Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

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Electronic paper
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Redetermination of 1,3,5-trichloro-2,4-dinitrobenzene

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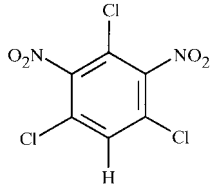
Received 21 August 2000
Accepted 5 October 2000

Data validation number: IUC0000283

The title compound, $C_6HCl_3N_2O_4$, is an intermediate in the synthesis of 1,3,5-trichloro-2,4,6-trinitrobenzene. The crystal structure at 153 K shows no major deviations from the previously reported structure at 295 K other than the expected contraction of the *a* and *c* axes and, correspondingly, the β angle.

Comment

The synthesis of 1,3,5-trichloro-2,4-dinitrobenzene, (I), was reported as early as 1887 (Jackson & Wing, 1887) although there have been a number of preparations reported subsequently (Huntress & Carten, 1940; Kaplan, 1964). Indeed, careful study of the kinetics of nitration of 1,3,5-trichlorobenzene to give both the title compound and 1,3,5-trichloro-2,4,6-trinitrobenzene has been carried out (Moodie *et al.*, 1985) and revealed that the title compound is an intermediate in the formation of the trinitro product. Our interest stemmed from the knowledge that 1,3,5-trichloro-2,4,6-trinitrobenzene may be converted to hexaaminobenzene and subsequently to a variety of substituted hexaazatriphenylenes and pyrazinoquinoxalines (Praefcke *et al.*, 1989; Rodgers, 1986; Kohne & Praefcke, 1985). During the course of synthesizing 1,3,5-trichloro-2,4,6-trinitrobenzene, we isolated colourless crystals of the title intermediate compound.



(I)

The structure of (I) has been reported previously at a temperature of 295 K (Wigand *et al.*, 1987) and the molecular structure and crystal packing at 153 K show no major deviations from that at ambient temperature. The crystal appears to show no phase change associated with the decrease in temperature, and the crystal system and space group remain unchanged. There is, however, a significant contraction of the *a* and *c* axes and, correspondingly, the β angle and volume of the unit cell.

Experimental

Compound (I) was prepared according to the literature method of Jackson & Wing (1887).

Crystal data

$C_6HCl_3N_2O_4$	D_x = 1.902 Mg m^{-3}
M_r = 271.44	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 999 reflections
a = 7.933 (7) Å	θ = 2–25°
b = 9.328 (6) Å	μ = 0.96 mm^{-1}
c = 12.814 (9) Å	T = 153 (2) K
β = 91.76 (4)°	Prism, colourless
V = 947.8 (12) Å^3	0.36 × 0.28 × 0.26 mm
Z = 4	

Data collection

Bruker AXS SMART diffractometer	R_{int} = 0.074
ω–2θ scans	θ_{max} = 26.46°
5687 measured reflections	$h = -7 \rightarrow 9$
1942 independent reflections	$k = -11 \rightarrow 11$
1315 reflections with $I > 2\sigma(I)$	$l = -14 \rightarrow 16$
	Intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R(F)$ = 0.054	$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.154	$(\Delta/\sigma)_{max} < 0.001$
S = 0.889	$\Delta\rho_{max}$ = 0.77 e Å^{-3}
1942 reflections	$\Delta\rho_{min}$ = −0.49 e Å^{-3}
136 parameters	

Crystal stablity was monitored by double collection of the first set of frames; there was no significant decay in the reflection intensities.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*.

We are grateful to the EC (contract ERBMRXCT98-0226) and Trinity College Academic Development Fund for financial support.

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