SUPPORTING INFORMATION

for

"X-Ray Crystallographic and Mass Spectrometric Probing of the Conformational and Ionophoric Properties of Stereoisomeric Hexatetrahydrofuranylhexane Segments"

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X-Ray Crystallographic Details for **23**.

The data collection crystal was a colorless chunk cut from a very large crystal. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 3.9, which means that 90% of the reflections were measured at least 3.9 times. Phi and omega scans with a frame width of 1.0˚ were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.038.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on 4948 intensities and 271 variables and resulted in agreement factors of R1(F) = 0.062 and wR2(F^2) = 0.116. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.043 for 3787 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.55 and -0.37 e/$Å^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

References

(1) DENZO: Otwinowski, Z. & Minor, W., Methods in Enzymology, Vol 276: Macromolecular Crystallography, part A, 307-326, (1997), Carter, Jr., C. W. & Sweet, R. M., Eds., Academic Press.

(2) SHELXS-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.

(3) SHELXL-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.

(4) WinGX-Version 1.70.01: Farrugia, L. J., J. Appl. Cryst., (1999), 32, 837-838.

(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 1. Crystallographic data for **23**.

Empirical formula	C24 H38 O6
Formula weight	422.54
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P\ 2_1/n$
Unit cell dimensions	a = 9.681(1) Å
	b = 14.382(1) Å
	c = 15.626(2) Å
	β= 97.944(4)°
Volume	2154.9(4) $Å^3$
Z	4
Density (calculated)	1.302 Mg/m^3
Absorption coefficient	0.092 mm^{-1}
F(000)	920
Crystal size	0.27 x 0.42 x 0.46 mm^3
Theta range for data collection	2.34 to 27.47°
Index ranges	-12<=h<=12, -18<=k<=18, -20<=l<=20
Reflections collected	39386
Independent reflections	4948 [R(int) = 0.038]
Completeness to theta = 27.47°	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4948 / 0 / 271
Goodness-of-fit on F^2	1.055
Final R indices [I>2sigma(I)]	R1 = 0.0433, wR2 = 0.1081
R indices (all data)	R1 = 0.0618, wR2 = 0.1161
Largest diff. peak and hole	0.550 and -0.369 e/$Å^3$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **23**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	6556(1)	1387(1)	3455(1)	30(1)
O(2)	10367(1)	1372(1)	3861(1)	25(1)
O(3)	10259(1)	2371(1)	2371(1)	21(1)
O(4)	9441(1)	1177(1)	1093(1)	20(1)
O(5)	10616(1)	-455(1)	1534(1)	24(1)
O(6)	13140(1)	95(1)	2597(1)	28(1)
C(1)	7942(1)	1038(1)	3721(1)	24(1)
C(2)	8039(2)	855(1)	4687(1)	32(1)
C(3)	6543(2)	581(1)	4774(1)	35(1)
C(4)	5711(2)	1216(1)	4129(1)	38(1)
C(5)	9037(1)	1718(1)	3449(1)	21(1)
C(6)	8884(2)	2697(1)	3841(1)	27(1)
C(7)	10130(2)	2799(1)	4547(1)	37(1)
C(8)	11176(2)	2134(1)	4257(1)	37(1)
C(9)	9083(1)	1771(1)	2453(1)	18(1)
C(10)	7790(1)	2306(1)	1983(1)	22(1)
C(11)	8360(2)	3219(1)	1668(1)	29(1)
C(12)	9905(2)	3032(1)	1687(1)	26(1)
C(13)	9297(1)	837(1)	1946(1)	18(1)
C(14)	7969(1)	219(1)	1811(1)	23(1)
C(15)	7681(2)	22(1)	840(1)	32(1)
C(16)	8487(2)	756(1)	432(1)	30(1)
C(17)	10630(1)	218(1)	2230(1)	19(1)
C(18)	10613(1)	-377(1)	3055(1)	25(1)
C(19)	10925(2)	-1368(1)	2794(1)	36(1)
C(20)	10549(2)	-1384(1)	1839(1)	48(1)
C(21)	12029(1)	746(1)	2285(1)	21(1)
C(22)	12423(1)	1142(1)	1440(1)	24(1)
C(23)	13982(2)	944(1)	1510(1)	35(1)
C(24)	14086(2)	25(1)	1978(1)	39(1)

Table 3. Bond lengths [Å] and angles [°] for **23**.

O(1)-C(1)	1.4395(17)
O(1)-C(4)	1.4410(17)
O(2)-C(8)	1.4361(18)
O(2)-C(5)	1.4455(16)
O(3)-C(12)	1.4344(16)
O(3)-C(9)	1.4488(15)
O(4)-C(16)	1.4224(16)
O(4)-C(13)	1.4436(15)
O(5)-C(20)	1.4223(18)
O(5)-C(17)	1.4552(16)
O(6)-C(24)	1.4249(18)
O(6)-C(21)	1.4583(16)
C(1)-C(2)	1.5227(19)
C(1)-C(5)	1.5444(19)
C(1)-H(1)	1.0000
C(2)-C(3)	1.526(2)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.508(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.5512(19)
C(5)-C(9)	1.5647(18)
C(6)-C(7)	1.524(2)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.507(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.5629(18)

C(9)-C(13)	1.5873(18)
C(10)-C(11)	1.533(2)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.516(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-C(14)	1.5535(17)
C(13)-C(17)	1.5806(18)
C(14)-C(15)	1.532(2)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.505(2)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-C(21)	1.5450(18)
C(17)-C(18)	1.5485(18)
C(18)-C(19)	1.525(2)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.487(3)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-C(22)	1.5326(19)
C(21)-H(21)	1.0000
C(22)-C(23)	1.525(2)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-C(24)	1.507(2)
C(23)-H(23A)	0.9900

C(23)-H(23B)	0.9900
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900

C(1)-O(1)-C(4)	109.33(11)
C(8)-O(2)-C(5)	109.20(11)
C(12)-O(3)-C(9)	110.56(10)
C(16)-O(4)-C(13)	113.00(10)
C(20)-O(5)-C(17)	111.72(11)
C(24)-O(6)-C(21)	109.37(11)
O(1)-C(1)-C(2)	105.83(11)
O(1)-C(1)-C(5)	110.36(11)
C(2)-C(1)-C(5)	115.54(12)
O(1)-C(1)-H(1)	108.3
C(2)-C(1)-H(1)	108.3
C(5)-C(1)-H(1)	108.3
C(1)-C(2)-C(3)	101.76(12)
C(1)-C(2)-H(2A)	111.4
C(3)-C(2)-H(2A)	111.4
C(1)-C(2)-H(2B)	111.4
C(3)-C(2)-H(2B)	111.4
H(2A)-C(2)-H(2B)	109.3
C(4)-C(3)-C(2)	102.10(12)
C(4)-C(3)-H(3A)	111.3
C(2)-C(3)-H(3A)	111.3
C(4)-C(3)-H(3B)	111.3
C(2)-C(3)-H(3B)	111.3
H(3A)-C(3)-H(3B)	109.2
O(1)-C(4)-C(3)	106.66(12)
O(1)-C(4)-H(4A)	110.4
C(3)-C(4)-H(4A)	110.4
O(1)-C(4)-H(4B)	110.4
C(3)-C(4)-H(4B)	110.4
H(4A)-C(4)-H(4B)	108.6
O(2)-C(5)-C(1)	105.23(11)
O(2)-C(5)-C(6)	105.29(10)

C(1)-C(5)-C(6)	111.11(11)
O(2)-C(5)-C(9)	108.30(10)
C(1)-C(5)-C(9)	114.77(11)
C(6)-C(5)-C(9)	111.46(11)
C(7)-C(6)-C(5)	105.32(12)
C(7)-C(6)-H(6A)	110.7
C(5)-C(6)-H(6A)	110.7
C(7)-C(6)-H(6B)	110.7
C(5)-C(6)-H(6B)	110.7
H(6A)-C(6)-H(6B)	108.8
C(8)-C(7)-C(6)	102.63(12)
C(8)-C(7)-H(7A)	111.2
C(6)-C(7)-H(7A)	111.2
C(8)-C(7)-H(7B)	111.2
C(6)-C(7)-H(7B)	111.2
H(7A)-C(7)-H(7B)	109.2
O(2)-C(8)-C(7)	105.36(12)
O(2)-C(8)-H(8A)	110.7
C(7)-C(8)-H(8A)	110.7
O(2)-C(8)-H(8B)	110.7
C(7)-C(8)-H(8B)	110.7
H(8A)-C(8)-H(8B)	108.8
O(3)-C(9)-C(10)	104.66(10)
O(3)-C(9)-C(5)	104.38(10)
C(10)-C(9)-C(5)	111.12(10)
O(3)-C(9)-C(13)	107.52(9)
C(10)-C(9)-C(13)	109.45(10)
C(5)-C(9)-C(13)	118.61(11)
C(11)-C(10)-C(9)	105.81(11)
C(11)-C(10)-H(10A)	110.6
C(9)-C(10)-H(10A)	110.6
C(11)-C(10)-H(10B)	110.6
C(9)-C(10)-H(10B)	110.6
H(10A)-C(10)-H(10B)	108.7
C(12)-C(11)-C(10)	103.83(11)
C(12)-C(11)-H(11A)	111.0

C(10)-C(11)-H(11A)	111.0
C(12)-C(11)-H(11B)	111.0
C(10)-C(11)-H(11B)	111.0
H(11A)-C(11)-H(11B)	109.0
O(3)-C(12)-C(11)	105.45(11)
O(3)-C(12)-H(12A)	110.7
C(11)-C(12)-H(12A)	110.7
O(3)-C(12)-H(12B)	110.7
C(11)-C(12)-H(12B)	110.7
H(12A)-C(12)-H(12B)	108.8
O(4)-C(13)-C(14)	104.62(10)
O(4)-C(13)-C(17)	105.68(9)
C(14)-C(13)-C(17)	110.11(11)
O(4)-C(13)-C(9)	102.18(10)
C(14)-C(13)-C(9)	112.91(10)
C(17)-C(13)-C(9)	119.62(10)
C(15)-C(14)-C(13)	105.90(11)
C(15)-C(14)-H(14A)	110.6
C(13)-C(14)-H(14A)	110.6
C(15)-C(14)-H(14B)	110.6
C(13)-C(14)-H(14B)	110.6
H(14A)-C(14)-H(14B)	108.7
C(16)-C(15)-C(14)	104.82(11)
C(16)-C(15)-H(15A)	110.8
C(14)-C(15)-H(15A)	110.8
C(16)-C(15)-H(15B)	110.8
C(14)-C(15)-H(15B)	110.8
H(15A)-C(15)-H(15B)	108.9
O(4)-C(16)-C(15)	108.26(12)
O(4)-C(16)-H(16A)	110.0
C(15)-C(16)-H(16A)	110.0
O(4)-C(16)-H(16B)	110.0
C(15)-C(16)-H(16B)	110.0
H(16A)-C(16)-H(16B)	108.4
O(5)-C(17)-C(21)	106.60(10)
O(5)-C(17)-C(18)	104.73(10)

C(21)-C(17)-C(18)	109.60(11)
O(5)-C(17)-C(13)	104.01(10)
C(21)-C(17)-C(13)	114.45(10)
C(18)-C(17)-C(13)	116.33(10)
C(19)-C(18)-C(17)	105.62(12)
C(19)-C(18)-H(18A)	110.6
C(17)-C(18)-H(18A)	110.6
C(19)-C(18)-H(18B)	110.6
C(17)-C(18)-H(18B)	110.6
H(18A)-C(18)-H(18B)	108.7
C(20)-C(19)-C(18)	104.58(12)
C(20)-C(19)-H(19A)	110.8
C(18)-C(19)-H(19A)	110.8
C(20)-C(19)-H(19B)	110.8
C(18)-C(19)-H(19B)	110.8
H(19A)-C(19)-H(19B)	108.9
O(5)-C(20)-C(19)	107.85(13)
O(5)-C(20)-H(20A)	110.1
C(19)-C(20)-H(20A)	110.1
O(5)-C(20)-H(20B)	110.1
C(19)-C(20)-H(20B)	110.1
H(20A)-C(20)-H(20B)	108.5
O(6)-C(21)-C(22)	105.45(10)
O(6)-C(21)-C(17)	107.64(10)
C(22)-C(21)-C(17)	117.14(11)
O(6)-C(21)-H(21)	108.8
C(22)-C(21)-H(21)	108.8
C(17)-C(21)-H(21)	108.8
C(23)-C(22)-C(21)	103.39(11)
C(23)-C(22)-H(22A)	111.1
C(21)-C(22)-H(22A)	111.1
C(23)-C(22)-H(22B)	111.1
C(21)-C(22)-H(22B)	111.1
H(22A)-C(22)-H(22B)	109.0
C(24)-C(23)-C(22)	101.37(12)
C(24)-C(23)-H(23A)	111.5

C(22)-C(23)-H(23A)	111.5
C(24)-C(23)-H(23B)	111.5
C(22)-C(23)-H(23B)	111.5
H(23A)-C(23)-H(23B)	109.3
O(6)-C(24)-C(23)	105.17(12)
O(6)-C(24)-H(24A)	110.7
C(23)-C(24)-H(24A)	110.7
O(6)-C(24)-H(24B)	110.7
C(23)-C(24)-H(24B)	110.7
H(24A)-C(24)-H(24B)	108.8

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for **23**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	20(1)	44(1)	27(1)	6(1)	8(1)	0(1)
O(2)	21(1)	34(1)	20(1)	-1(1)	0(1)	0(1)
O(3)	20(1)	20(1)	22(1)	0(1)	5(1)	-4(1)
O(4)	23(1)	24(1)	13(1)	-1(1)	2(1)	-4(1)
O(5)	29(1)	18(1)	27(1)	-4(1)	9(1)	-2(1)
O(6)	19(1)	32(1)	36(1)	9(1)	6(1)	4(1)
C(1)	22(1)	29(1)	22(1)	0(1)	7(1)	1(1)
C(2)	31(1)	44(1)	24(1)	7(1)	10(1)	0(1)
C(3)	35(1)	42(1)	31(1)	2(1)	18(1)	-2(1)
C(4)	28(1)	53(1)	38(1)	7(1)	18(1)	0(1)
C(5)	19(1)	25(1)	18(1)	-2(1)	3(1)	1(1)
C(6)	31(1)	28(1)	23(1)	-7(1)	8(1)	-1(1)
C(7)	42(1)	41(1)	28(1)	-11(1)	3(1)	-8(1)
C(8)	31(1)	49(1)	29(1)	-9(1)	-3(1)	-6(1)
C(9)	17(1)	19(1)	18(1)	-1(1)	4(1)	-2(1)
C(10)	22(1)	24(1)	22(1)	-1(1)	4(1)	2(1)
C(11)	34(1)	26(1)	27(1)	3(1)	1(1)	2(1)
C(12)	35(1)	20(1)	25(1)	1(1)	9(1)	-3(1)
C(13)	18(1)	20(1)	16(1)	1(1)	5(1)	-2(1)
C(14)	20(1)	24(1)	25(1)	-2(1)	5(1)	-5(1)
C(15)	31(1)	38(1)	28(1)	-9(1)	1(1)	-11(1)
C(16)	32(1)	37(1)	20(1)	-4(1)	-5(1)	-6(1)
C(17)	20(1)	19(1)	17(1)	-2(1)	5(1)	-1(1)
C(18)	23(1)	26(1)	26(1)	7(1)	7(1)	2(1)
C(19)	38(1)	24(1)	46(1)	9(1)	7(1)	2(1)
C(20)	76(1)	20(1)	51(1)	0(1)	22(1)	3(1)
C(21)	18(1)	20(1)	24(1)	1(1)	4(1)	2(1)
C(22)	22(1)	25(1)	28(1)	2(1)	8(1)	-2(1)
C(23)	25(1)	34(1)	49(1)	10(1)	17(1)	2(1)
C(24)	27(1)	35(1)	58(1)	10(1)	22(1)	8(1)

Table 5. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **23**.

	x	y	z	U(eq)
H(1)	8038	430	3423	29
H(2A)	8327	1420	5027	39
H(2B)	8699	344	4871	39
H(3A)	6364	-81	4622	42
H(3B)	6329	693	5366	42
H(4A)	4820	919	3888	46
H(4B)	5502	1808	4409	46
H(6A)	8900	3184	3395	32
H(6B)	7999	2746	4088	32
H(7A)	10491	3445	4576	45
H(7B)	9884	2621	5118	45
H(8A)	11709	2437	3837	44
H(8B)	11838	1916	4757	44
H(10A)	7325	1939	1490	27
H(10B)	7108	2430	2387	27
H(11A)	7915	3369	1075	35
H(11B)	8202	3740	2056	35
H(12A)	10096	2772	1129	31
H(12B)	10450	3611	1805	31
H(14A)	8128	-368	2140	27
H(14B)	7170	548	2007	27
H(15A)	8007	-607	708	39
H(15B)	6671	71	628	39
H(16A)	8999	472	-8	36
H(16B)	7841	1230	145	36
H(18A)	11332	-157	3524	30
H(18B)	9690	-343	3257	30
H(19A)	11925	-1518	2960	43
H(19B)	10357	-1821	3071	43
H(20A)	11206	-1784	1575	57
H(20B)	9595	-1635	1683	57

H(21)	12029	1263	2712	25
H(22A)	11903	825	932	29
H(22B)	12234	1817	1396	29
H(23A)	14287	890	932	42
H(23B)	14539	1430	1847	42
H(24A)	13819	-493	1571	46
H(24B)	15048	-82	2268	46

Table 6. Torsion angles [°] for **23**.

C(4)-O(1)-C(1)-C(2)	-13.93(16)
C(4)-O(1)-C(1)-C(5)	-139.61(13)
O(1)-C(1)-C(2)-C(3)	31.99(15)
C(5)-C(1)-C(2)-C(3)	154.43(12)
C(1)-C(2)-C(3)-C(4)	-37.12(16)
C(1)-O(1)-C(4)-C(3)	-10.37(17)
C(2)-C(3)-C(4)-O(1)	30.04(17)
C(8)-O(2)-C(5)-C(1)	-133.96(12)
C(8)-O(2)-C(5)-C(6)	-16.48(14)
C(8)-O(2)-C(5)-C(9)	102.85(12)
O(1)-C(1)-C(5)-O(2)	171.39(10)
C(2)-C(1)-C(5)-O(2)	51.40(15)
O(1)-C(1)-C(5)-C(6)	57.93(15)
C(2)-C(1)-C(5)-C(6)	-62.06(16)
O(1)-C(1)-C(5)-C(9)	-69.66(14)
C(2)-C(1)-C(5)-C(9)	170.35(12)
O(2)-C(5)-C(6)-C(7)	-5.83(14)
C(1)-C(5)-C(6)-C(7)	107.59(13)
C(9)-C(5)-C(6)-C(7)	-123.04(12)
C(5)-C(6)-C(7)-C(8)	24.32(16)
C(5)-O(2)-C(8)-C(7)	32.81(15)
C(6)-C(7)-C(8)-O(2)	-34.75(16)
C(12)-O(3)-C(9)-C(10)	-18.80(13)
C(12)-O(3)-C(9)-C(5)	-135.65(10)
C(12)-O(3)-C(9)-C(13)	97.54(11)
O(2)-C(5)-C(9)-O(3)	-57.36(12)
C(1)-C(5)-C(9)-O(3)	-174.57(10)
C(6)-C(5)-C(9)-O(3)	58.01(13)
O(2)-C(5)-C(9)-C(10)	-169.64(10)
C(1)-C(5)-C(9)-C(10)	73.14(14)
C(6)-C(5)-C(9)-C(10)	-54.27(14)
O(2)-C(5)-C(9)-C(13)	62.24(14)
C(1)-C(5)-C(9)-C(13)	-54.98(15)
C(6)-C(5)-C(9)-C(13)	177.61(10)

O(3)-C(9)-C(10)-C(11)	-0.89(13)
C(5)-C(9)-C(10)-C(11)	111.22(12)
C(13)-C(9)-C(10)-C(11)	-115.87(11)
C(9)-C(10)-C(11)-C(12)	18.49(14)
C(9)-O(3)-C(12)-C(11)	31.30(14)
C(10)-C(11)-C(12)-O(3)	-29.90(14)
C(16)-O(4)-C(13)-C(14)	-7.67(14)
C(16)-O(4)-C(13)-C(17)	108.58(12)
C(16)-O(4)-C(13)-C(9)	-125.57(11)
O(3)-C(9)-C(13)-O(4)	-55.74(11)
C(10)-C(9)-C(13)-O(4)	57.41(11)
C(5)-C(9)-C(13)-O(4)	-173.70(10)
O(3)-C(9)-C(13)-C(14)	-167.55(10)
C(10)-C(9)-C(13)-C(14)	-54.40(14)
C(5)-C(9)-C(13)-C(14)	74.49(14)
O(3)-C(9)-C(13)-C(17)	60.42(14)
C(10)-C(9)-C(13)-C(17)	173.56(10)
C(5)-C(9)-C(13)-C(17)	-57.54(15)
O(4)-C(13)-C(14)-C(15)	16.14(14)
C(17)-C(13)-C(14)-C(15)	-97.00(13)
C(9)-C(13)-C(14)-C(15)	126.44(12)
C(13)-C(14)-C(15)-C(16)	-18.46(16)
C(13)-O(4)-C(16)-C(15)	-4.14(16)
C(14)-C(15)-C(16)-O(4)	14.24(17)
C(20)-O(5)-C(17)-C(21)	117.98(13)
C(20)-O(5)-C(17)-C(18)	1.85(15)
C(20)-O(5)-C(17)-C(13)	-120.72(13)
O(4)-C(13)-C(17)-O(5)	-54.79(12)
C(14)-C(13)-C(17)-O(5)	57.66(12)
C(9)-C(13)-C(17)-O(5)	-169.10(10)
O(4)-C(13)-C(17)-C(21)	61.11(13)
C(14)-C(13)-C(17)-C(21)	173.56(10)
C(9)-C(13)-C(17)-C(21)	-53.20(15)
O(4)-C(13)-C(17)-C(18)	-169.37(11)
C(14)-C(13)-C(17)-C(18)	-56.92(14)
C(9)-C(13)-C(17)-C(18)	76.32(15)

O(5)-C(17)-C(18)-C(19)	12.61(13)
C(21)-C(17)-C(18)-C(19)	-101.41(13)
C(13)-C(17)-C(18)-C(19)	126.78(12)
C(17)-C(18)-C(19)-C(20)	-21.77(16)
C(17)-O(5)-C(20)-C(19)	-16.14(18)
C(18)-C(19)-C(20)-O(5)	23.37(18)
C(24)-O(6)-C(21)-C(22)	-4.34(15)
C(24)-O(6)-C(21)-C(17)	121.42(12)
O(5)-C(17)-C(21)-O(6)	-68.59(12)
C(18)-C(17)-C(21)-O(6)	44.23(13)
C(13)-C(17)-C(21)-O(6)	177.02(10)
O(5)-C(17)-C(21)-C(22)	49.91(14)
C(18)-C(17)-C(21)-C(22)	162.73(11)
C(13)-C(17)-C(21)-C(22)	-64.48(15)
O(6)-C(21)-C(22)-C(23)	-19.82(14)
C(17)-C(21)-C(22)-C(23)	-139.48(12)
C(21)-C(22)-C(23)-C(24)	35.01(16)
C(21)-O(6)-C(24)-C(23)	27.30(17)
C(22)-C(23)-C(24)-O(6)	-38.59(17)

X-Ray Crystallographic Details for **38**.

The data collection crystal was a colorless plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 3.9, which means that 90% of the reflections were measured at least 3.9 times. Phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.031.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4). Since the space group is $P2_1$, only one enantiomer is present in the crystal. The X-ray data cannot determine if the correct enantiomer was chosen for this structure. Since the synthetic procedure produced a racemic mixture, the correct enantiomer cannot be assigned from the chemistry.

There are two sets of disordered carbon atoms: C(3A) and C(3B) and C(23A) and C(23B). The occupancy factors of each set were refined: C(3A) and C(3B) refined to 0.42(2) and 0.58(2), respectively, and C(23A) and C(23B) refined to 0.80(1) and 0.20(1), respectively. SADI restraints were used for the bonds involving atoms C(23A) and C(23B).

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on 2603 intensities and 291 variables and resulted in agreement factors of R1(F) = 0.052 and wR2(F^2) = 0.119. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.045 for 2284 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.35 and -0.28 e/Å^3. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

References

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(2) SHELXS-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.
(3) SHELXL-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.
(4) WinGX-Version 1.70.01: Farrugia, L. J., J. Appl. Cryst., (1999), 32, 837-838.
(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 7. Crystallographic data for **38**.

Empirical formula	C24 H38 O6
Formula weight	422.54
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 10.648(1) Å
	b = 8.576(1) Å
	c = 12.780(2) Å
	β= 113.614(7)°
Volume	1069.3(2) $Å^3$
Z	2
Density (calculated)	1.312 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	460
Crystal size	0.08 x 0.31 x 0.38 mm^3
Theta range for data collection	2.09 to 27.47°
Index ranges	-13<=h<=13, -11<=k<=11, -16<=l<=16
Reflections collected	19571
Independent reflections	2603 [R(int) = 0.031]
Completeness to theta = 27.47°	99.8 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2603 / 3 / 291
Goodness-of-fit on F^2	1.059
Final R indices [I>2sigma(I)]	R1 = 0.0448, wR2 = 0.1141
R indices (all data)	R1 = 0.0524, wR2 = 0.1193
Largest diff. peak and hole	0.347 and -0.276 e/$Å^3$

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **38**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	11816(3)	9353(4)	8634(2)	38(1)
C(2)	12789(3)	8452(4)	9692(3)	47(1)
C(3A)	13890(9)	7894(15)	9345(10)	64(3)*
C(3B)	14227(7)	8761(11)	9692(6)	47(2)*
C(4)	13926(3)	8960(6)	8483(3)	63(1)
C(5)	11396(2)	10964(3)	8904(2)	32(1)
C(6)	10624(3)	10776(4)	9698(2)	38(1)
C(7)	11439(3)	11689(4)	10778(2)	36(1)
C(8)	12804(3)	12009(6)	10734(3)	61(1)
C(9)	10621(2)	12070(3)	7864(2)	30(1)
C(10)	11629(3)	12739(4)	7369(2)	42(1)
C(11)	11978(4)	14383(5)	7819(3)	56(1)
C(12)	11180(3)	14669(4)	8565(4)	58(1)
C(13)	9283(2)	11332(3)	6922(2)	28(1)
C(14)	9600(3)	10498(4)	5980(2)	37(1)
C(15)	8760(3)	9016(4)	5691(2)	46(1)
C(16)	8239(3)	8831(3)	6628(2)	41(1)
C(17)	8057(2)	12518(3)	6398(2)	31(1)
C(18)	8443(3)	14053(4)	5980(3)	46(1)
C(19)	7228(3)	14390(5)	4848(3)	57(1)
C(20)	6771(4)	12768(5)	4400(3)	56(1)
C(21)	7333(2)	12799(4)	7220(2)	35(1)
C(22)	6272(3)	11578(5)	7207(3)	49(1)
C(23A)	5081(3)	12481(6)	7210(4)	54(2)*
C(23B)	4854(11)	12438(17)	6411(13)	46(6)*
C(24)	5169(3)	13961(7)	6589(4)	72(1)
O(1)	12544(2)	9434(3)	7894(2)	49(1)
O(2)	12654(2)	11791(3)	9582(1)	37(1)
O(3)	10229(2)	13418(2)	8349(2)	38(1)
O(4)	8825(2)	10075(2)	7435(1)	30(1)
O(5)	7055(2)	11791(3)	5376(1)	40(1)

O(6)	6592(2)	14263(3)	6935(2)	50(1)

*Occupancy factors are as follows: C(3A) is 0.42(2), C(3B) is 0.58(2), C(23A) is 0.80(1) and C(23B) is 0.20(1).

Table 9. Bond lengths [Å] and angles [°] for **38**.

C(1)-O(1)	1.444(3)
C(1)-C(5)	1.533(4)
C(1)-C(2)	1.541(4)
C(1)-H(1)	1.0000
C(2)-C(3A)	1.488(12)
C(2)-C(3B)	1.553(8)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(2)-H(2C)	0.9900
C(2)-H(2D)	0.9900
C(3A)-C(4)	1.444(11)
C(3A)-H(3A)	0.9900
C(3A)-H(3B)	0.9900
C(3B)-C(4)	1.457(7)
C(3B)-H(3C)	0.9900
C(3B)-H(3D)	0.9900
C(4)-O(1)	1.418(3)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(4)-H(4C)	0.9900
C(4)-H(4D)	0.9900
C(5)-O(2)	1.455(3)
C(5)-C(6)	1.550(3)
C(5)-C(9)	1.571(4)
C(6)-C(7)	1.520(4)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.502(4)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-O(2)	1.429(3)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-O(3)	1.450(3)

C(9)-C(10)	1.558(3)
C(9)-C(13)	1.583(3)
C(10)-C(11)	1.512(5)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.529(5)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-O(3)	1.425(4)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-O(4)	1.445(3)
C(13)-C(14)	1.550(3)
C(13)-C(17)	1.576(3)
C(14)-C(15)	1.513(5)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.516(4)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-O(4)	1.440(3)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-O(5)	1.455(3)
C(17)-C(18)	1.538(4)
C(17)-C(21)	1.551(3)
C(18)-C(19)	1.533(4)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.509(6)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-O(5)	1.431(4)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-O(6)	1.449(4)

C(21)-C(22)	1.536(4)
C(21)-H(21)	1.0000
C(22)-C(23A)	1.488(5)
C(22)-C(23B)	1.621(12)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(22)-H(22C)	0.9900
C(22)-H(22D)	0.9900
C(23A)-C(24)	1.520(8)
C(23A)-H(23A)	0.9900
C(23A)-H(23B)	0.9900
C(23B)-C(24)	1.345(15)
C(23B)-H(23C)	0.9900
C(23B)-H(23D)	0.9900
C(24)-O(6)	1.422(4)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900
C(24)-H(24C)	0.9900
C(24)-H(24D)	0.9900

O(1)-C(1)-C(5)	112.6(2)
O(1)-C(1)-C(2)	104.5(2)
C(5)-C(1)-C(2)	114.1(2)
O(1)-C(1)-H(1)	108.5
C(5)-C(1)-H(1)	108.5
C(2)-C(1)-H(1)	108.5
C(3A)-C(2)-C(1)	103.5(4)
C(1)-C(2)-C(3B)	103.4(3)
C(3A)-C(2)-H(2A)	111.1
C(1)-C(2)-H(2A)	111.1
C(3A)-C(2)-H(2B)	111.1
C(1)-C(2)-H(2B)	111.1
H(2A)-C(2)-H(2B)	109.0
C(1)-C(2)-H(2C)	111.1
C(3B)-C(2)-H(2C)	111.1
C(1)-C(2)-H(2D)	111.1

C(3B)-C(2)-H(2D)	111.1
H(2C)-C(2)-H(2D)	109.1
C(4)-C(3A)-C(2)	106.7(6)
C(4)-C(3A)-H(3A)	110.4
C(2)-C(3A)-H(3A)	110.4
C(4)-C(3A)-H(3B)	110.4
C(2)-C(3A)-H(3B)	110.4
H(3A)-C(3A)-H(3B)	108.6
C(4)-C(3B)-C(2)	102.8(5)
C(4)-C(3B)-H(3C)	111.2
C(2)-C(3B)-H(3C)	111.2
C(4)-C(3B)-H(3D)	111.2
C(2)-C(3B)-H(3D)	111.2
H(3C)-C(3B)-H(3D)	109.1
O(1)-C(4)-C(3A)	104.3(4)
O(1)-C(4)-C(3B)	109.7(3)
O(1)-C(4)-H(4A)	110.9
C(3A)-C(4)-H(4A)	110.9
O(1)-C(4)-H(4B)	110.9
C(3A)-C(4)-H(4B)	110.9
H(4A)-C(4)-H(4B)	108.9
O(1)-C(4)-H(4C)	109.7
C(3B)-C(4)-H(4C)	109.7
O(1)-C(4)-H(4D)	109.7
C(3B)-C(4)-H(4D)	109.7
H(4C)-C(4)-H(4D)	108.2
O(2)-C(5)-C(1)	107.0(2)
O(2)-C(5)-C(6)	105.25(19)
C(1)-C(5)-C(6)	109.4(2)
O(2)-C(5)-C(9)	104.8(2)
C(1)-C(5)-C(9)	117.1(2)
C(6)-C(5)-C(9)	112.3(2)
C(7)-C(6)-C(5)	106.4(2)
C(7)-C(6)-H(6A)	110.5
C(5)-C(6)-H(6A)	110.5
C(7)-C(6)-H(6B)	110.5

C(5)-C(6)-H(6B)	110.5
H(6A)-C(6)-H(6B)	108.6
C(8)-C(7)-C(6)	105.6(2)
C(8)-C(7)-H(7A)	110.6
C(6)-C(7)-H(7A)	110.6
C(8)-C(7)-H(7B)	110.6
C(6)-C(7)-H(7B)	110.6
H(7A)-C(7)-H(7B)	108.7
O(2)-C(8)-C(7)	107.7(2)
O(2)-C(8)-H(8A)	110.2
C(7)-C(8)-H(8A)	110.2
O(2)-C(8)-H(8B)	110.2
C(7)-C(8)-H(8B)	110.2
H(8A)-C(8)-H(8B)	108.5
O(3)-C(9)-C(10)	104.1(2)
O(3)-C(9)-C(5)	105.32(19)
C(10)-C(9)-C(5)	110.6(2)
O(3)-C(9)-C(13)	108.57(18)
C(10)-C(9)-C(13)	113.2(2)
C(5)-C(9)-C(13)	114.2(2)
C(11)-C(10)-C(9)	107.0(2)
C(11)-C(10)-H(10A)	110.3
C(9)-C(10)-H(10A)	110.3
C(11)-C(10)-H(10B)	110.3
C(9)-C(10)-H(10B)	110.3
H(10A)-C(10)-H(10B)	108.6
C(10)-C(11)-C(12)	105.9(3)
C(10)-C(11)-H(11A)	110.6
C(12)-C(11)-H(11A)	110.6
C(10)-C(11)-H(11B)	110.6
C(12)-C(11)-H(11B)	110.6
H(11A)-C(11)-H(11B)	108.7
O(3)-C(12)-C(11)	106.7(3)
O(3)-C(12)-H(12A)	110.4
C(11)-C(12)-H(12A)	110.4
O(3)-C(12)-H(12B)	110.4

C(11)-C(12)-H(12B)	110.4
H(12A)-C(12)-H(12B)	108.6
O(4)-C(13)-C(14)	103.1(2)
O(4)-C(13)-C(17)	107.53(17)
C(14)-C(13)-C(17)	111.67(19)
O(4)-C(13)-C(9)	108.53(18)
C(14)-C(13)-C(9)	111.48(19)
C(17)-C(13)-C(9)	113.9(2)
C(15)-C(14)-C(13)	106.6(2)
C(15)-C(14)-H(14A)	110.4
C(13)-C(14)-H(14A)	110.4
C(15)-C(14)-H(14B)	110.4
C(13)-C(14)-H(14B)	110.4
H(14A)-C(14)-H(14B)	108.6
C(14)-C(15)-C(16)	105.2(2)
C(14)-C(15)-H(15A)	110.7
C(16)-C(15)-H(15A)	110.7
C(14)-C(15)-H(15B)	110.7
C(16)-C(15)-H(15B)	110.7
H(15A)-C(15)-H(15B)	108.8
O(4)-C(16)-C(15)	107.3(2)
O(4)-C(16)-H(16A)	110.2
C(15)-C(16)-H(16A)	110.2
O(4)-C(16)-H(16B)	110.2
C(15)-C(16)-H(16B)	110.2
H(16A)-C(16)-H(16B)	108.5
O(5)-C(17)-C(18)	104.9(2)
O(5)-C(17)-C(21)	106.87(19)
C(18)-C(17)-C(21)	112.1(2)
O(5)-C(17)-C(13)	106.8(2)
C(18)-C(17)-C(13)	114.14(19)
C(21)-C(17)-C(13)	111.39(19)
C(19)-C(18)-C(17)	104.3(3)
C(19)-C(18)-H(18A)	110.9
C(17)-C(18)-H(18A)	110.9
C(19)-C(18)-H(18B)	110.9

C(17)-C(18)-H(18B)	110.9
H(18A)-C(18)-H(18B)	108.9
C(20)-C(19)-C(18)	101.9(3)
C(20)-C(19)-H(19A)	111.4
C(18)-C(19)-H(19A)	111.4
C(20)-C(19)-H(19B)	111.4
C(18)-C(19)-H(19B)	111.4
H(19A)-C(19)-H(19B)	109.2
O(5)-C(20)-C(19)	106.6(3)
O(5)-C(20)-H(20A)	110.4
C(19)-C(20)-H(20A)	110.4
O(5)-C(20)-H(20B)	110.4
C(19)-C(20)-H(20B)	110.4
H(20A)-C(20)-H(20B)	108.6
O(6)-C(21)-C(22)	105.3(2)
O(6)-C(21)-C(17)	109.4(2)
C(22)-C(21)-C(17)	116.7(2)
O(6)-C(21)-H(21)	108.4
C(22)-C(21)-H(21)	108.4
C(17)-C(21)-H(21)	108.4
C(23A)-C(22)-C(21)	105.6(3)
C(21)-C(22)-C(23B)	101.1(6)
C(23A)-C(22)-H(22A)	110.6
C(21)-C(22)-H(22A)	110.6
C(23A)-C(22)-H(22B)	110.6
C(21)-C(22)-H(22B)	110.6
H(22A)-C(22)-H(22B)	108.8
C(21)-C(22)-H(22C)	111.6
C(23B)-C(22)-H(22C)	111.6
C(21)-C(22)-H(22D)	111.6
C(23B)-C(22)-H(22D)	111.6
H(22C)-C(22)-H(22D)	109.4
C(22)-C(23A)-C(24)	101.7(3)
C(22)-C(23A)-H(23A)	111.4
C(24)-C(23A)-H(23A)	111.4
C(22)-C(23A)-H(23B)	111.4

C(24)-C(23A)-H(23B)	111.4
H(23A)-C(23A)-H(23B)	109.3
C(24)-C(23B)-C(22)	103.3(8)
C(24)-C(23B)-H(23C)	111.1
C(22)-C(23B)-H(23C)	111.1
C(24)-C(23B)-H(23D)	111.1
C(22)-C(23B)-H(23D)	111.1
H(23C)-C(23B)-H(23D)	109.1
C(23B)-C(24)-O(6)	113.1(6)
O(6)-C(24)-C(23A)	105.7(3)
O(6)-C(24)-H(24A)	110.6
C(23A)-C(24)-H(24A)	110.6
O(6)-C(24)-H(24B)	110.6
C(23A)-C(24)-H(24B)	110.6
H(24A)-C(24)-H(24B)	108.7
C(23B)-C(24)-H(24C)	109.0
O(6)-C(24)-H(24C)	109.0
C(23B)-C(24)-H(24D)	109.0
O(6)-C(24)-H(24D)	109.0
H(24C)-C(24)-H(24D)	107.8
C(4)-O(1)-C(1)	110.7(2)
C(8)-O(2)-C(5)	112.0(2)
C(12)-O(3)-C(9)	112.26(19)
C(16)-O(4)-C(13)	110.87(18)
C(20)-O(5)-C(17)	110.8(2)
C(24)-O(6)-C(21)	109.0(3)

Table 10. Anisotropic displacement parameters ($Å^2$x 10^3) for **38**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	33(1)	43(2)	32(1)	1(1)	7(1)	3(1)
C(2)	50(2)	42(2)	41(2)	9(1)	9(1)	2(1)
C(3A)	42(4)	44(6)	73(6)	3(5)	-11(4)	4(4)
C(3B)	33(3)	52(5)	43(3)	5(3)	3(2)	14(3)
C(4)	41(2)	90(3)	55(2)	12(2)	15(1)	26(2)
C(5)	27(1)	40(2)	28(1)	-2(1)	11(1)	-4(1)
C(6)	40(1)	49(2)	27(1)	-6(1)	16(1)	-9(1)
C(7)	37(1)	40(2)	29(1)	-3(1)	11(1)	2(1)
C(8)	60(2)	82(3)	40(2)	-21(2)	21(1)	-26(2)
C(9)	24(1)	37(1)	32(1)	-1(1)	15(1)	0(1)
C(10)	31(1)	54(2)	47(1)	12(1)	23(1)	2(1)
C(11)	49(2)	50(2)	73(2)	7(2)	29(2)	-16(2)
C(12)	39(2)	40(2)	98(3)	-11(2)	31(2)	-9(1)
C(13)	27(1)	32(1)	28(1)	3(1)	14(1)	2(1)
C(14)	35(1)	51(2)	27(1)	3(1)	15(1)	8(1)
C(15)	48(2)	52(2)	36(1)	-9(1)	15(1)	4(2)
C(16)	54(2)	34(2)	34(1)	-7(1)	15(1)	-7(1)
C(17)	27(1)	34(1)	34(1)	4(1)	12(1)	2(1)
C(18)	37(1)	43(2)	61(2)	18(2)	23(1)	7(1)
C(19)	48(2)	62(2)	64(2)	34(2)	27(2)	20(2)
C(20)	54(2)	74(2)	37(1)	22(2)	15(1)	20(2)
C(21)	25(1)	41(1)	40(1)	-2(1)	14(1)	2(1)
C(22)	40(2)	58(2)	60(2)	-7(2)	31(1)	-11(2)
C(23A)	27(2)	80(3)	57(3)	-29(2)	20(2)	-8(2)
C(23B)	13(6)	81(13)	41(10)	-13(8)	8(5)	-1(6)
C(24)	34(2)	104(4)	78(2)	10(3)	22(2)	25(2)
O(1)	44(1)	68(2)	35(1)	7(1)	15(1)	25(1)
O(2)	28(1)	46(1)	33(1)	-5(1)	9(1)	-6(1)
O(3)	28(1)	36(1)	52(1)	-10(1)	19(1)	-4(1)
O(4)	33(1)	33(1)	25(1)	0(1)	11(1)	-4(1)
O(5)	35(1)	50(1)	30(1)	6(1)	7(1)	5(1)
O(6)	38(1)	47(1)	68(1)	-6(1)	24(1)	8(1)

Table 11. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **38**.

	x	y	z	U(eq)
H(1)	10968	8716	8246	45
H(2A)	13166	9144	10367	56
H(2B)	12312	7566	9870	56
H(2C)	12573	7325	9621	56
H(2D)	12733	8855	10398	56
H(3A)	14784	7887	10010	77
H(3B)	13688	6823	9033	77
H(3C)	14646	9713	10131	56
H(3D)	14849	7866	10017	56
H(4A)	14523	9865	8836	76
H(4B)	14268	8433	7959	76
H(4C)	14546	9756	8387	76
H(4D)	14082	7965	8161	76
H(6A)	9682	11194	9326	45
H(6B)	10571	9662	9880	45
H(7A)	10967	12677	10797	43
H(7B)	11555	11071	11465	43
H(8A)	13098	13091	10984	73
H(8B)	13505	11286	11248	73
H(10A)	12469	12093	7615	50
H(10B)	11193	12744	6524	50
H(11A)	11701	15140	7181	67
H(11B)	12975	14488	8275	67
H(12A)	11812	14692	9381	70
H(12B)	10690	15679	8366	70
H(14A)	9346	11171	5296	44
H(14B)	10590	10251	6260	44
H(15A)	9333	8112	5679	55
H(15B)	7985	9104	4935	55
H(16A)	7226	8893	6305	49
H(16B)	8521	7808	7009	49

H(18A)	8563	14903	6536	55
H(18B)	9301	13934	5860	55
H(19A)	6493	14964	4973	68
H(19B)	7516	14994	4322	68
H(20A)	5778	12759	3907	68
H(20B)	7278	12400	3946	68
H(21)	8050	12877	8016	42
H(22A)	5997	10919	6514	59
H(22B)	6652	10898	7888	59
H(22C)	6317	11377	7984	59
H(22D)	6392	10584	6863	59
H(23A)	5176	12708	7998	65
H(23B)	4208	11923	6796	65
H(23C)	4577	12161	5597	55
H(23D)	4105	12160	6649	55
H(24A)	4722	13810	5751	86
H(24B)	4716	14839	6802	86
H(24C)	4875	14353	7183	86
H(24D)	4653	14542	5876	86

Table 12. Torsion angles [°] for **38**.

O(1)-C(1)-C(2)-C(3A)	-8.8(6)
C(5)-C(1)-C(2)-C(3A)	-132.2(6)
O(1)-C(1)-C(2)-C(3B)	25.2(5)
C(5)-C(1)-C(2)-C(3B)	-98.3(4)
C(1)-C(2)-C(3A)-C(4)	25.6(9)
C(3B)-C(2)-C(3A)-C(4)	-68.2(9)
C(3A)-C(2)-C(3B)-C(4)	64.7(8)
C(1)-C(2)-C(3B)-C(4)	-29.6(6)
C(2)-C(3A)-C(4)-O(1)	-32.9(10)
C(2)-C(3A)-C(4)-C(3B)	71.1(10)
C(2)-C(3B)-C(4)-O(1)	23.9(7)
C(2)-C(3B)-C(4)-C(3A)	-62.9(8)
O(1)-C(1)-C(5)-O(2)	-66.5(2)
C(2)-C(1)-C(5)-O(2)	52.4(3)
O(1)-C(1)-C(5)-C(6)	-179.99(19)
C(2)-C(1)-C(5)-C(6)	-61.1(3)
O(1)-C(1)-C(5)-C(9)	50.7(3)
C(2)-C(1)-C(5)-C(9)	169.6(2)
O(2)-C(5)-C(6)-C(7)	6.1(3)
C(1)-C(5)-C(6)-C(7)	120.8(2)
C(9)-C(5)-C(6)-C(7)	-107.4(3)
C(5)-C(6)-C(7)-C(8)	-14.6(3)
C(6)-C(7)-C(8)-O(2)	18.0(4)
O(2)-C(5)-C(9)-O(3)	-67.5(2)
C(1)-C(5)-C(9)-O(3)	174.17(19)
C(6)-C(5)-C(9)-O(3)	46.3(3)
O(2)-C(5)-C(9)-C(10)	44.4(3)
C(1)-C(5)-C(9)-C(10)	-73.9(3)
C(6)-C(5)-C(9)-C(10)	158.2(2)
O(2)-C(5)-C(9)-C(13)	173.51(18)
C(1)-C(5)-C(9)-C(13)	55.1(3)
C(6)-C(5)-C(9)-C(13)	-72.7(3)
O(3)-C(9)-C(10)-C(11)	11.5(3)
C(5)-C(9)-C(10)-C(11)	-101.1(3)
C(13)-C(9)-C(10)-C(11)	129.3(2)
C(9)-C(10)-C(11)-C(12)	-0.1(3)
C(10)-C(11)-C(12)-O(3)	-11.6(4)
O(3)-C(9)-C(13)-O(4)	-95.9(2)
C(10)-C(9)-C(13)-O(4)	149.0(2)

C(5)-C(9)-C(13)-O(4)	21.3(3)
O(3)-C(9)-C(13)-C(14)	151.3(2)
C(10)-C(9)-C(13)-C(14)	36.2(3)
C(5)-C(9)-C(13)-C(14)	-91.6(2)
O(3)-C(9)-C(13)-C(17)	23.8(3)
C(10)-C(9)-C(13)-C(17)	-91.3(3)
C(5)-C(9)-C(13)-C(17)	140.98(19)
O(4)-C(13)-C(14)-C(15)	23.6(2)
C(17)-C(13)-C(14)-C(15)	-91.6(3)
C(9)-C(13)-C(14)-C(15)	139.8(2)
C(13)-C(14)-C(15)-C(16)	-12.4(3)
C(14)-C(15)-C(16)-O(4)	-3.5(3)
O(4)-C(13)-C(17)-O(5)	-73.5(2)
C(14)-C(13)-C(17)-O(5)	38.9(3)
C(9)-C(13)-C(17)-O(5)	166.21(18)
O(4)-C(13)-C(17)-C(18)	171.1(2)
C(14)-C(13)-C(17)-C(18)	-76.6(3)
C(9)-C(13)-C(17)-C(18)	50.8(3)
O(4)-C(13)-C(17)-C(21)	42.8(3)
C(14)-C(13)-C(17)-C(21)	155.2(2)
C(9)-C(13)-C(17)-C(21)	-77.4(3)
O(5)-C(17)-C(18)-C(19)	22.1(3)
C(21)-C(17)-C(18)-C(19)	-93.5(3)
C(13)-C(17)-C(18)-C(19)	138.6(2)
C(17)-C(18)-C(19)-C(20)	-32.6(3)
C(18)-C(19)-C(20)-O(5)	32.0(3)
O(5)-C(17)-C(21)-O(6)	-84.9(2)
C(18)-C(17)-C(21)-O(6)	29.5(3)
C(13)-C(17)-C(21)-O(6)	158.8(2)
O(5)-C(17)-C(21)-C(22)	34.5(3)
C(18)-C(17)-C(21)-C(22)	148.9(3)
C(13)-C(17)-C(21)-C(22)	-81.8(3)
O(6)-C(21)-C(22)-C(23A)	-14.3(3)
C(17)-C(21)-C(22)-C(23A)	-135.9(3)
O(6)-C(21)-C(22)-C(23B)	21.7(6)
C(17)-C(21)-C(22)-C(23B)	-99.9(6)
C(21)-C(22)-C(23A)-C(24)	30.0(4)
C(23B)-C(22)-C(23A)-C(24)	-57.3(9)
C(23A)-C(22)-C(23B)-C(24)	73.1(10)
C(21)-C(22)-C(23B)-C(24)	-28.2(10)

C(22)-C(23B)-C(24)-O(6)	25.0(12)
C(22)-C(23B)-C(24)-C(23A)	-61.1(7)
C(22)-C(23A)-C(24)-C(23B)	71.5(8)
C(22)-C(23A)-C(24)-O(6)	-36.1(4)
C(3A)-C(4)-O(1)-C(1)	27.7(7)
C(3B)-C(4)-O(1)-C(1)	-8.3(6)
C(5)-C(1)-O(1)-C(4)	113.0(3)
C(2)-C(1)-O(1)-C(4)	-11.4(4)
C(7)-C(8)-O(2)-C(5)	-14.9(4)
C(1)-C(5)-O(2)-C(8)	-111.0(3)
C(6)-C(5)-O(2)-C(8)	5.3(4)
C(9)-C(5)-O(2)-C(8)	124.0(3)
C(11)-C(12)-O(3)-C(9)	20.4(4)
C(10)-C(9)-O(3)-C(12)	-20.0(3)
C(5)-C(9)-O(3)-C(12)	96.4(3)
C(13)-C(9)-O(3)-C(12)	-140.9(3)
C(15)-C(16)-O(4)-C(13)	19.8(3)
C(14)-C(13)-O(4)-C(16)	-26.8(2)
C(17)-C(13)-O(4)-C(16)	91.2(2)
C(9)-C(13)-O(4)-C(16)	-145.1(2)
C(19)-C(20)-O(5)-C(17)	-19.2(3)
C(18)-C(17)-O(5)-C(20)	-2.1(3)
C(21)-C(17)-O(5)-C(20)	117.1(3)
C(13)-C(17)-O(5)-C(20)	-123.6(2)
C(23B)-C(24)-O(6)-C(21)	-11.5(9)
C(23A)-C(24)-O(6)-C(21)	28.3(4)
C(22)-C(21)-O(6)-C(24)	-8.9(3)
C(17)-C(21)-O(6)-C(24)	117.3(3)

X-Ray Crystallographic Details for **39**.

The data collection crystal was a colorless triangular plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 4.4, which means that 90% of the reflections were measured at least 4.4 times. Phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.049.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on 4833 intensities and 271 variables and resulted in agreement factors of R1(F) = 0.075 and wR2(F^2) = 0.129. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.047 for 3412 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.47 and -0.38 e/Å^3. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

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Table 13. Crystallographic data for **39**.

Empirical formula	C24 H38 O6
Formula weight	422.54
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 10.991(1) Å
	b = 13.996(2) Å
	c = 14.350(2) Å
	β= 106.475(6)°
Volume	2116.8(5) Å^3
Z	4
Density (calculated)	1.326 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	920
Crystal size	0.19 x 0.35 x 0.38 mm^3
Theta range for data collection	2.75 to 27.47°
Index ranges	-14<=h<=14, -18<=k<=18, -18<=l<=18
Reflections collected	44226
Independent reflections	4833 [R(int) = 0.049]
Completeness to theta = 27.47°	99.8 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4833 / 0 / 271
Goodness-of-fit on F^2	1.075
Final R indices [I>2sigma(I)]	R1 = 0.0474, wR2 = 0.1183
R indices (all data)	R1 = 0.0750, wR2 = 0.1294
Largest diff. peak and hole	0.469 and -0.375 e/Å^3

Table 14. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **39**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8145(2)	4011(1)	1083(1)	23(1)
C(2)	8167(2)	5002(1)	632(1)	29(1)
C(3)	8862(2)	5615(1)	1500(1)	33(1)
C(4)	8402(2)	5193(1)	2299(1)	31(1)
C(5)	6941(1)	3390(1)	687(1)	19(1)
C(6)	6806(2)	2646(1)	1455(1)	21(1)
C(7)	5829(2)	3082(1)	1898(1)	27(1)
C(8)	5009(2)	3652(1)	1058(1)	24(1)
C(9)	6778(1)	2915(1)	-342(1)	18(1)
C(10)	6478(1)	3671(1)	-1170(1)	21(1)
C(11)	5031(2)	3730(1)	-1532(1)	25(1)
C(12)	4603(2)	2759(1)	-1288(1)	25(1)
C(13)	7923(1)	2235(1)	-368(1)	19(1)
C(14)	8934(2)	2782(1)	-723(1)	25(1)
C(15)	10223(2)	2442(1)	-74(1)	34(1)
C(16)	9916(2)	1863(1)	729(1)	29(1)
C(17)	7531(1)	1277(1)	-955(1)	21(1)
C(18)	6708(2)	1424(1)	-2018(1)	26(1)
C(19)	7370(2)	855(2)	-2636(1)	59(1)
C(20)	8563(2)	539(2)	-1987(1)	51(1)
C(21)	6924(2)	543(1)	-415(1)	24(1)
C(22)	7778(2)	10(1)	448(1)	27(1)
C(23)	7031(2)	-909(1)	448(1)	31(1)
C(24)	6505(2)	-1104(1)	-623(1)	34(1)
O(1)	8295(1)	4193(1)	2103(1)	30(1)
O(2)	5870(1)	4028(1)	568(1)	21(1)
O(3)	5622(1)	2363(1)	-528(1)	20(1)
O(4)	8585(1)	1981(1)	619(1)	22(1)
O(5)	8702(1)	865(1)	-1044(1)	26(1)
O(6)	6363(1)	-204(1)	-1103(1)	38(1)

Table 15. Bond lengths [Å] and angles [°] for **39**.

C(1)-O(1)	1.4484(18)
C(1)-C(2)	1.534(2)
C(1)-C(5)	1.550(2)
C(1)-H(1)	1.0000
C(2)-C(3)	1.527(2)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.500(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(1)	1.426(2)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-O(2)	1.4488(18)
C(5)-C(6)	1.553(2)
C(5)-C(9)	1.582(2)
C(6)-C(7)	1.523(2)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.511(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-O(2)	1.4306(18)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-O(3)	1.4463(17)
C(9)-C(10)	1.555(2)
C(9)-C(13)	1.586(2)
C(10)-C(11)	1.529(2)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.512(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(12)-O(3)	1.4343(18)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-O(4)	1.4406(17)
C(13)-C(14)	1.551(2)
C(13)-C(17)	1.577(2)
C(14)-C(15)	1.534(2)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.523(2)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-O(4)	1.4358(19)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-O(5)	1.4482(18)
C(17)-C(21)	1.548(2)
C(17)-C(18)	1.550(2)
C(18)-C(19)	1.522(2)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.446(3)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-O(5)	1.395(2)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-O(6)	1.4504(19)
C(21)-C(22)	1.520(2)
C(21)-H(21)	1.0000
C(22)-C(23)	1.526(2)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-C(24)	1.506(3)
C(23)-H(23A)	0.9900
C(23)-H(23B)	0.9900

C(24)-O(6)	1.423(2)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900

O(1)-C(1)-C(2)	104.85(13)
O(1)-C(1)-C(5)	108.08(12)
C(2)-C(1)-C(5)	117.83(13)
O(1)-C(1)-H(1)	108.6
C(2)-C(1)-H(1)	108.6
C(5)-C(1)-H(1)	108.6
C(3)-C(2)-C(1)	103.23(13)
C(3)-C(2)-H(2A)	111.1
C(1)-C(2)-H(2A)	111.1
C(3)-C(2)-H(2B)	111.1
C(1)-C(2)-H(2B)	111.1
H(2A)-C(2)-H(2B)	109.1
C(4)-C(3)-C(2)	101.69(13)
C(4)-C(3)-H(3A)	111.4
C(2)-C(3)-H(3A)	111.4
C(4)-C(3)-H(3B)	111.4
C(2)-C(3)-H(3B)	111.4
H(3A)-C(3)-H(3B)	109.3
O(1)-C(4)-C(3)	105.23(13)
O(1)-C(4)-H(4A)	110.7
C(3)-C(4)-H(4A)	110.7
O(1)-C(4)-H(4B)	110.7
C(3)-C(4)-H(4B)	110.7
H(4A)-C(4)-H(4B)	108.8
O(2)-C(5)-C(1)	106.11(12)
O(2)-C(5)-C(6)	105.04(11)
C(1)-C(5)-C(6)	111.05(12)
O(2)-C(5)-C(9)	105.99(11)
C(1)-C(5)-C(9)	115.88(12)
C(6)-C(5)-C(9)	111.87(12)
C(7)-C(6)-C(5)	104.36(12)
C(7)-C(6)-H(6A)	110.9

C(5)-C(6)-H(6A)	110.9
C(7)-C(6)-H(6B)	110.9
C(5)-C(6)-H(6B)	110.9
H(6A)-C(6)-H(6B)	108.9
C(8)-C(7)-C(6)	102.14(12)
C(8)-C(7)-H(7A)	111.3
C(6)-C(7)-H(7A)	111.3
C(8)-C(7)-H(7B)	111.3
C(6)-C(7)-H(7B)	111.3
H(7A)-C(7)-H(7B)	109.2
O(2)-C(8)-C(7)	104.96(12)
O(2)-C(8)-H(8A)	110.8
C(7)-C(8)-H(8A)	110.8
O(2)-C(8)-H(8B)	110.8
C(7)-C(8)-H(8B)	110.8
H(8A)-C(8)-H(8B)	108.8
O(3)-C(9)-C(10)	103.76(11)
O(3)-C(9)-C(5)	104.92(11)
C(10)-C(9)-C(5)	111.76(12)
O(3)-C(9)-C(13)	109.63(12)
C(10)-C(9)-C(13)	112.88(12)
C(5)-C(9)-C(13)	113.13(11)
C(11)-C(10)-C(9)	105.74(12)
C(11)-C(10)-H(10A)	110.6
C(9)-C(10)-H(10A)	110.6
C(11)-C(10)-H(10B)	110.6
C(9)-C(10)-H(10B)	110.6
H(10A)-C(10)-H(10B)	108.7
C(12)-C(11)-C(10)	103.58(13)
C(12)-C(11)-H(11A)	111.0
C(10)-C(11)-H(11A)	111.0
C(12)-C(11)-H(11B)	111.0
C(10)-C(11)-H(11B)	111.0
H(11A)-C(11)-H(11B)	109.0
O(3)-C(12)-C(11)	107.24(12)
O(3)-C(12)-H(12A)	110.3

C(11)-C(12)-H(12A)	110.3
O(3)-C(12)-H(12B)	110.3
C(11)-C(12)-H(12B)	110.3
H(12A)-C(12)-H(12B)	108.5
O(4)-C(13)-C(14)	103.62(11)
O(4)-C(13)-C(17)	107.52(12)
C(14)-C(13)-C(17)	110.87(12)
O(4)-C(13)-C(9)	107.97(11)
C(14)-C(13)-C(9)	110.98(12)
C(17)-C(13)-C(9)	115.13(12)
C(15)-C(14)-C(13)	105.76(13)
C(15)-C(14)-H(14A)	110.6
C(13)-C(14)-H(14A)	110.6
C(15)-C(14)-H(14B)	110.6
C(13)-C(14)-H(14B)	110.6
H(14A)-C(14)-H(14B)	108.7
C(16)-C(15)-C(14)	105.05(13)
C(16)-C(15)-H(15A)	110.7
C(14)-C(15)-H(15A)	110.7
C(16)-C(15)-H(15B)	110.7
C(14)-C(15)-H(15B)	110.7
H(15A)-C(15)-H(15B)	108.8
O(4)-C(16)-C(15)	107.03(13)
O(4)-C(16)-H(16A)	110.3
C(15)-C(16)-H(16A)	110.3
O(4)-C(16)-H(16B)	110.3
C(15)-C(16)-H(16B)	110.3
H(16A)-C(16)-H(16B)	108.6
O(5)-C(17)-C(21)	107.42(12)
O(5)-C(17)-C(18)	104.56(11)
C(21)-C(17)-C(18)	111.47(13)
O(5)-C(17)-C(13)	105.76(12)
C(21)-C(17)-C(13)	112.79(12)
C(18)-C(17)-C(13)	114.10(13)
C(19)-C(18)-C(17)	105.20(13)
C(19)-C(18)-H(18A)	110.7

C(17)-C(18)-H(18A)	110.7
C(19)-C(18)-H(18B)	110.7
C(17)-C(18)-H(18B)	110.7
H(18A)-C(18)-H(18B)	108.8
C(20)-C(19)-C(18)	106.29(15)
C(20)-C(19)-H(19A)	110.5
C(18)-C(19)-H(19A)	110.5
C(20)-C(19)-H(19B)	110.5
C(18)-C(19)-H(19B)	110.5
H(19A)-C(19)-H(19B)	108.7
O(5)-C(20)-C(19)	110.76(16)
O(5)-C(20)-H(20A)	109.5
C(19)-C(20)-H(20A)	109.5
O(5)-C(20)-H(20B)	109.5
C(19)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	108.1
O(6)-C(21)-C(22)	104.19(13)
O(6)-C(21)-C(17)	107.39(12)
C(22)-C(21)-C(17)	118.67(13)
O(6)-C(21)-H(21)	108.7
C(22)-C(21)-H(21)	108.7
C(17)-C(21)-H(21)	108.7
C(21)-C(22)-C(23)	101.91(13)
C(21)-C(22)-H(22A)	111.4
C(23)-C(22)-H(22A)	111.4
C(21)-C(22)-H(22B)	111.4
C(23)-C(22)-H(22B)	111.4
H(22A)-C(22)-H(22B)	109.3
C(24)-C(23)-C(22)	101.74(13)
C(24)-C(23)-H(23A)	111.4
C(22)-C(23)-H(23A)	111.4
C(24)-C(23)-H(23B)	111.4
C(22)-C(23)-H(23B)	111.4
H(23A)-C(23)-H(23B)	109.3
O(6)-C(24)-C(23)	106.92(14)
O(6)-C(24)-H(24A)	110.3

C(23)-C(24)-H(24A)	110.3
O(6)-C(24)-H(24B)	110.3
C(23)-C(24)-H(24B)	110.3
H(24A)-C(24)-H(24B)	108.6
C(4)-O(1)-C(1)	110.57(12)
C(8)-O(2)-C(5)	110.09(11)
C(12)-O(3)-C(9)	112.92(11)
C(16)-O(4)-C(13)	110.62(11)
C(20)-O(5)-C(17)	111.13(12)
C(24)-O(6)-C(21)	110.03(12)

Table 16. Anisotropic displacement parameters ($Å^2 x 10^3$) for **39**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	25(1)	21(1)	22(1)	-2(1)	5(1)	-3(1)
C(2)	33(1)	27(1)	25(1)	2(1)	8(1)	-8(1)
C(3)	38(1)	23(1)	35(1)	-3(1)	5(1)	-8(1)
C(4)	32(1)	27(1)	30(1)	-6(1)	3(1)	-2(1)
C(5)	20(1)	16(1)	20(1)	1(1)	4(1)	2(1)
C(6)	24(1)	19(1)	19(1)	1(1)	3(1)	-2(1)
C(7)	33(1)	26(1)	22(1)	0(1)	10(1)	-3(1)
C(8)	26(1)	22(1)	27(1)	-2(1)	11(1)	0(1)
C(9)	18(1)	16(1)	19(1)	0(1)	3(1)	-2(1)
C(10)	25(1)	18(1)	19(1)	2(1)	4(1)	-1(1)
C(11)	26(1)	25(1)	22(1)	4(1)	4(1)	4(1)
C(12)	19(1)	26(1)	25(1)	3(1)	-2(1)	2(1)
C(13)	18(1)	21(1)	16(1)	2(1)	2(1)	0(1)
C(14)	23(1)	24(1)	28(1)	1(1)	7(1)	-1(1)
C(15)	20(1)	37(1)	42(1)	5(1)	6(1)	-1(1)
C(16)	18(1)	33(1)	30(1)	1(1)	-1(1)	0(1)
C(17)	18(1)	21(1)	23(1)	-1(1)	5(1)	4(1)
C(18)	29(1)	26(1)	22(1)	-3(1)	2(1)	3(1)
C(19)	47(1)	93(2)	31(1)	-22(1)	2(1)	26(1)
C(20)	54(1)	75(2)	24(1)	-3(1)	12(1)	28(1)
C(21)	23(1)	18(1)	28(1)	-4(1)	6(1)	-1(1)
C(22)	32(1)	23(1)	25(1)	2(1)	6(1)	-3(1)
C(23)	32(1)	23(1)	40(1)	3(1)	13(1)	-1(1)
C(24)	33(1)	19(1)	45(1)	-1(1)	5(1)	-2(1)
O(1)	41(1)	25(1)	21(1)	-3(1)	2(1)	-10(1)
O(2)	24(1)	18(1)	23(1)	2(1)	9(1)	3(1)
O(3)	16(1)	19(1)	21(1)	2(1)	0(1)	-1(1)
O(4)	19(1)	24(1)	19(1)	2(1)	0(1)	3(1)
O(5)	23(1)	30(1)	24(1)	-4(1)	7(1)	7(1)
O(6)	44(1)	20(1)	37(1)	0(1)	-8(1)	-7(1)

Table 17. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **39**.

	x	y	z	U(eq)
H(1)	8897	3641	1022	28
H(2A)	8630	4988	133	34
H(2B)	7296	5242	334	34
H(3A)	9794	5554	1639	40
H(3B)	8622	6297	1393	40
H(4A)	9015	5318	2941	37
H(4B)	7569	5466	2294	37
H(6A)	6506	2024	1145	25
H(6B)	7626	2549	1956	25
H(7A)	5331	2582	2113	32
H(7B)	6236	3500	2456	32
H(8A)	4367	3237	619	29
H(8B)	4568	4174	1296	29
H(10A)	6843	4299	-921	25
H(10B)	6832	3471	-1701	25
H(11A)	4705	4244	-1193	30
H(11B)	4741	3850	-2241	30
H(12A)	3831	2821	-1068	30
H(12B)	4407	2339	-1867	30
H(14A)	8848	2634	-1413	30
H(14B)	8840	3481	-656	30
H(15A)	10660	2040	-448	40
H(15B)	10773	2993	201	40
H(16A)	10117	1180	671	34
H(16B)	10420	2096	1374	34
H(18A)	5836	1182	-2105	32
H(18B)	6663	2109	-2195	32
H(19A)	6847	300	-2937	71
H(19B)	7519	1261	-3158	71
H(20A)	8598	-168	-1987	61
H(20B)	9272	782	-2219	61

H(21)	6236	867	-204	28
H(22A)	7892	372	1060	32
H(22B)	8619	-123	353	32
H(23A)	7591	-1434	782	37
H(23B)	6344	-814	761	37
H(24A)	7093	-1517	-852	40
H(24B)	5675	-1432	-756	40

Table 18. Torsion angles [°] for **39**.

O(1)-C(1)-C(2)-C(3)	22.55(16)
C(5)-C(1)-C(2)-C(3)	142.76(14)
C(1)-C(2)-C(3)-C(4)	-35.67(17)
C(2)-C(3)-C(4)-O(1)	36.45(17)
O(1)-C(1)-C(5)-O(2)	76.12(14)
C(2)-C(1)-C(5)-O(2)	-42.38(17)
O(1)-C(1)-C(5)-C(6)	-37.48(17)
C(2)-C(1)-C(5)-C(6)	-155.99(13)
O(1)-C(1)-C(5)-C(9)	-166.57(12)
C(2)-C(1)-C(5)-C(9)	74.93(18)
O(2)-C(5)-C(6)-C(7)	-13.93(15)
C(1)-C(5)-C(6)-C(7)	100.35(14)
C(9)-C(5)-C(6)-C(7)	-128.47(13)
C(5)-C(6)-C(7)-C(8)	30.50(16)
C(6)-C(7)-C(8)-O(2)	-36.87(16)
O(2)-C(5)-C(9)-O(3)	-64.17(13)
C(1)-C(5)-C(9)-O(3)	178.46(12)
C(6)-C(5)-C(9)-O(3)	49.78(15)
O(2)-C(5)-C(9)-C(10)	47.64(15)
C(1)-C(5)-C(9)-C(10)	-69.74(16)
C(6)-C(5)-C(9)-C(10)	161.59(12)
O(2)-C(5)-C(9)-C(13)	176.37(11)
C(1)-C(5)-C(9)-C(13)	59.00(17)
C(6)-C(5)-C(9)-C(13)	-69.68(15)
O(3)-C(9)-C(10)-C(11)	20.90(15)
C(5)-C(9)-C(10)-C(11)	-91.63(14)
C(13)-C(9)-C(10)-C(11)	139.51(13)
C(9)-C(10)-C(11)-C(12)	-26.11(16)
C(10)-C(11)-C(12)-O(3)	21.90(16)
O(3)-C(9)-C(13)-O(4)	-97.61(13)
C(10)-C(9)-C(13)-O(4)	147.26(12)
C(5)-C(9)-C(13)-O(4)	19.10(16)
O(3)-C(9)-C(13)-C(14)	149.46(12)
C(10)-C(9)-C(13)-C(14)	34.33(16)

C(5)-C(9)-C(13)-C(14)	-93.82(14)
O(3)-C(9)-C(13)-C(17)	22.50(16)
C(10)-C(9)-C(13)-C(17)	-92.63(15)
C(5)-C(9)-C(13)-C(17)	139.22(13)
O(4)-C(13)-C(14)-C(15)	23.26(16)
C(17)-C(13)-C(14)-C(15)	-91.81(15)
C(9)-C(13)-C(14)-C(15)	138.91(13)
C(13)-C(14)-C(15)-C(16)	-9.79(18)
C(14)-C(15)-C(16)-O(4)	-7.38(18)
O(4)-C(13)-C(17)-O(5)	-70.41(13)
C(14)-C(13)-C(17)-O(5)	42.21(15)
C(9)-C(13)-C(17)-O(5)	169.23(11)
O(4)-C(13)-C(17)-C(21)	46.72(15)
C(14)-C(13)-C(17)-C(21)	159.33(12)
C(9)-C(13)-C(17)-C(21)	-73.65(16)
O(4)-C(13)-C(17)-C(18)	175.24(12)
C(14)-C(13)-C(17)-C(18)	-72.15(16)
C(9)-C(13)-C(17)-C(18)	54.87(17)
O(5)-C(17)-C(18)-C(19)	13.25(19)
C(21)-C(17)-C(18)-C(19)	-102.50(17)
C(13)-C(17)-C(18)-C(19)	128.31(17)
C(17)-C(18)-C(19)-C(20)	-7.8(3)
C(18)-C(19)-C(20)-O(5)	-0.8(3)
O(5)-C(17)-C(21)-O(6)	-76.02(14)
C(18)-C(17)-C(21)-O(6)	37.96(17)
C(13)-C(17)-C(21)-O(6)	167.84(12)
O(5)-C(17)-C(21)-C(22)	41.62(18)
C(18)-C(17)-C(21)-C(22)	155.61(14)
C(13)-C(17)-C(21)-C(22)	-74.51(17)
O(6)-C(21)-C(22)-C(23)	-34.30(16)
C(17)-C(21)-C(22)-C(23)	-153.61(14)
C(21)-C(22)-C(23)-C(24)	38.08(16)
C(22)-C(23)-C(24)-O(6)	-28.83(17)
C(3)-C(4)-O(1)-C(1)	-23.38(17)
C(2)-C(1)-O(1)-C(4)	0.11(16)
C(5)-C(1)-O(1)-C(4)	-126.37(14)

C(7)-C(8)-O(2)-C(5)	29.59(15)
C(1)-C(5)-O(2)-C(8)	-127.23(12)
C(6)-C(5)-O(2)-C(8)	-9.54(15)
C(9)-C(5)-O(2)-C(8)	109.03(13)
C(11)-C(12)-O(3)-C(9)	-9.24(17)
C(10)-C(9)-O(3)-C(12)	-7.41(15)
C(5)-C(9)-O(3)-C(12)	110.00(13)
C(13)-C(9)-O(3)-C(12)	-128.23(13)
C(15)-C(16)-O(4)-C(13)	23.75(17)
C(14)-C(13)-O(4)-C(16)	-29.38(16)
C(17)-C(13)-O(4)-C(16)	88.06(14)
C(9)-C(13)-O(4)-C(16)	-147.15(12)
C(19)-C(20)-O(5)-C(17)	10.0(3)
C(21)-C(17)-O(5)-C(20)	104.08(16)
C(18)-C(17)-O(5)-C(20)	-14.46(19)
C(13)-C(17)-O(5)-C(20)	-135.23(16)
C(23)-C(24)-O(6)-C(21)	7.59(18)
C(22)-C(21)-O(6)-C(24)	17.15(17)
C(17)-C(21)-O(6)-C(24)	143.85(13)

X-Ray Crystallographic Details for **40**.

The data collection crystal was a clear, colorless plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 3.8, which means that 90% of the reflections were measured at least 3.8 times. Phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.043.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on 4867 intensities and 271 variables and resulted in agreement factors of R1(F) = 0.078 and wR2(F^2) = 0.144. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.051 for 3444 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.33 and -0.27 e/$Å^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

References

(1) DENZO: Otwinowski, Z. & Minor, W., Methods in Enzymology, Vol 276: Macromolecular Crystallography, part A, 307-326, (1997), Carter, Jr., C. W. & Sweet, R. M., Eds., Academic Press.

(2) SHELXS-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.

(3) SHELXL-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.

(4) WinGX-Version 1.70.01: Farrugia, L. J., J. Appl. Cryst., (1999), 32, 837-838.

(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 19. Crystallographic data for **40**.

Empirical formula	C24 H38 O6
Formula weight	422.54
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	P 2_1/n
Unit cell dimensions	a = 11.908(1) Å
	b = 14.174(1) Å
	c = 13.367(2) Å
	β= 109.920(4)°
Volume	2121.2(4) $Å^3$
Z	4
Density (calculated)	1.323 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	920
Crystal size	0.12 x 0.23 x 0.31 mm^3
Theta range for data collection	2.17 to 27.47°
Index ranges	-15<=h<=15, -16<=k<=18, -17<=l<=17
Reflections collected	39128
Independent reflections	4867 [R(int) = 0.043]
Completeness to theta = 27.47°	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4867 / 0 / 271
Goodness-of-fit on F^2	1.052
Final R indices [I>2sigma(I)]	R1 = 0.0512, wR2 = 0.1308
R indices (all data)	R1 = 0.0777, wR2 = 0.1445
Largest diff. peak and hole	0.330 and -0.267 e/$Å^3$

Table 20. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **40**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	5091(1)	2726(1)	1549(1)	27(1)
C(2)	6087(2)	2301(1)	1210(2)	34(1)
C(3)	7050(2)	3059(2)	1531(2)	46(1)
C(4)	6305(2)	3944(1)	1290(2)	44(1)
C(5)	3785(1)	2517(1)	849(1)	24(1)
C(6)	2936(2)	3271(1)	1026(1)	27(1)
C(7)	2744(2)	3972(1)	109(1)	34(1)
C(8)	2796(2)	3332(1)	-766(1)	36(1)
C(9)	3386(1)	1451(1)	923(1)	24(1)
C(10)	3960(2)	811(1)	272(1)	29(1)
C(11)	2928(2)	375(2)	-609(2)	56(1)
C(12)	1849(2)	920(1)	-629(2)	41(1)
C(13)	3589(1)	985(1)	2049(1)	24(1)
C(14)	3195(2)	-66(1)	1918(1)	30(1)
C(15)	4337(2)	-653(1)	2232(2)	38(1)
C(16)	5300(2)	21(1)	2863(2)	39(1)
C(17)	3043(1)	1489(1)	2827(1)	24(1)
C(18)	3434(2)	1000(1)	3929(1)	31(1)
C(19)	4337(2)	1679(1)	4669(2)	43(1)
C(20)	3968(2)	2613(1)	4142(1)	39(1)
C(21)	1676(1)	1615(1)	2376(1)	27(1)
C(22)	849(2)	761(1)	2102(2)	35(1)
C(23)	-304(2)	1173(1)	2176(2)	39(1)
C(24)	170(2)	1833(1)	3109(2)	40(1)
O(1)	5277(1)	3742(1)	1558(1)	37(1)
O(2)	3692(1)	2646(1)	-258(1)	30(1)
O(3)	2120(1)	1410(1)	352(1)	27(1)
O(4)	4870(1)	961(1)	2592(1)	26(1)
O(5)	3548(1)	2424(1)	3030(1)	28(1)
O(6)	1338(1)	2112(1)	3175(1)	33(1)

Table 21. Bond lengths [Å] and angles [°] for **40**.

C(1)-O(1)	1.458(2)
C(1)-C(2)	1.530(2)
C(1)-C(5)	1.546(2)
C(1)-H(1)	1.0000
C(2)-C(3)	1.522(3)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.507(3)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(1)	1.417(2)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-O(2)	1.4579(19)
C(5)-C(6)	1.544(2)
C(5)-C(9)	1.596(2)
C(6)-C(7)	1.533(2)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.498(3)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-O(2)	1.432(2)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-O(3)	1.4407(18)
C(9)-C(10)	1.565(2)
C(9)-C(13)	1.585(2)
C(10)-C(11)	1.515(3)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.492(3)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(12)-O(3)	1.421(2)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-O(4)	1.4492(18)
C(13)-C(14)	1.553(2)
C(13)-C(17)	1.574(2)
C(14)-C(15)	1.526(2)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.510(3)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-O(4)	1.428(2)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-O(5)	1.4414(19)
C(17)-C(21)	1.542(2)
C(17)-C(18)	1.550(2)
C(18)-C(19)	1.529(3)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.494(3)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-O(5)	1.423(2)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-O(6)	1.447(2)
C(21)-C(22)	1.525(2)
C(21)-H(21)	1.0000
C(22)-C(23)	1.525(2)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-C(24)	1.507(3)
C(23)-H(23A)	0.9900
C(23)-H(23B)	0.9900

C(24)-O(6)	1.420(2)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900

O(1)-C(1)-C(2)	105.08(13)
O(1)-C(1)-C(5)	108.27(13)
C(2)-C(1)-C(5)	117.84(14)
O(1)-C(1)-H(1)	108.4
C(2)-C(1)-H(1)	108.4
C(5)-C(1)-H(1)	108.4
C(3)-C(2)-C(1)	102.95(15)
C(3)-C(2)-H(2A)	111.2
C(1)-C(2)-H(2A)	111.2
C(3)-C(2)-H(2B)	111.2
C(1)-C(2)-H(2B)	111.2
H(2A)-C(2)-H(2B)	109.1
C(4)-C(3)-C(2)	101.29(15)
C(4)-C(3)-H(3A)	111.5
C(2)-C(3)-H(3A)	111.5
C(4)-C(3)-H(3B)	111.5
C(2)-C(3)-H(3B)	111.5
H(3A)-C(3)-H(3B)	109.3
O(1)-C(4)-C(3)	105.90(15)
O(1)-C(4)-H(4A)	110.6
C(3)-C(4)-H(4A)	110.6
O(1)-C(4)-H(4B)	110.6
C(3)-C(4)-H(4B)	110.6
H(4A)-C(4)-H(4B)	108.7
O(2)-C(5)-C(6)	103.92(12)
O(2)-C(5)-C(1)	107.65(12)
C(6)-C(5)-C(1)	110.54(13)
O(2)-C(5)-C(9)	105.09(12)
C(6)-C(5)-C(9)	114.93(12)
C(1)-C(5)-C(9)	113.80(12)
C(7)-C(6)-C(5)	104.72(13)
C(7)-C(6)-H(6A)	110.8

C(5)-C(6)-H(6A)	110.8
C(7)-C(6)-H(6B)	110.8
C(5)-C(6)-H(6B)	110.8
H(6A)-C(6)-H(6B)	108.9
C(8)-C(7)-C(6)	101.38(14)
C(8)-C(7)-H(7A)	111.5
C(6)-C(7)-H(7A)	111.5
C(8)-C(7)-H(7B)	111.5
C(6)-C(7)-H(7B)	111.5
H(7A)-C(7)-H(7B)	109.3
O(2)-C(8)-C(7)	105.27(14)
O(2)-C(8)-H(8A)	110.7
C(7)-C(8)-H(8A)	110.7
O(2)-C(8)-H(8B)	110.7
C(7)-C(8)-H(8B)	110.7
H(8A)-C(8)-H(8B)	108.8
O(3)-C(9)-C(10)	105.01(12)
O(3)-C(9)-C(13)	106.22(12)
C(10)-C(9)-C(13)	109.35(13)
O(3)-C(9)-C(5)	106.58(12)
C(10)-C(9)-C(5)	108.57(12)
C(13)-C(9)-C(5)	120.06(13)
C(11)-C(10)-C(9)	106.02(13)
C(11)-C(10)-H(10A)	110.5
C(9)-C(10)-H(10A)	110.5
C(11)-C(10)-H(10B)	110.5
C(9)-C(10)-H(10B)	110.5
H(10A)-C(10)-H(10B)	108.7
C(12)-C(11)-C(10)	105.58(15)
C(12)-C(11)-H(11A)	110.6
C(10)-C(11)-H(11A)	110.6
C(12)-C(11)-H(11B)	110.6
C(10)-C(11)-H(11B)	110.6
H(11A)-C(11)-H(11B)	108.8
O(3)-C(12)-C(11)	108.25(15)
O(3)-C(12)-H(12A)	110.0

C(11)-C(12)-H(12A)	110.0
O(3)-C(12)-H(12B)	110.0
C(11)-C(12)-H(12B)	110.0
H(12A)-C(12)-H(12B)	108.4
O(4)-C(13)-C(14)	105.09(12)
O(4)-C(13)-C(17)	106.31(12)
C(14)-C(13)-C(17)	109.43(13)
O(4)-C(13)-C(9)	106.21(12)
C(14)-C(13)-C(9)	110.56(13)
C(17)-C(13)-C(9)	118.30(13)
C(15)-C(14)-C(13)	106.53(14)
C(15)-C(14)-H(14A)	110.4
C(13)-C(14)-H(14A)	110.4
C(15)-C(14)-H(14B)	110.4
C(13)-C(14)-H(14B)	110.4
H(14A)-C(14)-H(14B)	108.6
C(16)-C(15)-C(14)	104.17(14)
C(16)-C(15)-H(15A)	110.9
C(14)-C(15)-H(15A)	110.9
C(16)-C(15)-H(15B)	110.9
C(14)-C(15)-H(15B)	110.9
H(15A)-C(15)-H(15B)	108.9
O(4)-C(16)-C(15)	108.04(14)
O(4)-C(16)-H(16A)	110.1
C(15)-C(16)-H(16A)	110.1
O(4)-C(16)-H(16B)	110.1
C(15)-C(16)-H(16B)	110.1
H(16A)-C(16)-H(16B)	108.4
O(5)-C(17)-C(21)	106.54(12)
O(5)-C(17)-C(18)	105.15(12)
C(21)-C(17)-C(18)	110.98(13)
O(5)-C(17)-C(13)	107.62(12)
C(21)-C(17)-C(13)	114.40(13)
C(18)-C(17)-C(13)	111.53(13)
C(19)-C(18)-C(17)	104.50(13)
C(19)-C(18)-H(18A)	110.9

C(17)-C(18)-H(18A)	110.9
C(19)-C(18)-H(18B)	110.9
C(17)-C(18)-H(18B)	110.9
H(18A)-C(18)-H(18B)	108.9
C(20)-C(19)-C(18)	102.93(15)
C(20)-C(19)-H(19A)	111.2
C(18)-C(19)-H(19A)	111.2
C(20)-C(19)-H(19B)	111.2
C(18)-C(19)-H(19B)	111.2
H(19A)-C(19)-H(19B)	109.1
O(5)-C(20)-C(19)	105.55(15)
O(5)-C(20)-H(20A)	110.6
C(19)-C(20)-H(20A)	110.6
O(5)-C(20)-H(20B)	110.6
C(19)-C(20)-H(20B)	110.6
H(20A)-C(20)-H(20B)	108.8
O(6)-C(21)-C(22)	104.14(13)
O(6)-C(21)-C(17)	106.87(13)
C(22)-C(21)-C(17)	120.82(14)
O(6)-C(21)-H(21)	108.1
C(22)-C(21)-H(21)	108.1
C(17)-C(21)-H(21)	108.1
C(21)-C(22)-C(23)	101.68(14)
C(21)-C(22)-H(22A)	111.4
C(23)-C(22)-H(22A)	111.4
C(21)-C(22)-H(22B)	111.4
C(23)-C(22)-H(22B)	111.4
H(22A)-C(22)-H(22B)	109.3
C(24)-C(23)-C(22)	101.56(14)
C(24)-C(23)-H(23A)	111.5
C(22)-C(23)-H(23A)	111.5
C(24)-C(23)-H(23B)	111.5
C(22)-C(23)-H(23B)	111.5
H(23A)-C(23)-H(23B)	109.3
O(6)-C(24)-C(23)	107.87(14)
O(6)-C(24)-H(24A)	110.1

C(23)-C(24)-H(24A)	110.1
O(6)-C(24)-H(24B)	110.1
C(23)-C(24)-H(24B)	110.1
H(24A)-C(24)-H(24B)	108.4
C(4)-O(1)-C(1)	109.85(13)
C(8)-O(2)-C(5)	110.73(12)
C(12)-O(3)-C(9)	112.10(12)
C(16)-O(4)-C(13)	111.97(12)
C(20)-O(5)-C(17)	110.22(12)
C(24)-O(6)-C(21)	109.38(12)

Table 22. Anisotropic displacement parameters ($Å^2 x 10^3$) for **40**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	27(1)	23(1)	32(1)	2(1)	10(1)	-3(1)
C(2)	25(1)	37(1)	43(1)	4(1)	14(1)	2(1)
C(3)	30(1)	55(1)	58(1)	0(1)	21(1)	-7(1)
C(4)	43(1)	43(1)	48(1)	2(1)	19(1)	-15(1)
C(5)	24(1)	24(1)	24(1)	1(1)	10(1)	-1(1)
C(6)	28(1)	26(1)	30(1)	-2(1)	14(1)	-3(1)
C(7)	35(1)	30(1)	39(1)	6(1)	14(1)	6(1)
C(8)	38(1)	37(1)	32(1)	8(1)	10(1)	6(1)
C(9)	22(1)	24(1)	25(1)	-2(1)	8(1)	0(1)
C(10)	31(1)	26(1)	31(1)	-3(1)	13(1)	4(1)
C(11)	40(1)	72(2)	51(1)	-33(1)	10(1)	2(1)
C(12)	35(1)	50(1)	33(1)	-17(1)	5(1)	-1(1)
C(13)	22(1)	21(1)	27(1)	-1(1)	8(1)	-1(1)
C(14)	35(1)	21(1)	34(1)	-2(1)	13(1)	-3(1)
C(15)	43(1)	23(1)	46(1)	2(1)	13(1)	6(1)
C(16)	37(1)	26(1)	49(1)	4(1)	9(1)	8(1)
C(17)	26(1)	21(1)	26(1)	1(1)	11(1)	-2(1)
C(18)	34(1)	26(1)	36(1)	8(1)	18(1)	4(1)
C(19)	44(1)	51(1)	30(1)	3(1)	8(1)	0(1)
C(20)	45(1)	37(1)	31(1)	-6(1)	8(1)	-8(1)
C(21)	28(1)	27(1)	29(1)	-2(1)	13(1)	-1(1)
C(22)	31(1)	35(1)	42(1)	-8(1)	16(1)	-8(1)
C(23)	30(1)	45(1)	44(1)	-5(1)	17(1)	-6(1)
C(24)	32(1)	44(1)	52(1)	-9(1)	23(1)	-6(1)
O(1)	30(1)	26(1)	55(1)	2(1)	14(1)	-5(1)
O(2)	34(1)	33(1)	27(1)	5(1)	15(1)	6(1)
O(3)	22(1)	30(1)	27(1)	-4(1)	7(1)	0(1)
O(4)	23(1)	23(1)	31(1)	3(1)	7(1)	3(1)
O(5)	32(1)	23(1)	30(1)	-2(1)	13(1)	-5(1)
O(6)	29(1)	33(1)	41(1)	-9(1)	18(1)	-4(1)

Table 23. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **40**.

	x	y	z	U(eq)
H(1)	5209	2516	2292	33
H(2A)	6381	1701	1588	41
H(2B)	5811	2186	433	41
H(3A)	7536	3016	2296	55
H(3B)	7581	3021	1101	55
H(4A)	6755	4479	1718	52
H(4B)	6078	4108	526	52
H(6A)	2170	2984	1002	32
H(6B)	3303	3587	1722	32
H(7A)	1959	4289	-76	41
H(7B)	3384	4453	283	41
H(8A)	2014	3024	-1116	43
H(8B)	3018	3690	-1307	43
H(10A)	4465	1192	-29	34
H(10B)	4461	315	733	34
H(11A)	3057	426	-1300	67
H(11B)	2838	-300	-458	67
H(12A)	1626	1374	-1228	49
H(12B)	1168	487	-726	49
H(14A)	2728	-216	2385	36
H(14B)	2693	-198	1172	36
H(15A)	4281	-1201	2671	45
H(15B)	4496	-882	1593	45
H(16A)	5489	-87	3634	47
H(16B)	6036	-79	2689	47
H(18A)	2742	904	4169	37
H(18B)	3807	380	3903	37
H(19A)	4283	1673	5392	52
H(19B)	5163	1517	4718	52
H(20A)	3327	2896	4360	46
H(20B)	4655	3052	4332	46

H(21)	1479	2025	1730	33
H(22A)	1156	243	2620	42
H(22B)	734	525	1376	42
H(23A)	-760	1517	1518	46
H(23B)	-816	677	2316	46
H(24A)	-354	2393	3007	48
H(24B)	194	1511	3773	48

Table 24. Torsion angles [°] for **40**.

O(1)-C(1)-C(2)-C(3)	24.48(18)
C(5)-C(1)-C(2)-C(3)	145.12(15)
C(1)-C(2)-C(3)-C(4)	-36.60(19)
C(2)-C(3)-C(4)-O(1)	36.6(2)
O(1)-C(1)-C(5)-O(2)	73.79(15)
C(2)-C(1)-C(5)-O(2)	-45.18(18)
O(1)-C(1)-C(5)-C(6)	-39.09(17)
C(2)-C(1)-C(5)-C(6)	-158.06(14)
O(1)-C(1)-C(5)-C(9)	-170.18(12)
C(2)-C(1)-C(5)-C(9)	70.85(18)
O(2)-C(5)-C(6)-C(7)	-17.90(16)
C(1)-C(5)-C(6)-C(7)	97.35(15)
C(9)-C(5)-C(6)-C(7)	-132.16(14)
C(5)-C(6)-C(7)-C(8)	33.31(17)
C(6)-C(7)-C(8)-O(2)	-36.97(17)
O(2)-C(5)-C(9)-O(3)	-70.36(14)
C(6)-C(5)-C(9)-O(3)	43.22(17)
C(1)-C(5)-C(9)-O(3)	172.12(12)
O(2)-C(5)-C(9)-C(10)	42.29(15)
C(6)-C(5)-C(9)-C(10)	155.87(13)
C(1)-C(5)-C(9)-C(10)	-75.23(16)
O(2)-C(5)-C(9)-C(13)	169.04(12)
C(6)-C(5)-C(9)-C(13)	-77.38(18)
C(1)-C(5)-C(9)-C(13)	51.52(18)
O(3)-C(9)-C(10)-C(11)	-4.53(19)
C(13)-C(9)-C(10)-C(11)	109.10(17)
C(5)-C(9)-C(10)-C(11)	-118.22(17)
C(9)-C(10)-C(11)-C(12)	13.4(2)
C(10)-C(11)-C(12)-O(3)	-17.9(2)
O(3)-C(9)-C(13)-O(4)	175.77(11)
C(10)-C(9)-C(13)-O(4)	62.93(15)
C(5)-C(9)-C(13)-O(4)	-63.45(16)
O(3)-C(9)-C(13)-C(14)	62.28(15)
C(10)-C(9)-C(13)-C(14)	-50.55(17)

C(5)-C(9)-C(13)-C(14)	-176.94(13)
O(3)-C(9)-C(13)-C(17)	-64.97(16)
C(10)-C(9)-C(13)-C(17)	-177.81(12)
C(5)-C(9)-C(13)-C(17)	55.81(19)
O(4)-C(13)-C(14)-C(15)	-9.93(17)
C(17)-C(13)-C(14)-C(15)	-123.73(15)
C(9)-C(13)-C(14)-C(15)	104.27(15)
C(13)-C(14)-C(15)-C(16)	18.41(19)
C(14)-C(15)-C(16)-O(4)	-20.7(2)
O(4)-C(13)-C(17)-O(5)	59.93(15)
C(14)-C(13)-C(17)-O(5)	172.94(12)
C(9)-C(13)-C(17)-O(5)	-59.27(17)
O(4)-C(13)-C(17)-C(21)	178.11(12)
C(14)-C(13)-C(17)-C(21)	-68.89(17)
C(9)-C(13)-C(17)-C(21)	58.91(18)
O(4)-C(13)-C(17)-C(18)	-54.91(16)
C(14)-C(13)-C(17)-C(18)	58.09(17)
C(9)-C(13)-C(17)-C(18)	-174.11(12)
O(5)-C(17)-C(18)-C(19)	-9.30(17)
C(21)-C(17)-C(18)-C(19)	-124.12(15)
C(13)-C(17)-C(18)-C(19)	107.06(15)
C(17)-C(18)-C(19)-C(20)	26.27(19)
C(18)-C(19)-C(20)-O(5)	-34.49(19)
O(5)-C(17)-C(21)-O(6)	-59.45(15)
C(18)-C(17)-C(21)-O(6)	54.49(16)
C(13)-C(17)-C(21)-O(6)	-178.24(12)
O(5)-C(17)-C(21)-C(22)	-178.02(14)
C(18)-C(17)-C(21)-C(22)	-64.07(19)
C(13)-C(17)-C(21)-C(22)	63.20(19)
O(6)-C(21)-C(22)-C(23)	36.16(17)
C(17)-C(21)-C(22)-C(23)	156.08(15)
C(21)-C(22)-C(23)-C(24)	-37.47(18)
C(22)-C(23)-C(24)-O(6)	26.4(2)
C(3)-C(4)-O(1)-C(1)	-22.1(2)
C(2)-C(1)-O(1)-C(4)	-1.82(18)
C(5)-C(1)-O(1)-C(4)	-128.58(15)

C(7)-C(8)-O(2)-C(5)	27.42(18)
C(6)-C(5)-O(2)-C(8)	-5.49(16)
C(1)-C(5)-O(2)-C(8)	-122.76(14)
C(9)-C(5)-O(2)-C(8)	115.62(14)
C(11)-C(12)-O(3)-C(9)	15.9(2)
C(10)-C(9)-O(3)-C(12)	-6.86(18)
C(13)-C(9)-O(3)-C(12)	-122.67(15)
C(5)-C(9)-O(3)-C(12)	108.22(15)
C(15)-C(16)-O(4)-C(13)	15.4(2)
C(14)-C(13)-O(4)-C(16)	-3.21(17)
C(17)-C(13)-O(4)-C(16)	112.76(14)
C(9)-C(13)-O(4)-C(16)	-120.41(14)
C(19)-C(20)-O(5)-C(17)	30.22(19)
C(21)-C(17)-O(5)-C(20)	105.24(15)
C(18)-C(17)-O(5)-C(20)	-12.62(17)
C(13)-C(17)-O(5)-C(20)	-131.64(14)
C(23)-C(24)-O(6)-C(21)	-3.9(2)
C(22)-C(21)-O(6)-C(24)	-20.58(18)
C(17)-C(21)-O(6)-C(24)	-149.51(14)

X-Ray Crystallographic Details for **51**.

The data collection crystal was a clear, colorless rectangular rod. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 4.0, which means that 90% of the reflections were measured at least 4.0 times. Phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.038.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4). There are two sets of disordered carbon atoms: C(15A) and C(15B) and C(23A) and C(23B). The first set was refined anisotropically, but the second set acquired odd looking ellipsoids. So the second set, C(23A) and C(23B), was kept isotropic. The occupancy factors of each set were refined: C(15A) and C(15B) refined to 0.600(8) and 0.400(8), respectively, and C(23A) and C(23B) refined to 0.553(9) and 0.447(9), respectively.

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on 4798 intensities and 281 variables and resulted in agreement factors of R1(F) = 0.079 and $wR2(F^2)$ = 0.127. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.049 for 3360 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.27 and -0.27 e/$Å^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

References

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(2) SHELXS-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.

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(4) WinGX-Version 1.70.01: Farrugia, L. J., J. Appl. Cryst., (1999), 32, 837-838.

(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 25. Crystallographic data for **51**.

Empirical formula	C24 H38 O6
Formula weight	422.54
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P\ 2_1/n$
Unit cell dimensions	a = 12.194(2) Å
	b = 14.252(2) Å
	c = 12.946(2) Å
	β= 109.525(8)°
Volume	2120.6(6) $Å^3$
Z	4
Density (calculated)	1.324 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	920
Crystal size	0.23 x 0.23 x 0.38 mm^3
Theta range for data collection	2.20 to 27.48°
Index ranges	-15<=h<=15, -18<=k<=18, -16<=l<=16
Reflections collected	21491
Independent reflections	4798 [R(int) = 0.038]
Completeness to theta = 27.48°	98.5 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4798 / 0 / 281
Goodness-of-fit on F^2	1.051
Final R indices [I>2sigma(I)]	R1 = 0.0491, wR2 = 0.1144
R indices (all data)	R1 = 0.0789, wR2 = 0.1269
Largest diff. peak and hole	0.266 and -0.273 $e/Å^3$

Table 26. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **51**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1744(1)	1688(1)	7159(1)	21(1)
C(2)	932(1)	829(1)	6881(1)	28(1)
C(3)	-63(2)	1105(1)	7289(2)	48(1)
C(4)	217(1)	2094(1)	7708(1)	30(1)
C(5)	3068(1)	1512(1)	7592(1)	20(1)
C(6)	3379(2)	952(1)	8686(1)	30(1)
C(7)	4214(2)	1582(1)	9551(1)	39(1)
C(8)	3984(2)	2549(1)	9042(1)	32(1)
C(9)	3563(1)	1047(1)	6746(1)	20(1)
C(10)	4865(1)	800(1)	7294(1)	26(1)
C(11)	4924(2)	-264(1)	7484(2)	38(1)
C(12)	3741(2)	-627(1)	6808(2)	40(1)
C(13)	3328(1)	1537(1)	5589(1)	19(1)
C(14)	3733(1)	858(1)	4840(1)	25(1)
C(15A)	2625(3)	314(3)	4180(3)	29(1)*
C(15B)	2678(4)	791(6)	3785(5)	33(2)*
C(16)	1668(1)	975(1)	4169(1)	33(1)
C(17)	3796(1)	2566(1)	5522(1)	20(1)
C(18)	3102(1)	3359(1)	5852(1)	22(1)
C(19)	2741(2)	4031(1)	4872(1)	30(1)
C(20)	2688(2)	3385(1)	3932(1)	34(1)
C(21)	5100(1)	2707(1)	6126(1)	25(1)
C(22)	5593(1)	3668(1)	5974(2)	34(1)
C(23A)	6917(3)	3419(2)	6320(4)	31(1)*
C(23B)	6726(4)	3490(3)	5760(5)	29(1)*
C(24)	6872(1)	2451(1)	5875(2)	34(1)
O(1)	1450(1)	2163(1)	8014(1)	26(1)
O(2)	3664(1)	2398(1)	7885(1)	24(1)
O(3)	2968(1)	157(1)	6484(1)	27(1)
O(4)	2082(1)	1608(1)	5066(1)	23(1)
O(5)	3609(1)	2726(1)	4370(1)	27(1)

O(6)	5774(1)	2046(1)	5743(1)	31(1)

*The site occupancy factors are as follows for the two disordered sets of carbon atoms:
C(15A) is 0.600(8), C(15B) is 0.400(8), C(23A) is 0.553(9), and C(23B) is 0.447(9).
Atoms C(23A) and C(23B) were refined only isotropically.

Table 27. Bond lengths [Å] and angles [°] for **51**.

C(1)-O(1)	1.4422(18)
C(1)-C(2)	1.539(2)
C(1)-C(5)	1.542(2)
C(1)-H(1)	1.0000
C(2)-C(3)	1.528(2)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.508(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(1)	1.4236(18)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-O(2)	1.4432(17)
C(5)-C(6)	1.558(2)
C(5)-C(9)	1.564(2)
C(6)-C(7)	1.528(2)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.513(3)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-O(2)	1.4320(18)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-O(3)	1.4440(17)
C(9)-C(10)	1.547(2)
C(9)-C(13)	1.589(2)
C(10)-C(11)	1.534(2)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.508(3)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(12)-O(3)	1.4312(19)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-O(4)	1.4460(16)
C(13)-C(14)	1.563(2)
C(13)-C(17)	1.587(2)
C(14)-C(15B)	1.534(5)
C(14)-C(15A)	1.544(3)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(14)-H(14C)	0.9900
C(14)-H(14D)	0.9900
C(15A)-C(16)	1.496(4)
C(15A)-H(15A)	0.9900
C(15A)-H(15B)	0.9900
C(15B)-C(16)	1.497(5)
C(15B)-H(15C)	0.9900
C(15B)-H(15D)	0.9900
C(16)-O(4)	1.4236(18)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(16)-H(16C)	0.9900
C(16)-H(16D)	0.9900
C(17)-O(5)	1.4496(17)
C(17)-C(21)	1.533(2)
C(17)-C(18)	1.554(2)
C(18)-C(19)	1.532(2)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.510(2)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-O(5)	1.4285(19)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-O(6)	1.4425(19)

C(21)-C(22)	1.535(2)
C(21)-H(21)	1.0000
C(22)-C(23B)	1.518(4)
C(22)-C(23A)	1.565(4)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(22)-H(22C)	0.9900
C(22)-H(22D)	0.9900
C(23A)-C(24)	1.489(4)
C(23A)-H(23A)	0.9900
C(23A)-H(23B)	0.9900
C(23B)-C(24)	1.493(4)
C(23B)-H(23C)	0.9900
C(23B)-H(23D)	0.9900
C(24)-O(6)	1.4149(19)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900
C(24)-H(24C)	0.9900
C(24)-H(24D)	0.9900

O(1)-C(1)-C(2)	104.36(12)
O(1)-C(1)-C(5)	107.24(11)
C(2)-C(1)-C(5)	117.98(13)
O(1)-C(1)-H(1)	109.0
C(2)-C(1)-H(1)	109.0
C(5)-C(1)-H(1)	109.0
C(3)-C(2)-C(1)	103.75(13)
C(3)-C(2)-H(2A)	111.0
C(1)-C(2)-H(2A)	111.0
C(3)-C(2)-H(2B)	111.0
C(1)-C(2)-H(2B)	111.0
H(2A)-C(2)-H(2B)	109.0
C(4)-C(3)-C(2)	104.70(14)
C(4)-C(3)-H(3A)	110.8
C(2)-C(3)-H(3A)	110.8
C(4)-C(3)-H(3B)	110.8

C(2)-C(3)-H(3B)	110.8
H(3A)-C(3)-H(3B)	108.9
O(1)-C(4)-C(3)	104.86(13)
O(1)-C(4)-H(4A)	110.8
C(3)-C(4)-H(4A)	110.8
O(1)-C(4)-H(4B)	110.8
C(3)-C(4)-H(4B)	110.8
H(4A)-C(4)-H(4B)	108.9
O(2)-C(5)-C(1)	109.10(12)
O(2)-C(5)-C(6)	105.12(11)
C(1)-C(5)-C(6)	108.73(12)
O(2)-C(5)-C(9)	106.03(11)
C(1)-C(5)-C(9)	114.62(11)
C(6)-C(5)-C(9)	112.73(12)
C(7)-C(6)-C(5)	105.41(13)
C(7)-C(6)-H(6A)	110.7
C(5)-C(6)-H(6A)	110.7
C(7)-C(6)-H(6B)	110.7
C(5)-C(6)-H(6B)	110.7
H(6A)-C(6)-H(6B)	108.8
C(8)-C(7)-C(6)	103.55(13)
C(8)-C(7)-H(7A)	111.0
C(6)-C(7)-H(7A)	111.0
C(8)-C(7)-H(7B)	111.0
C(6)-C(7)-H(7B)	111.1
H(7A)-C(7)-H(7B)	109.0
O(2)-C(8)-C(7)	105.36(14)
O(2)-C(8)-H(8A)	110.7
C(7)-C(8)-H(8A)	110.7
O(2)-C(8)-H(8B)	110.7
C(7)-C(8)-H(8B)	110.7
H(8A)-C(8)-H(8B)	108.8
O(3)-C(9)-C(10)	105.39(11)
O(3)-C(9)-C(5)	104.76(11)
C(10)-C(9)-C(5)	110.62(12)
O(3)-C(9)-C(13)	104.31(11)

C(10)-C(9)-C(13)	111.50(12)
C(5)-C(9)-C(13)	118.92(12)
C(11)-C(10)-C(9)	106.55(13)
C(11)-C(10)-H(10A)	110.4
C(9)-C(10)-H(10A)	110.4
C(11)-C(10)-H(10B)	110.4
C(9)-C(10)-H(10B)	110.4
H(10A)-C(10)-H(10B)	108.6
C(12)-C(11)-C(10)	105.15(13)
C(12)-C(11)-H(11A)	110.7
C(10)-C(11)-H(11A)	110.7
C(12)-C(11)-H(11B)	110.7
C(10)-C(11)-H(11B)	110.7
H(11A)-C(11)-H(11B)	108.8
O(3)-C(12)-C(11)	108.06(14)
O(3)-C(12)-H(12A)	110.1
C(11)-C(12)-H(12A)	110.1
O(3)-C(12)-H(12B)	110.1
C(11)-C(12)-H(12B)	110.1
H(12A)-C(12)-H(12B)	108.4
O(4)-C(13)-C(14)	104.64(11)
O(4)-C(13)-C(17)	104.54(11)
C(14)-C(13)-C(17)	110.18(12)
O(4)-C(13)-C(9)	107.59(11)
C(14)-C(13)-C(9)	108.50(12)
C(17)-C(13)-C(9)	120.21(11)
C(15B)-C(14)-C(13)	104.3(2)
C(15A)-C(14)-C(13)	104.81(15)
C(15A)-C(14)-H(14A)	110.8
C(13)-C(14)-H(14A)	110.8
C(15A)-C(14)-H(14B)	110.8
C(13)-C(14)-H(14B)	110.8
H(14A)-C(14)-H(14B)	108.9
C(15B)-C(14)-H(14C)	110.9
C(13)-C(14)-H(14C)	110.9
C(15B)-C(14)-H(14D)	110.9

C(13)-C(14)-H(14D)	110.9
H(14C)-C(14)-H(14D)	108.9
C(16)-C(15A)-C(14)	103.0(2)
C(16)-C(15A)-H(15A)	111.2
C(14)-C(15A)-H(15A)	111.2
C(16)-C(15A)-H(15B)	111.2
C(14)-C(15A)-H(15B)	111.2
H(15A)-C(15A)-H(15B)	109.1
C(16)-C(15B)-C(14)	103.4(3)
C(16)-C(15B)-H(15C)	111.1
C(14)-C(15B)-H(15C)	111.1
C(16)-C(15B)-H(15D)	111.1
C(14)-C(15B)-H(15D)	111.1
H(15C)-C(15B)-H(15D)	109.0
O(4)-C(16)-C(15A)	109.17(15)
O(4)-C(16)-C(15B)	106.0(2)
O(4)-C(16)-H(16A)	109.8
C(15A)-C(16)-H(16A)	109.8
O(4)-C(16)-H(16B)	109.8
C(15A)-C(16)-H(16B)	109.8
H(16A)-C(16)-H(16B)	108.3
O(4)-C(16)-H(16C)	110.5
C(15B)-C(16)-H(16C)	110.5
O(4)-C(16)-H(16D)	110.5
C(15B)-C(16)-H(16D)	110.5
H(16C)-C(16)-H(16D)	108.7
O(5)-C(17)-C(21)	106.48(12)
O(5)-C(17)-C(18)	104.85(11)
C(21)-C(17)-C(18)	109.42(12)
O(5)-C(17)-C(13)	105.39(11)
C(21)-C(17)-C(13)	115.04(12)
C(18)-C(17)-C(13)	114.71(12)
C(19)-C(18)-C(17)	105.01(12)
C(19)-C(18)-H(18A)	110.7
C(17)-C(18)-H(18A)	110.7
C(19)-C(18)-H(18B)	110.7

C(17)-C(18)-H(18B)	110.7
H(18A)-C(18)-H(18B)	108.8
C(20)-C(19)-C(18)	102.09(13)
C(20)-C(19)-H(19A)	111.4
C(18)-C(19)-H(19A)	111.4
C(20)-C(19)-H(19B)	111.4
C(18)-C(19)-H(19B)	111.4
H(19A)-C(19)-H(19B)	109.2
O(5)-C(20)-C(19)	105.37(12)
O(5)-C(20)-H(20A)	110.7
C(19)-C(20)-H(20A)	110.7
O(5)-C(20)-H(20B)	110.7
C(19)-C(20)-H(20B)	110.7
H(20A)-C(20)-H(20B)	108.8
O(6)-C(21)-C(17)	110.64(12)
O(6)-C(21)-C(22)	104.27(12)
C(17)-C(21)-C(22)	115.77(13)
O(6)-C(21)-H(21)	108.6
C(17)-C(21)-H(21)	108.6
C(22)-C(21)-H(21)	108.6
C(23B)-C(22)-C(21)	107.04(19)
C(21)-C(22)-C(23A)	99.85(17)
C(21)-C(22)-H(22A)	111.8
C(23A)-C(22)-H(22A)	111.8
C(21)-C(22)-H(22B)	111.8
C(23A)-C(22)-H(22B)	111.8
H(22A)-C(22)-H(22B)	109.5
C(23B)-C(22)-H(22C)	110.3
C(21)-C(22)-H(22C)	110.3
C(23B)-C(22)-H(22D)	110.3
C(21)-C(22)-H(22D)	110.3
H(22C)-C(22)-H(22D)	108.6
C(24)-C(23A)-C(22)	101.5(2)
C(24)-C(23A)-H(23A)	111.5
C(22)-C(23A)-H(23A)	111.5
C(24)-C(23A)-H(23B)	111.5

C(22)-C(23A)-H(23B)	111.5
H(23A)-C(23A)-H(23B)	109.3
C(24)-C(23B)-C(22)	103.5(3)
C(24)-C(23B)-H(23C)	111.1
C(22)-C(23B)-H(23C)	111.1
C(24)-C(23B)-H(23D)	111.1
C(22)-C(23B)-H(23D)	111.1
H(23C)-C(23B)-H(23D)	109.0
O(6)-C(24)-C(23A)	109.60(17)
O(6)-C(24)-C(23B)	108.43(19)
O(6)-C(24)-H(24A)	109.7
C(23A)-C(24)-H(24A)	109.7
O(6)-C(24)-H(24B)	109.7
C(23A)-C(24)-H(24B)	109.7
H(24A)-C(24)-H(24B)	108.2
O(6)-C(24)-H(24C)	110.0
C(23B)-C(24)-H(24C)	110.0
O(6)-C(24)-H(24D)	110.0
C(23B)-C(24)-H(24D)	110.0
H(24C)-C(24)-H(24D)	108.4
C(4)-O(1)-C(1)	105.29(11)
C(8)-O(2)-C(5)	110.10(11)
C(12)-O(3)-C(9)	112.82(12)
C(16)-O(4)-C(13)	111.63(11)
C(20)-O(5)-C(17)	110.10(11)
C(24)-O(6)-C(21)	108.46(12)

Table 28. Anisotropic displacement parameters ($Å^2$x 10^3) for **51**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$[$h^2a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	24(1)	20(1)	24(1)	-2(1)	13(1)	-1(1)
C(2)	24(1)	25(1)	36(1)	-5(1)	13(1)	-4(1)
C(3)	37(1)	35(1)	83(2)	-17(1)	36(1)	-9(1)
C(4)	25(1)	32(1)	39(1)	-2(1)	18(1)	0(1)
C(5)	21(1)	17(1)	23(1)	2(1)	8(1)	-2(1)
C(6)	33(1)	30(1)	31(1)	10(1)	16(1)	5(1)
C(7)	33(1)	59(1)	22(1)	8(1)	7(1)	2(1)
C(8)	32(1)	44(1)	20(1)	-7(1)	8(1)	-10(1)
C(9)	20(1)	16(1)	26(1)	1(1)	8(1)	-2(1)
C(10)	21(1)	29(1)	30(1)	6(1)	9(1)	5(1)
C(11)	40(1)	33(1)	44(1)	14(1)	20(1)	15(1)
C(12)	49(1)	21(1)	53(1)	7(1)	21(1)	10(1)
C(13)	16(1)	21(1)	21(1)	-2(1)	6(1)	2(1)
C(14)	22(1)	26(1)	28(1)	-6(1)	11(1)	2(1)
C(15A)	29(2)	25(2)	31(2)	-10(2)	9(1)	-2(1)
C(15B)	28(2)	36(4)	35(3)	-17(3)	10(2)	-4(2)
C(16)	26(1)	42(1)	30(1)	-16(1)	6(1)	-2(1)
C(17)	22(1)	21(1)	17(1)	0(1)	9(1)	2(1)
C(18)	25(1)	19(1)	25(1)	1(1)	12(1)	0(1)
C(19)	35(1)	24(1)	33(1)	8(1)	15(1)	7(1)
C(20)	42(1)	35(1)	25(1)	10(1)	12(1)	14(1)
C(21)	23(1)	23(1)	32(1)	0(1)	13(1)	1(1)
C(22)	30(1)	25(1)	50(1)	0(1)	18(1)	-5(1)
C(24)	21(1)	44(1)	38(1)	1(1)	13(1)	-3(1)
O(1)	26(1)	26(1)	31(1)	-7(1)	16(1)	-2(1)
O(2)	27(1)	25(1)	19(1)	-3(1)	7(1)	-8(1)
O(3)	28(1)	14(1)	42(1)	-1(1)	16(1)	-1(1)
O(4)	16(1)	28(1)	23(1)	-7(1)	4(1)	1(1)
O(5)	34(1)	31(1)	21(1)	5(1)	14(1)	8(1)
O(6)	24(1)	27(1)	51(1)	-3(1)	22(1)	-1(1)

Table 29. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **51**.

	x	y	z	U(eq)
H(1)	1552	2106	6502	26
H(2A)	642	710	6081	33
H(2B)	1337	261	7262	33
H(3A)	-819	1081	6685	57
H(3B)	-97	680	7883	57
H(4A)	-168	2555	7128	36
H(4B)	-39	2206	8347	36
H(6A)	2671	821	8874	36
H(6B)	3757	349	8627	36
H(7A)	4045	1561	10248	46
H(7B)	5033	1392	9694	46
H(8A)	3345	2861	9222	38
H(8B)	4690	2944	9310	38
H(10A)	5312	978	6811	32
H(10B)	5193	1137	7998	32
H(11A)	5538	-548	7242	45
H(11B)	5092	-409	8270	45
H(12A)	3788	-959	6152	48
H(12B)	3451	-1073	7244	48
H(14A)	4046	1214	4344	30
H(14B)	4341	424	5286	30
H(14C)	4417	1115	4687	30
H(14D)	3935	234	5188	30
H(15A)	2617	179	3428	34
H(15B)	2562	-284	4546	34
H(15C)	2722	1268	3244	39
H(15D)	2623	160	3452	39
H(16A)	1409	1329	3471	40
H(16B)	995	617	4228	40
H(16C)	1014	1257	3574	40
H(16D)	1396	384	4405	40

H(18A)	2409	3100	5989	27
H(18B)	3595	3684	6522	27
H(19A)	1973	4319	4773	36
H(19B)	3325	4533	4956	36
H(20A)	1927	3061	3661	41
H(20B)	2802	3742	3320	41
H(21)	5262	2600	6927	30
H(22A)	5439	4149	6460	41
H(22B)	5281	3883	5203	41
H(22C)	5037	4004	5347	41
H(22D)	5735	4055	6641	41
H(23A)	7335	3852	5984	37
H(23B)	7291	3429	7126	37
H(23C)	6672	3696	5015	35
H(23D)	7382	3820	6304	35
H(24A)	7000	2471	5159	40
H(24B)	7495	2064	6383	40
H(24C)	7180	2203	5311	40
H(24D)	7430	2292	6606	40

Table 30. Torsion angles [°] for **51**.

O(1)-C(1)-C(2)-C(3)	21.51(17)
C(5)-C(1)-C(2)-C(3)	140.34(15)
C(1)-C(2)-C(3)-C(4)	2.7(2)
C(2)-C(3)-C(4)-O(1)	-26.4(2)
O(1)-C(1)-C(5)-O(2)	-58.06(14)
C(2)-C(1)-C(5)-O(2)	-175.37(12)
O(1)-C(1)-C(5)-C(6)	56.07(15)
C(2)-C(1)-C(5)-C(6)	-61.24(17)
O(1)-C(1)-C(5)-C(9)	-176.76(11)
C(2)-C(1)-C(5)-C(9)	65.93(17)
O(2)-C(5)-C(6)-C(7)	-2.50(17)
C(1)-C(5)-C(6)-C(7)	-119.22(14)
C(9)-C(5)-C(6)-C(7)	112.54(15)
C(5)-C(6)-C(7)-C(8)	20.42(18)
C(6)-C(7)-C(8)-O(2)	-31.55(17)
O(2)-C(5)-C(9)-O(3)	178.95(10)
C(1)-C(5)-C(9)-O(3)	-60.63(15)
C(6)-C(5)-C(9)-O(3)	64.47(14)
O(2)-C(5)-C(9)-C(10)	65.85(14)
C(1)-C(5)-C(9)-C(10)	-173.74(12)
C(6)-C(5)-C(9)-C(10)	-48.64(16)
O(2)-C(5)-C(9)-C(13)	-65.09(15)
C(1)-C(5)-C(9)-C(13)	55.32(17)
C(6)-C(5)-C(9)-C(13)	-179.58(12)
O(3)-C(9)-C(10)-C(11)	-8.92(16)
C(5)-C(9)-C(10)-C(11)	103.78(15)
C(13)-C(9)-C(10)-C(11)	-121.50(14)
C(9)-C(10)-C(11)-C(12)	13.75(18)
C(10)-C(11)-C(12)-O(3)	-13.75(19)
O(3)-C(9)-C(13)-O(4)	57.92(13)
C(10)-C(9)-C(13)-O(4)	171.18(11)
C(5)-C(9)-C(13)-O(4)	-58.27(16)
O(3)-C(9)-C(13)-C(14)	-54.76(13)
C(10)-C(9)-C(13)-C(14)	58.50(15)

C(5)-C(9)-C(13)-C(14)	-170.95(12)
O(3)-C(9)-C(13)-C(17)	177.22(11)
C(10)-C(9)-C(13)-C(17)	-69.52(17)
C(5)-C(9)-C(13)-C(17)	61.03(17)
O(4)-C(13)-C(14)-C(15B)	13.4(4)
C(17)-C(13)-C(14)-C(15B)	-98.4(4)
C(9)-C(13)-C(14)-C(15B)	128.1(4)
O(4)-C(13)-C(14)-C(15A)	-20.4(3)
C(17)-C(13)-C(14)-C(15A)	-132.2(2)
C(9)-C(13)-C(14)-C(15A)	94.3(2)
C(15B)-C(14)-C(15A)-C(16)	-67.7(4)
C(13)-C(14)-C(15A)-C(16)	25.8(3)
C(15A)-C(14)-C(15B)-C(16)	67.9(5)
C(13)-C(14)-C(15B)-C(16)	-27.4(5)
C(14)-C(15A)-C(16)-O(4)	-22.7(4)
C(14)-C(15A)-C(16)-C(15B)	67.5(4)
C(14)-C(15B)-C(16)-O(4)	32.1(5)
C(14)-C(15B)-C(16)-C(15A)	-68.7(5)
O(4)-C(13)-C(17)-O(5)	-68.73(13)
C(14)-C(13)-C(17)-O(5)	43.20(14)
C(9)-C(13)-C(17)-O(5)	170.45(11)
O(4)-C(13)-C(17)-C(21)	174.31(11)
C(14)-C(13)-C(17)-C(21)	-73.77(15)
C(9)-C(13)-C(17)-C(21)	53.49(18)
O(4)-C(13)-C(17)-C(18)	46.06(15)
C(14)-C(13)-C(17)-C(18)	157.99(11)
C(9)-C(13)-C(17)-C(18)	-74.76(16)
O(5)-C(17)-C(18)-C(19)	-10.12(15)
C(21)-C(17)-C(18)-C(19)	103.76(14)
C(13)-C(17)-C(18)-C(19)	-125.22(13)
C(17)-C(18)-C(19)-C(20)	27.45(16)
C(18)-C(19)-C(20)-O(5)	-35.69(16)
O(5)-C(17)-C(21)-O(6)	-60.65(15)
C(18)-C(17)-C(21)-O(6)	-173.47(12)
C(13)-C(17)-C(21)-O(6)	55.69(17)
O(5)-C(17)-C(21)-C(22)	57.66(17)

C(18)-C(17)-C(21)-C(22)	-55.15(17)
C(13)-C(17)-C(21)-C(22)	174.00(13)
O(6)-C(21)-C(22)-C(23B)	-13.3(3)
C(17)-C(21)-C(22)-C(23B)	-135.1(3)
O(6)-C(21)-C(22)-C(23A)	-38.9(2)
C(17)-C(21)-C(22)-C(23A)	-160.7(2)
C(23B)-C(22)-C(23A)-C(24)	-72.4(5)
C(21)-C(22)-C(23A)-C(24)	36.5(3)
C(21)-C(22)-C(23B)-C(24)	-3.7(4)
C(23A)-C(22)-C(23B)-C(24)	73.4(5)
C(22)-C(23A)-C(24)-O(6)	-22.6(3)
C(22)-C(23A)-C(24)-C(23B)	69.6(5)
C(22)-C(23B)-C(24)-O(6)	20.2(4)
C(22)-C(23B)-C(24)-C(23A)	-76.9(5)
C(3)-C(4)-O(1)-C(1)	41.50(17)
C(2)-C(1)-O(1)-C(4)	-39.38(14)
C(5)-C(1)-O(1)-C(4)	-165.28(12)
C(7)-C(8)-O(2)-C(5)	31.80(16)
C(1)-C(5)-O(2)-C(8)	98.37(14)
C(6)-C(5)-O(2)-C(8)	-18.09(16)
C(9)-C(5)-O(2)-C(8)	-137.69(12)
C(11)-C(12)-O(3)-C(9)	8.64(19)
C(10)-C(9)-O(3)-C(12)	0.31(17)
C(5)-C(9)-O(3)-C(12)	-116.45(13)
C(13)-C(9)-O(3)-C(12)	117.86(13)
C(15A)-C(16)-O(4)-C(13)	10.3(3)
C(15B)-C(16)-O(4)-C(13)	-24.9(4)
C(14)-C(13)-O(4)-C(16)	6.71(16)
C(17)-C(13)-O(4)-C(16)	122.57(13)
C(9)-C(13)-O(4)-C(16)	-108.56(14)
C(19)-C(20)-O(5)-C(17)	31.16(17)
C(21)-C(17)-O(5)-C(20)	-128.71(13)
C(18)-C(17)-O(5)-C(20)	-12.79(16)
C(13)-C(17)-O(5)-C(20)	108.65(14)
C(23A)-C(24)-O(6)-C(21)	-2.0(3)
C(23B)-C(24)-O(6)-C(21)	-30.4(3)

C(17)-C(21)-O(6)-C(24)	151.72(13)
C(22)-C(21)-O(6)-C(24)	26.61(16)

X-Ray Crystallographic Details for **52**.

The data collection crystal was a clear, colorless prismatic rod. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 4.3, which means that 90% of the reflections were measured at least 4.3 times. Phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.039.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on 4893 intensities and 271 variables and resulted in agreement factors of R1(F) = 0.060 and wR2(F^2) = 0.116. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.043 for 3759 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.26 and -0.24 e/$Å^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

References

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Table 31. Crystallographic data for **52**.

Empirical formula	C24 H38 O6
Formula weight	422.54
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P\ 2_1/n$
Unit cell dimensions	a = 11.961(1) Å
	b = 14.493(1) Å
	c = 13.123(1) Å
	β= 110.603(4)°
Volume	2129.4(3) $Å^3$
Z	4
Density (calculated)	1.318 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	920
Crystal size	0.23 x 0.31 x 0.38 mm^3
Theta range for data collection	2.86 to 27.49°
Index ranges	-15<=h<=15, -18<=k<=18, -17<=l<=17
Reflections collected	45150
Independent reflections	4893 [R(int) = 0.039]
Completeness to theta = 27.49°	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4893 / 0 / 271
Goodness-of-fit on F^2	1.050
Final R indices [I>2sigma(I)]	R1 = 0.0431, wR2 = 0.1088
R indices (all data)	R1 = 0.0601, wR2 = 0.1164
Largest diff. peak and hole	0.259 and -0.240 e/$Å^3$

Table 32. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **52**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3336(1)	1636(1)	7844(1)	21(1)
C(2)	4228(1)	831(1)	8186(1)	29(1)
C(3)	5285(1)	1162(1)	7877(1)	40(1)
C(4)	4884(1)	2087(1)	7339(1)	30(1)
C(5)	1995(1)	1408(1)	7406(1)	20(1)
C(6)	1701(1)	830(1)	6343(1)	30(1)
C(7)	775(1)	1395(1)	5465(1)	35(1)
C(8)	960(1)	2365(1)	5919(1)	30(1)
C(9)	1529(1)	945(1)	8261(1)	20(1)
C(10)	211(1)	646(1)	7726(1)	26(1)
C(11)	223(2)	-404(1)	7591(1)	38(1)
C(12)	1489(2)	-707(1)	8219(1)	37(1)
C(13)	1740(1)	1466(1)	9379(1)	18(1)
C(14)	1316(1)	810(1)	10119(1)	23(1)
C(15)	2385(1)	734(1)	11179(1)	31(1)
C(16)	3428(1)	889(1)	10805(1)	29(1)
C(17)	1230(1)	2478(1)	9383(1)	18(1)
C(18)	1997(1)	3247(1)	9130(1)	20(1)
C(19)	2207(1)	3945(1)	10052(1)	24(1)
C(20)	2235(1)	3325(1)	10986(1)	26(1)
C(21)	-107(1)	2618(1)	8694(1)	21(1)
C(22)	-1027(1)	2211(1)	9149(1)	27(1)
C(23)	-1951(1)	2970(1)	8983(1)	39(1)
C(24)	-1192(1)	3820(1)	9106(1)	38(1)
O(1)	3614(1)	2087(1)	6980(1)	24(1)
O(2)	1326(1)	2253(1)	7076(1)	22(1)
O(3)	2204(1)	104(1)	8574(1)	26(1)
O(4)	3012(1)	1556(1)	9946(1)	21(1)
O(5)	1338(1)	2645(1)	10509(1)	21(1)
O(6)	-354(1)	3599(1)	8597(1)	28(1)

Table 33. Bond lengths [Å] and angles [°] for **52**.

C(1)-O(1)	1.4463(14)
C(1)-C(5)	1.5370(18)
C(1)-C(2)	1.5372(18)
C(1)-H(1)	1.0000
C(2)-C(3)	1.5341(19)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.512(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(1)	1.4234(16)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-O(2)	1.4433(15)
C(5)-C(6)	1.5578(17)
C(5)-C(9)	1.5690(17)
C(6)-C(7)	1.525(2)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.513(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-O(2)	1.4332(15)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-O(3)	1.4404(15)
C(9)-C(10)	1.5443(18)
C(9)-C(13)	1.5892(16)
C(10)-C(11)	1.534(2)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.513(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(12)-O(3)	1.4320(17)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-O(4)	1.4459(14)
C(13)-C(14)	1.5647(17)
C(13)-C(17)	1.5891(17)
C(14)-C(15)	1.5266(19)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.5093(19)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-O(4)	1.4354(15)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-O(5)	1.4574(13)
C(17)-C(21)	1.5483(17)
C(17)-C(18)	1.5529(17)
C(18)-C(19)	1.5284(17)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.5106(18)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-O(5)	1.4290(16)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-O(6)	1.4489(16)
C(21)-C(22)	1.5432(18)
C(21)-H(21)	1.0000
C(22)-C(23)	1.520(2)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-C(24)	1.505(2)
C(23)-H(23A)	0.9900
C(23)-H(23B)	0.9900

C(24)-O(6)	1.4226(17)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900

O(1)-C(1)-C(5)	107.54(9)
O(1)-C(1)-C(2)	104.17(9)
C(5)-C(1)-C(2)	118.20(11)
O(1)-C(1)-H(1)	108.9
C(5)-C(1)-H(1)	108.9
C(2)-C(1)-H(1)	108.9
C(3)-C(2)-C(1)	103.75(11)
C(3)-C(2)-H(2A)	111.0
C(1)-C(2)-H(2A)	111.0
C(3)-C(2)-H(2B)	111.0
C(1)-C(2)-H(2B)	111.0
H(2A)-C(2)-H(2B)	109.0
C(4)-C(3)-C(2)	104.19(11)
C(4)-C(3)-H(3A)	110.9
C(2)-C(3)-H(3A)	110.9
C(4)-C(3)-H(3B)	110.9
C(2)-C(3)-H(3B)	110.9
H(3A)-C(3)-H(3B)	108.9
O(1)-C(4)-C(3)	106.32(11)
O(1)-C(4)-H(4A)	110.5
C(3)-C(4)-H(4A)	110.5
O(1)-C(4)-H(4B)	110.5
C(3)-C(4)-H(4B)	110.5
H(4A)-C(4)-H(4B)	108.7
O(2)-C(5)-C(1)	109.01(10)
O(2)-C(5)-C(6)	105.12(10)
C(1)-C(5)-C(6)	109.10(10)
O(2)-C(5)-C(9)	106.18(9)
C(1)-C(5)-C(9)	114.26(10)
C(6)-C(5)-C(9)	112.68(10)
C(7)-C(6)-C(5)	105.25(11)
C(7)-C(6)-H(6A)	110.7

C(5)-C(6)-H(6A)	110.7
C(7)-C(6)-H(6B)	110.7
C(5)-C(6)-H(6B)	110.7
H(6A)-C(6)-H(6B)	108.8
C(8)-C(7)-C(6)	103.57(11)
C(8)-C(7)-H(7A)	111.0
C(6)-C(7)-H(7A)	111.0
C(8)-C(7)-H(7B)	111.0
C(6)-C(7)-H(7B)	111.0
H(7A)-C(7)-H(7B)	109.0
O(2)-C(8)-C(7)	105.06(11)
O(2)-C(8)-H(8A)	110.7
C(7)-C(8)-H(8A)	110.7
O(2)-C(8)-H(8B)	110.7
C(7)-C(8)-H(8B)	110.7
H(8A)-C(8)-H(8B)	108.8
O(3)-C(9)-C(10)	105.80(10)
O(3)-C(9)-C(5)	105.13(9)
C(10)-C(9)-C(5)	110.97(10)
O(3)-C(9)-C(13)	104.33(9)
C(10)-C(9)-C(13)	110.95(10)
C(5)-C(9)-C(13)	118.48(10)
C(11)-C(10)-C(9)	106.38(11)
C(11)-C(10)-H(10A)	110.5
C(9)-C(10)-H(10A)	110.5
C(11)-C(10)-H(10B)	110.5
C(9)-C(10)-H(10B)	110.5
H(10A)-C(10)-H(10B)	108.6
C(12)-C(11)-C(10)	105.85(12)
C(12)-C(11)-H(11A)	110.6
C(10)-C(11)-H(11A)	110.6
C(12)-C(11)-H(11B)	110.6
C(10)-C(11)-H(11B)	110.6
H(11A)-C(11)-H(11B)	108.7
O(3)-C(12)-C(11)	107.94(12)
O(3)-C(12)-H(12A)	110.1

C(11)-C(12)-H(12A)	110.1
O(3)-C(12)-H(12B)	110.1
C(11)-C(12)-H(12B)	110.1
H(12A)-C(12)-H(12B)	108.4
O(4)-C(13)-C(14)	104.36(9)
O(4)-C(13)-C(17)	104.50(9)
C(14)-C(13)-C(17)	110.51(9)
O(4)-C(13)-C(9)	108.30(9)
C(14)-C(13)-C(9)	107.68(10)
C(17)-C(13)-C(9)	120.31(9)
C(15)-C(14)-C(13)	105.01(10)
C(15)-C(14)-H(14A)	110.7
C(13)-C(14)-H(14A)	110.7
C(15)-C(14)-H(14B)	110.7
C(13)-C(14)-H(14B)	110.7
H(14A)-C(14)-H(14B)	108.8
C(16)-C(15)-C(14)	102.48(11)
C(16)-C(15)-H(15A)	111.3
C(14)-C(15)-H(15A)	111.3
C(16)-C(15)-H(15B)	111.3
C(14)-C(15)-H(15B)	111.3
H(15A)-C(15)-H(15B)	109.2
O(4)-C(16)-C(15)	104.96(10)
O(4)-C(16)-H(16A)	110.8
C(15)-C(16)-H(16A)	110.8
O(4)-C(16)-H(16B)	110.8
C(15)-C(16)-H(16B)	110.8
H(16A)-C(16)-H(16B)	108.8
O(5)-C(17)-C(21)	106.01(9)
O(5)-C(17)-C(18)	104.42(9)
C(21)-C(17)-C(18)	110.27(10)
O(5)-C(17)-C(13)	105.06(9)
C(21)-C(17)-C(13)	115.96(10)
C(18)-C(17)-C(13)	113.96(10)
C(19)-C(18)-C(17)	104.53(9)
C(19)-C(18)-H(18A)	110.8

C(17)-C(18)-H(18A)	110.8
C(19)-C(18)-H(18B)	110.8
C(17)-C(18)-H(18B)	110.8
H(18A)-C(18)-H(18B)	108.9
C(20)-C(19)-C(18)	101.45(10)
C(20)-C(19)-H(19A)	111.5
C(18)-C(19)-H(19A)	111.5
C(20)-C(19)-H(19B)	111.5
C(18)-C(19)-H(19B)	111.5
H(19A)-C(19)-H(19B)	109.3
O(5)-C(20)-C(19)	104.86(10)
O(5)-C(20)-H(20A)	110.8
C(19)-C(20)-H(20A)	110.8
O(5)-C(20)-H(20B)	110.8
C(19)-C(20)-H(20B)	110.8
H(20A)-C(20)-H(20B)	108.9
O(6)-C(21)-C(22)	104.91(10)
O(6)-C(21)-C(17)	108.52(10)
C(22)-C(21)-C(17)	116.99(10)
O(6)-C(21)-H(21)	108.7
C(22)-C(21)-H(21)	108.7
C(17)-C(21)-H(21)	108.7
C(23)-C(22)-C(21)	104.48(11)
C(23)-C(22)-H(22A)	110.9
C(21)-C(22)-H(22A)	110.9
C(23)-C(22)-H(22B)	110.9
C(21)-C(22)-H(22B)	110.9
H(22A)-C(22)-H(22B)	108.9
C(24)-C(23)-C(22)	101.32(12)
C(24)-C(23)-H(23A)	111.5
C(22)-C(23)-H(23A)	111.5
C(24)-C(23)-H(23B)	111.5
C(22)-C(23)-H(23B)	111.5
H(23A)-C(23)-H(23B)	109.3
O(6)-C(24)-C(23)	105.30(12)
O(6)-C(24)-H(24A)	110.7

C(23)-C(24)-H(24A)	110.7
O(6)-C(24)-H(24B)	110.7
C(23)-C(24)-H(24B)	110.7
H(24A)-C(24)-H(24B)	108.8
C(4)-O(1)-C(1)	104.66(9)
C(8)-O(2)-C(5)	110.16(9)
C(12)-O(3)-C(9)	113.04(10)
C(16)-O(4)-C(13)	110.74(9)
C(20)-O(5)-C(17)	110.08(9)
C(24)-O(6)-C(21)	109.63(10)

Table 34. Anisotropic displacement parameters ($Å^2 x 10^3$) for **52**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	23(1)	20(1)	22(1)	4(1)	13(1)	3(1)
C(2)	28(1)	28(1)	37(1)	11(1)	18(1)	9(1)
C(3)	32(1)	39(1)	57(1)	19(1)	26(1)	12(1)
C(4)	23(1)	34(1)	38(1)	11(1)	16(1)	4(1)
C(5)	23(1)	17(1)	21(1)	-2(1)	10(1)	1(1)
C(6)	38(1)	28(1)	30(1)	-12(1)	20(1)	-7(1)
C(7)	32(1)	52(1)	21(1)	-10(1)	10(1)	-11(1)
C(8)	27(1)	43(1)	18(1)	5(1)	7(1)	7(1)
C(9)	22(1)	16(1)	24(1)	-2(1)	11(1)	1(1)
C(10)	26(1)	27(1)	26(1)	-8(1)	13(1)	-6(1)
C(11)	44(1)	30(1)	44(1)	-16(1)	21(1)	-15(1)
C(12)	50(1)	18(1)	52(1)	-7(1)	28(1)	-8(1)
C(13)	17(1)	18(1)	19(1)	-1(1)	7(1)	0(1)
C(14)	26(1)	20(1)	26(1)	2(1)	13(1)	-1(1)
C(15)	32(1)	33(1)	30(1)	13(1)	13(1)	4(1)
C(16)	27(1)	31(1)	25(1)	11(1)	6(1)	5(1)
C(17)	20(1)	19(1)	14(1)	-1(1)	7(1)	1(1)
C(18)	23(1)	18(1)	21(1)	-1(1)	10(1)	0(1)
C(19)	26(1)	19(1)	27(1)	-5(1)	10(1)	-2(1)
C(20)	27(1)	27(1)	22(1)	-8(1)	7(1)	-5(1)
C(21)	20(1)	21(1)	21(1)	-2(1)	6(1)	2(1)
C(22)	21(1)	34(1)	27(1)	-5(1)	10(1)	-1(1)
C(23)	29(1)	56(1)	37(1)	5(1)	18(1)	14(1)
C(24)	43(1)	41(1)	30(1)	-3(1)	12(1)	21(1)
O(1)	24(1)	26(1)	28(1)	9(1)	14(1)	4(1)
O(2)	25(1)	24(1)	17(1)	1(1)	7(1)	6(1)
O(3)	32(1)	15(1)	36(1)	0(1)	18(1)	2(1)
O(4)	18(1)	22(1)	21(1)	5(1)	6(1)	2(1)
O(5)	23(1)	24(1)	17(1)	-5(1)	9(1)	-4(1)
O(6)	27(1)	24(1)	33(1)	0(1)	9(1)	8(1)

Table 35. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **52**.

	x	y	z	U(eq)
H(1)	3503	2072	8470	25
H(2A)	4478	714	8978	35
H(2B)	3877	260	7789	35
H(3A)	6014	1227	8531	47
H(3B)	5449	725	7366	47
H(4A)	5216	2594	7865	36
H(4B)	5157	2169	6715	36
H(6A)	2427	737	6156	36
H(6B)	1373	220	6428	36
H(7A)	917	1363	4766	42
H(7B)	-44	1174	5349	42
H(8A)	1584	2687	5720	36
H(8B)	209	2724	5637	36
H(10A)	-249	817	8195	31
H(10B)	-157	949	7010	31
H(11A)	-342	-702	7888	46
H(11B)	-6	-571	6813	46
H(12A)	1512	-1085	8854	45
H(12B)	1800	-1084	7747	45
H(14A)	617	1073	10254	27
H(14B)	1096	196	9774	27
H(15A)	2354	1211	11708	37
H(15B)	2424	116	11513	37
H(16A)	4127	1127	11409	34
H(16B)	3657	307	10534	34
H(18A)	2764	2996	9124	24
H(18B)	1565	3536	8416	24
H(19A)	2973	4276	10208	28
H(19B)	1547	4399	9883	28
H(20A)	3030	3033	11317	31
H(20B)	2052	3678	11554	31

H(21)	-262	2359	7950	25
H(22A)	-1398	1646	8747	32
H(22B)	-647	2058	9931	32
H(23A)	-2578	2936	8251	47
H(23B)	-2329	2945	9543	47
H(24A)	-1688	4355	8744	46
H(24B)	-777	3969	9884	46

Table 36. Torsion angles [°] for **52**.

O(1)-C(1)-C(2)-C(3)	26.11(14)
C(5)-C(1)-C(2)-C(3)	145.33(12)
C(1)-C(2)-C(3)-C(4)	-2.90(16)
C(2)-C(3)-C(4)-O(1)	-21.85(16)
O(1)-C(1)-C(5)-O(2)	-59.20(11)
C(2)-C(1)-C(5)-O(2)	-176.64(10)
O(1)-C(1)-C(5)-C(6)	55.08(13)
C(2)-C(1)-C(5)-C(6)	-62.36(14)
O(1)-C(1)-C(5)-C(9)	-177.79(9)
C(2)-C(1)-C(5)-C(9)	64.77(14)
O(2)-C(5)-C(6)-C(7)	-3.99(14)
C(1)-C(5)-C(6)-C(7)	-120.78(12)
C(9)-C(5)-C(6)-C(7)	111.21(12)
C(5)-C(6)-C(7)-C(8)	21.91(14)
C(6)-C(7)-C(8)-O(2)	-32.46(14)
O(2)-C(5)-C(9)-O(3)	179.77(9)
C(1)-C(5)-C(9)-O(3)	-60.05(12)
C(6)-C(5)-C(9)-O(3)	65.21(13)
O(2)-C(5)-C(9)-C(10)	65.83(12)
C(1)-C(5)-C(9)-C(10)	-173.98(10)
C(6)-C(5)-C(9)-C(10)	-48.72(14)
O(2)-C(5)-C(9)-C(13)	-64.23(13)
C(1)-C(5)-C(9)-C(13)	55.95(14)
C(6)-C(5)-C(9)-C(13)	-178.79(10)
O(3)-C(9)-C(10)-C(11)	-9.22(13)
C(5)-C(9)-C(10)-C(11)	104.29(12)
C(13)-C(9)-C(10)-C(11)	-121.79(11)
C(9)-C(10)-C(11)-C(12)	9.86(15)
C(10)-C(11)-C(12)-O(3)	-6.98(15)
O(3)-C(9)-C(13)-O(4)	54.73(11)
C(10)-C(9)-C(13)-O(4)	168.23(10)
C(5)-C(9)-C(13)-O(4)	-61.70(13)
O(3)-C(9)-C(13)-C(14)	-57.58(12)
C(10)-C(9)-C(13)-C(14)	55.92(13)

C(5)-C(9)-C(13)-C(14)	-174.01(10)
O(3)-C(9)-C(13)-C(17)	174.66(10)
C(10)-C(9)-C(13)-C(17)	-71.84(14)
C(5)-C(9)-C(13)-C(17)	58.23(15)
O(4)-C(13)-C(14)-C(15)	10.86(13)
C(17)-C(13)-C(14)-C(15)	-100.97(11)
C(9)-C(13)-C(14)-C(15)	125.81(11)
C(13)-C(14)-C(15)-C(16)	-27.96(14)
C(14)-C(15)-C(16)-O(4)	35.49(14)
O(4)-C(13)-C(17)-O(5)	-71.09(10)
C(14)-C(13)-C(17)-O(5)	40.65(12)
C(9)-C(13)-C(17)-O(5)	167.11(9)
O(4)-C(13)-C(17)-C(21)	172.25(9)
C(14)-C(13)-C(17)-C(21)	-76.01(12)
C(9)-C(13)-C(17)-C(21)	50.45(14)
O(4)-C(13)-C(17)-C(18)	42.60(12)
C(14)-C(13)-C(17)-C(18)	154.34(10)
C(9)-C(13)-C(17)-C(18)	-79.20(13)
O(5)-C(17)-C(18)-C(19)	-14.82(12)
C(21)-C(17)-C(18)-C(19)	98.67(11)
C(13)-C(17)-C(18)-C(19)	-128.89(10)
C(17)-C(18)-C(19)-C(20)	32.00(12)
C(18)-C(19)-C(20)-O(5)	-38.41(12)
O(5)-C(17)-C(21)-O(6)	76.07(11)
C(18)-C(17)-C(21)-O(6)	-36.40(12)
C(13)-C(17)-C(21)-O(6)	-167.80(9)
O(5)-C(17)-C(21)-C(22)	-42.30(13)
C(18)-C(17)-C(21)-C(22)	-154.77(10)
C(13)-C(17)-C(21)-C(22)	73.83(13)
O(6)-C(21)-C(22)-C(23)	15.11(13)
C(17)-C(21)-C(22)-C(23)	135.41(12)
C(21)-C(22)-C(23)-C(24)	-31.83(14)
C(22)-C(23)-C(24)-O(6)	38.08(14)
C(3)-C(4)-O(1)-C(1)	39.58(14)
C(5)-C(1)-O(1)-C(4)	-167.09(10)
C(2)-C(1)-O(1)-C(4)	-40.86(13)

C(7)-C(8)-O(2)-C(5)	31.69(14)
C(1)-C(5)-O(2)-C(8)	99.71(11)
C(6)-C(5)-O(2)-C(8)	-17.13(13)
C(9)-C(5)-O(2)-C(8)	-136.75(10)
C(11)-C(12)-O(3)-C(9)	1.15(15)
C(10)-C(9)-O(3)-C(12)	5.14(13)
C(5)-C(9)-O(3)-C(12)	-112.36(11)
C(13)-C(9)-O(3)-C(12)	122.27(11)
C(15)-C(16)-O(4)-C(13)	-30.46(14)
C(14)-C(13)-O(4)-C(16)	11.95(12)
C(17)-C(13)-O(4)-C(16)	128.04(10)
C(9)-C(13)-O(4)-C(16)	-102.57(11)
C(19)-C(20)-O(5)-C(17)	30.80(13)
C(21)-C(17)-O(5)-C(20)	-126.15(10)
C(18)-C(17)-O(5)-C(20)	-9.67(12)
C(13)-C(17)-O(5)-C(20)	110.56(11)
C(23)-C(24)-O(6)-C(21)	-30.18(14)
C(22)-C(21)-O(6)-C(24)	9.08(13)
C(17)-C(21)-O(6)-C(24)	-116.68(11)

X-Ray Crystallographic Details for **53**.

The data collection crystal was a colorless rhombus-shaped plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 3.7, which means that 90% of the reflections were measured at least 3.7 times. Phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.048.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on 4903 intensities and 271 variables and resulted in agreement factors of R1(F) = 0.078 and wR2(F^2) = 0.123. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.047 for 3347 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.35 and -0.21 e/$Å^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

References

(1) DENZO: Otwinowski, Z. & Minor, W., Methods in Enzymology, Vol 276: Macromolecular Crystallography, part A, 307-326, (1997), Carter, Jr., C. W. & Sweet, R. M., Eds., Academic Press.

(2) SHELXS-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.

(3) SHELXL-97: Sheldrick, G. M., Universitat Gottingen, Germany, 1997.

(4) WinGX-Version 1.70.01: Farrugia, L. J., J. Appl. Cryst., (1999), 32, 837-838.

(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 37. Crystallographic data for **53**.

Empirical formula	C24 H38 O6
Formula weight	422.54
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	P 2_1/c
Unit cell dimensions	a = 17.160(2) Å
	b = 8.814(1) Å
	c = 14.308(2) Å
	β= 96.688(5)°
Volume	2149.3(5) $Å^3$
Z	4
Density (calculated)	1.306 Mg/m^3
Absorption coefficient	0.092 mm^{-1}
F(000)	920
Crystal size	0.06 x 0.38 x 0.38 mm^3
Theta range for data collection	2.60 to 27.47°
Index ranges	-22<=h<=22, -11<=k<=11, -18<=l<=18
Reflections collected	40839
Independent reflections	4903 [R(int) = 0.048]
Completeness to theta = 27.47°	99.6 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4903 / 0 / 271
Goodness-of-fit on F^2	1.039
Final R indices [I>2sigma(I)]	R1 = 0.0470, wR2 = 0.1117
R indices (all data)	R1 = 0.0778, wR2 = 0.1231
Largest diff. peak and hole	0.349 and -0.213 e/$Å^3$

Table 38. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **53**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2393(1)	-349(2)	2522(1)	24(1)
C(2)	1847(1)	-1678(2)	2721(1)	32(1)
C(3)	1643(1)	-2448(2)	1771(1)	35(1)
C(4)	1861(1)	-1268(2)	1088(1)	31(1)
C(5)	3190(1)	-262(2)	3129(1)	23(1)
C(6)	3803(1)	606(2)	2619(1)	27(1)
C(7)	4290(1)	-631(2)	2215(1)	36(1)
C(8)	4249(1)	-1904(2)	2910(1)	37(1)
C(9)	3151(1)	332(2)	4172(1)	22(1)
C(10)	2790(1)	-858(2)	4792(1)	26(1)
C(11)	3352(1)	-922(2)	5698(1)	39(1)
C(12)	4123(1)	-682(2)	5332(1)	36(1)
C(13)	2820(1)	2029(2)	4233(1)	22(1)
C(14)	3336(1)	3004(2)	4964(1)	27(1)
C(15)	3920(1)	3784(2)	4402(1)	34(1)
C(16)	3435(1)	4023(2)	3460(1)	32(1)
C(17)	1936(1)	2196(2)	4413(1)	21(1)
C(18)	1815(1)	2023(2)	5463(1)	24(1)
C(19)	1027(1)	1209(2)	5459(1)	29(1)
C(20)	818(1)	673(2)	4446(1)	31(1)
C(21)	1611(1)	3743(2)	4031(1)	24(1)
C(22)	1346(1)	3838(2)	2965(1)	30(1)
C(23)	507(1)	4456(2)	2893(1)	40(1)
C(24)	463(1)	5102(2)	3862(1)	35(1)
O(1)	2542(1)	-557(1)	1554(1)	28(1)
O(2)	3486(1)	-1806(1)	3217(1)	30(1)
O(3)	3972(1)	369(1)	4562(1)	27(1)
O(4)	2901(1)	2770(1)	3343(1)	24(1)
O(5)	1468(1)	1015(1)	3941(1)	23(1)
O(6)	917(1)	4079(1)	4476(1)	27(1)

Table 39. Bond lengths [Å] and angles [°] for **53**.

C(1)-O(1)	1.4489(17)
C(1)-C(5)	1.5357(19)
C(1)-C(2)	1.546(2)
C(1)-H(1)	1.0000
C(2)-C(3)	1.524(2)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.504(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(1)	1.4207(18)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-O(2)	1.4524(18)
C(5)-C(6)	1.550(2)
C(5)-C(9)	1.590(2)
C(6)-C(7)	1.527(2)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.507(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-O(2)	1.4307(18)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-O(3)	1.4537(16)
C(9)-C(10)	1.549(2)
C(9)-C(13)	1.606(2)
C(10)-C(11)	1.523(2)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.493(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(12)-O(3)	1.4396(18)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-O(4)	1.4514(17)
C(13)-C(14)	1.550(2)
C(13)-C(17)	1.5734(19)
C(14)-C(15)	1.520(2)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.514(2)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-O(4)	1.4327(18)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-O(5)	1.4346(17)
C(17)-C(18)	1.5488(19)
C(17)-C(21)	1.549(2)
C(18)-C(19)	1.530(2)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-C(20)	1.527(2)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-O(5)	1.4289(17)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-O(6)	1.4449(17)
C(21)-C(22)	1.542(2)
C(21)-H(21)	1.0000
C(22)-C(23)	1.532(2)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-C(24)	1.508(2)
C(23)-H(23A)	0.9900
C(23)-H(23B)	0.9900

C(24)-O(6)	1.4259(18)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900

O(1)-C(1)-C(5)	107.59(11)
O(1)-C(1)-C(2)	105.00(11)
C(5)-C(1)-C(2)	116.88(12)
O(1)-C(1)-H(1)	109.0
C(5)-C(1)-H(1)	109.0
C(2)-C(1)-H(1)	109.0
C(3)-C(2)-C(1)	104.69(12)
C(3)-C(2)-H(2A)	110.8
C(1)-C(2)-H(2A)	110.8
C(3)-C(2)-H(2B)	110.8
C(1)-C(2)-H(2B)	110.8
H(2A)-C(2)-H(2B)	108.9
C(4)-C(3)-C(2)	102.91(13)
C(4)-C(3)-H(3A)	111.2
C(2)-C(3)-H(3A)	111.2
C(4)-C(3)-H(3B)	111.2
C(2)-C(3)-H(3B)	111.2
H(3A)-C(3)-H(3B)	109.1
O(1)-C(4)-C(3)	104.69(12)
O(1)-C(4)-H(4A)	110.8
C(3)-C(4)-H(4A)	110.8
O(1)-C(4)-H(4B)	110.8
C(3)-C(4)-H(4B)	110.8
H(4A)-C(4)-H(4B)	108.9
O(2)-C(5)-C(1)	106.37(12)
O(2)-C(5)-C(6)	104.73(11)
C(1)-C(5)-C(6)	111.53(12)
O(2)-C(5)-C(9)	106.22(11)
C(1)-C(5)-C(9)	114.52(11)
C(6)-C(5)-C(9)	112.63(12)
C(7)-C(6)-C(5)	104.86(12)
C(7)-C(6)-H(6A)	110.8

C(5)-C(6)-H(6A)	110.8
C(7)-C(6)-H(6B)	110.8
C(5)-C(6)-H(6B)	110.8
H(6A)-C(6)-H(6B)	108.9
C(8)-C(7)-C(6)	102.25(13)
C(8)-C(7)-H(7A)	111.3
C(6)-C(7)-H(7A)	111.3
C(8)-C(7)-H(7B)	111.3
C(6)-C(7)-H(7B)	111.3
H(7A)-C(7)-H(7B)	109.2
O(2)-C(8)-C(7)	105.80(12)
O(2)-C(8)-H(8A)	110.6
C(7)-C(8)-H(8A)	110.6
O(2)-C(8)-H(8B)	110.6
C(7)-C(8)-H(8B)	110.6
H(8A)-C(8)-H(8B)	108.7
O(3)-C(9)-C(10)	103.25(11)
O(3)-C(9)-C(5)	102.87(11)
C(10)-C(9)-C(5)	112.23(12)
O(3)-C(9)-C(13)	106.93(11)
C(10)-C(9)-C(13)	115.59(11)
C(5)-C(9)-C(13)	114.31(11)
C(11)-C(10)-C(9)	104.89(12)
C(11)-C(10)-H(10A)	110.8
C(9)-C(10)-H(10A)	110.8
C(11)-C(10)-H(10B)	110.8
C(9)-C(10)-H(10B)	110.8
H(10A)-C(10)-H(10B)	108.8
C(12)-C(11)-C(10)	101.25(13)
C(12)-C(11)-H(11A)	111.5
C(10)-C(11)-H(11A)	111.5
C(12)-C(11)-H(11B)	111.5
C(10)-C(11)-H(11B)	111.5
H(11A)-C(11)-H(11B)	109.3
O(3)-C(12)-C(11)	105.77(12)
O(3)-C(12)-H(12A)	110.6

C(11)-C(12)-H(12A)	110.6
O(3)-C(12)-H(12B)	110.6
C(11)-C(12)-H(12B)	110.6
H(12A)-C(12)-H(12B)	108.7
O(4)-C(13)-C(14)	103.69(11)
O(4)-C(13)-C(17)	107.07(10)
C(14)-C(13)-C(17)	108.88(11)
O(4)-C(13)-C(9)	107.50(11)
C(14)-C(13)-C(9)	112.04(11)
C(17)-C(13)-C(9)	116.73(11)
C(15)-C(14)-C(13)	104.72(12)
C(15)-C(14)-H(14A)	110.8
C(13)-C(14)-H(14A)	110.8
C(15)-C(14)-H(14B)	110.8
C(13)-C(14)-H(14B)	110.8
H(14A)-C(14)-H(14B)	108.9
C(16)-C(15)-C(14)	101.87(12)
C(16)-C(15)-H(15A)	111.4
C(14)-C(15)-H(15A)	111.4
C(16)-C(15)-H(15B)	111.4
C(14)-C(15)-H(15B)	111.4
H(15A)-C(15)-H(15B)	109.3
O(4)-C(16)-C(15)	105.94(12)
O(4)-C(16)-H(16A)	110.5
C(15)-C(16)-H(16A)	110.5
O(4)-C(16)-H(16B)	110.5
C(15)-C(16)-H(16B)	110.5
H(16A)-C(16)-H(16B)	108.7
O(5)-C(17)-C(18)	104.69(11)
O(5)-C(17)-C(21)	108.28(11)
C(18)-C(17)-C(21)	110.22(12)
O(5)-C(17)-C(13)	110.67(11)
C(18)-C(17)-C(13)	112.96(11)
C(21)-C(17)-C(13)	109.83(11)
C(19)-C(18)-C(17)	105.10(11)
C(19)-C(18)-H(18A)	110.7

C(17)-C(18)-H(18A)	110.7
C(19)-C(18)-H(18B)	110.7
C(17)-C(18)-H(18B)	110.7
H(18A)-C(18)-H(18B)	108.8
C(20)-C(19)-C(18)	105.01(11)
C(20)-C(19)-H(19A)	110.7
C(18)-C(19)-H(19A)	110.7
C(20)-C(19)-H(19B)	110.7
C(18)-C(19)-H(19B)	110.7
H(19A)-C(19)-H(19B)	108.8
O(5)-C(20)-C(19)	107.65(11)
O(5)-C(20)-H(20A)	110.2
C(19)-C(20)-H(20A)	110.2
O(5)-C(20)-H(20B)	110.2
C(19)-C(20)-H(20B)	110.2
H(20A)-C(20)-H(20B)	108.5
O(6)-C(21)-C(22)	105.19(11)
O(6)-C(21)-C(17)	107.84(11)
C(22)-C(21)-C(17)	116.68(12)
O(6)-C(21)-H(21)	109.0
C(22)-C(21)-H(21)	109.0
C(17)-C(21)-H(21)	109.0
C(23)-C(22)-C(21)	104.59(12)
C(23)-C(22)-H(22A)	110.8
C(21)-C(22)-H(22A)	110.8
C(23)-C(22)-H(22B)	110.8
C(21)-C(22)-H(22B)	110.8
H(22A)-C(22)-H(22B)	108.9
C(24)-C(23)-C(22)	102.70(12)
C(24)-C(23)-H(23A)	111.2
C(22)-C(23)-H(23A)	111.2
C(24)-C(23)-H(23B)	111.2
C(22)-C(23)-H(23B)	111.2
H(23A)-C(23)-H(23B)	109.1
O(6)-C(24)-C(23)	104.19(13)
O(6)-C(24)-H(24A)	110.9

C(23)-C(24)-H(24A)	110.9
O(6)-C(24)-H(24B)	110.9
C(23)-C(24)-H(24B)	110.9
H(24A)-C(24)-H(24B)	108.9
C(4)-O(1)-C(1)	106.09(11)
C(8)-O(2)-C(5)	110.68(11)
C(12)-O(3)-C(9)	111.06(11)
C(16)-O(4)-C(13)	111.70(11)
C(20)-O(5)-C(17)	110.06(10)
C(24)-O(6)-C(21)	106.07(10)

Table 40. Anisotropic displacement parameters ($Å^2 x 10^3$) for **53**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	25(1)	26(1)	23(1)	-1(1)	6(1)	-2(1)
C(2)	29(1)	36(1)	31(1)	0(1)	3(1)	-10(1)
C(3)	40(1)	26(1)	39(1)	-5(1)	8(1)	-6(1)
C(4)	33(1)	30(1)	30(1)	-7(1)	1(1)	-2(1)
C(5)	22(1)	18(1)	29(1)	1(1)	5(1)	2(1)
C(6)	23(1)	26(1)	32(1)	-4(1)	8(1)	-4(1)
C(7)	26(1)	34(1)	48(1)	-13(1)	14(1)	-4(1)
C(8)	27(1)	33(1)	50(1)	-11(1)	6(1)	5(1)
C(9)	15(1)	23(1)	28(1)	1(1)	1(1)	-1(1)
C(10)	23(1)	26(1)	27(1)	5(1)	1(1)	1(1)
C(11)	35(1)	42(1)	39(1)	14(1)	-4(1)	-1(1)
C(12)	30(1)	40(1)	36(1)	9(1)	-6(1)	4(1)
C(13)	21(1)	23(1)	22(1)	2(1)	3(1)	-1(1)
C(14)	24(1)	27(1)	30(1)	-5(1)	4(1)	-2(1)
C(15)	27(1)	29(1)	46(1)	-8(1)	6(1)	-7(1)
C(16)	30(1)	22(1)	47(1)	1(1)	16(1)	-5(1)
C(17)	19(1)	23(1)	20(1)	0(1)	3(1)	-2(1)
C(18)	24(1)	29(1)	21(1)	3(1)	4(1)	2(1)
C(19)	27(1)	34(1)	26(1)	7(1)	7(1)	1(1)
C(20)	23(1)	39(1)	32(1)	0(1)	10(1)	-7(1)
C(21)	22(1)	25(1)	25(1)	2(1)	8(1)	3(1)
C(22)	34(1)	32(1)	26(1)	8(1)	7(1)	7(1)
C(23)	32(1)	56(1)	31(1)	14(1)	5(1)	9(1)
C(24)	29(1)	42(1)	37(1)	11(1)	7(1)	12(1)
O(1)	30(1)	33(1)	23(1)	-2(1)	5(1)	-5(1)
O(2)	29(1)	22(1)	40(1)	-1(1)	7(1)	4(1)
O(3)	19(1)	29(1)	32(1)	4(1)	-1(1)	1(1)
O(4)	24(1)	23(1)	26(1)	3(1)	8(1)	-2(1)
O(5)	19(1)	27(1)	23(1)	0(1)	4(1)	-4(1)
O(6)	22(1)	35(1)	25(1)	4(1)	6(1)	9(1)

Table 41. Calculated hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **53**.

	x	y	z	U(eq)
H(1)	2107	629	2576	29
H(2A)	2120	-2392	3183	38
H(2B)	1368	-1298	2969	38
H(3A)	1951	-3389	1726	42
H(3B)	1077	-2694	1659	42
H(4A)	1977	-1745	493	38
H(4B)	1432	-524	946	38
H(6A)	3541	1252	2110	32
H(6B)	4138	1254	3066	32
H(7A)	4058	-935	1576	43
H(7B)	4837	-296	2190	43
H(8A)	4319	-2897	2607	44
H(8B)	4661	-1786	3449	44
H(10A)	2260	-542	4923	31
H(10B)	2752	-1861	4479	31
H(11A)	3240	-109	6141	47
H(11B)	3331	-1919	6013	47
H(12A)	4511	-256	5829	43
H(12B)	4328	-1652	5112	43
H(14A)	3013	3760	5257	32
H(14B)	3610	2358	5464	32
H(15A)	4100	4761	4692	41
H(15B)	4380	3130	4342	41
H(16A)	3144	4994	3455	39
H(16B)	3775	4041	2947	39
H(18A)	2244	1416	5803	29
H(18B)	1800	3028	5769	29
H(19A)	1075	336	5897	34
H(19B)	622	1910	5645	34
H(20A)	716	-432	4432	37
H(20B)	341	1200	4155	37

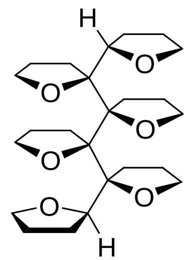
H(21)	2013	4546	4205	29
H(22A)	1690	4530	2654	36
H(22B)	1356	2823	2670	36
H(23A)	420	5252	2404	48
H(23B)	116	3637	2749	48
H(24A)	687	6138	3914	43
H(24B)	-87	5140	4008	43

Table 42. Torsion angles [°] for **53**.

O(1)-C(1)-C(2)-C(3)	-7.14(16)
C(5)-C(1)-C(2)-C(3)	-126.25(14)
C(1)-C(2)-C(3)-C(4)	-16.86(16)
C(2)-C(3)-C(4)-O(1)	35.99(16)
O(1)-C(1)-C(5)-O(2)	-76.13(14)
C(2)-C(1)-C(5)-O(2)	41.58(16)
O(1)-C(1)-C(5)-C(6)	37.49(16)
C(2)-C(1)-C(5)-C(6)	155.20(13)
O(1)-C(1)-C(5)-C(9)	166.87(11)
C(2)-C(1)-C(5)-C(9)	-75.42(17)
O(2)-C(5)-C(6)-C(7)	15.27(14)
C(1)-C(5)-C(6)-C(7)	-99.38(14)
C(9)-C(5)-C(6)-C(7)	130.25(13)
C(5)-C(6)-C(7)-C(8)	-29.82(15)
C(6)-C(7)-C(8)-O(2)	34.23(15)
O(2)-C(5)-C(9)-O(3)	65.54(13)
C(1)-C(5)-C(9)-O(3)	-177.37(11)
C(6)-C(5)-C(9)-O(3)	-48.54(15)
O(2)-C(5)-C(9)-C(10)	-44.78(14)
C(1)-C(5)-C(9)-C(10)	72.31(15)
C(6)-C(5)-C(9)-C(10)	-158.86(12)
O(2)-C(5)-C(9)-C(13)	-178.93(10)
C(1)-C(5)-C(9)-C(13)	-61.84(16)
C(6)-C(5)-C(9)-C(13)	66.99(15)
O(3)-C(9)-C(10)-C(11)	21.76(15)
C(5)-C(9)-C(10)-C(11)	131.84(13)
C(13)-C(9)-C(10)-C(11)	-94.62(14)
C(9)-C(10)-C(11)-C(12)	-34.99(17)
C(10)-C(11)-C(12)-O(3)	35.62(17)
O(3)-C(9)-C(13)-O(4)	93.25(12)
C(10)-C(9)-C(13)-O(4)	-152.46(11)
C(5)-C(9)-C(13)-O(4)	-19.89(15)
O(3)-C(9)-C(13)-C(14)	-20.04(15)
C(10)-C(9)-C(13)-C(14)	94.25(14)

C(5)-C(9)-C(13)-C(14)	-133.17(12)
O(3)-C(9)-C(13)-C(17)	-146.53(11)
C(10)-C(9)-C(13)-C(17)	-32.24(16)
C(5)-C(9)-C(13)-C(17)	100.34(13)
O(4)-C(13)-C(14)-C(15)	-23.22(15)
C(17)-C(13)-C(14)-C(15)	-136.96(12)
C(9)-C(13)-C(14)-C(15)	92.40(14)
C(13)-C(14)-C(15)-C(16)	33.69(16)
C(14)-C(15)-C(16)-O(4)	-32.27(15)
O(4)-C(13)-C(17)-O(5)	84.62(13)
C(14)-C(13)-C(17)-O(5)	-163.87(11)
C(9)-C(13)-C(17)-O(5)	-35.83(15)
O(4)-C(13)-C(17)-C(18)	-158.37(12)
C(14)-C(13)-C(17)-C(18)	-46.86(16)
C(9)-C(13)-C(17)-C(18)	81.18(15)
O(4)-C(13)-C(17)-C(21)	-34.88(14)
C(14)-C(13)-C(17)-C(21)	76.63(14)
C(9)-C(13)-C(17)-C(21)	-155.33(11)
O(5)-C(17)-C(18)-C(19)	-23.75(15)
C(21)-C(17)-C(18)-C(19)	92.50(14)
C(13)-C(17)-C(18)-C(19)	-144.23(13)
C(17)-C(18)-C(19)-C(20)	11.11(16)
C(18)-C(19)-C(20)-O(5)	5.42(17)
O(5)-C(17)-C(21)-O(6)	78.15(13)
C(18)-C(17)-C(21)-O(6)	-35.82(15)
C(13)-C(17)-C(21)-O(6)	-160.89(10)
O(5)-C(17)-C(21)-C(22)	-39.87(16)
C(18)-C(17)-C(21)-C(22)	-153.84(12)
C(13)-C(17)-C(21)-C(22)	81.09(15)
O(6)-C(21)-C(22)-C(23)	7.61(17)
C(17)-C(21)-C(22)-C(23)	127.07(14)
C(21)-C(22)-C(23)-C(24)	16.92(18)
C(22)-C(23)-C(24)-O(6)	-36.32(17)
C(3)-C(4)-O(1)-C(1)	-42.15(15)
C(5)-C(1)-O(1)-C(4)	155.50(12)
C(2)-C(1)-O(1)-C(4)	30.33(15)

C(7)-C(8)-O(2)-C(5)	-26.08(16)
C(1)-C(5)-O(2)-C(8)	124.65(12)
C(6)-C(5)-O(2)-C(8)	6.44(14)
C(9)-C(5)-O(2)-C(8)	-112.95(12)
C(11)-C(12)-O(3)-C(9)	-23.41(17)
C(10)-C(9)-O(3)-C(12)	0.66(15)
C(5)-C(9)-O(3)-C(12)	-116.25(13)
C(13)-C(9)-O(3)-C(12)	123.03(12)
C(15)-C(16)-O(4)-C(13)	18.78(15)
C(14)-C(13)-O(4)-C(16)	2.87(15)
C(17)-C(13)-O(4)-C(16)	117.91(12)
C(9)-C(13)-O(4)-C(16)	-115.93(12)
C(19)-C(20)-O(5)-C(17)	-21.74(16)
C(18)-C(17)-O(5)-C(20)	28.48(14)
C(21)-C(17)-O(5)-C(20)	-89.09(13)
C(13)-C(17)-O(5)-C(20)	150.47(11)
C(23)-C(24)-O(6)-C(21)	42.93(15)
C(22)-C(21)-O(6)-C(24)	-31.23(15)
C(17)-C(21)-O(6)-C(24)	-156.41(12)



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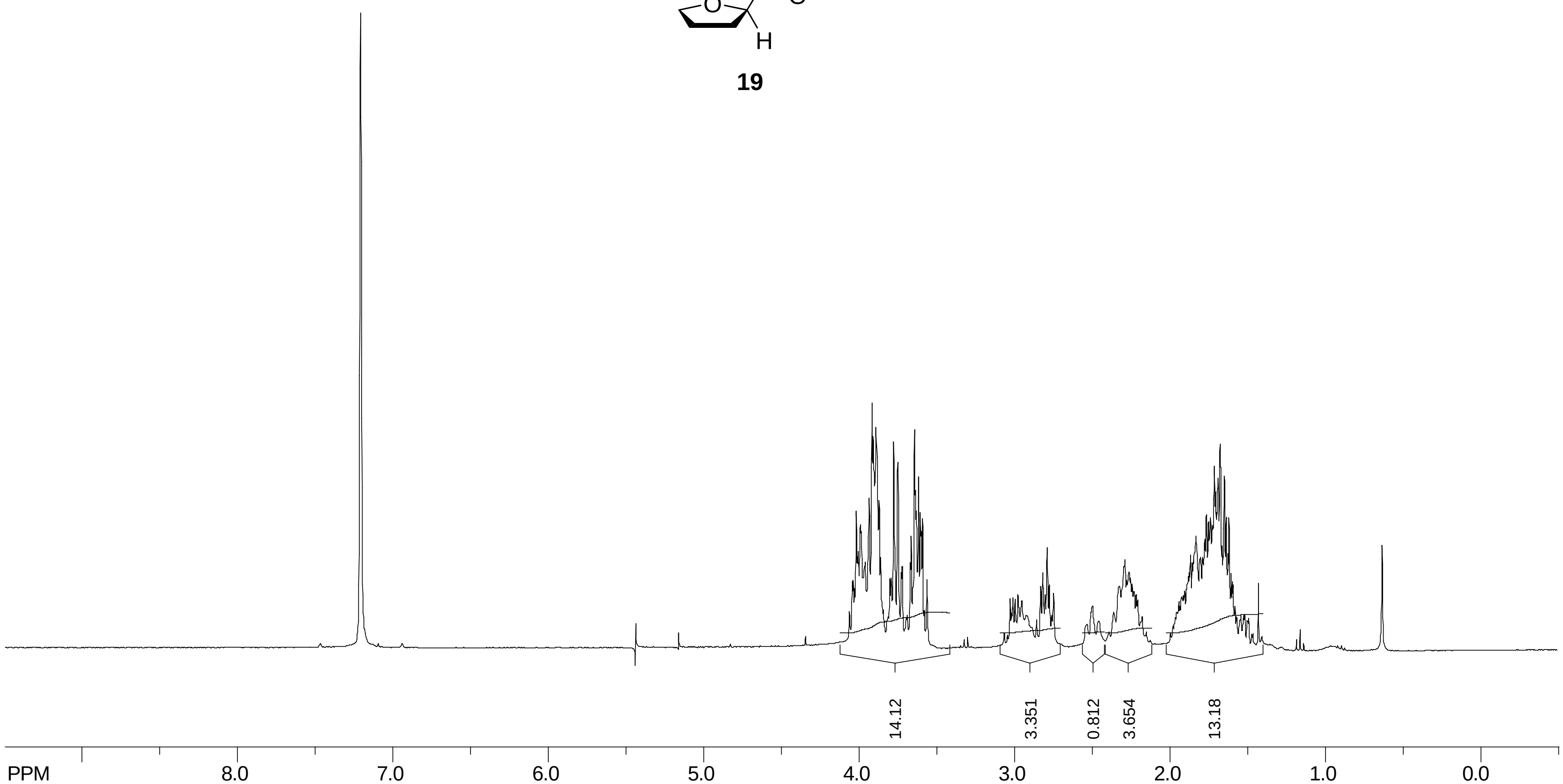
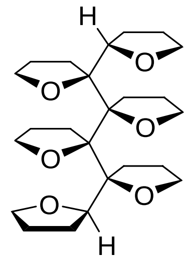
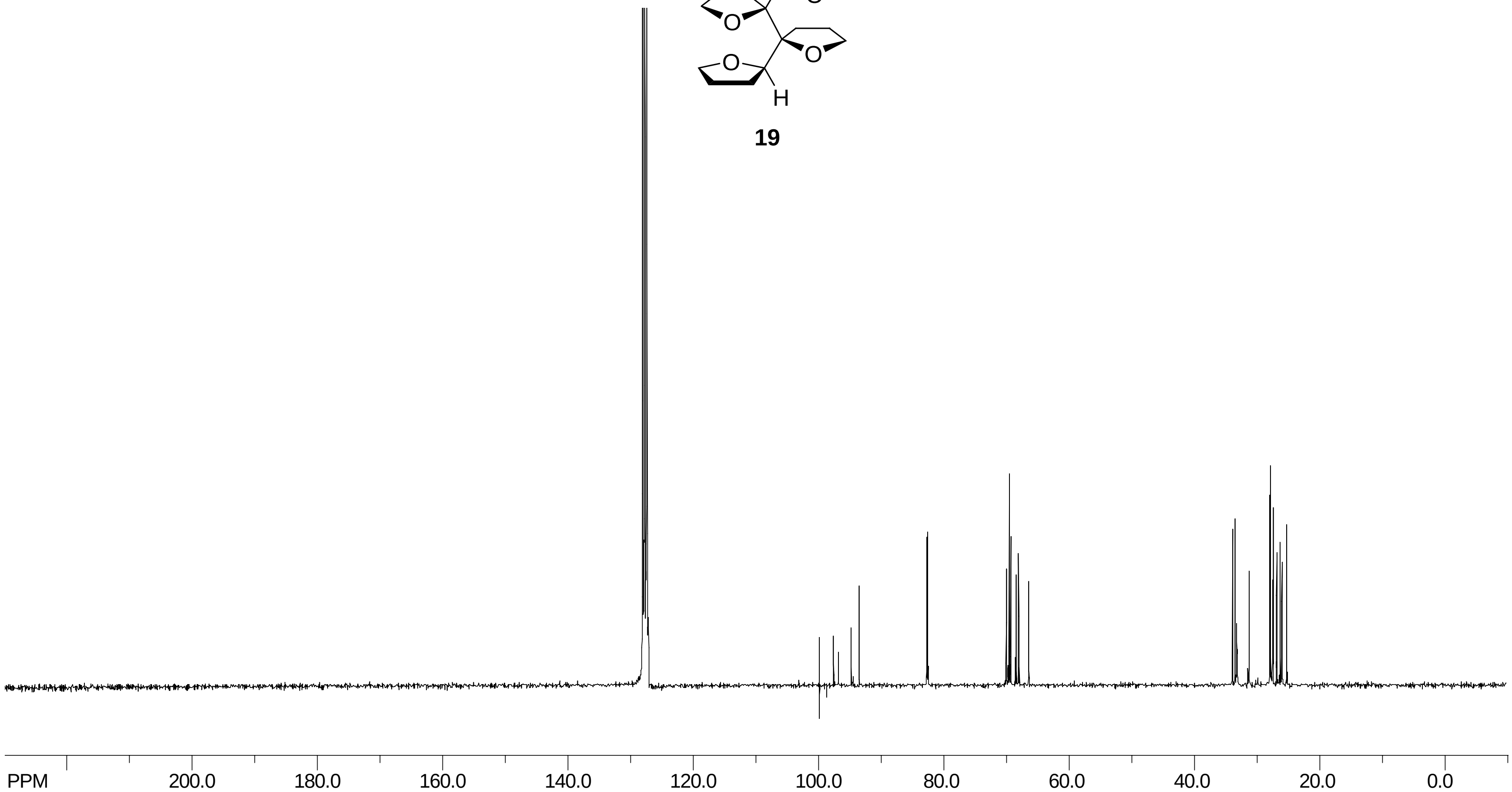


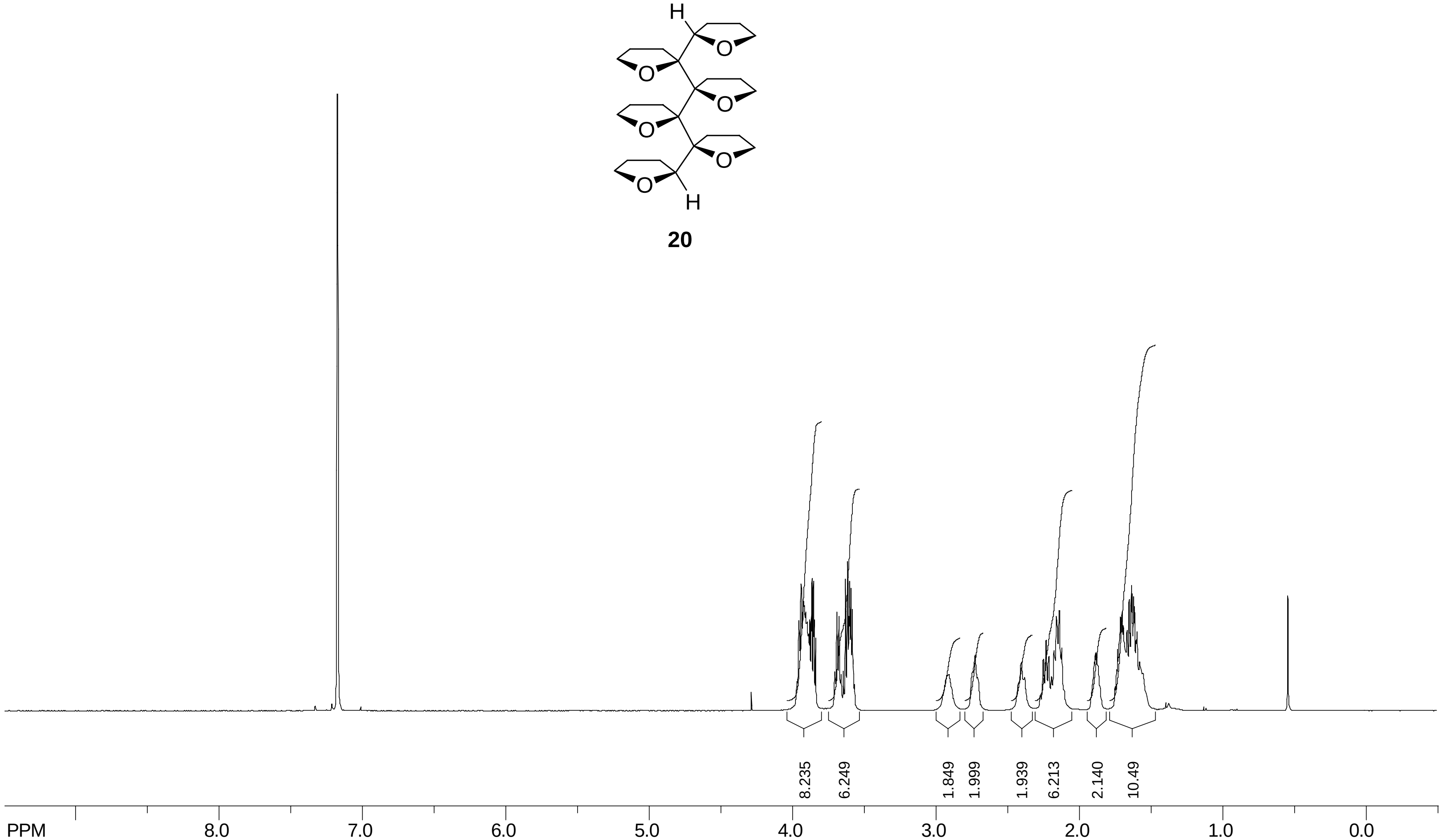
^{1}H NMR : 300 MHz, C_6D_6



19

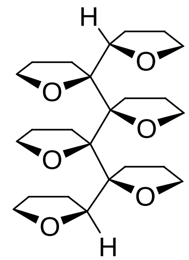


^{13}C NMR : 75 MHz, C_6D_6



20

^{1}H NMR : 500 MHz, C_6D_6



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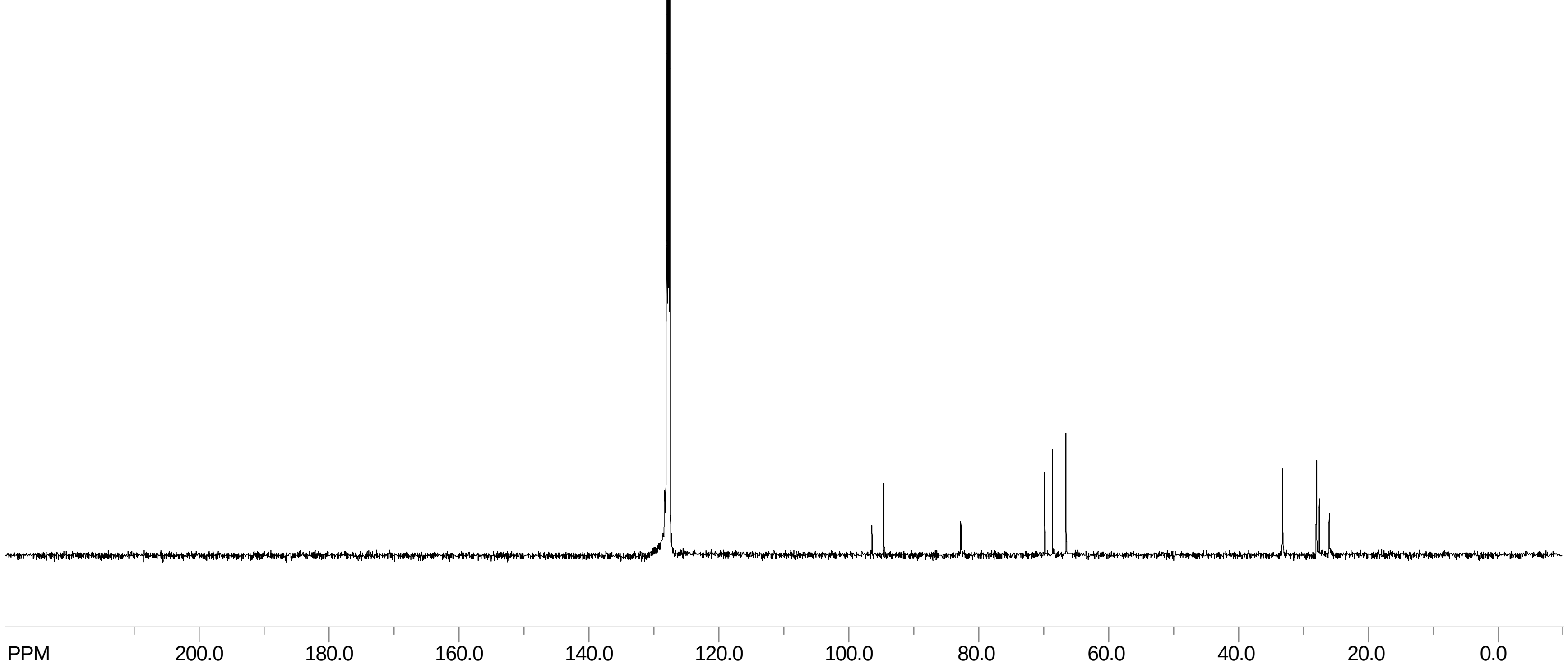
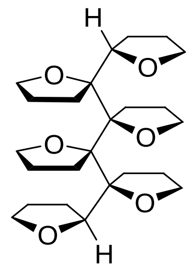


^{13} C NMR : 125 MHz, C_6D_6



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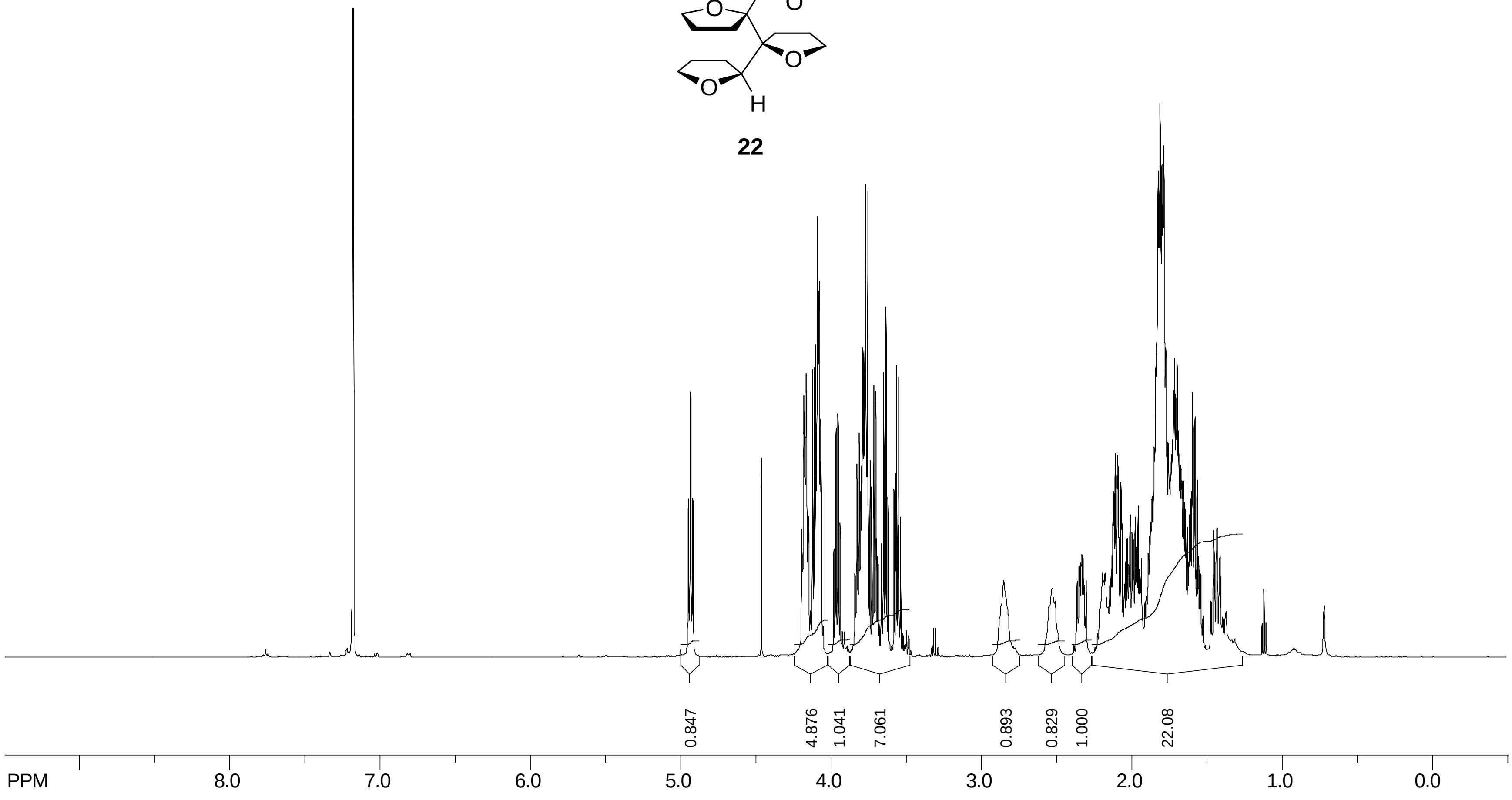
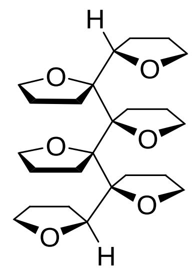
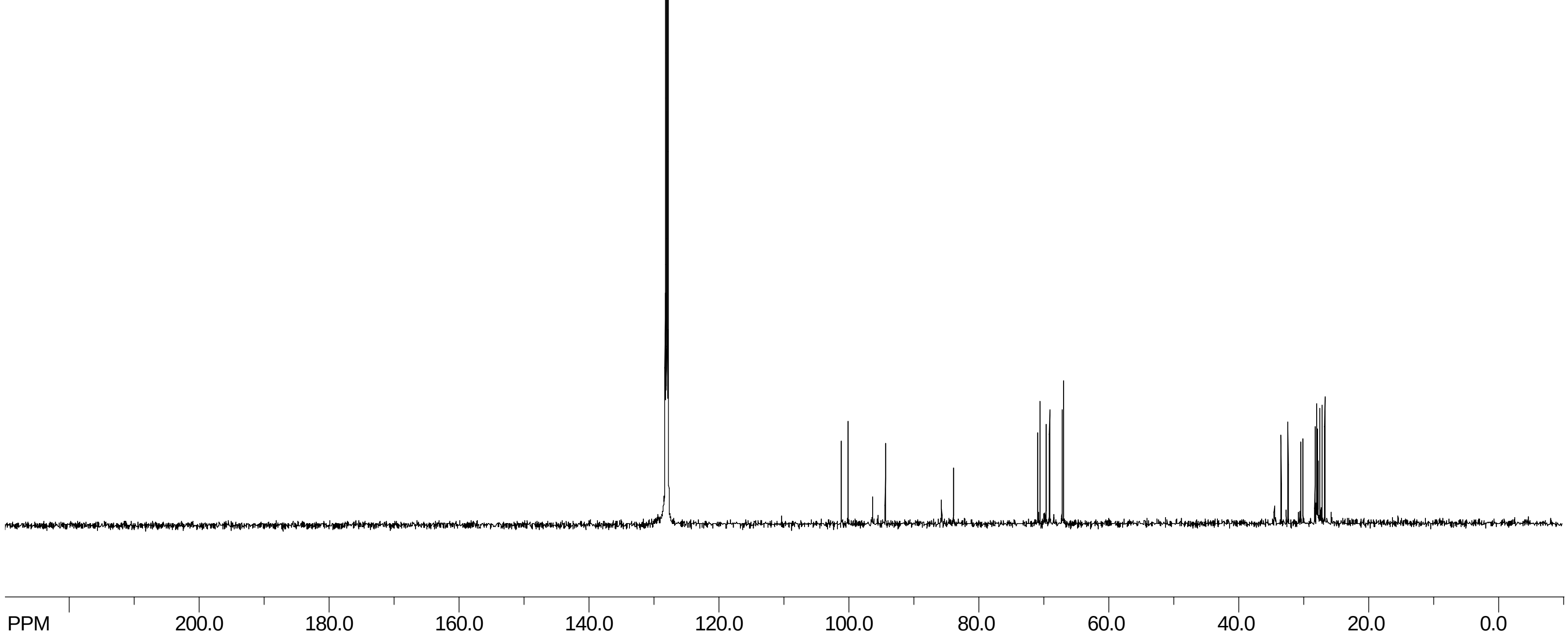


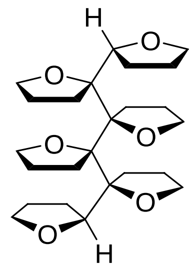
^{1}H NMR : 500 MHz, C_6D_6, 60 ^{o}C



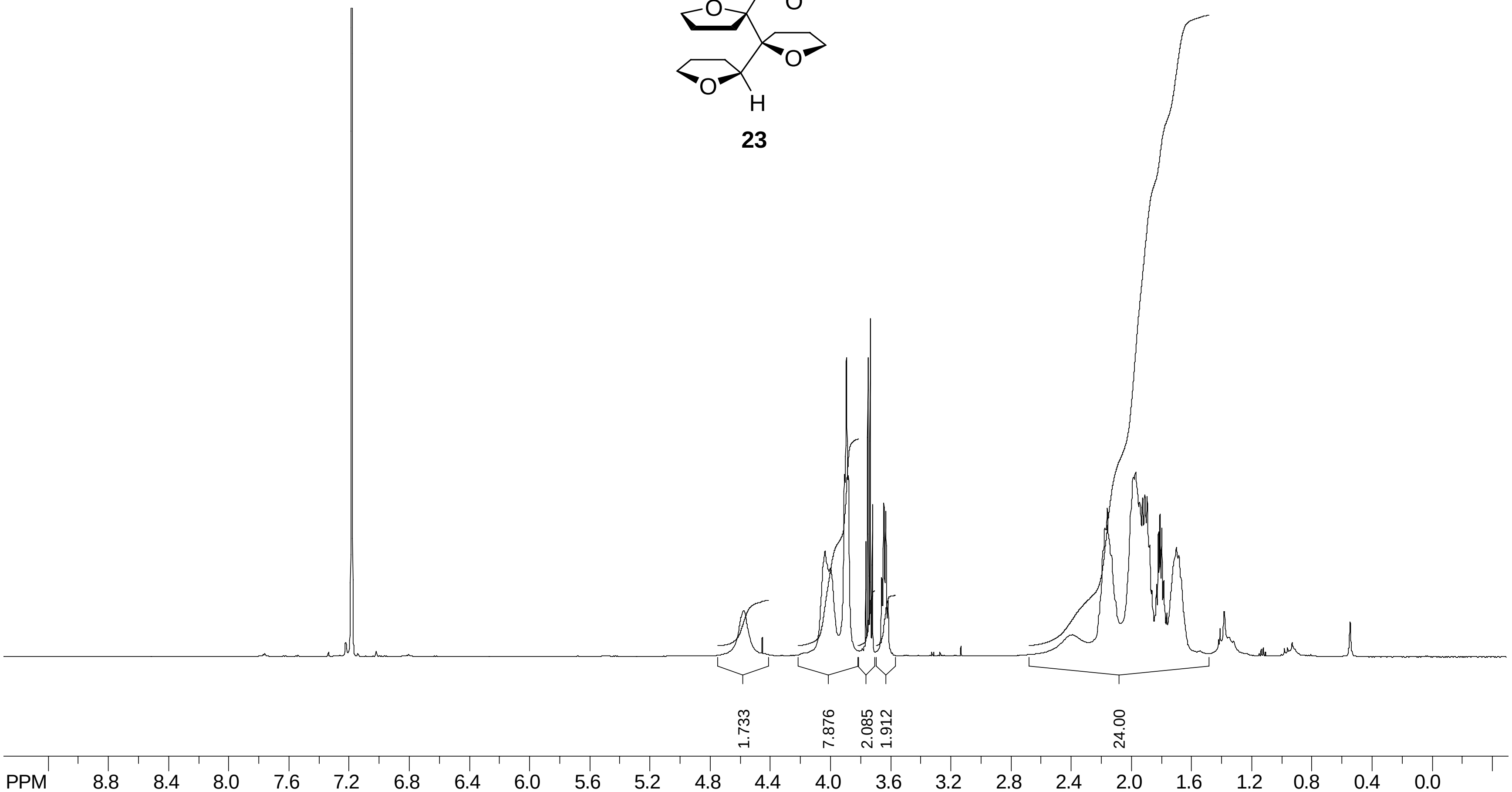
22



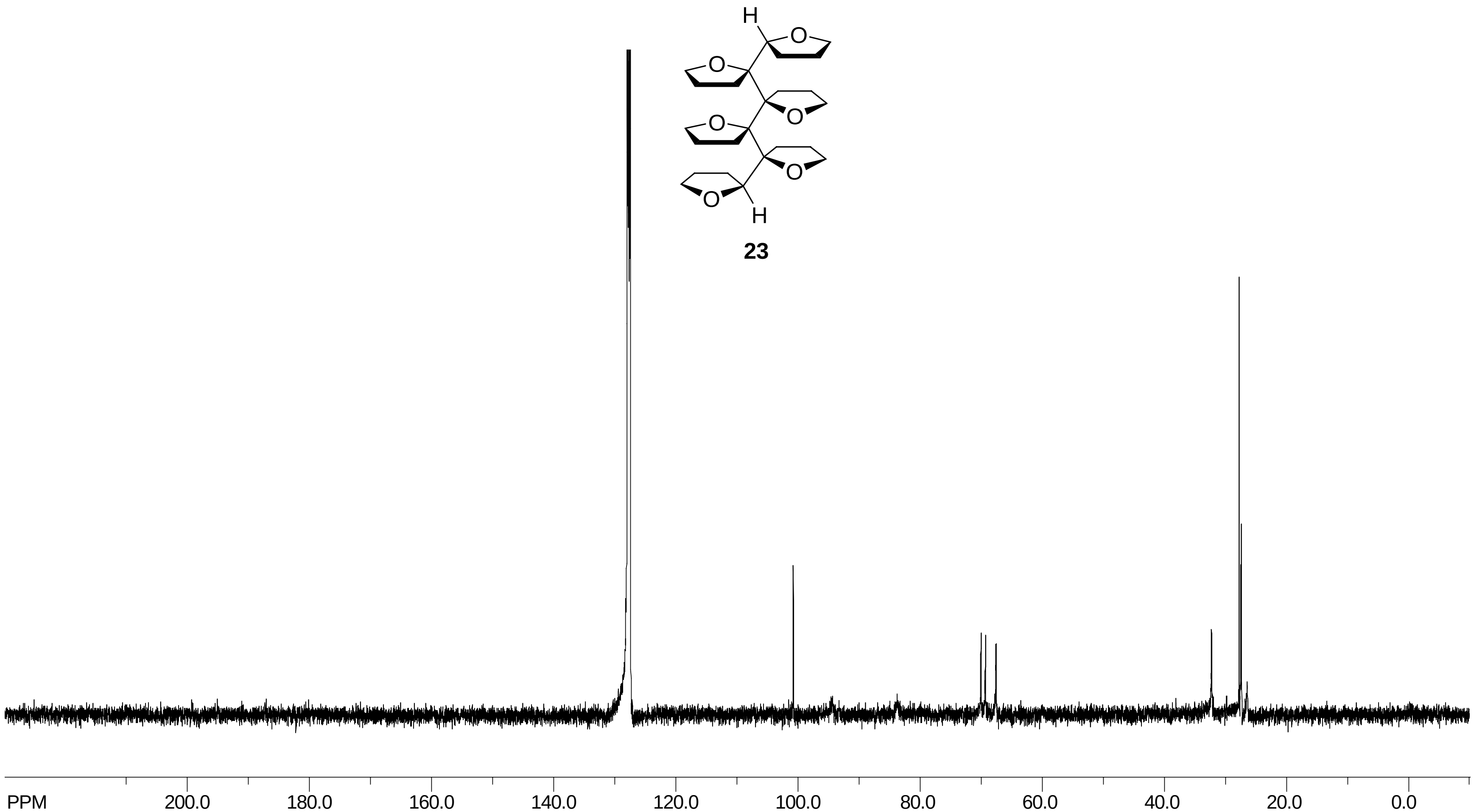
^{13}C NMR : 125 MHz, C_6D_6, 60 ^{o}C



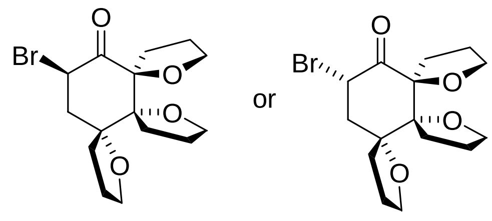
23



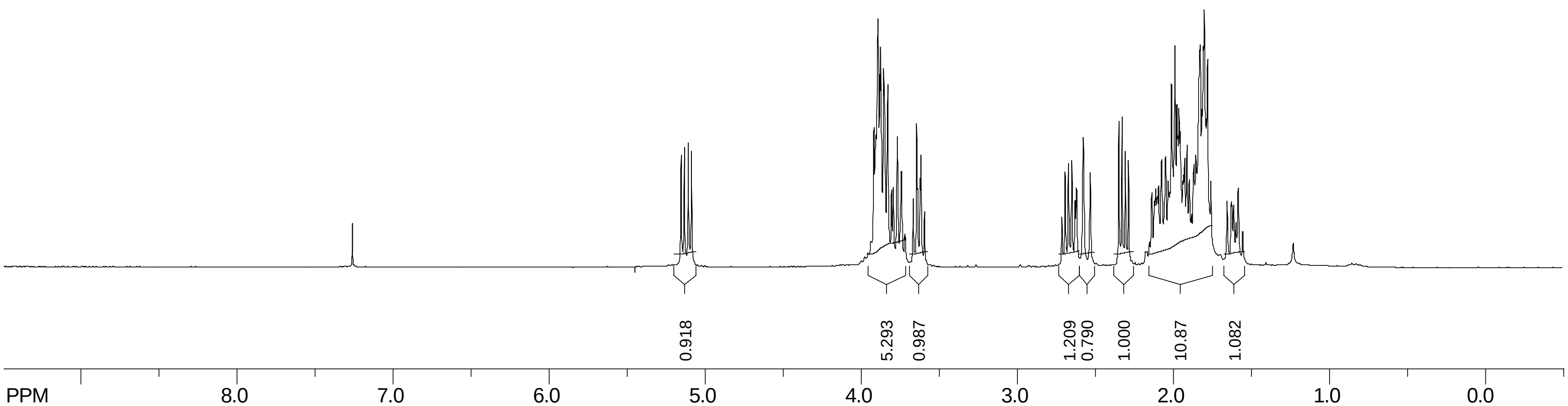
^{1}H NMR : 500 MHz, C_6D_6, 70 ^oC



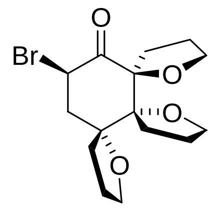
^{13}C NMR : 125 MHz, C_6D_6, 70 ^{o}C



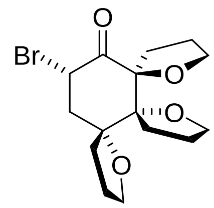
25 or **26**



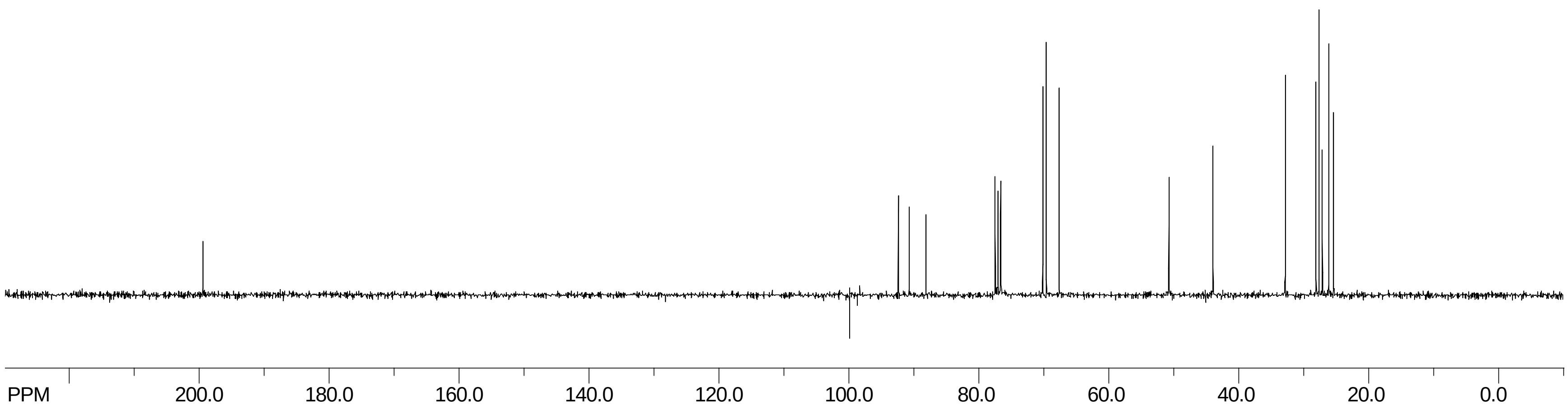
^{1}H NMR : 300 MHz, $CDCl_3$



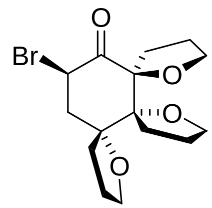
or



25 **26**



^{13}C NMR : 75 MHz, $CDCl_3$



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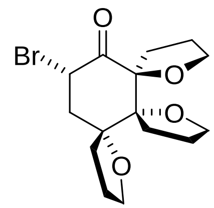
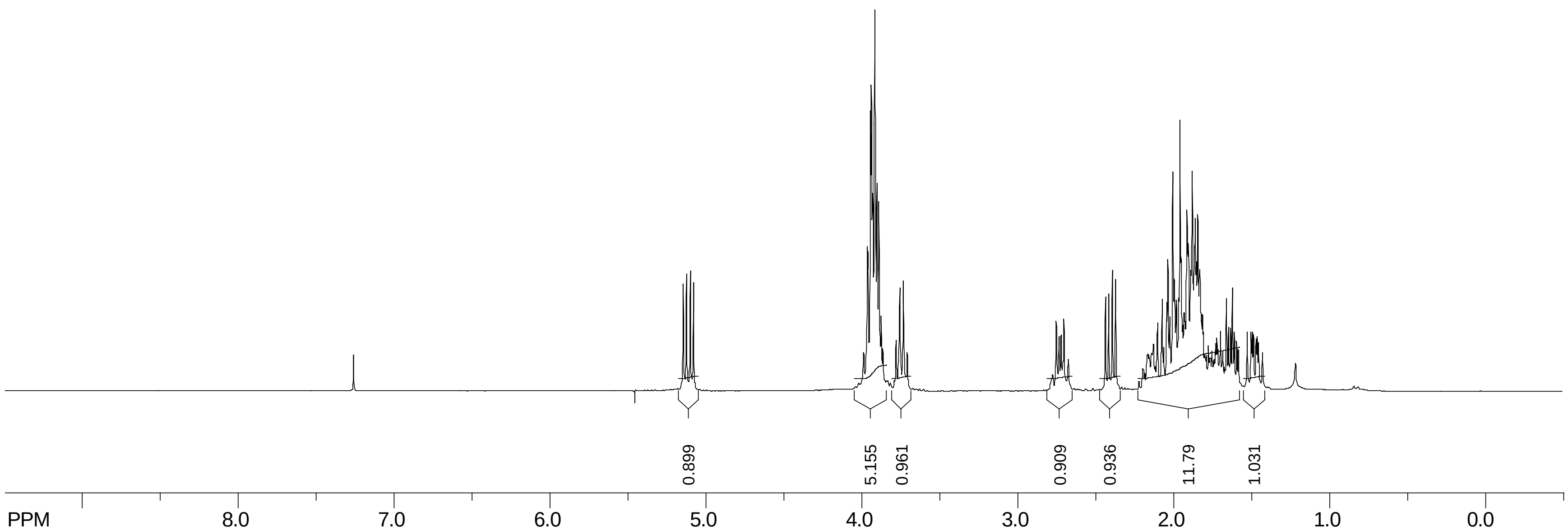
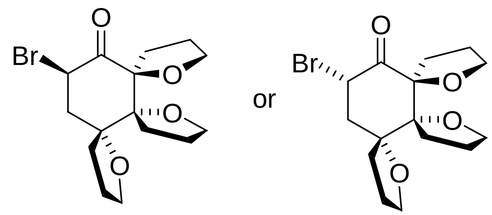


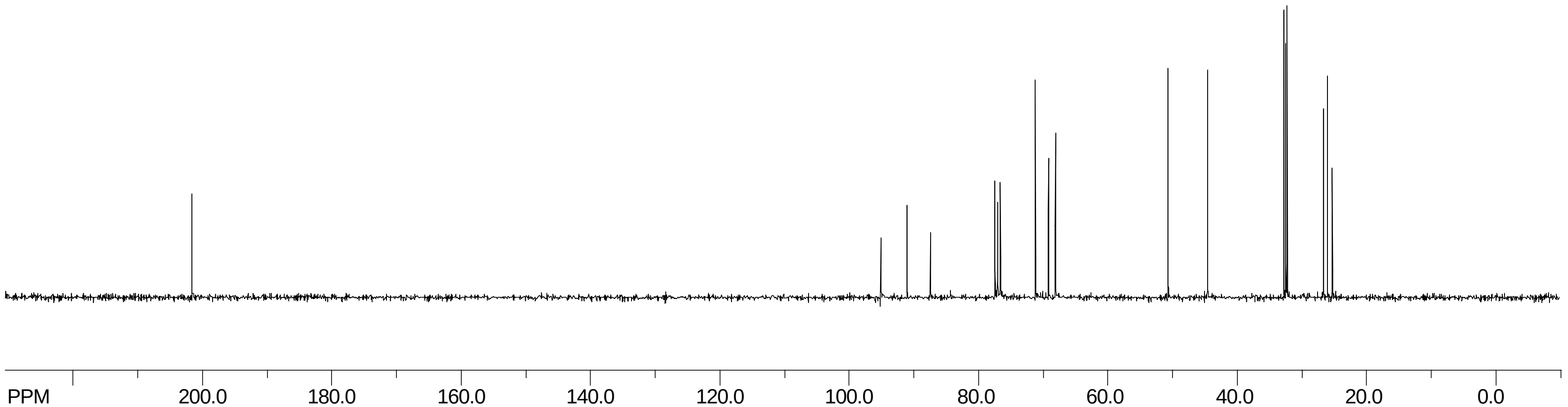
25 **26**



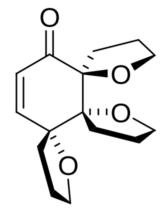
^{1}H NMR : 300 MHz, $CDCl_3$



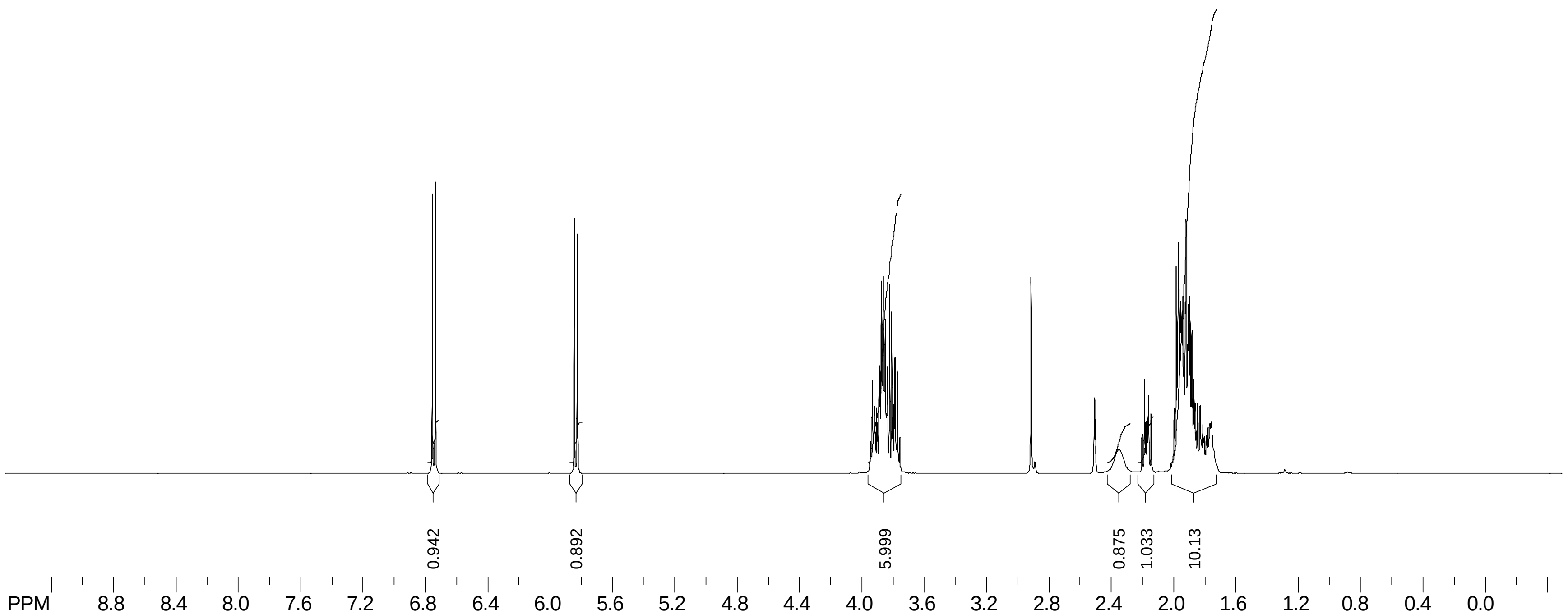
25 **26**



^{13}C NMR : 75 MHz, $CDCl_3$

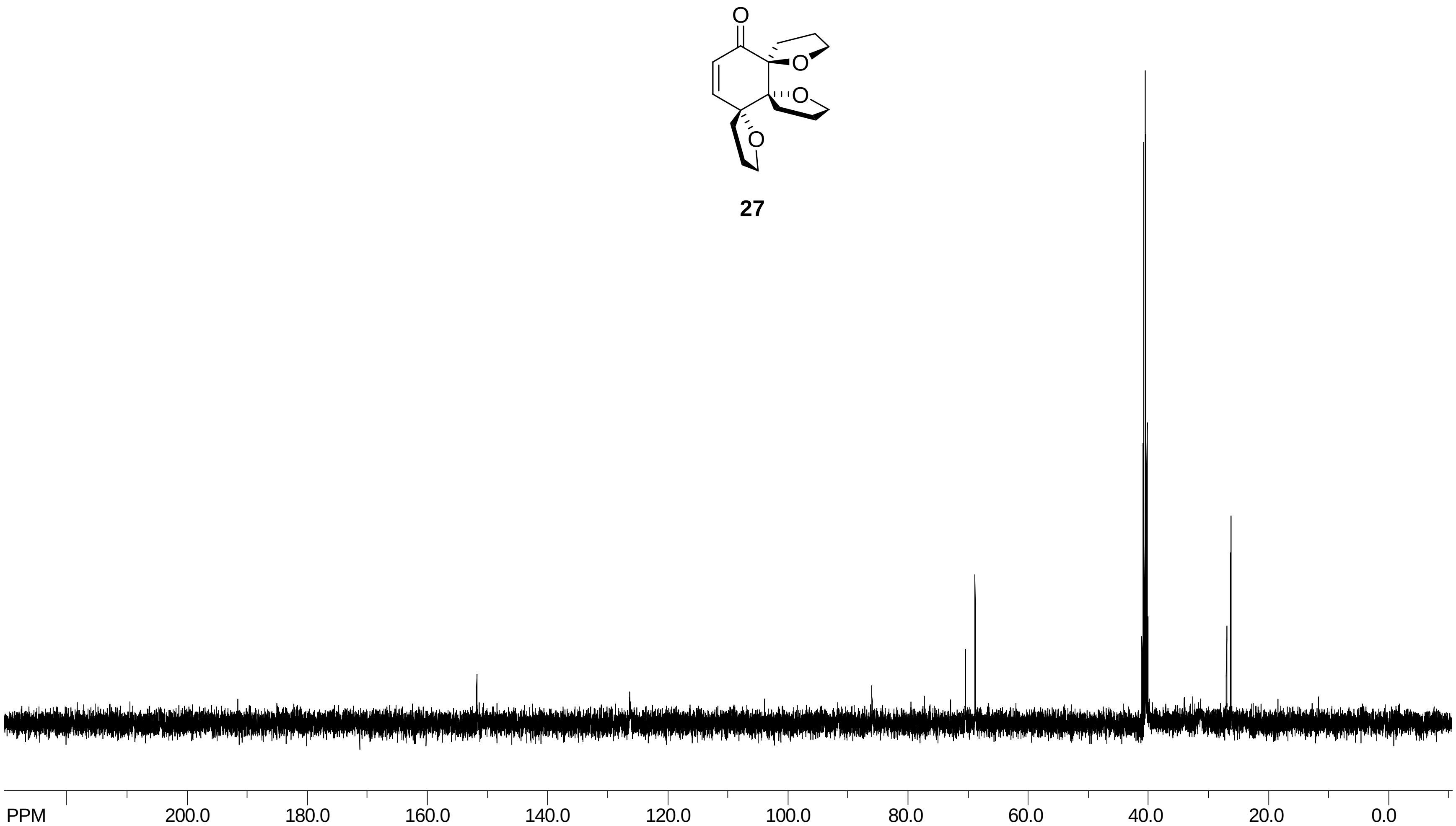


27

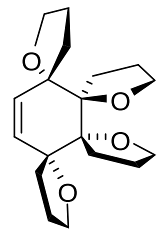


^{1}H NMR : 500 MHz, DMSO, 90 ^{o}C

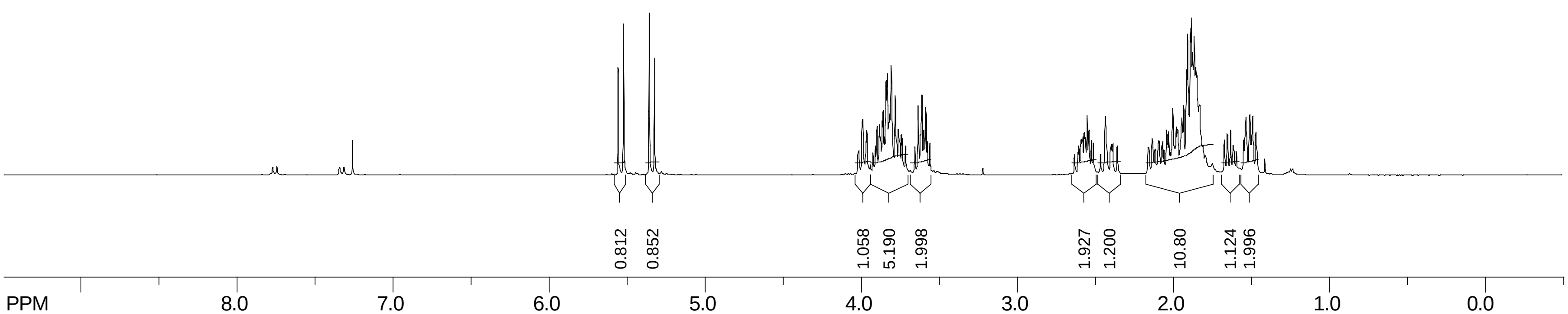
27



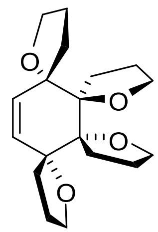
^{13}C NMR : 125 MHz, DMSO, 90 ^{o}C



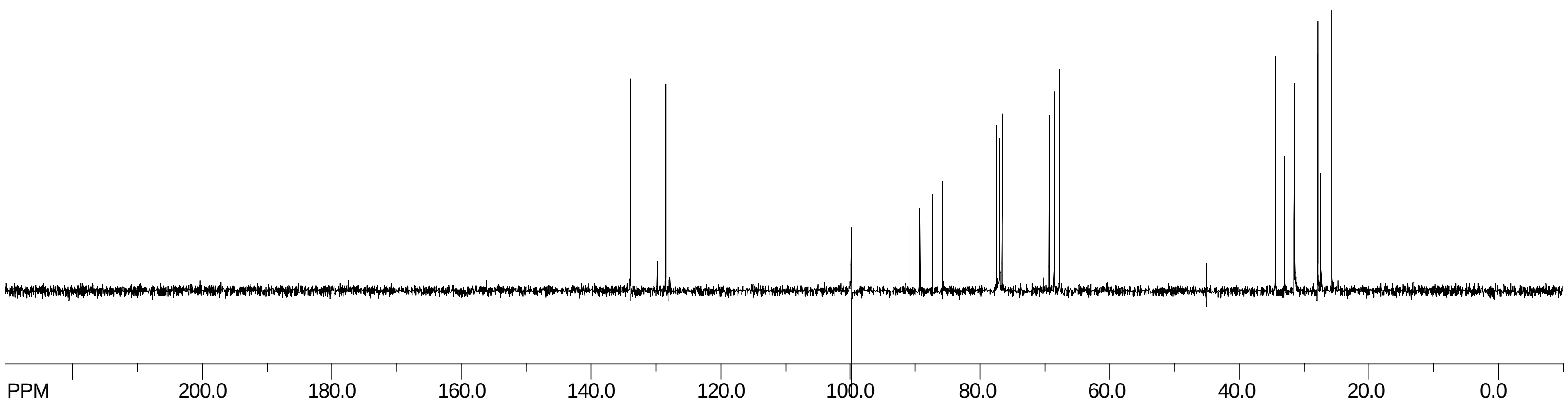
28



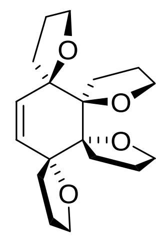
^{1}H NMR : 300 MHz, $CDCl_3$



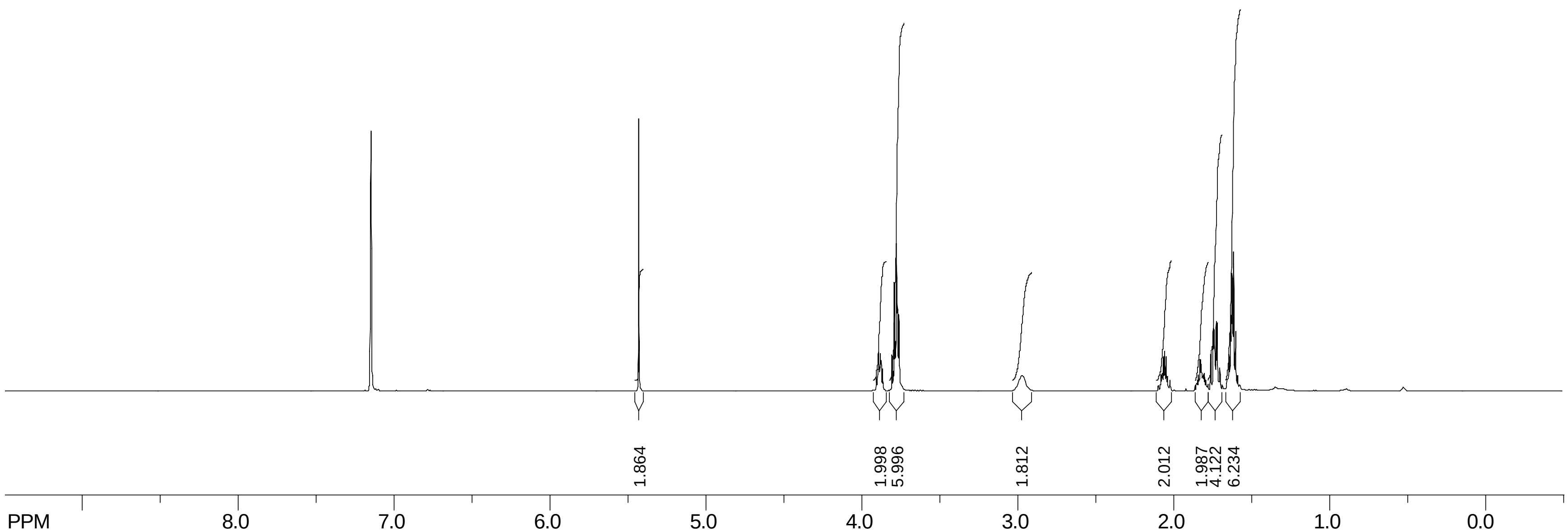
28



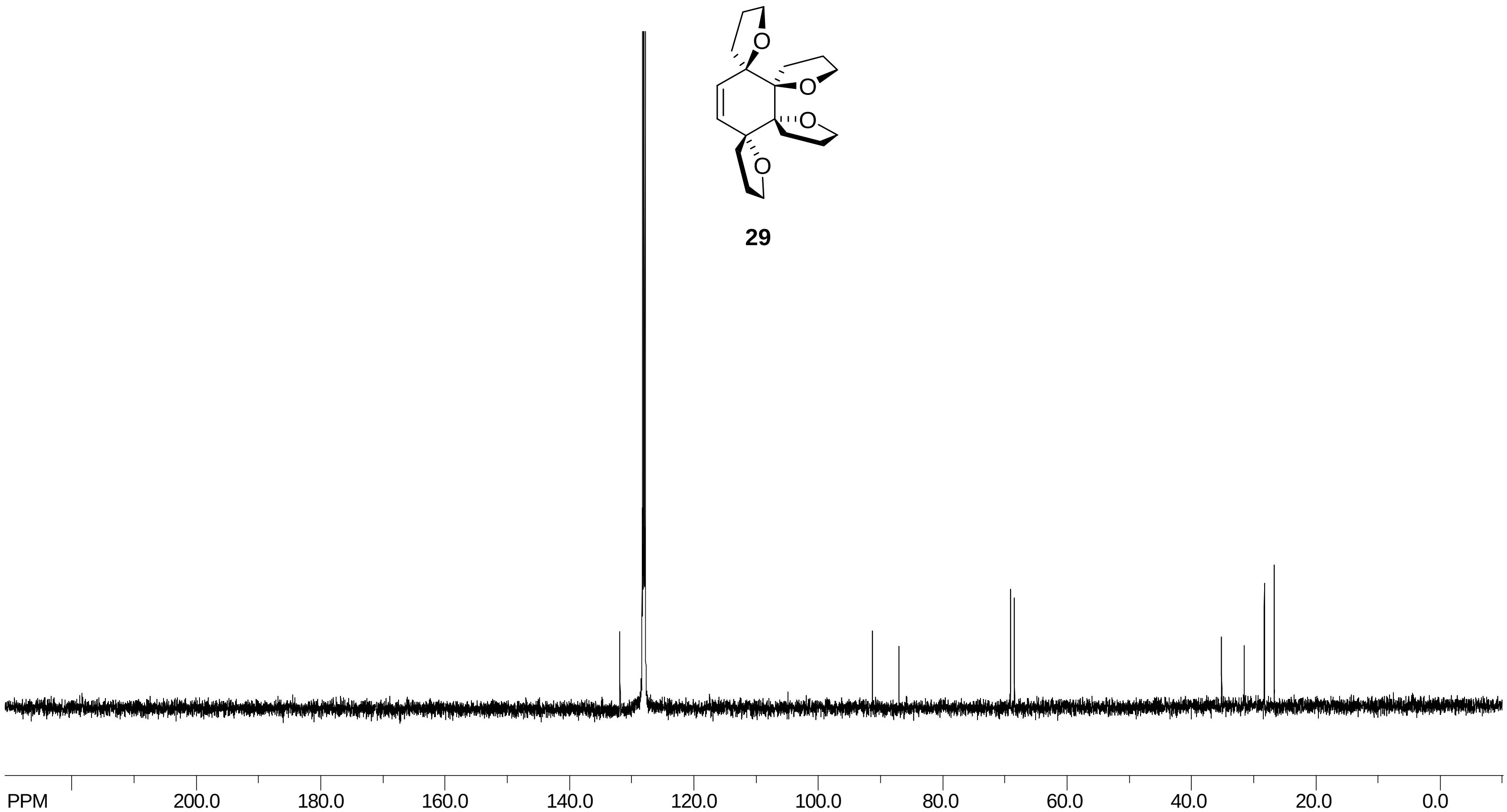
^{13}C NMR : 75 MHz, $CDCl_3$



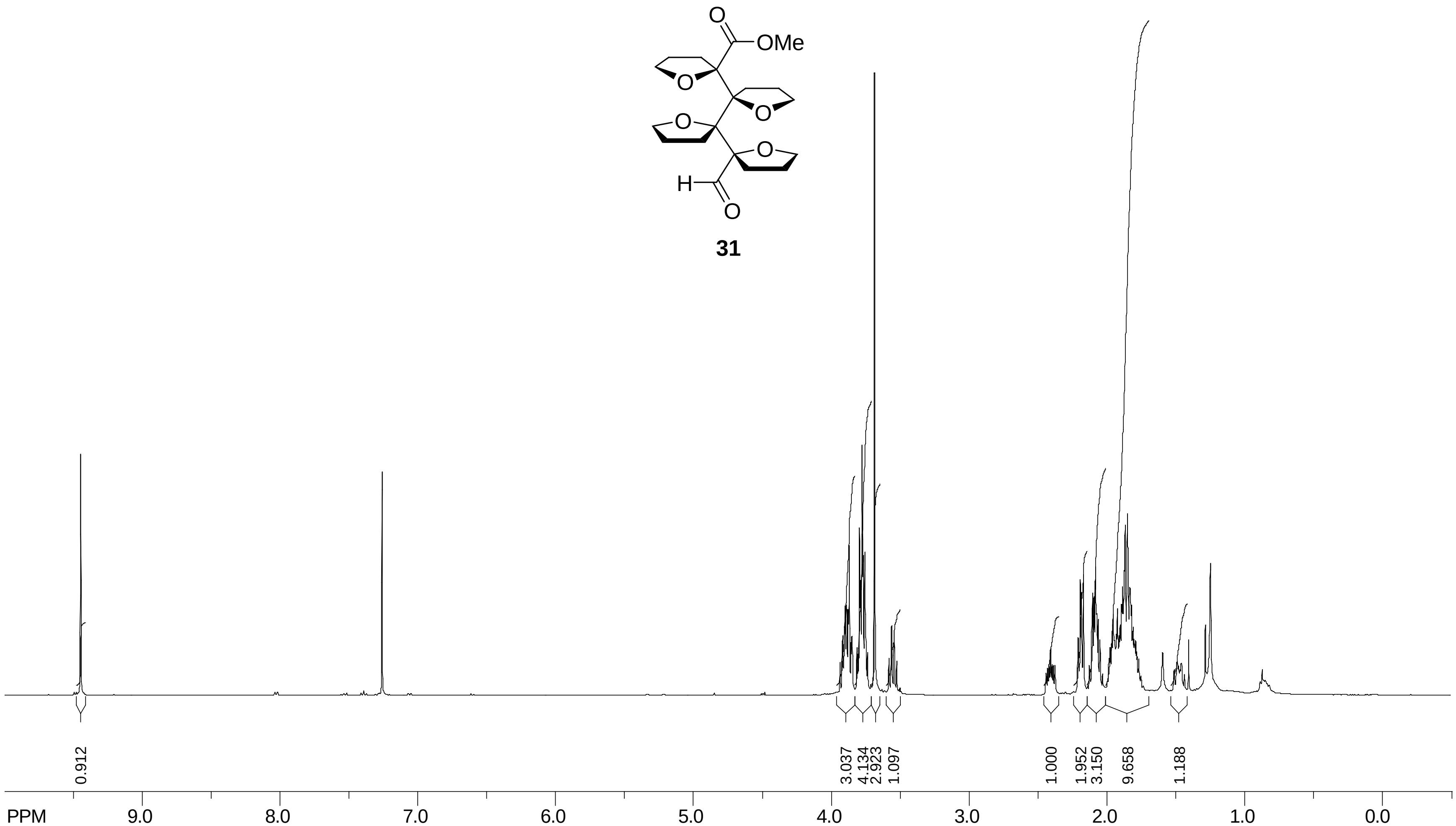
29



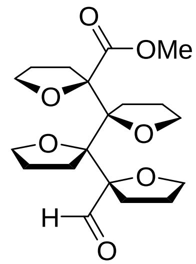
^{1}H NMR : 500 MHz, C_6D_6



^{13}C NMR : 125 MHz, C_6D_6



^{1}H NMR : 500 MHz, $CDCl_3$



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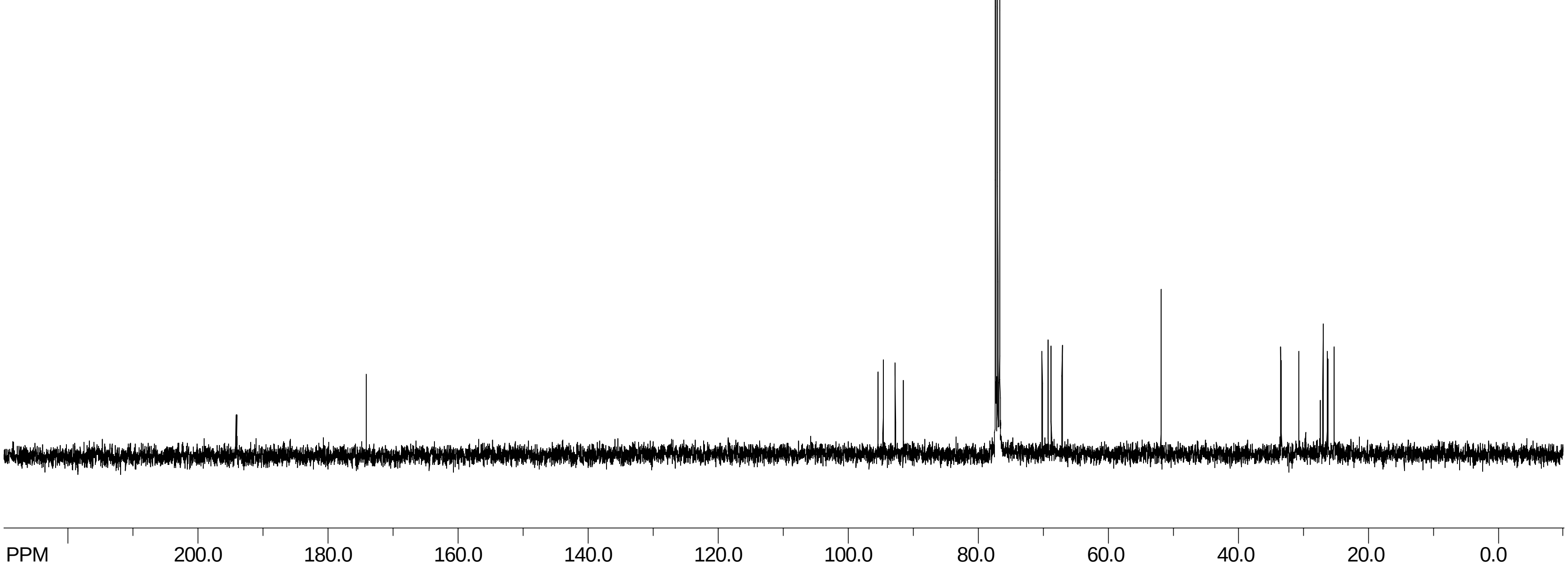
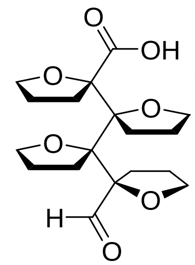
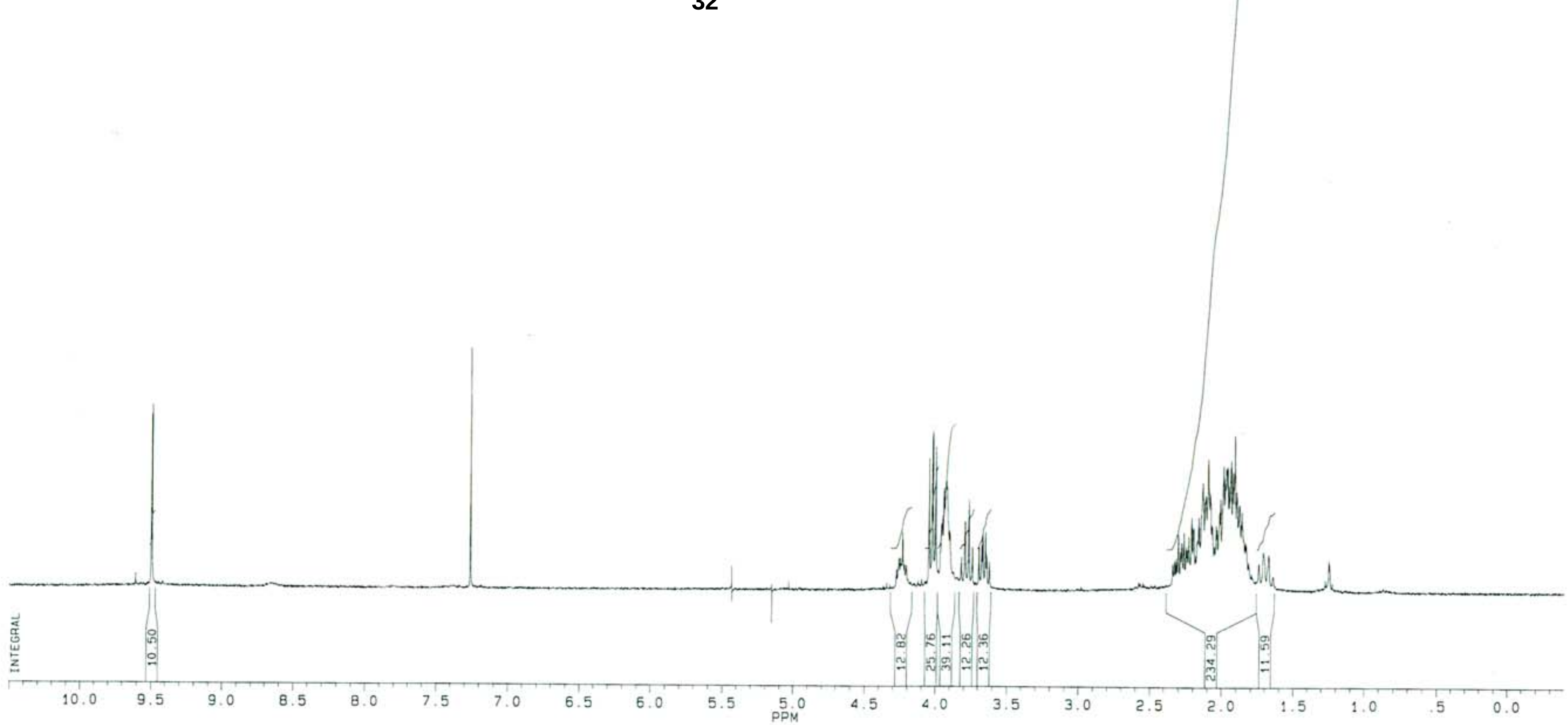


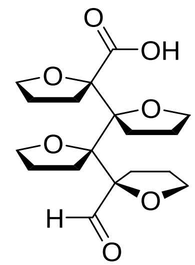
^{13}C NMR : 125 MHz, $CDCl_3$



32



^{1}H NMR : 300 MHz, $CDCl_3$



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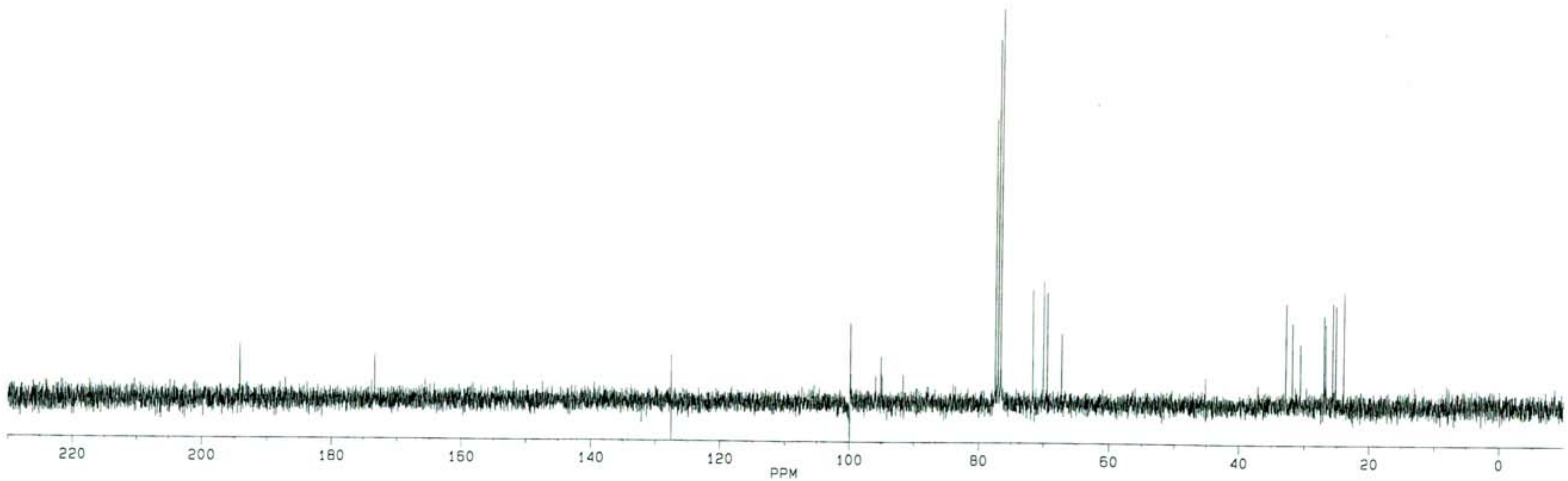
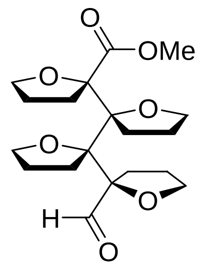


^{13}C NMR : 75 MHz, $CDCl_3$



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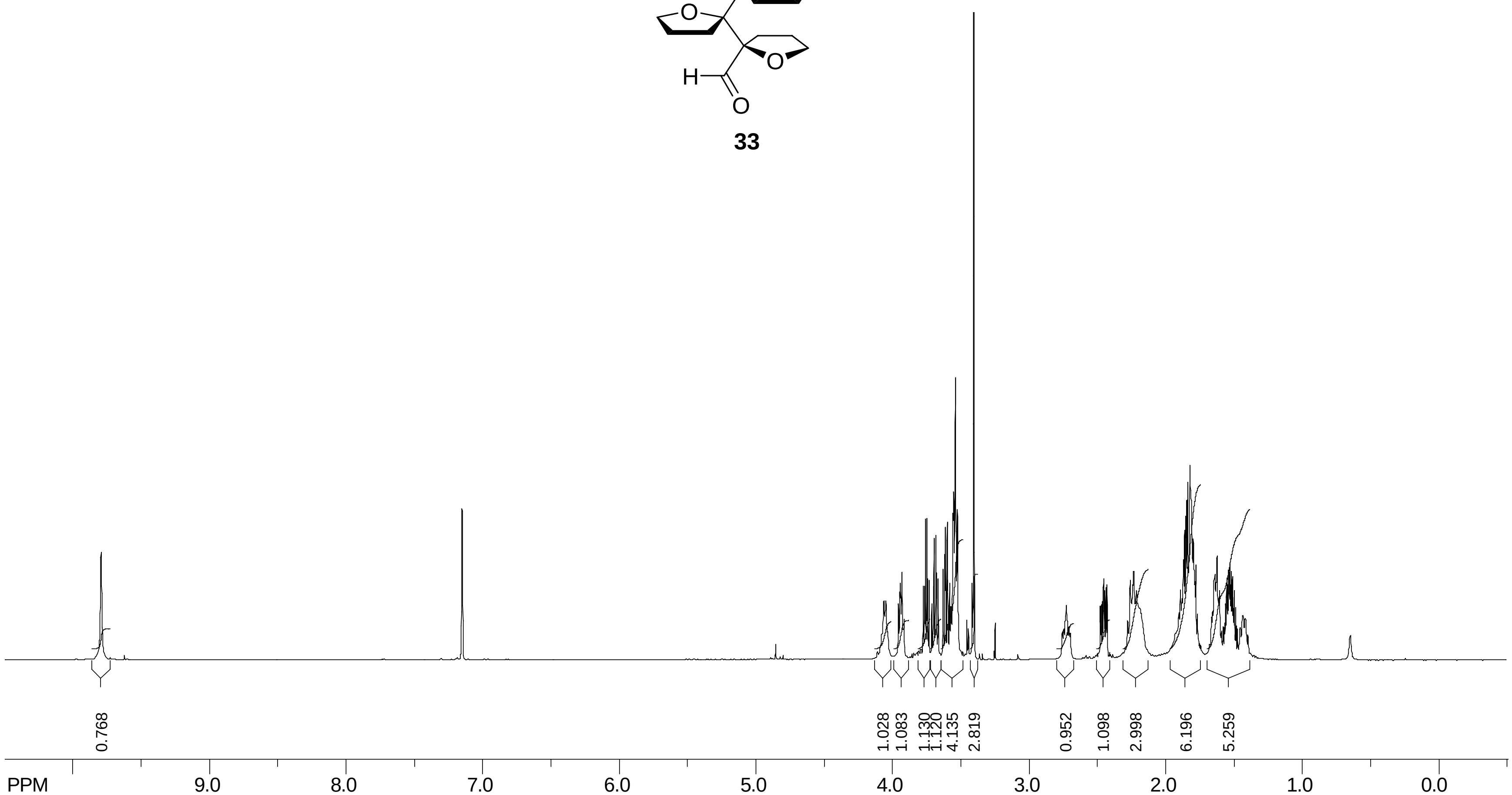
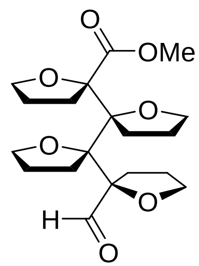


^{1}H NMR : 500 MHz, C_6D_6, 70 ^{o}C



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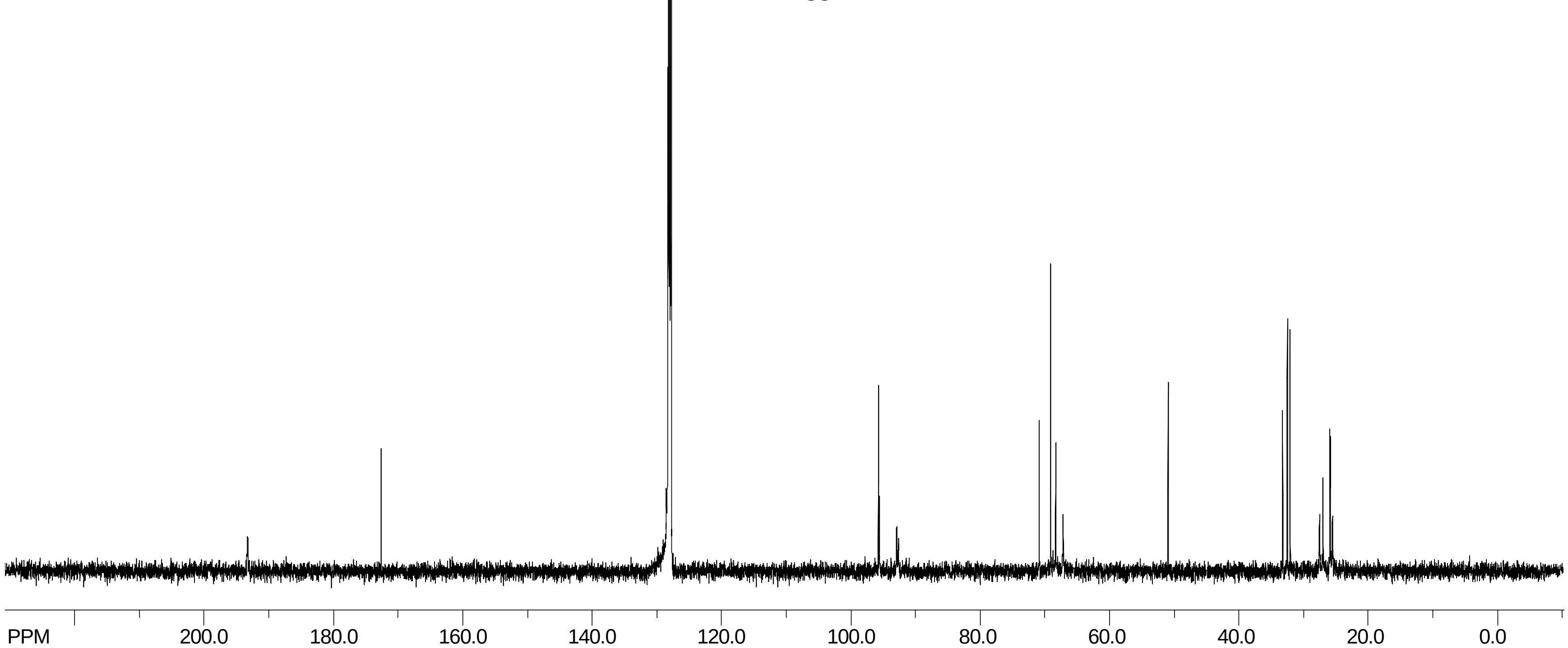
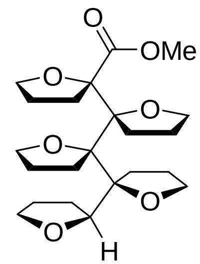


^{13}C NMR : 125 MHz, C_6D_6, 70 ^oC



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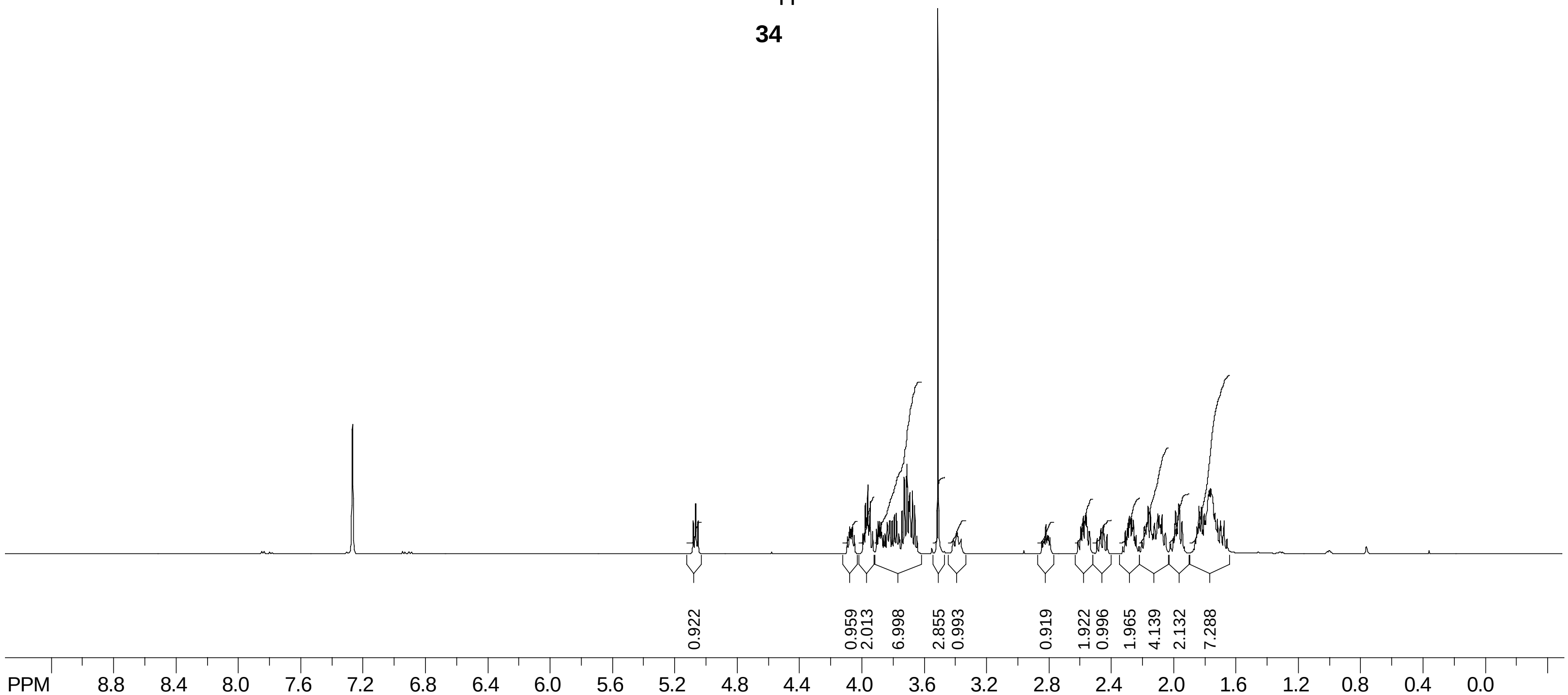
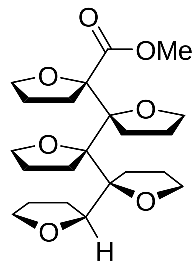


^{1}H NMR : 500 MHz, C_6D_6, 70 ^{o}C



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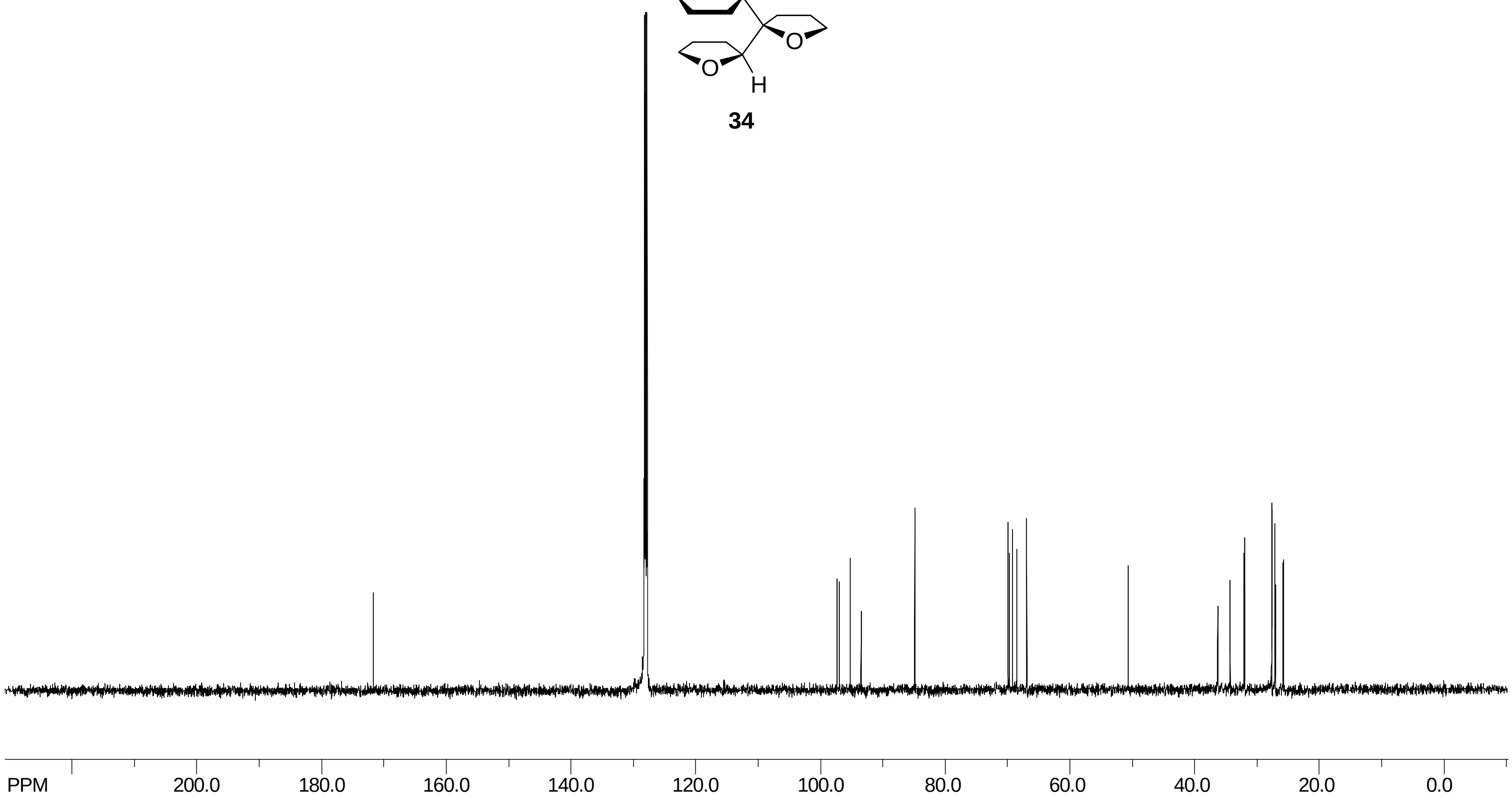
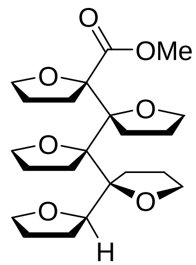


^{13}C NMR : 125 MHz, C_6D_6, 70 ^{o}C



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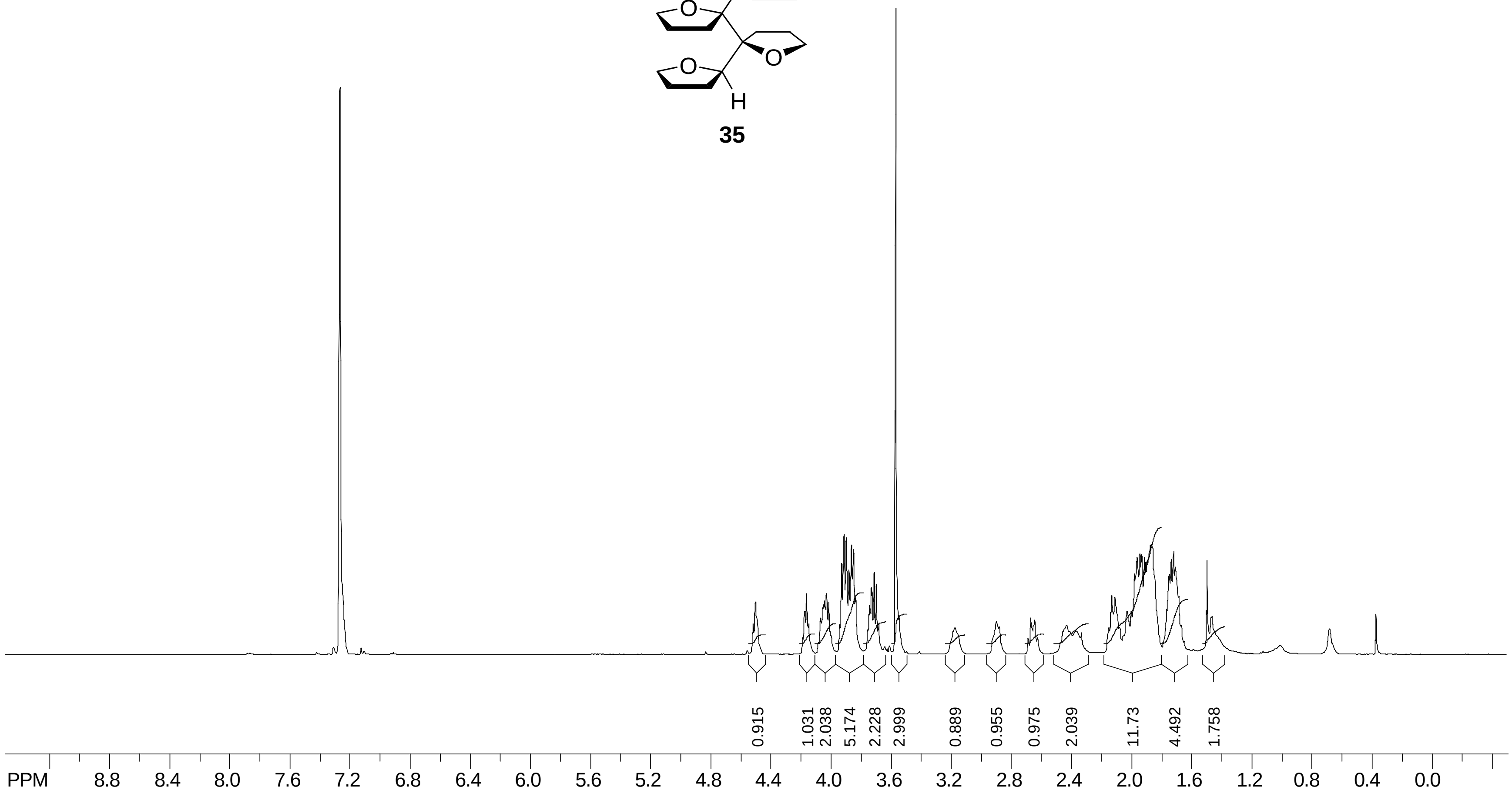
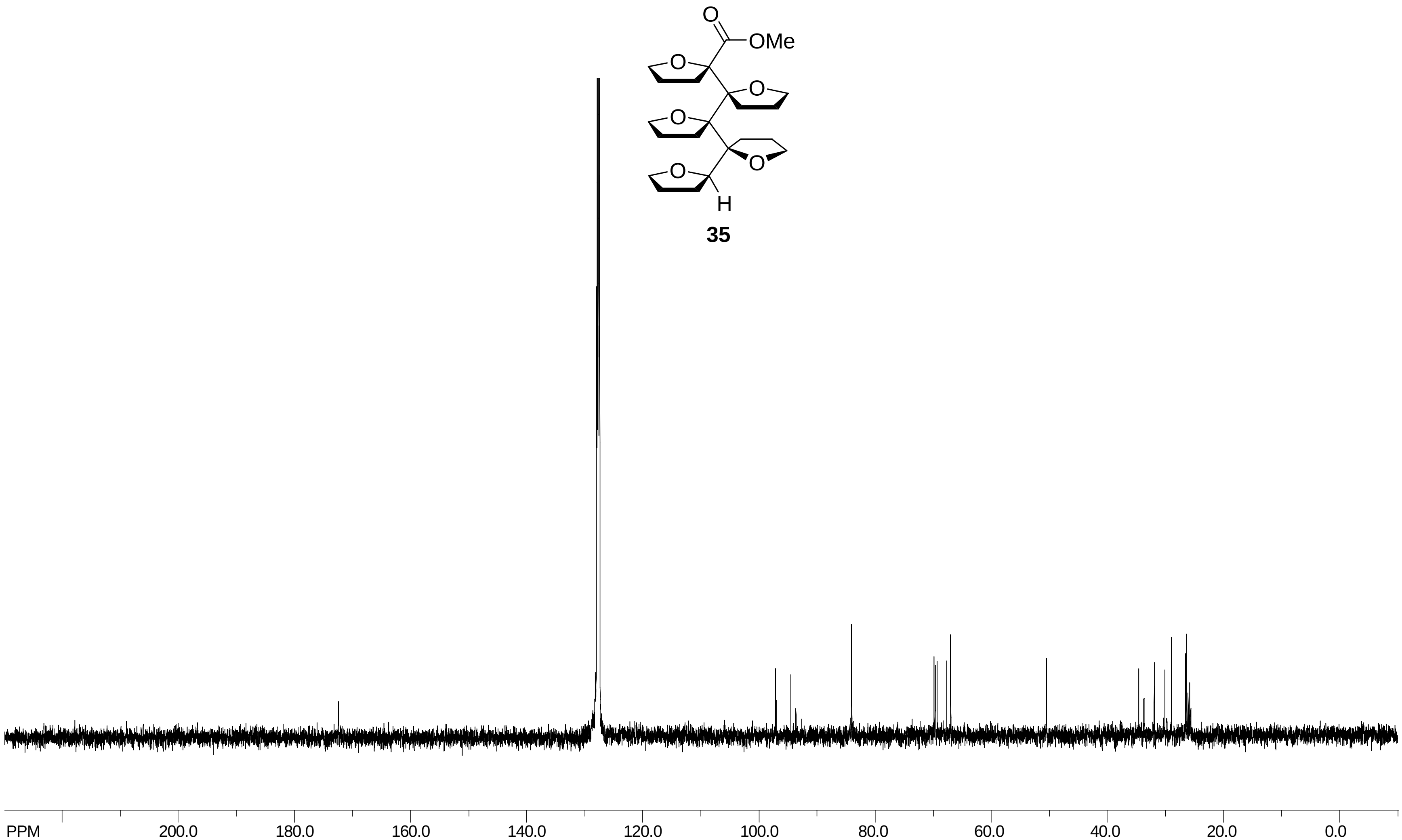
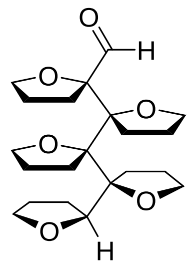


^{1}H NMR : 500 MHz, C_6D_6, 60 ^{o}C



^{13}C NMR : 125 MHz, C_6D_6, 60 ^oC



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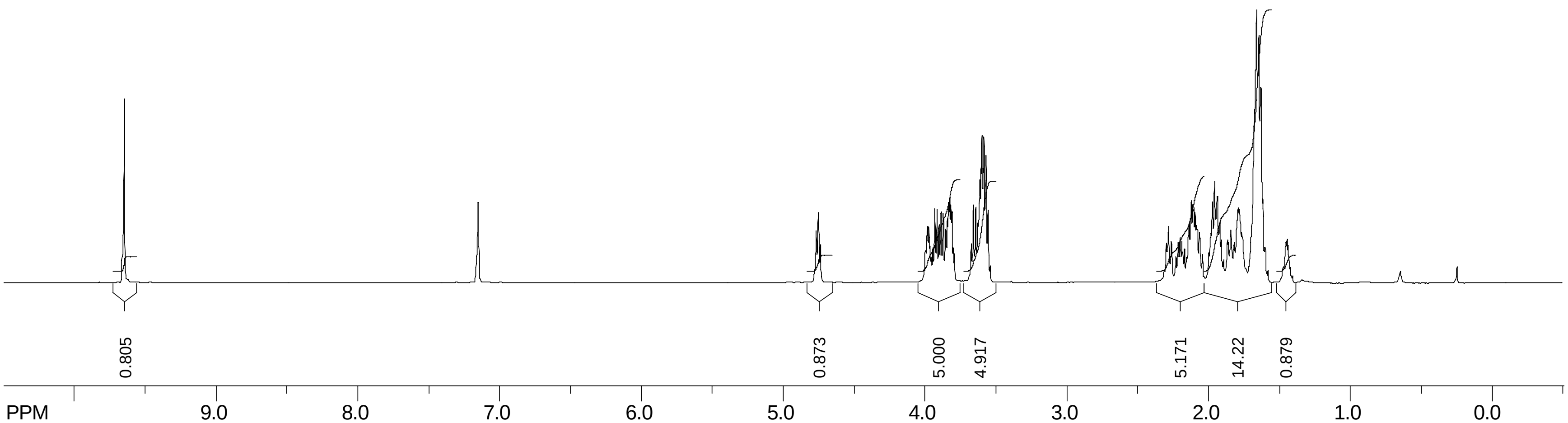
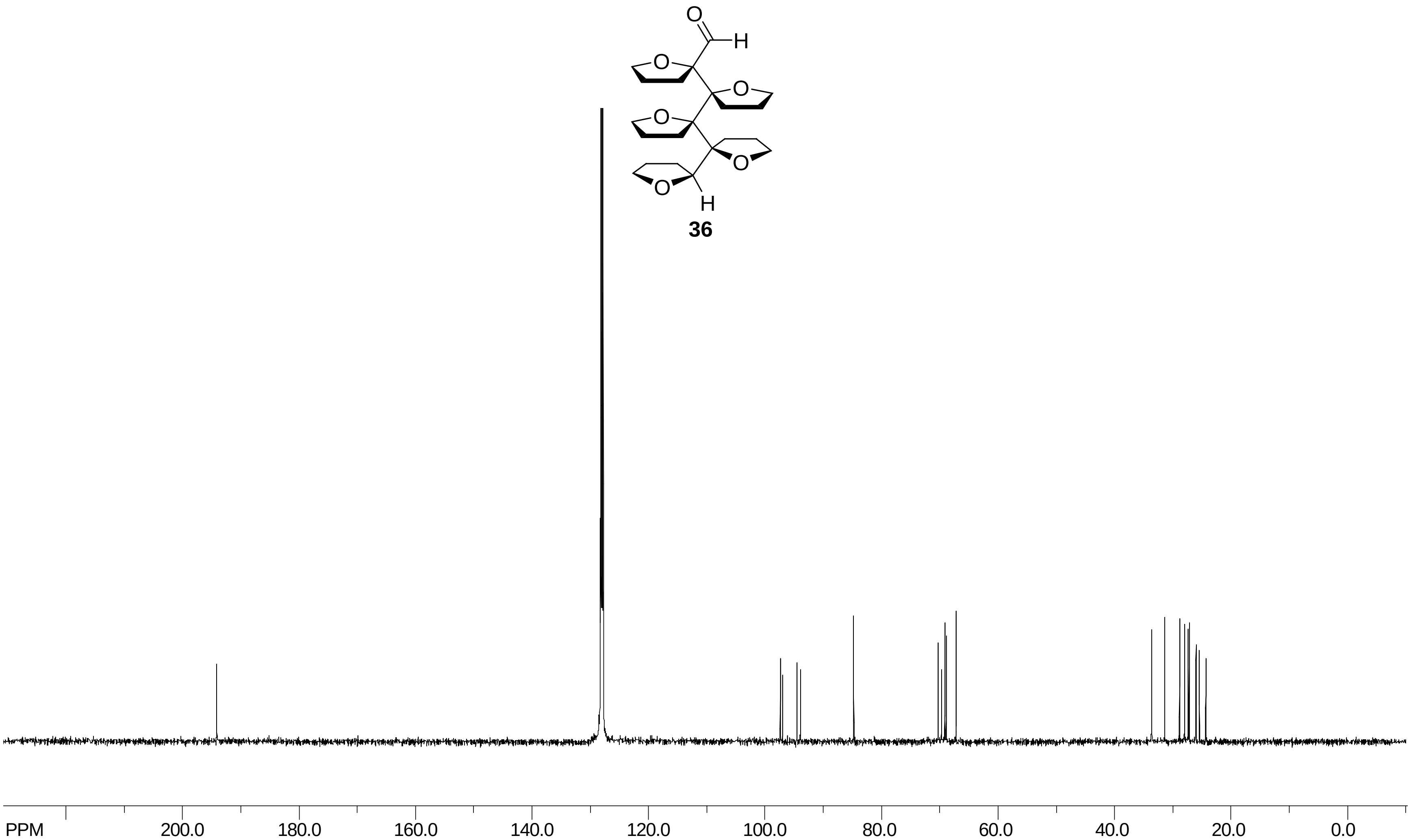
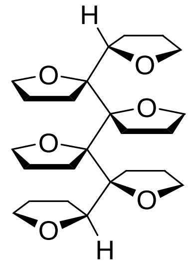


^{1}H NMR : 500 MHz, C_6D_6, 70 ^{o}C



^{13}C NMR : 125 MHz, C_6D_6, 70 ^oC



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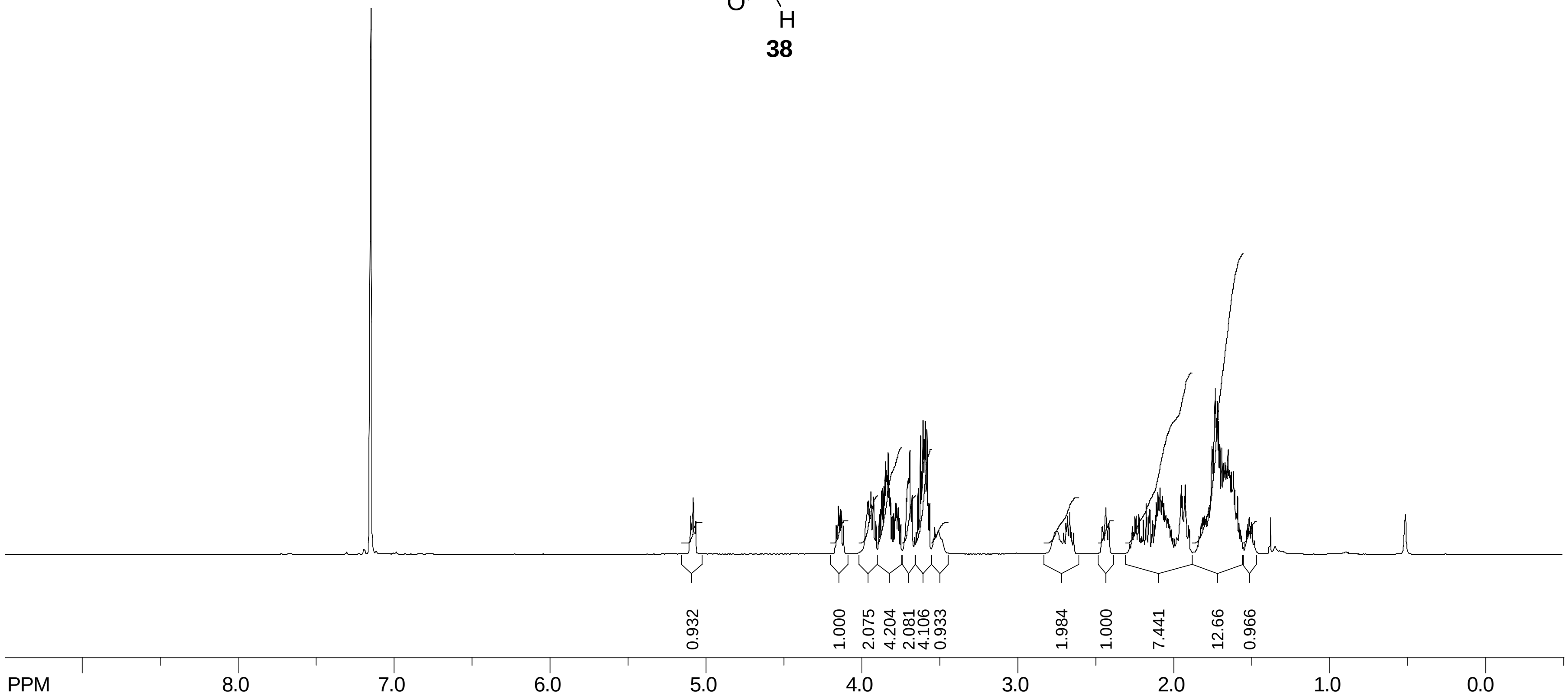
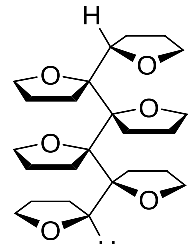


^{1}H NMR : 500 MHz, C_6D_6, 70 ^{o}C



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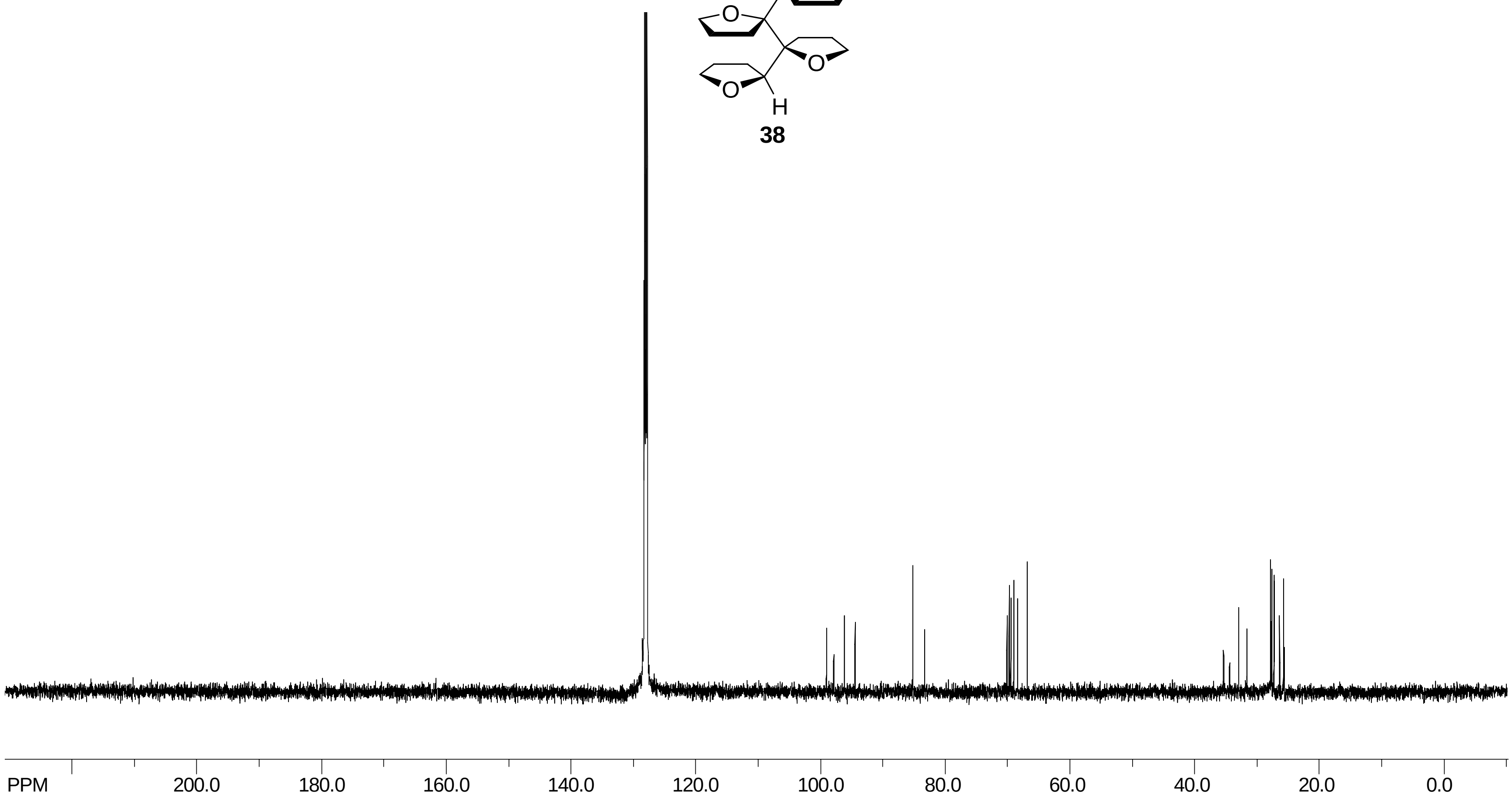
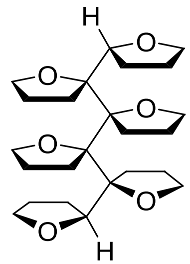


^{13}C NMR : 125 MHz, C_6D_6, 70 ^{o}C



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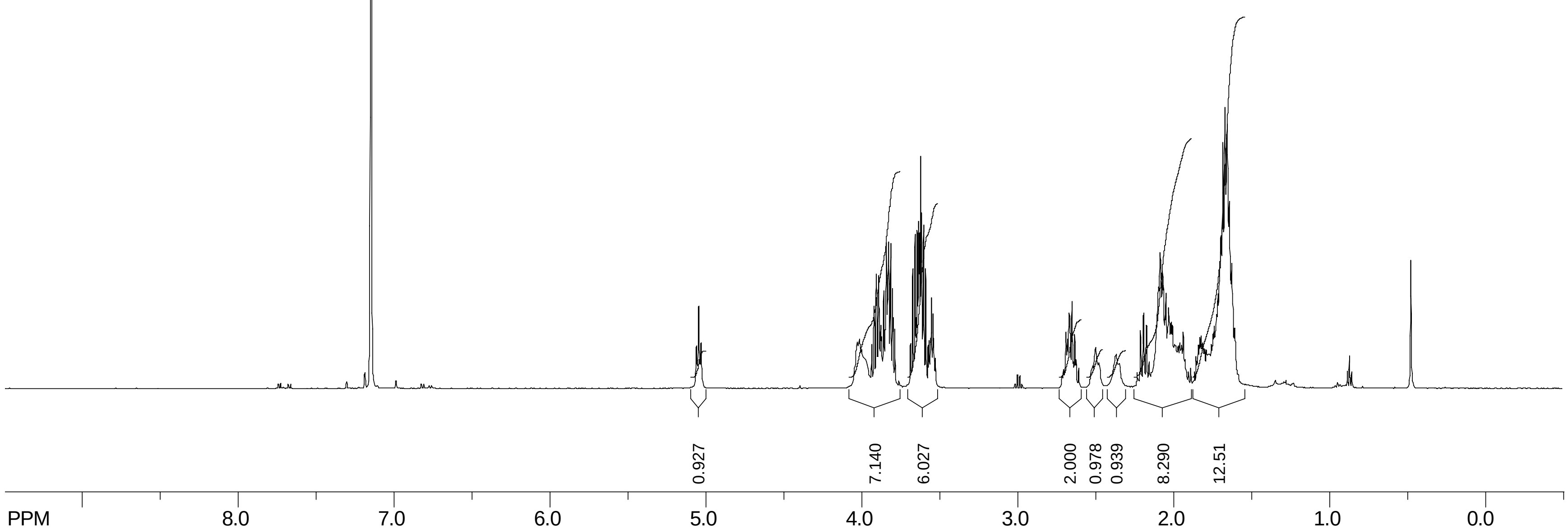
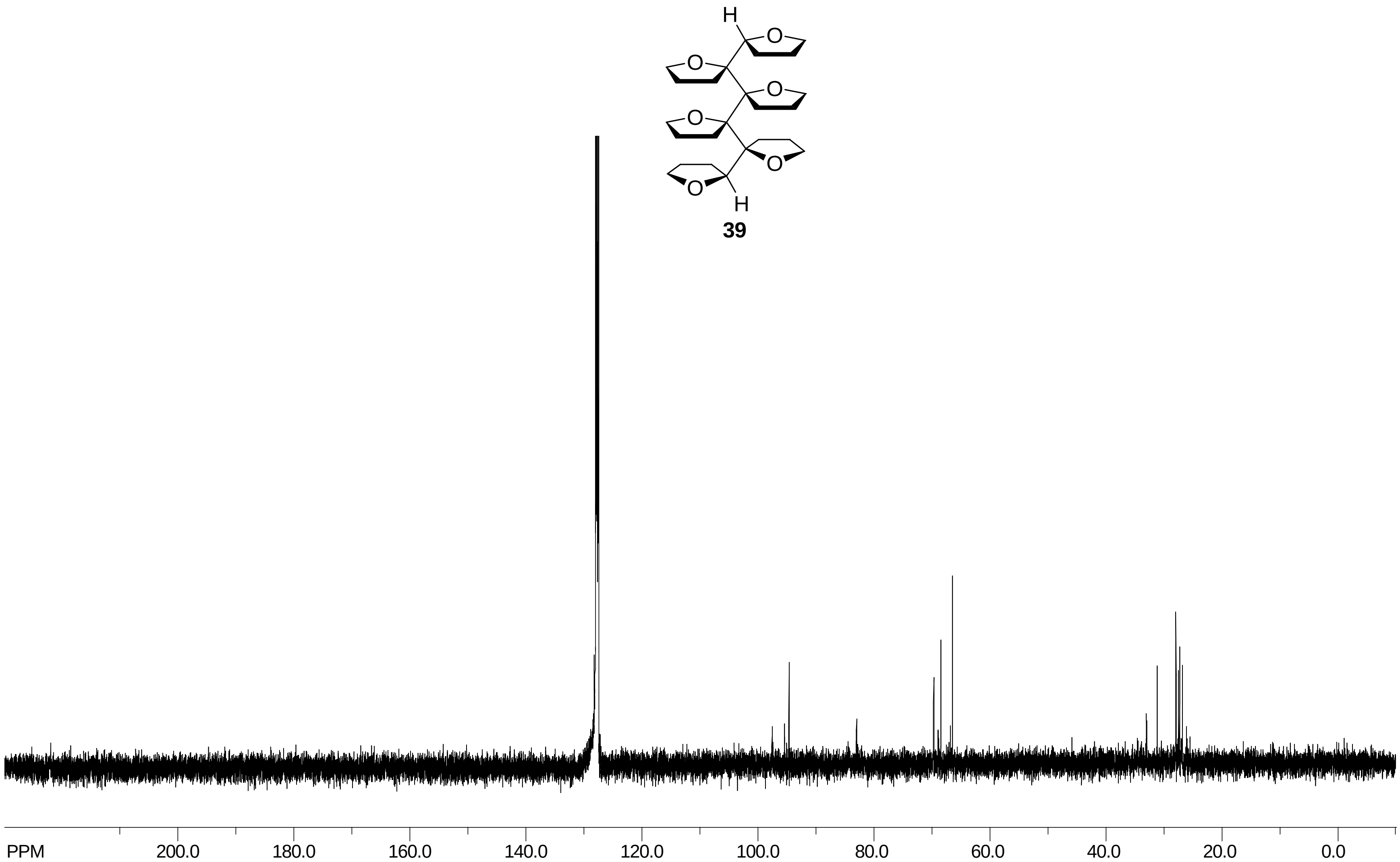
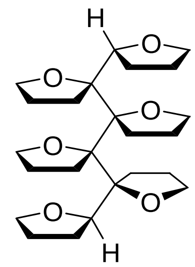


^{1}H NMR : 500 MHz, C_6D_6, 60 ^{o}C



^{13}C NMR : 125 MHz, C_6D_6, 70 ^oC



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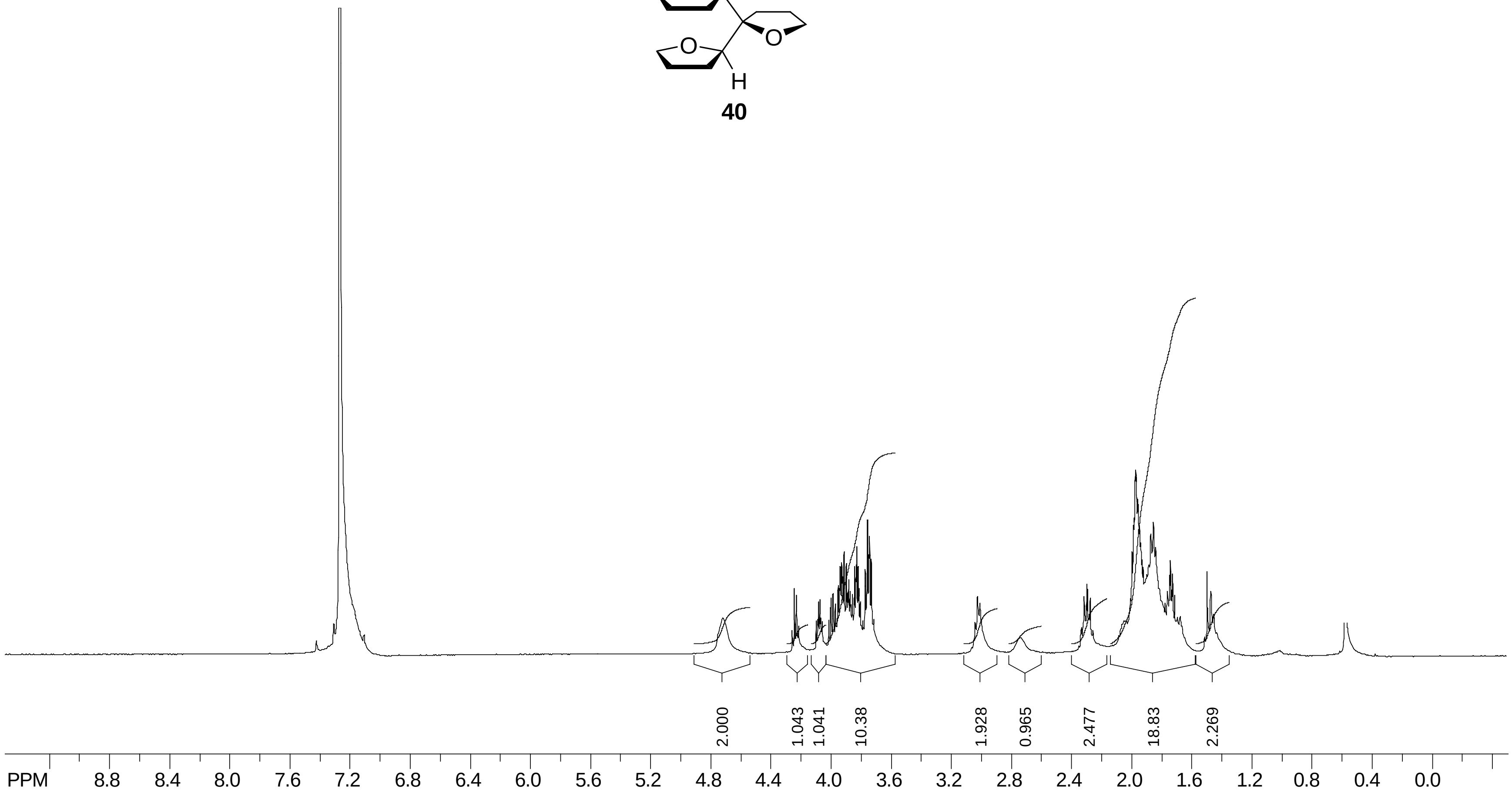
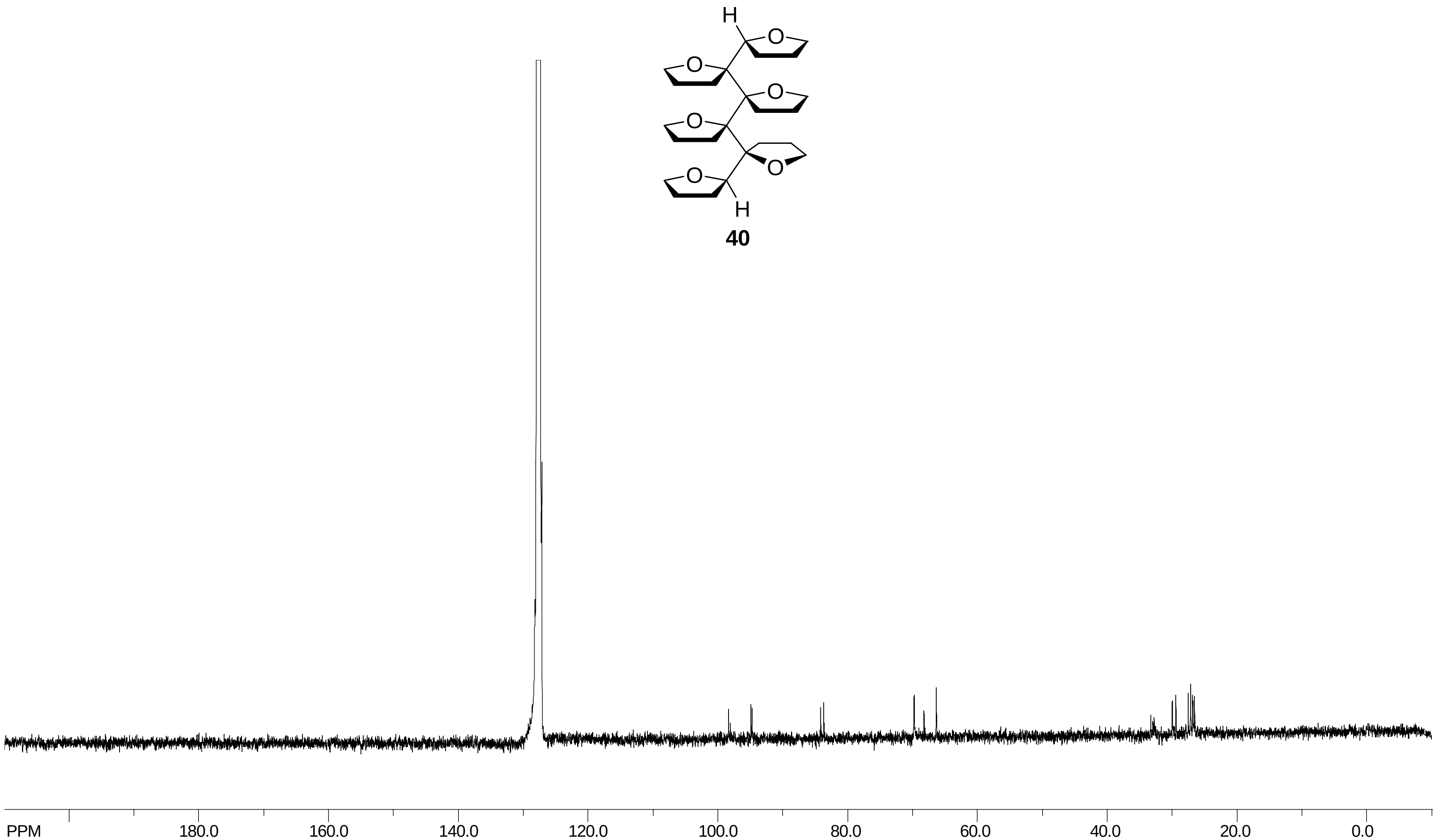


^{1}H NMR : 500 MHz, C_6D_6, 60 ^{o}C



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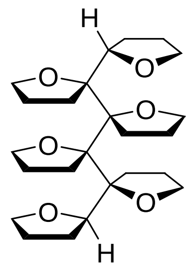
^{13}C NMR : 125 MHz, C_6D_6, 70 ^oC



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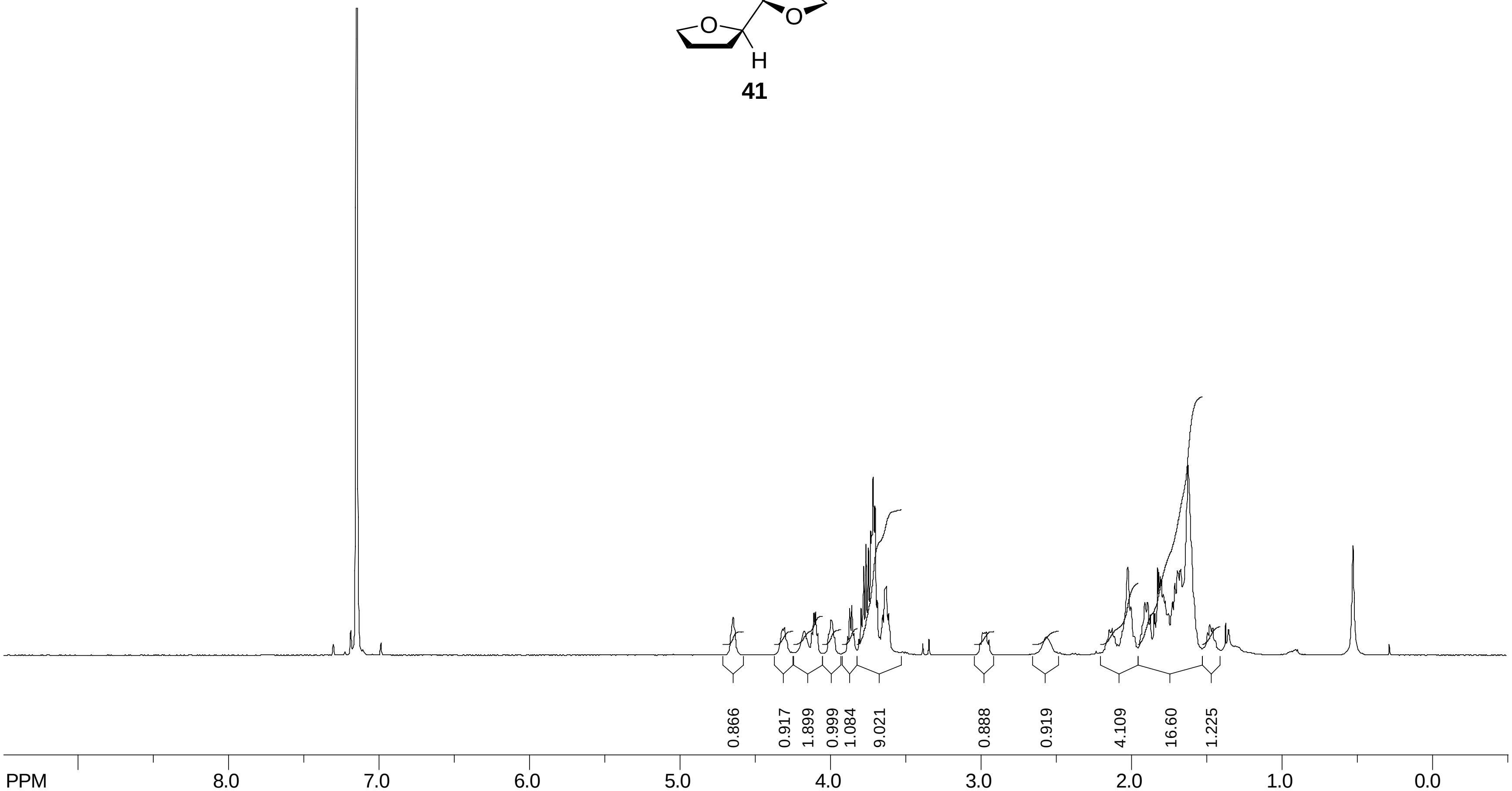
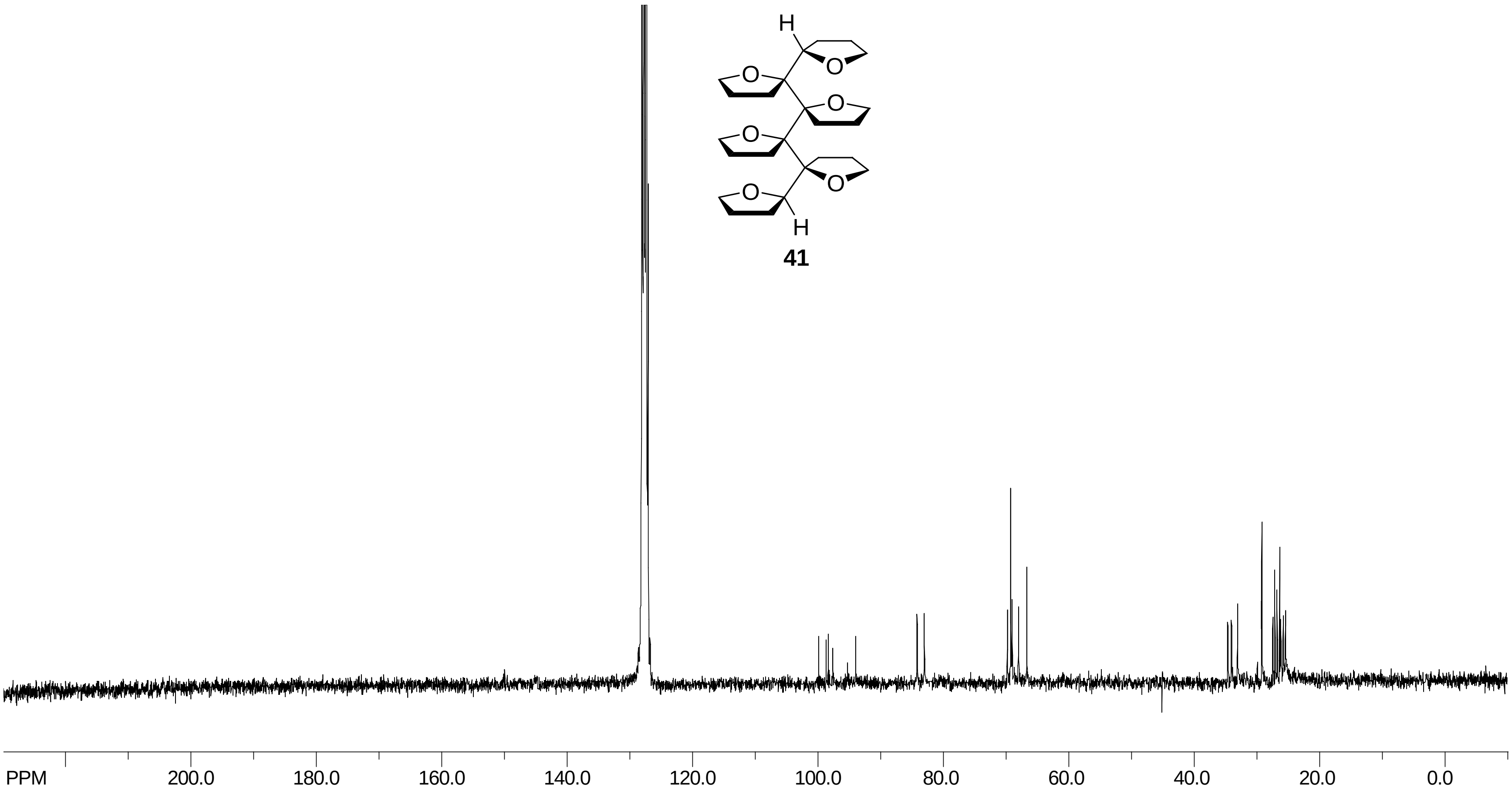


^{1}H NMR : 300 MHz, C_6D_6



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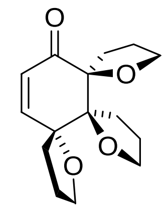
^{13}C NMR : 75 MHz, C_6D_6



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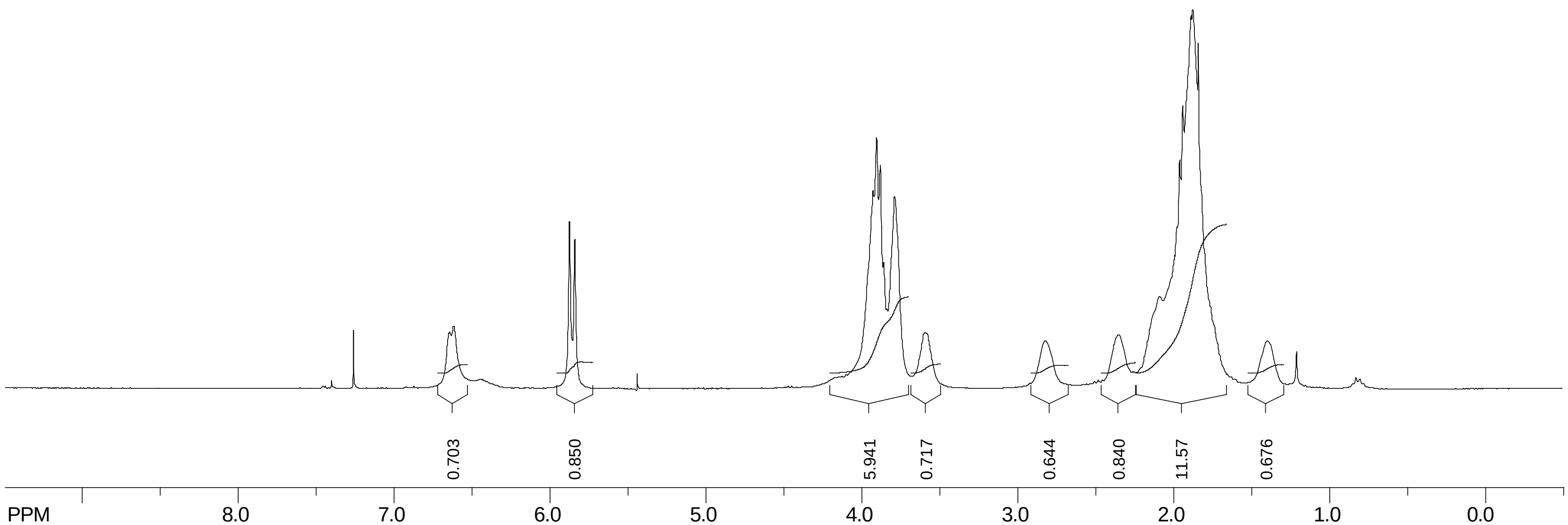
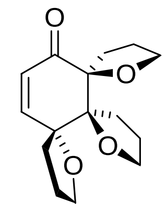


^{1}H NMR : 300 MHz, $CDCl_3$



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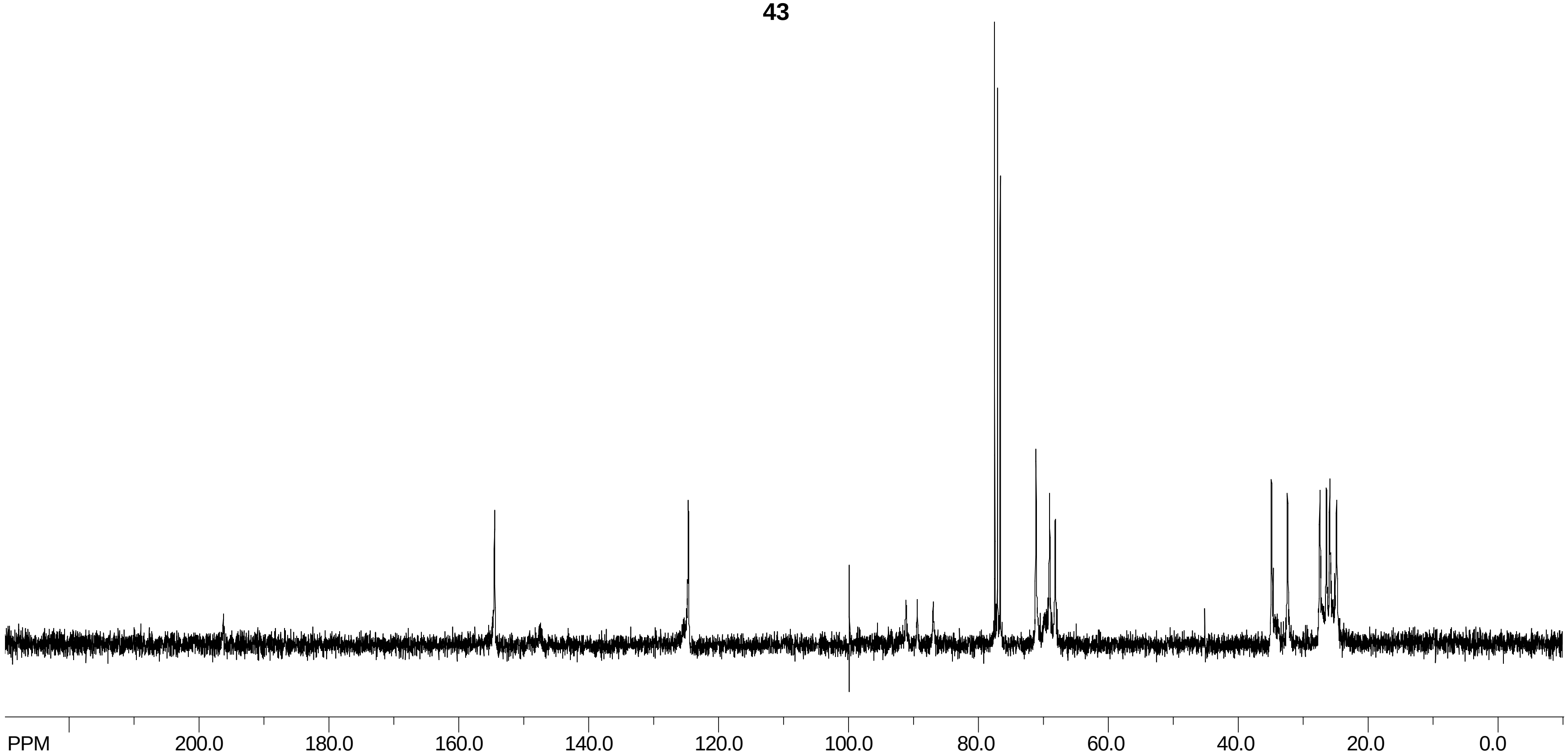
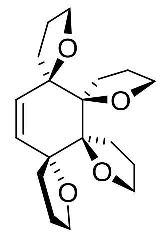


^{13}C NMR : 75 MHz, $CDCl_3$



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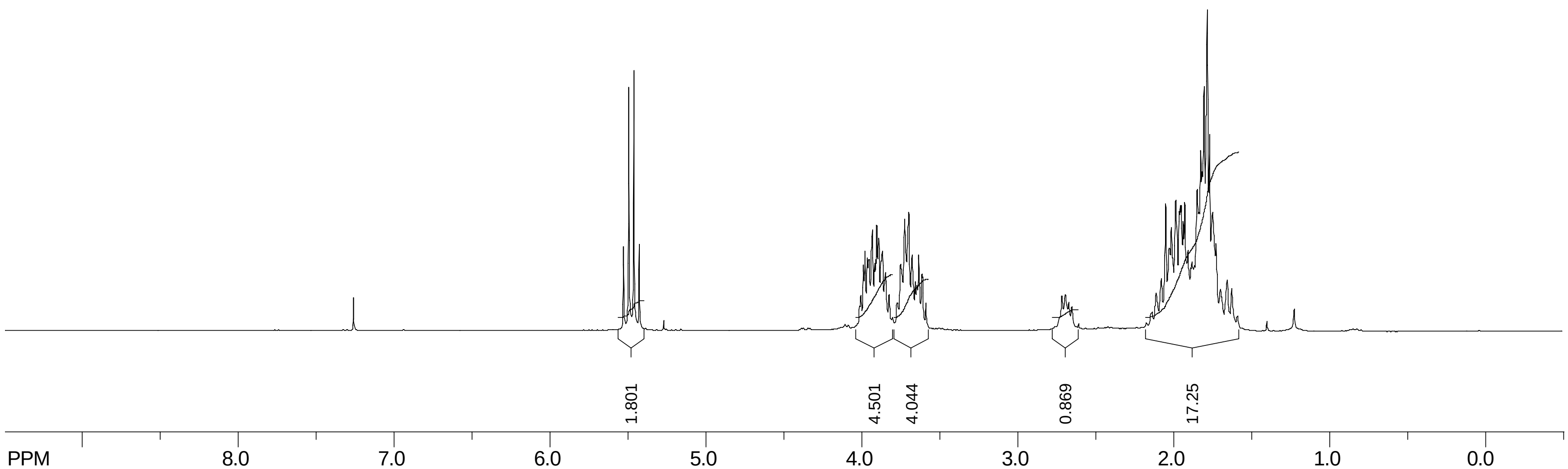
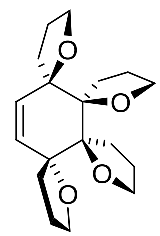


^{1}H NMR : 300 MHz, $CDCl_3$



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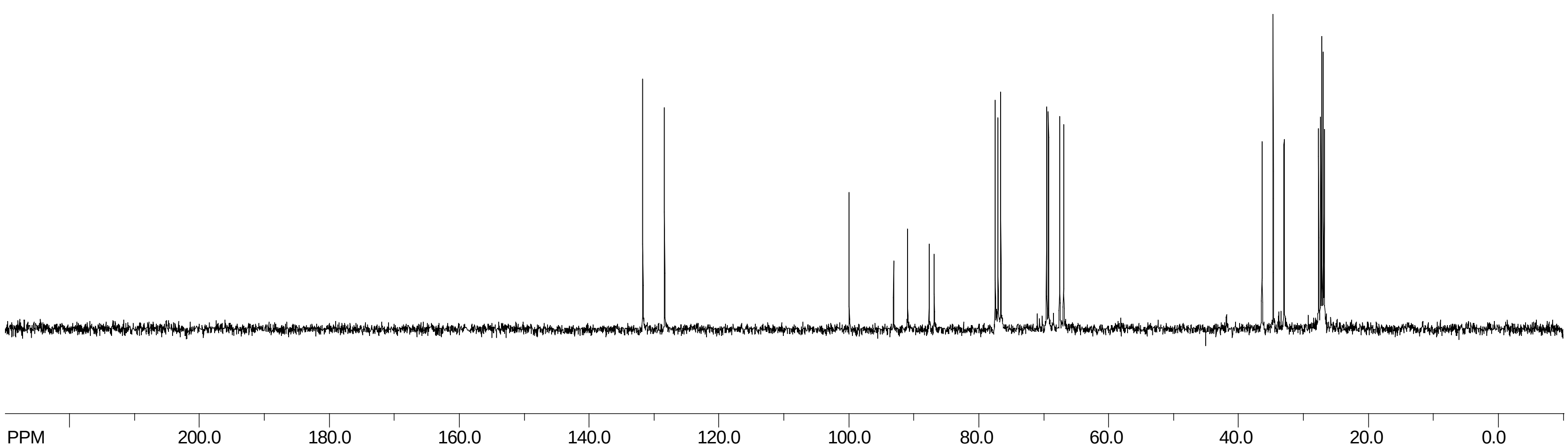
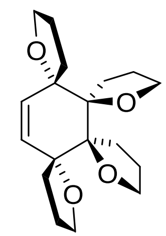


^{13}C NMR : 75 MHz, $CDCl_3$



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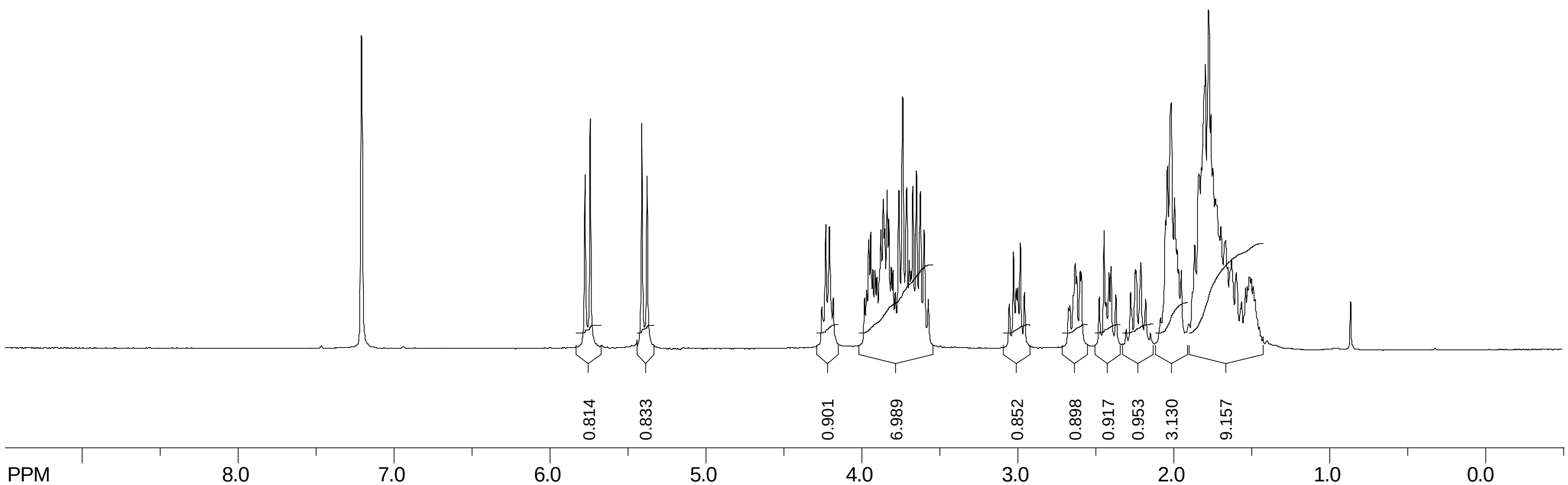
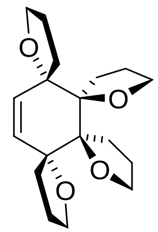


^{1}H NMR : 300 MHz, $CDCl_3$



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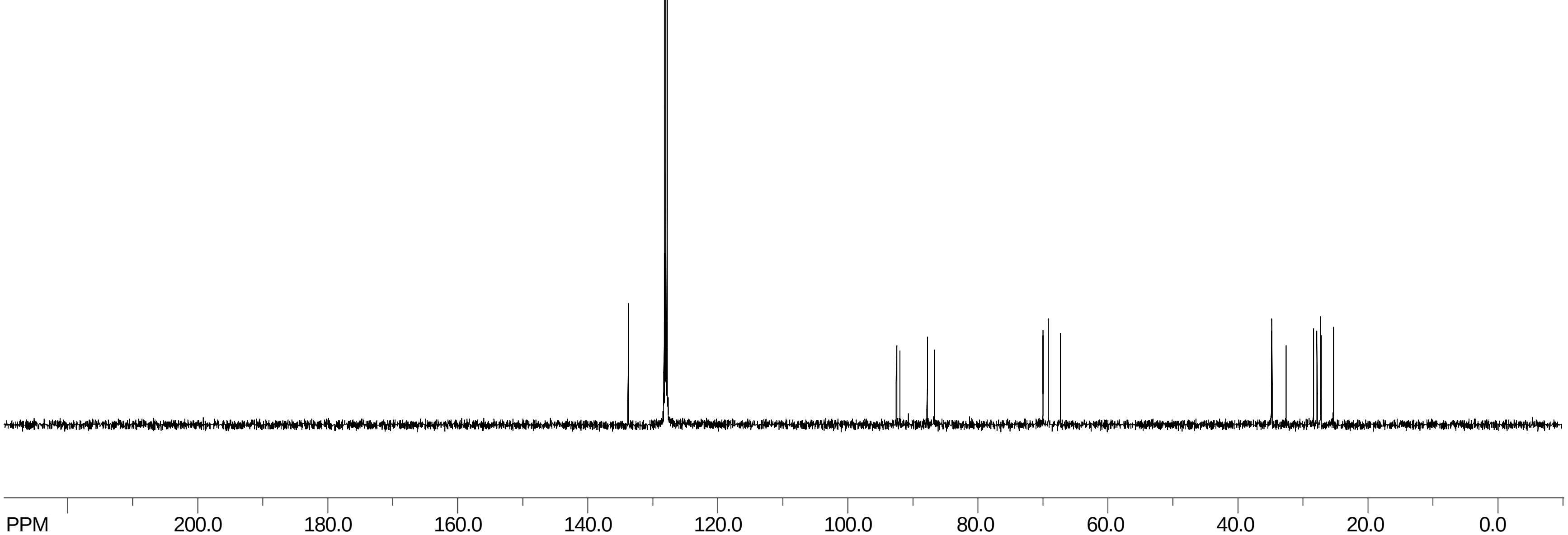
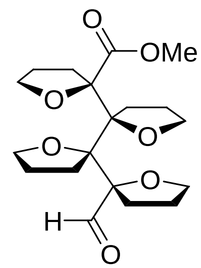


^{13}C NMR : 125 MHz, C_6D_6



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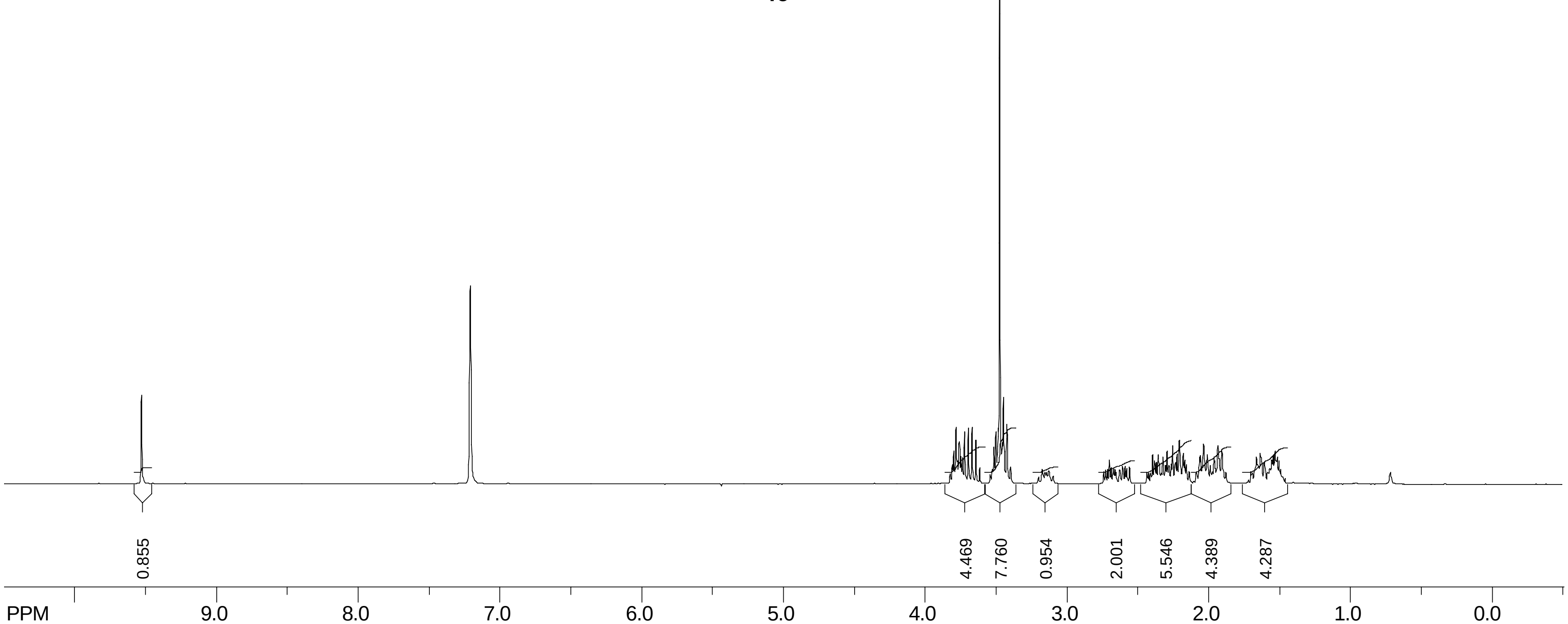
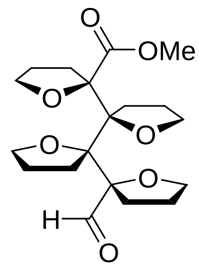


^{1}H NMR : 300 MHz, C_6D_6



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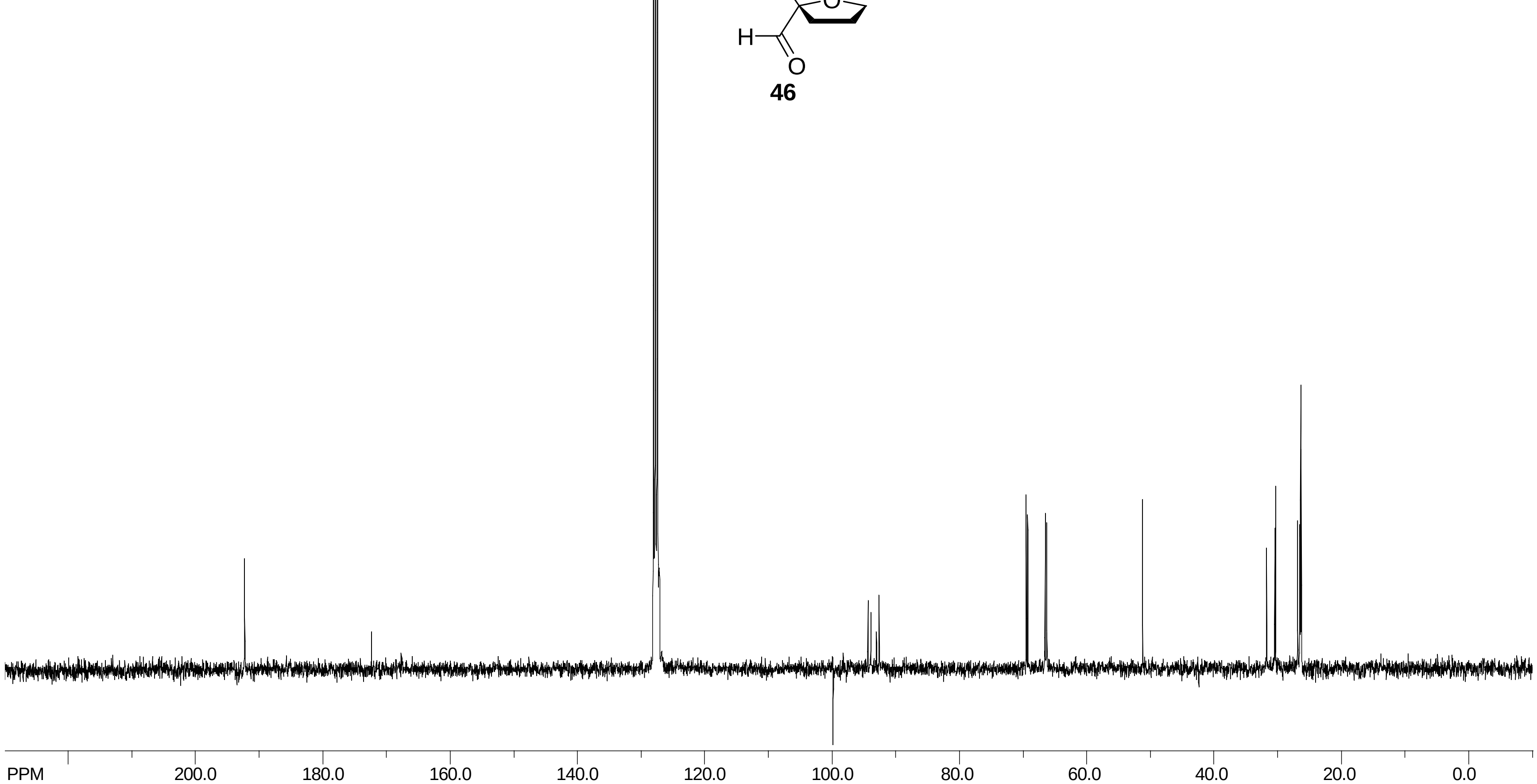
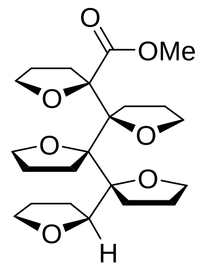


^{13}C NMR : 125 MHz, C_6D_6



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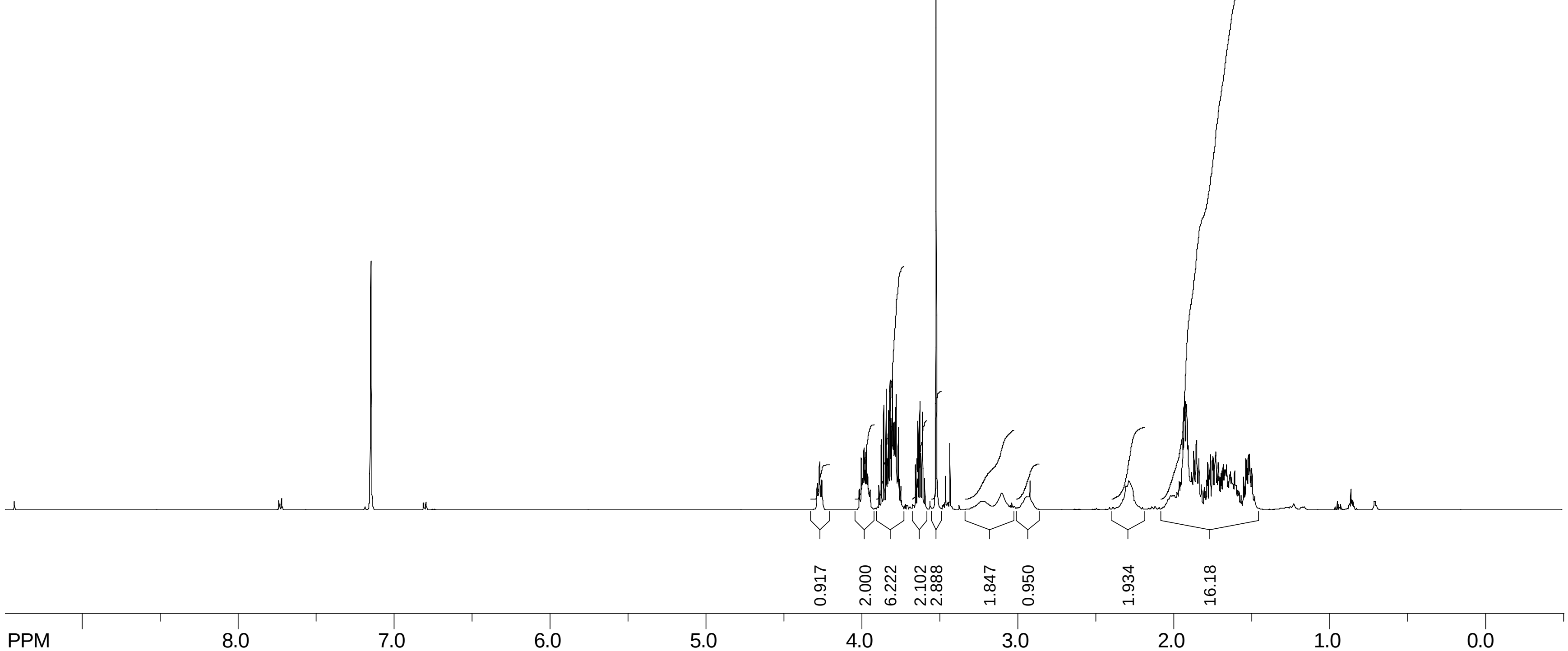
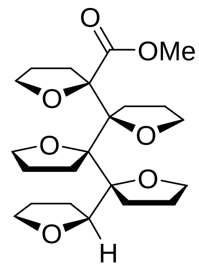


^{1}H NMR : 500 MHz, C_6D_6, 60 ^{o}C



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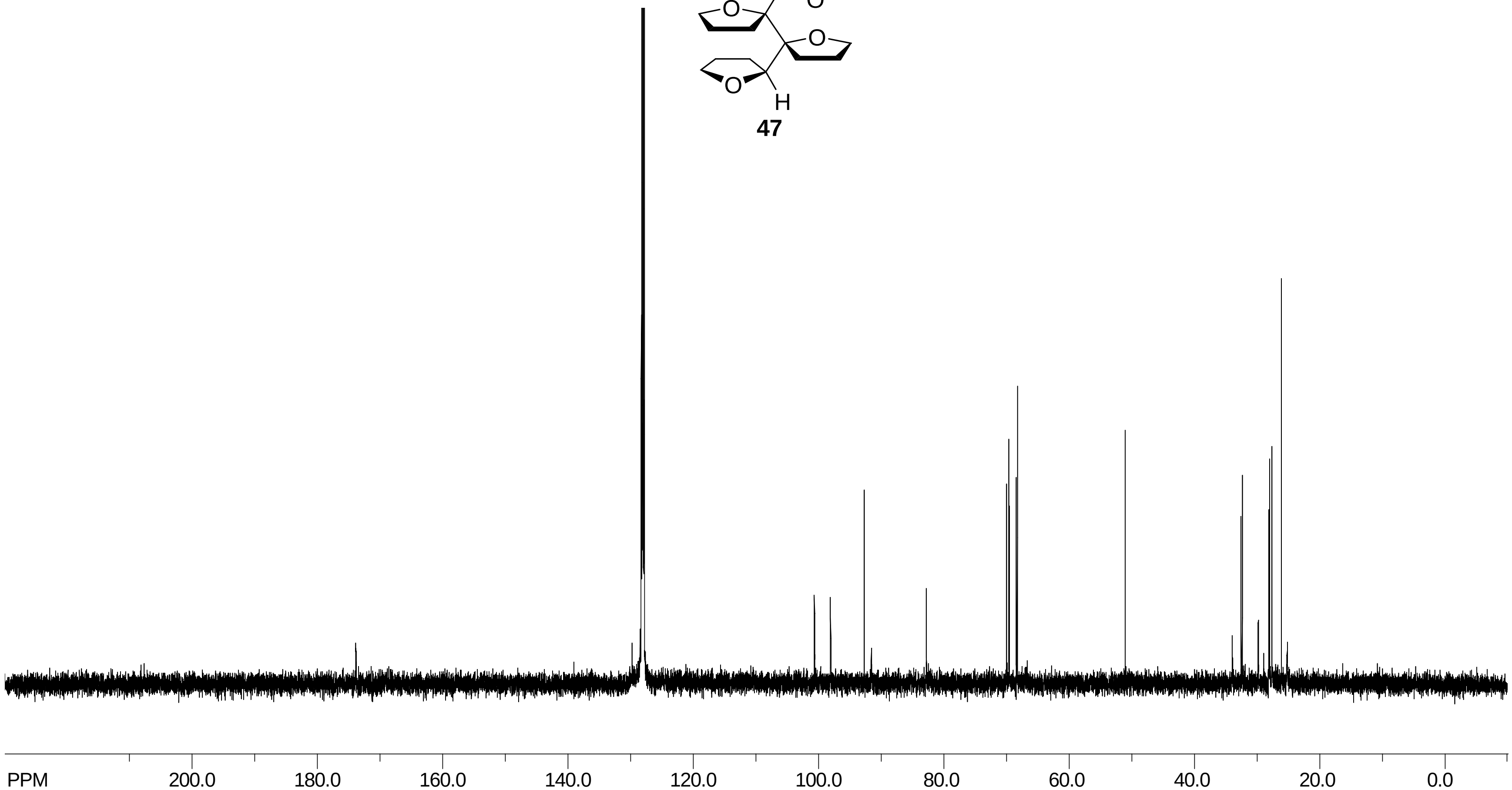
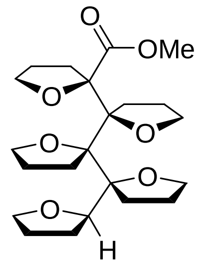


^{13}C NMR : 125 MHz, C_6D_6, 60 ^oC



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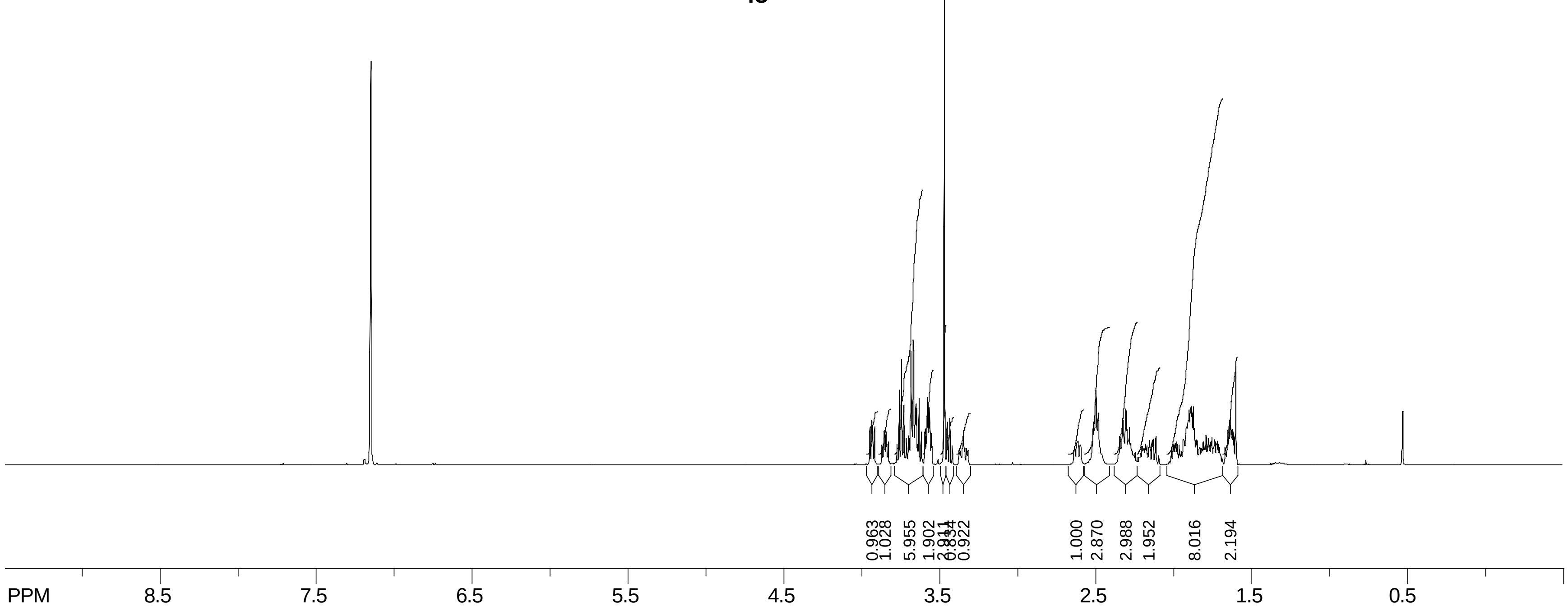
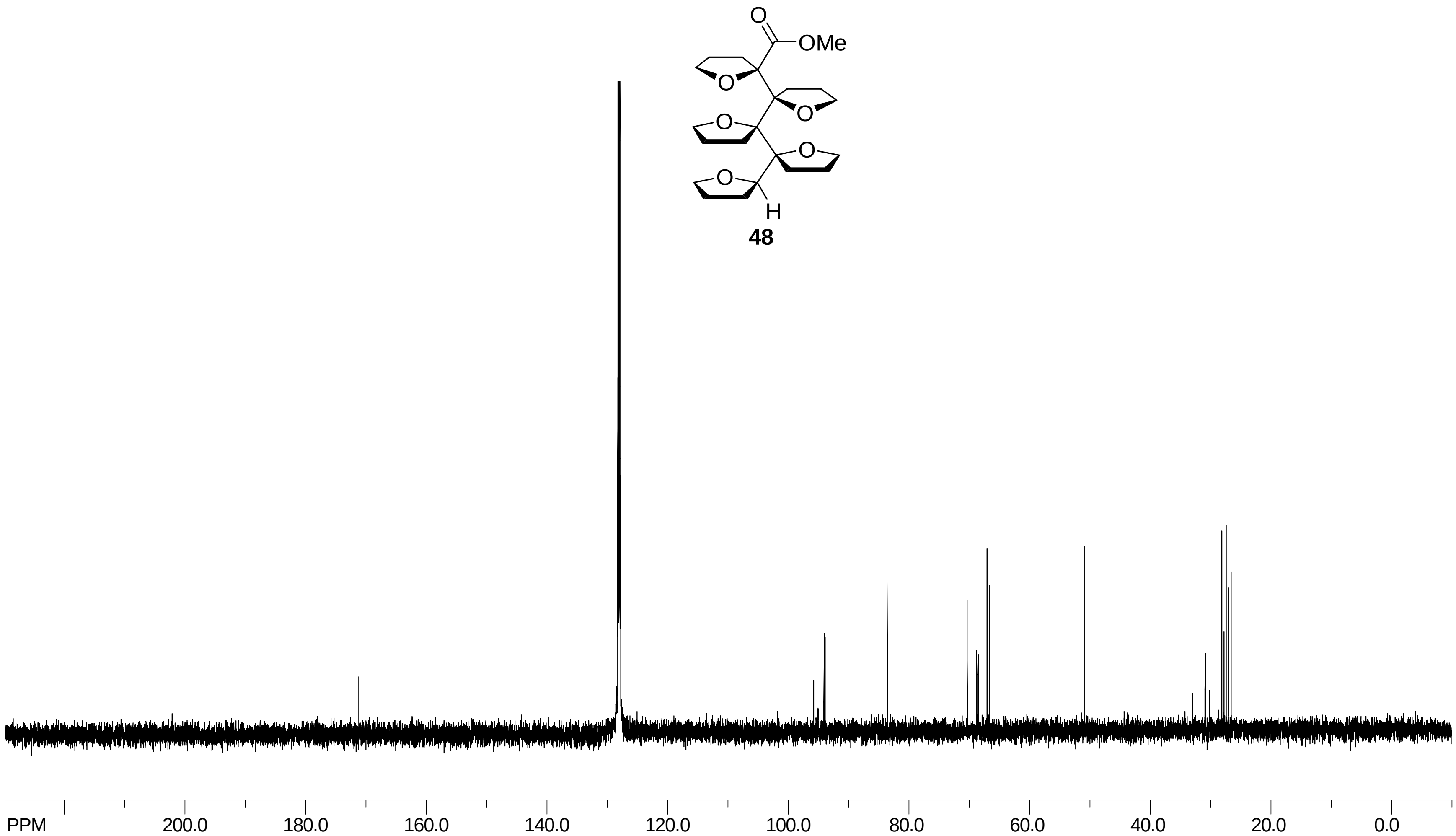
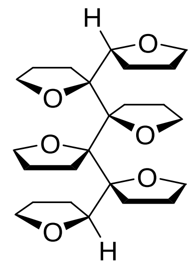


^{1}H NMR : 500 MHz, C_6D_6, 60 ^{o}C



^{13}C NMR : 125 MHz, C_6D_6, 60 ^oC



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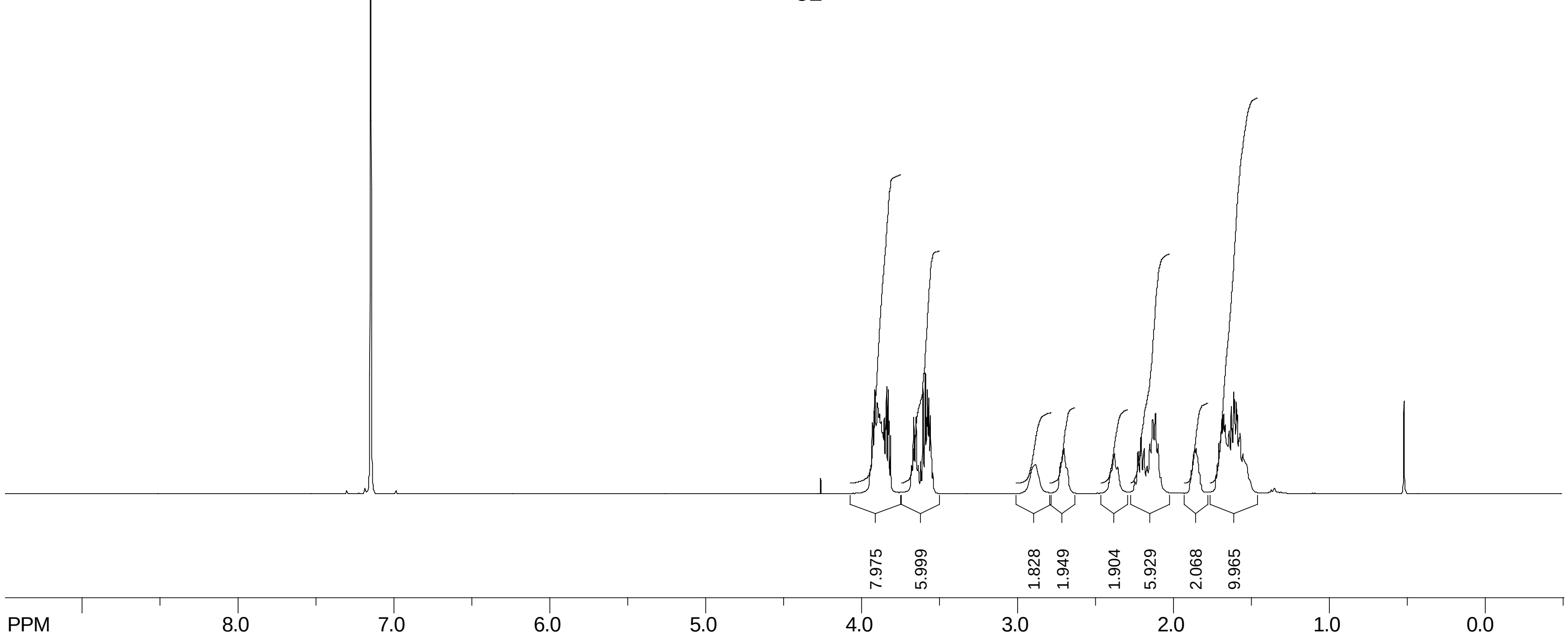
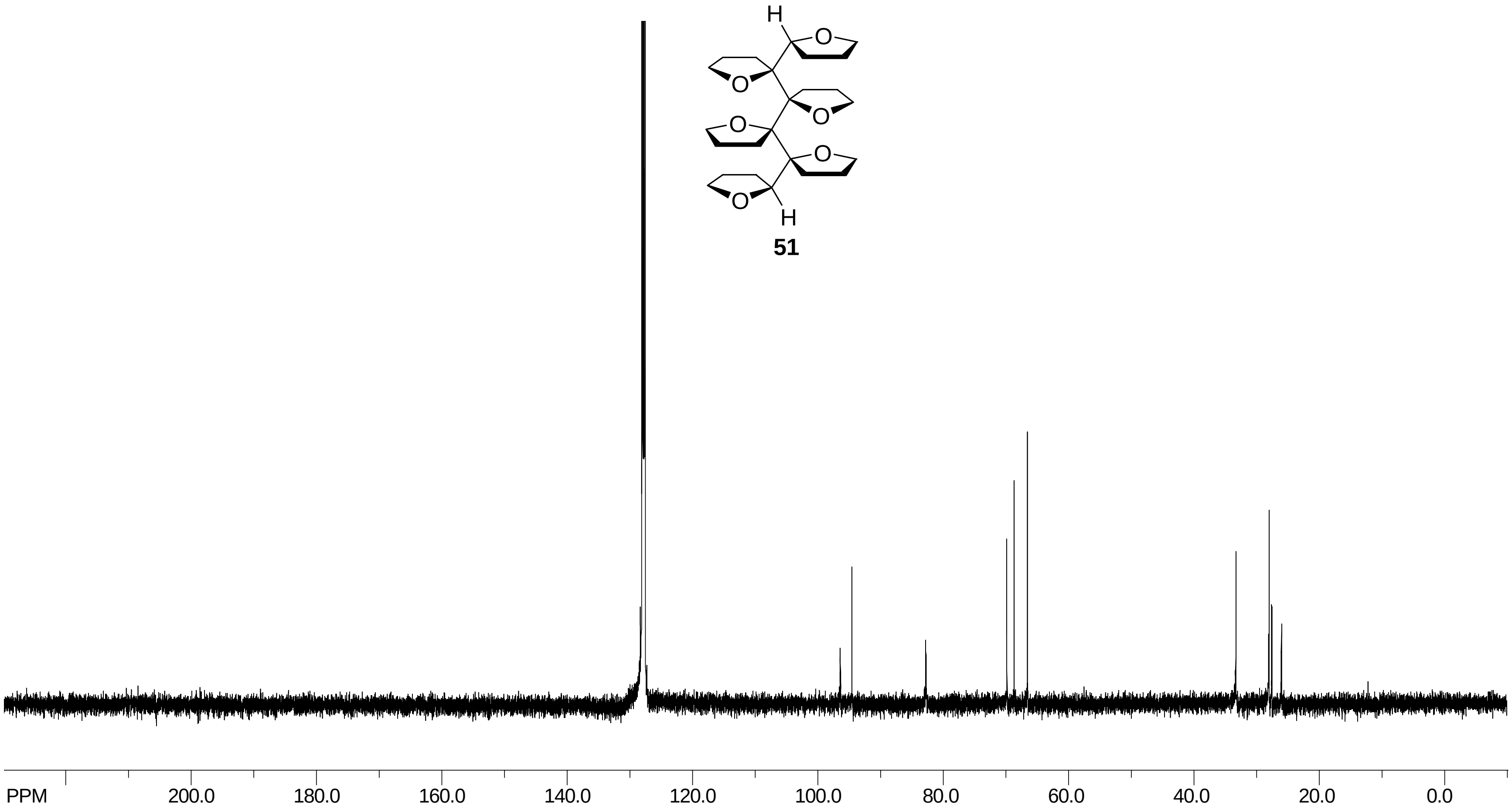
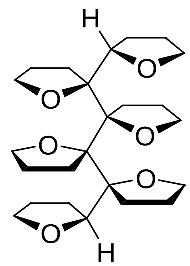


^{1}H NMR : 300 MHz, C_6D_6



^{13}C NMR : 75 MHz, C_6D_6



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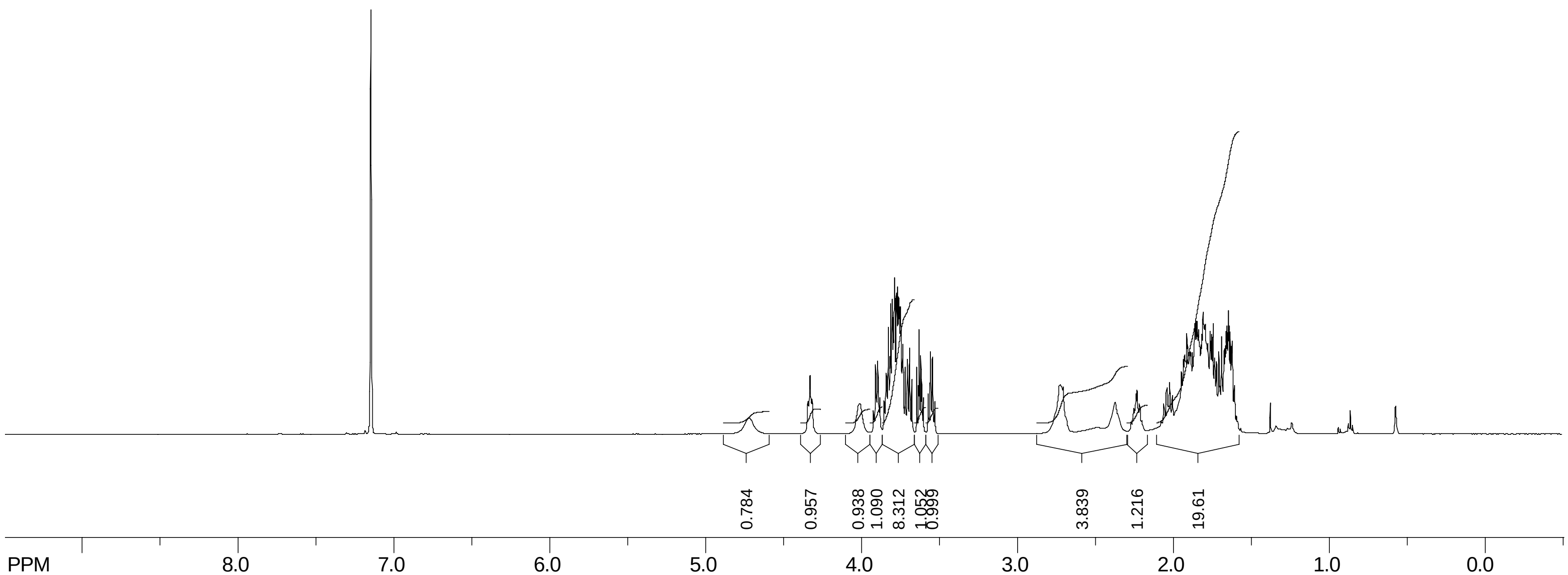
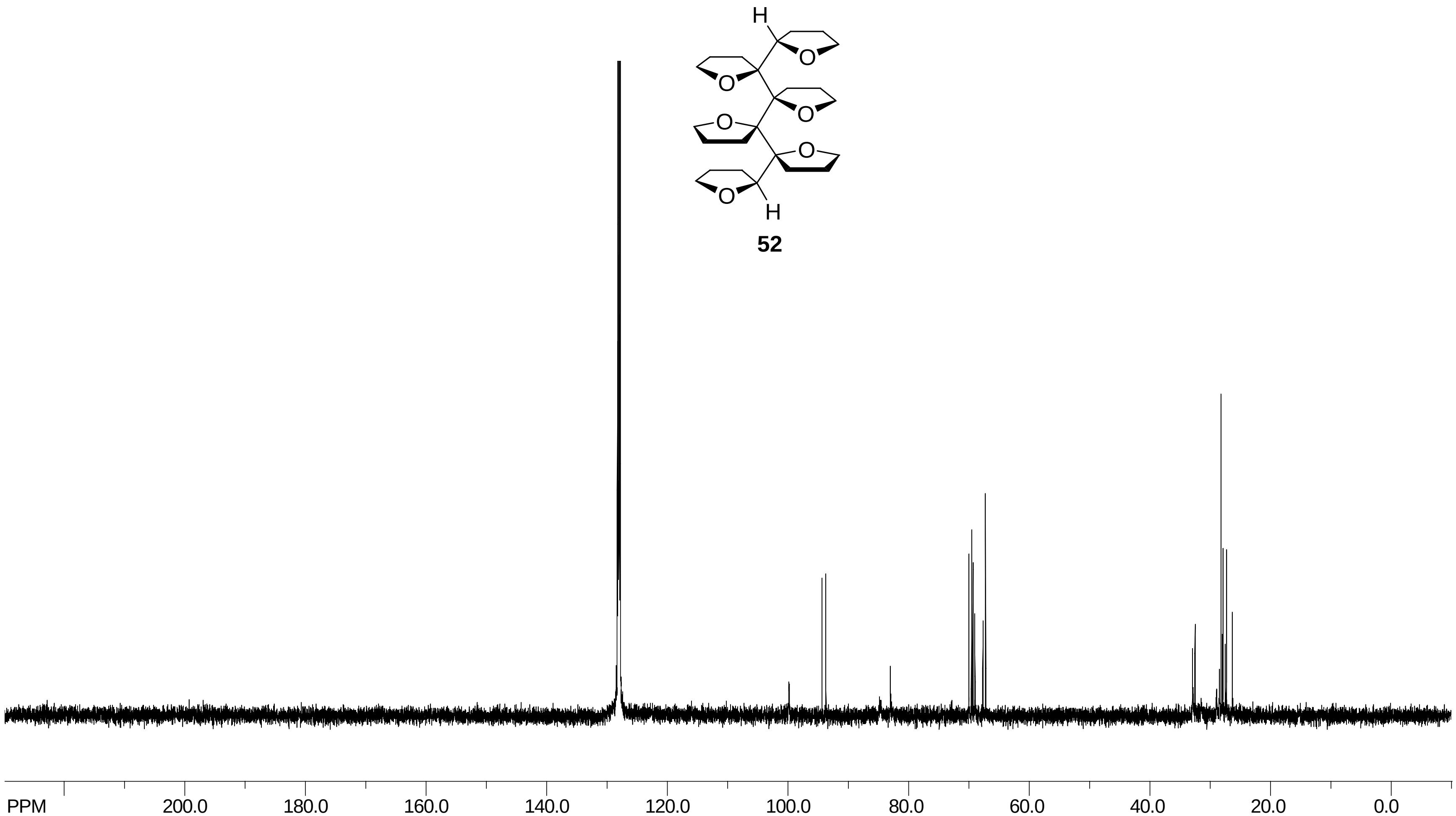
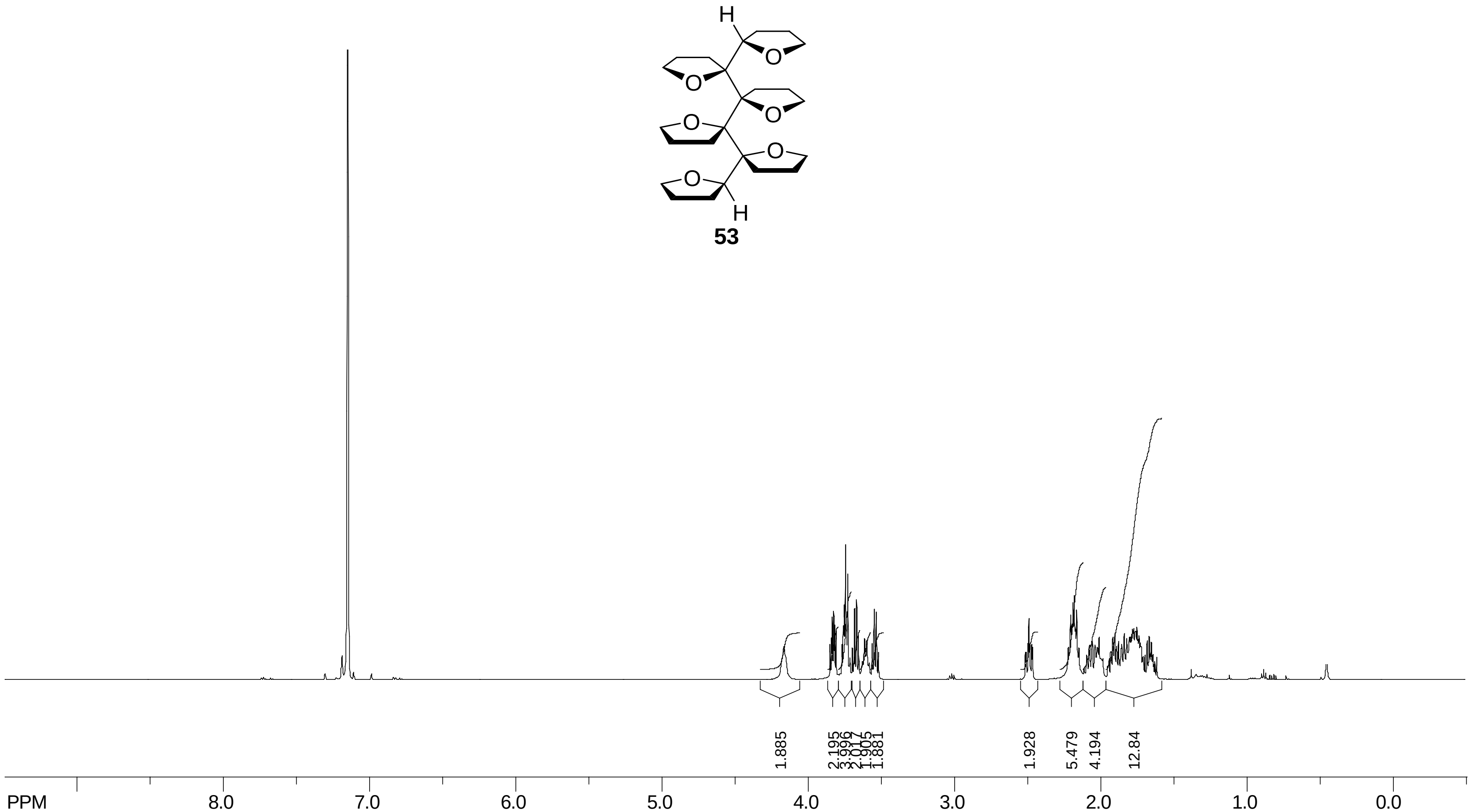


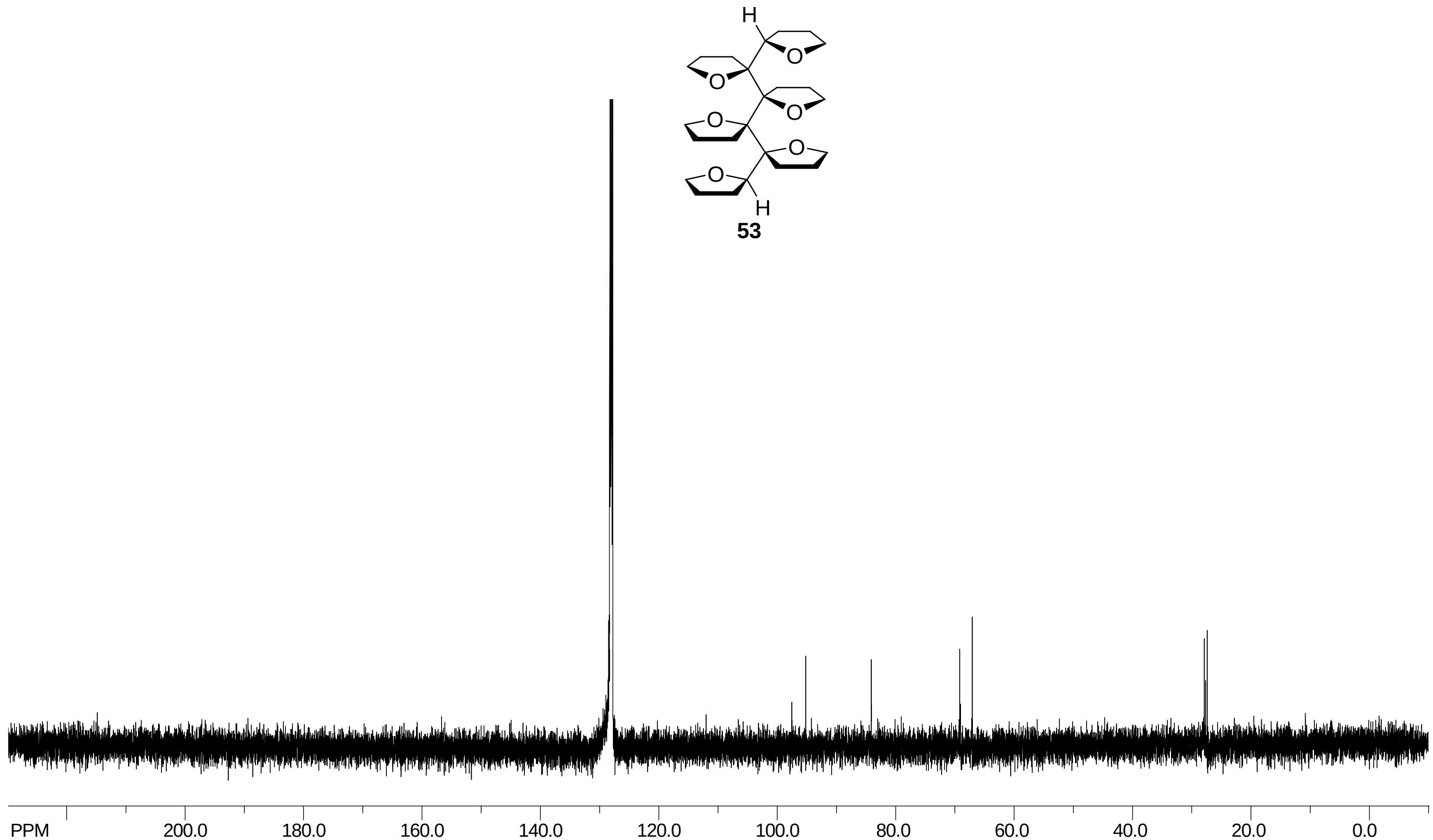
^{1}H NMR : 500 MHz, C_6D_6, 70 ^{o}C



^{13}C NMR : 125 MHz, C_6D_6, 70 ^{o}C



^{1}H NMR : 500 MHz, C_6D_6, 70 ^{o}C



^{13}C NMR : 125 MHz, C_6D_6, 70 ^oC