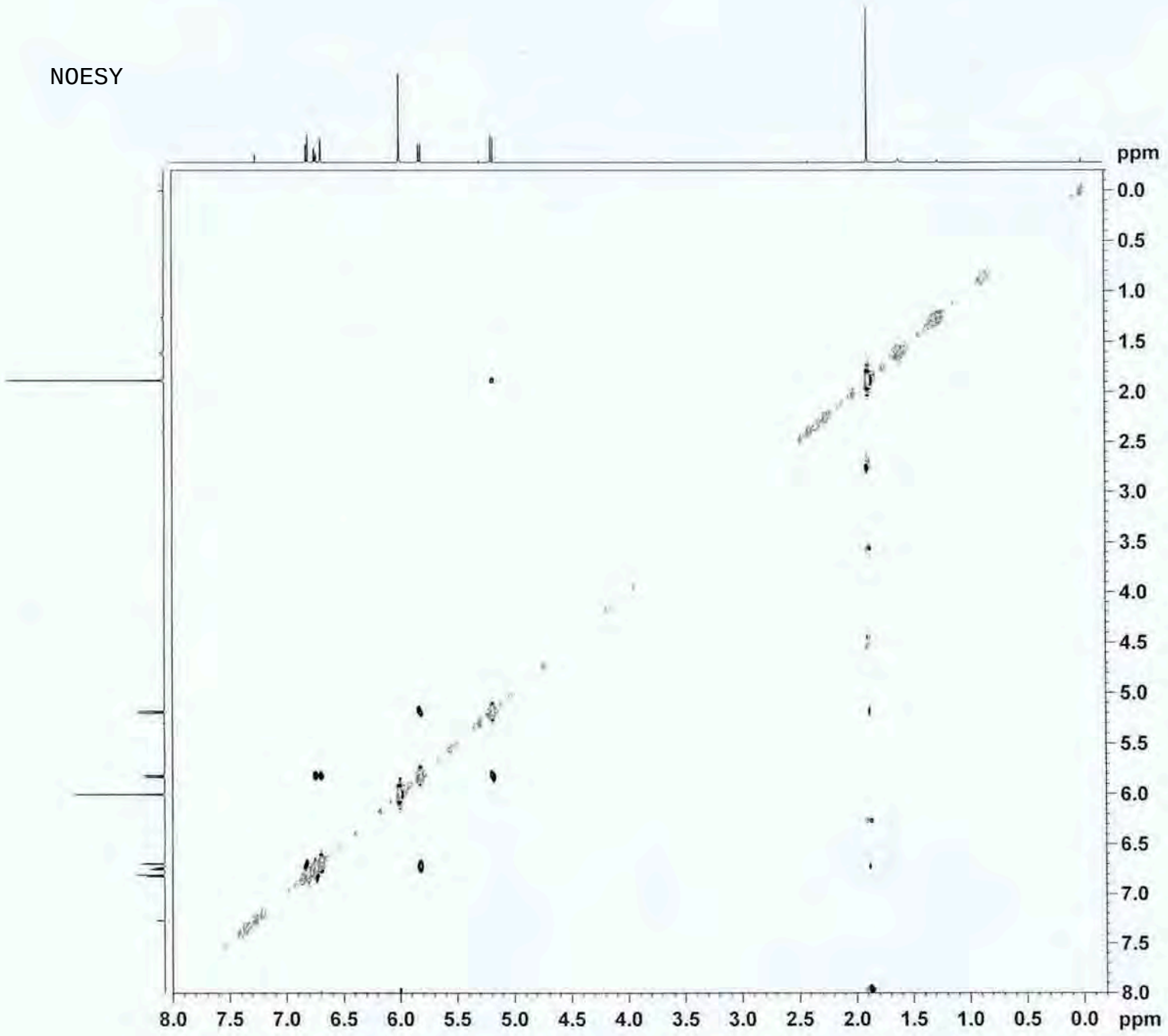
1H NMR



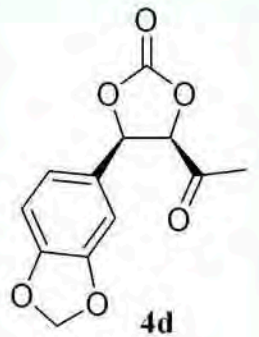
NAME	MB-110-P3
EXPNO	1
PROCNO	1
Date_	20100907
Time	12.33
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4380.841 Hz
FIDRES	0.133693 Hz
AQ	3.7399712 sec
RG	228
DW	114.133 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618227 MHz
SI	32768
SF	500.2600109 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

4d

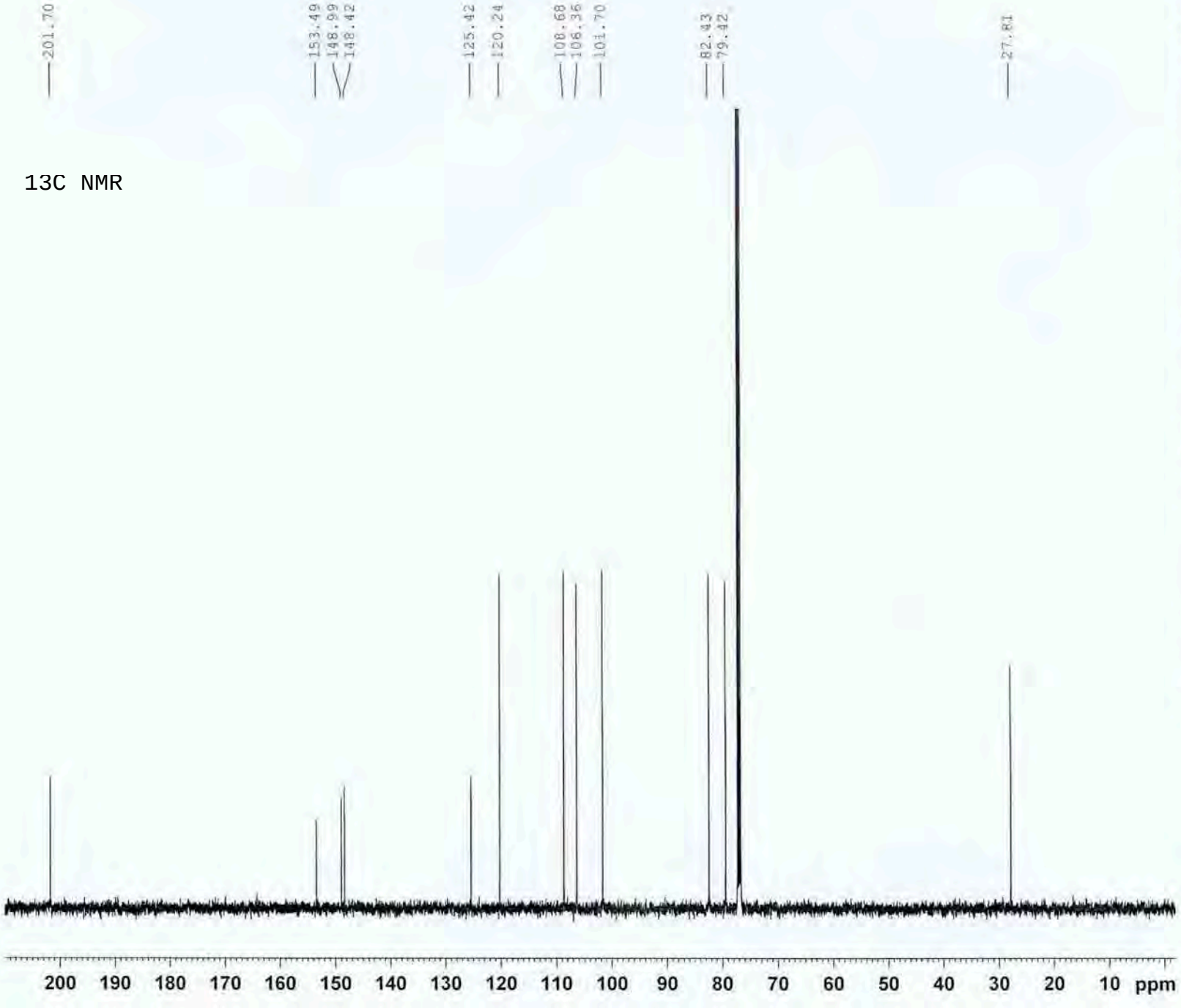
NOESY



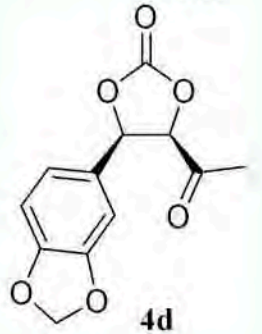
NAME	MB-110-F3
EXPNO	3
PROCNO	1
Date_	20100907
Time	12.47
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesyph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4380.841 Hz
FIDRES	4.278165 Hz
AQ	0.1169225 sec
RG	287
DW	114.133 usec
DE	6.50 usec
TE	297.9 K
D0	0.00010223 sec
D1	2.00000000 sec
D8	1.00000000 sec
IN0	0.00022825 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618229 MHz
ND0	1
TD	128
SFO1	500.2618 MHz
FIDRES	34.225334 Hz
SW	8.757 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600072 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600085 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0



4d

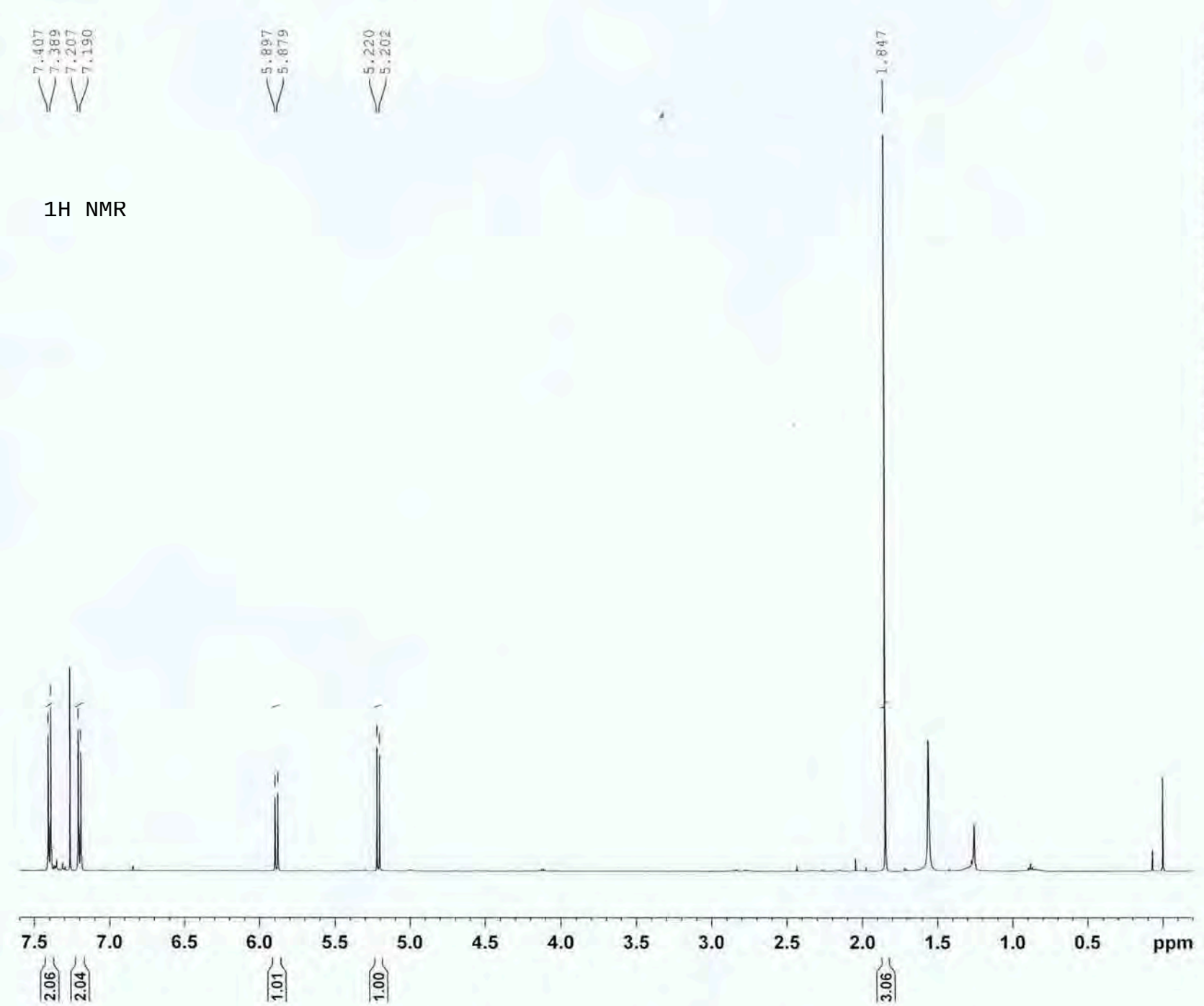


NAME	MB-110-F3
EXPNO	2
PROCNO	1
Date_	20100907
Time	12.41
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	258
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1620
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618229 MHz
SI	32768
SF	125.7904822 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

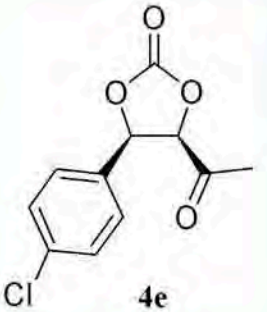


4d

1H NMR

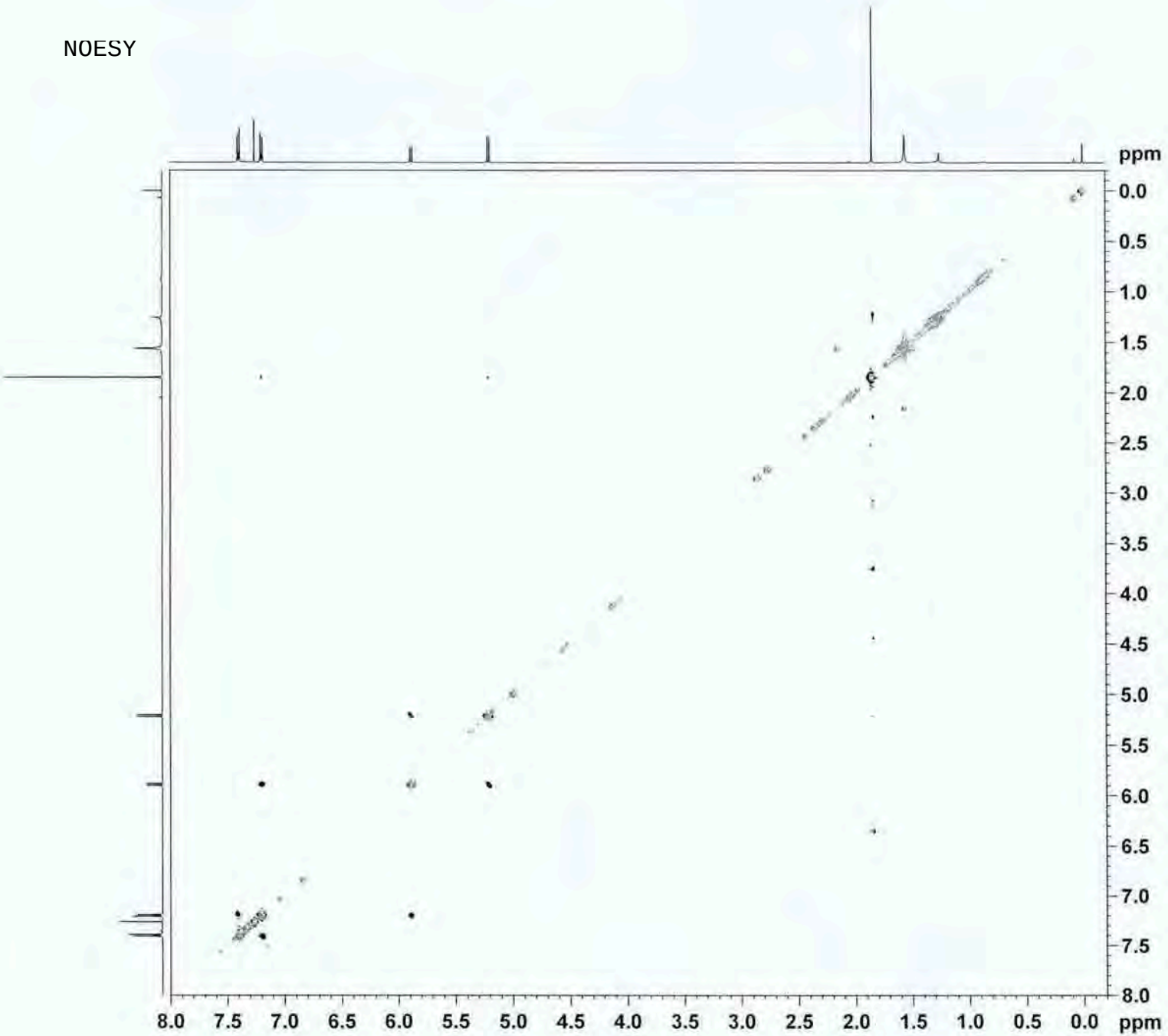


NAME	MB-109-F2
EXPNO	1
PROCNO	1
Date	20100907
Time	13.54
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4504.504 Hz
FIDRES	0.137467 Hz
AQ	3.6372981 sec
RG	322
DW	111.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2619001 MHz
SI	32768
SF	500.2600138 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

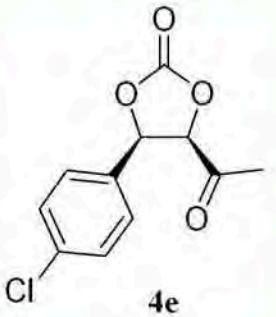


4e

NOESY

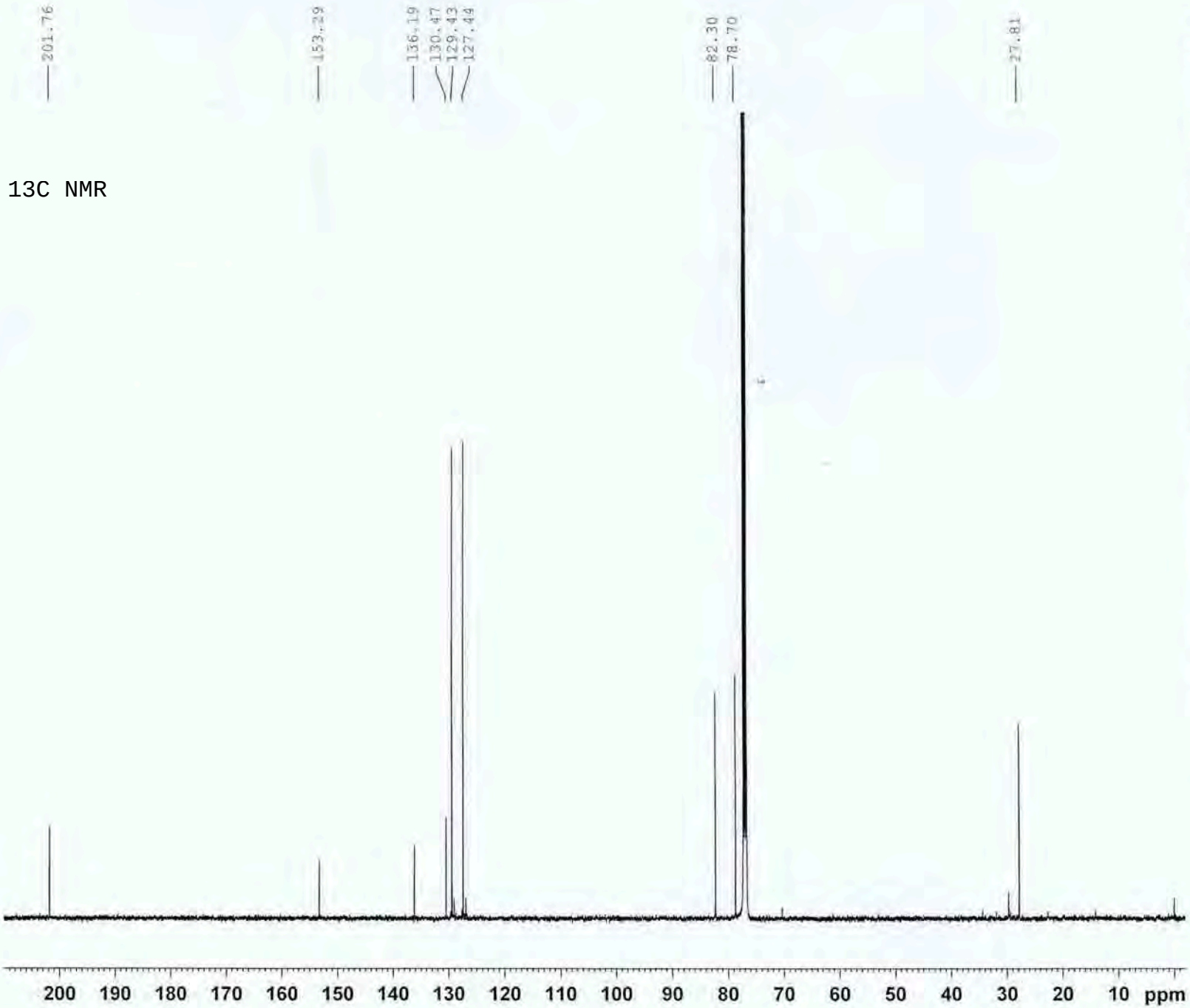


NAME	MB-109-F2	
EXPNO	2	
PROCNO	1	
Date_	20100907	
Time	13.59	
INSTRUM	spect	
PROBHD	5 mm BBO BB-1H	
PULPROG	noesyph	
TD	1024	
SOLVENT	CDCl3	
NS	16	
DS	16	
SWH	4504.504	Hz
FIDRES	4.398930	Hz
AQ	0.1137140	sec
RG	287	
DW	111.000	usec
DE	6.50	usec
TE	298.0	K
D0	0.00009910	sec
D1	2.00000000	sec
D8	1.00000000	sec
IN0	0.00022200	sec
========	CHANNEL f1 ========	
NUC1	1H	
P1	9.35	usec
PL1	0.00	dB
PL1W	27.37956238	W
SFO1	500.2619000	MHz
ND0	1	
TD	256	
SFO1	500.2619	MHz
FIDRES	17.595736	Hz
SW	9.004	ppm
FnMODE	States-TPPI	
SI	512	
SF	500.2600088	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	
PC	1.00	
SI	512	
MC2	States-TPPI	
SF	500.2600104	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	

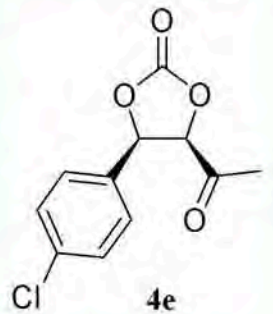


4e

13C NMR

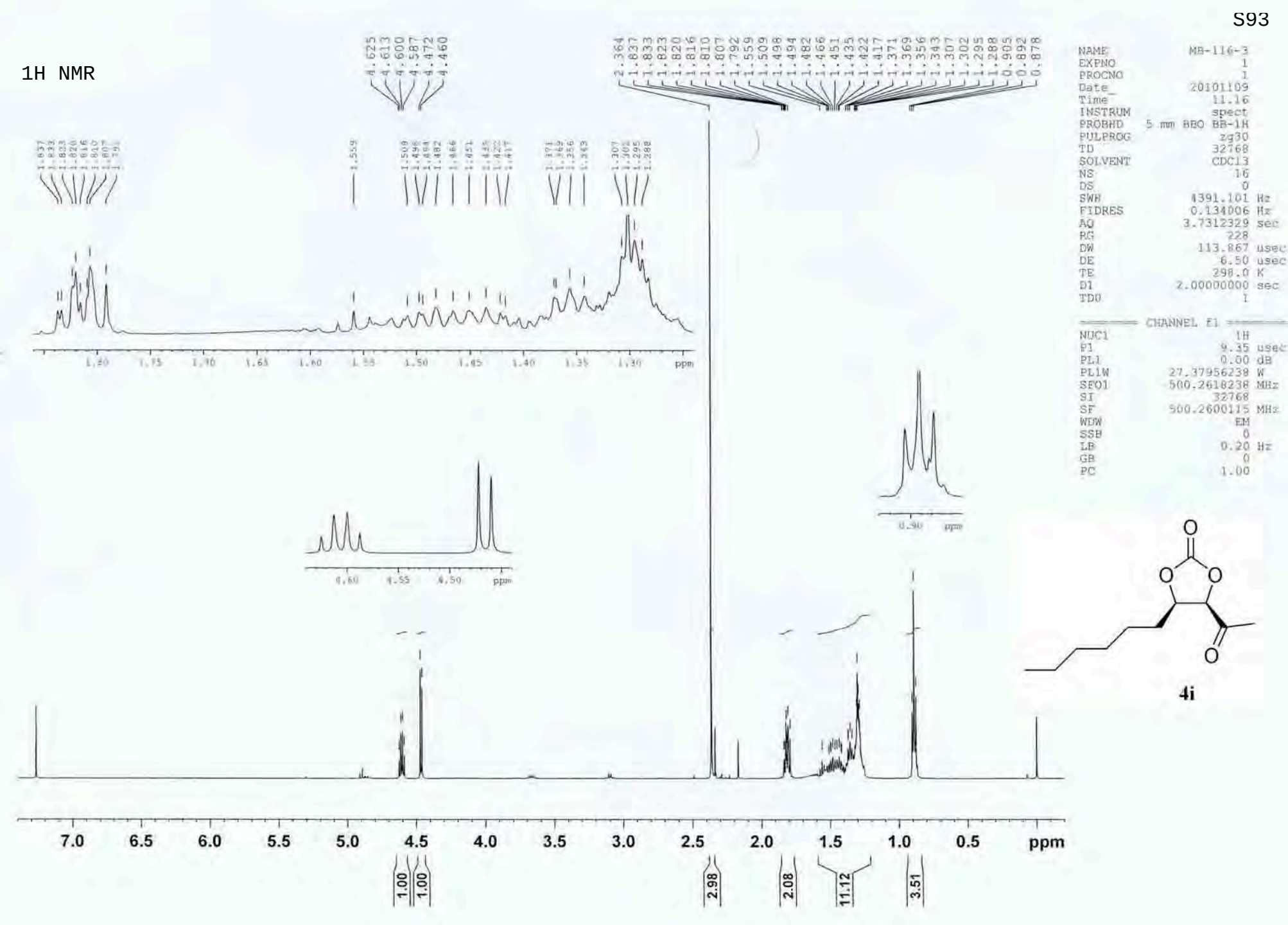


NAME	MB-109-F2
EXPNO	3
PROCNO	1
Date_	20100908
Time	10.25
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	23322
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1620
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2619000 MHz
SI	32768
SF	125.7904796 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



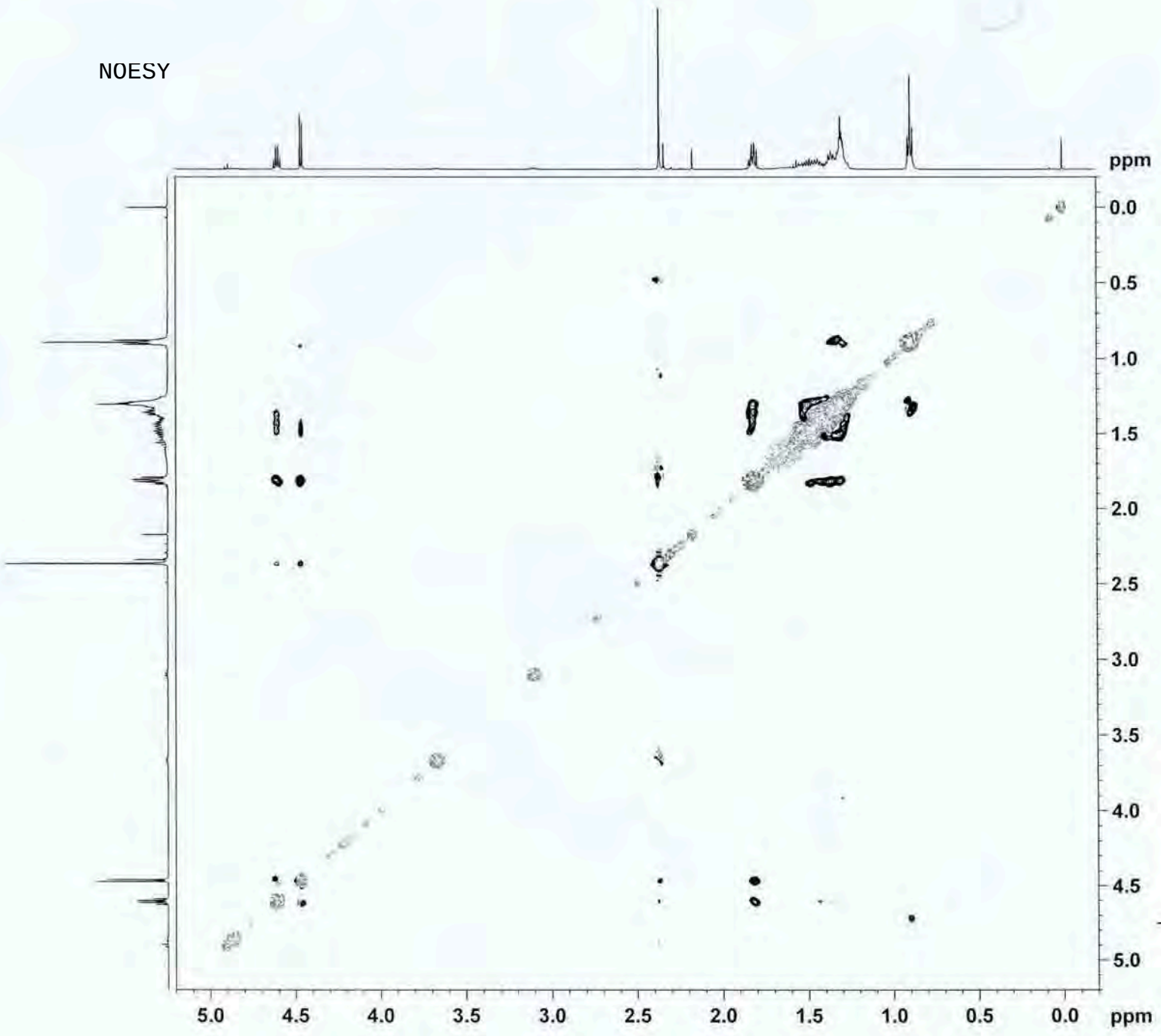
4e

1H NMR

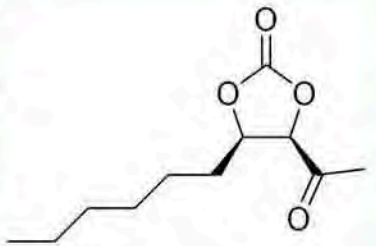

4.625
4.613
4.600
4.587
4.472
4.460
2.364
1.837
1.833
1.823
1.820
1.816
1.810
1.807
1.792
1.559
1.509
1.498
1.494
1.482
1.466
1.451
1.435
1.422
1.417
1.371
1.369
1.356
1.343
1.307
1.302
1.295
1.288
0.905
0.892
0.878
NAME MB-116-3
EXPNO 1
PROCNO 1
Date_ 20101109
Time 11.16
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 0
SWH 4391.101 Hz
FIDRES 0.134006 Hz
AQ 3.7312329 sec
RG 228
DW 113.867 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 9.35 usec
PL1 0.00 dB
PL1W 27.37956238 W
SFO1 500.2618238 MHz
SI 32768
SF 500.2600115 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
ppm
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
1.00
1.00
2.98
2.08
11.12
3.51

4i

NOESY



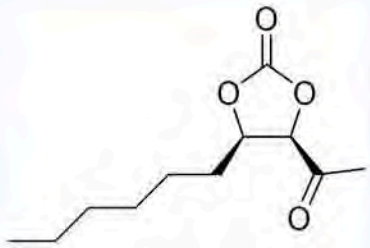
NAME	MB-116-3
EXPNO	2
PROCNO	1
Date_	20101109
Time	11.19
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesyph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4391.101 Hz
FIDRES	4.288184 Hz
AQ	0.1166495 sec
RG	128
DW	113.867 usec
DE	6.50 usec
TE	298.0 K
D0	0.00010196 sec
D1	2.00000000 sec
D8	1.00000000 sec
IN0	0.00022775 sec
========	CHANNEL f1 ========
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618239 MHz
ND0	1
TD	256
SFO1	500.2618 MHz
FIDRES	17.152727 Hz
SW	8.778 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600064 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600068 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0



4i

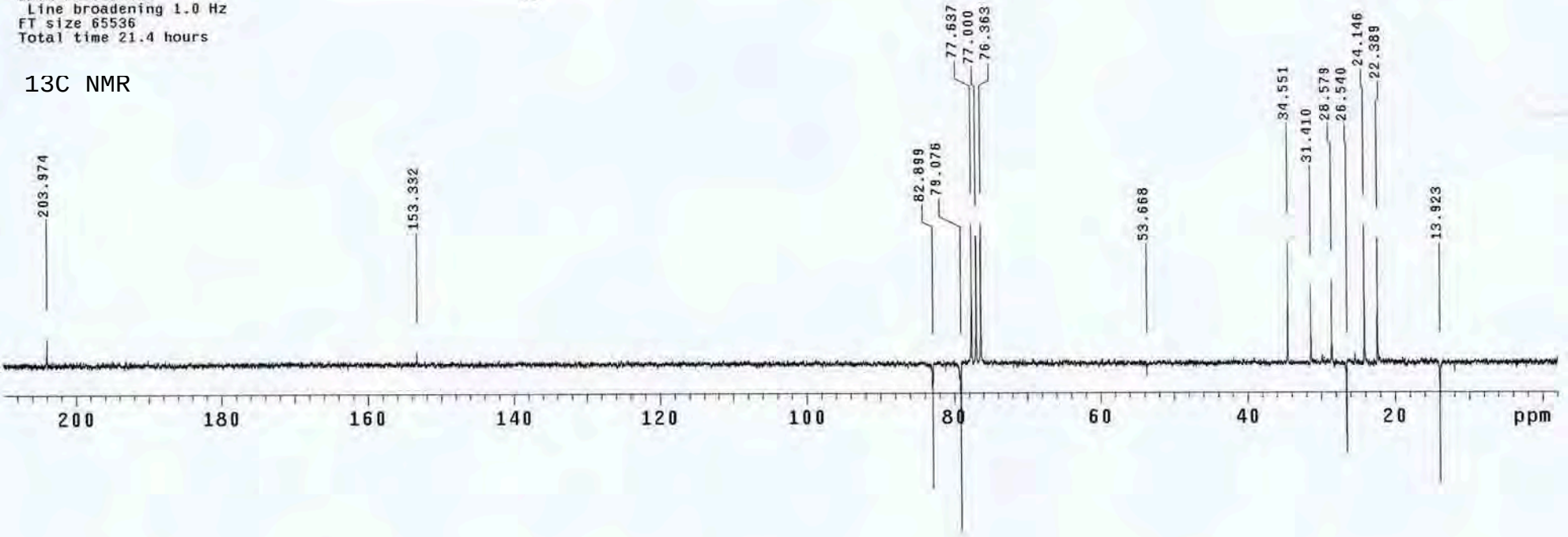
MB-116-3-1
Solvent: cdcl3
Ambient temperature
File: aptmb10631
GEMINI-200 "nmr"

PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827785 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 21.4 hours



4i

13C NMR

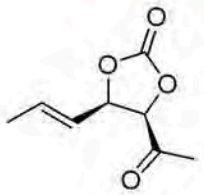


1H NMR

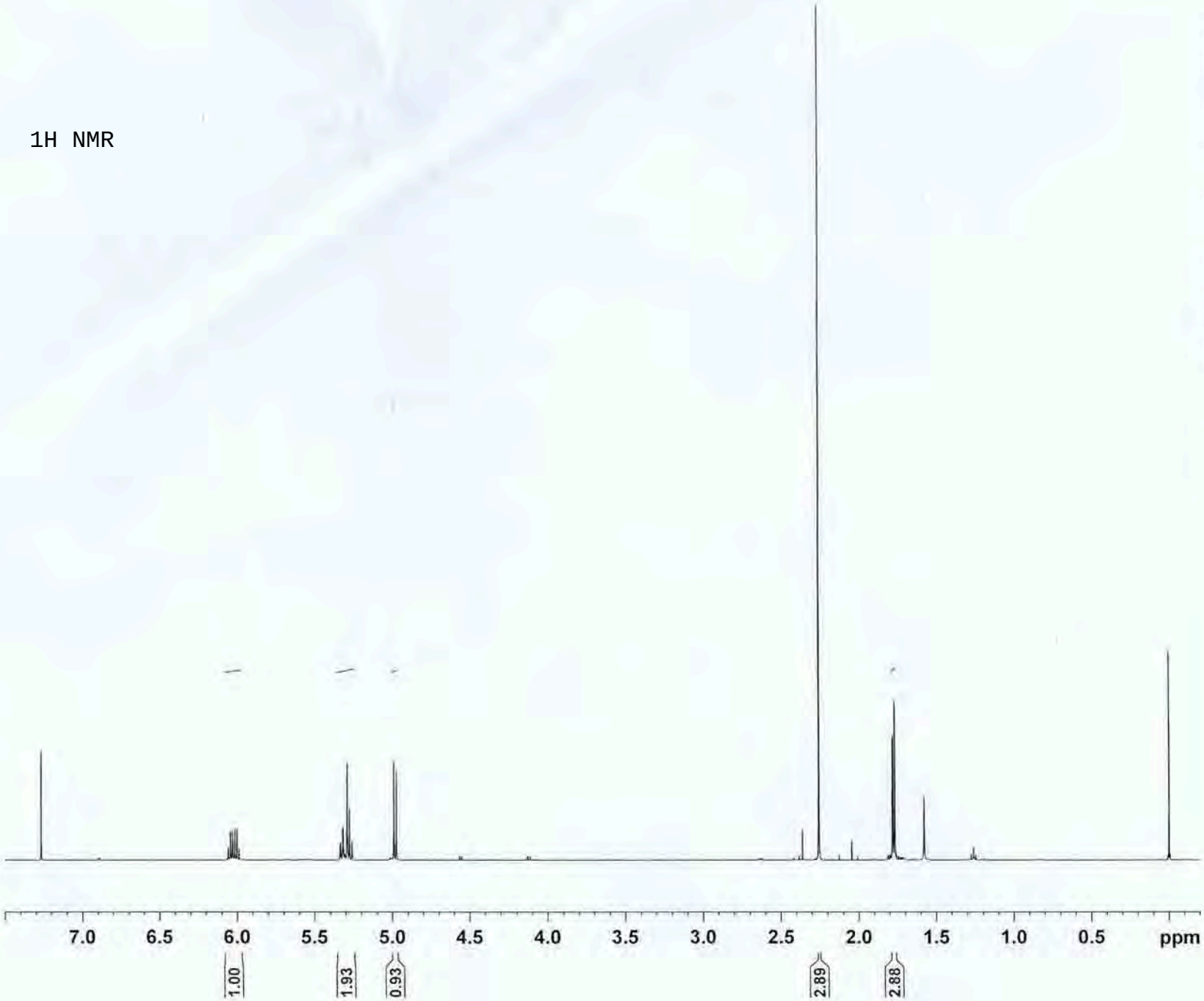
NAME	MB-117-F2
EXPNO	1
PROCNO	1
Date_	20101015
Time	12.43
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4360.465 Hz
FIDRES	0.133071 Hz
AQ	3.7574472 sec
RG	287
DW	114.667 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1

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

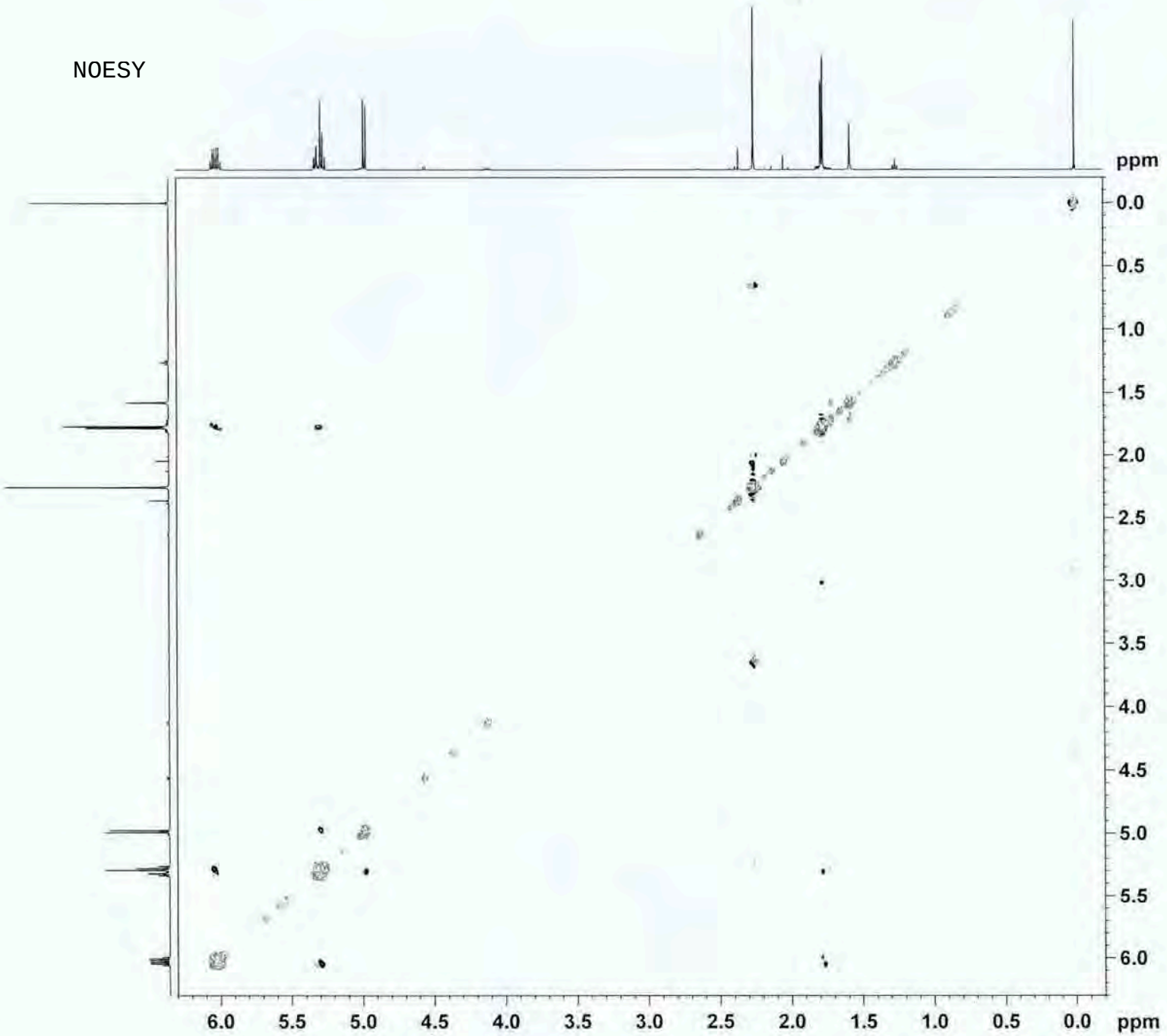
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618293 MHz
SI	32768
SF	500.2600119 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



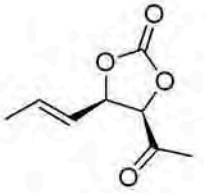
4j



NOESY

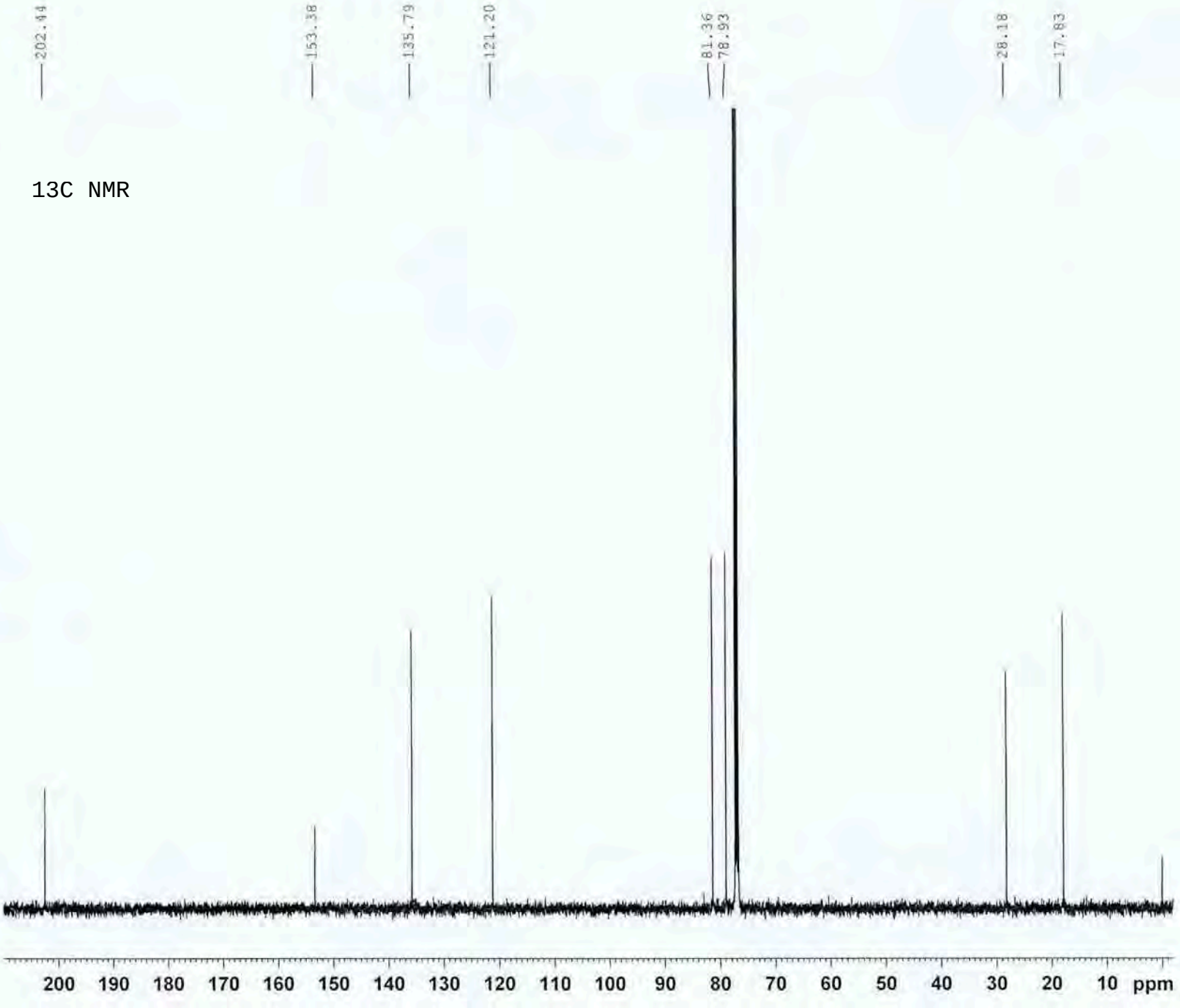


NAME	MB-117-P2
EXPNO	4
PROCNO	1
Date_	20101015
Time	13.56
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesyph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4360.465 Hz
FIDRES	4.258267 Hz
AQ	0.1174687 sec
RG	161
DW	114.667 usec
DE	6.50 usec
TE	298.0 K
D0	0.00010276 sec
D1	2.00000000 sec
D8	1.00000000 sec
IN0	0.00022935 sec
CHANNEL f1	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618295 MHz
NDO	1
TD	194
SFO1	500.2618 MHz
FIDRES	22.476713 Hz
SW	8.716 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600067 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600086 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

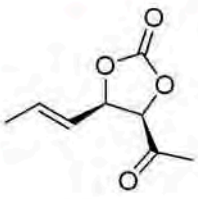


4j

13C NMR

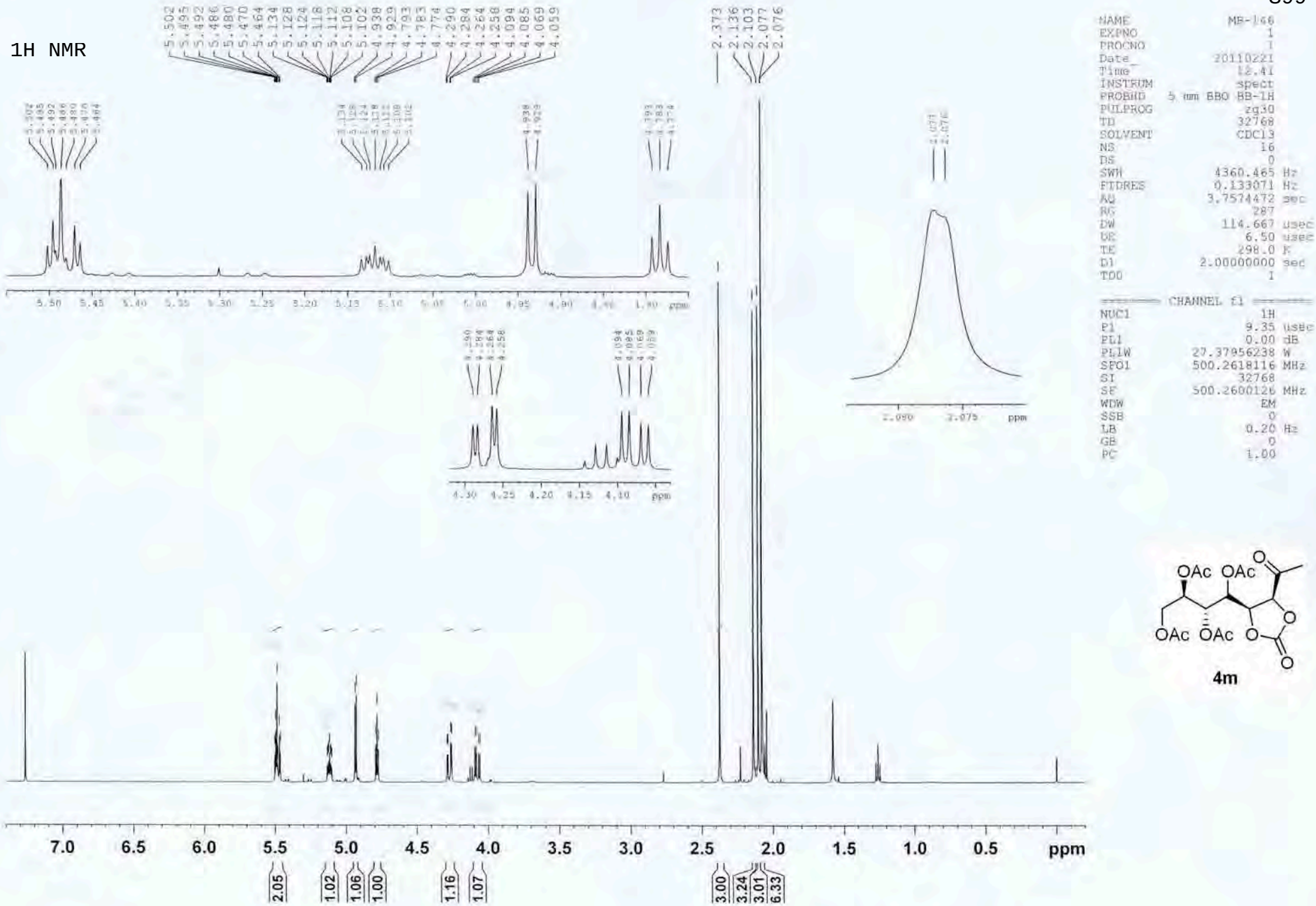


NAME	MB-117-F2	
EXPNO	3	
PROCNO	1	
Date_	20101015	
Time	13.15	
INSTRUM	spect	
PROBHD	5 mm BBO BB-1H	
PULPROG	zgpg30	
TD	32768	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	29761.904	Hz
FIDRES	0.908261	Hz
AQ	0.5505524	sec
RG	912	
DW	16.800	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
========	CHANNEL f1	========
NUC1	13C	
P1	11.50	usec
PL1	3.00	dB
PL1W	32.22848892	W
SFO1	125.8043140	MHz
========	CHANNEL f2	========
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	1.20	dB
PL12	18.40	dB
PL13	18.40	dB
PL2W	20.76952171	W
PL12W	0.39575511	W
PL13W	0.39575511	W
SFO2	500.2618295	MHz
SI	32768	
SF	125.7904805	MHz
WDW	EM	
SSB	0	
LB	1.50	Hz
GB	0	
PC	1.40	



4j

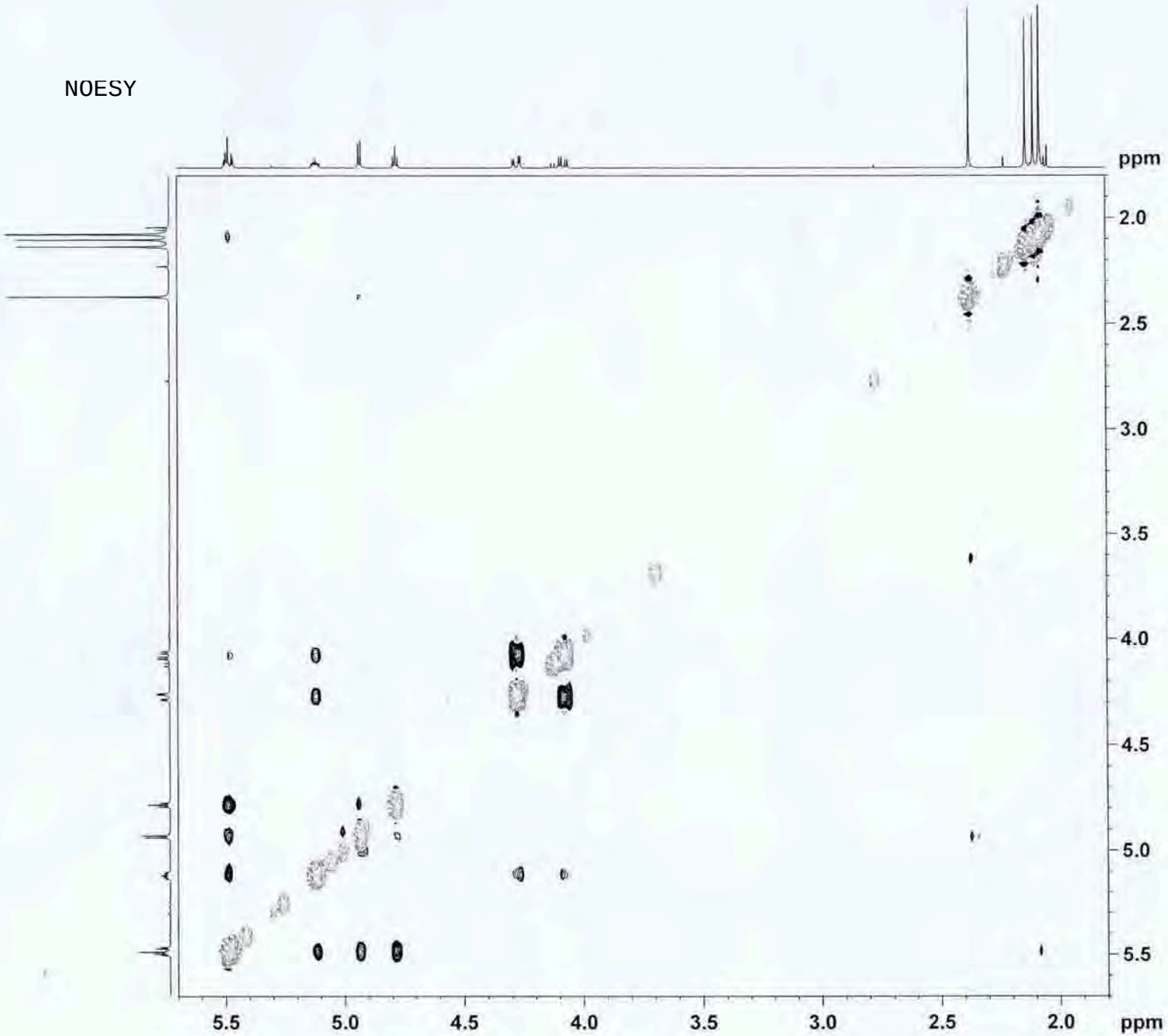
1H NMR



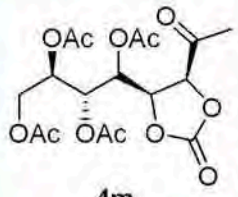
NAME	MB-146
EXPNO	1
PROCNO	1
Date_	20110221
Time	12.41
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4360.465 Hz
FIDRES	0.133071 Hz
AQ	3.7574472 sec
RG	287
DW	114.667 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
CHANNEL f1	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618116 MHz
SI	32768
SF	500.2600126 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

4m

NOESY

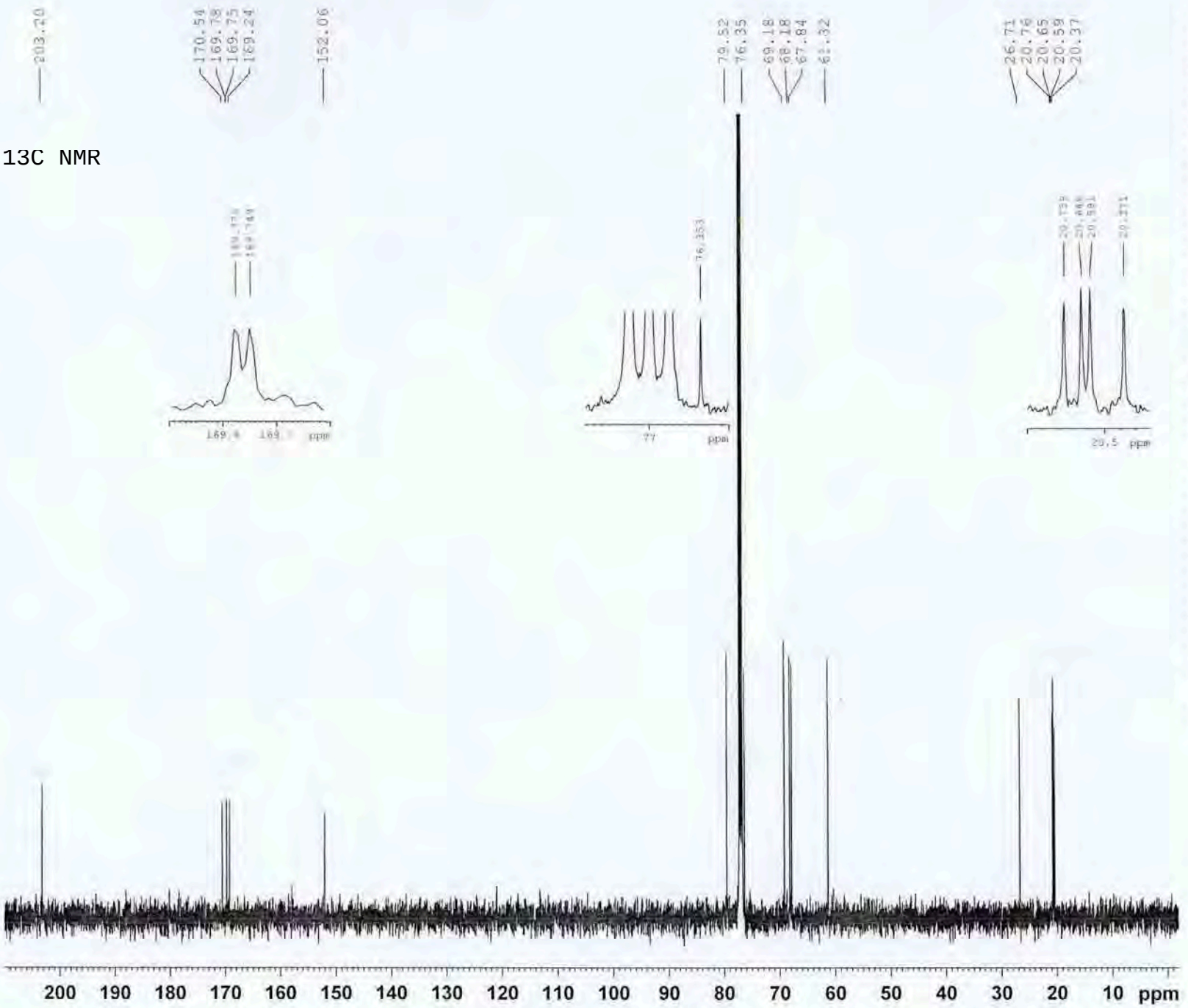


NAME	MB-146
EXPNO	5
PROCNO	1
Date_	20110221
Time	13.36
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesyph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4360.465 Hz
FIDRES	4.258267 Hz
AQ	0.1174687 sec
RG	228
DW	114.667 usec
DE	6.50 usec
TE	298.0 K
D0	0.00010276 sec
D1	2.00000000 sec
D8	1.00000000 sec
IN0	0.00022935 sec
CHANNEL f1	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618114 MHz
ND0	1
TD	128
SFO1	500.2618 MHz
FIDRES	34.066265 Hz
SW	8.716 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600082 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600095 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

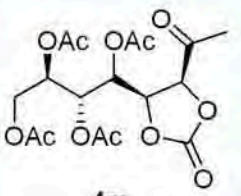


4m

13C NMR



NAME	MB-146
EXPNO	2
PROCNO	1
Date_	20110221
Time	12.48
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	518
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1620
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618114 MHz
SI	32768
SF	125.7904800 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

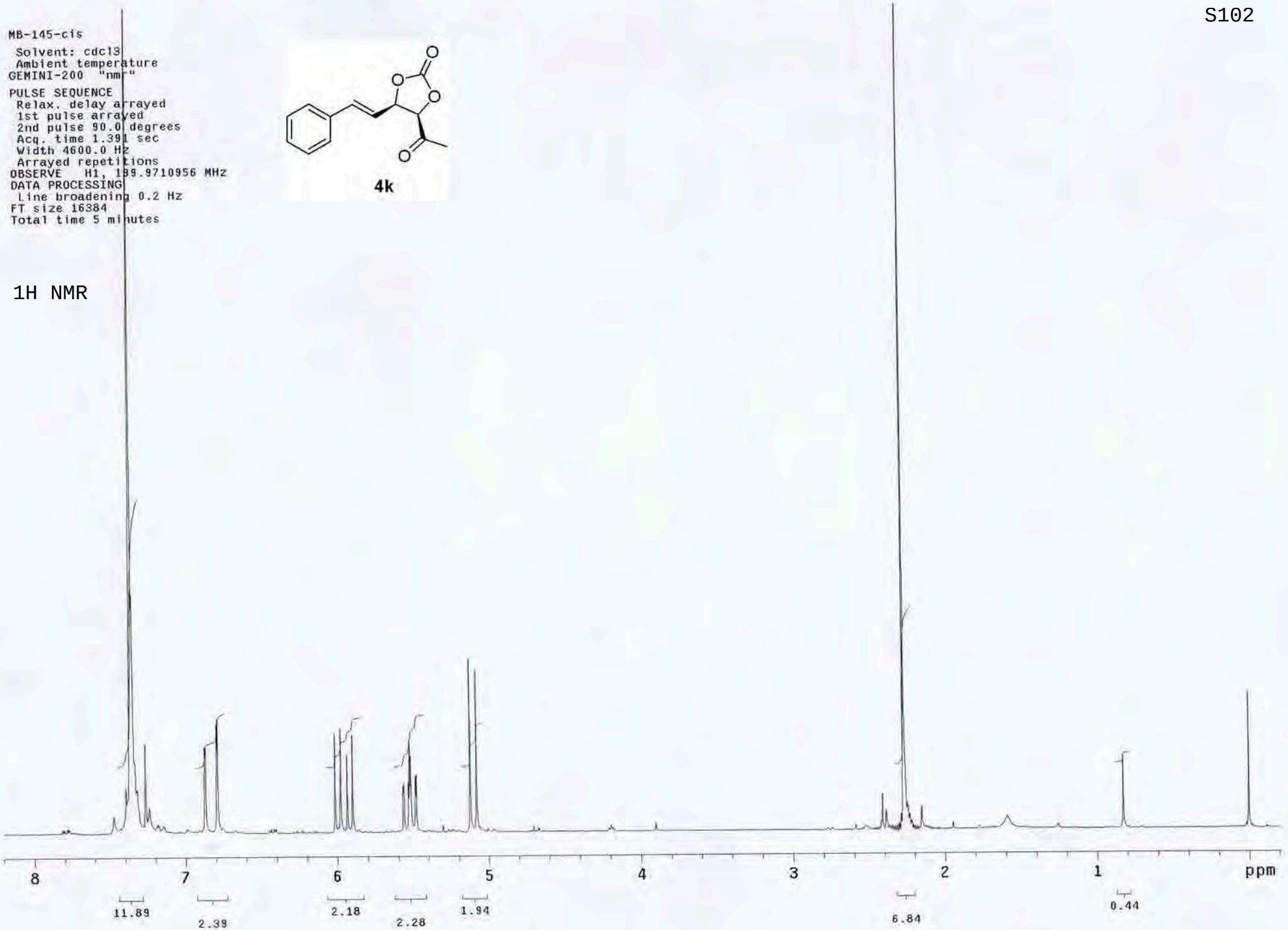


4m

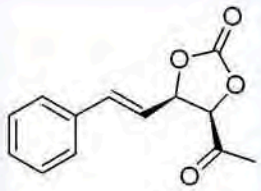
MB-145-cis
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710956 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 5 minutes

4k

1H NMR

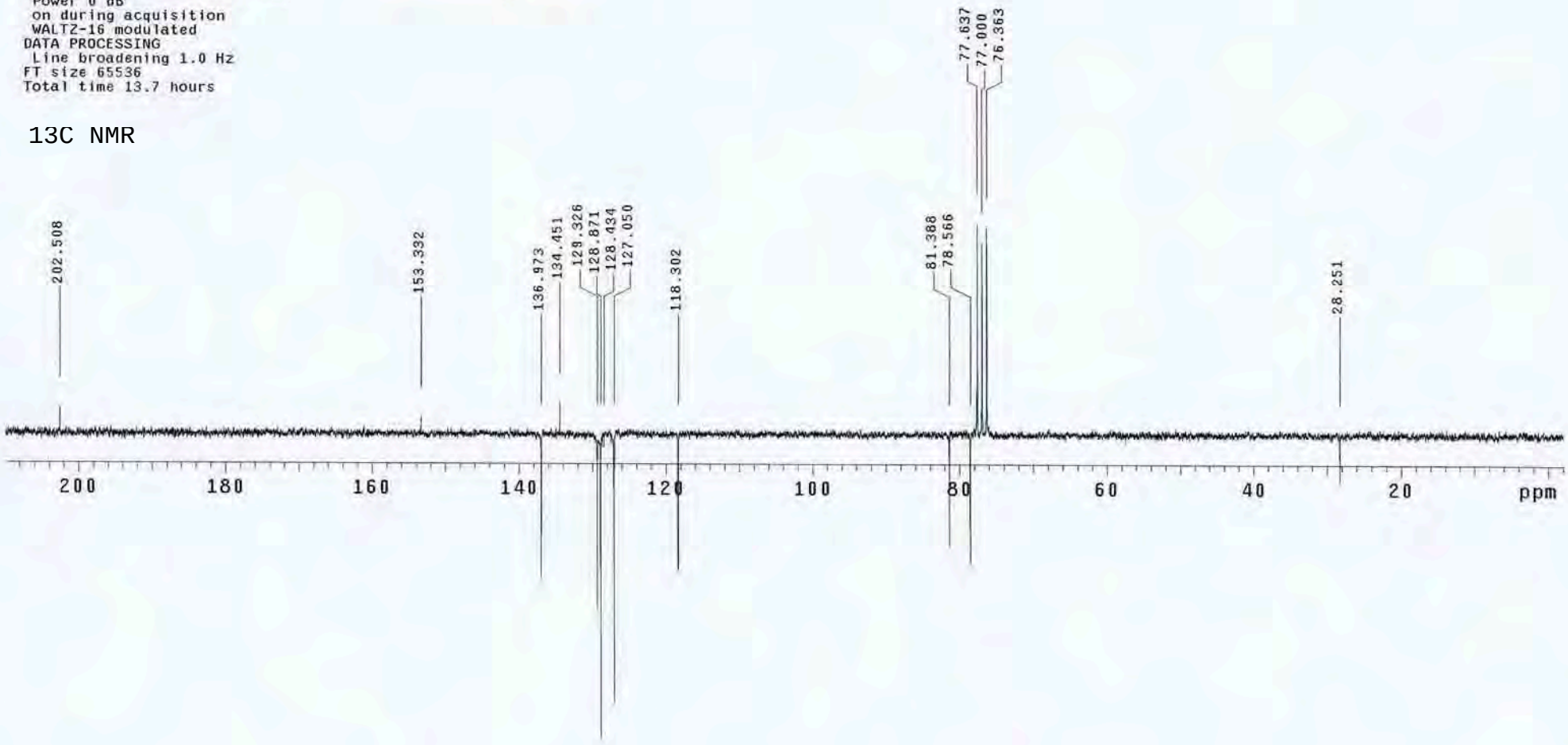


MB-145-cis
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827789 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 13.7 hours



4k

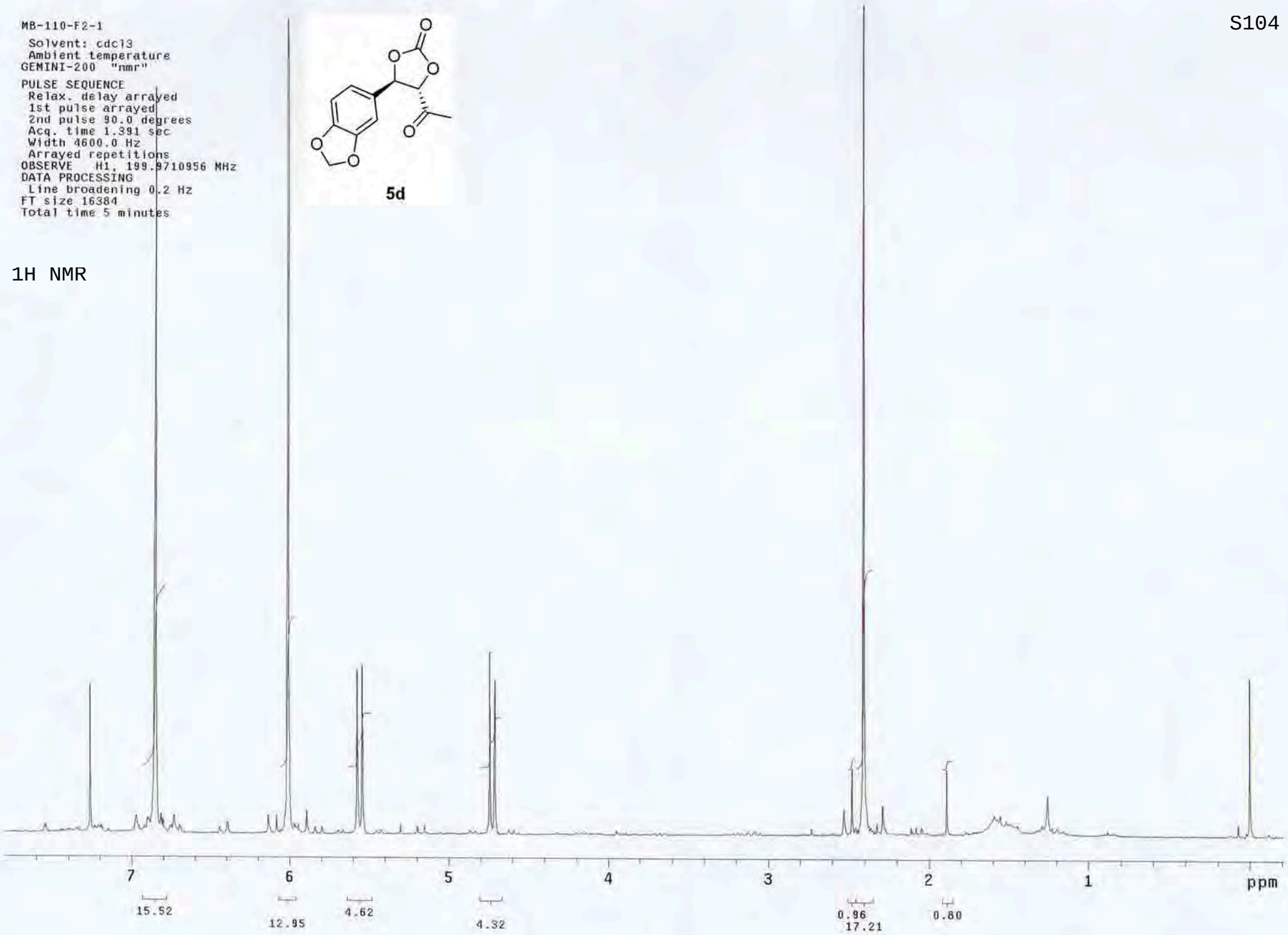
13C NMR



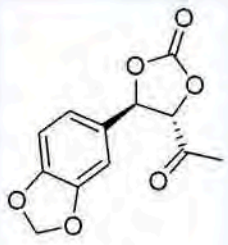
MB-110-F2-1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710956 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 5 minutes

5d

1H NMR

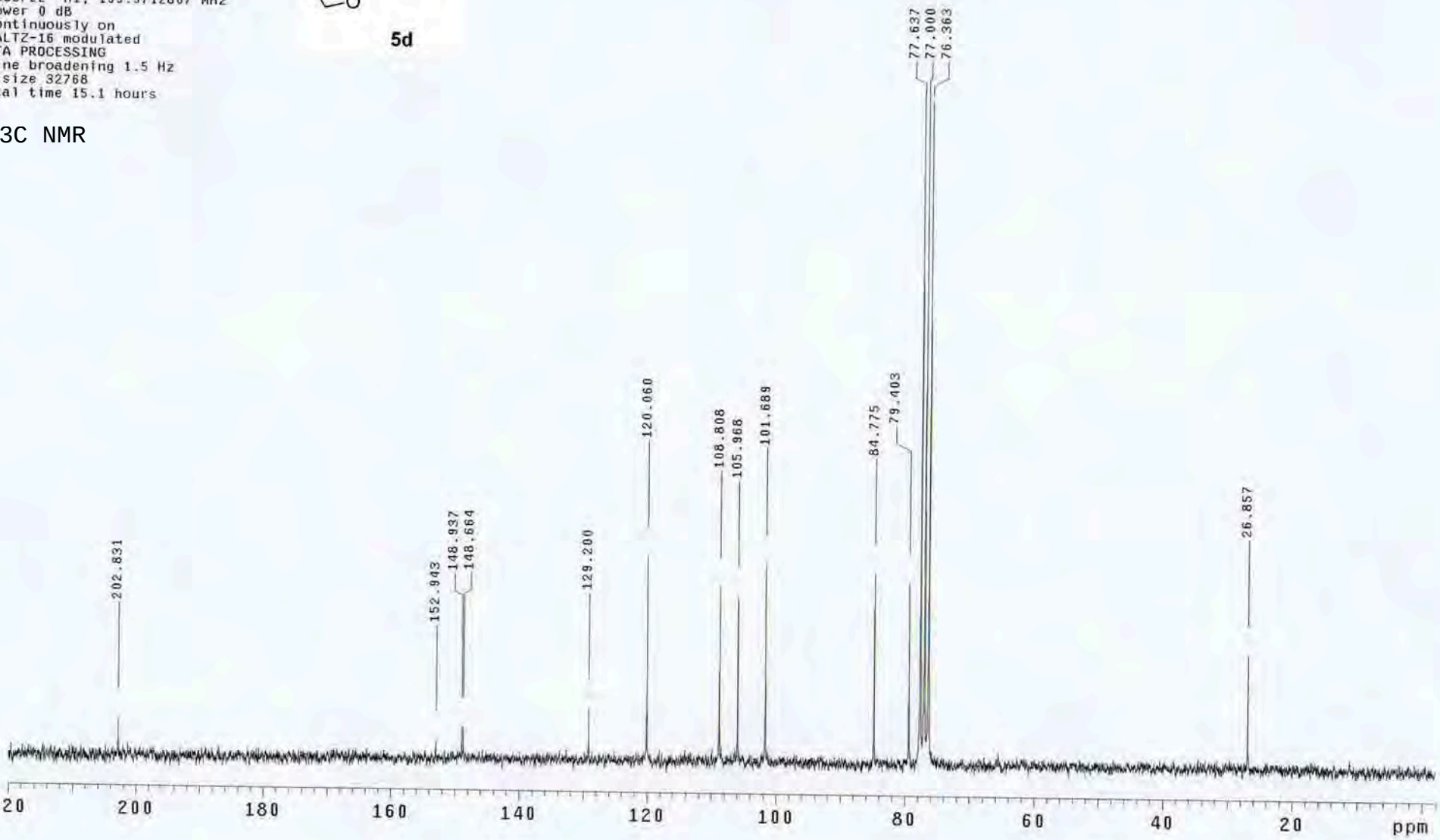


MB-110-F2-1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827773 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 15.1 hours

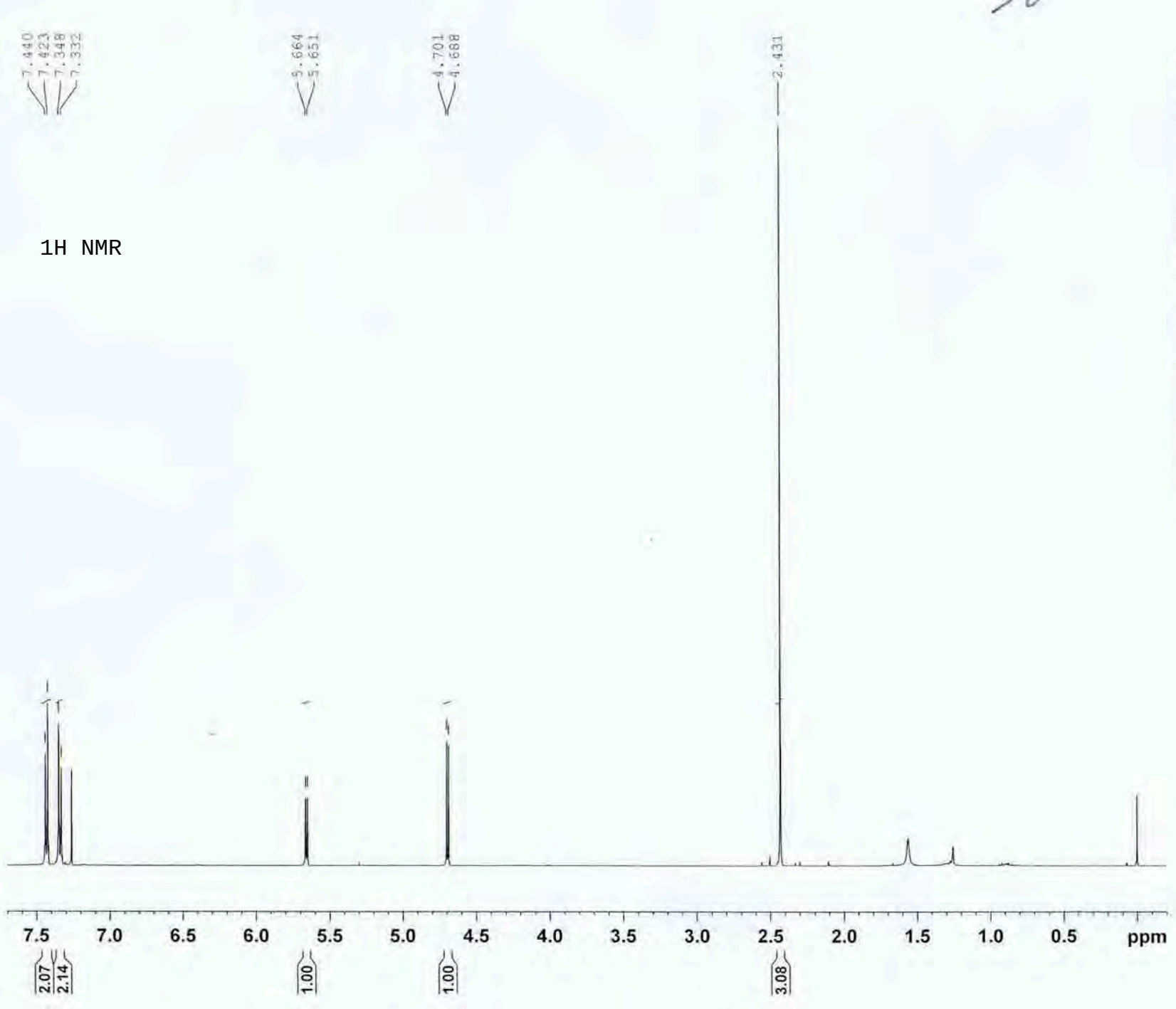


5d

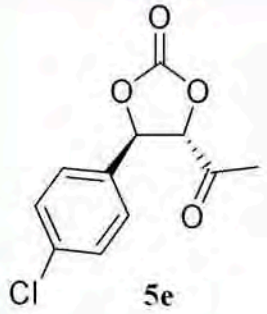
13C NMR



1H NMR

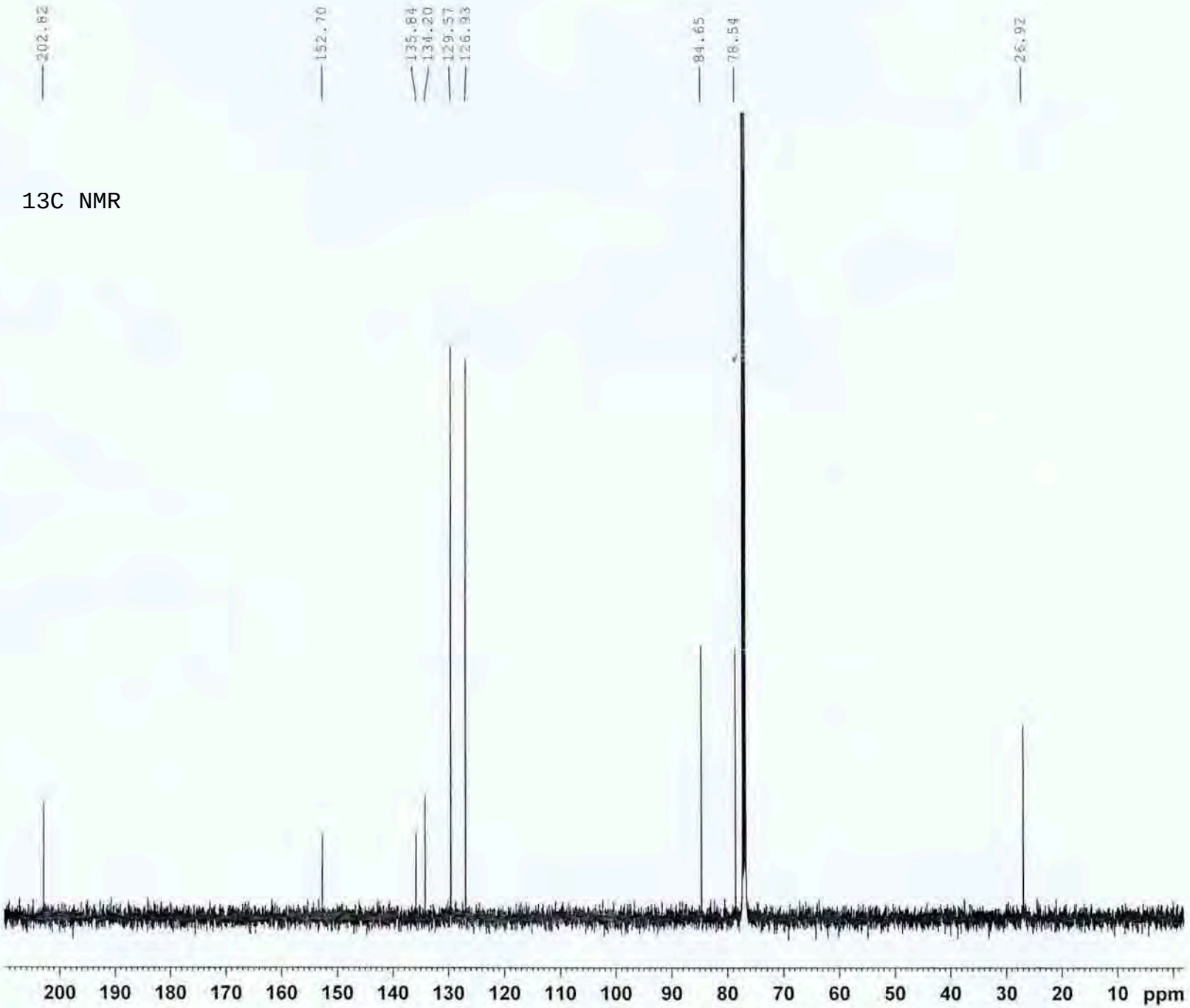


NAME	MB-109-F1
EXPNO	1
PROCNO	1
Date_	20100906
Time	13.51
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4496.403 Hz
FIDRES	0.137219 Hz
AQ	3.6438515 sec
RG	322
DW	111.200 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618905 MHz
SI	32768
SF	500.2600135 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

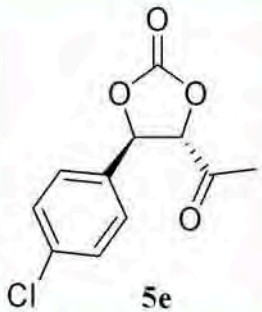


5e

13C NMR

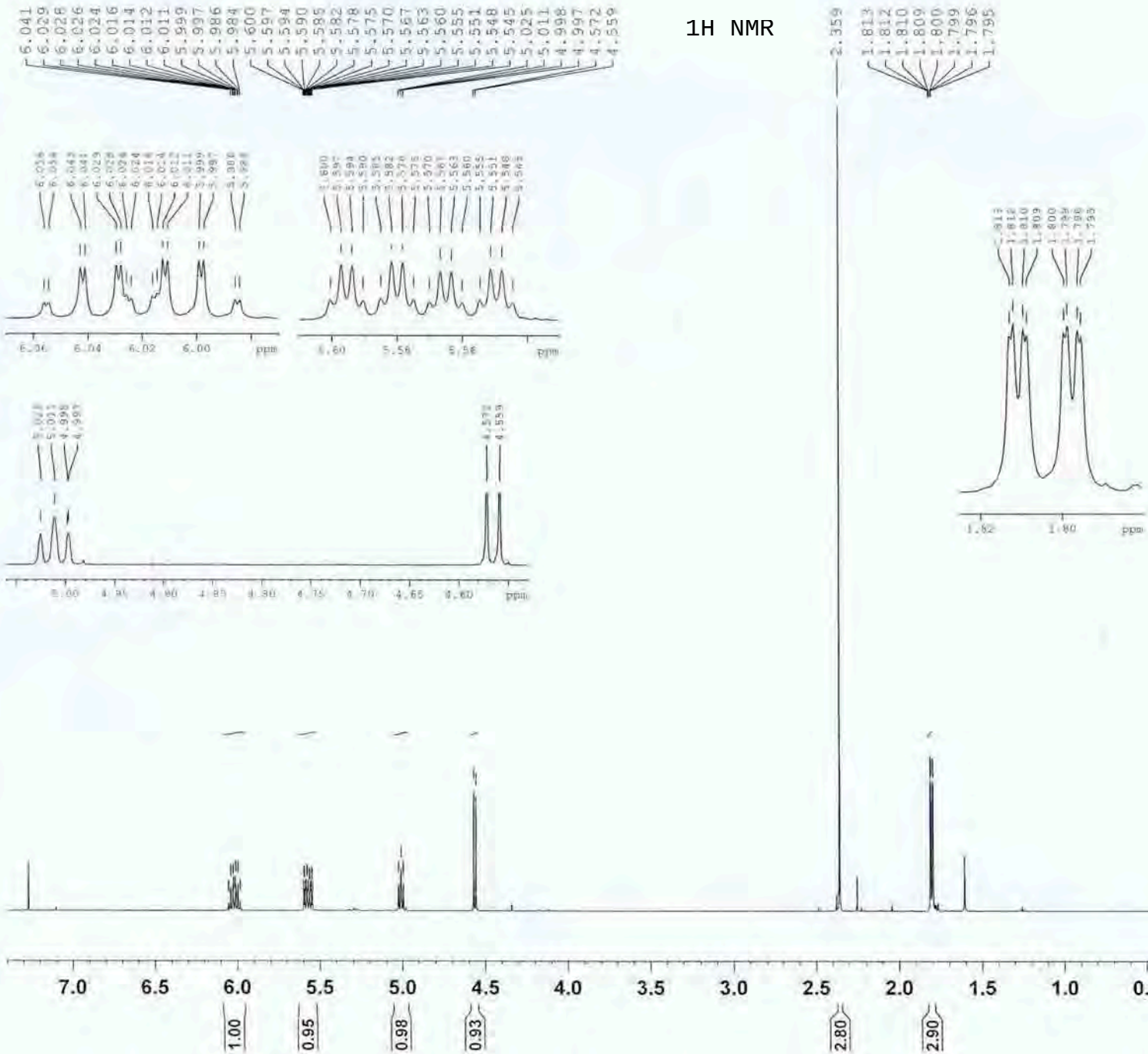


NAME	MB-109-F1
EXPNO	2
PROCNO	1
Date_	20100906
Time	13.55
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	514
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 se
RG	1620
DW	16.800 us
DE	6.50 us
TE	298.0 K
D1	2.00000000 se
D11	0.03000000 se
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 us
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MH
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 us
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618905 MH
SI	32768
SF	125.7904802 MH
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



5e

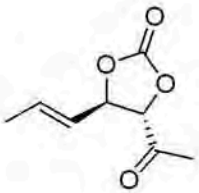
1H NMR



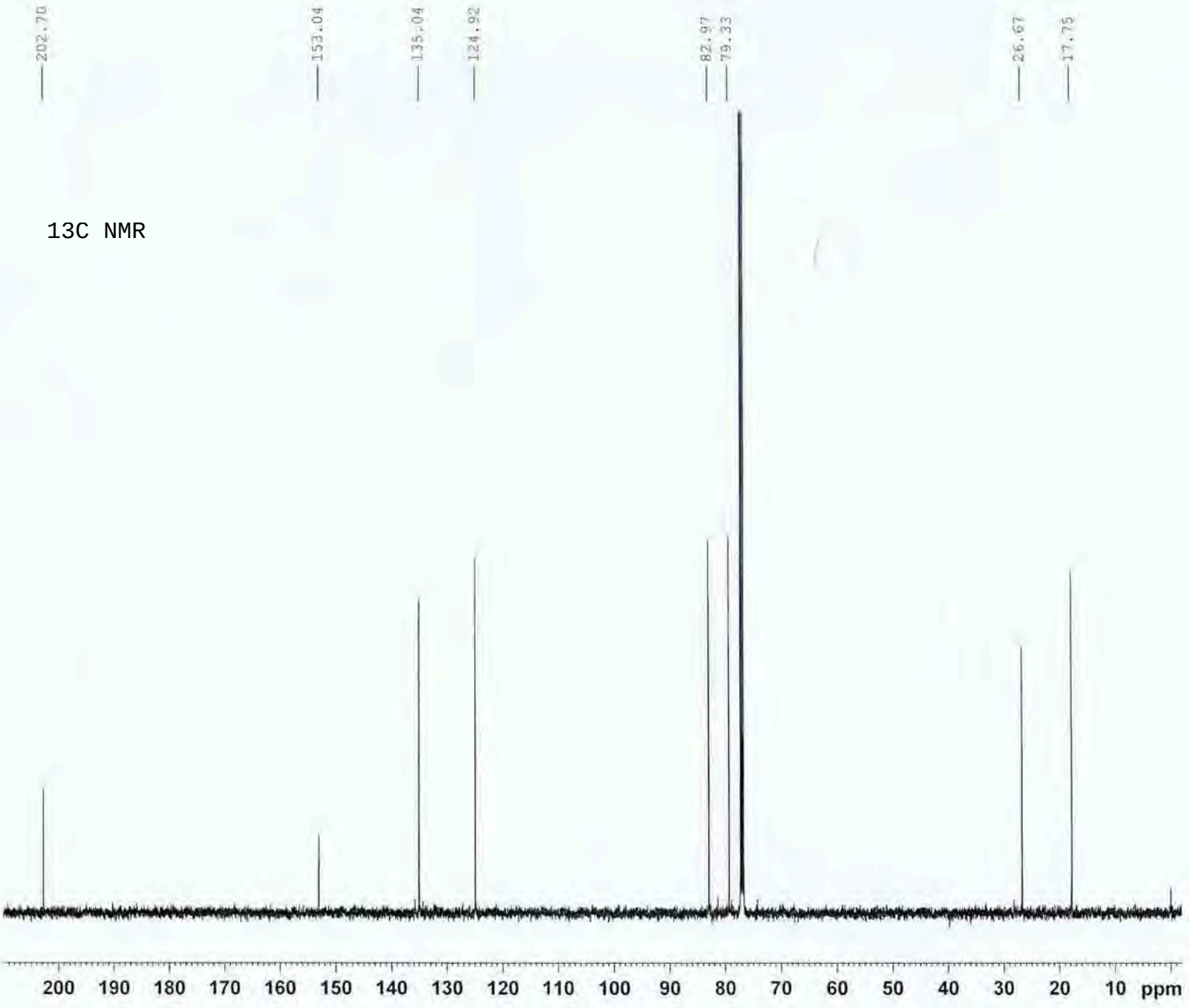
NAME	MB-117-F1
EXPNO	1
PROCNO	1
Date_	20101008
Time	10.57
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4370.629 Hz
FIDRES	0.133381 Hz
AQ	3.7487092 sec
RG	256
DW	114.400 use
DE	6.50 use
TE	298.0 K
D1	2.00000000 sec
TD0	1

CHANNEL f1

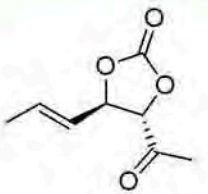
NUC1	1H
P1	9.35 use
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618329 MHz
SI	32768
SF	500.2600097 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



5j



NAME	MB-117-F1
EXPNO	2
PROCNO	1
Date_	20101008
Time	11.13
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	352
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
========	CHANNEL f2 ========
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618330 MHz
SI	32768
SF	125.7904820 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



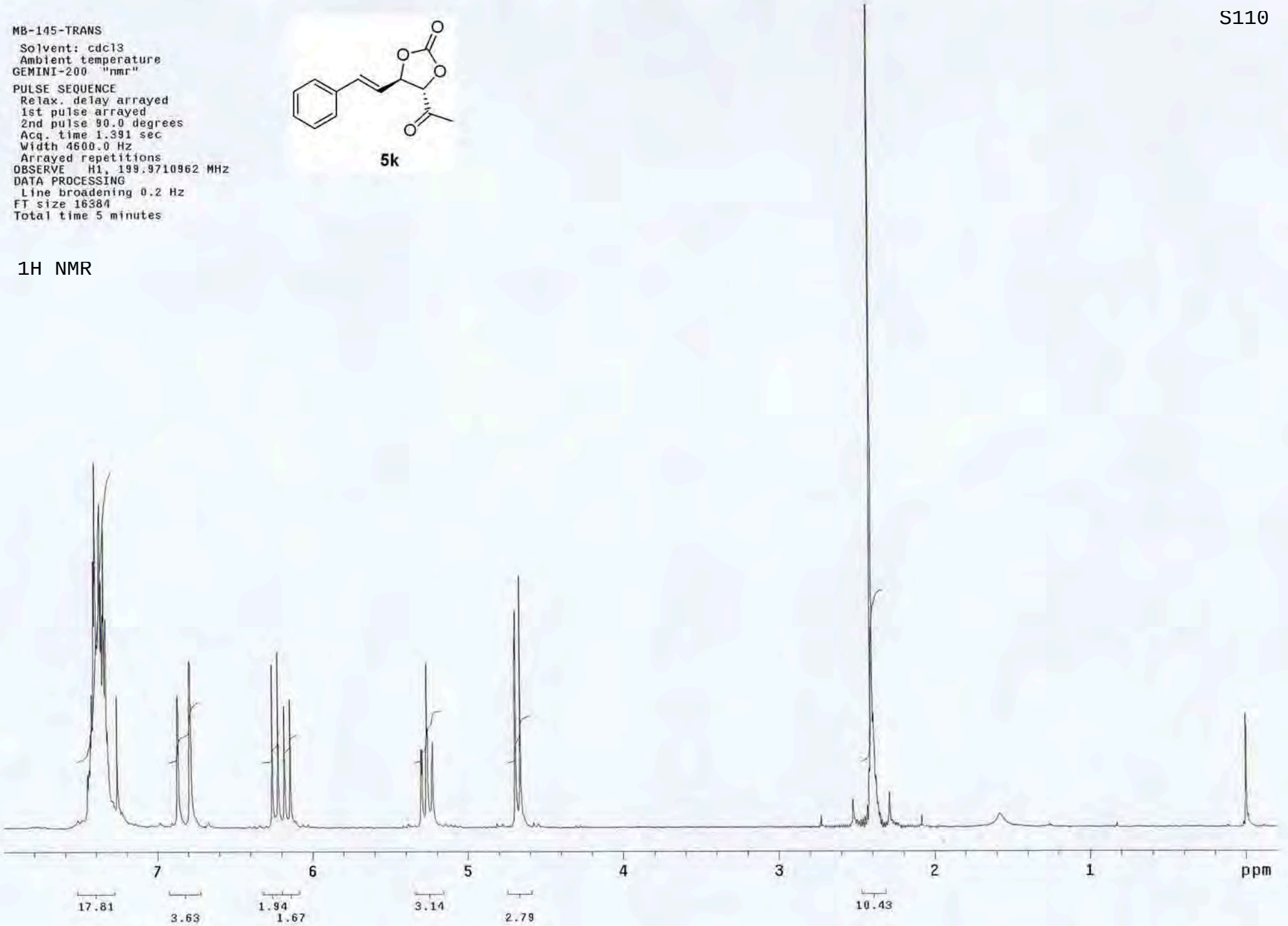
5j

MB-145-TRANS
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

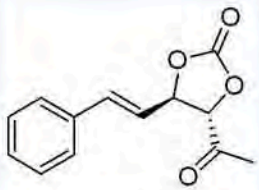
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710962 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 5 minutes

5k

1H NMR

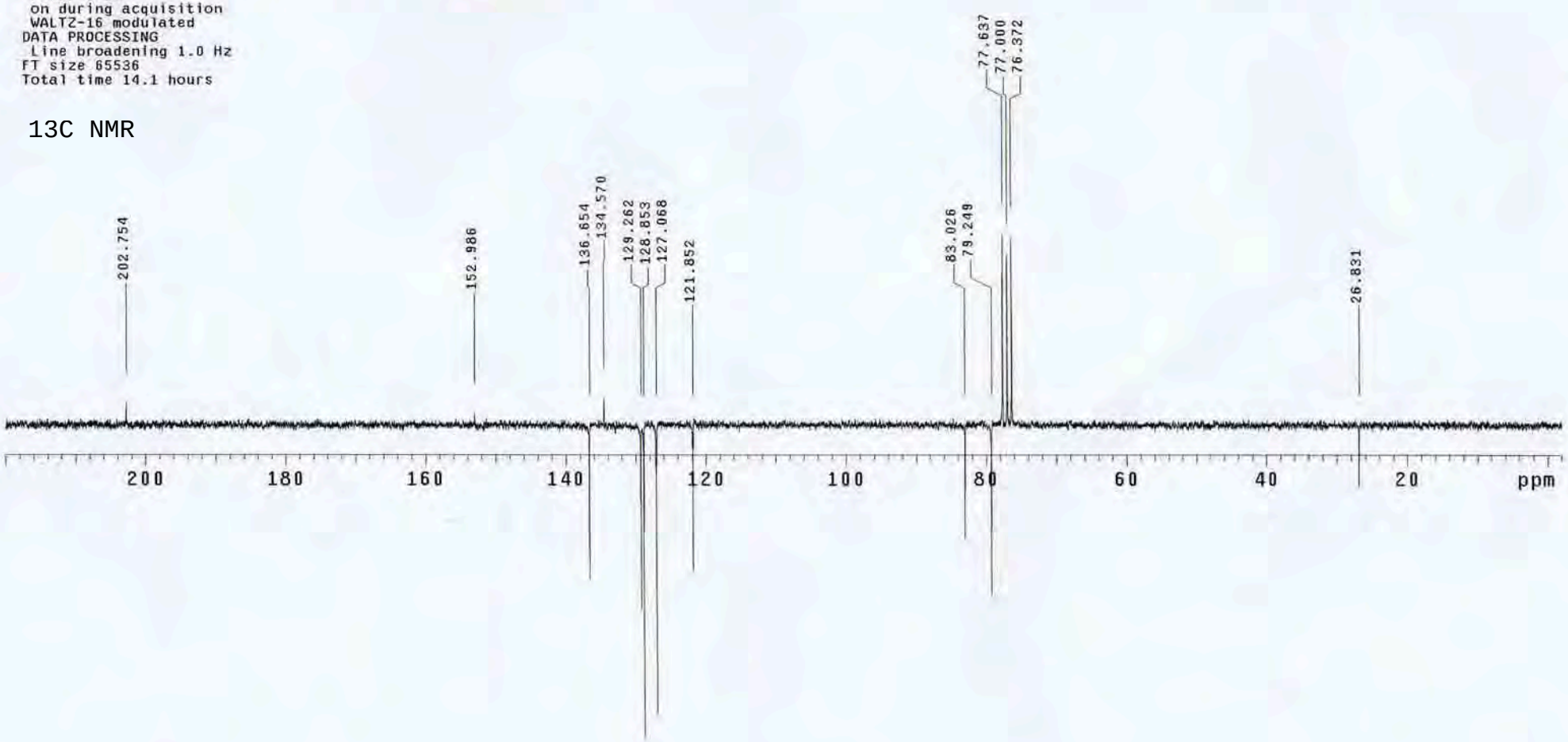


MB-145-TRANS
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827785 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 14.1 hours

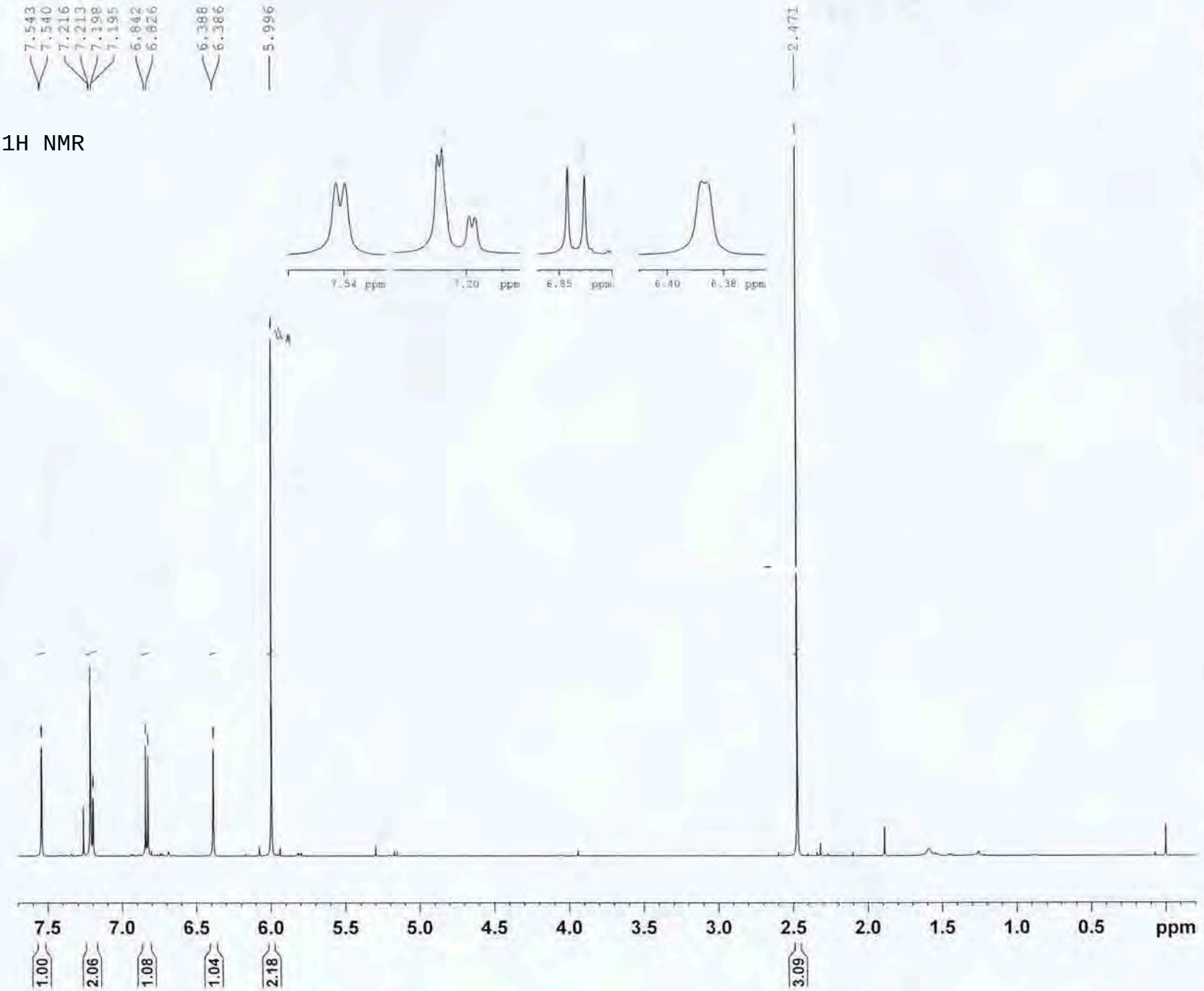


5k

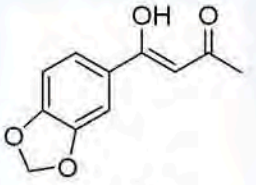
13C NMR



1H NMR

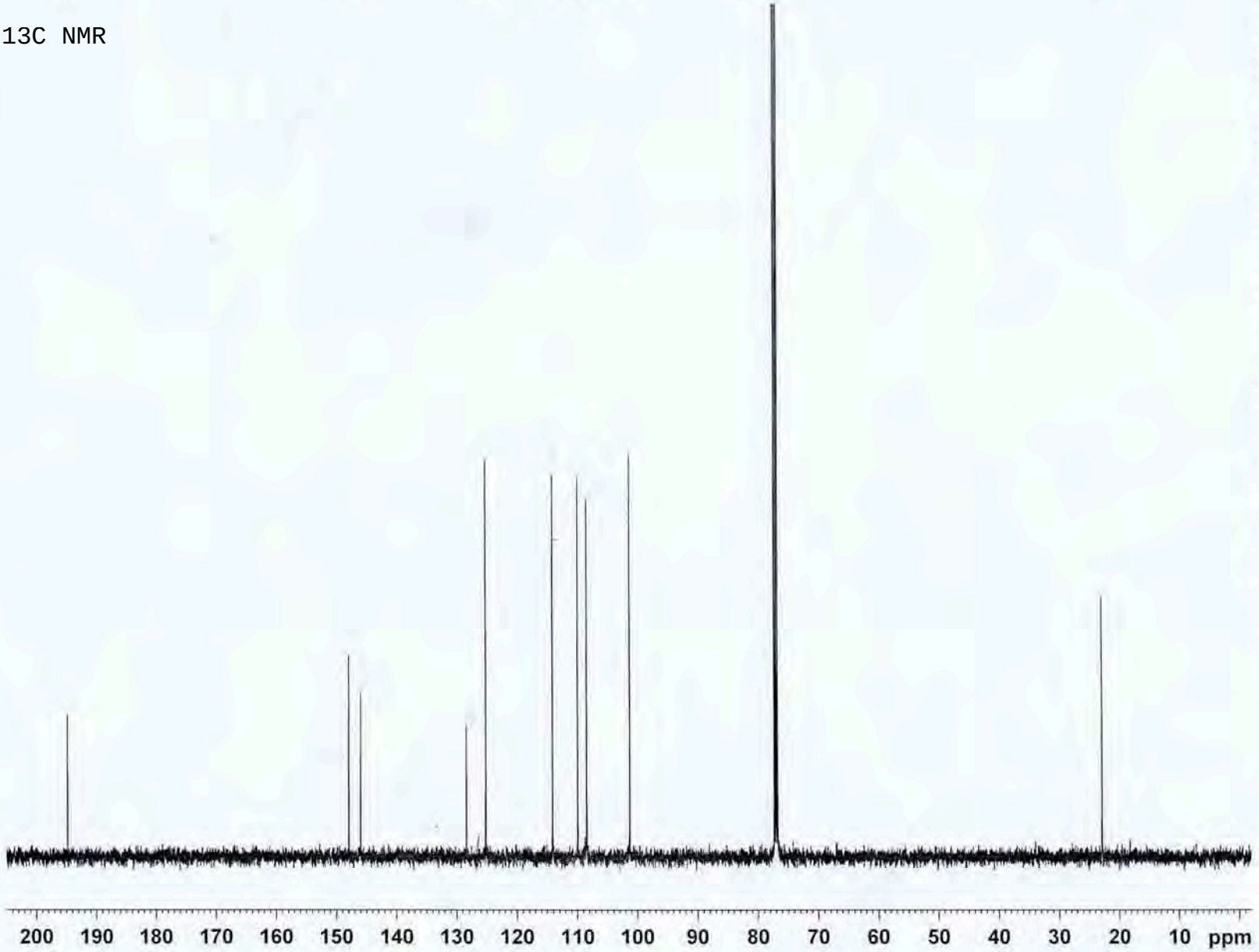


NAME	MB-110-F1-1
EXPNO	1
PROCNO	1
Date_	20110420
Time	10.58
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4734.849 Hz
FIDRES	0.144496 Hz
AQ	3.4603508 sec
RG	228
DW	105.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618965 MHz
SI	32768
SF	500.2600141 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

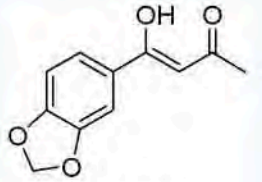


6d

13C NMR



NAME	MB-110-F1-1
EXPNO	2
PROCNO	1
Date_	20110420
Time	11.11
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	345
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	645
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618965 MHz
SI	32768
SF	125.7904810 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



6d