Jun Yuan*, Bingheng Wu, Yong Wang, Jianlong Wang, Duanlin Cao and Lizhen Chen

The crystal structure of 1-methyl-2-nitro-1*H*-imidazole 3-oxide, C₄H₅N₃O₃

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.18 × 0.15 × 0.11 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.14 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.4°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6195, 1157, 0.035
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1011
$N(\text{param})_{\text{refined}}$:	92
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

All of reagents were purchased with analysis grade. In representative experiments, 1.27 g (mmol) 1-methyl-2-nitroimidazole was dissolved in 10 mL ethanoic acid, and then 2 mL 30% H₂O₂ were added at 293 K. The target compound was obtained by the reaction at 323 K for 3 h. Crystals of the title compound were obtained by slow evaporation at 293 K.

Experimental details

Using Olex2 [2], the structure was solved with the ShelXT [3] structure solution program using Intrinsic Phasing and refined with the ShelXL [4] refinement package. All H atoms were positioned geometrically and treated as riding, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Comment

A series of studies on imidazole-N-oxide began in the 20th century. 1-Hydroxyimidazoles 3-oxides, 1-hydroxy-imidazol-N3-oxide, 1-alkyl(aryl)imidazole-N(3)-oxiden, benzimidazol-N-oxide had been synthesized one after the other [5–9]. For a long time no progress has been made. To the best of our knowledge the title compound is reported for the first time in this paper (cf. the figure).

There is one title molecule in the asymmetric unit. The title molecule is almost planar. All bond lengths and angles in the title molecule (see the figure) are in the expected ranges; this holds especially true for the nitron moiety [10].

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Abstract

C₄H₅N₃O₃, monoclinic, $P2_1/n$ (no. 14), $a = 8.4556(2)$ Å, $b = 7.0609(3)$ Å, $c = 9.5928(4)$ Å, $\beta = 90.12(2)^\circ$, $V = 572.73(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0339$, $wR_{\text{ref}}(F^2) = 0.0914$, $T = 140.0$ 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.

*Corresponding author: Jun Yuan, Shanxi College of Technology, Shuozhou 036000, Shanxi Province, P. R. China, E-mail: 575510418@qq.com. <https://orcid.org/0000-0003-2103-8000>

Bingheng Wu and Yong Wang, North University of China, Taiyuan 030051, Shanxi Province, P. R. China. <https://orcid.org/0000-0003-2992-9908> (Y. Wang)

Jianlong Wang, Duanlin Cao and Lizhen Chen, School of Chemical Engineering and Technology, North University of China, Taiyuan 030051, Shanxi Province, P. R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.46480 (18)	0.3852 (2)	0.16181 (15)	0.0249 (3)
H1A	0.560084	0.341222	0.113672	0.037*
H1B	0.384582	0.421395	0.092910	0.037*
H1C	0.423488	0.283271	0.220726	0.037*
C2	0.60316 (15)	0.56048 (19)	0.36043 (14)	0.0179 (3)
C3	0.43874 (16)	0.7244 (2)	0.23260 (14)	0.0214 (3)
H3	0.365145	0.758892	0.162067	0.026*
C4	0.49569 (16)	0.8415 (2)	0.33346 (14)	0.0215 (3)
H4	0.468880	0.971059	0.345950	0.026*
N1	0.70141 (13)	0.41344 (16)	0.41014 (12)	0.0212 (3)
N2	0.50423 (13)	0.55003 (16)	0.24911 (11)	0.0185 (3)
N3	0.59860 (13)	0.73886 (17)	0.41365 (11)	0.0184 (3)
O1	0.67393 (12)	0.80360 (15)	0.52122 (10)	0.0259 (3)
O2	0.79183 (14)	0.44742 (16)	0.50660 (12)	0.0360 (3)
O3	0.68966 (12)	0.25720 (14)	0.35269 (11)	0.0280 (3)

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References

1. Bruker. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, Wisconsin, USA, 2009.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, 42, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, C71, 3–8.
4. Sheldrick G. M. A short history of SHELX. *Acta Crystallogr.* 2008, A64, 112–122.
5. Wright J. B. The reaction between 2,3-butanedione monoxime and aldehyde oximes. the preparation of 1-hydroxyimidazoles 3-oxides. *J. Org. Chem.* 1964, 29, 1620–1621.
6. Volodarskii L. B., Lysak A. N., Koptug V. A. Synthesis of isomeric pairs of imidazole N-oxides. *Tetrahedron Lett.* 1965, 6, 1565–1568.
7. Volkamer K., Zimmermann H. W. Über arylsubstituierte 1-hydroxyimidazole und 1-hydroxyimidazol-N3-oxide. *Chem. Ber.* 1969, 102, 4177–4187.
8. Lettau H. Imidazol-N-oxide; synthese von 1-alkyl(aryl)imidazol-N(3)-oxiden. *Z. Chem.* 1970, 10, 431–432.
9. Lettau H. Imidazol- und benzimidazol-N-oxide. *Z. Chem.* 1970, 10, 211–216.
10. Laus G., Schwarzler A., Bentivoglio G., Hummel M., Kahlenberg V., Wurst K., Kristeva E., Schutz J., Kopacka H., Kreutz C., Bonn G., Andriyko Y., Nauer G., Schottenberger H. Synthesis and crystal structures of 1-alkoxy-3-alkylimidazolium salts including ionic liquids, 1-alkylimidazole 3-oxides and 1-alkylimidazole perhydrates. *Z. Naturforsch., B: Chem. Sci.* 2008, 63, 447–464.