

Data collection

Siemens SMART CCD	2522 independent reflections
System diffractometer	1964 reflections with
ω scans (1271 frames, 0.30°)	$I > 2\sigma(I)$
10 s, detector distance	$R_{\text{int}} = 0.060$
5.5 cm, detector angle	$\theta_{\text{max}} = 26.60^\circ$
23.0°)	$h = -17 \rightarrow 18$
Absorption correction: none	$k = -6 \rightarrow 8$
6368 measured reflections	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.452P]$
$R[F^2 > 2\sigma(F^2)] = 0.051$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.154$	$(\Delta/\sigma)_{\text{max}} = 0.033$
$S = 1.063$	$\Delta\rho_{\text{max}} = 0.376 \text{ e } \text{Å}^{-3}$
2522 reflections	$\Delta\rho_{\text{min}} = -0.171 \text{ e } \text{Å}^{-3}$
125 parameters	Extinction correction: none
H atoms treated by a	Scattering factors from
mixture of independent	<i>International Tables for</i>
and constrained refinement	<i>Crystallography</i> (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Si1—O1	1.6485 (16)	O1—C3	1.437 (3)
Si1—C6	1.852 (3)	C1—C2	1.168 (5)
Si1—C7	1.855 (3)	C2—C3	1.474 (5)
Si1—C8	1.874 (3)	C3—C5	1.527 (4)
O1—Si1—C6	111.92 (11)	C1—C2—C3	176.4 (3)
O1—Si1—C7	110.45 (12)	C2—C3—C4	110.2 (2)
O1—Si1—C8	103.73 (10)	C5—C3—C4	109.5 (2)
C3—O1—Si1	132.28 (18)	C4—C3—C5	113.8 (2)
C8—Si1—O1—C3	-168.9 (2)	C1—C2—C3—C5	66 (6)
Si1—O1—C3—C2	-18.2 (3)	C1—C2—C3—C4	-55 (6)
Si1—O1—C3—C5	103.2 (3)	O1—C3—C4—C4'	61.5 (4)
Si1—O1—C3—C4	-139.06 (19)	C2—C3—C4—C4'	-60.7 (4)
C1—C2—C3—O1	-174 (6)	C5—C3—C4—C4'	178.5 (3)

Symmetry code: (i) 2 - x, -y, 2 - z.

H-atom positions were calculated from idealized geometries and refined as riding on the corresponding C atoms.

Data collection: SMART (Siemens 1996a). Cell refinement: SAINT (Siemens 1996a). Data reduction: SAINT. Program(s) used to solve structure: SHELXS90 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997). Molecular graphics: SHELXTL (Siemens, 1996b). Software used to prepare material for publication: SHELXL97.

This work has been supported by the Swiss National Science Foundation (Project No. 20.43565.95). The diffraction data have been collected within an undergraduate trainee program, organized by Prof. H.-B. Bürgi, whom the authors thank for his generous support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1151). Services for accessing these data are described at the back of the journal.

References

- Allen, F. H., Bellard, S., Brice, M. D., Cartwright, B. A., Doubleday, A., Higgs, H., Hummelink, T., Hummelink-Peters, B. G., Kennard, O., Motherwell, W. D. S., Rodgers, J. R. & Watson, D. G. (1979). *Acta Cryst.* B35, 2331–2339.
- Boss, C. (1996). PhD thesis, Universität Bern, Switzerland.

- Boss, C. & Keese, R. (1996). *Helv. Chim. Acta*, 79, 2164–2175.
- Boss, C. & Keese, R. (1997). *Tetrahedron*, 53, 3111–3122.
- Boss, C., Keese, R. & Förtsch, M. (1996). *Acta Cryst.* C52, 2370–2372.
- Dale, J. (1978). *Stereochemie und Konformationsanalyse*, ch. 4. Weinheim: VCH.
- Meyer, M. & Keese, R. (1993). *Tetrahedron*, 49, 2055–2064.
- Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.
- Sheldrick, G. M. (1997). SHELXL97. Release 97-1. Program for the Refinement of Crystal Structures. University of Göttingen, Germany.
- Siemens (1996a). SMART and SAINT. Area Detector Control and Integration Software. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1996b). SHELXTL. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1998). C54, 384–386

Ammonium Hydrogen Tartronate

JUN-ICHIRO TAKA, SHINYA OGINO AND SETSUO KASHINO

Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700, Japan. E-mail: kashinos@cc.okayama-u.ac.jp

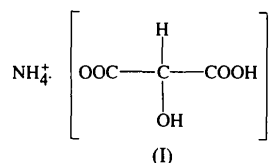
(Received 4 July 1997; accepted 3 November 1997)

Abstract

The title compound, ammonium hydrogen hydroxypropanedioate, NH₄⁺.C₃H₃O₅⁻, has a very short non-centrosymmetric O—H...O hydrogen bond [2.443 (2) Å] between the carboxyl and carboxylate groups. The hydroxyl group attached to C1 forms a bifurcated hydrogen bond with intra- and intermolecular O atoms. The NH₄⁺ cation forms six N—H...O hydrogen bonds including two bifurcated hydrogen bonds, by which a layer composed of the cations and anions is formed.

Comment

It has been found that acid salts of carboxylic acids form very short O—H...O hydrogen bonds (Macdonald *et al.*, 1972; Videnova *et al.*, 1980; Kroon & Kanters, 1982; Hermansson & Tellgren, 1983; Misaki *et al.*, 1986). The structure of ammonium hydrogen tartronate, (I), is expected to be suitable for a detailed investigation of the bonding behavior of the H atom, because the molecule contains only a few light atoms with low atomic numbers.



The molecular structure and crystal packing are shown in Fig. 1. The hydrogen-bond geometries are summarized in Table 1. The short O5—H5···O3 bond lies in a general position and, thus, it is not centrosymmetric. An elongated peak around the center of the O5···O3 bond was observed in a difference Fourier map drawn by omitting atom H5, and the least-squares refinement resulted in a long O5—H5 bond. The hydroxyl group, O1—H1O, links the layers formed by the cations and anions.

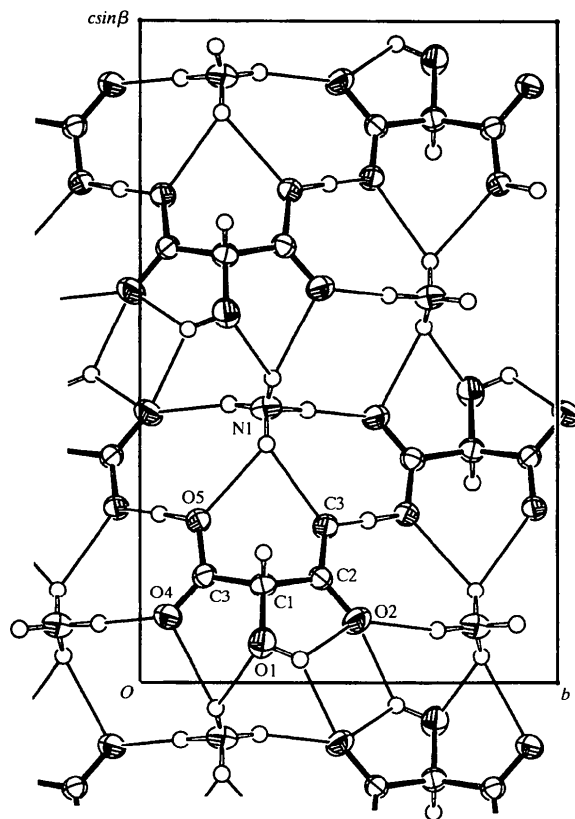


Fig. 1. Projection of the crystal structure viewed down the *a* axis with the numbering of atoms of the asymmetric unit. Hydrogen bonds are shown as thin solid lines.

The NH_4^+ ion donates hydrogen bonds to six O atoms, while the K^+ ion in potassium hydrogen tartronate, (II), in the same space group as (I) is coordinated by eight O atoms (Kroon & Kanters, 1982). This means that the hydrogen bonds govern the coordination around NH_4^+ more than the electrostatic interaction. This fact is reflected in the differences in the crystal structures and lattice constants between (I) and (II).

Experimental

Crystals were grown by slow evaporation from an aqueous solution containing equimolar amounts of tartronic acid (SIGMA

T-0657) and ammonia. A rather long crystal was used for diffraction measurement, because the crystals of (I) tended to grow as thin needles and crack when cut.

Crystal data

$\text{NH}_4^+ \cdot \text{C}_3\text{H}_3\text{O}_5^-$
 $M_r = 137.09$
 Monoclinic
 $P2_1/c$
 $a = 4.3226(6) \text{ \AA}$
 $b = 9.073(2) \text{ \AA}$
 $c = 14.284(2) \text{ \AA}$
 $\beta = 91.73(1)^\circ$
 $V = 560.0(2) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.626 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 9.5\text{--}11.0^\circ$
 $\mu = 0.158 \text{ mm}^{-1}$
 $T = 295 \text{ K}$
 Prismatic
 $0.75 \times 0.12 \times 0.10 \text{ mm}$
 Colorless

Data collection

Rigaku AFC-5R diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 1456 measured reflections
 1293 independent reflections
 1110 reflections with
 $I > 0.5\sigma(I)$
 $R_{\text{int}} = 0.012$

$\theta_{\text{max}} = 27.5^\circ$
 $h = 0 \rightarrow 5$
 $k = 0 \rightarrow 11$
 $l = -18 \rightarrow 18$
 3 standard reflections
 every 97 reflections
 intensity decay: none

Refinement

Refinement on F
 $R = 0.066$
 $wR = 0.055$
 $S = 1.22$
 1110 reflections
 111 parameters
 All H atoms refined
 $w = 1/\sigma^2(F)$
 $(\Delta/\sigma)_{\text{max}} = 0.05$

$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
 Extinction correction:
 $I_{\text{corr}} = I_0(1 + gI_c)$
 Extinction coefficient:
 0.328×10^{-5}
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O3 ⁱ	1.18 (3)	1.26 (3)	2.443 (2)	178 (3)
O1—H1O···O2 ⁱⁱ	0.90 (3)	2.06 (3)	2.775 (2)	136 (3)
O1—H1O···O2	0.90 (3)	2.17 (3)	2.654 (2)	113 (3)
N1—H1N···O2 ⁱ	0.95 (4)	2.10 (5)	3.044 (3)	169 (4)
N1—H2N···O3	0.88 (3)	2.21 (3)	2.892 (3)	135 (3)
N1—H2N···O5	0.88 (3)	2.24 (4)	2.910 (3)	133 (3)
N1—H3N···O4 ⁱⁱⁱ	1.08 (6)	1.82 (6)	2.901 (3)	177 (5)
N1—H4N···O4 ^{iv}	0.85 (4)	2.21 (4)	2.903 (3)	139 (3)
N1—H4N···O1 ^{iv}	0.85 (4)	2.40 (4)	3.129 (3)	145 (3)

Symmetry codes: (i) $-1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $-x, 1 - y, -z$; (iii) $-1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iv) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

Data collection and cell refinement were carried out with RASAI software (Rigaku Corporation, 1990). The structure was solved by direct methods using MITHRIL (Gilmore, 1984) and refined by full-matrix least-squares techniques using TEXSAN (Molecular Structure Corporation, 1985). H atoms were located from a difference Fourier map and refined isotropically. The displacement ellipsoids were drawn using ORTEPII (Johnson, 1976). Calculations were performed on a VAX 3100 computer using TEXSAN at the X-ray Laboratory of Okayama University, Japan.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1514). Services for accessing these data are described at the back of the journal.

References

- Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
 Hermansson, K. & Tellgren, R. (1983). *Acta Cryst.* **C39**, 1507–1510.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Kroon, J. & Kanters, J. A. (1982). *Croat. Chem. Acta*, **55**, 99–104.
 Macdonald, A. L., Speakman, J. C. & Hadži, D. (1972). *J. Chem. Soc. Perkin Trans. 2*, pp. 825–832.
 Misaki, S., Kashino, S. & Haisa, M. (1986). *Bull. Chem. Soc. Jpn.* **59**, 1059–1065.
 Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Rigaku Corporation (1990). *RASAIL. X-ray Data Collection Package*. Rigaku Corporation, Tokyo, Japan.
 Videnova, V., Baran, J., Glowiak, T. & Ratajczak, H. (1980). *Acta Cryst.* **B36**, 459–461.

Acta Cryst. (1998). **C54**, 386–387

1H-Indole-3-propionic Acid

NOBUO OKABE AND YUKARI ADACHI

Faculty of Pharmaceutical Sciences, Kinki University,
 Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan

(Received 18 July 1997; accepted 10 November 1997)

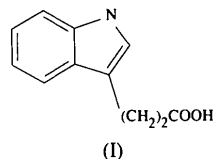
Abstract

The title compound, C₁₁H₁₁NO₂, has a fully extended propionic acid side chain. The molecules are held together as dimers by centrosymmetric pairs of intermolecular hydrogen bonds between the carboxyl groups. The O···O distance is 2.649 (2) Å.

Comment

The title compound, (I), has antibacterial activity against *Pseudomonas solanacearum* which causes a serious disease of many crop plants belonging to the family Solanaceae (Toyoda *et al.*, 1991; Matsuda *et al.*, 1993). It is also known as a hormone-type plant growth regulator like indole-3-acetic acid (Fargasova, 1994). Until now, its functional mechanism was unclear. Therefore, it is important to clarify its structure and conformation in order to investigate its function. Accordingly, the precise crystal structure of the title compound has been redetermined; its original structure determination was reported from film data, with an *R* value of 0.16 (Lahiri *et al.*, 1978). The non-

standard space group *P2*₁/*a* was chosen to agree with the original determination, which had unit-cell dimensions *a* = 12.38, *b* = 5.25, *c* = 14.39 Å and β = 95°.



The molecular structure of the title compound with the atomic labeling is shown in Fig. 1. The indole ring and the carboxyl group are in an *anti* conformation. The torsion angles, O(1)—C(12)—C(11)—C(10) −179.1 (1) and C(9)—C(3)—C(10)—C(11) −178.1 (2)°, indicate that the molecule has a nearly planar fully extended conformation. It may be important to take the planar conformation of indole-3-propionic acid into consideration for investigating the mechanism of the antibacterial or plant growth regulatory action. No stacking interactions between indole rings are observed. The crystal structure is stabilized by van der Waals interactions and hydrogen bonds between carboxyl groups, O(1)—H(3)···O(2)(1 − *x*, −*y*, −*z*) 2.649 (2) Å.

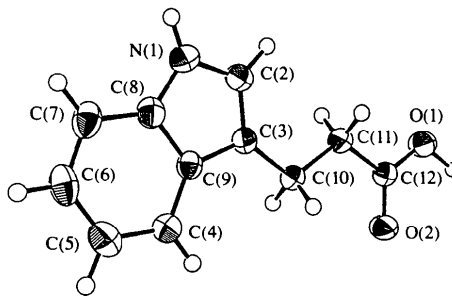


Fig. 1. *ORTEPII* (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Ellipsoids for non-H atoms correspond to 50% probability.

Experimental

The colorless pillar-shaped crystal used for analysis was obtained by the slow evaporation of a 90% ethanol solution of the title compound at room temperature.

Crystal data

C₁₁H₁₁NO₂
M_r = 189.21
 Monoclinic
*P2*₁/*a*
a = 12.346 (3) Å
b = 5.244 (3) Å
c = 14.362 (2) Å
 β = 94.77 (2)°
V = 926.6 (5) Å³
Z = 4
D_x = 1.356 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 20.89–24.11°
 μ = 0.088 mm⁻¹
T = 296 K
 Pillar
 0.4 × 0.3 × 0.2 mm
 Colorless