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Crystal structure of 2,4,7,9-tetranitro-10*H*-benzofuro[3,2-*b*]indole – dimethyl sulfoxide (1/1), $C_{16}H_{11}N_5O_{10}S$

Table 1: Data collection and handling.

Crystal:	Orange plate
Size:	0.10 × 0.10 × 0.05 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	2.21 mm ⁻¹
Diffractometer, scan mode:	XtaLAB Synergy, ω
$\theta_{\max}$, completeness:	74.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	25142, 3718, 0.054
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2863
$N(\text{param})_{\text{refined}}$:	312
Programs:	Rigaku [1], SHELX [2], Materials studio [3]

Source of material

The educt 2,4,7,9-tetranitro-10*H*-benzofuro[3,2-*b*]indole (TNBFI) was provided by Institute of Chemical Materials, China Academy of Engineering Physics. Dimethyl sulphoxide (DMSO) was purchased from Tianjin Kermel Chemical Reagent Co. Ltd. and used without further purification. Four milligram TNBFI was dissolved into 2.0 mL DMSO at 80 °C in a unsealed bottle with stirring for 30 min. The solution was heated at 80 °C continuously until H₂O (0.5 ml) was dripped in the mixed solution. The orange needle crystals of the title compound were obtained when the solution was kept at 25 °C for 24 h.

Experimental details

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

Comment

Energetic materials (EM) including explosives, propellants and pyrotechnics have been widely used in both

<https://doi.org/10.1515/ncrs-2020-0614>

Received November 27, 2020; accepted December 29, 2020;
published online January 25, 2021

Abstract

$C_{16}H_{11}N_5O_{10}S$, triclinic, $P\bar{1}$ (no. 2), $a = 4.6658(1)$ Å, $b = 12.2129(4)$ Å, $c = 17.0355(5)$ Å, $\alpha = 74.925(2)^\circ$, $\beta = 86.398(2)^\circ$, $\gamma = 88.040(2)^\circ$, $V = 935.31(5)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0642$, $wR_{\text{ref}}(F^2) = 0.1744$, $T = 295$ K.

CCDC no.: 2046356

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

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
O1	0.7951 (4)	0.43165 (15)	0.31488 (11)	0.0458 (5)
O2	1.9239 (5)	0.3650 (2)	−0.00069 (15)	0.0753 (7)
O3	1.7680 (7)	0.1949 (2)	0.02861 (18)	0.0950 (9)
O4	1.1784 (6)	0.5880 (2)	0.22642 (18)	0.0910 (9)
O5	1.5082 (6)	0.6371 (2)	0.13238 (16)	0.0829 (8)
O6	0.1325 (6)	0.4180 (2)	0.56342 (16)	0.0798 (7)
O7	−0.1466 (5)	0.2814 (2)	0.56302 (15)	0.0790 (8)
O8	0.2568 (5)	−0.0108 (2)	0.44020 (16)	0.0812 (8)
O9	0.6076 (6)	0.0290 (2)	0.35190 (18)	0.0896 (9)
N1	0.9873 (4)	0.19028 (18)	0.24979 (13)	0.0433 (5)
H1	0.964060	0.119593	0.253252	0.052 [*]
N2	1.7630 (5)	0.2906 (3)	0.03674 (16)	0.0604 (7)
N3	1.3448 (5)	0.5682 (2)	0.17416 (16)	0.0550 (6)
N4	0.0626 (5)	0.3389 (2)	0.53734 (15)	0.0574 (6)
N5	0.4294 (5)	0.0549 (2)	0.39884 (15)	0.0556 (6)
C1	1.1809 (5)	0.2608 (2)	0.19813 (16)	0.0424 (6)
C2	1.3744 (6)	0.2330 (2)	0.14110 (17)	0.0475 (6)
H2	1.384664	0.160430	0.133125	0.057 [*]
C3	1.5505 (6)	0.3174 (3)	0.09687 (16)	0.0479 (7)
C4	1.5421 (6)	0.4281 (3)	0.10644 (17)	0.0493 (7)
H4	1.666469	0.482535	0.075273	0.059 [*]
C5	1.3481 (5)	0.4543 (2)	0.16230 (16)	0.0438 (6)
C6	1.1582 (5)	0.3724 (2)	0.21019 (15)	0.0408 (6)
C7	0.9355 (5)	0.3630 (2)	0.27188 (16)	0.0415 (6)
C8	0.8393 (5)	0.2535 (2)	0.29440 (16)	0.0408 (6)
C9	0.6186 (5)	0.2491 (2)	0.35688 (15)	0.0428 (6)
C10	0.4283 (6)	0.1705 (2)	0.40691 (17)	0.0466 (6)
C11	0.2425 (6)	0.2006 (3)	0.46454 (17)	0.0493 (7)
H11	0.116441	0.148288	0.497343	0.059 [*]
C12	0.2490 (5)	0.3100 (3)	0.47215 (17)	0.0482 (7)
C13	0.4231 (6)	0.3937 (2)	0.42391 (17)	0.0479 (6)
H13	0.418582	0.467522	0.429548	0.057 [*]
C14	0.6032 (5)	0.3602 (2)	0.36705 (16)	0.0436 (6)
S1 ^a	0.7382 (3)	0.93793 (10)	0.19405 (8)	0.0610 (5)
O19 ^a	1.0041 (8)	0.9829 (4)	0.2189 (4)	0.0823 (18)
C15 ^a	0.6550 (19)	0.8132 (6)	0.2664 (6)	0.081 (2)
H15A ^a	0.810146	0.759253	0.267517	0.121 [*]
H15B ^a	0.482868	0.782981	0.252914	0.121 [*]
H15C ^a	0.625679	0.828220	0.318954	0.121 [*]
C16 ^a	0.845 (2)	0.8870 (10)	0.1109 (5)	0.145 (4)
H16A ^a	0.902004	0.949157	0.065755	0.218 [*]
H16B ^a	0.688209	0.848705	0.096359	0.218 [*]
H16C ^a	1.004202	0.834945	0.124268	0.218 [*]
S1 ^b	0.9324 (11)	0.8942 (4)	0.1822 (4)	0.128 (3)
O19 ^b	0.963 (3)	1.0129 (6)	0.1896 (11)	0.0823 (18)
C15 ^b	0.748 (5)	0.805 (2)	0.2640 (16)	0.081 (2)
H15D ^b	0.874217	0.778050	0.307548	0.121 [*]
H15E ^b	0.677240	0.741971	0.247695	0.121 [*]
H15F ^b	0.588888	0.846064	0.282318	0.121 [*]
C16 ^b	0.752 (4)	0.881 (3)	0.0996 (10)	0.145 (4)
H16D ^b	0.553073	0.901817	0.106399	0.218 [*]
H16E ^b	0.766237	0.803951	0.095878	0.218 [*]
H16F ^b	0.835238	0.930149	0.050667	0.218 [*]

^aOccupancy: 0.702(4), ^bOccupancy: 0.298(4).

civilian and military applications [4, 5]. The reactivity and the crystallinity in solvent is significantly important in the synthesis, purification and recrystallization of energetic materials [6, 7]. 2,4,7,9-Tetranitro-10H-benzofuro[3,2-*b*]indole (TNBFI) is an energetic material structurally related to hexanitrostilbene (HNS) and a thermally stable booster explosive. In our previous studies, we have reported the synthesis and solvation of TNBFI from the DMF solution of HNS with 4-(4-pyridyl) morpholine (PMP) and 1,1-bis(morpholino) ethylene (BME) as additives [8]. Here, the reactivity and crystallinity of TNBFI in other solvent such as dimethyl sulphoxide (DMSO) has been studied.

In the crystal structure of the title compound, one TNBFI molecule interacts with one DMSO molecule *via* one hydrogen bond (O⋯H = 1.90 Å; see the figure; the DMSO molecule features a disorder which is omitted in the figure). The TNBFI molecules stack along the *a* axis to form a TNBFI queue, and there are three π - π stacking interactions in the TNBFI queue with the distance of 3.502, 3.557 and 3.566 Å. The 3D supermolecular structure results from the H-bonding and π - π stacking interactions.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The Presidential Foundation of CAEP (Grant No. YZJLX2020004) and National Natural Science Foundation of China (No. 21975227).

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

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Supplementary Material: The online version of this article offers supplementary material (<https://doi.org/10.1515/ncrs-2020-0614>).