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The crystal structure of 1-(2-bromoethane)-4-amine-3,5-dinitropyrazole, $C_5H_6Br_1N_5O_4$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.16 × 0.11 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.51 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
$\theta_{\max}$, completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12,423, 1803, 0.057
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1507
$N(\text{param})_{\text{refined}}$:	136
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Abstract

$C_5H_6Br_1N_5O_4$, orthorhombic, *Pbca* (no. 61), $a = 11.4370(4)$ Å, $b = 6.9921(2)$ Å, $c = 22.8443(9)$ Å, $\beta = 90^\circ$, $V = 1826.83(11)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0357$, $wR_{\text{ref}}(F^2) = 0.1062$, $T = 100$ K.

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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.

Source of material

We added 3.46 g (20 mmol) of 4-amine-3,5-dinitropyrazole to 20 mL of *N,N*-dimethylformamide (DMF), heated to 90 °C and kept stirring to obtain 4-amine-3,5-dinitropyrazole completely soluble. Then 2 mL (23 mmol) 1,2-dibromoethane was added to the reaction solution and reacted for 1 h. After that the reaction solution was added to 60 mL distilled water. The obtained precipitate is a light yellow powder. Chromatographic column separation was used to obtain the title compound. Evaporation of a related solution at room temperature to obtain yellow crystals suitable for single crystal X-ray diffraction.

Experimental details

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

Comment

Polynitrazole compounds can be used in energetic materials [5, 6]. In addition, two nitroazole compounds are connected with a vinyl moiety that generates a large conjugate system and improves the thermal decomposition stability of the compound. Vinyl bridged azole energetic materials can be obtained from 1-(2-bromoethane)-azole compounds by replacing one bromine group of 1,2-dibromoethane by

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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}
Br1	0.40337 (3)	0.42963 (5)	0.59003 (2)	0.02683 (18)
C1	0.6272 (3)	0.7487 (5)	0.71297 (13)	0.0139 (6)
C2	0.7137 (3)	0.8049 (4)	0.67241 (15)	0.0153 (7)
C3	0.6451 (3)	0.8517 (5)	0.62394 (14)	0.0145 (7)
C4	0.4225 (3)	0.8352 (5)	0.60236 (15)	0.0186 (7)
H4A	0.421123	0.962312	0.583262	0.022 [*]
H4B	0.353803	0.827218	0.628587	0.022 [*]
C5	0.4123 (3)	0.6826 (5)	0.55595 (15)	0.0215 (8)
H5A	0.481031	0.689481	0.529654	0.026 [*]
H5B	0.341475	0.706317	0.532146	0.026 [*]
N1	0.8300 (2)	0.8102 (4)	0.67893 (13)	0.0217 (6)
H1A	0.874614	0.847661	0.649718	0.026 [*]
H1B	0.861801	0.776174	0.712422	0.026 [*]
N2	0.6460 (2)	0.6834 (4)	0.77107 (12)	0.0170 (6)
N3	0.6849 (3)	0.9281 (4)	0.57104 (13)	0.0189 (6)
N4	0.5292 (2)	0.8193 (4)	0.63761 (11)	0.0142 (6)
N5	0.5184 (2)	0.7562 (4)	0.69200 (12)	0.0150 (6)
O1	0.7909 (2)	0.9543 (4)	0.56654 (12)	0.0284 (6)
O2	0.6145 (2)	0.9666 (4)	0.53146 (12)	0.0326 (7)
O3	0.7487 (2)	0.6587 (4)	0.78622 (11)	0.0277 (6)
O4	0.5613 (2)	0.6549 (4)	0.80272 (10)	0.0213 (5)

another azole compounds. Its bromine group can be replaced by several nitrazoles (such as imidazole, pyrazole, 1,2,4-triazole) forms vinyl bridged azole energetic materials. Then further oxidation forms vinyl bridged azole energetic materials. In this paper, an important energetic intermediate a was synthesized from LLM-116 [7–10].

There is one molecule in the asymmetric unit of the title structure (see the figure). All bond lengths and angles are in the expected ranges. In detail the pyrazolyl moiety, the two nitro groups as well as the amino group are all in one plane.

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