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Structure of Piperazinium Adipate, $C_6H_8O_4^{2-}.C_4H_{12}N_2^{2+}$*

BY MICHELINE VANIER AND FRANÇOIS BRISSE

Département de Chimie, Université de Montréal, CP 6210, *Succ. A, Montréal, Québec, Canada H*3*C* 3*V*1

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Abstract. $M_r = 232{\cdot}28$, triclinic, $P\bar{1}$, $a = 5{\cdot}750\,(3)$, $b = 7{\cdot}463\,(2)$, $c = 7{\cdot}501\,(4)$ Å, $\alpha = 64{\cdot}49\,(3)$, $\beta = 81{\cdot}53\,(4)$, $\gamma = 80{\cdot}24\,(3)°$, $V = 285{\cdot}7$ Å^3, $Z = 1$, $D_m = 1{\cdot}35$, $D_x = 1{\cdot}336$ Mg m^{-3}, λ(Cu $K\bar{\alpha}$) = 1·54178 Å, $\mu = 0{\cdot}82$ mm^{-1}, $F(000) = 126$, $T = 293$ K. Final $R = 0{\cdot}050$ for 927 observed reflections. The structure comprises a piperazinium cation, in the chair conformation, situated on a crystallographic center of symmetry and an adipate anion also on a center of symmetry. This anion is in the *trans–trans–trans* conformation. The carboxy groups are tilted away from the plane of the methylenic C atoms by 30°.

Introduction. This paper is part of a series of structure determinations of piperazinium alkanedioates. The crystal structures of piperazinium glutarate (Vanier & Brisse, 1982) and piperazinium succinate (Vanier & Brisse, 1983) have recently been reported. This work has been undertaken to establish whether or not the observed regular variations of the densities and the melting points in the series of piperazinium alkanedioates (Vanier, 1982) can be structurally related.

Experimental. Preparation reported by Vanier (1982), colorless platelets (from 1-pentanol); D_m by flotation in chloroform/monochlorobenzene, m.p. = 523 K; 0·04 × 0·20 × 0·22 mm; Nonius CAD-4 diffractometer, graphite-monochromatized Cu $K\alpha$; orientation verified every 100 measurements, intensity check every hour using three standard reflections, largest intensity fluctuation: 2·5%; 1318 $(hkl, hk\bar{l}, h\bar{k}l, h\bar{k}\bar{l})$ with $2\theta \le 140{\cdot}0°$, 927 with $I \ge 1{\cdot}90\sigma(I)$, 391 unobserved; Lp correction, no absorption correction; direct methods (*MULTAN*, Main, Lessinger, Woolfson, Germain & Declercq, 1977); anisotropic full-matrix least-squares refinement based on F's, H(calculated) isotropic; final $R = 0{\cdot}050$, $R_w = 0{\cdot}060$; maximum $(\text{shift}/\sigma) = 0{\cdot}95$, mean $(\text{shift}/\sigma) = 0{\cdot}30$; final electron-density fluctuations $-0{\cdot}25$ to $+0{\cdot}27$ 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 those listed in Vanier & Brisse (1983).

Discussion. The final atomic coordinates are given in Table 1.† The atomic numbering is shown in Fig. 1 for the adipate anion, that of the piperazinium cation is given in Vanier & Brisse (1983). The bond distances, angles and torsion angles are given in Table 2.

The structure includes a piperazinium cation, located on a center of symmetry $(0\frac{1}{2}\frac{1}{2})$. This cation is in the chair conformation and all its dimensions compare well with previously established values (Vanier & Brisse, 1982). The adipate anion is also situated on a center of symmetry of the unit cell $(\frac{1}{2}00)$. The adipate anion is almost planar and is in the *trans–trans–trans* conformation. The actual values of the torsion angles are −177·4 (3), 180 and 177·4 (3)° for C(1)–C(2)–C(3)–C(3′), C(2)–C(3)–C(3′)–C(2′) and C(3)–C(3′)–C(2′)–C(1′) respectively. The carboxy groups are tilted by 30° from the plane of the C atoms. This conformation is similar to that of adipic acid (Housty & Hospital, 1965) but differs significantly from that of piperazinium succinate where all the atoms of the succinate anion are coplanar (Vanier & Brisse,

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* Structural Studies of Compounds with Aliphatic Chains. 9. Part 8: Vanier & Brisse (1983).

† Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38423 (6 pp.). Copies may be obtained from The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* (× 10^4 *for* O, N *and* C; × 10^3 *for* H *atoms*), U_{eq} (× 10^4 *for* O, N *and* C) *and* U_{iso} (× 10^3 *for* H) *with e.s.d.'s in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$/ $U_{\rm iso}$(Å^2)
O(1)	1713 (3)	7703 (3)	8053 (3)	429 (8)
O(2)	4952 (3)	6503 (3)	6796 (3)	408 (7)
C(1)	3894 (3)	7556 (3)	7653 (3)	308 (9)
C(2)	5284 (4)	8854 (4)	8084 (4)	466 (12)
C(3)	4325 (4)	9316 (3)	9822 (3)	326 (9)
C(1P)	9473 (4)	6152 (3)	2974 (3)	325 (8)
C(2P)	9613 (4)	2937 (3)	5856 (3)	327 (8)
N(1)	10313 (3)	3989 (3)	3722 (3)	289 (7)
H(21)	688 (5)	823 (4)	824 (4)	65 (8)
H(22)	516 (5)	1040 (4)	661 (4)	70 (9)
H(31)	448 (4)	790 (4)	1109 (4)	51 (7)
H(32)	261 (4)	979 (3)	974 (3)	38 (6)
H(11P)	1005 (4)	674 (3)	160 (3)	38 (6)
H(12P)	771 (4)	623 (3)	310 (3)	36 (6)
H(21P)	784 (4)	311 (3)	599 (3)	42 (7)
H(22P)	1028 (4)	153 (3)	635 (4)	44 (7)
H(1N)	960 (5)	338 (4)	302 (4)	57 (8)
H(2N)	1204 (4)	376 (3)	350 (3)	44 (7)

Table 2. *Bond distances* (Å), *angles and torsion angles* (°) *in piperazinium adipate*

Adipate anion		Piperazinium cation	
C(1)–O(1)	1·243 (3)	N(1)–C(1P)	1·480 (3)
C(1)–O(2)	1·248 (3)	N(1)–C(2P)	1·475 (3)
C(1)–C(2)	1·513 (4)	C(1P)–C(2P)	1·510 (3)
C(2)–C(3)	1·494 (4)		
C(3)–C(3')*	1·510 (5)		
O(1)–C(1)–O(2)	123·2 (2)	C(1P)–N(1)–C(2P)	111·4 (2)
O(1)–C(1)–C(2)	117·7 (2)	N(1)–C(1P)–C(2P')	110·4 (2)
O(2)–C(1)–C(2)	118·9 (2)	N(1)–C(2P)–C(1P')	110·7 (2)
C(1)–C(2)–C(3)	116·0 (2)		
C(2)–C(3)–C(3')	115·2 (3)		
O(1)–C(1)–C(2)–C(3)	31·9 (3)	N(1')–C(1P')–C(2P)–N(1)	−56·2 (3)
O(2)–C(1)–C(2)–C(3)	−153·0 (2)	C(2P)–N(1)–C(1P)–C(2P')	56·6 (3)
C(1)–C(2)–C(3)–C(3')	−177·4 (3)	C(1P)–N(1)–C(2P)–C(1P')	−56·7 (3)

* The primed atoms are centrosymmetrically related to the umprimed atoms having the same number. Since the atoms are symmetry-related, the e.s.d.'s include a factor of $\sqrt{2}$.

1983). A stereo pair showing the unit-cell content is given in Fig. 2. Each piperazinium cation is hydrogen-bonded to four different adipate anions and *vice versa*. These hydrogen-bonded structural units form a two-dimensional network parallel to (01$\bar{1}$). The geometry of the hydrogen-bonding scheme is as follows: N(1)···O(1) = 2·670 (3), H(1N)···O(1) = 1·68 (3), N(1)–H(1N) = 0·99 (3) Å and N(1)–H(1N)···O(1) = 177 (3)°; N(1)···O(2) = 2·678 (2), H(2N)···O(2) = 1·70 (2), N(1)–H(2N) = 0·98 (2) Å and N(1)–H(2N)···O(2) = 175 (3)°.

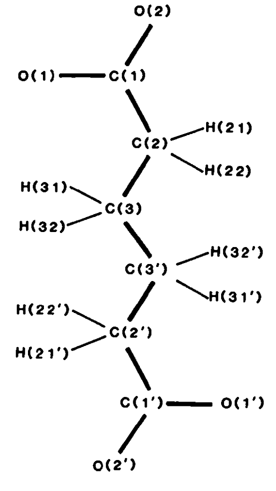

Fig. 1. Atomic numbering of the adipate anion.

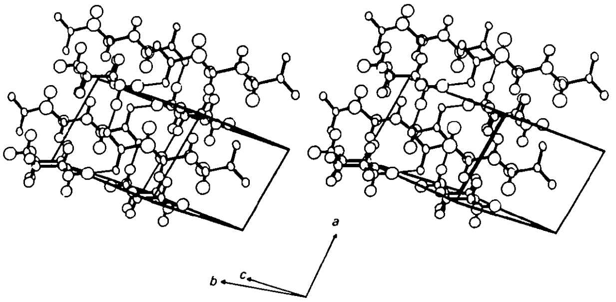

Fig. 2. Stereoview of the unit-cell content of piperazinium adipate.

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