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Conformational Aspects of *meso*-Tartaric Acid. IX.* Structure of (+)-(*R*)-α-Methylbenzylammonium *meso*-Tartrate Monohydrate, $2C_8H_{12}N^+.C_4H_4O_6^{2-}.H_2O$

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Abstract. $M_r = 410{\cdot}47$, triclinic, $P1$, $a = 9{\cdot}758\ (2)$, $b = 10{\cdot}552\ (7)$, $c = 11{\cdot}726\ (4)$ Å, $\alpha = 82{\cdot}37\ (4)$, $\beta = 66{\cdot}21\ (2)$, $\gamma = 88{\cdot}50\ (3)°$, $V = 1094{\cdot}5\ (9)$ Å^3, $Z = 2$, $D_m = 1{\cdot}243$(flotation), $D_x = 1{\cdot}245$ Mg m^{-3}, λ(Cu $K\bar{\alpha}$) = 1·5418 Å, μ(Cu $K\bar{\alpha}$) = 0·70 mm^{-1}, $F(000) = 440$, $T = 295$ K, $R = 0{\cdot}095$ for 3859 observed diffractometer data with $I > 2{\cdot}5\sigma(I)$. The *meso*-tartrate anion has a nearly centrosymmetric molecular conformation just as in the corresponding acid salt. The prominent pseudocentre observed in the structure applies to all atoms, except for the methyl groups of the cations and the water molecules. The *meso*-tartrate anions are sandwiched between α-methylbenzylammonium cations and water molecules.

Introduction. In view of the overwhelming evidence for the stability of the dissymmetric rotamers of *meso*-tartaric acid (Kroon, 1982; and references therein), which was formerly considered to be optically inactive due to intramolecular compensation, we thought it would be possible to convert this acid into one of its enantiomeric constituents. In our first attempt we co-crystallized *meso*-tartaric acid with the optically active base α-methylbenzylamine in 1:1 proportion. However, surprisingly, it turned out that in the necessarily dissymmetric crystal the *meso*-tartaric molecules had acquired an almost centrosymmetrical form (Kroon, 1982; Kroon, Duisenberg & Peerdeman, 1984). We also prepared a crystalline 1:2 complex of *meso*-tartaric acid with optically pure α-methylbenzylamine; this paper describes the structure of this complex.

Experimental. Irregularly prismatic crystal, dimensions approximately 0·5 mm, grown from an aqueous solution, exhibiting large mosaic spread. Enraf–Nonius CAD-4 diffractometer, Ni-filtered Cu $K\alpha$ radiation; cell measurement with setting angles of 14 reflections in the θ range from 30·2 to 31·0°; $\omega/2\theta$ scan of width $0{\cdot}60° + 0{\cdot}14°\tan\theta$ and variable speed; $2\theta_{\max} = 150°$, $h = -11$ to 11, $k = -12$ to 12, $l = -14$ to 14; average decay of standard reflections 3%; 8716 reflections measured; $R_{\rm int} = 0{\cdot}13$ from merging 4264 equivalent pairs, 405 of which were considered unobserved $[I \leq 2{\cdot}5\sigma(I)]$; Lp corrections; no absorption correction. Structure solved by direct methods (*MULTAN*80 system; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement on F; block-matrix least squares (three blocks) with anisotropic temperature factors for all non-H atoms. Since the structure is nearly centrosymmetric (except for the water molecules and the methyl groups) large parameter correlation occurred. To achieve convergence, highly correlated parameters were included in the same block (Dunitz, 1979; Franklin & Marsh, 1982). In addition, benzene groups were refined as rigid-body groups [with C(Ph)—C(Ph) distances 1·395 Å]; corresponding bonds of the α-C atoms in the base molecules were restrained to the same value [C(α)—N 1·50, C(α)—methyl 1·53, C(α)—C(Ph) 1·52 Å]. H atoms located from a three-dimensional difference Fourier map and by stereochemical considerations; temperature factors, equal to the isotropic temperature factors of their carrier atoms, were kept fixed during the subsequent refinement; phenyl and aliphatic H according to riding model; methyl and nitrogen H atoms as rigid groups and hydroxyl and water-molecule H atoms with Waser constraints; number of refined parameters 526. Weights according to $w = [\sigma^2(F) + k|F_o|^2]^{-1}$, $k = 0{\cdot}00031$ by demanding that uniform average values of $\sum w(|F_o| - |F_c|)^2$ were obtained when analysed in batches of increasing $|F_o|$ and $(\sin\theta)/\lambda$. Convergence at $R = 0{\cdot}095$, $wR = 0{\cdot}098$, $S = 0{\cdot}56$, $(\Delta/\sigma)_{\max} = 1{\cdot}46$, max. and min. peaks on final $\Delta\rho$ map 0·44 and −0·40 e Å^{-3}, respectively. Calculations carried out on the CDC-Cyber 175 computer of the University of Utrecht with programs of the *APOLLO* (data reduction and correction by A. L. Spek) and *EUCLID* [calculations of geometrical data and illustrations (Spek, 1982)] packages and on the in-house Eclipse 8/230 minicomputer using the programs of a modified version of *SHELX*76 (Sheldrick, 1976). Scattering factors for non-hydrogen atoms from

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Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965). We did not determine the absolute structure of the title compound by the anomalous diffraction technique. However, it could unambiguously be assigned on the basis of the absolute-configuration determination of (−)-α-methylbenzylamine (Busk, Dullforce & Sim, 1969).

Discussion. Positional and equivalent isotropic thermal-vibration parameters for the non-hydrogen atoms are given in Table 1.* The molecular structures of the anions and cations are shown in Fig. 1, which also gives the atom-numbering scheme. Bond lengths, bond angles and selected torsion angles, involving non-hydrogen atoms only, are listed in Table 2.

As in the case of the 1:1 *meso*-tartaric acid/(−)-α-methylbenzylammonium complex (Kroon, Duisenberg & Peerdeman, 1984) and in sodium *meso*-tartrate (Blankensteyn & Kroon, 1985) the conformations of the *meso*-tartaric acid molecules are nearly centrosymmetric. Apparently the crystal-packing conditions favour this particular conformation. The two independent *meso*-tartrate ions are related by a pseudocentre at 0·39, 0·75, 0·57. The carboxyl groups are rotated from the C—C(α)—O(H) planes by 5·9 (4) and 37·7 (4)° respectively in *meso*-tartrate anion (I), and by 3·8 (4) and 26·8 (4)° respectively in *meso*-tartrate anion (II). Planar glycolic acid halves in centrosymmetric molecules are energetically not favourable because of repulsions between the β-hydroxyl groups and the neighbouring carboxyl O atoms (Blankensteyn & Kroon, 1985; Kroon-Batenburg, de Vries & Kroon, 1985). Apparently in one half of each molecule this repulsion is released by the formation of weak intramolecular H atoms.

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *for the non-hydrogen atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
O(1)	0·4854 (4)	0·1129 (4)	0·5479 (4)	0·0713 (5)
O(2)	0·4340 (4)	−0·0949 (4)	0·6056 (4)	0·0705 (5)
O(3)	0·2521 (4)	0·1586 (4)	0·4907 (4)	0·0651 (5)
O(4)	0·1447 (4)	−0·1623 (4)	0·6599 (4)	0·0612 (5)
O(5)	−0·0208 (4)	−0·0826 (4)	0·5291 (4)	0·0629 (5)
O(6)	−0·0806 (4)	0·0857 (4)	0·6289 (4)	0·0621 (5)
O(7)	0·8084 (4)	0·6020 (4)	0·6093 (4)	0·0569 (5)
O(8)	0·8558 (4)	0·4255 (4)	0·5189 (4)	0·0609 (5)
O(9)	0·6186 (4)	0·6758 (4)	0·5036 (4)	0·0591 (5)
O(10)	0·5372 (4)	0·3392 (4)	0·6366 (4)	0·0574 (5)
O(11)	0·3322 (4)	0·6039 (4)	0·5707 (4)	0·0626 (5)
O(12)	0·3172 (4)	0·3951 (4)	0·5663 (4)	0·0652 (5)
O(13)	0·8491 (5)	0·8677 (5)	0·3532 (5)	0·0928 (5)
O(14)	0·6213 (6)	0·1623 (5)	0·8057 (5)	0·1077 (5)
N(1)	0·0135 (4)	0·3239 (4)	0·6603 (4)	0·0516 (5)
N(2)	0·7909 (4)	0·1895 (4)	0·4585 (4)	0·0577 (5)
N(3)	0·6586 (4)	0·8288 (4)	0·6857 (4)	0·0543 (5)
N(4)	0·1150 (4)	0·6804 (4)	0·4838 (4)	0·0507 (5)
C(1)	0·4108 (5)	0·0130 (5)	0·5647 (5)	0·0569 (5)
C(2)	0·2766 (5)	0·0284 (5)	0·5264 (5)	0·0531 (5)
C(3)	0·1321 (5)	−0·0298 (5)	0·6329 (4)	0·0506 (5)
C(4)	0·0005 (5)	−0·0079 (5)	0·5941 (4)	0·0490 (5)
C(5)	0·7811 (5)	0·5209 (5)	0·5500 (5)	0·0511 (5)
C(6)	0·6463 (5)	0·5429 (5)	0·5149 (4)	0·0499 (5)
C(7)	0·5074 (5)	0·4702 (5)	0·6189 (4)	0·0475 (5)
C(8)	0·3741 (5)	0·4913 (5)	0·5820 (5)	0·0493 (5)
C(9)	−0·0576 (5)	0·3328 (4)	0·7974 (4)	0·0621 (5)
C(10)	−0·0022 (3)	0·2264 (3)	0·8659 (3)	0·0622 (5)
C(11)	−0·1054 (3)	0·1311 (3)	0·9460 (3)	0·0816 (5)
C(12)	−0·0583 (3)	0·0283 (3)	1·0077 (3)	0·0941 (5)
C(13)	0·0920 (3)	0·0208 (3)	0·9894 (3)	0·0904 (5)
C(14)	0·1951 (3)	0·1160 (3)	0·9092 (3)	0·0792 (5)
C(15)	0·1480 (3)	0·2188 (3)	0·8475 (3)	0·0698 (5)
C(16)	0·8672 (5)	0·1954 (3)	0·3194 (4)	0·0658 (5)
C(17)	0·8001 (4)	0·3007 (3)	0·2598 (3)	0·0641 (5)
C(18)	0·6633 (4)	0·2736 (3)	0·2552 (3)	0·0777 (5)
C(19)	0·5918 (4)	0·3692 (3)	0·2074 (3)	0·0952 (5)
C(20)	0·6571 (4)	0·4918 (3)	0·1642 (3)	0·0947 (5)
C(21)	0·7938 (4)	0·5189 (3)	0·1687 (3)	0·0924 (5)
C(22)	0·8653 (4)	0·4233 (3)	0·2165 (3)	0·0757 (5)
C(23)	0·5946 (5)	0·8144 (5)	0·8263 (4)	0·0607 (5)
C(24)	0·4830 (3)	0·7022 (3)	0·8779 (3)	0·0593 (5)
C(25)	0·5242 (3)	0·5793 (3)	0·9094 (3)	0·0710 (5)
C(26)	0·4188 (3)	0·4782 (3)	0·9535 (3)	0·0850 (5)
C(27)	0·2721 (3)	0·5000 (3)	0·9660 (3)	0·0900 (5)
C(28)	0·2308 (3)	0·6229 (3)	0·9345 (3)	0·0910 (5)
C(29)	0·3362 (3)	0·7240 (3)	0·8905 (3)	0·0786 (5)
C(30)	0·1717 (5)	0·6824 (4)	0·3446 (4)	0·0621 (5)
C(31)	0·2951 (3)	0·7845 (3)	0·2807 (3)	0·0596 (5)
C(32)	0·4320 (3)	0·7729 (3)	0·2920 (3)	0·0692 (5)
C(33)	0·5421 (3)	0·8699 (3)	0·2341 (3)	0·0815 (5)
C(34)	0·5153 (3)	0·9784 (3)	0·1649 (3)	0·0886 (5)
C(35)	0·3784 (3)	0·9900 (3)	0·1537 (3)	0·1007 (5)
C(36)	0·2683 (3)	0·8930 (3)	0·2116 (3)	0·0800 (5)
C(37)	−0·0260 (6)	0·4678 (5)	0·8176 (5)	0·0710 (5)
C(38)	1·0366 (5)	0·2072 (6)	0·2810 (6)	0·0773 (5)
C(39)	0·7246 (5)	0·8059 (5)	0·8679 (5)	0·0713 (5)
C(40)	0·2225 (6)	0·5482 (5)	0·3125 (5)	0·0746 (5)

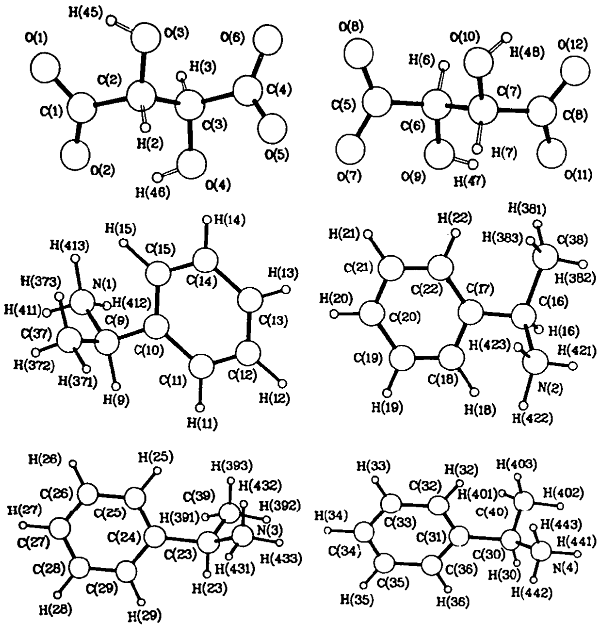

Fig. 1. Perspective views of the compound molecules, showing the atom labelling.

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* Lists of H-atom parameters, structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42215 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

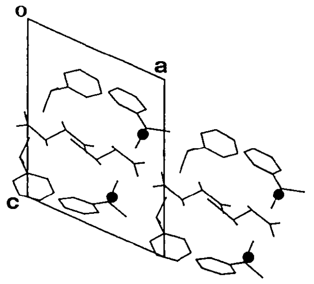

Fig. 2. Projection of the unit-cell contents down the *b* axis. Black spheres represent water molecules.

Table 2. *Bond lengths* (Å), *bond angles* (°), *selected torsion angles* (°) *and hydrogen-bond geometry, with e.s.d.'s in parentheses*

Anion (I)		Anion (II)	
C(1)–O(1)	1·245 (6)	C(5)–O(7)	1·267 (7)
C(1)–O(2)	1·227 (7)	C(5)–O(8)	1·234 (6)
C(1)–C(2)	1·545 (7)	C(5)–C(6)	1·533 (7)
C(2)–O(3)	1·425 (7)	C(6)–O(9)	1·420 (7)
C(2)–C(3)	1·532 (7)	C(6)–C(7)	1·544 (7)
C(3)–O(4)	1·405 (7)	C(7)–O(10)	1·411 (7)
C(3)–C(4)	1·529 (7)	C(7)–C(8)	1·530 (7)
C(4)–O(5)	1·242 (7)	C(8)–O(11)	1·254 (7)
C(4)–O(6)	1·255 (6)	C(8)–O(12)	1·239 (7)
Cation (I)		Cation (II)	
C(9)–N(1)	1·487 (7)	C(16)–N(2)	1·488 (7)
C(9)–C(10)	1·509 (6)	C(16)–C(17)	1·509 (5)
C(9)–C(37)	1·534 (7)	C(16)–C(38)	1·532 (6)
Cation (III)		Cation (IV)	
C(23)–N(3)	1·496 (7)	C(30)–N(4)	1·495 (7)
C(23)–C(24)	1·516 (6)	C(30)–C(31)	1·516 (5)
C(23)–C(39)	1·529 (7)	C(30)–C(40)	1·532 (7)

Anion (I)		Anion (II)	
O(1)–C(1)–C(2)	115·3 (5)	O(7)–C(5)–C(6)	117·7 (4)
O(2)–C(1)–C(2)	117·4 (5)	O(8)–C(5)–C(6)	118·0 (5)
O(1)–C(1)–O(2)	127·3 (5)	O(7)–C(5)–O(8)	124·3 (5)
O(3)–C(2)–C(3)	108·6 (4)	O(9)–C(6)–C(7)	109·6 (4)
C(1)–C(2)–C(3)	111·3 (5)	C(5)–C(6)–C(7)	109·2 (4)
O(3)–C(2)–C(1)	112·7 (4)	O(9)–C(6)–C(5)	109·3 (4)
C(2)–C(3)–C(4)	110·0 (4)	C(6)–C(7)–C(8)	108·7 (4)
O(4)–C(3)–C(2)	110·5 (4)	O(10)–C(7)–C(6)	110·1 (4)
O(4)–C(3)–C(4)	108·2 (4)	O(10)–C(7)–C(8)	111·8 (4)
O(5)–C(4)–O(6)	123·0 (5)	O(11)–C(8)–O(12)	126·0 (5)
O(6)–C(4)–C(3)	118·2 (5)	O(12)–C(8)–C(7)	116·8 (5)
O(5)–C(4)–C(3)	118·8 (4)	O(11)–C(8)–C(7)	117·3 (5)
Cation (I)		Cation (II)	
N(1)–C(9)–C(37)	108·3 (4)	N(2)–C(16)–C(38)	108·5 (4)
C(10)–C(9)–C(37)	114·6 (4)	C(17)–C(16)–C(38)	115·9 (4)
N(1)–C(9)–C(10)	108·9 (3)	N(2)–C(16)–C(17)	108·6 (3)
C(9)–C(10)–C(15)	122·0 (3)	C(16)–C(17)–C(22)	122·5 (3)
C(9)–C(10)–C(11)	117·9 (3)	C(16)–C(17)–C(18)	117·4 (3)
Cation (III)		Cation (IV)	
N(3)–C(23)–C(39)	108·2 (4)	N(4)–C(30)–C(40)	108·7 (4)
C(24)–C(23)–C(39)	115·7 (4)	C(31)–C(30)–C(40)	113·4 (4)
N(3)–C(23)–C(24)	108·9 (4)	N(4)–C(30)–C(31)	108·9 (4)
C(23)–C(24)–C(29)	118·1 (3)	C(30)–C(31)–C(36)	118·5 (3)
C(23)–C(24)–C(25)	122·0 (3)	C(30)–C(31)–C(32)	121·4 (3)

Anion (I)		Anion (II)	
C(1)–C(2)–C(3)–C(4)	177·6 (4)	C(5)–C(6)–C(7)–C(8)	−179·2 (4)
O(1)–C(1)–C(2)–O(3)	−6·4 (7)	O(7)–C(5)–C(6)–O(9)	26·8 (6)
O(2)–C(1)–C(2)–O(3)	174·9 (5)	O(8)–C(5)–C(6)–O(9)	−153·2 (5)
O(5)–C(4)–C(3)–O(4)	−37·9 (6)	O(11)–C(8)–C(7)–O(10)	176·1 (5)
O(6)–C(4)–C(3)–O(4)	142·6 (5)	O(12)–C(8)–C(7)–O(10)	−3·7 (7)
Cation (I)		Cation (II)	
N(1)–C(9)–C(10)–C(11)	−113·7 (4)	N(2)–C(16)–C(17)–C(18)	−79·7 (4)
C(37)–C(9)–C(10)–C(11)	124·9 (4)	C(38)–C(16)–C(17)–C(18)	157·9 (4)
Cation (III)		Cation (IV)	
N(3)–C(23)–C(24)–C(25)	93·4 (5)	N(4)–C(30)–C(31)–C(32)	67·1 (4)
C(39)–C(23)–C(24)–C(25)	−28·6 (6)	C(40)–C(30)–C(31)–C(32)	−54·1 (6)

Table 2 (*cont.*)

Hydrogen-bonding geometry (distances in Å, angles in deg)*

D–H···*A*	H···*A*	*D*···*A*	*D*–H···*A*
O(3)–H(45)···O(12)	2·24	2·923 (6)	132
O(3)–H(45)···O(1)	2·05	2·637 (6)	121
O(4)–H(46)···O(2)	2·04	2·723 (5)	134
O(9)–H(47)···O(11)	2·01	2·680 (5)	134
O(10)–H(48)···O(1)	2·15	2·861 (6)	136
O(10)–H(48)···O(12)	2·07	2·621 (6)	118
O(13)–H(491)···O(5^{ii})	1·97	2·928 (8)	176
O(13)–H(492)···O(9)	1·99	2·891 (6)	157
O(14)–H(501)···O(6^{iii})	2·04	2·978 (7)	167
O(14)–H(502)···O(10)	1·98	2·888 (7)	161
N(1)–H(411)···O(8^{iv})	1·86	2·787 (6)	141
N(1)–H(412)···O(6)	1·74	2·817 (6)	174
N(1)–H(413)···O(12)	1·75	2·797 (5)	164
N(2)–H(421)···O(6^{iii})	1·93	2·852 (7)	141
N(2)–H(422)···O(1)	1·76	2·832 (5)	167
N(2)–H(423)···O(8)	1·76	2·831 (6)	174
N(3)–H(431)···O(2^{i})	1·74	2·775 (6)	159
N(3)–H(432)···O(7)	1·75	2·828 (6)	173
N(3)–H(433)···O(5^{ii})	2·26	3·016 (5)	125
N(4)–H(441)···O(7^{iv})	1·95	2·838 (5)	137
N(4)–H(442)···O(5^{i})	1·75	2·811 (6)	168
N(4)–H(443)···O(11)	1·69	2·760 (6)	171

Symmetry code: (i) $x, y+1, z$; (ii) $x+1, y+1, z$; (iii) $x+1, y, z$; (iv) $x-1, y, z$.

* For H-bond geometries, involving H atoms, no e.s.d.'s can be calculated because of the refinement procedure followed (rigid body; riding model); in view of the e.s.d.'s of the non-H atoms the e.s.d.'s for the H···*A* distances can be valued at 0·05 Å, those for the *D*–H···*A* angles at 3°.

The base molecules (I) and (II) are also related by the pseudocentre as are molecules (III) and (IV). However, torsion angles about the C(α)–C(Ph) bond in (I) and (IV) are approximately equal as is the case for (II) and (III). The occurrence of only one antipode of the base makes the centre of symmetry only a partial one, and the dissymmetric stacking of the methyl groups prevents the pseudocentre from being operative on the water molecules.

The structure is characterized by a strong hydrogen-bonded network; details are given in Table 2. Two H atoms bonded to C(α) atoms and two C(Ph) H atoms are in the proximity (~2·75 Å) of each independent water molecule. A view of the structure along **b** is given in Fig. 2, which shows the layers of *meso*-tartrate anions, sandwiched between layers containing the cations and the water molecules.

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Structures of Two Crystalline Modifications of Bis(triphenylphosphoranediyl)ammonium 3-Isothiocyanato-4,7; 4,8; 5,6-tri-μ-hydro-3,4,5,5,6,7,8,9,10,11-decahydro-1,2-didebor[C_{2v}-($1v^6 2v^4 422$)-Δ^{18}-*closo*]undecaborate(1−) Dichloromethane Solvate (2/1),* $C_{36}H_{30}NP_2^+.B_9H_{13}(NCS)^-.0{\cdot}5CH_2Cl_2$

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Abstract. $M_r = 749{\cdot}5$, $\bar{\lambda}(\text{Mo}\,K\alpha) = 0{\cdot}71069$ Å. α-Form: monoclinic, $P2_1/a$, $a = 15{\cdot}405\,(3)$, $b = 16{\cdot}129\,(2)$, $c = 17{\cdot}811\,(1)$ Å, $\beta = 108{\cdot}523\,(12)°$, $U = 4196$ Å^3, $Z = 4$, $D_x = 1{\cdot}186$ Mg m^{-3}, $\mu = 0{\cdot}207$ mm^{-1}, $F(000) = 1564$, $T = 295$ K, final $R = 0{\cdot}0836$ for 3793 observed reflections. β-Form: triclinic, $P\bar{1}$, $a = 11{\cdot}385\,(7)$, $b = 13{\cdot}960\,(3)$, $c = 15{\cdot}228\,(4)$ Å, $\alpha = 65{\cdot}06\,(2)$, $\beta = 69{\cdot}73\,(3)$, $\gamma = 75{\cdot}52\,(3)°$, $U = 2043$ Å^3, $Z = 2$, $D_x = 1{\cdot}218$ Mg m^{-3}, $\mu = 0{\cdot}212$ mm^{-1}, $F(000) = 782$, $T = 185$ K, final $R = 0{\cdot}0690$ for 5137 observed reflections. The title compound exists in both monoclinic (α) and triclinic (β) modifications. The substituted borane anion has a 1,2-didebor-undecaborate architecture with the isothiocyanato ligand at the 3 position and μ-H functions in 4–7, 4–8 and 5–6 locations (as determined by a low-temperature study of the β form). Between the two crystal forms the cations are effectively superimposable, and are bent at nitrogen, P—N—P *ca* 137°.

Introduction. The structure of the $[B_9H_{13}(NCS)]^-$ ion, (1), has been inferred from high-field, variable-temperature NMR studies (Jacobsen, Morris & Reed, 1984); these suggested that the arrangement of borane H atoms is similar to that in $B_9H_{13}(NCMe)$, (2) (Wang, Simpson & Lipscomb, 1961) in which there are μ-4,7 and μ-5,6 H atoms, in contrast to that in the parent species $[B_9H_{14}]^-$, (3) (Greenwood, McGinnety & Owen, 1972) which has μ-3,6 and μ-3,7 H atoms. In view of our interest in the effects of bridging H atoms on the lengths of B—B connectivities in borane and substituted borane polyhedra, we have determined the structure of $[B_9H_{13}(NCS)]^-$ as its $[(Ph_3P)_2N]^+$ salt in the solid state. Initially the structure was determined from a room-temperature data set, but this study failed to locate all the borane H atoms. In repeating the experiment with a different crystal at low temperature we unexpectedly encountered a second crystalline modification. Results of both determinations are presented herein.

Experimental. Preparation and recrystallization from CH_2Cl_2/*n*-hexane (Jacobsen, Morris & Reed, 1984) yields the title compound as its dichloromethane solvate (2:1). Data are given for the α-form, with differences in respect of the β-form in curly brackets. Clear, colourless blocks, 0·03 × 0·02 × 0·04 {0·07 × 0·05 × 0·03} cm, mounted on glass fibre; preliminary Weissenberg photography; CAD-4 diffractometer; lattice parameters from 25 centred reflections, $13 < \theta < 14{\cdot}5°$ $\{12 < \theta < 16°\}$; graphite-monochromated Mo $K\alpha$; for data collection $\theta_{\max} = 25°$; ω–2θ scan in 96 steps; ω-scan width $0{\cdot}85° + 0{\cdot}35°\tan\theta$ $\{0{\cdot}8° + 0{\cdot}35°\tan\theta\}$; rapid prescan after which reflections with $I \geq \sigma(I)$ remeasured such that final net intensity had $I > 33\sigma(I)$ subject to a maximum measuring time of 60 s; no significant crystal decay or movement; 7292 {7186}

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* Nomenclature according to Casey, Evans & Powell (1983).

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