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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' -oligomethylenedibenzamide 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.

sequence of eight carbon atoms in the central part of the molecule are defined as planes *A*, *B* and *C*, respectively. The dihedral angles *A*–*B*, *A*–*C* and *B*–*C* have the values 29.2 (5), 20.6 (5) and 8.6 (8)°, respectively. These values are within 0.2° of those

observed in *N,N'*-hexamethylenedibenzamide and within 2° of those calculated from the data given by Harkema *et al.* (1980) for *N,N'*-tetramethylenedibenzamide. Each molecule is hydrogen-bonded to two others through four H bonds forming a ribbon parallel to **a**. This arrangement is shown by the stereopair in Fig. 2. The relevant bond distances and angles are as follows: N(1)···O(1) = 3.011 (3), N(1)–H(N) = 0.89 (3), H(N)···O(1) = 2.14 (3) Å and N(1)–H(N)···O(1) = 166 (2)°. The equivalent of the fiber repeat of Nylon 8T, *i.e.* the distance between the centers of two consecutive benzene rings, is 18.64 (4) Å (see Fig. 1).

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} (Å² $\times 10^4$ for O, N and C) and U_{iso} (Å² $\times 10^3$ for H)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}/U_{iso}
O(1)	–2643 (3)	3175 (4)	6135 (1)	579
N(1)	1594 (4)	2235 (4)	6066 (1)	443
C(1)	348 (5)	5181 (5)	6560 (1)	403
C(2)	–1277 (5)	7206 (5)	6631 (1)	486
C(3)	–789 (6)	8802 (6)	6933 (1)	563
C(4)	1322 (6)	8399 (6)	7174 (1)	582
C(5)	2947 (5)	6392 (7)	7109 (1)	587
C(6)	2478 (5)	4796 (6)	6800 (1)	490
C(7)	–342 (5)	3454 (5)	6236 (1)	423
C(8)	1059 (5)	489 (5)	5752 (1)	465
C(9)	3452 (5)	–988 (5)	5644 (1)	435
C(10)	2902 (5)	–2721 (5)	5306 (1)	442
C(11)	5275 (5)	–4152 (5)	5172 (1)	440
H(2)	–282 (5)	750 (5)	648 (1)	66 (9)
H(3)	–207 (5)	1025 (6)	700 (1)	74 (9)
H(4)	163 (6)	942 (6)	746 (1)	100 (11)
H(5)	459 (5)	610 (5)	728 (1)	68 (9)
H(6)	366 (5)	347 (5)	674 (1)	67 (9)
H(8(1))	–39 (5)	–66 (5)	583 (1)	72 (9)
H(8(2))	31 (5)	142 (6)	551 (1)	74 (9)
H(9(1))	502 (5)	21 (6)	557 (1)	67 (9)
H(9(2))	402 (5)	–193 (5)	588 (1)	59 (8)
H(10(1))	148 (5)	–389 (6)	539 (1)	72 (9)
H(10(2))	221 (5)	–172 (5)	506 (1)	68 (9)
H(11(1))	667 (5)	–300 (5)	511 (1)	57 (8)
H(11(2))	610 (5)	–510 (5)	541 (1)	64 (8)
H(N)	321 (4)	259 (5)	613 (1)	47 (7)

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

C(1)–C(2)	1.394 (4)	O(1)–C(7)	1.239 (3)
C(2)–C(3)	1.375 (4)	C(7)–N(1)	1.334 (3)
C(3)–C(4)	1.381 (4)	N(1)–C(8)	1.461 (4)
C(4)–C(5)	1.383 (5)	C(8)–C(9)	1.514 (4)
C(5)–C(6)	1.392 (4)	C(9)–C(10)	1.521 (4)
C(6)–C(1)	1.385 (4)	C(10)–C(11)	1.518 (4)
C(1)–C(7)	1.497 (4)	C(11)–C(11')*	1.526 (6)
C(1)–C(2)–C(3)	121.0 (3)	C(1)–C(7)–O(1)	120.4 (2)
C(2)–C(3)–C(4)	120.1 (3)	N(1)–C(7)–O(1)	122.0 (2)
C(3)–C(4)–C(5)	119.7 (3)	C(1)–C(7)–N(1)	117.6 (1)
C(4)–C(5)–C(6)	120.3 (3)	C(7)–N(1)–C(8)	120.6 (2)
C(5)–C(6)–C(1)	120.1 (3)	N(1)–C(8)–C(9)	111.9 (2)
C(6)–C(1)–C(2)	118.8 (3)	C(8)–C(9)–C(10)	111.5 (2)
C(2)–C(1)–C(7)	118.5 (2)	C(9)–C(10)–C(11)	113.7 (2)
C(6)–C(1)–C(7)	122.7 (2)	C(10)–C(11)–C(11')	113.4 (3)
O(1)–C(7)–C(1)–C(6)	149.5 (5)	O(1)–C(7)–N(1)–C(8)	–0.5 (5)
O(1)–C(7)–C(1)–C(2)	–28.1 (5)	C(7)–N(1)–C(8)–C(9)	–170.9 (4)
N(1)–C(7)–C(1)–C(6)	–29.9 (4)	N(1)–C(8)–C(9)–C(10)	–177.6 (4)
N(1)–C(7)–C(1)–C(2)	152.5 (4)	C(8)–C(9)–C(10)–C(11)	177.0 (3)
C(1)–C(7)–N(1)–C(8)	179.0 (4)	C(9)–C(10)–C(11)–C(11')	–178.9 (6)

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

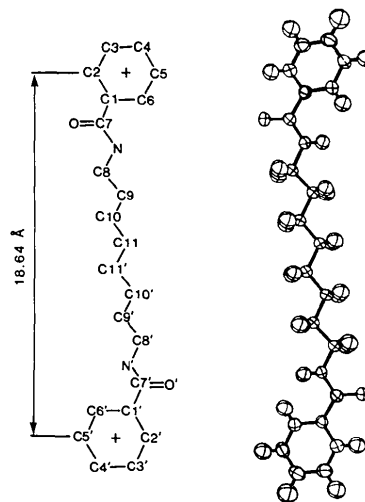


Fig. 1. View of the *N,N'*-octamethylenedibenzamide molecule, the atomic numbering adopted and the equivalent fiber repeat.

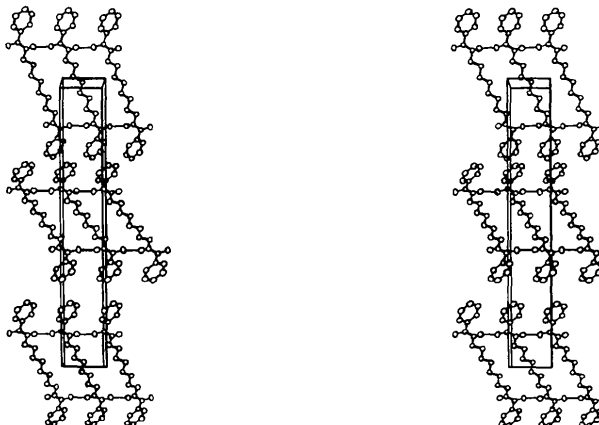


Fig. 2. Stereoview of the hydrogen-bonding system forming ribbons parallel to **a**. **a** is horizontal while **c** is vertical.

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Structure of *N*-Ethylmaleimide, C₆H₇NO₂

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Abstract. $M_r = 125.12$, monoclinic, $P2_1/c$, $a = 11.875$ (7), $b = 6.681$ (5), $c = 8.393$ (3) Å, $\beta = 103.73$ (4)°, $V = 646.84$ Å³, $Z = 4$, $D_x = 1.286$ Mg m⁻³, Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å, m.p. = 318.7 K, $F(000) = 264$, $\mu = 0.830$ mm⁻¹, room temperature, final $R = 0.067$ for 761 reflections with $I > 3\sigma(I)$. Thermal diffuse scattering is strongly associated with the intense low-angle reflections. The molecules are held in the crystalline lattice solely by van der Waals interactions, thus accounting for the high vapor pressure.

Introduction. *N*-Ethylmaleimide (NEM) reacts quickly and specifically with thiol groups. It is used to determine numbers of sulfhydryl groups in peptides and proteins, and the effects of sulfhydryl-group modifications on proteins (Means & Feeny, 1971).

Experimental. NEM was obtained in crystalline form, 99+% pure, from Mann Research Laboratories, Inc. To prevent sublimation of the crystal (m.p. 318.7 K), it was mounted inside a glass capillary tube with smaller crystals, and the tube sealed. Irregular rectangular prism, 1.2 × 0.4 × 0.3 mm, Enraf–Nonius CAD-4 diffractometer, cell measurement with reflections in the range $\theta = 8$ –52°, $\omega/2\theta$ scan of width 1.5° and variable speed, absorption correction by the empirical method of North, Phillips & Mathews (1968) with a maximum correction of 1.271 and a minimum correction of 1.003, maximum $(\sin\theta)/\lambda = 0.5614$ Å⁻¹, range in $h = -13$ to 13, $k = 0$ to 7, $l = -9$ to 9, average decay of standard reflections 2.0%, 2177 reflections measured, 951 unique reflections, $R_{\text{merge}} = 3.34\%$, 190 unobserved reflections with $I < 3\sigma(I)$. Data reduction, calculation of Fourier maps and least-squares refine-

ment performed with the programs of the XRAY70 system of Stewart, Kundell & Baldwin (1970).^{*} Structure solved by direct methods using *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). The non-hydrogen atoms were all located readily on the E map. Full-matrix least-squares refinement on $|F|$ was performed with $w = [\sigma^2(I)]^{-1}$ [from $\sigma^2(I)$ as defined in Fujinaga & James (1980)]. H atoms were located at an intermediate stage of refinement from a $\Delta\rho$ map. In the final cycles of refinement positional parameters for all atoms, anisotropic thermal-vibration parameters for non-hydrogen atoms, isotropic thermal-vibration parameters for H atoms and the structure factor scale factor were varied; $R_w = 0.067$. Maximum and average Δ/σ in final cycle 0.46 and 0.04 respectively. Max. and min. peaks on final $\Delta\rho$ map 0.23 and -0.22 e Å⁻³. Atomic scattering factors for non-hydrogen atoms from Cromer & Mann (1968), for H from Mason & Robertson (1966). No correction for secondary extinction was required.

Discussion. Positional and equivalent isotropic thermal-vibration parameters are given in Table 1 for non-hydrogen atoms, and a view of the molecule is given in Fig. 1.†

^{*} C. G. Broughton's map-display and molecule-manipulation program *M3*, which runs on an MMS-X interactive graphics system of Barry, Molnar & Rosenberger (1976), was invaluable in interpretation of Fourier maps, assignment of initial atomic coordinates and composition of figures.

† Lists of structure factors, anisotropic thermal parameters for non-hydrogen atoms, and coordinates and isotropic thermal parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38683 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.