Although most of the atoms of these rings do not superimpose on each other, the distance between their mean planes is 3·468 (2) Å, a value slightly larger than would be expected for an appreciable charge-transfer complex to be present. Dipole–dipole interactions may play an essential role in stabilizing this system. Couples of *n*-glide-related molecules form aggregates of four molecules around the twofold axes and the space between their protruding side chains may be filled by the hydroxyl group when assuming the minor conformation. The 0·25 occupancy factor attributed to O(99) may be accounted for by assuming a statistically disordered hydroxyl group for each set of four molecules so arranged. The four broken circles in Fig. 2 represent the four possible positions that may be occupied in turn by O(99).

The hydroxyl group plays an important role in the hydrogen-bond network, acting as a donor in both conformations. O(11) of a molecule at x, y, z forms an intermolecular hydrogen bond with the carbonylic O(4) of another molecule at $-\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}-z$ with the following geometrical features: O(11)···O(4) = 2·786 (3), H···O(4) = 1·87 (3) Å, O(11)—H···O(4) = 154 (0), C(4)—O(4)···H = 142 (0), C(4)—O(4)···O(11) = 151·0 (3), C(11)—O(11)···O(4) = 118·5 (3), [O(11)—H—O(4)—C(4)] = −168 (1)°, where H represents the hydrogen bound to O(11) found in the final difference Fourier. The hydroxyl O(11) at x, y, z is also an acceptor of an intermolecular hydrogen bond with the hydroxylic O(99) of another molecule at $-\frac{1}{2}+y, \frac{1}{2}+x, \frac{1}{2}+z$ with the following geometrical features: O(11)···O(99) = 2·86 (1) Å, C(11)—O(11)···O(99) = 117·9 (4) and O(4)···O(11)···O(99) = 100·8 (3)°; the values including the H atom have not been computed since this could not be detected from the final difference Fourier.

The disordered hydroxyl group does not cause weakening of the crystal packing forces. In fact, when an O(11) atom is replaced by an O(99) atom, the O(4)···O(11) hydrogen bond is replaced by the energetically similar O(11)···O(99) hydrogen bond. Furthermore, while the hydrogen bond between the hydroxylic and carbonylic groups allows the packing of the molecules in the *ab* plane, that between the hydroxyl groups of the two different conformers has a large component perpendicular to this plane.

The ^{13}C NMR spectrum was recorded on the Bruker WP 200 of the CNR Research Area (Montelibretti). Mr M. Viola is thanked for the drawings. Financial support from the Ministero della Pubblica Istruzione is also acknowledged.

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Structure of (*R*)-Norfenfluramine Dichloroacetate*

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Abstract. $C_{10}H_{13}F_3N^+.C_2HCl_2O_2^-$, $M_r = 332·16$, monoclinic, $P2_1$, $a = 13·359$ (5), $b = 6·810$ (3), $c = 8·447$ (3) Å, $\beta = 107·55$ (2)°, $V = 732·7$ (6) Å^3, $Z = 2$, D_m (by flotation) = 1·509, $D_x = 1·506$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 4·17$ cm^{-1}, $F(000) = 340$, $T = 294$ K, final $R = 0·058$ for 1793 observed reflections with $I > 3\sigma(I)$. The conformation of the protonated amine is extended. The poisoned faces (100) and ($\bar{1}00$) of the enantiomeric crystal in racemic supersaturated solutions contain the achiral anion and the group $-NH_3^+$.

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[*] Norfenfluramine is α-methyl-3-(trifluoromethyl)benzene-ethanamine (*Chemical Abstracts* name).

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Introduction. Optical resolution by preferential crystallization can be carried out if a mixture of enantiomers is a eutectic without solid solution (*i.e.* conglomerate). Usually the absence of a racemate is sufficient to allow separation by this procedure (Collet, Brienne & Jacques, 1980; Jacques, Collet & Wilen, 1981; Asai & Ikegami, 1982). This method of resolution has been studied recently for norfenfluramine and fenfluramine derivates (Coquerel, 1986). Among the ten conglomerates studied norfenfluramine dichloroacetate is the only one which cannot be used for optical resolution. This particular behaviour can be explained by the crystal growth processes in supersaturated and quasi-racemic solution. Strong growth-inhibiting phenomena take place on the (100) and ($\bar{1}00$) faces, and a heterogeneous nucleation occurs on the other faces. The purpose of this study is to elucidate the nature of the faces involved in the poisoning and heterogeneous nucleation phenomena, in the hope of avoiding them by adding selective impurities.

Experimental. The compound is prepared by direct neutralization of an ethanolic solution of (*R*)-norfenfluramine (Oril, France) by dichloroacetic acid (Prolabo Rhone Poulenc). Two recrystallizations in the same solvent at room temperature lead to good monocrystals: parallelepipedic crystal: $0{\cdot}2 \times 0{\cdot}3 \times 0{\cdot}5$ mm. The habit of the crystals is elongated along **b**; the most developed faces are (100) and ($10\bar{1}$) which are all parallel to **b**; the faces ($1\bar{1}0$) are also present but less developed.

X-ray measurements made with Philips PW1100 diffractometer using Mo $K\bar{\alpha}$ radiation. Unit-cell dimensions from least-squares refinement for 15 reflections, $9{\cdot}62 < \theta < 13{\cdot}84°$. Intensity data collected using ω–2θ scans, $(1{\cdot}5 + 0{\cdot}35\tan\theta)°$, $4 < 2\theta < 60°$, $-18 < h < 18$, $0 < k < 9$, $0 < l < 11$. Intensity of three standard reflections ($\bar{6}23$, $11\bar{4}$, $\bar{1}4\bar{2}$) monitored with no significant intensity variation; 2307 unique reflections measured; rejection of 514 reflections [$I < 3\sigma(I)$]. Correction for Lorentz–polarization and no absorption correction; neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974).

The structure has been solved by *MULTAN* (Declercq, Germain, Main & Woolfson, 1973; Woolfson, 1977) and refined with anisotropic thermal factors by full-matrix least-squares method (Busing, Martin & Levy, 1963). All the H atoms have been located by difference Fourier synthesis, the thermal parameters being kept isotropic. Final refinement with 1793 reflections; final $R = 0{\cdot}058$, $wR = 0{\cdot}058$ [$w = 1/\sigma^2(F)$], $S = 1{\cdot}06$, $(\Delta/\sigma)_{\max} = 0{\cdot}08$ in final least-squares cycle; final difference maps showed strongest peak (trough) of $+1{\cdot}00$ ($-1{\cdot}25$) e Å^{-3}. Computer NAS-9080 (CIRCE).

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

	x	*y*	*z*	B_{eq}*
C(1)	6614 (4)	3763 (14)	7448 (6)	2·9
C(2)	5914 (4)	3467 (14)	8350 (7)	3·4
C(3)	4846 (4)	3575 (15)	7583 (7)	3·7
C(4)	4453 (5)	3948 (14)	5902 (8)	4·0
C(5)	5127 (5)	4172 (15)	4980 (9)	4·1
C(6)	6211 (5)	4081 (14)	5753 (7)	3·8
C(7)	7783 (4)	3774 (14)	8338 (7)	3·3
C(8)	8082 (4)	5561 (13)	9456 (6)	2·4
C(9)	7891 (5)	7501 (14)	8551 (8)	3·6
C(10)	4090 (5)	3285 (19)	8552 (10)	5·8
C(11)	805 (4)	6408 (13)	5880 (6)	2·7
C(12)	603 (4)	5373 (13)	7363 (5)	2·2
F(1)	3432 (4)	4761 (15)	8409 (7)	9·0
F(2)	3501 (5)	1730 (16)	8063 (9)	11·7
F(3)	4537 (4)	3021†	10145 (6)	8·4
N	9219 (3)	5416 (12)	460 (4)	2·3
Cl(1)	1329 (1)	4888 (11)	4645 (2)	4·1
Cl(2)	1615 (1)	8511 (11)	6487 (2)	4·7
O(1)	560 (4)	3540 (12)	7328 (5)	3·9
O(2)	446 (3)	6461 (12)	8443 (4)	3·2

* $B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + 2\beta_{12}ab\cos\gamma + 2\beta_{13}ac\cos\beta + 2\beta_{23}bc\cos\alpha)$.

† Origin fixed.

Table 2. *Selected bond lengths* (Å), *angles* (°) *and torsion angles* (°) *involving non-*H *atoms, with e.s.d.'s in parentheses*

C(1)—C(2)	1·387 (5)	C(2)—C(1)—C(6)	118·27 (5)
C(1)—C(6)	1·386 (6)	C(1)—C(6)—C(5)	120·93 (5)
C(1)—C(7)	1·515 (6)	C(6)—C(5)—C(4)	119·79 (5)
C(2)—C(3)	1·380 (6)	C(5)—C(4)—C(3)	119·7 (2)
C(3)—C(4)	1·381 (7)	C(4)—C(3)—C(2)	120·76 (8)
C(3)—C(10)	1·492 (6)	C(3)—C(2)—C(1)	120·47 (6)
C(4)—C(5)	1·365 (6)	C(2)—C(1)—C(7)	119·52 (6)
C(5)—C(6)	1·399 (7)	C(6)—C(1)—C(7)	122·20 (6)
C(7)—C(8)	1·518 (8)	C(2)—C(3)—C(10)	120·69 (7)
C(8)—C(9)	1·509 (9)	C(4)—C(3)—C(10)	118·55 (1)
C(8)—N	1·503 (5)	C(1)—C(7)—C(8)	110·55 (3)
C(10)—F(1)	1·317 (9)	C(7)—C(8)—C(9)	114·46 (7)
C(10)—F(2)	1·310 (9)	C(7)—C(8)—N	109·26 (2)
C(10)—F(3)	1·310 (7)	C(9)—C(8)—N	109·33 (2)
C(11)—C(12)	1·531 (6)	C(3)—C(10)—F(1)	113·09 (8)
C(11)—Cl(1)	1·758 (6)	C(3)—C(10)—F(2)	112·06 (8)
C(11)—Cl(2)	1·775 (8)	C(3)—C(10)—F(3)	114·04 (6)
C(12)—O(1)	1·249 (7)	F(1)—C(10)—F(2)	105·51 (3)
C(12)—O(2)	1·241 (5)	F(2)—C(10)—F(3)	112·06 (8)
		F(3)—C(10)—F(1)	113·09 (8)
		Cl(1)—C(11)—Cl(2)	109·23 (4)
		Cl(1)—C(11)—C(12)	114·49 (5)
		Cl(2)—C(11)—C(12)	112·19 (6)
		O(1)—C(12)—O(2)	126·65 (13)
		C(6)—C(1)—C(7)—C(8)	110·5
		C(1)—C(7)—C(8)—N	174

Table 3. *Hydrogen-bond lengths* (Å)

		Equivalent positions of H atoms
Cl(1)—H′(C9)	2·65	$1-x, y-\frac{1}{2}, 2-z$
Cl(2)—H(C6)	2·88	$-x, \frac{1}{2}+y, -z$
Cl(2)—H(C8)	2·99	$1-x, \frac{1}{2}+y, 2-z$
F(1)—H(C2)	2·85	$1-x, \frac{1}{2}+y, 2-z$
F(2)—H(C8)	2·44	$1-x, y-\frac{1}{2}, 2-z$
O(1)—H″(C9)	2·83	$1-x, y-\frac{1}{2}, 2-z$
O(2)—H′(C7)	2·56	$1-x, \frac{1}{2}+y, 2-z$
O(2)—H′(C9)	2·83	$x-1, y, z$

Discussion. The atomic positional parameters and equivalent isotropic thermal parameters for non-hydrogen atoms are given in Table 1.* The molecular structure and the atom-numbering system of the title compound are given in Fig. 1. A stereoscopic view of the molecule drawn along the *c* axis is given in Fig. 2. The bond distances and angles are listed in Table 2. Sharp ionic bonds collinear with the helical binary axis are sketched in Fig. 3. The columns are held together by a close network of hydrogen bonds (Table 3). The N atom is in an sp^3 hybridization state as expected. The torsion angles (Table 2) of the side chain show that the chain is in a fully extended conformation, as in other amphetamines previously reported (Creese & Grunewald, 1983; Weintraub & Hopfinger, 1975). The plane C(1)C(7)C(8)N and the benzene ring make a dihedral angle of 110° which is nearly opposite (average 60°) to those observed in the hydrochlorides of fenfluramine (Grunewald, Creese & Extine, 1981; Mayer, Perez, Petit & Coquerel, 1982). The large thermal motion of the $-CF_3$ group can be partially explained by the strength of the hydrogen bond with the three fluorines. This result has been reported earlier in other structures which contain the trifluoromethyl group (Halfpenny & Small, 1981; Cheng, Koo, Mellor, Nyburg & Young, 1970).

The faces present in the common shape are (100), $(\bar{1}01)$ and (110). Fig. 2 shows that the (100) face is formed by the achiral anion and the protonated function $-NH_3^+$. Thus we can assume that the molecules of opposite chirality can be adsorbed irreversibly and are responsible for the growth blockage of this face in racemic solution. The adsorbed molecules are probably perpendicular to the (100) plane. Examination of the poisoned surface shows that this two-dimensional arrangement is similar to those observed in several racemates (Pedone & Benedetti, 1972).

In the slice $(\bar{1}01)$, however, cations lie flat. They take part in the growth but their participation is not restricted to only one chirality. We have established that simultaneous nucleation of both the enantiomers occurs on this face and thus prevents any possibility of resolution by preferential crystallization (Coquerel, 1986).

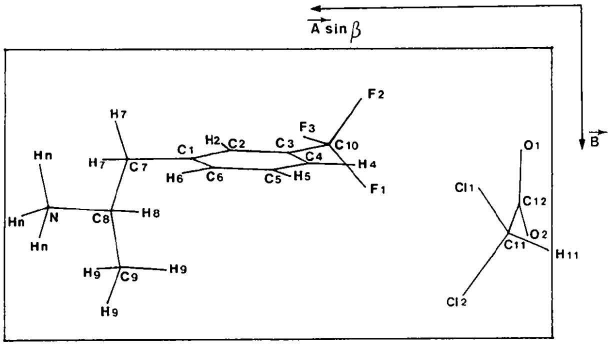

Fig. 1. Molecular structure and atom-numbering system of the dichloroacetate of (*R*)-norfenfluramine.

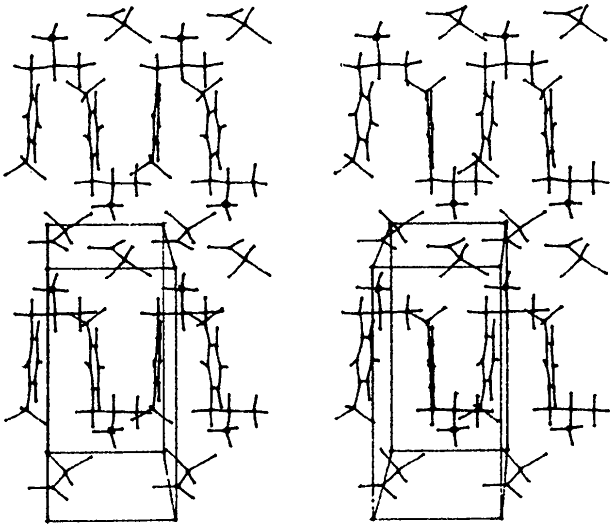

Fig. 2. Stereoscopic view down **c** of the crystal packing. **a** points down the page and **b** to the right.

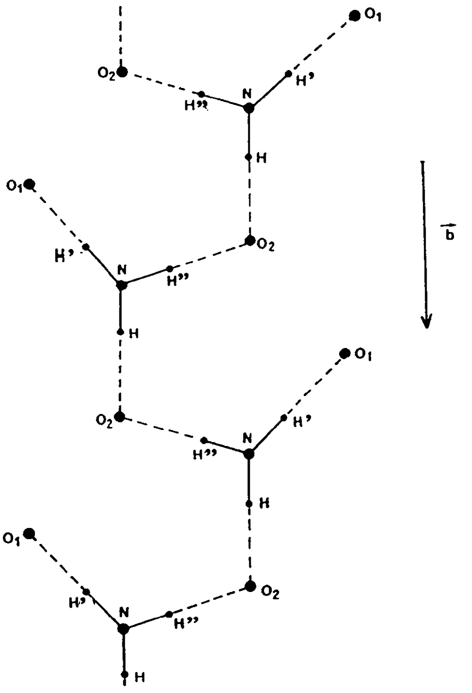

Fig. 3. Representation of strong bonds in the crystal structure.

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* Lists of anisotropic thermal parameters, H-atom parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44717 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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. $C_6F_4(CN)_2$, $M_r = 200\cdot10$, orthorhombic, $P2_12_12_1$, $a = 10\cdot826\ (3)$, $b = 12\cdot320\ (5)$, $c = 5\cdot819\ (5)$ Å, $Z = 4$, $V/Z = 194\cdot0\ (3)$ Å^3, $D_x = 1\cdot712\ (3)$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 1\cdot91$ cm^{-1}, $F(000) = 392$, $T = 295\ (2)$ K, $R = 0\cdot051$ 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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