phy on a silica gel column and eluted with petroleum ether. The elute was concentrated and further purified by repeated thin layer chromatography. Single crystals were obtained from ethanol solution and data were collected on the best crystal available.

Crystal data

$C_{12}H_6Cl_4$	Mo $K\alpha$ radiation
$M_r = 292.0$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 25
$C2/c$	reflections
$a = 12.909\,(3)$ Å	$\theta = 7.5–10°$
$b = 8.174\,(2)$ Å	$\mu = 0.967$ mm^{-1}
$c = 11.302\,(2)$ Å	$T = 296$ K
$\beta = 94.82\,(3)°$	Thin plate
$V = 1188.4\,(5)$ Å^3	$0.50 \times 0.30 \times 0.07$ mm
$Z = 4$	Colourless
$D_x = 1.632$ Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-6S diffractometer	550 observed reflections $[F > 4\sigma(F)]$
$\omega/2\theta$ scans	$R_{int} = 0.0880$
Absorption correction:	$\theta_{max} = 22.5°$
empirical *via* ψ scans	$h = 0 \to 13$
(*SHELXTL-Plus*;	$k = 0 \to 8$
Sheldrick, 1990)	$l = -12 \to 12$
$T_{min} = 0.5731$, $T_{max} = 1.000$	3 standard reflections monitored every 150
884 measured reflections	reflections
773 independent reflections	intensity decay: 0.3%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.003$
$R = 0.077$	$\Delta\rho_{max} = 0.70$ e Å^{-3}
$wR = 0.094$	$\Delta\rho_{min} = -0.38$ e Å^{-3}
$S = 0.97$	Extinction correction: none
550 reflections	Atomic scattering factors
73 parameters	from *International Tables*
H-atom parameters not refined	*for X-ray Crystallography* (1974, Vol. IV)
$w = 1/[\sigma^2(F) + 0.0135F^2]$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl(1)	0.6341 (2)	0.1181 (3)	0.4071 (2)	0.063 (1)
Cl(2)	0.3101 (2)	0.4524 (4)	0.7035 (2)	0.063 (1)
C(1)	0.4905 (5)	0.2829 (10)	0.6851 (6)	0.036 (2)
C(2)	0.5611 (6)	0.2119 (10)	0.6140 (6)	0.040 (2)
C(3)	0.5430 (6)	0.2096 (11)	0.4918 (8)	0.047 (3)
C(4)	0.4540 (6)	0.2756 (12)	0.4331 (7)	0.055 (3)
C(5)	0.3829 (6)	0.3475 (11)	0.5028 (7)	0.049 (3)
C(6)	0.4017 (5)	0.3541 (10)	0.6233 (7)	0.041 (3)

Table 2. *Selected geometric parameters (Å, °)*

Cl(1)—C(3)	1.746 (9)	Cl(2)—C(6)	1.746 (8)
C(1)—C(2)	1.392 (10)	C(1)—C(6)	1.416 (10)
C(1)—C(1')	1.467 (14)	C(2)—C(3)	1.382 (11)
C(3)—C(4)	1.386 (11)	C(4)—C(5)	1.389 (12)
C(5)—C(6)	1.364 (11)		

C(2)—C(1)—C(6)	115.4 (6)	C(2)—C(1)—C(1')	121.0 (7)
C(6)—C(1)—C(1')	123.6 (7)	C(1)—C(2)—C(3)	121.2 (7)
Cl(1)—C(3)—C(2)	119.2 (6)	Cl(1)—C(3)—C(4)	118.3 (7)
C(2)—C(3)—C(4)	122.5 (8)	C(3)—C(4)—C(5)	117.0 (7)
C(4)—C(5)—C(6)	120.8 (7)	Cl(2)—C(6)—C(1)	119.2 (6)
Cl(2)—C(6)—C(5)	117.7 (6)	C(1)—C(6)—C(5)	123.0 (7)

Symmetry code: (') $1 - x, y, \frac{3}{2} - z$.

Crystals of good quality were difficult to obtain and this was the main reason for the large R values. H atoms were not refined. They were located at calculated positions and assigned isotropic displacement parameters. Data collection and refinement used *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). The structure solution and refinement used *SHELXTL-Plus* (Sheldrick, 1990), which was also used for data reduction, molecular graphics and the preparation of material for publication.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1067). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$\alpha,\alpha,\alpha,2,3,4,5,6$-Octafluoroacetanilide

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Abstract

Acetanilide and its derivatives have been the subject of some interest because of certain anomalous properties

of the vibrational spectrum of the amide group [Careri, Buontempo, Galluzi, Scott, Gratton & Shyamsunder (1984). *Phys. Rev. B*, **30**, 4689–4702; Barthes (1995). *Nonlinear Excitations in Biomolecules*, edited by M. Peyrard, pp. 209–221. Berlin: Springer]. In the title compound, C$_8$HF$_8$NO, the molecules are linked through N—H···O hydrogen bonds, with an intermolecular N···O distance of 2.894 (9) Å. They form chains parallel to the *b* axis which are stacked one on top of another in the **c** direction.

Comment

Acetanilide and *N*-methylacetamide compounds are well known to be simple and suitable models for studying structural and physical properties of biological compounds such as polypeptides (Scott, 1992; Barthes, 1995). Single-crystal structures for such compounds have been determined (Brown, 1966; Wasserman, Ryan & Layne, 1985; Lapasset & Moret, 1990; Von Dreele & Eckert, 1990). As part of these various studies, we have undertaken a single-crystal X-ray analysis of the title compound (I) which belongs to this family of acetanilide derivatives in which the H atoms of both the phenyl ring and methyl group of the acetanilide have been substituted by F atoms.

(I)
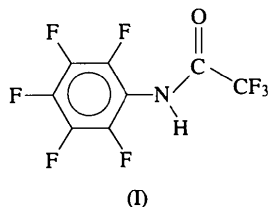
A view of (I) with atomic labelling scheme is shown in Fig. 1. The four atoms of the amide group (O=C—N—H) are coplanar [displacement 0.001 (1) Å from the mean plane] as are the six C atoms of the phenyl ring [mean displacement 0.004 (2) Å]. The interplanar angle between the amide group and the C atoms of the phenyl ring is 60.01 (2)°, indicating a strong interaction between these two parts of the molecule. The environment around atom C1 is slightly distorted from planarity with atoms N, F2 and F6 lying −0.051 (4), 0.016 (4) and −0.057 (4) Å, respectively, out of the phenyl ring plane; note the opposite displacement of the F atoms. The observed N—H bond length of 0.97 (3) Å is reasonable for a distance determined by X-ray analysis. All the C—C ring distances [range 1.366 (6)–1.385 (5) Å] are shorter than those in the parent acetanilide molecule (Brown, 1966). The internal ring angles differ by as much as 2.2 (3)° from 120°.

The molecular packing of (I) is shown in Fig. 2. This arrangement is characterized by N—H···O^i intermolecular hydrogen bonds between glide-related molecules [symmetry code: (i) $1 - x$, $y - \frac{1}{2}$, $\frac{1}{2} - z$]. Each molecule
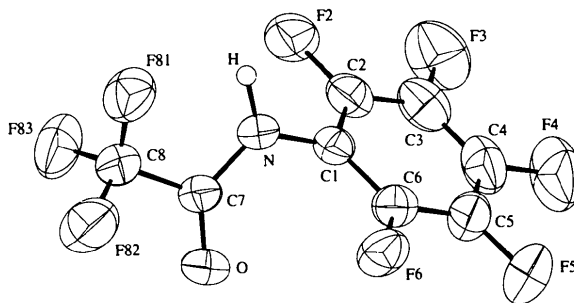

Fig. 1. An *ORTEP*II (Johnson, 1976; André & Michalowicz, 1991) drawing showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and the H atom is represented by a sphere of arbitrary size.

is involved in two hydrogen bonds to two neighbouring molecules forming chains parallel to the *b* axis; the intra-chain molecules (alongside one another) are related by a twofold screw axis, whereas molecules of two adjacent chains are related by a centre of symmetry. The N—H···O^i hydrogen bond has an N···O^i distance of 2.894 (9) Å, shorter than the value of 2.935 (3) Å found in acetanilide (Johnson, Eckert, Barthes, McMullan & Muller, 1995). The N—H···O^i bond angle of 145 (3)° is further from linearity than that in acetanilide [172.3 (4)°; Johnson *et al.*, 1995].
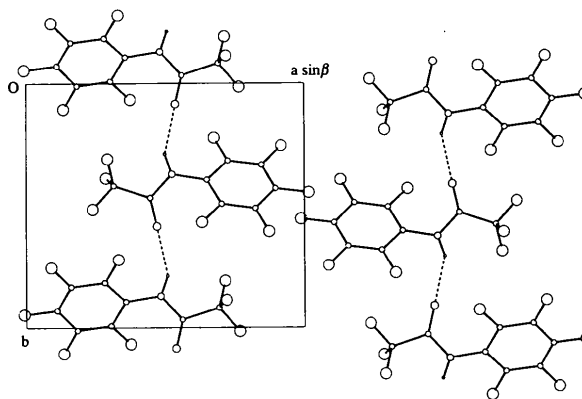
Fig. 2. The molecular packing viewed down the *a* axis, with intermolecular hydrogen bonds represented by dashed lines and O atoms plotted as full spheres.

Experimental

2,3,4,5,6-Pentafluoroaniline (Aldrich) and trifluoroacetic acid (Aldrich) were mixed and the removal of water by distillation resulted in the displacement of the reversible equilibrium

$$C_6F_5NH_2 + CF_3COOH \leftrightarrow C_6F_5NHCOCF_3 + H_2O$$
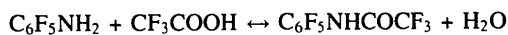
to the right. Single crystals for X-ray study were obtained by condensation of the vapour phase generated by a small temperature gradient in a sealed Pyrex tube.

Crystal data

C_8HF_8NO

$M_r = 279.10$

Monoclinic

$P2_1/c$

$a = 11.23\,(3)$ Å

$b = 9.66\,(3)$ Å

$c = 8.89\,(3)$ Å

$\beta = 98.20\,(3)°$

$V = 955\,(5)$ Å^3

$Z = 4$

$D_x = 1.942$ Mg m^{-3}

D_m not measured

Cu $K\alpha$ radiation

$\lambda = 1.54180$ Å

Cell parameters from 20 reflections

$\theta = 34$–$50°$

$\mu = 2.171$ mm^{-1}

$T = 293\,(2)$ K

Transparent prism

$0.46 \times 0.18 \times 0.16$ mm

Colourless

Data collection

Enraf–Nonius CAD-3 diffractometer

$\theta/2\theta$ scans

Absorption correction: gaussian (*SHELX*76; Sheldrick, 1976)

$T_{min} = 0.6293$, $T_{max} = 0.7811$

1395 measured reflections

1723 independent reflections

1269 observed reflections

$[I > 2\sigma(I)]$

$R_{int} = 0.0199$

$\theta_{max} = 65.97°$

$h = -13 \rightarrow 13$

$k = 0 \rightarrow 11$

$l = 0 \rightarrow 10$

1 standard reflection monitored every 60 reflections

intensity decay: none

Refinement

Refinement on F^2

$R(F) = 0.0476$

$wR(F^2) = 0.1234$

$S = 1.078$

1395 reflections

164 parameters

H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.6965P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.238$ e Å^{-3}

$\Delta\rho_{min} = -0.193$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)

Extinction coefficient: 0.0311 (19)

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
C1	0.6461 (3)	0.3944 (3)	0.2842 (3)	0.0450 (7)
C2	0.7214 (3)	0.3151 (3)	0.3871 (3)	0.0556 (8)
C3	0.8439 (3)	0.3317 (4)	0.4060 (4)	0.0677 (10)
C4	0.8935 (3)	0.4296 (4)	0.3229 (5)	0.0719 (11)
C5	0.8209 (3)	0.5107 (4)	0.2210 (4)	0.0626 (9)
C6	0.6983 (3)	0.4919 (3)	0.2013 (3)	0.0499 (7)
C7	0.4422 (3)	0.4672 (3)	0.2915 (3)	0.0471 (7)
C8	0.3106 (3)	0.4185 (3)	0.2776 (4)	0.0565 (8)
N	0.5205 (2)	0.3703 (2)	0.2628 (3)	0.0467 (6)
O	0.4668 (2)	0.5842 (2)	0.3299 (3)	0.0604 (6)
F2	0.6725 (2)	0.2210 (2)	0.4685 (2)	0.0744 (7)
F3	0.9148 (2)	0.2539 (3)	0.5064 (3)	0.1055 (9)
F4	1.0133 (2)	0.4464 (3)	0.3410 (3)	0.1099 (10)
F5	0.8695 (2)	0.6049 (3)	0.1391 (3)	0.0943 (8)
F6	0.6283 (2)	0.5666 (2)	0.0973 (2)	0.0655 (6)
F81	0.2891 (2)	0.3078 (2)	0.1924 (2)	0.0764 (7)
F82	0.2360 (2)	0.5144 (3)	0.2185 (4)	0.1094 (10)
F83	0.2840 (2)	0.3875 (3)	0.4138 (2)	0.0867 (8)

Table 2. *Selected geometric parameters* (Å, °)

C1—C6	1.377 (5)	C5—C6	1.375 (6)
C1—C2	1.385 (5)	C6—F6	1.335 (4)
C1—N	1.416 (5)	C7—O	1.201 (5)
C2—F2	1.328 (4)	C7—N	1.334 (4)
C2—C3	1.372 (6)	C7—C8	1.538 (6)
C3—F3	1.339 (5)	C8—F82	1.308 (5)
C3—C4	1.366 (6)	C8—F81	1.313 (5)
C4—F4	1.342 (5)	C8—F83	1.323 (5)
C4—C5	1.374 (6)	N—H	0.97 (3)
C5—F5	1.331 (5)		
C6—C1—C2	117.8 (3)	F6—C6—C5	119.7 (3)
C6—C1—N	122.2 (3)	F6—C6—C1	119.2 (3)
C2—C1—N	120.0 (3)	C5—C6—C1	121.1 (3)
F2—C2—C3	119.9 (3)	O—C7—N	125.7 (3)
F2—C2—C1	118.6 (3)	O—C7—C8	119.3 (3)
C3—C2—C1	121.5 (3)	N—C7—C8	114.9 (3)
F3—C3—C4	120.0 (4)	F82—C8—F81	106.9 (4)
F3—C3—C2	120.4 (4)	F82—C8—F83	108.2 (3)
C4—C3—C2	119.6 (3)	F81—C8—F83	107.3 (3)
F4—C4—C3	119.9 (4)	F82—C8—C7	111.6 (3)
F4—C4—C5	120.0 (4)	F81—C8—C7	113.0 (3)
C3—C4—C5	120.1 (4)	F83—C8—C7	109.6 (3)
F5—C5—C4	120.0 (4)	C7—N—C1	122.0 (3)
F5—C5—C6	120.2 (3)	C7—N—H	118 (2)
C4—C5—C6	119.9 (3)	C1—N—H	120 (2)

The H atom was located from difference Fourier maps and the isotropic displacement parameter set at $U = 0.05$ Å^2; the parameters were not refined. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined by full-matrix least-squares techniques using *SHELXL*93 (Sheldrick, 1993). Molecular graphics were obtained using *ORTEP*II (Johnson, 1976) adapted for Macintosh by André & Michalowicz (1991).

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