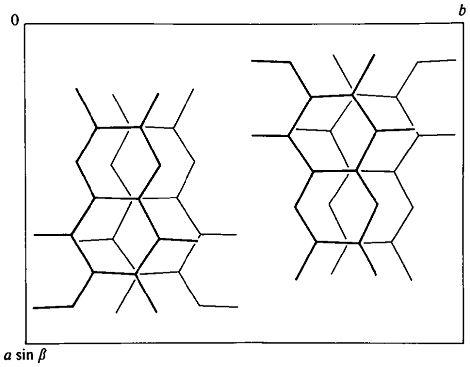

Fig. 2. The crystal structure projected down c.

between the C=C bond length and the CCC angle opposite the C=C bond (Coppens, 1973).

The isoquinoline ring is approximately planar with the largest deviation from the least-squares plane being 0·055 Å. C(16) and O(17) are definitely out of this plane, the deviations being 0·714 and 0·258 Å respectively. The methoxy group is inclined by 33·0° to this plane.

As shown in Fig. 2, the molecules are stacked in infinite columns along the *c* axis and the mean separation between the molecular planes is 3·41 Å. C(9) and O(13) of the carbonyl group are located just above O(13) and C(9), respectively, in the molecule related by the *c* glide plane. Intermolecular distances between these atoms are 3·424 and 3·493 Å respectively. These observations suggest that there is appreciable dipole–dipole interaction between the carbonyl groups.

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1,2-Diphenylbenzene (*o*-Terphenyl)

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Abstract. $C_{18}H_{14}$, $M_r = 230\cdot31$, orthorhombic, $P2_12_12_1$, $a = 18\cdot582$ (4), $b = 6\cdot024$ (2), $c = 11\cdot729$ (2) Å, $Z = 4$, $V = 1313\cdot0$ (6) Å^3, $D_x = 1\cdot165$ g cm^{-3}; λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·77 cm^{-1}. The structure was solved by direct methods and refined by the block-diagonal least-squares method. The final *R* was 0·080 for 1139 reflections. The two phenyl groups are twisted in the same direction with respect to the central ring; the dihedral angles between the mean plane of the central ring and those of the two phenyl rings are 62·1 and 42·5°.

Introduction. It has been found that *o*-terphenyl can easily form a stable glassy structure when it is supercooled below 240 K (Chang & Bestul, 1972). We have studied the physical properties of this compound in the glassy state. Although the crystal structure of *o*-terphenyl has been argued from magnetic susceptibility measurements (Clews & Lonsdale, 1937), its direct determination seems necessary in order to elucidate the structure in the glassy state and to correlate the physical properties with the structure.

Prismatic crystals were obtained from a methanol solution. Intensity data up to $2\theta = 50°$ were collected with a crystal of dimensions $0{\cdot}4 \times 0{\cdot}4 \times 0{\cdot}3$ mm, on a Rigaku four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation; an ω–2θ scan at 4° (2θ) min^{-1} was employed. Lorentz and polarization corrections were made as usual. A total of 1365 reflections were obtained, of which 226 with $|F_o| < 3{\cdot}0\sigma(|F_o|)$ were considered as unobserved. The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by the block-diagonal least-squares method. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where w is the weight as follows: $w = 0{\cdot}2$ for $|F_o| < 2{\cdot}41$ and $|F_o| > 24{\cdot}08$, and $w = (6{\cdot}9753 - 0{\cdot}90244|F_o| + 0{\cdot}03416F_o^2)^{-1}$ for $2{\cdot}41 \le |F_o| \le 24{\cdot}08$. All the hydrogen atoms were located on a difference map and their positional and isotropic thermal parameters were also refined. At the final stage of the refinement, ten reflections were excluded because the profiles of their peaks showed some splitting, probably due to a slight crack in the crystal. The final R value was 0·080 for 1139 reflections. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The positional parameters are given in Table 1.*

Table 1. *Final positional parameters (carbon atoms* $\times 10^4$; *hydrogen atoms* $\times 10^3$), *with their standard deviations in parentheses*

	x	y	z
C(1)	2619 (2)	3363 (7)	3579 (3)
C(2)	1932 (2)	3695 (9)	3168 (3)
C(3)	1449 (2)	5143 (10)	3632 (4)
C(4)	1695 (2)	6518 (8)	4538 (4)
C(5)	2365 (2)	6214 (7)	4963 (3)
C(6)	2848 (2)	4693 (6)	4513 (3)
C(7)	3562 (2)	4453 (7)	5064 (3)
C(8)	3768 (3)	2476 (8)	5579 (3)
C(9)	4440 (3)	2339 (10)	6098 (4)
C(10)	4897 (3)	4090 (12)	6105 (4)
C(11)	4694 (3)	6038 (9)	5590 (4)
C(12)	4033 (2)	6219 (8)	5079 (4)
C(13)	3104 (2)	1784 (7)	2983 (3)
C(14)	3810 (2)	2336 (8)	2708 (3)
C(15)	4256 (3)	875 (8)	2107 (4)
C(16)	4001 (3)	−1119 (8)	1732 (4)
C(17)	3280 (3)	−1706 (8)	1984 (4)
C(18)	2851 (2)	−266 (8)	2601 (3)
H(1)	170 (2)	262 (7)	246 (3)
H(2)	95 (2)	568 (5)	319 (3)
H(3)	134 (2)	776 (6)	483 (3)
H(4)	253 (2)	751 (6)	560 (3)
H(5)	336 (2)	97 (7)	552 (3)
H(6)	467 (2)	98 (9)	643 (4)
H(7)	536 (2)	400 (7)	644 (3)
H(8)	504 (3)	768 (10)	549 (4)
H(9)	394 (2)	766 (7)	464 (3)
H(10)	408 (2)	393 (7)	302 (4)
H(11)	488 (2)	151 (8)	195 (4)
H(12)	440 (2)	−256 (7)	121 (4)
H(13)	296 (2)	−326 (7)	169 (3)
H(14)	228 (2)	−62 (7)	278 (3)

Discussion. Bond lengths and angles are shown in Fig. 1, with the atom-numbering and ring-notation system used. The repulsion between the B and C rings makes

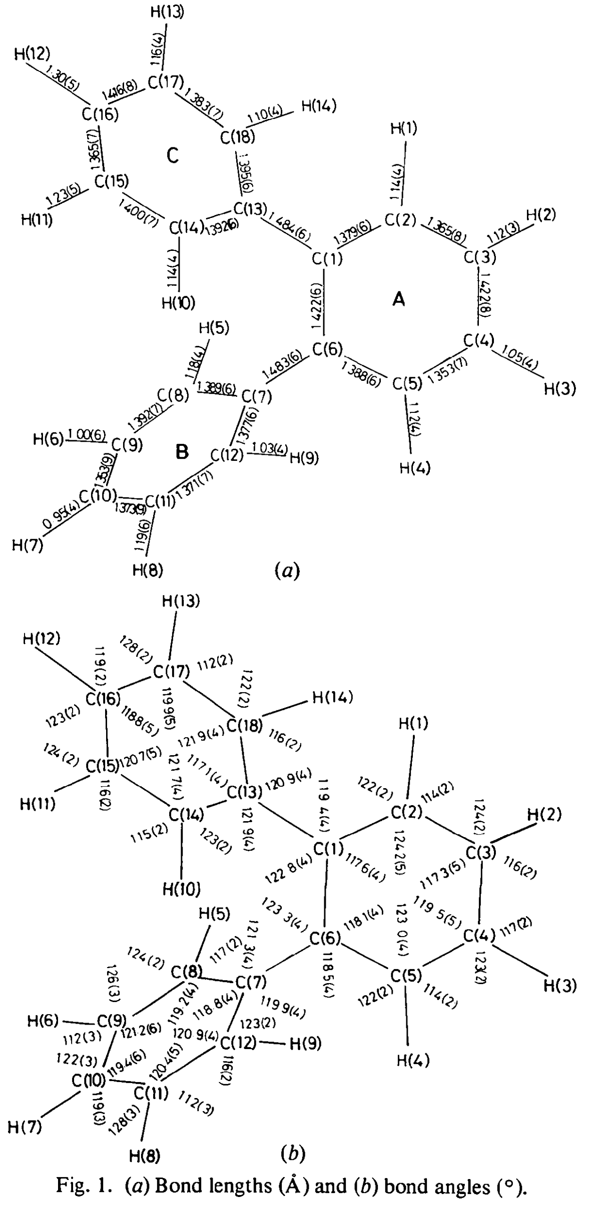

Fig. 1. (*a*) Bond lengths (Å) and (*b*) bond angles (°).

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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 33592 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

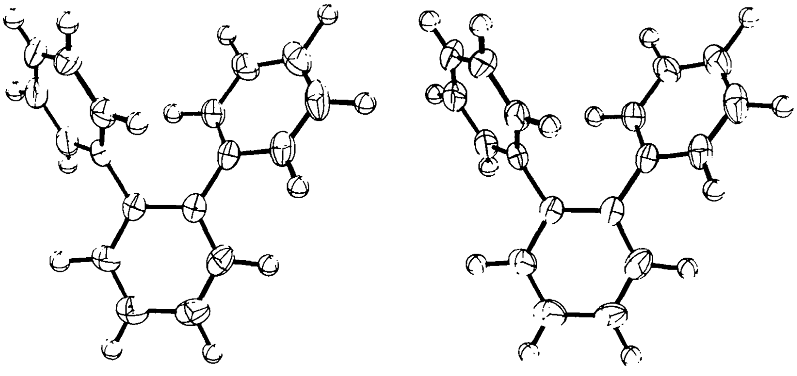

Fig. 2. Stereoscopic view with thermal ellipsoids at 30% probability.

Table 2. *Deviations of the atoms from the least-squares planes* (Å)

Coordinates X,Y,Z are in Å referred to **a**, **b** and **c**.

Plane I: $-0{\cdot}3347X - 0{\cdot}6928Y + 0{\cdot}6388Z + 0{\cdot}3527 = 0$
Plane II: $0{\cdot}3971X - 0{\cdot}3129Y - 0{\cdot}8628Z + 3{\cdot}3344 = 0$
Plane III: $-0{\cdot}3035X + 0{\cdot}4232Y - 0{\cdot}8537Z + 4{\cdot}2769 = 0$

Plane I		Plane II		Plane III	
C(1)	0·0015*	C(7)	−0·0015*	C(13)	−0·0058*
C(2)	−0·0172*	C(8)	0·0027*	C(14)	0·0124*
C(3)	0·0267*	C(9)	−0·0012*	C(15)	−0·0102*
C(4)	−0·0219*	C(10)	−0·0016*	C(16)	0·0014*
C(5)	0·0072*	C(11)	0·0029*	C(17)	0·0051*
C(6)	0·0036*	C(12)	−0·0013*	C(18)	−0·0029*
C(7)	0·0733	C(6)	−0·0152	C(1)	0·0739
C(13)	−0·0867				

* Atoms defining the least-squares plane. The dihedral angles between planes I and II, and between planes I and III are +62·1 and +42·5°, respectively.

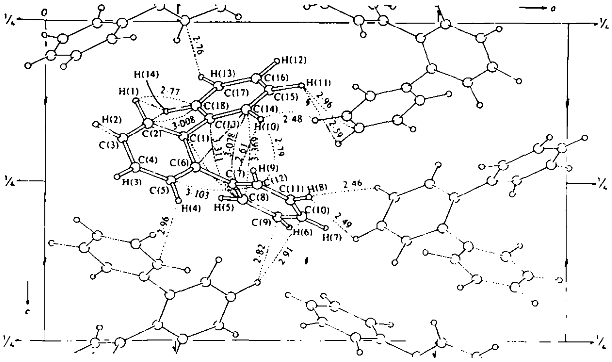

Fig. 3. The crystal structure projected along *b*. Distances not shown on the figure: H(1)···H(14) 2·26, H(14)···C(2) 2·72, C(1)···C(8) 3·217, C(6)···C(14) 3·112 Å.

the bond lengths C(1)–C(6) and C(3)–C(4) longer than the normal distance. As shown in Fig. 2, the *B* and *C* rings rotate around the C(6)–C(7) and C(1)–C(13) bonds respectively in the same direction to avoid steric hindrance between the rings. The equations of the three mean planes, I, II and III for rings *A*, *B* and *C* respectively, and the deviations of the atoms from the planes are listed in Table 2. The II and III planes make angles of +62·1 and +42·5° respectively with I. The C(7) and C(13) atoms are significantly displaced from plane I in opposite directions to avoid a short contact between them.

The lengths of the two C–C bonds joining the benzene rings are 1·483 and 1·484 Å, both of which are shorter than the corresponding ones in biphenyl, 1·497 Å, (Robertson, 1961) and in *p*-terphenyl, 1·496 Å, (Rietveld, Maslen & Clews, 1970). In both cases the benzene rings are almost coplanar, probably because of the electronic resonance interaction between the rings, which may cause large steric hindrance between the *ortho* hydrogen atoms. The C–C bond connecting the benzene rings would be expanded to avoid the steric repulsion. In the present compound, however, the benzene rings cannot be coplanar because of the large steric hindrance between the *ortho*-substituted phenyl rings, so that the C–C bonds are no longer expanded and are in fact shorter than those in biphenyl and *p*-terphenyl.

Fig. 3 shows the crystal structure viewed along the *b* axis, including several short distances between non-bonded atoms. There is no unusually short contact between the molecules. Among the intramolecular non-bonded distances, there are some unusually short distances: C(7)···C(14), 3·078, and H(1)···H(14), 2·26 Å. The repulsions between these atoms make it impossible for the dihedral angle between *C* and *A* to take a smaller value than the one observed, +42·5°. On the other hand, the C(8)···C(13) contact is loose compared with C(7)···C(14). This may imply that the rotation angle of *B* could decrease from the observed value, if it were not affected by the crystalline field. However, the $2p_z$ orbitals of C(8) and C(14) overlap with each other in this conformation. The repulsion between these orbitals may prevent the rotation angle from being smaller, though the C(8)···C(14) and C(8)···C(13) distances are not unusually short. Therefore, the observed angle of *C* is considered to be a 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.

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

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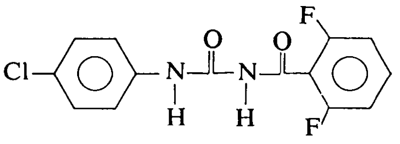
Abstract. $C_{14}H_9ClF_2N_2O_2$, $M_r = 310·7$, monoclinic, $P2_1/c$, $a = 7·096$ (4), $b = 7·696$ (4), $c = 24·988$ (11) Å, $\beta = 105·30$ (6)°, $Z = 4$, $D_m = 1·560$, $D_c = 1·568$ g cm^{-3}. The urea moiety is approximately planar, and makes dihedral angles of 13·3 and 44·5° with the planes of 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.



(I)

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.

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 $2\theta = 130°$ 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 $2\theta < 100°$ 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 ($\mu = 28·8$ cm^{-1}).