

Solid-State Compounds of Stereoisomers: *cis* and *trans* Isomers of 1,2-Cyclohexanediol and 2,3-Tetralindiol

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Synopsis: The structure of the 1:1 compound of *cis* and *trans*-2,3-tetralindiol, the existence of which has been known for nearly a century, has finally been determined. The likelihood of compound formation between stereoisomers is discussed.

Supplementary Material

1. Ellipsoid plots for enantiomerically pure *trans*-2,3-tetralindiol at 90, 110, and 173 K (1 pg)
 2. Ellipsoid plots for *trans*-1,2-cyclohexanediol at 299 K [*cis*; *trans* (*Pbca*); *trans* (*C2/c*)] and at 293 K (R,R) (1 pg)
 3. Information about the Rietveld refinements (2 pp)

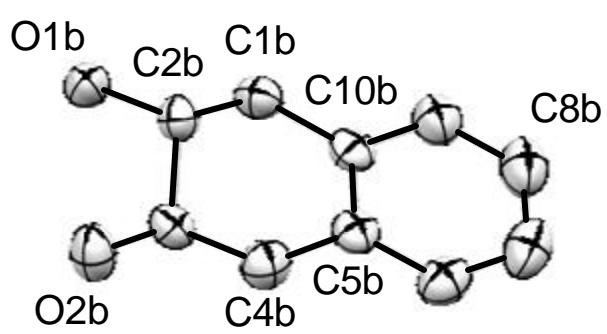
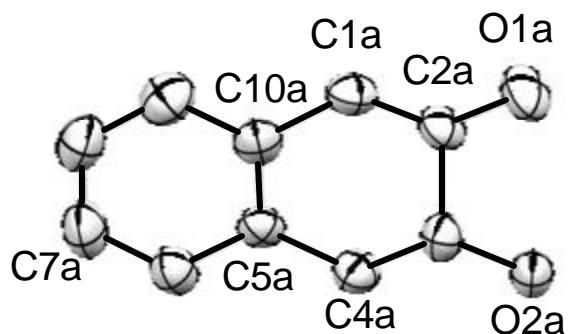
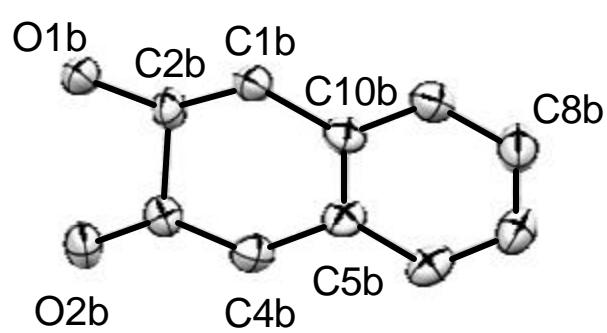
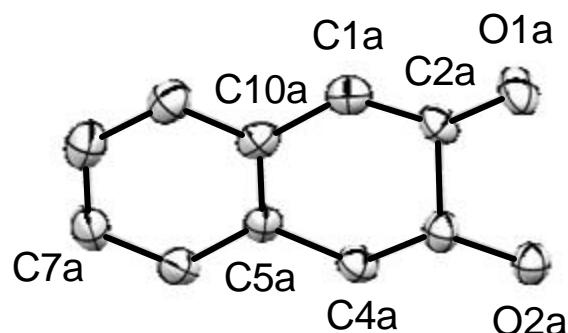
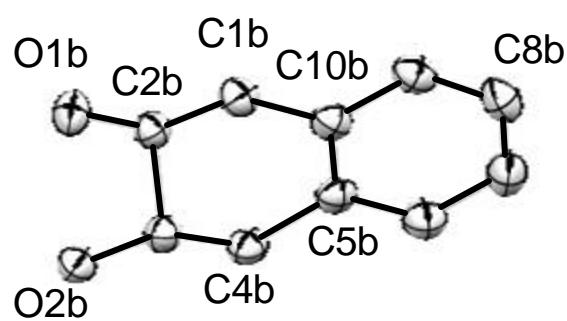
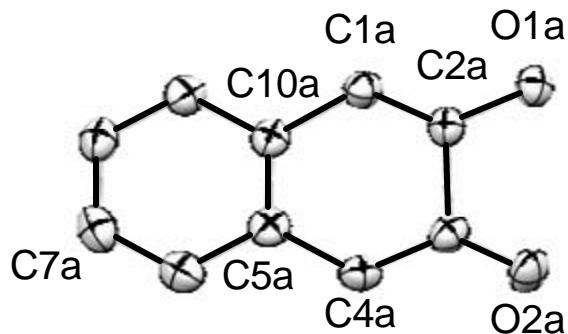
2,3-Tetralindiol

70% ellipsoids

P₂₁ at 90 K (S,S)

P₂₁ at 110 K (R,R)

P₂₁ at 173 K (R,R)



1,2-

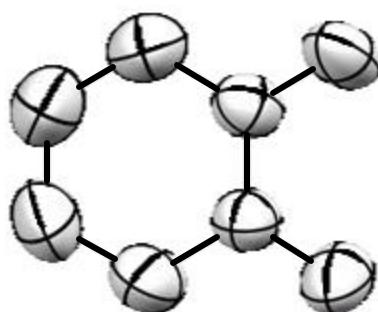
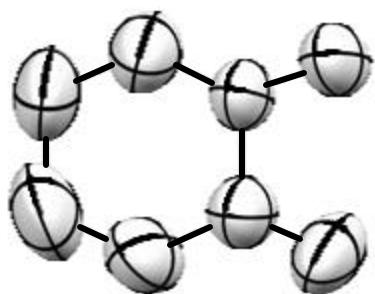
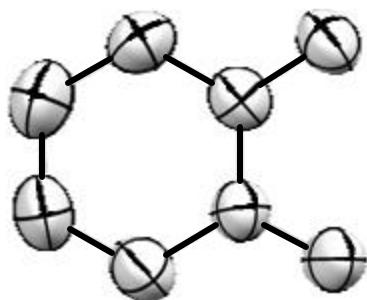
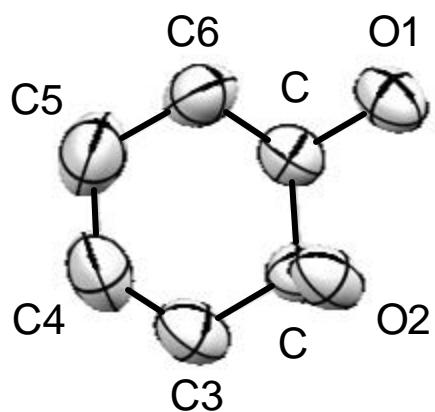
Pbca (*cis*) at 299 K

C2/c at 299 K

70%

Pbca (*trans*) at 299 K

*P3*₂1 at 293 K



Details of the Rietveld refinements used to determine the phases present in samples of 1,2-cyclohexanediol

Sample	<i>cis</i> -1,2-cyclohexanediol final ^a data set	<i>trans</i> -1,2-cyclohexanediol first data set	<i>trans</i> -1,2-cyclohexanediol second data set	<i>trans</i> -1,2-cyclohexanediol final data set	<i>cis</i> - and <i>trans</i> -cyclohexanediol first data set	<i>cis</i> - and <i>trans</i> -cyclohexanediol final data set
Sample Information and Overall Parameters						
Solvent used	as supplied	ethanol	ethanol	as supplied	ethanol	ethanol
μ^b , cm ⁻¹	5.06	5.06	5.06	5.06	5.00	5.00
Displacement ^c	-0.3064(9)	-0.309(1)	-0.2626(14)	-0.3064(13)	-0.4367(19)	-0.1773(12)
Coefficients of Background Polynomial ^d	102(2) -16(2) -61(4) 34(2)	89(1) -40(2) -50(6) 66(6)	235(5) -123(6) -202(6) 248(12)	158(6) -67(9) 4(14) 36(18)	189(3) -135(9) 15(12) 20(5)	320(5) -42(4) -231(11) 123(6)
Phase-Specific Parameters						
Pbca <i>cis</i> -1,2-cyclohexanediol						
Scale fact (*10 ³)	0.546(5)				0.434(15)	0.750(11)
Half-width W ^e	0.117(5)				0.12(2)	0.18(2)
Half-width V	0.03(1)				-0.02(6)	0.11(10)
Mixing (η) ^f	0.16(1)				0.42(7)	0.29(3)
B ^g overall, Å ²	2.8(2)				3.8(4)	4.0(5)
Pbca <i>trans</i> -1,2-cyclohexanediol						
Scale fact (*10 ³)		0.270(5)	0.732(8)	1.21(2)		
Half-width W		0.46(5)	0.32(5)	0.07(5)		
Half-width V		0.045(3)	0.128(8)	0.100(9)		
Mixing (η)		0.42(3)	0.42(2)	0.35(2)		
B _{overall} , Å ²		-1.6(2)	-3.6(2)	3.1(4)		
C2/c <i>trans</i> -1,2-cyclohexanediol						
Scale fact (*10 ³)		0.196(3)	0.326(7)	0.402(11)	0.290(12)	0.780(12)
Half-width W		0.9(2)	0.57(10)	2.7(3)	0.32(12)	0.19(11)
Half-width V		-0.00(3)	0.12(2)	-0.29(4)	0.03(3)	0.17(2)

Mixing (η)		0.64(2)	0.261(5)	0.44(5)	0.34(9)	0.40(3)
B _{overall} , Å ²		3.0(5)	-4.2(5)	-1.3(9)	-0.8(5)	2.3(5)
Goodness of Fit and Results^h						
R _p	10.70	8.08	14.75	12.84	8.15	8.27
R _{wp}	13.65	10.48	20.50	16.25	10.39	11.03
S	2.04	1.40	4.93	3.87	1.56	2.73
Durbin-Watson	0.75	0.11	0.13	0.37	1.11	0.67
% cis Pbca ⁱ	100%				59(2)%	48(1)%
% trans Pbca		57(1)%	69(1)%	74(1)%		
% trans C2/c		43(1)%	31(1)%	26(1)%	41(2)%	52(1)%

^a An additional *cis*-1,2-cyclohexanediol data set was also collected, and also refined satisfactorily as a single phase.

^b Equal to 0.75 x weighted average of single crystal values for early estimate of phase ratios, to account for sample air space, used in preprocessing collected intensities prior to Rietveld analysis. The actual impact of this correction was extremely small.

^c Specimen displacement, compensates for displacement of center of diffraction from center of instrument geometry.

^d Background is modeled as a cubic polynomial of the form $\Sigma c_n (2\theta - 40^\circ)^n$

^e Peak half widths in degrees 2θ are modeled as $H^2 = W + V \tan(\theta)$

^f Pseudo-Voight mixing parameter, controls the Gaussian vs. Lorentzian character of the peak profile model as $\eta L + (1-\eta)G$

^g Thermal displacement parameter applied to the entire phase, in addition to individual atomic displacement parameters derived from single crystal data, on the assumption grinding introduces additional displacements

^h R_p is $100 (\sum |y_i - y_c| / \sum |y_i|)$

R_{wp} is $100 (\sum ((1/y_i)(y_i - y_c)^2) / \sum y_i)^{1/2}$

S is $(\sum (1/y_i)(y_i - y_c)^2 / (N-P))^{1/2}$

ⁱ Weight percentages of each phase derived from scale factors, molecular weights and unit cell volumes by $W_p = S_p(ZMV)_p / \sum S_i(ZMV)_i$