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Structure of Piperazinium Succinate, $C_4H_4O_4^{2-}.C_4H_{12}N_2^{2+}$*

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Abstract. $M_r = 204{\cdot}23$, triclinic, $P\bar{1}$, $a = 5{\cdot}8225\,(11)$, $b = 6{\cdot}0027\,(9)$, $c = 6{\cdot}8943\,(15)$ Å, $\alpha = 95{\cdot}06\,(2)$, $\beta = 99{\cdot}54\,(2)$, $\gamma = 93{\cdot}77\,(2)°$, $V = 235{\cdot}91$ Å^3, $Z = 1$, $D_m = 1{\cdot}44$, $D_x = 1{\cdot}437$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}927$ mm^{-1}, $F(000) = 110$, $T = 293$ K. Final $R = 0{\cdot}037$ for 869 observed reflections. The structure consists of a completely planar succinate anion on a crystallographic center of symmetry and a piperazinium cation, in the chair conformation, also on a crystallographic center of symmetry. Each piperazinium cation forms four hydrogen bonds with four different succinate anions.

Introduction. The present study follows a report in which the densities and the melting points, in a series of piperazinium alkanedioates, were correlated to the parity of the number of C atoms in the alkanedioate chain (Vanier, 1982). The crystal structure of piperazinium glutarate, reported recently (Vanier & Brisse, 1982), revealed that the glutarate anion, with an odd number of C atoms, had a *gauche–trans* conformation. It is to find out if the above observed correlation may be linked to a structural differentiation that the structure determination of three alkanedioates, all having an even number of methylene groups, has been undertaken. We report here on the crystal structure of piperazinium succinate.

Experimental. Prepared by reaction between the diacid and a matched amount of piperazine in 2-propanol, recrystallization in dimethylformamide solution; D_m by flotation in chloroform/monochlorobenzene m.p. = 481 K; colorless thin platelets, 0·08 × 0·10 × 0·58 mm; Nonius CAD-4 diffractometer, graphite-monochromatized Cu $K\alpha$; intensity check every hour, orientation verified every 100 reflections using three standard reflections, largest intensity fluctuation: 2·1%; 973 unique reflections (hkl, $hk\bar{l}$, $h\bar{k}l$, $h\bar{k}\bar{l}$) with $2\theta \leq 150°$, 873 with $I \geq 1{\cdot}90\sigma(I)$, 100 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 $R = 0{\cdot}037$ (four reflections given zero weight in last refinement cycle), $R_w = 0{\cdot}038$, $w = 1$, for all measured reflections $R = 0{\cdot}046$; maximum (shift/σ) = 1·0, average (shift/σ) = 0·2; final electron-density fluctuations −0·21 to +0·26 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 modified versions of *NRC*-2 (data reduction), *NRC*-10 (bond distances and angles) and *NRC*-22 (mean planes) (Ahmed, Hall, Pippy & Huber, 1973), *FORDAP* (Fourier and Patterson maps, A. Zalkin), *NUCLS* (least-squares refinement, Doedens & Ibers, 1967), *MULTAN* (Main *et al.*, 1978), *ORTEP* (stereoviews, Johnson, 1965).

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* Structural Studies of Compounds with Aliphatic Chains. 8. For previous papers, see Vanier & Brisse (1982).

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Discussion. The final atomic coordinates are given in Table 1.* The atomic numbering is shown in Fig. 1 while the bond distances, angles and torsion angles are given in Table 2.

The structure consists of a piperazinium cation in the chair conformation, located on a center of symmetry $(\frac{1}{2}00)$ and a succinate anion also on a center of symmetry $(0\frac{1}{2}\frac{1}{2})$ of the unit cell. The succinate anion is almost planar, the carboxy groups being tilted away from the ethylene chain by only 1·2 (3)°. This compares well with the nearly planar conformations of succinic acid in the benzamide–succinic acid complex (Huang, Leiserowitz & Schmidt, 1973), in succinic acid itself (Broadley, Cruickshank, Morrison, Robertson & Shearer, 1959), and in diphenyl succinate (Brisse, Molhant & Pérez, 1979). A stereo pair showing the unit-cell content is given in Fig. 2. There are some short N···O contacts so that each piperazinium is hydrogen-bonded to four different succinate anions and *vice versa*. These hydrogen bonds connect anions and cations in a two-dimensional network. This layer-like organization is reflected in the crystal morphology. The hydrogen-bonding characteristics are as follows: N(1)···O(1) = 2·657 (2), H(1N)···O(1) = 1·71 (2), N(1)–H(1N) = 0·98 (2) Å and N(1)–H(1N)···O(1) = 161 (2)°; N(1)···O(2) = 2·730 (2), H(2N)···O(2) = 1·78 (2), N(1)–H(2N) = 0·96 (2) Å and N(1)–H(2N)···O(2) = 170 (2)°.

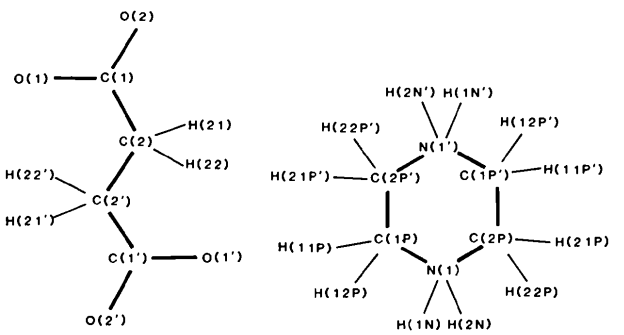

Fig. 1. Atomic numbering of the succinate and piperazinium ions.

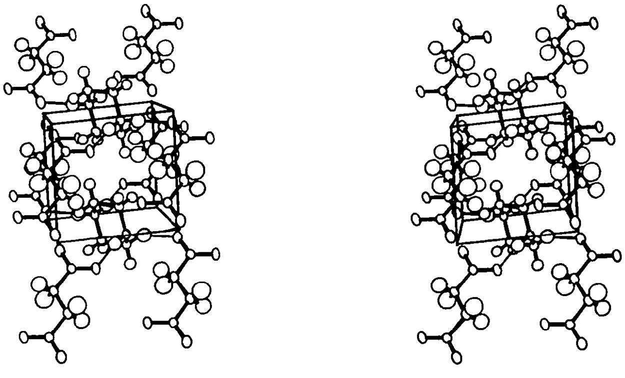

Fig. 2. Stereo pair of the unit-cell content of piperazinium succinate. The *a* axis is vertical and the *b* axis horizontal.

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*

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

	x	y	z	U_{eq}/ U_{iso}(Å^2)
O(1)	1290 (2)	341 (2)	2562 (2)	356 (4)
O(2)	2803 (2)	3158 (2)	1185 (2)	319 (4)
C(1)	2643 (3)	1137 (3)	1498 (2)	216 (4)
C(2)	4178 (3)	−480 (3)	616 (3)	308 (6)
N(1)	9317 (2)	3874 (2)	6606 (2)	233 (4)
C(1P)	7856 (3)	3588 (3)	4600 (2)	255 (5)
C(2P)	11830 (3)	4363 (3)	6483 (2)	253 (5)
H(21)	506 (5)	−119 (5)	175 (4)	87 (9)
H(22)	318 (5)	−175 (4)	−16 (4)	70 (8)
H(1N)	917 (4)	246 (4)	720 (3)	53 (6)
H(2N)	874 (3)	503 (3)	740 (3)	38 (5)
H(11P)	835 (3)	222 (3)	381 (3)	27 (5)
H(12P)	619 (4)	332 (3)	474 (3)	41 (5)
H(21P)	1280 (3)	466 (3)	787 (3)	39 (5)
H(22P)	1232 (3)	302 (3)	575 (3)	26 (5)

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

Succinate anion		Piperazinium cation	
C(1)–O(1)	1·261 (2)	N(1)–C(1P)	1·488 (2)
C(1)–O(2)	1·251 (2)	N(1)–C(2P)	1·491 (2)
C(1)–C(2)	1·517 (2)	C(1P)–C(2P′)	1·510 (3)
C(2)–C(2′)*	1·501 (4)		
O(1)–C(1)–O(2)	123·6 (2)	C(1P)–N(1)–C(2P)	110·8 (1)
O(1)–C(1)–C(2)	116·7 (1)	N(1)–C(1P)–C(2P′)	111·2 (2)
O(2)–C(1)–C(2)	119·7 (1)	N(1)–C(2P)–C(1P′)	110·3 (2)
C(1)–C(2)–C(2′)	116·7 (2)		
O(1)–C(1)–C(2)–C(2′)	−178·8 (3)	N(1′)–C(1P′)–C(2P)–N(1)	−56·7 (4)
O(2)–C(1)–C(2)–C(2′)	−0·3 (3)	C(2P)–N(1)–C(1P)–C(2P′)	−56·9 (3)
		C(1P)–N(1)–C(2P)–C(1P′)	56·5 (3)

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

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

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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 $2\theta \leq 140{\cdot}0°$, 927 with $I \geq 1{\cdot}90\sigma(I)$, 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 (shift/σ) = 0·95, mean (shift/σ) = 0·30; final electron-density fluctuations −0·25 to +0·27 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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