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The crystal structure of 1,2-bis(2,4-dinitro-1*H*-imidazol-1-yl)ethane – acetone (1/1), C₁₁H₁₂N₈O₉

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.11 × 0.04 × 0.03 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.14 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
$\theta_{\max}$, completeness:	26.4°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12,479, 3330, 0.066
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2134
$N(\text{param})_{\text{refined}}$:	255
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

The asymmetric unit of the title crystal 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

All the reagents were purchased with analysis grade. In representative experiments, 5 ml HNO₃ and 15 ml H₂SO₄ were dissolved in the solvent, then 14 g (50 mmol) bis (2-nitroimidazole-1-yl)ethidine were added at 293 K. The target compound was obtained by reaction at 348 K for 9 h. The raw product was dissolved in acetone and crystals of the title compound were obtained by slow evaporation at 293 K.

Experimental details

A suitable crystal was selected and mounted on a 'Bruker APEX-II CCD' diffractometer. Using Olex2 [2], the structure was solved with the XT [3] structure solution program and refined with the ShelXL [4] refinement package. All H atoms were positioned geometrically and treated as riding, with C–H bond 0.95 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Comment

Ethan-1,2-diyl bridged nitrogen-containing ligands are semi-rigid which means that they behave between rigidity

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Abstract

C₁₁H₁₂N₈O₉, monoclinic, $P2_1/c$ (no. 14), $a = 9.2565(11)$ Å, $b = 16.629(2)$ Å, $c = 11.2944(13)$ Å, $\beta = 108.945(4)^\circ$, $V = 1644.3(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0528$, $wR_{\text{ref}}(F^2) = 0.1427$, $T = 170.0$ K.

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	0.6983 (3)	0.51372 (15)	0.1016 (2)	0.0268 (6)
C2	0.6512 (3)	0.39977 (15)	0.1544 (3)	0.0314 (6)
C3	0.6840 (3)	0.44500 (15)	0.2605 (3)	0.0308 (6)
H3	0.685495	0.428009	0.341154	0.037*
C4	0.7554 (3)	0.58932 (15)	0.3110 (2)	0.0296 (6)
H4A	0.842547	0.618021	0.297623	0.036*
H4B	0.788247	0.570005	0.398605	0.036*
C5	0.6225 (3)	0.64739 (15)	0.2904 (3)	0.0301 (6)
H5A	0.660298	0.697807	0.336694	0.036*
H5B	0.580586	0.660766	0.200238	0.036*
C6	0.3621 (3)	0.58913 (15)	0.2531 (2)	0.0297 (6)
H6	0.328995	0.587998	0.164169	0.036*
C7	0.2819 (3)	0.56608 (15)	0.3291 (2)	0.0299 (6)
C8	0.4914 (3)	0.60475 (15)	0.4487 (2)	0.0271 (6)
C9	0.0429 (3)	0.75246 (18)	0.3969 (3)	0.0373 (7)
C10	0.0600 (4)	0.7258 (2)	0.5256 (3)	0.0636 (10)
H10A	0.069499	0.667106	0.530546	0.095*
H10B	−0.029871	0.742354	0.547345	0.095*
H10C	0.151706	0.750290	0.584312	0.095*
C11	−0.0051 (4)	0.83722 (18)	0.3633 (3)	0.0497 (8)
H11A	0.012380	0.851308	0.284810	0.074*
H11B	0.054610	0.873112	0.430117	0.074*
H11C	−0.113905	0.842997	0.352914	0.074*
N1	0.6090 (3)	0.31669 (14)	0.1414 (3)	0.0405 (6)
N2	0.7204 (3)	0.57971 (14)	0.0268 (2)	0.0337 (5)
N3	0.6591 (2)	0.44216 (13)	0.0546 (2)	0.0305 (5)
N4	0.7142 (2)	0.51976 (12)	0.22563 (19)	0.0258 (5)
N5	0.5000 (2)	0.61423 (12)	0.33179 (19)	0.0270 (5)
N6	0.6114 (3)	0.62834 (14)	0.5612 (2)	0.0340 (5)
N7	0.3620 (2)	0.57491 (12)	0.4522 (2)	0.0304 (5)
N8	0.1288 (3)	0.53470 (13)	0.2905 (2)	0.0375 (6)
O1	0.5749 (3)	0.28543 (12)	0.0376 (2)	0.0582 (7)
O2	0.6091 (3)	0.28152 (12)	0.2373 (2)	0.0530 (6)
O3	0.7026 (3)	0.56552 (13)	−0.08231 (18)	0.0535 (6)
O4	0.7541 (2)	0.64538 (12)	0.07616 (19)	0.0455 (5)
O5	0.7266 (2)	0.65834 (13)	0.55052 (19)	0.0477 (6)
O6	0.5908 (3)	0.61556 (16)	0.65989 (19)	0.0592 (7)
O7	0.0634 (3)	0.70678 (14)	0.3203 (2)	0.0568 (6)
O8	0.0801 (2)	0.50680 (12)	0.3710 (2)	0.0497 (6)
O9	0.0565 (2)	0.53832 (12)	0.1785 (2)	0.0467 (6)

and flexibility. Chemists have drawn great attention to the synthesis of bridged diimidazoles, such as methane-bridged ones [5–8].

The asymmetric unit of the title structure contains one target molecule and one acetone molecule (see the Figure). The nitroimidazole rings are almost flat, and the dihedral angle of the two nitroimidazole rings is 46.18°. In general, all bond lengths and angles are in the expected ranges in both molecules [9].

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