Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Trichloroacetic acid at 105 K

K. Rajagopal,[a] A. Mostad,[b] R. V. Krishnakumar,[c] M. Subha Nandhini[d] and S. Natarajan[d]*

[a]Department of Physics, Saraswathi Narayanan College, Madurai 625022, India, [b]Department of Chemistry, University of Oslo, PO Box 1033 Blindern, N-0315 Oslo 3, Norway, [c]Department of Physics, Thiagarajar College, Madurai 625009, India, and [d]Department of Physics, Madurai Kamaraj University, Madurai 625021, India

Correspondence e-mail: s_natarajan50@yahoo.com

Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.001 Å
R factor = 0.031
wR factor = 0.081
Data-to-parameter ratio = 43.7

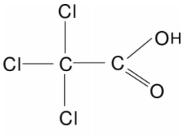
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 January 2003
Accepted 4 February 2003
Online 14 February 2003

In the crystal structure of the title compound, Cl_3CCOOH or $C_2HCl_3O_2$, molecules form centrosymmetric pairs leading to centrosymmetric dimers through strong O—H···O hydrogen bonds. In the absence of other strong intermolecular interactions, the Cl···O [3.029 (1) Å] and Cl···Cl [3.612 (1) Å] interactions presumably play an important role in the stabilization of the crystal structure.

Comment

Carboxylic acids are believed to have existed in the prebiotic earth (Miller & Orgel, 1974; Kvenvolden *et al.*, 1971) and exhibit characteristic intermolecular interactions and aggregation patterns. The carboxyl groups are known to participate strongly in the formation of hydrogen-bonding patterns, especially dimers, as they contain a donor and an acceptor site. Detailed analyses of molecular packing modes of carboxylic acids and some of their primary stacking features have already been made (Leiserowitz, 1976; Bernstein *et al.*, 1994). The concept of using carboxyl groups as primary building blocks in the design of crystals (Desiraju, 1989; MacDonald & Whitesides, 1994) is an important step towards the prediction of crystal structures; this is recognized to be a difficult problem, similar to that of predicting protein folding. In this context, the title compound, (I), represents a class of simple organic molecules of the type R—CO_2H (monocarboxylic acids) and accurate X-ray crystallographic data on such molecules may prove to be useful in the design of crystals, assuming a 'direct' relationship between the nature of R and the difficulty of design.



(I)

In our laboratory, we have been elucidating the crystal structures of amino acid–trichloroacetic acid complexes with a view to understanding the possible effects of halogen–halogen interactions in biomolecular aggregation patterns. A search of the Cambridge Structural Database (Version 5.21, April 2001; Allen, 2002) revealed that no crystallographic data are available for (I) in its pure form, probably due to its highly deliquescent nature. However, a more detailed literature survey points to an unpublished neutron diffraction study of (I) (Verbist *et al.*, 1972). This prompted us to obtain crystals of (I) and elucidate its crystal structure.

DOI: 10.1107/S1600536803002903

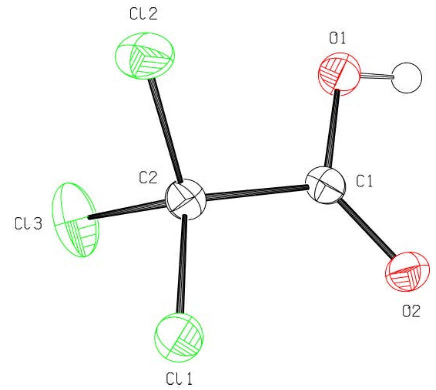


Figure 1
Molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

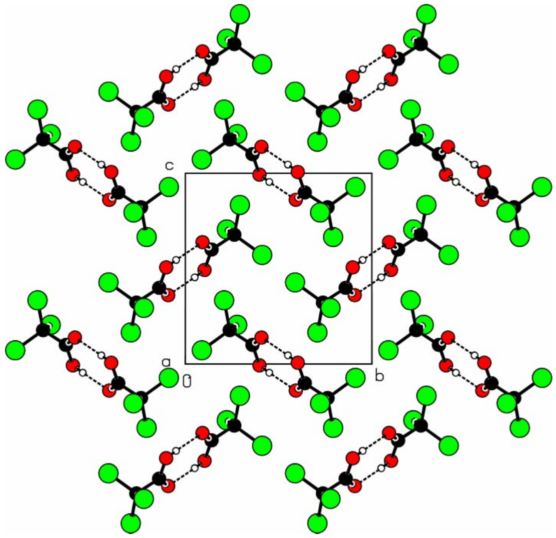


Figure 2
Packing of the molecules of (I), viewed down the *a* axis.

Fig. 1 shows the molecular structure of (I) and the atom-numbering scheme. The bond lengths in the carboxylic acid group, C1—O1(H) = 1.305 (1) Å and C1—O2 = 1.216 (1) Å, agree well with expected values. The packing of molecules of (I), viewed down the *a* axis, is shown in Fig. 2. The molecules form centrosymmetric pairs, leading to centrosymmetric dimers through strong O—H···O hydrogen bonds (Table 2), in agreement with the results of previous proton magnetic resonance studies (Goldman, 1958). In the absence of other strong intermolecular interactions, the observed Cl···O short contact [Cl2···O2 ($x - 1, y, z$) = 3.029 (1) Å] and the closest Cl···Cl interaction [Cl3···Cl3($-x + 1, -y, -z$) = 3.612 (1) Å] presumably play an important role in the stabilization of the crystal structure.

Experimental

Crystals of (I) were obtained from Aldrich in a sealed bottle. The bottle consisted of 'wet' crystals and a few good specimens were put in a dish along with some solid CO_2. A crystal was swiftly mounted on a goniometer head, where the temperature was maintained at 105 K. The crystal remained intact throughout the data collection.

Crystal data

$C_2HCl_3O_2$
$M_r = 163.38$
Monoclinic, $P2_1/c$
$a = 6.126$ (1) Å
$b = 9.573$ (1) Å
$c = 11.243$ (1) Å
$\beta = 119.23$ (2)°
$V = 575.35$ (16) Å^3
$Z = 4$

$D_x = 1.886$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1024 reflections
$\theta = 1.7$–27.5°
$\mu = 1.48$ mm^{-1}
$T = 105$ (2) K
Needle, colourless
0.32 × 0.16 × 0.12 mm

Data collection

Bruker SMART diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.743$, $T_{\max} = 0.843$
10 831 measured reflections
2969 independent reflections

2569 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 37.5°$
$h = -10 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.081$
$S = 1.03$
2969 reflections
68 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.2185P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.63$ e Å^{-3}
$\Delta\rho_{\min} = -0.67$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.3049 (11)	C1—C2	1.5518 (12)
O2—C1	1.2162 (11)		
O2—C1—O1	126.88 (8)	O1—C1—C2	111.86 (8)
O2—C1—C2	121.25 (8)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2$^{\rm i}$	0.77 (2)	1.89 (2)	2.6615 (10)	175 (2)

Symmetry code: (i) $1 - x, 1 - y, -z$.

The H atom was located from a difference Fourier map and was included in the refinement with an isotropic displacement parameter.

Data collection: *SMART-NT* (Bruker, 1999); cell refinement: *SAINT-NT* (Bruker, 1999); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

KR thanks the UGC for the FIP programme. The authors thank the UGC for the DRS programme and the Bioinformatics Centre, Madurai Kamaraj University, for providing the Cambridge Structural Database (Allen, 2002).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bernstein, J., Etter, M. C. & Leiserowitz, L. (1994). *Structure Correlation*, edited by H.-B. Bürgi & J. D. Dunitz, Vol. 2, pp. 431–507. Weinheim: VCH.

Bruker (1998). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (1999). *SMART-NT* (Version 5.0) and *SAINT-NT* (Version 5.0). Bruker AXS Inc., Madison, Wisconsin, USA.

Desiraju, G. R. (1989). In *Crystal Engineering: The Design of Organic Solids*. Amsterdam: Elsevier.

Goldman, M. (1958). *J. Phys. Chem. Solids.* **7**, 165–169.

Kvenvolden, K. A., Lawless, J. G. & Ponnamperuma, C. (1971). *Proc. Natl Acad. Sci. USA*, **68**, 486–490.

Leiserowitz, L. (1976). *Acta Cryst.* B**32**, 775–802.

MacDonald, J. C. & Whitesides, G. M. (1994). *Chem. Rev.* **94**, 2383–2420.

Miller, S. L & Orgel, L. E. (1974). *The Origins of Life on the Earth*, p. 83. New Jersey: Prentice-Hall.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (1999). *PLATON for Windows*. University of Utrecht, The Netherlands.

Verbist, J. J., Lehmann, M. S., Koetzle, T. F. & Hamilton, W. C. (1972). *Nature* (*London*), **235**, 328–329.