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Notebook references
P11474-87-A

TO: Mark Teasley, CRD, E328/264, x2145

FROM: R. Harlow, CRD, E228/316d, x2097

X-RAY CRYSTAL STRUCTURE ANALYSIS 96237
CYCLO-[-C_6H_4-CO-C_6H_4-O-]$_2$

CRYSTAL DATA: $O_4C_{26}H_{16}$, from THF/ether, colorless, rectangular plate, ~0.37 x 0.35 x 0.12mm, triclinic, P$\bar{1}$ (No. 2), a = 9.291(1), b = 13.371(2), c = 8.813(1)Å, α = 95.04(1), β = 117.69(1), γ = 98.16(1)°, T = -118° C, V = 944.8Å^3, Z = 2, FW = 392.41, Dc = 1.379g/cc, μ(Mo) = 0.87cm^{-1}.

DATA COLLECTION: Rigaku RU300, R-AXIS image plate area detector, MoKα radiation, anode power = 55Kv x 160ma, crystal to plate distance = 85.0mm, 105u pixel raster, number of frames = 30, oscillation range = 6.0° /frame, exposure = 8.0 min/frame, box sum integration, 2393 data collected,2.7° $\leq 2\theta \leq$ 48.4°, maximum h,k,l = 13 17 13, no absorption correction, 1977 unique reflections with I$\geq 3.0\sigma$(I).

SOLUTION AND REFINEMENT: Structure solved by direct methods (SHELXS), refinement by full-matrix least squares on F, scattering factors from Int. Tables for X-ray Crystallography, Vol IV, biweight $\propto [\sigma^2(I)+0.0009I^2]^{-1/2}$, (excluded 9), refined anisotropic: all non-hydrogen atoms, isotropic: H . 335 parameters, data/parameter ratio = 5.90, final R = 0.034, Rw = 0.036, error of fit = 1.07, max Δ/σ = 0.06, largest residual density = 0.12e/Å^3, near C8

RESULTS: The crystal structure consists of one independent molecule of the cyclo-keto-ether. All bond distances and angles fall within expected ranges. The molecule is shaped like an open mouth, with C9/C10 much closer to C22/C23 than C12/C13 are to C25/C26. In the opening process, the rings containing C1-C6 and C14-C19 bend slightly backward. The dihedral angle between rings containing C9-C13 and C21-C26 is 119.48 deg: the angle for rings C1-C6 and C14-C19 is 36.31 deg. There are no close intermolecular contacts but some of the rings pack roughly parallel to those of neighboring molecules.

TABLE I. Fractional Coordinates and Isotropic Thermal Parameters

ATOM	X	Y	Z	BISO
O(1)	-0.3583(2)	0.1615(1)	0.0710(2)	3.20(5)'
O(2)	0.2586(2)	0.5207(1)	0.5817(2)	3.22(5)'
O(3)	0.8689(2)	0.3444(1)	0.7809(2)	3.33(5)'
O(4)	0.2480(2)	-0.0146(1)	0.2720(2)	2.97(5)'
C(1)	-0.1461(2)	0.0643(1)	0.1755(2)	2.4(1)'
C(2)	0.0205(2)	0.0708(1)	0.2229(2)	2.5(1)'
C(3)	0.0836(2)	-0.0181(1)	0.2291(2)	2.5(1)'
C(4)	-0.0186(2)	-0.1134(1)	0.1851(2)	2.9(1)'
C(5)	-0.1846(2)	-0.1190(1)	0.1354(2)	3.1(1)'
C(6)	-0.2497(2)	-0.0312(1)	0.1310(2)	2.7(1)'
C(7)	-0.2125(2)	0.1607(1)	0.1695(2)	2.5(1)'
C(8)	-0.0947(2)	0.2560(1)	0.2847(2)	2.4(1)'
C(9)	0.0206(2)	0.2560(1)	0.4581(2)	2.6(1)'
C(10)	0.1347(2)	0.3436(1)	0.5606(2)	2.7(1)'
C(11)	0.1412(2)	0.4315(1)	0.4883(2)	2.6(1)'
C(12)	0.0237(2)	0.4347(1)	0.3203(2)	2.7(1)'
C(13)	-0.0945(2)	0.3476(1)	0.2202(2)	2.7(1)'
C(14)	0.4218(2)	0.5122(1)	0.6853(2)	2.7(1)'
C(15)	0.4855(2)	0.4307(1)	0.6508(2)	2.6(1)'
C(16)	0.6509(2)	0.4270(1)	0.7568(2)	2.5(1)'
C(17)	0.7524(2)	0.5084(1)	0.8923(2)	2.9(1)'
C(18)	0.6878(2)	0.5905(1)	0.9218(2)	3.2(1)'
C(19)	0.5222(2)	0.5928(1)	0.8210(2)	3.1(1)'
C(20)	0.7189(2)	0.3381(1)	0.7209(2)	2.6(1)'
C(21)	0.5990(2)	0.2422(1)	0.6083(2)	2.4(1)'
C(22)	0.4814(2)	0.1937(1)	0.6487(2)	2.5(1)'
C(23)	0.3665(2)	0.1073(1)	0.5405(2)	2.6(1)'
C(24)	0.3641(2)	0.0712(1)	0.3866(2)	2.4(1)'
C(25)	0.4818(2)	0.1168(1)	0.3463(2)	2.7(1)'
C(26)	0.6000(2)	0.2010(1)	0.4579(2)	2.7(1)'
H(2)	0.0935(23)	0.1369(14)	0.2469(22)	3.1(4)
H(5)	-0.2602(24)	-0.1861(15)	0.1008(23)	3.3(4)
H(6)	-0.3690(26)	-0.0344(13)	0.0942(24)	3.5(4)
H(9)	0.0174(21)	0.1924(14)	0.5043(22)	3.2(4)
H(10)	0.2205(24)	0.3459(14)	0.6827(26)	3.7(4)
H(12)	0.0289(23)	0.4980(15)	0.2758(24)	3.7(4)
H(13)	-0.1780(23)	0.3462(14)	0.0978(25)	3.5(4)
H(15)	0.4156(22)	0.3784(13)	0.5536(23)	2.8(4)
H(17)	0.8674(25)	0.5092(13)	0.9680(23)	3.3(4)
H(18)	0.7612(25)	0.6461(15)	1.0155(25)	3.6(4)
H(19)	0.4723(24)	0.6506(16)	0.8397(25)	4.2(4)
H(22)	0.4813(22)	0.2187(13)	0.7562(24)	3.4(4)
H(23)	0.2814(22)	0.0736(13)	0.5673(22)	3.0(4)
H(24)	0.0262(23)	-0.1737(14)	0.1873(23)	3.4(4)
H(25)	0.4785(21)	0.0889(13)	0.2430(24)	3.2(4)
H(26)	0.6846(24)	0.2333(14)	0.4338(23)	3.3(4)

TABLE II. Anisotropic Thermal Parameters:
exp[-19.739(U11hha*a*...+2(U12hka*b*...))]

ATOM	U11	U22	U33	U12	U13	U23
O(1)	0.0279(9)	0.0413(7)	0.0438(7)	0.0053(6)	0.0108(6)	0.0075(6)
O(2)	0.0263(8)	0.0323(7)	0.0511(7)	0.0075(6)	0.0098(6)	-0.0033(6)
O(3)	0.0274(9)	0.0383(7)	0.0515(8)	0.0083(6)	0.0114(6)	0.0064(6)
O(4)	0.0309(8)	0.0328(7)	0.0430(7)	0.0055(6)	0.0148(6)	-0.0033(6)
C(1)	0.0279(11)	0.0332(9)	0.0267(8)	0.0031(8)	0.0105(8)	0.0044(7)
C(2)	0.0320(12)	0.0325(10)	0.0297(9)	0.0030(8)	0.0147(9)	0.0048(8)
C(3)	0.0275(11)	0.0348(10)	0.0282(9)	0.0064(8)	0.0112(8)	0.0038(7)
C(4)	0.0381(12)	0.0292(10)	0.0367(9)	0.0055(9)	0.0154(9)	0.0028(8)
C(5)	0.0366(13)	0.0299(10)	0.0420(10)	-0.0019(9)	0.0155(9)	0.0030(8)
C(6)	0.0288(12)	0.0370(10)	0.0315(9)	0.0014(8)	0.0109(9)	0.0031(8)
C(7)	0.0278(11)	0.0370(10)	0.0305(9)	0.0057(8)	0.0137(9)	0.0088(8)
C(8)	0.0244(10)	0.0316(9)	0.0346(9)	0.0061(7)	0.0143(8)	0.0043(7)
C(9)	0.0299(11)	0.0357(10)	0.0355(10)	0.0075(8)	0.0164(9)	0.0081(8)
C(10)	0.0285(11)	0.0387(10)	0.0319(9)	0.0087(9)	0.0112(9)	0.0017(8)
C(11)	0.0234(11)	0.0305(9)	0.0407(10)	0.0055(8)	0.0140(9)	-0.0041(8)
C(12)	0.0287(11)	0.0307(10)	0.0417(10)	0.0076(8)	0.0149(9)	0.0045(8)
C(13)	0.0278(11)	0.0363(10)	0.0353(10)	0.0088(8)	0.0129(9)	0.0066(8)
C(14)	0.0269(11)	0.0348(10)	0.0363(9)	0.0086(8)	0.0122(8)	0.0039(8)
C(15)	0.0298(11)	0.0330(10)	0.0319(9)	0.0058(8)	0.0115(9)	0.0021(8)
C(16)	0.0272(11)	0.0335(9)	0.0320(9)	0.0051(8)	0.0115(8)	0.0047(8)
C(17)	0.0282(12)	0.0385(10)	0.0342(9)	0.0046(9)	0.0081(9)	0.0035(8)
C(18)	0.0369(13)	0.0369(11)	0.0352(10)	0.0044(9)	0.0091(9)	-0.0038(8)
C(19)	0.0383(13)	0.0342(10)	0.0396(10)	0.0073(9)	0.0153(9)	-0.0017(8)
C(20)	0.0267(12)	0.0358(10)	0.0340(9)	0.0097(8)	0.0118(9)	0.0086(8)
C(21)	0.0255(10)	0.0301(9)	0.0335(9)	0.0089(8)	0.0103(8)	0.0066(7)
C(22)	0.0298(11)	0.0339(10)	0.0304(9)	0.0077(8)	0.0125(8)	0.0039(8)
C(23)	0.0293(11)	0.0342(9)	0.0348(9)	0.0068(8)	0.0152(9)	0.0070(8)
C(24)	0.0249(10)	0.0279(9)	0.0338(9)	0.0069(8)	0.0093(8)	0.0030(7)
C(25)	0.0319(11)	0.0382(10)	0.0334(9)	0.0104(8)	0.0160(9)	0.0030(8)
C(26)	0.0281(11)	0.0393(10)	0.0395(10)	0.0088(9)	0.0181(9)	0.0087(8)

TABLE III. Interatomic Distances (A)

O(1)-C(7)	1.225 (2)
O(2)-C(11)	1.383 (2)
O(2)-C(14)	1.387 (2)
O(3)-C(20)	1.226 (2)
O(4)-C(3)	1.384 (2)
O(4)-C(24)	1.390 (2)
C(1)-C(2)	1.390 (2)
C(1)-C(6)	1.387 (2)
C(1)-C(7)	1.501 (2)
C(2)-C(3)	1.393 (2)
C(3)-C(4)	1.379 (2)
C(4)-C(5)	1.383 (3)
C(5)-C(6)	1.390 (3)
C(7)-C(8)	1.482 (2)
C(8)-C(9)	1.400 (2)
C(8)-C(13)	1.394 (2)
C(9)-C(10)	1.372 (3)
C(10)-C(11)	1.393 (2)
C(11)-C(12)	1.381 (2)
C(12)-C(13)	1.378 (2)
C(14)-C(15)	1.386 (2)
C(14)-C(19)	1.383 (3)
C(15)-C(16)	1.390 (2)
C(16)-C(17)	1.390 (2)
C(16)-C(20)	1.499 (2)
C(17)-C(18)	1.384 (3)
C(18)-C(19)	1.381 (3)
C(20)-C(21)	1.488 (2)
C(21)-C(22)	1.393 (2)
C(21)-C(26)	1.394 (2)
C(22)-C(23)	1.376 (2)
C(23)-C(24)	1.388 (2)
C(24)-C(25)	1.380 (2)
C(25)-C(26)	1.375 (3)
C(2)-H(2)	0.975 (18)
C(4)-H(24)	0.956 (19)
C(5)-H(5)	0.982 (19)
C(6)-H(6)	0.993 (20)
C(9)-H(9)	0.977 (18)
C(10)-H(10)	0.992 (19)
C(12)-H(12)	0.968 (19)
C(13)-H(13)	0.990 (19)
C(15)-H(15)	0.946 (18)
C(17)-H(17)	0.959 (20)
C(18)-H(18)	0.961 (20)
C(19)-H(19)	0.995 (20)
C(22)-H(22)	0.977 (19)
C(23)-H(23)	0.988 (18)
C(25)-H(25)	0.938 (19)
C(26)-H(26)	0.961 (19)

TABLE IV. Intramolecular Angles (Deg)

C(11)-O(2)-C(14)	118.0 (1)	C(20)-C(21)-C(22)	120.7 (2)
C(3)-O(4)-C(24)	117.2 (1)	C(20)-C(21)-C(26)	120.3 (2)
O(4)-C(3)-C(2)	121.7 (1)	C(22)-C(21)-C(26)	119.0 (2)
O(4)-C(3)-C(4)	117.7 (1)	C(21)-C(22)-C(23)	120.5 (2)
O(1)-C(7)-C(1)	121.5 (2)	C(22)-C(23)-C(24)	119.4 (2)
O(1)-C(7)-C(8)	120.8 (2)	C(23)-C(24)-C(25)	120.9 (2)
O(2)-C(11)-C(10)	122.4 (2)	C(24)-C(25)-C(26)	119.4 (2)
O(2)-C(11)-C(12)	116.8 (1)	C(21)-C(26)-C(25)	120.7 (2)
O(2)-C(14)-C(15)	122.6 (2)	C(1)-C(2)-H(2)	121 (1)
O(2)-C(14)-C(19)	116.4 (2)	C(3)-C(2)-H(2)	119 (1)
O(3)-C(20)-C(16)	121.1 (2)	C(3)-C(4)-H(24)	120 (1)
O(3)-C(20)-C(21)	120.8 (1)	C(5)-C(4)-H(24)	122 (1)
O(4)-C(24)-C(23)	121.2 (1)	C(4)-C(5)-H(5)	120 (1)
O(4)-C(24)-C(25)	117.9 (1)	C(6)-C(5)-H(5)	118 (1)
C(2)-C(1)-C(6)	119.6 (2)	C(1)-C(6)-H(6)	119 (1)
C(2)-C(1)-C(7)	119.6 (2)	C(5)-C(6)-H(6)	122 (1)
C(6)-C(1)-C(7)	120.8 (2)	C(8)-C(9)-H(9)	118 (1)
C(1)-C(2)-C(3)	120.2 (2)	C(10)-C(9)-H(9)	121 (1)
C(2)-C(3)-C(4)	120.6 (2)	C(9)-C(10)-H(10)	123 (1)
C(3)-C(4)-C(5)	118.8 (2)	C(11)-C(10)-H(10)	118 (1)
C(4)-C(5)-C(6)	121.6 (2)	C(11)-C(12)-H(12)	119 (1)
C(1)-C(6)-C(5)	119.3 (2)	C(13)-C(12)-H(12)	122 (1)
C(1)-C(7)-C(8)	117.7 (1)	C(8)-C(13)-H(13)	117 (1)
C(7)-C(8)-C(9)	121.2 (1)	C(12)-C(13)-H(13)	122 (1)
C(7)-C(8)-C(13)	120.1 (1)	C(14)-C(15)-H(15)	119 (1)
C(9)-C(8)-C(13)	118.6 (2)	C(16)-C(15)-H(15)	121 (1)
C(8)-C(9)-C(10)	120.6 (2)	C(16)-C(17)-H(17)	122 (1)
C(9)-C(10)-C(11)	119.4 (2)	C(18)-C(17)-H(17)	118 (1)
C(10)-C(11)-C(12)	120.7 (2)	C(17)-C(18)-H(18)	118 (1)
C(11)-C(12)-C(13)	119.3 (2)	C(19)-C(18)-H(18)	120 (1)
C(8)-C(13)-C(12)	120.9 (2)	C(14)-C(19)-H(19)	118 (1)
C(15)-C(14)-C(19)	120.9 (2)	C(18)-C(19)-H(19)	123 (1)
C(14)-C(15)-C(16)	120.2 (2)	C(21)-C(22)-H(22)	121 (1)
C(15)-C(16)-C(17)	118.9 (2)	C(23)-C(22)-H(22)	119 (1)
C(15)-C(16)-C(20)	120.1 (2)	C(22)-C(23)-H(23)	121 (1)
C(17)-C(16)-C(20)	120.9 (2)	C(24)-C(23)-H(23)	120 (1)
C(16)-C(17)-C(18)	120.1 (2)	C(24)-C(25)-H(25)	119 (1)
C(17)-C(18)-C(19)	121.3 (2)	C(26)-C(25)-H(25)	122 (1)
C(14)-C(19)-C(18)	118.5 (2)	C(21)-C(26)-H(26)	118 (1)
C(16)-C(20)-C(21)	118.1 (1)	C(25)-C(26)-H(26)	121 (1)

TABLE V. Intramolecular Non-Bonding Distances (A)

C(2)...C(9)	3.086 (2)	C(10)...C(15)	2.994 (3)
C(2)...C(23)	3.059 (3)	C(15)...C(22)	3.162 (2)

TABLE VI. Intermolecular Distances (A)

O(1)...C(6)a	3.332 (2)	O(3)...C(12)c	3.311 (2)
O(2)...C(12)b	3.231 (2)	C(11)...C(12)b	3.323 (2)
O(2)...C(13)b	3.343 (2)	C(24)...C(24)d	3.369 (3)

Symmetry Operation Codes

a	-1-X,-Y,-Z	c	1-X,1-Y,1-Z
b	-X,1-Y,1-Z	d	1-X,-Y,1-Z

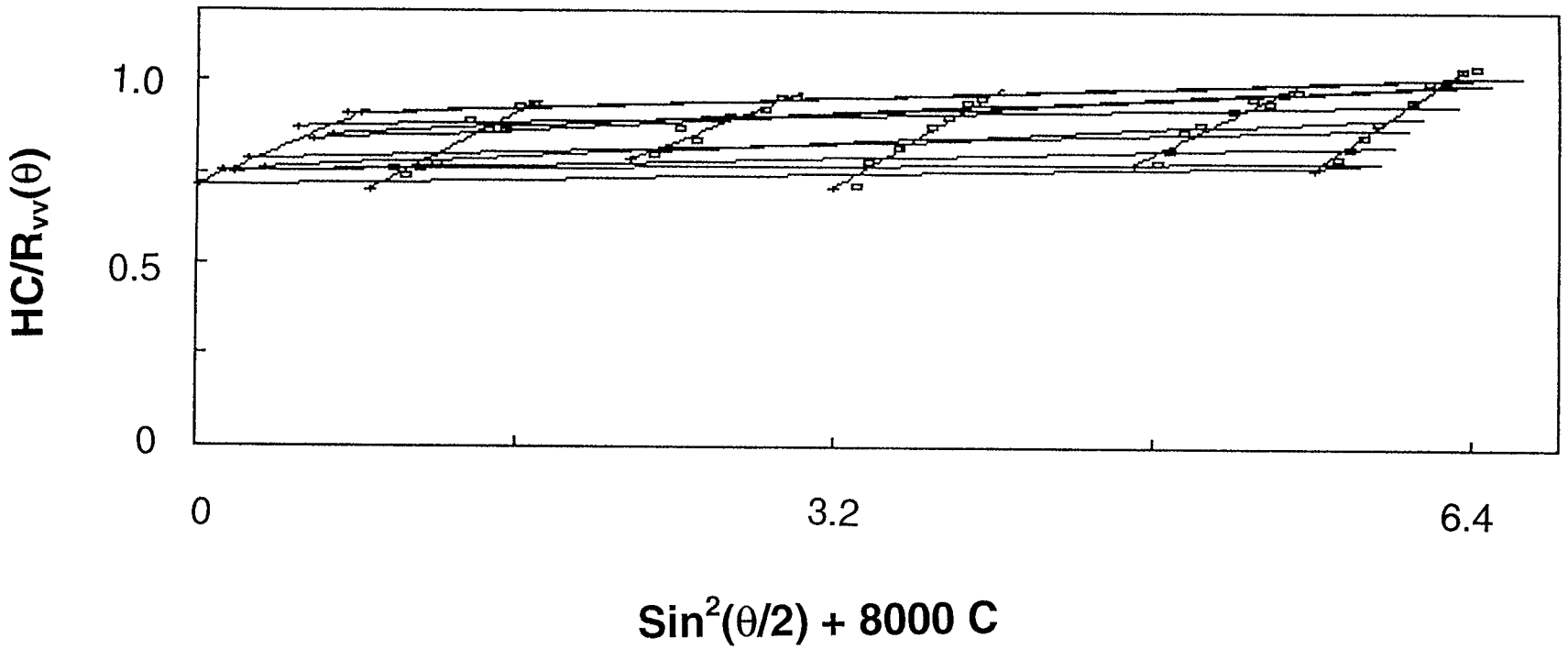


Figure 1: Zimm Plot for IsoPEK (P11227-81-A) in 2-Chlorophenol measured at 23°C. The solutions were dissolved at 135°C for 2 hours. M_w=1.40 (±0.08) x10^5 g/mol, R_g=24.7 nm, and A_2=4.05x10^{-4} cm^3mol/g^2 were obtained.

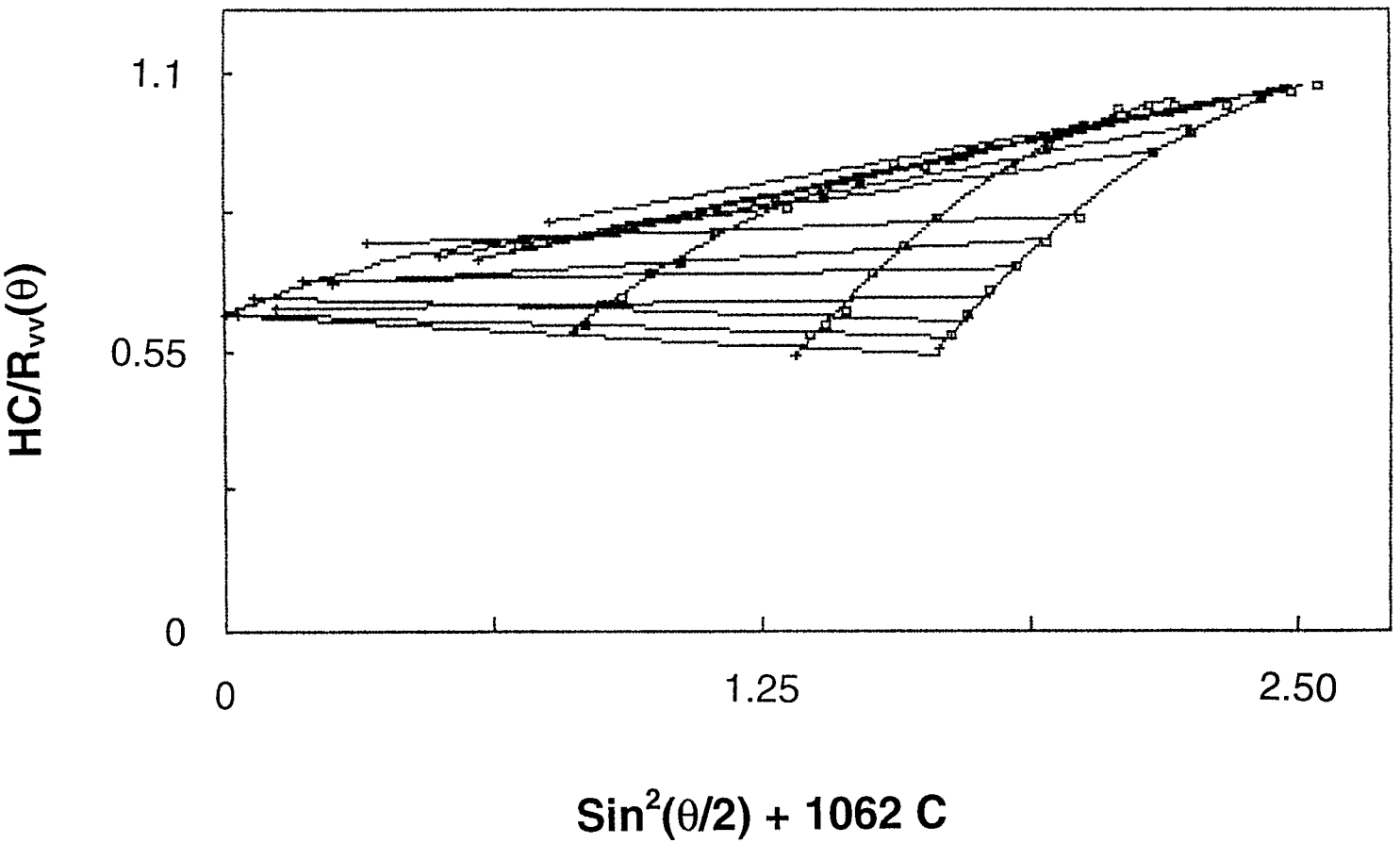
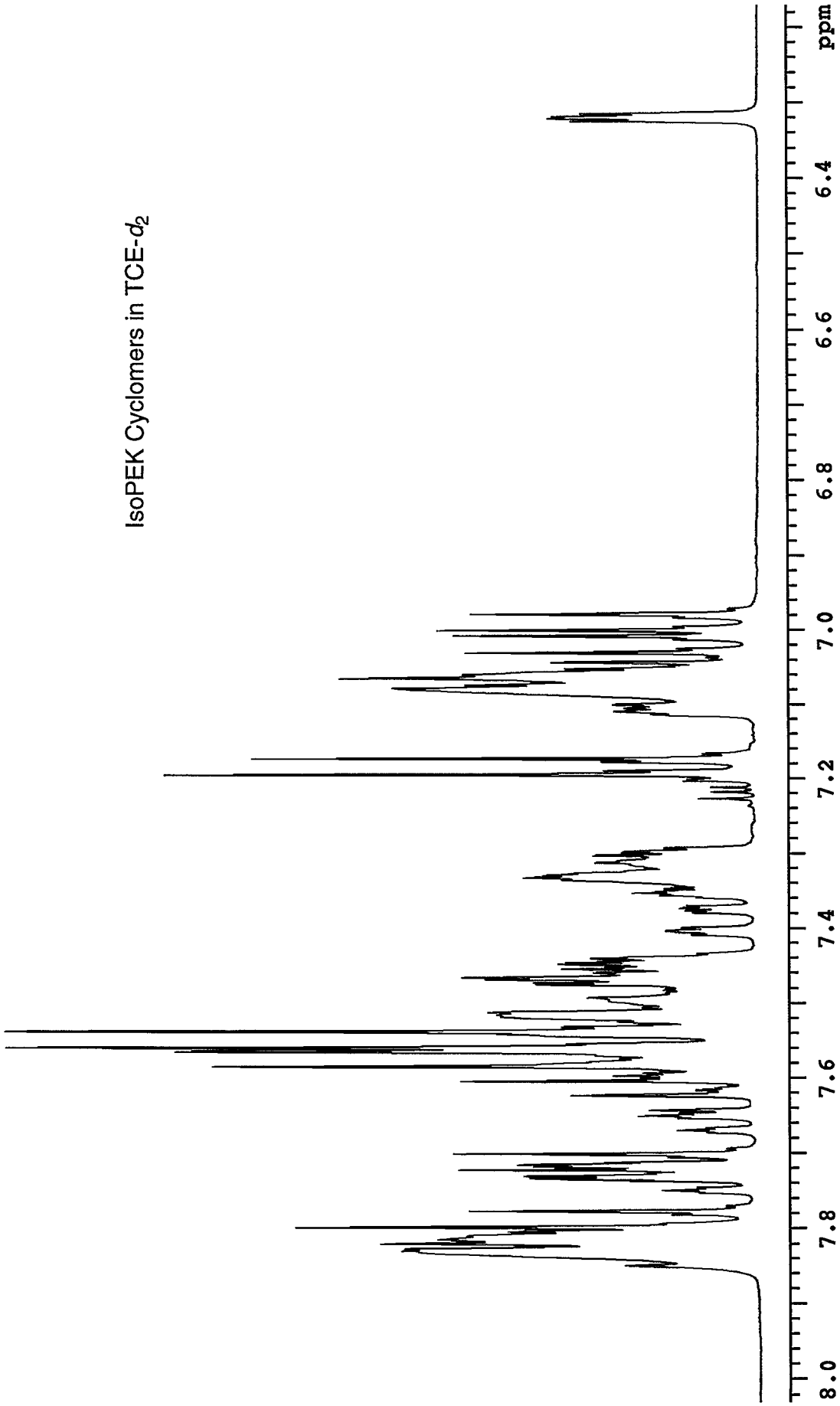


Figure 2. Zimm Plot for IsoPEK (P11227-101b) in 2-Chlorophenol measured at 23°C. The solutions were dissolved at 135°C for 2 hours. M_w=1.60 (±0.10) x10^5 g/mol, R_g=31.9 nm, and A_2=-2.56x10^{-4} cm^3mol/g^2 were obtained.



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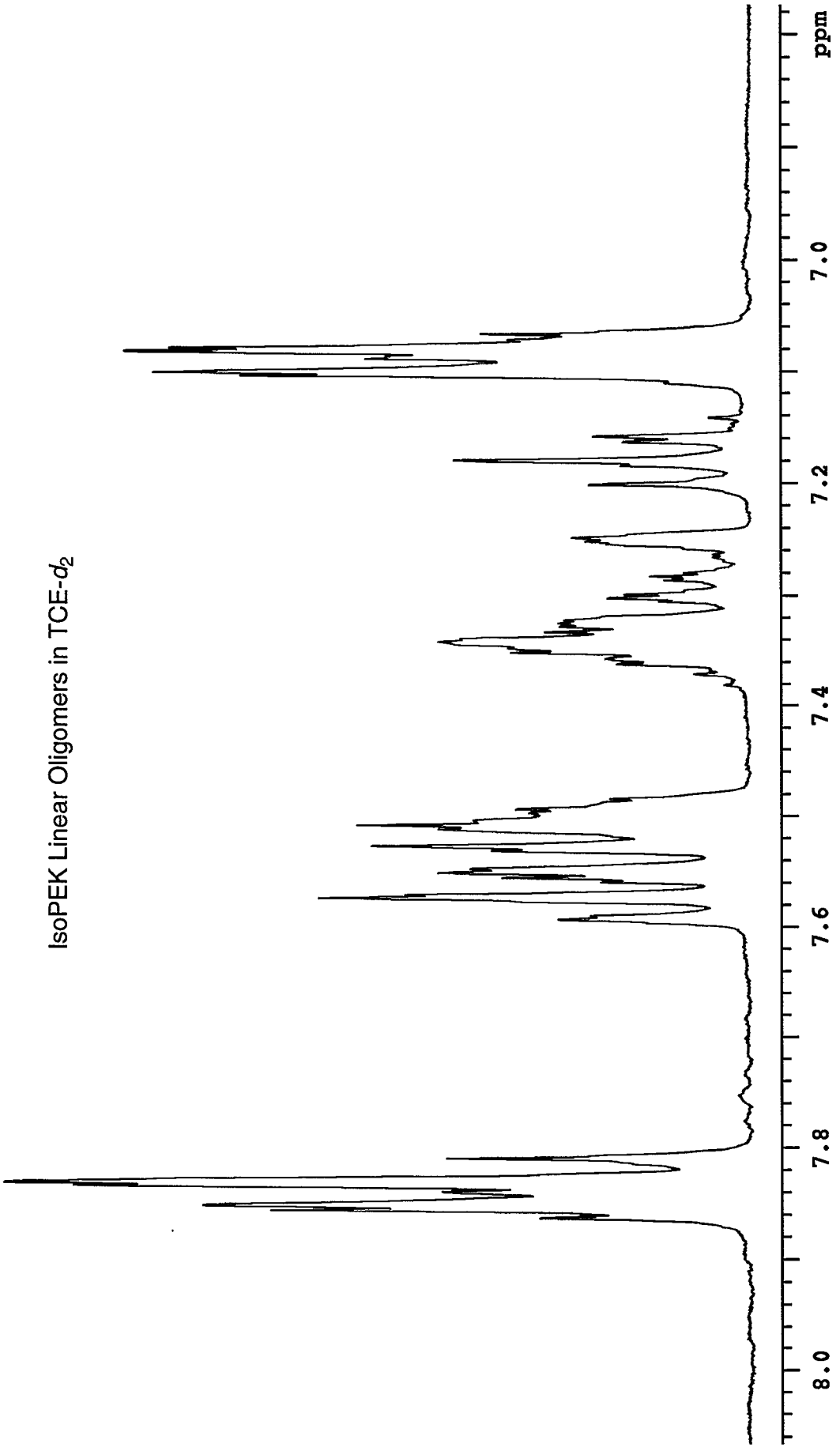

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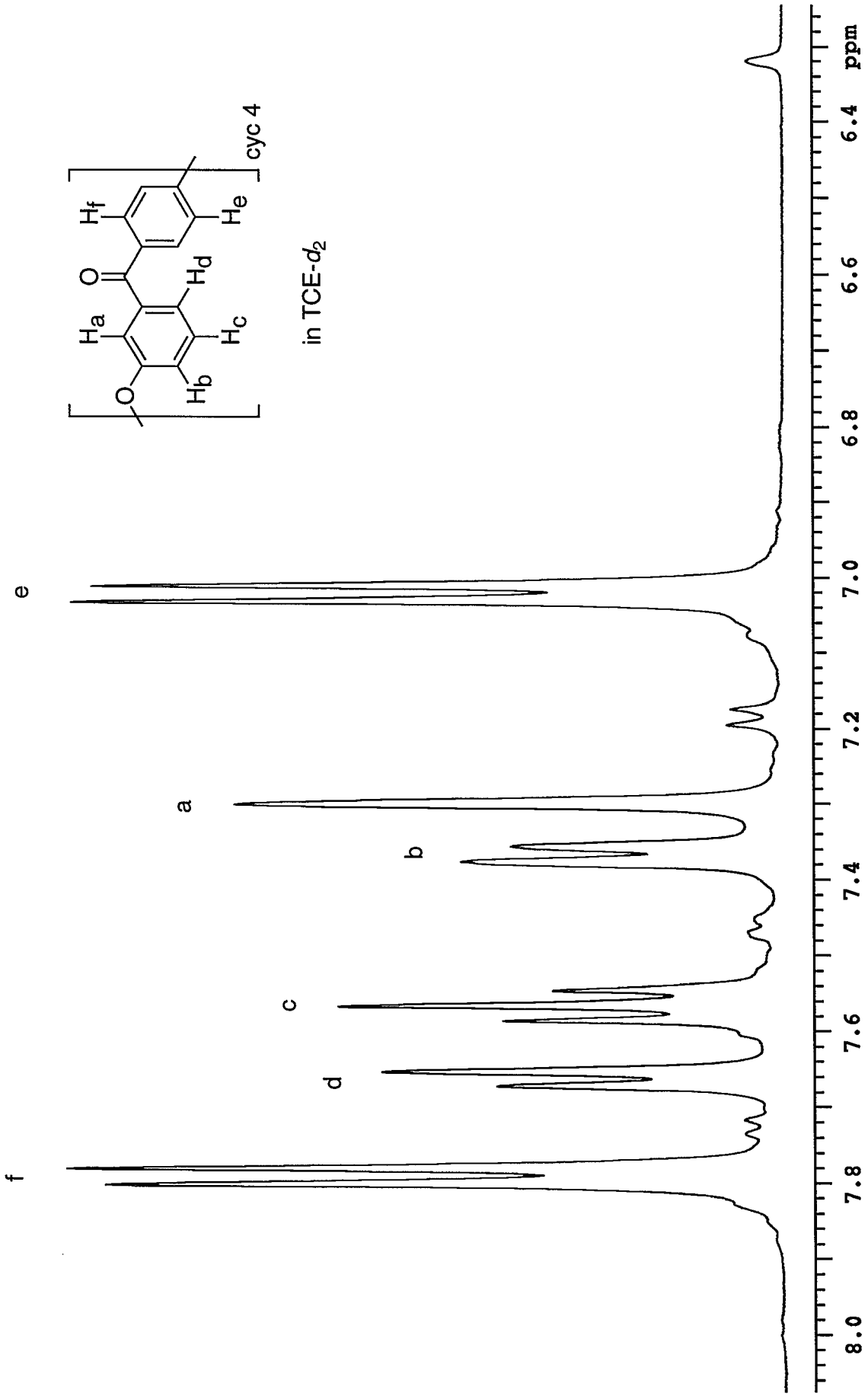
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H_f
H_e
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H_d
H_a
H_c
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ppm