threshold value for this molecule in any phase, whereas the rotation angle of *B* would depend upon that of *C*. This explanation seems to be supported by the fact that the atoms in *C* deviate much more from the mean plane than those in *B*.

In the liquid phase, the *B* and *C* rings could rotate easily around the C(6)—C(7) and C(1)—C(13) bonds, respectively. The *C* ring would take various rotation angles between 90 ± 47·5°, whereas *B* may have a relatively narrow range of angles depending on the conformation of *C*, and *vice versa*. When the liquid is supercooled and changes into the glassy state, intermolecular entangling of the phenyl groups would occur giving random rotation angles; these random rotation angles may be in the same range as in the liquid phase mentioned above.

The authors are grateful to Professor T. Tachibana for his keen interest and valuable discussions.

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1-(4-Chlorophenyl)-3-(2,6-difluorobenzoyl)urea

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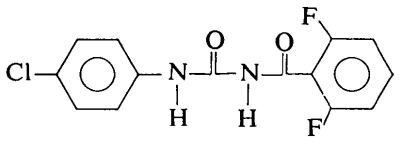
(*Received* 29 *March* 1978; *accepted* 9 *May* 1978)

Abstract. $C_{14}H_9ClF_2N_2O_2$, M_r = 310·7, monoclinic, $P2_1/c$, a = 7·096 (4), b = 7·696 (4), c = 24·988 (11) Å, β = 105·30 (6)°, Z = 4, D_m = 1·560, D_c = 1·568 g cm^{-3}. The urea moiety is approximately planar, and makes dihedral angles of 13·3 and 44·5° with the planes of the adjacent six-membered rings. The amide linkages in sequence have a *trans–cis–trans* conformation. The 3-carbonylurea group conforms to a planar pseudo-ring closed by an intramolecular hydrogen bond.

Introduction. The benzoyl-phenyl ureas represent a new class of insecticides which act by selectively interfering with the deposition of chitin in a large number of foraging insect species (Verloop & Ferrell, 1977). A wide variety of insecticidal properties can be achieved by replacement of the hydrogen atoms at the 2- and 6-positions of the benzoyl ring with halogen atoms and at the 4-position of the phenyl ring with an electron-withdrawing substituent. The title compound (I), commonly known as diflubenzuron, is a particularly potent larvicide having a short life-time in the environment and a low toxicity towards the majority of non-target organisms. Its crystal structure determination was undertaken to provide geometrical parameters for the effect of the halogen substituents on the molecular conformation.



(I)

Technical-grade diflubenzuron, obtained from the Thompson–Hayward Chemical Co., was recrystallized by evaporation of a dioxane–acetone solution. The crystals, m.p. 218 °C, were colourless needles elongated about *a*. One of these was cut to yield a specimen of dimensions 0·35 × 0·10 × 0·13 mm. This was mounted on a four-circle diffractometer with a^* parallel to the φ axis. The intensities of 2181 independent reflexions within the limiting sphere 2θ = 130° were measured with Ni-filtered Cu $K\alpha$ radiation and a scintillation counter. The θ–2θ scan mode was used with scans of 2° for 2θ < 100° and 3° otherwise. Background intensities were estimated from an experimentally determined function of θ. 445 reflexions were treated as unobserved, as their net intensities were less than either 30 counts or 10% of the corresponding background intensity. Absorption corrections, calculated by Gaussian integration, ranging from 1·22 to 1·68 were applied to the intensities (μ = 28·8 cm^{-1}).

Table 1. *Final atomic coordinates* ($\times 10^3$ *for* H *atoms*, $\times 10^4$ *otherwise) and isotropic thermal parameters for* H *atoms*

E.s.d.'s are given in parentheses.

	x	*y*	*z*	*B* (Å^2)
Cl(1)	−5839 (1)	4630 (1)	−1537 (0·3)	
C(1)	−3776 (4)	3948 (4)	−1034 (1)	
C(2)	−2112 (4)	3506 (5)	−1177 (1)	
C(3)	−498 (4)	2923 (4)	−781 (1)	
C(4)	−559 (3)	2771 (3)	−234 (1)	
N(4)	1014 (3)	2157 (3)	197 (1)	
C(5)	−2238 (4)	3252 (4)	−93 (1)	
C(6)	−3853 (4)	3835 (4)	−493 (1)	
C(7)	2618 (3)	1351 (3)	139 (1)	
O(7)	2953 (2)	1009 (3)	−303 (1)	
N(7)	4003 (3)	875 (3)	627 (1)	
C(8)	3964 (3)	1121 (3)	1166 (1)	
O(8)	2587 (2)	1775 (3)	1296 (1)	
C(9)	5734 (3)	508 (3)	1591 (1)	
C(10)	7631 (3)	820 (3)	1564 (1)	
F(10)	7893 (2)	1769 (2)	1135 (1)	
C(11)	9266 (3)	288 (4)	1957 (1)	
C(12)	9014 (4)	−609 (4)	2410 (1)	
C(13)	7178 (4)	−953 (4)	2470 (1)	
C(14)	5590 (3)	−385 (4)	2062 (1)	
F(14)	3807 (2)	−757 (2)	2121 (1)	
H(2)	−202 (4)	361 (4)	−154 (1)	4·8 (0·7)
H(3)	67 (4)	256 (4)	−88 (1)	4·2 (0·6)
H(4)	99 (4)	231 (4)	52 (1)	4·0 (0·6)
H(5)	−228 (4)	318 (4)	30 (1)	4·9 (0·7)
H(6)	−506 (4)	412 (4)	−40 (1)	4·0 (0·6)
H(7)	502 (3)	36 (3)	57 (1)	2·6 (0·5)
H(11)	1053 (4)	57 (4)	191 (1)	3·7 (0·6)
H(12)	1013 (4)	−97 (4)	269 (1)	3·6 (0·6)
H(13)	695 (4)	−158 (4)	279 (1)	4·1 (0·6)

The structure was determined by direct methods using the program *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974). Refinement was by a block-diagonal least-squares method, minimizing $\sum w(\Delta F)^2$. The hydrogen atoms were placed in chemically reasonable positions and were included in the refinement. The five most intense reflexions were judged to suffer extinction with intensities reduced to as much as 75% of the calculated value. These reflexions were omitted from the refinement process. Otherwise, the weighting scheme used in the final stages was $w^{-1} = 1 + [(|F_o| - 13)/20]^2$. With this weighting scheme the weighted residual showed no obvious dependence on $|F_o|$. In the final refinement cycle the largest coordinate shift of a non-hydrogen atom was 0·2 of the corresponding e.s.d., and the residual electron density in the final difference map was within ±0·16 e Å^{-3}. The final residual for observed reflexions was $R = 0{\cdot}042$ ($R_w = 0{\cdot}047$). The refined atomic positions are given in Table 1.*

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

E.s.d.'s of bond angles range from 0·17 to 0·28°.

C(1)–Cl(1)	1·740 (3)	C(2)–C(1)–Cl(1)	120·5
C(1)–C(2)	1·364 (4)	C(6)–C(1)–Cl(1)	119·1
C(1)–C(6)	1·370 (4)	C(6)–C(1)–C(2)	120·4
C(2)–C(3)	1·376 (4)	C(1)–C(2)–C(3)	120·3
C(3)–C(4)	1·384 (4)	C(2)–C(3)–C(4)	120·1
C(4)–C(5)	1·379 (4)	C(3)–C(4)–C(5)	118·9
C(5)–C(6)	1·382 (4)	C(3)–C(4)–N(4)	123·8
C(4)–N(4)	1·412 (3)	C(5)–C(4)–N(4)	117·2
C(7)–N(4)	1·337 (3)	C(4)–C(5)–C(6)	120·6
C(7)–N(7)	1·399 (3)	C(5)–C(6)–C(1)	119·6
C(7)–O(7)	1·218 (3)	C(4)–N(4)–C(7)	126·6
C(8)–N(7)	1·366 (3)	N(4)–C(7)–O(7)	125·0
C(8)–O(8)	1·218 (3)	N(4)–C(7)–N(7)	116·6
C(8)–C(9)	1·492 (3)	N(7)–C(7)–O(7)	118·3
C(9)–C(10)	1·387 (4)	C(7)–N(7)–C(8)	129·1
C(9)–C(14)	1·390 (3)	N(7)–C(8)–C(9)	115·2
C(10)–C(11)	1·370 (4)	N(7)–C(8)–O(8)	123·2
C(11)–C(12)	1·378 (4)	O(8)–C(8)–C(9)	121·6
C(12)–C(13)	1·375 (4)	C(8)–C(9)–C(10)	123·7
C(13)–C(14)	1·375 (4)	C(8)–C(9)–C(14)	121·6
C(10)–F(10)	1·349 (3)	C(10)–C(9)–C(14)	114·6
C(14)–F(14)	1·343 (3)	C(9)–C(10)–C(11)	124·2
N(4)–H(4)	0·83 (3)	C(9)–C(10)–F(10)	118·2
H(4)···O(8)	2·00 (3)	C(11)–C(10)–F(10)	117·6
N(4)···O(8)	2·695 (3)	C(10)–C(11)–C(12)	118·1
N(7)–H(7)	0·87 (2)	C(11)–C(12)–C(13)	121·1
H(7)···O(7)*	2·03 (2)	C(12)–C(13)–C(14)	118·3
N(7)···O(7)*	2·890 (3)	C(9)–C(14)–C(13)	123·7
H(7)···F(10)	2·41 (2)	C(9)–C(14)–F(14)	118·7
N(7)···F(10)	2·803 (3)	C(13)–C(14)–F(14)	117·6
O(8)···F(14)	2·802 (3)	N(4)–H(4)···O(8)	140 (2)
H(3)···O(7)	2·21 (3)	N(7)–H(7)···O(7)*	169 (2)
H(5)···F(10)†	2·33 (2)		
H(11)···F(14)‡	2·46 (2)		
C–H mean	0·96 (2)		
C–H range	0·94–0·99		

* Atoms related by center of symmetry at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$.
† Atoms related by translation of *a*.
‡ Atoms related by *c* glide.

The computer programs used were those of Ahmed, Hall, Pippy & Huber (1973). Scattering factors were taken from Stewart, Davidson & Simpson (1965) for H and from Hanson, Herman, Lea & Skillman (1964).

Discussion. Bond distances and angles are given in Table 2. An *ORTEP* plot (Johnson, 1965) of the molecule is given in Fig. 1. Diflubenzuron can be

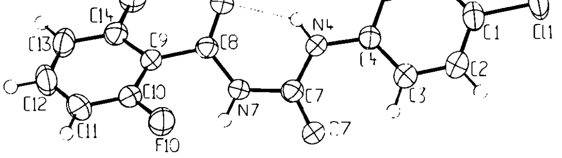


Fig. 1. Atom designations of the molecule. Vibration ellipsoids of the non-hydrogen atoms were drawn by *ORTEP* (Johnson, 1965) at the 50% probability level.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33605 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Distances* (Å ×10³) *of some atoms from certain mean planes*

Atoms defining the plane are underlined.

Plane *A*	C(1) 6, C(2) −2, C(3) −5, C(4) 9, C(5) −5, C(6) −2, Cl(1) 55, N(4) 30, C(7) 280, O(7) 542
Plane *B*	N(4) 0, C(7) 0, O(7) −1, N(7) 2, C(8) −1, O(8) 46, C(9) −52
Plane *C*	C(9) 5, C(10) −2, C(11) −1, C(12) 3, C(13) 0, C(14) −4, F(10) −73, F(14) 14, O(8) −738, N(7) 822, C(8) −20
Dihedral angles	*AB* 13·3°, *BC* 44·5°, *AC* 31·2°

considered to contain both *trans*- and *cis*-amide functions with the latter in part stabilized by the formation of a pseudo-ring closed by the N(4)—H(4)···O(8) intramolecular hydrogen bond. Unlike the structure of biuret (Craven, 1973) where the two C—N(imide) bond distances were equal [1·383 (3), 1·386 (3) Å], the C(7)—N(7) *cis*-amide linkage is longer than the corresponding C(7)—N(4) and C(8)—N(7) *trans*-amide linkages. Otherwise, the geometry of the urea pseudo-ring is very similar to that observed in biuret and in triuret (Carlström & Ringertz, 1965).

The difluorobenzoyl group is non-planar with the three adjacent non-hydrogen substituents displaced out of the ring plane so as to increase their separation. The ring is tilted 44·5° with respect to the urea pseudo-ring. In this conformation F(14) and F(10) are in van der Waals contact with O(8) and N(7) respectively. This conformation is partly stabilized by a weak interaction of 2·41 Å between F(10) and H(7). However, it is questionable whether H(7) should be considered as part of a bifurcated hydrogen bond especially in light of other short contacts including F(10)···H(5) and F(14)···H(11). The chlorophenyl ring is tilted out of the urea pseudo-ring probably to accommodate a close contact between H(3) and O(7). Table 3 shows the deviations from mean planes and lists the dihedral angles.

The molecules crystallize as dimers with N(7) and O(7) acting as intermolecular hydrogen-bond donors and acceptors to the corresponding atoms of molecules related by the center of symmetry at ½,½,½ as shown in Fig. 2. The chlorophenyl-urea moieties of molecules related by the center of symmetry at 0,0,0 overlap with a mean interplanar spacing of 3·5 Å.

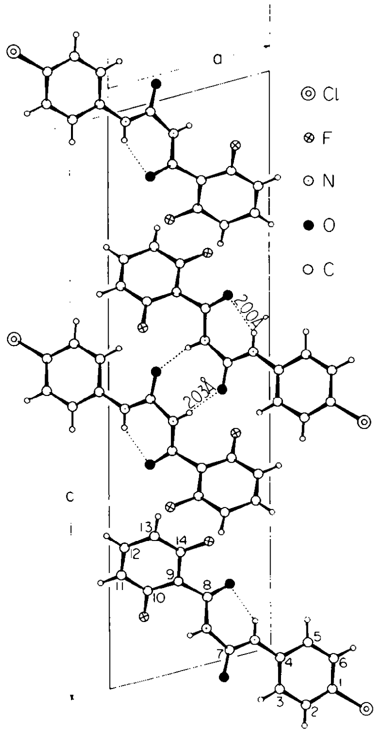

Fig. 2. Packing of the molecules viewed along *b*. Hydrogen bonds are indicated by dotted lines.

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