Table 5. $C-CO_2R$ *bond length for* (5), (6) *and* (7)

Ester group	Angle between plane of ester group and the plane of the enaminic system (°)	$C-CO_2R$ bond length (Å)
(5) 8-ester	45	1·470 (7)
(6) 6-ester	10	1·439 (7)
(6) 7-ester	70	1·504 (6)
(6) 8-ester	43	1·464 (7)
(7) 7-ester	0	1·443 (8)

(5) is compared in Table 5 with the equivalent parameters (from crystal structure data) for the 6- and 7-esters of (6) (Abbott, Acheson, Eisner, Watkin & Carruthers, 1976) and the 7-ester of (7) (Abbott, Acheson, Forder, Watkin & Carruthers, 1977). The 6-ester of (6) and the 7-ester of (7) are almost coplanar with an enaminic system and have shorter $C-CO_2R$ bonds while the 7-ester of (6), which is at 70° to the conjugated system, has a longer bond length (1·504 Å). There is a close similarity between the $C-CO_2R$ bond length for the 8-ester of (6) and the 8-ester of (5) which both make similar angles (43 and 45°) with their respective enaminic systems.

It may be concluded that mass spectrometry is an unreliable method for differentiating between the structural possibilities (1) and (2), and an alternative method using ^1H and ^{13}C NMR spectra which is based on the established structure (5) will be published elsewhere (Acheson & Wallis, 1980).

References

ABBOTT, P. J., ACHESON, R. M., EISNER, U., WATKIN, D. J. & CARRUTHERS, J. R. (1976). *J. Chem. Soc. Perkin Trans.* 1, pp. 1269–1278.

ABBOTT, P. J., ACHESON, R. M., FORDER, R. A., WATKIN, D. J. & CARRUTHERS, J. R. (1977). *Acta Cryst.* B**33**, 898–900.

ACHESON, R. M. & WALLIS, J. D. (1980). *J. Chem. Soc. Perkin Trans.* 1. In the press.

CARRUTHERS, J. R. (1975). *CRYSTALS User Manual.* Oxford Univ. Computing Laboratory.

CARRUTHERS, J. R. & WATKIN, D. J. (1979). *Acta Cryst.* A**35**, 698–699.

KATNER, A. S. & ZEIGE, E. A. (1971). *J. Chem. Soc. D*, pp. 864–865.

LOWN, J. W. & MA, J. C. N. (1967). *Can. J. Chem.* **45**, 953–967.

MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1977). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.

RUHEMANN, S. & STAPLETON, H. E. (1900). *J. Chem. Soc.* **77**, 804–810.

SASAKI, H., SAKATA, H. & IWANAMI, Y. (1964). *Nippon Kagaku Zasshi*, **85**, 704–707.

Acta Cryst. (1980). B**36**, 3182–3184

N,N'-Tetramethylenedibenzamide (TMDB)*

BY S. HARKEMA, G. J. VAN HUMMEL AND R. J. GAYMANS

Chemical Physics Laboratory, Twente University of Technology, PO Box 217, 7500 *AE Enschede, The Netherlands*

(*Received* 7 *July* 1980; *accepted* 1 *September* 1980)

Abstract. $C_{18}H_{20}N_2O_2$, monoclinic, $P2_1/c$, a = 5·118 (3), b = 5·324 (3), c = 28·410 (5) Å, β = 97·05 (5)°, Z = 2. The crystal structure was solved by direct methods. All H atoms have been located. R_w = 4·7%. The planes of the phenyl ring and the amide group are rotated with respect to each other due to steric hindrance. Hydrogen bonds connect molecules related by translation in the a direction.

Introduction. The present investigation reports the crystal structure of TMDB, a model compound of an aromatic–aliphatic polyamide. TMDB was prepared as described by Gaymans & Harkema (1977). Intensities were measured on a Philips PW 1100 diffractometer (Mo $K\alpha$ radiation, λ = 0·71069 Å, graphite monochromator). Reflections up to θ = 30° were measured with the $\omega/2\theta$ scan mode.

The number of reflections measured was 1612. All reflections were used in the refinement. No absorption correction was applied. Details of the solution of the structure, the weighting scheme, the scattering factors

* The Structure of Model Compounds of Aromatic and Aromatic–Aliphatic Polyamides. III. Part II: Harkema, Gaymans, van Hummel & Zylberlicht (1979).

0567-7408/80/123182-03$01.00

and the refinement are given in the first paper of this series (Harkema & Gaymans, 1977). The final value of R_w was 4·7%.* The asymmetric part of the unit cell contains one half of the molecule. The other half is generated by a center of symmetry.

Discussion. Final atomic positional parameters are given in Table 1. The numbering of the atoms is according to Fig. 1. Bond lengths and angles are collected in Table 2. A stereoscopic view of two molecules is given in Fig. 2. From this figure it can be seen that the tetramethylene part of the molecule adopts an extended zigzag chain conformation. Best planes have been fitted to the phenyl ring and the amide group (Table 3). The plane of the amide group is rotated with respect to the plane of the phenyl ring, which is planar within experimental accuracy. This rotation is caused by steric hindrance between H(3) and H(6). The angle between the two planes is 30·0°. Corresponding values for related compounds are: 24·6° in benzamide (Blake & Small, 1972); 29·1° in *N,N'*-(*p*-phenylene)dibenzamide (Harkema & Gaymans, 1977) and 30·6° in *N,N'*-diphenyl-

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35575 (12 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* (×10⁴) *and equivalent isotropic thermal parameters* (Å² ×10³)

	x	*y*	*z*	U_{eq}*
C(1)	1620 (4)	5263 (5)	2908 (1)	60 (1)
C(2)	3277 (4)	3259 (5)	2992 (1)	61 (1)
C(3)	3093 (4)	1670 (4)	3374 (1)	50 (1)
C(4)	1183 (3)	2091 (3)	3669 (1)	38 (1)
C(5)	−491 (4)	4120 (4)	3579 (1)	49 (1)
C(6)	−255 (5)	5702 (4)	3206 (1)	58 (1)
C(7)	769 (3)	374 (4)	4070 (1)	41 (1)
C(8)	2617 (4)	7380 (4)	4669 (1)	45 (1)
C(9)	5104 (4)	5890 (4)	4796 (1)	44 (1)
N	2858 (3)	9153 (3)	4286 (1)	45 (1)
O	−1453 (2)	95 (3)	4193 (1)	58 (1)
H(1)	1733 (35)	6333 (38)	2640 (7)	60 (7)
H(2)	4541 (41)	2900 (40)	2791 (7)	75 (7)
H(3)	4236 (34)	242 (34)	3429 (7)	51 (6)
H(4)	−1827 (35)	4410 (33)	3792 (7)	52 (6)
H(5)	−1441 (38)	7124 (39)	3134 (7)	68 (7)
H(6)	4403 (35)	−487 (33)	4208 (6)	46 (6)
H(7)	2164 (35)	−1705 (37)	4950 (7)	58 (7)
H(8)	1092 (36)	−3752 (35)	4562 (6)	58 (6)
H(9)	6538 (32)	−2996 (32)	4871 (6)	42 (5)
H(10)	5548 (35)	−5033 (37)	4511 (7)	60 (7)

* Defined according to Willis & Pryor (1975).

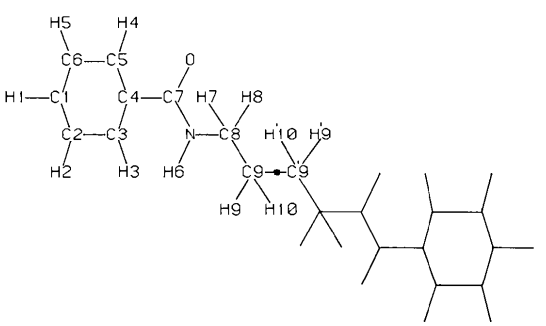


Fig. 1. Atomic arrangement of TMDB.

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

C(1)–C(2)	1·366 (4)	C(6)–H(5)	0·976 (20)
C(1)–C(6)	1·375 (3)	C(7)–N	1·334 (2)
C(1)–H(1)	0·957 (20)	C(7)–O	1·238 (2)
C(2)–C(3)	1·387 (4)	C(8)–C(9)	1·505 (3)
C(2)–H(2)	0·934 (21)	C(8)–N	1·459 (3)
C(3)–C(4)	1·383 (3)	C(8)–H(7)	0·986 (20)
C(3)–H(3)	0·960 (18)	C(8)–H(8)	1·004 (19)
C(4)–C(5)	1·383 (3)	C(9)–H(9)	0·947 (17)
C(4)–C(7)	1·497 (3)	C(9)–H(10)	0·996 (20)
C(5)–C(6)	1·372 (3)	C(9)–C'(9)	1·513 (3)
C(5)–H(4)	0·979 (18)	N–H(6)	0·868 (18)
C(2)–C(1)–C(6)	119·3 (2)	C(4)–C(7)–N	117·7 (2)
C(2)–C(1)–H(1)	120·5 (18)	C(4)–C(7)–O	120·5 (2)
C(6)–C(1)–H(1)	120·1 (17)	N–C(7)–O	121·6 (2)
C(1)–C(2)–C(3)	120·9 (2)	C(9)–C(8)–N	111·8 (2)
C(1)–C(2)–H(2)	120·9 (20)	C(9)–C(8)–H(7)	110·0 (13)
C(3)–C(2)–H(2)	118·1 (19)	C(9)–C(8)–H(8)	111·0 (13)
C(2)–C(3)–C(4)	119·7 (2)	N–C(8)–H(7)	109·5 (14)
C(2)–C(3)–H(3)	120·7(17)	N–C(8)–H(8)	107·2 (12)
C(4)–C(3)–H(3)	119·4 (16)	H(7)–C(8)–H(8)	106·9 (18)
C(3)–C(4)–C(5)	118·8 (2)	H(9)–C(9)–H(10)	104·2 (16)
C(3)–C(4)–C(7)	122·6 (2)	C(8)–C(9)–C'(9)	112·0 (2)
C(5)–C(4)–C(7)	118·4 (2)	C(8)–C(9)–H(9)	109·4 (12)
C(4)–C(5)–C(6)	120·7 (2)	C(8)–C(9)–H(10)	109·7 (13)
C(4)–C(5)–H(4)	118·2 (15)	C'(9)–C(9)–H(9)	110·0 (12)
C(6)–C(5)–H(4)	121·0 (16)	C'(9)–C(9)–H(10)	111·0 (14)
C(1)–C(6)–C(5)	120·3 (2)	C(7)–N–C(8)	121·4 (2)
C(1)–C(6)–H(5)	117·8 (17)	C(7)–N–H(6)	118·6 (17)
C(5)–C(6)–H(5)	121·7 (18)	C(8)–N–H(6)	119·7 (17)

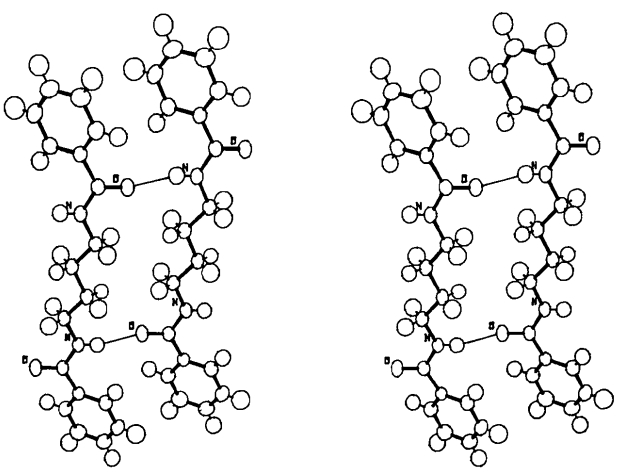


Fig. 2. Stereoscopic view (Johnson, 1965) of the crystal structure of TMDB.

Table 3. *Distances* (Å) *of atoms from different planes in the molecule*

Plane 1: plane fitted to the C atoms of the phenyl ring

$$0{\cdot}46914x + 0{\cdot}48589y + 2{\cdot}29650z = 1{\cdot}00000$$

Plane 2: plane through the C(7), N and O atoms of the amide group

$$0{\cdot}06545x + 0{\cdot}53452y + 2{\cdot}39539z = 1{\cdot}00000$$

	Plane 1	Plane 2		Plane 1	Plane 2
C(1)	0·003 (3)	–	N	–	0·000 (0)
C(2)	0·004 (3)	–	O	–	0·000 (0)
C(3)	−0·007 (2)	–	H(1)	0·03 (2)	–
C(4)	0·002 (2)	−0·012 (2)	H(2)	0·03 (2)	–
C(5)	0·005 (2)	–	H(3)	0·01 (2)	–
C(6)	−0·007 (3)	–	H(4)	0·00 (2)	–
C(7)	0·069 (2)	0·000 (0)	H(5)	0·01 (2)	–
C(8)	–	−0·033 (2)	H(6)	–	0·08 (2)

terephthalamide (Harkema, Gaymans, van Hummel & Zylberlicht, 1979). From Table 3 it can be concluded that C(7) and C(8) deviate significantly from the planes of the phenyl ring and the amide group respectively.

The molecules of TMDB are connected by N–H···O hydrogen bonds of length 2·999 (4) Å. Hydrogen bonds are formed between molecules related by translation along *a*. Each molecule is hydrogen bonded to two neighbors, giving rise to a one-dimensional hydrogen-bonding scheme.

References

BLAKE, C. C. F. & SMALL, R. W. H. (1972). *Acta Cryst.* B**28**, 2201–2206.

GAYMANS, R. J. & HARKEMA, S. (1977). *J. Polym. Sci. Polym. Phys. Ed.* **15**, 587–590.

HARKEMA, S. & GAYMANS, R. J. (1977). *Acta Cryst.* B**33**, 3609–3611.

HARKEMA, S., GAYMANS, R. J., VAN HUMMEL, G. J. & ZYLBERLICHT, D. (1979). *Acta Cryst.* B**35**, 506–508.

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

WILLIS, B. T. M. & PRYOR, A. W. (1975). *Thermal Vibrations in Crystallography*, pp. 101–102. Cambridge Univ. Press.

Acta Cryst. (1980). B**36**, 3184–3186

The Correct Structural Formula for Anthralin*

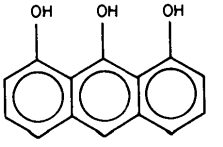
BY F. R. AHMED

Division of Biological Sciences, National Research Council of Canada, Ottawa, Canada K1A 0R6

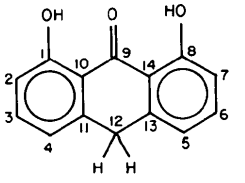
(*Received* 11 *July* 1980; *accepted* 11 *August* 1980)

Abstract. $C_{14}H_{10}O_3$, $M_r = 226{\cdot}23$, monoclinic, $A2/a$, $a = 20{\cdot}857\ (3)$, $b = 9{\cdot}264\ (3)$, $c = 11{\cdot}225\ (3)$ Å, $\beta = 104{\cdot}79\ (2)°$, $Z = 8$, $d_o = 1{\cdot}433$, $d_c = 1{\cdot}433$ Mg m^{-3}, $R = 0{\cdot}055$ and $R_w = 0{\cdot}056$ for 1118 observed reflexions. Anthralin (or dithranol) is confirmed as 1,8-dihydroxy-9-anthrone instead of 1,8,9-anthracenetriol which is the formula presently assigned in pharmaceutical literature. The molecule is almost planar with approximate mirror symmetry, and includes intramolecular O–H···O···H–O bonds associated with an elongated C=O length of 1·261 (4) Å at the central O atom.

Introduction. Anthralin is a parasiticide used as ointment or paint in the treatment of psoriasis, ringworm infections and other chronic dermatosis. It is listed as a reference standard in the current *United States Pharmacopeia* (1975), and under dithranol in the *British Pharmacopeia* (1973) with the structural formula 1,8,9-anthracenetriol or 1,8,9-trihydroxyanthracene, (I). Spectroscopic examination of anthralin by Avdovich & Neville (1980) led to the keto structure 1,8-dihydroxy-9-anthrone, (II), and the present X-ray analysis has confirmed this assignment.



(I)



(II)

X-ray measurements were carried out on a Nonius CAD-4 diffractometer with a thin-plate crystal 0·33 × 0·23 × 0·07 mm, and Ni-filtered Cu $K\alpha$ radiation at a take-off angle of 3·0°. The cell dimensions were derived by least squares from the angular settings of 15 centered reflexions ($\theta = 37$–$48°$). The intensities were recorded by ω–2θ scans for all reflexions within one hemisphere up to $\theta = 65°$ for scan ranges of $\Delta\theta = (0{\cdot}8$

* Issued as NRCC No. 18730.

0567-7408/80/123184-03$01.00