Slice (1$\bar{1}$0) exhibits a behaviour intermediate between (100), the stunted face, and ($\bar{1}$01), the *R* and *S* nucleation plane (Coquerel, Perez & Hartman, 1988).

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Tetrafluorophthalonitrile*

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Abstract. $C_6F_4(CN)_2$, $M_r = 200{\cdot}10$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}826\ (3)$, $b = 12{\cdot}320\ (5)$, $c = 5{\cdot}819\ (5)$ Å, $Z = 4$, $V/Z = 194{\cdot}0\ (3)$ Å^3, $D_x = 1{\cdot}712\ (3)$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}91$ cm^{-1}, $F(000) = 392$, $T = 295\ (2)$ K, $R = 0{\cdot}051$ for 860 reflections. The bond lengths and angles in the molecule are normal although the molecule deviates slightly from planarity. The only intermolecular contacts shorter than van der Waals distances are both between N atoms and ring C atoms in adjacent molecules, N1···C3 3·182 (3) and N2···C2, 3·178 (3) Å.

Introduction. As part of a study of intermolecular donor–acceptor interactions in crystals we have previously determined, among others, the crystal structures and packing of *p*-$C_6H_4(CN)_2$ (van Rij & Britton, 1977; see also Drück & Littke, 1978; Colapietro, Domenicano, Portalone, Schultz & Hargittai, 1984), *p*-$C_6F_4(CN)_2$ (van Rij & Britton, 1981; see also Dunitz, Schweizer & Seiler, 1982; Seiler, Schweizer & Dunitz, 1984), and all three isomers of $C_6Cl_4(CN)_2$ (Britton, 1981*a*,*b*). The nitrile group can act as a Lewis base and frequently does in intermolecular interactions. In *p*-$C_6H_4(CN)_2$, however, there is no good Lewis-acid site, and the primary interaction of the nitrile group occurs in an antiparallel alignment with an adjacent nitrile group. In *p*-$C_6F_4(CN)_2$ the Lewis-acid site occurs at the ring C atom attached to the CN group. In the $C_6Cl_4(CN)_2$ isomers there are two interactions (short contacts) between a nitrile group and the C atom attached to the CN group, but most of the interactions are between nitrile groups and Cl atoms in adjacent molecules. That is to say, the Cl atoms are better Lewis-acid sites than the C atoms. We report here an extension of this work to *o*-$C_6F_4(CN)_2$ with our particular interest being the packing arrangement.

Experimental. The compound was obtained from PCR Inc.; crystals suitable for diffraction experiments were present in the original sample. D_m was not measured. A crystal 0·25 × 0·25 × 0·4 mm was used for the data collection. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 24 reflections with $13 < \theta < 15°$ were used to determine the cell parameters. Systematic extinctions ($h00$, h odd; $0k0$, k odd; $00l$, l odd) uniquely determined the space group. Data were collected, using ω scans, in the range $0 < \theta < 28°$ for one octant (ranges: h, 0 to 14; k, 0 to 16; l, 0 to 7). 1116 independent reflections were measured of which the 860 with $I > \sigma(I)$ were used in the calculations. Three check

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

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reflections measured every 5000 s of exposure time showed no systematic change with time. No absorption corrections were made. The structure was solved by direct methods and refined with full-matrix least-squares refinement on F's. All the atoms were given anisotropic thermal parameters. Refinement converged with $R = 0{\cdot}051$, $wR = 0{\cdot}041$ and $S = 1{\cdot}429$; $w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I)_c + (0{\cdot}03I)^2$, where $\sigma(I)_c$ is the standard deviation in I based on counting statistics alone. $(\Delta/\sigma)_{\max}$ in the final cycle of refinement was 0·13. $(\Delta\rho)_{\max} = 0{\cdot}087$, $(\Delta\rho)_{\min} = 0{\cdot}114$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974). The computer programs used were all from the Enraf–Nonius *Structure Determination Package* and have been described by Frenz (1978).

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (B_{11} + B_{22} + B_{33})/3.$

	x	y	z	B_{eq} (Å^2)
F3	0·2682 (1)	0·2395 (1)	0·6009 (3)	6·1
F4	0·4404 (2)	0·2193 (1)	0·9310 (3)	6·4
F5	0·6435 (2)	0·3463 (1)	0·9254 (4)	6·6
F6	0·6758 (1)	0·4926 (1)	0·5898 (4)	6·2
N1	0·5151 (3)	0·5832 (2)	0·0939 (5)	6·9
N2	0·2206 (3)	0·3973 (2)	0·1026 (6)	7·2
C1	0·4887 (2)	0·4424 (2)	0·4167 (5)	3·9
C2	0·3824 (2)	0·3770 (2)	0·4208 (5)	3·8
C3	0·3680 (2)	0·3029 (2)	0·5939 (6)	4·3
C4	0·4563 (3)	0·2914 (2)	0·7632 (5)	4·4
C5	0·5592 (2)	0·3560 (2)	0·7605 (5)	4·4
C6	0·5750 (2)	0·4310 (2)	0·5885 (6)	4·2
C11	0·5038 (3)	0·5220 (2)	0·2381 (6)	4·9
C22	0·2909 (3)	0·3875 (2)	0·2455 (6)	5·0

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

The table is arranged so that equivalent values, for the isolated molecule, occur on the same line.

C(1)–C(2)	1·405 (3)		
C(2)–C(3)	1·368 (4)	C(6)–C(1)	1·375 (4)
C(3)–C(4)	1·380 (4)	C(5)–C(6)	1·372 (4)
C(4)–C(5)	1·370 (3)		
C(2)–C(22)	1·428 (4)	C(1)–C(11)	1·438 (4)
C(22)–N(2)	1·134 (4)	C(11)–N(1)	1·134 (4)
C(3)–F(3)	1·334 (3)	C(6)–F(6)	1·330 (2)
C(4)–F(4)	1·331 (3)	C(5)–F(5)	1·330 (3)
C(1)–C(2)–C(3)	119·3 (2)	C(6)–C(1)–C(2)	119·0 (2)
C(2)–C(3)–C(4)	121·0 (2)	C(5)–C(6)–C(1)	121·0 (2)
C(3)–C(4)–C(5)	119·7 (2)	C(4)–C(5)–C(6)	120·1 (3)
C(1)–C(2)–C(22)	120·3 (2)	C(2)–C(1)–C(11)	119·8 (3)
C(3)–C(2)–C(22)	120·5 (2)	C(6)–C(1)–C(11)	121·2 (2)
C(2)–C(3)–F(3)	120·4 (3)	C(1)–C(6)–F(6)	120·2 (2)
C(4)–C(3)–F(3)	118·6 (3)	C(5)–C(6)–F(6)	118·8 (3)
C(3)–C(4)–F(4)	120·2 (2)	C(6)–C(5)–F(5)	120·1 (2)
C(5)–C(4)–F(4)	120·1 (3)	C(4)–C(5)–F(5)	119·9 (3)
C(2)–C(22)–N(2)	178·0 (3)	C(1)–C(11)–N(1)	178·5 (3)

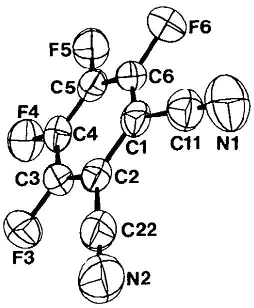

Fig. 1. o-$C_6F_4(CN)_2$. The atoms are shown in the same orientation as those at the center of Fig. 2. Thermal ellipsoids are shown at the 50% probability level.

Discussion. The final positional parameters are given in Table 1.* The thermal ellipsoids and the labelling of the atoms are shown in Fig. 1. Bond lengths and angles are given in Table 2; they are all normal within experimental error; pairs of distances and angles that should be identical in the isolated molecule agree satisfactorily. The molecule deviates slightly but significantly from planarity. The largest deviation is the C11 atom 0·019 (3) Å out of the plane of the benzene ring. There is no obvious pattern to the deviations; apparently small deviations due to intramolecular crowding are mixed with small deviations due to packing effects.

The packing is shown in Fig. 2. The shortest intermolecular contact is an F···F distance of 2·95 (1) Å, but this is slightly longer than the expected van der Waals contact distance. If we use the van der Waals radii of Pauling (1960), which are for C 1·70, for N 1·50 and for F 1·35 Å, only three distances are close to the expected values. These are N1···C3, 3·182 (3) Å, between two molecules related by a 2_1 axis parallel to **b**, and N2···C1, 3·195 (3), N2···C2, 3·178 (3) Å, between two molecules related by a 2_1 axis parallel to **c**. None of these distances is remarkably short; nevertheless, they appear to be the most significant intermolecular interactions in the structure. Both are shorter than the N···C distance of 3·31 Å in the *para* isomer (van Rij & Britton, 1981), but they are

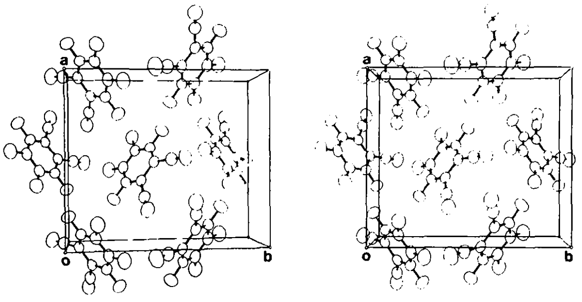

Fig. 2. The packing of o-$C_6F_4(CN)_2$. View approximately along **c**.

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* Lists of anisotropic thermal parameters, deviations from the mean plane, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44748 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

comparable to the 3·15 and 3·11 Å distances found in the N···C contacts in *m*- and *p*-$C_6Cl_4(CN)_2$ (Britton, 1981*a*). In the chlorine compound that is most directly analogous to this one, namely *o*-$C_6Cl_4(CN)_2$, the molecules pack in planar layers where all of the short contacts are between N and Cl atoms (Britton, 1981*b*).

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Structure of 3-*O*-Acetylgibberellin A_3

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Abstract. $C_{21}H_{24}O_7$, $M_r = 388·4$, orthorhombic, $P2_12_12_1$, $a = 13·279$ (10), $b = 15·615$ (10), $c = 9·427$ (8) Å, $V = 1954·7$ Å^3, $Z = 4$, $D_x = 1·319$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71069 Å, $\mu = 1·0$ cm^{-1}, $F(000) = 824$, $T = 293$ K, $R = 0·049$ for 1805 observed reflections. The gibberellane skeleton shows the conformational variety characteristic of many other gibberellin derivatives. Ring *A* is in a sofa conformation while the five-membered ring *B* assumes an intermediate conformation between an envelope and a half-chair. The lactone ring is in an ideal envelope conformation and shows a marked difference in its two C—O distances. Each molecule is hydrogen bonded to two others to form continuous spiral chains along the *c* axis.

Introduction. The gibberellins are a large group of plant hormones isolated from culture filtrates of the fungus *Gibberella fujikuroi.* Among these active fungal metabolites, gibberellin A_3 (or gibberellic acid) is known to be the most important metabolite of the fungus *Gibberella fujikuroi* and has been the subject of exhaustive chemical studies (Curtis & Cross, 1954; Stork & Newman, 1959; Grove, 1961; Sheppard, 1960). All gibberellins are diterpenoid acids based on the tetracyclic gibberellane skeleton. The structure and absolute stereochemistry of gibberellin A_3 have been determined from X-ray studies of some of its derivatives (Hartsuck & Lipscomb, 1963; McCapra, McPhail, Scott, Sim & Young, 1966). During the course of our continuing investigation of natural products from marine organisms, we encountered an extract from the marine boring sponge *Cliona caribboea* which was later identified as a gibberellin derivative, 3-*O*-acetylgibberellin A_3. The crystal structure was carried out to characterize the compound fully.

Experimental. Thick platy crystals grown from methanol solution, 0·90 × 0·35 × 0·12 mm; space group from systematic absences; CAD-4 single-crystal X-ray diffractometer, Mo $K\bar{\alpha}$ radiation (graphite monochromator), lattice parameters from setting of 48 reflections with $9 < \theta < 13°$. 2311 unique reflections with $2\theta \le 53°$; $0 \le h \le 14$, $0 \le k \le 15$, $0 \le l \le 11$; θ–2θ scan, scan width $(0·80 + 0·20 \tan\theta)°$, extended 25% on each side for background measurement, horizontal aperture $(3·0 + 0·86 \tan\theta)$ mm, vertical

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