

Fig. 2. *c*-axial projection of the cell contents.

Table 3. Inter-ring bond lengths (Å) and corresponding dihedral angles (°) in substituted perfluoroterphenyls

	Inter-ring bond length	Dihedral angle
Isomer (I)	1.50 (2)	63.3 (11)
Isomer (I)	1.49 (2)	65.6 (15)
Isomer (II)	1.47 (1)	78.3 (7)

The evidence for the proposed aryne mechanism for the formation of isomers (I) and (II) from 2-lithiononafluorobiphenyl has been further strengthened by the confirmation of the molecular structure of the title compound (I).

Isomer (II) is the only other substituted perfluoroterphenyl for which data are available for comparison (Bowen Jones & Brown, 1980). The dihedral angles between the central and terminal phenyl rings in (I) and (II), together with the corresponding inter-ring bond lengths, are listed in Table 3. The limited data may indicate a correlation between these two parameters in substituted perfluoroterphenyls, a smaller dihedral angle requiring a greater inter-ring bond length to minimize non-bonding interactions.

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Structure of *N,N'*-Di-*n*-hexyl-4-oxoheptanediamide, $C_{19}H_{36}N_2O_3$

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Abstract. $M_r = 340.53$, monoclinic, $C2/c$, $a = 44.435$ (7), $b = 5.1929$ (5), $c = 8.918$ (1) Å, $\beta = 99.88$ (2)°, $V = 2027.2$ (8) Å³, $Z = 4$, $D_m = 1.10$, $D_x = 1.116$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $F(000) = 752$, $T = 294$ K. Final $R = 0.045$ for 1635 observed

Mean C–F and intra-ring C–C lengths are similar in the two isomers, but the C–Br bond in (I) is 0.06 (3) Å shorter than in (II). There are no significantly short intermolecular contacts in either compound.

The phenyl rings in the title compound are planar to within ± 0.02 Å and the displacement of the Br atom from the plane of the central ring is 0.110 (9) Å compared with 0.050 (13) Å in its isomer.

Each isomer may be regarded as a disubstituted perfluorobiphenyl. The inter-ring bond length and dihedral angle in their biphenyl systems have been compared* with those of other substituted perfluorobiphenyls. Goodhand, Hamor & Hamor (1978) found no correlation between these two parameters in polyfluorobiphenyls, and the data for isomers (I) and (II) and for 2*H*,2'*H*-octafluorobiphenyl (Bowen Jones & Brown, 1980, 1982) confirm this.

We thank our colleague, Dr A. G. Massey, for suggesting this programme of structure analyses and for providing a sample of the title compound.

* See deposition footnote.

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reflections. The molecule presents a W shape with a twofold axis along the ketonic bond. Molecules consecutively related by inversion centres stack along *c*. The two hydrogen bonds per molecule run in the direction of the *c* axis.

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Introduction. An X-ray study of two series of monodisperse normal diamides *A*: $R\text{-NHCO}(\text{CH}_2)_{2n}\text{-CONH-R}$ and ketodiamides *B*: $R\text{-NHCO}(\text{CH}_2)_n\text{-CO}(\text{CH}_2)_n\text{-CONH-R}$, with $R = n$ -hexyl and the subindex reaching up to four is being carried out in our laboratory. In the crystal structure of the member with $n = 2$ the all-*trans* planar conformation has been found (Rueda & Fayos, 1982); on the basis of cell dimensions, the same conformation has been established for the other members of the *A* series (Ania, Rueda & Baltá Calleja, 1981). The presence of a ketonic group in the *B* series is expected to introduce conformational and packing changes through the members of the series. These changes would be mainly responsible for the observed differences in physical properties between the *A* and *B* series (Obeso, 1978) including the enhancement of molecular diamagnetism in the *B* series (Ania, Baltá Calleja & de Abajo, 1979).

Experimental. Synthesis of title compound reported elsewhere (Obeso, de Abajo & Fontán, 1977); colourless single crystals from dilute ethanolic solution, crystal $0.8 \times 0.1 \times 0.05$ mm used for data collection; 1811 independent reflections up to $\theta = 67^\circ$, four-circle diffractometer, graphite-monochromated $\text{Cu K}\alpha$ radiation, 2 min per reflection with scan of 2° in $\theta/2\theta$ mode, 1635 with $I > 2\sigma(I)$, absorption ignored; direct methods (*MULTAN*, Main, Lessinger, Woolfson, Germain & Declercq, 1977); H atoms by difference synthesis, full-matrix anisotropic (isotropic for H) weighted refinement converged to $R = 0.045$, $R_w = 0.046$, weighting scheme (Martínez-Ripoll & Cano, 1975) used which showed no trends in $\langle w\Delta^2F \rangle$ vs $\langle F_o \rangle$ and $\langle \sin\theta/\lambda \rangle$; calculations performed with XRAY 70 (Stewart, Kundell & Baldwin, 1970).

Discussion. Fractional coordinates and thermal parameters are given in Table 1.* There is a gradual increase in the thermal factors towards the chain end.

Fig. 1(a) shows two hydrogen-bonded molecules projected onto the *ab* plane. The two W-shaped molecules are related by an inversion centre. A twofold axis parallel to **b** passes along the central ketonic bond. This molecular conformation differs strongly from the planar extended chain found for the analogous member with $n = 2$ from the *A* series (Rueda & Fayos, 1982). Fig. 1(b) represents three molecules, projected onto the *ac* plane, linked by hydrogen bonds in the direction of the *c* axis. Inversion centres and a helical twofold axis are also indicated. The three twofold axes passing along the central ketonic groups are omitted for clarity.

All bond lengths and angles have normal values (Table 2a). The torsion angles are presented in Table

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38382 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($x \times 10^5$) (y and $z \times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for non-H atoms

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j \cos(a_i, a_j).$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(0)	0	3067 (4)	7500	48 (1)
O(0)	0	739 (3)	7500	82 (1)
C(1)	-2494 (3)	4590 (3)	8034 (2)	49 (1)
C(2)	-4825 (3)	2943 (3)	8642 (1)	48 (1)
C(3)	-6933 (3)	1510 (3)	7408 (1)	44 (1)
C(4)	-10734 (4)	-1891 (3)	6825 (1)	53 (1)
C(5)	-12885 (3)	-3396 (3)	7630 (1)	51 (1)
C(6)	-15177 (4)	-4911 (4)	6513 (2)	60 (1)
C(7)	-17454 (4)	-6482 (4)	7202 (2)	59 (1)
C(8)	-19718 (5)	7894 (5)	6023 (2)	80 (1)
C(9)	-22043 (7)	-9462 (7)	6671 (4)	98 (1)
O(3)	-7219 (3)	2136 (2)	6055 (1)	58 (1)
N	-8560 (3)	-384 (2)	7877 (1)	50 (1)

Table 2. Molecular geometry

(a) Bond lengths (Å) and angles (°)			
O(0)–C(0)	1.209 (3)	O(0)–C(0)–C(1)	121.73 (9)
C(0)–C(1)	1.504 (2)	C(0)–C(1)–C(2)	113.8 (1)
C(1)–C(2)	1.514 (2)	C(1)–C(2)–C(3)	113.2 (1)
C(2)–C(3)	1.513 (2)	C(2)–C(3)–O(3)	121.9 (1)
C(3)–O(3)	1.235 (1)	C(2)–C(3)–N	115.9 (1)
C(3)–N	1.330 (2)	O(3)–C(3)–N	122.1 (1)
N–C(4)	1.454 (2)	C(3)–N–C(4)	122.1 (1)
C(4)–C(5)	1.508 (2)	N–C(4)–C(5)	112.2 (1)
C(5)–C(6)	1.516 (2)	C(4)–C(5)–C(6)	111.5 (1)
C(6)–C(7)	1.510 (3)	C(5)–C(6)–C(7)	115.8 (1)
C(7)–C(8)	1.514 (3)	C(6)–C(7)–C(8)	113.0 (2)
C(8)–C(9)	1.506 (4)	C(7)–C(8)–C(9)	114.4 (2)
N–O(3)	2.938 (1)		
N–H...O(3)	H–N 0.91 (3)	N–H–O(3)	175 (2)
	H–O(3) 2.03 (3)		
(b) Torsion angles (°)			
C'(1)–C(0)–C(1)–C(2)	-176.6 (1)	N–C(4)–C(5)–C(6)	178.8 (1)
C(0)–C(1)–C(2)–C(3)	-72.7 (1)	C(4)–C(5)–C(6)–C(7)	-179.7 (1)
C(1)–C(2)–C(3)–N	165.6 (1)	C(5)–C(6)–C(7)–C(8)	178.6 (2)
C(2)–C(3)–N–C(4)	178.5 (1)	C(6)–C(7)–C(8)–C(9)	-179.5 (2)
C(3)–N–C(4)–C(5)	-164.4 (1)		

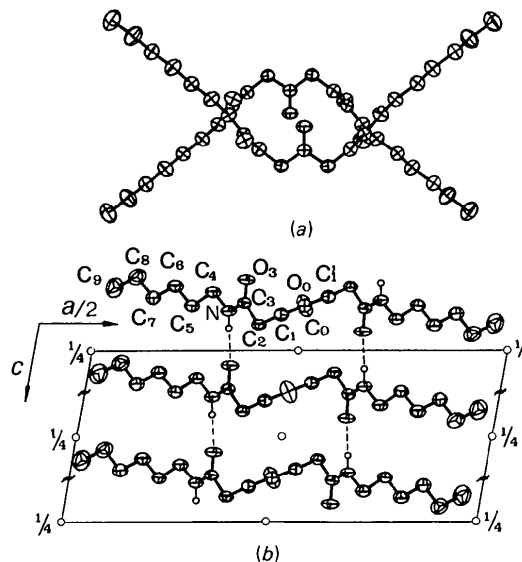


Fig. 1. (a) Two molecules projected onto the *ab* plane. (b) Projection onto the *ac* plane of three molecules showing the hydrogen bonds. The atoms of half the molecule are numbered.

2(b). They indicate a good planarity of the hexyl group which is twisted $15.6 (1)^\circ$ from the planar amidic group. The angle between the twofold axis and the normal to the hexyl plane is $51.7 (3)^\circ$.

From the above results and those recently reported for the analogous diamide member (Rueda & Fayos, 1982), we note that the inclusion of the ketonic group in the middle of the dicarboxylic acid drastically changes the shape and packing of the molecules. Nevertheless, the two hydrogen bonds per molecule are completely preserved. Indeed a shorter N...O distance is present for the ketonic member.

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12-Phenyl-5,12-dihydrodibenz[*b,g*]azocin-5-one, C₂₁H₁₅NO

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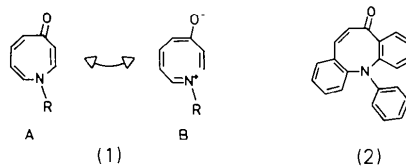
(Received 17 December 1982; accepted 4 February 1983)

Abstract. $M_r = 297.4$, monoclinic, $C2/c$, $a = 15.724 (12)$, $b = 9.222 (6)$, $c = 21.504 (16) \text{ \AA}$, $\beta = 95.91 (8)^\circ$, $U = 3102 \text{ \AA}^3$, $Z = 8$, $D_x = 1.274 \text{ Mg m}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71069 \text{ \AA}$, $\mu = 0.41 \text{ mm}^{-1}$, $F(000) = 1248$, $T = 294 (1) \text{ K}$. Final $R = 0.055$ for 1170 unique X-ray diffractometer data. The molecular structure has been elucidated.

Introduction. Our former results in studying synthesis, structural properties and chemical behaviour of cyclocyanines (Eicher, Abdesaken, Franke & Weber, 1975; Eicher & Franke, 1981) prompted us to direct synthetic efforts to the class of eight-membered cyclic merocyanines (1), especially with regard to the aspect of a probable participation of a resonance hybrid $A \leftrightarrow B$ in the ground-state description of the 1,4-dihydroazocin-4-one system (1).

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The title compound (2) was synthesized recently (Thimme, 1982) as the first member of the hitherto unknown class of azocin-4-ones. The above aspects of a probable bond delocalization in the eight-membered ring and the necessity of gaining rigorous proof of structure for the product of a multi-step synthesis (its spectroscopic data lacked specific information on detailed structural and conformational properties) made it desirable to determine the molecular structure of the dibenzazocinone (2).