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Structure of 1,3,5-Trichloro-2,4,6-trinitrobenzene

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Abstract

The three formula units in the asymmetric unit are stacked in almost planar layers. The average interlayer distance is 4.062 (6) Å, much greater than the normal spacing observed between benzene rings as a result of the large rotation of the nitro groups out of the ring planes by 72.3–88.2°. N and Cl atoms are within the benzene average planes [deviations 0.066 (4) and 0.064 (1) Å, respectively]. The three molecules are virtually connected by a pseudo-threefold axis, leading to a hexagonal habit and a strong twinning of the crystals.

Comment

In an effort to correlate the sensitivity of explosives with their structure, the Société Nationale des Poudres et Explosifs (SNPE, France) has undertaken a screening of trinitroaromatic compounds. The one at issue was synthetized, purified and named PICL3 by the SNPE. In nitroaromatic compounds, it is well known that a Cl atom induces strong twist angles of nitro groups in α positions (Table 3). In less-symmetrical compounds than PICL3, a deviation of Cl towards the less-rotated nitro group has been observed: in picryl chloride by Willis, Stewart, Ammon, Preston, Gluyas & Harris (1971); in 1,3-dichloro-2,4,6-trinitrobenzene by Holden & Dickinson (1967); and in 1-chloro-2,4-dinitrobenzene by Wilkins, Small & Gleghorn (1990). This feature is not observed in more symmetrical molecules such as 1,2,4,5-tetrachloro-3,6-dinitrobenzene, in which C—N torsion reaches 90°, or in 1,3,5-trichloro-2,4-dinitrobenzene (Wigand, Walz, Weiden & Weiss, 1987), or here in PICL3.

In all these compounds it is observed that the greater the twist at the C—N bond, the greater its length. In PICL3, where such twist angles (listed in Table 3) are very strong, these bonds are in the range 1.484–1.498 Å, among the highest observed in polynitroaromatics. The most common explanation is that strong torsion makes the resonance of NO_2 with the ring unlikely (*e.g.* Bhattacharjee & Ammon, 1981), although such resonance, even in a planar unit, has been recently challenged by Politzer, Lane, Jayasuriya & Domelsmith (1987), and such correlation challenged by Holden & Dickinson (1977). The three molecules of PICL3 show enlarged endocyclic angles (Table 2) at the C-bearing nitro groups, a feature explained by their strong withdrawing effect (Domenicano, Vaciago & Coulson, 1975).

Molecules are stacked in nearly planar sheets, almost parallel to $(11\bar{1})$ (Table 3). Average spacing between sheets is constant within experimental precision [4.062 (6) Å], but much greater than the normal spacing [3.4 Å] of benzene rings. There is no contact within a layer except O15···Cl35 [3.219 (3) Å]. No short O···O contact from layer to layer exists, but some C···O contacts are observed, the shortest (Table 3) involving C-bearing Cl atoms of the molecule 3, and O atoms of the two others,

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yielding the necessary link between sheets. This structure shows many rhombohedral features: crystals seems to be hexagonal prisms; a change of c in $-c$ leads to a virtually rhombohedral cell with $a \simeq 12$ Å and $\alpha \simeq 78°$; the three benzene rings are closely related by a threefold axis as are the whole of the molecules 1 and 2 (with the exception of the O atoms), but a substitution of Cl by NO_2 in the molecule 3 breaks down this symmetry. The strong twinning can be understood by considering rotations of some sheets around this pseudo-rhombohedral axis, which is virtually a diagonal of the lattice.

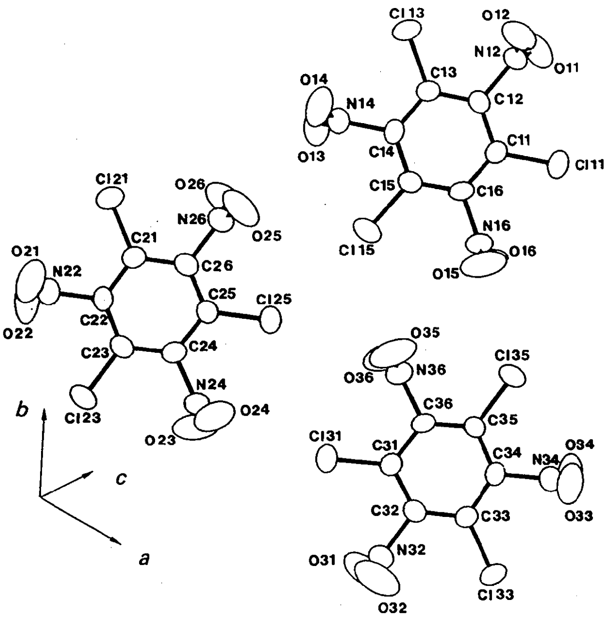

Fig. 1. An *ORTEP*II view (Johnson, 1976) of a sheet of the three molecules of PICL3 showing the atom numbering.

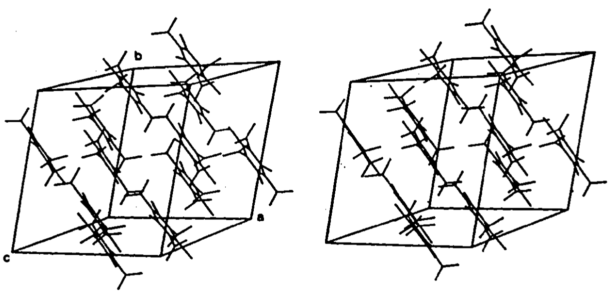

Fig. 2. A stereoscopic view of the structure of PICL3.

Experimental

Crystal data

$C_6Cl_3N_3O_6$
$M_r = 316.44$
Triclinic
$P\bar{1}$
$a = 12.137$ (2) Å
$b = 12.181$ (2) Å
$c = 11.694$ (2) Å
$\alpha = 100.39$ (1)°
$\beta = 100.10$ (1)°
$\gamma = 78.02$ (1)°
$V = 1646.3$ (4) Å^3
$Z = 6$
$D_x = 1.915$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.70930$ Å
Cell parameters from 25 reflections
$\theta = 7.5$-$17.28°$
$\mu = 0.859$ mm^{-1}
$T = 298$ K
Hexagonal prisms
0.32 × 0.24 × 0.24 mm
Colourless
Crystal source: Société Nationale des Poudres et Explosifs (France)

Data collection

CAD-4L diffractometer
ω scans of width (1.20 + 0.35tanθ)° and rate 0.97-3.29° min^{-1}
Absorption correction: none
3330 measured reflections
3114 independent reflections
2472 observed reflections $[F_o^2 \geq 3\sigma(F_o^2)]$

$R_{\text{int}} = 0.021$
$\theta_{\text{max}} = 22°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 12$
3 standard reflections ($\bar{1}45$, $3\bar{2}\bar{4}$, $\bar{4}51$) monitored every 100 reflections intensity variation: $<2\sigma(I)$

Refinement

Refinement on F $[\Sigma w(|F_o|-|F_c|)^2$ blocked full matrix, one cycle per molecule]
Final $R = 0.029$
$wR = 0.034$
$S = 1.33$
2472 reflections
164 parameters (one cycle per molecule)
$w = 1.27/\sigma^2(F)$
$(\Delta/\sigma)_{\text{max}} = 0.008$

$\Delta\rho_{\text{max}} = 0.18$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.2$ e Å^{-3}
Extinction correction: $F_c^* = F_c(1-xF_c^2/\sin\theta)$
Extinction coefficient: $x = 40$ (7) × 10^{-9}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Program(s) used for geometric calculations: *PARST* (Nardelli, 1983).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with their e.s.d.'s (Schomaker & Marsh,* 1983) *in parentheses*

$$B_{\text{eq}} = \frac{8\pi^2}{3}\Sigma_i\Sigma_j B_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C11	0.5169 (2)	0.7298 (3)	0.9926 (3)	3.31 (6)
C12	0.4364 (3)	0.8274 (3)	1.0120 (3)	3.22 (6)
C13	0.3367 (3)	0.8489 (2)	0.9340 (3)	3.16 (6)
C14	0.3200 (2)	0.7701 (3)	0.8347 (3)	3.29 (6)
C15	0.3985 (3)	0.6713 (3)	0.8123 (3)	3.49 (6)
C16	0.4958 (3)	0.6527 (2)	0.8925 (3)	3.36 (7)
Cl11	0.63764 (8)	0.70470 (8)	1.09119 (9)	5.44 (2)
Cl13	0.23806 (8)	0.96954 (7)	0.95862 (9)	5.09 (2)
Cl15	0.37551 (9)	0.57415 (8)	0.69008 (9)	5.96 (2)
N12	0.4565 (3)	0.9119 (2)	1.1189 (3)	3.94 (6)
N14	0.2147 (3)	0.7917 (2)	0.7494 (3)	4.08 (7)
N16	0.5812 (3)	0.5475 (3)	0.8699 (3)	3.88 (6)
O11	0.5274 (3)	0.9685 (2)	1.1202 (2)	6.60 (7)
O12	0.3997 (3)	0.9192 (2)	1.1949 (2)	6.21 (7)
O13	0.2246 (2)	0.8196 (2)	0.6582 (3)	6.19 (7)
O14	0.1270 (2)	0.7789 (3)	0.7760 (3)	6.69 (8)

O15	0.5678 (3)	0.4652 (3)	0.9044 (3)	7.62 (9)
O16	0.6545 (3)	0.5512 (2)	0.8166 (3)	7.08 (8)
C21	−0.0054 (3)	0.5150 (3)	0.2614 (3)	3.62 (7)
C22	−0.0174 (3)	0.4302 (3)	0.1665 (3)	3.50 (7)
C23	0.0653 (3)	0.3333 (3)	0.1516 (3)	3.56 (7)
C24	0.1593 (3)	0.3212 (2)	0.2367 (3)	3.22 (6)
C25	0.1748 (3)	0.4040 (3)	0.3339 (3)	3.35 (6)
C26	0.0918 (3)	0.5008 (3)	0.3434 (3)	3.40 (7)
Cl21	−0.10736 (9)	0.63451 (8)	0.27850 (10)	6.20 (2)
Cl23	0.04805 (9)	0.23051 (8)	0.03292 (9)	5.70 (2)
Cl25	0.29403 (8)	0.38811 (8)	0.43598 (9)	5.36 (2)
N22	−0.1219 (3)	0.4437 (3)	0.0779 (3)	4.34 (7)
N24	0.2478 (3)	0.2155 (3)	0.2252 (3)	3.89 (6)
N26	0.1068 (3)	0.5917 (3)	0.4455 (3)	4.26 (7)
O21	−0.2056 (3)	0.4247 (3)	0.1020 (3)	8.18 (9)
O22	−0.1153 (3)	0.4719 (4)	−0.0100 (3)	10.21 (12)
O23	0.3100 (3)	0.2069 (2)	0.1549 (3)	7.69 (8)
O24	0.2508 (3)	0.1498 (2)	0.2886 (3)	7.47 (8)
O25	0.0895 (3)	0.5764 (3)	0.5383 (3)	7.56 (9)
O26	0.1343 (3)	0.6740 (3)	0.4271 (3)	7.73 (9)
C31	0.6495 (3)	0.0946 (3)	0.5019 (3)	3.49 (6)
C32	0.7386 (3)	0.0026 (3)	0.4915 (3)	3.30 (6)
C33	0.8361 (3)	−0.0064 (3)	0.5727 (3)	3.33 (6)
C34	0.8419 (3)	0.0790 (3)	0.6685 (3)	3.28 (6)
C35	0.7562 (3)	0.1721 (3)	0.6823 (3)	3.42 (6)
C36	0.6615 (3)	0.1790 (3)	0.5983 (3)	3.43 (6)
Cl31	0.53012 (7)	0.10282 (8)	0.40029 (8)	5.50 (2)
Cl33	0.94652 (8)	−0.11777 (8)	0.55625 (8)	5.06 (2)
Cl35	0.76717 (8)	0.27444 (8)	0.80225 (8)	5.36 (2)
N32	0.7289 (3)	−0.0904 (3)	0.3892 (3)	4.34 (7)
N34	0.9443 (3)	0.0693 (3)	0.7605 (3)	4.51 (7)
N36	0.5695 (3)	0.2802 (3)	0.6083 (3)	4.78 (7)
O31	0.7505 (3)	−0.0746 (3)	0.2999 (3)	7.88 (9)
O32	0.7031 (3)	−0.1730 (3)	0.4070 (3)	9.22 (10)
O33	0.9421 (3)	0.0188 (3)	0.8388 (3)	7.60 (8)
O34	1.0180 (3)	0.1162 (3)	0.7516 (3)	8.20 (9)
O35	0.4979 (3)	0.2799 (3)	0.6639 (4)	8.71 (10)
O36	0.5747 (3)	0.3556 (3)	0.5597 (3)	9.53 (10)

Table 2. *Bond lengths* (Å) *and angles* (°)

C11—C12	1.386 (4)	C22—N22	1.490 (4)
C12—C13	1.391 (4)	C24—N24	1.498 (4)
C13—C14	1.381 (4)	C26—N26	1.489 (4)
C14—C15	1.386 (4)	N22—O21	1.178 (6)
C15—C16	1.380 (4)	N22—O22	1.161 (6)
C11—C16	1.384 (4)	N24—O23	1.188 (5)
C11—Cl11	1.708 (3)	N24—O24	1.175 (5)
C13—Cl13	1.707 (3)	N26—O25	1.194 (5)
C15—Cl15	1.705 (3)	N26—O26	1.185 (5)
C12—N12	1.488 (4)	C31—C32	1.391 (4)
C14—N14	1.485 (4)	C32—C33	1.379 (4)
C16—N16	1.486 (4)	C33—C34	1.387 (4)
N12—O11	1.206 (5)	C34—C35	1.378 (4)
N12—O12	1.197 (5)	C35—C36	1.374 (4)
N14—O13	1.208 (5)	C31—C36	1.391 (4)
N14—O14	1.207 (5)	C31—Cl31	1.703 (3)
N16—O15	1.197 (5)	C33—Cl33	1.708 (3)
N16—O16	1.184 (5)	C35—Cl35	1.706 (3)
C21—C22	1.382 (4)	C32—N32	1.498 (4)
C22—C23	1.389 (4)	C34—N34	1.494 (4)
C23—C24	1.378 (4)	C36—N36	1.484 (4)
C24—C25	1.391 (4)	N32—O31	1.176 (5)
C25—C26	1.384 (4)	N32—O32	1.176 (6)
C21—C26	1.386 (4)	N34—O33	1.198 (5)
C21—Cl21	1.710 (3)	N34—O34	1.185 (5)
C23—Cl23	1.708 (3)	N36—O35	1.174 (6)
C25—Cl25	1.708 (3)	N36—O36	1.181 (6)

C16—C11—C12	118.2 (3)	Cl25—C25—C26	121.2 (2)
Cl11—C11—C12	120.6 (2)	C25—C26—C21	121.9 (3)
Cl11—C11—C16	121.2 (2)	N26—C26—C21	119.0 (3)
C13—C12—C11	121.7 (3)	N26—C26—C25	119.0 (3)
N12—C12—C11	119.6 (3)	O21—N22—C22	117.5 (4)
N12—C12—C13	118.8 (2)	O22—N22—C22	118.0 (4)
C14—C13—C12	118.0 (2)	O22—N22—O21	124.5 (4)
Cl13—C13—C12	121.3 (2)	O23—N24—C24	117.1 (3)
Cl13—C13—C14	120.7 (2)	O24—N24—C24	116.9 (3)
C15—C14—C13	122.1 (3)	O24—N24—O23	126.0 (3)
N14—C14—C13	119.0 (2)	O25—N26—C26	117.7 (3)
N14—C14—C15	119.0 (3)	O26—N26—C26	116.7 (3)
C16—C15—C14	118.1 (3)	O26—N26—O25	125.6 (3)
Cl15—C15—C14	121.3 (2)	C36—C31—C32	117.5 (3)
Cl15—C15—C16	120.5 (2)	Cl31—C31—C32	121.0 (2)
C15—C16—C11	122.0 (3)	Cl31—C31—C36	121.5 (2)
N16—C16—C11	119.1 (3)	C33—C32—C31	122.2 (3)
N16—C16—C15	119.0 (3)	N32—C32—C31	119.2 (3)
O11—N12—C12	116.8 (3)	N32—C32—C33	118.6 (3)
O12—N12—C12	117.4 (3)	C34—C33—C32	117.6 (3)
O12—N12—O11	125.8 (3)	Cl33—C33—C32	121.6 (2)
O13—N14—C14	116.8 (3)	Cl33—C33—C34	120.8 (2)
O14—N14—C14	117.7 (3)	C35—C34—C33	122.4 (3)
O14—N14—O13	125.5 (3)	N34—C34—C33	118.9 (3)
O15—N16—C16	116.8 (3)	N34—C34—C35	118.7 (3)
O16—N16—C16	117.6 (3)	C36—C35—C34	118.2 (3)
O16—N16—O15	125.6 (3)	Cl35—C35—C34	120.5 (2)
C26—C21—C22	118.3 (3)	Cl35—C35—C36	121.3 (2)
Cl21—C21—C22	121.4 (2)	C35—C36—C31	122.1 (3)
Cl21—C21—C26	120.3 (2)	N36—C36—C31	118.4 (3)
C23—C22—C21	121.7 (3)	N36—C36—C35	119.5 (3)
N22—C22—C21	119.0 (3)	O31—N32—C32	116.9 (3)
N22—C22—C23	119.3 (3)	O32—N32—C32	116.6 (4)
C24—C23—C22	118.1 (3)	O32—N32—O31	126.5 (4)
Cl23—C23—C22	120.5 (2)	O33—N34—C34	116.9 (3)
Cl23—C23—C24	121.4 (2)	O34—N34—C34	116.8 (3)
C25—C24—C23	122.2 (3)	O34—N34—O33	126.2 (3)
N24—C24—C23	119.3 (3)	O35—N36—C36	118.1 (4)
N24—C24—C25	118.5 (3)	O36—N36—C36	116.8 (4)
C26—C25—C24	117.7 (3)	O36—N36—O35	125.1 (4)
Cl25—C25—C24	121.0 (2)		

Table 3. *Twist angles* (°), *dihedral angles* (°) *and short intermolecular contacts* (Å)

Signed twist angles [defined by Winkler & Dunitz (1971)]

C12—N12	72.3 (4)	C22—N22	−78.4 (5)	C32—N32	80.6 (4)
C14—N14	−74.5 (4)	C24—N24	74.2 (4)	C34—N34	−86.6 (4)
C16—N16	−88.2 (4)	C26—N26	−76.3 (4)	C36—N36	−86.9 (5)

Planes

1	$(11\bar{1})$	4	Benzene ring 2
2	Sheet of 18 C atoms	5	Benzene ring 3
3	Benzene ring 1		

Dihedral angles

1,2	0.39 (1)	2,3	1.35 (7)
1,3	1.00 (6)	2,4	0.65 (6)
1,4	0.69 (6)	2,5	2.57 (6)
1,5	2.54 (6)		

Line 6: cell diagonal from 0,0,1 to 1,1,0

Angles formed by line 6 and normal to the planes

6,1	2.94 (0)	6,4	3.22 (6)
6,2	3.32 (1)	6,5	4.00 (7)
6,3	2.15 (7)		

Shortest intermolecular contacts

Interlayer		Symmetry operation
C31···O13	3.052 (5)	$1-x,\ 1-y,\ 1-z$
C33···O24	3.031 (5)	$1-x,\ -y,\ 1-z$
C35···O26	3.068 (5)	$1-x,\ 1-y,\ 1-z$
C35···O12	3.046 (5)	$1-x,\ 1-y,\ 2-z$
Within the sheet		
O15···Cl35	3.219	$x,\ y,\ z$

All methods for recrystallization gave twinned hexagonal prisms. The less twinned were obtained by slow addition of water in a solution of PICL3 in ethanol. Prisms were checked by optical polarizing microscopy; uniformly coloured sections were cut with a razor blade.

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Lists of structure factors, anisotropic thermal parameters, interatomic and intermolecular contacts, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55946 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1015]

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Structure of *cis*-4-Cyclohexene-1,2-dicarboxylic Acid

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Abstract

The molecules are connected by two *inter*molecular hydrogen bonds [O···O distances 2.656 (2) and 2.641 (2) Å] forming infinite chains along [100]. No *intra*molecular hydrogen bond is present between the two neighbouring carboxylic groups. This can be explained by geometric considerations.

Comment

There exist several examples of dicarboxylic acids (or their acid salts) where short intramolecular hydrogen bonds are formed between adjacent carboxylic groups. The reasons for the occurrence or non-occurrence of the intramolecular hydrogen bond seem to be partly of geometric and partly of chemical nature (Küppers & Jessen, 1993). Since the title compound is a potential example where an intramolecular hydrogen bond might be found, an X-ray structure analysis was undertaken.

The atomic packing within the unit cell is shown by a stereoscopic *ORTEP*II plot (Johnson, 1976) in Fig. 1. The molecules do *not* form an *intra*molecular hydrogen bond. Instead, the molecules are interconnected by *inter*molecular hydrogen bonds of the 'cyclic dimer type' (Leiserowitz, 1976). Each of the two carboxylic groups of a molecule is linked to an equivalent (through inversion centre $\bar{1}$) carboxylic group of an adjacent molecule. The two intermolecular hydrogen bonds have lengths 2.656 (2) Å (O1—H7···O2) and 2.641 (2) Å (O3···H8—O4). Thus, the molecules build infinite zigzag chains, which extend along the [100] direction. Because of the double bond between C4 and C5, the atoms C3, C4, C5 and C6 roughly define a plane. The maximum deviation from the mean plane is 0.01 Å. As expected, the ring has a half-chair conformation. According to the *cis* configuration, one carboxylic group (C7, O1, O2) is in equatorial position [angle between the C7, O1, O2 plane and the mean plane defined by C3 to C6 is 6.6 (2)°]. The other carboxylic group (C8, O3, O4) is in an axial position; the respective angle is 85.0 (3)°. The torsion angle C7—C1—C2—C8 is 65.2 (2)°.

The fact that no intramolecular hydrogen bond is formed in the present compound can be explained through geometric considerations. Küppers & Jessen (1993) showed for several examples of dicarboxylic acids that the intramolecular hydrogen bond is not formed if the O···O distance between neighbouring —COOH groups (in unstrained molecules with properly rotated —COOH groups) is shorter than approximately 2.2 Å. In the range 2.2-2.4 Å, the molecules turn out to be able to adjust themselves by internal distortion of intramolecular angles, yielding widened O···O distances of about 2.4 Å. The molecules considered in the paper mentioned above are such that the four C atoms, which correspond to the present atoms C7, C1, C2 and C8, are coplanar. This is not the case in the molecule of this study. One can roughly estimate, however, the theoretical distance of neighbouring O atoms in a quasi-planar configuration if the COO groups are rotated around the C—C bond until two confronting O atoms lie within a plane defined by C7, C8 and the midpoint between C1 and C2. Assuming averaged distances

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