tropic thermal parameters; $R = 0.0778$, $wR = 0.0745$ for 280 parameters and 2280 reflections, $S = 1.443$, $(\Delta/\sigma)_{max} = 0.022$, largest peaks in the final difference map of 0.38 and -0.38 e Å^{-3}; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0.00071F_o^2]^{-1}$. All computer programs supplied by Nicolet (Nicolet Instrument Corporation, 1986) for Desktop 30 Microeclipse and Nova 4/C configuration; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). Fig. 1 is a drawing of the compound, Table 1 lists the atomic positional parameters, and Table 2 gives interatomic distances and angles.*

Related literature. Hymenoxin and related compounds have been reported previously (Thomas & Mabry, 1967; Gutierrez & Herz, 1988). 3,5-

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53380 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

dihydroxy-6,7,8-trimethoxyflavone (Hansel, Khaliefi & Peller, 1981) and 5-hydroxy-6,7,2',4',5'-pentamethoxyflavone (Al-Yaha, Hifnawy, Mossa, El-Feraly, McPhail & McPhail, 1989) exhibit highly oxygenated ring systems, and their X-ray structures can be used for comparison.

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Structure of Ethylenediammonium Terephthalate and Tetramethylenediammonium Terephthalate

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Abstract. Cu $K\alpha$, $\lambda = 1.54178$ Å, $T = 295$ K. Ethylenediammonium terephthalate (2T), $C_2H_{10}N_2^{2+}.C_8H_4O_4^{2-}$, $M_r = 226.22$, monoclinic, $P2_1/n$, $a = 8.381$ (8), $b = 9.236$ (4), $c = 7.471$ (4) Å, $\beta = 115.32$ (5)°, $V = 522.8$ (7) Å^3, $Z = 2$, $D_m = 1.438$ (2), $D_x = 1.438$ Mg m^{-3}, $\mu = 0.96$ mm^{-1}, $F(000) = 240$, $R = 0.042$ for 839 unique reflections. Tetramethylenediammonium terephthalate (4T), $C_4H_{14}N_2^{2+}.C_8H_4O_4^{2-}$, $M_r = 254.27$, triclinic, $P\bar{1}$, $a = 8.3490$ (8), $b = 11.760$ (2), $c = 8.2238$ (8) Å, $\alpha = 99.37$ (1), $\beta = 91.48$ (1), $\gamma = 125.027$ (7)°, $V = 645.3$ (1) Å^3, $Z = 2$, $D_m = 1.315$ (2), $D_x = 1.309$ Mg m^{-3}, $\mu = 0.83$ mm^{-1}, $F(000) = 272$, $R = 0.048$ for 2065 unique reflections. Both the cations and anions in 2T and two crystallographically independent cations in 4T

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have a center of symmetry. In these crystals, the cations and anions are held together by N—H···O hydrogen bonds to form three-dimensional networks.

Experimental. Experimental details are listed in Table 1. Both crystals obtained from aqueous solutions by slow evaporation at room temperature. D_m by flotation in benzene–CCl$_4$. Rigaku AFC-5 four-circle diffractometer equipped with rotating anode, ω–2θ scan method [scan speed 4° min^{-1} for 2T and 6° min^{-1} for 4T in ω, scan range in ω: (1.2 + 0.15tanθ)°], Ni-filtered Cu $K\alpha$ at 40 kV, 200 mA, background measured for 4 s on either side of the peak; three standard reflections recorded every 97 reflections, no variation in intensity. Lorentz and polarization corrections; no absorption correction. All the unique reflections used in structure analysis. The structures solved by *MULTAN*84 and refined

Table 1. *Experimental details*

	2T	4T
Crystal habit	Prismatic c	Prismatic c
Crystal size (mm)	$0.20 \times 0.20 \times 0.30$	$0.25 \times 0.30 \times 0.30$
Refinements for lattice parameters		
Number	20	20
2θ range (°)	25–40	33–41
Systematic absences	$h0l$ for $h + l$ odd	No condition
	$0k0$ for k odd	
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.5753	0.5753
Range of h, k, l	$-9 \le h \le 9$	$-9 \le h \le 9$
	$0 \le k \le 10$	$-13 \le k \le 13$
	$0 \le l \le 8$	$0 \le l \le 9$
Fluctuation of standard reflections		
$\Sigma(|F_o|/|F_o|_{initial})/3$	1.00–1.05	0.98–1.02
R_{int}	0.010 for 67	0.005 for 163
	$hk0$ reflections	$hk0$ reflections
Number of measured reflections	906	2230
Number of unique reflections	839	2067
Number of reflections with $|F_o| > \sigma(|F_o|)$	824	2006
Secondary extinction:		
number of reflections	5	25
g	7.29×10^{-5}	2.04×10^{-4}
R for unique reflections	0.042	0.048*
wR	0.064	0.090*
S	3.22	1.24*
$(\Delta/\sigma)_{max}$ for non-H atoms	0.1/0.4	0.3/1.2
$\Delta\rho_{max/min}$ (e Å^{-3})	$+0.26/-0.27$	$+0.39/-0.51$

* Excluding $\bar{2}11$ and $\bar{1}02$ reflections.

(non-H atoms anisotropically) by block-diagonal least-squares method, $\Sigma w(|F_o| - |F_c|)^2$ minimized with $w = 1.0/[\sigma(F_o)^2 + p|F_o| + q|F_o|^2]$ for $|F_o| > 0$, $w = r$ for $|F_o| = 0$ (for 2T $p = -0.0412$, $q = 0.0471$, $r = 5.6886$; for 4T $p = -0.0227$, $q = 0.0054$, $r = 10.8802$). The positions of H atoms determined from difference Fourier maps and refined isotropically. Correction for secondary extinction with $I_{corr} = I_o \times (1 + gIc)$. In the final refinement of 4T $\bar{2}11$ and $\bar{1}02$ reflections omitted because of poor agreement of the $|F_o|$ and $|F_c|$.

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs *MULTAN*84 (Main, Germain & Woolfson, 1984), *HBLS*-V and *DAPH* (Ashida, 1973), *MOLCON* (Fujii, 1979) and *ORTEP*II (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. Final atomic parameters are listed in Table 2.* The thermal ellipsoids are shown in Fig. 1 with the atomic numbering.

Table 2. *Final atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

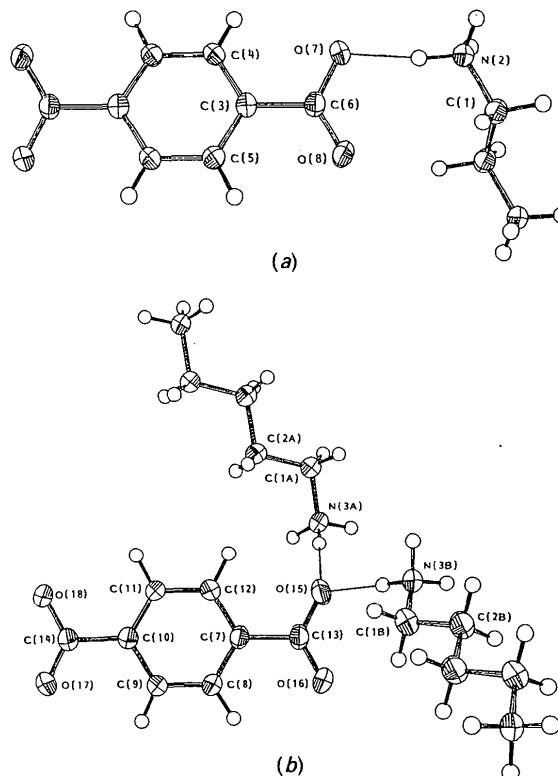|	x	y	z	B_{eq}(Å^2)
2T				
C(1)	0.0710 (2)	0.5410 (1)	-0.0147 (2)	2.21 (5)
N(2)	0.1098 (1)	0.6780 (1)	0.1001 (2)	2.18 (5)
C(3)	0.4094 (1)	0.5140 (1)	0.7936 (2)	1.95 (5)
C(4)	0.4574 (2)	0.6365 (1)	0.9152 (2)	2.06 (5)
C(5)	0.4536 (2)	0.3780 (1)	0.8821 (2)	2.15 (5)
C(6)	0.3112 (2)	0.5286 (1)	0.5711 (2)	1.98 (5)
O(7)	0.3116 (1)	0.6519 (1)	0.4983 (1)	2.57 (4)
O(8)	0.2339 (1)	0.4208 (1)	0.4728 (1)	2.66 (4)
4T				
C(1A)	0.1645 (2)	0.7030 (1)	0.0870 (1)	3.00 (5)
C(2A)	0.0799 (2)	0.5477 (1)	0.0750 (1)	2.88 (5)
N(3A)	0.3371 (1)	0.79929 (9)	0.2206 (1)	2.84 (4)
C(1B)	0.1035 (2)	0.9362 (1)	0.8320 (1)	3.63 (6)
C(2B)	0.0154 (2)	1.0100 (1)	0.9110 (1)	3.25 (5)
N(3B)	0.1244 (1)	0.94714 (9)	0.6552 (1)	2.99 (4)
C(7)	0.3896 (2)	0.6871 (1)	0.6752 (1)	2.59 (4)
C(8)	0.4969 (2)	0.6804 (1)	0.7997 (1)	2.85 (5)
C(9)	0.4809 (2)	0.5567 (1)	0.7995 (1)	3.01 (5)
C(10)	0.3529 (2)	0.4350 (1)	0.6758 (1)	2.54 (4)
C(11)	0.2440 (2)	0.4414 (1)	0.5517 (1)	2.89 (5)
C(12)	0.2624 (2)	0.5659 (1)	0.5508 (1)	3.06 (5)
C(13)	0.4077 (2)	0.8228 (1)	0.6768 (1)	3.07 (5)
C(14)	0.3357 (2)	0.3004 (1)	0.6783 (1)	2.75 (4)
O(15)	0.3082 (1)	0.82247 (9)	0.5585 (1)	4.33 (4)
O(16)	0.5143 (2)	0.92492 (9)	0.7972 (1)	4.17 (4)
O(17)	0.4371 (1)	0.30255 (9)	0.7946 (1)	3.93 (4)
O(18)	0.2193 (1)	0.19265 (8)	0.5649 (1)	3.54 (4)

(a)

(b)

Fig. 1. The thermal ellipsoids (50% probability) with atomic numbering. The H atoms are represented as spheres equivalent to $B = 1.0$ Å^2. Hydrogen bonds are shown by thin lines.

Table 3. *Bond lengths (Å) and angles (°) and geometry of hydrogen bonds*

2T

C(1)—C(1ⁱⁱ)	1·504 (3)	C(4)—C(5ⁱⁱⁱ)	1·379 (2)
C(1)—N(2)	1·485 (2)	C(3)—C(6)	1·513 (2)
C(3)—C(4)	1·398 (2)	C(6)—O(7)	1·263 (2)
C(3)—C(5)	1·394 (2)	C(6)—O(8)	1·242 (2)

N(2)—C(1)—C(1ⁱⁱ)	109·7 (1)	C(5)—C(3)—C(6)	120·7 (1)
C(4)—C(3)—C(5)	118·5 (1)	C(3)—C(6)—O(7)	117·0 (1)
C(3)—C(4)—C(5ⁱⁱⁱ)	120·3 (1)	C(3)—C(6)—O(8)	118·6 (1)
C(3)—C(5)—C(4ⁱⁱⁱ)	121·2 (1)	O(7)—C(6)—O(8)	124·4 (1)
C(4)—C(3)—C(6)	120·8 (1)		

4T

C(1A)—C(2A)	1·517 (2)	C(10)—C(11)	1·389 (2)
C(2A)—C(2Aⁱⁱ)	1·519 (3)	C(11)—C(12)	1·383 (2)
C(1A)—N(3A)	1·485 (2)	C(12)—C(7)	1·392 (2)
C(1B)—C(2B)	1·513 (2)	C(7)—C(13)	1·512 (2)
C(2B)—C(2Bⁱⁱⁱ)	1·525 (3)	C(10)—C(14)	1·509 (2)
C(1B)—N(3B)	1·486 (2)	C(13)—O(15)	1·261 (2)
C(7)—C(8)	1·385 (2)	C(13)—O(16)	1·247 (1)
C(8)—C(9)	1·382 (2)	C(14)—O(17)	1·248 (2)
C(9)—C(10)	1·393 (2)	C(14)—O(18)	1·255 (2)

N(3A)—C(1A)—C(2A)	112·3 (1)	C(12)—C(7)—C(13)	120·8 (1)
C(1A)—C(2A)—C(2Aⁱⁱ)	111·6 (1)	C(8)—C(7)—C(13)	120·4 (1)
N(3B)—C(1B)—C(2B)	111·7 (1)	C(9)—C(10)—C(14)	119·9 (1)
C(1B)—C(2B)—C(2Bⁱⁱⁱ)	110·7 (1)	C(11)—C(10)—C(14)	121·5 (1)
C(12)—C(7)—C(8)	118·7 (1)	C(7)—C(13)—O(15)	117·3 (1)
C(7)—C(8)—C(9)	120·9 (1)	C(7)—C(13)—O(16)	117·8 (1)
C(8)—C(9)—C(10)	120·6 (1)	C(10)—C(14)—O(17)	117·8 (1)
C(9)—C(10)—C(11)	118·5 (1)	C(10)—C(14)—O(18)	118·4 (1)
C(10)—C(11)—C(12)	120·8 (1)	O(15)—C(13)—O(16)	124·8 (2)
C(11)—C(12)—C(7)	120·5 (1)	O(17)—C(14)—O(18)	123·8 (1)

	Donor (N)	Acceptor (O)	N···O (Å)
2T			
(1)	N(2ⁱ)	O(7ⁱ)	2·732 (2)
(2)	N(2ⁱ)	O(7ⁱᵛ)	2·769 (2)
(3)	N(2ⁱ)	O(8ᵛ)	2·766 (1)
4T			
(4)	N(3Aⁱ)	O(15ⁱ)	2·778 (2)
(5)	N(3Aⁱ)	O(16ⁱᵛ)	2·761 (2)
(6)	N(3Aⁱ)	O(17ⁱ)	2·749 (2)
(7)	N(3Bⁱ)	O(15ⁱ)	2·721 (2)
(8)	N(3Bⁱ)	O(18ⁱⁱ)	2·750 (2)
(9)	N(3Bⁱ)	O(18ᵛⁱⁱ)	2·760 (2)

Symmetry codes: For 2T (i) x, y, z; (ii) $-x, 1-y, -z$; (iii) $1-x$, $1-y, 2-z$; (iv) $-\frac{1}{2}+x, \frac{3}{2}-y, -\frac{1}{2}+z$; (v) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$. For 4T (i) x, y, z; (ii) $-x, 1-y, -z$; (iii) $-x, 2-y, 2-z$; (iv) $1-x$, $2-y, 1-z$; (v) $1-x, 1-y, 1-z$; (vi) $-x, 1-y, 1-z$; (vii) $x, 1+y, z$.

Bond lengths and angles and geometry of the hydrogen bonds are listed in Table 3. The stereoscopic views of the crystal structures are shown in Fig. 2.

Related literature. The structures of the title compounds have been determined as an extension of the previous study on Nylon salts with aliphatic cation and aromatic anion (Moritani, Kashino & Haisa, 1990). The cations in 2T and 4T take *trans* zigzag conformations as found in hexamethylenediammonium terephthalate dihydrate (Moritani, Kashino & Haisa, 1990). In the anions C—O bonds which
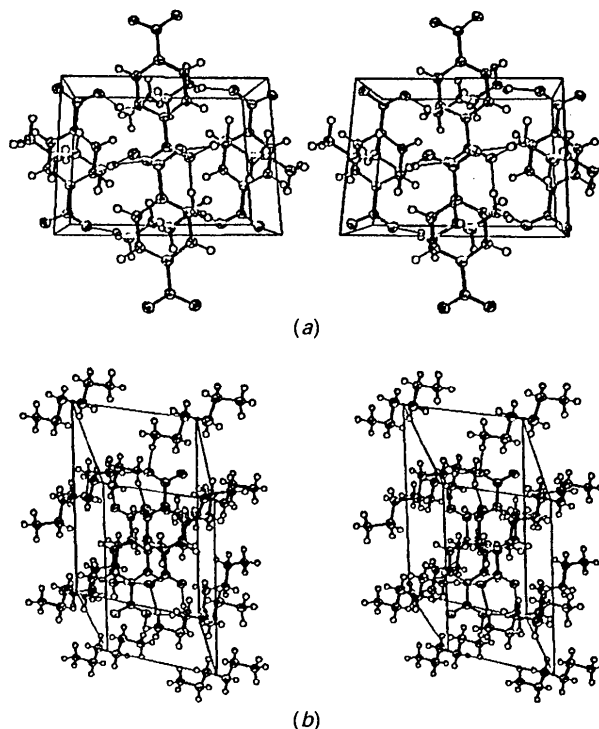

(a)

(b)

Fig. 2. Stereoscopic views of the crystal structures. Hydrogen bonds are shown by thin lines. (a) 2T. The c axis points upward, the b axis from left to right, and the a axis onto the plane of the paper. (b) 4T. The b axis points upward, the c axis from left to right, and the a axis into the plane of the paper.

accept two hydrogen bonds from the ammonium ions are longer than those which accept only one hydrogen bond, as found in tetramethylenediammonium adipate (Hirokawa & Ashida, 1962).

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