The structure of potassium hydrogen *meso*-tartrate was investigated by Kroon & Kanters (1972) with X-ray diffraction at 113 K and by Currie, Speakman, Kanters & Kroon (1975) with neutron diffraction. It is centrosymmetric, space group $P\bar{1}$ with two independent molecules in the asymmetric unit. As the *meso*-tartaric residues are dissymmetric, this structure is a racemate. It too shows a greater variety of intermolecular hydrogen bonds than the structure of the title compound. Two of the seven independent hydrogen bridges are symmetric across different inversion centres, connecting carboxy groups and having O···O distances of 2·483 (2) and 2·452 (2) Å. The one K^+ ion is surrounded by nine O atoms, the other by seven.

We wish to thank Mrs Eveline Müller for help with the calculations and the computer diagram and Professor W. Wendling for calculating and programming the standard deviation of the angle between two least-squares planes.

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(–)-*cis*-2,8-Dimethyl-1,2,3,4,4a,9b-hexahydro-2*H*$^+$-pyrido[4,3-*b*]indolium (*S*,*S*)-2,3-Di-*O*-benzoyltartrate(2–), $2C_{13}H_{19}N_2^+.C_{18}H_{12}O_8^{2-}$:A New Antiarrhythmic Drug

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Abstract. $M_r = 762·9$, monoclinic, $C2$, $a = 17·470$ (8), $b = 7·975$ (1), $c = 14·406$ (3) Å, $\beta = 94·10$ (3)°, $V = 2002$ Å^3, $Z = 2$, $D_m = 1·26$, $D_x = 1·266$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·82$ mm^{-1}, $F(000) = 812$, $T = 293$ K, final $R = 0·034$ for 1252 unique observed reflections. The crystal structure consists of groups of three ions: two cations and one anion linked by hydrogen bonds. The groups possess crystallographic C_2 symmetry and are held together by van der Waals forces only. The saturated six-membered ring in the cation exists in a flattened chair conformation while the five-membered ring adopts a symmetric C_s envelope conformation.

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Introduction. The title cation [hereafter (–)DB1021] is a new, structurally and chemically distinct, anti-arrhythmic drug, whose site and mechanism of action are currently being investigated. It may exist in a number of geometrical conformational and optical isomers but only the present isomer has been found to be active. Of particular interest is, therefore, the detailed conformation and absolute configuration of the active (–)-enantiomer of DB1021.

Experimental. Single crystals obtained from an aqueous alcoholic solution of (–)DB1021 and (–)-(*S*,*S*)-2,3-di-*O*-benzoyltartaric acid (hereafter *S*,*S*-DBTA),

Table 1. *Atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic thermal parameters*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
N(1)	7742 (1)	1704 (3)	1175 (1)	6·01
N(2)	7683 (1)	1287 (3)	3739 (1)	7·20
C(1)	7601 (1)	44 (4)	1638 (2)	8·00
C(2)	6962 (1)	265 (4)	2296 (2)	8·51
C(3)	7098 (1)	1684 (4)	2980 (2)	7·45
C(4)	7409 (1)	3306 (4)	2591 (2)	5·96
C(5)	8007 (1)	2949 (3)	1881 (1)	5·37
C(6)	7980 (1)	2814 (4)	4073 (1)	6·24
C(7)	8417 (1)	3134 (4)	4900 (2)	7·18
C(8)	8700 (1)	4735 (4)	5044 (2)	7·41
C(9)	8561 (1)	6035 (4)	4411 (2)	7·08
C(10)	8113 (1)	5681 (4)	3596 (2)	6·53
C(11)	7824 (1)	4084 (3)	3429 (1)	5·86
C(12)	8306 (1)	1517 (4)	445 (2)	7·29
C(13)	8878 (2)	7765 (5)	4577 (2)	9·29
O(1)	6518 (1)	649 (3)	−355 (1)	8·56
O(2)	6327 (1)	2885 (2)	509 (1)	5·93
O(3)	5050 (1)	332 (2)	−919 (1)	5·11
O(4)	5331 (1)	1441 (2)	−2270 (1)	7·67
C(14)	6112 (1)	1786 (3)	−70 (1)	5·51
C(15)	5254 (1)	1828 (3)	−408 (1)	4·56
C(16)	5136 (1)	267 (3)	−1826 (1)	4·72
C(17)	4961 (1)	−1434 (3)	−2199 (1)	4·22
C(18)	5118 (1)	−1792 (3)	−3109 (1)	5·08
C(19)	5002 (1)	−3386 (4)	−3458 (2)	6·48
C(20)	4714 (1)	−4621 (3)	−2913 (2)	6·66
C(21)	4544 (1)	−4276 (3)	−2019 (2)	5·90
C(22)	4664 (1)	−2682 (3)	−1658 (1)	4·96

colourless, 0·4 × 0·3 × 0·25 mm, D_m by flotation; systematic absences, hkl for $h+k=2n+1$, from Weissenberg photographs; Syntex $P2_1$ four-circle diffractometer; accurate unit-cell parameters by least-squares refinement of 16 reflections, $12 < \theta < 45°$; intensity data ($h=0$ to 18, $k=0$ to 8, $l=-15$ to 15) collected with Cu $K\alpha$ radiation, θ–2θ scan mode; two standard reflections measured every 98 reflections, no significant systematic fluctuation; intensities corrected for Lorentz–polarization effects but not for absorption; 1375 unique reflections, $3 < \theta < 55°$, 1252 with $I \geq 1{\cdot}96\sigma(I)$ considered as observed and included in the refinement; structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *YZARC* (Baggio, Woolfson, Declercq & Germain, 1978) and refined by Fourier and block-diagonal least-squares methods, difference electron density map showed positions of all H atoms, refinement continued on all positional parameters, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms; in final cycle $R = 0{\cdot}034$, $R_w = 0{\cdot}038$ for observed reflections only, max. shift/e.s.d. 0·16, function minimized $\sum w(\Delta F)^2$, where $w = 1$ if $|F_o| < 35$ and $w = 35/|F_o|$ if $|F_o| \geq 35$, max. final electron density difference peak 0·22 e Å^{-3}; scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974); all calculations except *MULTAN* and *YZARC* performed with the NRC system (Ahmed, Hall, Pippy & Huber, 1973).

Table 2. *Interatomic distances* (Å) *and valence angles* (°) *with e.s.d.'s in parentheses*

N(1)–C(1)	1·511 (4)	C(9)–C(13)	1·500 (5)
N(1)–C(5)	1·472 (3)	C(10)–C(11)	1·385 (4)
N(1)–C(12)	1·498 (3)	O(1)–C(14)	1·239 (3)
N(2)–C(3)	1·477 (3)	O(2)–C(14)	1·249 (3)
N(2)–C(6)	1·396 (4)	O(3)–C(15)	1·434 (3)
C(1)–C(2)	1·525 (4)	O(3)–C(16)	1·326 (2)
C(2)–C(3)	1·508 (4)	O(4)–C(16)	1·197 (3)
C(3)–C(4)	1·524 (4)	C(14)–C(15)	1·543 (3)
C(4)–C(5)	1·541 (3)	C(15)–C($15^{\rm i}$)	1·522 (3)
C(4)–C(11)	1·497 (3)	C(16)–C(17)	1·483 (3)
C(6)–C(7)	1·391 (3)	C(17)–C(18)	1·389 (3)
C(6)–C(11)	1·387 (4)	C(17)–C(22)	1·387 (3)
C(7)–C(8)	1·380 (5)	C(18)–C(19)	1·377 (4)
C(8)–C(9)	1·390 (4)	C(19)–C(20)	1·377 (4)
C(9)–C(10)	1·393 (3)	C(20)–C(21)	1·370 (3)
		C(21)–C(22)	1·384 (3)
C(1)–N(1)–C(5)	109·8 (3)	C(4)–C(11)–C(6)	107·3 (2)
C(1)–N(1)–C(12)	111·1 (2)	C(4)–C(11)–C(10)	132·1 (2)
C(5)–N(1)–C(12)	111·2 (2)	C(6)–C(11)–C(10)	120·2 (2)
C(3)–N(2)–C(6)	106·8 (2)	C(15)–O(3)–C(16)	119·8 (2)
N(1)–C(1)–C(2)	108·9 (2)	O(1)–C(14)–O(2)	125·6 (2)
C(1)–C(2)–C(3)	113·9 (2)	O(1)–C(14)–C(15)	118·6 (2)
N(2)–C(3)–C(2)	113·0 (2)	O(2)–C(14)–C(15)	115·7 (2)
N(2)–C(3)–C(4)	102·0 (2)	O(3)–C(15)–C(14)	110·1 (2)
C(2)–C(3)–C(4)	116·1 (2)	O(3)–C(15)–C($15^{\rm i}$)	105·1 (2)
C(3)–C(4)–C(5)	111·3 (2)	C(14)–C(15)–C($15^{\rm i}$)	111·3 (2)
C(3)–C(4)–C(11)	102·7 (2)	O(3)–C(16)–O(4)	123·5 (2)
C(5)–C(4)–C(11)	107·4 (2)	O(3)–C(16)–C(17)	111·0 (2)
N(1)–C(5)–C(4)	112·9 (2)	O(4)–C(16)–C(17)	125·6 (2)
N(2)–C(6)–C(7)	128·5 (2)	C(16)–C(17)–C(18)	118·7 (2)
N(2)–C(6)–C(11)	110·8 (2)	C(16)–C(17)–C(22)	121·9 (2)
C(7)–C(6)–C(11)	120·6 (2)	C(18)–C(17)–C(22)	119·4 (2)
C(6)–C(7)–C(8)	117·9 (2)	C(17)–C(18)–C(19)	120·0 (2)
C(7)–C(8)–C(9)	123·1 (3)	C(18)–C(19)–C(20)	120·2 (2)
C(8)–C(9)–C(10)	117·6 (3)	C(19)–C(20)–C(21)	120·3 (2)
C(8)–C(9)–C(13)	122·4 (3)	C(20)–C(21)–C(22)	120·0 (2)
C(10)–C(9)–C(13)	120·0 (3)	C(17)–C(22)–C(21)	120·1 (2)
C(9)–C(10)–C(11)	120·6 (3)		

Symmetry code: (i) $1-x, y, -z$.

Discussion. Atomic coordinates of non-H atoms and equivalent isotropic B's are listed in Table 1,* bond distances and angles in Table 2. The molecular packing and the atom numbering in *S*,*S*-DBTA are shown in Fig. 1. The numbering scheme for (−)DB1021 is shown in Fig. 2, which also displays the conformation of the molecule. The absolute configuration of (−)DB1021 is specified by the known configuration of (−)DBTA and Fig. 2 shows the correct enantiomorphic form. The two chiral centres are C(3)–*R*, C(4)–*R*.

The unit cell contains four molecules of (−)DB1021 and two molecules of *S*,*S*-DBTA; each of the latter lies on a twofold axis of symmetry. Together they form a 2:1 salt. An H atom, originally attached to one of the O atoms of the *S*,*S*-DBTA carboxyl group, was found in the difference synthesis bonded to N(1) of (−)DB1021; no H atom was found attached to carboxyl O. Further

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39671 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

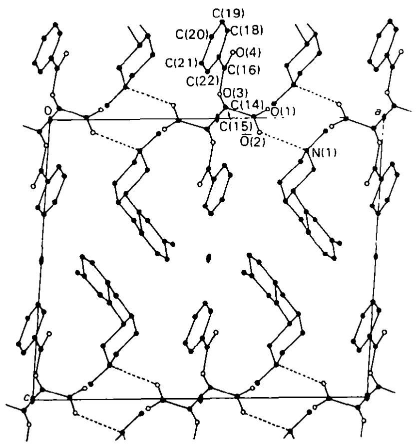

Fig. 1. The crystal structure projected along **b** and numbering scheme for *S,S*-DBTA. Hydrogen bonds are shown by broken lines.

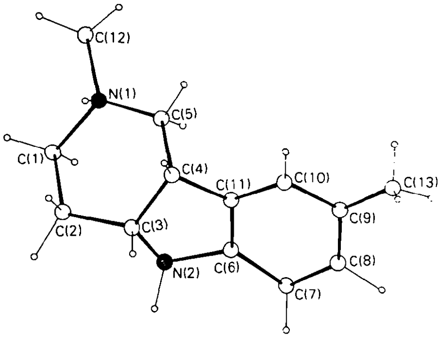

Fig. 2. View of the (−)DB1021 cation.

evidence for proton transfer comes from the carboxyl-group dimensions, C(14)—O(1) = 1·239 (3) and C(14)—O(2) = 1·249 (3) Å, and there is no doubt about the ionic nature of the carboxyl moieties. The intermolecular distances all indicate van der Waals interactions except for the distances between the protonated $N(1)^+$ and O(1) and O(2) of the deprotonated carboxyl group of *S,S*-DBTA, which are 3·077 (3) and 2·754 (2) Å respectively. An $N(1)^+$—H···O(2) hydrogen bond [H···O(2) = 1·76 (2) Å] is clearly indicated, while the second contact may be either part of a bifurcated hydrogen bond or just a van der Waals contact; because of the rather long $N(1)^+$···O(1) distance we prefer the latter possibility.

All bond lengths (Table 2) have values close to those generally expected. The mean values for $N(1)^+$—$C(sp^3)$, $C(sp^3)$—$C(sp^3)$ and $C(sp^3)$—$C(sp^2)$ in the (−)DB1021 cation are 1·494, 1·525 and 1·498 Å respectively. Valence angles at the atoms involved in the ring junctions are considerably distorted from ideal values, indicating strain. The unsaturated five-membered ring is *cis*-fused with the six-membered hetero ring and adopts an envelope form with small puckering [C(3) is 0·490 (2) Å from the mean plane through the four remaining atoms] and high mirror-plane symmetry as shown by the asymmetry parameter (Duax & Norton, 1975), $\Delta C_s[C(3)] = 3{\cdot}2°$. The six-membered hetero ring adopts a distorted chair conformation with the C(12) methyl attached equatorially. The distortion involves loss of rotation symmetry, $\Delta C_2[C(1), C(2)] = 21{\cdot}7°$, with retention of the orthogonal mirror plane, $\Delta C_s[N(1)] = 3{\cdot}0°$ [ring flattened at C(3)].

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The Structure of *N*-Aminophthalimide, $C_8H_6N_2O_2$

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Abstract. $M_r = 162{\cdot}15$, orthorhombic, $P2_12_12_1$, $a = 20{\cdot}472\ (14)$, $b = 6{\cdot}672\ (3)$, $c = 5{\cdot}254\ (3)$ Å, $V = 717{\cdot}5\ (7)$ Å^3, $Z = 4$, $D_m = 1{\cdot}486\ (8)$, $D_x = 1{\cdot}501\ (2)$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}21$ cm^{-1}, $F(000) = 336$, $T = 296$ K, $R = 0{\cdot}046$ for 779 observed reflections. With the exception of the amine hydrogens, the molecule is planar with an N—N bond length of 1·409 (3) Å. The terminal nitrogen

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