Fig. 1. View of the title complex with 50% thermal ellipsoids (Johnson, 1965).

Table 3. *Selected bond lengths* (Å) *and angles* (°)

Co—Cl(1)	2·2134 (5)	N(1)—C(1)	1·347 (2)
Co—Cl(2)	2·2144 (5)	N(1)—C(5)	1·351 (2)
Co—N(1)	2·042 (1)	N(2)—C(6)	1·351 (2)
Co—N(2)	2·039 (1)	N(2)—C(10)	1·348 (2)
Cl(1)—Co—Cl(2)	110·89 (2)	Co—N(1)—C(1)	126·8 (1)
Cl(1)—Co—N(1)	118·07 (4)	Co—N(1)—C(5)	113·2 (1)
Cl(1)—Co—N(2)	118·72 (4)	C(1)—N(1)—C(5)	119·9 (2)
Cl(2)—Co—N(1)	112·24 (4)	Co—N(2)—C(6)	113·7 (1)
Cl(2)—Co—N(2)	112·78 (4)	Co—N(2)—C(10)	126·6 (1)
N(1)—Co—N(2)	81·28 (6)	C(6)—N(2)—C(10)	119·7 (2)

Fronczek, Gupta, Puckett, Pantaleo & Kiefer (1982); structure of $[(C_{12}H_{12}N_2)_2Cu(I)]BF_4$: Burke, McMillin & Robinson (1980).

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lengths and bond angles are shown in Table 3.* Fig. 1 shows the molecule and the atomic numbering scheme.

Related literature. Preparation of Pd^{II}, Pt^{II}, Cu^{II} and Zn^{II} analogues: Newkome, Pantaleo, Puckett, Ziefle & Deutsch (1981); structure of Pd analogue: Newkome,

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths, bond angles, torsion angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51040 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Hygric Acid (I) and Stachydrine (II) as Their Hydrochlorides

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Abstract. (I) 1-Methyl-L-proline hydrochloride, $C_6H_{12}NO_2^+ \cdot Cl^-$, $M_r = 165 \cdot 6$, orthorhombic, $P2_12_12_1$, $a = 6 \cdot 717$ (3), $b = 10 \cdot 397$ (1), $c = 12 \cdot 140$ (1) Å, $V = 848$ (2) Å³, $Z = 4$, $D_m = 1 \cdot 28$, $D_x = 1 \cdot 297$ Mg m⁻³, Mo $K\bar{\alpha}$ radiation, $\lambda = 0 \cdot 7107$ Å, $\mu = 0 \cdot 346$ mm⁻¹, $F(000) = 352$, $T = 293$ (2) K, $R = 0 \cdot 061$ for 750 observed reflections. (II) 2-Carboxy-1,1-dimethyl-pyrrolidinium chloride, $C_7H_{14}NO_2^+ \cdot Cl^-$, $M_r = 179 \cdot 6$, orthorhombic, $P2_12_12_1$, $a = 6 \cdot 561$ (2), $b = 11 \cdot 671$ (6), $c = 11 \cdot 690$ (4) Å, $V = 895$ (2) Å³, $Z = 4$, $D_m = 1 \cdot 31$, $D_x = 1 \cdot 333$ Mg m⁻³, Mo $K\bar{\alpha}$ radiation, $\lambda = 0 \cdot 7107$ Å, $\mu = 0 \cdot 331$ mm⁻¹, $F(000) = 384$, $T = 293$ (2) K, $R = 0 \cdot 031$ for 1239 observed reflections. In the *N*-methylated and *N,N'*-dimethylated proline derivatives (I) and (II) the N atom lies out of the plane of the pyrrolidine ring, to the same side of the molecule as the

Table 1. *Fractional atomic coordinates and B_{eq} values (Å^2) for (I)*

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
Cl	−0·0295 (3)	0·4264 (2)	0·0657 (2)	3·92
O(1)	−0·6643 (9)	−0·2923 (5)	0·0093 (4)	4·51
O(2)	−0·6306 (12)	−0·1847 (7)	−0·1494 (6)	5·21
N(1)	−0·6956 (10)	−0·5238 (6)	−0·1040 (5)	3·24
C(2)	−0·6941 (13)	−0·3993 (7)	−0·1648 (6)	3·48
C(3)	−0·8941 (18)	−0·4021 (10)	−0·2223 (10)	5·19
C(4)	−0·9420 (22)	−0·5417 (11)	−0·2420 (11)	6·24
C(5)	−0·7900 (17)	−0·6172 (9)	−0·1808 (9)	4·82
C(6)	−0·6608 (11)	−0·2866 (7)	−0·0901 (6)	3·14
C(7)	−0·4913 (12)	−0·5658 (9)	−0·0641 (8)	5·63

Table 2. *Fractional atomic coordinates and B_{eq} values (Å^2) for (II)*

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
Cl	0·0884 (1)	0·1948 (1)	0·3186 (1)	3·07
O(1)	0·3124 (3)	0·1675 (2)	0·0264 (2)	4·39
O(2)	0·0297 (3)	0·0887 (2)	0·0955 (2)	4·14
N(1)	−0·0804 (3)	−0·0070 (1)	−0·1182 (2)	2·38
C(2)	0·1179 (3)	0·0575 (2)	−0·1009 (2)	2·45
C(3)	0·1233 (4)	0·1420 (2)	−0·2004 (2)	3·55
C(4)	0·0081 (5)	0·0816 (2)	−0·2972 (2)	3·69
C(5)	−0·0705 (5)	−0·0298 (2)	−0·2456 (2)	3·12
C(6)	0·1596 (3)	0·1112 (2)	0·0141 (2)	2·82
C(7)	−0·0869 (5)	−0·1178 (2)	−0·0523 (2)	3·27
C(8)	−0·2689 (4)	0·0624 (2)	−0·0926 (3)	3·24

carboxyl group, by 0·564 (6) and 0·671 (2) Å respectively. The closest interionic contact in the crystal lattice for (I) is the N—H···Cl interaction of 2·11 (4) Å whereas in (II) O—H···Cl of 1·94 (2) Å is found. The presence of the second methyl group in (II) has the result that the dihedral angle between the plane of the pyrrolidine ring and that through the carboxyl group has increased to 136·3 compared with 119·5° in (I).

Experimental. The isolation of (I) from *Melaleuca* genus will be reported elsewhere (Jones, Naidu, Paleg & Tiekink, 1988). (II) was prepared by the literature method (Cornforth & Henry, 1952). The compounds were crystallized as their hydrochlorides from CH_3-$OH/(C_2H_5)_2O$ solution; m.p. 437 for (I) and 499 K for (II). D_m by flotation. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω–2θ scan technique. Cell parameters on crystal 0·10 × 0·65 × 0·65 mm for (I) and 0·15 × 0·30 × 1·00 mm for (II) from least-squares procedure (de Boer & Duisenberg, 1984) on 25 reflections ($3 \le \theta \le 12°$; $7 \le \theta \le 13°$). Total of 1347 (1574) reflections ($1·0 \le \theta \le 25·0$; 27·5°) measured in the range $-7 \le h \le 0$, $-12 \le k \le 0$, $-14 \le l \le 6$ for (I); $-8 \le h \le 0$, $-15 \le k \le 0$, $-15 \le l \le 2$ for (II); some high-angle Friedel pairs were also included in both data sets. For (II), standards 215, 223, 324 monitored every 3600 s showed a linear decrease of 5% and were used to scale the data. No correction made for absorption. 1208 (1483) unique reflections ($R_{int}0·109$; 0·046), 750 (1239) satisfied $I \ge 2·5\sigma(I)$. Structures solved by direct methods (Sheldrick, 1986), full-matrix least-squares refinement of 128 (156) parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms located from difference map and refined except for methyl H in (I) which were included at their calculated positions. At convergence $R = 0·061$ (0·031), $wR = 0·061$ (0·030), $w = k/[\sigma^2(F) + gF^2]$ for $k = 1·35$ (26·2) and $g = 0·0031$ (0·0001), $S = 1·39$ (13·1), $(\Delta/\sigma)_{max} \le 0·001$, $(\Delta\rho)_{max} = 0·48$ (0·23), $(\Delta\rho)_{min} = -0·49$ (0·49) e Å^{-3}; no extinction correction. Scattering factors for all atoms given in *SHELX76*

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

|	(I), $Y = $H(8)	(II), $Y = $C(8)
O(1)—C(6)	1·208 (8)	1·207 (3)
O(2)—C(6)	1·297 (9)	1·304 (3)
N(1)—C(2)	1·490 (9)	1·517 (3)
N(1)—C(5)	1·49 (1)	1·515 (3)
C(2)—C(3)	1·51 (1)	1·525 (3)
C(2)—C(6)	1·50 (1)	1·508 (3)
C(3)—C(4)	1·51 (1)	1·533 (4)
C(4)—C(5)	1·49 (2)	1·523 (4)
N(1)—C(7)	1·52 (1)	1·505 (3)
N(1)—Y	1·05 (7)	1·508 (3)
O(2)—H	0·65 (8)	0·94 (4)
C(2)—N(1)—C(5)	105·0 (6)	100·5 (2)
C(2)—N(1)—C(7)	113·7 (7)	112·5 (2)
C(2)—N(1)—Y	112 (4)	114·2 (2)
C(5)—N(1)—C(7)	113·4 (7)	110·7 (2)
C(5)—N(1)—Y	108 (3)	108·9 (2)
C(7)—N(1)—Y	105 (4)	109·7 (2)
N(1)—C(2)—C(3)	101·9 (7)	103·8 (2)
N(1)—C(2)—C(6)	112·4 (5)	118·7 (2)
C(3)—C(2)—C(6)	115·2 (7)	114·0 (2)
C(2)—C(3)—C(4)	106·4 (8)	104·8 (2)
C(3)—C(4)—C(5)	106·4 (9)	105·5 (2)
C(4)—C(5)—N(1)	105·2 (7)	104·7 (2)
C(2)—C(6)—O(1)	124·2 (6)	118·9 (2)
C(2)—C(6)—O(2)	109·0 (6)	116·6 (2)
O(1)—C(6)—O(2)	126·7 (7)	124·4 (2)
C(6)—O(2)—H	102 (8)	112 (3)
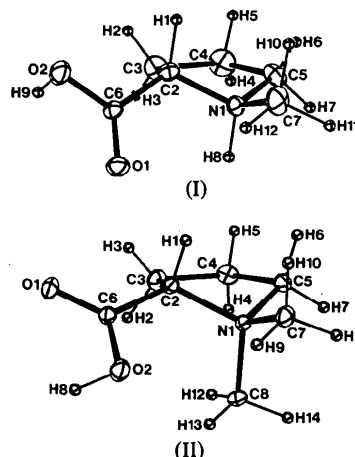
Fig. 1. Molecular structure and numbering scheme for (I) hygric acid and (II) stachydrine, showing 15% probability ellipsoids (Johnson, 1971).

(Sheldrick, 1976). All calculations on laboratory micro-Vax I computer system. Atomic parameters given in Tables 1 and 2, selected bond distances and angles in Table 3,* the numbering scheme used is shown in Fig. 1.

Related literature. Hygric acid (I), a previously unknown naturally occurring compound in higher plants, has been found to accumulate in various species of the *Melaleuca* genus (Jones, Naidu, Paleg & Tiekink, 1988). (I) has been implicated in the biosynthesis of stachydrine (II), a compound which occurs widely in higher plants (Delaveau, Koudogbo & Pousset, 1972) and in algae (Blunden, Gorden, McLean & Guiry, 1982).

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* Lists of structure factors, thermal parameters, H-atom parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44999 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,4-Diiodocubane

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Abstract. 1,4-Diiodopentacyclo[4.2.0.0^{2,5}.0^{3,8}.0^{4,7}]-octane, $C_8H_6I_2$, $M_r = 355.9$, monoclinic, $P2_1/c$, $a = 7.137$ (1), $b = 7.269$ (2), $c = 8.991$ (2) Å, $\beta = 111.69$ (2)°, $V = 433.4$ (3) Å^3, $Z = 2$, $D_x = 2.73$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å (graphite monochromator), $\mu = 72.6$ cm^{-1}, $F(000) = 320$, $T = 293$ K, final $R = 0.036$ for 714 reflections with $I > 3\sigma(I)$. The molecule possesses a center of symmetry; the two I atoms are positioned on opposite corners of the cube.

Experimental. The compound (I) was synthesized by one of the authors (Reddy, 1988). Crystals obtained from methyl chloride and hexane solution; 0.27 × 0.20 × 0.23 mm rectangular shape used for X-ray measurements; Enraf–Nonius CAD-4 diffractometer;

Mo radiation with incident-beam monochromator; cell parameters from 25 reflections automatically centered in the range $4.7 < \theta < 25.7°$; θ–2θ scan at variable θ speed of 1.03 to 8.24° min^{-1}; each scan recorded in 96 steps over the θ range of 1.5 × (1.2° + 0.35°tanθ) and subsequently processed with a modified Lehmann–Larsen profile analysis procedure (Lehmann & Larsen, 1974; Ammon, 1986); six standards measured at 200 data intervals; 923 data (includes standards and systematically absent reflections) measured from $\theta = 2$ to 25°; index range for h, k, $l = -8$ to 8, 0 to 8, −10 to 0; 830 unique reflections; 714 reflections with $I > 3\sigma(I)$; average change in standard intensities of 1.9% with a range of −1.4 to 4.3%; $R_{int} = 0.011$ (51 pairs). All crystallographic calculations performed with the