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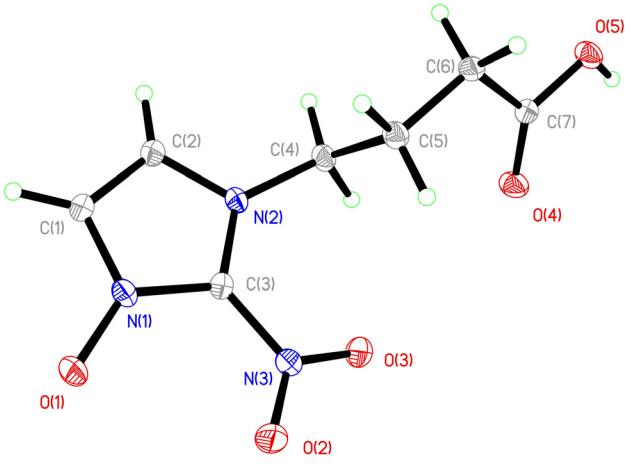
The crystal structure of 3-(3-carboxypropyl)-2-nitro-1*H*-pyrrole 1-oxide, $C_7H_9N_3O_5$



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Abstract

$C_7H_9N_3O_5$, monoclinic, $P2_1/n$ (no. 14), a = 4.7454(3) Å, b = 11.6805(8) Å, c = 15.9284(9) Å, β = 95.257(2) Å V = 879.18(10) Å^3, Z = 4, $R_{gt}(F)$ = 0.0462, $wR_{ref}(F^2)$ = 0.1093, T = 170.0 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

1 Source of material

All reagents were purchased with analysis grade. In representative experiments, 7.05 g hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$), 5.3 g sodium carbonate (Na_2CO_3), 14.5 g glyoxal ($C_2H_2O_2$), 9.7 g formaldehyde (CH_2O), 10.3 g aminobutyric acid ($C_4H_9NO_2$) were added in the ice bath, and reacted at 273 K for 12 h. Then we added 6.3 g nitric acid (HNO_3) and the target compound was obtained by reaction at 273 K for 12 h. The crystal of the title compound was obtained by slow evaporation at 293 K (Table 1).

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.08 × 0.05 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.14 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω scans
θ_{max}, completeness:	26.5°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9,902, 1,802, 0.088
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1,170
$N(param)_{refined}$:	137
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

2 Experimental details

A suitable crystal was selected and on a 'Bruker D8 VENTURE' diffractometer.[1] The crystal was kept at 170 K during data collection. Using Olex2,[2] the structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL[4] refinement package. All H atoms were positioned geometrically and treated as riding, and $U_{iso}(H) = 1.2U_{eq}(C)$.

3 Discussion

Derivatives of imidazole are used in many fields, especially nitroimidazoles and imidazole oxides.[5–7] Many of these compounds have been synthesized and researched, such as 1-alkyl(aryl)imidazol-N(3)-oxiden, 2-nitroimidazole, 5-nitroimidazole.[8–12]

The title compound has been reported for the first time in this paper (cf. the figure). The title structure contains butyric acid and imidazole-oxides. The imidazole ring is flat, and the angle between butyric acid and imidazole-oxides, is about 171.6°. All geometric parameters are in the expected ranges.[11,12]

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