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Supporting Information (27 pages)

SUPPORTING INFORMATION

Tetrabutylammonium Fluoride Promoted Novel Reactions of ortho-Carborane: Inter- and Intramolecular Addition to Aldehydes and Ketones, and Annulation via Enals and Enones

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Spectral data for the compounds (**3f-j, 4b-d, 4f-g, 7e-g**) were shown in below. For the compounds (**3c, 3e, 3g, 3h, 4b-g, 5b,** and **5f**), in which only high mass data are obtained in the experimental section, those ^{1}H NMR spectra are shown in the supporting information. Also X-ray crystallographic data for compound **7a** are shown in the supporting information.

1-(*o*-Carboranyl)-*p*-methoxybenzyl alcohol (3f). White crystal: mp 111 ℃; IR ($CHCl_3$) 3600, 3500~3150, 3150~2850, 2840, 2570, 1610, 1500, 1300, 1220, 1170, 1090, 1030 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.25 (d, J = 9.0 Hz, 2 H), 6.91 (d, J = 9.0 Hz, 2 H), 5.22 (d, J = 3.0 Hz, 1 H), 3.83 (s, 3 H), 3.78 (bs, 1 H), 2.48 (d, J = 3.0 Hz, 1 H). MS (EI) *m/z* 280 (M^+), 264 (M^+–OH), 121 (MeOPhC), 109 (MeOPh), 94 (OPh), 77 (Ph). HRMS (EI): Calcd for $C_{10}H_{20}O_2B_{10}$: *m/z* 282.2394. Found *m/z* 282.2395. Anal. Calcd for $C_{10}H_{20}O_2B_{10}$: C, 42.80; H, 7.19. Found: C, 42.50; H, 6.95 %.

1-(*o*-Carboranyl)-3-phenyl-1-propanol (3g). liquid; IR ($CHCl_3$) 3600, 3500~3150, 3100, 2940, 2860, 2600, 1600, 1500, 1460, 1390, 1100, 1060, 1020 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.34~7.16 (m, 5 H), 4.02 (m, 3 H), 2.87 (m, 1 H), 2.67 (m, 1 H), 2.10 (m, 2 H), 1.77 (m, 1 H). MS (EI) *m/z* 278 (M^+), 261 (M^+–OH). HRMS (EI): Calcd for $C_{11}H_{22}OB_{10}$: *m/z* 280.2601. Found *m/z* 280.2603.

1-(*o*-Carboranyl)-1-pentanol. (3h). Colorless needle; IR (KBr) 3650~3100, 2950, 2930, 2850, 2560, 1620, 1460, 1120, 1070, 1010 cm^{-1}; ^1H NMR δ 4.04 (m, 2 H), 1.97 (d, J = 7.0 Hz, 1 H), 1.76 (m. 1 H), 1.60~1.20 (m, 5 H), 0.92 (t, J = 7.0 Hz, 3 H). MS (EI) *m/z* 230 (M^+), 212 (M^+–H–OH), 197 (M^+–H–OH–Me), 182 (M^+–H–OH–C_2H_5), 172 (M^+–H–C_3H_7), 155 (M^+–H–C_3H_7OH), 142 (carborane). HRMS (EI): Calcd for $C_7H_{22}OB_{10}$: *m/z* 232.2601. Found *m/z* 232.2609.

1-(*o*-Carboranyl)-2-methyl-1-propanol (3i). Colorless crystal; mp 79 ℃; IR ($CHCl_3$) 3150~2900, 2600, 1740, 1020 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.04 (bs, 1 H), 3.96 (dd, J = 8.0, 2.5 Hz, 1 H), 2.08 (m, 1 H), 1.98 (d, J = 8.0 Hz, 1 H), 1.05 (d, J = 7.0 Hz, 3 H), 0.97 (d, J = 6.5 Hz, 3 H). MS (EI) *m/z* 215 (M^+–H), 200 (M^+–H–Me), 172 (M^+–H–iPr). HRMS (EI): Calcd for $C_6H_{19}OB_{10}$: *m/z* 217.2366. Found *m/z* 217.2401. Anal. Calcd for $C_6H_{19}OB_{10}$: C, 33.31; H, 9.25. Found C, 33.14; H, 8.86.

1-(*o*-Carboranyl)-1-ethanol (3j). Colorless oil; IR (neat) 3431, 3085, 2987, 2582, 1388, 1209, 1126, 1062 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.27 (q, J = 6.5 Hz, 1 H), 4.01 (bs, 1 H), 2.30 (s, 1 H), 1.38 (d, J = 6.5 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 79.45, 68.92, 58.57, 23.54; Anal. Calcd for $C_4H_{16}OB_{10}$: C, 25.52; H, 8.57. Found: C, 25.53; H, 8.50.

4-(*o*-Carboranyl)-butanal (4b). White solid: IR (KBr) 3055, 2966, 2943, 2851, 2802, 2574, 1712, 1454 cm^{-1}. ^1H NMR ($CDCl_3$) δ 9.69 (s, 1H), 3.60 (bs, 1H), 2.44 (t, J = 6.5 Hz, 2H). 2.19 (t, J = 8.5 Hz, 2H), 1.81-1.70 (m, 2H);); ^{13}C NMR ($CDCl_3$) δ 200.17, 74.63, 61.19, 42.35, 36.75, 21.38; HRMS (EI) Calcd for $C_6H_{18}OB_{10}$: *m/z* 216.2289. Found *m/z* 216.2292.

5-(*o*-Carboranyl)-pentanal (4c). White solid: IR (KBr) 3062, 2939, 2869, 2827, 2590, 1722, 1461, 1409, 1390, 1124 cm^{-1}. ^1H NMR ($CDCl_3$) δ 9.76 (t, J = 1.4 Hz, 1H), 3.58 (bs, 1H), 2.47 (dt, J = 6.5, 1.4 Hz, 2H). 2.21 (t, J = 9.0 Hz, 2H), 1.62-1.43 (m, 4H);); ^{13}C NMR ($CDCl_3$) δ 201.20, 74.81, 61.05, 43.19, 37.87, 28.59, 21.19; HRMS (EI) Calcd for $C_7H_{20}OB_{10}$: *m/z* 230.2445. Found *m/z* 230.2443.

6-(*o*-Carboranyl)-hexanal (4d). White solid: IR (KBr) 2939, 2866, 2823, 2723, 2592, 1724, 1463, 1407, 1390, 1066 cm^{-1}. ^1H NMR ($CDCl_3$) δ 9.75 (s, 1H), 3.55 (bs, 1H), 2.44 (t, J = 7.0, Hz, 2H). 2.19 (t, J = 8.5 Hz, 2H), 1.67-1.43 (m, 4H), 1.35-1.25 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 201.92, 75.16, 61.05, 43.37, 37.64, 28.28, 28.22, 21.28; HRMS (EI) Calcd for $C_8H_{22}OB_{10}$: *m/z* 244.2602. Found *m/z* 244.2612.

5-(*o*-Carboranyl)-2-pentanone (4f). White solid: IR (KBr) 2929, 2898, 2590, 1714, 1452, 1407, 1363, 1164 cm^{-1}. ^1H NMR ($CDCl_3$) δ 3.59 (bs, 1H), 2.43 (t, J = 6.0, Hz, 2H). 2.20 (t, J = 8.5 Hz, 2H), 2.13 (s, 3H), 1.86-1.65 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 206.96, 74.01, 60.99, 41.90, 37.09, 29.95, 22.86; HRMS (EI) Calcd for $C_7H_{20}OB_{10}$: *m/z* 230.2445. Found *m/z* 230.2461.

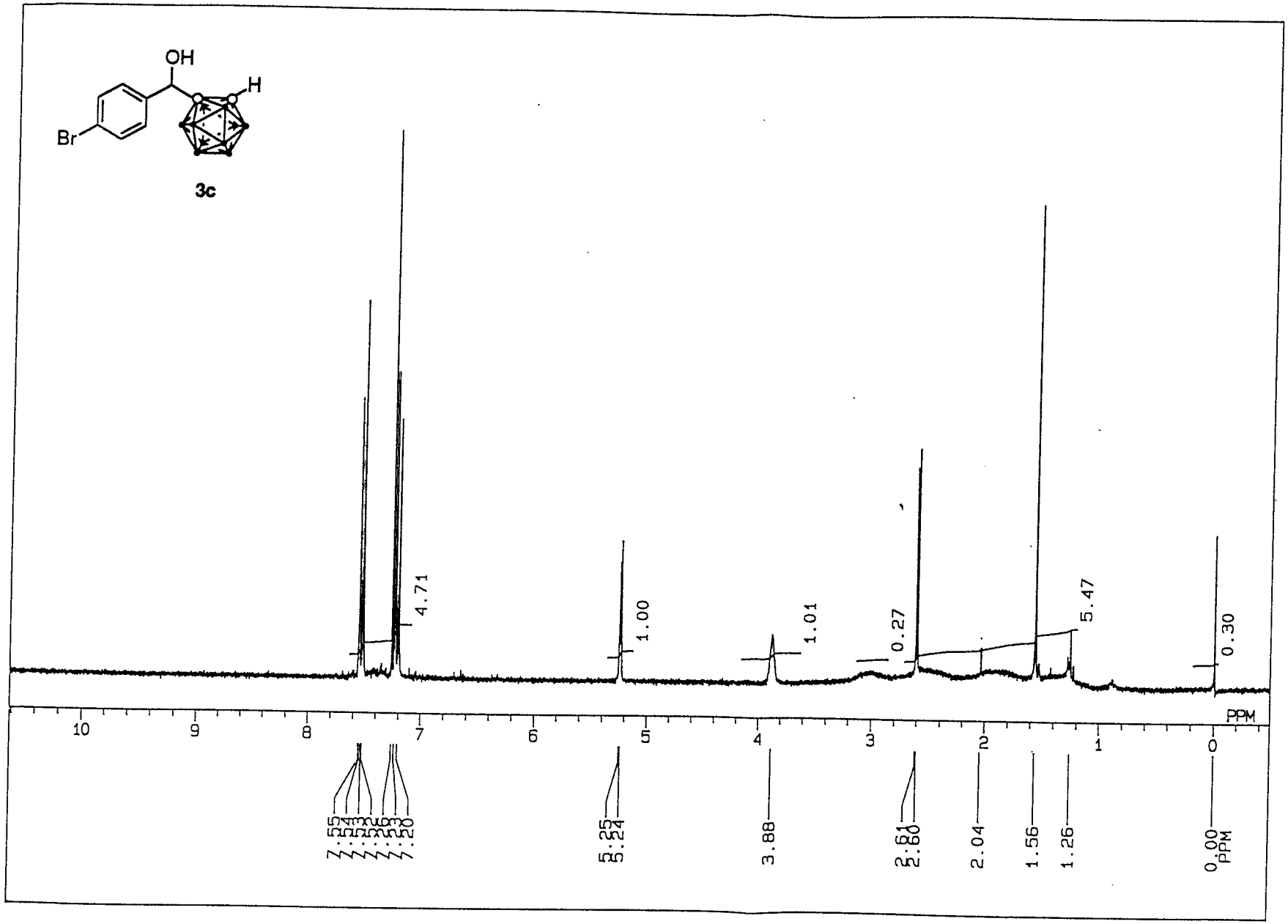
6-(*o*-Carboranyl)-2-hexanone (4g). White solid: IR (KBr) 3004, 2939, 2893, 2869, 2590, 1714, 1461, 1409, 1367, 1161 cm^{-1}. ^1H NMR ($CDCl_3$) δ 3.57 (bs,

1H), 2.44 (t, J = 6.5, Hz, 2H). 2.20 (t, J = 9.0 Hz, 2H), 2.12 (s, 3H), 1.56-1.39 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 207.82, 75.01, 60.99, 42.70, 37.87, 29.92, 28.56, 22.72.

4-Methyl-1,2-carboracyclopentan-3-ol (7e): White solid: IR (CCl_4) 3583, 3363, 2970, 2588, 665 cm^{-1}. ^1H NMR ($CDCl_3$) anti isomer; δ 4.31 (t, J = 6.0 Hz, 1H), 2.60 (m, 2H), 2.28 (d, J = 5.6 Hz, 1H), 2.06 (dd, J = 12.8, 8.8 Hz, 1H), 1.20 (d, J = 7.2 Hz, 3H), syn isomer; δ 4.55 (dd, J = 7.6, 6.0 Hz, 1H), 3.01 (m, 1H), 2.60 (m, 1H), 2.18 (d, J = 6.0 Hz, 1H), 2.13 (dd, J = 13.6, 9.2 Hz, 1H), 1.05 (d, J = 7.2 Hz, 3H). Anal. Calcd for $C_6H_{18}OB_{10}$: C, 33.63; H, 8.47. Found: C, 33.36; H, 8.47.

3,5-Dimethyl-1,2-carboracyclopentan-3-ol (7f): White solid: IR (KBr) 3565, 3487, 2856, 2594, 1452, 1385 cm^{-1}. ^1H NMR ($CDCl_3$) anti isomer; δ 3.08-2.93 (m, 1H), 2.45 (dd, J = 14.5, 7.7 Hz, 1H), 2.05 (s, 1H), 1.95 (dd, J = 14.5, 10.0 Hz, 1H), 1.55 (s, 3H), 1.11 (d, J = 6.5 Hz, 3H), syn isomer; δ 2.87-2.77 (m, 1H), 2.60 (dd, J = 14.5, 8.5 Hz, 1H), 2.00 (m, 1H), 1.62 (s, 3H), 1.18 (d, J = 6.5 Hz, 3H). Anal. Calcd for $C_7H_{26}OB_{10}$: C, 36.82; H, 8.83. Found: C, 36.81; H, 8.85.

3-Methyl-5-phenyl-1,2-carboracyclopentan-3-ol (7g): White solid: IR (CCl_4) 3583, 3467, 2580, 2310, 2343, 665 cm^{-1}. ^1H NMR ($CDCl_3$) anti isomer; δ 7.24-7.41 (m, 5H), 4.21 (dd, J = 10.5, 8.0 Hz, 1H), 2.75 (dd, J = 14.0, 10.5 Hz, 1H), 2.67 (dd, J = 14.0, 8.0 Hz, 1H), 2.13 (s, 1H), 1.69 (s, 3H), syn isomer; δ 7.24-7.41 (m, 5H), 4.04 (t, J = 8.0 Hz, 1H), 2.77 (m, 2H), 2.17 (s, 1H), 1.74 (s, 3H). Anal. Calcd for $C_{12}H_{21}OB_{10}$: C,49.80; H, 7.31. Found: C, 49.57; H, 7.28.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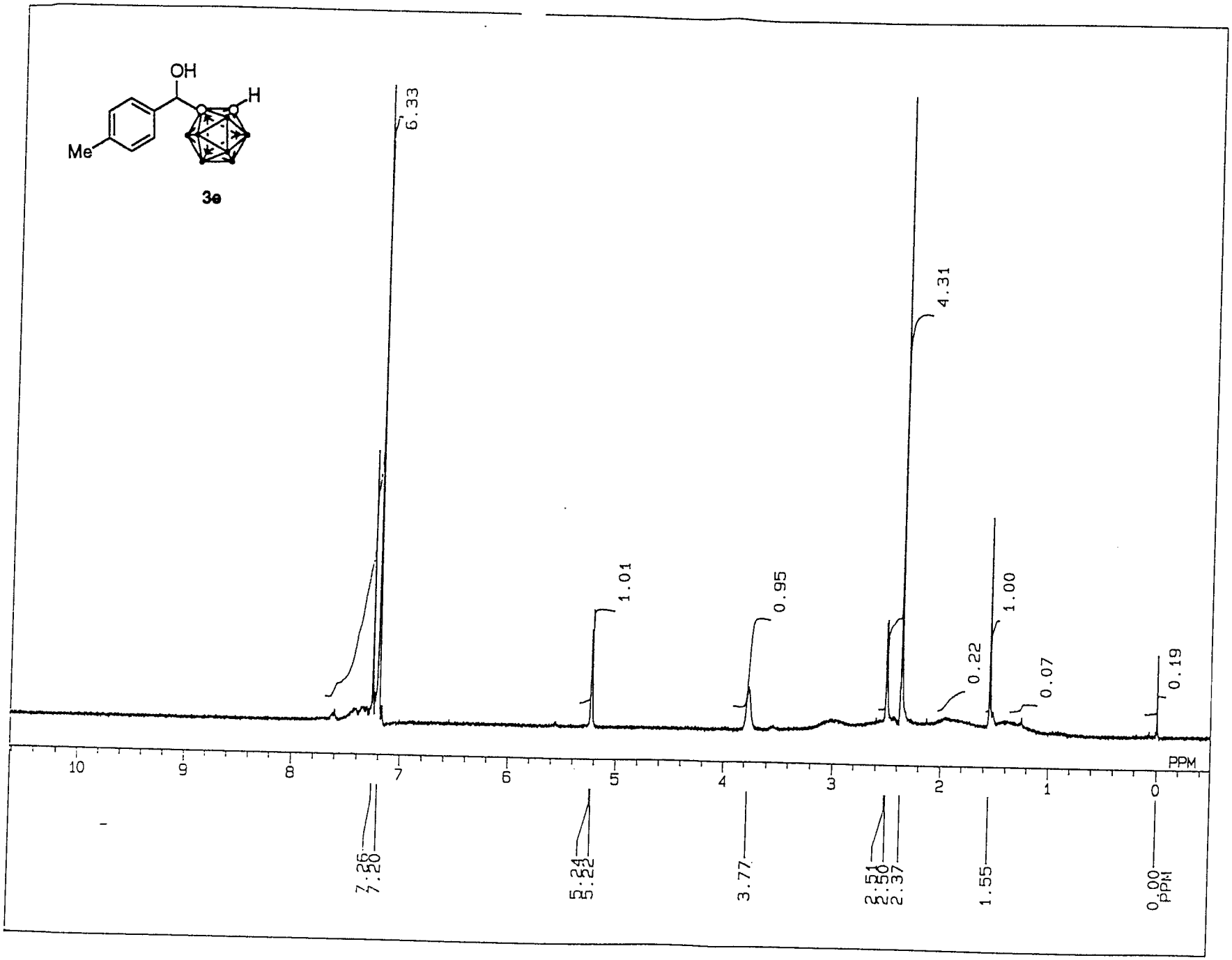
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8
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6
5
4
3
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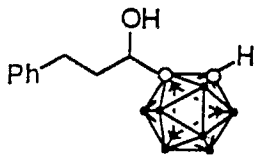

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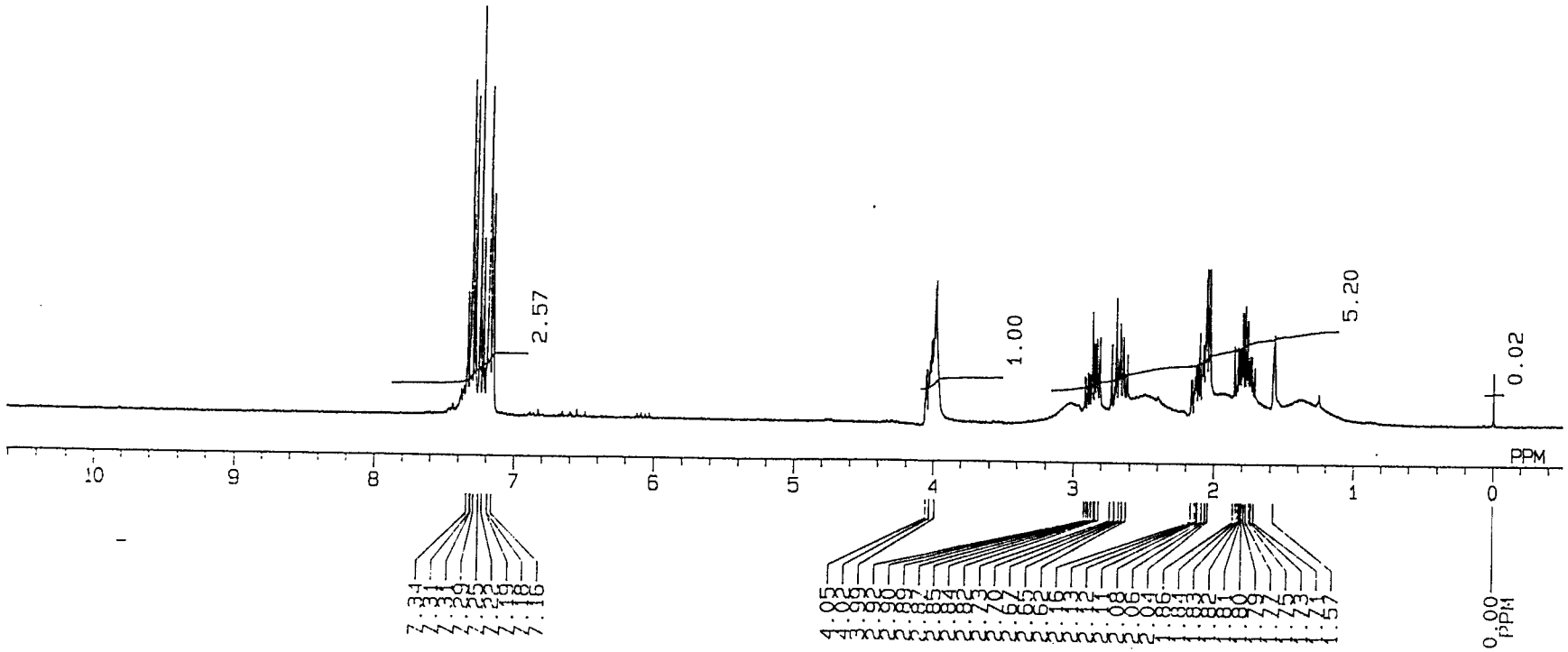

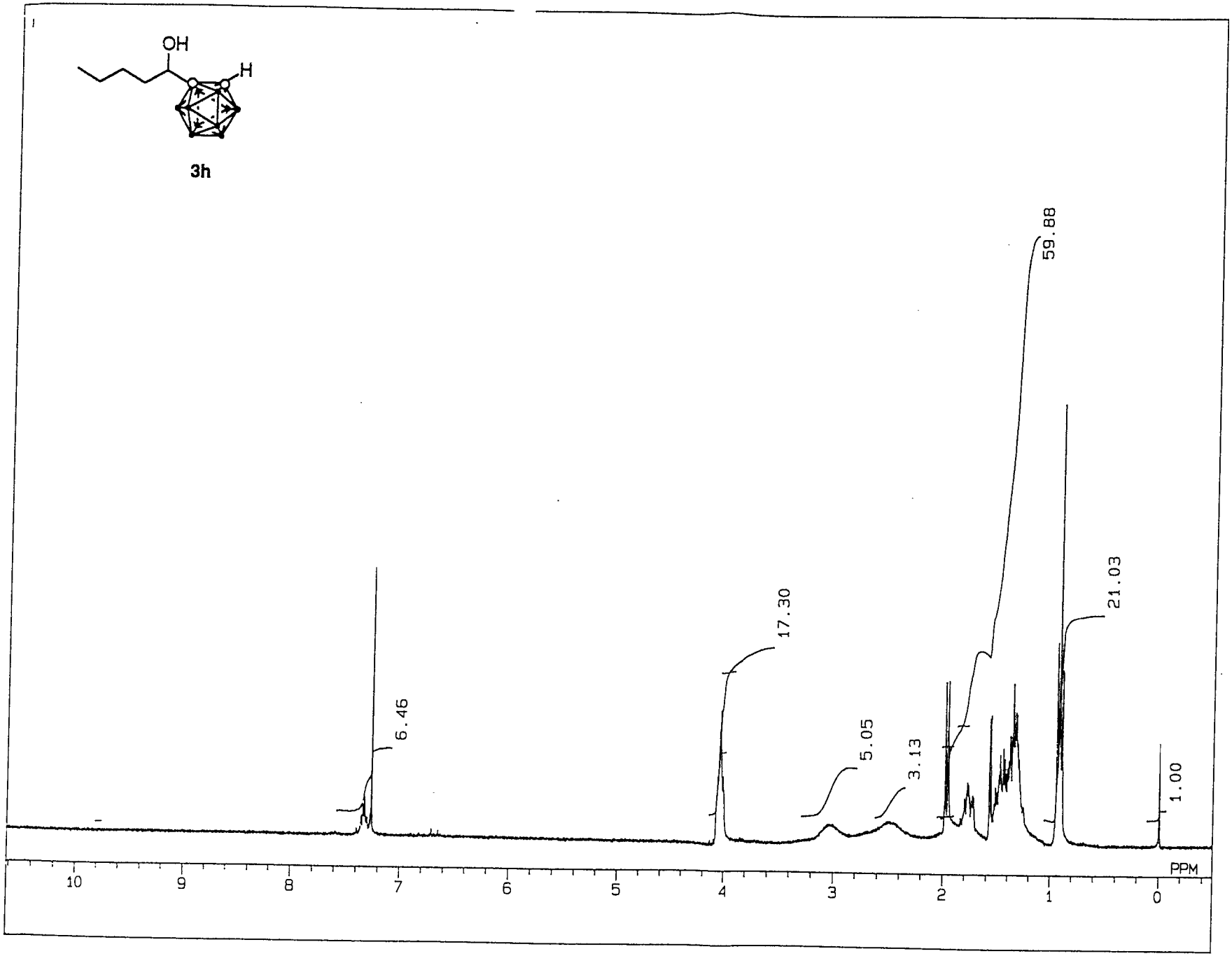
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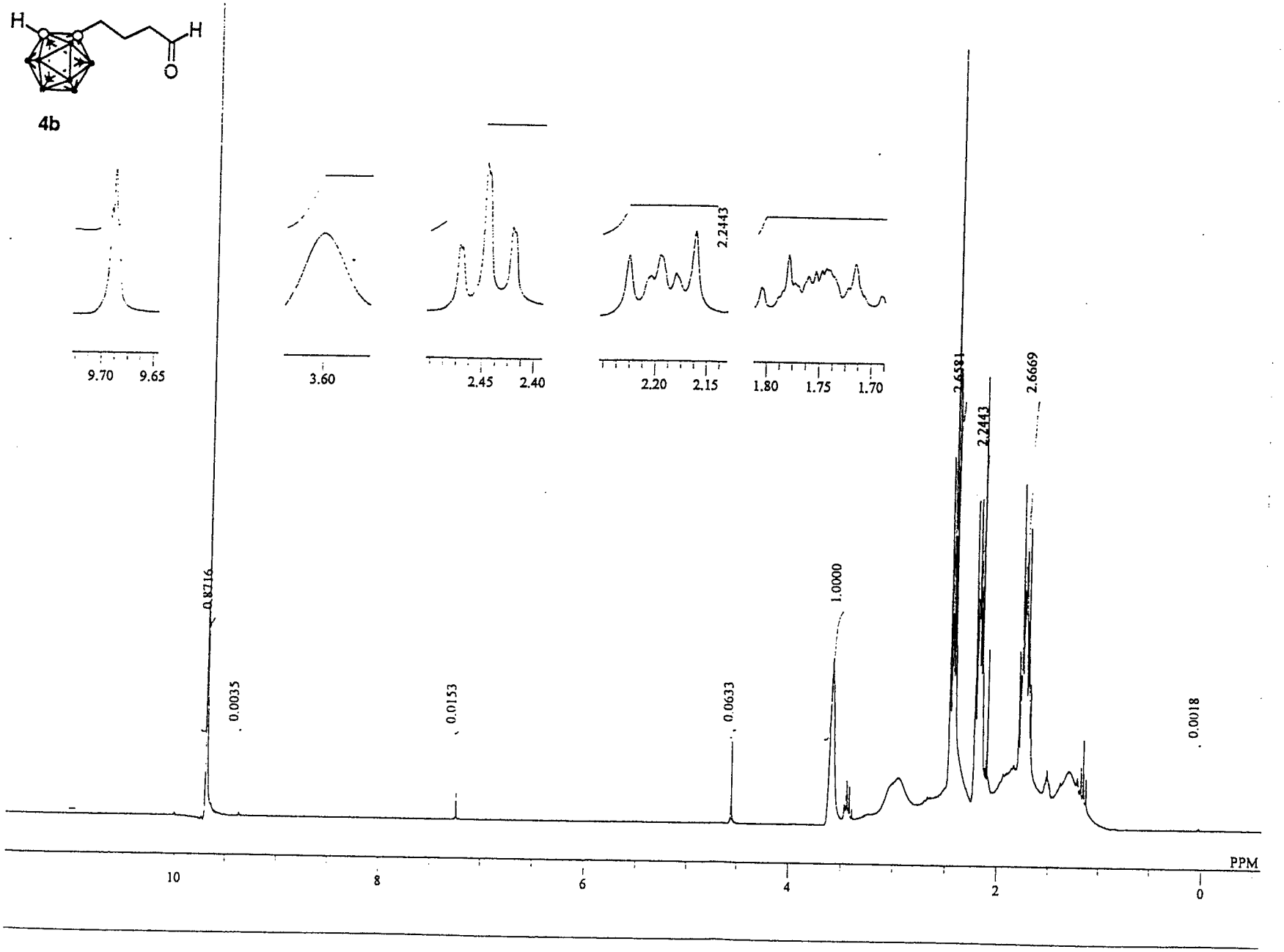
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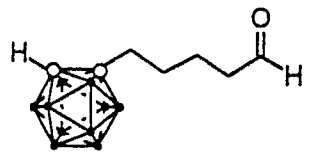

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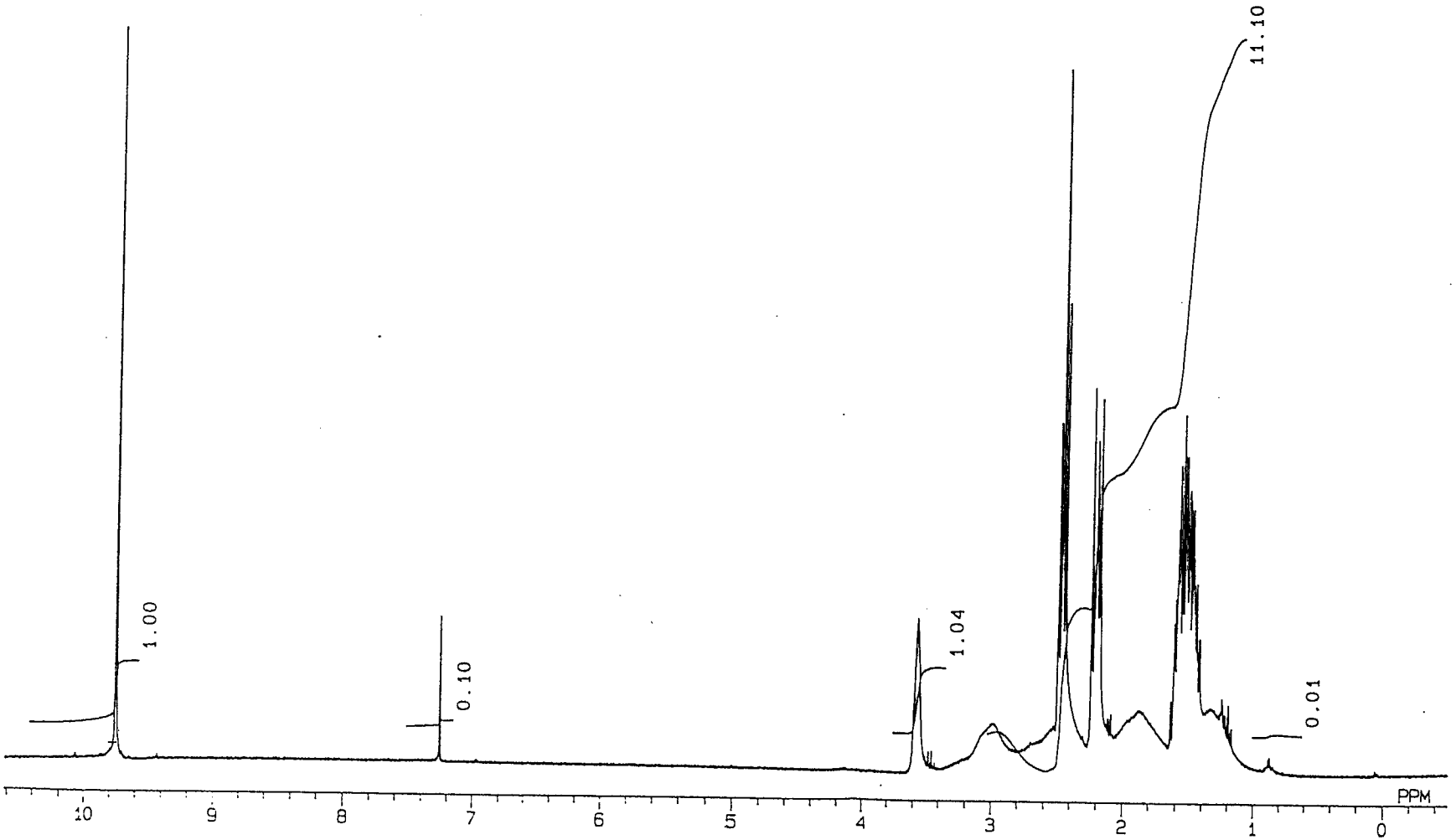

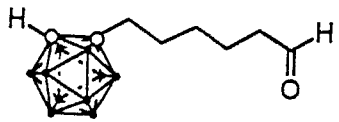

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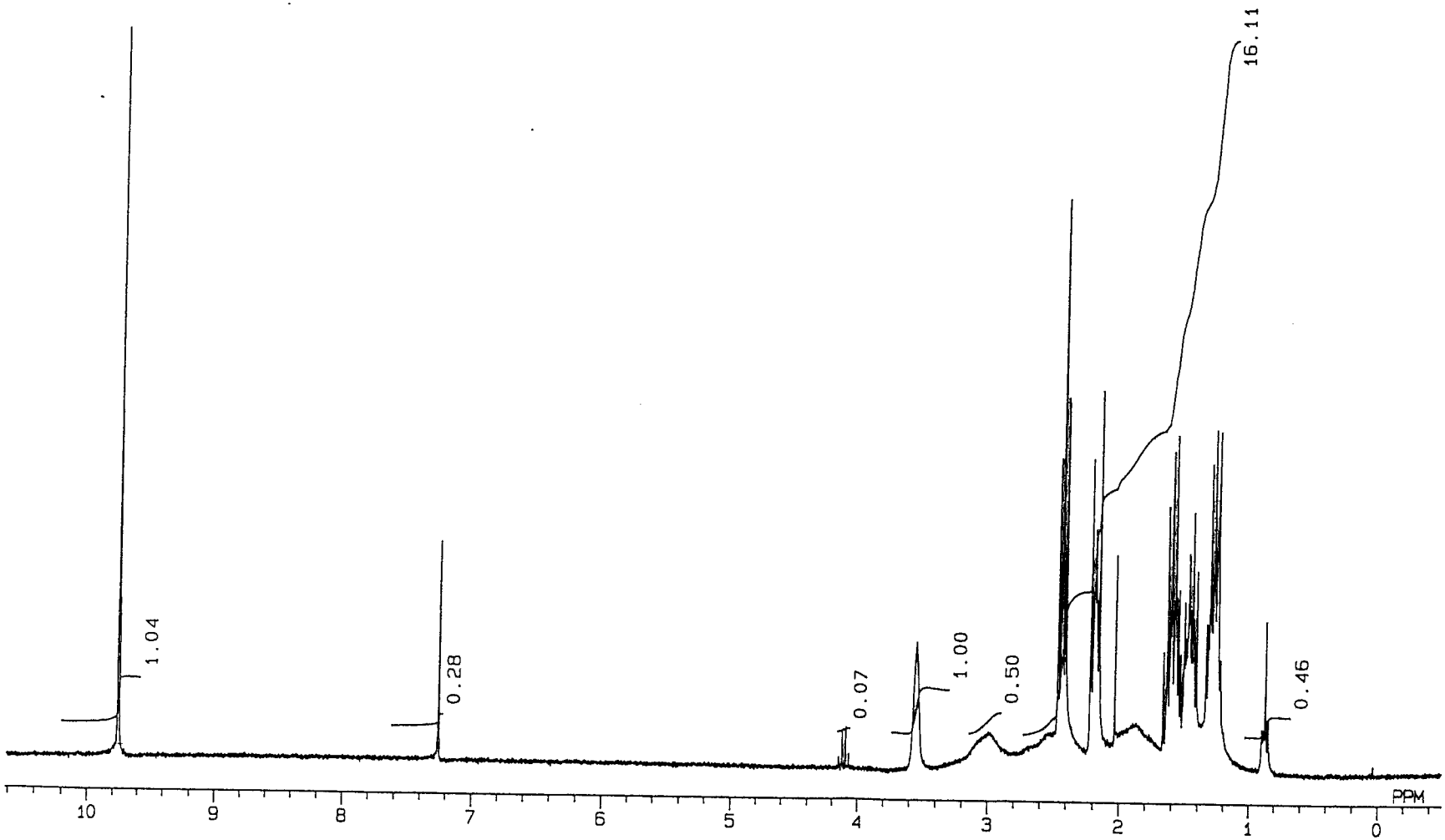

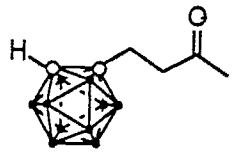

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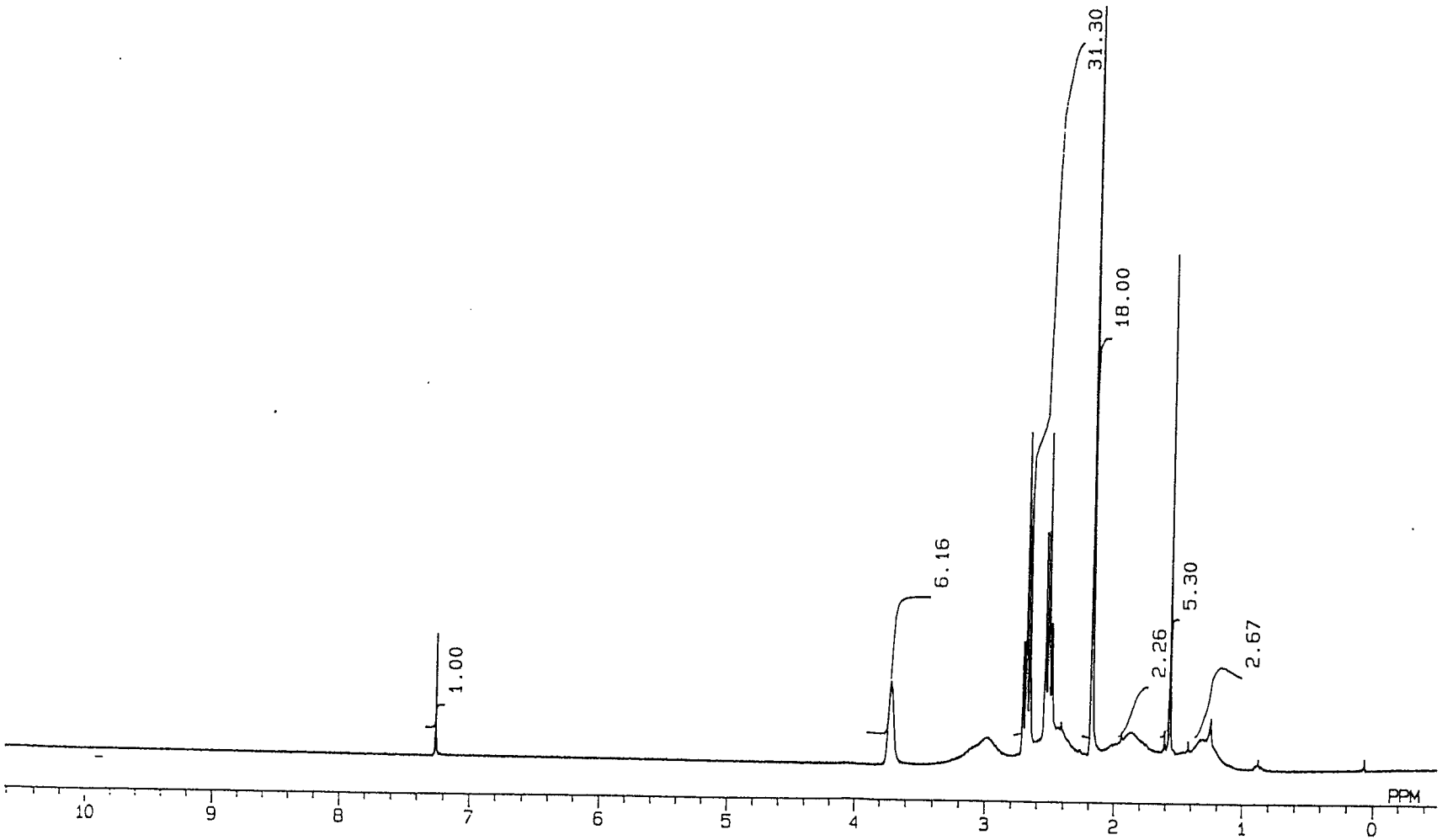

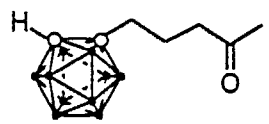

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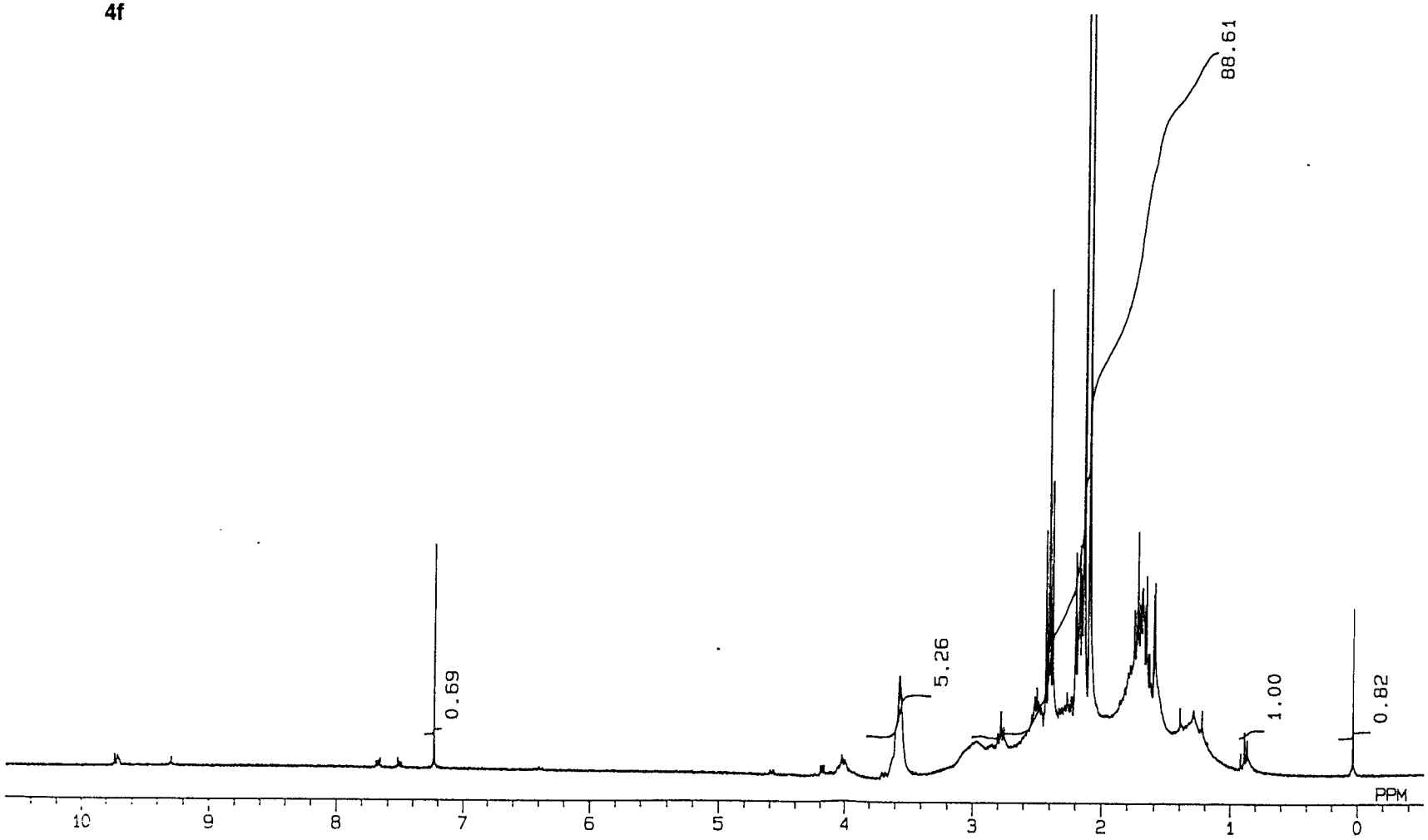

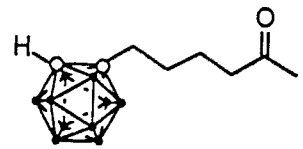

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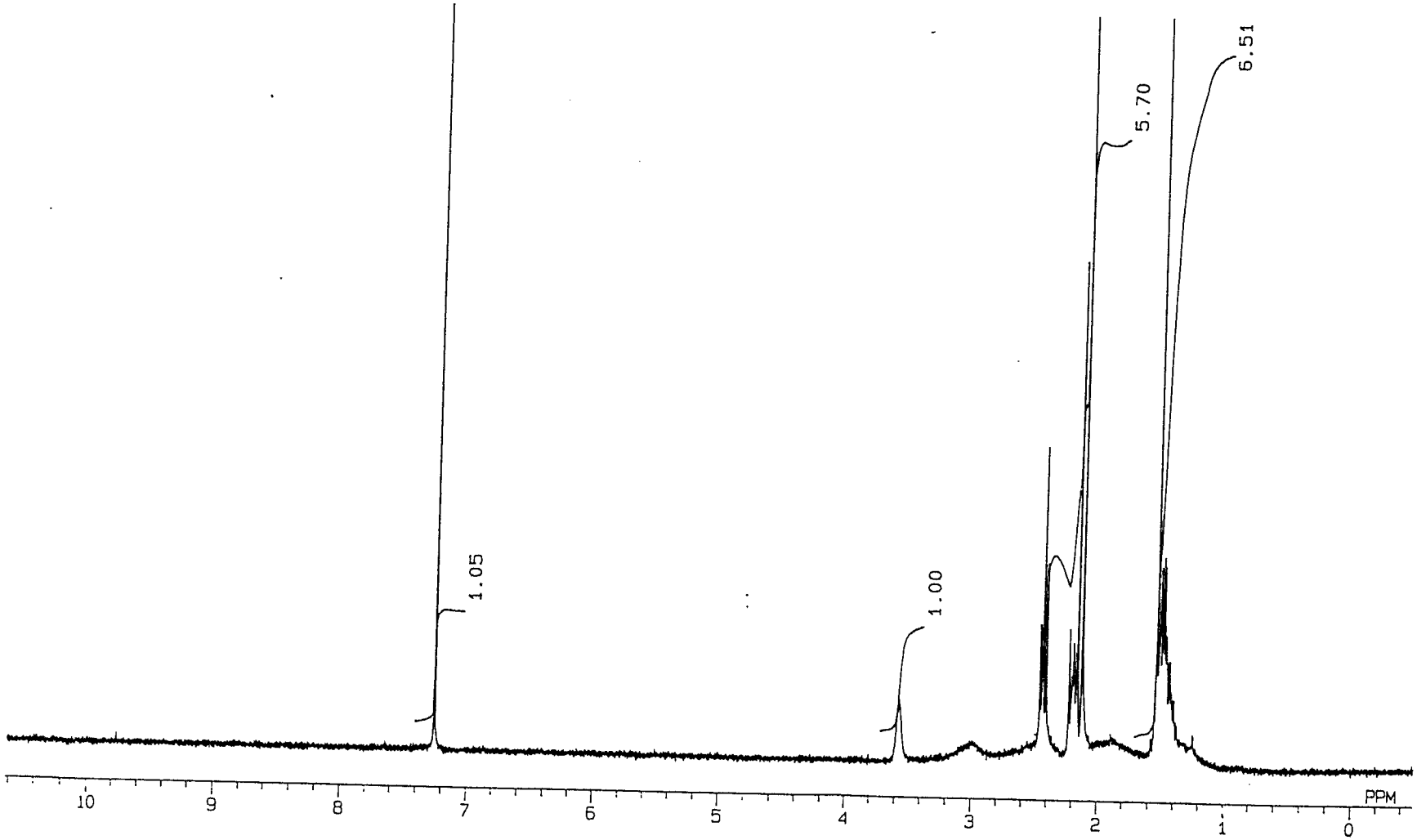

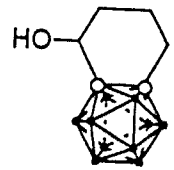

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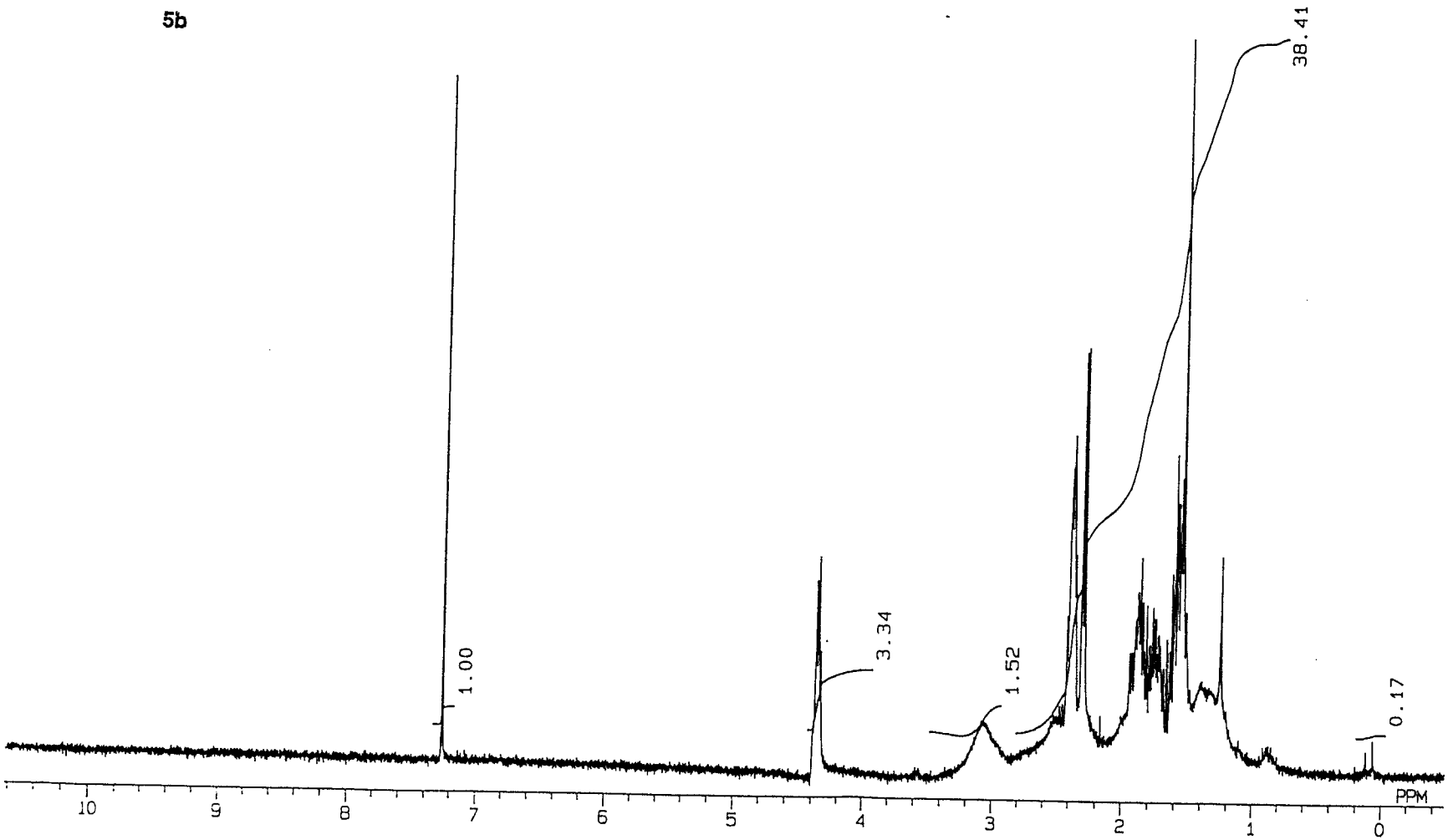

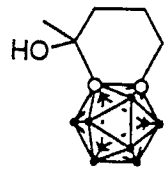

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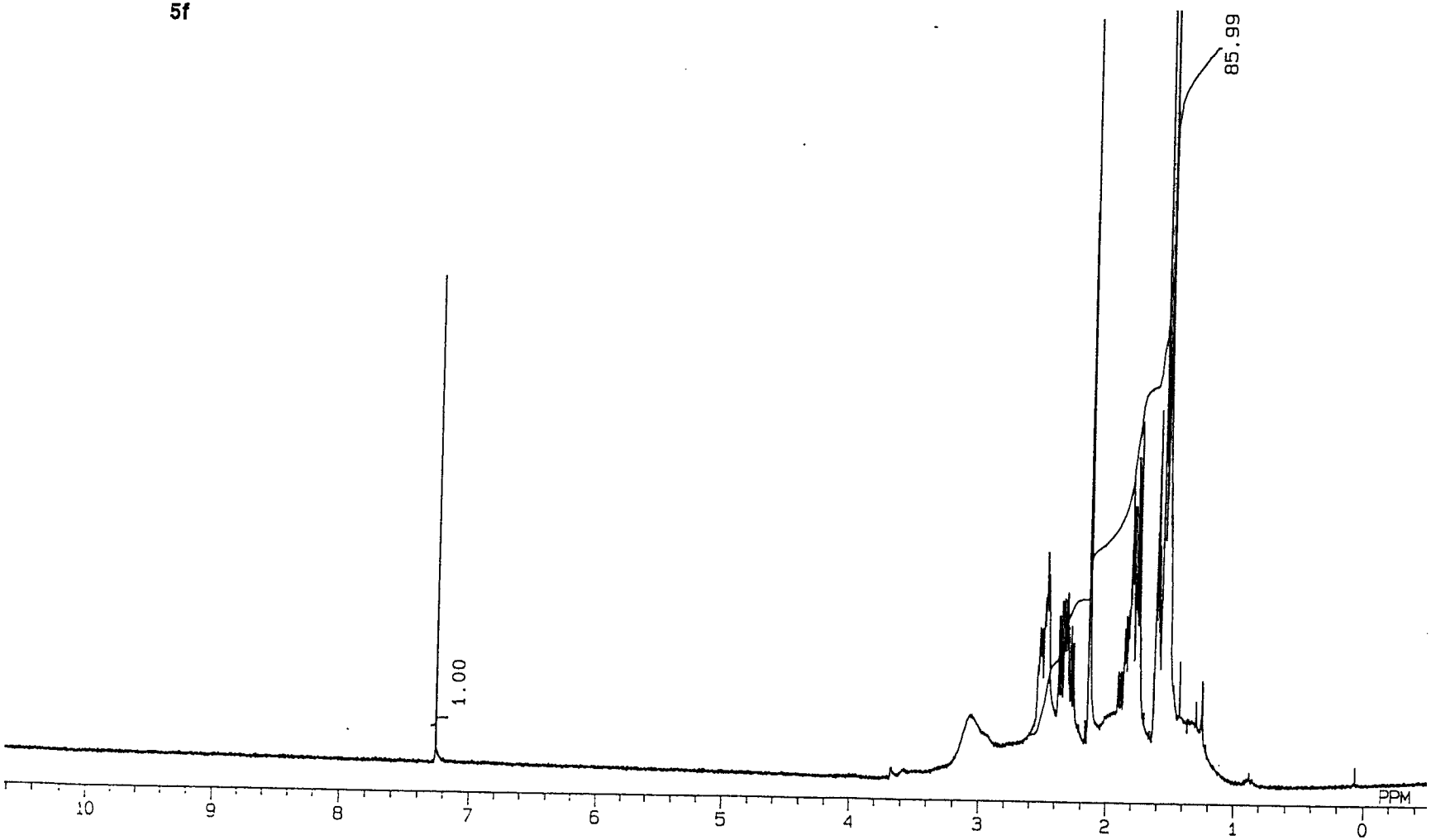

Summary of Structure Determination of Compounds 7a.

A. Crystal Data

Empirical Formula	$C_6H_{18}OB_{10}$
Formula Weight	214.31
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.70 X 0.60 X 0.60 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (30.4 - 45.8°)
Omega Scan Peak Width at Half-height	0.34°
Lattice Parameters	a = 24.945(2) Å b = 22.690(2) Å c = 14.038(1) Å V = 7945(2) Å^3
Space Group	Pbca (#61)
Z value	24
D_{calc}	1.075 g/cm^3
D_{obs}	1.000 g/cm^3
F_{000}	2688.00
μ(CuKα)	3.84 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R
Radiation	CuKα (λ = 1.54178 Å) graphite monochromated

Attenuator	Ni foil (factors = 1.00, 3.58, 13.03, 45.22)
Take-off Angle	6.0°
Detector Aperture	6.0 mm horizontal 6.0 mm vertical
Crystal to Detector Distance	258 mm
Voltage, Current	40kV, 180mA
Temperature	13.0°C
Scan Type	ω-2θ
Scan Rate	4.0°/min (in ω) (up to 2 scans)
Scan Width	$(1.10 + 0.30 \tan \theta)°$
$2\theta_{max}$	125.6°
No. of Reflections Measured	Total: 7044
Corrections	Lorentz-polarization Decay (4.18% decline)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0050
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>4.00σ(I))	3515
No. Variables	460
Reflection/Parameter Ratio	7.64
Residuals: R; Rw	0.079 ; 0.075
Goodness of Fit Indicator	6.71
Max Shift/Error in Final Cycle	0.27

Maximum peak in Final Diff. Map	0.23 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.34 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}
O1	0.4525(2)	-0.0470(2)	-0.1571(3)	7.1(1)
O2	0.4001(2)	-0.0305(2)	0.0092(3)	6.2(1)
O3	0.5562(1)	-0.0627(2)	-0.1010(3)	5.9(1)
C1	0.3911(2)	-0.1071(2)	0.1291(4)	4.4(2)
C2	0.3628(2)	-0.0608(3)	0.0669(4)	5.0(2)
C3	0.3247(2)	-0.0960(3)	0.0047(4)	5.8(2)
C4	0.3115(2)	-0.1559(3)	0.0491(5)	6.3(2)
C5	0.3582(2)	-0.1687(3)	0.1165(4)	5.1(2)
C6	0.3020(3)	-0.2039(3)	-0.0232(5)	9.8(3)
C7	0.3883(2)	-0.0258(3)	-0.2843(4)	5.0(2)
C8	0.3941(3)	-0.0430(3)	-0.3959(5)	5.5(2)
C9	0.4285(3)	-0.0986(3)	-0.4022(6)	7.5(2)
C10	0.4543(3)	-0.1036(3)	-0.3038(6)	7.5(2)
C11	0.4201(3)	-0.0725(3)	-0.2279(5)	6.5(2)
C12	0.4698(3)	-0.0988(3)	-0.4804(5)	9.8(3)
C13	0.6032(2)	-0.1503(2)	-0.1523(4)	4.5(2)
C14	0.6552(2)	-0.1801(2)	-0.0993(4)	4.3(1)
C15	0.6498(3)	-0.1675(2)	0.0076(4)	5.1(2)
C16	0.6020(3)	-0.1240(3)	0.0161(4)	6.1(2)
C17	0.5685(2)	-0.1216(2)	-0.0753(4)	5.0(2)
C18	0.7000(3)	-0.1452(3)	0.0543(4)	7.2(2)
B1	0.3513(3)	-0.1298(3)	0.2228(5)	5.8(2)
B2	0.3954(4)	-0.1649(4)	0.2988(5)	7.5(3)
B3	0.4611(3)	-0.1577(4)	0.2556(6)	7.1(3)

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

atom	x	y	z	B_{eq}
B4	0.4641(3)	-0.1964(4)	0.1460(6)	7.2(3)
B5	0.4209(3)	-0.1626(3)	0.0646(5)	5.5(2)
B6	0.4002(4)	-0.2282(3)	0.1235(6)	7.1(3)
B7	0.4577(3)	-0.1192(4)	0.1459(6)	6.1(2)
B8	0.4251(4)	-0.2249(4)	0.2420(7)	7.8(3)
B9	0.4151(3)	-0.0986(3)	0.2426(6)	6.2(2)
B10	0.3563(4)	-0.2076(4)	0.2188(6)	7.0(3)
B11	0.3853(3)	0.0494(3)	-0.2700(6)	6.1(2)
B12	0.3282(3)	0.0031(4)	-0.2595(6)	7.2(3)
B13	0.3242(4)	0.0686(4)	-0.3267(7)	7.4(3)
B14	0.2934(3)	0.0035(4)	-0.3706(8)	8.4(3)
B15	0.3297(3)	0.0499(4)	-0.4470(7)	7.9(3)
B16	0.3363(4)	-0.0278(4)	-0.4553(7)	8.4(3)
B17	0.3933(3)	0.0171(4)	-0.4680(6)	7.1(3)
B18	0.4276(3)	0.0181(3)	-0.3568(6)	5.7(2)
B19	0.3865(3)	0.0762(3)	-0.3887(7)	7.3(3)
B20	0.3323(3)	-0.0559(4)	-0.3382(7)	7.0(3)
B21	0.6051(3)	-0.2267(3)	-0.1395(5)	5.1(2)
B22	0.5839(3)	-0.1924(3)	-0.2472(6)	6.2(2)
B23	0.6278(3)	-0.2530(3)	-0.2497(6)	5.9(2)
B24	0.6379(4)	-0.1907(4)	-0.3243(5)	6.8(2)
B25	0.6935(3)	-0.2230(3)	-0.2678(6)	6.2(2)
B26	0.6882(3)	-0.1445(3)	-0.2763(5)	6.0(2)
B27	0.7106(3)	-0.1780(3)	-0.1697(5)	5.2(2)

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

atom	x	y	z	B_{eq}
B28	0.6650(3)	-0.1179(3)	-0.1679(5)	4.9(2)
B29	0.6741(3)	-0.2444(3)	-0.1524(5)	5.2(2)
B30	0.6190(3)	-0.1256(3)	-0.2640(5)	5.6(2)
H1	0.3101	-0.1000	0.2291	10.1439
H2	0.3870	-0.1650	0.3710	0.1140
H3	0.5031	-0.1532	0.3014	10.1439
H4	0.5006	-0.2195	0.1094	10.1439
H5	0.4202	-0.1526	-0.0197	10.1439
H6	0.3842	-0.2740	0.0781	10.1439
H7	0.4878	-0.0831	0.1207	10.1439
H8	0.4363	-0.2742	0.2723	10.1439
H9	0.4216	-0.0467	0.2769	10.1439
H10	0.3219	-0.2432	0.2483	10.1439
H11	0.3465	-0.0276	0.1214	10.1439
H12	0.2704	-0.1528	0.0993	10.1439
H13	0.2925	-0.0738	-0.0060	6.7121
H14	0.2700	-0.1944	-0.0574	11.1037
H15	0.3413	-0.1020	-0.0570	6.7121
H16	0.2974	-0.2397	0.0097	11.1037
H17	0.3313	-0.2053	-0.0645	11.1037
H18	0.4010	0.0000	0.0168	10.1439
H19	0.4071	0.0711	-0.1948	10.1439
H20	0.3100	-0.0020	-0.1930	0.1140
H21	0.3022	0.1129	-0.2907	10.1439

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

atom	x	y	z	B_{eq}
H22	0.2469	-0.0034	-0.3701	10.1439
H23	0.3019	0.0730	-0.5055	10.1439
H24	0.3224	-0.0533	-0.5175	10.1439
H25	0.4203	0.0131	-0.5499	10.1439
H26	0.4769	0.0127	-0.3505	10.1439
H27	0.3251	-0.1119	-0.3179	10.1439
H28	0.4020	0.1246	-0.3978	10.1439
H29	0.3971	-0.0994	-0.1975	8.0005
H30	0.4572	-0.1438	-0.2844	9.2251
H31	0.4894	-0.0863	-0.3018	9.2251
H32	0.4041	-0.1422	-0.4140	10.1439
H33	0.4930	-0.0640	-0.4737	12.0929
H34	0.4529	-0.0969	-0.5396	12.0929
H35	0.4924	-0.1324	-0.4763	12.0929
H36	0.4701	-0.0724	-0.1312	8.2517
H37	0.5744	-0.2483	-0.0819	10.1439
H38	0.5430	-0.1970	-0.2490	0.1140
H39	0.6190	-0.3039	-0.2703	10.1439
H40	0.7147	-0.1145	-0.3297	10.1439
H41	0.7206	-0.2503	-0.3090	10.1439
H42	0.7534	-0.1703	-0.1407	10.1439
H43	0.6913	-0.2829	-0.1088	10.1439
H44	0.6720	-0.0755	-0.1283	10.1439
H45	0.6320	-0.1940	-0.3970	0.1140

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

atom	x	y	z	B_{eq}
H46	0.6017	-0.0807	-0.2899	10.1439
H47	0.5328	-0.1493	-0.0611	10.1439
H48	0.5799	-0.1269	0.0840	10.1439
H49	0.6185	-0.0866	0.0189	10.1439
H50	0.6337	-0.2065	0.0434	10.1439
H51	0.7157	-0.1087	0.0196	10.1439
H52	0.6920	-0.1305	0.1208	10.1439
H53	0.7316	-0.1682	0.0399	10.1439
H54	0.5391	-0.0481	-0.0586	6.7325

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	0.074(3)	0.113(4)	0.083(3)	0.023(3)	-0.024(3)	0.019(3)
O2	0.105(4)	0.046(3)	0.086(3)	-0.006(2)	0.006(3)	0.004(2)
O3	0.078(3)	0.054(3)	0.094(3)	0.017(2)	0.002(3)	-0.023(2)
C1	0.052(4)	0.052(4)	0.065(4)	-0.001(3)	-0.002(3)	-0.010(3)
C2	0.070(4)	0.051(4)	0.069(4)	0.007(4)	-0.003(4)	0.000(4)
C3	0.058(4)	0.086(5)	0.079(5)	0.007(4)	-0.009(4)	0.014(4)
C4	0.068(5)	0.089(5)	0.081(5)	-0.021(4)	-0.016(4)	0.005(4)
C5	0.064(4)	0.056(4)	0.075(5)	-0.011(4)	-0.011(4)	0.005(4)
C6	0.142(7)	0.117(6)	0.113(7)	-0.045(6)	-0.056(6)	-0.001(6)
C7	0.053(4)	0.073(4)	0.065(5)	0.010(4)	0.003(4)	0.011(4)
C8	0.074(5)	0.069(4)	0.068(5)	-0.003(4)	-0.007(4)	-0.007(4)
C9	0.095(6)	0.073(5)	0.115(7)	0.000(5)	-0.002(6)	-0.022(5)
C10	0.091(6)	0.073(5)	0.123(7)	0.029(4)	0.002(5)	0.001(5)
C11	0.072(5)	0.081(5)	0.095(6)	0.015(4)	0.012(5)	0.032(5)
C12	0.106(6)	0.153(8)	0.113(6)	0.011(6)	0.028(5)	-0.053(6)
C13	0.055(4)	0.044(3)	0.072(4)	0.001(3)	0.002(4)	-0.003(3)
C14	0.057(4)	0.049(4)	0.059(4)	0.003(3)	0.003(3)	0.002(3)
C15	0.082(5)	0.059(4)	0.055(4)	0.000(4)	0.002(4)	0.004(4)
C16	0.091(5)	0.080(5)	0.061(4)	0.017(4)	0.011(4)	-0.010(4)
C17	0.057(4)	0.052(4)	0.080(5)	0.006(3)	0.008(4)	-0.016(4)
C18	0.098(6)	0.100(6)	0.078(5)	0.007(5)	-0.015(4)	-0.016(4)
B1	0.068(5)	0.088(6)	0.065(6)	-0.001(5)	0.008(5)	-0.002(5)
B2	0.108(7)	0.125(8)	0.053(5)	-0.007(7)	0.000(5)	0.015(6)
B3	0.073(6)	0.102(7)	0.094(7)	-0.002(5)	-0.022(5)	0.007(6)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
B4	0.084(6)	0.092(7)	0.099(7)	0.038(5)	0.004(6)	0.006(6)
B5	0.067(5)	0.064(5)	0.077(6)	0.014(4)	0.002(5)	-0.005(5)
B6	0.112(7)	0.054(5)	0.103(7)	0.001(5)	-0.011(7)	0.002(5)
B7	0.052(5)	0.084(6)	0.095(7)	-0.004(4)	-0.010(5)	-0.003(5)
B8	0.116(8)	0.080(6)	0.098(8)	0.019(6)	-0.012(7)	0.013(6)
B9	0.084(6)	0.085(6)	0.066(6)	-0.013(5)	-0.013(5)	-0.006(5)
B10	0.110(8)	0.078(6)	0.079(7)	-0.023(6)	-0.008(6)	0.021(5)
B11	0.084(6)	0.069(5)	0.080(6)	0.004(5)	0.000(5)	-0.006(5)
B12	0.075(6)	0.113(7)	0.085(6)	0.021(6)	0.038(5)	0.029(6)
B13	0.085(7)	0.094(7)	0.104(8)	0.024(6)	0.002(6)	0.017(6)
B14	0.051(5)	0.127(8)	0.142(9)	-0.002(6)	-0.013(6)	0.041(8)
B15	0.074(6)	0.117(8)	0.108(8)	0.004(6)	-0.029(6)	0.034(7)
B16	0.085(7)	0.129(9)	0.105(8)	-0.024(7)	-0.039(6)	-0.012(7)
B17	0.087(7)	0.109(7)	0.074(6)	-0.005(6)	-0.005(5)	0.019(6)
B18	0.070(5)	0.063(5)	0.082(6)	-0.016(4)	0.000(5)	0.002(5)
B19	0.090(7)	0.069(6)	0.118(8)	-0.003(5)	-0.017(6)	0.022(6)
B20	0.062(5)	0.078(6)	0.126(8)	-0.022(5)	-0.015(6)	0.016(6)
B21	0.071(5)	0.043(4)	0.082(6)	0.001(4)	-0.002(5)	-0.001(4)
B22	0.059(5)	0.073(5)	0.103(7)	-0.001(4)	-0.007(5)	-0.022(5)
B23	0.081(6)	0.059(5)	0.085(6)	0.016(5)	0.003(5)	-0.008(5)
B24	0.122(8)	0.091(6)	0.045(5)	0.015(6)	-0.001(5)	-0.016(5)
B25	0.090(6)	0.077(6)	0.071(6)	0.013(5)	0.014(5)	-0.008(5)
B26	0.091(6)	0.073(5)	0.062(5)	-0.007(5)	0.018(5)	0.005(4)
B27	0.056(4)	0.064(5)	0.079(6)	-0.004(4)	0.012(4)	-0.001(5)
B28	0.068(5)	0.053(5)	0.065(5)	-0.007(4)	0.003(4)	0.009(4)
B29	0.069(5)	0.051(4)	0.076(6)	0.006(4)	0.008(5)	-0.007(4)
B30	0.096(6)	0.061(5)	0.054(5)	-0.003(5)	-0.003(5)	0.004(4)

The general temperature factor expression:

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