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

C(1)–C(2)	1·538 (5)	C(5)–C(8)	1·523 (6)
C(1)–C(7)	1·499 (6)	C(6)–C(7)	1·295 (6)
C(1)–C(8)	1·506 (6)	C(9)–C(10)	1·374 (5)
C(2)–P(3)	1·812 (4)	C(9)–C(14)	1·398 (5)
P(3)–C(4)	1·822 (5)	C(10)–C(11)	1·381 (5)
P(3)–C(9)	1·808 (4)	C(11)–C(12)	1·379 (6)
P(3)–O(15)	1·482 (3)	C(12)–C(13)	1·361 (6)
C(4)–C(5)	1·532 (6)	C(13)–C(14)	1·377 (6)
C(5)–C(6)	1·494 (6)		
C(7)–C(1)–C(2)	111·5 (3)	C(6)–C(5)–C(8)	100·7 (4)
C(2)–C(1)–C(8)	110·2 (4)	C(5)–C(6)–C(7)	111·2 (5)
C(7)–C(1)–C(8)	101·3 (4)	C(1)–C(7)–C(6)	110·2 (5)
C(1)–C(2)–P(3)	114·6 (3)	C(1)–C(8)–C(5)	101·8 (4)
C(2)–P(3)–C(4)	105·9 (2)	P(3)–C(9)–C(10)	125·6 (3)
C(2)–P(3)–C(9)	108·9 (2)	P(3)–C(9)–C(14)	116·2 (3)
C(4)–P(3)–C(9)	107·3 (2)	C(10)–C(9)–C(14)	118·0 (4)
C(2)–P(3)–O(15)	112·8 (2)	C(9)–C(10)–C(11)	121·6 (4)
C(4)–P(3)–O(15)	111·7 (2)	C(10)–C(11)–C(12)	119·7 (5)
C(9)–P(3)–O(15)	110·0 (2)	C(11)–C(12)–C(13)	119·4 (5)
P(3)–C(4)–C(5)	115·2 (3)	C(12)–C(13)–C(14)	121·4 (5)
C(4)–C(5)–C(6)	110·1 (4)	C(9)–C(14)–C(13)	119·9 (5)
C(4)–C(5)–C(8)	110·2 (4)		

C(1), C(5), C(4), and C(2) effectively coplanar. In the *exo*-phenyl isomer the projection of the plane of the phenyl ring essentially bisects the C(2)—P(3)—C(4) bond angle, whereas the considerable steric crowding in the *endo*-isomer causes the phenyl group to assume an orientation which results in the projection of its plane falling roughly parallel with the P(3)—C(2) bond vector. As a consequence of this, non-bonded intramolecular interaction occurs between the *endo*-hydrogen bonded to C(2) and the *ortho*-hydrogen bonded to C(10), these H atoms being only 2·28 (5) Å apart.

Whilst the bond lengths and bond angles in the *exo* and *endo*-isomers show a close similarity, there are some interesting and significant differences in the torsion and flap angles.* Thus, the magnitude of the angle between the planes C(2)—P(3)—C(4) and C(1)—C(2)—C(4)—C(5) decreases from 31° in the *exo*-isomer to 17·9 (4)° in the *endo*-isomer, presumably due to the distorting forces induced by the intramolecular interaction between H(101) and H(21) mentioned in the previous paragraph. To our knowledge no other phosphorinane ring structure so far studied has exhibited such a degree of flattening at the phosphorus end. In addition, the [3.2.1] ring system which in the *exo*-isomer approximates closely to a mirror plane, shows less evidence of such symmetry in the *endo*-isomer, due also to this intramolecular interaction.

We gratefully thank UPM, Dhahran, for a grant CY/CRYSTR/52 and Professor S.E. Cremer for providing crystals.

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* See previous footnote.

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N,N'-Diphenylhexanediamide (DPHD),* $C_{18}H_{20}N_2O_2$

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Abstract. $M_r = 296{\cdot}4$, orthorhombic, *Pbca*, $a = 15{\cdot}228\ (4)$, $b = 11{\cdot}265\ (3)$, $c = 9{\cdot}489\ (3)$ Å, $V = 1627{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}21$ Mg m^{-3}, $\lambda(\text{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}60$ mm^{-1}, $F(000) = 632$. Final $R =$ 4·2% for 686 observed reflexions. Molecules are connected by hydrogen bonds to four other molecules. The hydrogen-bonding scheme found is compared with that of related diamides. Two different types of hydrogen bonding are found. From the available data it is concluded that torsion angles can be influenced by hydrogen bonding.

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* The Structure of Model Compounds of Aromatic and Aromatic–Aliphatic Polyamides. IV. Part III: Harkema, van Hummel & Gaymans (1980).

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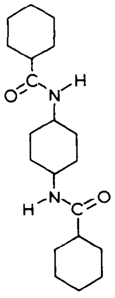
Introduction. DPHD can be considered to be a model compound for certain aromatic–aliphatic polyamides. In order to obtain detailed structural information on the units of which aromatic–aliphatic polyamides are composed, a single-crystal study of DPHD was carried out.

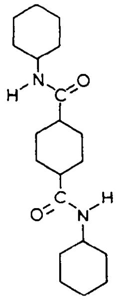
Experimental. DPHD prepared as described by Gaymans & Harkema (1977), crystals suitable for X-ray diffraction experiments obtained by crystallization from dimethylacetamide, intensities measured on a Philips PW1100 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scan mode, $3 < \theta < 58°$, 781 reflexions measured of which 686 with $I > \sigma(I)$ used for determination of crystal structure, no absorption correction applied; structure solved by *MULTAN* (Germain, Main & Woolfson, 1971), refined by full-matrix least squares (Busing, Martin & Levy, 1962), weight assigned to each reflexion $w = [\sigma(F_o) + 0{\cdot}01 \mid F_o \mid]^{-2}$, where $\sigma(F_o)$ is the e.s.d. of the structure factor (F_o) obtained from counting statistics, scattering factors for C, N and O from *International Tables for X-ray Crystallography* (1974), all H atoms found from a difference Fourier synthesis; for H scattering factor taken from Stewart, Davidson & Simpson (1965), parameters refined in last cycles: scale factor, extinction parameter, positional parameters, anisotropic thermal parameters for non-H atoms, isotropic thermal parameters for H, final $R = E_w = 4{\cdot}2\%$.

The asymmetric part of the unit cell contains one half molecule, the other half being generated by a center of symmetry.

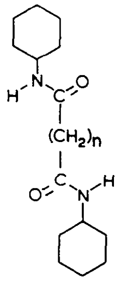
Discussion. Final atomic parameters are given in Table 1.* The atomic numbering is shown in Fig. 1. Bond lengths and angles and torsion angles are in Table 2. A stereoscopic view (Johnson, 1965) showing the molecular packing is given in Fig. 2. As in the related molecule *N,N'*-tetramethylenedibenzamide (TMDB) (Harkema, van Hummel & Gaymans, 1980), the tetramethylene part of the molecule has the extended zigzag chain conformation, the independent torsion angles being −174·5 (3) and 180·0 (0)°. The phenyl group is planar within experimental accuracy and C(8) and N deviate significantly from the planes of the amide group and the phenyl ring respectively.* The angle between the two planes is 28·9 (1)°. The angle found can be compared with the corresponding angle in the related compounds *N,N'*-diphenylterephthalamide (DPTP) (Harkema, Gaymans, van Hummel & Zylberlicht, 1979) and *N,N'*-(*p*-phenylene)dibenzamide (PPDB) (Harkema & Gaymans, 1977). The values found in these compounds are 30·4 and 35·9° respectively. The main reason for this rotation seems to be the repulsion between H(3) and O.



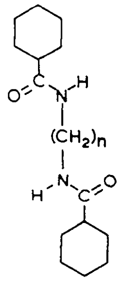
PPDB



DPTP



n = 4 DPHD



n = 2 DMDB
n = 4 TMDB

Molecules of DPHD are connected by N—H···O hydrogen bonds of length 2·840 (4) Å, with an N—H···O angle of 166 (3)°. Each molecule is hydrogen-bonded to four neighbouring molecules, which are symmetry-related by glide planes. The hydrogen-bonding scheme found here is the same as found in the related diamides *N,N'*-dimethylenedibenzamide (DMDB) (Palmer & Brisse, 1980) and PPDB (second polymorph) (Adams, Fratini & Wiff, 1978). A second hydrogen-bonding scheme, in which each molecule is hydrogen-bonded to two neighbours related by translation, is found in PPDB,

Table 1. *Fractional atomic coordinates* (×10⁴) *and equivalent isotropic thermal parameters* (Å² ×10³)

	x	y	z	U_{eq}*
C(1)	1829 (2)	−1884 (3)	783 (5)	69 (2)
C(2)	1375 (2)	−1358 (3)	−300 (5)	61 (2)
C(3)	1133 (2)	−176 (3)	−233 (4)	49 (1)
C(4)	1354 (2)	486 (2)	943 (4)	38 (1)
C(5)	1820 (2)	−46 (3)	2017 (4)	50 (1)
C(6)	2052 (2)	−1224 (3)	1935 (5)	66 (2)
C(7)	950 (2)	2491 (3)	113 (4)	44 (1)
C(8)	727 (3)	3720 (3)	650 (5)	62 (2)
C(9)	74 (3)	4367 (2)	−236 (5)	47 (1)
N	1088 (2)	1681 (2)	1131 (3)	40 (1)
O	1036 (2)	2282 (2)	−1143 (3)	63 (1)

* Defined according to Willis & Pryor (1975).

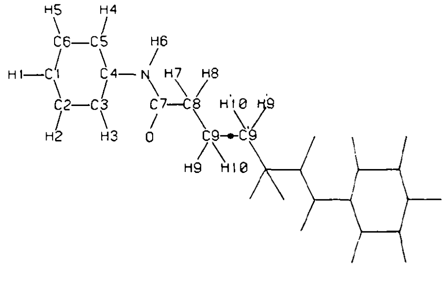


Fig. 1. Atomic arrangement of DPHD.

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* Lists of structure factors, anisotropic thermal parameters for the non-H atoms, positional and isotropic thermal parameters for the H atoms, complete lists of bond lengths and angles, and results of mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38239 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

C(1)–C(2)	1·373 (6)	C(5)–C(6)	1·376 (5)
C(1)–C(6)	1·365 (6)	C(7)–C(8)	1·513 (5)
C(2)–C(3)	1·383 (4)	C(7)–N	1·345 (5)
C(3)–C(4)	1·383 (5)	C(7)–O	1·221 (5)
C(4)–C(5)	1·379 (5)	C(8)–C(9)	1·492 (6)
C(4)–N	1·417 (3)	C(9)–C′(9)	1·511 (4)
C(2)–C(1)–C(6)	119·3 (5)	C(1)–C(6)–C(5)	120·5 (5)
C(1)–C(2)–C(3)	121·0 (4)	C(8)–C(7)–N	114·5 (3)
C(2)–C(3)–C(4)	119·4 (4)	C(8)–C(7)–O	121·9 (4)
C(3)–C(4)–C(5)	119·2 (4)	N–C(7)–O	123·6 (4)
C(3)–C(4)–N	123·0 (4)	C(7)–C(8)–C(9)	114·0 (4)
C(5)–C(4)–N	117·8 (3)	C(8)–C(9)–C′(9)	113·2 (4)
C(4)–C(5)–C(6)	120·6 (4)	C(4)–N–C(7)	126·8 (4)
C(6)–C(1)–C(2)–C(3)	−0·7 (4)	C(5)–C(4)–N–C(7)	152·2 (3)
C(2)–C(1)–C(6)–C(5)	0·4 (4)	C(4)–C(5)–C(6)–C(1)	0·4 (4)
C(1)–C(2)–C(3)–C(4)	0·1 (4)	C(8)–C(7)–N–C(4)	−176·1 (3)
C(2)–C(3)–C(4)–C(5)	0·7 (3)	N–C(7)–C(8)–C(9)	−143·9 (3)
C(2)–C(3)–C(4)–N	−176·2 (3)	O–C(7)–C(8)–C(9)	39·5 (4)
C(3)–C(4)–C(5)–C(6)	−0·9 (4)	O–C(7)–N–C(4)	0·5 (3)
C(3)–C(4)–N–C(7)	−30·9 (3)	C(7)–C(8)–C(9)–C′(9)	−174·5 (3)
N–C(4)–C(5)–C(6)	176·1 (3)		

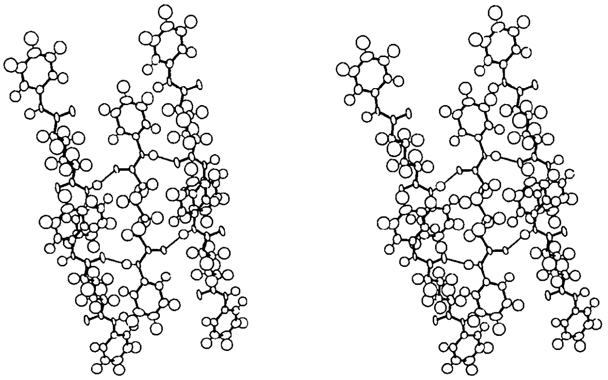

Fig. 2. Stereoscopic view of the crystal structure of DPHD.

DPTP and TMDB. Hydrogen bonds in the first group are shorter [N–H···O distances: DPHD 2·840 (4), DMDB 2·85 and PPDB (II) 2·857 (1) Å] than the hydrogen bonds in the second group [N–H···O distances: PPDB 3·111 (4), DPTP 3·118 (2) and TMDB 2·999 (4) Å]. The overall packing of the molecules, as judged from the cell volume, is better for the second group [cell volumes: PPDB 759, PPDB (II) 818 (× 2) Å^3]. Therefore it seems that stronger hydrogen bonding is possible at the expense of the efficiency of the overall packing. In the case of short hydrogen bonds, large deviations from ideal values of the torsion angles of the bonds between the amide group and the central (phenyl, tetramethylene or dimethylene) group of the molecule are found. For instance the torsion angle N–C(7)–C(8)–C(9) has the value −143·9 (3)° in DPHD. The corresponding C(7)–N–C(8)–C(9) torsion angle in TMDB is 169·5 (2)°, much closer to the expected *trans* value of 180°. Similar effects are found in DMDB and PPDB (II).

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Structure of 1,1′-Fluoroboranediylbis(3-fluorodimethylsilyl-2,2,4,4,6,6-hexamethylcyclotrisilazane), $C_{16}H_{50}BF_3N_6Si_8$

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(*Received* 6 *October* 1982; *accepted* 18 *November* 1982)

Abstract. $M_r = 619·1$, orthorhombic, *Pbcn*, $a = 18·764$ (2), $b = 14·633$ (2), $c = 13·098$ (2) Å, $U = 3596·4$ Å^3, $Z = 4$, $D_x = 1·143$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·32$ mm^{-1}, $F(000) = 1328$, $T =$

0108-2701/83/030387-03$01.50