

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

S—O(1)	1.431 (4)	C(2)—C(3)	1.522 (8)
S—O(2)	1.431 (4)	C(2)—C(7)	1.557 (8)
S—O(3)	1.584 (3)	C(2)—C(8)	1.568 (8)
S—C(1)	1.795 (6)	C(3)—C(4)	1.526 (9)
O(3)—C(12)	1.472 (6)	C(4)—C(5)	1.553 (9)
O(4)—C(13)	1.418 (6)	C(5)—C(6)	1.518 (8)
O(4)—C(14)	1.431 (6)	C(5)—C(8)	1.561 (8)
O(5)—C(13)	1.418 (6)	C(6)—C(7)	1.575 (8)
O(5)—C(15)	1.443 (6)	C(8)—C(9)	1.503 (8)
O(6)—C(3)	1.199 (7)	C(8)—C(10)	1.540 (8)
O(7)—C(23)	1.375 (8)	C(11)—C(12)	1.517 (8)
O(7)—C(26)	1.400 (7)	C(12)—C(13)	1.555 (7)
C(1)—C(2)	1.524 (8)	C(13)—C(18)	1.530 (8)
O(1)—S—O(2)	119.6 (2)	C(4)—C(5)—C(8)	101.0 (5)
O(1)—S—O(3)	104.0 (2)	C(6)—C(5)—C(8)	103.1 (5)
O(1)—S—C(1)	111.4 (3)	C(5)—C(6)—C(7)	103.1 (5)
O(2)—S—O(3)	109.0 (2)	C(2)—C(7)—C(6)	103.6 (4)
O(2)—S—C(1)	106.5 (2)	C(2)—C(8)—C(5)	93.9 (4)
O(3)—S—C(1)	105.5 (2)	C(2)—C(8)—C(9)	113.3 (5)
S—O(3)—C(12)	120.7 (3)	C(2)—C(8)—C(10)	112.5 (5)
C(13)—O(4)—C(14)	116.3 (4)	C(5)—C(8)—C(9)	113.2 (5)
C(13)—O(5)—C(15)	114.0 (4)	C(5)—C(8)—C(10)	114.9 (5)
C(23)—O(7)—C(26)	117.8 (4)	C(9)—C(8)—C(10)	108.6 (5)
S—C(1)—C(2)	117.5 (4)	O(3)—C(12)—C(11)	110.1 (4)
C(1)—C(2)—C(3)	115.6 (4)	O(3)—C(12)—C(13)	105.0 (4)
C(1)—C(2)—C(7)	117.7 (5)	C(11)—C(12)—C(13)	112.5 (5)
C(1)—C(2)—C(8)	114.3 (4)	O(4)—C(13)—O(5)	111.9 (4)
C(3)—C(2)—C(7)	105.5 (4)	O(4)—C(13)—C(12)	109.1 (4)
C(3)—C(2)—C(8)	98.8 (4)	O(4)—C(13)—C(18)	106.7 (4)
C(7)—C(2)—C(8)	102.6 (4)	O(5)—C(13)—C(12)	104.1 (4)
O(6)—C(3)—C(2)	126.5 (5)	O(5)—C(13)—C(18)	112.6 (4)
O(6)—C(3)—C(4)	126.7 (5)	C(12)—C(13)—C(18)	112.4 (4)
C(2)—C(3)—C(4)	106.7 (5)	O(7)—C(23)—C(22)	114.5 (5)
C(3)—C(4)—C(5)	101.9 (5)	O(7)—C(23)—C(24)	125.6 (6)
C(4)—C(5)—C(6)	108.2 (5)		

the dihedral angle between the C(13)—O(5)—C(15) and C(13)—O(4)—C(14) planes being 87(1)°. Four intramolecular distances less than 2.9 Å are observed for the O atoms of these methoxy groups. The values of O(4)...C(15), O(4)...C(19), O(5)...O(3) and O(5)...C(14) are 2.863 (6), 2.687 (6), 2.691 (5) and 2.810 (6) Å, respectively. The naphthyl moiety is essentially planar with deviations less than 0.05 Å for each atom in the least-squares plane. The torsion angle C(24)—C(23)—O(7)—C(26) is 2(1)°. The bond angle C(24)—C(23)—O(7) of 125.6(6)° is greater than C(22)—C(23)—O(7) of 114.5(5)° because of the steric interaction between C(24) and C(26) [2.821(9) Å].

The crystal structure consists of discrete molecules separated by normal van der Waals distances.* The shortest intermolecular distance is 3.185(8) Å for O(6)...C(14)(-½+x, ½-y, -z).

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* See deposition footnote.

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water and *N,N*-dimethylformamide. 1,2-rearrangement of the 6-methoxy-2-naphthyl group took place during the course of the hydrolysis. The absolute configuration around the asymmetric C atom of (II) could be deduced as *R* based on the known absolute configuration of (+)-(*R*)-α-(6-methoxy-2-naphthyl)propionic acid (Kitajima, Mitamura, Kobayashi, Tsuchihashi, Ohba & Saito, 1982). Thus it was revealed that the inversion of the absolute configuration around the asymmetric C atom occurs with the aryl-group rearrangement.

The torsion angle O(3)—C(12)—C(13)—C(18) is 60.9(5)°. The two bulky groups of 6-methoxy-2-naphthyl [C(18)] and 2-oxo-10-bornanesulfonate [O(3)] are in a *gauche* position with respect to the C(12)—C(13) bond axis. However, there is no short contact between these groups. The two methoxy groups bonded to the C(13) atom are bent into a spiral shape,

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The Structure of *N,N'*-Hexamethylenedibenzamide, C₂₀H₂₄N₂O₂

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Abstract. $M_r = 324.43$, monoclinic, $P2_1/c$, $a = 1.247 \text{ Mg m}^{-3}$, $\lambda(\text{Cu K}\alpha) = 1.54178 \text{ \AA}$, $\mu(\text{Cu K}\alpha) = 5.136 (2)$, $b = 5.346 (1)$, $c = 31.497 (12)$, $\beta = 0.61 \text{ mm}^{-1}$, $F(000) = 348$, $T = 295 \text{ K}$, $R = 0.044$ for 92.81 (3)°, $V = 863.8 \text{ \AA}^3$, $Z = 2$, $D_m = 1.23$, $D_x = 1219$ observed reflections. The structure of *N,N'*-

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hexamethylenedibenzamide is constituted of five nearly planar groups of atoms: the central $\text{NH}-(\text{CH}_2)_6-\text{NH}$ sequence, two amide groups which are $8.8 (4)^\circ$ from this plane and two benzene rings tilted by $29.3 (6)^\circ$ from the amide planes. Each molecule is hydrogen bonded through four hydrogen bonds to two other molecules. The molecules so linked form a ladder-like ribbon extending in the \mathbf{a} direction.

Introduction. In order to be able to provide a reasonable starting structure for the aromatic nT Nylons, $[-\text{C}_6\text{H}_4-\text{CO}-\text{NH}(\text{CH}_2)_n-\text{NH}-\text{CO}-]$, whose member poly(hexamethyleneterephthalamide) is the best known (Nylon 6T), we have undertaken a systematic study of their related model compounds. These are the N,N' -oligomethylenedibenzamides of the type $\text{C}_6\text{H}_5-\text{CO}-\text{NH}-(\text{CH}_2)_n-\text{NH}-\text{CO}-\text{C}_6\text{H}_5$. The crystal structures for various values of n have already been reported; $n = 2$ (Palmer & Brisse, 1980), $n = 4$ (Harkema, van Hummel & Gaymans, 1980), $n = 7$ (Brisson & Brisse, 1982). In all these compounds, the $\text{NH}-(\text{CH}_2)_n-\text{NH}$ sequence is nearly planar and is almost in the fully extended conformation. Although the dihedral angle between the phenyl ring and the amide group has values between 20 and 30° for all the above mentioned compounds, the relative orientation of the amide group with respect to the $\text{NH}-(\text{CH}_2)_n-\text{NH}$ plane does not remain constant as n changes. There are two distinct orientations. For $n = 2$ and 7 , the dihedral angle is about 90° while for $n = 4$ this angle is near 10° . Because of this variation we decided to establish the crystal structure of N,N' -hexamethylenedibenzamide ($n = 6$) since its structure is more likely to be related to that of Nylon 6T.

Experimental. Slow addition of benzoyl chloride to an ether solution of 1,6-hexanediamine in presence of pyridine (Gaymans & Harkema, 1977), recrystallization in 100% ethanol, density by flotation in ZnCl_2 solution, m.p. = 434 K , colorless crystals, $0.12 \times 0.20 \times 0.20 \text{ mm}$, Nonius CAD-4 diffractometer, graphite-monochromatized $\text{Cu K}\alpha$, orientation verified every 100 reflections, intensity check every hour using three reference reflections, largest intensity fluctuation 2.1%, 1632 hkl , hkl ($h = 0-6$, $k = 0-6$, $l = \overline{38}-38$) with $2\theta \leq 140^\circ$, 1219 with $I \geq 1.90\sigma(I)$, 413 unobserved, L_p correction, no absorption correction; direct methods (MULTAN, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), anisotropic block-diagonal least-squares refinement based on F , H (calculated) isotropic, final $R = 0.044$ (observed), $R = 0.061$ (all measured), $R_w = 0.076$, $w = 1$, maximum (shift/ σ) = 0.85 , mean (shift/ σ) = 0.28 , final electron density fluctuations $-0.25, +0.18 \text{ e \AA}^{-3}$, scattering factors for O, N and C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); programs used in this work are modified versions of NRC-2 (data reduction),

NRC-10 (bond distances and angles), NRC-22 (mean planes) (Ahmed, Hall, Pippy & Huber, 1973), FORDAP (Fourier & Patterson maps, A. Zalkin), NUCLS (least-squares refinement, Doedens & Ibers, 1967), MULTAN (Main *et al.*, 1978), ORTEP (stereoviews, Johnson, 1965).*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38636 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and their e.s.d.'s ($\times 10^4$ for O, N and C; $\times 10^3$ for H), U_{eq} ($\text{\AA}^2 \times 10^4$ for O, N and C) and U_{iso} ($\text{\AA}^2 \times 10^3$ for H)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
O(1)	2907 (2)	8375 (3)	9012 (1)	570
N(1)	7168 (3)	9327 (3)	9087 (1)	431
C(1)	5725 (3)	6378 (3)	8541 (1)	390
C(2)	7759 (4)	6787 (4)	8276 (1)	498
C(3)	8099 (4)	5180 (5)	7934 (1)	589
C(4)	6453 (4)	3176 (4)	7862 (1)	589
C(5)	4456 (4)	2753 (4)	8127 (1)	563
C(6)	4076 (4)	4352 (4)	8463 (1)	483
C(7)	5159 (3)	8102 (4)	8899 (1)	414
C(8)	6763 (3)	11074 (4)	9433 (1)	457
C(9)	9185 (3)	12546 (4)	9555 (1)	440
C(10)	8781 (3)	14284 (3)	9928 (1)	439
H(2)	897 (3)	827 (4)	833 (1)	57 (5)
H(3)	957 (4)	554 (4)	774 (1)	72 (6)
H(4)	671 (4)	208 (4)	760 (1)	81 (7)
H(5)	318 (4)	129 (4)	806 (1)	83 (7)
H(6)	263 (4)	408 (4)	866 (1)	63 (6)
H(81)	529 (3)	1221 (4)	934 (1)	60 (6)
H(82)	618 (4)	1014 (4)	970 (1)	66 (6)
H(91)	978 (4)	1352 (4)	931 (1)	66 (6)
H(92)	1067 (3)	1142 (4)	963 (1)	59 (6)
H(101)	732 (4)	1549 (4)	985 (1)	63 (6)
H(102)	814 (4)	1330 (4)	1017 (1)	60 (6)
H(N)	882 (3)	895 (4)	902 (1)	55 (5)

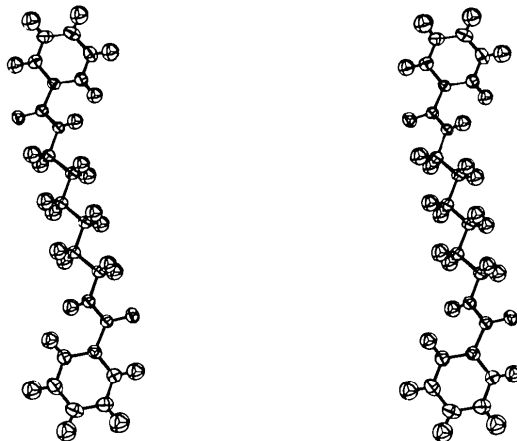


Fig. 1. Stereopair showing one molecule of N,N' -hexamethylenedibenzamide.

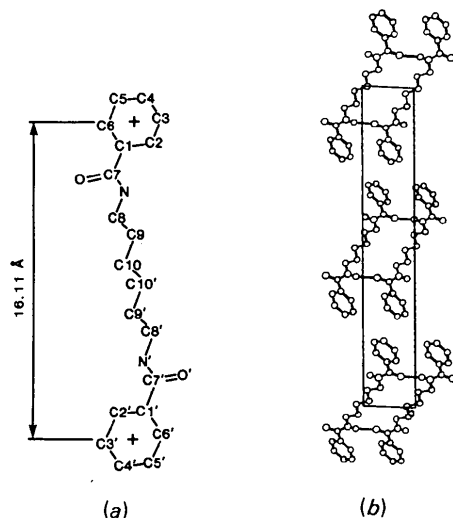


Fig. 2. (a) Atomic numbering used for *N,N'*-hexamethylenedibenzamide, and the measure of the equivalent fiber repeat. (b) Association of *N,N'*-hexamethylenedibenzamide through hydrogen bonds forming a ladder-like ribbon parallel to **a**. **a** is horizontal and **c** is vertical.

Table 2. Bond distances (Å), angles and torsion angles (°) in *N,N'*-hexamethylenedibenzamide

C(1)–C(2)	1.386 (2)	C(7)–O(1)	1.235 (2)
C(2)–C(3)	1.396 (3)	C(7)–N(1)	1.336 (2)
C(3)–C(4)	1.377 (3)	N(1)–C(8)	1.458 (3)
C(4)–C(5)	1.373 (3)	C(8)–C(9)	1.506 (3)
C(5)–C(6)	1.382 (3)	C(9)–C(10)	1.520 (3)
C(6)–C(1)	1.389 (3)	C(10)–C(10')*	1.518 (5)
C(1)–C(7)	1.496 (2)		
C(1)–C(2)–C(3)	119.6 (2)	C(1)–C(7)–O(1)	120.6 (2)
C(2)–C(3)–C(4)	120.5 (2)	N(1)–C(7)–O(1)	121.9 (2)
C(3)–C(4)–C(5)	120.0 (2)	C(1)–C(7)–N(1)	117.5 (2)
C(4)–C(5)–C(6)	120.1 (2)	C(7)–N(1)–C(8)	120.7 (2)
C(5)–C(6)–C(1)	120.7 (2)	N(1)–C(8)–C(9)	112.3 (2)
C(6)–C(1)–C(2)	119.2 (2)	C(8)–C(9)–C(10)	112.0 (2)
C(2)–C(1)–C(7)	122.5 (2)	C(9)–C(10)–C(10')	113.5 (3)
C(6)–C(1)–C(7)	118.3 (2)		
O(1)–C(7)–C(1)–C(6)	–27.9(4)	C(1)–C(7)–N(1)–C(8)	178.9 (4)
O(1)–C(7)–C(1)–C(2)	149.5 (4)	O(1)–C(7)–N(1)–C(8)	–0.8 (4)
N(1)–C(7)–C(1)–C(6)	152.5 (4)	C(7)–N(1)–C(8)–C(9)	–171.2 (4)
N(1)–C(7)–C(1)–C(2)	–30.2 (4)	N(1)–C(8)–C(9)–C(10)	–177.6 (4)
		C(8)–C(9)–C(10)–C(10')	177.5 (6)

* The primed atoms are centrosymmetrically related to the unprimed atom having the same number. Since the atoms are symmetry related, the e.s.d.'s have been multiplied by a factor of $\sqrt{2}$.

Discussion. The final atomic coordinates are given in Table 1. A stereopair showing the molecule is given in Fig. 1. The atomic numbering is shown in Fig. 2(a) while bond distances, angles and torsion angles are given in Table 2.

Comparing the geometry of this compound to that of the related hexamethylene dibenzoate (Pérez & Brisse, 1977), in which NH is replaced by O, one notes that the $C(sp^3)–C(sp^3)$ distances in the methylenic sequence of the dibenzamide are not as short as in the benzoate. The

shortest and mean distances are 1.506 (3) and 1.515 (4) Å (benzamide) and 1.485 (4) and 1.501 Å (benzoate). The molecule of *N,N'*-hexamethylenedibenzamide is shown in Fig. 1 where one may recognize the following planar groups of atoms: the aromatic plane [atoms C(1) to C(6)], *A*, the amide plane composed of C(1), C(7), N(1) and O(1), *B*, and the methylene plane, *C*, including C(8), C(9), C(10) and the centrosymmetrically related atoms. The dihedral angles *A–B*, *A–C* and *B–C* have the values of 29.3 (6), 20.6 (5) and 8.8 (4)°, respectively. It is then clear that *N,N'*-hexamethylenedibenzamide has a conformation quite similar to that of the tetramethylene analog (Harkema *et al.*, 1980). Although the methylenic sequence is planar and in the fully extended conformation in both the benzoate and the benzamide, the replacement of the oxygen atom (benzoate) by NH (benzamide) results in a totally different situation since the dihedral angles *A–B*, *A–C* and *B–C* in the benzoate have values of 3.3 (5), 2.5 (5) and 5.8 (3)°, respectively.

This of course is brought about by the H bonding that links each *N,N'*-hexamethylenedibenzamide to two adjacent molecules. This H bonding is characterized by the following quantities: $N(1)\cdots O(1) = 3.012$ (3), $N(1)–H(N) = 0.91$ (2), $H(N)\cdots O(1) = 2.12$ (2) Å, $N(1)–H(N)\cdots O(1) = 165$ (1)°. The molecules so bonded form a ribbon or a ladder-like association parallel to the **a** direction, as shown in Fig. 2(b).

Although the structure of Nylon 6*T* has not yet been established, Quynn (1970) managed to record a fiber diagram for this poly(terephthalamide). Then, Morgan & Kwolek (1975) obtained the value of $c = 15.6$ Å for the fiber repeat of this polymer. The equivalent fiber repeat, that is the distance between the centers of the benzene rings, calculated from the model compound, is $c = 16.11$ Å (see Fig. 2a). It is possible to match the experimental fiber repeat of Nylon 6*T* simply by varying the torsion angle C(7)–N(1)–C(8)–C(9) from its value of $–171.2$ (4)° to $–93$ or $+99$ °. In this latter case the molecular conformation is similar to that of *N,N'*-ethylenedibenzamide (Palmer & Brisse, 1980).

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The Structure of N,N' -Octamethylenedibenzamide, $C_{22}H_{28}N_2O_2$

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Abstract. $M_r = 352.48$, monoclinic, $P2_1/c$, $a = 5.141$ (3), $b = 5.364$ (2), $c = 34.715$ (12) Å, $\beta = 90.57$ (4)°, $V = 957.3$ Å³, $Z = 2$, $D_m = 1.24$, $D_x = 1.222$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu(\text{Cu } K\alpha) = 0.58$ mm⁻¹, $F(000) = 380$, $T = 295$ K, $R = 0.046$ for 1245 observed reflections. The structure of N,N' -octamethylenedibenzamide is similar to that of the hexamethylene analog. The central NH—(CH₂)₈—NH sequence is planar. The two amide groups are tilted by 8.6 (8)° from the methylenic sequence and the benzene rings are inclined by 29.2 (5)° from the plane of the amide groups. The molecules are hydrogen bonded and form ribbons parallel to the **a** direction.

Introduction. The nT Nylons of the type [—C₆H₄—CO—NH—(CH₂) _{n} —NH—CO—] have the molecules of N,N' -oligomethylenedibenzamides, C₆H₅—CO—NH—(CH₂) _{n} —NH—CO—C₆H₅, as model compounds. These can be sorted into two categories. In the first one, which includes compounds with $n = 2$ and 7 (Palmer & Brisse, 1980; Brisson & Brisse, 1982), the amide plane is at 90° from the planar NH—(CH₂) _{n} —NH sequence. The second category, composed of compounds with $n = 4$ and 6 (Harkema, van Hummel & Gaymans, 1980; Pineault & Brisse, 1983), is characterized by a dihedral angle close to 10°. We have undertaken to establish the crystal structure of the title compound to try to rationalize the conformations adopted by the N,N' -oligomethylene-dibenzamide molecules as the number of methylene groups increases.

Experimental. Synthesis described elsewhere (Pineault, 1982), colorless crystals, 0.08 × 0.40 × 0.40 mm, from ethanol, m.p. = 445 K, density by flotation in ZnCl₂ solution, Nonius CAD-4 diffractometer, graphite monochromatized Cu $K\alpha$, orientation verified every 100

reflections, intensity check every hour with three standard reflections, largest intensity fluctuation 1.4%, 1633 hkl , hkl (h 0–5, k 0–6, l 42–42) with $2\theta \leq 140.0^\circ$, 1249 with $I \geq 2.50\sigma(I)$, 384 unobserved, Lp correction, no absorption correction; direct methods (*MULTAN*, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), anisotropic block-diagonal least-squares refinement based on F , H (calculated) isotropic, final $R = 0.046$ (observed, four reflections were given zero weight in the last cycles of the least-squares refinement), $R = 0.064$ (all data), $R_w = 0.048$, $w = 1$, maximum (shift/ σ) = 0.90, mean (shift/ σ) = 0.35, final electron density fluctuations -0.18 , $+0.27$ e Å⁻³, scattering factors for O, N and C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); programs used in this work are the same as in Pineault & Brisse (1983).*

Discussion. The final atomic coordinates are given in Table 1. The atomic numbering is shown in Fig. 1 while the bond distances, angles and torsion angles are given in Table 2.

The molecule consists of two halves related by a crystallographic center of symmetry situated at the midpoint of the central C—C bond (see Fig. 1).

Except for the number of methylene groups, this compound is identical to the tetra- and hexamethylene analogs whose structures were reported earlier. The similarity is noticeable in the conformation adopted by these molecules and in the hydrogen-bonding scheme. In order to identify the dihedral angles of interest, the benzene ring, the amide group of atoms and the

* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38637 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.