$w = 1/\sigma^2(F)$ with $\sigma^2(F)$ given by the expression $[\sigma^2(I_c) + (0.04F)^2]^{1/2}$, 1677 reflections, 186 variables, $S = 1.847$, $(\Delta/\sigma)_{max} < 0.0$. A final difference Fourier map showed no features outside the range of -0.17 and 0.20 e Å^{-3}. Values of the neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the molecule and Fig. 2 is a stereoview of an overlay between this TPPO fragment and the three known TPPO polymorphs. The long P—C bonds allow considerable conformational flexibility and, in the present case, two independent rings are forced into near parallelism by the hydrogen bonding.

Related literature. TPPO has been shown to be useful for the formation of cocrystalline solids which are often better formed and of higher quality than the substrates themselves (Etter & Baures, 1988). There are three known polymorphs of TPPO, an orthorhombic form (space group *Pbca*) reported by Bandoli, Bortolozzo, Clemente, Croatto & Panattoni (1970), a monoclinic modification (space group

$P2_1/a$) by Ruban & Zabel (1976) and a second monoclinic modification (space group $P2_1/c$) by Spek (1987). The rotation angles for the three phenyl rings fall within the range for the stereoisomerism of triphenylphosphine oxide fragments and related compounds (Bye, Schweizer & Dunitz, 1982).

PWB thanks Dr Drake Eggleston (Smith Kline and French Laboratories) for his assistance in the structure determination and JVS thanks Mr William Comstock (NHLBI-LC) for mass spectroscopy.

---

* 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 52439 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BANDOLI, G., BORTOLOZZO, G., CLEMENTE, D. A., CROATTO, U. & PANATTONI, C. (1970). *J. Chem. Soc. A*, pp. 2778–2780.
BYE, E., SCHWEIZER, B. & DUNITZ, J. D. (1982). *J. Am. Chem. Soc.* **104**, 5893–5898.
ETTER, M. C. (1989). Private communication.
ETTER, M. C. & BAURES, P. W. (1988). *J. Am. Chem. Soc.* **110**, 639–640.
International Tables for X-ray Crystallography. (1974). Vol. IV, p. 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
RUBAN, G. & ZABEL, V. (1976). *Cryst. Struct. Commun.* **5**, 671–677.
SPEK, A. L. (1987). *Acta Cryst.* C43, 1233–1235.
WALKER, N. & STUART, D. (1983). *Acta Cryst.* A39, 158–166.

Structure of a New Form of Betaine Hydrochloride

BY WAI-HING YIP, WANG RU-JI AND THOMAS C. W. MAK*

Department of Chemistry, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong

Abstract. 1-Carboxy-N,N,N-trimethylmethanaminium chloride, $C_5H_{12}NO_2^+.Cl^-$, $M_r = 153.63$, monoclinic, Cm, $a = 10.578$ (1), $b = 6.9383$ (8), $c = 5.7052$ (8) Å, $\beta = 111.45$ (1)°, $V = 389.72$ (8) Å^3, $Z = 2$, $D_m = 1.357$, $D_x = 1.309$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 4.24$ cm^{-1}, $F(000) = 164$, $T = 294$ K, $R_F = 0.048$ for 820 independent reflections. The packing mode in this new form (I) is different from that in the known form (II) which belongs to space group $P2_1/c$ with $V = 776$ Å^3 and $Z = 4$ [Fischer,

Templeton & Zalkin (1970). *Acta Cryst.* B26, 1392–1397]. Although the molecule, which contains an O—H⋯Cl hydrogen bond, occupies a site of symmetry m in (I) and has only approximate mirror symmetry in (II), the measured bond lengths and angles in both crystalline forms agree with one another within experimental error.

Experimental. In an attempt to prepare a tin complex of betaine (1-carboxy-N,N,N-trimethylmethanaminium hydroxide inner salt), $(CH_3)_3NCH_2COO$, $SnCl_4.5H_2O$ (0.8972 g, 2.56 mmol) and betaine

---

* To whom correspondence should be addressed.

Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^3)$ *for form* (I) *of betaine hydrochloride*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cl(1)	5000	0	5000	46 (1)
O(1)	2193 (3)	0	4890 (6)	53 (1)
O(2)	1302 (3)	0	671 (7)	55 (1)
C(1)	1182 (3)	0	2680 (7)	37 (1)
C(2)	-136 (3)	0	3150 (6)	33 (1)
N(1)	-1404 (3)	0	837 (5)	31 (1)
C(3)	-1521 (3)	1780 (4)	-725 (5)	44 (1)
C(4)	-2577 (4)	0	1744 (9)	46 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *of the betaine hydrochloride molecule*

Symmetry transformation: (i) x, $-y$, z. Corresponding values for form (II) are enclosed in square brackets.

O(1)—C(1)	1·322 (4) [1·316 (2)]	O(2)—C(1)	1·999 (6) [1·193 (2)]
C(1)—C(2)	1·513 (6) [1·500 (3)]	C(2)—N(1)	1·498 (4) [1·497 (2)]
N(1)—C(3)	1·502 (3) [1·496 (3)]	N(1)—C(4)	1·509 (6) [1·504 (3)]
N(1)—C(3i)	1·502 (3) [1·498 (3)]	O(1)···Cl(1)	2·946 (3) [2·955 (2)]
O(1)—C(1)—O(2)	125·4 (4) [125·1 (2)]	O(1)—C(1)—C(2)	107·9 (3) [109·0 (2)]
O(2)—C(1)—C(2)	126·6 (3) [126·0 (2)]	C(1)—C(2)—N(1)	115·5 (3) [116·4 (2)]
C(2)—N(1)—C(3)	112·1 (2) [111·7 (2)]	C(2)—N(1)—C(4)	106·3 (3) [107·0 (2)]
C(3)—N(1)—C(4)	107·7 (2) [108·3 (2)]	C(2)—N(1)—C(3i)	112·1 (2) [111·4 (1)]
C(3)—N(1)—C(3i)	110·7 (3) [109·8 (2)]	C(4)—N(1)—C(3i)	107·7 (2) [108·5 (2)]
C(1)—O(1)···Cl(1)	118·6 (2) [116·6 (2)]		

monohydrate (1·384 g, 10·24 mmol) were dissolved in 12 ml water. After the clear aqueous solution was allowed to stand at room temperature for three weeks, colorless granular crystals of (I) were deposited. A crystal of dimensions 0·44 × 0·44 × 0·44 mm was mounted on a Nicolet *R3m*/V diffractometer; density measured by flotation in *n*-hexane/CCl₄; data collected in the ω–2θ scan mode ($3\cdot0 \le 2\theta \le 80°$) at room temperature (294 K); variable scan rate (3·01–15·63° min⁻¹); graphite-monochromatized Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($20\cdot3 \le 2\theta \le 30\cdot6°$); 1128 independent reflections measured ($0 \le h \le 19$, $0 \le k \le 12$, $-10 \le l \le 10$), of which 820 reflections were considered observed $[|F_o| \ge 6\sigma(|F_o|)]$. Two standard reflections (60$\bar{2}$ and 420) monitored after every 125 data measurements showed only random fluctuations within 1% of their mean values during data collection. Lorentz–polarization corrections and ψ-scan-based empirical absorption corrections (transmission factors 0·746–0·921) were applied. Structure solved by direct-phase determination. The H atom of the carboxy group was derived from a difference Fourier map, held fixed, and its isotropic factor was allowed to vary. The methyl groups were treated as rigid groups (C—H bonds fixed at 0·96 Å), and their H atoms were assigned the same isotropic temperature factor of $U = 0\cdot06$ Å² in structure-factor calculations. Final $R_F = 0\cdot048$ and $wR_F = 0\cdot062$ with $w = [\sigma^2(F_o) + 0\cdot0012|F_o|^2]^{-1}$ for 51 parameters and 820 reflections, $S = 1\cdot411$, $(\Delta/\sigma)_{max} = 0\cdot001$; largest peaks in

the final difference map of 0·68 and $-0\cdot47$ e Å⁻³. Computations were performed on a DEC Micro-VAXII computer with the *SHELXTL-Plus* program package (Sheldrick, 1982, 1985). Analytic expressions of atomic scattering factors were employed, and anomalous-dispersion corrections were incorporated (*International Tables for X-ray Crystallography* 1974). Table 1 lists the atomic parameters,* and Table 2 gives the bond lengths and angles of the [(CH₃)₃NCH₂COOH]Cl molecule in the present crystalline form (I) and form (II) as determined by Fischer, Templeton & Zalkin (1970). Fig. 1 illustrates the mode of molecular packing in (I), which is quite different from that in (II).

Related literature. The title compound is of interest in view of its biological activity in metabolism, mainly as a transmethylating agent in complex lipids (Vogt, 1966; Abdel Kader, Abdel Hay, Kamel, Talaat & Fayek, 1970). Clastre (1964) published a preliminary structural report for this compound based on two-dimensional projections, and Fischer *et al.* (1970) carried out an independent study to provide an accurate structure [space group $P2_1/c$, $a =$
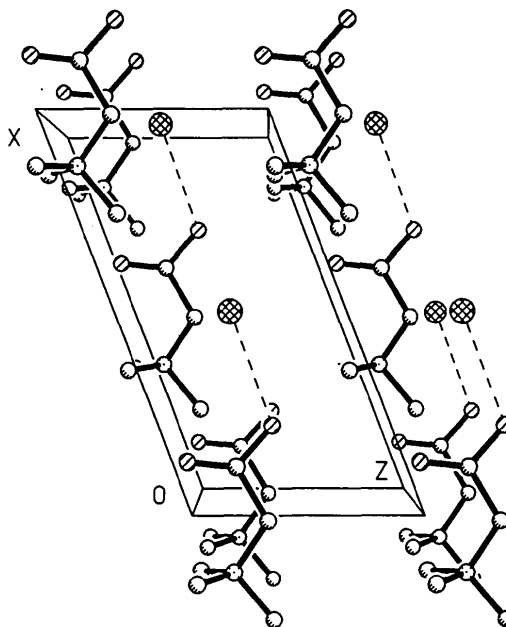

Fig. 1. Molecular packing in the crystal structure of form (I) of the title compound. The origin of the unit cell lies at the lower left corner, with *a* pointing upwards at a slant, *b* towards the reader, and *c* from left to right.

$7 \cdot 428$ (2), $b = 9 \cdot 108$ (5), $c = 11 \cdot 550$ (3) Å, $\beta = 96 \cdot 71$ (3)°, $V = 776$ Å^3, $Z = 4$; $R_F = 0 \cdot 026$ for 782 Cu $K\alpha$ data]. Both groups worked with needle-shaped crystals of form (II) prepared from crystallization in aqueous methanol.

References

ABDEL KADER, M. M., ABDEL HAY, A., KAMEL, G., TALAAT, M. & FAYEK, K. I. (1970). *Acta Biol. Med. Ger.* **24**, 1–11.

CLASTRE, J. (1964). *C. R. Acad. Sci.* **259**, 3267–3269.
FISCHER, M. S., TEMPLETON, D. H. & ZALKIN, A. (1970). *Acta Cryst.* **B26**, 1392–1397.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 55, 99, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
SHELDRICK, G. M. (1982). *Computational Crystallography*, edited by D. SAYRE, pp. 506–514. Oxford Univ. Press.
SHELDRICK, G. M. (1985). *Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.
VOGT, H. (1966). *Arch. Gefluegelkd.* **30**, 283–298.

Structure of (2R,3R)-3-Acetoxy-5,7-dihydroxy-6-methylflavanone

By M. C. Cheng, Y. S. Cheng, C. F. Chang and Yu Wang*

Department of Chemistry, National Taiwan University, Taipei, Taiwan

Abstract. $C_{18}H_{16}O_6$, $M_r = 328 \cdot 32$, monoclinic, $I2$, $a = 14 \cdot 488$ (5), $b = 8 \cdot 034$ (1), $c = 14 \cdot 341$ (5) Å, $\beta = 110 \cdot 76$ (3)°, $V = 1561$ (2) Å^3, $Z = 4$, $D_x = 1 \cdot 40$, $D_m = 1 \cdot 39$ g cm^{-3}, $\mu = 1 \cdot 0$ cm^{-1}, $F(000) = 680$, $T = 298$ K, $\lambda(Mo\ K\alpha) = 0 \cdot 71069$ Å, final $R = 0 \cdot 050$ for 1827 reflections. The structure of a new flavanone (extracted from the heartwood of *Pinus morrisonicola*) was characterized by X-ray diffraction. All single- and double-bond characters are as expected for a flavanone structure. The saturated heterocyclic ring in the molecule is not planar. The acetoxy and phenyl groups make a torsion angle of $53 \cdot 0°$.

Experimental. Crystal (Fang, Chang & Cheng, 1987) $0 \cdot 2 \times 0 \cdot 3 \times 0 \cdot 4$ mm, CAD-4 diffractometer. Unit cell: 25 reflections, 2θ range $19 \cdot 58$ to $23 \cdot 56°$. D_m by flotation (*n*-hexane/CCl$_4$). $2\theta_{max} = 60°$. Ranges of h, k, l: -20 to 20, 0 to 11, 0 to 20, respectively. 2θ scan range $(1 \cdot 4 + 0 \cdot 7\tan\theta)°$. Three standard reflections monitored every hour: variation $< 2\%$. 2449 unique reflections, 1827 observed with $I > 1 \cdot 5\sigma(I)$. $R(F) = 0 \cdot 050$, $wR(F) = 0 \cdot 037$, $S = 3 \cdot 8$. Weighting scheme from counting statistics. Structure solved by direct method using the *MULTAN* program. H atoms calculated according to the ideal geometry. Only one of the methyl H atoms and the hydroxyl H atoms are found in a difference Fourier map after isotropic refinement. $(\Delta/\sigma)_{max} = 0 \cdot 01$. Peak in final map $< \pm 0 \cdot 18$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computing programs: *NRCC SDP VAX Package* (Gabe, 1985), *MULTAN* and *ORTEP* from

Enraf–Nonius (1979) *Structure Determination Package*. Atomic parameters are given in Table 1,† bond distances and angles in Table 2. A drawing of the molecule is shown in Fig. 1.

---

† Lists of anisotropic temperature factors of the non-hydrogen atoms, positional and isotropic thermal parameters of the H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51919 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic parameters and B_{eq} values*

E.s.d.'s refer to the last digit printed.

$$B_{eq} = \tfrac{8}{3}\pi^2 \Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O1	0·5622 (2)	0·68310	0·1532 (2)	4·6 (2)
C2	0·6382 (3)	0·6817 (6)	0·1178 (3)	3·7 (2)
C3	0·6592 (3)	0·5313 (6)	0·0843 (3)	3·9 (2)
C4	0·7368 (3)	0·5259 (6)	0·0500 (3)	3·8 (2)
C5	0·7966 (3)	0·6618 (6)	0·0502 (3)	3·9 (2)
C6	0·7718 (3)	0·8111 (6)	0·0829 (3)	4·2 (2)
C7	0·6905 (3)	0·8258 (6)	0·1155 (3)	3·6 (2)
C8	0·6584 (3)	0·9852 (6)	0·1389 (3)	3·9 (2)
C9	0·5616 (3)	0·9777 (6)	0·1550 (3)	4·0 (2)
C10	0·5598 (3)	0·8229 (6)	0·2151 (3)	3·9 (2)
C11	0·4720 (3)	0·8035 (6)	0·2456 (3)	4·0 (2)
C12	0·3784 (3)	0·8293 (7)	0·1813 (3)	6·4 (3)
C13	0·2993 (3)	0·8061 (10)	0·2103 (4)	8·1 (3)
C14	0·3120 (3)	0·7580 (9)	0·3038 (4)	7·6 (3)
C15	0·4049 (3)	0·7280 (9)	0·3693 (3)	8·3 (4)
C16	0·4861 (3)	0·7505 (7)	0·3419 (3)	6·0 (3)
O17	0·7601 (2)	0·3811 (4)	0·0152 (2)	4·7 (1)
C18	0·8825 (3)	0·6468 (6)	0·0153 (3)	5·4 (2)
O19	0·8242 (2)	0·9495 (5)	0·0792 (2)	5·8 (2)
O20	0·7028 (2)	1·1170 (5)	0·1417 (2)	5·2 (2)
O21	0·5474 (2)	1·1218 (5)	0·2066 (2)	4·7 (1)
C22	0·4695 (3)	1·2193 (6)	0·1588 (3)	4·3 (2)
O23	0·4173 (2)	1·1944 (5)	0·0749 (2)	6·0 (2)
C24	0·4587 (3)	1·3559 (6)	0·2231 (3)	5·7 (3)