

We thank Dr W. J. Muizebelt (Akzo Research Laboratories) for donating the crystals and Dr A. J. M. Duisenberg for collecting the data.

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Acta Cryst. (1991). **C47**, 2472–2474

Structure of 5-Chloro-2,4,6-trifluoroisophthalonitrile*

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(Received 15 April 1991; accepted 10 May 1991)

Abstract. $C_6ClF_3(CN)_2$, $M_r = 216.55$, monoclinic, $P2_1/c$, $a = 10.266$ (3), $b = 14.848$ (6), $c = 11.537$ (3) Å, $\beta = 108.83$ (3)°, $Z = 8$ (two molecules in the asymmetric unit), $V = 1664$ (2) Å³, $D_x = 1.728$ (2) g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 4.60$ cm⁻¹, $F(000) = 848$, $T = 297$ (2) K, $R = 0.053$ for 2259 unique observed reflections with $I > \sigma(I)$. The bond lengths and angles are normal, except that one ring is slightly puckered and in both molecules about half of the substituents are bent out of the mean plane of the ring by 1.0 to 2.5°. Three short intermolecular distances can be seen in the packing.

Experimental. The compound was obtained from Dr Robert Battershell of the Diamond Shamrock Corp.; crystals suitable for X-ray diffraction were found in the original sample. A colorless crystal $0.10 \times 0.25 \times 0.60$ mm, mounted in air, was used for the data collection. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 22 reflections with $10 < \theta < 14^\circ$ were used to determine the cell parameters. Systematic extinctions ($h0l$, l odd; $0k0$, k odd) uniquely determined the space group. Data were collected, using $\omega/2\theta$ scans, in the range $0 < \theta < 25^\circ$ for all of one octant and half of a second (ranges: h , -12 to 4 ; k , 0 to 17 ; l , -13 to 13). The intensities of 4429 different reflections were measured. Three check reflections measured every 6000 s of exposure time showed a linear decrease in intensity of 45% over the

course of the data collection; a correction was made for this decay. After absorption corrections were made using the program *DIFABS* (Walker & Stuart, 1983; transmission factors 0.73 to 1.10), equivalent reflections were combined to give 2924 independent reflections ($R_{int} = 0.037$) of which the 2259 with $I > \sigma(I)$ were used in the calculations. The structure was solved by direct methods (*MITHRIL*, Gilmore, 1984) and refined with full-matrix least-squares refinement on F^2 's. There are two molecules in the asymmetric unit. All atoms were given anisotropic thermal parameters. Refinement converged with $R = 0.053$, $wR = 0.056$, and $S = 1.22$; $w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I)_c + (0.05I)^2$, where $\sigma(I)_c$ is the standard deviation in I based on counting statistics alone. In the final cycle of refinement ($\Delta/\sigma)_{max} = 0.03$, $(\Delta\rho)_{max} = 0.23$ and $(\Delta\rho)_{min} = -0.31$ e Å⁻³. Atomic scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The computer programs used were from *TEXSAN* (Molecular Structure Corporation, 1985). The final positional parameters are given in Table 1.†

† Lists of anisotropic thermal parameters, complete bond angles, deviations from the mean planes of the benzene rings, intermolecular distances, observed and calculated structure factors, and a figure showing the thermal ellipsoids in molecule *B* have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54237 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* 5-Chloro-2,4,6-trifluoro-1,3-benzenedicarbonitrile.

Table 1. *Positional and isotropic equivalent thermal parameters*

$$B_{eq} = (8\pi^2/3)\sum_i U_i a_i^* a_i$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
C1A	0.8122 (3)	0.3335 (3)	0.4829 (3)	3.4 (1)
C2A	0.7432 (3)	0.3059 (2)	0.5618 (3)	3.5 (1)
C3A	0.6775 (3)	0.3655 (3)	0.6151 (3)	3.8 (1)
C4A	0.6827 (3)	0.4555 (3)	0.5874 (3)	3.9 (1)
C5A	0.7512 (3)	0.4872 (2)	0.5104 (3)	3.8 (1)
C6A	0.8140 (3)	0.4245 (3)	0.4604 (3)	3.7 (2)
C11A	0.8816 (4)	0.2704 (3)	0.4278 (4)	4.5 (2)
N1A	0.9359 (4)	0.2224 (3)	0.3845 (4)	7.0 (2)
F2A	0.7424 (2)	0.2185 (1)	0.5877 (2)	5.1 (1)
C13A	0.6049 (4)	0.3356 (3)	0.6970 (4)	4.7 (2)
N3A	0.5488 (4)	0.3126 (3)	0.7587 (4)	6.8 (2)
F4A	0.6171 (2)	0.5148 (2)	0.6362 (2)	5.8 (1)
C15A	0.7569 (1)	0.59955 (8)	0.4793 (1)	6.61 (6)
F6A	0.8834 (2)	0.4522 (1)	0.3871 (2)	5.0 (1)
C1B	0.6363 (3)	0.9100 (2)	0.4803 (3)	3.4 (1)
C2B	0.6493 (3)	0.8376 (2)	0.5574 (3)	3.3 (1)
C3B	0.7600 (3)	0.8280 (2)	0.6622 (3)	3.3 (1)
C4B	0.8613 (3)	0.8941 (2)	0.6877 (3)	3.3 (1)
C5B	0.8558 (3)	0.9667 (2)	0.6124 (3)	3.4 (1)
C6B	0.7419 (3)	0.9729 (2)	0.5096 (3)	3.7 (1)
C11B	0.5177 (4)	0.9215 (3)	0.3731 (3)	4.4 (2)
N1B	0.4222 (4)	0.9322 (3)	0.2921 (3)	6.2 (2)
F2B	0.5529 (2)	0.7743 (1)	0.5289 (2)	4.7 (1)
C13B	0.7727 (4)	0.7525 (3)	0.7428 (3)	4.1 (2)
N3B	0.7853 (4)	0.6941 (3)	0.8086 (4)	6.4 (2)
F4B	0.9682 (2)	0.8858 (1)	0.7893 (2)	4.66 (9)
C15B	0.9839 (1)	1.04481 (7)	0.6454 (1)	5.17 (5)
F6B	0.7304 (2)	1.0427 (2)	0.4349 (2)	5.7 (1)

Table 2. *Bond lengths (\AA) and angles (°)*

C1A—C2A	1.384 (5)	C1B—C2B	1.375 (5)
C1A—C6A	1.377 (5)	C1B—C6B	1.387 (5)
C1A—C11A	1.444 (5)	C1B—C11B	1.439 (5)
C2A—C3A	1.372 (5)	C2B—C3B	1.374 (5)
C2A—F2A	1.332 (4)	C2B—F2B	1.326 (4)
C3A—C4A	1.379 (5)	C3B—C4B	1.390 (5)
C3A—C13A	1.449 (5)	C3B—C13B	1.435 (5)
C4A—C5A	1.381 (5)	C4B—C5B	1.374 (5)
C4A—F4A	1.338 (4)	C4B—F4B	1.328 (4)
C5A—C6A	1.362 (5)	C5B—C6B	1.374 (5)
C5A—C15A	1.712 (4)	C5B—C15B	1.702 (3)
C6A—F6A	1.335 (4)	C6B—F6B	1.330 (4)
C11A—N1A	1.117 (5)	C11B—N1B	1.126 (4)
C13A—N3A	1.105 (5)	C13B—N3B	1.133 (5)
C2A—C1A—C6A	117.1 (3)	C2B—C1B—C6B	117.6 (3)
C1A—C2A—C3A	122.2 (3)	C1B—C2B—C3B	122.0 (3)
C2A—C3A—C4A	117.4 (3)	C2B—C3B—C4B	117.6 (3)
C3A—C4A—C5A	123.0 (3)	C3B—C4B—C5B	123.0 (3)
C4A—C5A—C6A	116.7 (3)	C4B—C5B—C6B	116.6 (3)
C1A—C6A—C5A	123.6 (3)	C1B—C6B—C5B	123.1 (3)
C1A—C11A—N1A	179.1 (5)	C1B—C11B—N1B	177.2 (4)
C3A—C13A—N3A	179.5 (4)	C3B—C13B—N3B	178.2 (4)

The thermal ellipsoids and the labelling of the atoms are shown for molecule *A* in Fig. 1; a similar figure for molecule *B* is included in the Supplementary Material. Bond lengths and angles are given in Table 2; they are normal within experimental error. The ring in molecule *A* is planar within experimental error, but the substituents are bent out of the mean plane of the ring by +0.4, +1.3, -0.1, -1.4, +0.4, and +1.4° on atoms C1A to C6A, respectively, with e.s.d.'s of approximately 0.3°. The ring in molecule *B* is slightly puckered, with the substituents bent out of

the mean plane of the ring by -2.6, +2.4, +0.4, -1.2, +1.0, and -0.3° on atoms C1B to C6B, respectively.

The packing is shown in Fig. 2. With the van der Waals radii given by Nyburg & Faerman (1985) plus 1.70 Å as the radius for carbon, there are six intermolecular distances shorter than the expected van der Waals distances. Only three of these, however, involve a shortening of more than 0.1 Å. These are: the 1A nitrile pointing towards the middle of a C4B—F4B bond with an N1A...C4B distance of 3.022 (5) Å; the 1B nitrile pointing towards the middle of a C6A—F6A bond with an N1B...C6A distance of 3.134 (5) Å; an N3B...C15B contact of 3.162 (4) Å where the C13B—N3B...C15B angle is 124.5 (4)° and the N3B...C15B—C5B angle is 176.8 (5)°.

Related literature. Structures of tetrafluorophthalonitrile (Britton, 1988), tetrafluoroterephthalonitrile (van Rij & Britton, 1981; Dunitz, Schweizer, & Seiler, 1982; Seiler, Schweizer, & Dunitz, 1984), tetrachlorophthalonitrile (Britton, 1981a), tetrachloroisophthalonitrile and -terephthalonitrile (Britton, 1981b).

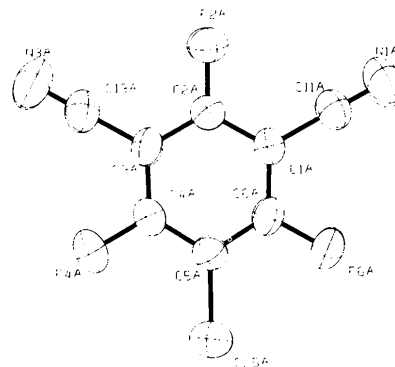


Fig. 1. $\text{ClF}_3\text{C}_6(\text{CN})_2$, molecule *A*. Thermal ellipsoids are shown at the 50% probability level. Molecule *B* does not look significantly different.

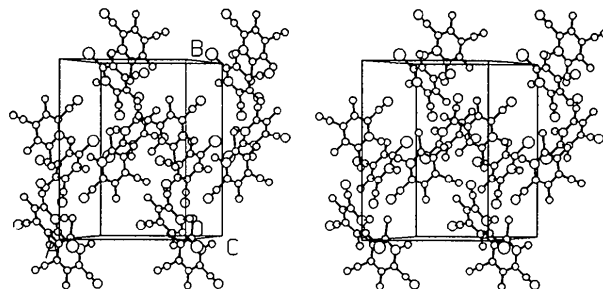


Fig. 2. The packing of $\text{ClF}_3\text{C}_6(\text{CN})_2$. The atoms involved in the short intermolecular contacts, N1A...C4B, N1B...C6A, and N3B...C15B, are shown slightly larger than the remaining atoms.

I thank Dr Robert Battershell for the sample of the compound.

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Acta Cryst. (1991). **C47**, 2474–2476

Redetermination of the Structure of Guanine Hydrochloride Monohydrate

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(Received 30 July 1990; accepted 1 May 1991)

Abstract. $C_5H_6N_5O^+ \cdot Cl^- \cdot H_2O$, $M_r = 205.6$, monoclinic, $P2_1/c$, $a = 4.479$ (1), $b = 9.995$ (1), $c = 19.304$ (2) Å, $\beta = 107.90$ (1)°, $V = 819.1$ (2) Å³, $Z = 4$, $D_m = 1.664$, $D_x = 1.667$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 4.38$ cm⁻¹, $F(000) = 424$, $T = 293$ (1) K, $R = 0.034$, $wR = 0.043$ for 1736 observed reflections with $I > 1.96 \sigma(I)$. The guanine molecule is non-planar with a dihedral angle of 1.7 (1.5)° between the imidazole and the pyrimidine rings. The base rings are protonated at N(7) and form a hydrogen network with two modes of pairing [N(7)—H7···O(1) and N(10)—H101···N(3)]. Several interactions of the type $D—H \cdots A$ are noted between purine cations, water and chlorine anions.

Experimental. Crystals were grown by cooling a solution of the title compound in 18% hydrochloric acid in a special crystallizer with reversible stirring of the crystal seeds (starting temperature was about 308 K, cooling gradient 0.1 K per day). As grown the crystals are isometric, colourless and very often twinned, with maximal crystal dimensions of $5 \times 5 \times 3$ mm. The density was determined by flotation in toluene–methylene iodide mixtures. A crystal of size $0.2 \times 0.3 \times 0.45$ mm was selected and mounted on an Enraf–Nonius CAD-4 diffractometer. The unit-cell parameters were determined by a least-squares fit of the adjusted angular settings of 25 reflections with $17 < \theta < 18^\circ$ constrained to a monoclinic cell. The space group was determined by systematic absences ($0k0$

with k odd, $h0l$ with l odd) in the diffractometer-collected intensity data. Intensities were collected with graphite-monochromated Mo $K\alpha$ radiation. The $\omega-2\theta$ technique was used with a scan width of $(0.75 + 0.35 \tan \theta)^\circ$ extended 25% on each side for background measurement. The set of 2234 unique reflections, 1736 of which had $I > 1.96 \sigma(I)$, was obtained from 2810 reflections measured in the range $1 < 2\theta < 60^\circ$ ($0 < h < 8$, $0 < k < 13$, $-30 < l < 30$, $\sin \theta / \lambda < 0.7027$ Å⁻¹, $R_{int} = 0.02$). The angular positions of three standard reflections (0,0,18, 0,10,0, 5,2,14) were checked after measuring 500 reflections, the intensity of reflection 121 was monitored after every 6000 s. The position of the crystal was stable and the monitored intensities showed a very slight decrease of about 0.2%. The intensity data were corrected for Lorentz and polarization but not for absorption effects.

The structure was solved from Patterson and $\Delta\rho$ maps and refined on F by the full-matrix least-squares method with anisotropic thermal motion. All H-atom positions were found from $\Delta\rho$ maps and included in the model. Final refinement was performed with 151 variables including all positional parameters, anisotropic displacement parameters for the non-H atoms, isotropic displacement parameters for the H atoms, one scale factor and the isotropic secondary-extinction coefficient of 4.3×10^{-7} . The final agreement factors were $R = 0.034$, $wR = 0.043$ and $S = 1.42$. The ratio of the maximum least-