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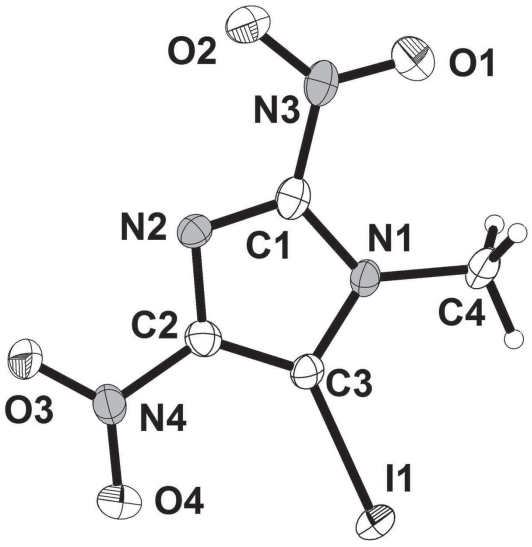
The crystal structure of 1-methyl-2,4-dinitro-5-iodoimidazole, $C_4H_3IN_4O_4$



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Abstract

$C_4H_3IN_4O_4$, orthorhombic, *Pbca* (no. 61), $a = 9.9513(6)$ Å, $b = 11.3365(4)$ Å, $c = 14.3295(12)$ Å, $V = 1616.55(17)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0279$, $wR_{ref}(F^2) = 0.0684$, $T = 105.6$ K.

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

Source of material

The title compound was prepared by nitrifying 1-menthyl-2,4,5-triiodoimidazole. It was recrystallized from acetone solution at room temperature to give colorless crystals suitable for single-crystal X-ray diffraction.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.30 × 0.25 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	39.5 cm^{-1}
Diffractometer, scan mode:	Xcalibur, ω-scans
$2\theta_{max}$, completeness:	52°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4081, 1585, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1465
$N(param)_{refined}$:	119
Programs:	CrysAlis^{PRO} [8], SHELX [9], OLEX2 [10]

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

Atom	x	y	z	U_{iso}*/U_{eq}
I1	0.07674(3)	0.75983(2)	0.287870(18)	0.01716(12)
N1	0.1556(3)	0.9159(3)	0.1234(2)	0.0126(7)
C4	0.2290(4)	0.9996(3)	0.1853(3)	0.0193(9)
H4A	0.1875	1.0778	0.1811	0.029*
H4B	0.3231	1.0046	0.1656	0.029*
H4C	0.2247	0.9715	0.2499	0.029*
N4	−0.0396(3)	0.6637(3)	0.0563(2)	0.0156(7)
C3	0.0916(4)	0.8142(3)	0.1503(3)	0.0121(8)
C2	0.0405(4)	0.7684(3)	0.0682(3)	0.0140(8)
O4	−0.0608(3)	0.6024(3)	0.1253(2)	0.0261(7)
O3	−0.0825(3)	0.6424(3)	−0.02254(19)	0.0208(7)
O2	0.1504(3)	1.0180(2)	−0.11046(19)	0.0180(6)
O1	0.2522(3)	1.0967(2)	0.0084(2)	0.0214(6)
N2	0.0690(3)	0.8360(3)	−0.0068(2)	0.0120(7)
N3	0.1866(3)	1.0214(3)	−0.0291(2)	0.0152(5)
C1	0.1378(4)	0.9227(3)	0.0290(3)	0.0142(6)

Experimental details

All H atoms were positioned geometrically and treated as riding, with C—H bond lengths constrained to 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$. The diff. electron densities of 1.571 and -0.840 are caused by a partial disorder of I/NO_2.

Discussion

Polynitroimidazole systems have been used as antifungal, antibacterial, antiviral, antitumor drugs and their intermediates [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, 1-methyl-2,4,5-trinitroimidazole was synthesized by the nitration of 1-methyl-2,4,5-triiodoimidazole [6, 7]. The result showed two iodine atoms were substituted by nitro groups, while the position of another iodine could not been confirmed. In order to determine the molecular structure, the single crystal was cultured and X-ray diffraction experiment is completed.

As shown in the figure, the asymmetric unit of the title compound consists of one molecule. The imidazole ring is almost planar, the two nitro groups are twisted with the imidazole planar, making dihedral angles of 6.0 (N3/O1,O2) and 3.6 (N4/O3,O4)°, respectively. All bond lengths and angles are in the expected ranges.

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