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2-Fluoro-6-iodobenzonitrile

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Key indicators

Single-crystal X-ray study
T = 174 K
Mean σ(C–C) = 0.009 Å
R factor = 0.043
wR factor = 0.109
Data-to-parameter ratio = 14.8

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

The title compound packs with two crystallographically independent molecules, both symmetry 1, with the same bond lengths and angles within experimental error. The molecules form a four-membered cyclamer held together by I···CN intermolecular Lewis acid–base interactions. The two independent N···I distances are 3.263 (6) and 3.344 (6) Å. The independent molecules are not related by any pseudosymmetry.

Comment

In the structure of *p*-iodobenzonitrile (Schlemper & Britton, 1965; Desiraju & Harlow, 1989) the CN group on one molecule points directly toward the I atom in the next molecule giving a linear C—I···NC arrangement. Similar arrangements, sometimes only approximately linear, occur in a number of other crystals (see Ojala *et al.*, 1999), but in *o*-iodobenzonitrile (Lam & Britton, 1974) the important intermolecular interactions are between I atoms; there are no I···CN contacts at all. To look at another example of an *o*-iodobenzonitrile, the structure of 2-fluoro-6-iodobenzonitrile (I) has been determined and is reported here

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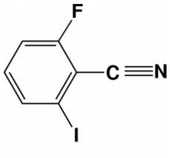

(I)

There are two molecules in the asymmetric unit; using the notation suggested by Zorky and coworkers (Zorky *et al.*, 1967; Belsky *et al.*, 1995; Zorky, 1996) the structure is in $P\overline{1}$, $Z = 4$ (1^2). Drawings showing the atom labelling of the two independent molecules are given in Fig. 1. The bond lengths and angles are all normal and agree between the two molecules within experimental uncertainty.

A cyclamer is formed by the association of four monomers through weak intermolecular CN···I Lewis acid–base interactions. This can be seen in Fig. 2. These interactions are summarized in Table 1. The CN···I distances are what would be expected from the non-spherical radii of Nyburg & Faerman (1985). The angles at the I atoms are approximately linear, as expected for this type of interaction, but the angles at the N atoms are closer to trigonal. This is a less common arrangement but has been observed before (see Table 4 in Ojala *et al.*, 1999).

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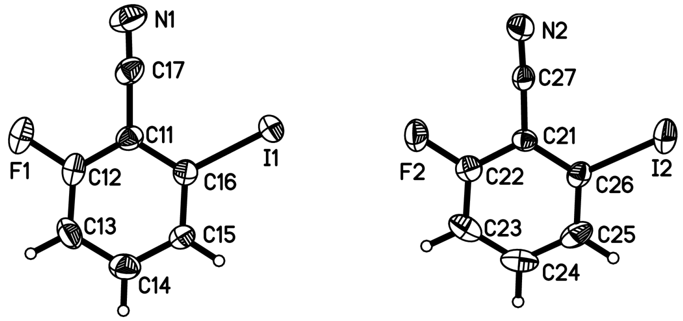

Figure 1
The two independent $C_6H_3F(CN)I$ molecules. Displacement ellipsoids are shown at the 50% probability level.

In addition to the CN···I interactions, there are two weak C—H···F interactions and two weak C—H···N interactions. These are also shown in Fig. 2. The metric data for these interactions are given in Table 2. None of the four interaction distances is especially short; they are very weak interactions. Nevertheless, they all involve attractive interactions and play their part in the overall packing. For discussions of C—H···F interactions, see Howard *et al.* (1996); Dunitz & Taylor, 1997; Thalladi *et al.* (1998). For a more wide-ranging discussion of C—H···*X* interactions, see Desiraju & Steiner, 1999).

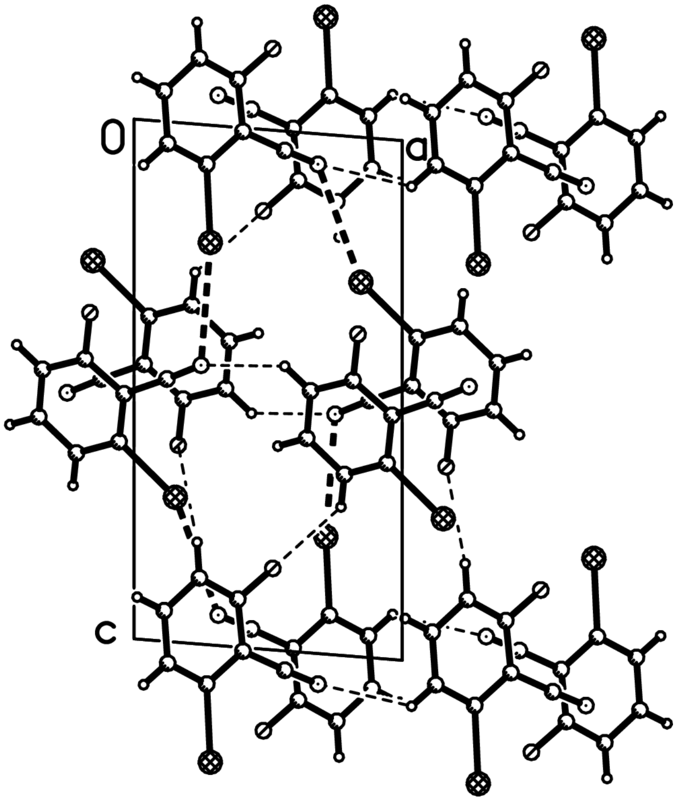

Figure 2
The packing of $C_6H_3F(CN)I$ viewed along the *b* axis. The CN···I interactions are shown as heavy dashed lines. One tetrameric cyclamer can be traced out in the figure. The light dashed lines show the N···H and F···H interactions.

Experimental

The compound was obtained from Lancaster Synthesis, Inc. The crystals grew as plates from acetone, with {001} as the prominent form.

Crystal data

C_7H_3FIN
$M_r = 247.00$
Triclinic, $P\overline{1}$
$a = 8.0270$ (8) Å
$b = 8.0435$ (8) Å
$c = 13.8493$ (13) Å
$\alpha = 82.232$ (2)°
$\beta = 82.359$ (2)°
$\gamma = 62.242$ (2)°
$V = 781.52$ (15) Å^3
$Z = 4$
$D_x = 2.099$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4333 reflections
$\theta = 4.0$–24.8°
$\mu = 4.04$ mm^{-1}
$T = 174$ (2) K
Plates, colorless
0.40 × 0.40 × 0.10 mm

Data collection

Siemens SMART area-detector diffractometer
ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996; Blessing, 1995)
$T_{\min} = 0.23$, $T_{\max} = 0.67$
5149 measured reflections
2689 independent reflections
2241 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.042$
$\theta_{\max} = 24.9$°
$h = -9 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -16 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.109$
$S = 0.98$
2689 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 1.07$ e Å^{-3}
$\Delta\rho_{\min} = -1.49$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0042 (9)

Table 1
Distances and angles (Å,°) in the C—N···I—C contacts.

N	I	C—N···I	N···I	N···I—C
N1	I2	119.0 (5)	3.344 (6)	154.9 (6)
N2	I1^{i}	114.9 (5)	3.267 (6)	172.6 (7)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Table 2
Intermolecular hydrogen contacts C—H···*X*—*Y* (Å,°).

C	H	*X*	*Y*	C—H	H···*X*	C···*X*	C—H···*X*	H···*X*—*Y*
C13	H13	F2^{i}	C22^{i}	0.95	2.63	3.550 (7)	164.5	144
C25	H25	F1^{ii}	C12^{ii}	0.95	2.60	3.164 (6)	119	175
C15	H15	N1^{iii}	C17^{iii}	0.95	2.77	3.464 (7)	130	167
C23	H23	N2^{iv}	C27^{iv}	0.95	2.63	3.274 (7)	134	160

Symmetry codes: (i) $1 + x, y - 1, 1 + z$; (ii) $-x, 1 - y, 2 - z$; (iii) $1 - x, y - 1, z$; (iv) $x - 1, y, z$.

The matrix ($\overline{1}\overline{1}0/1\overline{1}0/001$) converts the triclinic unit cell to a *C*-centered cell that is metrically close to monoclinic. However, if the symmetry is assumed to be monoclinic, $R_{\text{int}} = 0.432$, which rules out the monoclinic alternative.

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*.

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