Crystal structure of 2-methyl-pyridine-*N*-oxide, C_6H_7NO

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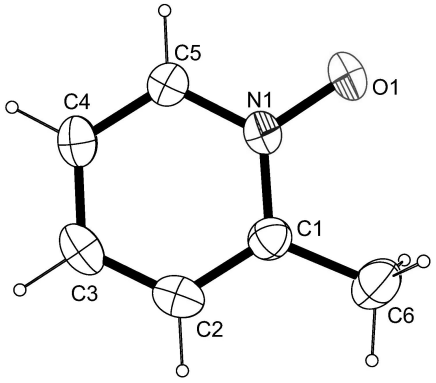

Abstract

C_6H_7NO, orthorhombic, *Pbca* (no. 61), a = 5.7327(6) Å, b = 13.347(1) Å, c = 14.593(2) Å, V = 1116.6 Å^3, Z = 8, $R(F)$ = 0.060, $wR_{ref}(F^2)$ = 0.122, T = 200 K.

Source of material

The compound was obtained commercially (Aldrich). Crystals suitable for the X-ray diffraction study were taken directly from the provided batch and mounted as quickly as possible to account for the low melting point as well as the hydrophilicity of the compound.

Experimental details

Hydrogen atoms bound to aromatic carbon atoms were placed in calculated positions with d(C—H) = 0.95 Å and were included in the refinement in the riding model approximation with U(H) set to 1.2 U_{eq}(C). The hydrogen atoms of the methyl group were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density (HFIX 137 [1]).

Discussion

The coordination chemistry of picolyl oxides is vastly unexplored. To allow for comparative studies of bond lengths and angles in envisioned coordination compounds, we determined the molecular and crystal structure of the title compound. Structural analyses for other pyridine-derived *N*-oxides are apparent in the literature [2,3]. The molecule shows non-crystallographic C_s symmetry. The length of the N—O bond is in good agreement with other aromatic *N*-oxides whose crystal structural data has been deposited with the Cambridge Structural Database [4]. In the crystal structure, C–H···O contacts whose range falls by 0.3 Å below the sum of van-der-Waals radii are present. These stem from the hydrogen atom in *ortho*-position to the intracyclic nitrogen atom and give rise to the formation of centrosymmetric dimers. In terms of graph-set analysis [5,6], the descriptor for these contacts is $R_2^2(8)$ on the unitary level. The lack of further pronounced intermolecular contacts is in good agreement with the low melting point of the title compound. π-Stacking is not a prominent feature in the crystal structure, with the shortest intercentroid distance between two aromatic systems found at 4.921(1) Å.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.404 × 0.409 × 0.484 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.90 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ/ω
$2\theta_{max}$:	55.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8338, 1313
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1034
$N(param)_{refined}$:	74
Programs:	SIR97 [7], SHELXL-97 [1], ORTEP-3 [8], Mercury [9], PLATON [10]

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

Atom	Site	x	y	z	U_{iso}
H(2)	8*c*	0.2934	0.1815	0.1910	0.046
H(3)	8*c*	0.5410	0.3116	0.1491	0.046
H(4)	8*c*	0.8848	0.2741	0.0693	0.044
H(5)	8*c*	0.9744	0.1090	0.0353	0.041
H(6A)	8*c*	0.2847	–0.0514	0.1038	0.067
H(6B)	8*c*	0.1995	0.0041	0.1951	0.067
H(6C)	8*c*	0.4248	–0.0666	0.1977	0.067

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}
O(1)	8*c*	0.7427(3)	–0.04359(8)	0.07185(9)	0.0506(9)	0.0219(5)	0.0640(8)	0.0028(5)	0.0125(7)	–0.0038(5)
N(1)	8*c*	0.6880(3)	0.04874(9)	0.09229(9)	0.0336(8)	0.0237(6)	0.0358(7)	0.0012(6)	0.0008(6)	0.0000(5)
C(1)	8*c*	0.4859(3)	0.0683(1)	0.1378(1)	0.032(1)	0.0343(8)	0.0298(7)	–0.0020(7)	0.0009(6)	–0.0006(6)
C(2)	8*c*	0.4334(3)	0.1669(1)	0.1588(1)	0.036(1)	0.0428(9)	0.0352(8)	0.0075(8)	0.0011(7)	–0.0061(7)

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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(3)	8*c*	0.5791(4)	0.2443(1)	0.1342(1)	0.049(1)	0.0276(7)	0.0396(8)	0.0087(8)	–0.0053(8)	–0.0042(6)
C(4)	8*c*	0.7820(4)	0.2219(1)	0.0874(1)	0.045(1)	0.0258(7)	0.0393(8)	–0.0020(7)	–0.0021(7)	0.0031(6)
C(5)	8*c*	0.8344(3)	0.1242(1)	0.0672(1)	0.0328(9)	0.0304(8)	0.0389(8)	–0.0021(7)	0.0039(7)	0.0013(6)
C(6)	8*c*	0.3360(4)	–0.0188(1)	0.1605(1)	0.043(1)	0.048(1)	0.0440(9)	–0.0126(9)	0.0056(8)	0.0001(8)

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