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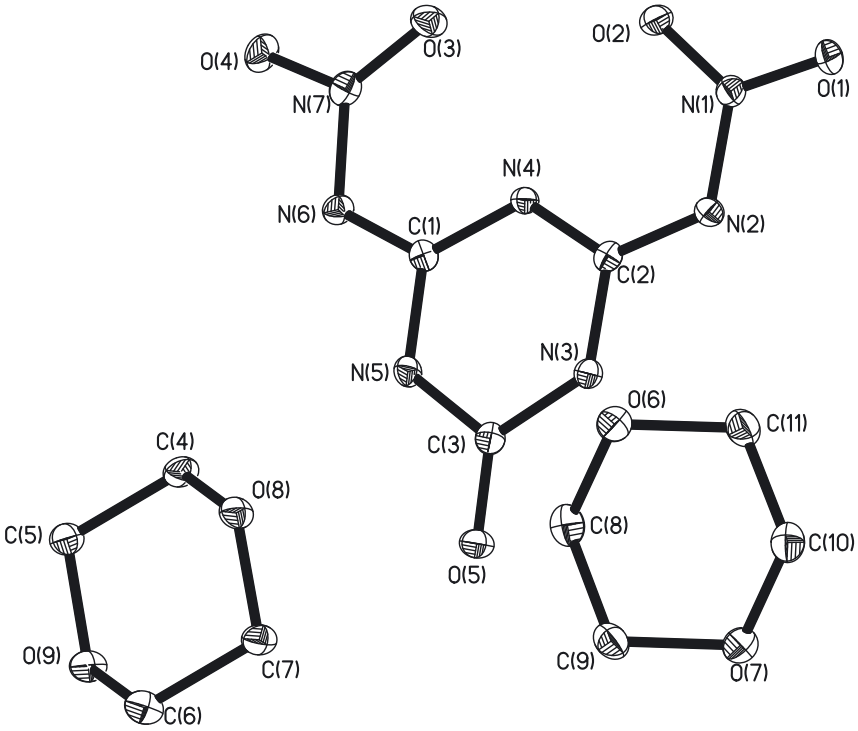
The crystal structure of the co-crystal 1,4-dioxane–4,6-bis(nitroimino)-1,3,5-triazinan-2-one(2/1), $C_{11}H_{19}N_7O_9$



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Abstract

$C_{11}H_{19}N_7O_9$, orthorhombic, $P2_12_12_1$ (no. 19), a = 6.2578(2) Å, b = 15.4188(5) Å, c = 17.6841(7) Å, V = 1706.30(1) Å^3, Z = 4, $R_{gt}(F)$ = 0.0428, w$R_{ref}(F^2)$ = 0.0972, T = 100 K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.11 × 0.06 × 0.04 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,486, 3502, 0.079
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2831
$N(param)_{refined}$:	244
Programs:	Olex2 [1], SHELX [2, 3], Bruker [4]

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

1 Source of material

Two grams of 4,6-dinitroamino-1,3,5-triazin-2-one (DNAM) were dissolved in 40 mL of 1,4-dioxane. A saturated solution

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3145 (5)	0.65177 (19)	0.61137 (16)	0.0174 (7)
C2	0.2544 (5)	0.60105 (18)	0.48542 (16)	0.0172 (7)
C3	0.5839 (5)	0.5649 (2)	0.54905 (16)	0.0196 (7)
C4	0.6510 (6)	0.6040 (2)	0.80989 (17)	0.0242 (8)
H4A	0.564828	0.552031	0.822060	0.029*
H4B	0.554023	0.654731	0.808406	0.029*
C5	0.8167 (6)	0.6174 (2)	0.86962 (17)	0.0244 (8)
H5A	0.894566	0.672070	0.859677	0.029*
H5B	0.746360	0.622447	0.919568	0.029*
C6	1.0647 (6)	0.5349 (2)	0.79828 (15)	0.0247 (8)
H6A	1.161386	0.484118	0.799936	0.030*
H6B	1.150957	0.586726	0.785690	0.030*
C7	0.8969 (6)	0.5212 (2)	0.73849 (17)	0.0236 (8)
H7A	0.966388	0.515003	0.688473	0.028*
H7B	0.817325	0.467054	0.749195	0.028*
C8	0.3845 (6)	0.3971 (2)	0.67253 (19)	0.0305 (8)
H8A	0.287055	0.361780	0.704099	0.037*
H8B	0.457571	0.439267	0.705906	0.037*
C9	0.5475 (6)	0.3391 (2)	0.63607 (19)	0.0292 (8)
H9A	0.650459	0.374711	0.607004	0.035*
H9B	0.627671	0.307401	0.675617	0.035*
C10	0.3197 (6)	0.3238 (2)	0.53139 (18)	0.0293 (8)
H10A	0.244986	0.281476	0.498634	0.035*
H10B	0.414950	0.359175	0.499036	0.035*
C11	0.1587 (6)	0.3816 (2)	0.5687 (2)	0.0306 (8)
H11A	0.075407	0.412756	0.529660	0.037*
H11B	0.058277	0.346011	0.598901	0.037*
N1	−0.0437 (5)	0.63214 (16)	0.41302 (14)	0.0223 (6)
N2	0.1473 (4)	0.58939 (16)	0.42189 (14)	0.0198 (6)
N3	0.4487 (5)	0.56287 (16)	0.48759 (13)	0.0190 (6)
H3	0.491756	0.534826	0.447014	0.023*
N4	0.1917 (4)	0.64440 (16)	0.54822 (13)	0.0174 (6)
H4	0.064973	0.669105	0.548172	0.021*
N5	0.5087 (4)	0.61395 (16)	0.60920 (13)	0.0186 (6)
H5	0.593209	0.620817	0.648519	0.022*
N6	0.2672 (4)	0.69151 (16)	0.67551 (14)	0.0198 (6)
N7	0.0748 (5)	0.73603 (17)	0.68003 (14)	0.0222 (6)
O1	−0.1405 (4)	0.61465 (15)	0.35466 (13)	0.0338 (6)
O2	−0.1137 (4)	0.68593 (13)	0.45958 (11)	0.0243 (5)
O3	−0.0568 (4)	0.73989 (14)	0.62717 (11)	0.0261 (5)
O4	0.0447 (4)	0.77171 (15)	0.74119 (12)	0.0279 (6)
O5	0.7536 (4)	0.52795 (14)	0.55024 (11)	0.0228 (5)
O6	0.2631 (4)	0.44280 (14)	0.61683 (13)	0.0291 (6)
O7	0.4451 (4)	0.27824 (14)	0.58640 (12)	0.0287 (6)
O8	0.7514 (4)	0.59301 (14)	0.73689 (11)	0.0236 (5)
O9	0.9653 (4)	0.54651 (14)	0.87105 (11)	0.0240 (5)

was obtained at a temperature of 333 K. It was cooled to room temperature in a water bath and the precipitated solid was filtered, washed and dried. The result was in the form of needle crystals.

2 Experimental details

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

3 Comment

In recent years, triazine-rich nitrogen compounds have been widely used in the field of energy-containing materials and have become a hot spot for research due to their advantages of high density, good stability and high energy [5]. Among them, 4,6-dinitroamino-1,3,5-triazin-2-one (DNAM) is a typical triazine nitrogen-rich compound with good thermal stability. Through a series of performance tests on DNAM, including density, impact sensitivity, friction sensitivity, vacuum stability and thermal stability properties, the results show that DNAM is expected to be a high-energy, blunt-sensitive monolithic explosive and burn rate modifier. The above properties make 4,6-dinitroamino-1,3,5-triazin-2-one an important application prospect in the field of energy containing materials [6–9].

The 4,6-dinitroamino-1,3,5-triazin-2-one molecule in the crystal structure of the product is mixed up with two dioxane molecules due to the use of dioxane as a solvent. The bond lengths and angles of the title compound were within the normal range compared to expectations. The structure of the title compound is slightly different from the crystal structure of DNAM [10]. The dihedral angle of nitro N7–O4–O3 with the nitrogen-containing six-membered ring decreased from 10.05° to 2.71° and the dihedral angle of nitro N1–O2–O3 with the nitrogen-containing six-membered ring decreased from 10.05° to 9.54°.

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