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Crystal structure of 3,4-dinitropyrazole, $C_3H_2N_4O_4$

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Abstract

$C_3H_2N_4O_4$, monoclinic, $P2_1/c$ (No. 14), $a = 9.7013(13)$ Å, $b = 12.0797(10)$ Å, $c = 9.7587(7)$ Å, $\beta = 93.962(11)^\circ$, $V = 1140.88(19)$ Å³, $Z = 8$, $R_{gt}(F) = 0.0408$, $wR_{ref}(F^2) = 0.1001$, $T = 105.3$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared by nitrifying pyrazole. It was recrystallized from an ether solution at room temperature to give colorless crystals 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 $U_{iso}(H) = 1.2U_{eq}(C)$, and N–H bond 0.88 Å and $U_{iso}(H) = 1.2U_{eq}(N)$.

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Table 1: Data collection and handling.

Crystal:	Colourless, block size $0.40 \times 0.30 \times 0.15$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	1.7 cm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
$2\theta_{\max}$, completeness:	58.8° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4485, 2220, 0.032
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1813
$N(\text{param})_{\text{refined}}$:	199
Programs:	CrysAlis ^{PRO} [7], SHELX [8], OLEX2 [9]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O8	0.98864(14)	0.04174(11)	0.15219(13)	0.0211(3)
O5	1.34800(14)	0.01273(12)	0.49371(14)	0.0248(3)
N7	1.24991(16)	0.05974(13)	0.53994(16)	0.0181(4)
O6	1.20603(16)	0.04178(13)	0.65237(14)	0.0304(4)
O7	1.10443(15)	−0.05297(11)	0.31190(15)	0.0290(4)
N8	1.06131(17)	0.03449(13)	0.26130(16)	0.0193(4)
N5	1.11548(16)	0.30497(13)	0.39480(16)	0.0185(4)
H5	1.1058	0.3774	0.3945	0.022*
N6	1.19176(17)	0.25006(13)	0.49508(16)	0.0188(4)
C6	1.0569(2)	0.24066(15)	0.29750(19)	0.0170(4)
H6	0.9989	0.2630	0.2200	0.020*
C5	1.09794(19)	0.13433(15)	0.33243(18)	0.0165(4)
C4	1.18020(19)	0.14587(15)	0.45587(19)	0.0161(4)
O1	0.71020(16)	0.18052(12)	0.16963(14)	0.0286(4)
N3	0.74380(16)	0.17825(13)	0.05119(16)	0.0186(4)
O3	0.52217(15)	0.28926(11)	−0.10410(14)	0.0241(3)
O4	0.42190(16)	0.20911(12)	−0.28425(15)	0.0311(4)
N2	0.71922(17)	−0.00932(13)	−0.02898(16)	0.0187(4)
N4	0.50158(17)	0.20899(13)	−0.17954(16)	0.0196(4)
O2	0.82813(15)	0.23929(12)	0.00179(15)	0.0283(4)
N1	0.63877(16)	−0.06168(13)	−0.12936(15)	0.0180(4)
H1	0.6445	−0.1331	−0.1459	0.022*
C2	0.5731(2)	0.10865(15)	−0.14446(18)	0.0167(4)
C3	0.5507(2)	0.00509(15)	−0.20010(19)	0.0179(4)
H3	0.4860	−0.0147	−0.2735	0.022*
C1	0.67850(19)	0.09471(15)	−0.03969(19)	0.0163(4)

Discussion

Polynitropyrazole systems have been investigated extensively because of their biological activity. [1–3]. Recently, these so called "high energy density materials" have attracted renewed attention in conjunction with their favorable detonation performance [4, 5]. As a promising candidate, 3,4-dinitropyrazole was synthesized by the nitration of pyrazole [6]. We here report the crystal structure of the title compound (Fig. 1).

As shown in the figure, 3,4-dinitropyrazole crystallizes with two crystallographic independent molecules in the asymmetric unit. The two pyrazole rings are approximately parallel. Owing to space steric effect, the dihedral angle of the two adjacent nitro groups are 76.5 and 69.4° in the two pyrazole rings, respectively.

All geometric parameters are in the expected ranges.

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