Crystal structure of 4-bromo-2,6-bis(hydroxymethyl)phenol, $C_8H_9BrO_3$

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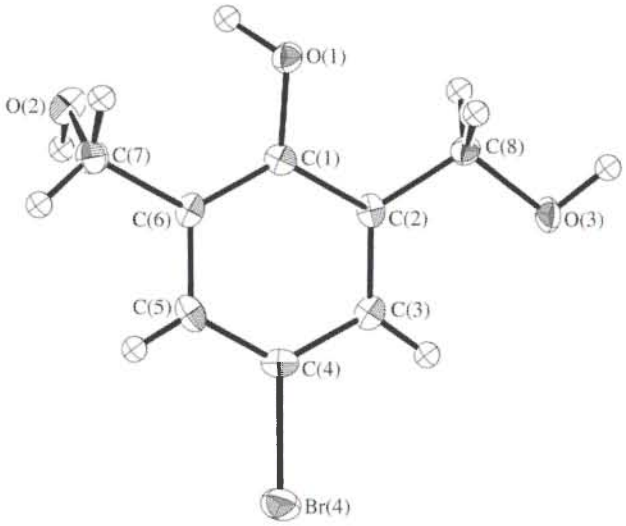

Abstract

$C_8H_9BrO_3$, monoclinic, $P12_1/c1$ (No. 14), a = 7.365(4) Å, b = 14.479(4) Å, c = 8.433(2) Å, β = 112.72(3)°, V = 829.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.029, $wR_{ref}(F)$ = 0.026, T = 173 K.

Source of material

The title compound was prepared from 4-bromophenol and formaldehyde in the presence of sodium hydroxide according to reference [1]. Colorless needles were obtained from recrystallization from ethyl acetate; mp 423 K – 424 K.

Discussion

The mean deviation through the least-squares plane defined by the six aromatic carbons, the bromide, the phenolic oxygen and the two methylene carbons is 0.008 Å with the O(2) and O(3) atoms lying 1.312(2) Å and 0.162(2) Å out of this plane, respectively. All hydroxyl groups are involved in both acceptor and donor hydrogen- bonding interactions. Centrosymmetrically related molecules associate via O(1)–H···O(2)*i* interactions implying that while the two molecules are parallel, they are are not co-planar as the O(2)H group is not co-planar with the aromatic ring as seen in the C1–C6–C7–O2 torsion angle of 69.7(3)°.; d(H···O(2)*i*) = 1.75Å, d(O(1)···O(2)*i*) = 2.663(3) Å and the O(1)–H···O(2)*i* angle is 152°. This association leads to the formation of 12-membered rings. Association with a translationally-related centrosymmetric molecule occurs via O(2)–H···O(3)*ii* interactions such that d(H···O(3)*ii*) = 1.76 Å, d(O(2)···O(3)*ii*) = 2.773(3) Å and ∠O(2)–H···O(3)*ii* = 163°. Sixteen-membered rings result from this pairing of molecules. This also has the result that the two aromatic rings are approximately superimposed and are in close proximity as seen in the average C···C separation of 3.73 Å. Finally, O(3)–H···O(1)*iii* interactions occur: d(H···O(1)*ii*) = 1.78 Å, d(O(3)···O(1)*iii*) = 2.802(3) Å and ∠O(3)–H···O(1)*iii* = 171°. Hence, each molecule is associated with a total of four symmetry- related molecules. The overall structure may be described as being comprised of corrugated layers. Symmetry operations: (*i*) 1–*x*, –*y*, 2–*z*; (*ii*) 1–*x*, –*y*, 1–*z*; and (*iii*) *x*, –0.5–y, –0.5+*z*.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.07 × 0.23 × 0.37 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	49.31 cm^{-1}
Diffractometer, scan mode:	AFC7R, ω/2θ
$2\theta_{max}$:	55.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2125, 1982
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1327
$N(param)_{refined}$:	109
Programs:	teXsan [2], DIRDIF92 [3], DIFABS [4]

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

Atom	Site	x	y	z	U_{iso}
H(1o)	4*e*	0.4543	–0.0870	0.8637	0.023
H(2o)	4*e*	0.5765	0.1339	0.8712	0.023
H(3o)	4*e*	0.3701	–0.2663	0.2643	0.023
H(3)	4*e*	0.2132	–0.0361	0.1959	0.023
H(5)	4*e*	0.1642	0.1584	0.5059	0.023
H(7a)	4*e*	0.2055	0.1334	0.7980	0.023
H(7b)	4*e*	0.2776	0.0290	0.8787	0.023
H(8a)	4*e*	0.4888	–0.1962	0.5286	0.023
H(8b)	4*e*	0.2425	–0.2285	0.4664	0.023

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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}
Br(4)	4*e*	0.05852(4)	0.14483(2)	0.14077(4)	0.0279(2)	0.0279(1)	0.0265(1)	0.0101(2)	0.0102(1)	0.0068(1)
O(1)	4*e*	0.3889(3)	–0.1121(1)	0.7455(2)	0.024(1)	0.017(1)	0.0160(9)	–0.0026(8)	0.0090(8)	–0.0002(7)
O(2)	4*e*	0.4958(3)	0.0893(1)	0.9164(2)	0.019(1)	0.028(1)	0.0192(9)	–0.0104(8)	0.0083(8)	–0.0021(8)
O(3)	4*e*	0.3364(3)	–0.1970(1)	0.2630(2)	0.042(1)	0.015(1)	0.022(1)	0.0012(9)	0.0218(9)	–0.0015(8)
C(1)	4*e*	0.3135(4)	–0.0497(2)	0.6131(3)	0.008(1)	0.018(1)	0.020(1)	–0.001(1)	0.008(1)	0.001(1)
C(2)	4*e*	0.2907(3)	–0.0824(2)	0.4500(3)	0.007(1)	0.016(1)	0.021(1)	–0.004(1)	0.010(1)	–0.002(1)
C(3)	4*e*	0.2140(4)	–0.0240(2)	0.3110(3)	0.009(1)	0.023(1)	0.018(1)	–0.003(1)	0.009(1)	–0.002(1)
C(4)	4*e*	0.1627(4)	0.0654(2)	0.3353(3)	0.010(1)	0.023(1)	0.019(1)	0.000(1)	0.007(1)	0.006(1)
C(5)	4*e*	0.1830(4)	0.0974(2)	0.4953(3)	0.012(1)	0.017(1)	0.030(1)	0.002(1)	0.013(1)	–0.001(1)
C(6)	4*e*	0.2606(3)	0.0398(2)	0.6373(3)	0.007(1)	0.023(1)	0.018(1)	–0.002(1)	0.009(1)	–0.003(1)
C(7)	4*e*	0.2894(4)	0.0768(2)	0.8120(3)	0.018(2)	0.021(1)	0.022(1)	0.004(1)	0.011(1)	–0.001(1)
C(8)	4*e*	0.3502(4)	–0.1804(2)	0.4344(3)	0.021(2)	0.016(1)	0.015(1)	–0.002(1)	0.009(1)	0.001(1)

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