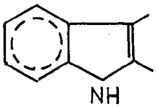
The average P—C (benzene) bond, 1·813 Å, is longer than P—C (indolyl) 1·737 Å. The bond distances and angles show high delocalization of π electrons over the whole indolyl moiety. All the indolyl distances are typical of partially conjugated bonds. The bond lengths C2—C3 and C32—C33 (average 1·412 Å) are longer than the aromatic C—C bond. In any case the description of the indolyl moiety by the resonance structure with the double bond between C2—C3 would be misleading. The six-membered ($\chi^2 = 21$, 13) and five-membered rings ($\chi^2 = 1$, 10) of the indolyl moieties form dihedral angles of 0·5 (1) and 0·9 (1)° for molecules 1 and 2 respectively. The deviations from the mean plane of the five-membered rings are: for phosphorus 0·015 (1), −0·020 (2) Å, and for sulfur 0·122 (1), −0·109 (1) Å. The torsion angles are S10—C2—C3—P11 4·1 (5), S40—C32—C33—P41 4·6 (5) and S10—C2—C3—C4 179·1 (3), S40—C32—C33—C34 175·3 (3)°.



Comparison of intramolecular dihedral angles (Table 2) shows a similar conformation for both molecules. Projection of the crystal structure along the *b* axis (Fig. 3) shows that the dominant feature of the crystal structure is dimerization of two symmetry independent molecules, formed by two nonequivalent hydrogen bonds N1^i—H1^i···S40 and N31—H31···S10^i, where (i) = $x + \frac{1}{2}$, $-y + \frac{1}{2}$, $z + \frac{1}{2}$. The corresponding characteristics are N1^i···S40 3·366 (3), N1^i—H1^i 0·75 (4) Å, N1^i—H1^i···S40 164 (5)°, N31···S10^i 3·308 (2), N31—H31 0·89 (5) Å, N31—H31···S10^i 169 (4)°.

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Structure of Hexamethylenediammonium Terephthalate Dihydrate

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Abstract. $C_6H_{18}N_2^{2+}.C_8H_4O_4^{2-}.2H_2O$, M_r = 318·38, monoclinic, $P2_1/n$, a = 20·541 (2), b = 7·2953 (5), c = 11·376 (1) Å, β = 100·588 (6)°, V = 1675·8 (2) Å^3, Z = 4, D_m = 1·267 (1), D_x = 1·262 Mg m^{-3}, Cu $K\alpha$, λ = 1·5418 Å, μ = 0·83 mm^{-1}, $F(000)$ = 688, T = 295 K, R = 0·056 for 2672 unique reflections. Hexamethylenediammonium cations and terephthalate anions are held together by two kinds of N—H···O hydrogen bonds to form a ribbon of these ions along **a**. The ribbons are stacked along **b** to form a sheet parallel to (001) and the sheets are joined by N—H···O and O—H···O hydrogen bonds to form a three-dimensional network.

Introduction. Hexamethylenediammonium terephthalate (6T) is one of the intermediates in the manufacture of Nylon 6T (Morgan & Kwolek, 1975; Pineault & Brisse, 1983*a*). The structure of 6T has been studied in order to elucidate a characteristic feature of the molecular arrangement in the crystals of the Nylon salt composed of the aliphatic cation and aromatic anion.

Experimental. Crystals obtained from an aqueous solution by slow evaporation at room temperature: colorless prisms elongated along **b**. D_m by flotation in benzene–CCl_4. Systematic absences: $h0l$ for $h + l$ odd, $0k0$ for k odd. A crystal with dimensions 0·20 × 0·28 × 0·28 mm mounted on a Rigaku AFC-5 four-circle diffractometer. Lattice parameters determined with 15 reflections in the range 39 < 2θ < 41° by the

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least-squares method. Intensities measured up to $(\sin\theta)/\lambda = 0{\cdot}5753\ \text{Å}^{-1}$ by the ω–2θ scan method [scan speed 6° min^{-1} in ω, scan range in ω: $(1{\cdot}2 + 0{\cdot}15\tan\theta)°$, Ni-filtered Cu $K\alpha$ at 40 kV, 200 mA, rotating anode]. Background measured for 3 s on either side of the peak; three standard reflections recorded every 97 reflections; the fluctuation within 1·5% in F. Lorentz and polarization corrections; no absorption correction. 2823 reflections measured. $R_{\text{int}} = 0{\cdot}009$ for 150 $hk0$ reflections. All 2673 unique reflections (ranging over $h = -23$ to 23, $k = 0$ to 8, $l = 0$ to 13) used in the structure analysis; the $|F_o|$ values of 2515 reflections were larger than $\sigma(F_o)$. The structure solved by *RANTAN* (Yao, 1981) incorporated in *MULTAN*84 and refined (non-H atoms anisotropically) by full-matrix least-squares method, $\sum w(|F_o| - |F_c|)^2$ minimized with $w = 1{\cdot}0/[\sigma(F_o)^2 - 0{\cdot}4790|F_o| + 0{\cdot}0235|F_o|^2]$ for $|F_o| > 0$, $w = 2{\cdot}9782$ for $|F_o| = 0$. The positions of H atoms determined from difference Fourier maps and refined isotropically. Correction for secondary extinction with $I_{\text{corr}} = I_o(1 + 7{\cdot}11 \times 10^{-5} \times I_c)$ for 37 strongest reflections. In the final refinement 021 reflection omitted because of poor agreement of $|F_o| = 160{\cdot}6$ and $|F_c| = 298{\cdot}7$. The final $R = 0{\cdot}056$ for 2672 unique reflections; $wR = 0{\cdot}053$, $S = 2{\cdot}09$. $(\Delta/\sigma)_{\max} = 0{\cdot}65$. $\Delta\rho$ in final difference Fourier map $-0{\cdot}50$ to $+0{\cdot}47\ \text{e Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs *MULTAN*84 (Main, Germain & Woolfson, 1984), *FMLS* and *DAPH* (Ashida, 1973), *MOLCON* (Fujii, 1979) and *ORTEP* (Johnson, 1971). Computations carried out at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University, and at the Okayama University Computer Center.

Discussion. Final atomic parameters are listed in Table 1.* The thermal ellipsoids are shown in Fig. 1 with the atomic numbering. Bond lengths and angles and geometry of the hydrogen bonds are listed in Table 2. The crystal structure viewed down the b axis is shown in Fig. 2.

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{\text{eq}} = 4/3\textstyle\sum_i \beta_{ii}/a_i^{*2}.$$

	x	y	z	$B_{\text{eq}}(\text{Å}^2)$
C(1)	0·66644 (6)	0·1065 (2)	0·2545 (1)	3·35 (5)
C(2)	0·59247 (5)	0·1152 (2)	0·2088 (1)	3·22 (5)
C(3)	0·55210 (5)	0·1033 (2)	0·3080 (1)	3·13 (5)
C(4)	0·47782 (5)	0·1147 (2)	0·2590 (1)	3·21 (5)
C(5)	0·43627 (5)	0·1014 (2)	0·3564 (1)	3·07 (5)
C(6)	0·36291 (5)	0·1230 (2)	0·3069 (1)	3·19 (5)
N(7)	0·70341 (4)	0·1056 (1)	0·15370 (9)	3·54 (5)
N(8)	0·32231 (4)	0·1088 (1)	0·40236 (9)	3·11 (4)
C(9)	0·94680 (5)	0·1087 (1)	0·2296 (1)	2·56 (5)
C(10)	0·99099 (5)	0·0926 (2)	0·15176 (9)	2·72 (5)
C(11)	1·05870 (5)	0·0992 (2)	0·1929 (1)	2·83 (5)
C(12)	1·08353 (5)	0·1224 (1)	0·31430 (9)	2·58 (5)
C(13)	1·03929 (5)	0·1379 (2)	0·3924 (1)	3·07 (5)
C(14)	0·97150 (5)	0·1316 (2)	0·3510 (1)	3·11 (5)
C(15)	0·87308 (5)	0·1013 (2)	0·1828 (1)	2·91 (5)
C(16)	1·15726 (5)	0·1295 (2)	0·3623 (1)	2·91 (5)
O(17)	0·83566 (4)	0·0977 (2)	0·25689 (8)	5·12 (5)
O(18)	0·85406 (4)	0·0977 (1)	0·07109 (7)	3·66 (4)
O(19)	1·19517 (4)	0·1297 (2)	0·28729 (8)	4·89 (4)
O(20)	1·17675 (4)	0·1324 (1)	0·47214 (8)	4·15 (4)
O(21)	1·19604 (5)	0·2187 (1)	0·05646 (8)	4·39 (4)
O(22)	0·82239 (5)	0·2846 (2)	0·4822 (1)	5·83 (5)

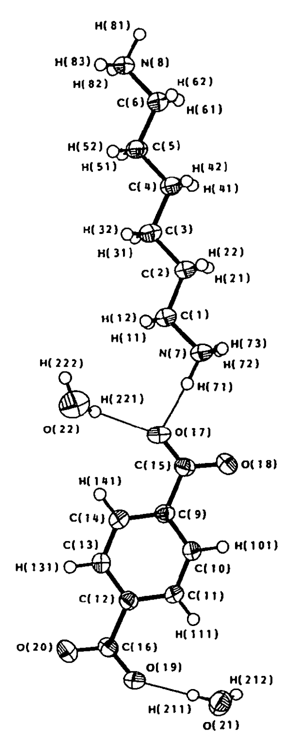

Fig. 1. The thermal ellipsoids (50% probability) with atomic numbering. The H atoms are represented as spheres equivalent to $B = 1{\cdot}0\ \text{Å}^2$. Hydrogen bonds are shown by thin lines.

The non-H atoms and H(71) and H(81) in the cation take a *trans* zigzag conformation and show maximum deviation of only 0·041 (1) Å from planarity. The bond lengths and angles compare well with those found in the crystal structures of hexamethylenediammonium adipate (Brown, 1966) and hexamethylenediammonium phthalate trihydrate (Jagannathan, Subramanian, Srinivasan & Trotter, 1984); C(2)—C(3), C(3)—C(4) and C(4)—C(5) bonds are longer than C(1)—C(2) and C(5)—C(6) bonds because of the electron-withdrawing effect of the N atoms.

The benzene ring of the anion is planar within 0·003 (1) Å. In general, the larger the dihedral angle

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52555 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *and hydrogen-bond geometry*

C(1)—C(2)	1·514 (2)	C(12)—C(13)	1·387 (2)
C(2)—C(3)	1·521 (2)	C(13)—C(14)	1·386 (2)
C(3)—C(4)	1·527 (2)	C(14)—C(9)	1·391 (2)
C(4)—C(5)	1·521 (2)	C(9)—C(15)	1·511 (2)
C(5)—C(6)	1·517 (2)	C(12)—C(16)	1·513 (2)
C(1)—N(7)	1·487 (2)	C(15)—O(17)	1·241 (2)
C(6)—N(8)	1·489 (2)	C(15)—O(18)	1·259 (1)
C(9)—C(10)	1·385 (2)	C(16)—O(19)	1·256 (2)
C(10)—C(11)	1·384 (2)	C(16)—O(20)	1·240 (1)
C(11)—C(12)	1·392 (2)		

N(7)—C(1)—C(2)	111·0 (1)	C(13)—C(14)—C(9)	120·1 (1)
C(1)—C(2)—C(3)	113·1 (1)	C(14)—C(9)—C(15)	120·9 (1)
C(2)—C(3)—C(4)	111·7 (1)	C(10)—C(9)—C(15)	120·3 (1)
C(3)—C(4)—C(5)	112·8 (1)	C(11)—C(12)—C(16)	121·5 (1)
C(4)—C(5)—C(6)	112·0 (1)	C(13)—C(12)—C(16)	119·7 (1)
C(5)—C(6)—N(8)	111·9 (1)	C(9)—C(15)—O(17)	117·9 (1)
C(14)—C(9)—C(10)	118·9 (1)	C(9)—C(15)—O(18)	117·5 (1)
C(9)—C(10)—C(11)	121·1 (1)	C(12)—C(16)—O(19)	117·3 (1)
C(10)—C(11)—C(12)	120·1 (1)	C(12)—C(16)—O(20)	118·7 (1)
C(11)—C(12)—C(13)	118·8 (1)	O(17)—C(15)—O(18)	124·7 (1)
C(12)—C(13)—C(14)	121·0 (1)	O(19)—C(16)—O(20)	124·0 (1)

	Donor (*D*)	Acceptor (*A*)	*D*···*A* (Å)	H···*A* (Å)	*D*—H···*A* (°)
(1)	N(7^{i})	O(17^{i})	2·755 (2)	1·75 (2)	174 (1)
(2)	N(7^{i})	O(22^{ii})	2·802 (2)	1·80 (2)	176 (1)
(3)	N(7^{i})	O(20^{iii})	2·793 (1)	1·83 (2)	166 (1)
(4)	N(8^{i})	O(19^{iv})	2·702 (1)	1·69 (1)	173 (1)
(5)	N(8^{i})	O(21^{ii})	2·920 (1)	2·05 (1)	155 (1)
(6)	N(8^{i})	O(18^{v})	2·870 (1)	1·90 (2)	175 (1)
(7)	O(21^{i})	O(19^{i})	2·708 (1)	1·84 (2)	175 (1)
(8)	O(21^{i})	O(18^{vi})	2·818 (1)	1·91 (2)	177 (1)
(9)	O(22^{i})	O(17^{i})	2·960 (2)	2·13 (2)	153 (2)
(10)	O(22^{i})	O(21^{v})	2·873 (1)	2·06 (2)	164 (2)

Symmetry codes: (i) x, y, z; (ii) $3/2 - x$, $-1/2 + y$, $1/2 - z$; (iii) $-1/2 + x$, $1/2 - y$, $-1/2 + z$; (iv) $-1 + x$, y, z; (v) $-1/2 + x$, $1/2 - y$, $1/2 + z$; (vi) $2 - x$, $-y$, $-z$.

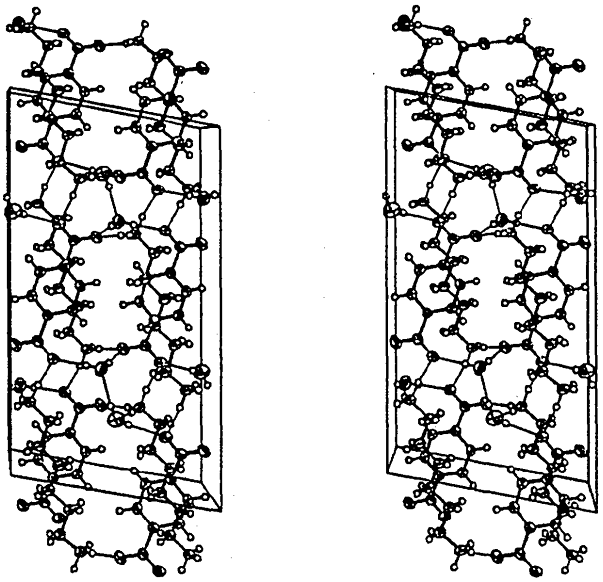

Fig. 2. Stereoscopic view of the molecular packing in the unit cell. The *a* axis points upward, the *c* axis from left to right, and the *b* axis onto the plane of the paper. Hydrogen bonds are shown by thin lines.

between the carboxylate group and the benzene ring, the longer the bond between the aromatic C and the carboxyl C (Takusagawa, Hirotsu & Shimada, 1971; Kashino, Sasaki & Haisa, 1973; Moritani, Sasahara, Kashino & Haisa, 1987). However, in the present compound the bonds are rather long [1·511 (2) and 1·5134 (2) Å] in spite of the small angles of 6·80 (4) and 6·29 (4)°.

The cations and anions lie nearly on a plane at $y = 1/8$ and are held together by the strongest N—H···O hydrogen bonds (1) and (4) in Table 2 to form a ribbon of ions along **a**. The long molecular axes of the aliphatic cation and aromatic anion make an angle of 146·8 (1)° which is close to the corresponding angles of *N*,*N'*-tetramethylenedibenzamide (Harkema, van Hummel & Gaymans, 1980), *N*,*N'*-hexamethylenedibenzamide (Pineault & Brisse, 1983*a*) and *N*,*N'*-octamethylenedibenzamide (Pineault & Brisse, 1983*b*) [144·8 (3), 146·3 (3)°, respectively], though in these compounds the aliphatic and aromatic portions are linked by a covalent bond.

The ribbons are stacked along **b** by van der Waals and ionic interactions and hydrogen bonds (2), (5), (7), (8) and (9) to form a sheet parallel to (001). These sheets are stacked along **c** by hydrogen bonds (3), (6) and (10). The water molecules' rôle is to tighten the structure by hydrogen bonding between the ribbons and sheets and to fill up the vacant spaces among the sheets.

The present structure is close to a *C*2/*c* structure. That is, the centers of gravity of the cationic and anionic moieties lie approximately at (1/2, 1/8, 1/4) and (1, 1/8, 1/4), respectively, and O(21) and O(22) of water molecules are related by a pseudo twofold axis. If the moieties at these positions have twofold symmetry and the water molecules are related by a twofold axis by disordering, the space group would be *C*2/*c*. However, in the actual structure the twofold symmetry of the moieities is lost by packing requirements in the sheet, and the water molecules are not disordered. Although the molecules having 2/*m* or *mmm* symmetry frequently have symmetry $\bar{1}$ in the crystals (Müller, 1978), the moieties in the present crystals have no $\bar{1}$ symmetry, because no reasonable packing with $P2_1/c$ or its minimal supergroup can be obtained by adding $\bar{1}$ to the moieties.

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Acta Cryst. (1990). C46, 849–852

Structure of 6-Methoxy-1-[(4-methoxyphenyl)amino]xanthen-9-one

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Abstract. $C_{21}H_{17}NO_4$, $M_r = 347.37$, orthorhombic, $P2_12_12_1$, $a = 7.060$ (2), $b = 14.024$ (4), $c = 34.005$ (10) Å, $V = 3366.8$ Å^3, $Z = 8$ (two molecules per asymmetric unit), $D_x = 1.37$, $D_m = 1.332$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 0.69$ mm^{-1}, $F(000) = 1456$, $T = 293$ K, $R = 0.036$, $wR = 0.034$ for 1925 reflections with $I > 3\sigma(I)$. The pyran ring in both molecules is in a boat conformation. There is an internal hydrogen bond N—H···O linking the nitrogen of the 4-methoxy-*N*-substituted aniline and the carbonyl oxygen of the pyranoid ring in each molecule.

Introduction. Xanthone derivatives have been well documented as biogenic precursors (Vinod & Gupta, 1979) and neuroleptic (Lassen, Bogeso, Hansen, Buss & Bigler, 1980) and antiallergic (Pfister, 1980) agents. The title compound is a condensation product obtained by treating 2-bromo-6-methoxycoumaran-3-one with a 4-substituted aromatic amine (Chandrasekhar & Ramadas, unpublished) in glacial acetic acid medium. The crystal structure analysis of this condensation product was undertaken in order to confirm the assigned structure.

Experimental. Pale pink prismatic single crystals were grown by slow evaporation of the compound in hexane. The density was measured by flotation. Intensity data from a crystal of approximate dimensions 0.40 × 0.45 × 0.66 mm were collected up to $(\sin\theta/\lambda) = 0.63$ Å^{-1} with ω–2θ scan using Ni-filtered Cu $K\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer. Cell parameters were refined by least-squares calculations from 20 reflections with $35 < \theta < 45°$. Two strong reflections ($2\overline{2}\overline{9}$, $2\overline{4}\overline{6}$) monitored periodically during data collection showed that the crystal was stable. Index range of unique data $0 \leq h \leq 7$, $0 \leq k \leq 15$, $0 \leq l \leq 37$. 2995 reflections were collected of which 1925 unique reflections with $I > 3\sigma(I)$ were considered observed. $R_{\text{int}} = 0.000$, $R_{\text{sigma}} = 0.0214$. Lp correction applied, absorption and extinction neglected.

Structure solution by multi-solution technique using *SHELXS*86 (Sheldrick, 1986). Refinement on F by least-squares method with *SHELX*76 (Sheldrick, 1976). H-atom positions from difference Fourier maps at intermediate stages. Final refinement with non-H atoms anisotropic and H atoms isotropic converged to $R = 0.036$ and $wR = 0.034$ with $w = K\{[\sigma(F_o)]^2 + g(|F_o|)^2\}^{-1}$, where $K = 4.5716$ and $g = 0.000064$. Quantity minimized was $\sum w(|F_o| - K|F_c|)^2$. (Shift/e.s.d.)$_{\max} = 0.019$. Maximum and minimum heights in final difference synthesis were 0.13 and -0.21 e Å^{-3}. Atomic scattering factors were from *SHELX*76 and all calculations were performed using a Siemens 7580-E computer.

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