Chen Lizhen, Liu Yunzhang, Wang Jianlong*, Chen Jun and Pan Hongxia

The crystal structure of propan-2-one O-(2,4,6-trinitrophenyl) oxime, C₉H₈N₄O₇

Table 1: Data collection and handling.

Crystal:	Yellow column
Size:	0.26 × 0.13 × 0.06 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.14 mm ⁻¹
Diffractometer, scan mode:	PHOTON 100 CMOS, ω
$\theta_{\max}$, completeness:	26.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	22229, 2358, 0.080
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1881
$N(\text{param})_{\text{refined}}$:	183
Programs:	Bruker [1], SHELX [2, 3]

$V = 2391.5(2) \text{ \AA}^3$, $Z = 8$, $R_{\text{gt}}(F) = 0.0397$, $wR_{\text{ref}}(F^2) = 0.0825$,
 $T = 173(2) \text{ K}$.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Using *N,N*-dimethylformamide as solvent, triethylamine as deacidification agent, ethyl acetohydroxanoate and 2,4,6-trinitrochlorobenzene reacted at 10 °C for 2 h to give 2,4,6-trinitrobenzenesulfonyl acetyl hydroxamate (EPAH), recrystallized from ethanol to obtain EPAH. Then, using 1,4-dioxane as a solvent, EPAH was reacted with perchloric acid at room temperature for 1.5 h, and filtered to obtain a yellow solid. The title compound was isolated from the yellow solid by column chromatography, and then recrystallized from chloroform at room temperature to obtain a yellow crystal suitable for single crystal X-ray diffraction.

Experimental details

All H atoms were positioned geometrically and treated as riding, with C–H bond lengths constrained to 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the hydrogen atoms of benzene ring, C–H bond 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl group.

Comment

3,4,5-trinitro-1H-pyrazole (TNP) is widely studied because it is the only pyrazole compound with full carbon nitrification.

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Abstract

C₉H₈N₄O₇, orthorhombic, *Aea2* (no. 41; old symbol *Aba2*), $a = 21.0595(11) \text{ \AA}$, $b = 14.8565(8) \text{ \AA}$, $c = 7.6437(4) \text{ \AA}$,

*Corresponding author: Wang Jianlong, School of Chemical Engineering and Technology, North University of China, Taiyuan 030051, Shanxi Province, P.R. China, e-mail: 619379961@qq.com

Chen Lizhen and Liu Yunzhang: School of chemical engineering and technology, North University of China, Taiyuan 030051, Shanxi Province, P.R. China

Chen Jun and Pan Hongxia: Hubei Oriental Chemical Industry Co.LTD, China North Industries Group Corporation Limited, Xiangyang 441404, Hubei Province, P.R. China

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.72042(15)	0.03319(19)	0.6271(4)	0.0194(7)
C2	0.71704(13)	−0.05561(18)	0.6892(4)	0.0181(6)
C3	0.77024(14)	−0.10623(19)	0.7243(4)	0.0203(6)
H3	0.7667	−0.1662	0.7661	0.024*
C4	0.82871(14)	−0.0673(2)	0.6970(4)	0.0218(7)
C5	0.83530(15)	0.0201(2)	0.6385(4)	0.0214(7)
H5	0.8761	0.0462	0.6230	0.026*
C6	0.78106(15)	0.06829(19)	0.6034(4)	0.0214(7)
C7	0.57142(15)	0.11391(19)	0.6906(5)	0.0261(7)
C8	0.5560(2)	0.1464(3)	0.5117(6)	0.0462(11)
H8A	0.5903	0.1853	0.4696	0.069*
H8B	0.5162	0.1804	0.5146	0.069*
H8C	0.5514	0.0948	0.4329	0.069*
C9	0.52183(16)	0.1205(3)	0.8297(6)	0.0416(10)
H9A	0.5375	0.0923	0.9373	0.062*
H9B	0.4832	0.0895	0.7909	0.062*
H9C	0.5122	0.1840	0.8523	0.062*
N1	0.62352(12)	0.07988(16)	0.7390(4)	0.0233(6)
N2	0.65497(11)	−0.10040(16)	0.7082(4)	0.0224(6)
N3	0.88636(13)	−0.12163(19)	0.7215(4)	0.0306(7)
N4	0.78921(14)	0.16144(17)	0.5423(4)	0.0263(6)
O1	0.66920(10)	0.08483(14)	0.5932(3)	0.0257(5)
O2	0.61783(10)	−0.09371(15)	0.5866(3)	0.0297(5)
O3	0.64571(10)	−0.14297(15)	0.8419(3)	0.0298(5)
O4	0.87968(12)	−0.20115(16)	0.7571(4)	0.0418(7)
O5	0.93755(10)	−0.08382(17)	0.7043(4)	0.0448(7)
O6	0.83271(12)	0.20450(16)	0.6059(4)	0.0416(6)
O7	0.75232(13)	0.18958(14)	0.4311(4)	0.0374(6)

TNP and its derivatives can be widely used in pesticide, medicine, dye, energy-containing materials and other fields, which has far-reaching research significance and broad development prospects [4–6]. The title compounds obtained in the synthesis of amination reagents derived from ATNP have great effect on the improvement of amination reagents [7, 8]. Here we report the structure of the title compound (the figure).

In the crystal structure of the title compound, 2,4,6-trinitrophenyl substituted for the hydrogen atom on the hydroxyl group of acetone oxime, similar to the structure of EPAH [9]. The dihedral angle formed by the plane of the two nitro groups adjacent to the acetone oxime group and the plane of the benzene ring is larger. However, the dihedral angle formed by the plane of the nitro group that forms the

para position with the acetone oxime group and the plane of the benzene ring is smaller. This may be due to the presence of acetone oxime group caused [10, 11]. Bond lengths and angles are all in the expected ranges [12].

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