Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,5,6-Trichloro-4-iodoisophthalonitrile

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Received 18 December 2003
Accepted 5 January 2004
Online 17 January 2004

The molecule of the title compound, $C_8Cl_3IN_2$, is disordered in the crystal. For the major component, there is an I···N intermolecular contact of 3.06 (3) Å and two Cl···N contacts of 3.10 (3) and 3.19 (3) Å. In all three contacts, the angle is approximately linear at the halogen atom and bent at the nitrogen.

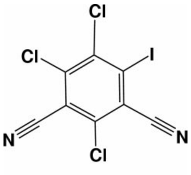
Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.02 Å
Disorder in main residue
R factor = 0.050
wR factor = 0.095
Data-to-parameter ratio = 10.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The structure of tetrachloroisophalonitrile was determined previously (Britton, 1981). The structure of the title compound, (I), has been determined to examine the effect on the packing caused by the replacement of one Cl atom by an I atom. The I atom is a much stronger Lewis acid than the Cl atom and the question was whether the expected stronger I···N—C interaction would lead to a significant change in the packing.



(I)

Fig. 1 shows the atom labeling and anisotropic displacement ellipsoids for the compound. All of the bond distances and angles are normal. The molecule is disordered by rotation about an axis passing approximately through Cl12 and Cl15. The effect of this disorder is to exchange an I atom and a Cl atom. The minor component is shown in outline in Fig. 1. The major component of the disorder had an abundance of 0.735 (3).

The molecules pack to form double ribbons, which are close to planar [molecular tilt 1.2 (1)° out of the plane of the

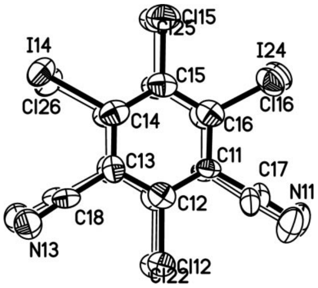


Figure 1
The molecule of (I). Displacement ellipsoids are drawn at the 50% probability level. The molecule is disordered with an exchange of Cl and I. The minor component of the disorder is shown in outline; only two of the minor component atoms are labeled.

DOI: 10.1107/S1600536804000133

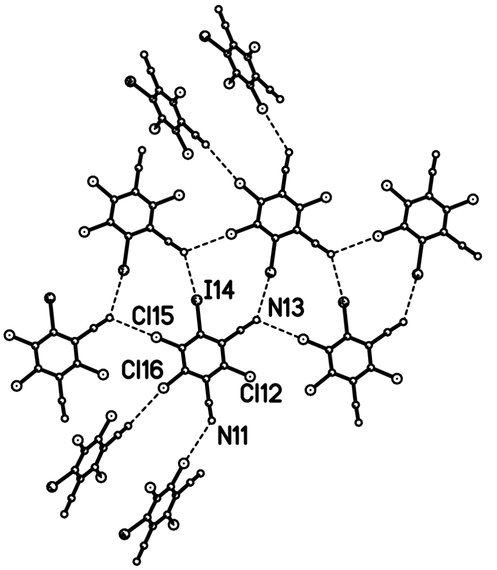


Figure 2
The packing of $ICl_3C_6(CN)_2$, viewed normal to the molecular plane. The I···C and Cl···NC contacts are shown as dashed lines. Only the major component of the disorder is shown.

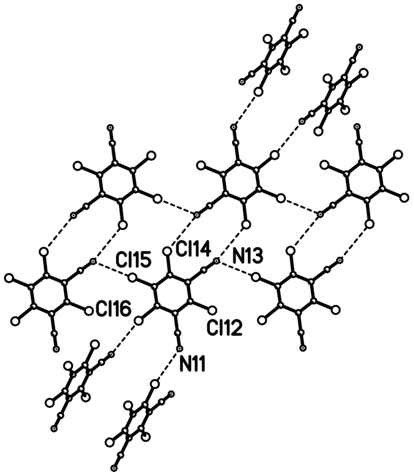


Figure 3
The packing of $Cl_4C_6(CN)_2$, viewed normal to the molecular plane. The Cl···NC contacts are shown as dashed lines.

ribbon]. Fig. 2 shows a portion of the ribbon (using the major component only) plus contacts with adjacent ribbons. The two halves of the ribbon are related by a 2_1 axis. Table 1 gives the metric values for the different contacts for each of the four possible orientations of the disorder: major–major, major–minor, minor–major, and minor–minor, which occur, respectively, in 54.0, 19.5, 19.5, and 7.0% of the contacts. In all three contacts, the angular arrangement is close to linear at the halogen and bent at the nitrogen. Ideally, for a Lewis acid–base interaction the angles would be near linear at both atoms, but bending at the nitrogen is well known in other structures [see Britton (2002) for a number of examples]. The contact distances in the major–major component are typical for these sorts of contacts. The I···N contact at 3.06 (3) Å appears shorter than either of the Cl···N contacts [3.10 (3) and 3.19 (3) Å], although not beyond experimental error.

The major component of the disorder has the I···N contact within the ribbon while the minor component has it between adjacent ribbons. Given the large degree of disorder the two different arrangements must be close in energy. The distances and angles, especially those involving the minor component, have unusually large s.u. values as a consequence of the disorder and cannot be used to explain the disorder in any detail.

The original intent was to look for changes with respect to the structure of the corresponding $Cl_4C_6(CN)_2$ (Britton, 1981). The packings are similar, as can be seen in Fig. 3, which shows the view for the Cl_4 structure corresponding to Fig. 2. The difference in the ribbon is that here the two halves of the ribbon are related by a center of symmetry. This makes the Cl···N interaction within the ribbon less than ideal, with a C—Cl···N interaction that is far from linear. The metric data for this structure are also included in Table 1.

To make the overall similarity more apparent, Fig. 4 shows the two structures viewed edge-on to one set of ribbons. Although the two space groups are quite different, $P4_32_12$ *versus* $P2_1/a$, the unit-cell dimensions are similar except that the long axis in the Cl_4 structure must be doubled. The doubling arises since 2_1 axes relate adjacent ribbons in the Cl_4 structure, while 4_1 axes relate them in the ICl_3 structure. The cause for this difference is not apparent. Either structure would appear to be reasonable for both compounds.

Experimental

The compound was supplied by Dr Robert Battershell of the Diamond–Shamrock Corporation. Crystals grown from a variety of solvents gave tetragonal plates of inadequate quality for data collection. A very thin, but useable, crystal was finally obtained from acetone.

Crystal data

$C_8Cl_3IN_2$
$M_r = 357.35$
Tetragonal, $P4_32_12$
$a = 6.3657\ (16)$ Å
$c = 52.125\ (13)$ Å
$V = 2112.2\ (9)$ Å^3
$Z = 8$
$D_x = 2.247$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 900 reflections
$\theta = 3.2$–$29.5°$
$\mu = 3.75$ mm^{-1}
$T = 297\ (2)$ K
Plate, colorless
$0.15 \times 0.10 \times 0.01$ mm

Data collection

Siemens SMART area-detector diffractometer
ω scans
Absorption correction: analytical (*XPREP* in *SHELXTL*; Sheldrick, 1994)
$T_{\min} = 0.70$, $T_{\max} = 0.96$
18634 measured reflections

1872 independent reflections
1406 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.098$
$\theta_{\max} = 25.0°$
$h = -7 \rightarrow 7$
$k = -7 \rightarrow 7$
$l = -61 \rightarrow 61$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.095$
$S = 1.16$
1872 reflections
171 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0065P)^2 + 9.13P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.43$ e Å^{-3}
$\Delta\rho_{\min} = -0.48$ e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = $-0.02\ (8)$

Table 1
Distances and angles (Å,°) in the I···N and Cl···N contacts in $ICl_3C_6(CN)_2$ and $Cl_4C_6(CN)_2$[a].

X	N	C—X···N	X···N	X···N—C
I14	N13^{i}	169 (1)	3.06 (3)	139 (3)
I14	N21^{i}	164 (2)	2.83 (8)	144 (8)
Cl26	N13^{i}	169 (1)	3.36 (3)	140 (2)
Cl26	N21^{i}	165 (2)	3.13 (8)	146 (8)
Cl14	N13	134	3.39	165
Cl15	N13^{ii}	179 (2)	3.10 (3)	125 (2)
Cl15	N21^{ii}	174 (1)	3.27 (10)	115 (7)
Cl25	N13^{ii}	176 (3)	3.16 (4)	128 (2)
Cl25	N21^{ii}	173 (5)	3.31 (10)	117 (7)
Cl15	N13	177	3.07	122
Cl16	N11^{iii}	172 (1)	3.19 (3)	135 (2)
Cl16	N23^{iii}	166 (2)	3.34 (7)	139 (5)
I24	N11^{iii}	168 (1)	2.99 (3)	131 (2)
I24	N23^{iii}	163 (2)	3.14 (7)	135 (5)
Cl16	N13	167	314	137

Note: (*a*) the entries in each set of contacts comprise the interaction between the major components, then that between the major and minor components, then the minor–major, then the minor–minor, and finally the corresponding values in the $Cl_4C_6(CN)_2$ structure. The latter are given without error estimates. Symmetry codes: (i) $1+y, x, -z$; (ii) $1+x, 1+y, z$; (iii) $\frac{1}{2}+x, \frac{5}{2}-y, \frac{1}{4}-z$.

A complete sphere of data was collected. All possible Friedel pairs were measured of which 634 (51%) of the 1238 pairs contained information about which enantiomer was present. The refinement converged with a Flack (1983) parameter of −0.02 (8), indicating that the reported enantiomer is the correct one. The molecules are disordered. The two orientations of the disordered molecules were restrained to have the same interatomic distances with an s.u. of 0.001 Å. Atoms of the two orientations that overlapped were required to have identical anisotropic displacement parameters.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The author thanks Dr Robert Battershell for providing the sample of the title compound.

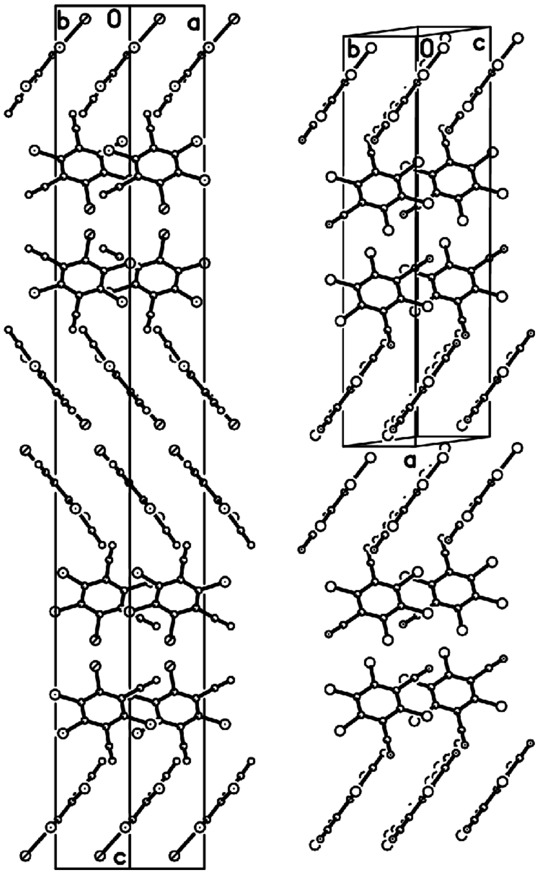


Figure 4
A comparison of the packings in $ICl_3C_6(CN)_2$ (left) and $Cl_4C_6(CN)_2$ (right). The left view is normal to (110); the right, to (011).

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