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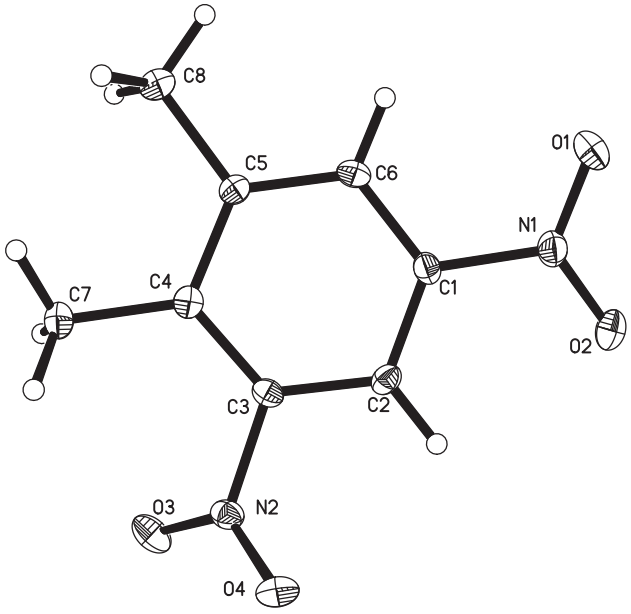
Crystal structure of 1,2-dimethyl-3,5-dinitrobenzene, $C_8H_8N_2O_4$



https://doi.org/10.1515/ncrs-2018-0093
Received March 15, 2018; accepted June 19, 2018; available online June 30, 2018

Abstract

$C_8H_8N_2O_4$, monoclinic, $P2_1/n$ (no. 14), $a = 14.6418(13)$ Å, $b = 3.8852(4)$ Å, $c = 15.7884(15)$ Å, $\beta = 108.417(3)°$, $V = 852.14(14)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0505$, $wR_{ref}(F^2) = 0.1159$, $T = 173(2)$ K.

CCDC no.: 1850364

The asymmetric unit of the 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.

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	$0.37 \times 0.20 \times 0.07$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	Photon 100 Detector, ω
θ_{max}, completeness:	25.4°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4688, 1556, 0.059
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1059
$N(param)_{refined}$:	129
Programs:	Bruker [5], SHELX [6], Olex2 [7]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.89018(17)	0.5728(7)	0.59829(16)	0.0210(6)
C2	0.82359(17)	0.4472(7)	0.63513(15)	0.0209(6)
H2	0.8403	0.3934	0.6969	0.025*
C3	0.73106(17)	0.4029(6)	0.57781(16)	0.0196(6)
C4	0.70172(17)	0.4776(6)	0.48635(16)	0.0195(6)
C5	0.77280(17)	0.6066(6)	0.45200(16)	0.0203(6)
C6	0.86628(17)	0.6550(7)	0.50862(16)	0.0219(6)
H6	0.9140	0.7449	0.4856	0.026*
C7	0.59891(17)	0.4388(7)	0.42663(17)	0.0266(6)
H7A	0.5870	0.1985	0.4074	0.040*
H7B	0.5880	0.5873	0.3742	0.040*
H7C	0.5550	0.5054	0.4595	0.040*
C8	0.74948(19)	0.6960(8)	0.35423(17)	0.0303(7)
H8A	0.8080	0.7751	0.3427	0.045*
H8B	0.7009	0.8789	0.3387	0.045*
H8C	0.7244	0.4917	0.3179	0.045*
N1	0.98979(15)	0.6254(6)	0.65640(15)	0.0290(6)
N2	0.66063(15)	0.2775(6)	0.62040(14)	0.0235(5)
O1	1.04325(13)	0.7993(6)	0.62793(13)	0.0436(6)
O2	1.01401(14)	0.4903(6)	0.73024(13)	0.0450(6)
O3	0.60282(13)	0.0551(5)	0.58169(14)	0.0358(5)
O4	0.66485(13)	0.4010(6)	0.69287(12)	0.0367(5)

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Source of material

The title compound was prepared in accordance with literature [1]. The raw product was purified by column chromatography and recrystallized from ethyl acetate solution at room temperature to give colorless plate crystals suitable for single-crystal X-ray diffraction.

Experimental details

H atoms were positioned geometrically with C—H = 0.95 and 0.98 Å for aromatic and methyl, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2$ (or 1.5 for methyl group) times $U_{eq}(C)$.

Comment

Nitro derivatives of the *o*-xylene are important intermediates for preparating pharmaceuticals and agrichemicals [2, 3]. The title compound is a dinitro derivative of the *o*-xylene, which was prepared by nitration of *o*-xylene.

As shown in the figure, the asymmetric unit of the title compound consists of one molecule. The aryl ring is planar, the two nitro groups are twisted against the best plane of the aryl ring, making dihedral angles of 14.4 (N1/O1,O2) and 42.6 (N2/O3,O4)°, respectively. All bond lengths and angles are in the expected ranges.

Acknowledgements: We thank the Center of Testing and Analysis, Huaiyin Normal University, for support.

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