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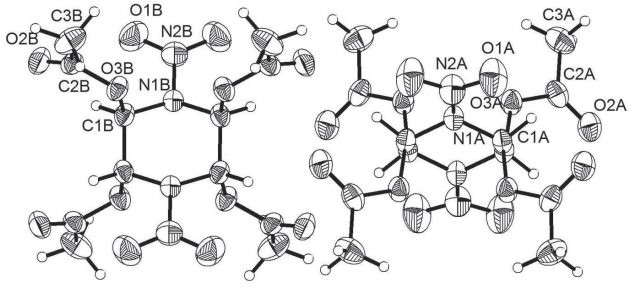
The crystal structure of 1,4-dinitro-2,3,5,6-tetraacetoxy-piperazine, $C_{12}H_{16}N_4O_{12}$



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Abstract

$C_{12}H_{16}N_4O_{12}$, orthorhombic, *Pbam* (no. 55), $a = 16.0836(17)$ Å, $b = 8.0165(7)$ Å, $c = 14.7754(15)$ Å, $V = 1905.1(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0499$, $wR_{ref}(F^2) = 0.1606$, $T = 173$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A glyoxal solution (6.00 mL; 40%) was added to the aqueous solution (15.00 mL) of ammonium sulfamate (12.50 g). The mixture was stirred at low temperatures about 1 day. After filtering and drying, 1.60 g white solid named ammonium hexasulfonate hexaazaisowurtzitane (HSIW) was obtained.

A mixture of HSIW, 98% nitric acid, acetic anhydride, acetic acid and ammonium nitrate was heated at 323 K for 4 h. The mixture was poured into deionized water and filtered at room temperature. The product was dried under vacuum drying oven and a white solid (1,4-dinitro-2,3,5,6-tetraacetoxy-piperazine) was obtained. The colorless transparent crystals were obtained by slow evaporation from a dichloromethane solution in a quiet environment at room temperature.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.17 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	SMART, φ and ω
θ_{max}, completeness:	25.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6208, 1808, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1064
$N(param)_{refined}$:	136
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1A	0.45236(18)	0.5000(4)	0.08493(18)	0.0344(7)
H1A	0.4310	0.4308	0.1362	0.041*
C2A	0.3870(2)	0.7102(4)	0.1742(2)	0.0410(8)
C3A	0.3712(2)	0.8914(4)	0.1779(2)	0.0564(10)
H3AA	0.4087	0.9429	0.2222	0.085*
H3AB	0.3134	0.9114	0.1959	0.085*
H3AC	0.3812	0.9404	0.1181	0.085*
C1B	0.49881(19)	0.5944(3)	0.41559(17)	0.0334(7)
H1B	0.4643	0.6354	0.3639	0.040*
C2B	0.6037(2)	0.7272(4)	0.3263(2)	0.0379(8)
C3B	0.6931(2)	0.7720(4)	0.3263(3)	0.0562(10)
H3BA	0.7024	0.8645	0.3685	0.084*
H3BB	0.7098	0.8059	0.2653	0.084*
H3BC	0.7260	0.6753	0.3453	0.084*
N1A	0.4226(2)	0.4344(4)	0.0000	0.0349(8)
N2A	0.3420(3)	0.3688(5)	0.0000	0.0518(11)
O3A	0.42633(13)	0.6693(2)	0.09543(12)	0.0394(6)
N1B	0.4649(2)	0.6537(4)	0.5000	0.0325(8)
N2B	0.4336(3)	0.8168(5)	0.5000	0.0465(10)
O3B	0.58290(12)	0.6524(2)	0.40608(12)	0.0370(6)
O1A	0.31051(17)	0.3396(4)	0.07333(17)	0.0748(9)
O2A	0.36960(14)	0.6085(3)	0.23149(15)	0.0514(7)
O1B	0.42011(18)	0.8797(3)	0.42660(16)	0.0659(8)
O2B	0.55467(15)	0.7516(3)	0.26650(14)	0.0472(6)

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Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.5\ U_{eq}(C)$ for methyl H atoms and 1.2 $U_{eq}(C)$ for all other H atoms. All the non-hydrogen atoms were refined anisotropically.

Comment

Piperazine is a typical heterocyclic compound, and its derivatives have important applications in the fields of medicines and materials [4–9].

The molecular structure of the title compound (*cf*. the figure) has two nitro groups and four acetoxy groups, which are bonded to two nitrogen atoms and four carbon atoms on the piperazine ring, respectively. All bond lengths and angles are in normal ranges.

From the crystallographic data, it can be found that the molecule of the title structure are centrosymmetric and mirror-symmetric (located around a 2/*m* site for both crystallographically independent molecules (0.5, 0.5, 0; 0.5, 0.5, 0.5; *cf*. the figure)). The molecules are connected *via* non-classical hydrogen bonds and generally by van-der-Waals interactions.

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