

mutually displaced by 0.36 Å. In keeping with the relatively short C=O bond, the N...O distance is 0.10–0.15 Å longer than the corresponding hydrogen-bonded distance in the other lactam structures mentioned in Table 3.

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Structures of Salsoline Hydrochloride Hydrate and Salsolidine Hydrochloride Dihydrate

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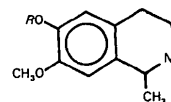
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Abstract. C₁₁H₁₆NO₂⁺.Cl⁻.H₂O: *M_r* = 247.72, orthorhombic, *P*2₁2₁2₁, *a* = 7.856 (2), *b* = 23.246 (6), *c* = 6.854 (2) Å, *V* = 1252 (1) Å³, *D_x* = 1.314 Mg m⁻³ for *Z* = 4, *μ* = 2.67 mm⁻¹, *F*(000) = 528, diffractometer data λ(Cu Kα) = 1.5418 Å, *T* = 293 K, 1185 reflections, 1086 with *I* > 3σ(*I*), *R* = 0.042. C₁₂H₁₈NO₂⁺.Cl⁻.2H₂O: *M_r* = 279.76, orthorhombic, *P*2₁2₁2₁, *a* = 7.293 (3), *b* = 12.605 (7), *c* = 15.708 (8) Å, *V* = 1444 (2) Å³, *D_x* = 1.287 Mg m⁻³ for *Z* = 4, *μ* = 2.42 mm⁻¹, *F*(000) = 600, 1373 reflections, 1251 with *I* > 3σ(*I*), *R* = 0.053. There is extensive hydrogen bonding involving the phenolic OH, methoxy oxygens, water molecules, ammonium NH₂⁺s and chloride anions.

Introduction. Salsoline [1,2,3,4-tetrahydro-7-methoxy-1-methylisoquinolin-6-ol (I)] and its methyl ether

salsolidine [1,2,3,4-tetrahydro-6,7-dimethoxy-1-methylisoquinoline (II)] are found in the upper part of the west Mediterranean plant *Salsola kali* L. and *S. longifolia* Forsk (Chenopodiaceae). They have been isolated also from some European species of the genus *Salsola* (Orechoff & Proskurnina, 1934; Khalimatov & Rustamov, 1963; Rizaev, Rustamov, Muslimov & Khalimatov, 1967). These alkaloids have been found effective in the treatment of hypertension by reducing blood pressure and stimulating respiration (Gvishiani, 1939; Wastl, 1946; Krylov, Nauchn & Ryzausk, 1962). The crystal structures of the hydrochlorides of salsoline (III) and salsolidine (IV) are reported. Both crystal forms are hydrated.



- (I): *R* = H
 (II): *R* = CH₃
 (III): (I) + HCl + H₂O
 (IV): (II) + HCl + 2H₂O

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Experimental. Cell parameters and intensity measurements made with a Picker FACS-I diffractometer, Cu K α radiation, graphite monochromator. Salsoline hydrochloride hydrate (III): needles from methanol, 0.35 \times 0.40 \times 0.15 mm crystal, cell parameters from 14 reflections centered at $\pm 2\theta$, 2θ - θ scan, 2θ scan speed 2° min⁻¹, 2θ scan width 1.50 + 0.3 tan θ , 10 s backgrounds, h, k, l range of 0-9, 0-26, 0-7; four standards every 100 reflections, average and maximum intensity deviations of 0.4%, 1.0%, 1185 reflections measured to $2\theta_{\max} = 126^\circ$, 1185 unique data, 1086 $I > 3\sigma(I)$; $8 \times 8 \times 8$ Gaussian quadrature absorption correction, $A_{\min}^* = 1.427$, $A_{\max}^* = 2.400$; structure solved and refined with XTAL program package (Stewart & Hall, 1983); ring hydrogen atom positions calculated, other (except hydrogen atoms of the water molecule which remained undetermined) located from difference map. Salsolidine hydrochloride dihydrate (IV): needles from nitromethane, 0.25 \times 0.20 \times 0.20 mm crystal; cell parameters from ten reflections centered at $\pm 2\theta$; 2θ - θ scan, 2θ scan speed 2° min⁻¹, 2θ scan width 1.65 + 0.3 tan θ , 10 s backgrounds; h, k, l range of 0-8, 0-14, 0-8; four standards every 100 reflections, average and maximum intensity deviations of 0.4% and 1.4%; 1373 reflections measured to $2\theta_{\max} = 126^\circ$, 1278 unique data, 1160 $I > 3\sigma(I)$; structure solved with MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); ring hydrogen atom positions calculated, others (except hydrogen atoms of the water molecules, which remained undetermined) located from a difference map.

Structure refinement by full-matrix least squares, $\sum w(F_o - F_c)^2$ minimized with $w = [1/\sigma(F)]^2$, reflections with $I_c < 3\sigma(I)$ omitted; anisotropic temperature factors for C, O, N and Cl, hydrogen atoms refined with isotropic terms [coordinates not varied in (IV)]; average and maximum shift/e.s.d. in final least-squares cycle of (III) 0.02 and 0.26, (IV) 0.12 and 0.25; minimum and maximum electron density in final difference map of (III) -0.19 and 0.27 e \AA^{-3} , (IV) -0.27 and 0.26 e \AA^{-3} ; final R , wR and S for (III) 0.042, 0.042, 0.77 and for (IV) 0.053, 0.064, 3.72. C, N, O, Cl form factors from Cromer & Mann (1968), H from Stewart, Davidson & Simpson (1965). All calculations were performed on a UNIVAC 1100/82 computer with the XTAL system (Stewart & Hall, 1983) and XRAY76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) of crystallographic programs. Atomic coordinates and thermal parameters are listed in Tables 1 and 2† for (III) and (IV) respectively.

† Lists of structure factors, anisotropic thermal parameters, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43433 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. ORTEP drawings (Johnson, 1976) for structures (III) and (IV) are shown in Figs. 1 and 2. Bond lengths, angles and the torsion angles for the nitrogen-containing ring are given in Table 3 for (III) and in Table 4 for (IV). The average values of the bond lengths in the benzene ring are 1.387 (7) \AA in (III) and 1.385 (8) \AA in (IV). These values are in fair agreement with the C-C distance (1.392 \AA) in benzene (Cox, Cruickshank & Smith, 1958). The average values of bond lengths and bond angles in the heterocyclic ring are similar at 1.496 (8), 1.496 (8) \AA and 115.4 (5), 114.0 (5)° in (III) and (IV), though the individual values of distances and angles vary to some extent. These average values agree well with the values reported for similar types of compounds (Pavkovic, Glowinski, Feng & Brown, 1981; Brennan, Garafalo & Williams, 1979). The maximum and average deviations

Table 1. Fractional coordinates, equivalent isotropic temperature factors (\AA^2) and e.s.d.'s (in parentheses) for (III)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	0.0541 (6)	-0.1368 (2)	0.1035 (8)	0.045 (3)
C(3)	0.2265 (7)	-0.1757 (2)	-0.1761 (9)	0.065 (3)
C(4)	0.1712 (8)	-0.1277 (2)	-0.3133 (9)	0.065 (3)
C(4a)	0.1051 (6)	-0.0755 (2)	-0.2007 (8)	0.045 (3)
C(5)	0.1017 (6)	-0.0219 (2)	-0.2924 (8)	0.052 (3)
C(6)	0.0419 (6)	0.0257 (2)	-0.1988 (7)	0.046 (3)
C(7)	-0.0201 (6)	0.0204 (2)	-0.0074 (7)	0.043 (3)
C(8)	-0.0167 (7)	-0.0320 (2)	0.0836 (8)	0.046 (3)
C(8a)	0.0456 (6)	-0.0806 (2)	-0.0099 (8)	0.039 (3)
C(9)	-0.1062 (7)	-0.1511 (2)	0.2223 (9)	0.069 (4)
C(10)	-0.1287 (9)	0.0695 (3)	0.2718 (11)	0.081 (4)
O(1)	0.0460 (5)	0.0783 (1)	-0.2910 (5)	0.062 (2)
O(2)	-0.0795 (5)	0.0710 (1)	0.0719 (5)	0.062 (2)
O(3)	-0.0166 (5)	0.1849 (1)	-0.1338 (6)	0.076 (3)
N	0.0862 (6)	-0.1867 (2)	-0.0318 (7)	0.055 (3)
Cl	0.2393 (2)	-0.27556 (5)	0.2593 (2)	0.055 (1)

Table 2. Fractional coordinates, equivalent isotropic temperature factors (\AA^2) and e.s.d.'s (in parentheses) for (IV)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	0.0573 (9)	0.5050 (4)	-0.0921 (4)	0.04 (1)
C(3)	0.006 (1)	0.4004 (5)	0.0400 (4)	0.07 (2)
C(4)	0.079 (1)	0.4882 (5)	0.0975 (4)	0.07 (1)
C(4a)	0.0855 (9)	0.5959 (4)	0.0528 (4)	0.05 (2)
C(5)	0.100 (1)	0.6865 (4)	0.1010 (4)	0.05 (1)
C(6)	0.1081 (9)	0.7844 (4)	0.0642 (3)	0.04 (2)
C(7)	0.1001 (9)	0.7921 (4)	-0.0258 (3)	0.04 (1)
C(8)	0.0869 (9)	0.7030 (4)	-0.0740 (4)	0.04 (1)
C(8a)	0.0794 (9)	0.6016 (4)	-0.0358 (3)	0.03 (1)
C(9)	0.178 (1)	0.5055 (5)	-0.1728 (4)	0.054 (7)
C(10)	0.100 (1)	0.9058 (5)	-0.1477 (4)	0.06 (2)
C(11)	0.143 (1)	0.8718 (5)	0.1969 (4)	0.06 (2)
O(1)	0.1181 (7)	0.8788 (3)	0.1059 (2)	0.04 (1)
O(2)	0.1052 (7)	0.8936 (3)	-0.0570 (2)	0.05 (1)
O(3)	-0.0036 (7)	1.0884 (3)	0.0196 (3)	0.055 (8)
O(4)	0.1105 (9)	1.1630 (4)	0.1847 (3)	0.090 (9)
N	0.1096 (8)	0.4063 (4)	-0.0426 (3)	0.045 (4)
Cl	-0.0117 (3)	0.2116 (1)	-0.1498 (1)	0.055 (9)

for the atoms of the benzene ring [C(4a), C(5), C(6), C(7), C(8) and C(8a)] from their least-squares plane are 0.007 (4) [for C(6)], 0.003 (4) Å in (III) and 0.004 (4) [for C(7)], 0.003 (4) Å in (IV). As expected, the two heterocyclic ring atoms [C(1), C(4)] linked to the benzene ring are close to the benzene plane, with deviations of 0.082 (4), -0.033 (4) Å in (III) and 0.046 (3), -0.007 (3) Å in (IV). The heterocyclic ring atoms N and C(3) are displaced above and below this plane with deviations of -0.230 (4), 0.898 (4) Å in (III) and -0.382 (3), 0.418 (3) Å in (IV). It should be noted that the more symmetrical displacement in (IV) occurs in the molecule with the more symmetrical pattern of methoxy substitution. The heterocyclic ring conformations in the two structures are similar and the torsion angles for the N-C(3) bond are -66.1 (6)° for (III) and -69.6 (6)° for (IV). The exocyclic angles at C(6) and C(7) of the benzene rings show the effects of the methyl substituents on oxygen. The methoxy groups are approximately coplanar with the benzene nuclei, and ring...methyl nonbonded contacts account for about a 10° difference in exocyclic angles at C(7) in (III) and C(6) and C(7) in (IV). In (III) the C(6) substituent is OH, and here the exocyclic angles differ by only 1°.

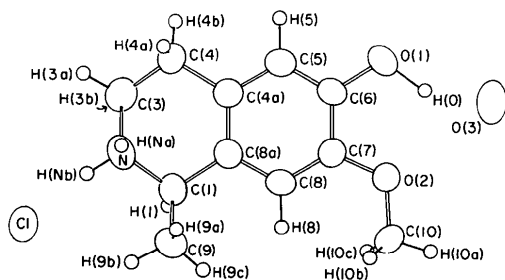


Fig. 1. An ORTEP drawing of (III) with the Cl, C, N and O atoms depicted as 50% probability boundary ellipses. H atoms are shown as 0.1 Å radius circles.

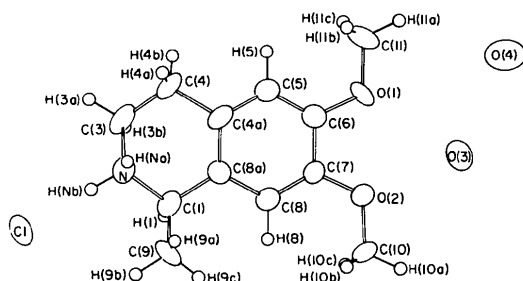


Fig. 2. An ORTEP drawing of (IV) with the Cl, C, N and O atoms depicted as 50% probability boundary ellipses. H atoms are shown as 0.1 Å radius circles.

ORTEP drawings showing the packing of the molecules are given in Figs. 3 and 4. In both structures, the positions of the hydrogen atoms in the water molecules could not be found in difference maps. However, on the basis of nonbonded contacts, the following schemes of hydrogen bonding have been deduced.

In structure (III), the single water molecule appears to participate in three H-bonding interactions. It serves as a donor to two chloride anions, and as an acceptor for the hydroxyl [O(1)-H]. The intermolecular contacts to the O(3) water molecule are O(3)-H...Cl (at $\frac{1}{2}-x, -y, -\frac{1}{2}+z$) = 3.119 (4), O(3)-H...Cl (at $-x,$

Table 3. Bond lengths (Å), angles (°), torsion angles (°) and *e.s.d.*'s (in parentheses) for (III)

C(1)-C(8a)	1.523 (7)	C(5)-C(6)	1.363 (7)
C(1)-C(9)	1.536 (8)	C(6)-C(7)	1.405 (7)
C(1)-N	1.506 (7)	C(6)-O(1)	1.377 (6)
C(3)-C(4)	1.521 (8)	C(7)-C(8)	1.369 (7)
C(3)-N	1.502 (8)	C(7)-O(2)	1.376 (6)
C(4)-C(4a)	1.530 (7)	C(8)-C(8a)	1.387 (6)
C(4a)-C(8a)	1.394 (7)	C(10)-O(2)	1.424 (8)
C(5)-C(4a)	1.395 (7)		
C(8a)-C(1)-C(9)	114.8 (4)	C(7)-C(6)-O(1)	120.9 (4)
C(8a)-C(1)-N	110.7 (4)	C(6)-C(7)-C(8)	119.7 (4)
C(9)-C(1)-N	107.3 (4)	C(6)-C(7)-O(2)	114.3 (4)
C(4)-C(3)-N	108.8 (4)	C(8)-C(7)-O(2)	125.9 (5)
C(3)-C(4)-C(4a)	111.5 (5)	C(7)-C(8)-C(8a)	121.3 (5)
C(4)-C(4a)-C(5)	119.2 (5)	C(1)-C(8a)-C(4a)	122.5 (4)
C(4)-C(4a)-C(8a)	121.3 (4)	C(1)-C(8a)-C(8)	118.5 (4)
C(5)-C(4a)-C(8a)	119.5 (4)	C(8)-C(8a)-C(4a)	118.9 (4)
C(6)-C(5)-C(4a)	121.3 (5)	C(7)-O(2)-C(10)	116.8 (4)
C(5)-C(6)-C(7)	119.3 (4)	C(1)-N-C(3)	113.4 (4)
C(5)-C(6)-O(1)	119.8 (4)		
C(1)-N-C(3)-C(4)	-66.1 (6)	C(1)-C(8a)-C(4a)-C(4)	4.5 (7)
C(3)-C(4)-C(4a)-C(8a)	-22.0 (7)	C(3)-N-C(1)-C(8a)	47.4 (6)
N-C(1)-C(8a)-C(4a)	-15.6 (6)	N-C(3)-C(4)-C(4a)	49.0 (6)

Table 4. Bond lengths (Å), angles (°), torsion angles (°) and *e.s.d.*'s (in parentheses) for (IV)

C(1)-C(8a)	1.513 (8)	C(5)-C(6)	1.364 (8)
C(1)-C(9)	1.543 (9)	C(6)-C(7)	1.418 (8)
C(1)-N	1.517 (8)	C(6)-O(1)	1.361 (7)
C(3)-C(4)	1.522 (9)	C(7)-C(8)	1.358 (8)
C(3)-N	1.503 (9)	C(7)-O(2)	1.371 (6)
C(4)-C(4a)	1.529 (8)	C(8)-C(8a)	1.413 (8)
C(4a)-C(8a)	1.394 (8)	C(10)-O(2)	1.433 (7)
C(5)-C(4a)	1.375 (8)	C(11)-O(1)	1.443 (8)
C(8a)-C(1)-C(9)	114.6 (5)	C(7)-C(6)-O(1)	115.0 (5)
C(8a)-C(1)-N	109.5 (5)	C(6)-C(7)-C(8)	120.1 (5)
C(9)-C(1)-N	106.3 (5)	C(6)-C(7)-O(2)	114.8 (5)
C(4)-C(3)-N	107.7 (6)	C(8)-C(7)-O(2)	125.1 (5)
C(3)-C(4)-C(4a)	112.6 (5)	C(7)-C(8)-C(8a)	120.9 (5)
C(4)-C(4a)-C(5)	119.1 (5)	C(1)-C(8a)-C(4a)	123.0 (5)
C(4)-C(4a)-C(8a)	120.2 (5)	C(1)-C(8a)-C(8)	118.9 (5)
C(5)-C(4a)-C(8a)	120.6 (5)	C(8)-C(8a)-C(4a)	118.0 (5)
C(6)-C(5)-C(4a)	121.4 (5)	C(6)-O(1)-C(11)	115.5 (5)
C(5)-C(6)-C(7)	118.9 (5)	C(7)-O(2)-C(10)	117.0 (4)
C(5)-C(6)-O(1)	126.1 (5)	C(1)-N-C(3)	110.9 (5)
C(1)-N-C(3)-C(4)	-69.6 (6)	C(1)-C(8a)-C(4a)-C(4)	3.0 (9)
C(3)-C(4)-C(4a)-C(8a)	-18.3 (9)	C(3)-N-C(1)-C(8a)	53.0 (7)
N-C(1)-C(8a)-C(4a)	-19.6 (8)	N-C(3)-C(4)-C(4a)	49.6 (7)

$\frac{1}{2}+y$, $\frac{1}{2}-z$) = 3.240 (5) Å; Cl—O(3)—Cl angle 106.1 (4)°; O(1)—H...O(3) = 2.746 (5) and O(1)—H...O(3) = 1.88 (4) Å (the underline indicates the atom used in the calculation of the given distance). As one might expect, both of the hydrogens on nitrogen are involved in strong nonbonded interactions with chloride. The distances are N—H(a)...Cl = 3.260 (5), N—H(a)...Cl (at $-\frac{1}{2}+x$, $-\frac{1}{2}-y$, $-z$) = 2.30 (5), N—H(b)...Cl = 3.114 (5) and N—H(b)...Cl = 2.20 (5) Å. The intermolecular interactions with chloride have a distorted tetrahedral geometry, the angles varying from 73.2 (5) to 138.9 (5)° with an average of 105.6°. Hydrogen-bonding interactions through water and chloride provide a means of tying together the tetrahydroquinolinium cations at both the ammonium and hydroxyl ends (Fig. 3).

Structure (IV), with two water molecules and no phenolic hydroxyl, has a different intermolecular hydrogen-bonding pattern from (III). Water O(3) presumably serves as a bifurcated H-bond donor to the two methoxy oxygen atoms O(1) and O(2), a donor to chloride, and acceptors from nitrogen and water O(4).

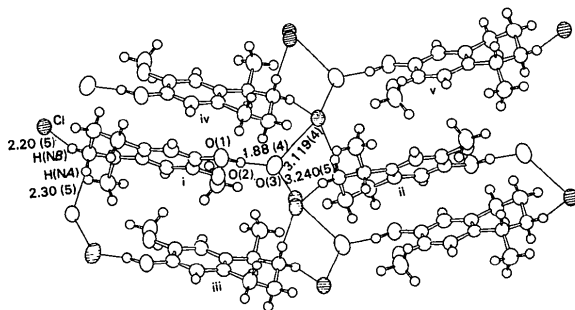


Fig. 3. Packing view of (III) down *c* rotated 3° around *b*. (i) x, y, z ; (ii) $-x, \frac{1}{2}+y, -\frac{1}{2}-z$; (iii) $\frac{1}{2}-x, -y, -\frac{1}{2}+z$; (iv) $\frac{1}{2}-x, -y, -\frac{1}{2}+z$; (v) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.

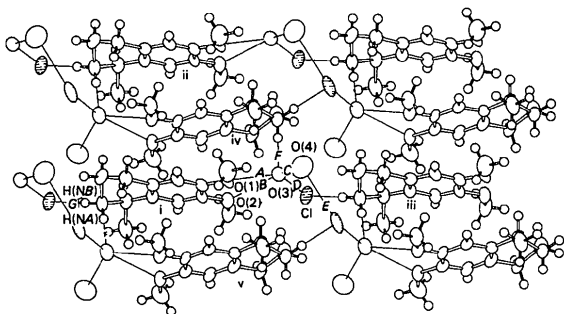


Fig. 4. Packing view of (IV) down *c* rotated 20° around *a*. Contacts: A = 3.099 (6), B = 2.847 (6), C = 2.882 (7), D = 3.082 (5), E = 3.223 (7), F = 1.85 and G = 2.11 Å. (i) x, y, z ; (ii) $-1+x, y, z$; (iii) $x, 1+y, z$; (iv) $-\frac{1}{2}+x, \frac{3}{2}-y, -z$; (v) $\frac{1}{2}+x, \frac{3}{2}-y, -z$.

The distances involving water O(3) are O(3)—H...O(1) = 3.099 (6), O(3)—H...O(2) = 2.847 (6), O(3)—H...Cl (at $x, 1+y, z$) = 3.082 (5), O(3)...H(b)—N (at $-\frac{1}{2}+x, \frac{3}{2}-y, -z$) = 2.844 (8), O(3)...H(b)—N = 1.85 Å and O(3)...H—O(4) = 2.882 (7) Å. In addition to this last contact, the O(4) water also serves as a donor to chloride with O(4)—H...Cl (at $\frac{1}{2}+x, \frac{3}{2}-y, -z$) = 3.223 (7) Å. The O(3)—O(4)—Cl (at $\frac{1}{2}+x, \frac{3}{2}-y, -z$) angle is 104.7 (5)°. If one further calls the mid-point of O(1) and O(2) O₁₂, the O₁₂—O(3)—Cl (at $x, 1+y, z$) angle is 120.6 (5)°. O₁₂, O(4), N (at $-\frac{1}{2}+x, \frac{3}{2}-y, -z$) and Cl (at $x, 1+y, z$) form a distorted tetrahedron around O(3), the six angles at O(3) varying from 94.5 (5) to 120.6 (5)° with an average of 104.9 (5)°. Unlike the NH₂ moiety in (III), only one of the two nitrogen hydrogens in (IV) is linked to a chloride, with N—H(a)...Cl = 3.104 (5) and N—H(a)...Cl = 2.11 Å; the other ammonium hydrogen atom, H(b), is a donor to water O(3) (see above). Water O(3) serves as a hydrogen-bonding bridge between N⁺ and Cl⁻ (Fig. 4).

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