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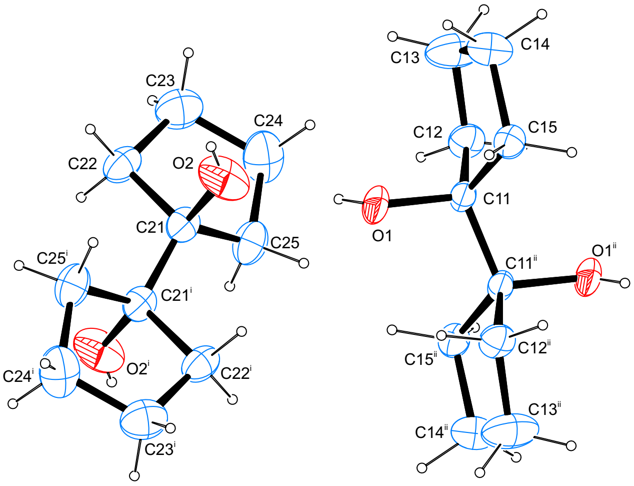
The crystal structure of bi-1,1′-cyclopentane-1,1′-diol, $C_{10}H_{18}O_2$



https://doi.org/10.1515/ncrs-2020-0511
Received October 3, 2020; accepted October 29, 2020;

Abstract
$C_{10}H_{18}O_2$, monoclinic, $C2/c$ (no. 15), a = 10.0025(5) Å, b = 18.8285(9) Å, c = 11.2384(6) Å, β = 115.327(2)°, $V = 1913.11(17)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0596$, $wR_{ref}(F^2) = 0.1606$, $T = 200$ K.

CCDC no.: 2041285

The asymmetric unit of the title structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The compound was synthesized upon pinacolic coupling of cyclopentanedione according to standard procedures [7]. Crystals were obtained upon free evaporation of the extracted solution of the final product.

Table 1: Data collection and handling.

Crystal:	Yellow blocks
Size:	0.59 × 0.49 × 0.41 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	23032, 2394, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2138
$N(param)_{refined}$:	113
Programs:	Bruker [1, 2], SHELX [3], WinGX/ORTEP [4], Mercury [5], PLATON [6]

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.99 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C).

All oxygen-bound H atoms were located on a difference Fourier map and refined freely. Due to their orientational disorder over two positions their site occupancy factors were set to 0.5.

Comment

Chelate ligands have found widespread use in coordination chemistry due to the increased stability of coordination compounds they can form in comparison to monodentate ligands. The stability of these compounds is enhanced further if the denticity of the ligand is increased upon incorporation of more and more potential donor sites [8]. One major factor influencing on the chelation ability of a ligand with vicinal donor sites is the interplay of the spatial pretense on the immediate backbone of the ligand next to the donor sites as well as the size and preferred coordination number and polyhedron of the central atom.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.46914 (13)	0.55853 (7)	0.61658 (13)	0.0381 (3)
H1A[a]	0.512103	0.557668	0.699199	0.046 (12)*
H1B[a]	0.458121	0.598732	0.606709	0.047 (13)*
O2	0.41901 (16)	0.70573 (8)	0.61734 (16)	0.0508 (4)
H2A[a]	0.395496	0.663810	0.591127	0.053 (14)*
H2B[a]	0.452627	0.708597	0.681774	0.041 (14)*
C11	0.55449 (15)	0.51900 (7)	0.56298 (14)	0.0228 (3)
C12	0.66149 (18)	0.56954 (8)	0.54064 (16)	0.0310 (3)
H12A	0.701044	0.547822	0.482052	0.037*
H12B	0.612199	0.614890	0.501522	0.037*
C13	0.7838 (2)	0.58112 (12)	0.6783 (2)	0.0569 (6)
H13A	0.879306	0.589935	0.674139	0.068*
H13B	0.760628	0.622496	0.720504	0.068*
C14	0.7923 (2)	0.51402 (11)	0.75559 (19)	0.0420 (4)
H14A	0.789134	0.525696	0.840243	0.050*
H14B	0.885257	0.488118	0.774205	0.050*
C15	0.65807 (17)	0.46869 (8)	0.66974 (15)	0.0283 (3)
H15A	0.688537	0.428601	0.629862	0.034*
H15B	0.608722	0.449434	0.722662	0.034*
C21	0.33227 (18)	0.75472 (8)	0.51522 (15)	0.0289 (3)
C22	0.3910 (2)	0.82982 (8)	0.55963 (17)	0.0352 (4)
H22A	0.412378	0.837127	0.653302	0.042*
H22B	0.317999	0.865813	0.505795	0.042*
C23	0.5330 (2)	0.83506 (12)	0.5397 (2)	0.0498 (5)
H23A	0.539795	0.882085	0.503315	0.060*
H23B	0.620992	0.828432	0.624457	0.060*
C24	0.5238 (3)	0.77594 (11)	0.4429 (2)	0.0520 (5)
H24A	0.536037	0.795680	0.366524	0.062*
H24B	0.601851	0.739994	0.486402	0.062*
C25	0.3702 (2)	0.74274 (9)	0.39870 (18)	0.0382 (4)
H25A	0.297354	0.766432	0.318532	0.046*
H25B	0.372474	0.691417	0.380487	0.046*

[a] Occupancy: 0.5.

A remarkable example in this context is the situation encountered for spirocyclic phenylarsoranes where the change from pinacol as chelator [9] to perfluoropinacol resulted in a completely changed coordination pattern in the resultant product [10]. In order to understand the rules behind these – sometimes drastic – changes it is advisable to explore the metrical parameters apparent for ligands to be applied in such studies. In continuation of our interest in the structures of diols [11–16] the molecular and crystal structure of the title compound were determined.

The structure solutions shows the presence of two cyclopentane rings joined *via* a single bond. Both bridging carbon atoms bear a hydroxyl group. The asymmetric unit consists of two half molecules. Each molecule is located around an inversion center. The C–C bond between the two central carbon atoms in each molecule was measured at 1.541(3) Å and 1.544(3) Å which is in good agreement with other compounds featuring two connected cycloalkyl moieties [17]. A conformational analysis of the five-membered rings according to Cremer & Pople [18, 19] shows these to adopt a ${}^1T_2$ (${}^{C11}T_{C12}$) conformation in one molecule as well as a ${}^1E$ (${}^{C21}E$) conformation in the second molecule.

In the crystal, classical hydrogen bonds of the O–H···O type are observed that appear in the form of cooperative cyclic D···A sets. In terms of graph-set analysis [20, 21], the descriptor for these contacts is $R_4^4(8)$ on the binary level. In total, the molecules are connected to a three-dimensional network in the crystal.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: National Research Foundation.
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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