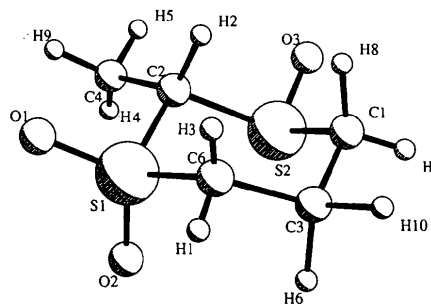


Table 1. Fractional coordinates and B_{eq} values with their standard deviations
$$B_{eq} = 8\pi^2 U_{eq} = 8\pi^2/3 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

	x	y	z	B_{eq} (\AA^2)
S(1)	0.5775 (1)	0.34572 (7)	0.5608 (2)	2.81 (4)
S(2)	0.4468 (1)	0.47063 (7)	0.8954 (2)	2.99 (5)
O(1)	0.6043 (4)	0.3354 (2)	0.3292 (5)	4.3 (2)
C(1)	0.3193 (5)	0.3753 (3)	0.9216 (8)	3.4 (2)
C(2)	0.4880 (4)	0.4551 (3)	0.6013 (7)	2.7 (2)
C(3)	0.3932 (5)	0.2817 (3)	0.8824 (8)	3.3 (2)
C(4)	0.5853 (6)	0.5319 (3)	0.5186 (9)	4.4 (2)
C(6)	0.4409 (5)	0.2671 (3)	0.6444 (8)	3.6 (2)
O(2)	0.7012 (3)	0.3396 (2)	0.7066 (6)	4.0 (1)
O(3)	0.3544 (3)	0.5562 (2)	0.9034 (6)	4.1 (1)

Table 2. Selected bond lengths (\AA) and angles ($^\circ$)

S(1)	O(1)	1.432 (3)	S(2)	C(2)	1.834 (4)		
S(1)	C(2)	1.798 (4)	S(2)	O(3)	1.497 (3)		
S(1)	C(6)	1.754 (5)	C(1)	C(3)	1.533 (6)		
S(1)	O(2)	1.426 (3)	C(3)	C(6)	1.520 (7)		
S(2)	C(1)	1.807 (4)	C(2)	C(4)	1.505 (6)		
O(1)	S(1)	C(2)	107.6 (2)	S(2)	C(1)	C(3)	112.9 (3)
O(1)	S(1)	C(6)	109.5 (2)	S(2)	C(2)	C(4)	110.5 (3)
O(1)	S(1)	O(2)	117.9 (2)	C(1)	C(3)	C(6)	113.2 (4)
C(2)	S(1)	C(6)	102.7 (2)	S(1)	C(2)	S(2)	109.4 (2)
C(2)	S(1)	O(2)	108.8 (2)	S(1)	C(2)	C(4)	110.3 (3)
C(6)	S(1)	O(2)	109.3 (2)	S(1)	C(6)	C(3)	112.5 (3)
C(1)	S(2)	C(2)	96.9 (2)				
C(1)	S(2)	O(3)	106.2 (2)				
C(2)	S(2)	O(3)	104.3 (2)				

Fig. 1. One molecule of the title compound, illustrating the structure and the atom numbering (*PLUTO*; Motherwell & Clegg, 1978).

References

- Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. Molecular Structure Corporation, Texas, USA.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- PAGE, P. C. B. & NAMWINDWA, E. S. (1991). *Syn. Lett.* pp. 80–83.
- SHELDRIK, G. M. (1986). *SHELXS86*. Program for crystal structure solution. Univs. of Göttingen, Germany.

Acta Cryst. (1992). **C48**, 2040–2042

Structure of Tetrafluoroisophthalonitrile*

BY DOYLE BRITTON

Department of Chemistry, University of Minnesota, Minneapolis, MN 55455, USA

(Received 3 December 1991; accepted 26 February 1992)

Abstract. $C_6F_4(CN)_2$, $M_r = 200.10$, orthorhombic, *Pbca*, $a = 13.343$ (7), $b = 21.991$ (5), $c = 10.462$ (3) \AA , $Z = 16$ (two molecules in the asymmetric unit), $V = 3070$ (4) \AA^3 , $D_x = 1.732$ (2) g cm^{-3} , Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA , $\mu = 1.68$ cm^{-1} , $F(000) = 1568$, $T = 172$ (3) K, $R = 0.056$ for 2671 unique observed reflections with $I > \sigma(I)$. The bond lengths and angles are normal, except that both rings are slightly puckered and in both molecules both nitrile groups are bent out of the mean plane of the ring by 2 to 3°. Five 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 $0.40 \times 0.45 \times 0.45$ 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. 25 reflections with $11 < \theta < 27^\circ$ were used to determine the cell parameters. Systematic extinctions ($0kl$, k odd; $h0l$, l odd; $hk0$, h odd) uniquely determined the space group. Data were collected, using ω scans, in the range $0 < \theta < 28^\circ$ for one quadrant (ranges: h , 0 to 17; k , 0 to 29; l , –12 to 12). The intensities of 7174 different reflections were measured. Three check reflections measured every 4500 s of exposure time showed no systematic change over the course of the data collection. The structure was solved by direct methods (*MITHRIL*; Gilmore, 1984) and refined with isotropic thermal parameters. After absorption corrections were made, using the program *DIFABS* (Walker & Stuart, 1983; transmis-

* 2,4,5,6-Tetrafluoro-1,3-benzenedicarbonitrile

Table 1. Positional and isotropic equivalent thermal parameters

$$B_{\text{eq}} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
C1A	0.2800 (2)	0.3971 (1)	0.2827 (2)	2.1 (1)
C2A	0.2493 (2)	0.4374 (1)	0.1892 (2)	2.03 (9)
C3A	0.1577 (2)	0.4673 (1)	0.1953 (2)	2.1 (1)
C4A	0.0973 (2)	0.4556 (1)	0.3004 (2)	2.5 (1)
C5A	0.1249 (2)	0.4153 (1)	0.3943 (2)	2.8 (1)
C6A	0.2156 (2)	0.3864 (1)	0.3846 (2)	2.5 (1)
C11A	0.3773 (2)	0.3691 (1)	0.2777 (2)	2.5 (1)
N1A	0.4545 (2)	0.3467 (1)	0.2741 (2)	3.2 (1)
C33A	0.1288 (2)	0.5109 (1)	0.1005 (2)	2.6 (1)
N3A	0.1052 (2)	0.5457 (1)	0.0261 (2)	3.6 (1)
F2A	0.3093 (1)	0.44843 (6)	0.0904 (1)	2.86 (6)
F4A	0.0096 (1)	0.48418 (7)	0.3105 (1)	3.57 (7)
F5A	0.0632 (1)	0.40393 (8)	0.4932 (1)	4.20 (8)
F6A	0.2426 (1)	0.34728 (7)	0.4752 (1)	3.72 (7)
C1B	0.4094 (2)	0.2920 (1)	0.6562 (2)	2.0 (1)
C2B	0.4693 (2)	0.3400 (1)	0.6935 (2)	2.1 (1)
C3B	0.5603 (2)	0.3526 (1)	0.6357 (2)	2.2 (1)
C4B	0.5900 (2)	0.3152 (1)	0.5355 (2)	2.2 (1)
C5B	0.5331 (2)	0.2667 (1)	0.4970 (2)	2.2 (1)
C6B	0.4438 (2)	0.2556 (1)	0.5562 (2)	2.1 (1)
C11B	0.3172 (2)	0.2781 (1)	0.7206 (2)	2.2 (1)
N1B	0.2456 (2)	0.2651 (1)	0.7731 (2)	3.2 (1)
C33B	0.6246 (2)	0.4009 (1)	0.6787 (3)	3.1 (1)
N3B	0.6763 (2)	0.4382 (1)	0.7136 (3)	4.8 (1)
F2B	0.4385 (1)	0.37543 (6)	0.7894 (1)	2.92 (6)
F4B	0.6766 (1)	0.32633 (7)	0.4764 (1)	3.19 (7)
F5B	0.5641 (1)	0.23110 (6)	0.4012 (1)	3.13 (7)
F6B	0.3877 (1)	0.20911 (6)	0.5173 (1)	3.05 (6)

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

C1A—C2A	1.383 (3)	C1B—C2B	1.382 (3)
C1A—C6A	1.389 (3)	C1B—C6B	1.395 (3)
C1A—C11A	1.436 (3)	C1B—C11B	1.435 (3)
C2A—C3A	1.389 (3)	C2B—C3B	1.385 (3)
C2A—F2A	1.330 (2)	C2B—F2B	1.335 (2)
C3A—C4A	1.388 (3)	C3B—C4B	1.389 (3)
C3A—C33A	1.432 (3)	C3B—C33B	1.439 (3)
C4A—C5A	1.374 (3)	C4B—C5B	1.371 (3)
C4A—F4A	1.331 (3)	C4B—F4B	1.333 (3)
C5A—C6A	1.370 (3)	C5B—C6B	1.365 (3)
C5A—F5A	1.346 (3)	C5B—F5B	1.337 (2)
C6A—F6A	1.331 (3)	C6B—F6B	1.330 (3)
C11A—N1A	1.143 (3)	C11B—N1B	1.139 (3)
C33A—N3A	1.137 (3)	C33B—N3B	1.131 (3)
C2A—C1A—C6A	117.8 (2)	C2B—C1B—C6B	117.4 (2)
C1A—C2A—C3A	122.2 (2)	C1B—C2B—C3B	122.5 (2)
C2A—C3A—C4A	117.4 (2)	C2B—C3B—C4B	117.5 (2)
C3A—C4A—C5A	122.0 (2)	C3B—C4B—C5B	121.6 (2)
C4A—C5A—C6A	118.9 (2)	C4B—C5B—C6B	119.3 (2)
C1A—C6A—C5A	121.7 (2)	C1B—C6B—C5B	121.7 (2)
C1A—C11A—N1A	179.7 (3)	C1B—C11B—N1B	177.4 (3)
C3A—C33A—N3A	179.3 (3)	C3B—C33B—N3B	178.7 (3)

sion factors 0.73 to 1.10), equivalent reflections were combined to give 3689 independent reflections ($R_{\text{int}} = 0.045$) of which the 2671 with $I > \sigma(I)$ were used in the calculations. The structure was refined with full-matrix least-squares techniques on F . There are two molecules in the asymmetric unit. All atoms were given anisotropic thermal parameters. Refinement converged with $R = 0.056$, $wR = 0.042$, and $S = 1.20$; $w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I_c) + (0.03I)^2$, where $\sigma(I_c)$ is the standard deviation in I based on counting statistics alone. In the

final cycle of refinement $(\Delta/\sigma)_{\text{max}} = 0.01$, $(\Delta\rho)_{\text{max}} = 0.36$ and $(\Delta\rho)_{\text{min}} = -0.28 \text{ e \AA}^{-3}$. 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.* The thermal ellipsoids and the labeling of the atoms are shown for molecule *A* in Fig. 1; a similar figure for molecule *B* has been deposited. Bond lengths and angles are given in Table 2; they are normal within experimental error. The rings in both molecules are slightly puckered, and the substituents are bent out of the mean plane of the ring by $+3.2$, $+0.3$, $+1.8$, $+1.1$, -1.1 and -0.7° on atoms C1A

* 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 55255 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0414]

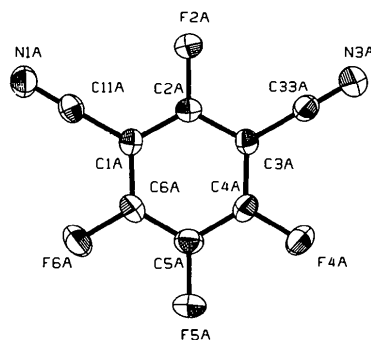


Fig. 1. $\text{C}_6\text{F}_4(\text{CN})_2$, molecule *A*. Thermal ellipsoids are shown at the 50% probability level. Molecule *B* is not significantly different.

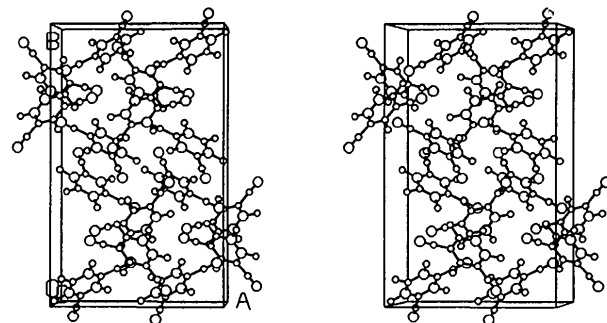


Fig. 2. The packing of $\text{C}_6\text{F}_4(\text{CN})_2$. The atoms involved in the short intermolecular distances, $\text{C4A}\cdots\text{N1A}\cdots\text{C5B}$, $\text{N3A}\cdots\text{C6A}$, $\text{N1B}\cdots\text{C4B}$, $\text{N3B}\cdots\text{C2A}$, are shown slightly larger than the remaining atoms.

to C6A, respectively, and by +1.8, +0.2, +2.8, -0.8, +0.3, and -0.4° on atoms C1B to C6B, respectively, with e.s.d.'s of approximately 0.2°.

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 C, there are eight intermolecular distances shorter than the expected van der Waals distances, but only five of these involve a shortening of more than 0.1 Å. N1A is close to ring atoms in two different rings with an N1A...C4A distance of 3.158 (3) Å and an N1A...C5B distance of 3.104 (3) Å. N3A is in a similar situation but only the N3A...C6A distance of 3.184 (3) Å is short. The 1B nitrile group is more or less pointing towards a ring atom with an N1B...C4B distance of 3.089 (3) Å and a C11B—N1B...C4B angle of 141.9 (2)°. The 3B nitrile group also points toward a ring atom with an N3B...C2A distance of 3.083 (3) Å and a C33B—N3B...C2A angle of 160.9 (2)°.

Related literature. Structures of tetrafluorophthalonitrile (Britton, 1988), tetrafluoroterephthalonitrile

(Van Rij & Britton, 1981; Dunitz, Schweizer & Seiler, 1982; Seiler, Schweizer & Dunitz, 1984), and 5-chloro-2,4,6-trifluoroisophthalonitrile (Britton, 1991).

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

References

- BRITTON, D. (1988). *Acta Cryst.* **C44**, 1020–1022.
 BRITTON, D. (1991). *Acta Cryst.* **C47**, 2472–2474.
 DUNITZ, J. D., SCHWEIZER, W. B. & SEILER, P. (1982). *Helv. Chim. Acta*, **66**, 123–133.
 GILMORE, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
 Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.
 NYBURG, S. C. & FAERMAN, C. H. (1985). *Acta Cryst.* **B41**, 274–279.
 SEILER, P., SCHWEIZER, W. B. & DUNITZ, J. D. (1984). *Acta Cryst.* **B40**, 319–327.
 VAN RIJ, C. & BRITTON, D. (1981). *Cryst. Struct. Commun.* **10**, 175–178.
 WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1992). **C48**, 2042–2043

2-[(*o*-Hydroxyphenyl)imino]methylpyridine

BY LAWRENCE B. ORR JR, EDITH J. PARSONS* AND WILLIAM T. PENNINGTON*

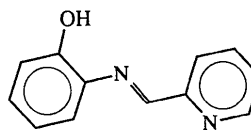
Department of Chemistry, Clemson University, Clemson, SC 29634-1905, USA

(Received 26 September 1991; accepted 31 January 1992)

Abstract. 2-[(2-Pyridylmethylene)amino]phenol, C₁₂H₁₀N₂O (1), *M_r* = 198.22, orthorhombic, *P*2₁2₁2₁, *a* = 4.722 (2), *b* = 12.284 (5), *c* = 17.638 (11) Å, *V* = 1023.0 (9) Å³, *Z* = 4, *D_x* = 1.29 Mg m⁻³, graphite-monochromated Mo *K*α radiation, λ = 0.71073 Å, μ = 0.08 mm⁻¹, *F*(000) = 416, *T* = 294 (1) K, *R* = 0.048 for 608 observed reflections. The relative planarity of the molecule (mean deviation = 0.049 Å) results in a close interaction between the proton at the *ortho* position of the phenyl ring [H(5)] and the proton of the imino group [H(6)]. This steric crowding is alleviated by expansion of the N(1)—C(6)—C(5) angle and by displacement of the two protons by 0.13 Å to opposite sides of the molecular plane. The *ortho*-hydroxy proton is involved in intramolecular hydrogen bonding to the imino N atom [H(1)...N(1) = 2.03 (8); O(1)...N(1) = 2.686 (7) Å; O(1)—H(1)...N(1) = 122 (7)°] and intermolecular

hydrogen bonding to the *ortho*-hydroxy O atom of an adjacent molecule [H(1)...O(1') = 2.30 (9); O(1)...O(1') = 2.987 (7) Å; O(1)—H(1)...O(1') = 126 (8)°]. The intermolecular hydrogen bonding links molecules into infinite chains; adjacent molecules in a chain are related by a 2₁ screw operation along the *a* axis.

Experimental. The title compound (1) was synthesized by literature methods (Pitt, Bao, Thompson, Wani, Rosenkrantz & Metterville, 1986). A yellow platey crystal, 0.05 × 0.40 × 0.60 mm, was used for data collection on a Nicolet R3m/V diffractometer,



(1)

* Authors to whom correspondence should be addressed.