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# Crystal structure of DL- $\alpha$ -(methylaminomethyl) benzyl alcohol, $C_9H_{13}NO$

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#### **Abstract**

 $C_9H_{13}NO$ , orthorhombic, *Pbcn* (no. 60), a = 29.554(4) Å, b = 7.4930(11) Å, c = 8.2800(10) Å, V = 1833.6(4) Å<sup>3</sup>, Z = 8,  $R_{gt}(F) = 0.0737$ ,  $wR_{ref}(F^2) = 0.1646$ , T = 200 K.

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The molecular 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 title compound was obtained commercially (ACROS). Crystals suitable for the diffraction study were obtained upon repeated slow sublimation of the compound at room temperature.

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Table 1: Data collection and handling.

Crystal:	Colourless rod		
Size:	$0.58 \times 0.21 \times 0.15~\text{mm}$		
Wavelength:	Mo <i>K</i> α radiation (0.71073 Å)		
μ:	$0.07 \; \text{mm}^{-1}$		
Diffractometer, scan mode:	Bruker APEX-II, $oldsymbol{arphi}$ and $oldsymbol{\omega}$		
$ heta_{max}$ , completeness:	28.2°, 99%		
$N(hkl)_{\text{measured}}$ , $N(hkl)_{\text{unique}}$ , $R_{\text{int}}$ :	14,634, 2212, 0.038		
Criterion for $I_{obs}$ , $N(hkl)_{gt}$ :	$I_{\rm obs} > 2 \ \sigma(I_{\rm obs}), 1614$		
N(param) <sub>refined</sub> :	106		
Programs:	Bruker [1,2], SHELX [3],		
	WinGX/ORTEP [4], Mercury [5],		
	PLATON [6]		

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters  $(\mathring{A}^2)$ .

Atom	X	Y	z	$U_{\rm iso}*/U_{\rm eq}$
01	0.05354 (5)	0.3666 (2)	0.10111 (19)	0.0433 (4)
H1	0.0511	0.4566	0.0412	0.065*
N1	0.04979 (7)	0.3113 (3)	0.4387 (2)	0.0403 (5)
H71	0.0231 (8)	0.323 (3)	0.376 (3)	0.048 (7)*
C1	0.13570 (7)	0.3290 (3)	0.1002 (3)	0.0378 (5)
C2	0.13387 (10)	0.2038 (4)	-0.0231 (3)	0.0521 (7)
H2	0.1056	0.1526	-0.0524	0.063*
С3	0.17290 (12)	0.1526 (5)	-0.1041 (4)	0.0740 (9)
Н3	0.1713	0.0659	-0.1876	0.089*
C4	0.21366 (11)	0.2264 (5)	-0.0640 (4)	0.0754 (10)
H4	0.2404	0.1904	-0.1190	0.090*
C5	0.21597 (9)	0.3527 (5)	0.0559 (4)	0.0700 (9)
H5	0.2442	0.4058	0.0825	0.084*
C6	0.17713 (8)	0.4028 (4)	0.1380 (3)	0.0530 (7)
H6	0.1791	0.4893	0.2216	0.064*
C7	0.09381 (7)	0.3803 (3)	0.1941 (3)	0.0359 (5)
H7	0.0971	0.5059	0.2331	0.043*
C8	0.08810 (8)	0.2571 (3)	0.3392 (3)	0.0391 (5)
H8A	0.0834	0.1333	0.3011	0.047*
H8B	0.1160	0.2593	0.4051	0.047*
C9	0.04157 (10)	0.1851 (4)	0.5710 (3)	0.0581 (7)
H9A	0.0396	0.0637	0.5274	0.087*
H9B	0.0131	0.2157	0.6252	0.087*
H9C	0.0665	0.1916	0.6487	0.087*

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## **Experimental details**

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms, C–H 0.99 Å for methylene groups, C–H 1.00 Å for the methine group) and were included in the refinement in the riding model approximation, with U(H) set to  $1.2U_{\rm eq}(C)$ .

The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the *SHELX* program suite[2]), with U(H) set to  $1.5U_{\rm eq}(C)$ .

The H atom of the hydroxyl groups was allowed to rotate with a fixed angle around the C–O bond to best fit the experimental electron density (HFIX 147 in the *SHELX* program suite[2]), with U(H) set to  $1.5U_{\rm eq}(O)$ .

The nitrogen-bound H atom was located on a difference Fourier map and refined freely.

## 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 [7]. Aminols are particularly interesting in this aspect as they offer two different donor sites of markedly diverging acidity as potential bonding partners. Upon variation of the substituion pattern on the hydrocarbon backbone, the acidity of the two donor sites can be varied over a wide range and they may serve as probes for establishing the rules in which pKa range coordination to various central atoms of variable Lewis acidity can be observed. Furthermore, the spatial requirements of the substitution pattern can also be exploited to enable unusual coordination numbers. At the beginning of a study about a series of homo- and heteroleptic coordination compounds derived from simple vicinal aminols, the title compound was chosen as it only features one hydrogen atom on the nitrogen atom which we deemed to limit and simplify the coordination behaviour of this ligand and because very similar aminoles have been found to form stable coordination compounds with copper [8–10]. The crystal and molecular structures of similar aminoles have been reported earlier [11–18].

The title compound is the N-methylated derivative of aminomethyl-benzyl alcohol. The C–O and C–N bond lengths are in good agreement with the most-common values found for other aminoles whose geometrical data has been deposited with the Cambridge Structural Database [19]. The two  $\sigma$ -donor sites are present in a nearly perfectly-staggered conformation with the corresponding O–C–C–N angle found at  $-61.2(2)^{\circ}$ . The phenyl ring is orientated perpendicular to the nitrogen-containing side chain with the least-squares planes as defined by the

carbon atoms of the aromatic moiety on the one hand and the non-hydrogen atoms of the  $CH_2$ – $CH_2$ –NH– $CH_3$  chain on the other hand enclosing an angle of 86.32(12)°.

In the crystal structure, classical hydrogen bonds of the O–H···N and N–H···O type are observed. The latter form a cooperative system. In terms of graph-set analysis [20, 21], the descriptor for these interactions is DD at the unary and  $R_4^4(8)$  at the binary level. These hydrogen bonds connect the molecules to double strands that propagate along the crystallographic c axis.  $\pi$ –stacking is not a prominent feature in the crystal structure of the title compound as the shortest distance between two centers of gravity has been measured at 5.3122(19) Å.

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