1580 measured reflections
1579 independent reflections
1310 observed reflections
[$I > 2.5\sigma(I)$]

2 standard reflections
monitored every 50
reflections
intensity variation: 3.0%

Refinement

Refinement on *F*
$R = 0.041$
$wR = 0.047$
$S = 3.92$
1310 reflections
127 parameters
All H-atom parameters
refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *International Tables*
for X-ray Crystallography
(1974, Vol. IV, Table
2.2B)

Table 7. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* AMEMONO

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
N1	0.30316 (23)	0.28987 (22)	0.36718 (18)	0.0374 (9)
C1	0.3439 (3)	0.4186 (3)	0.25940 (25)	0.0517 (14)
C2	0.3167 (3)	0.1075 (3)	0.32383 (21)	0.0332 (10)
O2	0.37205 (21)	0.06273 (19)	0.18203 (14)	0.0441 (9)
C21	0.3656 (4)	−0.1192 (3)	0.11716 (23)	0.0536 (14)
N3	0.28479 (22)	−0.01775 (21)	0.40813 (17)	0.0340 (9)
C4	0.2262 (3)	0.0379 (3)	0.55264 (20)	0.0308 (10)
N4	0.19334 (24)	−0.09096 (22)	0.63486 (18)	0.0392 (10)
C5	0.2032 (3)	0.2275 (3)	0.61155 (20)	0.0355 (10)
N5	0.1429 (3)	0.30054 (24)	0.75182 (19)	0.0458 (10)
O5	0.10369 (23)	0.19212 (21)	0.84278 (16)	0.0549 (10)
C6	0.2457 (3)	0.3593 (3)	0.51437 (21)	0.0391 (12)
O6	0.2321 (3)	0.52428 (21)	0.55220 (18)	0.0624 (11)
O1*W*	0.0973 (3)	0.70649 (24)	0.87500 (18)	0.0751 (13)

Table 8. *Selected geometric parameters* (Å, °) *for* AMEMONO

N1—C1	1.482 (3)	C4—N4	1.3140 (24)
N1—C2	1.3605 (24)	C4—C5	1.434 (3)
N1—C6	1.3965 (25)	C5—N5	1.3518 (24)
C2—O2	1.3182 (22)	C5—C6	1.444 (3)
C2—N3	1.3002 (24)	N5—O5	1.2663 (24)
O2—C21	1.4594 (25)	C6—O6	1.2257 (23)
N3—C4	1.3573 (23)		
N4···O6^i	2.8337 (23)	O*W*1···N5	3.067 (3)
N4···O5	2.6433 (23)	O*W*1···O5^{ii}	2.8784 (23)
C1—N1—C2	121.08 (16)	N3—C4—C5	121.18 (16)
C1—N1—C6	119.14 (15)	N4—C4—C5	122.24 (16)
C2—N1—C6	119.76 (15)	C4—C5—N5	126.66 (17)
N1—C2—O2	112.82 (16)	C4—C5—C6	118.68 (16)
N1—C2—N3	126.47 (17)	N5—C5—C6	114.66 (16)
O2—C2—N3	120.70 (16)	C5—N5—O5	117.80 (16)
C2—O2—C21	117.18 (15)	N1—C6—C5	116.03 (15)
C2—N3—C4	117.84 (15)	N1—C6—O6	119.41 (18)
N3—C4—N4	116.57 (15)	C5—C6—O6	124.55 (18)

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, 1-y, 2-z$.

Data collection and cell refinement: Nicolet *P*3 software. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Structure refinement: *NRCVAX*. Program used to solve structures: *SHELX*86 (Sheldrick, 1985). Programs used to produce molecular graphics: *PLUTON*92 (Spek, 1992).

JNL thanks Professor George Ferguson for his help.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1089). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Baker, B. R., Joseph, J. P. & Schaud, R. E. (1954). *J. Org. Chem.* **19**, 631–633.

Brown, D. J. (1970). Editor. *The Pyrimidines*, Suppl. 1. *The Chemistry of Heterocyclic Compounds*, No. 16, consulting editor A. Weissberger. New York: Wiley Interscience.

Dattagupta, J. K., Kroger, M. & Saenger, W. (1977). *Chem. Ber.* **110**, 353–354.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Hendrix, J. (1915). *J. Biol. Chem.* **20**, 153–155.

Hueso-Ureña, F., Moreno-Carretero, M. N., Romero-Molina, M. A., Salas-Peregrin, J. M., Sanchez-Sanchez, M. P., Alvarez de Cienfuegos, G. & Faure, R. (1992). *J. Inorg. Biochem.* In the press.

Kivekäs, R., Sundberg, M. R., Ruiz, J. & Colacio, E. (1991). *Acta Cryst.* C**47**, 1512–1515.

Ladd, M. F. (1979). *Structure and Bonding in Solid State Chemistry*. New York: John Wiley.

Low, J. N., Howie, R. A., Hueso-Ureña, F. & Moreno-Carretero, M. N. (1992). *Acta Cryst.* C**48**, 145–147.

Moreno-Carretero, M. N., Salas-Peregrin, J. M., Colacio-Rodriguez, E., Sanchez-Sanchez, M. P. & Nieto-Garcia, F. (1986). *Acta Cryst.* C**42**, 407–410.

Romero-Molina, M. A., Moreno-Carretero, M. N., Ruiz-Sanchez, J., Sanchez-Sanchez, M. P. & Nieto-Garcia, F. (1986). *Inorg. Chem.* **25**, 1498–1501.

Romero-Molina, M. A., Salas-Peregrin, J. M., Lopez-Garzon, R., Gutierrez-Valero, M. D., Pannerselvam, K., Chacko, K. K., Aoki, K. & Yamazaki, H. (1990). *Inorg. Chim. Acta*, **172**, 253–257.

Sheldrick, G. M. (1985). *SHELX*86. *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Spek, A. L. (1992). *PLUTON*92. *Molecular Graphics Program*. Univ. of Utrecht, The Netherlands.

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Acetamide Hydrochloride

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Abstract

The title compound, acetamide hydrochloride (I), $C_2H_6NO^+.Cl^-$, has a planar network of infinite hydrogen bonds connecting the $C_2H_6NO^+$ cation to three different Cl^- anions and *vice versa*, *via* one O—H···Cl and two N—H···Cl hydrogen bonds.

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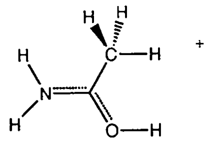
Comment

All atoms, except one H atom of the methyl group, are strictly coplanar and located on the mirror plane at $y = \frac{1}{4}$. The exchanging protons in acetamide hydrochloride are all involved in a planar hydrogen-bonding network. The three hydrogen bonds around the Cl^- anion, namely, O—H6···Cl, N—H4···Cl and N—H5···Cl, have angles about Cl of 118 (2), 108 (2) and 134 (1)°, respectively. The geometry of the O/N—H···Cl interactions is virtually linear indicating the subtle balance among the three hydrogen bonds. Acetamide nitric acid (Gubin *et al.*, 1980) exhibits an infinite chain of hydrogen bonds connecting, alternately, the $C_2H_6NO^+$ cations and the NO_3^- anions in the solid state. Bis(acetamide) nitric acid (Gubin, Buranbaev & Nurakhmetov, 1988) has a very strong O—H···O hydrogen bond connecting two acetamide units. Tri(acetamide) nitric acid (Gubin *et al.*, 1986) has one localized O—H···O hydrogen bond connecting two acetamide units as in the bis(acetamide) system. For the latter two compounds, there are also weaker hydrogen bonds involving acetamide and acetamide/nitrate.



(I)

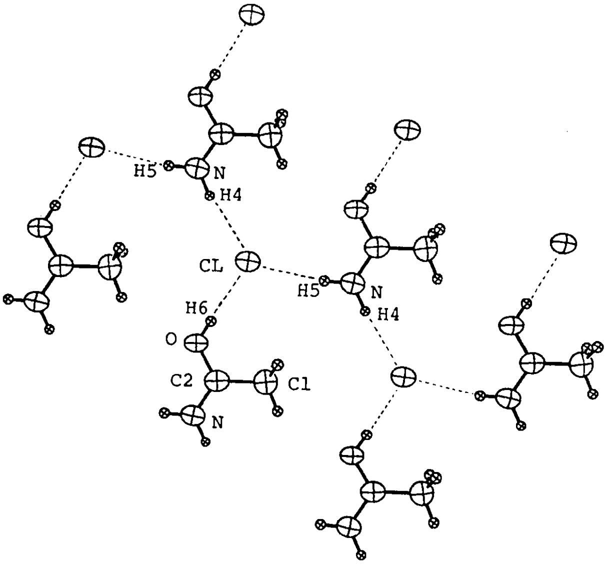


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the planar hydrogen-bonding network of acetamide hydrochloride. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Crystals of the title compound (I) were prepared by adding trimethylsilyl chloride (93 µl, 1 mmol) and water (27 µl, 1.5 mmol) sequentially to acetonitrile (0.4 ml) at 298 K. After standing for 12 h at room temperature, colorless moisture-sensitive crystals (m.p. 395–396 K) of acetamide hydrochloride were precipitated. ^1H NMR ($CDCl_3$ + 20% MeOH-d_4, TMS): δ 2.41 (*s*, 3H), 6.8–7.3 (*s*, broad, 2H), 8.0–10.5 (*s*, broad, 1H); ^{13}C NMR ($CDCl_3$ + 20% MeOH-d_4, TMS): δ 18.94, 178.48; MS: *m/z* 60 (M^+-35). Elemental analysis: calculated for C_2H_6ClNO, C 25.15, H 6.33, N 14.66%; found, C 25.20, H 6.59, N 14.62%. A prismatic crystal, obtained from slow evaporation of acetonitrile solution at 298K, was sealed in a lithium glass capillary for all the measurements.

Crystal data

$C_2H_6NO^+.Cl^-$
$M_r = 95.53$
Monoclinic
$P2_1/m$
$a = 5.8376$ (9) Å
$b = 6.4746$ (10) Å
$c = 6.2534$ (10) Å
$\beta = 104.089$ (5)°
$V = 229.24$ (6) Å^3
$Z = 2$
$D_x = 1.384$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 15.00$–$39.04°$
$\mu = 0.66$ mm^{-1}
$T = 298$ K
Prism
0.50 × 0.50 × 0.45 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (ψ scan)
$T_{min} = 0.914$, $T_{max} = 0.998$
365 measured reflections
332 independent reflections

315 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.015$
$\theta_{max} = 22.5°$
$h = -6 \rightarrow 6$
$k = 0 \rightarrow 6$
$l = 0 \rightarrow 6$
7 standard reflections
frequency: 120 min
intensity variation: 6%

Refinement

Refinement on F
$R = 0.030$
$wR = 0.042$
$S = 3.14$
315 reflections
48 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.0001F^2]$
$(\Delta/\sigma)_{max} = 0.040$

$\Delta\rho_{max} = 0.140$ e Å^{-3}
$\Delta\rho_{min} = -0.300$ e Å^{-3}
Extinction correction: secondary
Extinction coefficient: 1.50 (22)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

U_{iso} for H atoms; $U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	U_{iso}/U_{eq}
Cl	0.73632 (12)	1/4	0.03971 (12)	0.046 (6)
C(1)	0.1825 (7)	1/4	−0.3864 (6)	0.047 (2)
C(2)	0.3541 (5)	1/4	−0.5244 (5)	0.036 (1)
N	0.2911 (6)	1/4	−0.7364 (5)	0.046 (1)
O	0.5795 (4)	1/4	−0.4355 (4)	0.049 (1)

H(1)	0.021 (8)	1/4	−0.470 (7)	0.066 (9)
H(2)	0.215 (5)	0.375 (4)	−0.286 (4)	0.077 (7)
H(4)	0.135 (8)	1/4	−0.810 (7)	0.054 (8)
H(5)	0.398 (8)	1/4	−0.810 (7)	0.065 (10)
H(6)	0.619 (7)	1/4	−0.304 (8)	0.066 (11)

Table 2. *Selected geometric parameters* (Å, °)

C(1)—C(2)	1.473 (5)	C(2)—O	1.297 (4)
C(1)—H(1)	0.96 (4)	N—H(4)	0.92 (4)
C(1)—H(2)	1.02 (3)	N—H(5)	0.86 (5)
C(2)—N	1.287 (4)	O—H(6)	0.80 (5)
Cl···H(4^{i})	2.29 (4)	Cl···H(6)	2.09 (5)
Cl···H(5^{ii})	2.39 (5)		
C(1)—C(2)—N	122.6 (3)	N—C(2)—O	116.6 (3)
C(1)—C(2)—O	120.8 (3)		
H(4^{i})···Cl···H(5^{ii})	134.1 (14)	Cl^{iii}···H(4)—N	174 (3)
H(4^{i})···Cl···H(6)	118.0 (15)	Cl^{iv}···H(5)—N	171 (4)
H(5^{ii})···Cl···H(6)	108.0 (15)	Cl···H(6)—O	177 (4)

Symmetry codes: (i) $1 + x, y, 1 + z$; (ii) $x, y, 1 + z$; (iii) $x - 1, y, z - 1$; (iv) $x, y, z - 1$.

Computations were carried out on a MicroVAX 3600 computer with the *NRC* package (Gabe, Lee & Le Page, 1985). Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *NRC*. Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *NRC*. Molecular graphics: *ORTEP* (Johnson, 1976). Software used to prepare material for publication: *NRC*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1105). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf-Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.

Gabe, E. J., Lee, F. L. & Le Page, Y. (1985). *Crystallograhic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 167–174. Oxford: Clarendon Press.

Gubin, A. I., Buranbaev, M. Zh. & Nurakhmetov, N. N. (1988). *Kristallografiya*, 33, 506–508.

Gubin, A. I., Yanovskii, A. I., Struchkov, Yu. T., Beremzhanov, B. A., Nurakhmetov, N. N. & Buranbaev, M. Zh. (1980). *Cryst. Struct. Commun.* **9**, 745–748.

Gubin, A. I., Yanovskii, A. I., Struchkov, Yu. T., Nurakhmetov, N. N., Buranbaev, M. Zh. & Beremzhanov, B. A. (1986). *Izv. Akad. Nauk Kaz. SSR Ser. Khim.* pp. 78–81.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

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2,3,4,5,6-Penta-*O*-acetyl-D-galactonic Acid 1′-Phenyl-2′-ethoxycarbonylvinyl Ester

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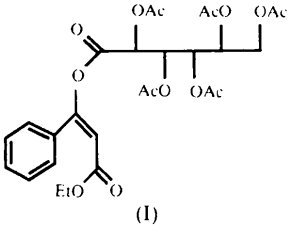
Abstract

In the galactonic acid part of the title compound, $C_{27}H_{32}O_{14}$, two C—O bonds are aligned 1,3-parallel with an O···O distance of 2.884 (2) Å. The absolute structure, determined crystallographically, corresponds to the known chirality of the title compound.

Comment

The title compound (I) was synthesized by reacting penta-*O*-acetyl-D-galactonyl chloride with ethyl benzoylacetate in the presence of a base in an aprotic solvent (Meisel, 1991). In order to characterize the resulting reaction product unambiguously, an X-ray structure determination was undertaken. Suitable single crystals, melting at 403–405 K, were obtained from ethanol. The positions of all H atoms were refined with distance restraints for the C—H distances (Sheldrick, 1993). All other distances and angles are generally as expected. Friedel opposites were collected after each reflection. These Friedel pairs are regarded as symmetry independent and were not merged. Since the title compound crystallizes in a polar space group, polar axis restraints were applied by the method of Flack & Schwarzenbach (1988).



(I)

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