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An Antihistaminic Agent: Soventol Hydrochloride Monohydrate Ethanol Solvate

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Abstract. $C_{19}H_{24}N_2.HCl.H_2O.C_2H_5OH$, triclinic, $P\bar{1}$, $a = 7.317$ (4), $b = 8.214$ (4), $c = 18.778$ (8) Å, $\alpha = 85.76$ (2), $\beta = 80.03$ (2), $\gamma = 83.90$ (2)°, $V = 1104$ Å^3, $Z = 2$, $D_m = 1.157$, $D_x = 1.146$ g cm^{-3}, μ(Mo $K\alpha$) = 1·93 cm^{-1}. The crystals, grown from ethanol, are colourless prisms which become opaque when exposed to air. The ethanol and water molecules of crystallization are involved in hydrogen bonding with the chloride ion and the piperidine nitrogen. The two phenyl rings are twisted by 95·0° with respect to each other. The structure was solved by *MULTAN* and refined to an *R* of 5·4%.

Introduction. Intensity data from a single crystal, 0·35 × 0·35 × 0·20 mm, were recorded at room temperature on a Nonius CAD-4 computer-controlled diffractometer with graphite-monochromatized Mo radiation and the ω–2θ scan mode. Measurements of 3458 independent reflexions, including 969 with negative intensities, were made with $\theta < 24°$. All reflexions were included in the structure refinement, the unobserved (mentioned above) were given $I = 0$ and appeared in the refinement with $\Delta F = -F_c$. Two standard reflexions were measured regularly throughout the data collection and the intensity of both dropped by 22%. 15% of this decay was in the first half of data collection when the surface of the crystal became opaque. Once the crystal became coated, the intensities decayed at a much slower rate. Lp corrections were applied, but an absorption correction was ignored. Scattering factors computed from numerical Hartree–Fock wave functions (Cromer & Mann, 1968) were used for the non-hydrogen atoms; for H, the values given by Stewart, Davidson & Simpson (1965) were used. The structure was solved by direct methods with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974). The most probable *E* map contained all the 21 non-hydrogen atoms in the molecule as well as the Cl$^-$ ion and the two O atoms. The C peaks in the ethanol molecule, found in a subsequent difference Fourier map, appeared as elongated ellipsoids with the largest axis along the *x* direction (the ethanol molecule lies roughly in the *yz* plane). The C end of the ethanol molecule was refined in two positions, each position given an occupancy of 0·5. The H parameters were included in the final cycles. Full-matrix least-squares refinement was performed with *CRYLSQ* of XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Final atomic parameters are listed in Table 1 (the H parameters can be obtained from the author).†

Discussion. Fig. 1 is a drawing of the soventol molecule and shows the atomic numbering. The bond distances and angles are listed in Table 2. The mean C(sp^2)—H bond distance is 0·93 Å, compared with 0·97 Å for C(sp^3)—H. All individual C—H distances have a

Table 1. *Fractional coordinates* (×10^4) *for the non-hydrogen atoms*

Numbers in parentheses in this and subsequent tables are the estimated deviations of the last digits.

	x	*y*	*z*
Cl$^-$	1463 (2)	2196 (2)	3836 (1)
O(1)	8089 (6)	7255 (6)	4548 (3)
O(2)	1517 (7)	5991 (6)	3709 (3)
N(1)	6546 (5)	3640 (4)	2013 (2)
N(2)	7217 (5)	2290 (5)	4216 (2)
C(1)	6220 (6)	3200 (5)	2791 (2)
C(2)	7087 (7)	1486 (6)	2981 (3)
C(3)	6598 (7)	1044 (6)	3784 (3)
C(4)	6342 (7)	3957 (6)	4038 (3)
C(5)	6834 (7)	4439 (6)	3240 (3)
C(6)	6842 (8)	1853 (8)	5013 (3)
C(7)	5424 (6)	3098 (5)	1570 (2)
C(8)	5801 (7)	3401 (6)	813 (3)
C(9)	4695 (8)	2847 (7)	376 (3)
C(10)	3188 (7)	1983 (7)	653 (3)
C(11)	2796 (7)	1697 (6)	1392 (3)
C(12)	3862 (6)	2241 (5)	1843 (2)
C(13)	8415 (6)	4059 (6)	1687 (3)
C(14)	8557 (6)	5826 (5)	1426 (2)
C(15)	10287 (7)	6321 (6)	1108 (3)
C(16)	10482 (8)	7929 (7)	884 (3)
C(17)	8983 (8)	9082 (6)	967 (3)
C(18)	7265 (8)	8618 (6)	1276 (3)
C(19)	7056 (6)	7001 (6)	1508 (3)
C(20)	1815 (12)	6535 (10)	2960 (4)
C(21)	1439 (32)	8165 (29)	2835 (12)
C(22)	2614 (21)	8151 (18)	2896 (8)

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32555 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

standard deviation of about 0·05 Å. The piperidine ring has been found to assume the chair conformation with parameters similar to those in another methyl-substituted piperidine antihistaminic agent (Birknes, 1977). The structure of piperidine hydrochloride (Datta Gupta & Saha, 1970) has roughly the same parameters, with the exception of C(1)–C(2) and C(1)–C(5) for which Datta Gupta & Saha report a length of 1·569 Å. The molecules are held together by a three-dimensional network of hydrogen bonds which are roughly in two planes and lie around a centre of inversion (0·0, 0·5, 0·5). The two planes are connected through two $Cl^-\cdots O(1)$ hydrogen bonds. The

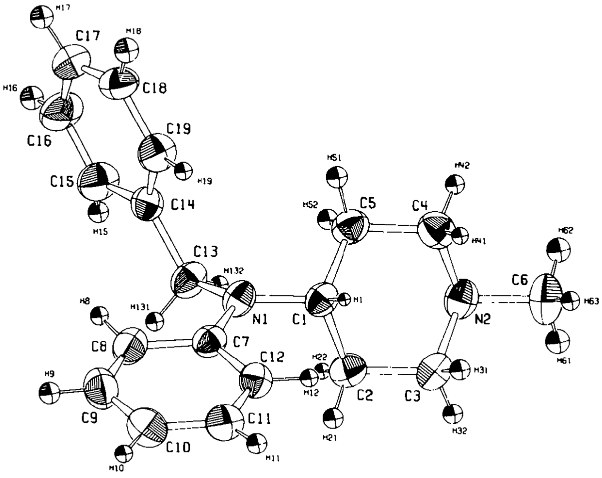

Fig. 1. Drawing of the soventol molecule showing the thermal-vibration ellipsoids (enclosing 40% probability) and the atomic numbering used in this paper. Figs. 1 and 2 were drawn with *ORTEP* (Johnson, 1970).

Table 2. *Bond distances* (Å) *and angles* (°) *in the soventol molecule*

C(1)–C(2)	1·525 (7)	C(1)–C(2)–C(3)	111·0 (4)
C(2)–C(3)	1·515 (7)	C(2)–C(3)–N(2)	110·4 (4)
N(2)–C(3)	1·503 (8)	C(3)–N(2)–C(4)	110·3 (4)
N(2)–C(4)	1·489 (7)	C(3)–N(2)–C(6)	112·1 (4)
C(4)–C(5)	1·513 (7)	C(4)–N(2)–C(6)	111·6 (4)
C(1)–C(5)	1·518 (8)	N(2)–C(4)–C(5)	111·5 (4)
N(2)–C(6)	1·497 (7)	C(1)–C(5)–C(4)	110·2 (4)
C(1)–N(1)	1·463 (6)	C(1)–N(1)–C(7)	119·9 (4)
N(1)–C(7)	1·391 (7)	C(1)–N(1)–C(13)	117·3 (4)
N(1)–C(13)	1·464 (7)	C(7)–N(1)–C(13)	118·5 (4)
C(7)–C(8)	1·409 (7)	N(1)–C(7)–C(8)	120·8 (4)
C(8)–C(9)	1·378 (9)	N(1)–C(7)–C(12)	122·8 (4)
C(9)–C(10)	1·380 (9)	C(8)–C(7)–C(12)	116·4 (4)
C(10)–C(11)	1·374 (8)	C(7)–C(8)–C(9)	120·6 (4)
C(11)–C(12)	1·374 (8)	C(8)–C(9)–C(10)	122·2 (5)
C(7)–C(12)	1·406 (8)	C(9)–C(10)–C(11)	117·6 (5)
C(13)–C(14)	1·505 (7)	C(10)–C(11)–C(12)	121·7 (5)
C(14)–C(15)	1·392 (8)	C(7)–C(12)–C(11)	121·5 (4)
C(15)–C(16)	1·371 (8)	N(1)–C(13)–C(14)	115·6 (4)
C(16)–C(17)	1·367 (9)	C(13)–C(14)–C(19)	123·0 (4)
C(17)–C(18)	1·371 (9)	C(13)–C(14)–C(15)	119·1 (4)
C(18)–C(19)	1·383 (7)	C(15)–C(14)–C(19)	117·9 (4)
C(14)–C(19)	1·379 (8)	C(14)–C(15)–C(16)	120·7 (4)
		C(15)–C(16)–C(17)	120·8 (5)
N(1)–C(1)–C(2)	113·1 (3)	C(16)–C(17)–C(18)	119·4 (5)
N(1)–C(1)–C(5)	113·4 (4)	C(17)–C(18)–C(19)	120·3 (5)
C(2)–C(1)–C(5)	109·9 (4)	C(14)–C(19)–C(18)	120·9 (4)

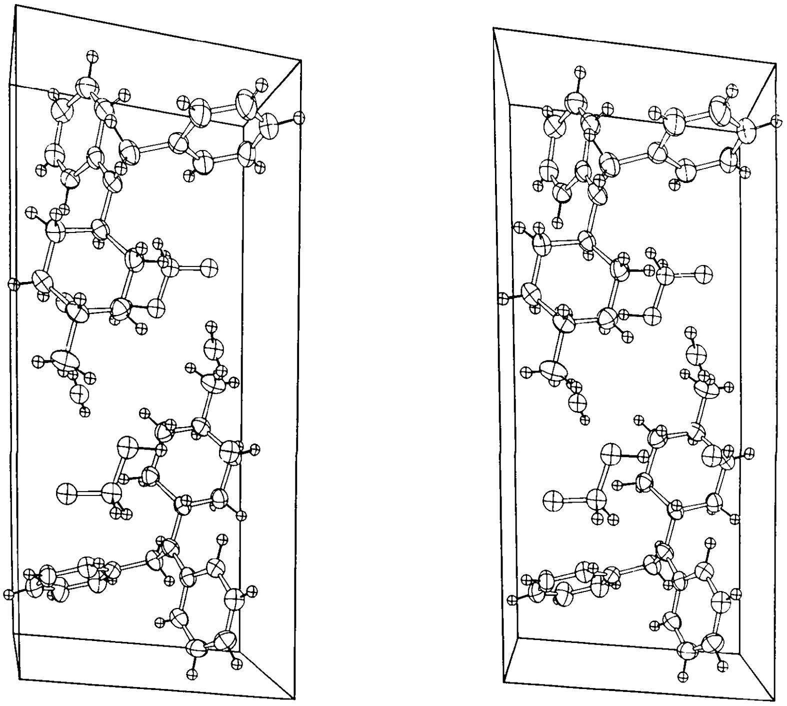

Fig. 2. Stereo-drawing showing the packing of the molecules in the unit cell. The *a* axis is pointing away from the viewer, the *b* axis runs right to left, and the *c* axis bottom to top.

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

D–H···A	Position of acceptor atom	D···A	H···A	∠D–H···A
O(1)–H(101)···Cl⁻	$\bar{x}+1, \bar{y}+1, \bar{z}+1$	3·177 (6)	2·44 (9)	159 (9)
O(1)–H(102)···O(2)	$x+1, y, z$	2·859 (12)	1·88 (9)	171 (8)
O(2)–H(3)···Cl⁻	x,y,z	3·113 (5)	2·10 (7)	177 (5)
N(2)–H(2)···Cl⁻	$x+1, y, z$	3·060 (6)	2·15 (5)	174 (5)

hydrogen-bond distances (Table 3) are similar to those found in cyproheptadine hydrochloride (Birknes, 1977). The N(2)···Cl⁻ distance of 3·060(6) Å is comparable with the 3·088 Å found by Datta Gupta & Saha (1970) in the structure of piperidine hydrochloride. The large thermal parameters associated with the ethanol atoms and the high σ values for the bond lengths and valence angles indicate that the ethanol molecule is not rigidly fixed in its cavity. The crystal packing of the unit cell is shown in Fig. 2. The H atoms on the methyl end of the ethanol did not show up in the difference map and they are omitted from Fig. 2.

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{(η-Cyclohexadiene)(η-cyclopentadiene)[1,2-bis(diphenylphosphino)ethane]-molybdenum(V)} Hexafluorophosphate, Sulphur Dioxide

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Abstract. $\{(\eta\text{-}C_5H_5)(\eta\text{-}C_6H_8)Mo[(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2]\}PF_6,SO_2$, $M_r = 848{\cdot}6$, monoclinic, $P2_1/c$, $a = 10{\cdot}880$ (7), $b = 15{\cdot}205$ (8), $c = 22{\cdot}36$ (1) Å, $\beta = 97{\cdot}5$ (1)°, $V = 3666{\cdot}8$ Å^3, $D_c = 1{\cdot}54$ g cm^{-3}, $Z = 4$. Patterson and Fourier methods were used to determine the structure from four-circle diffractometer intensity measurements. The final R was 0·061. The geometry of the cation is similar to that in bent bis(cyclopentadienyls). The SO_2 molecules occupy holes in the structure.

Introduction. Crystals were provided by Dr J. Segal and Dr M. L. H. Green (Segal, Green, Daran & Prout, 1976). The compounds are sensitive to air, and were mounted under dry nitrogen in glass capillaries.

The crystal system was determined from precession photographs (Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å). Systematic extinctions $h0l$, $l = 2n + 1$, $0k0$, $k = 2n + 1$ indicated space group $P2_1/c$. A crystal (0·3 × 0·3 × 0·3 mm) was set up on a Hilger & Watts PDP8-controlled four-circle diffractometer and cell dimensions and orientation matrix were obtained by a least-squares fit to the setting angles of 17 reflexions.

The intensities of each independent reflexion with $\sin\theta/\lambda < 0{\cdot}59$ were measured by an $\omega/2\theta$ scan with ordinate analysis (Watson, Shotton, Cox & Muirhead, 1970). Mo $K\alpha$ radiation from a graphite monochromator was used. 3513 independent reflexions with $I > 4\sigma$, where σ is the standard deviation based on simple counting statistics, were used in subsequent