molecular F⋯F and F⋯O distances are 3.02 and 3.03 Å, respectively. The O⋯H⋯O hydrogen bond, 2.648 (3) Å, is not significantly different from the bond in the trichloroacetic acid dimer, 2.666 (5) Å (Jönsson & Hamilton, 1972). It is also comparable to the O⋯H⋯O bond in the non-substituted acetic acid chain structure, 2.625 (5) Å (Nähringbauer, 1970), 2.631 (8) Å (Jönsson, 1971). The dimer is nearly planar. The deviations of the atoms from the plane through one of the carboxyl groups and from the least-squares plane through O(1), O(2), C(1) and C(2) are given in Table 2 and are very similar to those found in the trichloroacetic acid dimer. The CF₃COOH molecule has a perfectly staggered conformation. The torsion angle C(1)⋯C(2)⋯C(1)⋯O(1) is 29.1 (3)°, compared to 11.5 (4)° and 6.3 (1.6)° in CC₁₃COOH and CH₃COOH, respectively. C(1)⋯C(2), 1.526 (5) Å, is significantly longer, and the O(1)⋯C(1)⋯O(2) bond angle, 128.1 (3)°, larger than the corresponding bond and angle in acetic acid, 1.478 (6) Å and 121.3 (5)°.

Similar observations were made for the chloro-substituted acetic acid. The opening of the O–C–O angle is most probably associated with the different hydrogen bonding: dimer and chain, respectively. The CF₃ group is similar to CF₃ groups found in other structures (e.g. Lundgren, 1978) with an average C–F bond of 1.259 Å, and average C–C–F and F–C–F angles of 110.7° and 108.3°, respectively.

We thank Professor Ivar Olovsson for the facilities placed at our disposal and Mr Hilding Karlsson for skilled technical assistance. This work has been supported by grants from the Swedish Natural Science Research Council, which are gratefully acknowledged.

References


1-Methylimidazolium Oxalurate

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Abstract. (C₄H₇N₂)⁺.(C₃H₃N₂O₄)⁻, m.p. 423 K, Pī, a = 7.718 (4), b = 8.062 (4), c = 9.603 (4) Å, a = 69.34 (4), β = 62.52 (4), γ = 67.22 (4)°, Z = 2, Dm = 1.490 Mg m⁻³. The crystal structure determination was based on the X-ray intensities (Mo Kα) of 1779 reflections collected by diffractometer. Parameter refinement by the full-matrix least-squares method gave R = 0.058. The longer carboxylate C–O bond (1.259 Å) involves the O atom which forms the salt bridge (NH⋯O, 2.78 Å) and another hydrogen bond. © 1979 International Union of Crystallography
Introduction. Crystals of the oxalurate salt were obtained from a solution of 1-methylimidazole and parabanic acid in absolute ethanol. Parabanic acid (imidazoletrione) was hydrolyzed (Andrews & Sell, 1955) presumably with water absorbed from the atmosphere. This reaction also occurred when efforts to form a crystal complex of parabanic acid with 9-ethyladenine led to a remarkable crystal structure containing the variance from counting statistics. There were no

The atomic parameters are fractional coordinates (x 104 for non

phase problem was solved by symbolic addition and

corrections for X-ray absorption or extinction. The

0.20 mm, and 0/2t~ scans. The variance in an intensity

with sin 0/2 < 0.59 ,/~-~, using a crystal 0.35 × 0.25 ×

controlled four-circle diffractometer and graphite-

known as the adenine complex.

parabanic acid, oxalurate anion, 9-ethyladininium cation and water (Shieh & Voet, 1975), hereafter

form a crystal complex of parabanic acid with 9-ethyl-

obtained from a solution of 1-methylimidazole and

E.s.d.'s in parentheses refer to the least significant digit in the

parameter value.
but significant differences in bond lengths and angles (Fig. 1) which follow the same pattern as was observed in comparing the imidazolium cation with imidazole (Craven, McMullan, Bell & Freeman, 1977). Notable in Fig. 1 is the opening of the ring angle at N(3) by 4.5° as a result of protonation.

The structure of the oxalurate anion is similar to that observed in the adenine complex (Shieh & Voet, 1975), including the intramolecular NH⋯O hydrogen bond (Fig. 2). When the anion is subdivided the atoms within each of the urea, peptide and carboxylate fragments are almost coplanar (Table 2) as in the adenine complex. The torsion angles in the bonds linking these fragments (Table 3) show that the anion is slightly puckered from planarity, but there is less puckering than in the adenine complex. Most oxalurate bond lengths and angles agree with those in the adenine complex, although the difference (0.015 Å) in the N(3)⋯C(4) length may be significant. There is an interesting difference in the carboxylate C–O bonds in the two structures, which appears to be related to the hydrogen bonding. In the adenine complex, both O atoms form two hydrogen bonds and the C–O bond lengths (1.238, 1.241 Å) are the same within the experimental error (σ = 0.002 Å). In the 1-methylimidazolium salt the C–O bond with O(6), which forms both the salt bridge and a second hydrogen bond (Fig. 3), is longer (1.259 vs 1.230 Å) than the C–O bond with O(5) which is not hydrogen bonded.

There are three intermolecular hydrogen bonds in the crystal structure (Fig. 3). The shortest is the salt bridge between the cation N(3)H group and the anion O(6).

### Table 3. Torsion angles (°)

The e.s.d.'s in torsion angles are 2° for angles involving H otherwise 0.3°.

<table>
<thead>
<tr>
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<th>Adenine complex</th>
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<td>119</td>
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<table>
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<tr>
<td>C(2)–N(3)–C(4)–C(5)</td>
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<tr>
<td>N(3)–C(4)–C(5)–O(5)</td>
<td>−167.3</td>
<td>−167.3</td>
</tr>
</tbody>
</table>

Fig. 1. Bond lengths (Å) and angles (°) in (a) 1-methylimidazole (Wang & Craven, 1979), (b) 1-methylimidazolium cation (present work). Largest circles represent nitrogen atoms, N(1) and N(3).

Fig. 2. Bond lengths (Å) and angles (°) in oxalurate anion in (a) 1-methylimidazolium salt (present work), (b) adenine complex (Shieh & Voet, 1975).

Fig. 3. The crystal structure of 1-methylimidazolium oxalurate. Atoms are represented by 25% probability thermal ellipsoids.
The N—O distance is 2.78 Å and the N—H—O angle is 158°. The dihedral angle between the planes of the bridged molecules is 80°. There is also a hydrogen bond N(1)H(1)···O(6) which links oxalurate anions head to tail to form ribbons parallel to the a axis. This has an N—O distance of 2.91 Å and the NH—O angle is 175°. The oxalurate ribbons are crosslinked through pairs of N(3)H(3)···O(2) hydrogen bonds which are relatively weak. The N—O distances are 3.05 Å and NH—O angles are 155°.

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**cyclo(-L-Prolyl-D-tert-leucyl-)**

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(Received 13 October 1978; accepted 1 November 1978)

**Abstract.** C₁₁H₁₈N₂O₂, orthorhombic, P₂₁₂₁₂, Z = 4, FW 210.3, a = 10.942 (2), b = 16.438 (4), c = 6.452 (1) Å, D₀ = 1.20, Dₐ = 1.14 Mg m⁻³, λ(Cu Kα) = 1.5418 Å (graphite monochromator); the final R = 0.046 on 794 observed reflexions [F² > 3σ(F²)] in the range 0 < 2θ ≤ 68°. The molecules are linked by hydrogen bonds to form chains [N(2)—H(1)···O(1): N(2)—O(2) = 2.894 (4), H(1)—O(1) = 2.053 (4) Å, ∠N(2)—H(1)—O(1) = 153 (3)°].

**Introduction.** Colourless transparent prismatic crystals of the title compound were prepared by Pospíšek & Bláha (1976) at the Institute of Organic Chemistry and Biochemistry, Praha. Our main interest was in the molecular conformation of the 2,5-piperazinedione ring with the bulky side chain of the tert-butyl group.

Measurement of integrated intensities of 2364 reflexions with h ≥ 0, k ≥ 0, l ≥ 0 was made on a Syntex P₂₁ four-circle diffractometer. The scan range 〈2θ(1) = 1°, 2θ(1) + 1°〉 and a variable scan rate from 1.2 to 29.3° min⁻¹ were used. Three standard reflexions showed no unusual fluctuations. The data were corrected for background, Lorentz and polarization effects and for coincidence of pulses. The quality of the measurement was tested by

\[ \sum_{hkl} \frac{|I_{hkl} - I_{hkl}|}{I_{hkl}} \]

for both dependent sets of reflexions.

The structure was solved by direct methods with the tangent formula. For the set with the best figures of merit an E map was calculated and all non-hydrogen atoms were identified. They were refined anisotropically by full-matrix least squares to R = 0.102. An attempt to locate H atoms from a difference synthesis failed. We calculated positions for the H atoms with an isotropic temperature factor B = 3.4 Å². Positional and thermal parameters (anisotropic for non-hydrogen and isotropic for H atoms) were refined in three blocks (the last cycle in one block) to a final R = 0.046. Weights 1/σ(F²) were used. The final difference synthesis displayed no features >0.2 e Å⁻³.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34026 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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