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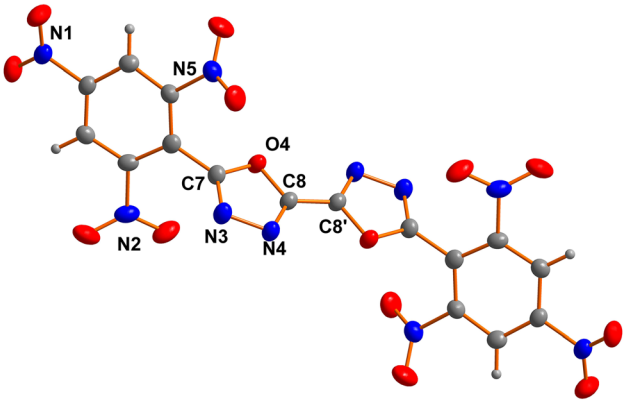
Crystal structure of 5,5′-bis(2,4,6-trinitrophenyl)-2,2′-bi(1,3,4-oxadiazole), $C_{16}H_4N_{10}O_{14}$



https://doi.org/10.1515/ncrs-2024-0399
Received October 9, 2024; accepted November 4, 2024;
published online March 26, 2025

Abstract

$C_{16}H_4N_{10}O_{14}$, monoclinic, $P2_1/c$ (no. 14), a = 7.3407(4) Å, b = 6.0054(3) Å, c = 22.8359(11) Å, β = 93.758(2)°, V = 1004.53(9) Å^3, Z = 2, $R_{gt}(F)$ = 0.0429, w$R_{ref}(F^2)$ = 0.1017, T = 170 K.

CCDC no.: 2387374

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:	Colourless block
Size:	0.16 × 0.12 × 0.08 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.17 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8940, 2036, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1,677
$N(param)_{refined}$:	181
Programs:	Olex2,[1] SHELX[2,3]

1 Source of materials

The nitromethane was purchased from commercial sources and used without further purification, and 5,5′-bis(2,4,6-trinitrophenyl)-2,2′-bis(1,3,4-oxadiazole) was synthesized by our laboratory. The 5,5′-bis(2,4,6-trinitrophenyl)-2,2′-bis(1,3,4-oxadiazole) (0.028 g, 0.05 mmol) was dissolved in nitromethane (15 ml) to obtain a clear colorless solution. After filtration, the obtained solution was slowly evaporated at room temperature for 4 months to give colorless and clear crystals, which were isolated by filtration and dried in air.

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. Crystal data, data collection and structure refinement details are summarized in Table 1. Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

3 Comment

Heat-resistant explosive has the excellent characteristics of high decomposition temperature, stability and reliability in high temperature or high vacuum environment,[4] and can meet the requirements of special military applications, such as aerospace special materials,[5,6] deep sea operations and space exploration.[7–10] With the development of the times, traditional explosives RDX and HMX have been unable to fully meet the current application requirements, and the research and development of high-performance heat-resistant explosives has gradually become a research hotspot in the field of energetic materials. At present, the methods to improve the thermal sensitivity of explosives include: (1) introducing amino group (–NH_2);[11,12] (2) forming a conjugate system;[13] (3) salt formation.[14] In recent years, a series of heat-resistant

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4633 (3)	0.2703 (4)	0.33625 (10)	0.0251 (5)
C2	0.2880 (3)	0.3152 (4)	0.31427 (10)	0.0259 (5)
H2	0.231062	0.229455	0.283258	0.031*
C3	0.1982 (3)	0.4902 (4)	0.33915 (10)	0.0256 (5)
C4	0.2795 (3)	0.6252 (4)	0.38312 (10)	0.0254 (5)
C5	0.4596 (3)	0.5722 (4)	0.40147 (10)	0.0253 (5)
C6	0.5522 (3)	0.3929 (4)	0.38021 (10)	0.0266 (5)
H6	0.672126	0.355839	0.395253	0.032*
C7	0.1784 (3)	0.8099 (4)	0.40875 (10)	0.0256 (5)
C8	0.0371 (3)	0.9804 (4)	0.47180 (10)	0.0259 (5)
N1	0.5573 (3)	0.0768 (3)	0.31244 (9)	0.0317 (5)
N2	0.0051 (3)	0.5206 (3)	0.31926 (10)	0.0335 (5)
N3	0.1186 (3)	0.9894 (3)	0.38361 (8)	0.0307 (5)
N4	0.0234 (3)	1.1039 (3)	0.42567 (8)	0.0297 (4)
N5	0.5627 (3)	0.7170 (3)	0.44411 (9)	0.0318 (5)
O1	0.7236 (2)	0.0715 (3)	0.31911 (9)	0.0511 (5)
O2	0.4623 (3)	−0.0667 (3)	0.28766 (8)	0.0421 (5)
O3	−0.0414 (3)	0.4609 (3)	0.26927 (9)	0.0497 (5)
O4	0.1335 (2)	0.7896 (3)	0.46532 (7)	0.0282 (4)
O5	−0.0972 (2)	0.5994 (3)	0.35414 (9)	0.0449 (5)
O6	0.5326 (3)	0.9168 (3)	0.44097 (8)	0.0448 (5)
O7	0.6749 (3)	0.6294 (3)	0.47835 (9)	0.0503 (5)

compounds have been developed with the continuous efforts of energy containing workers at home and abroad, and have been applied in practice. In the title structure two 1,3,4-oxadiazole bridge two 2,4,6-trinitrophenyl groups to form a conjugated system. As a new type of heat-resistant explosive being insoluble in water, TKX-55 melting point above 350 °C has become a potential candidate for application of heat-resistant explosive due to its excellent performance and stable underwater work ability. At present, the main characterization methods of TKX-55 are: infrared spectroscopy, mass spectrometry, elemental analysis, nuclear magnetism, etc. There are relatively few reports on its crystal structure. The precise structure information of TKX-55 can be obtained by single crystal analysis (see the figure). By studying its spatial configuration, atomic bond length, bond angle and other parameters, the reason of its heat resistance and the mechanism of its poor solubility can be analyzed. Geometric parameters are all in the expected ranges.

Acknowledgments: We are grateful to the support of the Testing and Analysis Center of Huaiyin Normal University.

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

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

Research funding: National Natural Science Foundation of China (22072034).

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