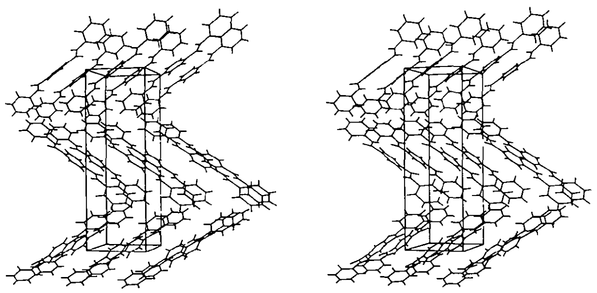

Fig. 2. A stereoscopic view of the packing.

Significant differences exist with twist angles observed in related compounds. The value of 30·4° observed here can be compared with the 17·6 in acetanilide (Brown, 1966) and 35·9° in PPDB. The corresponding values of the angle 30·6° are 24·6 in benzamide (Blake & Small, 1972) and 29·1° in PPDB.

The molecules of DPTP are linked by N—H···O hydrogen bonds of length 3·118 (2) Å. As in PPDB each molecule is hydrogen bonded to two other molecules. The molecules connected in this way are related to each other by translation along *c*. The hydrogen-bonding scheme found in both DPTP and PPDB contrasts with that found in a second polymorph of PPDB (Adams, Fratini & Wiff, 1978) in which each molecule is hydrogen bonded to four others.

A stereoscopic view of the crystal structure prepared by *POP*1 (van de Waal, 1978) is given in Fig. 2.

References

ADAMS, W. W., FRATINI, A. V. & WIFF, D. R. (1978). *Acta Cryst.* B**34**, 954–956.
BLAKE, C. C. F. & SMALL, R. W. H. (1972). *Acta Cryst.* B**28**, 2201–2206.
BROWN, C. J. (1966). *Acta Cryst.* **21**, 442–445.
DEWAR, R. B. K. (1970). *Crystallographic Computing*, edited by F. R. AHMED, pp. 63–65. Copenhagen: Munksgaard.
GAYMANS, R. J. & HARKEMA, S. (1977). *J. Polym. Sci. Polym. Phys. Ed.* **15**, 587–590.
HARKEMA, S. & GAYMANS, R. J. (1977). *Acta Cryst.* B**33**, 3609–3611.
WAAL, B. W. VAN DE (1978). *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, working sessions and program market, pp. 33–34. Delft Univ. Press.

Acta Cryst. (1979). B**35**, 508–510

Trifluoroacetic Acid*

BY INGER NAHRINGBAUER,† JAN-OLOF LUNDGREN AND ERIK KROGH ANDERSEN‡

Institute of Chemistry, University of Uppsala, Box 531, *S*-751 21 *Uppsala, Sweden*

(*Received* 21 *October* 1978; *accepted* 6 *November* 1978)

Abstract. $C_2HF_3O_2$, monoclinic, $P2_1/c$, $Z = 4$, $a = 8.060$ (1), $b = 4.762$ (1), $c = 9.959$ (1) Å, $\beta = 107.64$ (1)°, $D_x = 2.079$ Mg m^{-3} at 83 K, m.p. 257·9 K. Intensities were recorded at 83 K. Refinement with 524 observed data gave $R(F) = 0.047$, $R_w(F) = 0.060$. The structure is composed of hydrogen-bonded centrosymmetric dimers packing with normal van der Waals separations. The O—H···O bond is 2·648 (3) Å.

Introduction. The present investigation was undertaken to study the geometry of the CF_3COOH molecule and its bonding in the solid state. Formic and acetic acids have been found to form infinite chains in the crystalline state (Nahringbauer, 1978, 1970) whereas the chloro-substituted acetic acid (Jönsson & Hamilton, 1972) and higher carboxylic acids (Strieter, Templeton, Scheuerman & Sass, 1962) occur as dimers. The preference of the dimeric form to the chain form might be a steric effect due to the substitution of the CH_3 group by the larger CCl_3 or more bulky aliphatic groups. However, the substitution of only one H atom by F can give the dimeric form. The monofluoroacetic acid structure is composed of centrosymmetric dimers formed by carboxyl-group coupling (Kanters & Kroon, 1972). The size of the CF_3 group is intermediate between those of CH_3 and CCl_3; the structure determination of trifluoroacetic acid is thus a natural extension of the previous studies.

Crystals were grown by zone melting from commercially available CF_3COOH (*pro analysi*) sealed in thin-walled glass capillaries. Cell dimensions and intensities

* Hydrogen Bond Studies. CXXXV. Part CXXXIV: Lundgren, Tellgren & Olovsson (1978).

† Present address: Institute of Inorganic and Physical Pharmaceutical Chemistry, Biomedical Centre, University of Uppsala, Box 574, S-751 23 Uppsala, Sweden.

‡ Permanent address: Department of Chemistry, University of Odense, DK-5000 Odense, Denmark.

0567-7408/79/020508-03$01.00

were measured at 83 K on a Stoe–Philips semi-automatic two-circle diffractometer [Cu $K\alpha$ radiation monochromatized with a graphite crystal, $\lambda(\text{Cu } K\alpha_1) = 1{\cdot}54051$, $\lambda(\text{Cu } K\alpha_2) = 1{\cdot}54433$ Å]. The cell parameters were determined by least-squares methods from 2θ values of 36 high-angle reflexions from two crystals mounted around [100] and [010], respectively. Intensities for layers $0kl$ to $7kl$ were recorded from a cylindrical crystal with a diameter of 0·16 mm and a length of 0·32 mm mounted around [100]. The measurements were made in the equi-inclination ω scan mode. 663 independent reflexions were measured. Three $0kl$ test reflexions were monitored at regular intervals together with sets of three test reflexions within each layer. The intensities of the $0kl$ test reflexions decreased linearly to about 80% of their initial values. An appropriate correction was applied to all intensities. A complementary set of 455 independent reflexions (layers $hk0$ to $hk6$) was similarly measured at 83 K from a crystal mounted around [001]. This set was later used only to determine the relative scale factors between layers in the previous data set. The data sets were corrected for Lorentz, polarization and absorption effects [$\mu(\text{Cu } K\alpha) = 2{\cdot}57$ mm^{-1}]. E.s.d.'s were obtained from Poisson counting statistics, the scatter observed in the test reflexions, and the uncertainty in the scaling function (McCandlish, Stout & Andrews, 1975).

The structure was solved by direct methods. The positions of the non-hydrogen atoms were found from an E map, and the H atoms from a subsequent difference synthesis. Full-matrix least-squares refinement minimizing $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = \sigma(F_o^2)/4F_o$, included 69 parameters: one scale factor, an isotropic extinction parameter, positional and anisotropic thermal parameters for the eight independent atoms (an isotropic temperature factor was refined for H). The refinement including 524 reflexions with $F_o^2 > 3\sigma(F_o^2)$ gave $R(F) = \sum(|F_o| - |F_c|)/\sum|F_o| = 0{\cdot}047$ and $R_w(F) = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0{\cdot}060$. The standard deviation of an observation of unit weight was $s = 1{\cdot}18$. The refined value of the isotropic extinction parameter g was $0{\cdot}9\,(2) \times 10^3$ (Coppens & Hamilton, 1970). Scattering factors for C, F and O were those given by Hanson, Herman, Lea & Skillman (1964). The spherical scattering factors of Stewart, Davidson & Simpson (1965) were used for H. Anomalous-dispersion corrections were included for the non-hydrogen atoms (Cromer & Liberman, 1970). Final positional parameters are given in Table 1.* The programs used with IBM 370/155 and IBM 1800 computers have been described by Lundgren (1976).

Table 1. *Atomic coordinates* ($\times 10^5$)

	x	y	z
C(1)	15043 (46)	4106 (63)	39064 (31)
C(2)	28709 (45)	8479 (66)	31488 (31)
O(1)	3219 (31)	20745 (46)	37497 (22)
O(2)	18690 (36)	−17608 (46)	47403 (24)
F(1)	21753 (28)	21421 (42)	19102 (19)
F(2)	35511 (28)	−15487 (41)	28930 (20)
F(3)	41411 (27)	24551 (44)	39463 (21)
H	90330 (780)	20050 (1290)	49216 (730)

Discussion. The structure is composed of hydrogen-bonded centrosymmetric dimers packing with normal van der Waals separations (Fig. 1). The shortest inter-

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34045 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

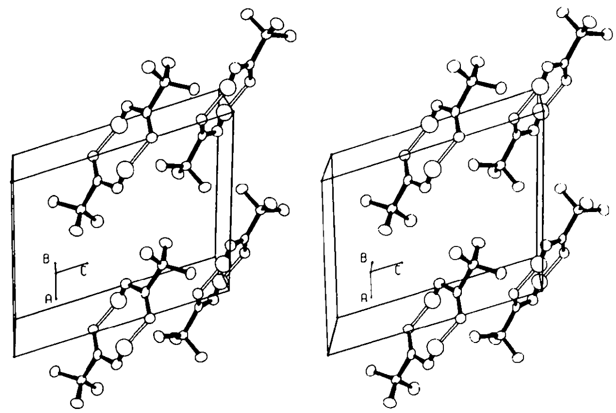

Fig. 1. Stereoscopic *ORTEP* drawing (Johnson, 1965) of the crystal structure of trifluoroacetic acid. Covalent bonds are filled and hydrogen bonds are open. Thermal ellipsoids are scaled to enclose 50% probability.

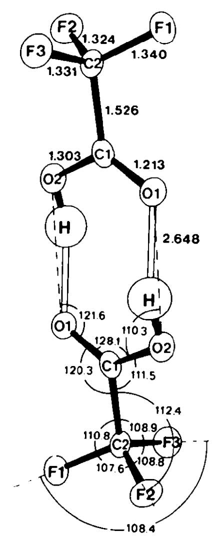

Fig. 2. Bond distances (Å) and angles (°) in the trifluoroacetic acid dimer. The e.s.d.'s of the angles are 0·2–0·3° and of the covalent bonds 0·004–0·005 Å. The e.s.d. of the O···O bond is 0·003 Å. The O—H distance is 0·90 (6) Å and the O—H···O angle is 161 (6)°.

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, (Fig. 2) 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 (Nahringbauer, 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 CF_3COOH molecule has a perfectly staggered conformation. The torsion angle F(1)—C(2)—C(1)—O(1) is 29·1 (3)°, compared to 11·5 (4)° and 6·3 (1·6)° in CCl_3COOH and CH_3COOH, respectively. C(1)—C(2), 1·526 (5) Å, is significantly longer, and the O(1)—C(1)—O(2) 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 CF_3 group is similar to CF_3 groups found in other structures (*e.g.* Lundgren, 1978) with an average C—F bond of 1·332 Å, and average C—C—F and F—C—F angles of 110·7 and 108·3°, respectively.

Table 2. *Least-squares planes*

Atoms defining the plane and its equation

I: O(1), O(2), C(1)

$$-3{\cdot}0703x - 2{\cdot}7228y - 5{\cdot}7464z + 2{\cdot}8184 = 0$$

II: O(1), O(2), C(1), C(2)

$$-2{\cdot}7403x - 2{\cdot}6854y - 6{\cdot}1167z + 2{\cdot}9345 = 0$$

Displacements from plane (Å)

	Plane I	Plane II
O(1)	0·000	−0·004 (2)
O(2)	0·000	−0·004 (2)
C(1)	0·000	0·023 (3)
C(2)	−0·103 (3)	−0·006 (3)
O(1′)	−0·110 (2)	−0·243 (2)
O(2′)	−0·110 (2)	−0·244 (2)
C(1′)	−0·110 (2)	−0·270 (2)

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

Coppens, P. & Hamilton, W. C. (1970). *Acta Cryst.* A**26**, 71–83.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Hanson, H. P., Herman, F., Lea, J. D. & Skillman, S. (1964). *Acta Cryst.* **17**, 1040–1044.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Jönsson, P.-G. (1971). *Acta Cryst.* B**27**, 893–898.

Jönsson, P.-G. & Hamilton, W. C. (1972). *J. Chem. Phys.* **56**, 4433–4439.

Kanters, J. A. & Kroon, J. (1972). *Acta Cryst.* B**28**, 1946–1949.

Lundgren, J.-O. (1976). *Crystallographic Computer Programs*. Report UUIC-B13-4-03. Inst. of Chemistry, Univ. of Uppsala, Sweden.

Lundgren, J.-O. (1978). *Acta Cryst.* B**34**, 2432–2435.

Lundgren, J.-O., Tellgren, R. & Olovsson, I. (1978). *Acta Cryst.* B**34**, 2945–2947.

McCandlish, L. E., Stout, G. H. & Andrews, L. C. (1975). *Acta Cryst.* A**31**, 245–249.

Nahringbauer, I. (1970). *Acta Chem. Scand.* **24**, 453–462.

Nahringbauer, I. (1978). *Acta Cryst.* B**34**, 315–318.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Strieter, F. J., Templeton, D. H., Scheuerman, R. F. & Sass, R. L. (1962). *Acta Cryst.* **15**, 1233–1239.

Acta Cryst. (1979). B**35**, 510–513

1-Methylimidazolium Oxalurate

By Albert C. Wang and B. M. Craven

Crystallography Department, University of Pittsburgh, Pittsburgh, Pennsylvania 15260, *USA*

(*Received* 22 *August* 1978; *accepted* 7 *November* 1978)

Abstract. $(C_4H_7N_2)^+.(C_3H_3N_2O_4)^-$, m.p. 423 K, $P\bar{1}$, $a = 7{\cdot}718$ (4), $b = 8{\cdot}062$ (4), $c = 9{\cdot}603$ (4) Å, $\alpha = 69{\cdot}34$ (4), $\beta = 62{\cdot}52$ (4), $\gamma = 67{\cdot}22$ (4)°, $Z = 2$, $D_m = 1{\cdot}490$ Mg m^{-3}. The crystal structure determination was based on the X-ray intensities (Mo $K\alpha$) of 1779 reflections collected by diffractometer. Parameter refinement by the full-matrix least-squares method gave $R = 0{\cdot}058$. The longer carboxylate C—O bond (1·259 *vs* 1·230 Å) involves the O atom which forms the salt bridge (NH···O, 2·78 Å) and another hydrogen bond.

0567-7408/79/020510-04$01.00