Crystal structure of 2,4-bis(4-chlorophenyl)pentane-2,4-diol, $C_{17}H_{18}Cl_2O_2$

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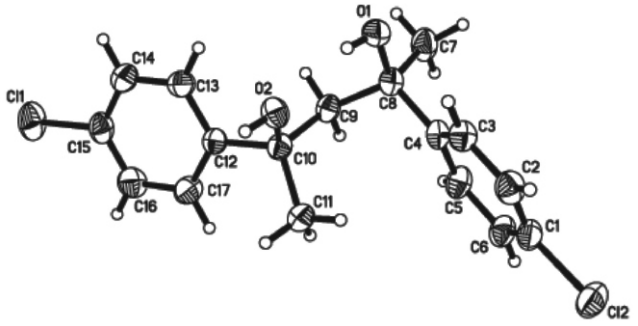

Abstract

$C_{17}H_{18}Cl_2O_2$, monoclinic, *C*12/*c*1 (no. 15), a = 17.9261(7) Å, b = 9.5904(4) Å, c = 19.0663(7) Å, β = 95.694(1)°, V = 3261.7 Å^3, Z = 8, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.103, T = 296 K.

Source of material

The Grignard reagent was prepared from magnesium (1.1 M) and 4-chloro-1-bromobenzene (1.0 M) in THF under protection of N_2. The mixture was cooled to room temperature. Isopropenyl acetate (0.4 M) dissolved in THF was added dropwise to a solution of the above prepared Grignard reagent. After the mixture was stirred under N_2 overnight, the reaction was terminated by the addition of saturated NH_4Cl solution. The organic product was extracted with ether. The combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated in vacuo to give a crude oil. This product was recrystallized and chromatographed on silica gel to afford the title compound. Crystals suitable for X-ray diffraction were grown from a mixture of petroleum and ester (3:1).

Discussion

In the title crystal structure, the angle ∠C8–C9–C10 is 119.5°, the dihedral angle between the two benzene rings is 152.30° and the OH distances of the two hydroxyl groups are 0.820 Å. All bond lengths are within normal ranges. There are potential intramolecular and intermolecular hydrogen bonding interactions (O–H···O). The distances and angles between donor and acceptor are 2.600(2) Å and 143° for O1–H1A···O2, and 2.755(2) Å and 167° for O2–H2A···O1, respectively (symmetry code: ½–x,–½+y,¾–z).

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.50 × 0.50 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.99 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9466, 3165
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2422
$N(param)_{refined}$:	195
Programs:	SHELXS-97, SHELXL-97, SHELXTL [1]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	8*f*	0.2504	0.9166	0.4873	0.073
H(3)	8*f*	0.2442	0.9967	0.6006	0.065
H(5)	8*f*	0.0271	0.8996	0.5867	0.071
H(6)	8*f*	0.0330	0.8177	0.4739	0.075
H(7A)	8*f*	0.0729	1.1729	0.7243	0.105
H(7B)	8*f*	0.0267	1.1076	0.6582	0.105
H(7C)	8*f*	0.0934	1.2093	0.6484	0.105
H(9A)	8*f*	0.0541	0.8738	0.7137	0.060
H(9B)	8*f*	0.0991	0.9510	0.7763	0.060
H(11A)	8*f*	0.1602	0.6981	0.6482	0.089
H(11B)	8*f*	0.0883	0.6438	0.6800	0.089
H(11C)	8*f*	0.1668	0.5795	0.7051	0.089
H(13)	8*f*	0.1885	0.8685	0.8764	0.076
H(14)	8*f*	0.1668	0.7894	0.9866	0.082
H(16)	8*f*	0.0516	0.4689	0.8991	0.082
H(17)	8*f*	0.0734	0.5480	0.7883	0.074
H(1)	8*f*	0.2282	1.0043	0.7226	0.088
H(2A)	8*f*	0.2561	0.7504	0.7661	0.080

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.1424(1)	0.8617(2)	0.46987(9)	0.077(1)	0.057(1)	0.043(1)	0.012(1)	0.0068(9)	0.0061(8)
C(2)	8*f*	0.2052(1)	0.9135(2)	0.5073(1)	0.062(1)	0.065(1)	0.059(1)	0.0047(9)	0.020(1)	0.0055(9)
C(3)	8*f*	0.2013(1)	0.9615(2)	0.5753(1)	0.050(1)	0.059(1)	0.054(1)	–0.0005(8)	0.0059(8)	0.0037(8)
C(4)	8*f*	0.13486(9)	0.9581(2)	0.60622(9)	0.0478(9)	0.053(1)	0.0431(9)	0.0038(7)	0.0036(7)	0.0103(7)
C(5)	8*f*	0.0724(1)	0.9034(2)	0.5669(1)	0.049(1)	0.080(1)	0.049(1)	0.0024(9)	0.0029(8)	0.0072(9)
C(6)	8*f*	0.0754(1)	0.8547(2)	0.4993(1)	0.062(1)	0.073(1)	0.049(1)	0.003(1)	–0.0092(9)	0.0054(9)
C(7)	8*f*	0.0756(1)	1.1374(2)	0.6775(1)	0.084(1)	0.070(1)	0.058(1)	0.024(1)	0.009(1)	0.009(1)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(8)	8*f*	0.12950(9)	1.0137(2)	0.68036(9)	0.0489(9)	0.054(1)	0.0452(9)	0.0030(8)	0.0027(7)	0.0045(7)
C(9)	8*f*	0.10392(9)	0.9042(2)	0.73187(9)	0.0477(9)	0.060(1)	0.0416(9)	0.0034(8)	0.0070(7)	0.0024(8)
C(10)	8*f*	0.15178(9)	0.7726(2)	0.74815(9)	0.0466(9)	0.0520(9)	0.0424(9)	–0.0022(7)	0.0044(7)	0.0014(7)
C(11)	8*f*	0.1408(1)	0.6634(2)	0.68998(9)	0.073(1)	0.060(1)	0.045(1)	–0.0026(9)	0.0055(9)	–0.0028(8)
C(12)	8*f*	0.13400(9)	0.7164(2)	0.81995(9)	0.0500(9)	0.0497(9)	0.0433(9)	–0.0003(8)	0.0061(7)	0.0026(7)
C(13)	8*f*	0.1611(1)	0.7871(2)	0.8804(1)	0.087(1)	0.055(1)	0.049(1)	–0.013(1)	0.010(1)	–0.0006(8)
C(14)	8*f*	0.1485(1)	0.7399(2)	0.9467(1)	0.092(2)	0.068(1)	0.045(1)	–0.004(1)	0.007(1)	–0.0019(9)
C(15)	8*f*	0.1089(1)	0.6204(2)	0.9527(1)	0.067(1)	0.068(1)	0.050(1)	0.010(1)	0.0185(9)	0.0123(9)
C(16)	8*f*	0.0798(1)	0.5490(2)	0.8944(1)	0.072(1)	0.069(1)	0.068(1)	–0.013(1)	0.023(1)	0.009(1)
C(17)	8*f*	0.0928(1)	0.5971(2)	0.8280(1)	0.063(1)	0.068(1)	0.054(1)	–0.015(1)	0.0093(9)	–0.0021(9)
Cl(1)	8*f*	0.09751(4)	0.55548(7)	1.03633(3)	0.1102(5)	0.1062(5)	0.0602(3)	0.0113(4)	0.0325(3)	0.0271(3)
Cl(2)	8*f*	0.14738(4)	0.80551(7)	0.38364(3)	0.1216(5)	0.0881(4)	0.0491(3)	0.0153(4)	0.0123(3)	–0.0045(3)
O(1)	8*f*	0.20029(7)	1.0687(1)	0.70903(7)	0.0635(8)	0.0516(7)	0.0582(8)	–0.0073(6)	–0.0039(6)	0.0024(6)
O(2)	8*f*	0.22887(6)	0.8170(1)	0.75468(7)	0.0460(7)	0.0534(7)	0.0604(8)	0.0026(5)	0.0063(6)	0.0093(6)

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References

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