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Crystal structure of (((1*E*,1'*E*)-1,2-phenylenebis(methaneylylidene))bis(hydrazin-1-yl-2-ylidene))bis(aminomethaniminium) dinitrate $C_{10}H_{16}N_{10}O_6$

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Abstract

$C_{10}H_{16}N_{10}O_6$, monoclinic, $C2/c$ (no. 15), $a = 12.5131(7)$ Å, $b = 16.4675(9)$ Å, $c = 8.1488(4)$ Å, $\beta = 107.361(5)^\circ$, $V = 1602.64(15)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0380$, $wR_{ref}(F^2) = 0.1180$, $T = 170(1)$ K.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.21 × 0.19 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.13 mm ⁻¹
Diffractometer, scan mode:	XtaLAB AFC10 (RCD3), ω
θ_{max} , completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	6206, 1814, 0.018
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1504
$N(param)_{refined}$:	118
Programs:	CrysAlis ^{PRO} [1], SHELX [2], Olex2 [3]

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

o-Phthalaldehyde (1.34 g, 0.01 mol) was placed in 15 ml water at room temperature, added into aminoguanidine nitrate (2.74 g, 0.02 mol) solution containing 5 ml water and 10 ml ethanol, heated and stirred for 8 h, cooled to room temperature. Then the precipitate was removed, and the filtrate was left standing to precipitate colorless crystals. Hint: high explosives – like the title compound require considerable safety precautions during handling and experimentation.

Experimental details

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

Comment

A Schiff base is a kind of compound containing C=N double bond, which is formed by nucleophilic addition of a

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.43858 (11)	0.08299 (7)	0.39475 (16)	0.0626 (4)
O2	0.29845 (9)	0.14651 (7)	0.22751 (14)	0.0572 (3)
O3	0.41800 (8)	0.21029 (6)	0.43416 (13)	0.0459 (3)
N5	0.38518 (10)	0.14587 (7)	0.35101 (15)	0.0404 (3)
N1	0.31107 (8)	0.41989 (7)	0.46977 (13)	0.0329 (3)
N2	0.27173 (9)	0.34731 (6)	0.39198 (13)	0.0348 (3)
H2	0.305134	0.302214	0.428197	0.042*
N3	0.12392 (10)	0.41776 (8)	0.21309 (16)	0.0469 (3)
H3A	0.063823	0.418853	0.127457	0.056*
H3B	0.147901	0.461448	0.270001	0.056*
N4	0.14428 (10)	0.28163 (7)	0.17214 (15)	0.0456 (3)
H4A	0.084417	0.281227	0.086066	0.055*
H4B	0.181802	0.237589	0.203253	0.055*
C1	0.45083 (11)	0.63784 (8)	0.68171 (15)	0.0340 (3)
H1	0.417279	0.686778	0.637564	0.041*
C2	0.40334 (10)	0.56593 (8)	0.61214 (15)	0.0320 (3)
H2A	0.338501	0.566471	0.519237	0.038*
C3	0.45138 (9)	0.49142 (7)	0.67943 (14)	0.0282 (3)
C4	0.40110 (10)	0.41653 (8)	0.59438 (15)	0.0304 (3)
H4	0.434708	0.366690	0.631107	0.037*
C5	0.17863 (11)	0.34993 (8)	0.25660 (17)	0.0364 (3)

carbonyl group in aldehyde or ketone and an amino group in primary amine. In recent years, Schiff bases and their complexes have been widely used in photochromism [4], catalysis [5], medicine [6], analytical detection [7] and many other fields. There are many kinds of Schiff bases, including single Schiff base, double Schiff base, macrocyclic Schiff base and so on. Symmetrical bis Schiff bases play an important role in coordination chemistry [8] because of their special structure. Symmetrical Schiff base is a good ligand because it has many coordination sites. Therefore, we synthesized the title compound, which has never been reported before.

The asymmetric unit of the title structure contains one half of the organic cation and one nitrate anion (see the

figure). Bond lengths and angles are in the expected ranges.

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