Supplementary Material (ESI) for Chemical Communications

Supporting Information

Remote induction of asymmetry in [13]-macro-dilactone topology by a single stereogenic center

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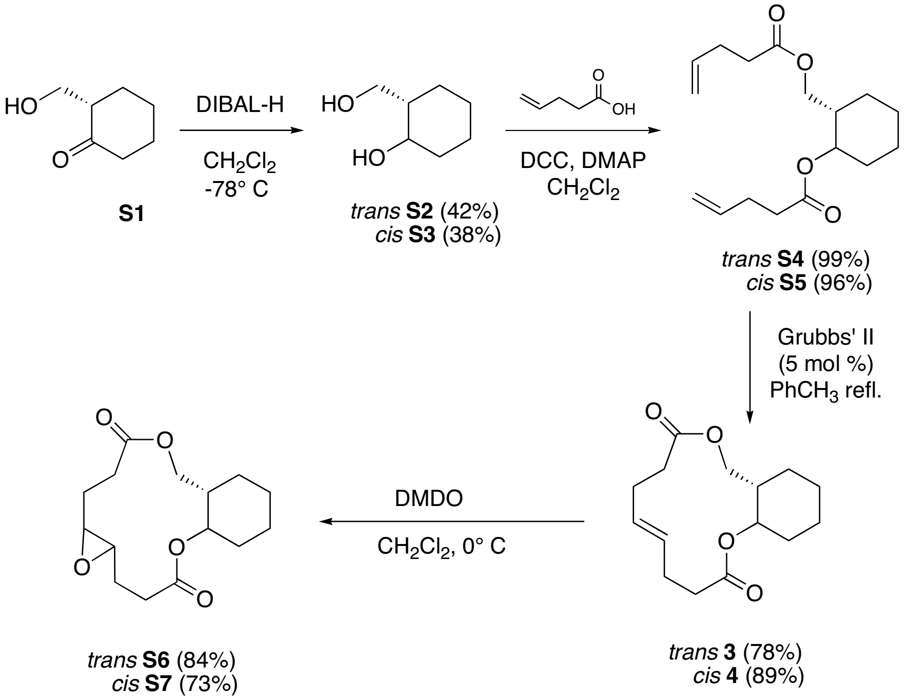
Total Pages: 69

Contents

1. Additional experimental procedures.

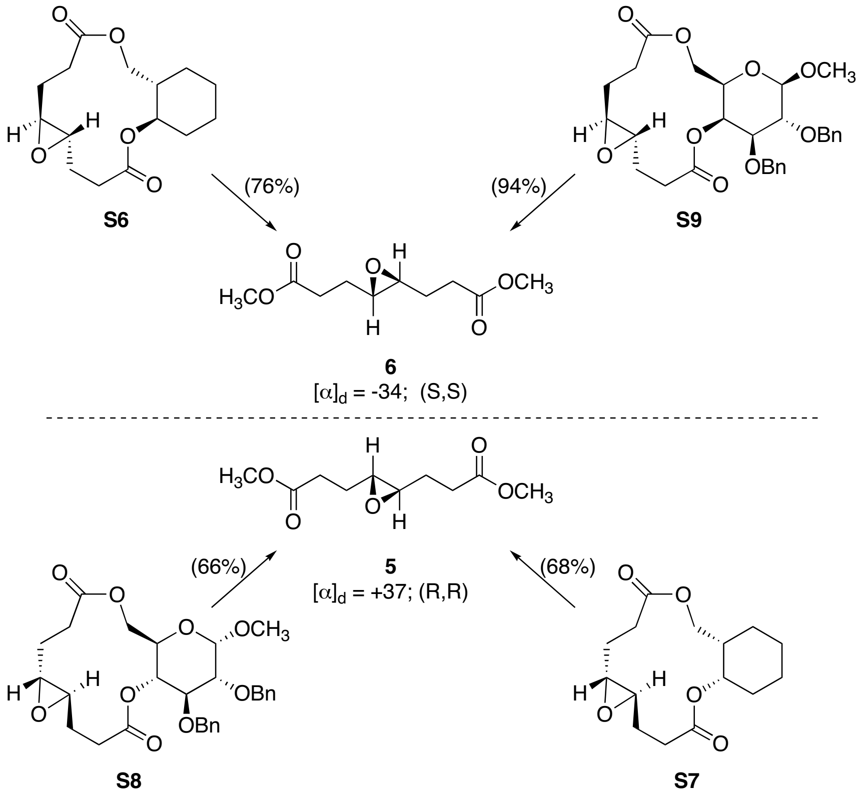
1a. Preparation of 3 and 4 and subsequent epoxidation.



Scheme S-1. Preparation of cyclohexane-fused epoxy macrodiolides **S6** and **S7**.

The synthesis of **3** and **4** was achieved from diastereomeric 1,3-diols **S2** and **S3** using the diacylation/RCM protocol we previously reported (see Scheme S-1).[1] Using known chemistry the (*S*)-proline catalyzed aldol condensation of cyclohexanone with formaldehyde in DMSO gave β-hydroxyketone **S1**[2] and established the stereochemical complement to the D-pyranose C-5 [carbohydrate nomenclature] bridgehead carbon in **1** and **2** (structures shown in manuscript). A diastereomeric mixture of the desired 1,3-diols **S2** and **S3** were obtained with the DIBAL-H reduction of **S1**. Separation of **S2** and **S3** was achieved via gravity column chromatography using 7% MeOH in CH_2Cl_2 as eluent. Stereochemistry of the 1,3 diols was assigned in accordance with distinct 2D NOESY signatures. The *cis* cyclohexane intermediates displayed nOe interactions between the protons on C1 and C2, while the *trans* cyclohexane derivatives did not. The remaining steps shown in scheme S-1 were performed in parallel for the *cis/trans* isomers. DCC-mediated diacylation with 4-pentenoic acid gave diesters **S4** and **S5** (99% and 96% yields). Ring closing metathesis (RCM) was performed on the diesters to yield *cis*- and *trans*-fused [13]-macro-dilactones **3** (78%) and **4** (89%) in single isomeric forms, respectively. As in the carbohydrate-derived series, the *cis*-fused bicycle (**4**) was a white solid while the *trans*-fused bicycle (**3**) was a clear, colorless syrup. Despite the apparent similarities to the carbohydrate analogues, the olefin geometries of **3** and **4** were unable to be verified through ^3J coupling constant values due to overlap of the vinylic protons in the ^1H spectra. The investigation continued under the assumption that the olefins were in an *E* (*trans*) configuration as we had observed previously for pyranose-fused [13]-macro-dilactones. Dimethyldioxirane (DMDO) epoxidation of **3** and **4** gave tricycles **S6** and **S7** in 92% and 76% yields. The epoxidation proved to be diastereoselective in each case.

1b. Determination of epoxide stereochemistry of S6 and S7.

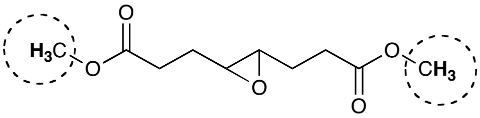


Scheme S-2. Transesterification of tricycles **S6** and **S7** were conducted in parallel under Zemplén conditions ($NaOCH_3$/MeOH) to afford one of two enantiomeric epoxy daughter fragments **5** and **6** as identified by comparison to previously characterized **5** and **6** afforded from the transesterification of **S8** and **S9**.

We next set about confirming the stereochemical configuration of the epoxidation products **S6** and **S7** based on our earlier work. The approach we took mirrored the one used to determine stereochemistry of epoxidation in the pyranose-fused [13]-macro-dilactones. Transesterification of the major diastereomers isolated from chromatography of **S6** and **S7** conducted under Zemplén conditions yielded respective optically active daughter fragments **6** and **5** (scheme S-2). The absolute configurations of **6** and **5** were determined to be the expected *S*,*S* and *R*,*R*, respectively. The absolute configurations were assigned by comparison to polarimetry and chiral shift reagent data (see part 1c.). We have previously reported on the assignment of absolute configuration of **5** and **6** prepared from carbohydrate-derived analogues **S8** and **S9**.[1] The earlier assignment was supported by chiral HPLC, and ^{1}H NMR chemical shift reagent experiments. X-ray crystallographic data for **S9**. The present results jointly confirmed the *E* olefin geometry of **3** and **4** (*Z* olefins would have generated *meso* epoxy-dioates) as well as the minimal effect the carbohydrate distinctions have on the facial selectivity.

1c. Chiral shift reagent data (^{1}H):

Each sample analyzed was exposed to complexation with chiral shift reagent europium tris[3-(heptafluoropropylhydroxymethylene)-(+)-camphorate] in $CDCl_3$ and ^{1}H NMR spectrum for 'racemate' was collected at 500 MHz and spectra for 'via **S6**' and 'via **S7**' were collected at 300 MHz.



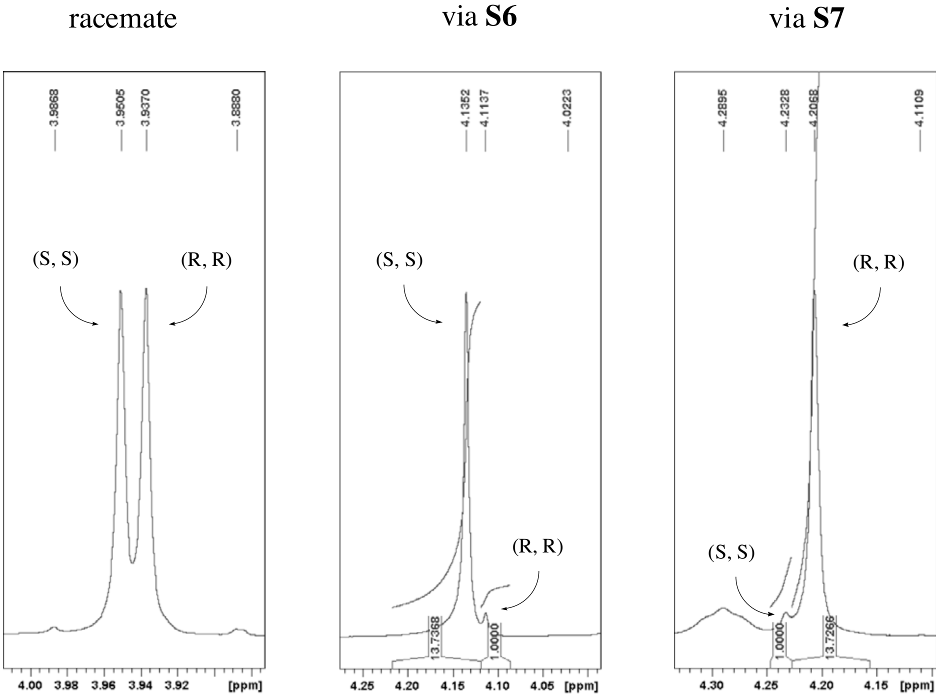
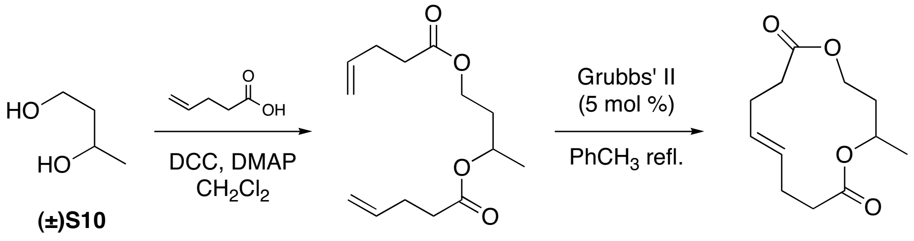


Figure S:1. The methoxy group proton signal from the ^{1}H NMR spectra of three samples of 4,5-epoxydioate as shown when complexed with $Eu(hfc)_3$-(+) additive. Racemate). as prepared and analyzed previously in reference 1. Via **S6**). epoxy fragment as accessed from transesterification of **S6**. Via **S7**). epoxy fragment as accessed from transesterification of **S7**.
Conditions:
Racemate[1]*: 5.1 mg of epoxide and 5.1 mg of* $Eu(hfc)_3$*-(+) dissolved in* $CDCl_3$
Via ***S6****: 3.9 mg of epoxide and 4.3 mg of* $Eu(hfc)_3$*-(+) dissolved in* $CDCl_3$
Via ***S7****: 3.6 mg of epoxide and 5.6 mg of* $Eu(hfc)_3$*-(+) dissolved in* $CDCl_3$

Note: Upon complexation of the epoxides derived from either **S6** or **S7** to chiral shift reagent europium tris[3-(heptafluoropropylhydroxymethylene)-(+)-camphorate] in $CDCl_3$ a very small amount (ca. 13:1) of the opposite enantiomer was revealed. Because the ratio of enantiomers observed appeared to be in identical proportions for **S6** and **S7**, we presumed that this arose from the enantioselective limitations of the preparation of β-hydroxyketone **S1**.

1d. Preparation of (±)7:



(±)S10

(±)S11 (87%)

(±)7 (72%)

Scheme S-3. Preparation of methyl substituted macrodiolides **7** and **ent-7**.

Macrodiolides **7** and **ent-7** were prepared together as a racemate from racemic diol **S10** in a manner analagous to the preparation of **3** and **4** from diols **S2** and **S3**, respectively (scheme S-3).

2. Experimental:

Preparation of diols S2 and S3:

β-hydroxyketone **S1** (0.0860 g, 0.671 mmol) was dissolved in dry CH_2Cl_2 (5 mL) and cooled to –78 °C with stirring under N_2. Added dropwise over about 25 minutes was 2.68 mL of a solution of DIBAL-H in CH_2Cl_2 (1.0 M, 4 eq.). After another 40 min the reaction mixture was quenched with dropwise addition of H_2O (2-3 mL). The resultant emulsion was broken with the addition of conc. HCl (~2 mL) and the biphasic mixture was allowed to stir for ~5 min. The organic layer was collected and due to the poor partition coefficient of these compounds for the organic phase the remaining aqueous layer was extracted with 5 x 25 mL CH_2Cl_2. The organic layers were combined, dried with Na_2SO_4, concentrated, and the residue was purified by gravity column chromatography using 7% MeOH in DCM giving *trans* **S2** (0.0412 g, 42%) as a clear, colorless oil, and *cis* **S3** (0.0384 g, 38%) as a clear colorless oil.

(1*R*,2*S*)-2-(hydroxymethyl)cyclohexanol (S2). R_f 0.38 (7:93, MeOH:CH_2Cl_2); $[\alpha]_D$ –3.84 (*c* 1.91, $CHCl_3$); IR (KBr) cm^{-1}:3343.22, 2927.93, 2856.06, 1449.40, 1351.83, 1261.73, 1192.99, 1087.93, 1063.38, 1040.23, 1014.84, 974.10, 801.50; ^1H NMR ($CDCl_3$): 500 MHz δ 3.81 (d, J = 3.0 Hz, 1H), 3.71-3.65 (m, 2H), 3.60 (ddd, J = 9.4, 9.4, 2.8 Hz, 1H), 3.47 (dddd, J = 9.8, 9.8, 3.5, 3.5 Hz, 1H), 1.94-1.88 (m, 1H), 1.74-1.72 (m, 1H), 1.66-1.63 (m, 1H), 1.59-1.55 (m, 1H), 1.54-1.47 (m, 1H), 1.31-1.15 (m, 3H), 0.89 (dddd, J =12.6, 12.6, 12.6, 3.8 Hz, 1H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 76.5, 68.7, 46.2, 35.4, 27.4, 25.2, 24.6.

(1*S*,2*S*)-2-(hydroxymethyl)cyclohexanol (S3). R_f 0.34 (7:93, MeOH:CH_2Cl_2); $[\alpha]_D$ +24.36 (*c* 1.26, $CHCl_3$); IR (KBr) cm^{-1}: 3351.74, 2929.98, 2857.08, 1447.20, 1261.49, 1190.89, 1093.80, 1020.71, 973.85, 804.67; ^1H NMR ($CDCl_3$): 500 MHz δ 4.13 (s, 1H), 3.76-3.67 (m, 2H), 2.92 (brd s, 1H), 2.81 (brd s, 1H), 1.78 (dddd, J = 13.3, 4.4, 4.4, 4.4 Hz, 1H), 1.68-1.58 (m, 4H), 1.54-1.38 (m, 3H), 1.33-1.25 (m, 1H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 70.0, 66.3, 42.6, 33.1, 25.1, 23.8, 20.7.

General procedure for diester preparation:

To an ice cold, stirred solution of the appropriate diol in dry CH_2Cl_2 (10 mL) under N_2, 2.2 eq. of 4-pentenoic acid, 0.6 eq. of DMAP, and 2.4 equivalents of DCC were added in that order. The reaction was allowed to warm to room temperature and stir overnight. The reaction mixture was diluted with 10 mL hexanes and filtered through a pad of celite which and washed with 100 mL of EtOAc. The organic layer was concentrated under reduced pressure and purified by column chromatography using 17:3 hexanes:EtOAc as eluent to give the corresponding diester.

(1*R*, 2*S*)-(methyl)cyclohexane-1,7-diyl-dipent-4-enoate (S4). R_f 0.65 (4:1, hexanes:EtOAc); $[\alpha]_D$ –38.70 (*c* 2.83, $CHCl_3$); IR (KBr) cm^{-1}: 3080.12, 2936.71, 2861.58, 1737.14, 1641.54, 1450.18, 1355.03, 1259.50, 1173.57, 1100.06, 1063.07, 1012.64, 913.97, 802.02; ^{1}H NMR ($CDCl_3$): 500 MHz δ 5.84-5.75 (m, 2H), 5.06 (s, 1H), 5.02 (s, 1H), 5.00 (d, J = 0.8 Hz, 1H), 4.98 (dd, J = 10.0 Hz, 1H), 4.64 (ddd, J = 10.2, 10.2, 4.3 Hz, 1H), 4.05-3.98 (m, 2H), 2.41-2.34 (m, 8H), 2.03-2.01 (m, 1H), 1.85-1.68 (m, 4H), 1.34-1.20 (m, 4H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 173.2, 172.6, 136.9, 136.8, 115.6 (2), 73.3, 65.2, 41.8, 33.9, 33.6, 31.9, 29.1, 29.0, 28.4, 25.0, 24.5; EMS $[M+Na]^+$ *m/z* calcd for $C_{17}H_{26}O_4Na^+$ 317.1723, found 317.1716.

(1*S*, 2*S*)-(methyl)cyclohexane-1,7-diyl-dipent-4-enoate (S5). R*f* 0.65 (4:1, hexanes:EtOAc); $[\alpha]_D$ +45.88 (*c* 3.28, $CHCl_3$); IR (KBr) cm^{-1}: 3080.31, 2936.10, 2860.85, 1736.75, 1641.77, 1448.70, 1355.80, 1259.74, 1173.27, 1129.89, 1182.27, 992.57, 914.59, 804.84; ^{1}H NMR ($CDCl_3$): 500 MHz δ 5.86-5.76 (m, 2H), 5.10 (s, 1H), 5.06 (dd, J = 6.7, 1.2 Hz, 1H), 5.03 (dd, J = 6.6, 1.5 Hz, 1H), 5.00 (dd, J = 5.4, 0.8 Hz, 1H), 4.98 (dd, J = 5.5, 1.0 Hz, 1H), 3.97-3.88 (m, 2H), 2.43-2.33 (m, 8H), 1.93-1.87 (m, 2H), 1.74 (ddd, J = 12.6, 3.5, 3.5 Hz, 1H), 1.56-1.25 (m, 6H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 173.2, 172.5, 136.9, 136.8, 115.6 (2), 69.4, 65.2, 39.5, 34.0, 33.6, 29.8, 29.2, 29.0, 24.8, 24.1, 20.8; EMS $[M+Na]^+$ *m/z* calcd for $C_{17}H_{26}O_4Na^+$ 317.1723, found 317.1715.

Butane-2,3-diyl-dipent-4-enoate ((±)S11). ^{1}H NMR ($CDCl_3$): 400 MHz δ; 5.85-5.75 (m, 2H), 5.06 (d, J = 1.5 Hz, 1H), 5.03-4.97 (m, 4H), 4.13-4.08 (m, 2H), 2.41-2.32 (m, 8H), 1.93-1.79 (m, 2H), 1.23 (d, J = 6.3 Hz, 3H); ^{13}C NMR ($CDCl_3$): 100 MHz δ; 173.0, 172.6, 136.8, 115.6, 67.9, 60.8, 35.0, 33.9, 33.6, 29.0 (2), 20.2.

RCM macrocyclization:

The RCM was conducted as described previously.[1]

(1*R*, 13*S*)-2,11-dioxabicyclo[11.4.0]heptadec-6-ene-3,10-dione (3). R*f* 0.52 (4:1, hexanes:EtOAc); $[\alpha]_D$ −6.98 (*c* 1.95, $CHCl_3$); IR (KBr) cm^{-1}: 2958.71, 2859.79, 1732.82, 1443.91, 1354.73, 1259.88, 1236.43, 1173.11, 1135.76, 1086.71, 1086.71, 1027.81, 960.67, 922.97, 801.69; ^{1}H NMR ($CDCl_3$): 300 MHz δ 5.48-5.32 (m, 2H), 4.64 (ddd, J = 10.8, 10.8, 3.8 Hz, 1H), 4.32 (dd, J = 11.2, 3.6 Hz, 1H), 3.66 (d, J = 11.2 Hz, 1H), 2.45-2.12 (m, 9H), 1.78-1.68 (m, 4H), 1.53-1.17 (m, 4H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 174.4, 172.9, 130.8, 130.0, 74.1, 65.7, 41.5, 34.8, 34.4, 32.0, 30.0, 29.4, 28.0, 25.9, 24.8; EMS $[M+Na]^+$ *m/z* calcd for $C_{15}H_{22}O_4Na^+$ 289.1410, found 289.1404.

(1*S*, 13*S*)-2,11-dioxabicyclo[11.4.0]heptadec-6-ene-3,10-dione (4). m.p. 81-84 °C; R*f* 0.52 (4:1, hexanes:EtOAc); $[\alpha]_D$ +20.95 (*c* 2.50 , $CHCl_3$); IR (KBr) cm^{-1}: 2940.15, 2859.19, 1733.66, 1438.60, 1355.71, 1260.41, 1241.95, 1175.60, 1127.02, 1086.63, 1018.34, 974.13, 870.13, 801.14; ^{1}H NMR ($CDCl_3$): 300 MHz δ 5.48-5.31 (m, 2H), 5.08 (s, 1H), 3.90 (dd, J = 11.2, 11.2 Hz, 1H), 3.76 (dd, J = 10.9, 5.5 Hz, 1H), 2.46-2.08 (m, 9H), 1.95 (brd s, 1H), 1.74 (d, J = 4.7 Hz, 1H), 1.52-1.24 (m, 6H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 174.1, 173.0, 130.8, 130.0, 68.9, 65.0, 37.5, 34.7, 34.4, 29.4, 29.2, 28.1, 24.9, 24.3, 20.7; EMS $[M+Na]^+$ *m/z* calcd for $C_{15}H_{22}O_4Na^+$ 289.1410, found 289.1405.

2-methyl-1,5-dioxacyclotridec-9-ene-6,13-dione ((±)7). ^{1}H NMR ($CDCl_3$): 300 MHz δ; 5.49-5.33 (m, 2H), 5.04 (m, 1H), 4.22 (ddd, J = 11.5, 11.5, 3.5 Hz, 1H), 3.92 (ddd, J = 11.2, 5.1, 2.9 Hz, 1H), 2.46-2.12 (m, 8H), 1.99 (dddd, J = 15.0, 11.6, 5.0, 3.4 Hz, 1H), 1.76 (dddd, J = 14.5, 11.2, 3.4, 3.4 Hz, 1H), 1.26 (d, J = 6.2 Hz, 3H); ^{13}C NMR ($CDCl_3$): 100 MHz δ; 174.1, 173.0, 130.5, 130.0, 67.3, 60.5, 34.7, 34.4, 33.6, 29.1, 28.0, 20.7.

General procedure for epoxidation:

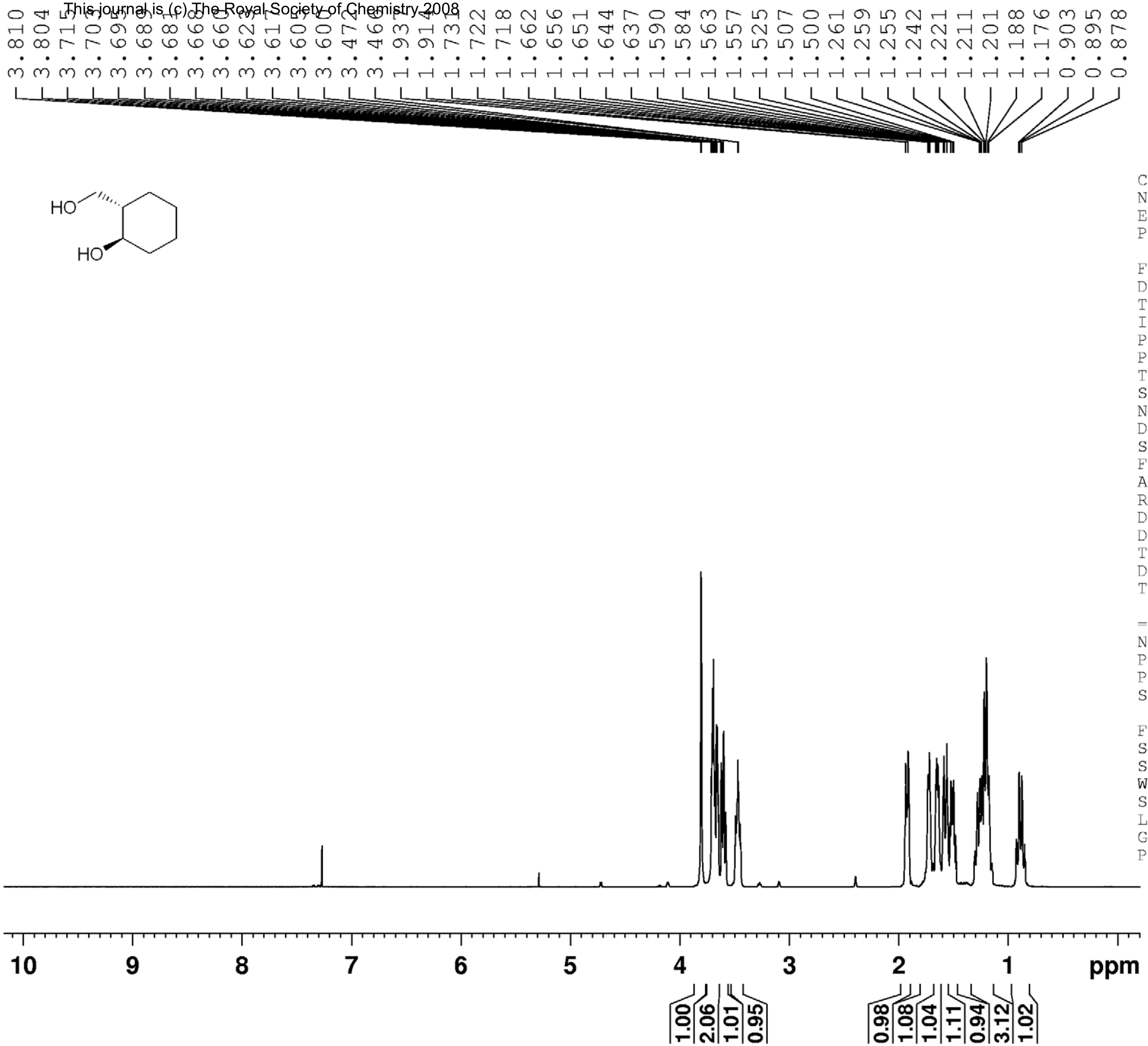
The DMDO epoxidation was conducted as described previously.[1]

(1*R*, 6*S*, 7*S*, 13*S*)-2,11,18-trioxatricyclo[12.4.0.0^{6,7}]octadecane-3,10-dione (S6). m.p. 132-134 °C; R*f* 0.30 (4:1, hexanes:EtOAc); $[\alpha]_D$ +33.50 (*c* 1.94, $CHCl_3$); IR (KBr) cm^{-1}: 2945.24, 2863.10, 1732.39, 1429.36, 1360.12, 1261.91, 1226.82, 1192.36, 1095.55, 1064.45, 977.53, 885.09, 798.39, 702.62; ^{1}H NMR ($CDCl_3$): 300 MHz δ 4.86-4.76 (m, 2H), 3.50 (d, J = 11.0 Hz, 1H), 2.77 (ddd, J = 9.9, 2.4, 2.4 Hz, 1H), 2.69 (ddd, 6.9, 2.9, 2.9 Hz, 1H), 2.49-2.35 (m, 4H), 2.24-2.10 (m, 2H), 1.98-1.94 (m, 1H), 1.77-1.73 (m, 4H), 1.61-1.17 (m, 6H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 173.0, 172.5, 73.8, 65.3, 58.6, 58.5, 42.1, 32.1, 30.3, 29.7, 29.6, 27.2, 26.6, 25.8, 24.9; EMS $[M+Na]^+$ *m/z* calcd for $C_{15}H_{22}O_5Na^+$ 305.1359, found 305.1354.

(1*S*, 6*R*, 7*R*, 13*S*)-2,11,18-trioxatricyclo[12.4.0.0^{6,7}]octadecane-3,10-dione (S7). m.p. 122-124 °C; R_f 0.30 (4:1, hexanes:EtOAc); $[\alpha]_D$ +0.96 (*c* 2.01, $CHCl_3$); IR (KBr) cm^{-1}: 2942.48, 2859.69, 1732.66, 1424.57, 1362.17, 1262.65, 1232.07, 1187.98, 1100.06, 1021.10, 983.92, 883.32, 801.60, 677.85; ^{1}H NMR ($CDCl_3$): 300 MHz δ 5.26 (s, 1H), 4.43 (dd, J = 11.3, 11.3 Hz, 1H), 3.59 (dd, J = 11.0, 5.8 Hz, 1H), 2.74 (ddd, J = 9.8, 2.4, 2.4 Hz, 1H), 2.68 (ddd, J = 9.7, 2.3, 2.3 Hz, 1H), 2.50-2.31 (m, 4H), 2.19-2.08 (m, 2H), 2.08-1.95 (m, 1H), 1.95-1.86 (m, 1H), 1.83-1.70 (m, 1H), 1.61-1.25 (m, 8H); ^{13}C NMR ($CDCl_3$): 100 MHz δ 172.8, 172.51, 68.0, 64.4, 58.8, 58.7, 37.9, 29.6, 29.5 (2), 27.1. 26.7. 24.9. 24.1. 20.7; EMS $[M+Na]^+$ *m/z* calcd for $C_{15}H_{22}O_5Na^+$ 305.1359, found 305.1356.

Notes and References:

1. W. S. Fyvie and M. W. Peczuh, *J. Org. Chem.* 2008, **73**, 3626.
2. J. Casas, H. Sundén and A. Cordova, *Tetrahedron Lett*. 2004, **45**, 6117.



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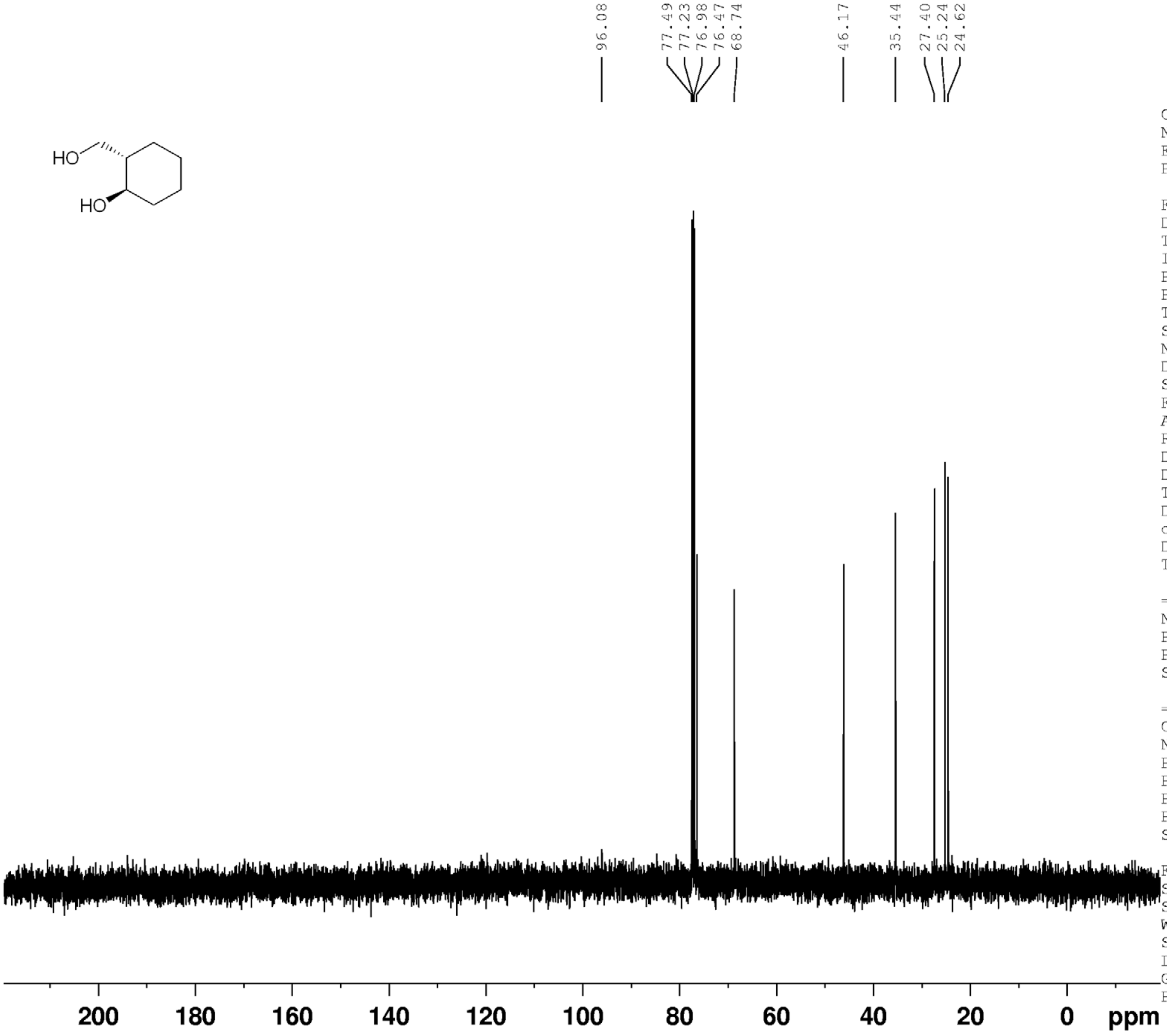

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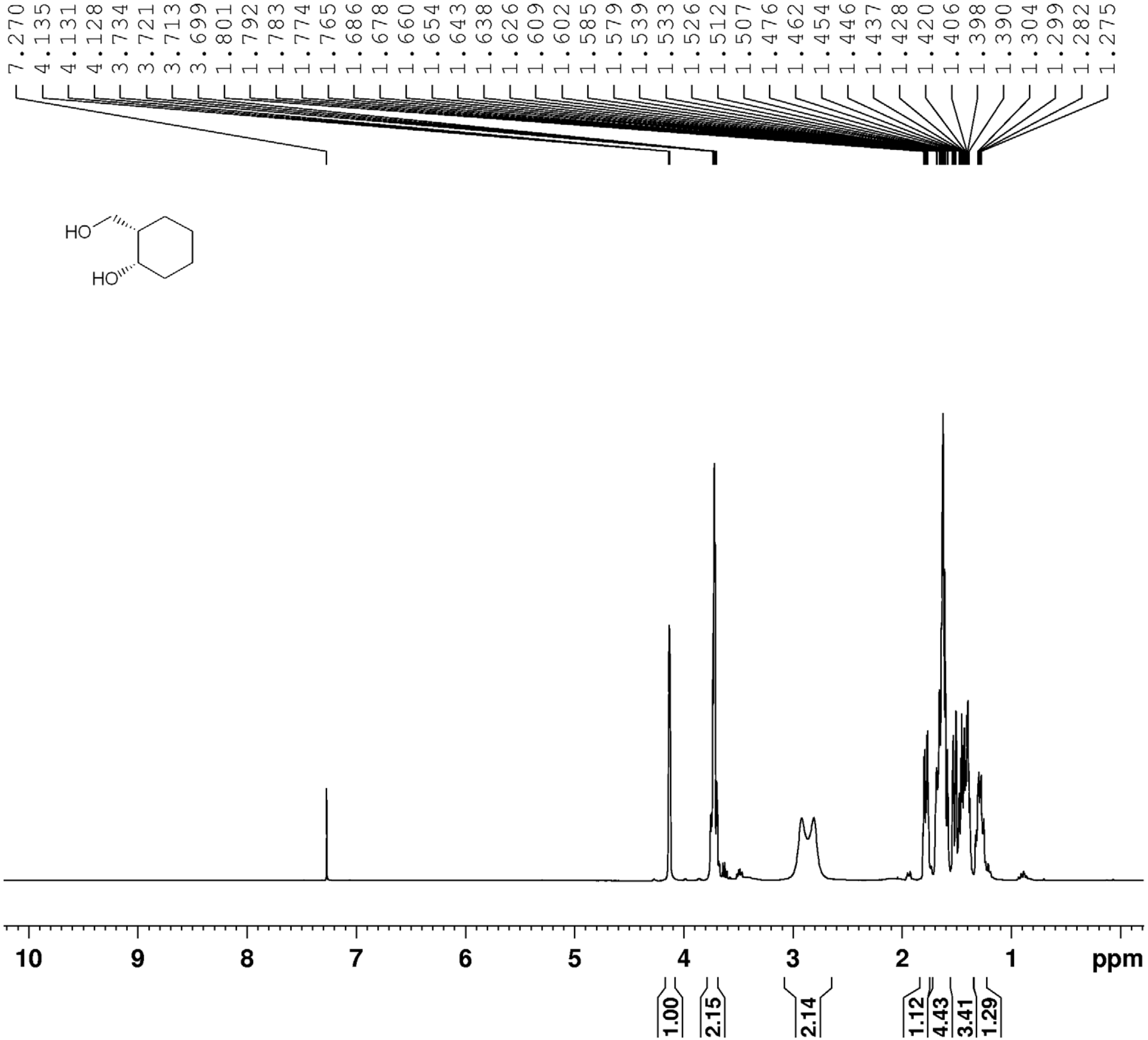

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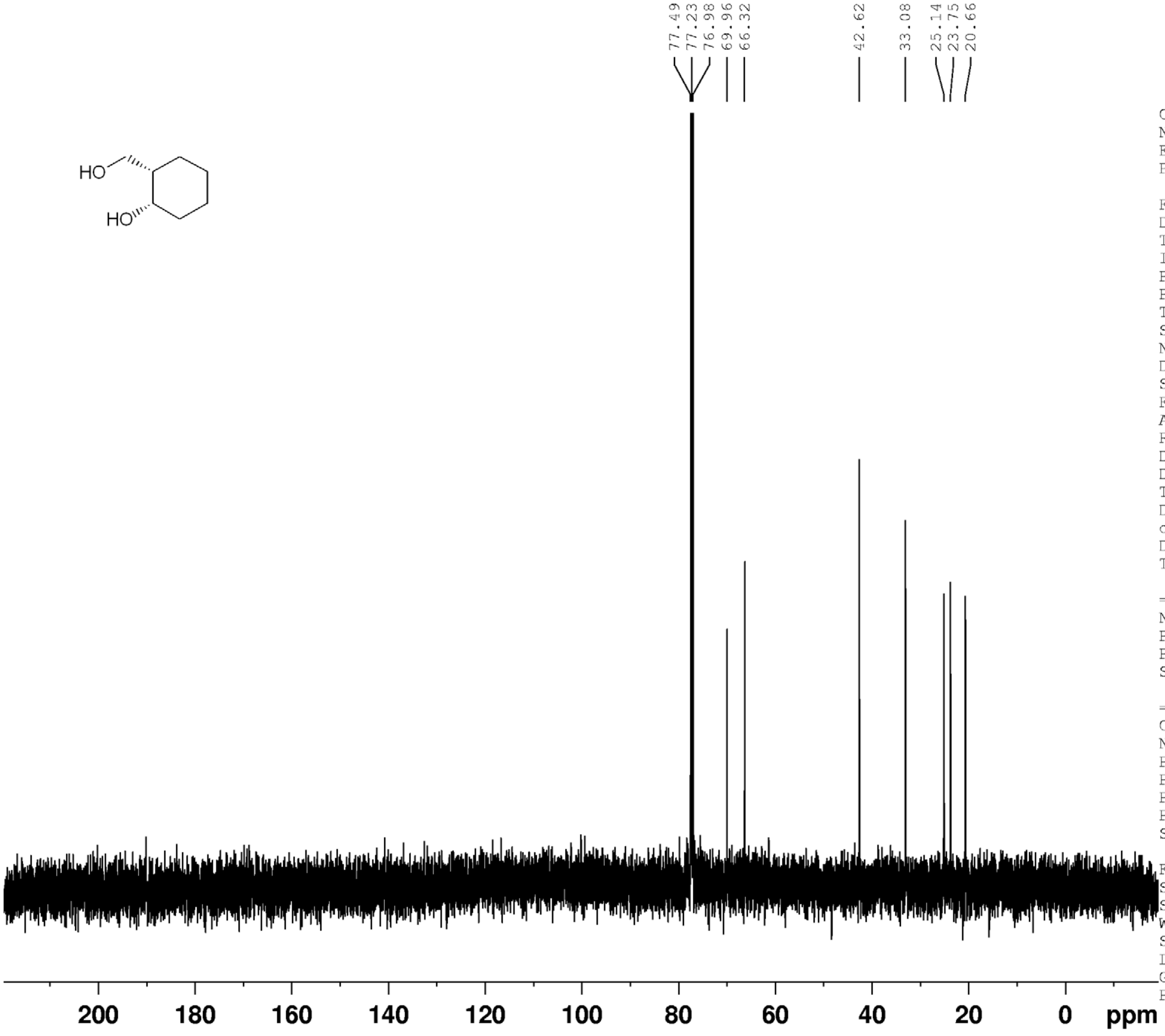

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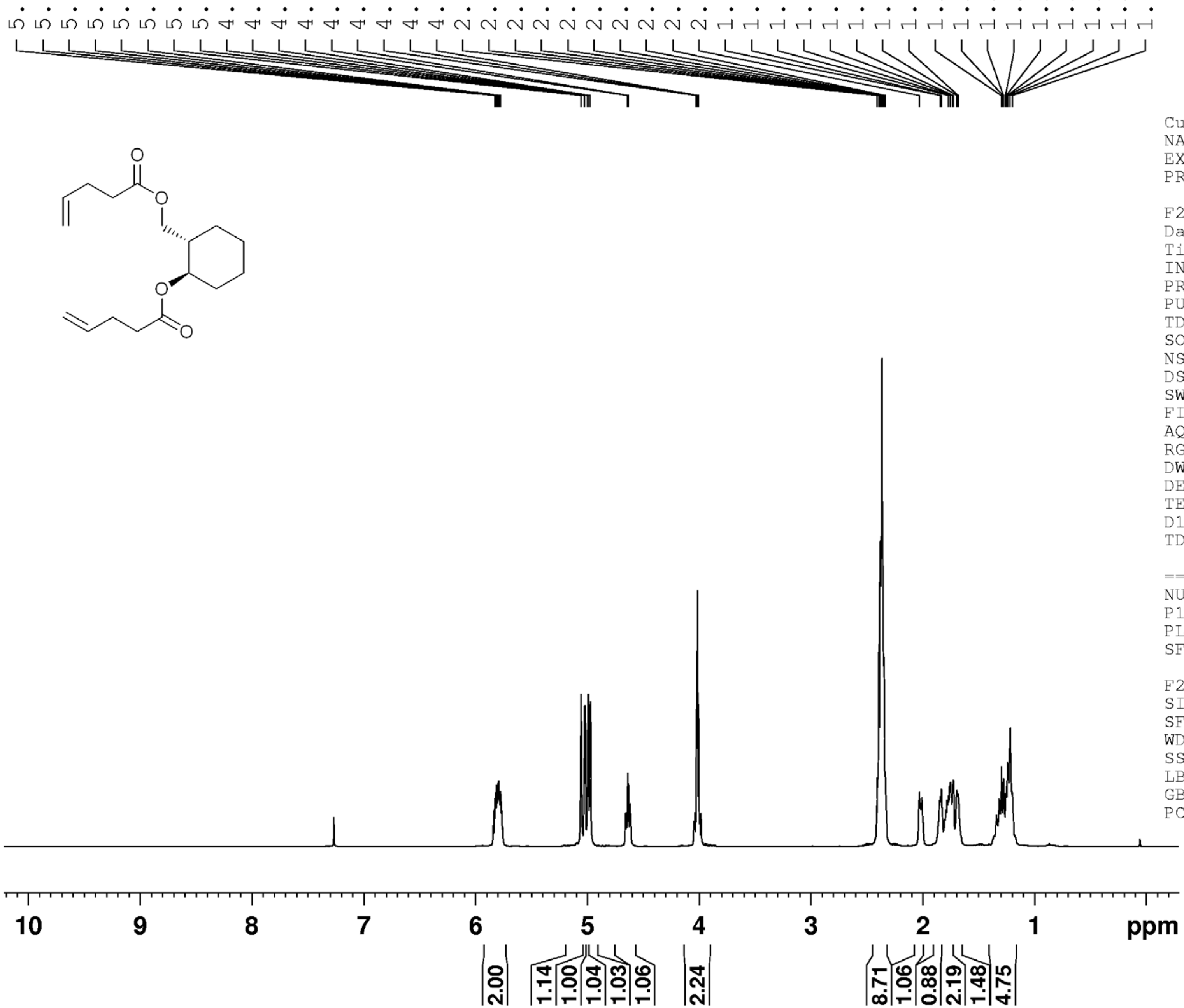
DS                     4
SWH            30030.029 Hz
FIDRES          0.916444 Hz
AQ             0.5456539 sec
RG                 32768
DW                16.650 usec
DE                  6.00 usec
TE                 298.2 K
D1            1.50000000 sec
d11           0.03000000 sec
DELTA         1.39999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.75 usec
PL1                 0.00 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PL2                -1.00 dB
PL12               14.69 dB
PL13               17.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577646 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



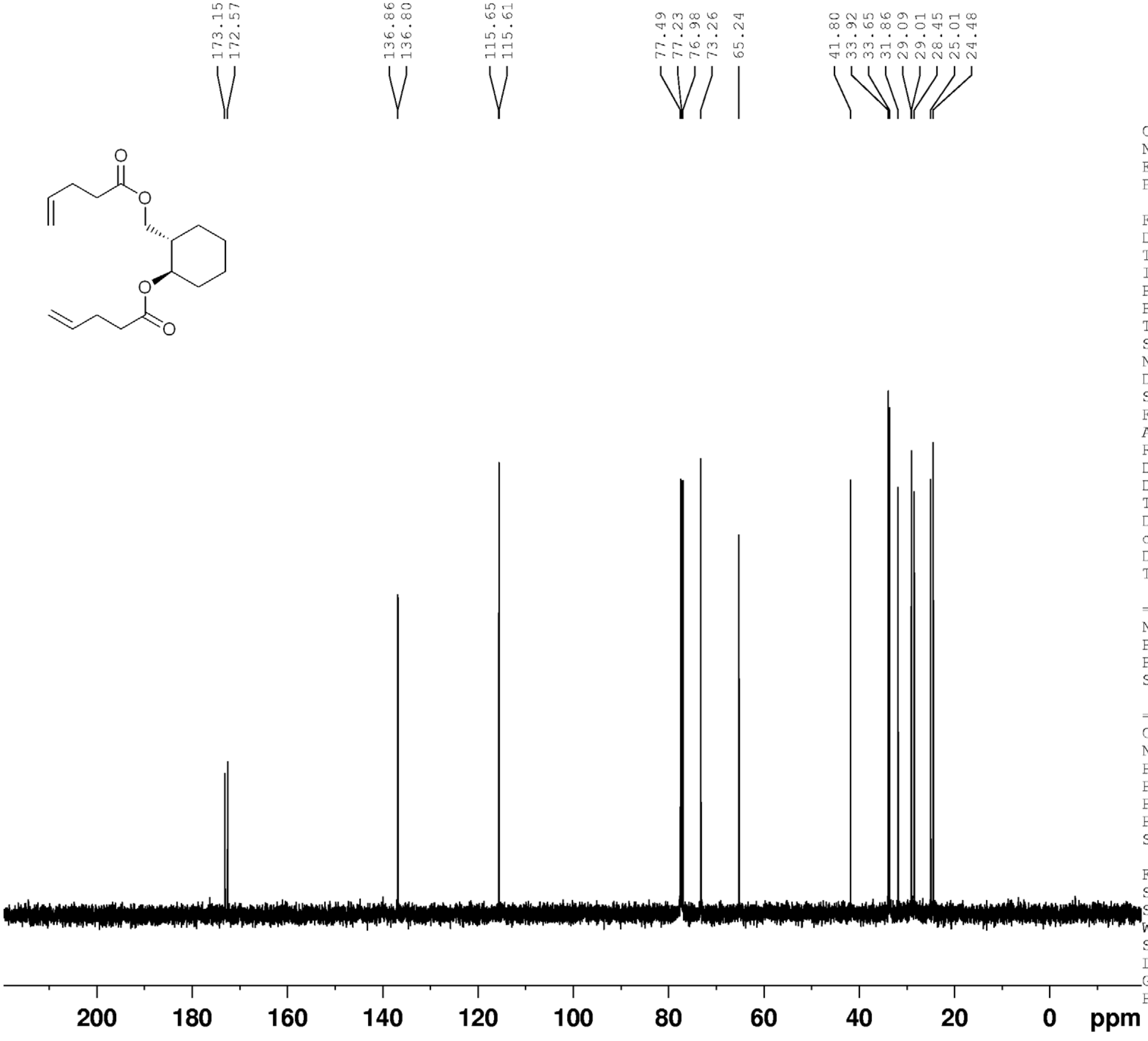
```
Current Data Parameters
NAME      wsf012208b_500
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080121
Time               20.36
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5860696 sec
RG                    57
DW                48.400 usec
DE                  6.00 usec
TE                 298.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -1.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF           500.1300079 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Supplementary Material (ESI) for Chemical Communications



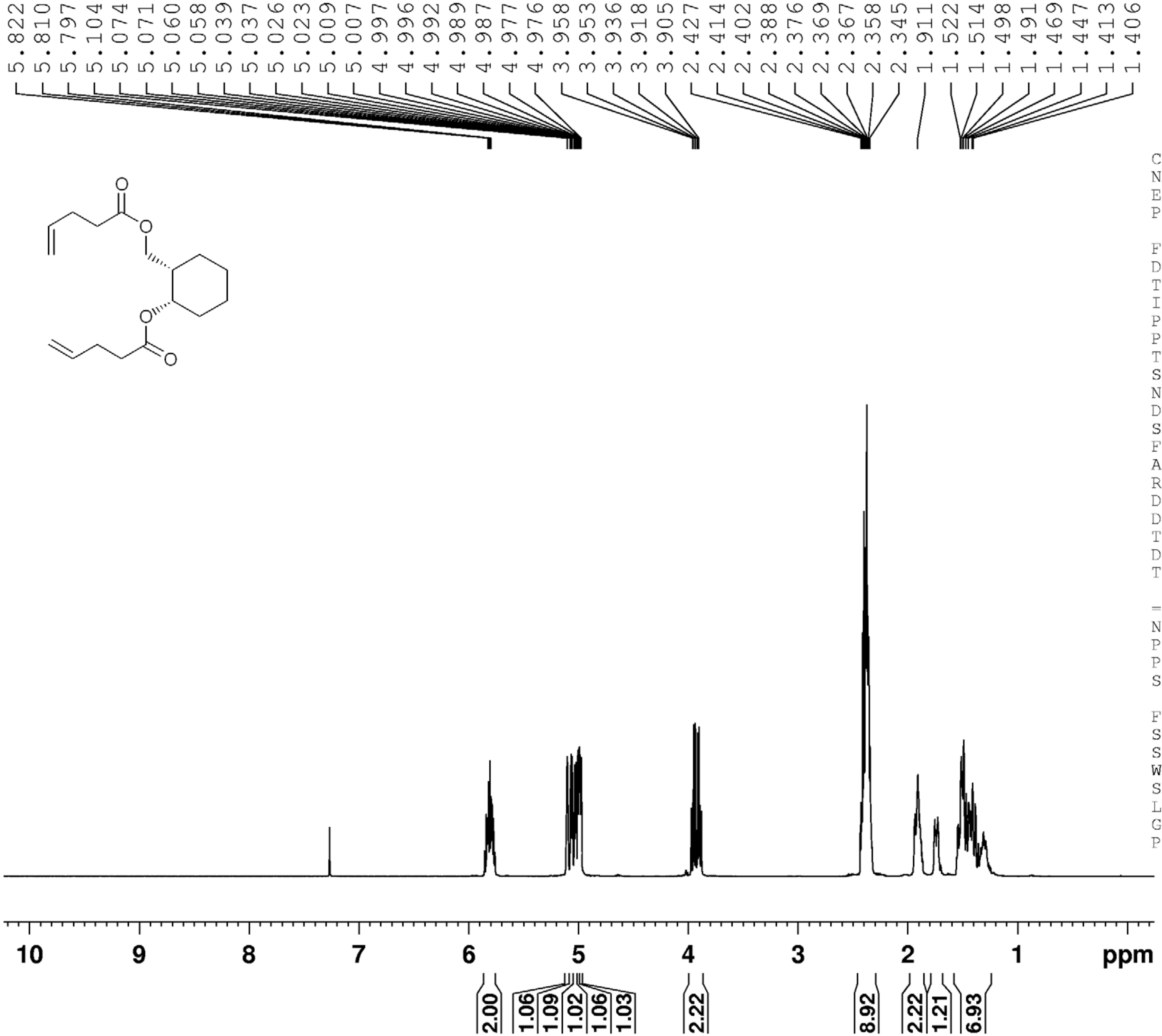
```
Current Data Parameters
NAME      wsf012208b_500
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080121
Time               20.39
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    70
DS                     4
SWH            30030.029 Hz
FIDRES          0.916444 Hz
AQ             0.5456539 sec
RG                 32768
DW                16.650 usec
DE                  6.00 usec
TE                 298.2 K
D1            1.50000000 sec
d11           0.03000000 sec
DELTA         1.39999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.75 usec
PL1                 0.00 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PL2                -1.00 dB
PL12               14.69 dB
PL13               17.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577670 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

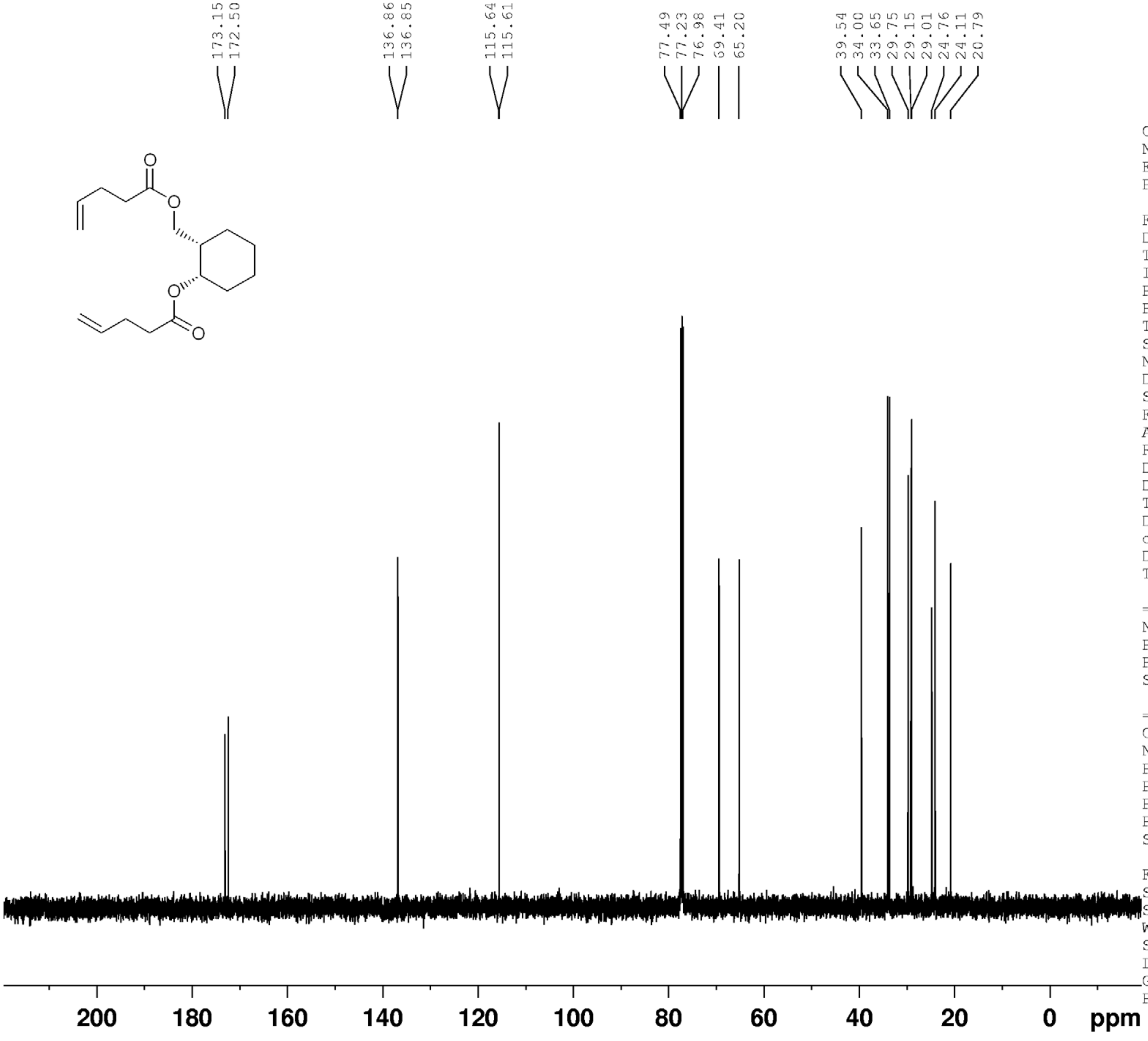


```
Current Data Parameters
NAME      wsf012208a_500
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080121
Time               18.37
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     2
SWH            10330.578 Hz
FIDRES          0.315264 Hz
AQ             1.5860696 sec
RG                  71.8
DW                48.400 usec
DE                  6.00 usec
TE                 298.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -1.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF           500.1300081 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



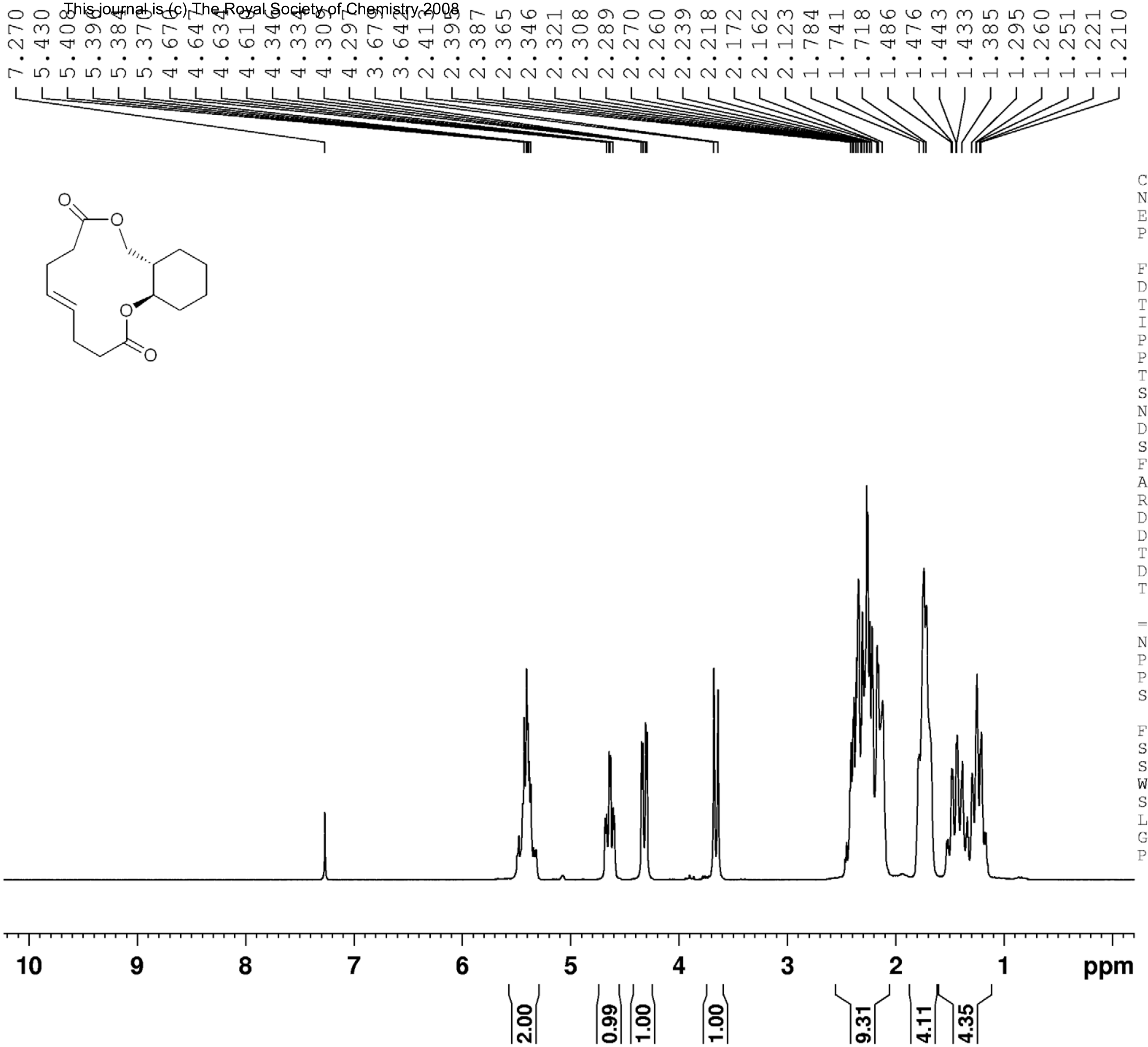
```
Current Data Parameters
NAME      wsf012208a_500
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080121
Time               18.42
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                    72
DS                     4
SWH            30030.029 Hz
FIDRES          0.916444 Hz
AQ             0.5456539 sec
RG                 32768
DW                16.650 usec
DE                  6.00 usec
TE                 298.2 K
D1            1.50000000 sec
d11           0.03000000 sec
DELTA         1.39999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.75 usec
PL1                 0.00 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PL2                -1.00 dB
PL12               14.69 dB
PL13               17.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577657 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

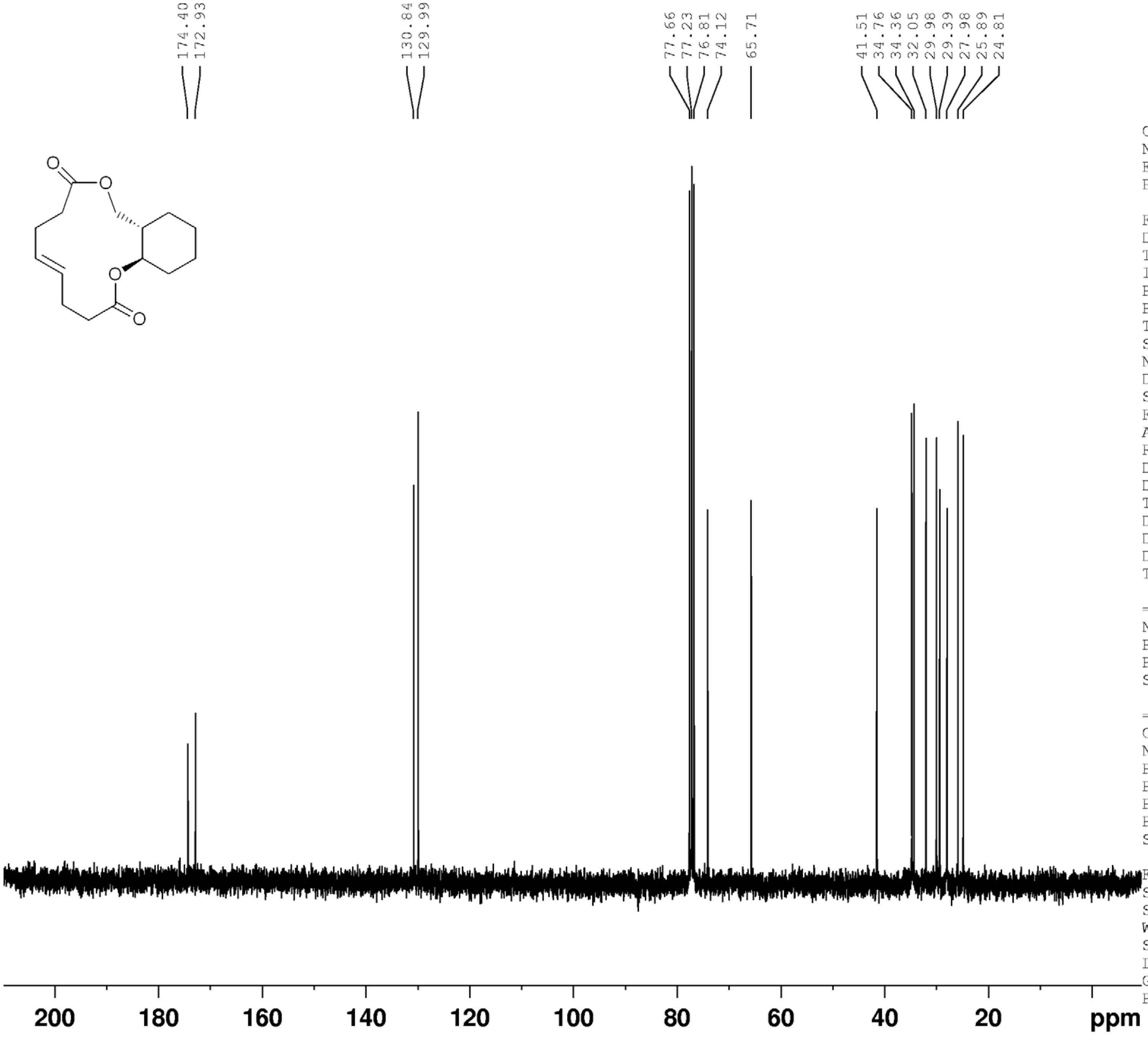


```
Current Data Parameters
NAME          wsf012608a
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080126
Time               21.11
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             4194.631 Hz
FIDRES          0.128010 Hz
AQ             3.9059956 sec
RG                  90.5
DW               119.200 usec
DE                  6.00 usec
TE                 298.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300035 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



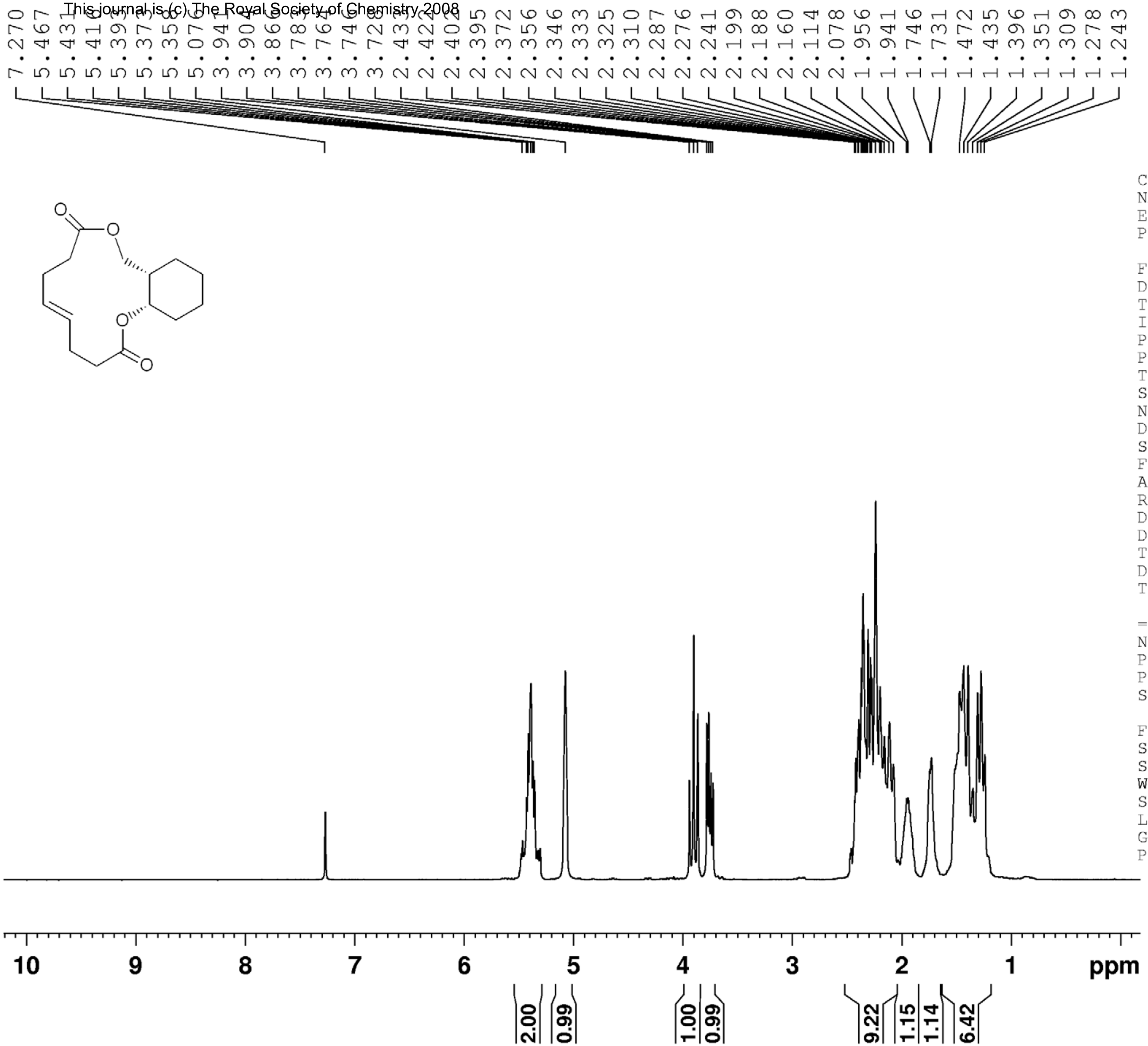
```
Current Data Parameters
NAME             wsf012608a
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080126
Time                  21.16
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      130
DS                        4
SWH               16556.291 Hz
FIDRES             0.505258 Hz
AQ                0.9896436 sec
RG                    32768
DW                   30.200 usec
DE                     6.00 usec
TE                    299.7 K
D1               2.00000000 sec
D11              0.03000000 sec
DELTA            1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.50 usec
PL1                   -2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -2.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF               75.4677344 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

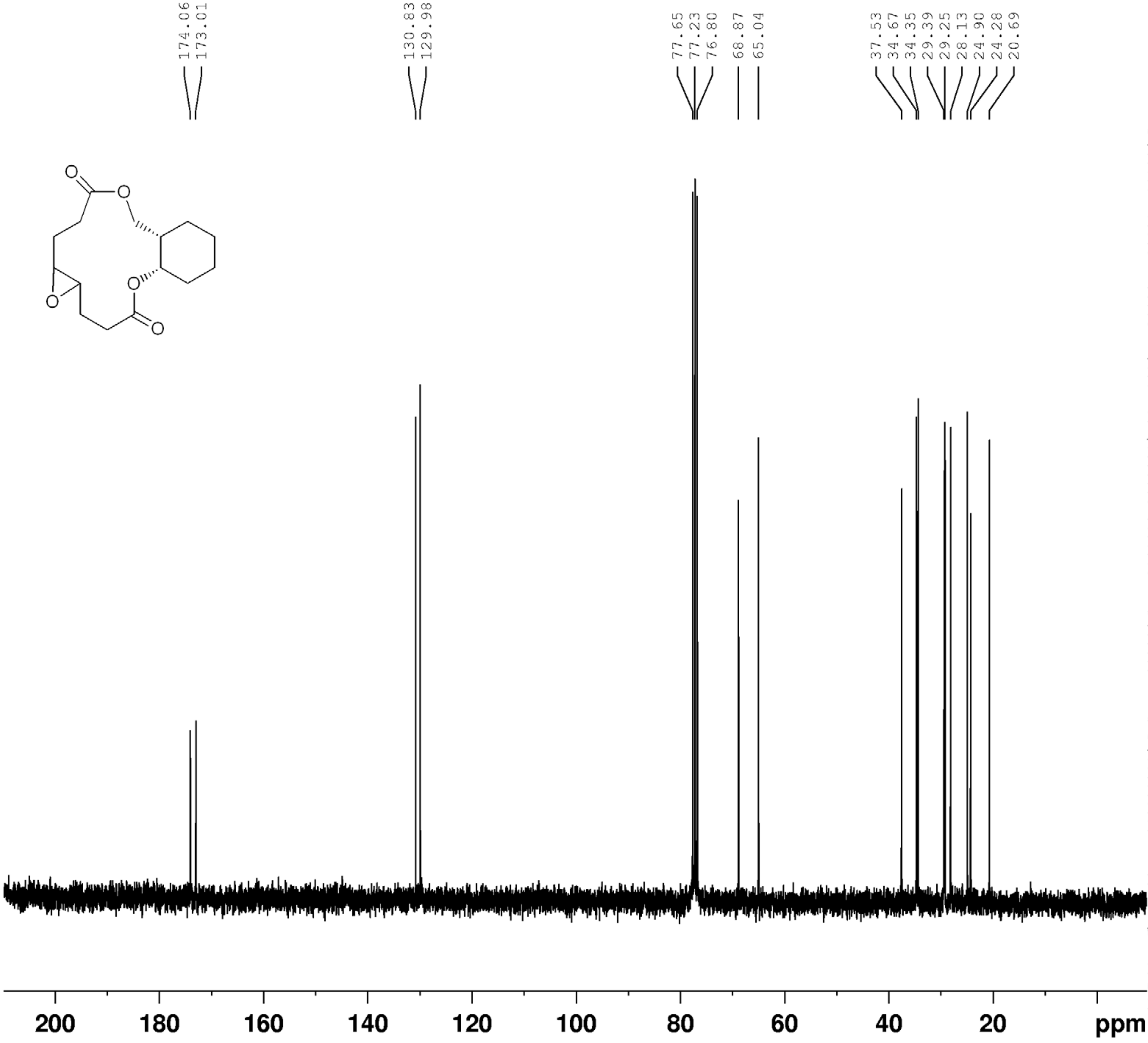


```
Current Data Parameters
NAME          wsf012608b
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080127
Time                0.14
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             4194.631 Hz
FIDRES          0.128010 Hz
AQ             3.9059956 sec
RG                  90.5
DW               119.200 usec
DE                  6.00 usec
TE                 298.7 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300034 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



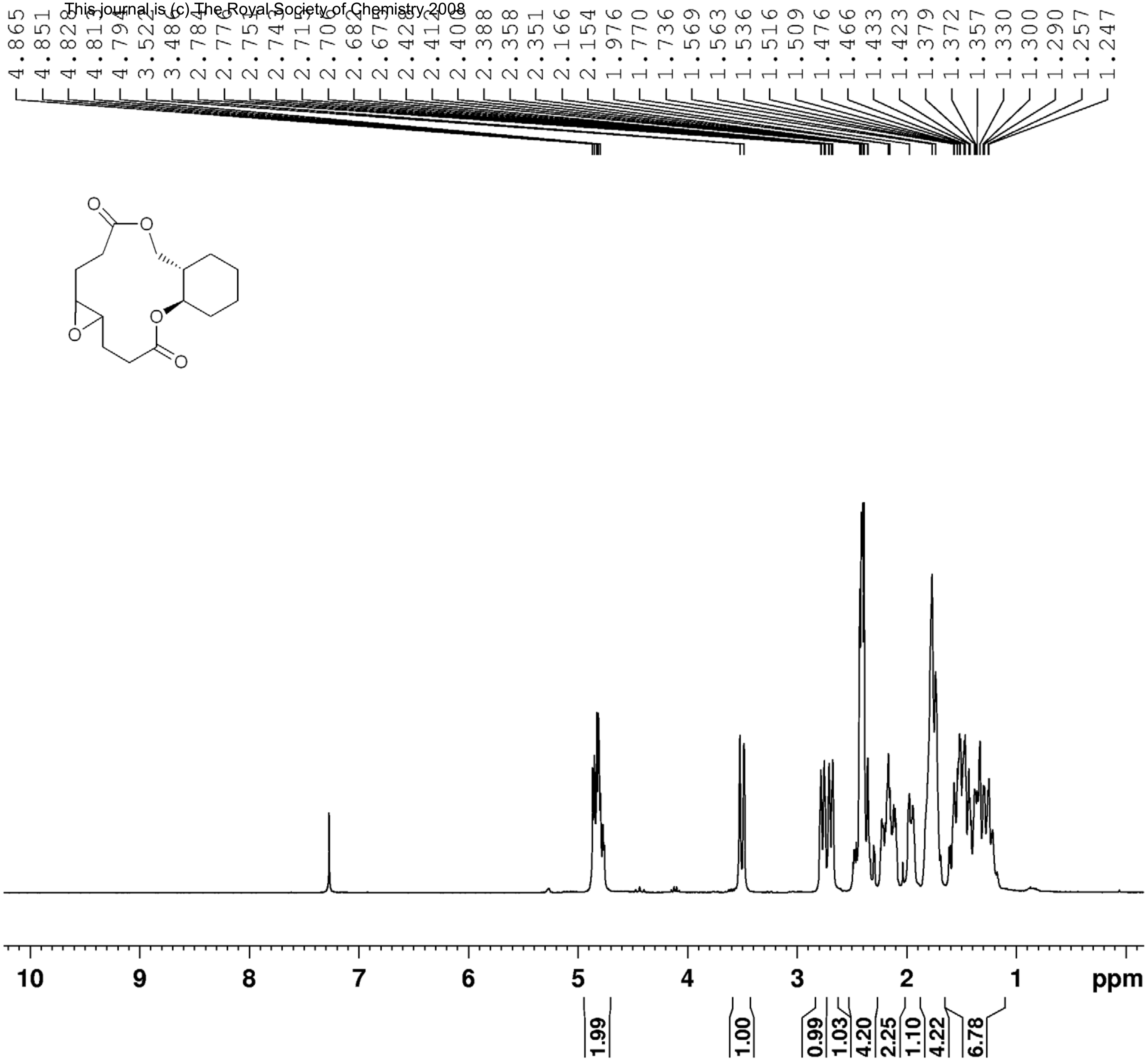
```
Current Data Parameters
NAME             wsf012608b
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080127
Time                   0.19
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      156
DS                        4
SWH               16556.291 Hz
FIDRES             0.505258 Hz
AQ                0.9896436 sec
RG                    32768
DW                   30.200 usec
DE                     6.00 usec
TE                    300.3 K
D1               2.00000000 sec
D11              0.03000000 sec
DELTA            1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.50 usec
PL1                   -2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -2.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF              75.4677336 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

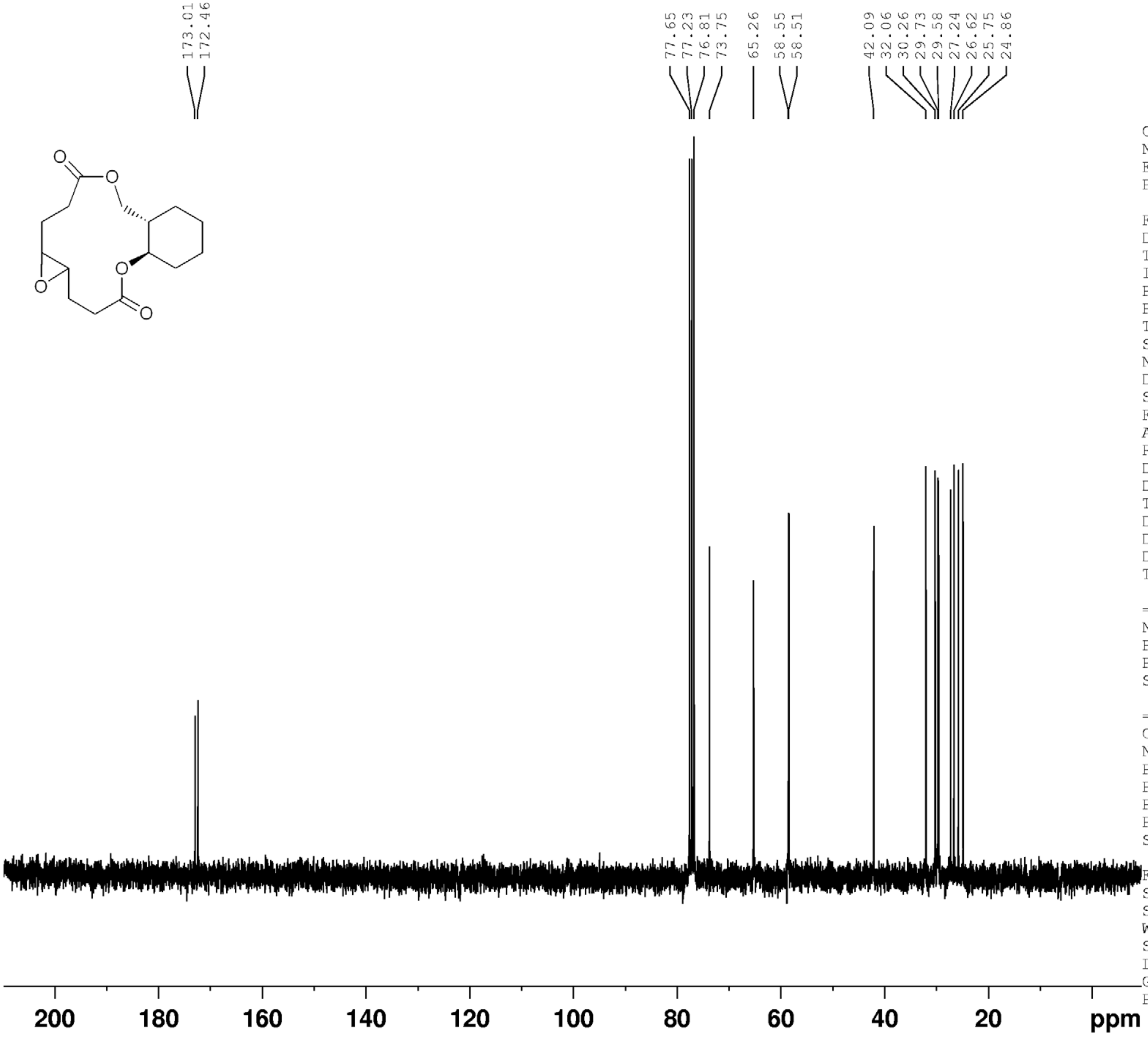


```
Current Data Parameters
NAME          wsf012908a
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080129
Time               22.03
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             4194.631 Hz
FIDRES          0.128010 Hz
AQ             3.9059956 sec
RG                  90.5
DW               119.200 usec
DE                  6.00 usec
TE                 297.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300034 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



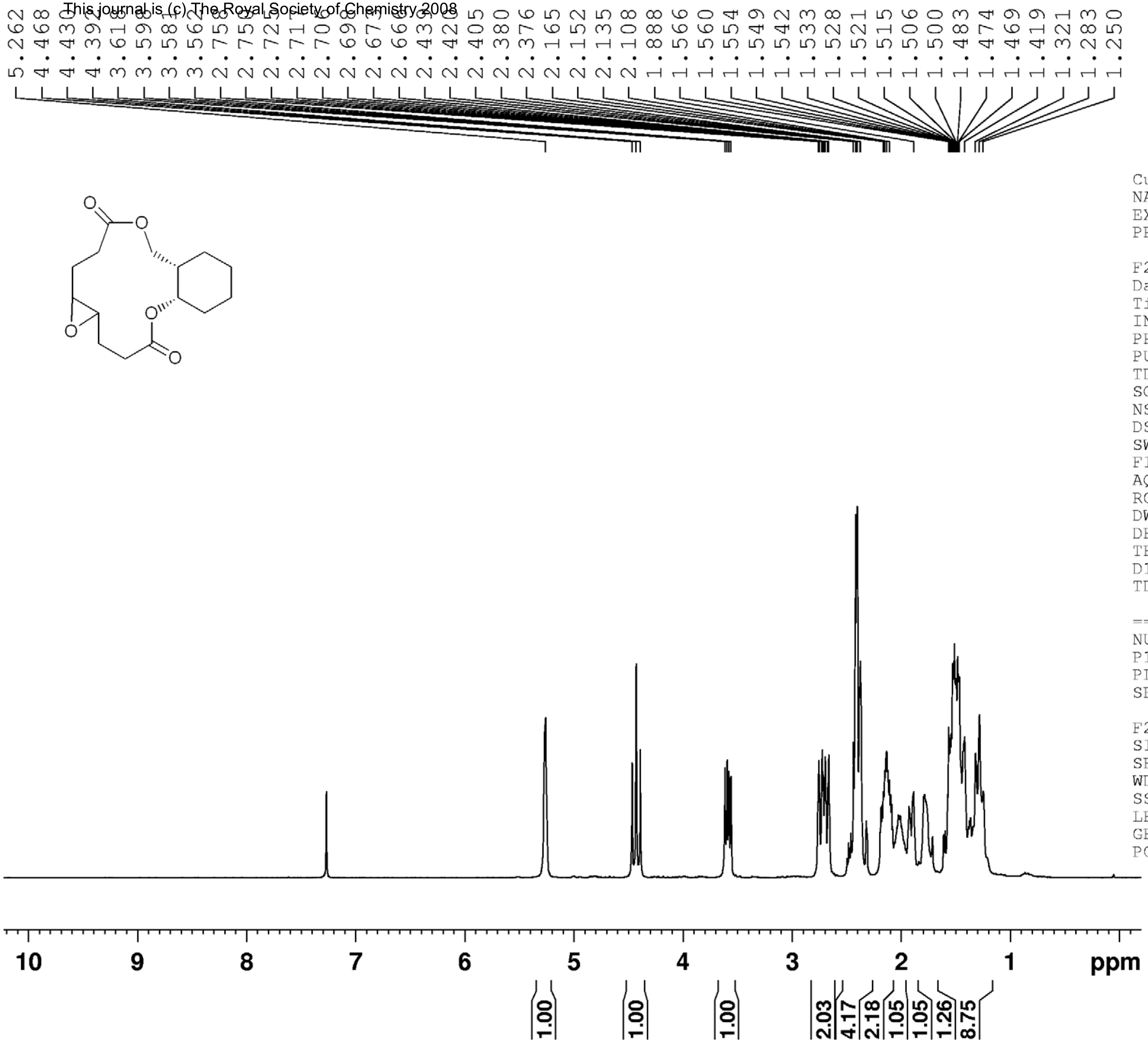
```
Current Data Parameters
NAME             wsf012908a
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080129
Time                  22.11
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     123
DS                       4
SWH              16556.291 Hz
FIDRES            0.505258 Hz
AQ               0.9896436 sec
RG                   32768
DW                  30.200 usec
DE                    6.00 usec
TE                   299.1 K
D1              2.00000000 sec
D11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.50 usec
PL1                  -2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  -2.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF              75.4677346 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

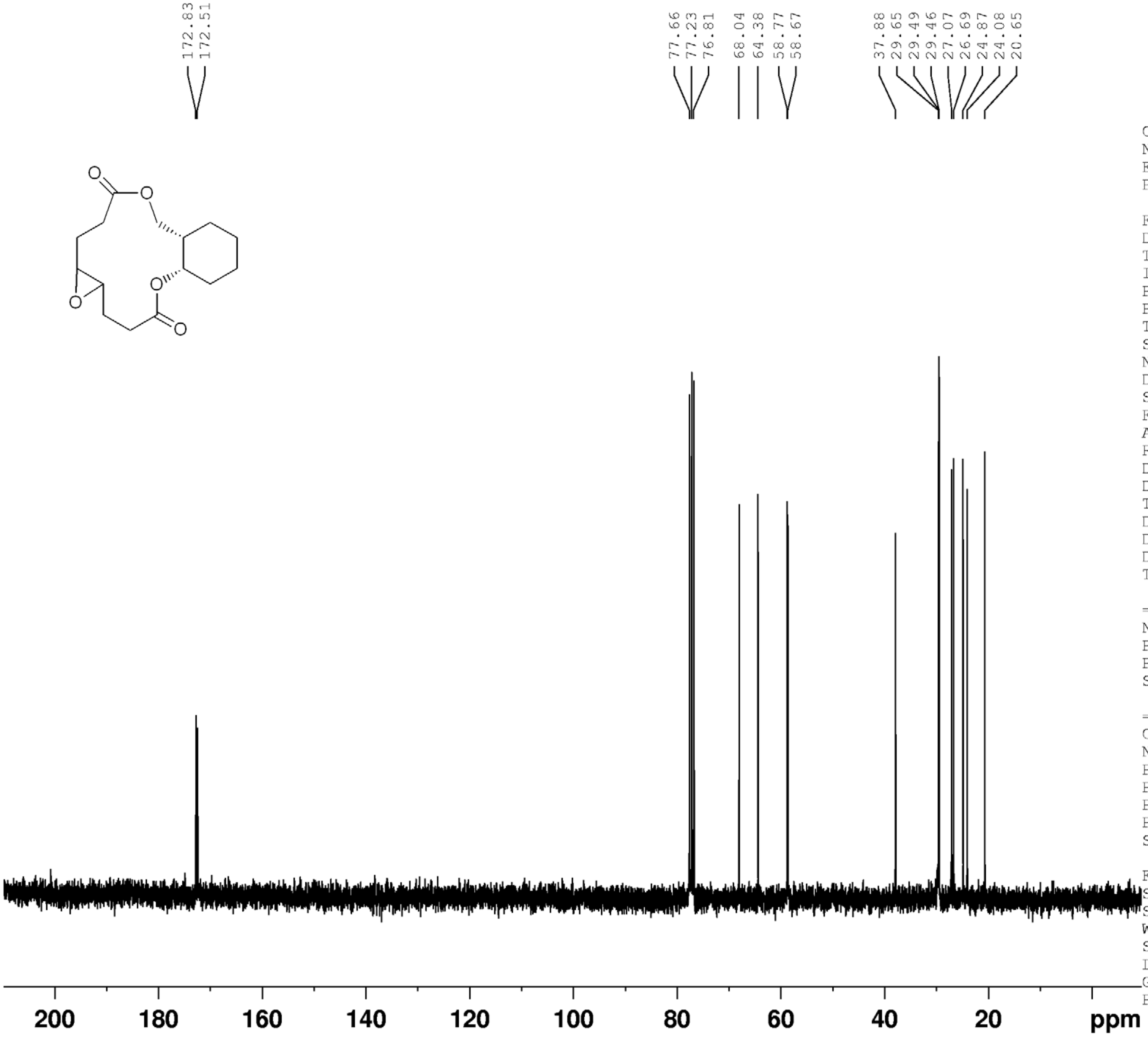


```
Current Data Parameters
NAME          wsf012908b
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080129
Time               23.46
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             4194.631 Hz
FIDRES          0.128010 Hz
AQ             3.9059956 sec
RG                  80.6
DW               119.200 usec
DE                  6.00 usec
TE                 298.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300034 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



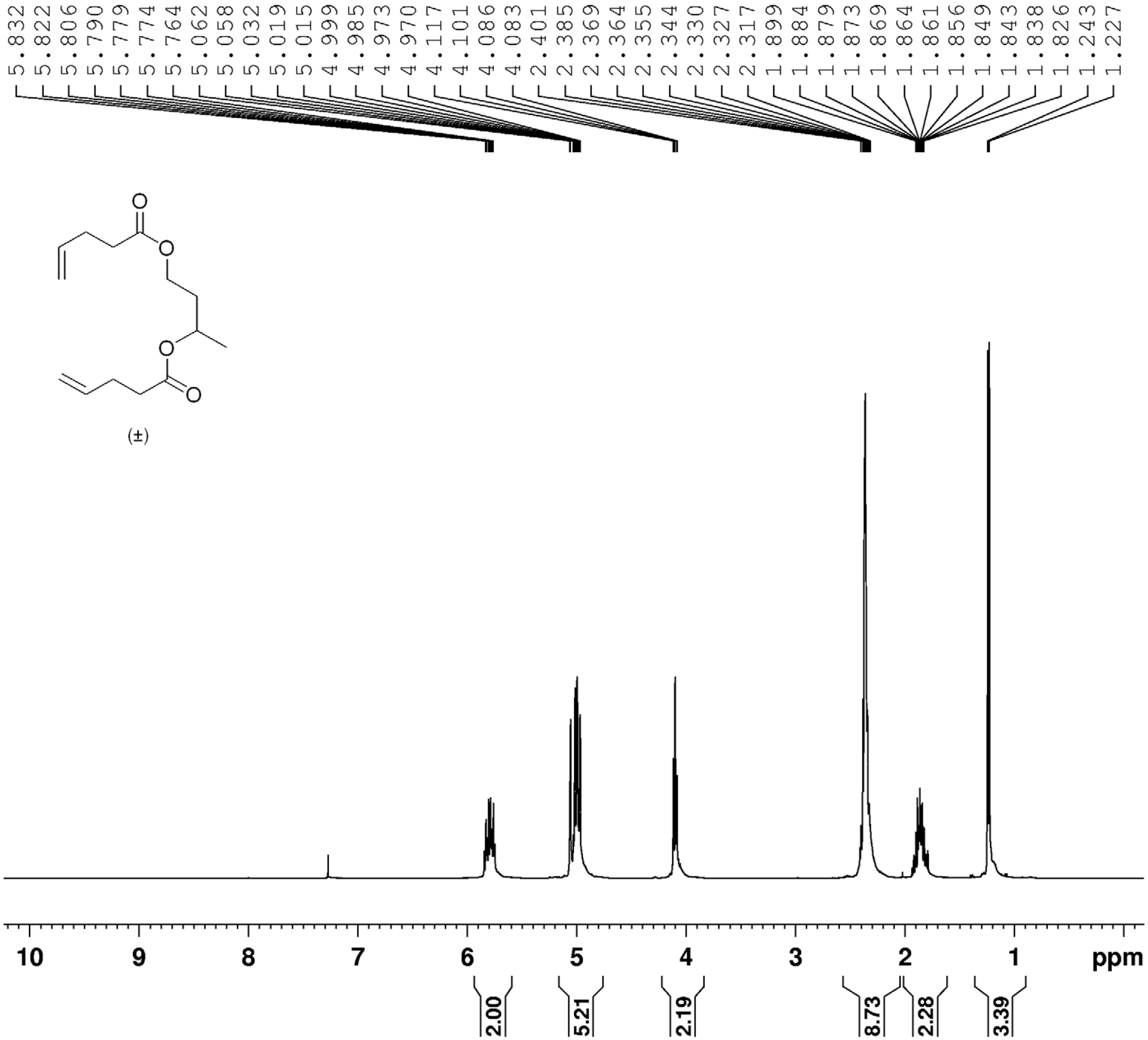
```
Current Data Parameters
NAME             wsf012908b
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080129
Time                  23.51
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      135
DS                        4
SWH               16556.291 Hz
FIDRES             0.505258 Hz
AQ                0.9896436 sec
RG                    32768
DW                   30.200 usec
DE                     6.00 usec
TE                    299.4 K
D1               2.00000000 sec
D11              0.03000000 sec
DELTA            1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.50 usec
PL1                   -2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -2.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF               75.4677351 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

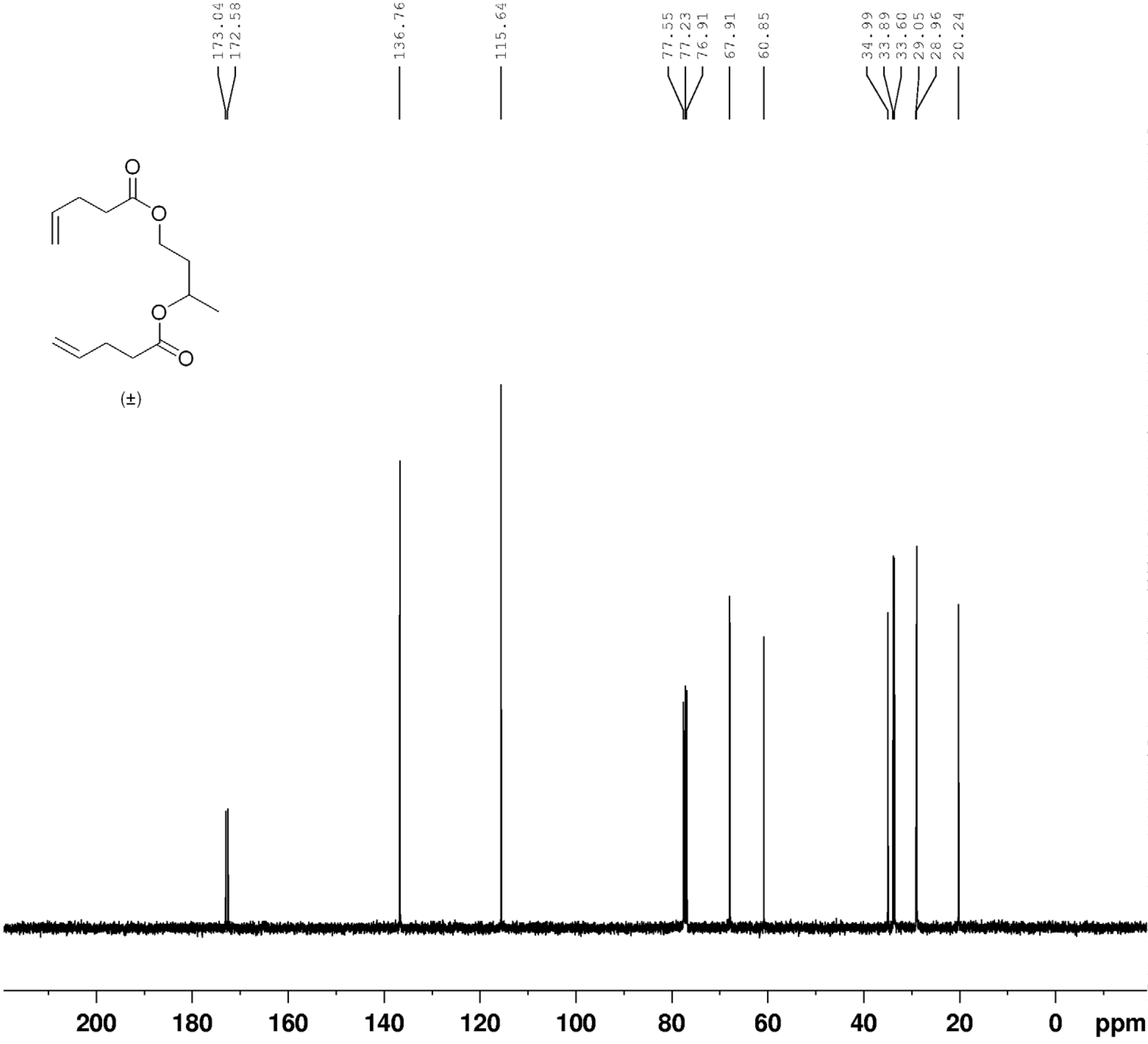


```
Current Data Parameters
NAME          wsf032008a
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080320
Time               20.34
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             5592.841 Hz
FIDRES          0.170680 Hz
AQ             2.9295092 sec
RG                  45.3
DW                89.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.20 usec
PL1                -2.00 dB
SFO1         400.1464009 MHz

F2 - Processing parameters
SI                 32768
SF           400.1440025 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



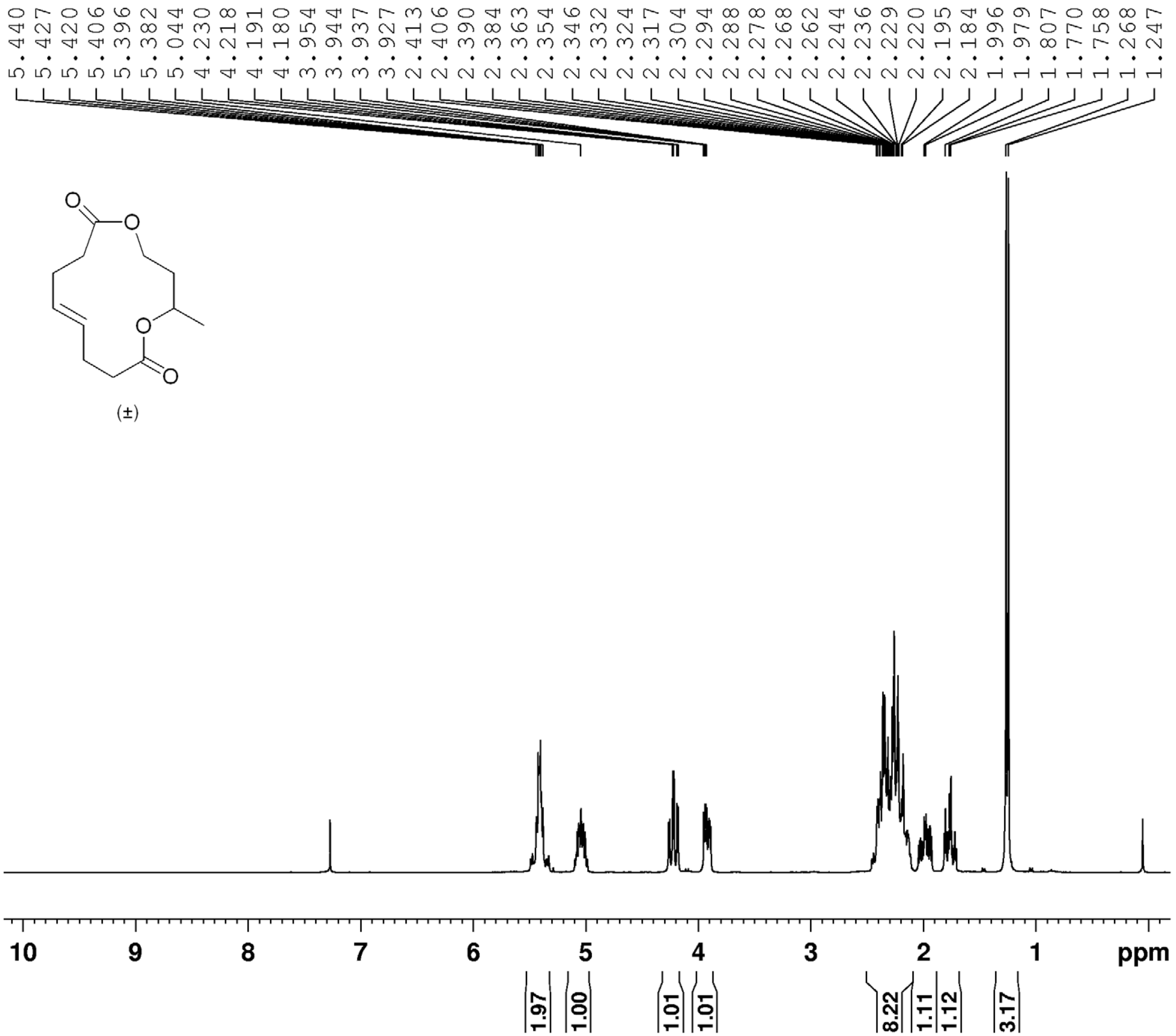
```
Current Data Parameters
NAME             wsf032008a
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20080320
Time                  20.41
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     152
DS                       4
SWH              23980.814 Hz
FIDRES            0.365918 Hz
AQ               1.3664756 sec
RG                   32768
DW                  20.850 usec
DE                    6.00 usec
TE                   300.0 K
D1              1.50000000 sec
d11             0.03000000 sec
DELTA           1.39999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.30 usec
PL1                  -3.00 dB
SFO1           100.6263505 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               110.00 usec
PL2                   0.00 dB
PL12                 19.00 dB
PL13                 19.00 dB
SFO2           400.1456006 MHz

F2 - Processing parameters
SI                   32768
SF             100.6162724 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

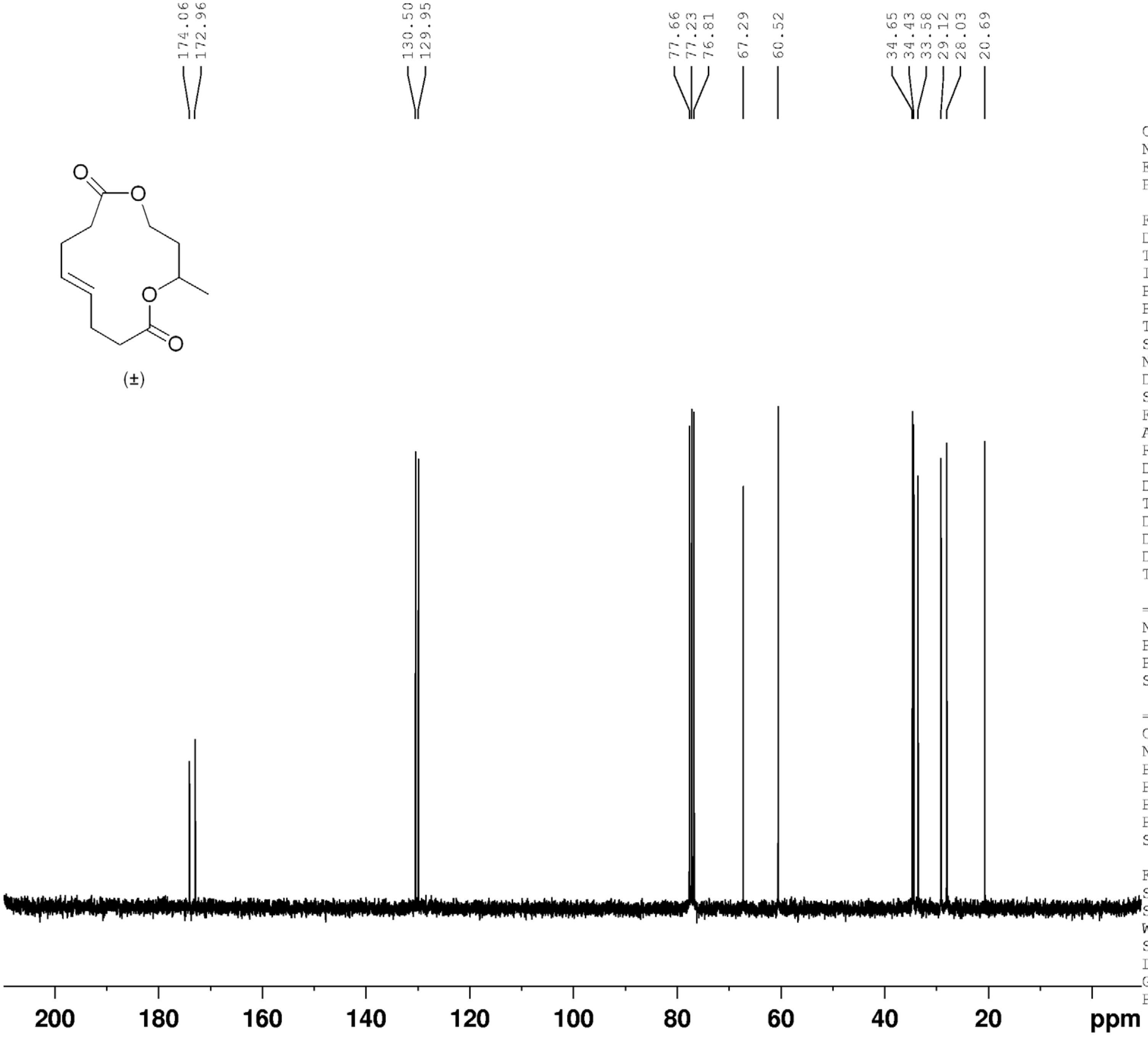


```
Current Data Parameters
NAME          wsf032508c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080326
Time                0.50
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             4194.631 Hz
FIDRES          0.128010 Hz
AQ             3.9059956 sec
RG                  90.5
DW               119.200 use
DE                  6.00 use
TE                 300.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                  8.20 use
PL1                 0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300033 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME           wsf032508c
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080326
Time                 1.04
INSTRUM             spect
PROBHD    5 mm Multinucl
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   194
DS                     4
SWH            16556.291 Hz
FIDRES          0.505258 Hz
AQ             0.9896436 sec
RG                 32768
DW                30.200 usec
DE                  6.00 usec
TE                 300.4 K
D1            2.00000000 sec
D11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                -2.00 dB
SFO1          75.4752953 MHz

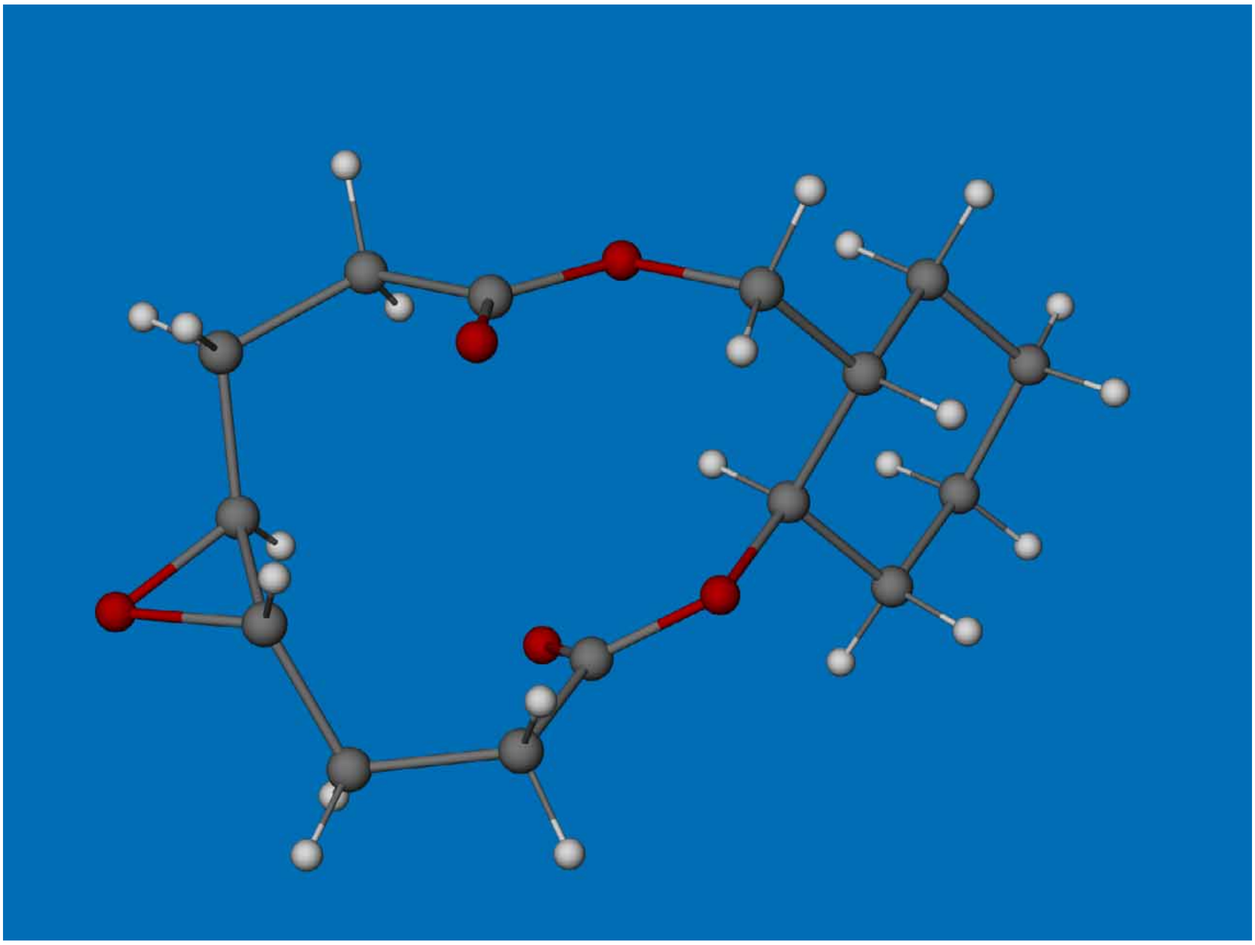
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               15.00 dB
PL13               15.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677337 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

YALE CHEMICAL INSTRUMENTATION CENTER

X-Ray Structure Report
Reference Number: UCONN_MP07

April 2, 2008



YALE CHEMICAL INSTRUMENTATION CENTER

X-Ray Structure Report
Reference Number: UCONN_MP07

April 2, 2008

Data Collection

A colorless plate crystal of $C_{15}H_{22}O_5$ having approximate dimensions of 0.30 x 0.20 x 0.10 mm^3 was mounted with epoxy cement on the tip of a fine glass fiber. All measurements were made on a Nonius KappaCCD diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

$a = 7.0375(14)$ Å $\quad \alpha = 90$ °
$b = 11.004(2)$ Å $\quad \beta = 102.95(3)$ °
$c = 9.6494(19)$ Å $\quad \gamma = 90$ °
$V = 728.3(3)$ Å^3

For Z = 2 and F.W. = 282.33, the calculated density is 1.287 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be $P2_1$ (#4).

The data were collected at a temperature of 173(2) K to a maximum 2θ value of 57.96 °. Five omega scans consisting of 37, 29, 34, 29, and 32 data frames, respectively, were collected with a frame width of 2.0 ° and a detector-to-crystal distance, Dx, of 36.0 mm. Each frame was exposed twice (for the purpose of de-zingering) for a total of 20 s. The data frames were processed and scaled using the DENZO software package.[1]

Data Reduction

A total of 3617 reflections were collected of which 3617 were unique and observed (R_{int} = 0.000, Friedel pairs not merged). The linear absorption coefficient, μ, for Mo-Kα radiation is 0.96 cm^{-1}, and no absorption correction was applied. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were treated as idealized contributions. The final cycle of full-matrix least-squares refinement[3] on F was based on 3617 observed reflections ($I > 2.00\sigma(I)$) and 181 variable parameters and converged with unweighted and weighted agreement factors of:

$$R = \Sigma \,||Fo| - |Fc|| \,/\, \Sigma\, |Fo| = 0.0507$$
$$R_W = \{\Sigma[w\,(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]\}^{1/2} = 0.0811$$

The maximum and minimum peaks on the final difference Fourier map corresponded to 0.177 and –0.206 $e^-/Å^3$ respectively.

REFERENCES

(1) Z. Otwinowski and W. Minor, "Processing of X-Ray Diffraction Data Collected in Oscillation Mode," Methods in Enzymology, vol. 276: Macromolecular Crystallography, part A, 307-326, 1997, C.W. Carter, Jr. & R.M. Sweet, Eds., Academic Press.

(2) *SHELXTL*, v.6.12, Bruker-AXS, Madison, WI, 2001.

(3) Least Squares function minimized:
$\Sigma w(F_o^2 - F_c^2)^2$

Structural Description

The compound crystallized in the chiral monoclinic space group $P2_1$ with one molecule in the asymmetric unit and two molecules in the unit cell.

Torsion angles of interest in the molecule are as follows: C(3)-C(4)-C(5)-C(6) = 151.8 °, O(1)-C(11)-C(10)-C(9) = 51.0 °, C(1)-C(2)-C(3)-C(4) = 73.7 °, C(5)-C(6)-C(7)-C(8) = 69.4 °.

An ambiguous Flack parameter was calculated due to the lack of substantially weighted atoms and thus the absolute configuration of the molecule could not be determined. There are no significant intermolecular contacts. ORTEPs, packing diagrams and full crystallographic tables follow.

Figure 1

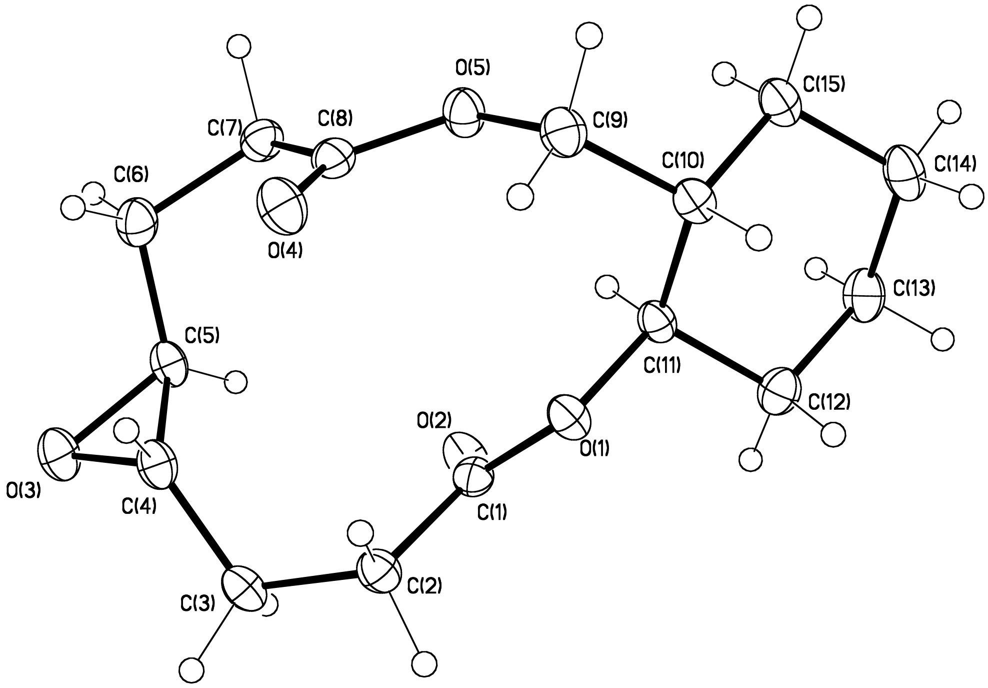


Figure 2

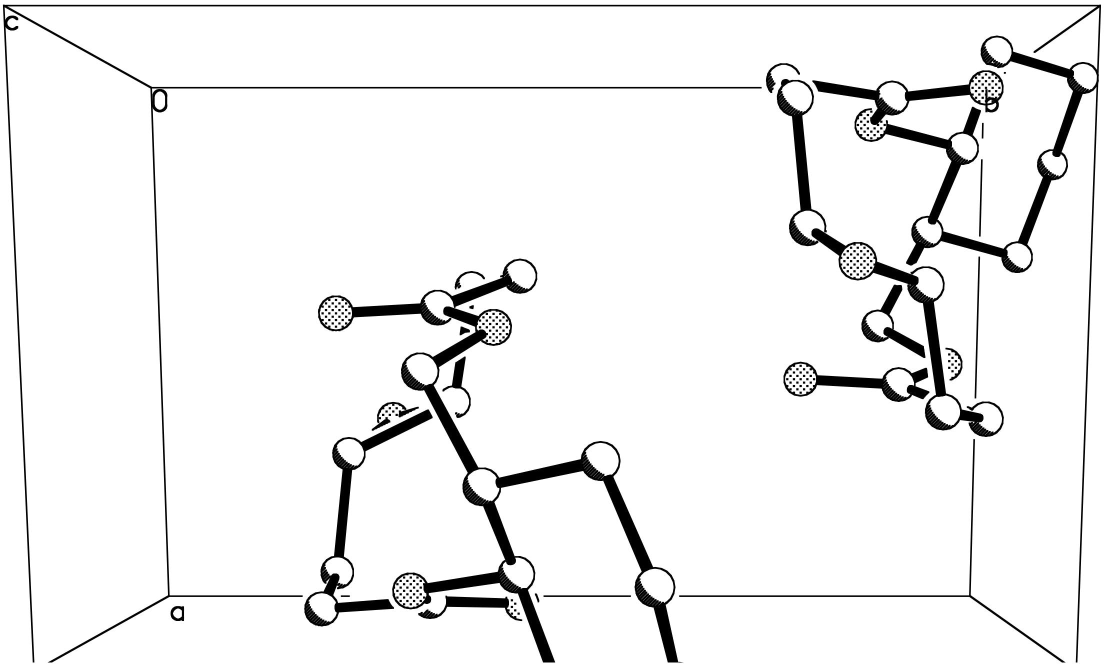
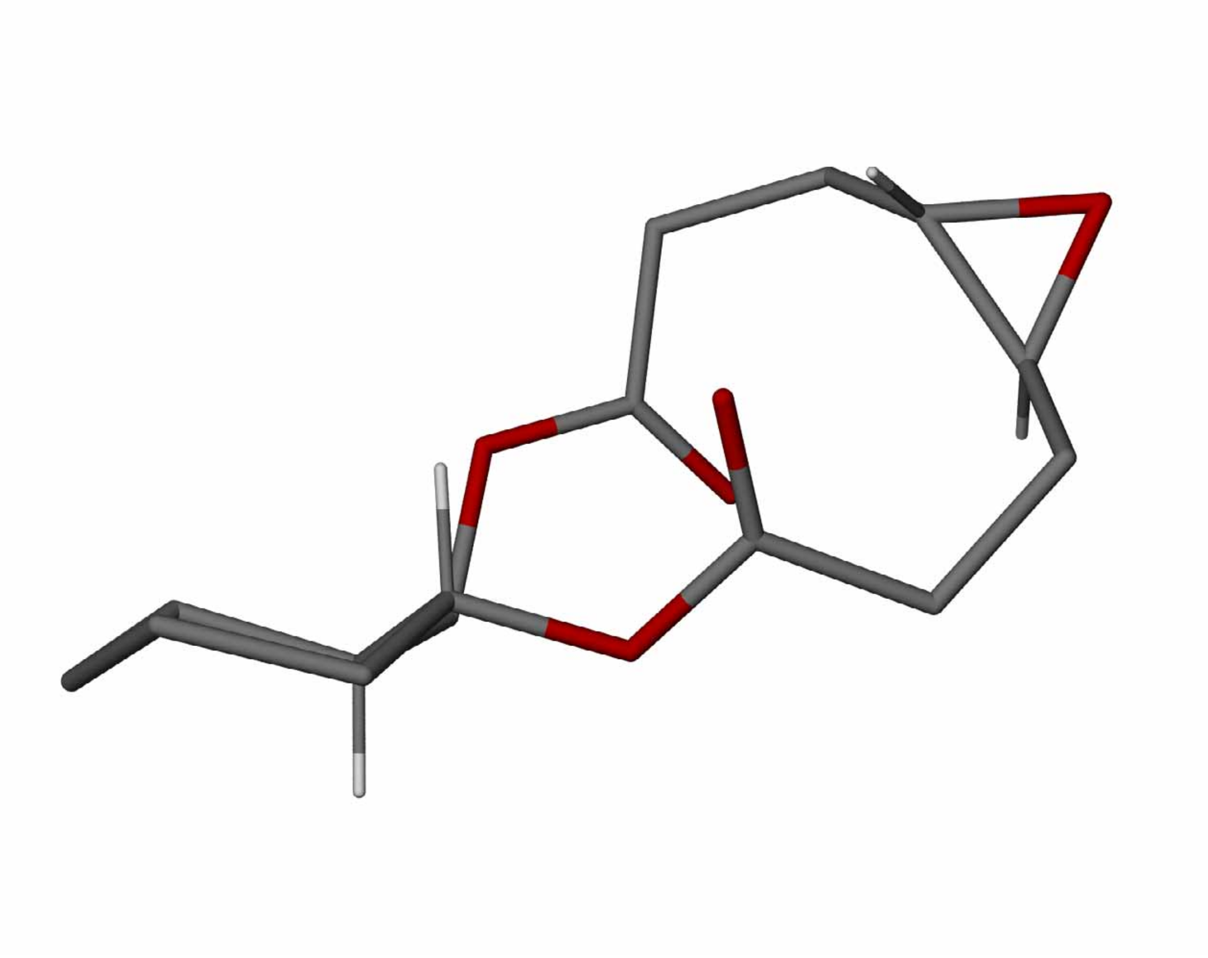
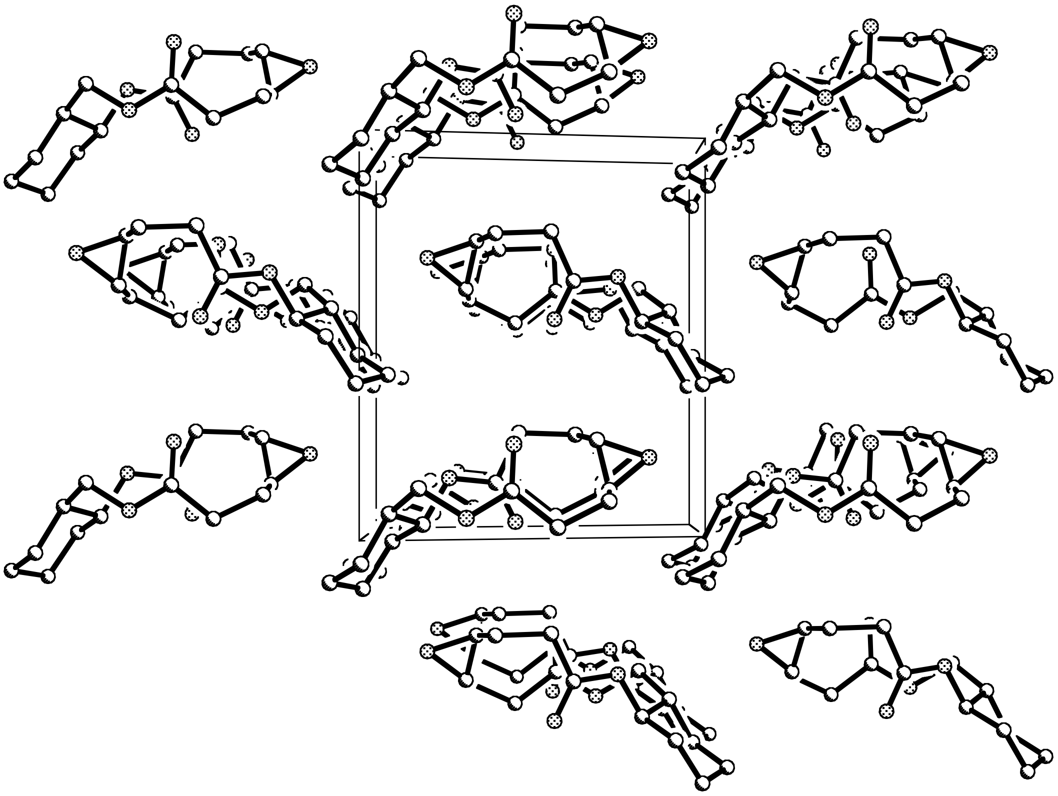


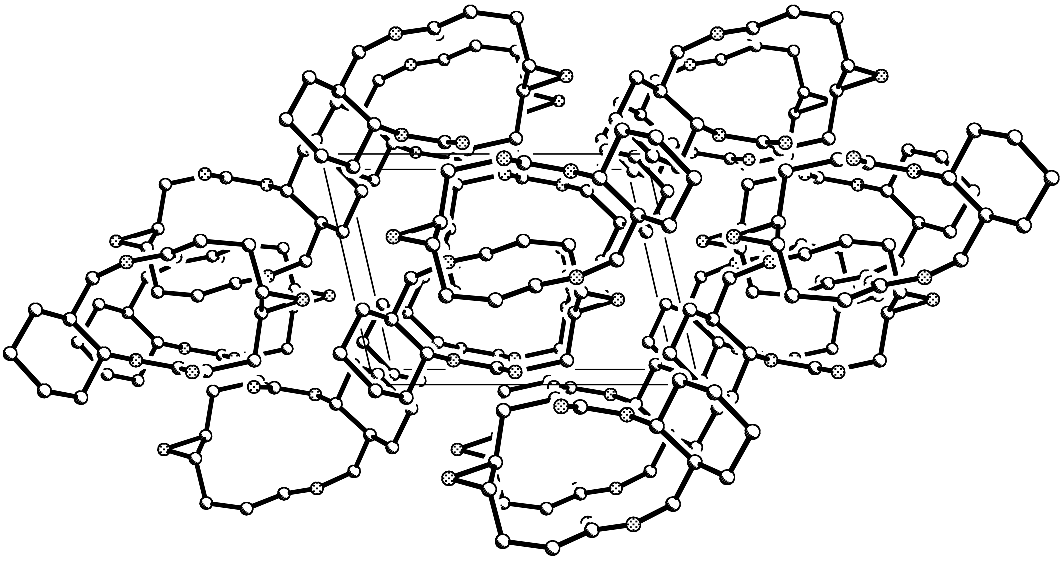
Figure 3



Packing diagram – View down the a-axis



Packing diagram – View down the b-axis



Packing diagram – View down the c-axis

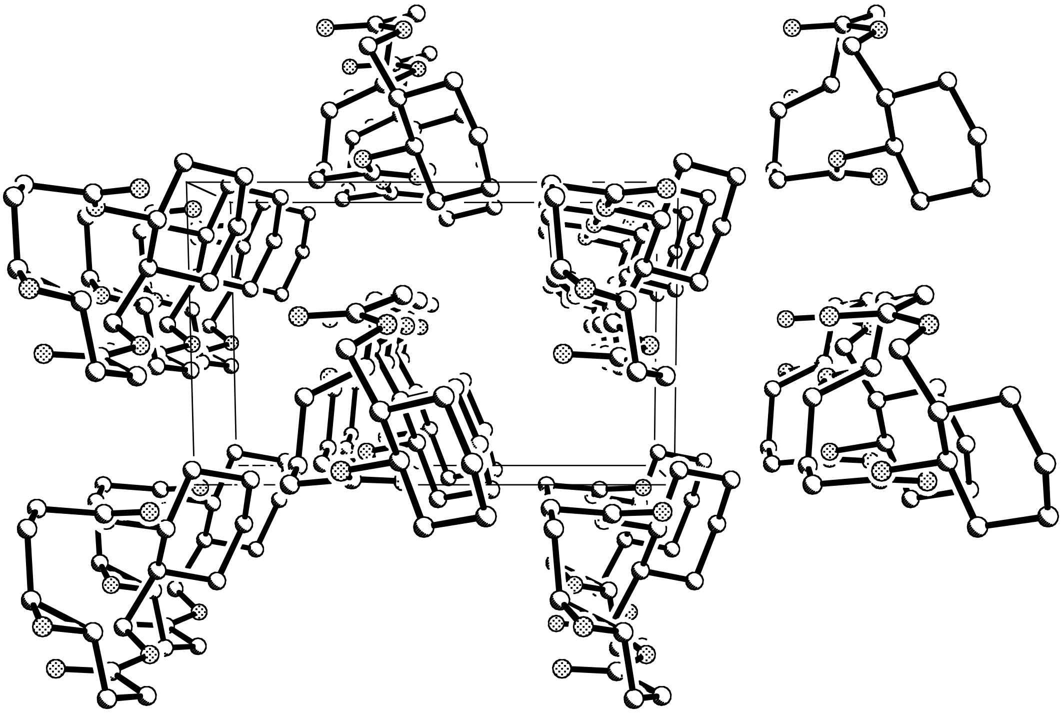


Table 1. Crystal data and structure refinement for uconn_mp07.

Identification code	uconn_mp07	
Empirical formula	$C_{15} H_{22} O_5$	
Formula weight	282.33	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 7.0375(14) Å	α= 90°.
	b = 11.004(2) Å	β= 102.95(3)°.
	c = 9.6494(19) Å	γ = 90°.
Volume	728.3(3) Å^3	
Z	2	
Density (calculated)	1.287 g/cm^3	
Absorption coefficient	0.96 cm^{-1}	
F(000)	304	
Crystal size	0.30 x 0.20 x 0.10 mm^3	
Theta range for data collection	2.97 to 28.98°.	
Index ranges	-9<=h<=9, -15<=k<=14, -13<=l<=13	
Reflections collected	3617	
Independent reflections	2167 [R(int) = 0.0000]	
Completeness to theta = 28.98°	98.5 %	
Absorption correction	None	
Max. and min. transmission	0.9905 and 0.9718	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3617 / 1 / 181	
Goodness-of-fit on F^2	1.009	
Final R indices [I>2sigma(I)]	R1 = 0.0507, wR2 = 0.0811	
R indices (all data)	R1 = 0.1130, wR2 = 0.0956	
Absolute structure parameter	-0.3(10)	
Largest diff. peak and hole	0.177 and -0.206 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for uconn_mp07. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	967(2)	8471(1)	2564(1)	30(1)
O(2)	525(2)	9561(1)	4435(1)	39(1)
O(3)	3649(2)	7955(1)	8203(1)	41(1)
O(4)	5572(2)	7613(1)	4426(1)	41(1)
O(5)	5322(2)	9327(1)	3138(1)	35(1)
C(1)	634(3)	8597(2)	3877(2)	28(1)
C(2)	418(3)	7381(2)	4532(2)	31(1)
C(3)	894(3)	7426(2)	6156(2)	34(1)
C(4)	3042(3)	7539(2)	6744(2)	32(1)
C(5)	3988(3)	8730(2)	7072(2)	32(1)
C(6)	6073(3)	8945(2)	7019(2)	38(1)
C(7)	6240(3)	9534(2)	5616(2)	33(1)
C(8)	5668(3)	8695(2)	4365(2)	31(1)
C(9)	4607(3)	8644(2)	1832(2)	34(1)
C(10)	2811(3)	9278(2)	958(2)	29(1)
C(11)	1319(3)	9582(2)	1832(2)	28(1)
C(12)	-555(3)	10048(2)	906(2)	36(1)
C(13)	-173(4)	11176(2)	92(2)	40(1)
C(14)	1402(4)	10947(2)	-737(2)	42(1)
C(15)	3257(4)	10437(2)	219(2)	35(1)

Table 3. Bond lengths [Å] and angles [°] for uconn_mp07.

O(1)-C(1)	1.346(2)	O(2)-C(1)-C(2)	125.17(18)
O(1)-C(11)	1.461(2)	O(1)-C(1)-C(2)	111.08(19)
O(2)-C(1)	1.200(2)	C(1)-C(2)-C(3)	112.54(18)
O(3)-C(5)	1.446(2)	C(4)-C(3)-C(2)	111.23(17)
O(3)-C(4)	1.451(2)	O(3)-C(4)-C(5)	59.29(13)
O(4)-C(8)	1.196(3)	O(3)-C(4)-C(3)	116.51(17)
O(5)-C(8)	1.347(2)	C(5)-C(4)-C(3)	121.70(19)
O(5)-C(9)	1.457(2)	O(3)-C(5)-C(4)	59.62(13)
C(1)-C(2)	1.503(3)	O(3)-C(5)-C(6)	116.59(18)
C(2)-C(3)	1.528(3)	C(4)-C(5)-C(6)	122.1(2)
C(3)-C(4)	1.496(3)	C(5)-C(6)-C(7)	111.55(17)
C(4)-C(5)	1.473(3)	C(8)-C(7)-C(6)	113.06(19)
C(5)-C(6)	1.499(3)	O(4)-C(8)-O(5)	123.8(2)
C(6)-C(7)	1.528(3)	O(4)-C(8)-C(7)	125.56(19)
C(7)-C(8)	1.501(3)	O(5)-C(8)-C(7)	110.65(19)
C(9)-C(10)	1.522(3)	O(5)-C(9)-C(10)	109.00(18)
C(10)-C(11)	1.524(3)	C(9)-C(10)-C(11)	112.56(16)
C(10)-C(15)	1.528(3)	C(9)-C(10)-C(15)	114.15(19)
C(11)-C(12)	1.507(3)	C(11)-C(10)-C(15)	108.30(17)
C(12)-C(13)	1.525(3)	O(1)-C(11)-C(12)	110.55(16)
C(13)-C(14)	1.526(4)	O(1)-C(11)-C(10)	107.26(16)
C(14)-C(15)	1.526(3)	C(12)-C(11)-C(10)	111.52(16)
		C(11)-C(12)-C(13)	110.54(19)
C(1)-O(1)-C(11)	116.95(16)	C(12)-C(13)-C(14)	111.59(19)
C(5)-O(3)-C(4)	61.10(13)	C(13)-C(14)-C(15)	111.57(19)
C(8)-O(5)-C(9)	116.96(17)	C(14)-C(15)-C(10)	110.6(2)
O(2)-C(1)-O(1)	123.7(2)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å^{2}x 10^3) for uconn_mp07. The anisotropic displacement factor exponent takes the form: $-2\pi^2$[h^2 $a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	41(1)	25(1)	26(1)	-2(1)	11(1)	-4(1)
O(2)	59(1)	26(1)	35(1)	0(1)	21(1)	7(1)
O(3)	54(1)	46(1)	24(1)	1(1)	9(1)	1(1)
O(4)	53(1)	30(1)	40(1)	5(1)	13(1)	4(1)
O(5)	41(1)	29(1)	31(1)	2(1)	3(1)	-4(1)
C(1)	25(1)	32(1)	27(1)	-1(1)	8(1)	3(1)
C(2)	35(1)	27(1)	32(1)	1(1)	9(1)	-2(1)
C(3)	44(2)	27(1)	33(1)	4(1)	14(1)	-2(1)
C(4)	43(2)	32(1)	23(1)	-1(1)	8(1)	3(1)
C(5)	41(2)	32(1)	22(1)	2(1)	8(1)	2(1)
C(6)	41(2)	38(1)	30(1)	0(1)	2(1)	-2(1)
C(7)	30(1)	30(1)	38(1)	1(1)	4(1)	-2(1)
C(8)	26(1)	33(1)	36(1)	4(1)	8(1)	2(1)
C(9)	39(1)	35(1)	30(1)	-2(1)	9(1)	1(1)
C(10)	37(1)	24(1)	26(1)	-2(1)	8(1)	-4(1)
C(11)	35(1)	23(1)	25(1)	2(1)	7(1)	-3(1)
C(12)	36(2)	33(1)	35(1)	-3(1)	3(1)	0(1)
C(13)	48(2)	32(1)	37(1)	4(1)	4(1)	9(1)
C(14)	59(2)	33(1)	32(1)	7(1)	8(1)	-1(1)
C(15)	46(2)	32(1)	28(1)	2(1)	9(1)	-6(1)

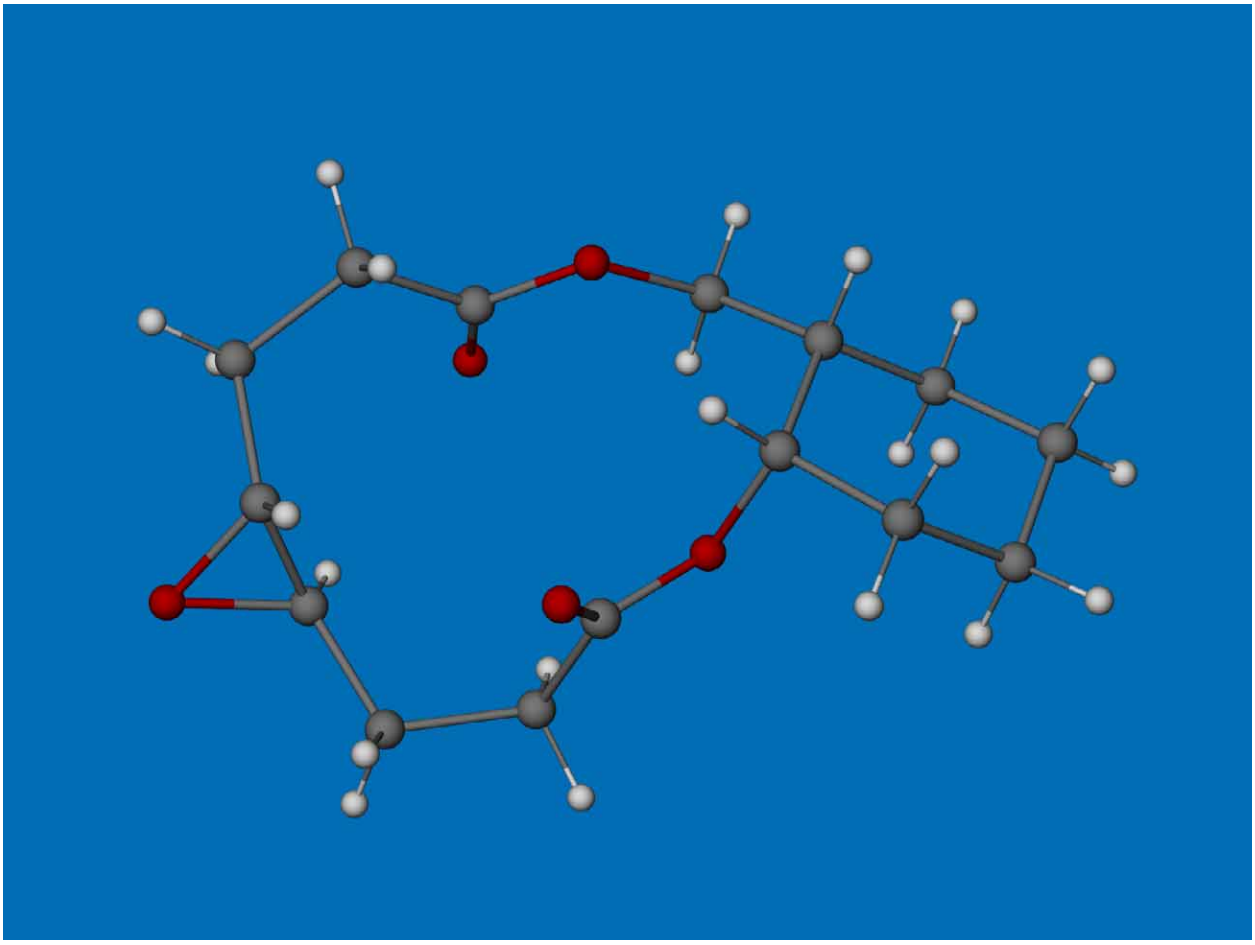
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for uconn_mp07.

	x	y	z	U(eq)
H(2A)	1294	6790	4218	38
H(2B)	-938	7091	4189	38
H(3A)	220	8127	6474	41
H(3B)	411	6677	6530	41
H(4A)	3867	6889	6453	39
H(5A)	3108	9451	6849	38
H(6A)	6781	8161	7132	45
H(6B)	6693	9479	7818	45
H(7A)	5396	10263	5450	40
H(7B)	7602	9803	5694	40
H(9A)	4270	7806	2063	41
H(9B)	5633	8598	1280	41
H(10A)	2174	8698	197	35
H(11A)	1873	10215	2553	33
H(12A)	-1484	10249	1505	43
H(12B)	-1150	9408	225	43
H(13A)	239	11852	770	48
H(13B)	-1395	11422	-577	48
H(14A)	1710	11719	-1166	50
H(14B)	905	10366	-1517	50
H(15A)	3833	11050	942	42
H(15B)	4221	10257	-360	42

YALE CHEMICAL INSTRUMENTATION CENTER

X-Ray Structure Report
Reference Number: UCONN_MP08

April 7, 2008



YALE CHEMICAL INSTRUMENTATION CENTER

X-Ray Structure Report
Reference Number: UCONN_MP08

April 7, 2008

Data Collection

A colorless plate crystal of $C_{15}H_{22}O_5$ having approximate dimensions of 0.15 x 0.15 x 0.08 mm^3 was mounted with epoxy cement on the tip of a fine glass fiber. All measurements were made on a Nonius KappaCCD diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 6.4153(13) Å	$\alpha = 90^{o}$
b = 12.269(3) Å	$\beta = 90^{o}$
c = 18.605(4) Å	$\gamma = 90^{o}$
V = 1464.4(5) Å^3	

For Z = 4 and F.W. = 282.33, the calculated density is 1.281 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be $P2_12_12_1$ (#19).

The data were collected at a temperature of 173(2) K to a maximum 2θ value of 57.94 °. Three omega scans consisting of 37, 29, and 22 data frames, respectively, were collected with a frame width of 2.0 ° and a detector-to-crystal distance, Dx, of 35.0 mm. Each frame was exposed twice (for the purpose of de-zingering) for a total of 60 s. The data frames were processed and scaled using the DENZO software package.[1]

Data Reduction

A total of 3801 reflections were collected of which 3801 were unique and observed (R_{int} = 0.000, Friedel pairs not merged). The linear absorption coefficient, μ, for Mo-Kα radiation is 0.95 cm^{-1}, and no absorption correction was applied. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were treated as idealized contributions. The final cycle of full-matrix least-squares refinement[3] on F was based on 3801 observed reflections ($I > 2.00\sigma(I)$) and 181 variable parameters and converged with unweighted and weighted agreement factors of:

$$R = \Sigma \,||Fo| - |Fc|| \,/\, \Sigma\, |Fo| = 0.0586$$
$$R_W = \{\Sigma[w\,(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]\}^{1/2} = 0.0833$$

The maximum and minimum peaks on the final difference Fourier map corresponded to 0.205 and –0.235 $e^-/Å^3$ respectively.

REFERENCES

(1) Z. Otwinowski and W. Minor, "Processing of X-Ray Diffraction Data Collected in Oscillation Mode," Methods in Enzymology, vol. 276: Macromolecular Crystallography, part A, 307-326, 1997, C.W. Carter, Jr. & R.M. Sweet, Eds., Academic Press.

(2) *SHELXTL*, v.6.12, Bruker-AXS, Madison, WI, 2001.

(3) Least Squares function minimized:
$$\Sigma w(F_o^2 - F_c^2)^2$$

Structural Description

The compound crystallized in the chiral orthorhombic space group $P2_12_12_1$ with one molecule in the asymmetric unit and four molecules in the unit cell.

Torsion angles of interest in the molecule are as follows: C(3)-C(4)-C(5)-C(6) = 155.2 °, O(1)-C(11)-C(10)-C(9) = 51.3 °, C(1)-C(2)-C(3)-C(4) = 73.3 °, C(5)-C(6)-C(7)-C(8) = 67.2 °.

An ambiguous Flack parameter was calculated due to the lack of substantially weighted atoms and thus the absolute configuration of the molecule could not be determined. There are no significant intermolecular contacts. ORTEPs, packing diagrams and full crystallographic tables follow.

Figure 1

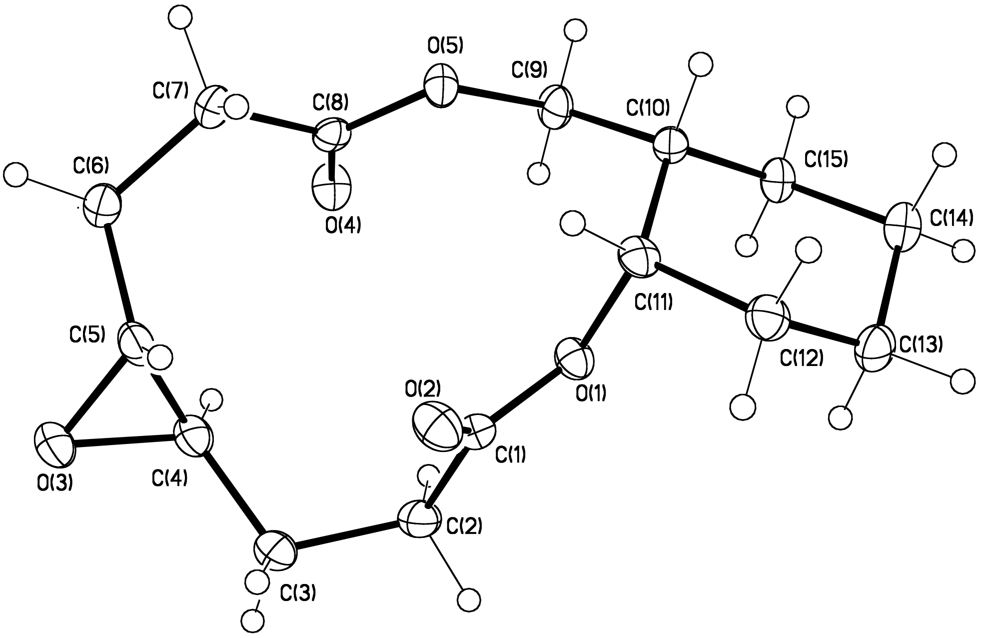


Figure 2

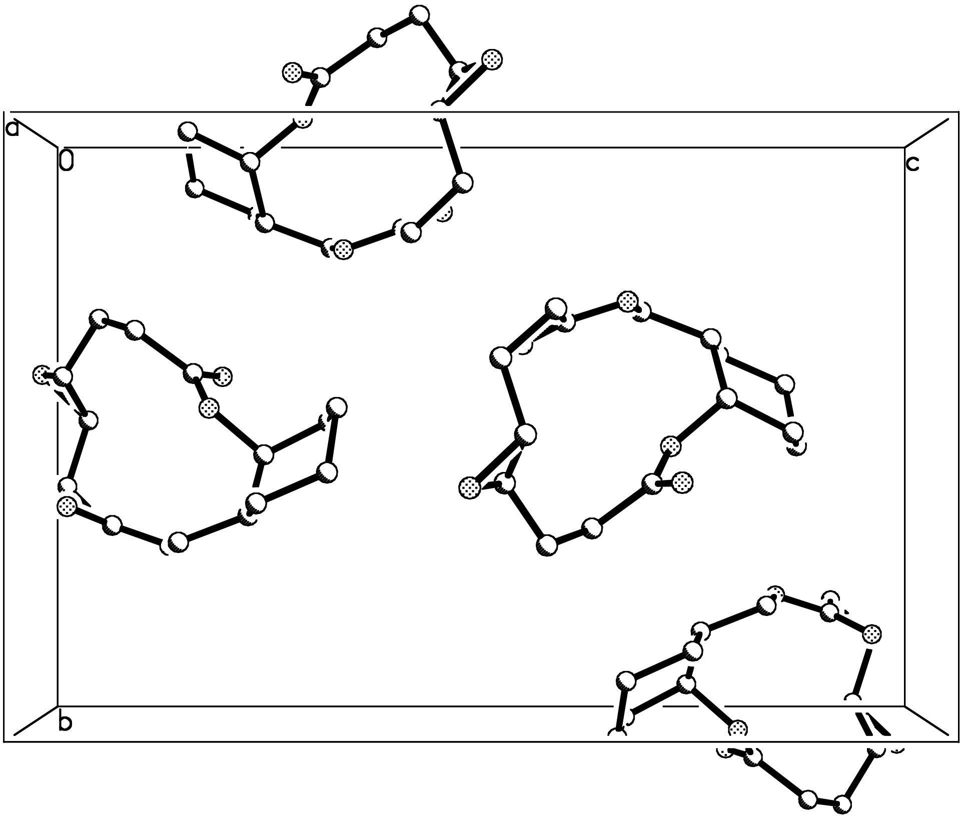
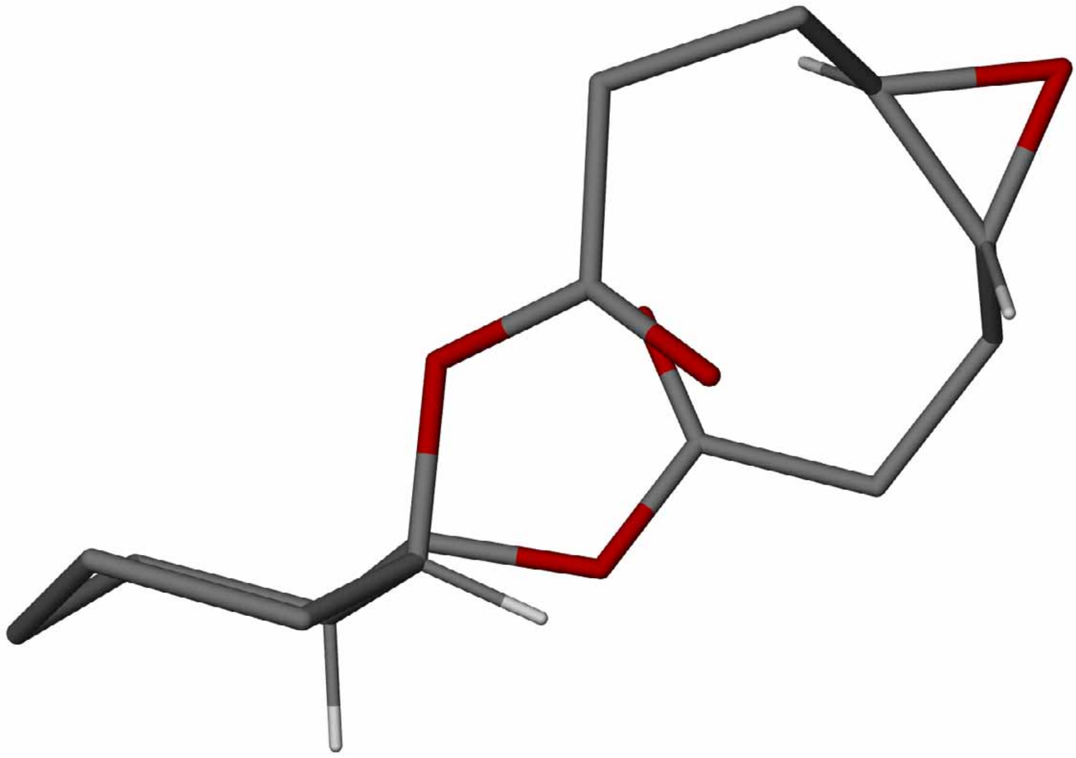
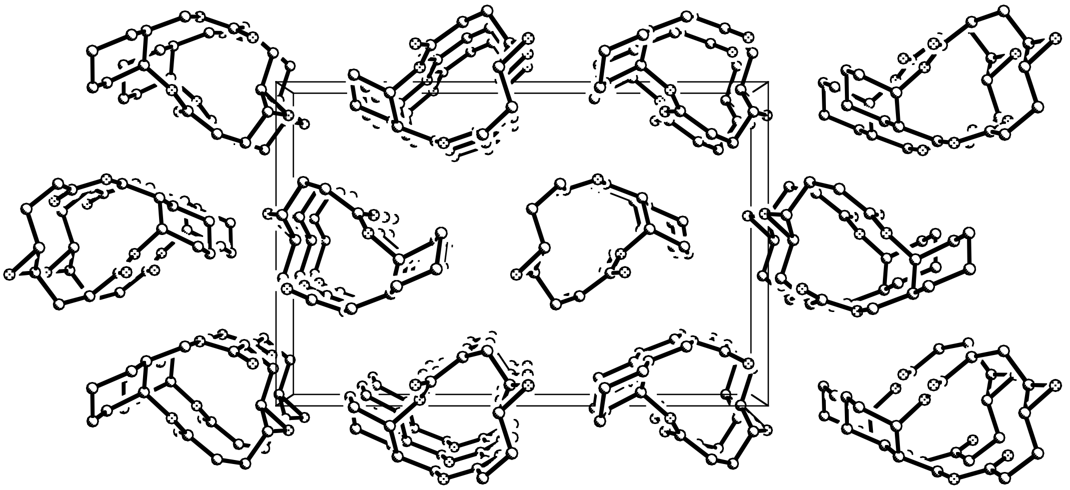


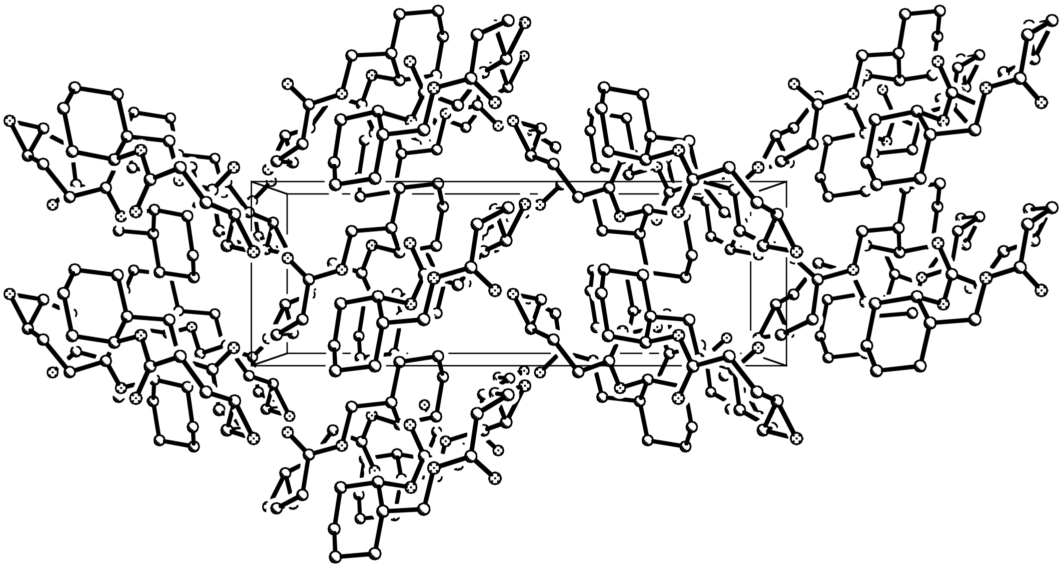
Figure 3



Packing diagram – View down the a-axis



Packing diagram – View down the b-axis



Packing diagram – View down the c-axis

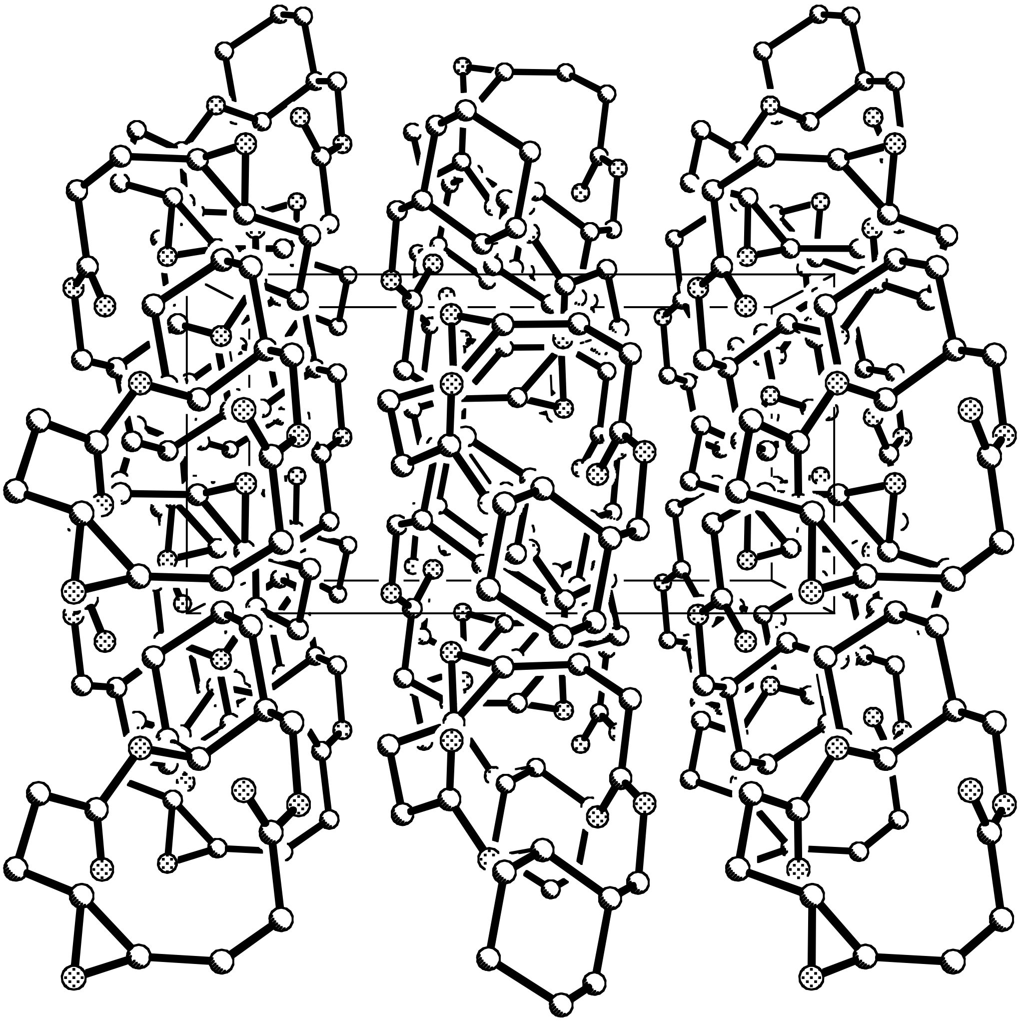


Table 1. Crystal data and structure refinement for uconn_mp08.

Identification code	uconn_mp08	
Empirical formula	$C_{15} H_{22} O_5$	
Formula weight	282.33	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 6.4153(13) Å	α= 90°.
	b = 12.269(3) Å	β= 90°.
	c = 18.605(4) Å	γ = 90°.
Volume	1464.4(5) $Å^3$	
Z	4	
Density (calculated)	1.281 g/cm^3	
Absorption coefficient	0.95 cm^{-1}	
F(000)	608	
Crystal size	0.15 x 0.15 x 0.08 mm^3	
Theta range for data collection	3.32 to 28.97°.	
Index ranges	-8<=h<=8, -16<=k<=16, -25<=l<=25	
Reflections collected	3801	
Independent reflections	2021 [R(int) = 0.0000]	
Completeness to theta = 28.97°	99.4 %	
Absorption correction	None	
Max. and min. transmission	0.9924 and 0.9859	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3801 / 0 / 181	
Goodness-of-fit on F^2	1.001	
Final R indices [I>2sigma(I)]	R1 = 0.0586, wR2 = 0.0833	
R indices (all data)	R1 = 0.1525, wR2 = 0.1022	
Absolute structure parameter	0.2(12)	
Largest diff. peak and hole	0.205 and -0.235 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for uconn_mp08. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	6662(2)	4687(1)	2028(1)	29(1)
O(2)	3324(3)	4135(1)	2060(1)	38(1)
O(3)	1174(3)	4080(1)	-114(1)	49(1)
O(4)	5892(2)	6329(1)	435(1)	35(1)
O(5)	5231(2)	6997(1)	1538(1)	29(1)
C(1)	5039(4)	4095(2)	1793(1)	28(1)
C(2)	5622(4)	3378(2)	1169(1)	34(1)
C(3)	3754(4)	3150(2)	678(1)	38(1)
C(4)	3180(4)	4130(2)	240(1)	35(1)
C(5)	1517(4)	4884(2)	444(1)	32(1)
C(6)	1475(4)	6050(2)	196(1)	30(1)
C(7)	2392(4)	6817(2)	757(1)	28(1)
C(8)	4664(4)	6668(2)	874(1)	25(1)
C(9)	7416(3)	6889(2)	1725(1)	31(1)
C(10)	7619(4)	6447(2)	2485(1)	25(1)
C(11)	6238(4)	5462(2)	2614(1)	28(1)
C(12)	6629(4)	4914(2)	3333(1)	34(1)
C(13)	8934(4)	4688(2)	3466(1)	36(1)
C(14)	10219(4)	5719(2)	3390(1)	35(1)
C(15)	9920(4)	6212(2)	2640(1)	31(1)

Table 3. Bond lengths [Å] and angles [°] for uconn_mp08.

O(1)-C(1)	1.342(3)
O(1)-C(11)	1.471(3)
O(2)-C(1)	1.208(3)
O(3)-C(4)	1.447(3)
O(3)-C(5)	1.449(3)
O(4)-C(8)	1.207(3)
O(5)-C(8)	1.351(3)
O(5)-C(9)	1.451(3)
C(1)-C(2)	1.504(3)
C(2)-C(3)	1.533(3)
C(3)-C(4)	1.499(3)
C(4)-C(5)	1.462(3)
C(5)-C(6)	1.503(3)
C(6)-C(7)	1.523(3)
C(7)-C(8)	1.485(3)
C(9)-C(10)	1.520(3)
C(10)-C(11)	1.518(3)
C(10)-C(15)	1.531(3)
C(11)-C(12)	1.519(3)
C(12)-C(13)	1.524(3)
C(13)-C(14)	1.517(3)
C(14)-C(15)	1.532(3)
C(1)-O(1)-C(11)	116.56(17)
C(4)-O(3)-C(5)	60.65(14)
C(8)-O(5)-C(9)	116.87(18)
O(2)-C(1)-O(1)	123.4(2)
O(2)-C(1)-C(2)	124.6(2)
O(1)-C(1)-C(2)	112.0(2)
C(1)-C(2)-C(3)	111.9(2)
C(4)-C(3)-C(2)	111.7(2)
O(3)-C(4)-C(5)	59.74(15)
O(3)-C(4)-C(3)	115.6(2)
C(5)-C(4)-C(3)	123.1(2)
O(3)-C(5)-C(4)	59.61(15)
O(3)-C(5)-C(6)	115.22(19)
C(4)-C(5)-C(6)	122.4(2)
C(5)-C(6)-C(7)	111.81(19)
C(8)-C(7)-C(6)	113.8(2)
O(4)-C(8)-O(5)	123.1(2)
O(4)-C(8)-C(7)	125.7(2)
O(5)-C(8)-C(7)	111.2(2)
O(5)-C(9)-C(10)	109.75(18)
C(11)-C(10)-C(9)	112.40(19)
C(11)-C(10)-C(15)	112.5(2)
C(9)-C(10)-C(15)	108.99(19)
O(1)-C(11)-C(10)	106.86(18)
O(1)-C(11)-C(12)	109.63(18)
C(10)-C(11)-C(12)	113.29(19)
C(11)-C(12)-C(13)	112.5(2)
C(14)-C(13)-C(12)	111.1(2)
C(13)-C(14)-C(15)	110.3(2)
C(10)-C(15)-C(14)	111.51(19)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for uconn_mp08. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	27(1)	33(1)	28(1)	-5(1)	1(1)	0(1)
O(2)	32(1)	40(1)	42(1)	-7(1)	2(1)	-6(1)
O(3)	63(1)	38(1)	48(1)	-1(1)	-27(1)	-9(1)
O(4)	29(1)	48(1)	27(1)	-1(1)	3(1)	0(1)
O(5)	24(1)	37(1)	25(1)	-2(1)	-3(1)	3(1)
C(1)	33(2)	22(1)	31(1)	1(1)	-5(1)	2(1)
C(2)	43(2)	26(2)	34(1)	-2(1)	-2(1)	2(1)
C(3)	49(2)	29(2)	35(1)	-6(1)	-8(1)	1(1)
C(4)	42(2)	33(2)	28(1)	-4(1)	-6(1)	-3(1)
C(5)	32(1)	34(2)	30(1)	1(1)	-7(1)	-9(1)
C(6)	28(1)	34(2)	30(1)	4(1)	-2(1)	-1(1)
C(7)	27(1)	32(2)	26(1)	6(1)	-2(1)	1(1)
C(8)	30(1)	21(1)	23(1)	6(1)	2(1)	-1(1)
C(9)	19(1)	38(2)	35(1)	2(1)	-4(1)	-2(1)
C(10)	26(1)	25(1)	24(1)	-1(1)	-3(1)	2(1)
C(11)	30(1)	31(1)	24(1)	-7(1)	4(1)	4(1)
C(12)	42(2)	34(2)	26(1)	0(1)	1(1)	-2(1)
C(13)	45(2)	38(2)	25(1)	5(1)	-2(1)	3(1)
C(14)	32(1)	45(2)	30(1)	3(1)	-5(1)	2(1)
C(15)	26(1)	39(2)	28(1)	2(1)	-4(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for uconn_mp08.

	x	y	z	U(eq)
H(2A)	6177	2679	1353	41
H(2B)	6737	3736	887	41
H(3A)	4097	2538	352	45
H(3B)	2545	2929	975	45
H(4A)	4336	4454	-51	41
H(5A)	824	4734	915	38
H(6A)	17	6266	95	36
H(6B)	2278	6117	-256	36
H(7A)	2134	7578	604	34
H(7B)	1658	6702	1219	34
H(9A)	8108	7609	1692	37
H(9B)	8110	6389	1383	37
H(10A)	7159	7034	2822	30
H(11A)	4746	5693	2588	34
H(12A)	5852	4217	3350	40
H(12B)	6089	5386	3722	40
H(13A)	9117	4388	3956	43
H(13B)	9434	4137	3118	43
H(14A)	11711	5549	3466	42
H(14B)	9787	6254	3759	42
H(15A)	10725	6899	2605	37
H(15B)	10467	5701	2274	37

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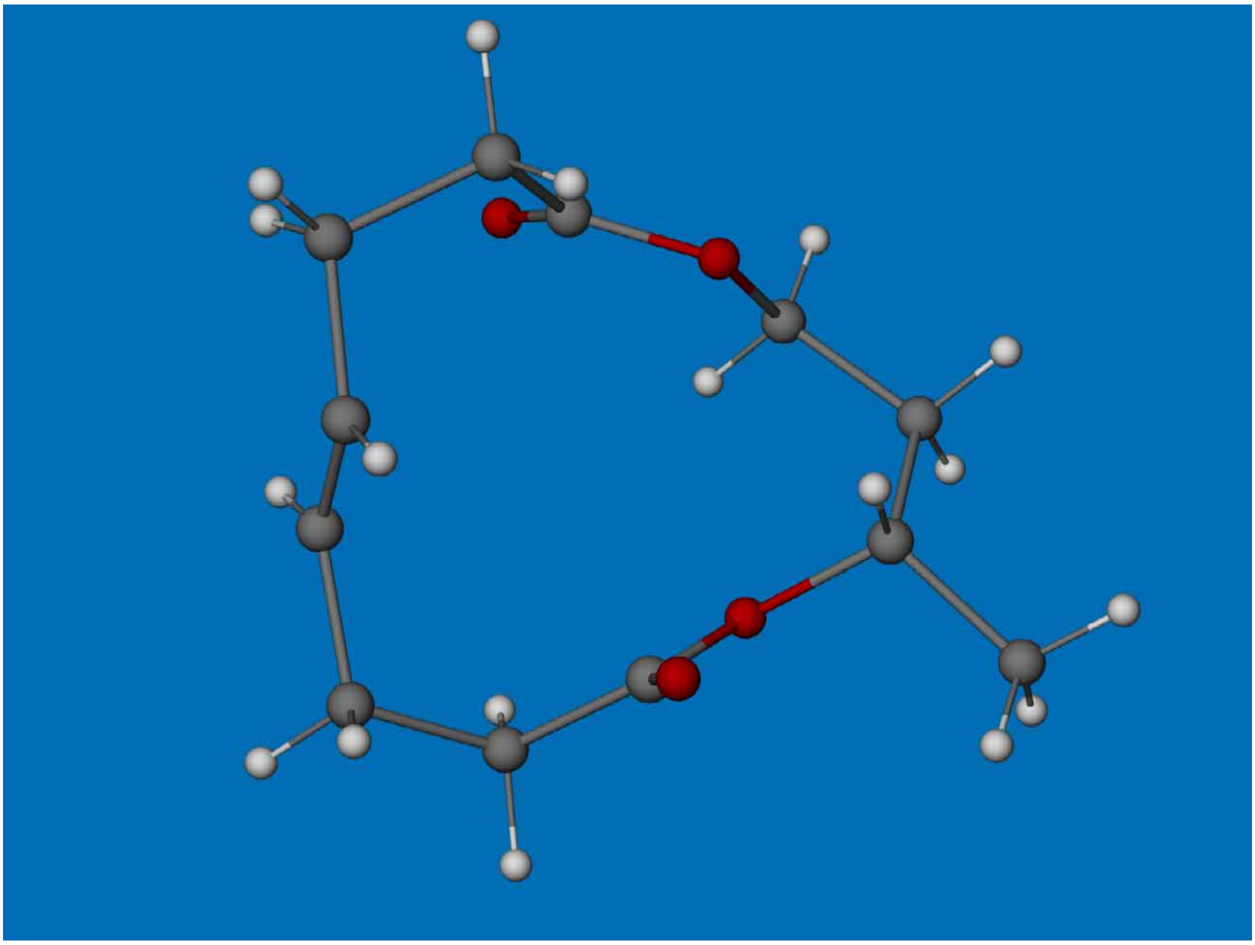
X-Ray Structure Report
Reference Number: UCONN_MP09

April 7, 2008



YALE CHEMICAL INSTRUMENTATION CENTER

X-Ray Structure Report
Reference Number: UCONN_MP09

April 7, 2008

Data Collection

A colorless plate crystal of $C_{12}H_{18}O_4$ having approximate dimensions of 0.20 x 0.10 x 0.08 mm^3 was mounted with epoxy cement on the tip of a fine glass fiber. All measurements were made on a Nonius KappaCCD diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

$a = 7.8101(16)$ Å $\alpha = 90^{o}$
$b = 18.266(4)$ Å $\beta = 112.26(3)^{o}$
$c = 9.2291(18)$ Å $\gamma = 90^{o}$
$V = 1218.5(4)$ Å^3

For Z = 4 and F.W. = 226.26, the calculated density is 1.233 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be $P2_1/n$ (#14).

The data were collected at a temperature of 173(2) K to a maximum 2θ value of 58.00 °. Four omega scans consisting of 75, 75, 39, and 54 data frames, respectively, were collected with a frame width of 1.0 ° and a detector-to-crystal distance, Dx, of 35.0 mm. Each frame was exposed twice (for the purpose of de-zingering) for a total of 80 s. The data frames were processed and scaled using the DENZO software package.[1]

Data Reduction

A total of 5332 reflections were collected of which 3180 were unique and observed (R_{int} = 0.0614). The linear absorption coefficient, μ, for Mo-Kα radiation is 0.92 cm^{-1}, and no absorption correction was applied. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were treated as idealized contributions. The final cycle of full-matrix least-squares refinement[3] on F was based on 3180 observed reflections ($I > 2.00\sigma(I)$) and 145 variable parameters and converged with unweighted and weighted agreement factors of:

$$R = \Sigma\ ||Fo| - |Fc|| / \Sigma\ |Fo| = 0.586$$
$$R_W = \{\Sigma[w\ (F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2} = 0.1110$$

The maximum and minimum peaks on the final difference Fourier map corresponded to 0.204 and –0.226 $e^-/Å^3$ respectively.

REFERENCES

(1) Z. Otwinowski and W. Minor, "Processing of X-Ray Diffraction Data Collected in Oscillation Mode," Methods in Enzymology, vol. 276: Macromolecular Crystallography, part A, 307-326, 1997, C.W. Carter, Jr. & R.M. Sweet, Eds., Academic Press.

(2) *SHELXTL*, v.6.12, Bruker-AXS, Madison, WI, 2001.

(3) Least Squares function minimized:

$$\Sigma w(F_o^2 - F_c^2)^2$$

Structural Description

The compound crystallized in the monoclinic space group $P2_1/n$ with one molecule in the asymmetric unit and four molecules in the unit cell.

Torsion angles of interest in the molecule are as follows: C(3)-C(4)-C(5)-C(6) = 174.8 °, C(12)-C(11)-O(1)-C(1) = 87.5 °, C(12)-C(11)-C(10)-C(9) = 169.9 °.

There are no significant intermolecular contacts. ORTEPs, packing diagrams and full crystallographic tables follow.

Figure 1

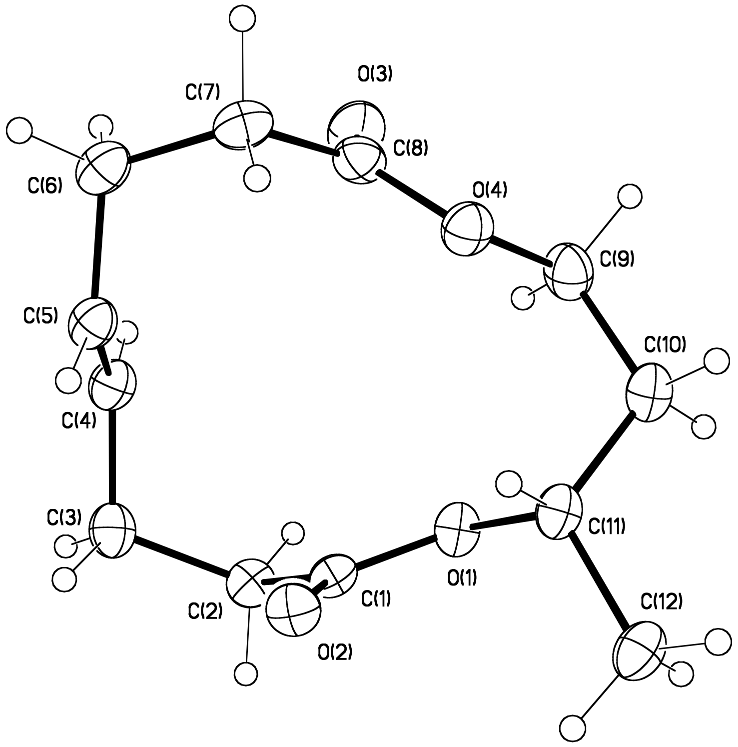


Figure 2

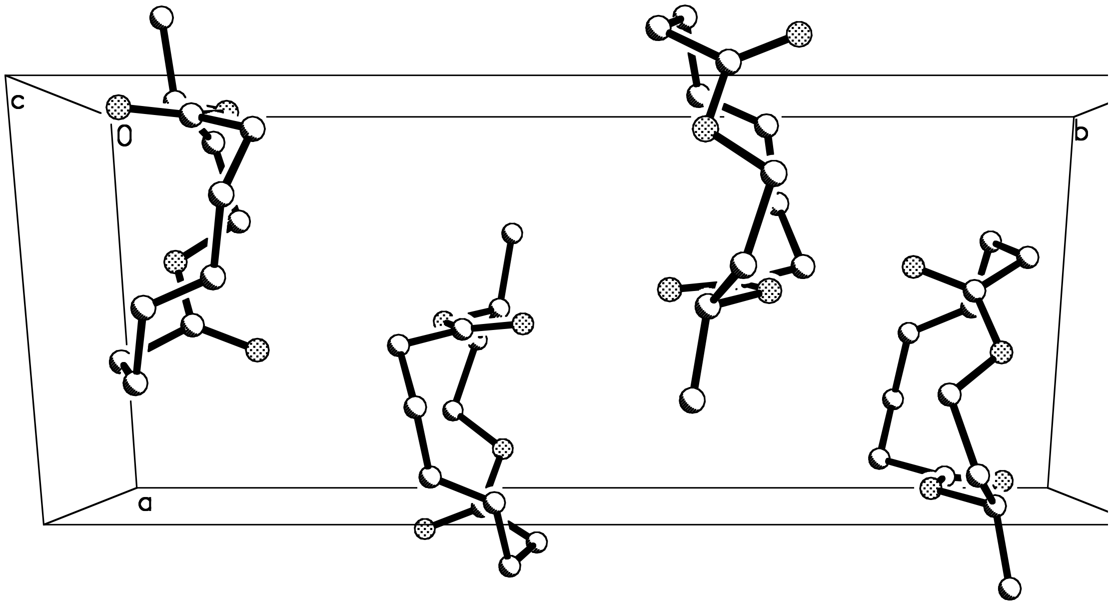
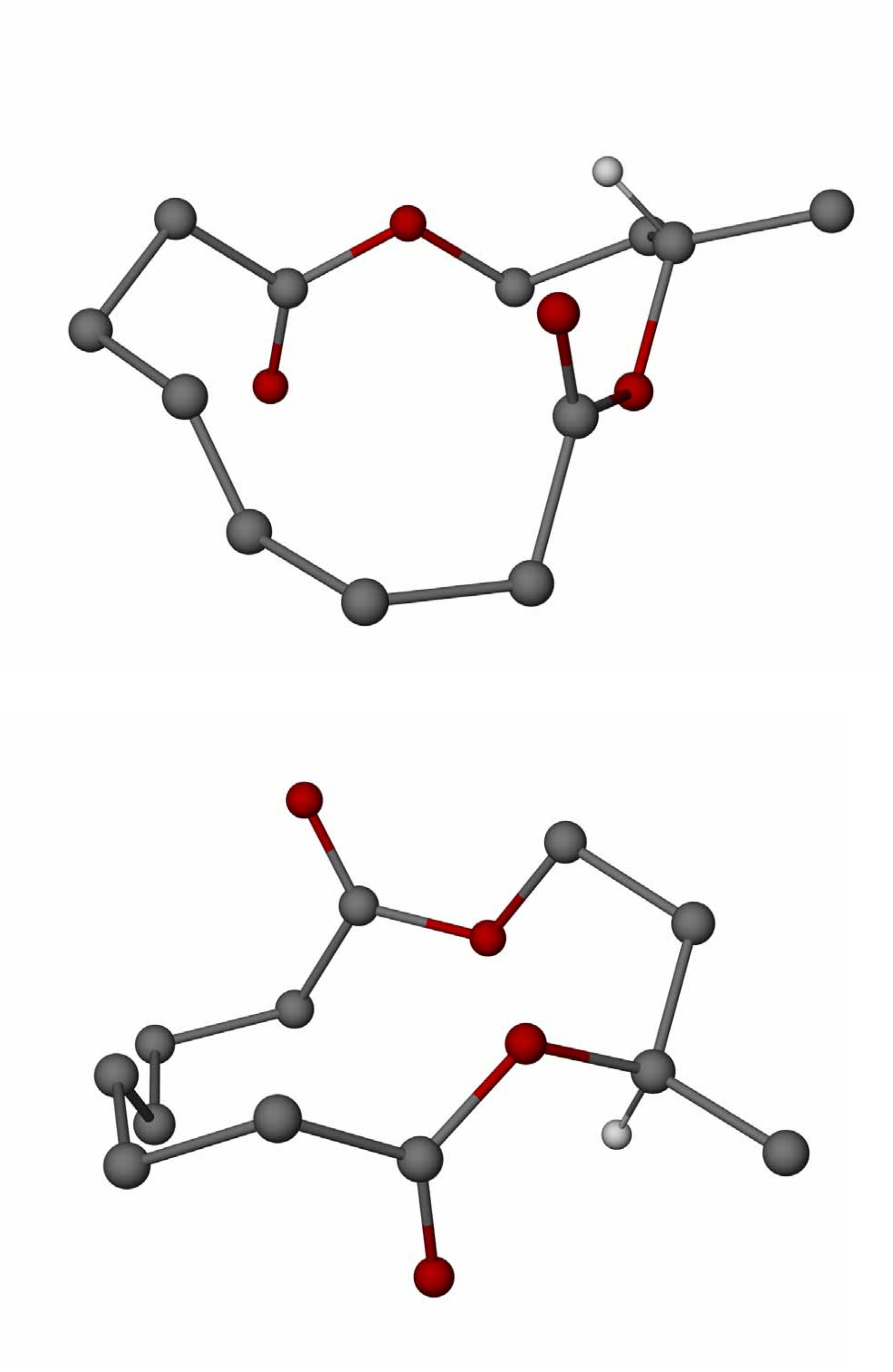
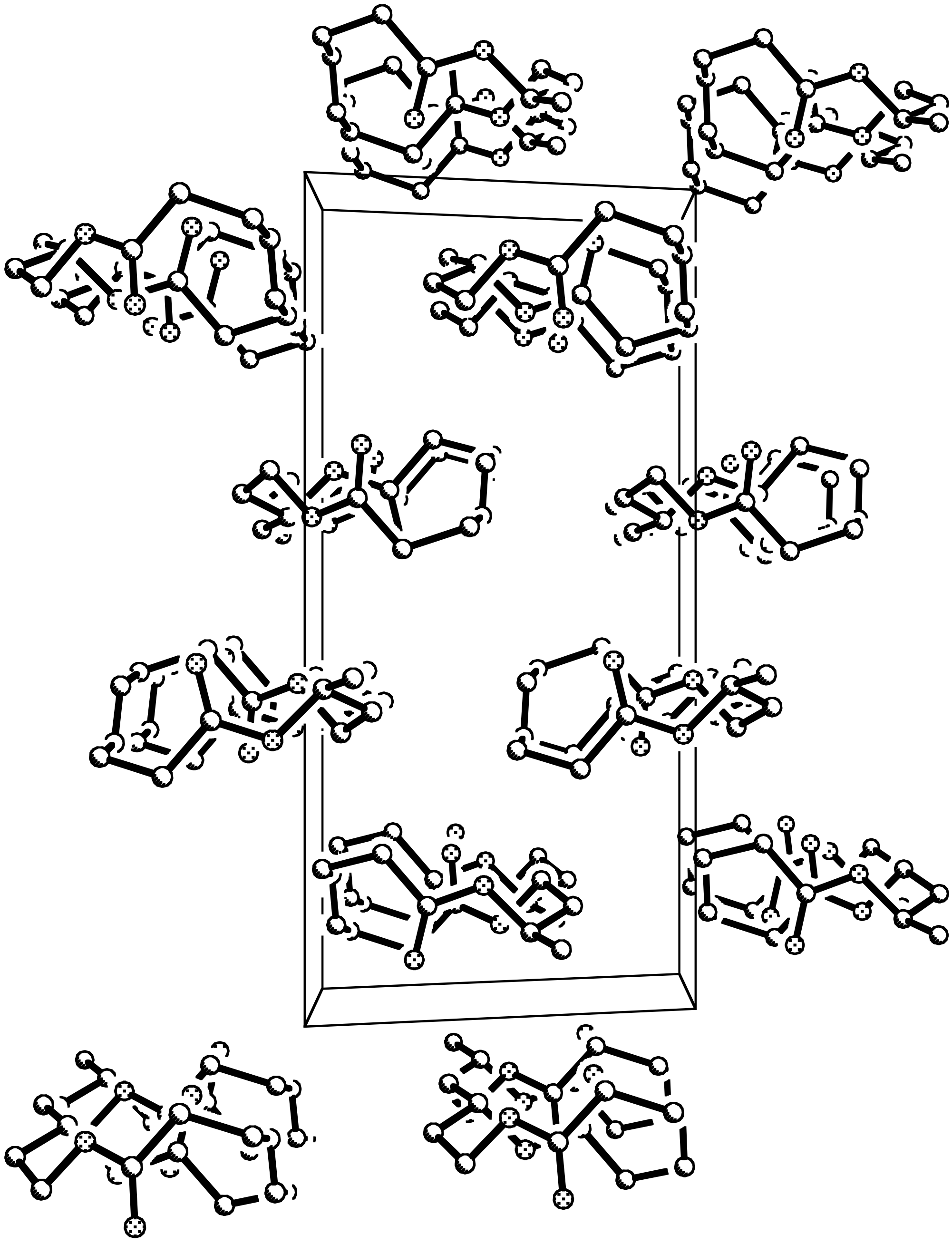


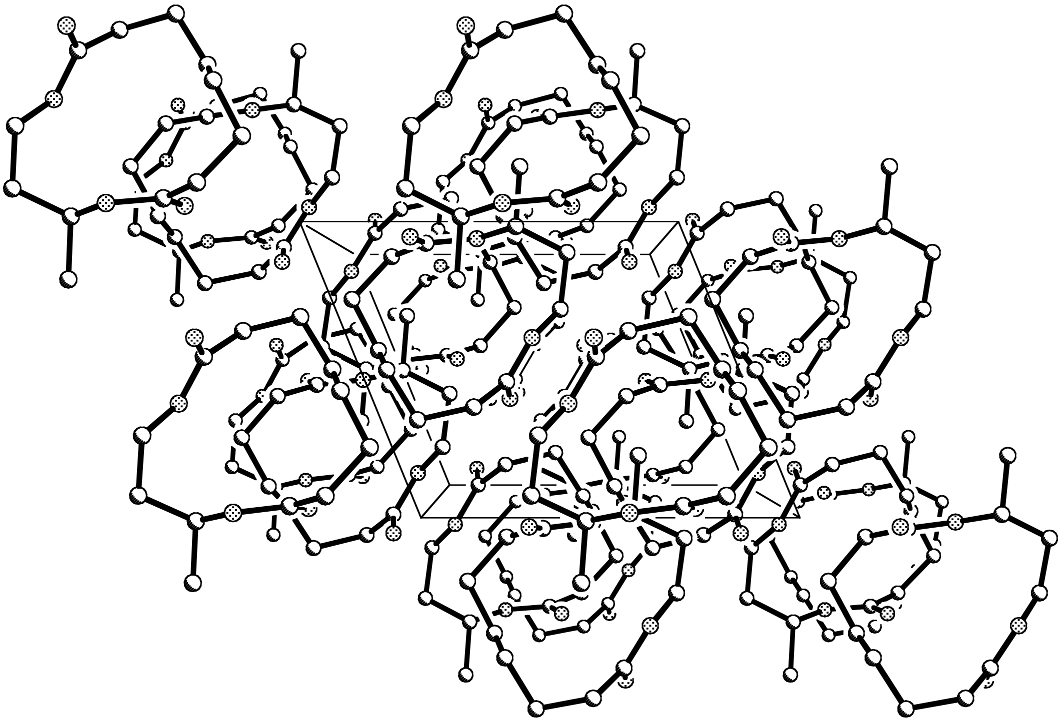
Figure 3



Packing diagram – View down the a-axis



Packing diagram – View down the b-axis



Packing diagram – View down the c-axis

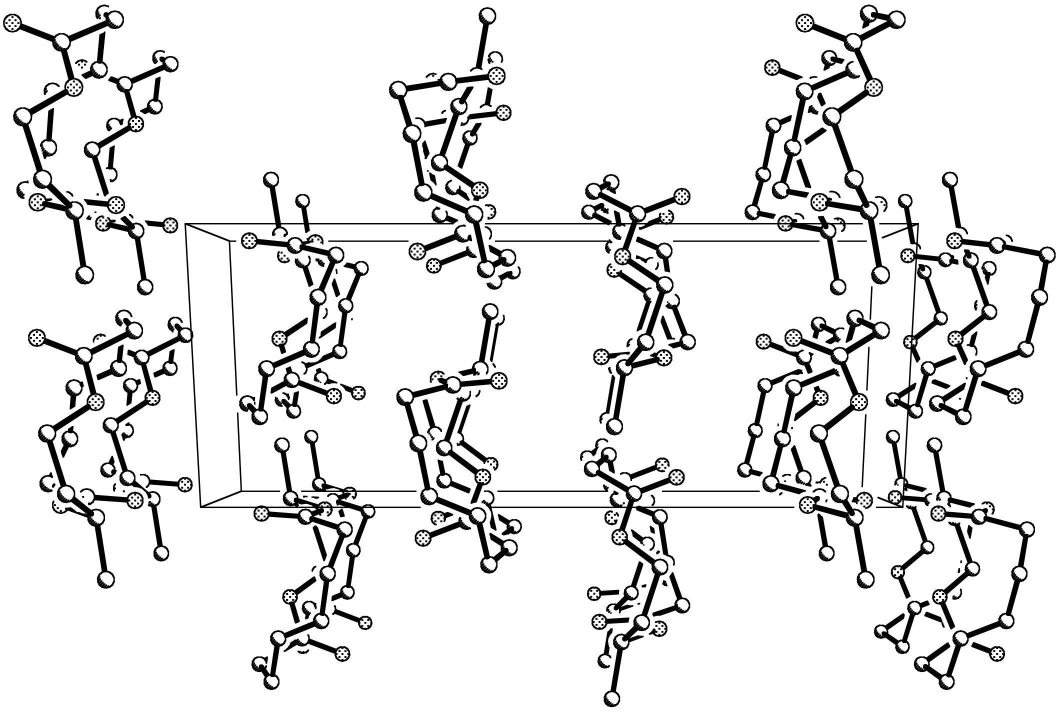


Table 1. Crystal data and structure refinement for uconn_mp09.

Identification code	uconn_mp09	
Empirical formula	$C_{12} H_{18} O_4$	
Formula weight	226.26	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 7.8101(16) Å	α= 90°.
	b = 18.266(4) Å	β= 112.26(3)°.
	c = 9.2291(18) Å	γ = 90°.
Volume	1218.5(4) $Å^3$	
Z	4	
Density (calculated)	1.233 g/cm^3	
Absorption coefficient	0.92 cm^{-1}	
F(000)	488	
Crystal size	0.20 x 0.10 x 0.08 mm^3	
Theta range for data collection	2.23 to 29.00°.	
Index ranges	-10<=h<=10, -24<=k<=23, -12<=l<=12	
Reflections collected	5332	
Independent reflections	3180 [R(int) = 0.0614]	
Completeness to theta = 29.00°	98.0 %	
Absorption correction	None	
Max. and min. transmission	0.9927 and 0.9819	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3180 / 0 / 145	
Goodness-of-fit on F^2	1.003	
Final R indices [I>2sigma(I)]	R1 = 0.0586, wR2 = 0.1110	
R indices (all data)	R1 = 0.1480, wR2 = 0.1376	
Largest diff. peak and hole	0.204 and -0.226 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for uconn_mp09. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	377(2)	1573(1)	5531(1)	38(1)
O(2)	422(2)	714(1)	7294(1)	40(1)
O(3)	6024(2)	1804(1)	6431(2)	59(1)
O(4)	3887(2)	975(1)	5122(2)	44(1)
C(1)	566(3)	1345(1)	6972(2)	35(1)
C(2)	970(3)	1967(1)	8115(2)	41(1)
C(3)	2504(3)	1762(1)	9682(2)	44(1)
C(4)	4318(3)	1637(1)	9514(2)	41(1)
C(5)	5006(3)	994(1)	9425(2)	40(1)
C(6)	6735(3)	851(1)	9130(2)	45(1)
C(7)	6286(3)	588(1)	7452(2)	45(1)
C(8)	5423(3)	1191(1)	6312(2)	43(1)
C(9)	2870(3)	1537(1)	4027(2)	48(1)
C(10)	902(3)	1275(1)	3216(2)	44(1)
C(11)	26(3)	1005(1)	4329(2)	37(1)
C(12)	-2028(3)	867(1)	3558(2)	45(1)

Table 3. Bond lengths [Å] and angles [°] for uconn_mp09.

O(1)-C(1)	1.347(2)	C(8)-O(4)-C(9)	116.46(15)
O(1)-C(11)	1.467(2)	O(2)-C(1)-O(1)	123.56(16)
O(2)-C(1)	1.207(2)	O(2)-C(1)-C(2)	124.40(17)
O(3)-C(8)	1.204(2)	O(1)-C(1)-C(2)	112.04(16)
O(4)-C(8)	1.343(2)	C(1)-C(2)-C(3)	111.01(16)
O(4)-C(9)	1.449(2)	C(4)-C(3)-C(2)	111.79(17)
C(1)-C(2)	1.501(3)	C(5)-C(4)-C(3)	124.80(19)
C(2)-C(3)	1.534(3)	C(4)-C(5)-C(6)	126.05(19)
C(3)-C(4)	1.500(3)	C(5)-C(6)-C(7)	111.21(17)
C(4)-C(5)	1.306(3)	C(8)-C(7)-C(6)	110.56(17)
C(5)-C(6)	1.499(3)	O(3)-C(8)-O(4)	123.05(19)
C(6)-C(7)	1.529(3)	O(3)-C(8)-C(7)	124.37(19)
C(7)-C(8)	1.495(3)	O(4)-C(8)-C(7)	112.58(17)
C(9)-C(10)	1.511(3)	O(4)-C(9)-C(10)	107.86(16)
C(10)-C(11)	1.516(3)	C(9)-C(10)-C(11)	113.78(16)
C(11)-C(12)	1.509(3)	O(1)-C(11)-C(12)	109.61(16)
		O(1)-C(11)-C(10)	106.23(15)
C(1)-O(1)-C(11)	116.58(14)	C(12)-C(11)-C(10)	113.98(16)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å^{2}x 10^3) for uconn_mp09. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	46(1)	28(1)	36(1)	-2(1)	11(1)	0(1)
O(2)	44(1)	31(1)	47(1)	2(1)	18(1)	-3(1)
O(3)	54(1)	56(1)	62(1)	4(1)	15(1)	-21(1)
O(4)	42(1)	43(1)	44(1)	-3(1)	14(1)	-5(1)
C(1)	28(1)	34(1)	40(1)	0(1)	10(1)	3(1)
C(2)	43(1)	34(1)	45(1)	-3(1)	17(1)	3(1)
C(3)	53(1)	40(1)	37(1)	-6(1)	14(1)	0(1)
C(4)	41(1)	39(1)	39(1)	-6(1)	9(1)	-6(1)
C(5)	39(1)	35(1)	41(1)	4(1)	11(1)	0(1)
C(6)	37(1)	41(1)	51(1)	2(1)	10(1)	-2(1)
C(7)	34(1)	46(1)	55(1)	-3(1)	16(1)	-3(1)
C(8)	38(1)	48(1)	46(1)	-6(1)	19(1)	-12(1)
C(9)	56(2)	48(1)	42(1)	6(1)	18(1)	-5(1)
C(10)	49(1)	42(1)	36(1)	2(1)	9(1)	-2(1)
C(11)	42(1)	29(1)	35(1)	-3(1)	9(1)	-1(1)
C(12)	41(1)	41(1)	45(1)	-2(1)	6(1)	5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for uconn_mp09.

	x	y	z	U(eq)
H(2A)	-168	2096	8291	49
H(2B)	1356	2401	7673	49
H(3A)	2647	2160	10447	53
H(3B)	2143	1312	10093	53
H(4A)	5019	2055	9470	50
H(5A)	4349	579	9560	48
H(6A)	7484	475	9872	54
H(6B)	7478	1306	9314	54
H(7A)	7435	423	7337	54
H(7B)	5426	167	7226	54
H(9A)	3438	1626	3249	58
H(9B)	2894	2000	4590	58
H(10A)	882	873	2490	53
H(10B)	145	1681	2583	53
H(11A)	654	542	4837	45
H(12A)	-2498	697	4344	68
H(12B)	-2261	493	2744	68
H(12C)	-2658	1322	3084	68