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The crystal structure of *N,N'*-((nitroazanediy)bis(methylene))diacetamide, $C_6H_{12}O_4N_4$

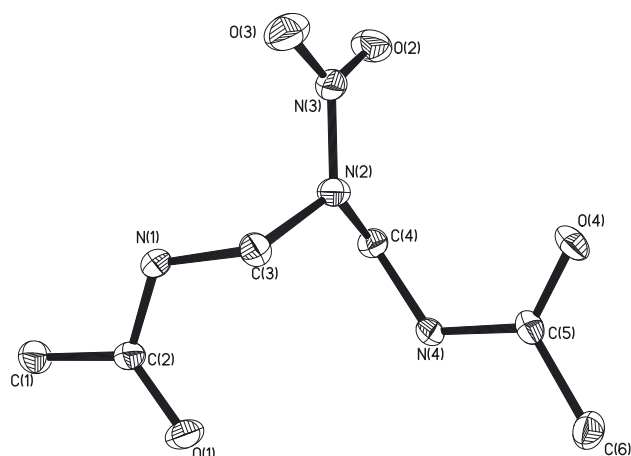


Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.11 × 0.06 × 0.04 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω -scans
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5326, 1725, 0.046
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1607
$N(\text{param})_{\text{refined}}$:	129
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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Abstract

$C_6H_{12}O_4N_4$, monoclinic, *Cc* (no. 9), $a = 9.8171(4)$ Å, $b = 16.1119(6)$ Å, $c = 6.9125(5)$ Å, $\beta = 121.443^\circ$, $V = 932.81(9)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0346$, $wR_{\text{ref}}(F^2) = 0.0894$, $T = 170$ K.

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The 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.

1 Source of material

10 g of 98 % nitric acid and 5 g of ammonium nitrate were added to the reactor. The temperature of the reactor was

lowered to 278 K. Then 1 g of 3,7-diacetyl-1,3,5,7-tetraazabicyclo [3.3.1] nonane (DAPT) was added to the reactor through a small number of times. The temperature of the reactor was raised to 298 K. Reaction lasted for 5 h at 29 K. After the end of the reaction, the reaction solution was diluted with 100 mL ice water. The diluted reaction solution was regulated to neutral by adding NaOH. Evaporate the water using a rotary evaporator and get the white solid. The organic matter in white solids was dissolved using acetone. Filter insoluble impurities and evaporate the filtrate at room temperature to obtain colorless blocky crystals.

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. All the non-hydrogen atoms were refined anisotropically.

3 Discussion

1,3,5,7-Tetranitro-1,3,5,7-tetraazacyclooctane (HMX) is a military explosive with good explosive performance [5]. The 3,7-diacetyl-1,3,5,7-tetraazabicyclo [3.3.1] nonane (DAPT) is an important intermediate product for HMX [6–9]. The title compound was obtained during the process of preparing HMX by nitrating DAPT with nitric acid and ammonium nitrate. The title compound is an organic intermediate with

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.5488 (2)	0.54037 (12)	0.3160 (4)	0.0406 (5)
O4	0.2846 (2)	0.71459 (12)	0.7250 (3)	0.0358 (5)
O2	0.5706 (3)	0.85622 (12)	0.6909 (4)	0.0436 (5)
N4	0.4483 (3)	0.62699 (13)	0.6896 (4)	0.0272 (5)
H4	0.467893	0.575593	0.668143	0.033*
O3	0.4453 (3)	0.86217 (13)	0.3228 (4)	0.0470 (6)
N2	0.4752 (3)	0.74053 (13)	0.4884 (4)	0.0292 (5)
N1	0.5764 (2)	0.67633 (13)	0.2731 (4)	0.0271 (5)
H1	0.632481	0.715427	0.257338	0.033*
N3	0.5003 (3)	0.82384 (13)	0.5026 (4)	0.0315 (5)
C2	0.6236 (3)	0.59689 (16)	0.2923 (4)	0.0280 (6)
C1	0.7699 (3)	0.58043 (18)	0.2829 (5)	0.0359 (7)
H1A	0.846894	0.548935	0.417426	0.054*
H1B	0.817757	0.633262	0.278710	0.054*
H1C	0.740928	0.548285	0.146279	0.054*
C3	0.4359 (3)	0.69903 (16)	0.2778 (4)	0.0282 (6)
H3A	0.373015	0.648435	0.259488	0.034*
H3B	0.368701	0.736316	0.148491	0.034*
C4	0.5515 (3)	0.69248 (16)	0.7017 (4)	0.0291 (6)
H4A	0.653175	0.668569	0.729798	0.035*
H4B	0.576079	0.729793	0.829743	0.035*
C5	0.3218 (3)	0.64297 (17)	0.7105 (4)	0.0278 (6)
C6	0.2305 (4)	0.56889 (19)	0.7123 (5)	0.0376 (7)
H6A	0.173966	0.582966	0.790192	0.056*
H6B	0.304571	0.523026	0.791886	0.056*
H6C	0.153053	0.552187	0.556045	0.056*

research value for studying the reaction mechanism of nitrating DAPT to synthesize HMX.

The asymmetric unit of the title compound is a *N,N'*-(nitroazanediy)bis(methylene)diacetamide molecule. The bond lengths and angles are in the expected ranges [11]. All non hydrogen atoms of the molecule are on three planes. The four atoms of O3, O2, N3 and N2 are on the first plane. The five atoms of C1, C2, O1, N1 and C3 are on the second plane. The five atoms of C4, N4, C5, O4 and C6 are on the last plane. The corresponding bond length and bond angle on two pentaatomic planes are almost identical. The dihedral angle of the plane of O3 O2 N3 N2 and the plane of C1 C2 O1 N1 C3 is 85.6°. The dihedral angle of the plane of O3 O2 N3 N2 and the plane of C4 N4 C5 O4 C6 is 84.2°. According to the above two dihedral angles, two pentaatomic planes are approximately parallel.

During the nitration process of DAPT, the bridge bond breaks. The title compound molecules have undergone structural changes. Firstly, the angle of C3 N2 C4 increases

from 113.2° to 119.2°. Secondly, the bonds of C3 N2 and C4 N2 may have rotated. This causes the two pentaatomic planes, reducing the spatial hindrance between the groups. In the end, the two pentaatomic planes are almost parallel but there is a clear angle in the DAPT molecule [10].

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

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