there might also hold here. An attractive intramolecular O(2)···C(11)=O(3) interaction (Bürgi, Dunitz & Shefter, 1974) might be the driving force towards such a distortion. Although the lone-pair orbital of O(2) cannot point exactly towards C(11), the tendency is to reduce the O(2)···C(11)=O(3) approach angle (116·5°) to the optimal value (100–110°) (Bürgi, Dunitz & Shefter, 1974).

The unsubstituted six-membered ring adopts the chair conformation. Torsion angles (Fig. 1) are related by 'mirror symmetry' passing through C(6) and C(9). The six-membered rings in [4.4.4]propellane (Ermer, Gerdil & Dunitz, 1971) also adopt the chair conformation but the 'mirror symmetry' of the torsion angles passes through C(7) and C(10) (the notation is according to the present work), parallel to the C(1) and C(6) bridging atoms. The difference is attributed to the distortion described above.

The five-membered ring has an envelope shape with C(6) as the flap. Mean-plane calculations show that C(1), C(11), C(12), and N are planar (out of plane displacement range: −0·03 to 0·03 Å) while C(6) is found to lie −0·42 Å from that plane; O(4) and C(13) are displaced by 0·11 Å towards the epoxides.

The packing of the molecules is shown in Fig. 3. Intermolecular distances shorter than the sum of the van der Waals radii were not observed.

The author thanks Professor D. Ginsburg and Dr I. J. Landheer for providing the sample.

References

BIRNBAUM, G. I. (1973). *Acta Cryst.* B**29**, 1426–1432.

BÜRGI, H. B., DUNITZ, J. D. & SHEFTER, E. (1974). *Acta Cryst.* B**30**, 1517–1527.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

ERMER, O., GERDIL, R. & DUNITZ, J. D. (1971). *Helv. Chim. Acta*, **54**, 2476–2487.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

KAFTORY, M. & DUNITZ, J. D. (1976). *Acta Cryst.* B**32**, 619–622.

LANDHEER, I. J. & GINSBURG, D. (1977). To be published.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN* 74, *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York/Louvain.

PAQUETTE, L. A., LIOTTA, D. C., LIAO, C. C., WALLIS, T. G., EICKMAN, N., CLARDY, J. & GLEITER, R. (1976). *J. Am. Chem. Soc.* **98**, 6413–6417.

SANDS, D. E. (1972). *Acta Cryst.* B**28**, 2463–2468.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

VOGEL, E., BREUER, A., SOMMERFELD, C. D., DAVIS, R. E. & LIU, L. K. (1977). *Angew. Chem. Int. Ed. Engl.* **16**, 169–170.

Acta Cryst. (1978). B**34**, 954–956

N,*N'*-Dibenzoyl-*p*-phenylenediamine

BY W. W. ADAMS*

Air Force Materials Laboratory, Wright–Patterson Air Force Base, Ohio 45433, *USA*

AND A. V. FRATINI† AND D. R. WIFF‡

University of Dayton, Dayton, Ohio 45469, *USA*

(*Received* 12 *August* 1977; *accepted* 20 *October* 1977)

Abstract. $C_{20}H_{16}N_2O_2$, orthorhombic, *Pbca*, $Z = 4$, $a = 9{\cdot}540$ (7), $b = 18{\cdot}273$ (9), $c = 9{\cdot}387$ (3) Å, $V = 1636$ Å^3, $D_x = 1{\cdot}285$, $D_m = 1{\cdot}29$ g cm^{-3} (by flotation in aqueous CsCl), λ(Mo $K\alpha$) = 0·7107 Å, $R = 0{\cdot}089$ for 1876 reflections. Centrosymmetric molecules are linked together by N—H···O hydrogen bonds. The angle between the phenylenediamine segment and the amide plane, expressed as the torsion angle C(7)—N—C(8)—C(10), is −56·3°. The amide plane is nearly flat with a torsion angle about the C(7)—N bond [O—C(7)—N—C(8)] of +1·4°.

Introduction. The compound was prepared by Dr Fred Arnold of the Air Force Materials Laboratory and recrystallized by vacuum sublimation at 330 °C to yield single crystals in the form of clear plates. Weissenberg and precession photographs were used to identify the crystal system and space group. A platelet with a

---

* To whom correspondence should be addressed.

† Department of Chemistry.

‡ Research Institute.

volume of 0·022 mm^3 was then mounted on a Picker FACS-I diffractometer with the longest direction (corresponding to **c**) parallel to the φ axis. Precise cell constants were obtained by a least-squares fit of angular positions of 14 Friedel pairs (31° < 2θ < 36°).

The θ–2θ scan mode was used to measure 1876 independent reflections with Nb-filtered Mo $K\alpha$ radiation to a maximum 2θ of 55°. Stationary backgrounds were measured at equal angles above and below the respective $K\alpha$ peak of base width 1·2°. The diffractometer control programs used were adapted by Lenhert (1975) for a DF-32 disk. A dispersion factor allowing for $\alpha_1\alpha_2$ splitting was applied at large 2θ values. Three standard reflections were monitored every 80 measurements to check the crystal alignment and stability; no decrease in intensity of the standards was observed.

The structure was readily determined from phases obtained with the *NORMSF*, *SINGEN* and *PHASE* programs of the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Full-matrix anisotropic least-squares refinement of nonhydrogen atoms was carried out with *CRYLSQ* of XRAY 72, followed by a difference Fourier synthesis in which all H atoms were located. Isotropic temperature factors were used for H atom refinement. The weight of the reflection was assigned as $1/\sigma^2(F)$, where $\sigma(F)$ was calculated from counting statistics as described by Miller, Lenhert & Joesten (1972). The final R index for all measured reflections $(\Sigma||F_o| - |F_c||/\Sigma|F_o|)$ was 0·089. The magnitude of $[\Sigma\, w(F_o - F_c)^2/(m - n)]^{1/2}$, where m is the number of reflections and n is the number of parameters refined, was 1·28. The maximum shift/error ratios were 0·04 and 0·17 for nonhydrogen and H atoms respectively. A final difference synthesis showed no peaks above 0·35 e Å^{-3}. Table 1 lists the final positional parameters with e.s.d.'s.* Atomic scattering factors for nonhydrogen atoms were those of Cromer & Mann (1968) and for H those of Stewart, Davidson & Simpson (1965).

Table 1. *Positional parameters* (×10^4)

Estimated standard deviations are in parentheses.

	x	y	z
O	−1215 (1)	4014 (1)	1693 (1)
N	954 (2)	4312 (1)	2501 (2)
C(1)	735 (2)	3610 (1)	318 (2)
C(2)	2064 (2)	3765 (1)	−191 (2)
C(3)	2592 (2)	3373 (1)	−1335 (2)
C(4)	1825 (2)	2829 (1)	−1974 (2)
C(5)	501 (2)	2668 (1)	−1459 (2)
C(6)	−59 (2)	3061 (1)	−322 (2)
C(7)	65 (2)	4001 (1)	1550 (2)
C(8)	461 (2)	4667 (1)	3767 (2)
C(9)	966 (2)	4429 (1)	5083 (2)
C(10)	−499 (2)	5237 (1)	3687 (2)
H(2)	2613 (15)	4167 (8)	183 (17)
H(3)	3380 (20)	3556 (9)	−1700 (18)
H(4)	2264 (18)	2480 (11)	−2758 (20)
H(5)	−76 (18)	2260 (10)	−1813 (18)
H(6)	−1083 (16)	2948 (8)	69 (18)
H(9)	1707 (15)	3985 (8)	5134 (16)
H(10)	−906 (16)	5413 (7)	2746 (17)
H(11)	1929 (19)	4220 (10)	2471 (21)

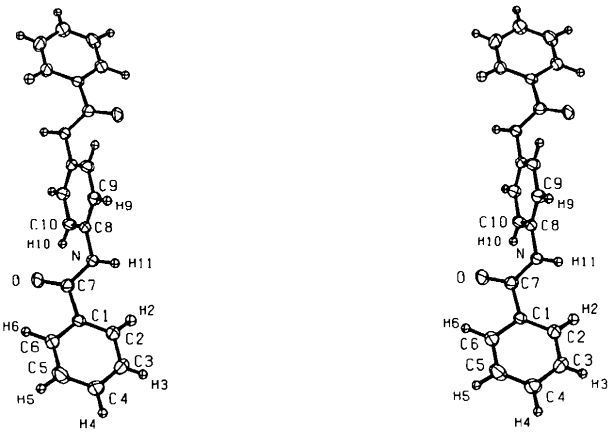
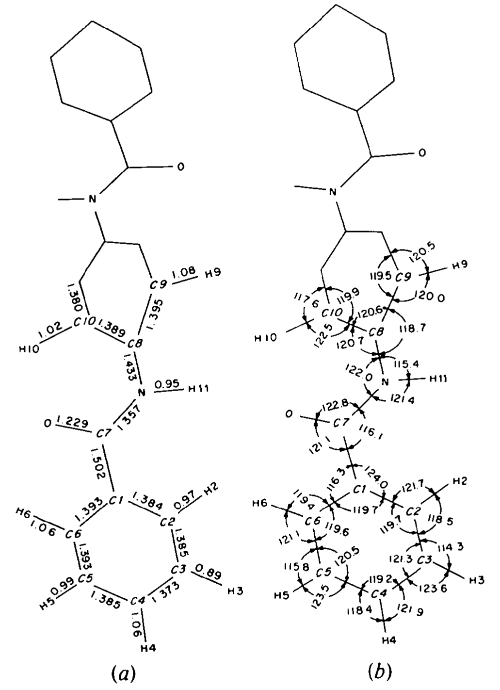


Fig. 1. Stereoview of *N,N'*-dibenzoyl-*p*-phenylenediamine.



(*a*) (*b*)

Fig. 2. (*a*) Bond distances (Å) and (*b*) bond angles (°) in the molecule.

---

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33093 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

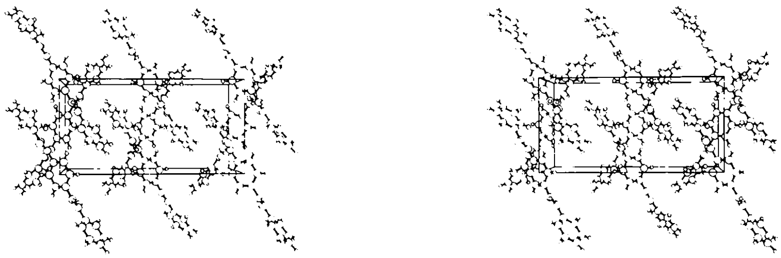


Fig. 3. Stereoview of the packing of molecules in the crystal (*a* axis projection, with the *b* axis horizontal and the *c* axis vertical).

Discussion. The aromatic polyamides belong to a class of polymers which have unique thermal and mechanical properties compared to other kinds of polyamides (Black, 1973; Frank, 1970). Fibers made from this group of polymers often exhibit high tensile strength, high elastic modulus and good thermal resistance. Tashiro, Kobayashi & Tadokoro (1977), in an attempt to develop new methods for X-ray analysis of polymers, have combined fiber diffraction data with interaction energy calculations in the solution of the structures of several aromatic polyamides, including poly(*p*-phenyleneterephthalamide). Inherent in this method is the need for accurate geometrical parameters for the various repeating units comprising the polymer chain. We have conducted X-ray diffraction studies on several low moleculer weight aromatic amides (one of which is presented here) in order to obtain accurate molecular parameters and to study certain aspects of the molecular structure which relate to the rheological and mechanical properties of these systems.

A stereoscopic view of the molecule is shown in Fig. 1. The bond distances and angles are presented in Fig. 2. The average estimated standard deviations for the nonhydrogen bond lengths are 0·001 Å, and 0·01 Å for those bonds involving hydrogen. The corresponding values for the bond angles are 0·1 and 0·2° respectively.

The amide group [C(1),C(7),O,N,C(8)] is nearly flat with atoms N and C(8) having the largest deviations from the least-squares plane of +0·02 and −0·02 Å respectively. The torsion angle about the C(7)—N bond [O—C(7)—N—C(8)] is +1·4°. The benzene planes of the benzoyl and *p*-phenylenediamine segments tilt at angles of −22·7 and −56·3°, expressed as the torsion angles C(6)—C(1)—C(7)—O and C(7)—N—C(8)—C(10) respectively. Coplanarity of the amide plane and benzene rings is precluded in part by intramolecular repulsions between the *ortho* H atoms on the benzoyl moiety, H(6) and H(2), with the oxygen and amide H(11); the intramolecular contacts are 2·25 Å for H(2)···H(11) and 2·48 Å for O···H(6).

Centrosymmetric molecules are linked together by N—H(11)···O hydrogen bonds directed approximately parallel to the *a* axis; the N···O distance is 2·857 (1) Å and the N—H(11)···O angle is 154·7 (1)°. When viewed along the *a* axis, molecules can be seen from Fig. 3 to lie parallel to (100) and to overlap in a staggered arrangement, in which each molecule is hydrogen-bonded to two different neighboring molecules. This type of hydrogen bonding differs from the layering of hydrogen-bonded chains of molecules reported for poly(*p*-phenyleneterephthalamide) (Northolt, 1974).

References

Black, W. B. (1973). *J. Macromol. Sci. Chem.* **7**, 3–41.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

Frank, F. C. (1970). *Proc. R. Soc. London Ser. A*, **319**, 127–136.

Lenhert, P. G. (1975). *J. Appl. Cryst.* **8**, 568–570.

Miller, P. T., Lenhert, P. G. & Joesten, M. D. (1972). *Inorg. Chem.* **11**, 2221–2227.

Northolt, M. G. (1974). *Eur. Polym. J.* **10**, 799–804.

Stewart, J. M., Kruger, G. J., Ammon, H. L., Dickinson, C. & Hall, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Tashiro, K., Kobayashi, M. & Tadokoro, H. (1977). *Macromolecules*, **10**, 413–420.