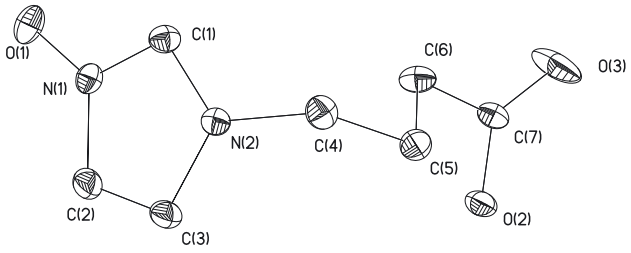
Song Le*, He Dan, Hu Wanghua and Chen LiZhen

The crystal structure of 1-(3-carboxypropyl)-1*H*-imidazole-3-oxide, $C_7H_{10}N_2O_3$



https://doi.org/10.1515/ncrs-2024-0100
Received March 1, 2024; accepted March 27, 2024;
published online April 11, 2024

Abstract

$C_7H_{10}N_2O_3$, monoclinic, *Cc* (no. 9), a = 8.8611(4) Å, b = 13.8991(7) Å, c = 7.7194(5) Å, β = 124.527(1)°, V = 783.27(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0493, $wR_{ref}(F^2)$ = 0.1257, T = 170 K.

CCDC no.: 2343328

The molecular structure is shown in the figure (hydrogen atoms are omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.07 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2664, 1316, 0.058
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1235
$N(param)_{refined}$:	114
Programs:	Olex2 [1], SHELX [2, 3], Bruker [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9469 (3)	0.6337 (2)	0.7812 (4)	0.0338 (7)
O2	0.1219 (4)	0.62114 (17)	0.6234 (4)	0.0339 (7)
N1	0.7800 (4)	0.5947 (2)	0.6450 (4)	0.0239 (6)
N2	0.4960 (3)	0.5796 (2)	0.3886 (4)	0.0218 (6)
O3	0.0543 (6)	0.7741 (2)	0.5381 (6)	0.0643 (11)
C2	0.7301 (5)	0.5017 (3)	0.6492 (6)	0.0311 (8)
H2A	0.806522	0.453029	0.746315	0.037*
C1	0.6374 (5)	0.6412 (2)	0.4857 (5)	0.0237 (7)
H1	0.635804	0.706355	0.447420	0.028*
C3	0.5515 (5)	0.4923 (3)	0.4891 (6)	0.0279 (8)
H3	0.478486	0.436109	0.453218	0.033*
C7	0.1333 (5)	0.7015 (2)	0.5454 (5)	0.0271 (8)
C6	0.2514 (5)	0.6957 (3)	0.4628 (5)	0.0300 (8)
H6A	0.378841	0.680930	0.580463	0.036*
H6B	0.251528	0.758970	0.404084	0.036*
C4	0.3087 (4)	0.6063 (2)	0.2164 (5)	0.0252 (7)
H4A	0.257573	0.555836	0.106612	0.030*
H4B	0.312133	0.667260	0.152359	0.030*
C5	0.1845 (5)	0.6186 (2)	0.2923 (5)	0.0260 (7)
H5A	0.175625	0.556382	0.348249	0.031*
H5B	0.060129	0.635977	0.170940	0.031*
H2	0.062 (6)	0.625 (4)	0.685 (7)	0.049 (15)*

1 Source of material

Add formaldehyde, glyoxal, butyric acid, and hydroxylamine to the reactor. The temperature of the reactor is 298 K. Turn on the mixer at a speed of 600 rpm. The raw materials undergo condensation reaction in the reactor. After the reaction, a white solid is obtained. Dissolve the white solid in deionized water and pour it into an evaporating dish. Place the dish at room temperature to evaporate deionized water to obtain colorless block crystals.

***Corresponding author: Song Le**, School of Energy and Power Engineering, North University of China, Taiyuan 030051, Shanxi Province, P.R. China, E-mail: songle@nuc.edu.cn. https://orcid.org/0000-0003-4211-9280

He Dan and Hu Wanghua, Gansu Yin Guang Chemical Industry Group Co. Ltd., Baiyin 7309004, Gansu Province, P.R. China

Chen LiZhen, School of Chemistry and Chemical Engineering, North University of China, Taiyuan 030051, Shanxi Province, P.R. China. https://orcid.org/0000-0003-3605-4142

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Imidazole compounds have always been a hot research topic in the field of organic chemistry [5–8]. The title compound is obtained by condensation reaction of formaldehyde, glyoxal, butyric acid, and hydroxylamine. The title compound is an imidazole oxide and has high research value as an organic intermediate [9–10].

The asymmetric unit of the title compound is 1-(3-carboxypropyl)-1H-imidazole-3-oxide molecule. The bond length and angle are within the expected range [11]. Almost all non hydrogen atoms in a molecule are in three planes. The seven atoms of O1, N1, C1, C2, N2, C3, and C4 are in the first plane. The four atoms C4, C5, C6, and C7 are in the second plane. The four atoms C6, C7, O2, and O3 are in the third plane. N1, C1, N2, C3, and C2 form imidazole structures similar to those reported in references.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the Natural Science Foundation for Young Scientists of Shanxi Province – China (grant number 202303021212178) and the Center of Testing and Analysis, Shanghai Institute.

References

1. 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.
2. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. BRUKER. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, WI, USA, 2016.
5. Zhongzhen Y., Dongrun T., Haifang W. The crystal structure of 1-(carboxypropyl)-1*H*-imidazole-3-oxide. *Z. Kristallogr. N. Cryst. Struct.* 2024, *239*, 141–142.
6. Xiao L., Hongxia P., Qiang L. The crystal structure of imidazzolium, $C_3H_5O_3N_3$. *Z. Kristallogr. N. Cryst. Struct.* 2022, *237*, 995–996.
7. Cai C., Yumin Y., Zhiwei W. The crystal structure of 5-amino-1-methyl-4-nitroimidazole, $C_4H_6O_2N_4$. *Z. Kristallogr. N. Cryst. Struct.* 2023, *238*, 963–964.
8. Lian P., Zhang L., Su H. A novel energetic cocrystal composed of CL-20 and 1-methyl-2,4,5-trinitroimidazole with high energy and low sensitivity. *Acta Crystallogr.* 2022, *B78*, 133–139.
9. Martinez C. S. The crystal structure of imidazole at–150 °C. *Acta Crystallogr.* 1966, *20*, 783–789.
10. Paliwoda D., Dziubek K. F., Katrusiak A. Imidazole hidden polar phase. *Cryst. Growth Des.* 2012, *12*, 4302–4305.
11. Jun Y., Bingheng W., Yong W., Jianlong W., Duanlin C., Lizhen C. The crystal structure of 1-propyl-2-nitro-imidazole oxide, $C_6H_9N_3O_3$. *Z. Kristallogr. N. Cryst. Struct.* 2023, *238*, 917–918.