9529 measured reflections
8968 independent reflections

3 standard reflections
frequency: 120 min
intensity decay: 0.2%

Refinement

Refinement on F
$R = 0.057$
$wR = 0.039$
$S = 1.243$
6584 reflections
674 parameters
H atoms refined isotropically
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$
$(\Delta/\sigma)_{\max} = 0.028$

$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Extinction correction: isotropic (Zachariasen, 1963)
Extinction coefficient: $1.9(3) \times 10^{-8}$
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

O1*A*—C3*A*	1.419 (5)	O1*B*—C3*B*	1.397 (5)
O1*A*—C4*A*	1.416 (5)	O1*B*—C4*B*	1.417 (5)
O2*A*—C7*A*	1.423 (5)	O2*B*—C7*B*	1.413 (5)
O2*A*—C8*A*	1.424 (5)	O2*B*—C8*B*	1.417 (5)
C1*A*—C2*A*	1.577 (6)	C1*B*—C2*B*	1.576 (6)
C5*A*—C6*A*	1.585 (6)	C5*B*—C6*B*	1.576 (6)
C10*A*—C11*A*	1.314 (6)	C10*B*—C11*B*	1.309 (6)
C15*A*—C16*A*	1.305 (6)	C15*B*—C16*B*	1.318 (6)
C3*A*—O1*A*—C4*A*	115.1 (3)	C3*B*—O1*B*—C4*B*	116.4 (3)
C7*A*—O2*A*—C8*A*	114.2 (3)	C7*B*—O2*B*—C8*B*	115.6 (3)
Cl9*A*—C13*A*—Cl10*A*	107.2 (2)	Cl9*B*—C13*B*—Cl10*B*	107.3 (2)
Cl11*A*—C18*A*—Cl12*A*	108.3 (2)	Cl11*B*—C18*B*—Cl12*B*	107.6 (2)
C8*A*—C1*A*—C2*A*—C3*A*	8.1 (5)	C8*B*—C1*B*—C2*B*—C3*B*	1.5 (6)
C9*A*—C1*A*—C2*A*—C12*A*	1.0 (4)	C9*B*—C1*B*—C2*B*—C12*B*	1.3 (4)
C4*A*—C5*A*—C6*A*—C7*A*	2.5 (5)	C4*B*—C5*B*—C6*B*—C7*B*	2.9 (5)
C14*A*—C5*A*—C6*A*—C17*A*	0.2 (4)	C14*B*—C5*B*—C6*B*—C17*B*	1.0 (4)

C—H distances are in the range 0.89 (3)–1.03 (3) Å, while $B_{\rm iso}$ values for H atoms are in range the 1.0 (7)–6 (1) Å^2.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF IN* in *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1349). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 647–649

Adenosine-5′-carboxylic Acid

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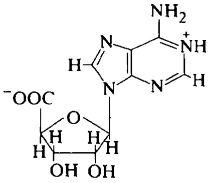
Abstract

The two molecules in the asymmetric unit of adenosine-5′-carboxylic acid, $C_{10}H_{11}N_5O_5$, exist as zwitterions with N1 protonated and the carboxyl groups ionized. Both molecules are in an *anti* conformation with glycosyl torsion angles of −161.4 (3) and −155.5 (3)°. The ribose moieties adopt a C3-*endo*–C2-*exo* twist conformation. The pseudo-rotation parameters are P =

0.01 (1) and 6.58 (1)°, and τ_m = 36.2 (2) and 34.6 (2)°, for molecules *A* and *B*, respectively. The carboxyl groups of *A* and *B* are not in the standard g^+, g^- or *t* conformations. Both Watson–Crick sites, N1 and N6, of the adenine bases are involved in a pair of hydrogen bonds with the dissociated carboxyl groups, forming a cyclic tetramer. The adenine base of molecule *A* stacks on the ribose O4′ atom of a symmetry-related *B* molecule at a distance of 2.88 Å; the adenine base of *B* stacks in an analogous way at a distance of 2.91 Å.

Comment

As part of our systematic X-ray studies on ribose/deoxyribose nucleosides and nucleotides (Padiyar & Seshadri, 1996; Mande *et al.*, 1994; Krishnan & Seshadri, 1992), we report here the crystal structure of adenosine-5′-carboxylic acid, A5CA.



A5CA

The atomic numbering and conformation of the two A5CA molecules in the asymmetric unit is shown in Fig. 1. A5CA exists as a zwitterion with N1 protonated and the carboxyl group ionized. Both molecules *A* and *B* have an *anti* conformation as indicated by the glycosyl torsion angles (C4—N9—C1′—O4′) for *A* and *B* of −161.2 (3) and −155.1 (4)°, respectively. The ribose moieties of molecules *A* and *B* adopt a C3′-*endo*–C2′-*exo* twist conformation. Atoms C3′ and C2′ are displaced by 0.30 (4) and 0.30 (4) Å (molecule *A*), and 0.39 (4) and 0.19 (4) Å (molecule *B*), respectively, on either side of the plane formed by atoms C1′, C4′ and O4′. The pseudo-rotation parameters (Altona & Sundaralingam, 1972) are *P* = 0.01 (1) and 6.58 (1)°, and τ_m = 36.2 (2) and 34.6 (2)°, for molecules *A* and *B*, respectively. The two carboxyl O atoms, O5′1 and O5′2, of both molecules are not in the usual g^+, g^- or *t* conformations displayed by the O5′ atom in unmodified nucleosides and nucleotides, but are about 35° away from these configurations, close to the values reported for thymidine-5′-carboxylic acid (Suck *et al.*, 1974). The adenine bases are planar: the largest deviations are for atoms C5*A* and C6*B*, which are displaced by 0.018 (4) and 0.010 (4) Å from their respective mean planes.

The adenine base of molecule *A* stacks on the ribose O4′ atom of a symmetry-related *B* molecule at a distance of 2.88 Å (O4′*A*···C2*B*); the analogous distance for the adenine base of molecule *B* is 2.91 Å (O4′*B*···N1*A*).

A noteworthy feature of the hydrogen bonding in A5CA, which is summarized in Table 1, is that both the Watson–Crick sites (N1 and N6) of the adenine bases of molecules *A* and *B* are involved in a pair of hydrogen bonds with the dissociated carboxyl groups, thus forming a cyclic tetramer. Other structures displaying a pair of hydrogen bonds between N1 and N6 of the adenine base and the carboxyl group include 5′-deoxy-5′-adenosineacetic acid (Ishida *et al.*, 1983) and *N*-(9-β-D-ribofuranosylpurine-6-yl)glycylalanine (Narayanan *et al.*, 1976).

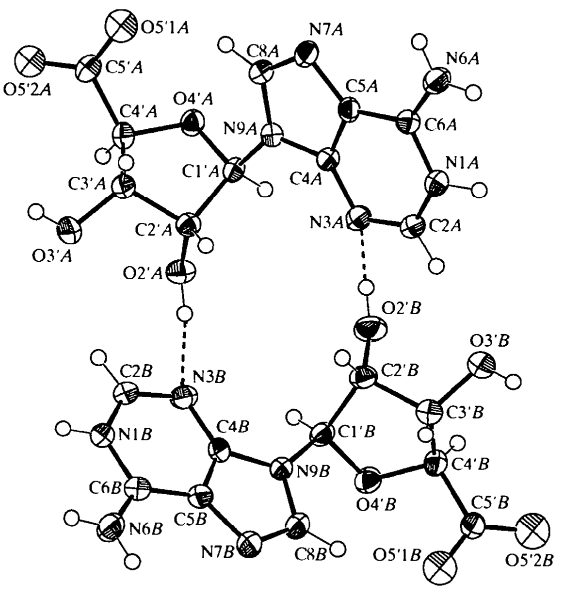


Fig. 1. The atomic numbering and molecular conformation of A5CA shown with 50% probability displacement ellipsoids.

Experimental

Crystals of A5CA were grown by direct evaporation from an aqueous solution of the compound containing a trace of ammonium hydroxide.

Crystal data

$C_{10}H_{11}N_5O_5$
M_r = 281.23
Orthorhombic
$P2_12_12_1$
a = 14.435 (2) Å
b = 18.025 (2) Å
c = 8.5494 (9) Å
V = 2224.4 (4) Å^3
Z = 8
D_x = 1.68 Mg m^{-3}
D_m not measured

Mo *K*α radiation
λ = 0.71067 Å
Cell parameters from 25 reflections
θ = 6–12°
μ = 0.138 mm^{-1}
T = 293 (2) K
Parallelepiped
0.35 × 0.20 × 0.20 mm
Colourless

Data collection

Rigaku AFC-7 diffractometer
ω–2θ scans
Absorption correction: none

θ_{max} = 24.99°
h = 0 → 17
k = 0 → 21
l = 0 → 10

2236 measured reflections
2236 independent reflections
1590 reflections with
$I > 2\sigma(I)$

3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.038$
$wR(F^2) = 0.098$
$S = 1.081$
2236 reflections
274 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.1143P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.208$ e Å^{-3}
$\Delta\rho_{\min} = -0.246$ e Å^{-3}
Extinction correction:
*SHELXL*93 (Sheldrick, 1993
Extinction coefficient:
0.0017 (6)
Scattering factors from
International Tables for Crystallography (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N6A—H1N6A···O5′2$A^{\rm i}$	0.86	2.13	2.942 (4)	157.6
N6A—H2N6A···O5′2$B^{\rm ii}$	0.86	2.01	2.766 (4)	146.3
N6B—H1N6B···O5′2$B^{\rm iii}$	0.86	2.04	2.892 (4)	171.1
N6B—H2N6B···O5′2$A^{\rm iv}$	0.86	2.12	2.844 (4)	141.2
N1A—HN1A···O5′1$A^{\rm i}$	0.86	1.78	2.596 (4)	157.4
N1B—HN1B···O5′1$B^{\rm iii}$	0.86	1.73	2.567 (4)	164.8
O2′A—HO2′A···N3B	0.90	1.94	2.831 (4)	169.3
O2′B—HO2′B···N3A	0.90	2.12	2.957 (4)	153.6
O3′A—HO3′A···N7$B^{\rm iii}$	0.86	2.07	2.855 (4)	151.2
O3′B—HO3′B···N7$A^{\rm i}$	0.77	2.23	2.950 (4)	155.1

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{5}{2} - z$; (ii) $1 - x, y - \frac{1}{2}, \frac{5}{2} - z$; (iii) $2 - x, y - \frac{1}{2}, \frac{5}{2} - z$; (iv) $2 - x, \frac{1}{2} + y, \frac{5}{2} - z$.

Data collection: *AFC/MSC* (Rigaku, 1991). Cell refinement: *AFC/MSC*. Data reduction: *AFC/MSC*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *INSIGHT*II (Biosym Technologies, 1995) and *Xtal_GX* (Hall & du Boulay, 1995). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1070). Services for accessing these data are described at the back of the journal.

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Ammonium Saccharin

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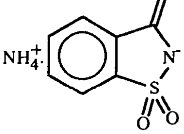
Abstract

The crystal structure of ammonium saccharin, NH_4^+.$C_7H_4NO_3S^-$, consists of ammonium and *o*-sulfobenzoimidate ion pairs linked by hydrogen bonds into a two-dimensional layer structure.

Comment

The artificial sweetener saccharin (3*H*-benzisothiazol-3-one 1,1-dioxide) in its deprotonated form can form complexes with a large number of metal ions (Schulze & Illegen, 1997). The ammonium saccharin salt, (I), has been prepared with the intention of using it for reaction with organometallic halides, the alternative reagent sodium saccharin being insoluble in alcoholic solvents.



(I)

The 3-oxo-3*H*-benzisothiazolyl group is flat and bond dimensions involving the anion are similar to those found in both the parent acid (Bart, 1968; Okaya, 1969) and the *N*,*N*-dimethyltolylammonium salt, which is air sensitive (Courseille *et al.*, 1991). The *N*,*N*-dimethyltolylammonium salt has only one hydrogen bond, *i.e.* between the N and carbonyl O atoms [2.767 (3) Å],