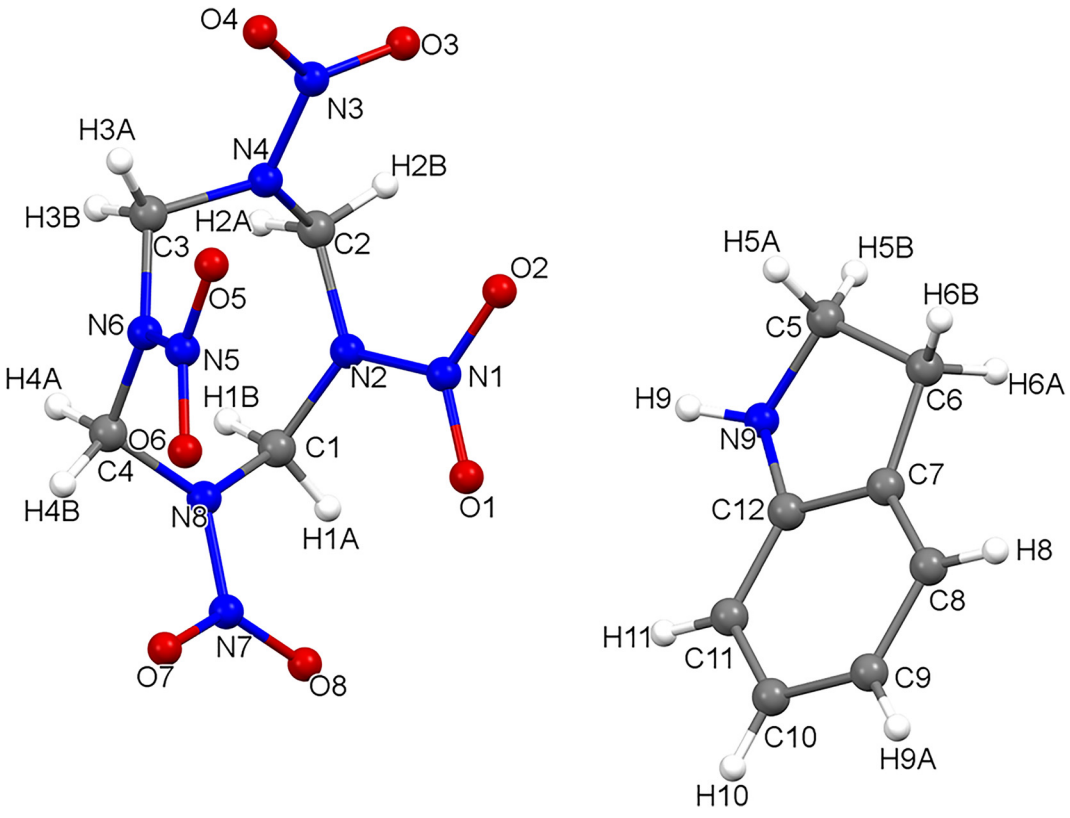
Penghua Liu, Jiangtao Xing*, Zhiqiang Wang and Baomin Wang*

Crystal structure of the cocrystal 1,3,5,7-tetranitro-1,3,5,7-tetrazoctane — 2,3-dihydroindole (1/1), $C_{12}H_{17}N_9O_8$



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Abstract

$C_{12}H_{17}N_9O_8$, orthorhombic, $Pna2_1$ (no. 33), a = 11.376(4) Å, b = 7.966(2) Å, c = 19.076(6) Å, V = 1728.6(9) Å^3, Z = 4, $R_{gt}(F)$ = 0.0373, $wR_{ref}(F^2)$ = 0.0908, T = 296 K.

***Corresponding authors: Jiangtao Xing**, College of Ordnance Engineering, Naval University of Engineering, Wuhan 430000, P. R. China, E-mail: 908000734@qq.com; and **Baomin Wang**, School of Environment and Safety Engineering, North University of China, Taiyuan 030051, P. R. China, E-mail: 1347915616@qq.com
Penghua Liu and Zhiqiang Wang, School of Environment and Safety Engineering, North University of China, Taiyuan 030051, P. R. China.
https://orcid.org/0000-0002-5766-839X (P. Liu)

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

Table 1: Data collection and handling.

Crystal:	Block
Size	0.11 × 0.10 × 0.09 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.14 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8177, 2868, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2608
$N(param)_{refined}$:	262
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.8248 (3)	−0.2308 (4)	−0.47495 (19)	0.0764 (10)
O2	−0.7422 (3)	−0.4766 (5)	−0.4762 (2)	0.0767 (10)
O3	−0.7354 (3)	−0.8300 (4)	−0.53740 (18)	0.0670 (9)
O4	−0.7177 (2)	−0.8092 (3)	−0.65001 (18)	0.0633 (8)
O5	−0.7143 (3)	−0.4438 (4)	−0.6994 (2)	0.0773 (10)
O6	−0.7975 (3)	−0.1991 (4)	−0.6932 (3)	0.0865 (12)
O7	−0.9795 (3)	0.0106 (3)	−0.64065 (19)	0.0695 (9)
O8	−1.0027 (3)	−0.0065 (3)	−0.5281 (2)	0.0815 (11)
N1	−0.8242 (3)	−0.3819 (4)	−0.48659 (17)	0.0514 (8)
N2	−0.9242 (2)	−0.4502 (3)	−0.51380 (14)	0.0345 (6)
N3	−0.7635 (2)	−0.7724 (3)	−0.5946 (2)	0.0447 (7)
N4	−0.8538 (2)	−0.6568 (3)	−0.59558 (16)	0.0343 (5)
N5	−0.7985 (3)	−0.3527 (4)	−0.6893 (2)	0.0524 (8)
N6	−0.9027 (2)	−0.4259 (3)	−0.67167 (14)	0.0345 (6)
N7	−0.9955 (2)	−0.0670 (3)	−0.5864 (2)	0.0506 (8)
N8	−1.0067 (2)	−0.2407 (3)	−0.59208 (18)	0.0414 (6)
C1	−1.0227 (3)	−0.3400 (4)	−0.5289 (2)	0.0386 (8)
H1A	−1.0342	−0.2648	−0.4895	0.046*
H1B	−1.0933	−0.4072	−0.5339	0.046*
C2	−0.9212 (3)	−0.6272 (4)	−0.53184 (18)	0.0355 (7)
H2A	−1.0009	−0.6676	−0.5384	0.043*
H2B	−0.8863	−0.6898	−0.4935	0.043*
C3	−0.9034 (3)	−0.6071 (4)	−0.66258 (17)	0.0340 (7)
H3A	−0.9836	−0.6479	−0.6658	0.041*
H3B	−0.8585	−0.6586	−0.7001	0.041*
C4	−1.0043 (3)	−0.3196 (4)	−0.6605 (2)	0.0385 (8)
H4A	−1.0749	−0.3865	−0.6661	0.046*
H4B	−1.0054	−0.2328	−0.6962	0.046*
N9	−0.6252 (4)	−0.2716 (5)	−0.3394 (2)	0.0743 (11)
H9	−0.6730	−0.3002	−0.3723	0.089*
C5	−0.5291 (7)	−0.3883 (6)	−0.3281 (3)	0.103 (2)
H5A	−0.5060	−0.4413	−0.3718	0.123*
H5B	−0.5513	−0.4747	−0.2948	0.123*
C6	−0.4306 (4)	−0.2821 (7)	−0.2995 (3)	0.0759 (16)
H6A	−0.4278	−0.2890	−0.2487	0.091*
H6B	−0.3555	−0.3181	−0.3183	0.091*
C7	−0.4588 (3)	−0.1082 (4)	−0.32259 (18)	0.0420 (8)
C8	−0.3960 (4)	0.0374 (5)	−0.3219 (2)	0.0570 (10)
H8	−0.3197	0.0405	−0.3045	0.068*
C9	−0.4501 (5)	0.1822 (5)	−0.3482 (2)	0.0672 (13)
H9A	−0.4083	0.2826	−0.3484	0.081*
C10	−0.5630 (5)	0.1802 (5)	−0.3736 (2)	0.0632 (12)
H10	−0.5965	0.2780	−0.3913	0.076*
C11	−0.6265 (4)	0.0332 (5)	−0.3730 (2)	0.0541 (9)
H11	−0.7034	0.0309	−0.3894	0.065*
C12	−0.5742 (3)	−0.1108 (4)	−0.34762 (16)	0.0380 (7)

Source of material

2.960 g (0.01 mol) of raw β-HMX was added to 5 mL of indoline and was stirred at 320 rpm. After 3 h, the powders were filtered and washed with demonized water before collection.

Experimental details

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

Comment

Energetic materials (explosives, propellants and pyrotechnics) are a class of compounds with large amounts of chemical energy that can decompose and release energy in a very short time while producing high temperature and high pressure and large amounts of gas [5, 6]. At the same time, as an important part of material science, it plays an important role in military and civil fields. The compound 1,3,5,7-tetranitro-1,3,5,7-tetrazoctane (HMX) is one of the best elemental explosives with high density (1.905 g·cm^{-3}) and high energy (detonation velocity 9110 m·s^{-1}, detonation pressure 39 GPa). It has been widely used in many fields such as high-power missile warheads, initiators and high-energy rocket propellants [7]. Solvents are important for the synthesis, eutectic modification and recrystallization of energetic materials [8].

As shown in the figure, the crystal structure of the title compound was formed by 1,3,5,7-tetranitro-1,3,5,7-tetrazoctane (HMX) and 2,3-dihydroindole through intermolecular hydrogen bonding force. There are two kinds of intermolecular hydrogen bonding forces, with bond lengths of 2.554 and 2.669 Å, respectively.

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