Supporting Information

High Chelation-Control of Three Contiguous Stereogenic Centers in the Reformatsky Reactions of Indium Enolates with α-Hydroxy Ketones: Unexpected Stereochemistry of Lactone Formation

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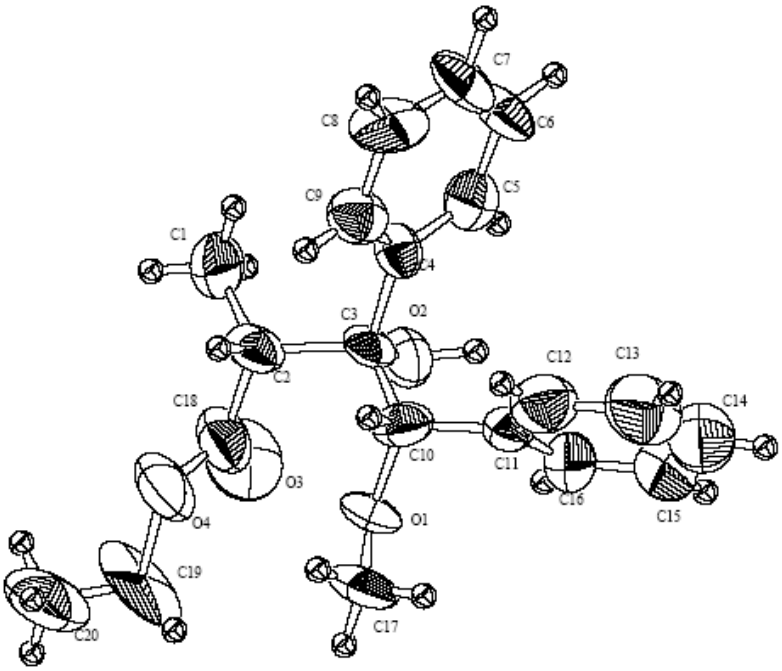
Contents of Supporting Information:

Determination of the relative stereochemistry:

a) Determination of stereochemistry of Reformatsky-type reactions of branched α-halo esters with α-alkoxy ketones:

Determination of stereochemistry of isomer 3a: The column chromatography purification of the reaction of **1a** with **2a** in the presence of indium afforded the major isomer **3a** in pure form as colorless solid. Repetitive crystallization afforded suitable crystalline for X-ray structure analysis. The X-ray structure analysis thus unambiguously revealed the stereochemistry of **3a**.

For X-ray analysis details see page S58.



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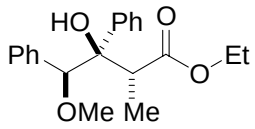
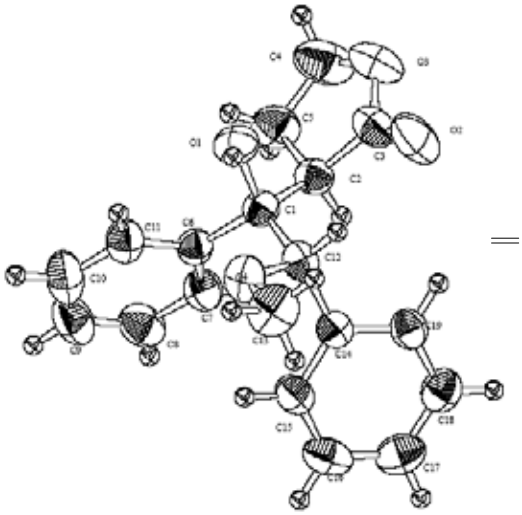


3a

Determination of stereochemistry of isomers 3b and 4b: The repetitive column chromatography purification of the reaction of **1a** with **2b** in the presence of indium afforded both isomers in pure form (**3b**, major isomer) and (**4b** minor isomer) as colorless solids. Repetitive crystallization afforded suitable crystalline for X-ray structure analyses. The X-ray structure analyses thus unambiguously revealed the stereochemistry of **3b** and **4b**.

For X-ray analysis details see pages S73 and S87.



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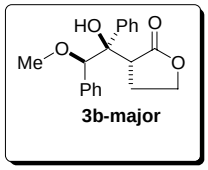
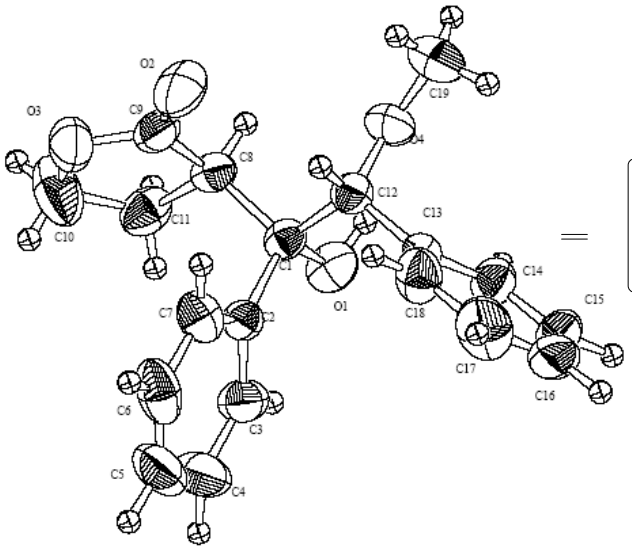


3b-major



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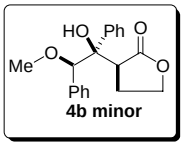
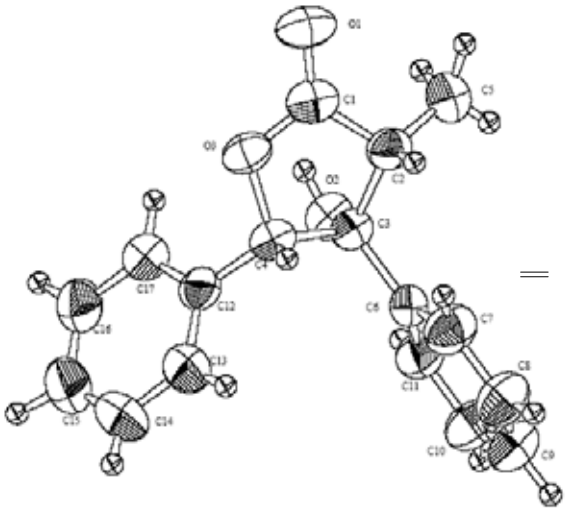


4b minor

b) Determination of stereochemistry of Reformatsky-type reactions of branched α-halo esters with α-hydroxy ketones:

Determination of stereochemistry of isomer 8a: The column chromatography purification of the reaction of **5a** with **2a** in the presence of indium afforded the isomer **8a** as colorless solid. Repetitive crystallization afforded suitable crystalline for X-ray structure analysis. The X-ray structure analysis thus unambiguously revealed the stereochemistry of **8a**.

For X-ray analysis details see page S101.



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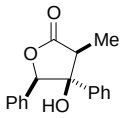
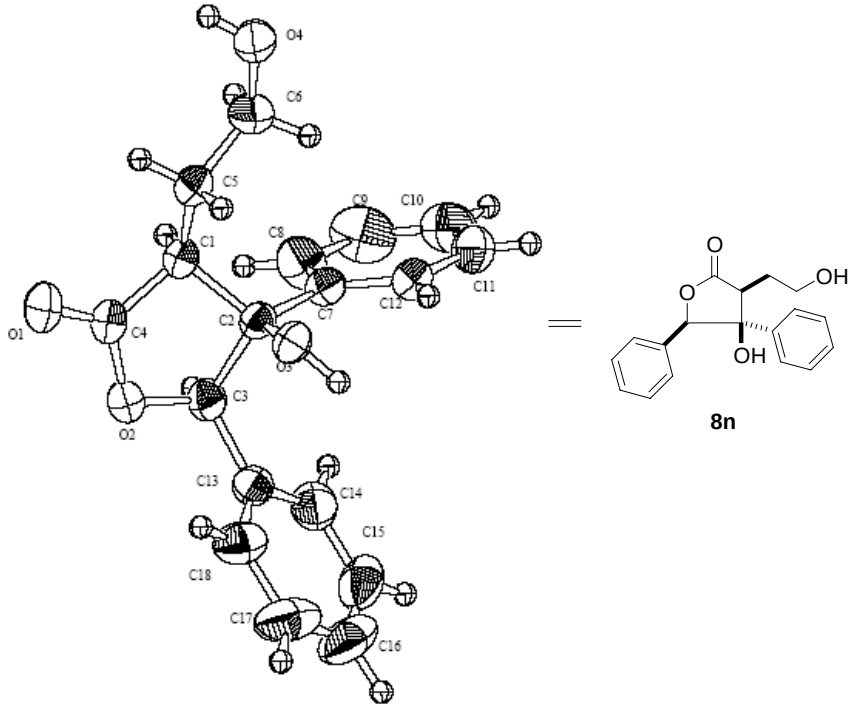


8a

Determination of stereochemistry of isomer 8n: The column chromatography purification of the reaction of **5a** with **2b** in the presence of indium afforded major isomer in pure form (**8n**, major isomer) as colorless solid. Repetitive crystallization afforded suitable crystalline for X-ray structure analysis. The X-ray structure analysis thus unambiguously revealed the stereochemistry of **8n**.

For X-ray analysis details see page S114.



8n

Experimental Section

General. Ultrasonication was carried out using Eyela (MUS 10) ultrasonic cleaner (38 kHz, 120 W). Melting points are uncorrected. IR spectra were recorded as thin films or KBr pellets on a HORIBA FT-720 spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on JEOL JNM-GSX-270 or 400 (270/67.9 or 400/100 MHz) spectrometers, respectively with TMS as internal or external standard. Carbon types were determined in DEPT ^{13}C NMR spectral analyses. Mass spectra were recorded on a JEOL JMS-DS303 spectrometer. Column chromatography was performed on silica gel (100-200 mesh, Wako silica gel). Reactions were carried out either under ultrasonication (bath temperature about 10-25 °C, ice was added time to time to the ultrasonication water bath) or at reflux temperature under an inert atmosphere. Solutions were dried with anhydrous magnesium sulfate. Reagents were added to the reaction flask through syringe. Analytical thin layer chromatography (TLC) was performed on silica plates and components were visualized by observation under iodine or UV light. Yields were determined from ^{1}H NMR spectra using internal standards or after isolation in column chromatography. Ratios of diastereomers were determined from H^{1} NMR of crude reaction mixture.

General procedures for indium-employed Reformatsky-type reaction of α-alkoxy ketones with halo esters:

In or In(I)X system (Method A): To a solution of an appropriate α-alkoxy ketone (0.5 or 1 mmol) and an appropriate α- halo ester (mmol as denoted in the tables, added *via* syringe) in dry THF or toluene (2 mL) was added fresh In or indium(I) halide (mmol as denoted in the tables) at room temperature. The resulting reaction mixture was subjected

to ultrasonication at rt or stirred at rt or dipped into a preheated hot bath (66-70 °C) under an inert atmosphere and stirred for an appropriate period (see Tables for specific entry). To this mixture cold water (5-7 mL) was added and the contents were subjected to ultrasonication for 2 min. Further, extraction was carried out using diethyl ether (3 X 15 mL). The combined organic layers were concentrated in vacuum and subjected to column to silica gel (5-30% EtOAc-Hexane mixture) chromatographic purification to afford the pure products.

In-InX_3 Combined system (Method B): To a solution of an appropriate ketone (0.5 mmol) and an appropriate α- halo ester (mmol as denoted in the tables, added *via* syringe) in toluene (2 mL) was added fresh indium metal powder (mmol as denoted in the tables, see the tables for specific entries) and InX_3 (mmol as denoted in the tables) The resulting reaction mixture was subjected to ultrasonication for an appropriate time (see tables for specific entries) and the rest of the procedure followed as given in method A.

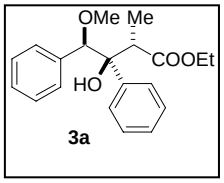
General procedure for In-employed Reformatsky-type reactions of α-hydroxy ketones with α-halo esters; synthesis of stereochemical lactones:

Method C: To a solution of an appropriate ketone (1 mmol) and an appropriate α- halo ester (2 mmol, added *via* syringe) in dry THF (2-3 mL) was added fresh indium metal powder (1.2 mmol) or indium(I) halide (1.2 mmol) at room temperature. The resulting reaction mixture was stirred at RT or refluxed (66-70 °C) under an inert atmosphere for an appropriate period (see Schemes for specific entries). After this, the reaction mixture was allowed to cool and stand at room temperature. To this mixture cold water (5-7 mL)

was added and the contents were subjected to ultrasonication for 2 min. Further, extraction was carried out using diethyl ether (3 X 15 mL). The combined organic layers were concentrated in vacuum and subjected to column to silica gel (5-30% EtOAc-Hexane mixture) chromatographic purification or recrystallization to afford the pure products.

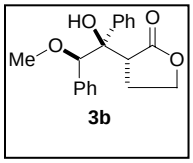
Spectral data as follows

(2S*)-3-[(3S*,4R*)-Ethyl-3-hydroxy-4-methoxy-2-methyl-3,4-diphenylbutyrate (3a): Silica gel column chromatographic purification afforded major isomer in pure form. Minor isomers were isolated as mixtures. Colorless solid, mp 101-102 °C; IR: (deposit from $CHCl_3$) 3451, 2985, 1708 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.25-6.92 (10 H, m, arom-*H*), 4.57 (1 H, s, O*H*), 4.34 (1 H, s, OC*H*), 4.34-4.20 (2 H, m, OCH_2), 3.19 (3 H, m, OCH_3), 3.09 (1 H, q, J = 7.2 Hz, C*H*), 1.38 (3 H, t, J = 7.2 Hz, CH_3), 0.92 (3 H, d, J = 7.2 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.0, 141.0, 136.7, 128.7, 127.7, 127.2, 127.0, 126.6, 125.6, 90.2, 80.3, 60.6, 56.9, 46.3, 14.2 13.7; MS: (CI) *m*/*z* 329 (M^++1, 12), 311 (26), 297 (100), 283 (11), 227 (6), 211 (1), 207 (10), 195 (9%); HRMS: (CI) calcd for $C_{20}H_{25}O_4$ 329.1753, found *m*/*z* 329.1752 (M^++1).



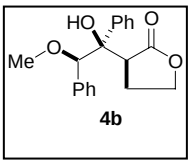
(3R*)-3-[(1R*,2R*)-1-Hydroxy-2-methoxy-1,2-diphenylethyl]-dihydrofuran-2-one (3b): Repetitive silica gel column chromatographic purification (two times) afforded both major and minor isomers in pure forms. Isomers **3b** and **4b**, were isolated as a colorless

solid (repetitive recrystallization from diethyl/hexane (recrystallized from hexane/ether (~35 mg of compound in a solution of ~1.5 mL diethyl ether and ~1 mL hexane was subjected to ultrasonication until a clear (diethyl ether can be used if needed more) solution is obtained and allowed to stand at rt for ~1 day to afford the crystals), **3b**: mp 122-124 °C; IR: (KBr) 3525, 1751, 1492, 1446, 1099 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$); 7.27-7.13 (6 H, m, arom-*H*), 7.04-7.02 (2 H, m, arom-*H*), 6.79-6.77 (2 H, m, arom-*H*), 5.43 (1 H, s, OC*H*), 4.37-4.31 (1 H, m, OCH_2), 4.23 (1 H, d, *J* = 1.6 Hz, O*H*), 4.18-4.11 (1 H, m, OCH_2), 3.25 (3 H, s, OCH_3), 3.19-3.14 (1 H, s, C*H*), 2.10-1.96 (2 H, m, CH_2); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.4, 139.4, 136.1, 128.9, 127.9, 127.6, 127.5, 127.0, 126.8, 84.9, 80.2, 67.1, 57.3, 44.7, 25.8; MS: (CI) *m*/*z* 313 (M^++1, 48), 295 (43), 282 (20), 281 (100), 227 (22), 195 (32), 87 (36%); HRMS: (CI) calcd for $C_{19}H_{21}O_4$ 313.1440, found *m*/*z* 313.1435 (M^++1).



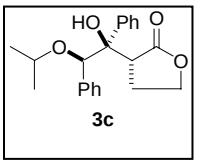
3b

(3S*)-3-[(1R*,2R*)-1-Hydroxy-2-methoxy-1,2-diphenylethyl]-dihydrofuran-2-one (4b): Colorless solid, mp 167-169 °C; IR: (KBr) 3567, 1743, 1492, 1450, 1384 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$); 7.36-7.05 (10 H, m, arom-*H*), 5.36 (1 H, s, OC*H*), 4.10-4.04 (1 H, m, OCH_2), 3.64-3.54 (2 H, m), 3.40 (3 H, s, OCH_3), 3.37 (1 H, s, O*H*), 2.42-2.33 (1 H, m, CH_2), 2.18-2.09 (1 H, m, CH_2); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.5, 139.6, 136.0, 128.9, 128.0, 127.6, 127.5, 127.5, 125.9, 83.8, 79.4, 66.6, 57.0, 47.3, 24.4; MS: (CI) *m*/*z* 313 (M^++1, 18), 295 (46), 282 (20), 281 (100), 227 (8), 87 (6%); HRMS: (CI) calcd for $C_{19}H_{21}O_4$ 313.1440, found *m*/*z* 313.1443 (M^++1).



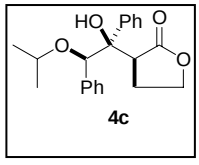
4b

(3R*)-3-[(1R*,2R*)-1-Hydroxy-2-isopropoxy-1,2-diphenylethyl]-dihydrofuran-2-one (3c): Repetitive silica gel column chromatographic purification (two times) afforded both major and minor isomers in pure forms. **3c**:Colorless solid, mp 122-124 °C; IR: (KBr) 3521, 1758, 1450, 1346, 921 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$); 7.25-7.02 (8 H, m, arom-*H*), 6.75-6.74 (2 H, m, arom-*H*), 5.68 (1 H, s, OC*H*), 4.36-4.31 (1 H, m, OCH_2), 4.26 (1 H, d, *J* = 1.6 Hz, O*H*), 4.17-4.12 (1 H, m, OCH_2), 3.54-3.48 (1 H, m, OCH_2), 3.19-3.15 (1 H, s, C*H*), 2.12-1.96 (2 H, m, CH_2), 1.21 (3 H, d, *J* = 6.0 Hz, CH_3), 1.01 (3 H, d, *J* = 6.0 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.5, 139.8, 137.7, 128.9, 127.7, 127.6, 127.4, 126.8, 126.7, 80.0, 79.8, 69.9, 67.0, 44.4, 25.9, 23.2, 21.3 (; MS: (CI) *m*/*z* 341 (M^++1, 52), 323 (12), 282 (19), 281 (100), 255 (15), 211 (1), 195 (20), 149 (8%); HRMS: (CI) calcd for $C_{21}H_{25}O_4$ 341.1753, found *m*/*z* 341.1750 (M^++1).



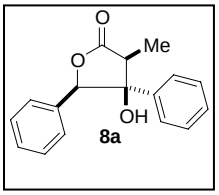
3c

(3S*)-3-[(1R*,2R*)-1-Hydroxy-2-isopropoxy-1,2-diphenylethyl]-dihydrofuran-2-one (4c): Colorless solid, mp 144-146 °C; IR: (KBr) 3567, 2973, 1754, 1184 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$); 7.35-7.05 (10 H, m, arom-*H*), 5.60 (1 H, s, OC*H*), 4.07-4.01 (1 H, m, OCH_2), 3.76 (1 H, s, O*H*), 3.74-3.67 (1 H, m), 3.59-3.49 (2 H, m), 2.42-2.32 (1 H, m, CH_2), 2.24-2.14 (1 H, m, CH_2), 1.30 (3 H, d, *J* = 6.0 Hz, CH_3), 1.10 (3 H, d, *J* = 6.0 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.4, 139.5, 137.4, 129.0, 127.3, 127.2, 127.1, 126.0, 79.3, 79.3, 69.8, 66.4, 47.3, 24.5, 23.5, 21.4; MS: (CI) *m*/*z* 341 (M^++1, 5), 323 (11), 282 (19), 281 (100), 255 (8), 203 (2), 195 (10), 149 (5%); HRMS: (CI) calcd for $C_{21}H_{25}O_4$ 341.1753, found *m*/*z* 341.1748 (M^++1).



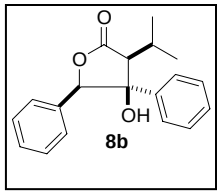
4c

[3R*,4R*,5S*] 4-Hydroxy-3-methyl-4,5-diphenyldihydrofuran-2-one (8a): Colorless solid, mp 120-122 °C; IR: (deposit from $CHCl_3$) 3563, 1781 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.46-7.26 (8 H, m, arom-*H*), 7.07 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.81 (1 H, s, OC*H*), 3.19 (1 H, qd, J_1 = 7.2 Hz, J_2 = 1.6 Hz, C*H*), 1.66 (1 H, d, *J* = 1.6 Hz, O*H*), 1.19 (3 H, d, *J* = 7.2 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 176.5, 138.9, 131.7, 129.1, 128.8, 128.6, 128.2, 126.4, 125.4, 86.7, 81.4, 48.8, 6.8; MS: (EI) *m*/*z* 268 (M^+, 0.3), 165 (1), 135 (10), 134 (100), 133 (38), 105 (22), 77 (13), 55 (4%); HRMS: (EI) calcd for $C_{17}H_{16}O_3$ 268.1099, found *m*/*z* 268.1097 (M^+). Anal. Calcd for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found: C, 75.99; H, 5.98.



8a

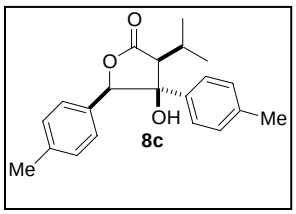
[3R*,4R*,5S*] 4-Hydroxy-3-isopropyl-4,5-diphenyldihydrofuran-2-one (8b): Colorless solid, mp 135-137 °C; IR: 3432, 1754 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.43-7.25 (8 H, m, arom-*H*), 6.94 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.58 (1 H, s, OC*H*), 3.01 (1 H, dd, J_1 = 7.4 Hz, J_2 = 1.6 Hz, C*H*), 2.23-2.18 (1 H, m, C*H*), 1.70 (1 H, d, *J* = 1.6 Hz, O*H*), 1.21 (3 H, d, *J* = 6.8 Hz, CH_3), 0.78 (3 H, d, *J* = 6.8 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 175.4, 140.4, 131.2, 129.2, 128.6, 128.6, 127.8, 126.5, 125.3, 87.2, 82.4, 57.9, 26.1, 22.0, 21.2; MS: (EI) *m*/*z* 296 (M^+, 0.2), 163 (12), 162 (100), 147 (81), 129 (3), 105 (21), 77 (12), 69 (12%); HRMS: (EI) calcd for $C_{19}H_{20}O_3$ 296.1412, found *m*/*z* 296.1413 (M^+). Anal. Calcd for $C_{19}H_{20}O_3$: C, 77.00; H, 6.80. Found: C, 76.81; H, 6.72.



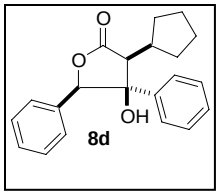
8b

[3R*,4R*,5S*] 4-Hydroxy-3-isopropyl-4,5-di-*p*-tolyldihydrofuran-2-one (8c): Colorless solid, mp 158-160 °C; IR: 3444, 1766 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.24 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.19 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.08 (2 H, d, *J* = 8.4 Hz, arom-*H*), 6.84 (2 H, d, *J* = 8.4 Hz, arom-*H*), 5.53 (1 H, s, OC*H*), 2.96 (1 H,

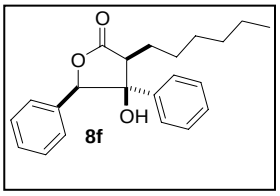
dd, J_1 = 7.4 Hz, J_2 = 1.6 Hz, C*H*), 2.38 (3 H, s, CH_3), 2.31 (3 H, s, CH_3), 2.24-2.16 (1 H, m), 1.67 (1 H, d, *J* = 1.6 Hz, O*H*), 1.20 (3 H, d, *J* = 6.8 Hz, CH_3), 0.78 (3 H, d, *J* = 6.8 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 175.6, 139.0, 137.5, 137.4, 129.3, 129.2, 128.1, 126.4, 125.2, 87.3, 82.3, 57.8, 26.0, 22.1, 21.2, 21.1, 21.0; MS: (EI) *m*/*z* 324 (M^+, 0.3), 195 (2), 177 (9), 176 (69), 162 (12), 161 (100), 119 (22), 91 (10), 69 (14%); HRMS: (EI) calcd for $C_{21}H_{24}O_3$ 324.1725, found *m*/*z* 324.1718 (M^+).



[3R*,4R*,5S*] 3-Cyclopentyl-4-hydroxy-4,5-diphenyldihydrofuran-2-one (8d): Colorless solid, mp 154-156 °C; IR: (deposit from $CHCl_3$) 3444, 1781 cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$); 7.43-7.25 (8 H, m, arom-*H*), 6.96 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.66 (1 H, s, OC*H*), 3.11 (1 H, dd, J_1 = 11.0 Hz, J_2 = 1.6 Hz, C*H*), 2.40-2.36 (1 H, m), 2.25-2.21 (1 H, m), 1.67 (1 H, d, *J* = 1.6 Hz, O*H*), 1.57-1.29 (6 H, m), 0.75-0.69 (1 H, m); ^{13}C NMR: (100 MHz, $CDCl_3$); 175.6, 140.2, 131.4, 129.1, 128.6, 128.6, 127.9, 126.4, 125.5, 87.3, 82.1, 57.8, 36.7, 31.4, 31.3, 24.5, 24.4; MS: (EI) *m*/*z* 322 (M^+, 0.3), 189 (15), 167 (4), 159 (10), 145 (5), 120 (27), 105 (26), 91 (5%); HRMS: (EI) calcd for $C_{21}H_{22}O_3$ 322.1569, found *m*/*z* 322.1564 (M^+).

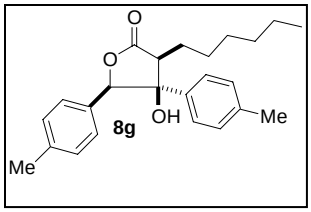


[3R*,4R*,5S*] 3-Hexyl-4-hydroxy-4,5-diphenyldihydrofuran-2-one (8f):Colorless solid, mp 119-121 °C; IR: 3451, 1766 (KBr) cm^{-1}; ^{1}H NMR: (400 MHz, $CDCl_3$); 7.45-7.26 (8 H, m, arom-*H*), 7.01 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.73 (1 H, s, OC*H*), 3.11 (1 H, t, *J* = 6.8 Hz, C*H*), 1.90-1.83 (1 H, m), 1.68-1.58 (1 H, m), 1.67 (1 H, s, O*H*), 1.33-1.07 (8 H, m), 0.79 (3 H, t, *J* = 7.2 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 176.4,

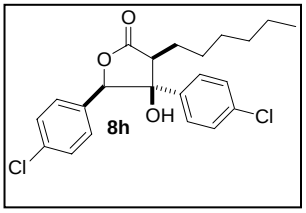


139.5, 131.6, 129.1, 128.7, 128.6, 128.1, 126.4, 125.3, 87.0, 81.4, 53.0, 31.3, 30.0, 27.2, 23.7, 22.5, 14.0; MS: (EI) *m*/*z* 338 (M^+, 0.6), 205 (16), 204 (100), 165 (3), 161 (4), 133 (80), 120 (36), 105 (30%); HRMS: (EI) calcd for $C_{22}H_{26}O_3$ 338.1882, found *m*/*z* 338.1879 (M^+).

[3R*,4R*,5S*] 3-Hexyl-4-hydroxy-4,5-di-*p*-tolyldihydrofuran-2-one (8g) :Colorless solid, mp 137-139 °C; IR: 3432, 1770 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.27 (2 H, d, *J* = 8.0 Hz, arom-*H*), 7.20 (2 H, d, *J* = 8.0 Hz, arom-*H*), 7.09 (2 H, d, *J* = 8.0 Hz, arom-*H*), 6.90 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.67 (1 H, s, OC*H*), 3.06 (1 H, t, *J* = 6.8 Hz, C*H*), 2.38 (3 H, s, CH_3), 2.31 (3 H, s, CH_3), 1.88-1.81 (1 H, m), 1.65-1.59 (1 H, m), 1.62 (1 H, s, O*H*), 1.32-1.12 (8 H, m), 0.79 (3 H, t, *J* = 7.2 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 176.7, 139.0, 137.7, 136.6, 129.4, 129.3, 128.5, 126.3, 125.2, 87.1, 81.3, 52.9, 31.4, 29.0, 27.2, 23.7, 22.5, 21.2, 21.0, 14.0; MS: (EI) *m*/*z* 366 (M^+, 0.5), 219 (15), 218 (96), 195 (6), 189 (1), 175 (4), 161 (2), 148 (11), 147 (100), 134 (54), 119 (36%); HRMS: (EI) calcd for $C_{24}H_{30}O_3$ 366.2195, found *m*/*z* 366.2202 (M^+).

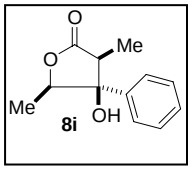


[3R*,4R*,5S*] 4,5-Bis-(4-chlorophenyl)-3-Hexyl-4-hydroxydihydrofuran-2-one (8h): Colorless solid, mp 140-142 °C; IR: 3448, 1766 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.41 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.35 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.27 (2 H, d, *J* = 8.4 Hz, arom-*H*), 6.95 (2 H, d, *J* = 8.4 Hz, arom-*H*), 5.62 (1 H, s, OC*H*), 3.08 (1 H, t, *J* = 6.8 Hz, C*H*), 1.89-1.81 (1 H, m), 1.69 (1 H, s, O*H*), 1.64-1.57 (1 H, m), 1.29-1.12 (8 H, m), 0.81 (3 H, t, *J* = 7.2 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 176.0, 137.8, 135.3, 134.4, 129.8, 129.1, 128.9, 127.8, 126.8, 86.4, 81.3, 52.9, 31.3, 29.0, 27.2, 23.6, 22.5,



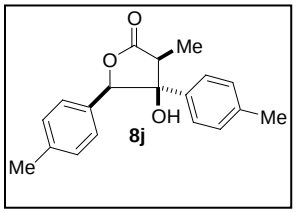
14.0; MS: (EI) *m*/*z* 406 (M^+, 0.2), 240 (28), 238 (83), 203 (5), 195 (4), 169 (32), 167 (100), 156 (18), 154 (58), 139 (31), 125 (4), 55 (16%); HRMS: (EI) calcd for $C_{22}H_{24}Cl_2O_3$ 406.1102, found *m*/*z* 406.1108 (M^+).

[3R*,4R*,5S*] 4-Hydroxy-3,5-dimethyl-4-phenyldihydrofuran-2-one (8i): Colorless solid; mp 89-91 °C IR: (deposit from $CHCl_3$) 3440, 1770 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.51-7.27 (5 H, m, arom-*H*), 4.66 (1 H, q, *J* = 6.4 Hz, OC*H*), 3.14 (1 H, t, *J* = 6.4 Hz, C*H*), 2.72 (1 H, s, O*H*), 1.32 (3 H, d, *J* = 6.4 Hz, CH_3), 1.14 (3 H, d, *J* = 6.8 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.3, 138.6, 128.7, 128.1, 125.2, 83.3, 81.0, 47.6, 11.4, 6.6; MS: (EI) *m*/*z* 206 (M^+, 0.4), 206 (10), 134 (100), 133 (50), 105 (42), 77 (17), 55 (6), 51 (4%); HRMS: (EI) calcd for $C_{12}H_{14}O_3$ 206.0943, found *m*/*z* 206.0948 (M^+).



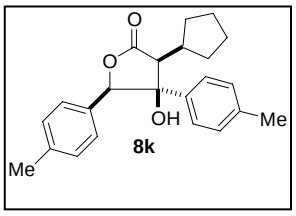
8i

[3R*,4R*,5S*] 4-Hydroxy-3-methyl-4,5-di-*p*-tolyldihydrofuran-2-one (8j): Colorless solid, mp 147-149 °C; IR: (deposit from $CHCl_3$) 3556, 1778 cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.28 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.22 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.10 (2 H, d, *J* = 8.4 Hz, arom-*H*), 6.96 (2 H, d, *J* = 8.4 Hz, arom-*H*), 5.75 (1 H, s, OC*H*), 3.14 (1 H, qd, J_1 = 6.4 Hz, J_2 = 1.6 Hz, C*H*), 2.38 (3 H, s, CH_3), 2.31 (3 H, s, CH_3), 1.62 (1 H, d, *J* = 1.6 Hz, O*H*), 1.17 (3 H, d, *J* = 7.2 Hz, CH_3); ^{13}C NMR: (100 MHz, $CDCl_3$); 176.8, 139.0, 137.9, 136.6, 129.4, 129.3, 128.6, 126.4, 125.3, 86.8, 81.2, 48.7, 21.2, 21.0, 6.8; MS: (EI) *m*/*z* 296 (M^+, 1), 195 (8), 149 (10), 148 (100), 133 (21), 119 (18), 91 (10), 65 (2%); HRMS: (EI) calcd for $C_{19}H_{20}O_3$ 296.1412, found *m*/*z* 296.1418 (M^+).

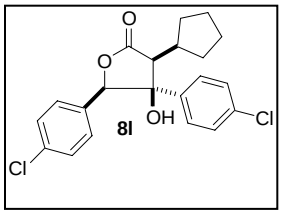


8j

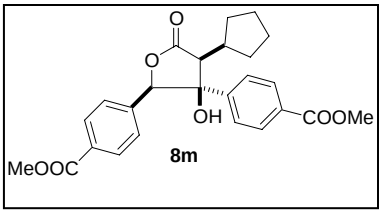
[3R*,4R*,5S*] 3-Cyclopentyl-4-hydroxy-4,5-di-*p*-tolyldihydrofuran-2-one (8k): Colorless solid, mp 177-179 °C; IR: 3367, 1762 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.25 (2 H, d, *J* = 8.0 Hz, arom-*H*), 7.19 (2 H, d, *J* = 8.0 Hz, arom-*H*), 7.07 (2 H, d, *J* = 8.0 Hz, arom-*H*), 6.85 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.56 (1 H, s, OC*H*), 3.06 (1 H, d, *J* = 9.6 Hz, C*H*), 2.43-2.15 (2 H, m), 2.38 (3 H, s, CH_3), 2.31 (3 H, s, CH_3), 1.66 (1 H, s, O*H*), 1.55-1.24 (6 H, m), 0.79-0.69 (1 H, m); ^{13}C NMR: (100 MHz, $CDCl_3$); 175.9, 138.9, 137.5, 137.3, 129.2, 128.4, 126.3, 125.4, 87.4, 82.0, 57.7, 36.7, 31.4, 31.3, 24.5, 24.4, 21.2, 21.1; MS: (EI) *m*/*z* 350 (M^+, 0.3), 203 (15), 202 (100), 187 (46), 173 (12), 159 (5), 145 (4), 134 (37), 119 (44), 91 (13%); HRMS: (EI) calcd for $C_{23}H_{26}O_3$ 350.1882, found *m*/*z* 350.1886 (M^+).



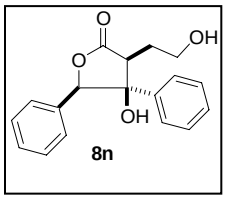
[3R*,4R*,5S*] 4,5-Bis-(4-chlorophenyl)-3-cyclopentyl-4-hydroxydihydrofuran-2-one (8l): Colorless solid, mp 181-183 °C; IR: 3428, 1754 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.39 (2 H, d, *J* = 8.8 Hz, arom-*H*), 7.34 (2 H, d, *J* = 8.8 Hz, arom-*H*), 7.26 (2 H, d, *J* = 8.8 Hz, arom-*H*), 6.91 (2 H, d, *J* = 8.8 Hz, arom-*H*), 5.55 (1 H, s, OC*H*), 3.06 (1 H, d, *J* = 9.2 Hz, C*H*), 2.39-2.15 (2 H, m), 1.75 (1 H, s, O*H*), 1.60-1.28 (6 H, m), 0.73-0.66 (1 H, m); ^{13}C NMR: (100 MHz, $CDCl_3$); 175.0, 138.5, 135.3, 134.2, 129.8, 129.0, 128.9, 127.8, 126.9, 86.6, 81.9, 57.7, 36.6, 31.5, 31.3, 24.4, 24.3; MS: (EI) *m*/*z* 390 (M^+, 0.2), 224 (32), 223 (15), 222 (100), 193 (12), 187 (20), 169 (2), 156 (17), 154 (51), 141 (15), 139 (37%); HRMS: (EI) calcd for $C_{21}H_{20}Cl_2O_3$ 390.0789, found *m*/*z* 390.0793 (M^+).



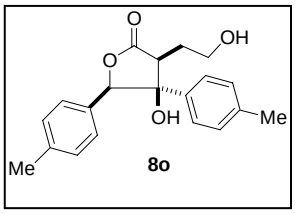
[3R*,4R*,5S*] 4,5-Bis-(4-methylcarboxylate phenyl)-3-cyclopentyl-4-hydroxydihydrofuran-2-one (8m): Colorless solid, mp 190-192 °C; IR: (deposit from $CHCl_3$) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 8.08 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.86 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.55 (2 H, d, *J* = 8.4 Hz, arom-*H*), 7.01 (2 H, d, *J* = 8.4 Hz, arom-*H*), 5.69 (1 H, s, OC*H*), 3.95 (3 H, s, OCH_3), 3.86 (3 H, s, OCH_3), 3.17 (1 H, d, *J* = 9.2 Hz, C*H*), 2.41-2.04 (2 H, m), 2.28 (1 H, s, O*H*), 1.56-1.26 (6 H, m), 0.67-0.59 (1 H, m); ^{13}C NMR: (100 MHz, $CDCl_3$); 175.0, 166.4, 166.3, 145.1, 136.6, 130.7, 130.0, 129.9, 129.6, 126.4, 125.7, 86.8, 82.5, 58.0, 52.3, 52.2, 36.6, 31.4, 31.3, 24.3, 24.3; MS: (CI) *m*/*z* 439 (M^++1, 100), 421 (28), 408 (21), 407 (76), 327 (10), 283 (78), 246 (22), 187 (3), 165 (6), 163 (5), 111 (3%); HRMS: (CI) calcd for $C_{25}H_{27}O_7$ 439.1757, found *m*/*z* 439.1759 (M^+).



[3R*,4R*,5S*] 4-Hydroxy-3-(2-hydroxyethyl)-4,5-diphenyldihydrofuran-2-one (8n): Colorless solid, mp 157-159 °C; IR: 3498, 3394, 1766 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.43-7.26 (8 H, m, arom-*H*), 7.03 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.74 (1 H, s, OC*H*), 3.72-3.59 (2 H, m), 3.46 (1 H, t, *J* = 6.8 Hz), 2.55 (1 H, s, O*H*), 2.14-2.06 (1 H, m), 2.01 (1 H, t, *J* = 4.8 Hz, O*H*) 1.99-1.87 (1 H, m); ^{13}C NMR: (100 MHz, $CDCl_3$); 176.8, 139.5, 131.6, 129.1, 128.8, 128.4, 128.3, 126.5, 125.5, 87.8, 81.5, 60.0, 51.2, 26.4; MS: (EI) *m*/*z* 298 (M^+, 0.1), 191 (10), 164 (66), 134 (10), 133 (100), 115 (3), 105 (32), 77 (17%); HRMS: (EI) calcd for $C_{18}H_{18}O_4$ 298.1205, found *m*/*z* 298.1196 (M^+).

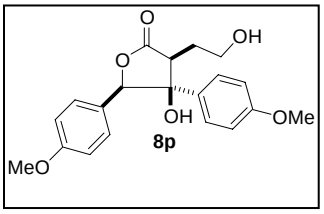


[3R*,4R*,5S*] 4-Hydroxy-3-(2-hydroxyethyl)-4,5-di-*p*-tolyldihydrofuran-2-one (8o):

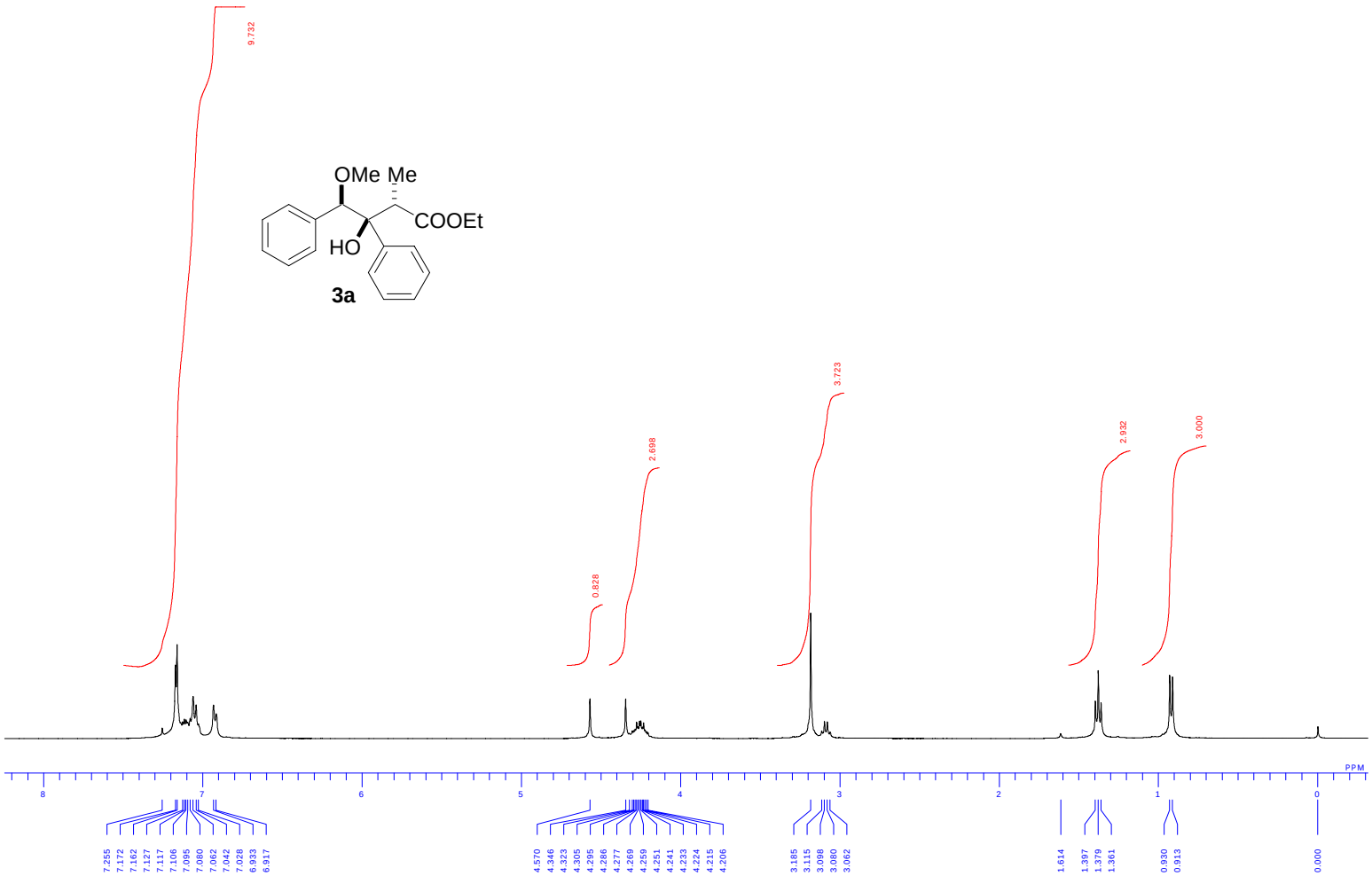


Colorless solid, mp 133-135 °C; IR: 3490, 3394, 1762 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.28 (2 H, d, *J* = 8.0 Hz, arom-*H*), 7.20 (2 H, d, *J* = 8.0 Hz, arom-*H*), 7.09 (2 H, d, *J* = 8.0 Hz, arom-*H*), 6.91 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.68 (1 H, s, OC*H*), 3.70-3.50 (2 H, m), 3.39 (1 H, t, *J* = 6.8 Hz), 2.49 (1 H, s, O*H*), 2.38 (3 H, s, CH_3), 2.31 (3 H, s, CH_3), 2.19 (1 H, br s, O*H*) 2.11-2.02 (1 H, m), 1.90 -1.78 (1 H, m); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.1, 138.9, 138.0, 136.0, 129.5, 129.1, 126.4, 125.3, 87.8, 81.3, 60.1, 51.2, 26.4, 21.2, 21.0; MS: (EI) *m*/*z* 326 (M^+, 0.1), 205 (10), 178 (60), 148 (11), 147 (100), 119 (30), 89 (1), 55 (16%); HRMS: (EI) calcd for $C_{20}H_{22}O_4$ 326.1518, found *m*/*z* 326.1511 (M^+). Data for the predominant isomer is given here.

[3R*,4R*,5S*] 4-Hydroxy-3-(2-hydroxyethyl)-4,5-bis-(4-methoxyphenyl)-dihydrofuran-2-one (8p):



Colorless solid, mp 126-128 °C; IR: 3401, 1758 (KBr) cm^{-1}; ^1H NMR: (400 MHz, $CDCl_3$); 7.28 (2 H, d, *J* = 8.0 Hz, arom-*H*), 7.37 (2 H, d, *J* = 8.0 Hz, arom-*H*), 6.98 (2 H, d, *J* = 8.0 Hz, arom-*H*), 6.79 (2 H, d, *J* = 8.0 Hz, arom-*H*), 5.59 (1 H, s, OC*H*), 4.84 (1 H, s, O*H*), 3.80 (3 H, s, OCH_3), 3.75 (3 H, s, OCH_3), 3.62-3.49 (3 H, m), 3.32-3.31 (1 H, m, O*H*), 2.03-2.1.95 (1 H, m), 1.84 -1.76 (1 H, m); ^{13}C NMR: (100 MHz, $CDCl_3$); 177.5, 159.1, 158.5, 130.4, 127.6, 126.2, 112.6, 112.2, 87.5, 80.9, 58.1, 53.7, 53.6, 48.3, 25.7; MS: (EI) *m*/*z* 358 (M^+, 0.5), 227 (8), 194 (65), 164 (10), 163 (100), 137 (26), 135 (35), 109 (3%); HRMS: (EI) calcd for $C_{20}H_{22}O_6$ 358.1416, found *m*/*z* 358.1408 (M^+).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3.000
PPM
8
7
6
5
4
3
2
1
0
7.255
7.172
7.162
7.127
7.117
7.106
7.095
7.080
7.062
7.042
7.028
6.933
6.917
4.570
4.346
4.323
4.305
4.295
4.286
4.277
4.269
4.259
4.251
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4.233
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4.215
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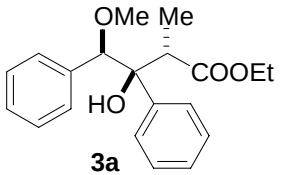
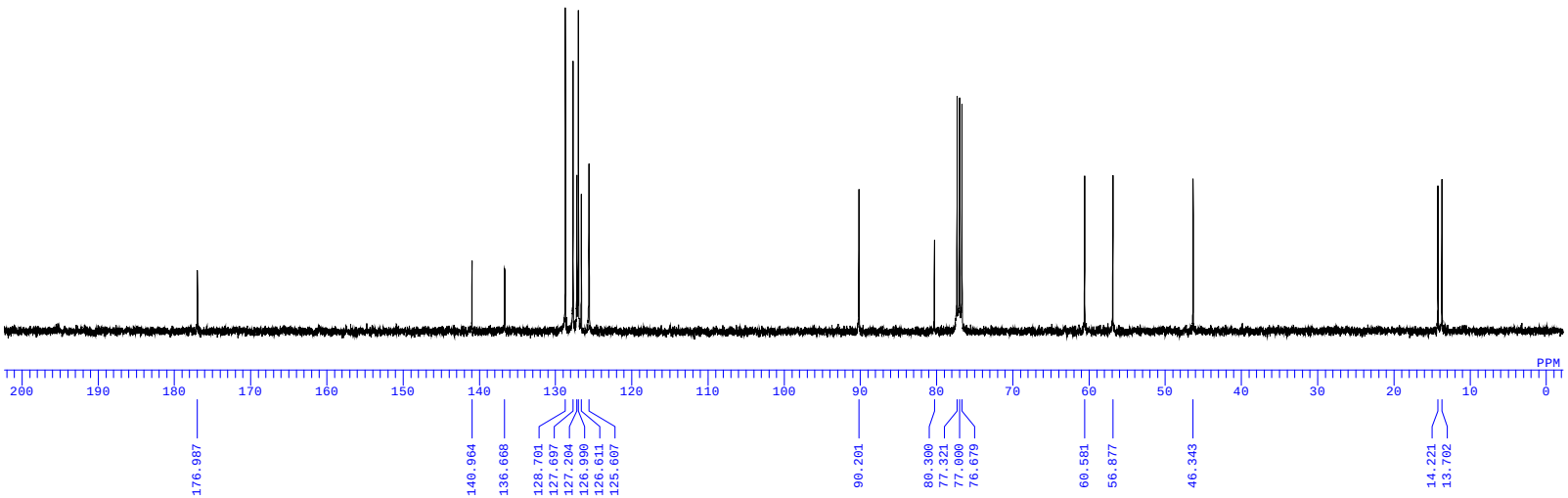
0.000


OMe
Me
COOEt
HO

3a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60
50
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30
20
10
0
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140.964
136.668
128.701
127.697
127.204
126.990
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125.607
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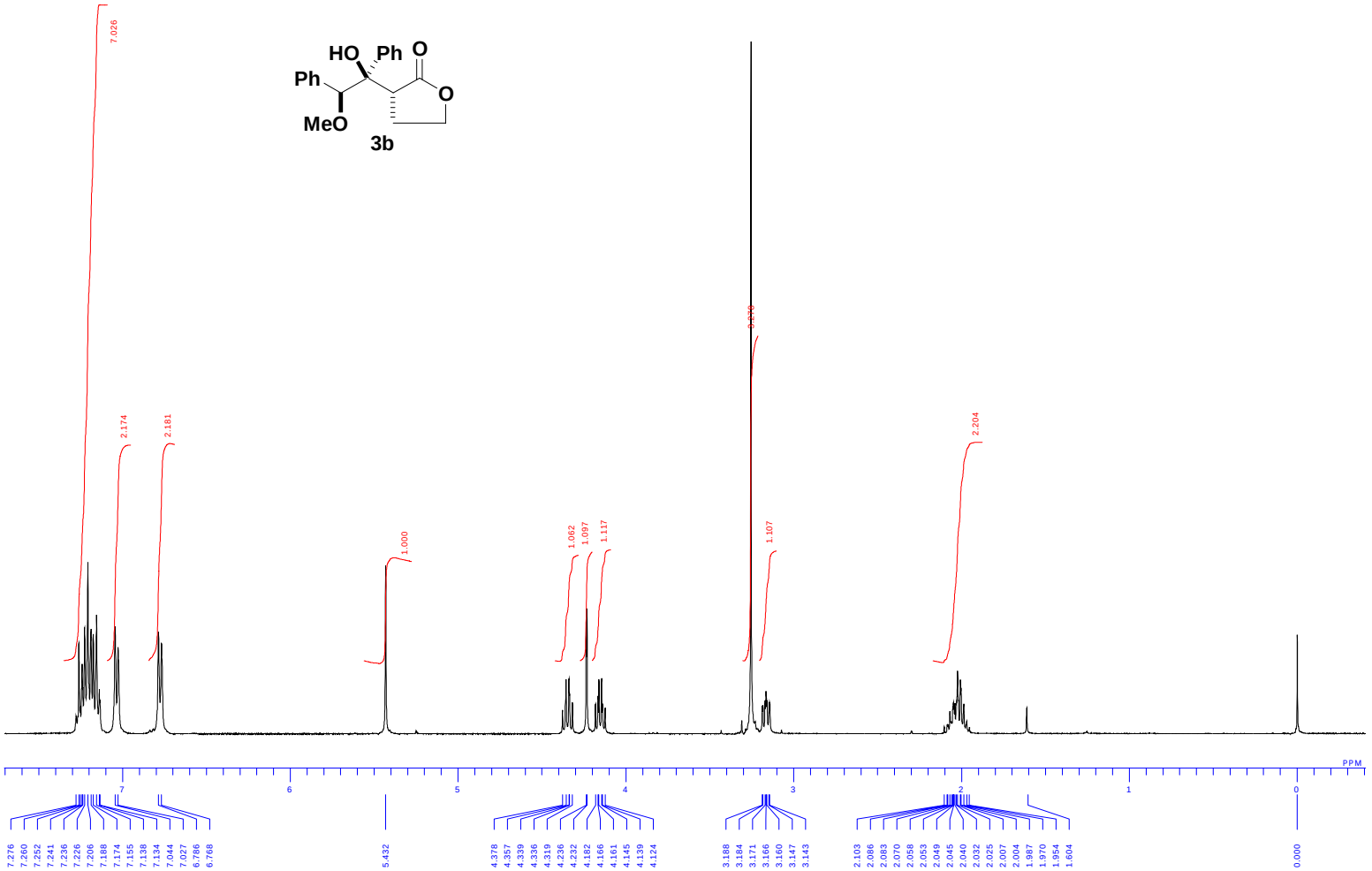
60.581
56.877
46.343
14.221
13.702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Ph
O
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1.000
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1.097
1.117
3.000
1.107
2.204
PPM
7
6
5
4
3
2
1
0
7.276
7.260
7.252
7.241
7.236
7.226
7.206
7.188
7.174
7.155
7.138
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2.058
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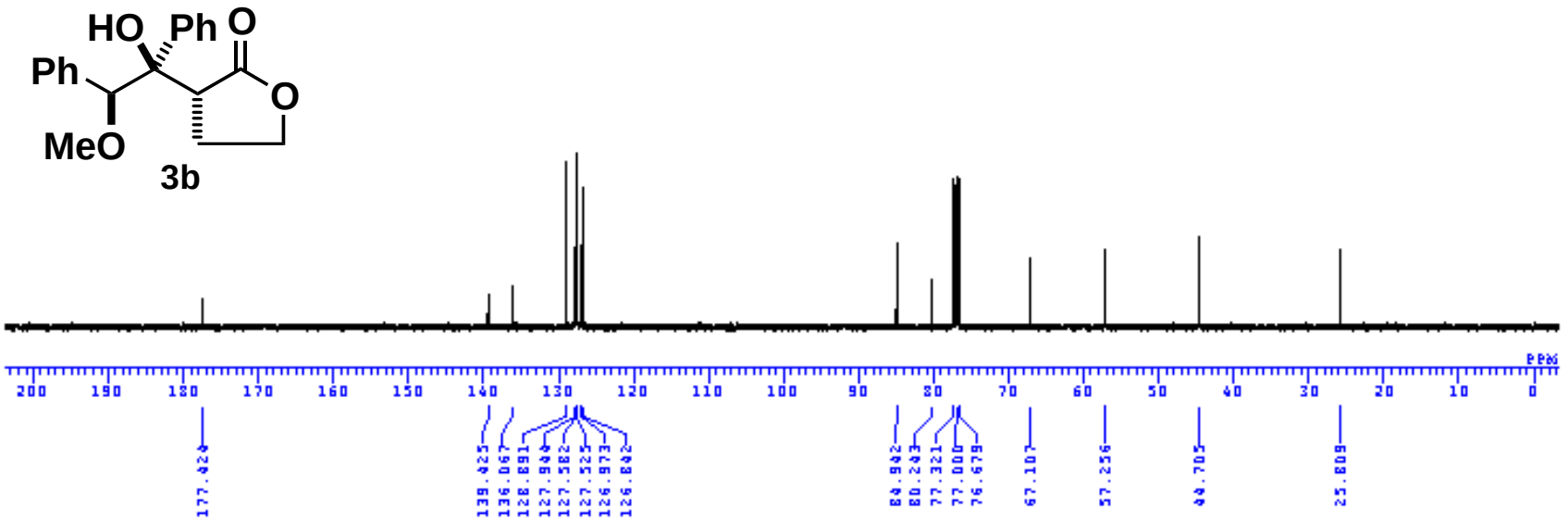
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MeO
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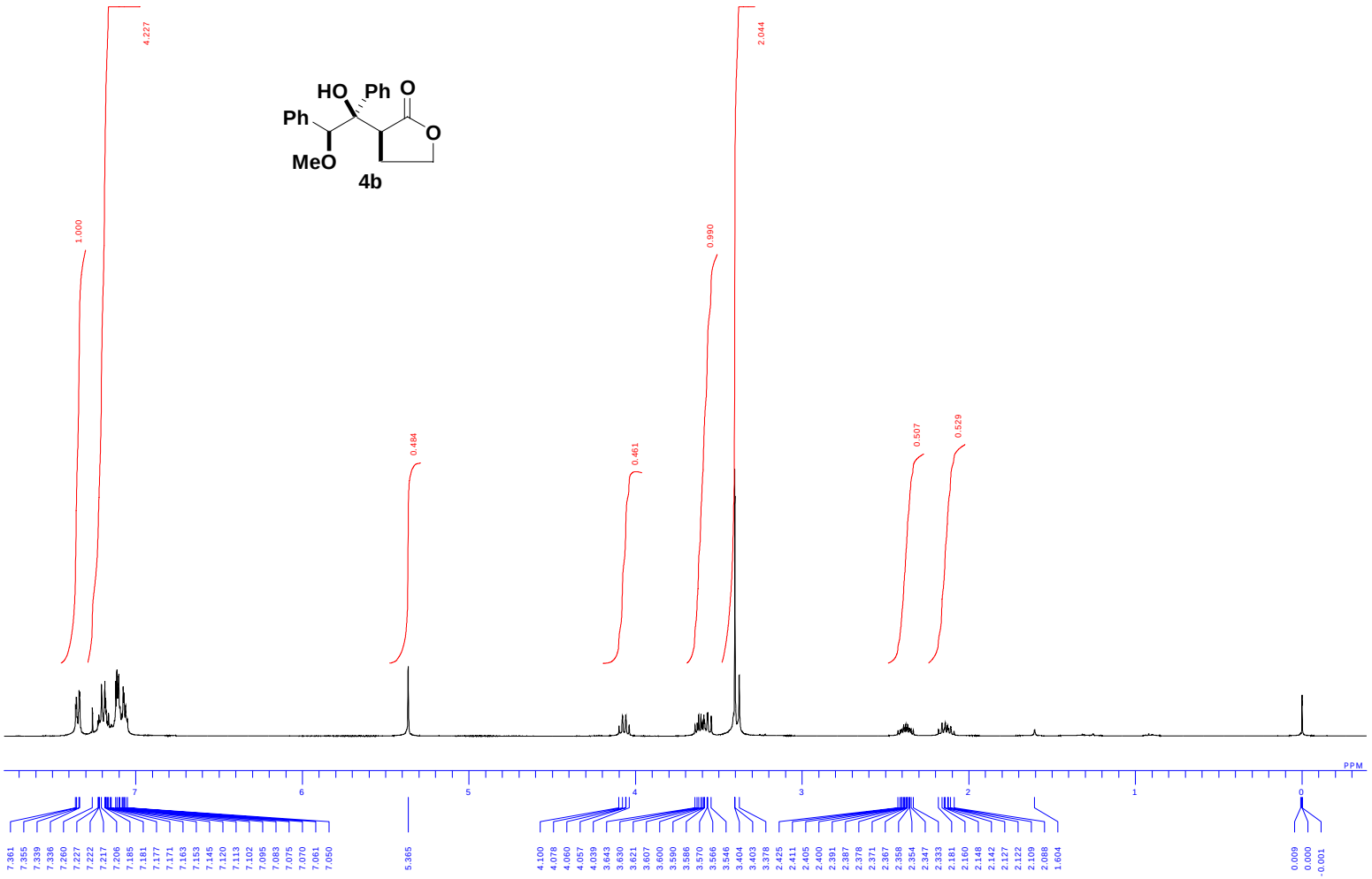
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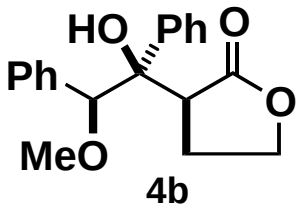
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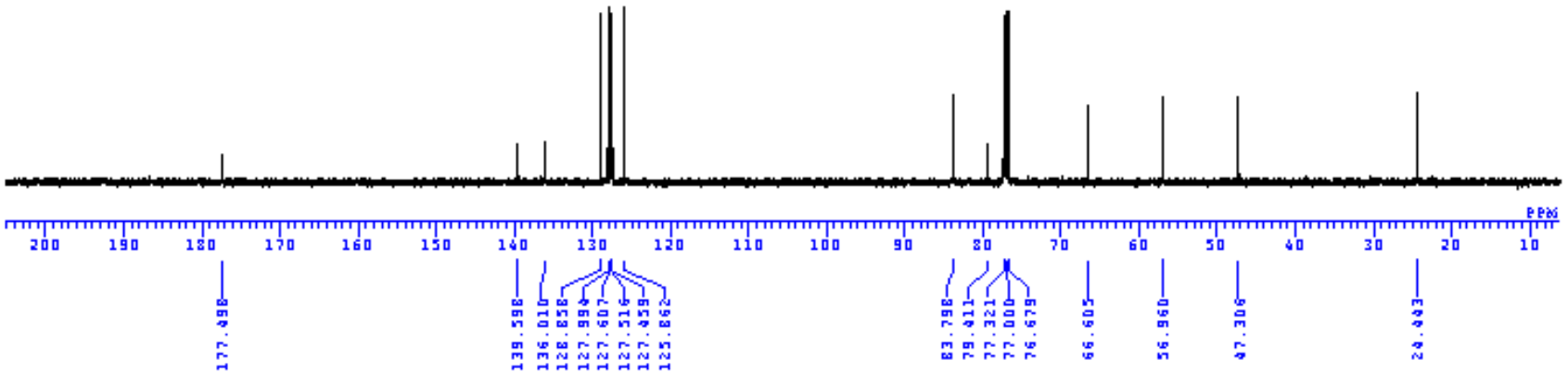
4b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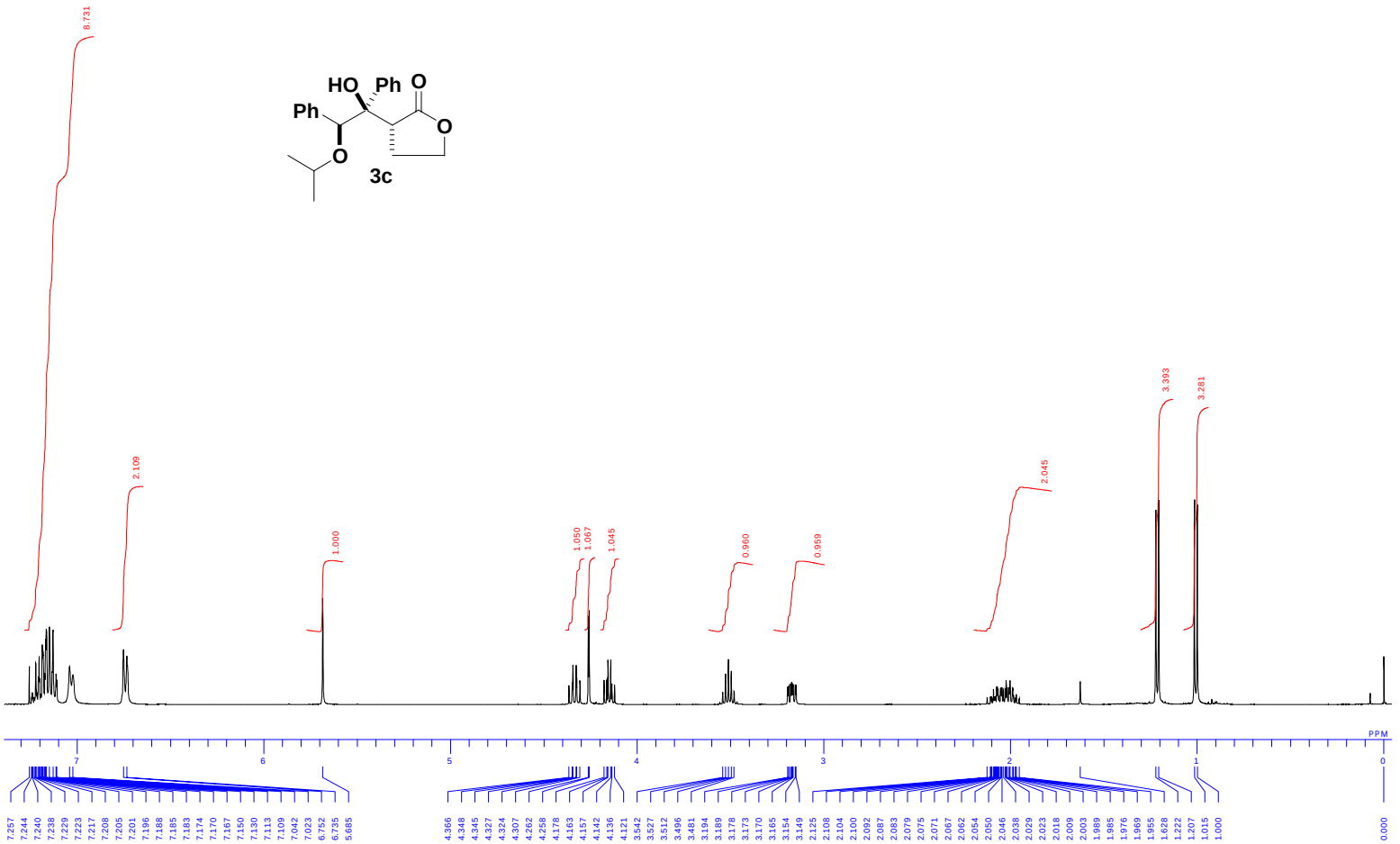
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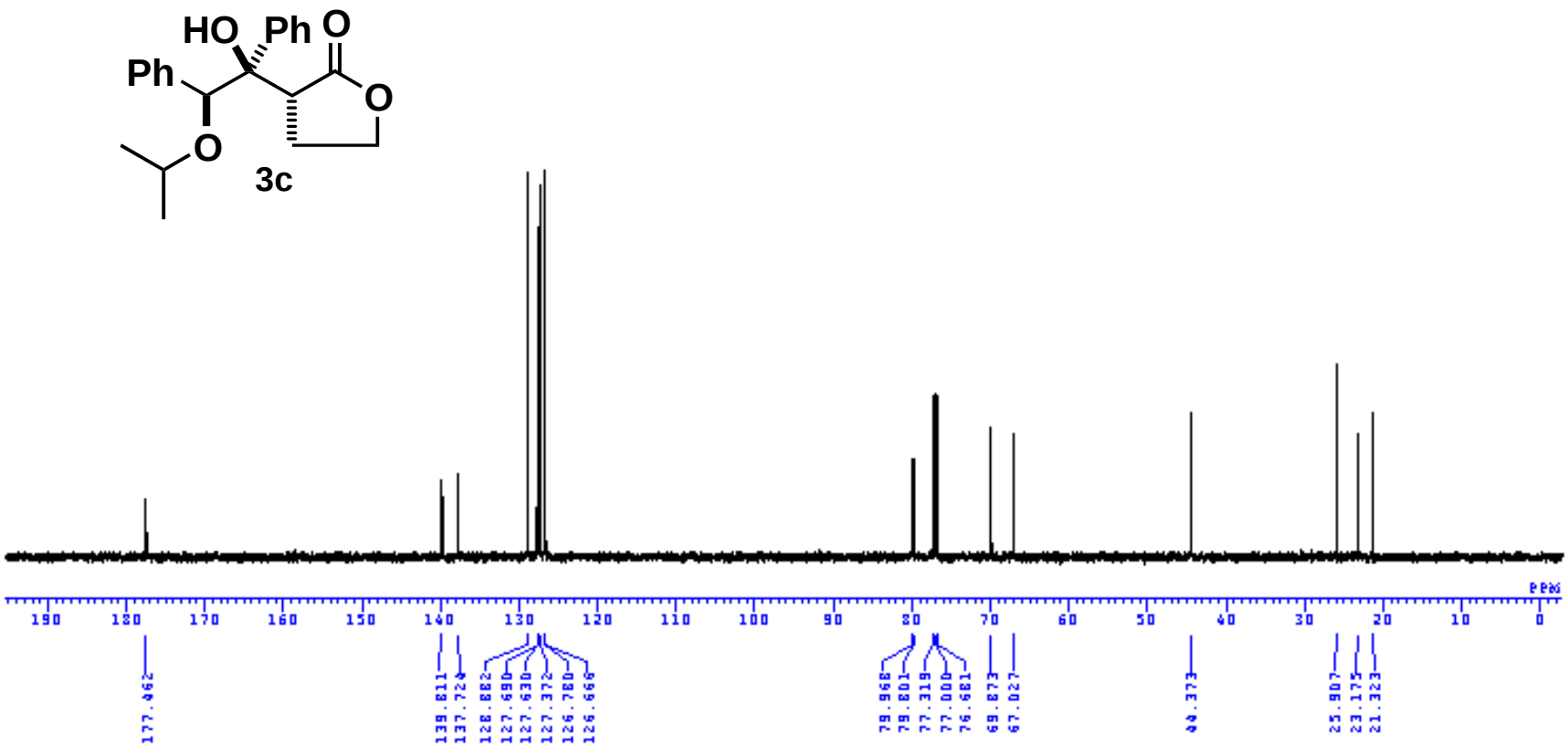
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3c
PPM
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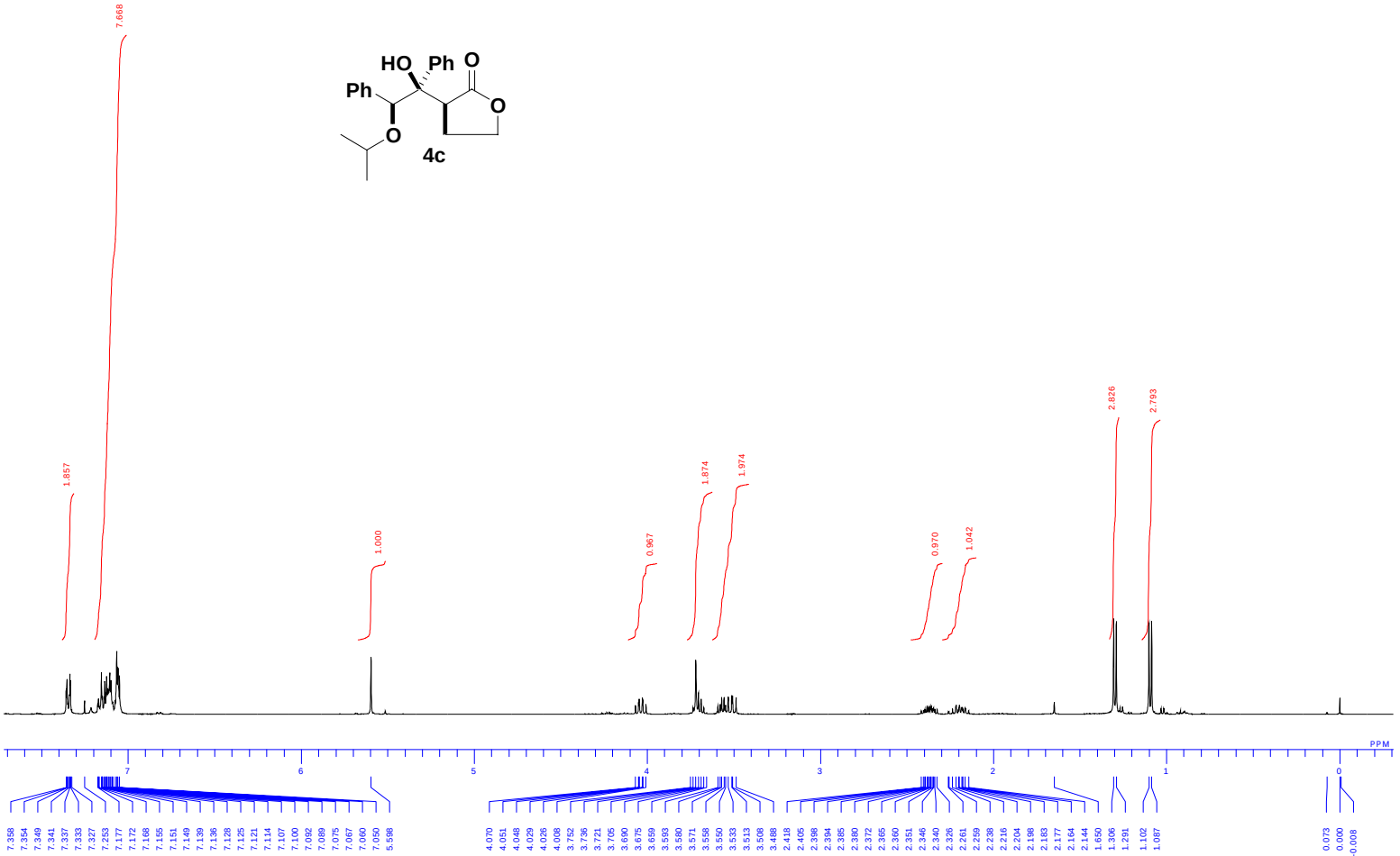
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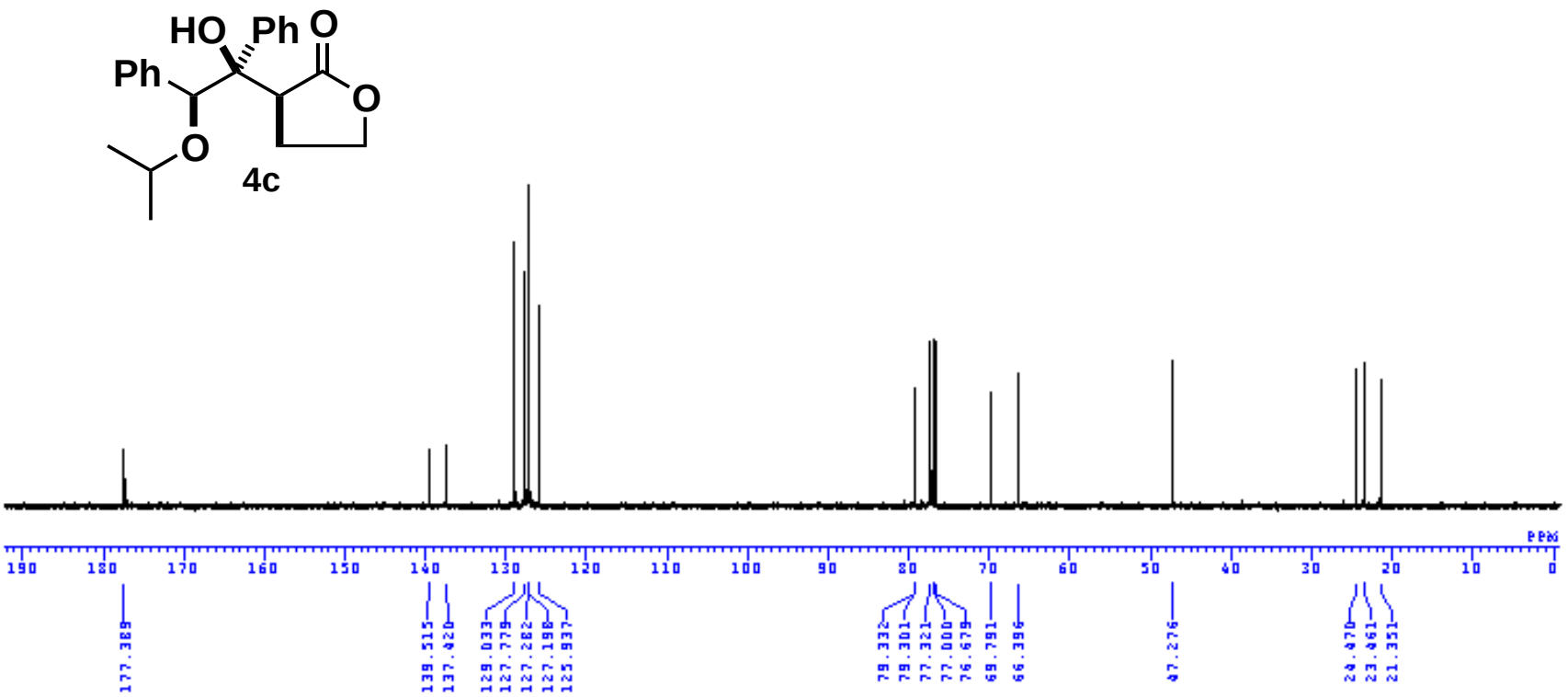
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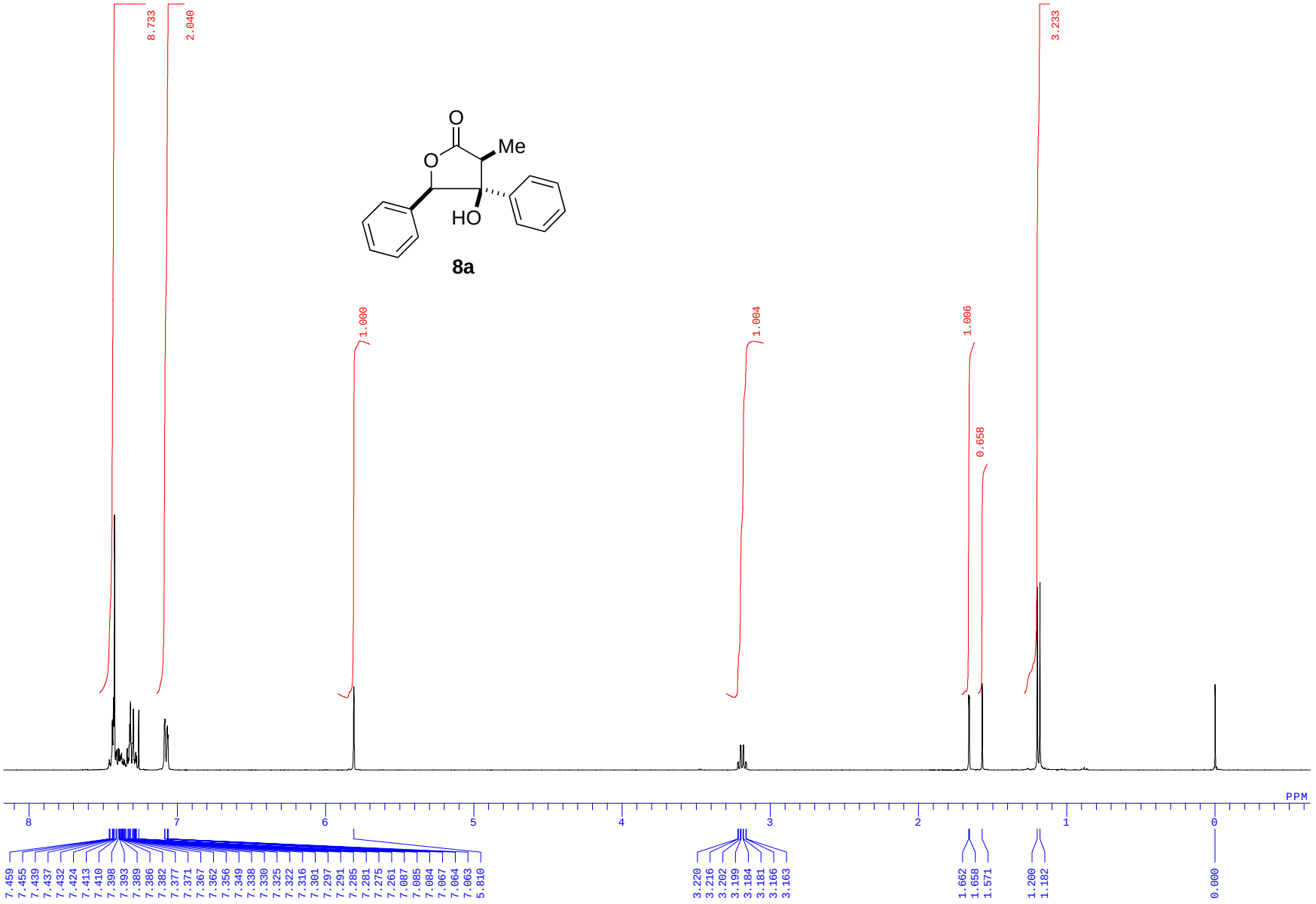
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HO
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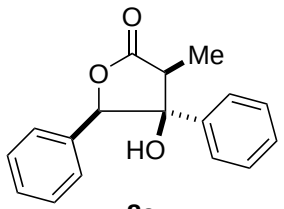
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HO

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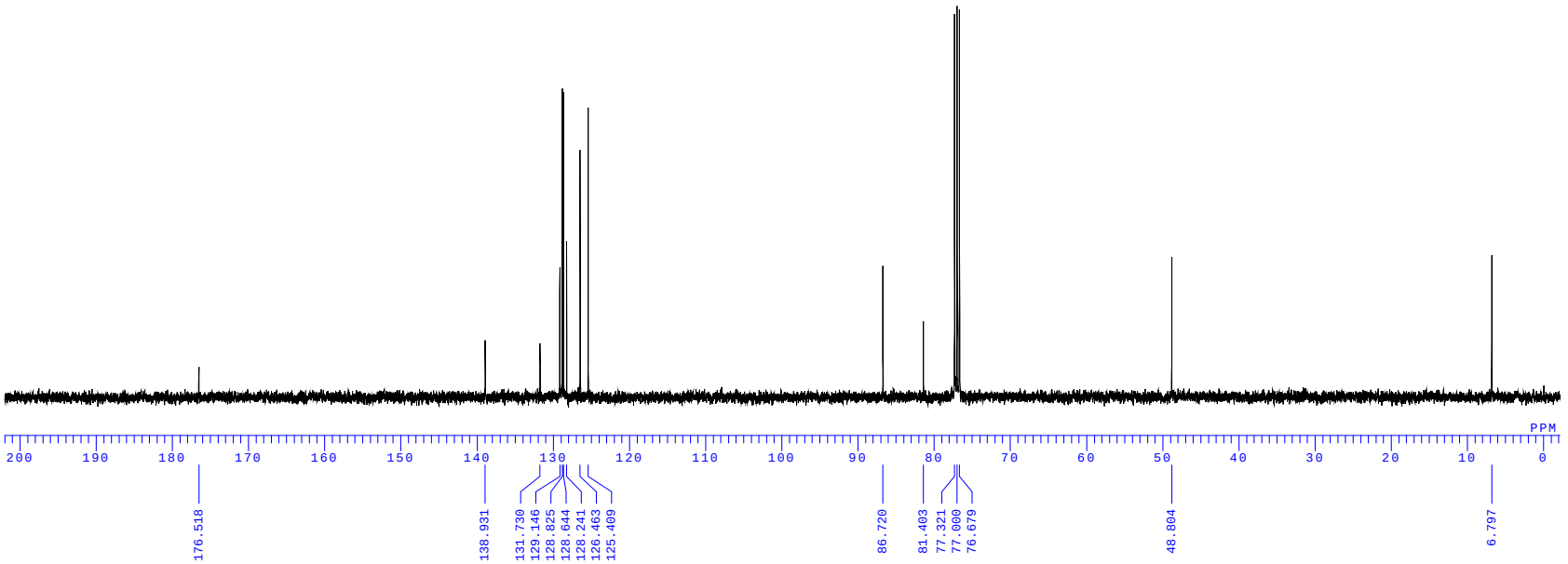
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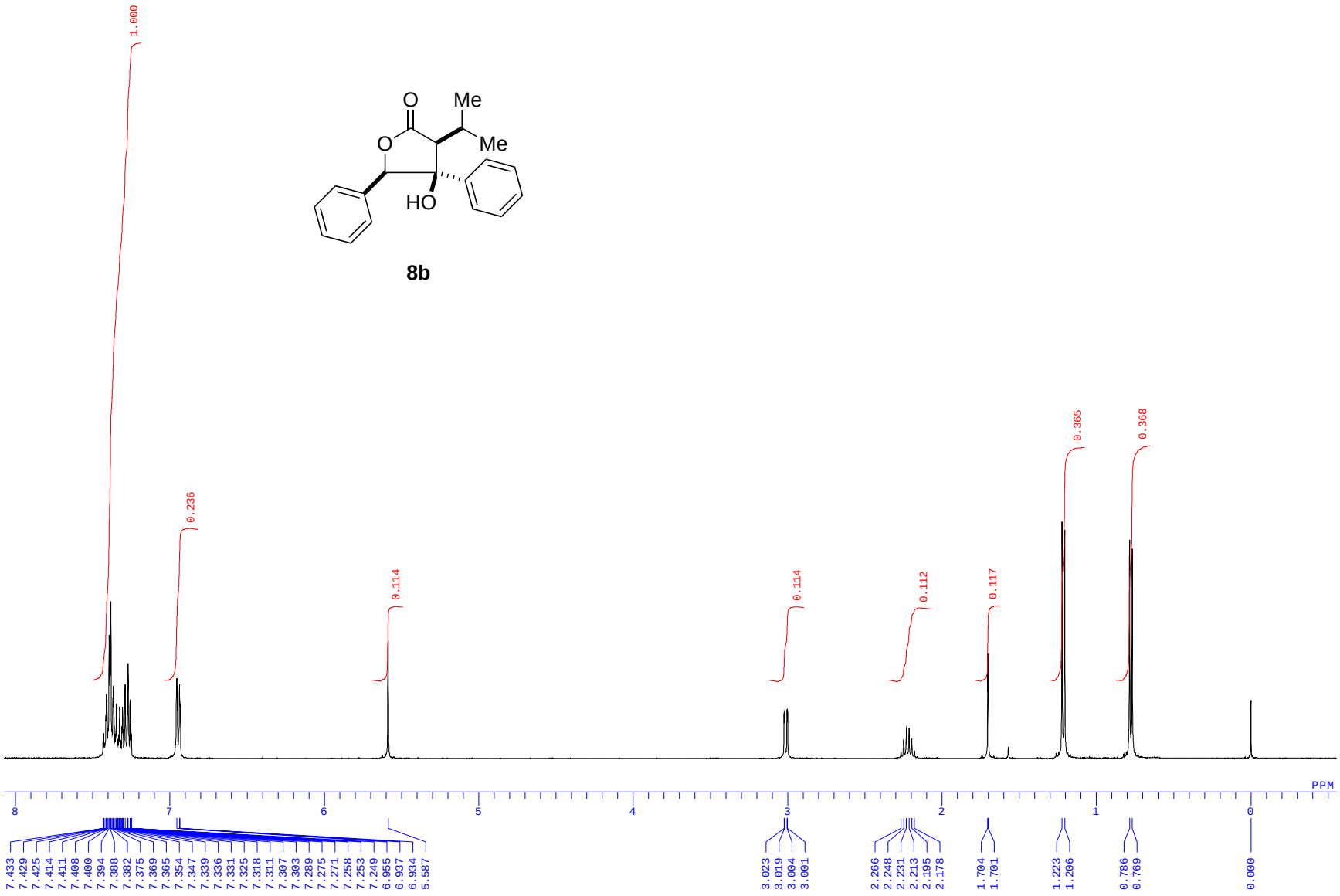
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Me
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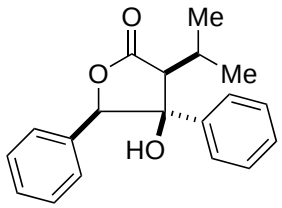
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HO

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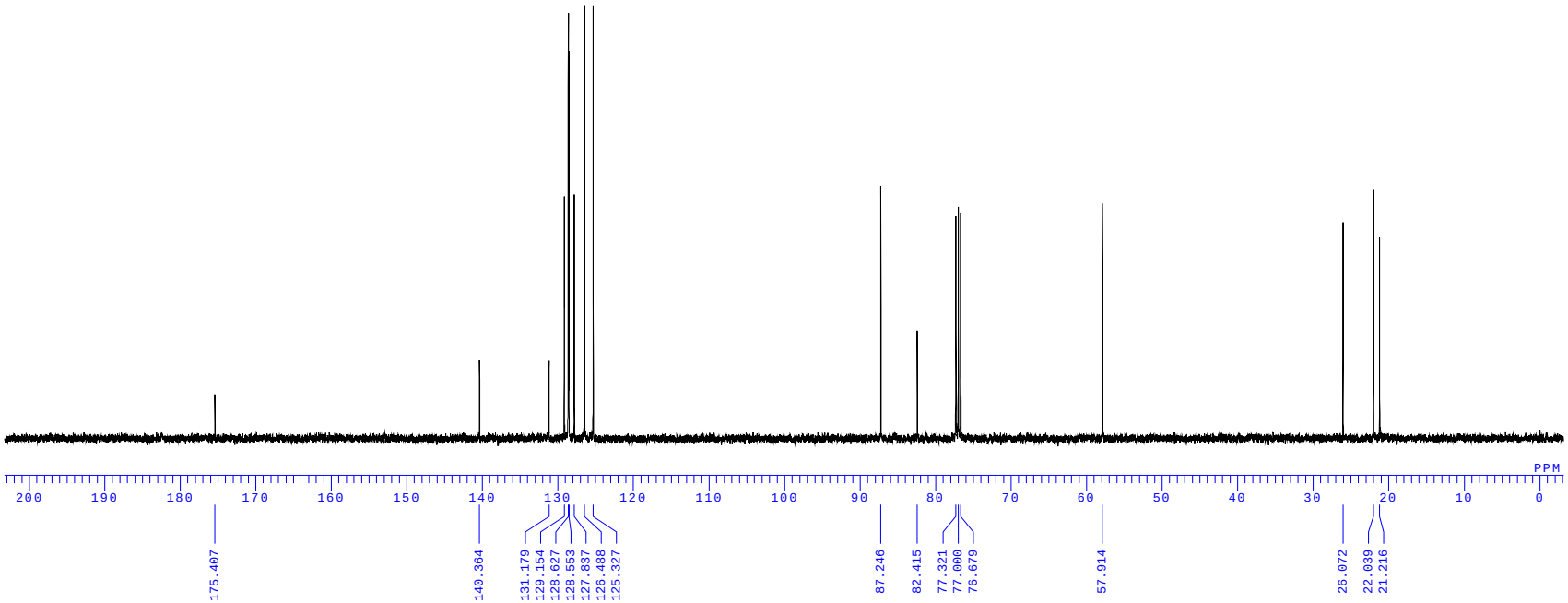
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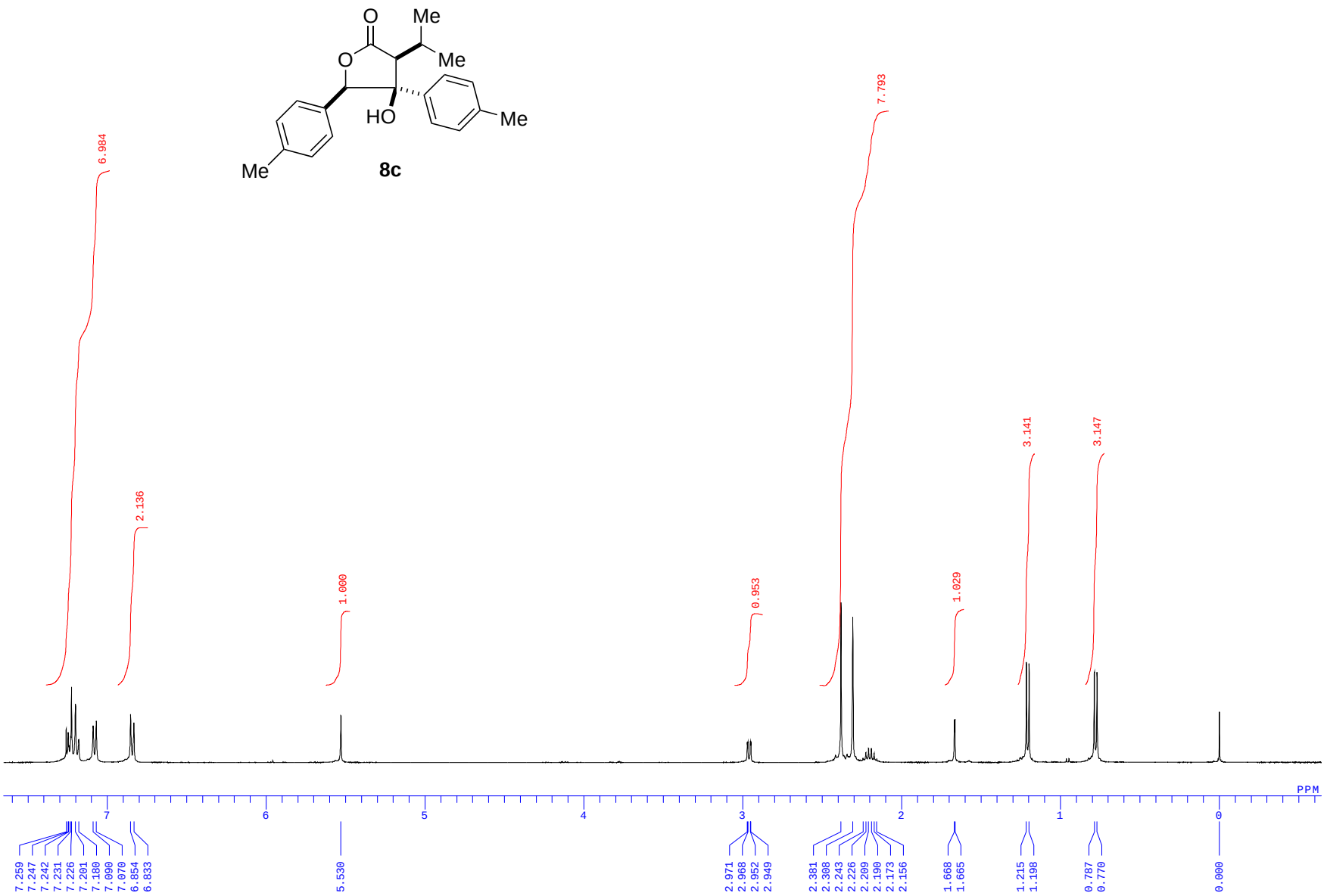
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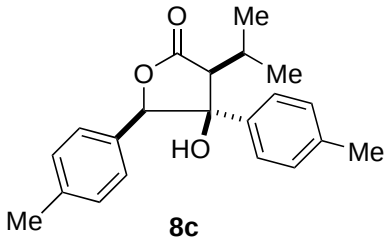
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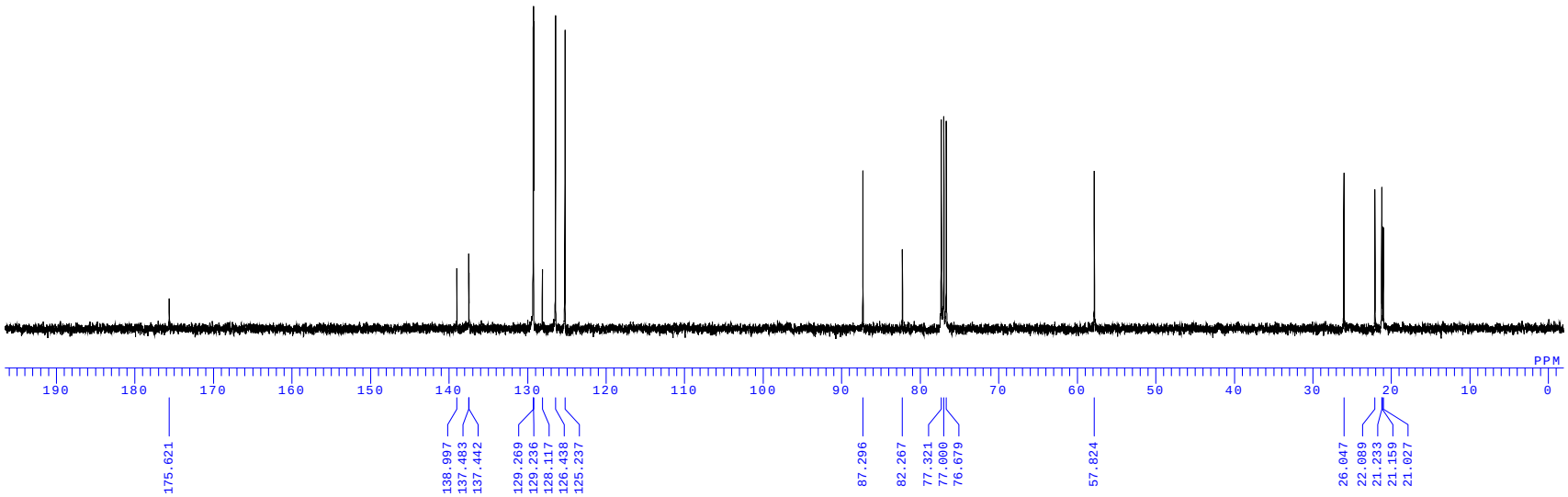
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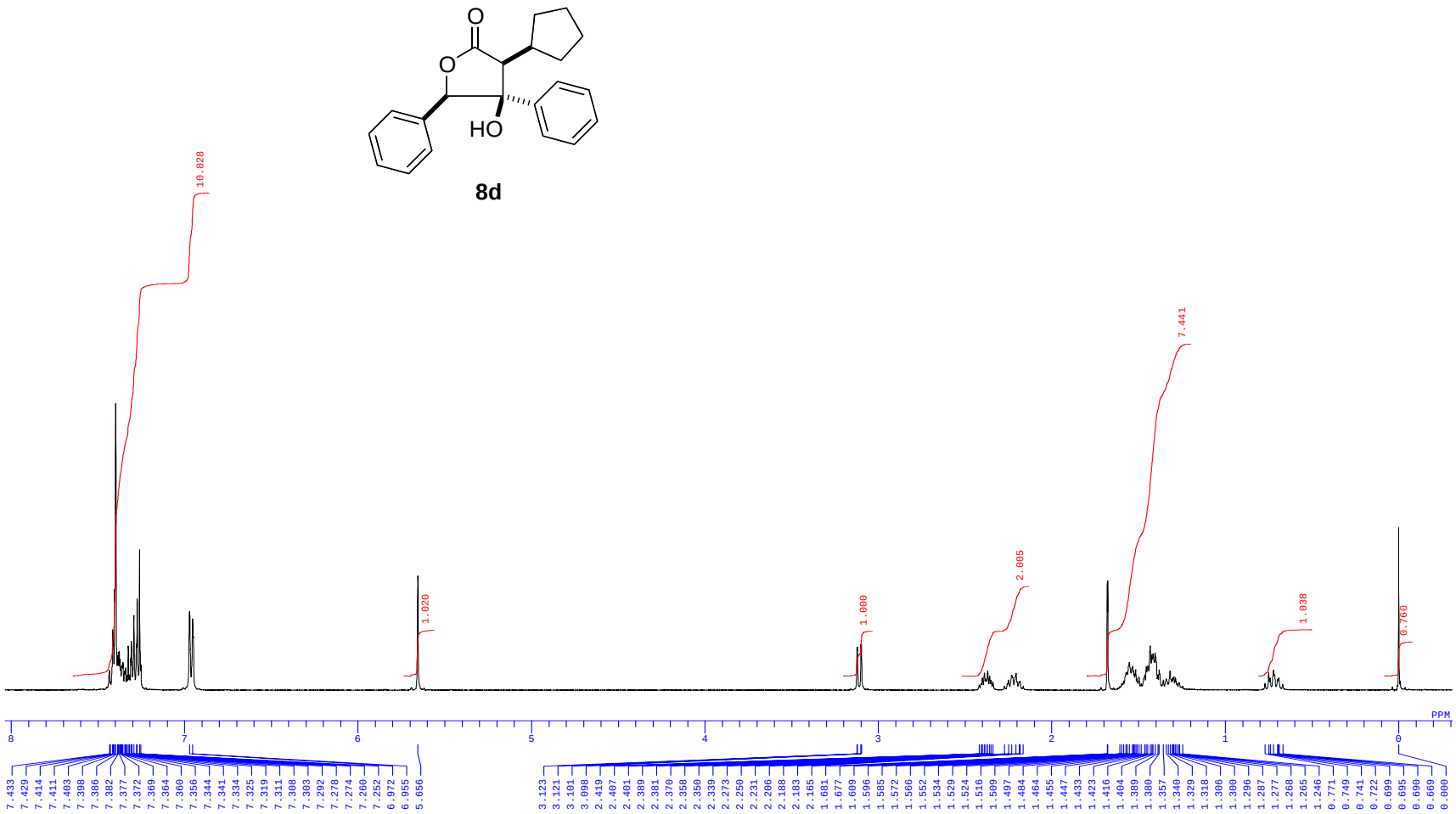
21.027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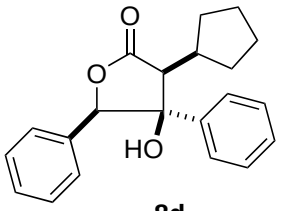
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HO

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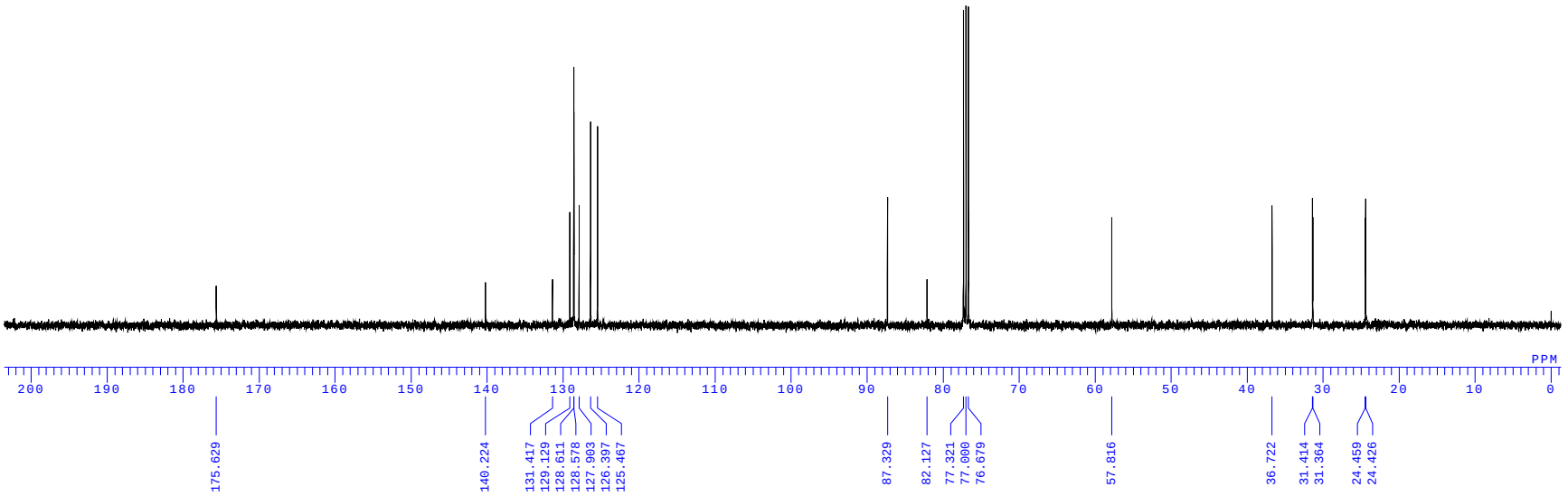
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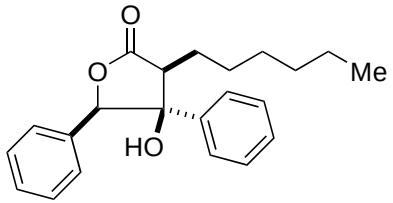
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Me
HO

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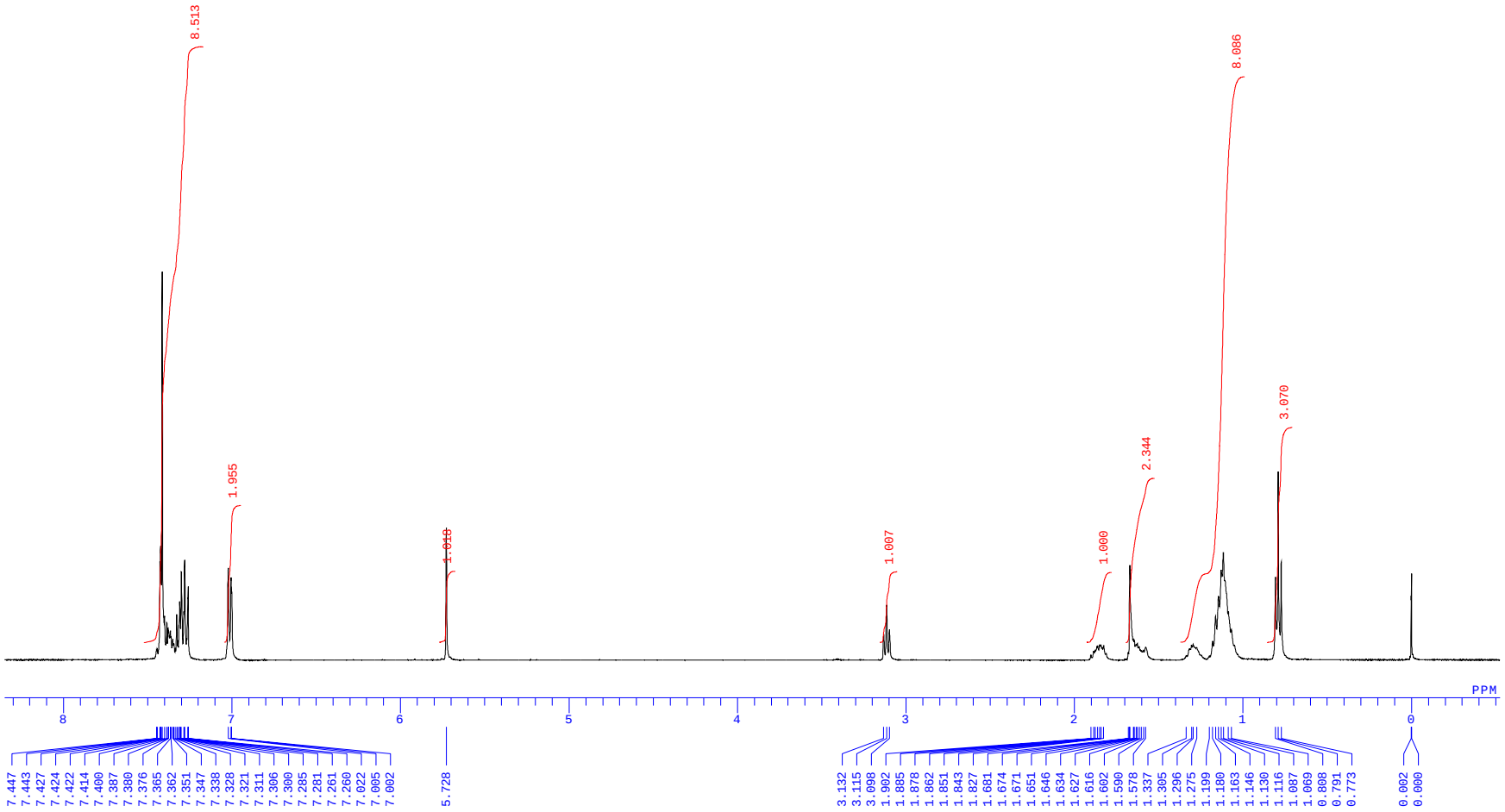
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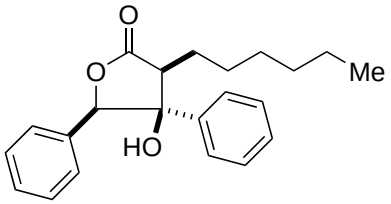
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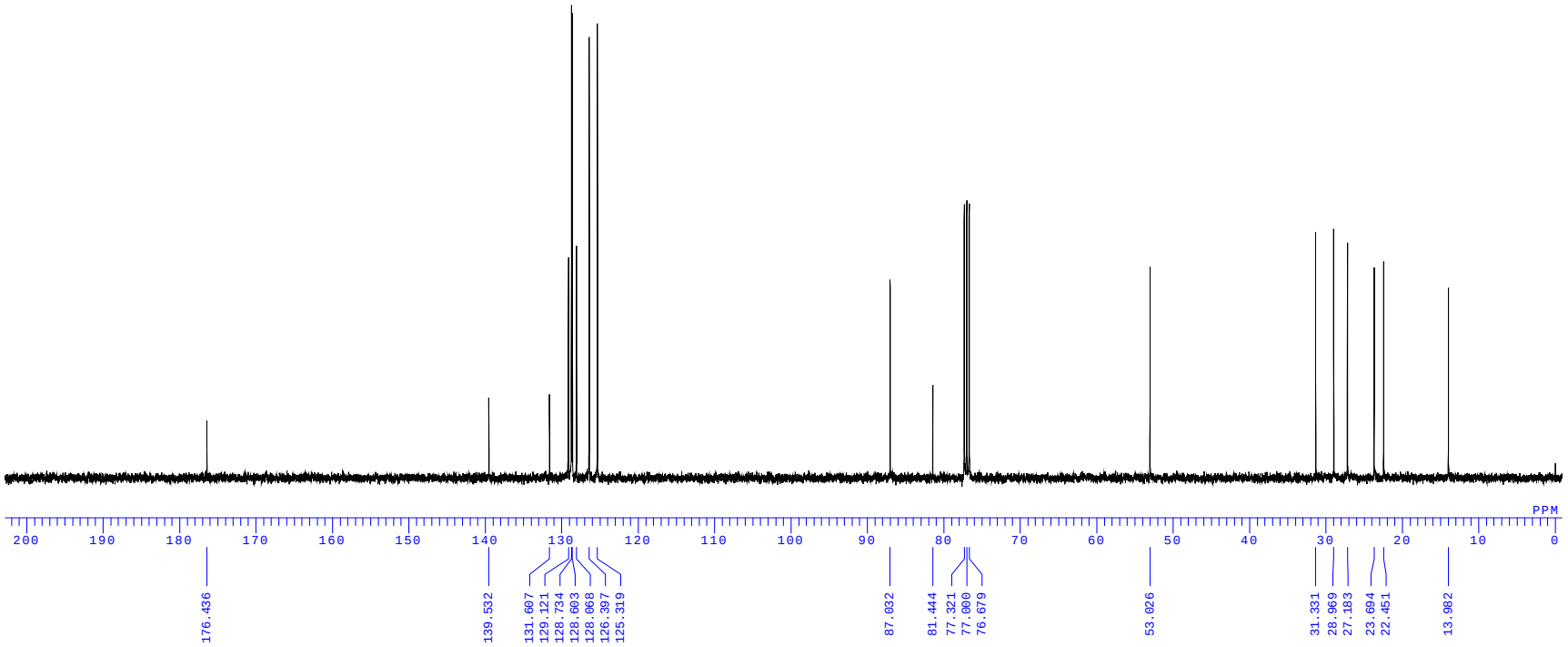
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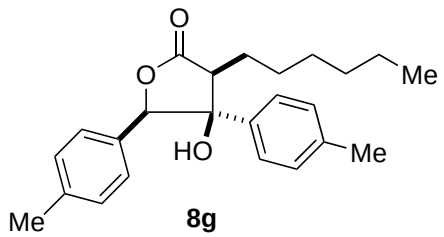
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HO
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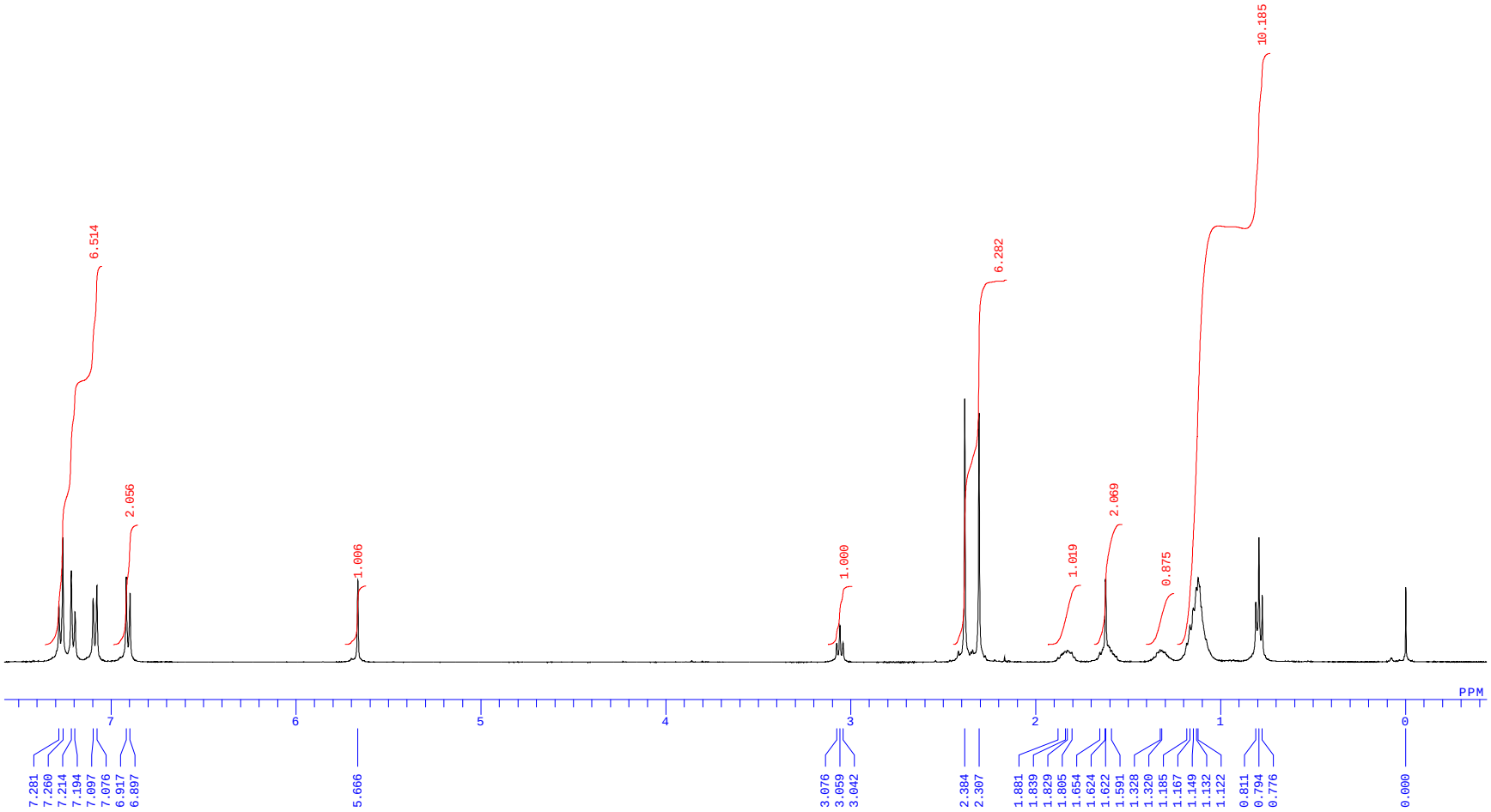
Me

8g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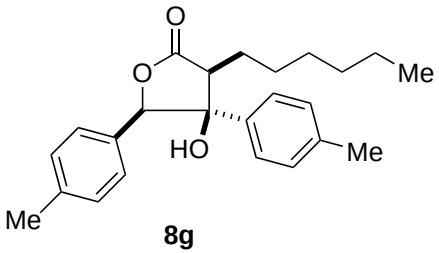
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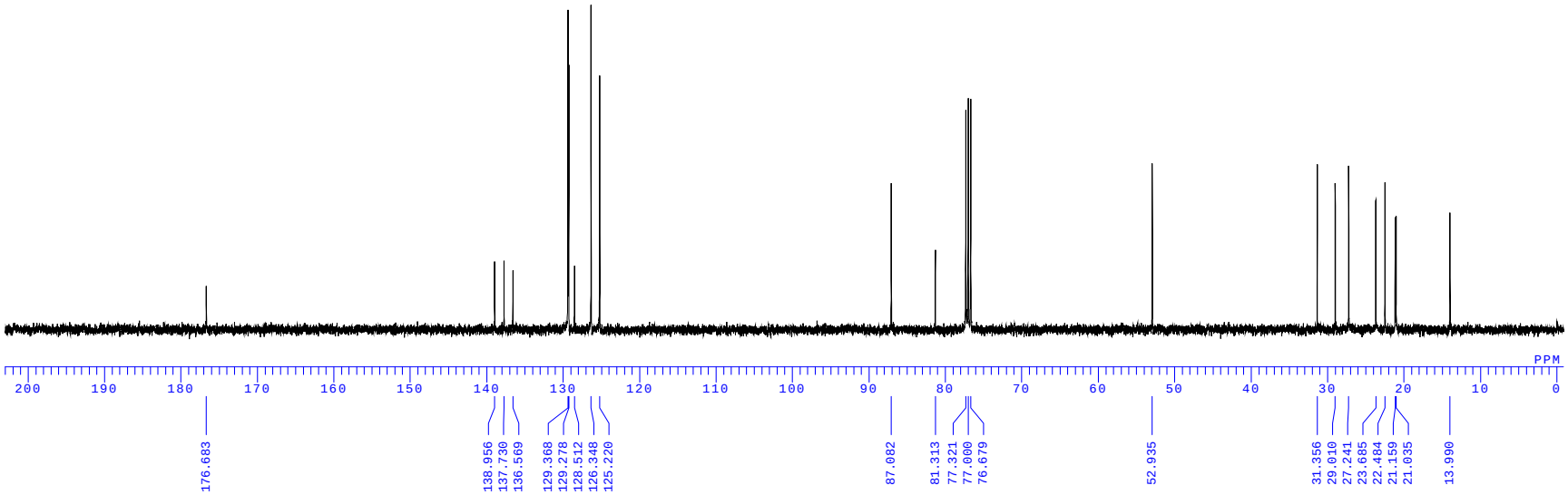
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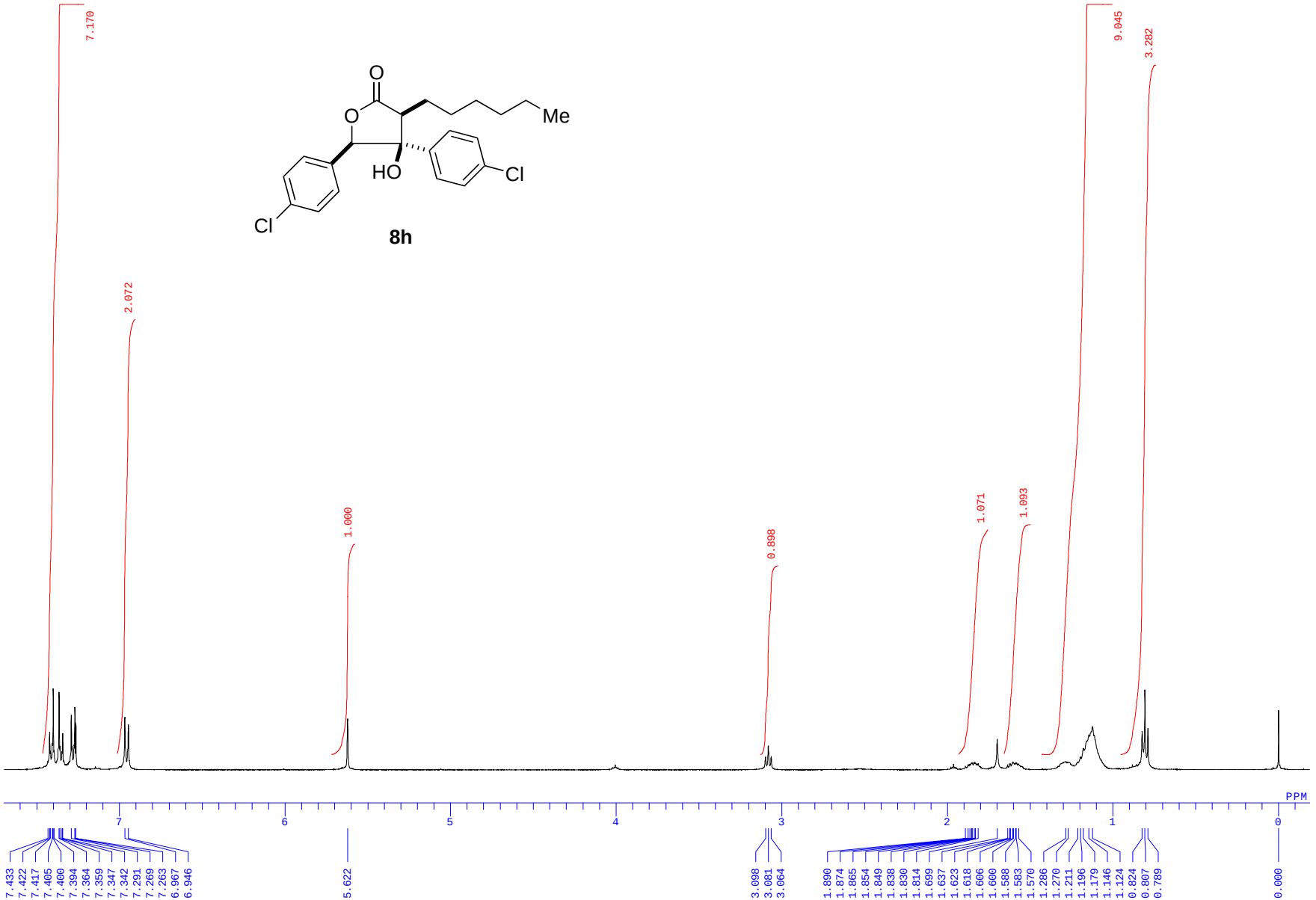
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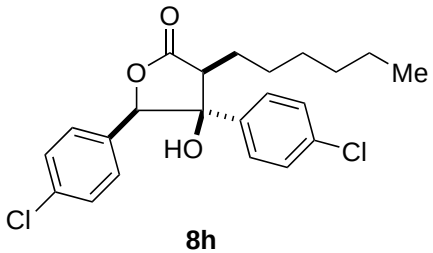
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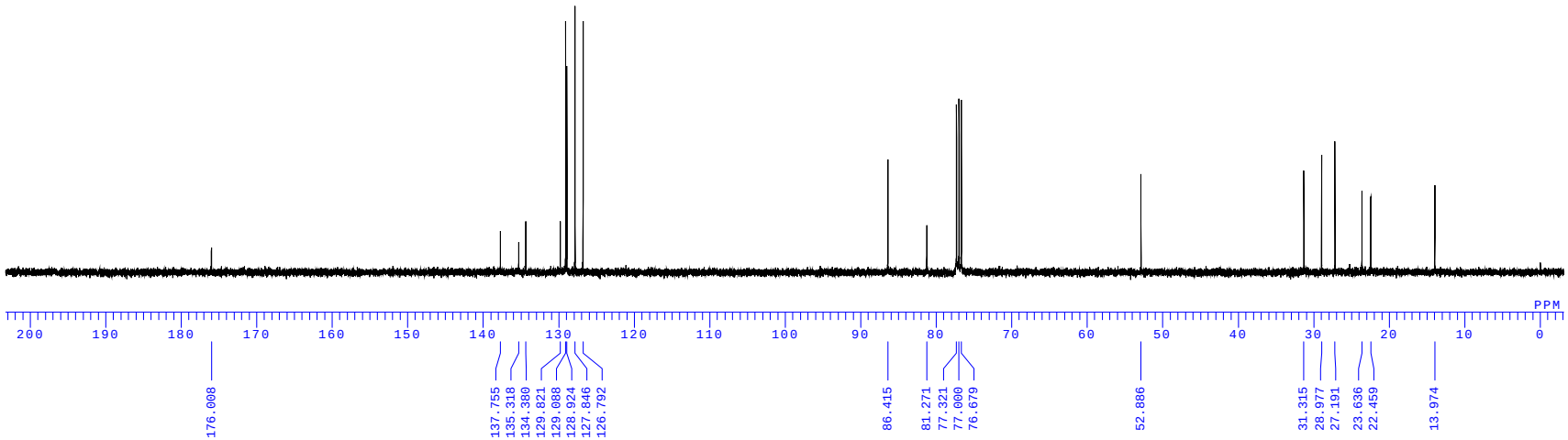
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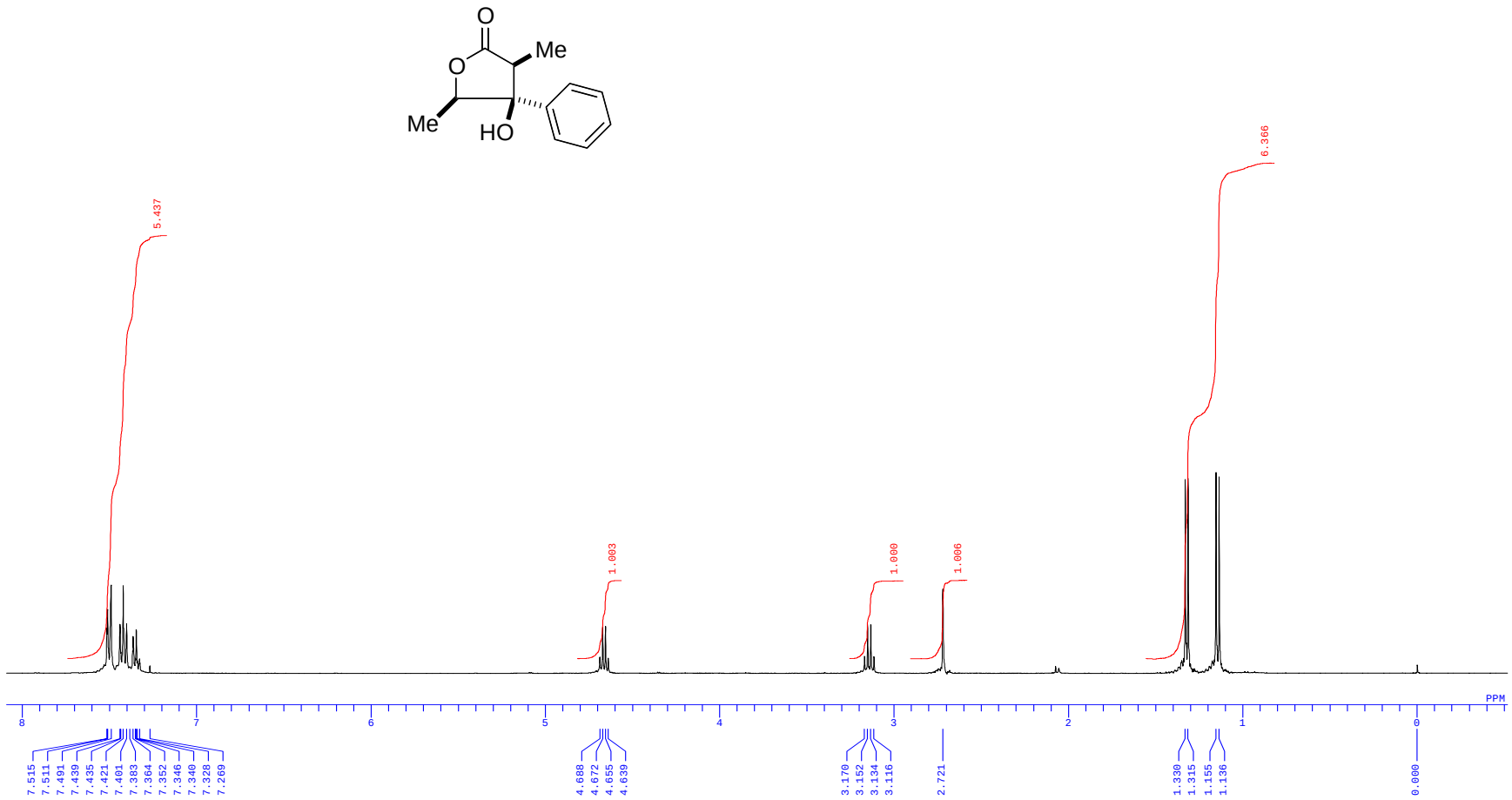
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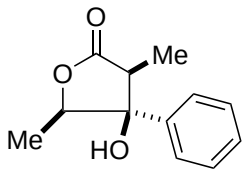
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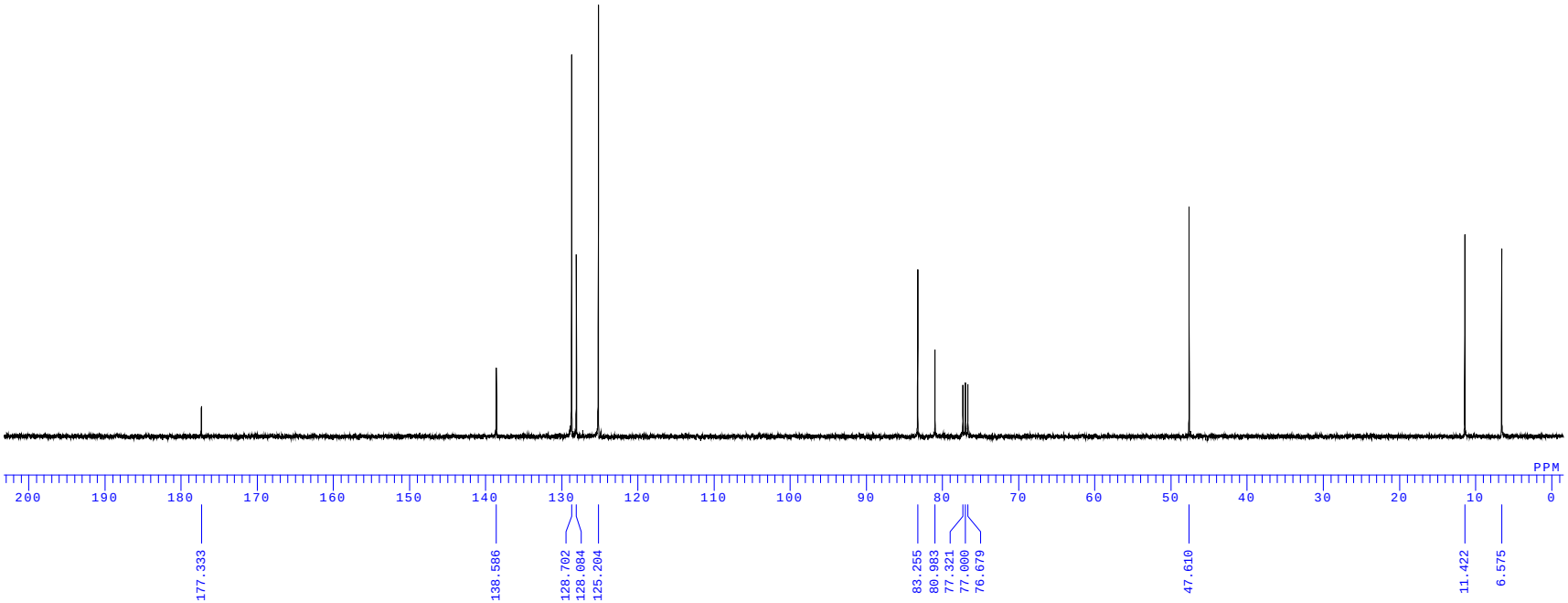
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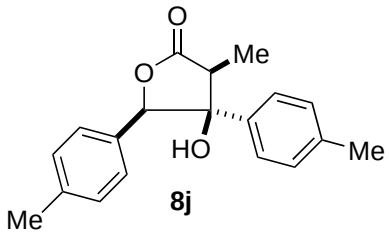
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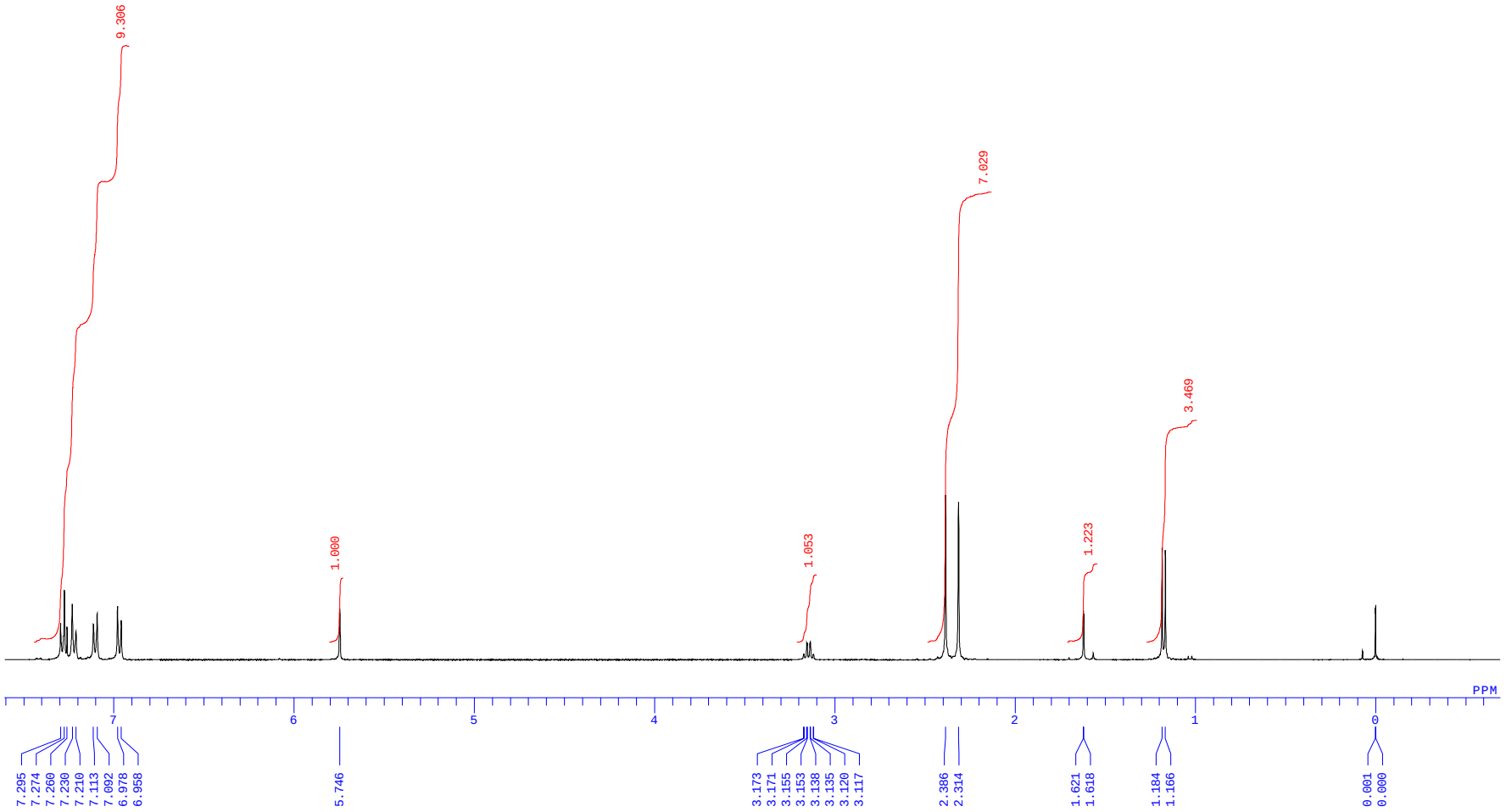
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6.958
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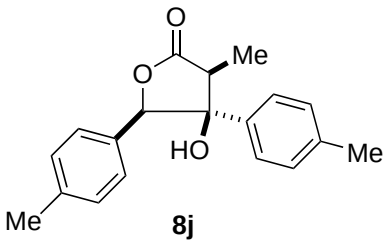
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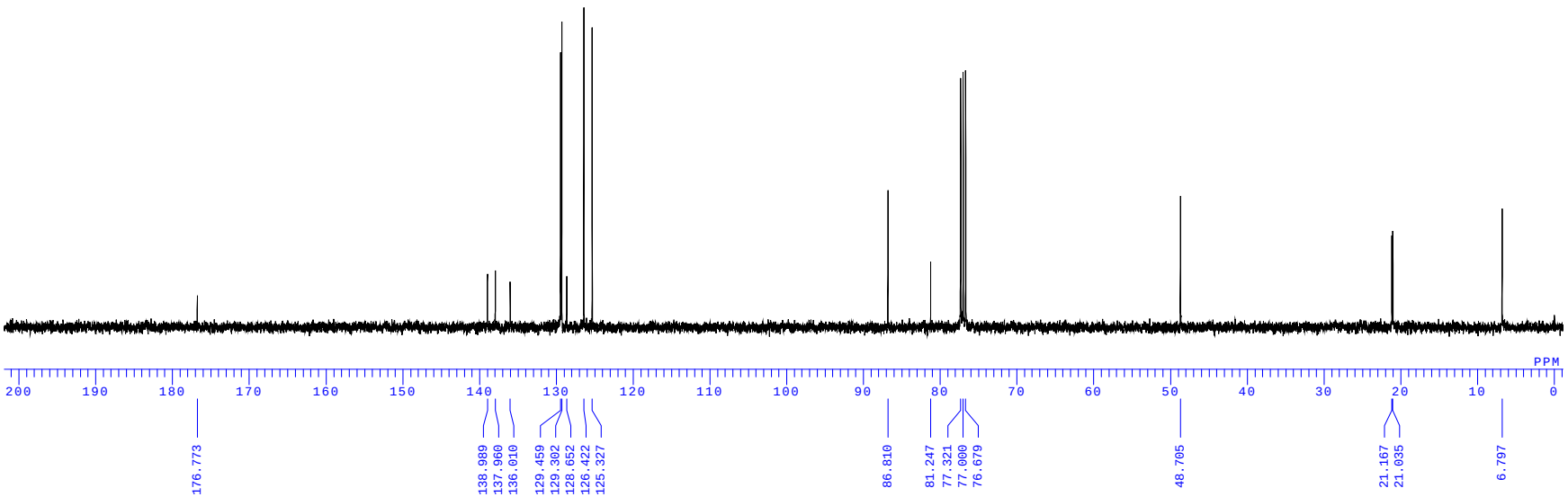
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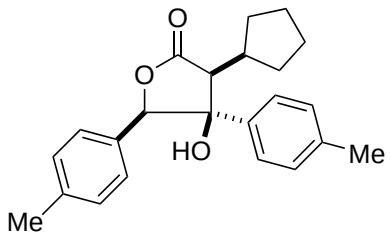
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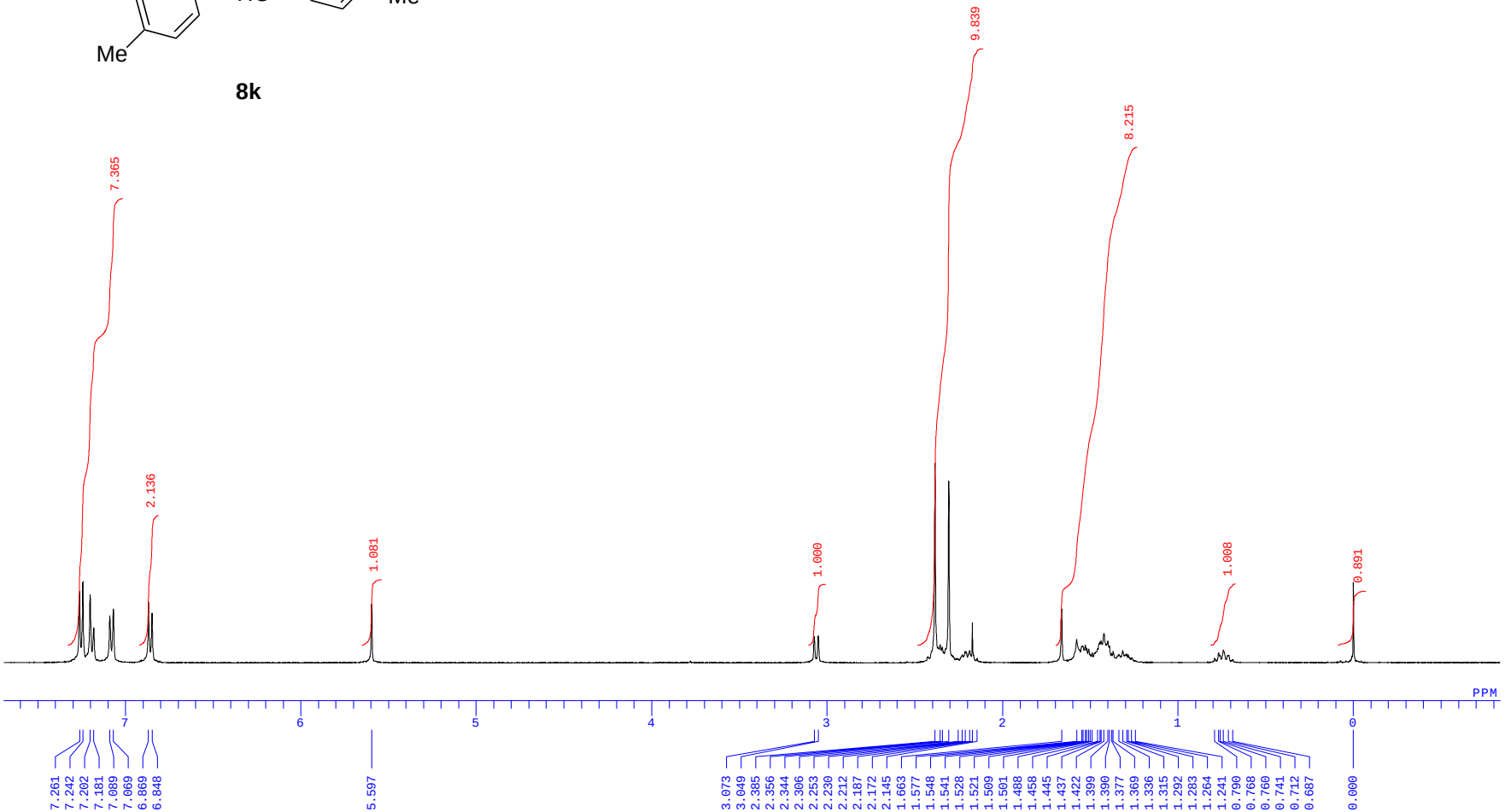
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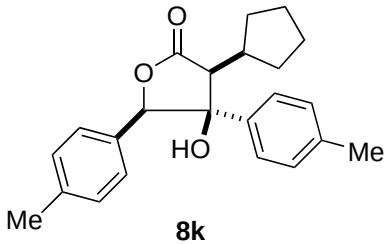
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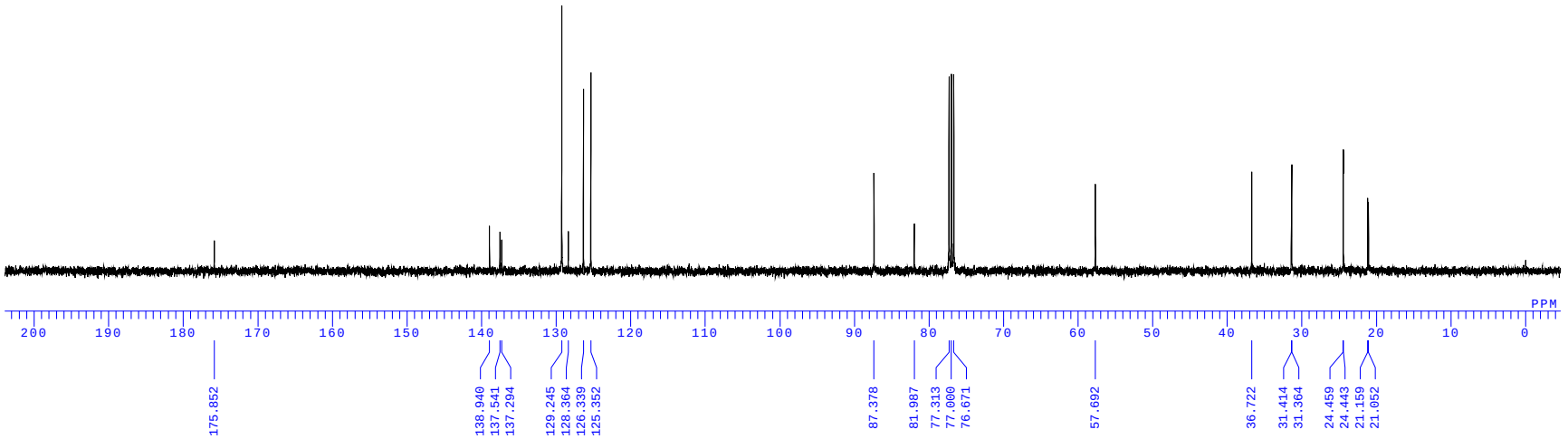
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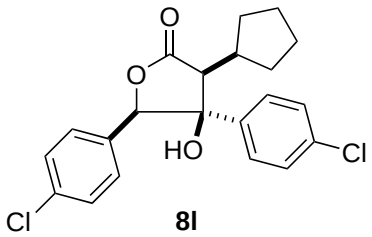
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Cl
Cl

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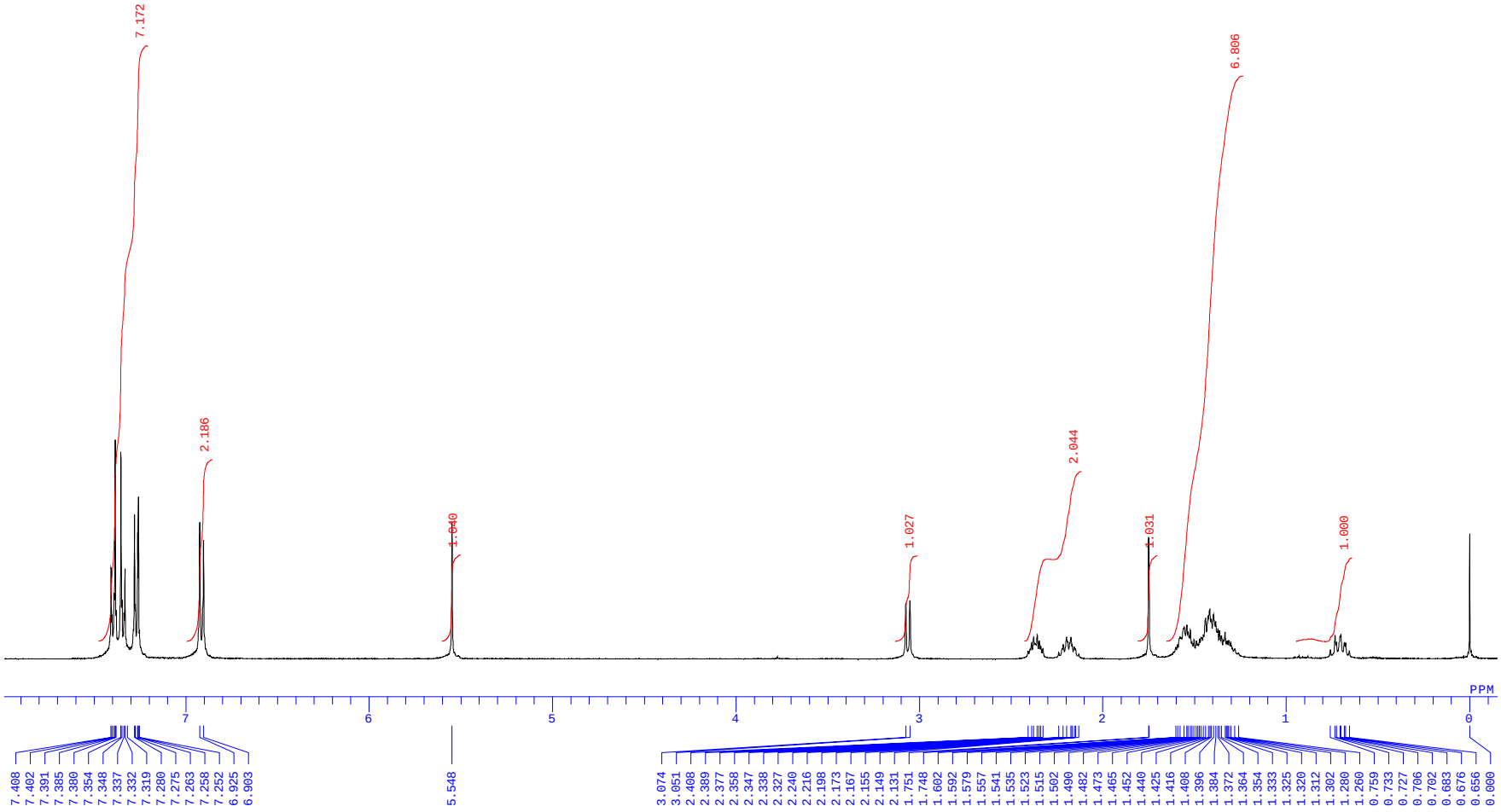
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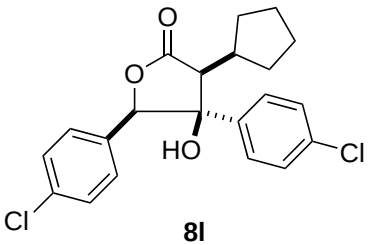
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HO
Cl
Cl

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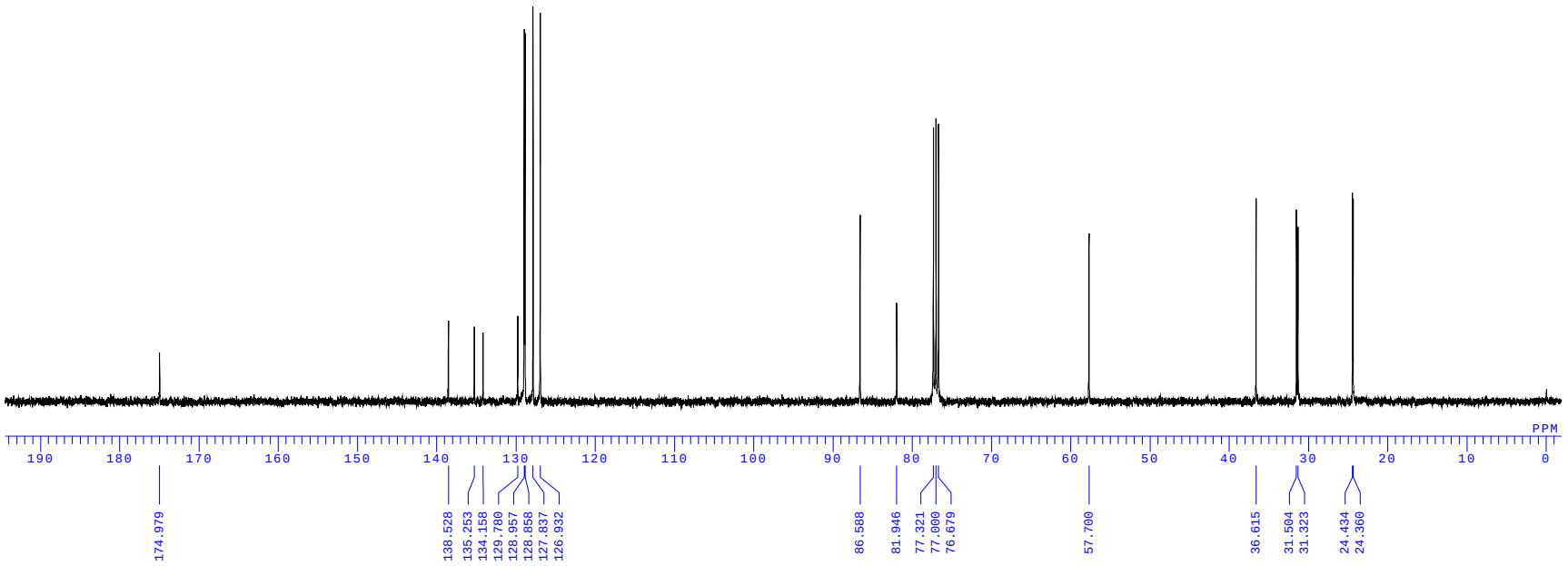
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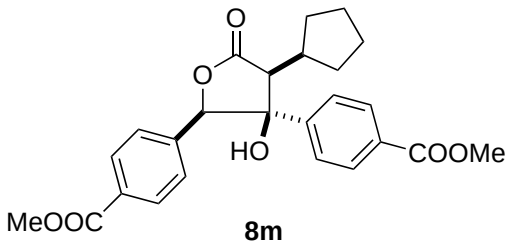
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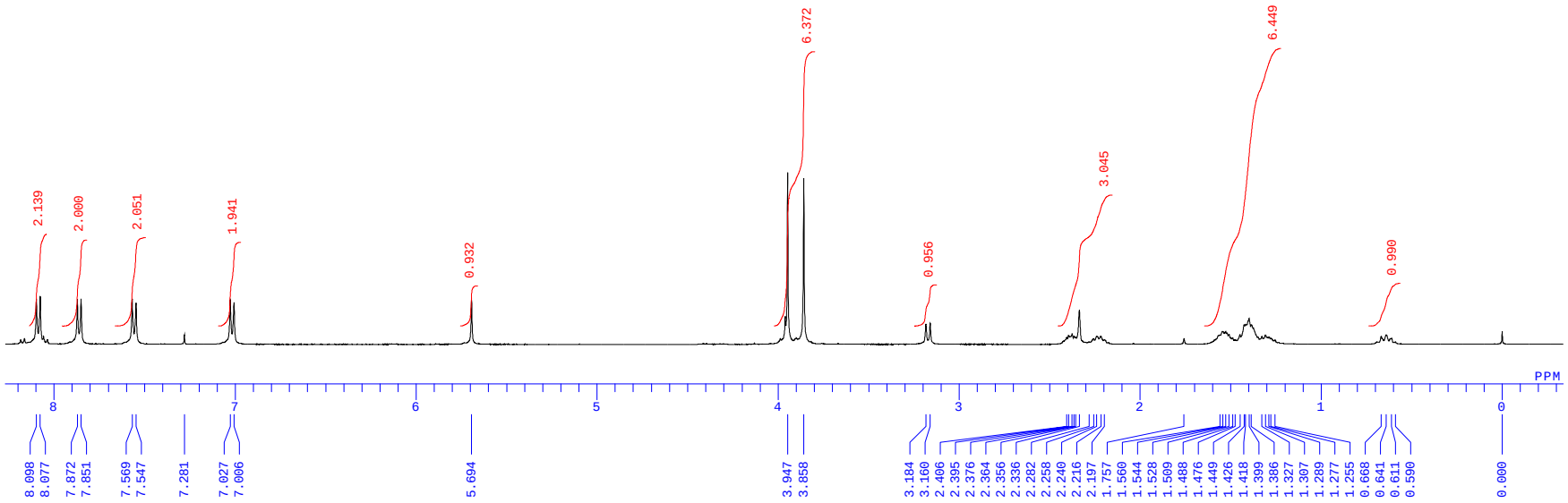
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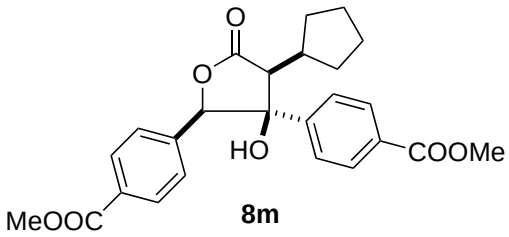
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COOMe
MeOOC

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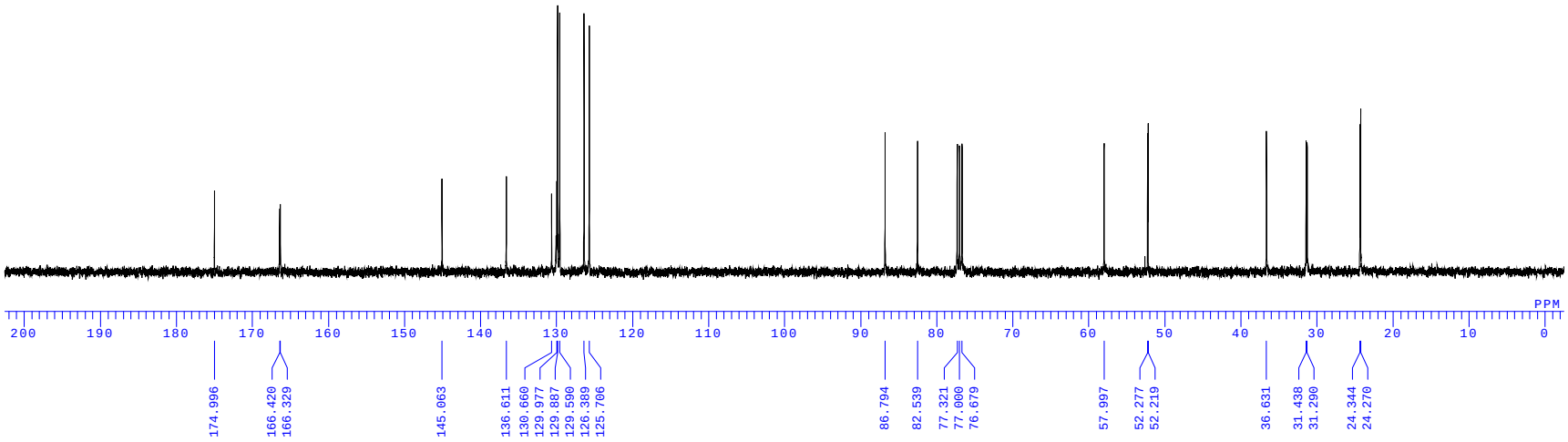
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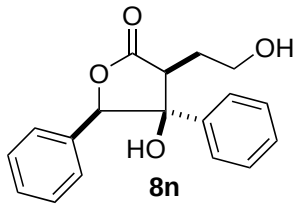
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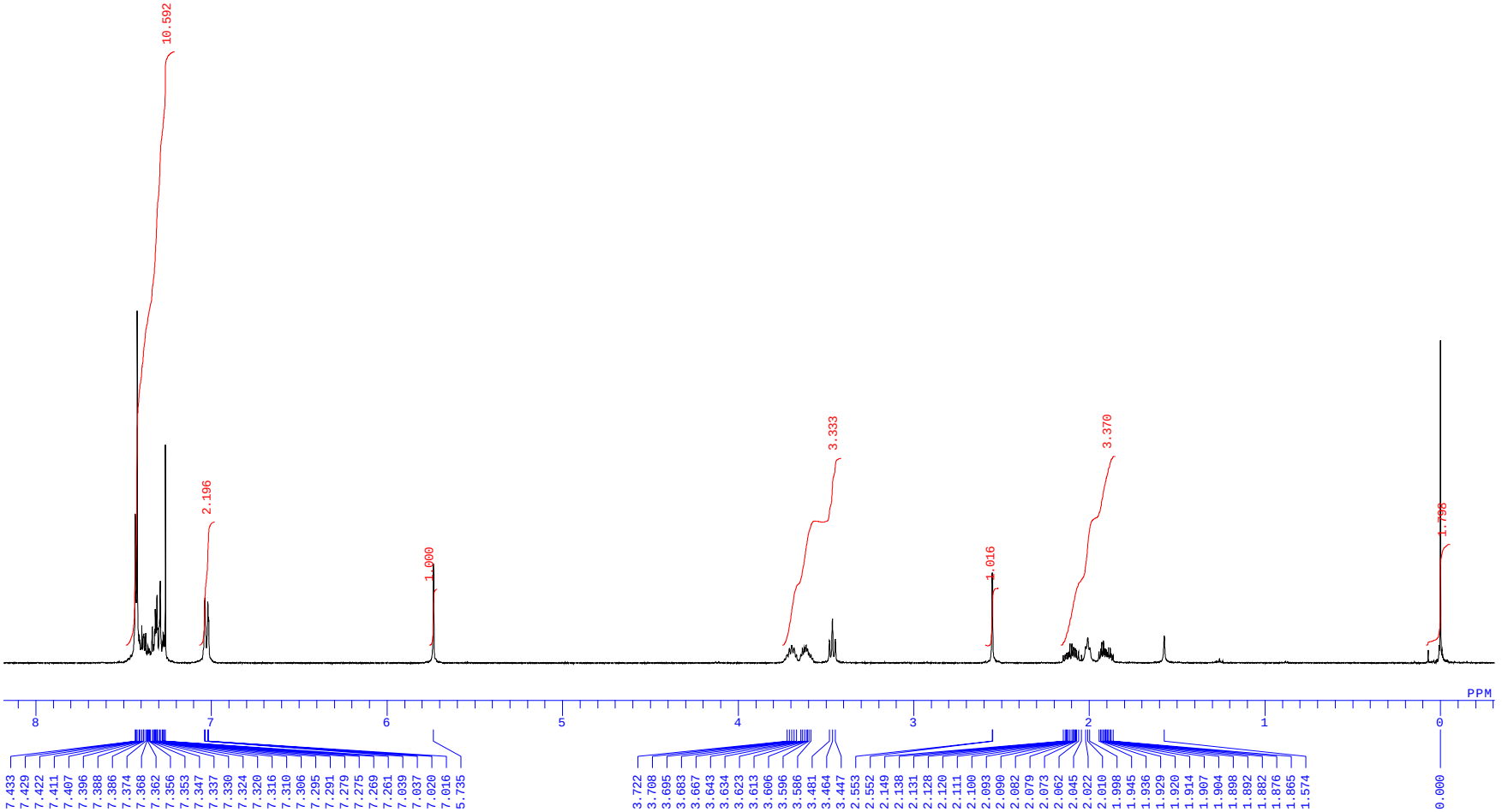
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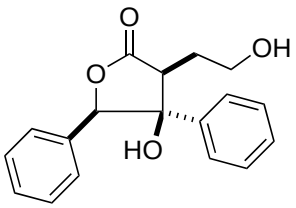
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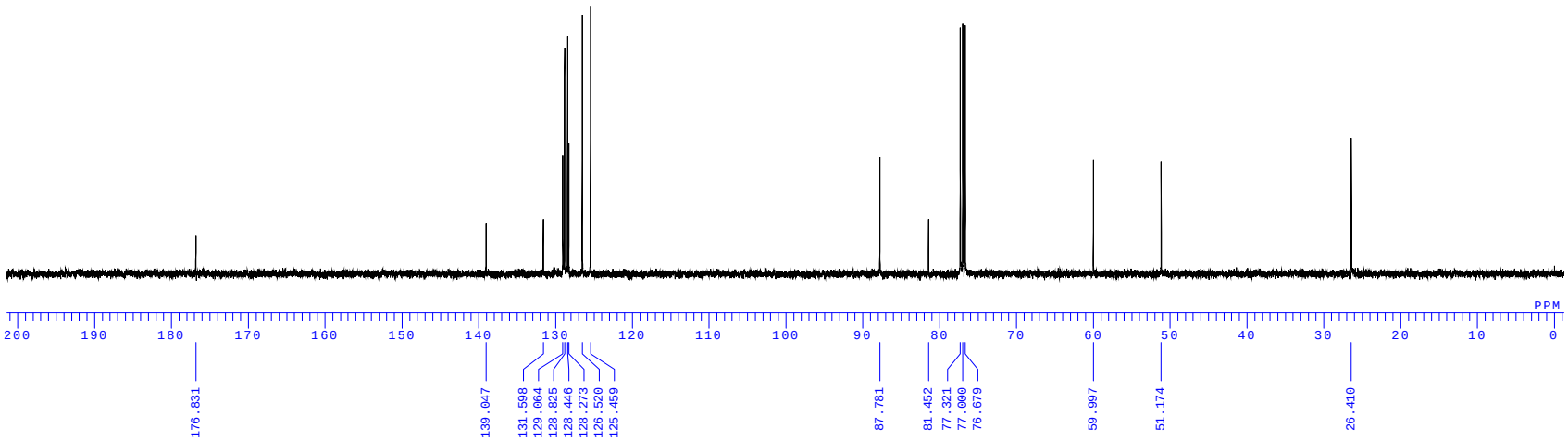
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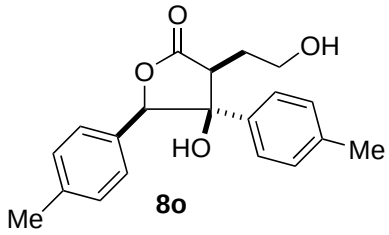
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Me

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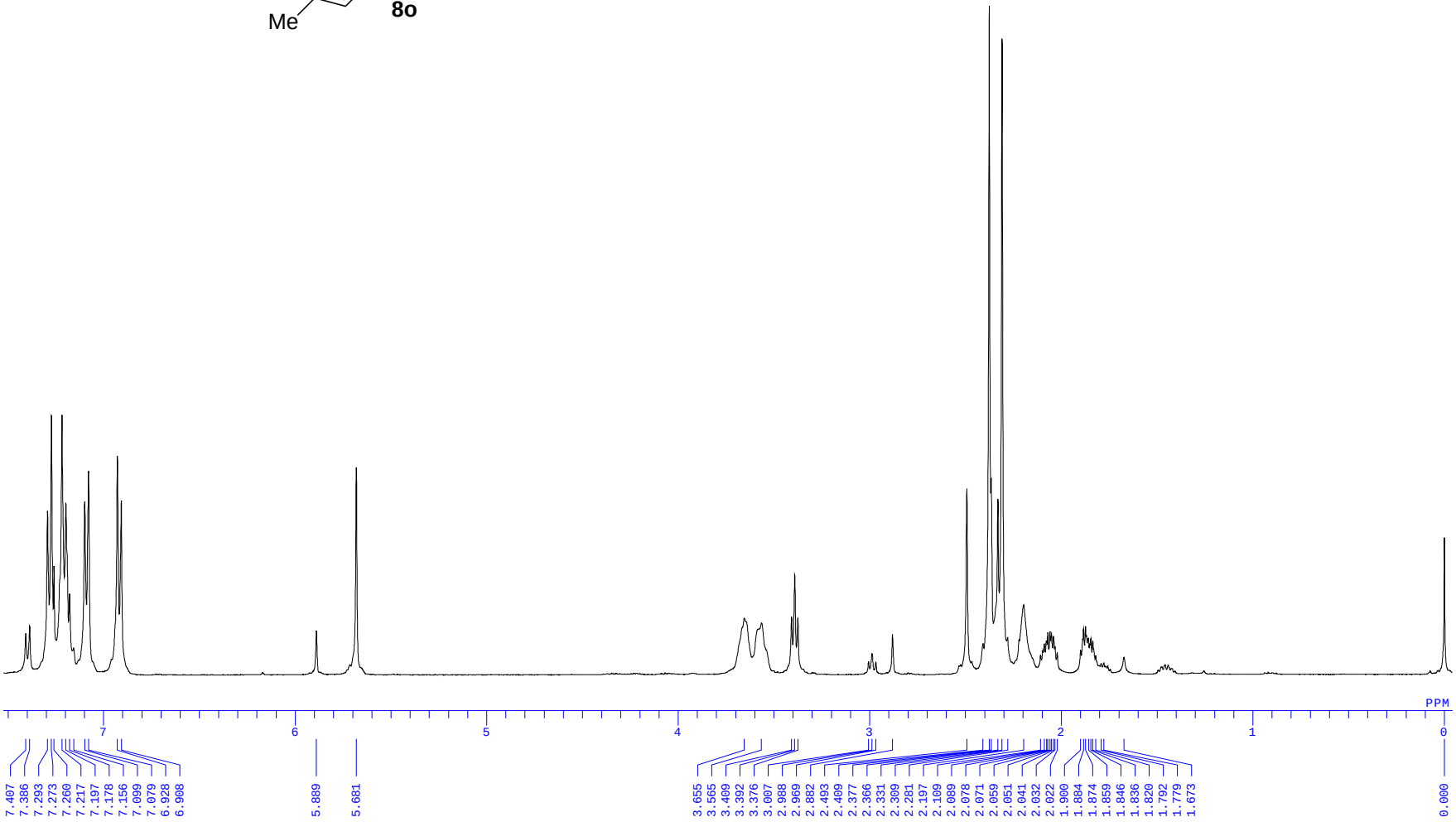
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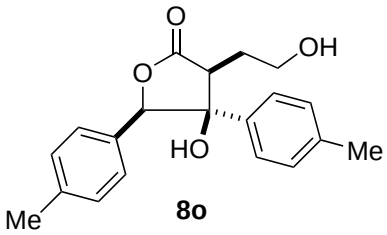
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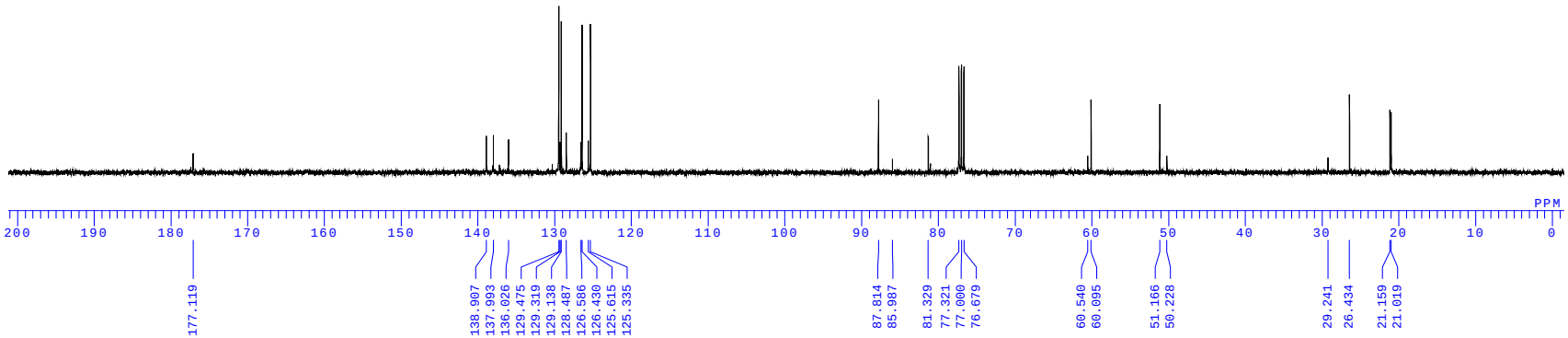
OH
HO
Me
Me

8o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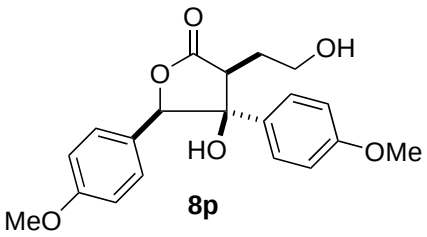
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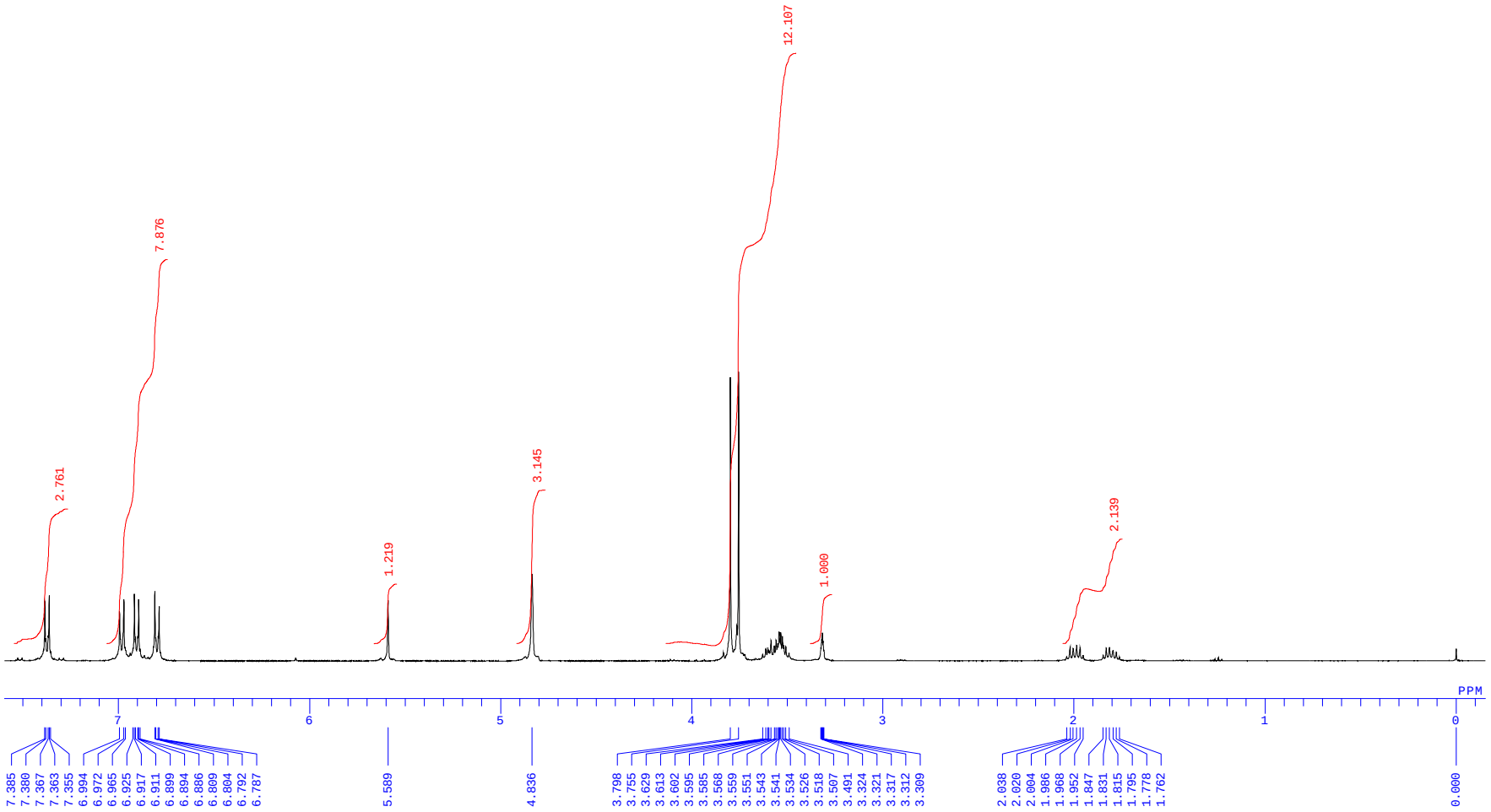
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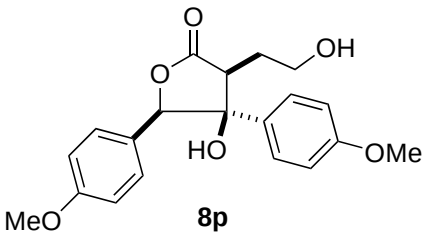
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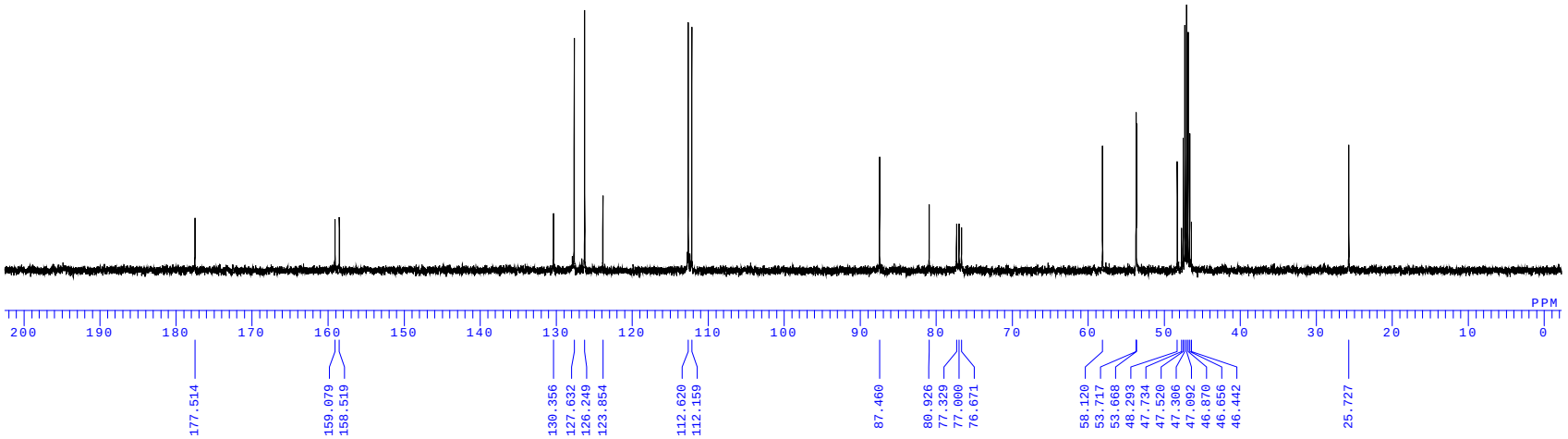
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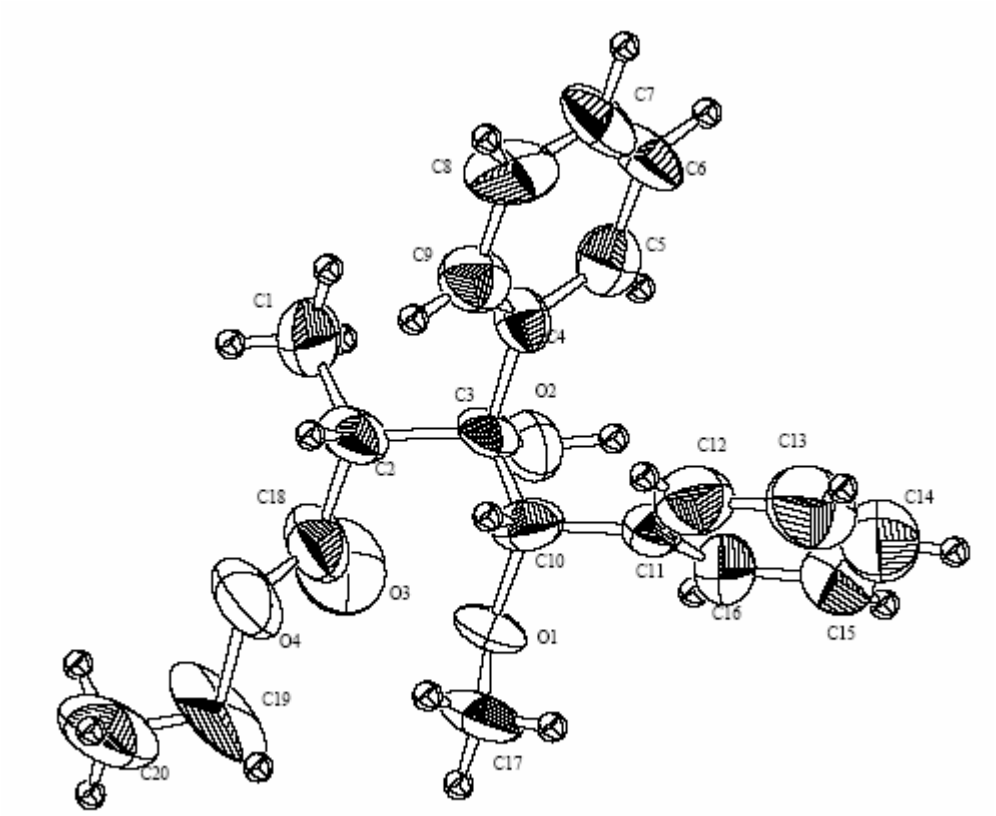
X-ray Structure Report **3a**



Experimental

Data Collection

A colorless prismatic crystal of $C_{20}H_{24}O_4$ having approximate dimensions of 0.40 x 0.40 x 0.40 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 24 carefully centered reflections in the range $20.79 < 2\theta < 28.15^{\circ}$ corresponded to a C-centered monoclinic cell with dimensions:

a = 37.51(3) Å
b = 9.04(2) Å β = 103.8(2)°
c = 11.09(2) Å
V = 3654(12) Å^3

For Z = 8 and F.W. = 328.41, the calculated density is 1.19 g/cm^3. Based on the systematic absences of:

hkl: h+k ± 2n
h0l: l ± 2n

packing considerations, a statistical analysis ofintensity distribution,and the successful solution and refinement of thestructure, the space group was determined to be:

C2/c (#15)

The data were collected at a temperature of 23 ± 1°C using the ω-2θ scan technique to a maximum 2θ value of 50.1°.Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.76° with a take-off angle of 6.0°. Scans of (1.84 + 0.30 tan θ)° were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 3074 reflections which were collected, 2821 were unique (R_{int} = 0.104). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.8 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.38 to 0.94.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 2145 observed reflections ($I > -0.30\sigma(I)$, $2\theta < 50.07$) and 217 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (Fo^2 - Fc^2) / \Sigma Fo^2 = 0.237$$

$$R_w = [(\Sigma\ w\ (Fo^2 - Fc^2)^2\ /\ \Sigma\ w\ (Fo^2)^2)]^{1/2} = 0.267$$

$$R1 = \Sigma\ ||Fo| - |Fc||\ /\ \Sigma\ |Fo| = 0.109 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight[4] was 1.86. The weighting scheme was based on counting statistics and included a factor (p = 0.055) to downweight the intense reflections. Plots of $\Sigma\ w\ (Fo^2 - Fc^2)^2$ versus Fo^2reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.53 and -0.50 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma\ w(F_o^2-F_c^2)^2$ where

$w = 1/[\sigma^2(Fo^2)] = [\sigma^2_C(Fo^2) + (p\ (Max(Fo^2,0)+2Fc^2)/3)^2]^{-1}$

$\sigma_C(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$[\Sigma\ w(|F_o|-|F_c|)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations

N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{20}H_{24}O_4$
Formula Weight	328.41
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.40 X 0.40 X 0.40 mm
Crystal System	monoclinic
Lattice Type	C-centered
No. of Reflections Used for Unit Cell Determination (2θ range)	24 (20.8 - 28.1°)
Omega Scan Peak Width at Half-height	0.76°
Lattice Parameters	a = 37.51(3) Å b = 9.04(2) Å c = 11.09(2) Å β = 103.8(2)° V = 3654(12) Å^3
Space Group	C2/c (#15)
Z value	8
D_{calc}	1.194 g/cm^3
F_{000}	1408.00
μ(MoKα)	0.82 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.65, 12.19, 42.58)
Temperature	23.0 °C
Collimator Size	1.0 mm
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm

Scan Type	ω-2θ
Scan Rate	16.0^{o}/min (in ω) (up to 5 scans)
Scan Width	(1.84 + 0.30 tan θ)^{o}
$2\theta_{max}$	50.1^{o}
No. of Reflections Measured	Total: 3074 Unique: 2821 (R_{int} = 0.104)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.3752 - 0.9359)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	1/σ^2(Fo^2)
p-factor	0.0550
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I>-0.30σ(I), 2θ < 50.07^{o})	2145
No. Variables	217
Reflection/Parameter Ratio	9.88
Residuals: R; Rw	0.237 ; 0.267
Residuals: R1	0.109
No. of Reflections to calc R1	863
Goodness of Fit Indicator	1.86
Max Shift/Error in Final Cycle	0.003
Maximum peak in Final Diff. Map	0.53 e^-/Å^3
Minimum peak in Final Diff. Map	-0.50 e^-/Å^3

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.1743(2)	0.6725(7)	0.2178(6)	6.2(2)
O(2)	0.1462(2)	0.4801(6)	0.0213(5)	5.2(2)
O(3)	0.2161(2)	0.4127(10)	0.1573(8)	9.3(3)
O(4)	0.2168(2)	0.4276(9)	0.3584(8)	8.8(2)
C(1)	0.1579(3)	0.204(1)	0.1723(9)	7.0(3)
C(2)	0.1617(2)	0.3601(10)	0.2232(8)	5.0(3)
C(3)	0.1350(2)	0.4743(9)	0.1331(9)	4.2(2)
C(4)	0.0961(3)	0.4152(9)	0.1096(9)	4.3(3)
C(5)	0.0738(3)	0.413(1)	-0.0070(10)	5.9(3)
C(6)	0.0368(3)	0.364(1)	-0.032(1)	6.7(3)
C(7)	0.0235(3)	0.328(1)	0.064(1)	6.5(3)
C(8)	0.0432(3)	0.324(1)	0.184(1)	7.4(4)
C(9)	0.0813(3)	0.3749(10)	0.2073(9)	5.6(3)
C(10)	0.1364(3)	0.6254(9)	0.1967(8)	5.1(3)
C(11)	0.1123(3)	0.7410(10)	0.122(1)	4.6(3)
C(12)	0.0825(4)	0.789(1)	0.163(1)	7.2(3)
C(13)	0.0594(4)	0.901(2)	0.097(2)	9.6(5)
C(14)	0.0664(6)	0.958(2)	-0.006(2)	9.9(5)
C(15)	0.0982(4)	0.919(1)	-0.043(1)	8.2(4)
C(16)	0.1190(3)	0.809(1)	0.023(1)	5.9(3)
C(17)	0.1831(3)	0.780(1)	0.317(1)	7.8(3)
C(18)	0.2012(3)	0.408(1)	0.239(1)	6.8(3)
C(19)	0.2540(3)	0.495(2)	0.384(2)	17.8(7)
C(20)	0.2764(4)	0.427(2)	0.480(1)	10.9(5)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(1)	0.1333	0.1706	0.1661	7.8
H(2)	0.1745	0.1427	0.2265	7.8
H(3)	0.1627	0.2053	0.0927	7.8
H(4)	0.1552	0.3617	0.3296	7.1
H(5)	0.1283	0.5442	-0.0349	5.7
H(6)	0.0848	0.4367	-0.0760	7.1
H(7)	0.0201	0.3622	-0.1117	7.7
H(8)	-0.0031	0.2930	0.0479	7.8
H(9)	0.0302	0.2985	0.2444	8.4
H(10)	0.0964	0.3752	0.2910	6.0
H(11)	0.1310	0.6137	0.2801	6.0
H(12)	0.1804	0.7369	0.3902	9.3
H(13)	0.1676	0.8624	0.2944	9.3
H(14)	0.2082	0.8106	0.3254	9.3
H(15)	0.1404	0.7768	-0.0022	7.3
H(16)	0.1042	0.9575	-0.1156	9.6
H(17)	0.0546	1.0326	-0.0532	11.9
H(18)	0.0392	0.9370	0.1207	11.3
H(19)	0.0780	0.7468	0.2345	6.9
H(20)	0.2502	0.6031	0.4166	20.2
H(21)	0.2627	0.5116	0.3158	20.2
H(22)	0.2680	0.4417	0.5509	11.6
H(23)	0.2993	0.4563	0.4825	11.6
H(24)	0.2720	0.3251	0.4525	11.6

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}

$B_{eq} = 8/3\ PI^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.032(4)	0.088(5)	0.104(5)	-0.025(4)	-0.010(4)	0.004(4)
O(2)	0.061(5)	0.081(4)	0.056(4)	0.003(4)	0.011(4)	0.012(3)
O(3)	0.065(6)	0.167(8)	0.141(8)	0.012(5)	0.059(6)	0.027(6)
O(4)	0.050(5)	0.161(7)	0.110(6)	-0.016(5)	-0.010(5)	0.052(5)
C(1)	0.068(8)	0.076(7)	0.114(9)	0.016(7)	0.009(7)	0.018(6)
C(2)	0.038(6)	0.081(7)	0.071(7)	-0.012(5)	0.010(5)	0.000(6)
C(3)	0.024(6)	0.071(6)	0.063(7)	-0.012(5)	0.005(5)	0.006(6)
C(4)	0.044(7)	0.066(6)	0.053(6)	0.009(5)	0.011(6)	0.005(5)
C(5)	0.078(9)	0.075(7)	0.068(7)	-0.002(7)	0.013(7)	0.014(6)
C(6)	0.038(7)	0.117(9)	0.086(9)	-0.007(7)	-0.009(6)	-0.004(7)
C(7)	0.031(6)	0.115(9)	0.093(9)	0.009(7)	-0.004(7)	-0.014(8)
C(8)	0.042(8)	0.093(8)	0.16(1)	-0.016(6)	0.058(8)	-0.008(8)
C(9)	0.054(7)	0.071(7)	0.087(8)	0.003(5)	0.015(6)	-0.010(6)
C(10)	0.065(8)	0.058(6)	0.073(7)	-0.023(6)	0.020(6)	0.000(5)
C(11)	0.039(7)	0.048(6)	0.081(8)	-0.002(5)	0.004(6)	-0.001(6)
C(12)	0.082(9)	0.072(7)	0.116(10)	-0.003(7)	0.016(8)	-0.012(7)
C(13)	0.07(1)	0.10(1)	0.18(2)	0.023(9)	0.02(1)	-0.03(1)
C(14)	0.14(2)	0.09(1)	0.13(1)	0.00(1)	-0.02(1)	-0.006(9)
C(15)	0.10(1)	0.087(9)	0.11(1)	-0.011(9)	-0.020(9)	0.001(8)
C(16)	0.067(8)	0.068(7)	0.085(8)	0.007(6)	0.008(7)	0.013(6)
C(17)	0.046(7)	0.100(8)	0.129(10)	-0.029(6)	-0.017(7)	-0.012(8)
C(18)	0.050(8)	0.117(9)	0.084(9)	0.003(7)	0.006(7)	0.031(8)
C(19)	0.024(8)	0.37(3)	0.25(2)	-0.08(1)	-0.04(1)	0.16(2)
C(20)	0.064(9)	0.18(1)	0.16(1)	-0.042(9)	-0.003(9)	0.00(1)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}

The general temperature factor expression:
$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(10)	1.45(1)	O(1)	C(17)	1.44(1)
O(2)	C(3)	1.402(9)	O(2)	H(5)	0.99
O(3)	C(18)	1.18(1)	O(4)	C(18)	1.32(1)
O(4)	C(19)	1.49(1)	C(1)	C(2)	1.51(1)
C(1)	H(1)	0.96	C(1)	H(2)	0.94
C(1)	H(3)	0.94	C(2)	C(3)	1.61(1)
C(2)	C(18)	1.51(1)	C(2)	H(4)	1.26
C(3)	C(4)	1.52(1)	C(3)	C(10)	1.53(1)
C(4)	C(5)	1.36(1)	C(4)	C(9)	1.38(1)
C(5)	C(6)	1.42(1)	C(5)	H(6)	0.98
C(6)	C(7)	1.32(1)	C(6)	H(7)	0.96
C(7)	C(8)	1.36(1)	C(7)	H(8)	1.02
C(8)	C(9)	1.47(1)	C(8)	H(9)	0.94
C(9)	H(10)	0.96	C(10)	C(11)	1.50(1)
C(10)	H(11)	1.00	C(11)	C(12)	1.37(1)
C(11)	C(16)	1.34(1)	C(12)	C(13)	1.42(2)
C(12)	H(19)	0.93	C(13)	C(14)	1.34(2)
C(13)	H(18)	0.92	C(14)	C(15)	1.40(2)
C(14)	H(17)	0.90	C(15)	C(16)	1.36(1)
C(15)	H(16)	0.95	C(16)	H(15)	0.95
C(17)	H(12)	0.93	C(17)	H(13)	0.94
C(17)	H(14)	0.97	C(19)	C(20)	1.34(2)
C(19)	H(20)	1.06	C(19)	H(21)	0.90
C(20)	H(22)	0.93	C(20)	H(23)	0.90

Table 3. Bond Lengths(Å) (continued)

atom	atom	distance	atom	atom	distance
C(20)	H(24)	0.97			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(10)	O(1)	C(17)	110.9(7)	C(3)	O(2)	H(5)	106.2
C(18)	O(4)	C(19)	114(1)	C(2)	C(1)	H(1)	108.8
C(2)	C(1)	H(2)	109.0	C(2)	C(1)	H(3)	108.5
H(1)	C(1)	H(2)	109.8	H(1)	C(1)	H(3)	109.3
H(2)	C(1)	H(3)	111.4	C(1)	C(2)	C(3)	112.3(7)
C(1)	C(2)	C(18)	108.2(9)	C(1)	C(2)	H(4)	109.8
C(3)	C(2)	C(18)	109.5(7)	C(3)	C(2)	H(4)	110.1
C(18)	C(2)	H(4)	106.7	O(2)	C(3)	C(2)	107.6(7)
O(2)	C(3)	C(4)	109.8(7)	O(2)	C(3)	C(10)	112.9(7)
C(2)	C(3)	C(4)	108.1(7)	C(2)	C(3)	C(10)	109.8(7)
C(4)	C(3)	C(10)	108.6(8)	C(3)	C(4)	C(5)	121.2(9)
C(3)	C(4)	C(9)	120.6(9)	C(5)	C(4)	C(9)	118.0(9)
C(4)	C(5)	C(6)	122(1)	C(4)	C(5)	H(6)	117.3
C(6)	C(5)	H(6)	119.7	C(5)	C(6)	C(7)	117(1)
C(5)	C(6)	H(7)	125.4	C(7)	C(6)	H(7)	117.0
C(6)	C(7)	C(8)	125(1)	C(6)	C(7)	H(8)	118.5
C(8)	C(7)	H(8)	116.3	C(7)	C(8)	C(9)	116(1)
C(7)	C(8)	H(9)	116.4	C(9)	C(8)	H(9)	127.1
C(4)	C(9)	C(8)	120.4(10)	C(4)	C(9)	H(10)	120.2
C(8)	C(9)	H(10)	119.3	O(1)	C(10)	C(3)	105.1(7)
O(1)	C(10)	C(11)	109.0(7)	O(1)	C(10)	H(11)	107.0
C(3)	C(10)	C(11)	114.8(7)	C(3)	C(10)	H(11)	109.8
C(11)	C(10)	H(11)	110.7	C(10)	C(11)	C(12)	118(1)
C(10)	C(11)	C(16)	124(1)	C(12)	C(11)	C(16)	117.1(10)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(11)	C(12)	C(13)	120(1)	C(11)	C(12)	H(19)	118.4
C(13)	C(12)	H(19)	121.2	C(12)	C(13)	C(14)	119(1)
C(12)	C(13)	H(18)	123.4	C(14)	C(13)	H(18)	116.9
C(13)	C(14)	C(15)	120(1)	C(13)	C(14)	H(17)	128.1
C(15)	C(14)	H(17)	110.8	C(14)	C(15)	C(16)	116(1)
C(14)	C(15)	H(16)	122.8	C(16)	C(15)	H(16)	119.9
C(11)	C(16)	C(15)	125(1)	C(11)	C(16)	H(15)	116.0
C(15)	C(16)	H(15)	118.8	O(1)	C(17)	H(12)	109.7
O(1)	C(17)	H(13)	108.4	O(1)	C(17)	H(14)	108.0
H(12)	C(17)	H(13)	112.0	H(12)	C(17)	H(14)	110.1
H(13)	C(17)	H(14)	108.6	O(3)	C(18)	O(4)	125(1)
O(3)	C(18)	C(2)	123(1)	O(4)	C(18)	C(2)	110(1)
O(4)	C(19)	C(20)	110(1)	O(4)	C(19)	H(20)	104.2
O(4)	C(19)	H(21)	114.7	C(20)	C(19)	H(20)	105.4
C(20)	C(19)	H(21)	117.2	H(20)	C(19)	H(21)	103.7
C(19)	C(20)	H(22)	109.3	C(19)	C(20)	H(23)	107.8
C(19)	C(20)	H(24)	99.5	H(22)	C(20)	H(23)	116.3
H(22)	C(20)	H(24)	109.7	H(23)	C(20)	H(24)	112.7

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(10)	C(3)	O(2)	-59.0(9)	O(1)	C(10)	C(3)	C(2)	61.1(8)
O(1)	C(10)	C(3)	C(4)	179.0(7)	O(1)	C(10)	C(11)	C(12)	-130.9(9)
O(1)	C(10)	C(11)	C(16)	44(1)	O(2)	C(3)	C(2)	C(1)	-63.9(9)
O(2)	C(3)	C(2)	C(18)	56(1)	O(2)	C(3)	C(4)	C(5)	-17(1)
O(2)	C(3)	C(4)	C(9)	168.6(8)	O(2)	C(3)	C(10)	C(11)	60(1)
O(3)	C(18)	O(4)	C(19)	15(1)	O(3)	C(18)	C(2)	C(1)	58(1)
O(3)	C(18)	C(2)	C(3)	-63(1)	O(4)	C(18)	C(2)	C(1)	-114(1)
O(4)	C(18)	C(2)	C(3)	123.0(9)	C(1)	C(2)	C(3)	C(4)	54(1)
C(1)	C(2)	C(3)	C(10)	172.9(8)	C(2)	C(3)	C(4)	C(5)	-134.4(9)
C(2)	C(3)	C(4)	C(9)	51(1)	C(2)	C(3)	C(10)	C(11)	-179.2(8)
C(2)	C(18)	O(4)	C(19)	-171(1)	C(3)	C(4)	C(5)	C(6)	-177.6(9)
C(3)	C(4)	C(9)	C(8)	177.4(8)	C(3)	C(10)	O(1)	C(17)	-157.3(7)
C(3)	C(10)	C(11)	C(12)	111.6(10)	C(3)	C(10)	C(11)	C(16)	-73(1)
C(4)	C(3)	C(2)	C(18)	174.9(9)	C(4)	C(3)	C(10)	C(11)	-61(1)
C(4)	C(5)	C(6)	C(7)	4(1)	C(4)	C(9)	C(8)	C(7)	-3(1)
C(5)	C(4)	C(3)	C(10)	106(1)	C(5)	C(4)	C(9)	C(8)	3(1)
C(5)	C(6)	C(7)	C(8)	-5(1)	C(6)	C(5)	C(4)	C(9)	-3(1)
C(6)	C(7)	C(8)	C(9)	4(1)	C(9)	C(4)	C(3)	C(10)	-67.6(10)
C(10)	C(3)	C(2)	C(18)	-66(1)	C(10)	C(11)	C(12)	C(13)	178.3(9)
C(10)	C(11)	C(16)	C(15)	-176.8(9)	C(11)	C(10)	O(1)	C(17)	79.2(9)
C(11)	C(12)	C(13)	C(14)	1(1)	C(11)	C(16)	C(15)	C(14)	-3(1)
C(12)	C(11)	C(16)	C(15)	-1(1)	C(12)	C(13)	C(14)	C(15)	-6(2)
C(13)	C(12)	C(11)	C(16)	2(1)	C(13)	C(14)	C(15)	C(16)	7(2)
C(18)	O(4)	C(19)	C(20)	-137(1)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	H(24)$^{1)}$	3.37	O(2)	H(4)$^{2)}$	2.65
O(2)	H(11)$^{2)}$	2.73	O(2)	H(12)$^{2)}$	2.92
O(2)	H(10)$^{2)}$	3.07	O(3)	H(22)$^{2)}$	2.83
O(3)	H(14)$^{3)}$	2.95	O(3)	H(20)$^{2)}$	3.23
O(3)	H(12)$^{2)}$	3.24	O(3)	H(20)$^{3)}$	3.26
O(4)	H(24)$^{4)}$	3.06	O(4)	H(22)$^{4)}$	3.50
C(1)	H(13)$^{5)}$	3.36	C(1)	H(21)$^{3)}$	3.42
C(1)	H(23)$^{3)}$	3.44	C(1)	H(12)$^{2)}$	3.47
C(2)	H(5)$^{6)}$	3.34	C(2)	H(15)$^{6)}$	3.55
C(5)	H(10)$^{2)}$	3.21	C(5)	H(19)$^{2)}$	3.25
C(5)	H(17)$^{5)}$	3.53	C(5)	H(11)$^{2)}$	3.56
C(6)	H(17)$^{5)}$	3.09	C(6)	H(8)$^{7)}$	3.34
C(6)	H(19)$^{2)}$	3.47	C(6)	C(7)$^{7)}$	3.56(1)
C(7)	H(9)$^{8)}$	3.27	C(7)	H(17)$^{5)}$	3.30
C(7)	H(7)$^{7)}$	3.35	C(8)	H(9)$^{8)}$	3.05
C(8)	H(6)$^{6)}$	3.49	C(8)	H(18)$^{5)}$	3.57
C(9)	H(6)$^{6)}$	2.92	C(9)	H(5)$^{6)}$	3.07
C(9)	H(16)$^{6)}$	3.58	C(10)	H(5)$^{6)}$	3.43
C(10)	H(6)$^{6)}$	3.57	C(12)	H(16)$^{9)}$	3.32
C(12)	H(8)$^{7)}$	3.40	C(12)	H(6)$^{6)}$	3.53
C(13)	H(8)$^{7)}$	2.92	C(13)	H(16)$^{9)}$	3.47
C(14)	H(8)$^{7)}$	3.24	C(14)	H(1)$^{10)}$	3.37
C(15)	H(10)$^{2)}$	3.23	C(15)	H(1)$^{10)}$	3.29
C(16)	H(10)$^{2)}$	3.01	C(16)	H(4)$^{2)}$	3.19

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(16)	H(23)$^{1)}$	3.35	C(17)	H(23)$^{11)}$	3.22
C(17)	H(22)$^{11)}$	3.25	C(17)	H(3)$^{6)}$	3.33
C(17)	H(2)$^{10)}$	3.43	C(17)	H(21)$^{1)}$	3.48
C(17)	H(20)$^{11)}$	3.55	C(17)	C(20)$^{11)}$	3.58(2)
C(19)	H(14)$^{3)}$	3.43	C(19)	H(2)$^{1)}$	3.47
C(20)	H(24)$^{4)}$	3.11	C(20)	H(14)$^{11)}$	3.17
C(20)	H(15)$^{3)}$	3.36	C(20)	H(2)$^{4)}$	3.40
C(20)	H(13)$^{11)}$	3.44	C(20)	H(12)$^{11)}$	3.59
H(1)	H(16)$^{6)}$	3.11	H(1)	H(13)$^{5)}$	3.25
H(1)	H(17)$^{5)}$	3.57	H(2)	H(13)$^{5)}$	2.67
H(2)	H(21)$^{3)}$	2.78	H(2)	H(22)$^{4)}$	2.97
H(2)	H(23)$^{3)}$	3.20	H(2)	H(23)$^{4)}$	3.27
H(2)	H(14)$^{5)}$	3.34	H(2)	H(20)$^{3)}$	3.57
H(3)	H(12)$^{2)}$	2.54	H(3)	H(23)$^{3)}$	2.89
H(3)	H(21)$^{3)}$	3.25	H(3)	H(13)$^{2)}$	3.41
H(3)	H(20)$^{3)}$	3.42	H(3)	H(16)$^{5)}$	3.57
H(4)	H(5)$^{6)}$	2.17	H(4)	H(15)$^{6)}$	2.42
H(4)	H(6)$^{6)}$	3.56	H(4)	H(16)$^{6)}$	3.59
H(5)	H(10)$^{2)}$	2.14	H(5)	H(11)$^{2)}$	2.52
H(5)	H(12)$^{2)}$	3.43	H(6)	H(10)$^{2)}$	2.36
H(6)	H(19)$^{2)}$	2.64	H(6)	H(11)$^{2)}$	2.66
H(6)	H(9)$^{2)}$	3.46	H(7)	H(7)$^{12)}$	3.08
H(7)	H(19)$^{2)}$	3.22	H(7)	H(17)$^{5)}$	3.25
H(7)	H(8)$^{7)}$	3.29	H(7)	H(18)$^{7)}$	3.49

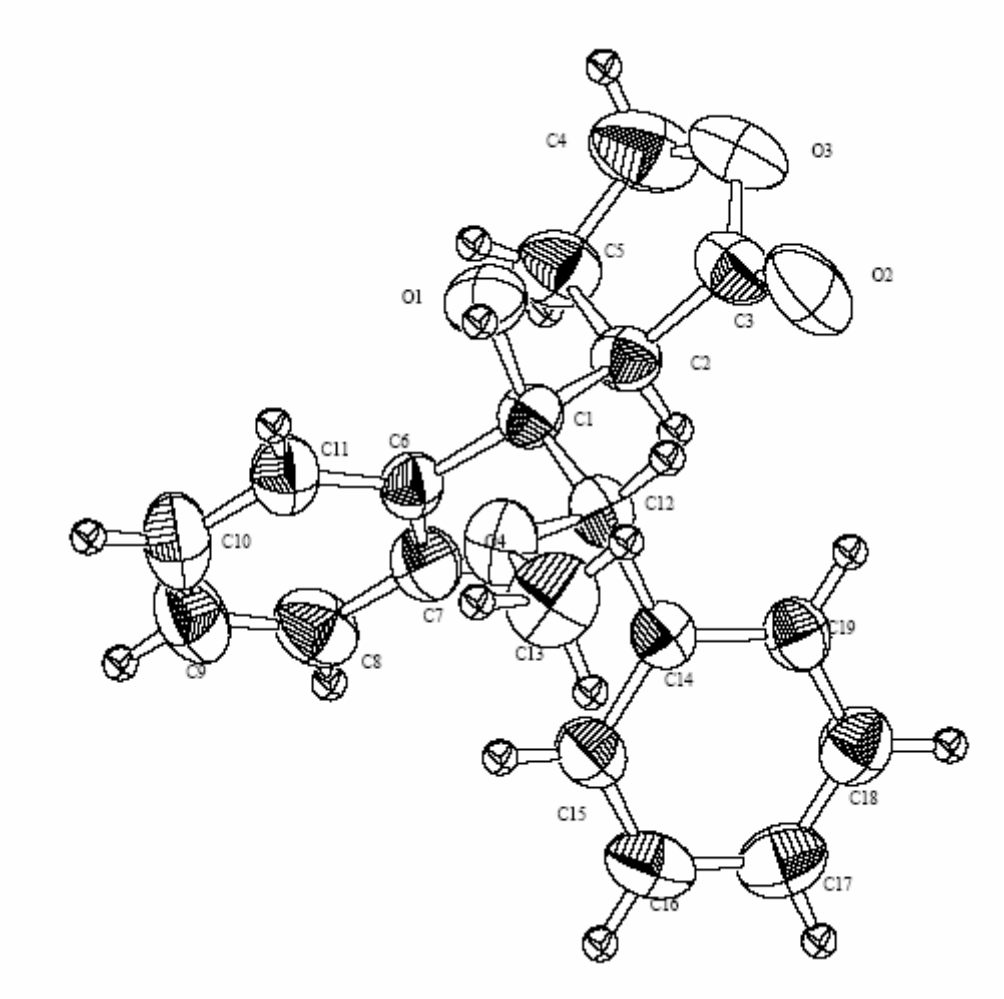
Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
H(7)	H(9)[2)]	3.52	H(8)	H(9)[8)]	2.73
H(8)	H(18)[7)]	2.91	H(8)	H(17)[7)]	3.53
H(8)	H(17)[5)]	3.56	H(8)	H(18)[5)]	3.59
H(9)	H(9)[8)]	2.30	H(9)	H(18)[5)]	3.59
H(10)	H(15)[6)]	2.84	H(10)	H(16)[6)]	3.17
H(12)	H(23)[11)]	3.12	H(12)	H(20)[11)]	3.29
H(12)	H(22)[11)]	3.47	H(13)	H(23)[11)]	2.98
H(13)	H(22)[11)]	3.15	H(13)	H(16)[9)]	3.23
H(13)	H(21)[1)]	3.42	H(14)	H(22)[11)]	2.67
H(14)	H(21)[1)]	2.79	H(14)	H(20)[11)]	3.02
H(14)	H(23)[11)]	3.06	H(14)	H(24)[1)]	3.34
H(15)	H(23)[1)]	2.75	H(15)	H(24)[1)]	3.23
H(16)	H(19)[13)]	3.18	H(16)	H(18)[13)]	3.47
H(16)	H(23)[1)]	3.57	H(17)	H(19)[13)]	3.36
H(17)	H(18)[14)]	3.43	H(17)	H(18)[13)]	3.54
H(20)	H(20)[11)]	3.24	H(21)	H(22)[2)]	3.02
H(22)	H(24)[4)]	2.84	H(24)	H(24)[4)]	2.55

Symmetry operations

(1)	-X+1/2,Y+1/2,-Z+1/2	(2)	X,-Y+1,Z-1/2
(3)	-X+1/2,Y-1/2,-Z+1/2	(4)	-X+1/2,-Y+1/2,-Z+1
(5)	X,Y-1,Z	(6)	X,-Y+1,Z+1/2
(7)	-X,-Y+1,-Z	(8)	-X,Y,-Z+1/2
(9)	X,-Y+2,Z+1/2	(10)	X,Y+1,Z
(11)	-X+1/2,-Y+3/2,-Z+1	(12)	-X,Y,-Z-1/2
(13)	X,-Y+2,Z-1/2	(14)	-X,-Y+2,-Z

X-ray Structure Report **3b**



Experimental

Data Collection

A colorless prismatic crystal of $C_{19}H_{20}O_4$ having approximate dimensions of 0.50 x 0.40 x 0.40 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $27.63 < 2\theta < 29.90^{\circ}$ corresponded to a primitive monoclinic cell with dimensions:

a = 9.238(2) Å
b = 13.245(2) Å β = 93.79(1)°
c = 13.418(2) Å
V = 1638.3(4) Å^3

For Z = 4 and F.W. = 312.36, the calculated density is 1.27 g/cm^3. The systematic absences of:

h0l: h ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/a$ (#14)

The data were collected at a temperature of 23 ± 1°C using the ω-2θ scan technique to a maximum 2θ value of 60.0°.Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.32° with a take-off angle of 6.0°. Scans of (1.47 + 0.30 tan θ)° were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 5241 reflections which were collected, 4771 were unique ($R_{int} = 0.018$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.9 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.96 to 1.00.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement[3] was based on 2882 observed reflections ($I > 0.80\sigma(I)$, $2\theta < 60.01$) and 288 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (Fo^2 - Fc^2) / \Sigma Fo^2 = 0.127$$

$$R_w = [(\Sigma\ w\ (Fo^2 - Fc^2)^2\ /\ \Sigma\ w\ (Fo^2)^2)]^{1/2} = 0.134$$

$$R1 = \Sigma\ ||Fo| - |Fc||\ /\ \Sigma\ |Fo| = 0.053 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight[4] was 1.20. The weighting scheme was based on counting statistics and included a factor (p = 0.065) to downweight the intense reflections. Plots of $\Sigma\ w\ (Fo^2 - Fc^2)^2$ versus Fo^2reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.22 and -0.32 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma\ w(F_o^2-F_c^2)^2$ where

$w = 1/[\sigma^2(Fo^2)] = [\sigma^2_C(Fo^2) + (p\ (Max(Fo^2,0)+2Fc^2)/3)^2]^{-1}$

$\sigma_C(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$[\Sigma\ w(|F_o|-|F_c|)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations

N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{19}H_{20}O_4$
Formula Weight	312.36
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.50 X 0.40 X 0.40 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (27.6 - 29.9°)
Omega Scan Peak Width at Half-height	0.32°
Lattice Parameters	a = 9.238(2) Å b = 13.245(2) Å c = 13.418(2) Å β = 93.79(1)° V = 1638.3(4) Å^3
Space Group	$P2_1/a$ (#14)
Z value	4
D_{calc}	1.266 g/cm^3
F_{000}	664.00
μ(MoKα)	0.88 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.22, 10.70, 35.51)
Temperature	23.0 °C
Collimator Size	1.0 mm
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm

Scan Type	ω-2θ
Scan Rate	16.0^{o}/min (in ω) (up to 5 scans)
Scan Width	(1.47 + 0.30 tan θ)^{o}
$2\theta_{max}$	60.0^{o}
No. of Reflections Measured	Total: 5241 Unique: 4771 (R_{int} = 0.018)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9649 - 0.9997)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	1/σ^2(Fo^2)
p-factor	0.0650
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I>0.80σ(I), 2θ < 60.01^{o})	2882
No. Variables	288
Reflection/Parameter Ratio	10.01
Residuals: R; Rw	0.127 ; 0.134
Residuals: R1	0.053
No. of Reflections to calc R1	2068
Goodness of Fit Indicator	1.20
Max Shift/Error in Final Cycle	0.003
Maximum peak in Final Diff. Map	0.22 e^-/Å^3
Minimum peak in Final Diff. Map	-0.32 e^-/Å^3

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.4701(2)	-0.0776(2)	0.1289(1)	4.80(4)
O(2)	0.7361(2)	-0.0665(2)	-0.0160(1)	6.86(5)
O(3)	0.6726(2)	-0.2272(1)	-0.0223(1)	6.58(5)
O(4)	0.5648(2)	0.0927(1)	0.2251(1)	4.64(4)
C(1)	0.6045(2)	-0.0786(2)	0.1885(1)	3.45(4)
C(2)	0.6966(2)	-0.1589(2)	0.1391(2)	3.76(5)
C(3)	0.7046(3)	-0.1416(2)	0.0271(2)	4.72(5)
C(4)	0.6460(5)	-0.3101(3)	0.0456(2)	7.50(9)
C(5)	0.6358(4)	-0.2657(2)	0.1454(2)	5.49(7)
C(6)	0.5783(2)	-0.1089(1)	0.2958(1)	3.53(4)
C(7)	0.6860(3)	-0.1539(2)	0.3587(2)	4.35(5)
C(8)	0.6591(3)	-0.1785(2)	0.4565(2)	5.28(6)
C(9)	0.5271(4)	-0.1569(2)	0.4931(2)	5.99(7)
C(10)	0.4209(3)	-0.1128(2)	0.4316(2)	6.08(7)
C(11)	0.4452(3)	-0.0891(2)	0.3340(2)	4.65(6)
C(12)	0.6711(2)	0.0292(2)	0.1854(2)	3.67(4)
C(13)	0.5851(4)	0.1974(2)	0.2052(3)	6.05(8)
C(14)	0.8190(2)	0.0408(1)	0.2392(1)	3.39(4)
C(15)	0.8351(3)	0.0658(2)	0.3401(2)	4.27(5)
C(16)	0.9718(3)	0.0761(2)	0.3877(2)	5.14(6)
C(17)	1.0939(3)	0.0618(2)	0.3357(2)	5.27(6)
C(18)	1.0798(3)	0.0387(2)	0.2356(2)	4.78(6)
C(19)	0.9440(2)	0.0287(2)	0.1878(2)	3.92(5)
H(1)	0.444(3)	-0.023(2)	0.126(2)	6.6(8)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(2)	0.797(2)	-0.156(1)	0.166(1)	3.4(4)
H(3)	0.737(5)	-0.361(4)	0.036(3)	15.4(8)
H(4)	0.556(4)	-0.332(3)	0.013(3)	11.0(8)
H(5)	0.686(4)	-0.305(3)	0.192(3)	9.1(8)
H(6)	0.539(4)	-0.261(3)	0.161(3)	9.3(8)
H(7)	0.777(2)	-0.171(2)	0.334(2)	4.1(5)
H(8)	0.737(3)	-0.208(2)	0.499(2)	6.2(6)
H(9)	0.513(3)	-0.173(2)	0.559(2)	8.1(7)
H(10)	0.328(3)	-0.096(2)	0.460(2)	6.7(6)
H(11)	0.369(3)	-0.054(2)	0.292(2)	5.4(6)
H(12)	0.674(2)	0.046(2)	0.116(2)	4.0(4)
H(13)	0.497(4)	0.230(3)	0.226(2)	8.3(7)
H(14)	0.675(3)	0.225(2)	0.242(2)	7.6(7)
H(15)	0.596(4)	0.206(2)	0.133(3)	8.4(8)
H(16)	0.749(3)	0.080(2)	0.375(2)	4.6(5)
H(17)	0.979(3)	0.096(2)	0.455(2)	6.9(6)
H(18)	1.187(3)	0.070(2)	0.371(2)	7.4(7)
H(19)	1.167(3)	0.032(2)	0.199(2)	6.5(6)
H(20)	0.935(2)	0.011(2)	0.117(2)	4.7(5)

$B_{eq} = 8/3\ PI^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0508(9)	0.065(1)	0.064(1)	0.0051(8)	-0.0091(8)	-0.0013(8)
O(2)	0.127(2)	0.086(1)	0.0495(10)	0.000(1)	0.019(1)	0.0036(9)
O(3)	0.110(1)	0.084(1)	0.0547(10)	0.008(1)	-0.0033(10)	-0.0239(10)
O(4)	0.0580(9)	0.0433(8)	0.076(1)	0.0117(7)	0.0138(8)	0.0035(7)
C(1)	0.039(1)	0.046(1)	0.045(1)	0.0034(9)	0.0002(8)	0.0001(8)
C(2)	0.048(1)	0.047(1)	0.047(1)	0.0031(9)	0.0005(9)	-0.0051(9)
C(3)	0.064(1)	0.067(2)	0.048(1)	0.013(1)	0.003(1)	-0.008(1)
C(4)	0.135(3)	0.071(2)	0.078(2)	-0.003(2)	-0.005(2)	-0.018(2)
C(5)	0.086(2)	0.048(1)	0.074(2)	-0.002(1)	0.004(2)	-0.012(1)
C(6)	0.047(1)	0.039(1)	0.049(1)	-0.0026(8)	0.0081(9)	-0.0012(8)
C(7)	0.059(1)	0.055(1)	0.052(1)	0.003(1)	0.007(1)	0.0029(10)
C(8)	0.090(2)	0.059(1)	0.051(1)	-0.002(1)	0.003(1)	0.006(1)
C(9)	0.108(2)	0.066(2)	0.057(2)	-0.005(2)	0.029(2)	0.006(1)
C(10)	0.083(2)	0.074(2)	0.079(2)	0.000(2)	0.038(2)	0.001(1)
C(11)	0.058(1)	0.056(1)	0.064(1)	0.001(1)	0.016(1)	-0.001(1)
C(12)	0.051(1)	0.045(1)	0.044(1)	0.0065(9)	0.0060(9)	0.0046(9)
C(13)	0.081(2)	0.046(1)	0.103(2)	0.014(1)	0.006(2)	0.005(1)
C(14)	0.051(1)	0.0359(10)	0.043(1)	0.0015(8)	0.0050(9)	0.0039(8)
C(15)	0.064(1)	0.055(1)	0.044(1)	-0.001(1)	0.007(1)	-0.0019(10)
C(16)	0.089(2)	0.057(1)	0.047(1)	-0.001(1)	-0.009(1)	-0.002(1)
C(17)	0.060(2)	0.059(1)	0.078(2)	0.001(1)	-0.012(1)	-0.001(1)
C(18)	0.051(1)	0.060(1)	0.071(2)	0.002(1)	0.006(1)	0.000(1)
C(19)	0.055(1)	0.048(1)	0.047(1)	0.0024(10)	0.008(1)	0.0042(9)

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.433(2)	O(1)	H(1)	0.77(3)
O(2)	C(3)	1.196(3)	O(3)	C(3)	1.337(3)
O(3)	C(4)	1.458(4)	O(4)	C(12)	1.423(2)
O(4)	C(13)	1.427(3)	C(1)	C(2)	1.540(3)
C(1)	C(6)	1.529(3)	C(1)	C(12)	1.556(3)
C(2)	C(3)	1.528(3)	C(2)	C(5)	1.527(3)
C(2)	H(2)	0.97(2)	C(4)	C(5)	1.471(4)
C(4)	H(3)	1.09(5)	C(4)	H(4)	0.96(4)
C(5)	H(5)	0.91(4)	C(5)	H(6)	0.93(4)
C(6)	C(7)	1.395(3)	C(6)	C(11)	1.388(3)
C(7)	C(8)	1.390(3)	C(7)	H(7)	0.95(2)
C(8)	C(9)	1.374(4)	C(8)	H(8)	0.97(3)
C(9)	C(10)	1.371(4)	C(9)	H(9)	0.93(3)
C(10)	C(11)	1.380(4)	C(10)	H(10)	0.98(3)
C(11)	H(11)	0.99(3)	C(12)	C(14)	1.510(3)
C(12)	H(12)	0.96(2)	C(13)	H(13)	0.98(3)
C(13)	H(14)	1.01(3)	C(13)	H(15)	0.99(3)
C(14)	C(15)	1.392(3)	C(14)	C(19)	1.393(3)
C(15)	C(16)	1.383(3)	C(15)	H(16)	0.97(2)
C(16)	C(17)	1.378(4)	C(16)	H(17)	0.94(3)
C(17)	C(18)	1.376(4)	C(17)	H(18)	0.96(3)
C(18)	C(19)	1.377(3)	C(18)	H(19)	0.97(3)
C(19)	H(20)	0.98(2)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	H(1)	106(2)	C(3)	O(3)	C(4)	111.7(2)
C(12)	O(4)	C(13)	113.7(2)	O(1)	C(1)	C(2)	104.4(2)
O(1)	C(1)	C(6)	110.1(2)	O(1)	C(1)	C(12)	107.8(2)
C(2)	C(1)	C(6)	110.5(2)	C(2)	C(1)	C(12)	113.1(2)
C(6)	C(1)	C(12)	110.8(2)	C(1)	C(2)	C(3)	112.6(2)
C(1)	C(2)	C(5)	113.6(2)	C(1)	C(2)	H(2)	110(1)
C(3)	C(2)	C(5)	103.6(2)	C(3)	C(2)	H(2)	104(1)
C(5)	C(2)	H(2)	111(1)	O(2)	C(3)	O(3)	121.3(2)
O(2)	C(3)	C(2)	129.0(2)	O(3)	C(3)	C(2)	109.7(2)
O(3)	C(4)	C(5)	106.9(2)	O(3)	C(4)	H(3)	103(2)
O(3)	C(4)	H(4)	96(2)	C(5)	C(4)	H(3)	116(2)
C(5)	C(4)	H(4)	115(2)	H(3)	C(4)	H(4)	114(3)
C(2)	C(5)	C(4)	105.9(2)	C(2)	C(5)	H(5)	113(2)
C(2)	C(5)	H(6)	108(2)	C(4)	C(5)	H(5)	109(2)
C(4)	C(5)	H(6)	110(2)	H(5)	C(5)	H(6)	109(3)
C(1)	C(6)	C(7)	122.1(2)	C(1)	C(6)	C(11)	119.8(2)
C(7)	C(6)	C(11)	118.1(2)	C(6)	C(7)	C(8)	120.4(2)
C(6)	C(7)	H(7)	120(1)	C(8)	C(7)	H(7)	119(1)
C(7)	C(8)	C(9)	120.5(3)	C(7)	C(8)	H(8)	118(1)
C(9)	C(8)	H(8)	120(1)	C(8)	C(9)	C(10)	119.3(2)
C(8)	C(9)	H(9)	118(1)	C(10)	C(9)	H(9)	121(1)
C(9)	C(10)	C(11)	120.9(3)	C(9)	C(10)	H(10)	117(1)
C(11)	C(10)	H(10)	121(1)	C(6)	C(11)	C(10)	120.7(3)
C(6)	C(11)	H(11)	119(1)	C(10)	C(11)	H(11)	119(1)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
O(4)	C(12)	C(1)	104.4(2)	O(4)	C(12)	C(14)	112.7(2)
O(4)	C(12)	H(12)	106(1)	C(1)	C(12)	C(14)	115.2(2)
C(1)	C(12)	H(12)	105(1)	C(14)	C(12)	H(12)	111(1)
O(4)	C(13)	H(13)	105(1)	O(4)	C(13)	H(14)	111(1)
O(4)	C(13)	H(15)	108(1)	H(13)	C(13)	H(14)	112(2)
H(13)	C(13)	H(15)	111(2)	H(14)	C(13)	H(15)	107(2)
C(12)	C(14)	C(15)	121.6(2)	C(12)	C(14)	C(19)	120.3(2)
C(15)	C(14)	C(19)	118.0(2)	C(14)	C(15)	C(16)	120.5(2)
C(14)	C(15)	H(16)	118(1)	C(16)	C(15)	H(16)	120(1)
C(15)	C(16)	C(17)	120.4(2)	C(15)	C(16)	H(17)	118(1)
C(17)	C(16)	H(17)	121(1)	C(16)	C(17)	C(18)	119.8(2)
C(16)	C(17)	H(18)	117(1)	C(18)	C(17)	H(18)	122(1)
C(17)	C(18)	C(19)	120.0(2)	C(17)	C(18)	H(19)	119(1)
C(19)	C(18)	H(19)	120(1)	C(14)	C(19)	C(18)	121.3(2)
C(14)	C(19)	H(20)	119(1)	C(18)	C(19)	H(20)	119(1)

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(1)	C(2)	C(3)	52.0(2)	O(1)	C(1)	C(2)	C(5)	-65.4(2)
O(1)	C(1)	C(6)	C(7)	154.3(2)	O(1)	C(1)	C(6)	C(11)	-27.6(3)
O(1)	C(1)	C(12)	O(4)	59.6(2)	O(1)	C(1)	C(12)	C(14)	-176.2(2)
O(2)	C(3)	O(3)	C(4)	176.6(3)	O(2)	C(3)	C(2)	C(1)	51.2(4)
O(2)	C(3)	C(2)	C(5)	174.4(3)	O(3)	C(3)	C(2)	C(1)	-130.1(2)
O(3)	C(3)	C(2)	C(5)	-6.9(3)	O(3)	C(4)	C(5)	C(2)	-14.6(4)
O(4)	C(12)	C(1)	C(2)	174.5(2)	O(4)	C(12)	C(1)	C(6)	-60.9(2)
O(4)	C(12)	C(14)	C(15)	30.8(3)	O(4)	C(12)	C(14)	C(19)	-147.8(2)
C(1)	C(2)	C(5)	C(4)	135.5(3)	C(1)	C(6)	C(7)	C(8)	178.6(2)
C(1)	C(6)	C(11)	C(10)	-177.8(2)	C(1)	C(12)	O(4)	C(13)	-164.3(2)
C(1)	C(12)	C(14)	C(15)	-88.9(2)	C(1)	C(12)	C(14)	C(19)	92.6(2)
C(2)	C(1)	C(6)	C(7)	39.5(3)	C(2)	C(1)	C(6)	C(11)	-142.4(2)
C(2)	C(1)	C(12)	C(14)	-61.4(2)	C(2)	C(3)	O(3)	C(4)	-2.2(3)
C(3)	O(3)	C(4)	C(5)	10.9(4)	C(3)	C(2)	C(1)	C(6)	170.3(2)
C(3)	C(2)	C(1)	C(12)	-64.9(2)	C(3)	C(2)	C(5)	C(4)	13.0(3)
C(5)	C(2)	C(1)	C(6)	52.9(2)	C(5)	C(2)	C(1)	C(12)	177.7(2)
C(6)	C(1)	C(12)	C(14)	63.3(2)	C(6)	C(7)	C(8)	C(9)	-1.4(4)
C(6)	C(11)	C(10)	C(9)	-0.4(4)	C(7)	C(6)	C(1)	C(12)	-86.6(2)
C(7)	C(6)	C(11)	C(10)	0.4(3)	C(7)	C(8)	C(9)	C(10)	1.4(4)
C(8)	C(7)	C(6)	C(11)	0.5(3)	C(8)	C(9)	C(10)	C(11)	-0.5(4)
C(11)	C(6)	C(1)	C(12)	91.5(2)	C(12)	C(14)	C(15)	C(16)	-179.9(2)
C(12)	C(14)	C(19)	C(18)	-179.9(2)	C(13)	O(4)	C(12)	C(14)	69.9(3)
C(14)	C(15)	C(16)	C(17)	0.1(3)	C(14)	C(19)	C(18)	C(17)	-0.5(3)
C(15)	C(14)	C(19)	C(18)	1.5(3)	C(15)	C(16)	C(17)	C(18)	1.0(4)

Table 5. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(16)	C(15)	C(14)	C(19)	-1.3(3)	C(16)	C(17)	C(18)	C(19)	-0.8(4)

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	H(3)$^{1)}$	2.55(5)	O(1)	O(2)$^{2)}$	3.030(3)
O(1)	H(5)$^{1)}$	3.22(4)	O(1)	H(19)$^{3)}$	3.34(3)
O(1)	C(4)$^{1)}$	3.461(5)	O(1)	H(12)$^{2)}$	3.49(2)
O(2)	H(1)$^{2)}$	2.46(3)	O(2)	H(19)$^{4)}$	2.71(3)
O(2)	H(3)$^{5)}$	2.75(5)	O(2)	H(4)$^{6)}$	3.25(4)
O(2)	H(20)$^{4)}$	3.49(2)	O(2)	C(18)$^{4)}$	3.517(3)
O(2)	C(4)$^{5)}$	3.596(4)	O(3)	H(15)$^{2)}$	2.82(3)
O(3)	H(15)$^{7)}$	2.82(3)	O(3)	H(13)$^{2)}$	3.06(3)
O(3)	C(13)$^{2)}$	3.327(4)	O(3)	H(14)$^{7)}$	3.41(3)
O(3)	C(13)$^{7)}$	3.572(4)	O(3)	H(12)$^{7)}$	3.59(2)
O(4)	H(9)$^{8)}$	3.21(3)	C(2)	H(6)$^{6)}$	3.33(4)
C(3)	H(1)$^{2)}$	3.24(3)	C(3)	H(4)$^{6)}$	3.29(4)
C(3)	H(15)$^{2)}$	3.50(3)	C(3)	H(15)$^{7)}$	3.55(3)
C(4)	H(20)$^{7)}$	3.27(2)	C(4)	H(12)$^{7)}$	3.41(2)
C(4)	H(15)$^{2)}$	3.45(3)	C(4)	H(20)$^{1)}$	3.47(2)
C(4)	H(15)$^{7)}$	3.49(3)	C(4)	H(19)$^{1)}$	3.58(3)
C(5)	H(2)$^{1)}$	3.33(2)	C(8)	H(18)$^{9)}$	3.00(3)
C(8)	H(10)$^{6)}$	3.36(3)	C(8)	H(17)$^{10)}$	3.49(3)
C(9)	H(8)$^{1)}$	3.23(3)	C(9)	H(18)$^{9)}$	3.32(3)
C(9)	H(17)$^{10)}$	3.34(3)	C(9)	H(16)$^{8)}$	3.35(2)
C(10)	H(8)$^{1)}$	3.09(3)	C(10)	H(16)$^{8)}$	3.15(2)
C(10)	H(18)$^{3)}$	3.31(3)	C(10)	H(7)$^{1)}$	3.39(2)
C(11)	H(18)$^{3)}$	3.25(3)	C(11)	H(5)$^{1)}$	3.28(4)
C(11)	H(19)$^{3)}$	3.44(3)	C(11)	H(7)$^{1)}$	3.54(2)

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(12)	H(3)$^{5)}$	3.46(5)	C(13)	H(9)$^{8)}$	3.36(3)
C(13)	C(18)$^{11)}$	3.519(4)	C(13)	H(4)$^{2)}$	3.59(4)
C(14)	H(13)$^{12)}$	3.46(4)	C(15)	H(10)$^{8)}$	3.19(3)
C(15)	H(13)$^{12)}$	3.49(4)	C(16)	H(17)$^{9)}$	3.12(3)
C(16)	H(13)$^{12)}$	3.38(3)	C(16)	H(9)$^{13)}$	3.40(3)
C(16)	H(8)$^{9)}$	3.48(3)	C(16)	H(10)$^{8)}$	3.56(3)
C(17)	H(11)$^{14)}$	3.06(3)	C(17)	H(14)$^{12)}$	3.20(3)
C(17)	H(13)$^{12)}$	3.22(3)	C(17)	H(8)$^{9)}$	3.26(3)
C(17)	H(10)$^{14)}$	3.37(3)	C(18)	H(11)$^{14)}$	2.99(3)
C(18)	H(13)$^{12)}$	3.15(3)	C(18)	H(14)$^{12)}$	3.25(3)
C(18)	H(5)$^{6)}$	3.32(4)	C(19)	H(4)$^{5)}$	3.26(4)
C(19)	H(13)$^{12)}$	3.26(3)	H(1)	H(3)$^{1)}$	2.69(6)
H(1)	H(19)$^{3)}$	2.89(4)	H(1)	H(12)$^{2)}$	3.37(4)
H(1)	H(5)$^{1)}$	3.46(5)	H(2)	H(6)$^{6)}$	2.50(4)
H(2)	H(4)$^{6)}$	3.27(4)	H(3)	H(12)$^{7)}$	2.57(5)
H(3)	H(15)$^{7)}$	2.95(6)	H(3)	H(20)$^{7)}$	3.02(5)
H(3)	H(19)$^{1)}$	3.24(6)	H(3)	H(6)$^{6)}$	3.55(6)
H(4)	H(20)$^{7)}$	2.71(5)	H(4)	H(15)$^{2)}$	2.86(5)
H(4)	H(20)$^{1)}$	3.01(5)	H(4)	H(13)$^{2)}$	3.47(5)
H(4)	H(12)$^{7)}$	3.52(4)	H(5)	H(11)$^{6)}$	2.82(4)
H(5)	H(19)$^{1)}$	3.02(5)	H(5)	H(6)$^{6)}$	3.43(5)
H(6)	H(20)$^{1)}$	3.48(4)	H(6)	H(7)$^{1)}$	3.58(4)
H(7)	H(10)$^{6)}$	3.53(3)	H(8)	H(18)$^{9)}$	2.59(4)
H(8)	H(10)$^{6)}$	2.78(4)	H(8)	H(17)$^{9)}$	3.04(4)

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
H(8)	H(9)$^{6)}$	3.06(4)	H(8)	H(16)$^{10)}$	3.27(4)
H(8)	H(17)$^{10)}$	3.35(4)	H(9)	H(16)$^{8)}$	2.90(4)
H(9)	H(13)$^{8)}$	2.99(4)	H(9)	H(17)$^{10)}$	3.07(4)
H(9)	H(18)$^{9)}$	3.18(4)	H(9)	H(14)$^{8)}$	3.35(4)
H(10)	H(16)$^{8)}$	2.38(4)	H(10)	H(18)$^{3)}$	2.79(4)
H(10)	H(17)$^{8)}$	3.13(4)	H(11)	H(19)$^{3)}$	2.46(4)
H(11)	H(18)$^{3)}$	2.62(4)	H(13)	H(14)$^{11)}$	3.06(4)
H(13)	H(19)$^{11)}$	3.55(4)	H(14)	H(18)$^{11)}$	3.23(4)
H(14)	H(19)$^{11)}$	3.27(4)	H(17)	H(17)$^{9)}$	2.84(5)
H(20)	H(20)$^{4)}$	3.44(5)			

Symmetry operations

(1)	X-1/2,-Y-1/2,Z	(2)	-X+1,-Y,-Z
(3)	X-1,Y,Z	(4)	-X+2,-Y,-Z
(5)	-X+3/2,Y+1/2,-Z	(6)	X+1/2,-Y-1/2,Z
(7)	-X+3/2,Y-1/2,-Z	(8)	-X+1,-Y,-Z+1
(9)	-X+2,-Y,-Z+1	(10)	-X+3/2,Y-1/2,-Z+1
(11)	X-1/2,-Y+1/2,Z	(12)	X+1/2,-Y+1/2,Z
(13)	-X+3/2,Y+1/2,-Z+1	(14)	X+1,Y,Z

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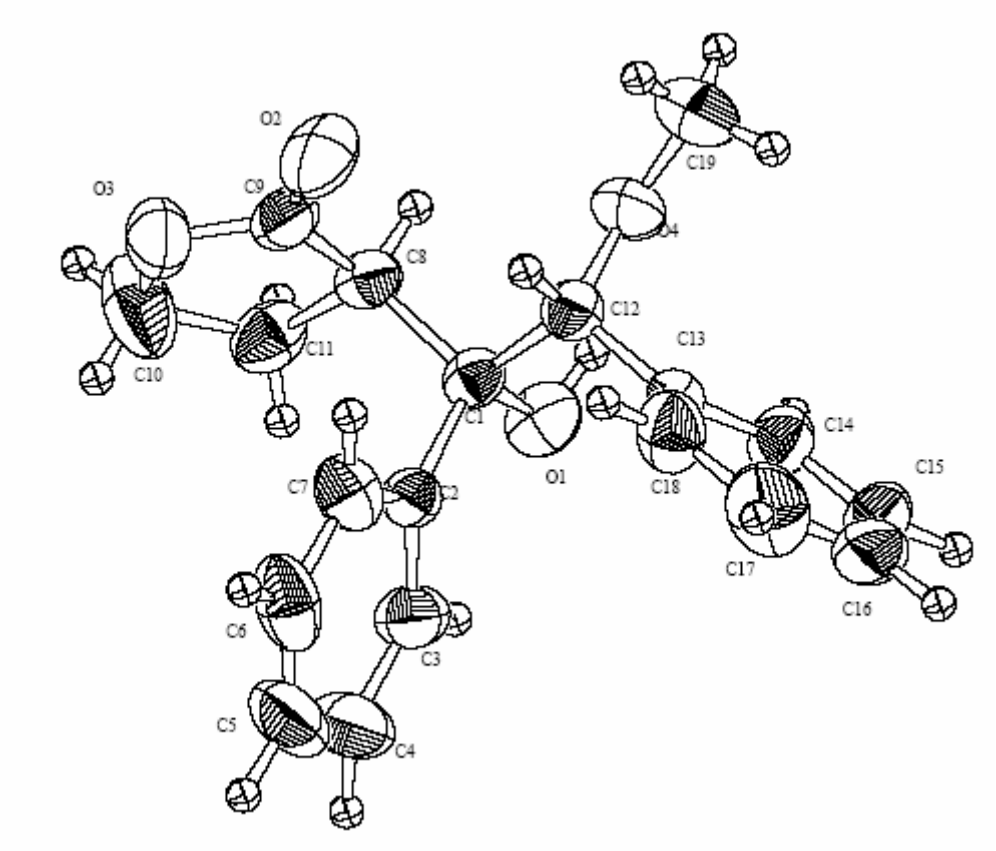

Experimental

Data Collection

A colorless prismatic crystal of $C_{19}H_{20}O_4$ having approximate dimensions of 0.50 x 0.50 x 0.50 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $27.12 < 2\theta < 29.38^{\circ}$ corresponded to a primitive orthorhombic cell with dimensions:

a = 16.779(3) Å
b = 23.256(3) Å
c = 8.243(3) Å
V = 3216(1) $Å^3$

For Z = 8 and F.W. = 312.36, the calculated density is 1.29 g/cm^3. The systematic absences of:

0kl: k ± 2n
h0l: l ± 2n
hk0: h ± 2n

uniquely determine the space group to be:

Pbca (#61)

The data were collected at a temperature of 23 ± 1°C using the ω-2θ scan technique to a maximum 2θ value of 60.0°.Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.32° with a take-off angle of 6.0°. Scans of $(1.73 + 0.30 \tan \theta)^{\circ}$ were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 5270 reflections which were collected, 4696 were unique ($R_{int} = 0.000$). The intensities of three representative reflection were measured after every 150 reflections. Over the course of data collection, the standards increased by 0.9%. A linear correction factor was applied to the data to account for this phenomenon.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.9 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.97 to 1.00.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were included in fixed positions. The final cycle of full-matrix least-squares refinement[3] was based on 2894 observed reflections ($I > 0.50\sigma(I)$, $2\theta < 60.00$) and 280 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (Fo^2 - Fc^2) / \Sigma Fo^2 = 0.127$$

$$R_w = [(\Sigma\ w\ (Fo^2 - Fc^2)^2 / \Sigma\ w\ (Fo^2)^2)]^{1/2} = 0.150$$

$$R1 = \Sigma\ ||Fo| - |Fc|| / \Sigma\ |Fo| = 0.056 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight[4] was 1.24. The weighting scheme was based on counting statistics and included a factor (p = 0.063) to downweight the intense reflections. Plots of $\Sigma\ w\ (Fo^2 - Fc^2)^2$ versus Fo^2reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.26 and -0.33 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma\ w(F_O^2-F_C^2)^2$ where

$w = 1/[\sigma^2(Fo^2)] = [\sigma^2_C(Fo^2) + (p\ (Max(Fo^2,0)+2Fc^2)/3)^2]^{-1}$

$\sigma_C(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$[\Sigma\ w(|F_O|-|F_C|)^2/(N_O-N_V)]^{1/2}$

where: N_O = number of observations

N_V = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{19}H_{20}O_4$
Formula Weight	312.36
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.50 X 0.50 X 0.50 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (27.1 - 29.4°)
Omega Scan Peak Width at Half-height	0.32°
Lattice Parameters	a = 16.779(3) Å b = 23.256(3) Å c = 8.243(3) Å V = 3216(1) Å^3
Space Group	Pbca (#61)
Z value	8
D_{calc}	1.290 g/cm^3
F_{000}	1328.00
μ(MoKα)	0.90 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.22, 10.70, 35.51)
Temperature	23.0 °C
Collimator Size	1.0 mm
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm

Scan Type	ω-2θ
Scan Rate	16.0^{o}/min (in ω) (up to 5 scans)
Scan Width	(1.73 + 0.30 tan θ)^{o}
$2\theta_{max}$	60.0^{o}
No. of Reflections Measured	Total: 5270 Unique: 4696 (R_{int} = 0.000)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9683 - 0.9994) Decay (0.89% increase)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	1/σ^2(Fo^2)
p-factor	0.0630
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I>0.50σ(I), 2θ < 60.00^{o})	2894
No. Variables	280
Reflection/Parameter Ratio	10.34
Residuals: R; Rw	0.127 ; 0.150
Residuals: R1	0.056
No. of Reflections to calc R1	1747
Goodness of Fit Indicator	1.24
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.26 e^-/Å^3
Minimum peak in Final Diff. Map	-0.33 e^-/Å^3

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.3756(1)	0.07819(8)	0.4386(2)	4.39(5)
O(2)	0.4622(1)	0.25535(9)	0.3351(3)	5.91(5)
O(3)	0.5656(1)	0.22082(9)	0.4683(3)	5.28(5)
O(4)	0.2897(1)	0.17247(9)	0.3750(2)	4.91(5)
C(1)	0.4122(1)	0.12417(10)	0.3508(3)	3.16(5)
C(2)	0.4755(1)	0.09842(10)	0.2420(3)	3.21(5)
C(3)	0.4965(2)	0.0406(1)	0.2575(4)	4.60(7)
C(4)	0.5563(2)	0.0169(2)	0.1643(5)	6.42(10)
C(5)	0.5958(2)	0.0498(2)	0.0553(5)	6.6(1)
C(6)	0.5763(2)	0.1069(2)	0.0368(4)	5.72(9)
C(7)	0.5162(2)	0.1310(1)	0.1299(3)	4.30(7)
C(8)	0.4499(2)	0.1643(1)	0.4817(3)	3.53(6)
C(9)	0.4902(2)	0.2176(1)	0.4174(4)	4.09(6)
C(10)	0.5849(2)	0.1737(2)	0.5738(5)	7.4(1)
C(11)	0.5138(2)	0.1361(1)	0.5857(4)	4.63(7)
C(12)	0.3465(1)	0.1561(1)	0.2545(3)	3.52(5)
C(13)	0.3084(1)	0.12271(10)	0.1190(3)	3.26(5)
C(14)	0.2536(2)	0.0792(1)	0.1506(4)	4.40(7)
C(15)	0.2171(2)	0.0497(1)	0.0269(5)	5.48(9)
C(16)	0.2344(2)	0.0628(2)	-0.1307(5)	6.12(10)
C(17)	0.2879(2)	0.1055(2)	-0.1648(4)	6.8(1)
C(18)	0.3246(2)	0.1355(2)	-0.0398(4)	4.94(8)
C(19)	0.2345(2)	0.2151(2)	0.3221(7)	7.1(1)
H(1)	0.334(2)	0.095(2)	0.473(5)	6.9(9)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(2)	0.406(2)	0.178(1)	0.546(3)	3.5(5)
H(5)	0.497(2)	0.133(2)	0.692(5)	8.3(8)
H(6)	0.523(2)	0.100(2)	0.561(4)	6.1(7)
H(7)	0.469(2)	0.019(1)	0.336(4)	5.5(7)
H(8)	0.574(2)	-0.025(2)	0.182(5)	9.4(9)
H(9)	0.640(2)	0.033(2)	-0.009(4)	8.0(8)
H(10)	0.602(2)	0.133(1)	-0.033(4)	6.6(8)
H(11)	0.502(1)	0.169(1)	0.115(3)	3.7(6)
H(12)	0.369(1)	0.1900(9)	0.210(3)	2.2(4)
H(13)	0.237(2)	0.071(1)	0.259(4)	6.2(7)
H(14)	0.176(2)	0.018(1)	0.048(4)	6.7(7)
H(15)	0.208(2)	0.043(1)	-0.216(4)	6.9(8)
H(16)	0.300(2)	0.122(2)	-0.267(5)	8.3(8)
H(17)	0.360(2)	0.164(1)	-0.062(3)	3.5(5)
H(18)	0.264(3)	0.248(2)	0.280(5)	8.9(9)
H(19)	0.206(3)	0.225(2)	0.410(6)	9.9(9)
H(20)	0.201(3)	0.201(2)	0.235(6)	10.2(9)
H(21)	0.6249	0.1566	0.5236	10.2
H(22)	0.6075	0.1855	0.6776	14.6

$B_{eq} = 8/3\ PI^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.067(1)	0.050(1)	0.050(1)	-0.0138(10)	0.012(1)	0.0080(9)
O(2)	0.085(1)	0.044(1)	0.096(2)	-0.009(1)	-0.027(1)	0.014(1)
O(3)	0.049(1)	0.060(1)	0.091(2)	-0.0074(9)	-0.006(1)	0.007(1)
O(4)	0.053(1)	0.068(1)	0.066(1)	0.0126(9)	0.007(1)	-0.022(1)
C(1)	0.044(1)	0.037(1)	0.039(1)	-0.005(1)	0.004(1)	0.001(1)
C(2)	0.041(1)	0.044(1)	0.037(1)	0.003(1)	-0.004(1)	-0.005(1)
C(3)	0.065(2)	0.048(2)	0.062(2)	0.007(1)	-0.007(2)	-0.008(2)
C(4)	0.074(2)	0.080(2)	0.090(3)	0.029(2)	-0.004(2)	-0.026(2)
C(5)	0.054(2)	0.118(3)	0.078(2)	0.030(2)	-0.002(2)	-0.033(3)
C(6)	0.044(2)	0.127(3)	0.046(2)	-0.003(2)	0.004(1)	0.002(2)
C(7)	0.048(2)	0.066(2)	0.049(2)	0.004(1)	0.001(1)	0.008(2)
C(8)	0.051(1)	0.040(1)	0.043(1)	-0.001(1)	0.004(1)	-0.005(1)
C(9)	0.058(2)	0.040(1)	0.058(2)	-0.002(1)	-0.008(1)	-0.007(1)
C(10)	0.053(2)	0.089(3)	0.139(3)	0.006(2)	-0.013(2)	0.039(3)
C(11)	0.075(2)	0.053(2)	0.049(2)	-0.003(2)	-0.015(2)	-0.001(1)
C(12)	0.043(1)	0.039(1)	0.052(1)	-0.003(1)	0.005(1)	-0.002(1)
C(13)	0.034(1)	0.040(1)	0.050(2)	0.0023(10)	0.002(1)	-0.006(1)
C(14)	0.054(2)	0.048(1)	0.066(2)	-0.007(1)	-0.005(2)	0.002(2)
C(15)	0.064(2)	0.047(2)	0.097(3)	-0.004(2)	-0.021(2)	-0.012(2)
C(16)	0.068(2)	0.083(2)	0.081(3)	0.017(2)	-0.021(2)	-0.043(2)
C(17)	0.072(2)	0.136(3)	0.051(2)	0.002(2)	0.009(2)	-0.022(2)
C(18)	0.052(2)	0.083(2)	0.053(2)	-0.010(2)	0.007(1)	-0.003(2)
C(19)	0.066(2)	0.084(3)	0.121(4)	0.028(2)	-0.003(2)	-0.031(3)

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.430(3)	O(1)	H(1)	0.85(4)
O(2)	C(9)	1.204(3)	O(3)	C(9)	1.335(3)
O(3)	C(10)	1.436(4)	O(4)	C(12)	1.428(3)
O(4)	C(19)	1.425(4)	C(1)	C(2)	1.513(3)
C(1)	C(8)	1.561(4)	C(1)	C(12)	1.549(4)
C(2)	C(3)	1.396(4)	C(2)	C(7)	1.377(4)
C(3)	C(4)	1.379(5)	C(3)	H(7)	0.95(3)
C(4)	C(5)	1.353(6)	C(4)	H(8)	1.03(4)
C(5)	C(6)	1.377(6)	C(5)	H(9)	0.99(4)
C(6)	C(7)	1.385(4)	C(6)	H(10)	0.93(3)
C(7)	H(11)	0.93(3)	C(8)	C(9)	1.509(4)
C(8)	C(11)	1.521(4)	C(8)	H(2)	0.96(3)
C(10)	C(11)	1.482(5)	C(10)	H(21)	0.88
C(10)	H(22)	0.98	C(11)	H(5)	0.92(4)
C(11)	H(6)	0.89(3)	C(12)	C(13)	1.503(4)
C(12)	H(12)	0.95(2)	C(13)	C(14)	1.391(4)
C(13)	C(18)	1.370(4)	C(14)	C(15)	1.374(4)
C(14)	H(13)	0.96(3)	C(15)	C(16)	1.366(5)
C(15)	H(14)	1.02(4)	C(16)	C(17)	1.367(5)
C(16)	H(15)	0.96(4)	C(17)	C(18)	1.388(5)
C(17)	H(16)	0.94(4)	C(18)	H(17)	0.91(3)
C(19)	H(18)	0.98(4)	C(19)	H(19)	0.90(5)
C(19)	H(20)	0.97(5)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	H(1)	100(2)	C(9)	O(3)	C(10)	111.2(2)
C(12)	O(4)	C(19)	113.9(3)	O(1)	C(1)	C(2)	107.8(2)
O(1)	C(1)	C(8)	105.7(2)	O(1)	C(1)	C(12)	108.2(2)
C(2)	C(1)	C(8)	111.2(2)	C(2)	C(1)	C(12)	112.7(2)
C(8)	C(1)	C(12)	110.9(2)	C(1)	C(2)	C(3)	120.3(2)
C(1)	C(2)	C(7)	121.9(2)	C(3)	C(2)	C(7)	117.8(3)
C(2)	C(3)	C(4)	121.2(3)	C(2)	C(3)	H(7)	117(1)
C(4)	C(3)	H(7)	121(1)	C(3)	C(4)	C(5)	120.1(4)
C(3)	C(4)	H(8)	120(2)	C(5)	C(4)	H(8)	119(2)
C(4)	C(5)	C(6)	120.1(3)	C(4)	C(5)	H(9)	120(2)
C(6)	C(5)	H(9)	119(2)	C(5)	C(6)	C(7)	120.1(4)
C(5)	C(6)	H(10)	124(2)	C(7)	C(6)	H(10)	114(2)
C(2)	C(7)	C(6)	120.7(3)	C(2)	C(7)	H(11)	119(1)
C(6)	C(7)	H(11)	120(1)	C(1)	C(8)	C(9)	115.5(2)
C(1)	C(8)	C(11)	114.7(2)	C(1)	C(8)	H(2)	106(1)
C(9)	C(8)	C(11)	103.7(2)	C(9)	C(8)	H(2)	104(1)
C(11)	C(8)	H(2)	111(1)	O(2)	C(9)	O(3)	120.4(2)
O(2)	C(9)	C(8)	128.5(3)	O(3)	C(9)	C(8)	111.1(2)
O(3)	C(10)	C(11)	108.1(3)	O(3)	C(10)	H(21)	103.3
O(3)	C(10)	H(22)	113.7	C(11)	C(10)	H(21)	112.2
C(11)	C(10)	H(22)	114.9	H(21)	C(10)	H(22)	104.0
C(8)	C(11)	C(10)	106.0(3)	C(8)	C(11)	H(5)	110(2)
C(8)	C(11)	H(6)	113(2)	C(10)	C(11)	H(5)	110(2)
C(10)	C(11)	H(6)	114(2)	H(5)	C(11)	H(6)	100(3)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
O(4)	C(12)	C(1)	104.2(2)	O(4)	C(12)	C(13)	111.8(2)
O(4)	C(12)	H(12)	108(1)	C(1)	C(12)	C(13)	115.8(2)
C(1)	C(12)	H(12)	108(1)	C(13)	C(12)	H(12)	107(1)
C(12)	C(13)	C(14)	121.2(2)	C(12)	C(13)	C(18)	120.9(2)
C(14)	C(13)	C(18)	117.9(3)	C(13)	C(14)	C(15)	121.3(3)
C(13)	C(14)	H(13)	120(1)	C(15)	C(14)	H(13)	117(1)
C(14)	C(15)	C(16)	119.9(3)	C(14)	C(15)	H(14)	122(1)
C(16)	C(15)	H(14)	117(1)	C(15)	C(16)	C(17)	119.8(3)
C(15)	C(16)	H(15)	119(2)	C(17)	C(16)	H(15)	120(2)
C(16)	C(17)	C(18)	120.3(4)	C(16)	C(17)	H(16)	127(2)
C(18)	C(17)	H(16)	111(2)	C(13)	C(18)	C(17)	120.8(3)
C(13)	C(18)	H(17)	118(1)	C(17)	C(18)	H(17)	120(1)
O(4)	C(19)	H(18)	109(2)	O(4)	C(19)	H(19)	105(2)
O(4)	C(19)	H(20)	111(2)	H(18)	C(19)	H(19)	111(3)
H(18)	C(19)	H(20)	107(3)	H(19)	C(19)	H(20)	111(3)

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(1)	C(2)	C(3)	-9.0(3)	O(1)	C(1)	C(2)	C(7)	173.6(2)
O(1)	C(1)	C(8)	C(9)	-178.1(2)	O(1)	C(1)	C(8)	C(11)	61.4(3)
O(1)	C(1)	C(12)	O(4)	55.9(2)	O(1)	C(1)	C(12)	C(13)	-67.3(3)
O(2)	C(9)	O(3)	C(10)	175.8(3)	O(2)	C(9)	C(8)	C(1)	57.5(4)
O(2)	C(9)	C(8)	C(11)	-176.2(3)	O(3)	C(9)	C(8)	C(1)	-124.8(3)
O(3)	C(9)	C(8)	C(11)	1.5(3)	O(3)	C(10)	C(11)	C(8)	-0.8(4)
O(4)	C(12)	C(1)	C(2)	174.9(2)	O(4)	C(12)	C(1)	C(8)	-59.7(2)
O(4)	C(12)	C(13)	C(14)	-44.2(3)	O(4)	C(12)	C(13)	C(18)	133.6(3)
C(1)	C(2)	C(3)	C(4)	-177.2(3)	C(1)	C(2)	C(7)	C(6)	177.1(3)
C(1)	C(8)	C(11)	C(10)	126.4(3)	C(1)	C(12)	O(4)	C(19)	164.5(3)
C(1)	C(12)	C(13)	C(14)	74.9(3)	C(1)	C(12)	C(13)	C(18)	-107.3(3)
C(2)	C(1)	C(8)	C(9)	65.2(3)	C(2)	C(1)	C(8)	C(11)	-55.3(3)
C(2)	C(1)	C(12)	C(13)	51.7(3)	C(2)	C(3)	C(4)	C(5)	-0.1(5)
C(2)	C(7)	C(6)	C(5)	0.2(5)	C(3)	C(2)	C(1)	C(8)	106.4(3)
C(3)	C(2)	C(1)	C(12)	-128.4(2)	C(3)	C(2)	C(7)	C(6)	-0.4(4)
C(3)	C(4)	C(5)	C(6)	-0.1(6)	C(4)	C(3)	C(2)	C(7)	0.3(4)
C(4)	C(5)	C(6)	C(7)	0.1(5)	C(7)	C(2)	C(1)	C(8)	-71.0(3)
C(7)	C(2)	C(1)	C(12)	54.2(3)	C(8)	C(1)	C(12)	C(13)	177.1(2)
C(8)	C(9)	O(3)	C(10)	-2.1(4)	C(9)	O(3)	C(10)	C(11)	1.8(4)
C(9)	C(8)	C(1)	C(12)	-61.0(3)	C(9)	C(8)	C(11)	C(10)	-0.4(3)
C(11)	C(8)	C(1)	C(12)	178.5(2)	C(12)	C(13)	C(14)	C(15)	178.2(3)
C(12)	C(13)	C(18)	C(17)	-178.4(3)	C(13)	C(12)	O(4)	C(19)	-69.8(3)
C(13)	C(14)	C(15)	C(16)	0.1(5)	C(13)	C(18)	C(17)	C(16)	0.4(5)
C(14)	C(13)	C(18)	C(17)	-0.6(5)	C(14)	C(15)	C(16)	C(17)	-0.3(5)

Table 5. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(15)	C(14)	C(13)	C(18)	0.3(4)	C(15)	C(16)	C(17)	C(18)	0.0(6)

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	H(14)$^{1)}$	2.57(4)	O(1)	H(16)$^{2)}$	2.92(4)
O(1)	H(15)$^{1)}$	3.39(3)	O(1)	C(15)$^{1)}$	3.434(4)
O(1)	H(8)$^{3)}$	3.47(4)	O(2)	H(17)$^{4)}$	2.68(3)
O(2)	H(5)$^{5)}$	2.92(4)	O(2)	H(11)$^{4)}$	2.97(3)
O(2)	H(2)$^{5)}$	2.99(2)	O(2)	H(22)$^{5)}$	3.08
O(2)	C(11)$^{5)}$	3.369(4)	O(2)	C(10)$^{5)}$	3.406(5)
O(2)	C(8)$^{5)}$	3.467(4)	O(2)	O(3)$^{5)}$	3.530(3)
O(2)	C(9)$^{5)}$	3.532(4)	O(2)	C(18)$^{4)}$	3.583(4)
O(3)	H(19)$^{6)}$	2.86(5)	O(3)	H(20)$^{7)}$	2.86(5)
O(3)	H(11)$^{4)}$	3.02(3)	O(3)	H(22)$^{5)}$	3.31
O(3)	H(10)$^{4)}$	3.47(3)	O(4)	H(16)$^{2)}$	3.18(4)
O(4)	H(10)$^{8)}$	3.53(3)	C(3)	H(14)$^{7)}$	3.45(3)
C(3)	H(9)$^{9)}$	3.52(4)	C(4)	H(14)$^{7)}$	3.11(3)
C(4)	H(13)$^{7)}$	3.34(3)	C(4)	C(4)$^{9)}$	3.395(8)
C(4)	C(5)$^{9)}$	3.493(5)	C(5)	H(13)$^{7)}$	2.86(3)
C(5)	H(15)$^{10)}$	3.37(4)	C(5)	H(8)$^{9)}$	3.50(4)
C(6)	H(5)$^{11)}$	3.20(4)	C(6)	H(13)$^{7)}$	3.29(3)
C(6)	H(22)$^{11)}$	3.52	C(6)	H(19)$^{7)}$	3.52(4)
C(6)	H(20)$^{7)}$	3.57(4)	C(8)	H(16)$^{2)}$	3.41(4)
C(9)	H(11)$^{4)}$	3.10(3)	C(9)	H(17)$^{4)}$	3.52(3)
C(9)	H(22)$^{5)}$	3.58	C(10)	H(19)$^{6)}$	3.12(5)
C(10)	H(20)$^{7)}$	3.27(5)	C(10)	H(10)$^{2)}$	3.39(3)
C(11)	H(10)$^{2)}$	3.48(3)	C(11)	H(8)$^{3)}$	3.54(4)
C(13)	H(21)$^{8)}$	3.39	C(14)	H(15)$^{1)}$	3.10(3)

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(14)	H(21)$^{8)}$	3.16	C(14)	H(9)$^{9)}$	3.38(4)
C(15)	H(21)$^{8)}$	2.96	C(15)	H(9)$^{9)}$	3.09(4)
C(15)	H(15)$^{1)}$	3.27(4)	C(15)	H(1)$^{12)}$	3.50(4)
C(15)	H(6)$^{8)}$	3.54(3)	C(16)	H(21)$^{8)}$	2.99
C(16)	H(13)$^{12)}$	3.27(3)	C(16)	H(9)$^{9)}$	3.29(4)
C(16)	H(8)$^{9)}$	3.36(4)	C(16)	H(9)$^{13)}$	3.43(4)
C(16)	H(14)$^{12)}$	3.58(3)	C(16)	H(22)$^{8)}$	3.58
C(17)	H(8)$^{9)}$	2.98(4)	C(17)	H(1)$^{11)}$	3.10(4)
C(17)	H(21)$^{8)}$	3.20	C(17)	H(18)$^{5)}$	3.45(4)
C(17)	H(2)$^{11)}$	3.53(3)	C(17)	H(22)$^{8)}$	3.56
C(18)	H(18)$^{5)}$	3.25(4)	C(18)	H(8)$^{9)}$	3.30(4)
C(18)	H(21)$^{8)}$	3.39	C(19)	H(22)$^{14)}$	3.14
C(19)	H(10)$^{8)}$	3.41(3)	H(1)	H(16)$^{2)}$	2.30(6)
H(1)	H(14)$^{1)}$	2.71(5)	H(1)	H(15)$^{2)}$	3.54(5)
H(1)	H(9)$^{8)}$	3.57(6)	H(2)	H(16)$^{2)}$	2.70(5)
H(2)	H(17)$^{2)}$	3.34(3)	H(2)	H(12)$^{4)}$	3.40(3)
H(2)	H(18)$^{4)}$	3.51(5)	H(5)	H(10)$^{2)}$	2.88(5)
H(5)	H(8)$^{3)}$	2.96(5)	H(5)	H(17)$^{2)}$	3.16(5)
H(5)	H(16)$^{2)}$	3.33(6)	H(5)	H(7)$^{3)}$	3.57(5)
H(5)	H(11)$^{2)}$	3.59(5)	H(6)	H(7)$^{3)}$	2.88(5)
H(6)	H(8)$^{3)}$	3.18(5)	H(6)	H(14)$^{7)}$	3.32(5)
H(7)	H(7)$^{3)}$	3.03(6)	H(7)	H(14)$^{1)}$	3.11(5)
H(7)	H(15)$^{1)}$	3.31(5)	H(7)	H(9)$^{9)}$	3.47(5)
H(8)	H(14)$^{7)}$	2.98(5)	H(8)	H(16)$^{9)}$	3.16(6)

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
H(8)	H(13)$^{7)}$	3.56(5)	H(8)	H(17)$^{9)}$	3.56(5)
H(9)	H(15)$^{10)}$	2.54(6)	H(9)	H(13)$^{7)}$	2.77(5)
H(9)	H(14)$^{9)}$	3.33(6)	H(10)	H(22)$^{11)}$	2.69
H(10)	H(19)$^{7)}$	2.94(5)	H(10)	H(20)$^{7)}$	3.36(5)
H(10)	H(15)$^{10)}$	3.43(5)	H(10)	H(13)$^{7)}$	3.50(5)
H(13)	H(15)$^{1)}$	2.80(4)	H(13)	H(14)$^{1)}$	3.47(5)
H(14)	H(15)$^{1)}$	3.09(5)	H(14)	H(21)$^{8)}$	3.38
H(15)	H(21)$^{8)}$	3.39	H(16)	H(18)$^{5)}$	3.11(5)
H(17)	H(18)$^{5)}$	2.90(5)	H(18)	H(22)$^{14)}$	3.07
H(18)	H(19)$^{5)}$	3.26(6)	H(19)	H(22)$^{14)}$	2.76
H(19)	H(21)$^{14)}$	3.13	H(19)	H(20)$^{4)}$	3.18(6)
H(20)	H(21)$^{8)}$	2.69	H(20)	H(22)$^{14)}$	3.15

Symmetry operations

(1)	-X+1/2,-Y,Z+1/2	(2)	X,Y,Z+1
(3)	-X+1,-Y,-Z+1	(4)	X,-Y+1/2,Z+1/2
(5)	X,-Y+1/2,Z-1/2	(6)	X+1/2,-Y+1/2,-Z+1
(7)	X+1/2,Y,-Z+1/2	(8)	X-1/2,Y,-Z+1/2
(9)	-X+1,-Y,-Z	(10)	X+1/2,Y,-Z-1/2
(11)	X,Y,Z-1	(12)	-X+1/2,-Y,Z-1/2
(13)	X-1/2,Y,-Z-1/2	(14)	X-1/2,-Y+1/2,-Z+1

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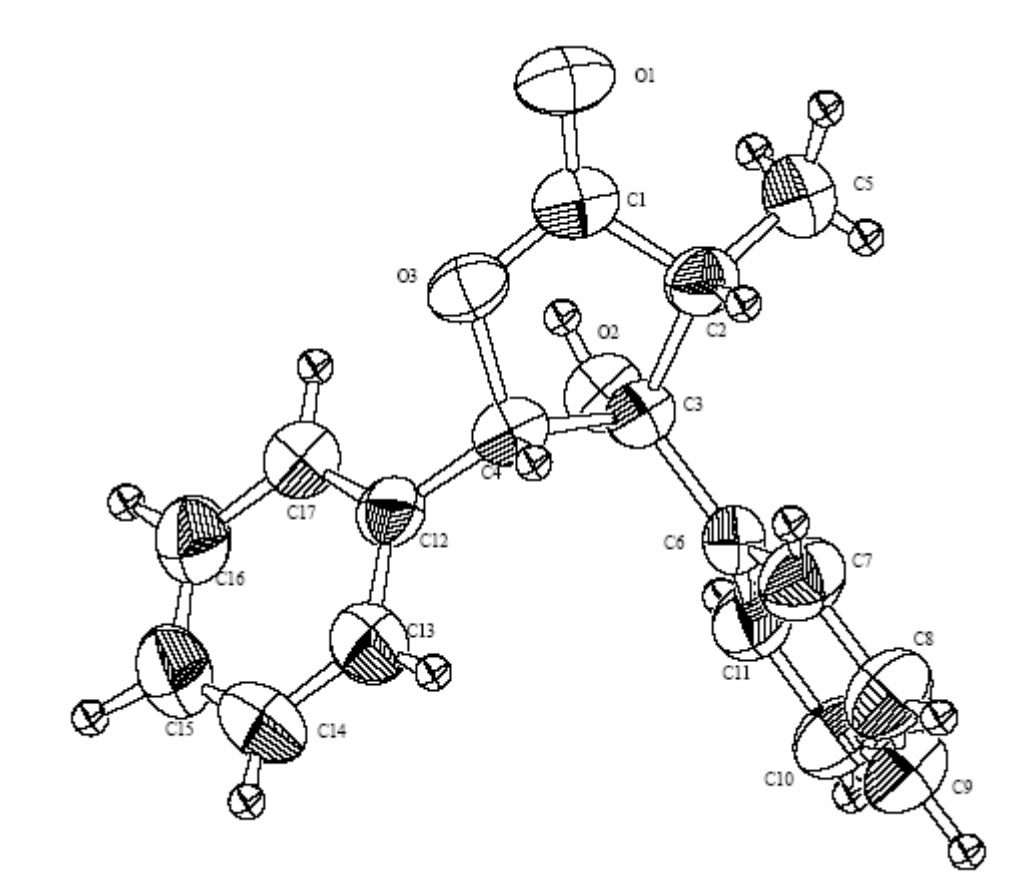

Experimental

Data Collection

A colorless prismatic crystal of $C_{17}H_{16}O_3$ having approximate dimensions of 0.38 x 0.15 x 0.25 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $20.66 < 2\theta < 23.59^{\circ}$ corresponded to a C-centered monoclinic cell with dimensions:

a = 27.767(3) Å
b = 6.363(3) Å $\beta = 119.888(7)^{\circ}$
c = 18.784(3) Å
V = 2877(1) Å^3

For Z = 8 and F.W. = 268.31, the calculated density is 1.24 g/cm^3. Based on the systematic absences of:

hkl: h+k $\pm$ 2n
h0l: l $\pm$ 2n

packing considerations, a statistical analysis ofintensity distribution,and the successful solution and refinement of thestructure, the space group was determined to be:

C2/c (#15)

The data were collected at a temperature of 23 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 60.0°.Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.34° with a take-off angle of 6.0°. Scans of $(1.52 + 0.30 \tan \theta)^{\circ}$ were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 4626 reflections which were collected, 4189 were unique (R_{int} = 0.017). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.8 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement[3] was based on 2445 observed reflections ($I > 0.50\sigma(I)$, $2\theta < 60.02$) and 245 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (Fo^2 - Fc^2) / \Sigma Fo^2 = 0.119$$

$$R_w = [(\Sigma\ w\ (Fo^2 - Fc^2)^2 / \Sigma\ w\ (Fo^2)^2)]^{1/2} = 0.153$$

$$R1 = \Sigma\ ||Fo| - |Fc|| / \Sigma\ |Fo| = 0.054 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight[4] was 1.20. The weighting scheme was based on counting statistics and included a factor (p = 0.055) to downweight the intense reflections. Plots of $\Sigma\ w\ (Fo^2 - Fc^2)^2$ versus Fo^2reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.39 and -0.41 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma\ w(F_o^2-F_c^2)^2$ where

$w = 1/[\sigma^2(Fo^2)] = [\sigma^2_c(Fo^2) + (p\ (Max(Fo^2,0)+2Fc^2)/3)^2]^{-1}$

$\sigma_c(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$[\Sigma\ w(|F_o|-|F_c|)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations

N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{17}H_{16}O_3$
Formula Weight	268.31
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.38 X 0.15 X 0.25 mm
Crystal System	monoclinic
Lattice Type	C-centered
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (20.7 - 23.6°)
Omega Scan Peak Width at Half-height	0.34°
Lattice Parameters	a = 27.767(3) Å b = 6.363(3) Å c = 18.784(3) Å β = 119.888(7)° V = 2877(1) Å^3
Space Group	C2/c (#15)
Z value	8
D_{calc}	1.239 g/cm^3
F_{000}	1136.00
μ(MoKα)	0.84 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R (rotating anode)
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.65, 12.19, 42.58)
Temperature	23.0 °C
Collimator Size	1.0 mm
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm

Scan Type	ω-2θ
Scan Rate	16.0^{o}/min (in ω) (up to 5 scans)
Scan Width	(1.52 + 0.30 tan θ)^{o}
$2\theta_{max}$	60.0^{o}
No. of Reflections Measured	Total: 4626 Unique: 4189 (R_{int} = 0.017)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	1/σ^2(Fo^2)
p-factor	0.0550
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I>0.50σ(I), 2θ < 60.02^{o})	2445
No. Variables	245
Reflection/Parameter Ratio	9.98
Residuals: R; Rw	0.119 ; 0.153
Residuals: R1	0.054
No. of Reflections to calc R1	1284
Goodness of Fit Indicator	1.20
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.39 e^-/Å^3
Minimum peak in Final Diff. Map	-0.41 e^-/Å^3

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.45955(9)	0.3006(3)	0.4687(1)	4.92(6)
O(2)	0.59281(10)	0.4775(3)	0.6797(2)	4.11(6)
O(3)	0.54714(9)	0.1926(3)	0.5309(1)	4.15(5)
C(1)	0.5012(1)	0.2551(5)	0.5315(2)	4.11(8)
C(2)	0.5106(1)	0.2554(6)	0.6174(2)	4.12(8)
C(3)	0.5743(1)	0.2657(5)	0.6701(2)	3.42(7)
C(4)	0.5911(1)	0.1401(6)	0.6149(2)	3.70(8)
C(5)	0.4766(2)	0.4197(10)	0.6309(3)	6.6(1)
C(6)	0.5989(1)	0.1722(5)	0.7558(2)	3.74(8)
C(7)	0.5834(2)	-0.0251(7)	0.7686(3)	5.6(1)
C(8)	0.6061(2)	-0.1053(9)	0.8482(3)	7.1(1)
C(9)	0.6446(2)	0.0074(10)	0.9144(3)	7.2(1)
C(10)	0.6611(2)	0.1966(9)	0.9020(2)	6.6(1)
C(11)	0.6383(1)	0.2814(7)	0.8236(2)	4.85(9)
C(12)	0.6468(1)	0.1789(5)	0.6237(2)	3.95(8)
C(13)	0.6881(2)	0.0322(8)	0.6644(3)	6.0(1)
C(14)	0.7405(2)	0.0639(10)	0.6758(3)	7.7(2)
C(15)	0.7521(2)	0.2368(10)	0.6454(3)	7.2(1)
C(16)	0.7116(2)	0.3850(8)	0.6040(3)	6.1(1)
C(17)	0.6589(2)	0.3563(7)	0.5933(2)	5.06(10)
H(1)	0.499(1)	0.121(5)	0.623(2)	3.8(7)
H(2)	0.576(1)	0.536(6)	0.634(2)	5.5(8)
H(3)	0.585(1)	-0.005(5)	0.617(2)	2.5(6)
H(4)	0.480(2)	0.559(8)	0.614(3)	10.7(7)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(5)	0.436(2)	0.403(9)	0.590(3)	12.5(7)
H(6)	0.481(2)	0.418(8)	0.676(3)	8.7(8)
H(7)	0.556(2)	-0.102(7)	0.722(3)	8.2(8)
H(8)	0.594(2)	-0.231(7)	0.858(3)	8.2(8)
H(9)	0.659(2)	-0.058(7)	0.968(3)	8.6(8)
H(10)	0.690(2)	0.286(6)	0.952(2)	7.6(8)
H(11)	0.647(1)	0.422(6)	0.822(2)	5.1(8)
H(12)	0.678(1)	-0.077(5)	0.684(2)	5.3(8)
H(13)	0.765(2)	-0.034(7)	0.697(3)	9.0(8)
H(14)	0.788(2)	0.275(7)	0.648(2)	8.6(8)
H(15)	0.721(2)	0.517(7)	0.582(2)	7.4(8)
H(16)	0.630(1)	0.456(5)	0.563(2)	5.1(8)

$B_{eq} = 8/3\ PI^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.056(1)	0.046(1)	0.056(1)	-0.011(1)	0.006(1)	0.004(1)
O(2)	0.062(1)	0.041(1)	0.049(1)	-0.008(1)	0.025(1)	-0.006(1)
O(3)	0.057(1)	0.050(1)	0.037(1)	-0.002(1)	0.014(1)	-0.004(1)
C(1)	0.051(2)	0.036(2)	0.054(2)	-0.014(2)	0.015(2)	-0.002(2)
C(2)	0.047(2)	0.052(2)	0.052(2)	-0.004(2)	0.021(2)	0.009(2)
C(3)	0.045(2)	0.039(2)	0.042(2)	-0.006(1)	0.019(1)	-0.001(1)
C(4)	0.052(2)	0.040(2)	0.042(2)	-0.003(2)	0.018(2)	-0.001(2)
C(5)	0.072(3)	0.121(4)	0.072(3)	0.026(3)	0.046(3)	0.016(3)
C(6)	0.046(2)	0.056(2)	0.045(2)	0.003(2)	0.026(2)	0.002(2)
C(7)	0.076(3)	0.064(3)	0.068(3)	-0.005(2)	0.033(2)	0.011(2)
C(8)	0.104(4)	0.089(4)	0.089(4)	0.011(3)	0.057(3)	0.040(3)
C(9)	0.093(4)	0.126(5)	0.057(3)	0.025(3)	0.040(3)	0.027(3)
C(10)	0.078(3)	0.122(4)	0.040(2)	0.005(3)	0.021(2)	-0.002(3)
C(11)	0.061(2)	0.070(3)	0.049(2)	-0.003(2)	0.024(2)	-0.004(2)
C(12)	0.055(2)	0.056(2)	0.039(2)	0.006(2)	0.024(2)	-0.003(2)
C(13)	0.073(3)	0.087(3)	0.080(3)	0.022(3)	0.047(2)	0.017(3)
C(14)	0.070(3)	0.128(5)	0.105(4)	0.042(3)	0.051(3)	0.031(3)
C(15)	0.064(3)	0.136(5)	0.085(3)	0.001(3)	0.047(2)	0.005(3)
C(16)	0.066(3)	0.102(4)	0.074(3)	-0.007(3)	0.041(2)	0.009(3)
C(17)	0.063(3)	0.073(3)	0.059(2)	0.005(2)	0.032(2)	0.008(2)

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.205(4)	O(2)	C(3)	1.421(3)
O(3)	C(1)	1.341(4)	O(3)	C(4)	1.474(3)
C(1)	C(2)	1.502(4)	C(2)	C(3)	1.538(4)
C(2)	C(5)	1.513(6)	C(3)	C(4)	1.553(4)
C(3)	C(6)	1.523(4)	C(4)	C(12)	1.494(4)
C(6)	C(7)	1.386(5)	C(6)	C(11)	1.383(5)
C(7)	C(8)	1.397(6)	C(8)	C(9)	1.370(7)
C(9)	C(10)	1.348(7)	C(10)	C(11)	1.390(5)
C(12)	C(13)	1.377(5)	C(12)	C(17)	1.379(5)
C(13)	C(14)	1.378(6)	C(14)	C(15)	1.350(7)
C(15)	C(16)	1.374(6)	C(16)	C(17)	1.387(5)

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(2)	H(2)	0.83(4)	C(2)	H(1)	0.93(3)
C(4)	H(3)	0.94(3)	C(5)	H(4)	0.97(5)
C(5)	H(5)	1.00(5)	C(5)	H(6)	0.79(5)
C(7)	H(7)	0.95(4)	C(8)	H(8)	0.91(4)
C(9)	H(9)	0.97(5)	C(10)	H(10)	1.05(4)
C(11)	H(11)	0.94(3)	C(13)	H(12)	0.90(3)
C(14)	H(13)	0.86(5)	C(15)	H(14)	1.02(4)
C(16)	H(15)	1.03(4)	C(17)	H(16)	0.96(3)

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(3)	C(4)	110.0(2)	O(1)	C(1)	O(3)	121.1(3)
O(1)	C(1)	C(2)	128.3(3)	O(3)	C(1)	C(2)	110.6(3)
C(1)	C(2)	C(3)	102.7(3)	C(1)	C(2)	C(5)	113.1(3)
C(3)	C(2)	C(5)	118.5(3)	O(2)	C(3)	C(2)	110.5(3)
O(2)	C(3)	C(4)	111.2(2)	O(2)	C(3)	C(6)	107.1(2)
C(2)	C(3)	C(4)	100.4(2)	C(2)	C(3)	C(6)	115.4(2)
C(4)	C(3)	C(6)	112.3(2)	O(3)	C(4)	C(3)	103.6(2)
O(3)	C(4)	C(12)	109.8(3)	C(3)	C(4)	C(12)	118.7(3)
C(3)	C(6)	C(7)	121.5(3)	C(3)	C(6)	C(11)	120.6(3)
C(7)	C(6)	C(11)	117.9(3)	C(6)	C(7)	C(8)	120.1(4)
C(7)	C(8)	C(9)	120.8(5)	C(8)	C(9)	C(10)	119.3(4)
C(9)	C(10)	C(11)	121.0(5)	C(6)	C(11)	C(10)	120.9(4)
C(4)	C(12)	C(13)	118.6(3)	C(4)	C(12)	C(17)	122.8(3)
C(13)	C(12)	C(17)	118.6(4)	C(12)	C(13)	C(14)	120.6(5)
C(13)	C(14)	C(15)	120.7(5)	C(14)	C(15)	C(16)	119.9(4)
C(15)	C(16)	C(17)	119.9(5)	C(12)	C(17)	C(16)	120.3(4)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	O(2)	H(2)	108(2)	C(1)	C(2)	H(1)	102(1)
C(3)	C(2)	H(1)	107(1)	C(5)	C(2)	H(1)	110(1)
O(3)	C(4)	H(3)	101(1)	C(3)	C(4)	H(3)	111(1)
C(12)	C(4)	H(3)	109(1)	C(2)	C(5)	H(4)	113(3)
C(2)	C(5)	H(5)	110(3)	C(2)	C(5)	H(6)	112(3)
H(4)	C(5)	H(5)	96(4)	H(4)	C(5)	H(6)	112(5)
H(5)	C(5)	H(6)	109(4)	C(6)	C(7)	H(7)	118(2)
C(8)	C(7)	H(7)	121(2)	C(7)	C(8)	H(8)	121(2)
C(9)	C(8)	H(8)	117(2)	C(8)	C(9)	H(9)	116(2)
C(10)	C(9)	H(9)	123(2)	C(9)	C(10)	H(10)	120(2)
C(11)	C(10)	H(10)	118(2)	C(6)	C(11)	H(11)	123(2)
C(10)	C(11)	H(11)	115(2)	C(12)	C(13)	H(12)	114(2)
C(14)	C(13)	H(12)	125(2)	C(13)	C(14)	H(13)	118(3)
C(15)	C(14)	H(13)	120(3)	C(14)	C(15)	H(14)	128(2)
C(16)	C(15)	H(14)	111(2)	C(15)	C(16)	H(15)	119(2)
C(17)	C(16)	H(15)	120(2)	C(12)	C(17)	H(16)	118(1)
C(16)	C(17)	H(16)	121(1)				

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(1)	O(3)	C(4)	-178.1(3)	O(1)	C(1)	C(2)	C(3)	-159.6(3)
O(1)	C(1)	C(2)	C(5)	-30.8(5)	O(2)	C(3)	C(2)	C(1)	84.9(3)
O(2)	C(3)	C(2)	C(5)	-40.5(4)	O(2)	C(3)	C(4)	O(3)	-83.0(3)
O(2)	C(3)	C(4)	C(12)	39.0(4)	O(2)	C(3)	C(6)	C(7)	172.1(3)
O(2)	C(3)	C(6)	C(11)	-9.0(4)	O(3)	C(1)	C(2)	C(3)	20.9(4)
O(3)	C(1)	C(2)	C(5)	149.8(3)	O(3)	C(4)	C(3)	C(2)	33.9(3)
O(3)	C(4)	C(3)	C(6)	157.0(2)	O(3)	C(4)	C(12)	C(13)	-136.8(3)
O(3)	C(4)	C(12)	C(17)	43.8(4)	C(1)	O(3)	C(4)	C(3)	-23.0(3)
C(1)	O(3)	C(4)	C(12)	-150.8(3)	C(1)	C(2)	C(3)	C(4)	-32.6(3)
C(1)	C(2)	C(3)	C(6)	-153.5(3)	C(2)	C(1)	O(3)	C(4)	1.4(3)
C(2)	C(3)	C(4)	C(12)	155.9(3)	C(2)	C(3)	C(6)	C(7)	48.7(4)
C(2)	C(3)	C(6)	C(11)	-132.4(3)	C(3)	C(4)	C(12)	C(13)	104.4(4)
C(3)	C(4)	C(12)	C(17)	-75.1(4)	C(3)	C(6)	C(7)	C(8)	-179.1(4)
C(3)	C(6)	C(11)	C(10)	-179.8(3)	C(4)	C(3)	C(2)	C(5)	-158.0(3)
C(4)	C(3)	C(6)	C(7)	-65.5(4)	C(4)	C(3)	C(6)	C(11)	113.4(3)
C(4)	C(12)	C(13)	C(14)	-178.3(4)	C(4)	C(12)	C(17)	C(16)	179.3(3)
C(5)	C(2)	C(3)	C(6)	81.1(4)	C(6)	C(3)	C(4)	C(12)	-80.9(4)
C(6)	C(7)	C(8)	C(9)	-1.3(7)	C(6)	C(11)	C(10)	C(9)	-1.2(6)
C(7)	C(6)	C(11)	C(10)	-0.9(5)	C(7)	C(8)	C(9)	C(10)	-0.8(7)
C(8)	C(7)	C(6)	C(11)	2.0(6)	C(8)	C(9)	C(10)	C(11)	2.0(7)
C(12)	C(13)	C(14)	C(15)	-1.7(7)	C(12)	C(17)	C(16)	C(15)	-0.3(6)
C(13)	C(12)	C(17)	C(16)	-0.1(6)	C(13)	C(14)	C(15)	C(16)	1.2(8)
C(14)	C(13)	C(12)	C(17)	1.2(6)	C(14)	C(15)	C(16)	C(17)	-0.2(7)

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(2)[1)]	2.799(3)	O(1)	C(1)[1)]	3.030(4)
O(1)	O(3)[2)]	3.144(3)	O(1)	C(4)[2)]	3.179(4)
O(1)	O(1)[1)]	3.199(4)	O(1)	O(3)[1)]	3.230(3)
O(1)	C(2)[1)]	3.553(4)	O(1)	C(3)[1)]	3.585(4)
O(3)	C(1)[2)]	3.118(4)	O(3)	O(3)[2)]	3.344(4)
C(1)	C(1)[1)]	3.322(6)	C(1)	C(1)[2)]	3.444(6)

Symmetry operations

(1) -X+1,-Y+1,-Z+1 (2) -X+1,-Y,-Z+1

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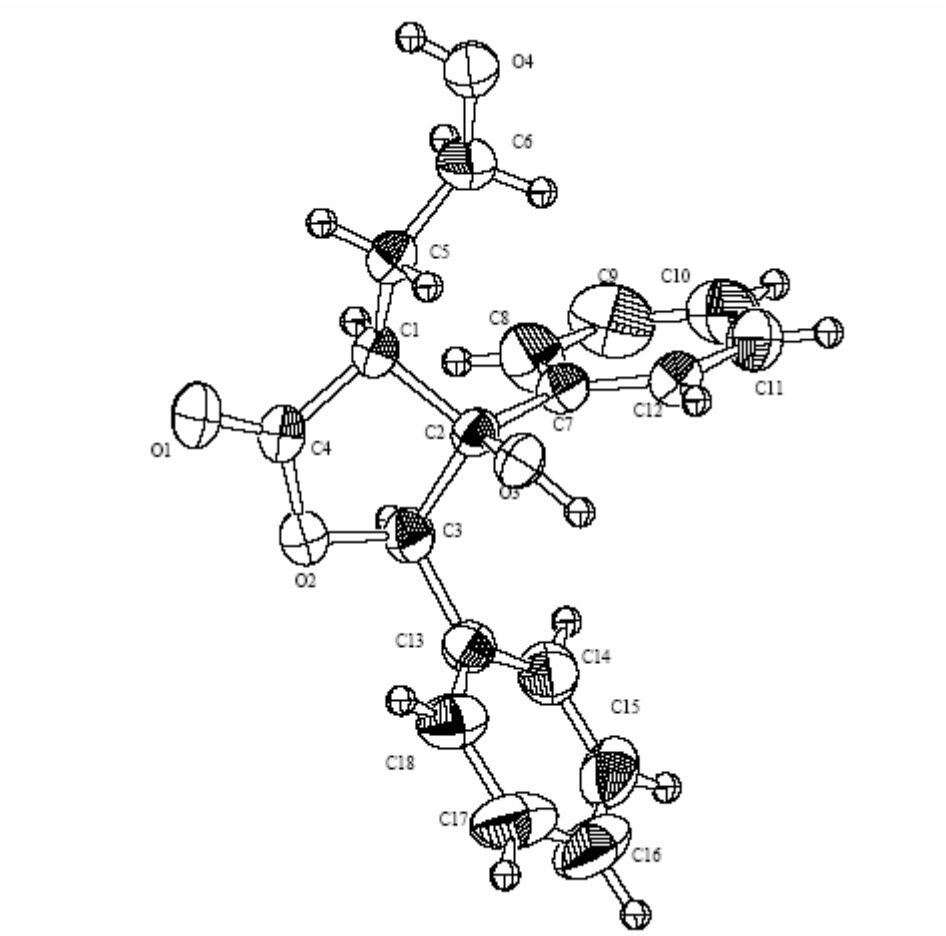

Experimental

Data Collection

A colorless prism crystal of $C_{18}H_{18}O_4$ having approximate dimensions of 0.40 x 0.40 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations which were exposed for 3.0 minutes.The camera radius was 127.40 mm.Readout was performed in the 0.100 mm pixel mode.

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

a = 8.7871(3) Å
b = 12.6298(4) Å β = 96.098(1)°
c = 13.6796(5) Å
V = 1509.57(9) Å^3

For Z = 4 and F.W. = 298.34, the calculated density is 1.31 g/cm^3. The systematic absences of:

h0l: h+l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/n$ (#14)

The data were collected at a temperature of 23 ± 1°C to a maximum 2θ value of 61.0°.A total of 74 images, corresponding to 222.0 ° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 1.00 minutes per degree. The camera radius was 127.40 mm.Readout was performed in the 0.100 mm pixel mode.Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 16397 reflections which were collected, 4569 were unique (R_{int} = 0.051).

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.9 cm^{-1}. A symmetry-related absorption correction using the program ABSCOR[1] was applied which resulted in transmission factors ranging from 0.53 to 0.99. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were included in fixed positions. The final cycle of full-matrix least-squares refinement[4] was based on 2717 observed reflections ($I > 2.90\sigma(I)$, $2\theta < 60.97$) and 267 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (Fo^2 - Fc^2) / \Sigma Fo^2 = 0.120$$

$$R_w = [(\Sigma w (Fo^2 - Fc^2)^2 / \Sigma w (Fo^2)^2)]^{1/2} = 0.161$$

$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.068$ for $I > 3.0\sigma(I)$ data

The standard deviation of an observation of unit weight[5] was 2.35. The weighting scheme was based on counting statistics and included a factor (p = 0.050) to downweight the intense reflections. Plots of $\Sigma \, w \, (Fo^2 - Fc^2)^2$ versus Fo^2reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.32 and -0.23 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[9]. All calculations were performed using the teXsan[10] crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(2) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994). J. Appl. Cryst. 27, 435.

(3) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least-Squares:

Function minimized: $\Sigma \, w(F_o^2 - F_c^2)^2$ where

$w = 1/[\sigma^2(Fo^2)] = [\sigma^2_c(Fo^2) + (p \, (Max(Fo^2,0)+2Fc^2)/3)^2]^{-1}$

$\sigma_c(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

(5) Standard deviation of an observation of unit weight:

$[\Sigma \, w(|F_o| - |F_c|)^2/(N_o - N_v)]^{1/2}$

where: N_o = number of observations

N_v = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{18}H_{18}O_4$
Formula Weight	298.34
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.40 X 0.40 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	17946 (3.0 - 61.0°)
Indexing Images	3 oscillations at 3.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 8.7871(3) Å b = 12.6298(4) Å c = 13.6796(5) Å β = 96.098(1)° V = 1509.57(9) Å^3
Space Group	$P2_1/n$ (#14)
Z value	4
D_{calc}	1.313 g/cm^3
F_{000}	632.00
μ(MoKα)	0.92 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Temperature	23.0 °C
Voltage, Current	50 kV, 40 mA
Collimator Size	0.8 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	74 exposures at 1.0 minutes per degree
Oscillation Range (ϕ=0.0°,χ=45.0°)	ω 130.0 - 190.0° with 3.0° step

Oscillation Range (ϕ=180.0^{o},χ=45.0^{o})	ω 0.0 - 162.0^{o} with 3.0^{o} step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	61.0^{o}
No. of Reflections Measured	Total: 16397 Unique: 4569 (R_{int} = 0.051)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.5277 - 0.9908)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	$1/\sigma^2(Fo^2)$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations (I>2.90σ(I), 2θ < 60.97^{o})	2717
No. Variables	267
Reflection/Parameter Ratio	10.18
Residuals: R; Rw	0.120 ; 0.161
Residuals: R1	0.068
No. of Reflections to calc R1	2717
Goodness of Fit Indicator	2.35
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.32 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.23 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.4359(3)	0.2850(2)	0.5116(1)	5.65(5)
O(2)	0.3863(2)	0.1728(1)	0.3881(1)	4.33(4)
O(3)	0.6455(2)	0.2322(1)	0.2828(1)	3.11(3)
O(4)	0.6907(3)	0.5962(2)	0.3369(2)	5.35(5)
C(1)	0.4627(3)	0.3432(2)	0.3437(2)	3.30(5)
C(2)	0.4925(2)	0.2661(2)	0.2606(1)	2.76(4)
C(3)	0.3804(3)	0.1754(2)	0.2813(2)	3.39(5)
C(4)	0.4297(3)	0.2679(2)	0.4249(2)	4.05(5)
C(5)	0.5827(3)	0.4258(2)	0.3756(2)	3.82(5)
C(6)	0.5904(4)	0.5131(2)	0.3019(2)	4.53(6)
C(7)	0.4582(2)	0.3090(2)	0.1570(1)	3.05(4)
C(8)	0.3124(3)	0.3449(2)	0.1243(2)	4.81(6)
C(9)	0.2783(4)	0.3807(3)	0.0297(2)	6.05(8)
C(10)	0.3892(4)	0.3838(2)	-0.0335(2)	5.52(7)
C(11)	0.5339(4)	0.3504(2)	-0.0019(2)	4.72(6)
C(12)	0.5691(3)	0.3126(2)	0.0928(2)	3.48(5)
C(13)	0.4182(2)	0.0690(2)	0.2420(2)	3.48(5)
C(14)	0.3698(3)	0.0464(2)	0.1441(2)	4.78(6)
C(15)	0.4100(4)	-0.0486(3)	0.1039(3)	6.27(9)
C(16)	0.4949(4)	-0.1220(3)	0.1601(4)	6.95(10)
C(17)	0.5403(4)	-0.1001(2)	0.2561(3)	6.28(9)
C(18)	0.5032(3)	-0.0048(2)	0.2976(2)	4.58(6)
H(1)	0.368(3)	0.376(2)	0.322(2)	3.8(5)
H(2)	0.668(3)	0.183(2)	0.235(2)	5.2(6)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(3)	0.279(3)	0.194(2)	0.256(2)	3.3(5)
H(4)	0.692(3)	0.393(2)	0.388(2)	4.3(5)
H(5)	0.560(3)	0.459(2)	0.434(2)	5.2(6)
H(6)	0.6253	0.4842	0.2432	5.5
H(7)	0.491(3)	0.541(2)	0.280(2)	5.0(6)
H(8)	0.655(4)	0.628(3)	0.380(2)	5.6(8)
H(9)	0.238(3)	0.344(2)	0.167(2)	4.7(6)
H(10)	0.183(4)	0.399(3)	0.011(2)	6.2(8)
H(11)	0.359(3)	0.411(3)	-0.098(2)	6.2(7)
H(12)	0.609(4)	0.354(2)	-0.043(2)	5.8(7)
H(13)	0.668(3)	0.287(2)	0.115(2)	4.7(6)
H(14)	0.314(4)	0.097(3)	0.100(3)	7.1(9)
H(15)	0.378(4)	-0.061(3)	0.034(2)	6.6(8)
H(16)	0.524(4)	-0.187(3)	0.132(2)	7.5(9)
H(17)	0.605(4)	-0.154(3)	0.294(2)	7.8(9)
H(18)	0.542(3)	0.013(2)	0.364(2)	4.4(6)

$B_{eq} = 8/3\ PI^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.109(2)	0.065(1)	0.0474(10)	-0.008(1)	0.0365(10)	-0.0085(9)
O(2)	0.075(1)	0.0450(9)	0.0493(9)	-0.0092(8)	0.0288(8)	-0.0026(8)
O(3)	0.0358(7)	0.0429(8)	0.0396(8)	0.0044(6)	0.0044(6)	-0.0038(7)
O(4)	0.093(1)	0.049(1)	0.067(1)	-0.0243(10)	0.038(1)	-0.0178(9)
C(1)	0.046(1)	0.039(1)	0.044(1)	0.0027(9)	0.0167(9)	-0.0028(10)
C(2)	0.0338(10)	0.0335(10)	0.0383(10)	0.0014(8)	0.0075(8)	-0.0032(8)
C(3)	0.039(1)	0.045(1)	0.045(1)	-0.0033(9)	0.0119(9)	-0.002(1)
C(4)	0.062(1)	0.048(1)	0.049(1)	0.000(1)	0.026(1)	-0.004(1)
C(5)	0.062(1)	0.042(1)	0.043(1)	-0.006(1)	0.017(1)	-0.009(1)
C(6)	0.074(2)	0.043(1)	0.057(1)	-0.012(1)	0.016(1)	-0.006(1)
C(7)	0.041(1)	0.036(1)	0.039(1)	0.0011(8)	0.0034(8)	-0.0024(9)
C(8)	0.049(1)	0.068(2)	0.065(2)	0.009(1)	0.006(1)	0.015(1)
C(9)	0.066(2)	0.079(2)	0.078(2)	0.008(2)	-0.022(2)	0.017(2)
C(10)	0.101(2)	0.060(2)	0.045(1)	-0.003(2)	-0.010(2)	0.005(1)
C(11)	0.086(2)	0.056(2)	0.039(1)	-0.001(1)	0.013(1)	-0.002(1)
C(12)	0.050(1)	0.042(1)	0.042(1)	0.0001(10)	0.0081(10)	-0.0051(10)
C(13)	0.040(1)	0.041(1)	0.053(1)	-0.0117(9)	0.0128(10)	-0.008(1)
C(14)	0.068(2)	0.060(2)	0.055(1)	-0.021(1)	0.012(1)	-0.012(1)
C(15)	0.087(2)	0.080(2)	0.076(2)	-0.039(2)	0.036(2)	-0.041(2)
C(16)	0.067(2)	0.063(2)	0.140(3)	-0.020(2)	0.040(2)	-0.051(2)
C(17)	0.054(2)	0.046(2)	0.139(3)	0.003(1)	0.010(2)	-0.014(2)
C(18)	0.054(2)	0.045(1)	0.073(2)	-0.004(1)	0.001(1)	-0.008(1)

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(4)	1.201(3)	O(2)	C(3)	1.457(3)
O(2)	C(4)	1.342(3)	O(3)	C(2)	1.413(2)
O(4)	C(6)	1.421(3)	C(1)	C(2)	1.540(3)
C(1)	C(4)	1.513(3)	C(1)	C(5)	1.515(3)
C(2)	C(3)	1.556(3)	C(2)	C(7)	1.516(3)
C(3)	C(13)	1.498(3)	C(5)	C(6)	1.501(4)
C(7)	C(8)	1.388(3)	C(7)	C(12)	1.380(3)
C(8)	C(9)	1.374(4)	C(9)	C(10)	1.369(5)
C(10)	C(11)	1.366(4)	C(11)	C(12)	1.385(4)
C(13)	C(14)	1.391(4)	C(13)	C(18)	1.373(4)
C(14)	C(15)	1.381(4)	C(15)	C(16)	1.373(6)
C(16)	C(17)	1.360(6)	C(17)	C(18)	1.385(4)

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(3)	H(2)	0.94(3)	O(4)	H(8)	0.81(3)
C(1)	H(1)	0.95(3)	C(3)	H(3)	0.96(2)
C(5)	H(4)	1.04(3)	C(5)	H(5)	0.94(3)
C(6)	H(6)	0.96	C(6)	H(7)	0.96(3)
C(8)	H(9)	0.92(3)	C(9)	H(10)	0.88(3)
C(10)	H(11)	0.96(3)	C(11)	H(12)	0.91(3)
C(12)	H(13)	0.94(3)	C(14)	H(14)	0.98(4)
C(15)	H(15)	0.98(3)	C(16)	H(16)	0.95(4)
C(17)	H(17)	0.99(4)	C(18)	H(18)	0.97(3)

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	O(2)	C(4)	109.5(2)	C(2)	C(1)	C(4)	101.9(2)
C(2)	C(1)	C(5)	118.7(2)	C(4)	C(1)	C(5)	113.8(2)
O(3)	C(2)	C(1)	105.4(2)	O(3)	C(2)	C(3)	110.1(2)
O(3)	C(2)	C(7)	113.5(2)	C(1)	C(2)	C(3)	99.8(2)
C(1)	C(2)	C(7)	115.6(2)	C(3)	C(2)	C(7)	111.5(2)
O(2)	C(3)	C(2)	104.1(2)	O(2)	C(3)	C(13)	110.7(2)
C(2)	C(3)	C(13)	115.3(2)	O(1)	C(4)	O(2)	120.8(2)
O(1)	C(4)	C(1)	128.2(2)	O(2)	C(4)	C(1)	110.9(2)
C(1)	C(5)	C(6)	113.1(2)	O(4)	C(6)	C(5)	112.7(2)
C(2)	C(7)	C(8)	120.2(2)	C(2)	C(7)	C(12)	121.5(2)
C(8)	C(7)	C(12)	118.3(2)	C(7)	C(8)	C(9)	120.8(3)
C(8)	C(9)	C(10)	120.4(3)	C(9)	C(10)	C(11)	119.5(3)
C(10)	C(11)	C(12)	120.7(3)	C(7)	C(12)	C(11)	120.3(2)
C(3)	C(13)	C(14)	118.2(2)	C(3)	C(13)	C(18)	122.6(2)
C(14)	C(13)	C(18)	119.2(2)	C(13)	C(14)	C(15)	119.8(3)
C(14)	C(15)	C(16)	120.7(3)	C(15)	C(16)	C(17)	119.3(3)
C(16)	C(17)	C(18)	121.0(4)	C(13)	C(18)	C(17)	120.1(3)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	O(3)	H(2)	108(1)	C(6)	O(4)	H(8)	109(2)
C(2)	C(1)	H(1)	104(1)	C(4)	C(1)	H(1)	106(1)
C(5)	C(1)	H(1)	110(1)	O(2)	C(3)	H(3)	107(1)
C(2)	C(3)	H(3)	109(1)	C(13)	C(3)	H(3)	109(1)
C(1)	C(5)	H(4)	111(1)	C(1)	C(5)	H(5)	110(1)
C(6)	C(5)	H(4)	107(1)	C(6)	C(5)	H(5)	105(1)
H(4)	C(5)	H(5)	108(2)	O(4)	C(6)	H(6)	108.7
O(4)	C(6)	H(7)	110(1)	C(5)	C(6)	H(6)	108.9
C(5)	C(6)	H(7)	111(1)	H(6)	C(6)	H(7)	104.0
C(7)	C(8)	H(9)	119(1)	C(9)	C(8)	H(9)	119(1)
C(8)	C(9)	H(10)	117(2)	C(10)	C(9)	H(10)	121(2)
C(9)	C(10)	H(11)	116(1)	C(11)	C(10)	H(11)	124(1)
C(10)	C(11)	H(12)	120(1)	C(12)	C(11)	H(12)	119(1)
C(7)	C(12)	H(13)	117(1)	C(11)	C(12)	H(13)	122(1)
C(13)	C(14)	H(14)	122(2)	C(15)	C(14)	H(14)	117(2)
C(14)	C(15)	H(15)	117(2)	C(16)	C(15)	H(15)	121(2)
C(15)	C(16)	H(16)	120(2)	C(17)	C(16)	H(16)	120(2)
C(16)	C(17)	H(17)	117(2)	C(18)	C(17)	H(17)	121(2)
C(13)	C(18)	H(18)	119(1)	C(17)	C(18)	H(18)	120(1)

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(4)	O(2)	C(3)	176.8(2)	O(1)	C(4)	C(1)	C(2)	160.0(3)
O(1)	C(4)	C(1)	C(5)	31.1(4)	O(2)	C(3)	C(2)	O(3)	74.9(2)
O(2)	C(3)	C(2)	C(1)	-35.6(2)	O(2)	C(3)	C(2)	C(7)	-158.2(2)
O(2)	C(3)	C(13)	C(14)	158.8(2)	O(2)	C(3)	C(13)	C(18)	-23.8(3)
O(2)	C(4)	C(1)	C(2)	-21.3(3)	O(2)	C(4)	C(1)	C(5)	-150.3(2)
O(3)	C(2)	C(1)	C(4)	-80.8(2)	O(3)	C(2)	C(1)	C(5)	45.0(2)
O(3)	C(2)	C(3)	C(13)	-46.6(3)	O(3)	C(2)	C(7)	C(8)	179.7(2)
O(3)	C(2)	C(7)	C(12)	0.3(3)	O(4)	C(6)	C(5)	C(1)	173.4(2)
C(1)	C(2)	C(3)	C(13)	-157.1(2)	C(1)	C(2)	C(7)	C(8)	-58.3(3)
C(1)	C(2)	C(7)	C(12)	122.2(2)	C(1)	C(4)	O(2)	C(3)	-2.0(3)
C(2)	C(1)	C(5)	C(6)	72.4(3)	C(2)	C(3)	O(2)	C(4)	24.4(2)
C(2)	C(3)	C(13)	C(14)	-83.4(3)	C(2)	C(3)	C(13)	C(18)	94.0(3)
C(2)	C(7)	C(8)	C(9)	-177.8(3)	C(2)	C(7)	C(12)	C(11)	178.9(2)
C(3)	C(2)	C(1)	C(4)	33.3(2)	C(3)	C(2)	C(1)	C(5)	159.2(2)
C(3)	C(2)	C(7)	C(8)	54.7(3)	C(3)	C(2)	C(7)	C(12)	-124.7(2)
C(3)	C(13)	C(14)	C(15)	176.4(2)	C(3)	C(13)	C(18)	C(17)	-177.1(2)
C(4)	O(2)	C(3)	C(13)	148.9(2)	C(4)	C(1)	C(2)	C(7)	153.0(2)
C(4)	C(1)	C(5)	C(6)	-167.8(2)	C(5)	C(1)	C(2)	C(7)	-81.2(3)
C(7)	C(2)	C(3)	C(13)	80.3(2)	C(7)	C(8)	C(9)	C(10)	-1.6(5)
C(7)	C(12)	C(11)	C(10)	-0.5(4)	C(8)	C(7)	C(12)	C(11)	-0.6(4)
C(8)	C(9)	C(10)	C(11)	0.4(5)	C(9)	C(8)	C(7)	C(12)	1.6(4)
C(9)	C(10)	C(11)	C(12)	0.6(5)	C(13)	C(14)	C(15)	C(16)	1.2(4)
C(13)	C(18)	C(17)	C(16)	0.6(5)	C(14)	C(13)	C(18)	C(17)	0.2(4)
C(14)	C(15)	C(16)	C(17)	-0.4(5)	C(15)	C(14)	C(13)	C(18)	-1.1(4)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(15)	C(16)	C(17)	C(18)	-0.5(5)					

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(4)[1)]	2.876(3)	O(2)	C(11)[2)]	3.595(4)
O(3)	O(4)[3)]	2.864(2)	O(3)	C(10)[4)]	3.451(3)
O(3)	C(17)[5)]	3.564(3)	O(4)	C(12)[5)]	3.523(3)
C(10)	C(11)[6)]	3.448(4)	C(10)	C(10)[6)]	3.587(6)
C(11)	C(16)[7)]	3.599(4)			

Symmetry operations

(1)	-X+1,-Y+1,-Z+1	(2)	X-1/2,-Y+1/2,Z+1/2
(3)	-X+3/2,Y-1/2,-Z+1/2	(4)	X+1/2,-Y+1/2,Z+1/2
(5)	-X+3/2,Y+1/2,-Z+1/2	(6)	-X+1,-Y+1,-Z
(7)	-X+1,-Y,-Z		