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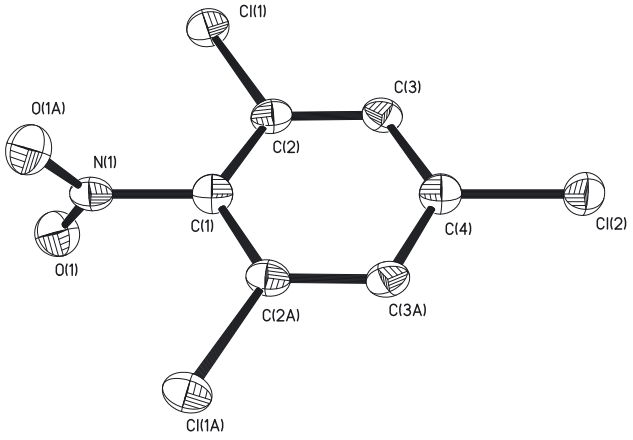
The crystal structure of 1,3,5-trichloro-2-nitrobenzene



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Abstract

$C_6H_2NCl_3O_2$, monoclinic, $C2/c$ (no. 15), a = 8.161(4) Å, b = 12.192(6) Å, c = 8.903(4) Å, β = 110.664(10)°, V = 828.8(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0513, $wR_{ref}(F^2)$ = 0.1384, T = 193 K.

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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:	White block
Size:	0.13 × 0.10 × 0.08 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.06 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE TXS PHOTON II, φ and ω
θ_{max}, completeness:	26.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2194, 855, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 657
$N(param)_{refined}$:	57
Programs:	Olex2,[1] SHELX,[2,3] Bruker[4]

1 Source of material

Slowly add 9.67 g of 1,3,5-trichlorobenzene(TCB) to 6.43 g of fuming nitric acid and 3.33 g of concentrated sulfuric acid, and react at 323.15 K for 30 min. After the reaction was completed, it was cooled to room temperature and poured into 100 mL of ice water to precipitate a white solid. The solid was filtered, washed, and dried to obtain 10.84 g, which was then recrystallized from ethanol to obtain a white solid. The solid was dissolved in ethyl acetate and slowly evaporated to obtain white block crystals.

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Nitrobenzene compounds are a research hotspot in the field of energetic materials. The title compound is a byproduct isolated during the synthesis of 1,3,5-trichloro- 2,4-dinitrobenzene (TCDNB) using 1,3,5-trichlorobenzene (TCB) as raw material and nitric acid sulfuric acid as nitration agent, proving that the title compound is an important intermediate for the synthesis of TCDNB.

The crystal structure of the title compound was analyzed and refined by Shelxl program.[1–4] The asymmetric unit of the title compound is half 1,3,5-trichloro-2-nitrobenzene molecule (see the Figure). The dihedral angle formed by the nitro group and the benzene ring plane is 81.93°. Compared with TCDNB, the planar dihedral angle formed by one nitro group and the benzene ring is 86.15°, and the planar dihedral angle formed by the other nitro group and the benzene ring is 76.04°. This is because the

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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}
Cl1	0.21552 (13)	0.41931 (8)	0.47939 (12)	0.0482 (4)
Cl2	0.500000	0.79882 (11)	0.750000	0.0501 (4)
O1	0.5659 (4)	0.2670 (2)	0.6651 (4)	0.0546 (8)
N1	0.500000	0.3139 (4)	0.750000	0.0380 (10)
C3	0.3713 (5)	0.6028 (3)	0.6289 (4)	0.0368 (8)
H3	0.283775	0.642158	0.547244	0.044*
C1	0.500000	0.4332 (4)	0.750000	0.0376 (11)
C2	0.3732 (5)	0.4897 (3)	0.6297 (4)	0.0360 (8)
C4	0.500000	0.6575 (5)	0.750000	0.0399 (12)

addition of the second nitro group causes the C–N bond to rotate in order to maintain a stable state of the TCDNB molecule, resulting in a change in the dihedral angle. Geometric parameters are all in the expected ranges.[5–10]

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