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*
N6*A*—H1N6*A*···O5′2*A*^{i}	0.86	2.13	2.942 (4)	157.6
N6*A*—H2N6*A*···O5′2*B*^{ii}	0.86	2.01	2.766 (4)	146.3
N6*B*—H1N6*B*···O5′2*B*^{iii}	0.86	2.04	2.892 (4)	171.1
N6*B*—H2N6*B*···O5′2*A*^{iv}	0.86	2.12	2.844 (4)	141.2
N1*A*—HN1*A*···O5′1*A*^{i}	0.86	1.78	2.596 (4)	157.4
N1*B*—HN1*B*···O5′1*B*^{iii}	0.86	1.73	2.567 (4)	164.8
O2′*A*—HO2′*A*···N3*B*	0.90	1.94	2.831 (4)	169.3
O2′*B*—HO2′*B*···N3*A*	0.90	2.12	2.957 (4)	153.6
O3′*A*—HO3′*A*···N7*B*^{iii}	0.86	2.07	2.855 (4)	151.2
O3′*B*—HO3′*B*···N7*A*^{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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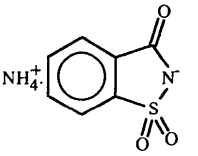
Abstract

The crystal structure of ammonium saccharin, $NH_4^+ \cdot C_7H_4NO_3S^-$, 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],

the distance being much shorter than for the hydrogen bonds found in the present ammonium salt. The ammonium ion forms hydrogen bonds to the sulfonyl O atoms of adjacent saccharin ions [N···O 2.881 (2) and 2.942 (2) Å]; these are somewhat longer than those between the ammonium ion and the carbonyl O atoms [N···O 2.785 (2) and 2.834 (2) Å], in agreement with the fact that the carbonyl group is a more basic site than the sulfonyl group. These four hydrogen bonds link the ion pairs into a layer structure.

The saccharin ion confers significant Lewis acceptor properties to tin in the triphenylstannyl derivative, allowing *N*-triphenylstannyl saccharin (Ng *et al.*, 1992) to form O-bonded adducts with ethanol (Ng *et al.*, 1989) and a number of O-donor ligands (Ng, 1996). Interestingly, with the cyclic ketone 1,2-diphenylcyclopropenone as the donor, the saccharin ion isomerizes to the 3-oxybenzisothiazole 1,1-dioxide ion (Ng *et al.*, 1993), which bonds to tin through its O-atom end.

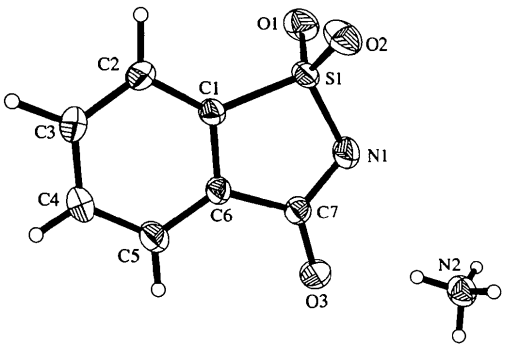


Fig. 1. *ORTEP*II (Johnson, 1976) plot of ammonium saccharin at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

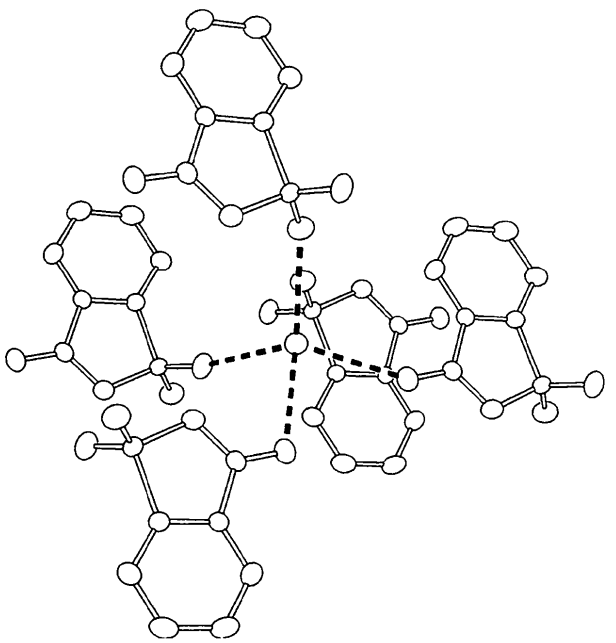


Fig. 2. Hydrogen bonding in ammonium saccharin.

Experimental

Aqueous ammonium hydroxide was added to saccharin dissolved in ethanol to precipitate ammonium saccharin, which was collected and recrystallized from ethanol.

Crystal data

$NH_4^+.C_7H_4NO_3S^-$
M_r = 200.21
Triclinic
$P\bar{1}$
a = 7.1513 (6) Å
b = 8.2054 (6) Å
c = 8.3385 (6) Å
α = 117.264 (6)°
β = 96.049 (6)°
γ = 100.497 (6)°
V = 417.56 (6) Å^3
Z = 2
D_x = 1.592 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 12–13°
μ = 0.361 mm^{-1}
T = 298 (2) K
Triangular block
0.55 × 0.55 × 0.55 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scans (North *et al.*, 1968)
T_{min} = 0.723, T_{max} = 0.820
2457 measured reflections
2421 independent reflections

2180 reflections with $I > 2\sigma(I)$
R_{int} = 0.002
θ_{max} = 29.96°
h = 0 → 10
k = −11 → 11
l = −11 → 11
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.101
S = 1.067
2421 reflections
151 parameters
H atoms were located and refined
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.1022P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.17 (1)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *CAD*-4 *VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX/PC*. Data reduction: *Xtal*3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). 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: KA1255). Services for accessing these data are described at the back of the journal.

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Absolute Configuration of (+)-1-Phenyl-1,2,3,4-tetrahydroisoquinoline Hydrochloride

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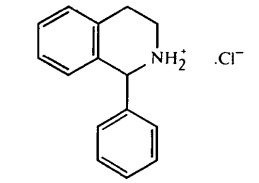
Abstract

(+)-1-Phenyl-1,2,3,4-tetrahydroisoquinoline with an $[\alpha]_D^{25}$ value of +47.58° in CCl_4 (*c* 2.83) has been synthesized and the crystal structure of its hydrochloride, $C_{15}H_{16}N^+.Cl^-$, determined by X-ray methods. The anomalous dispersion effect indicates that the absolute configuration is *S*.

Comment

1-Phenyl-1,2,3,4-tetrahydroisoquinoline (PTIQ) is a basic compound for the development of new drugs. The absolute configuration of (−)-PTIQ has been assigned to be *S* (Yamato *et al.*, 1990). However, our related compound suggested that (+)-PTIQ has the *S* configuration (Nakahara *et al.*, 1997). In order to confirm the absolute configuration, an X-ray diffraction study of the title compound, (I), was undertaken.



(I)

The optically resolved PTIQ we prepared had an $[\alpha]_D^{25}$ value of +47.58° in CCl_4 (*c* 2.83) indicating that it is the enantiomer of (−)-PTIQ (Leithe, 1929; Yamato *et al.*, 1990). Compound (I) crystallizes in the monoclinic space group $P2_1$ and the asymmetric unit contains two independent molecules, *A* and *A'*, having almost the same three-dimensional structure, with an r.m.s. deviation of 0.026 Å. They are related by a pseudo-twofold symmetry; their fractional atomic coordinates are related by the following equations: $x = -0.9995x' - 0.0117y' - 0.0389z' - 0.4600$, $y = -0.0027x' + 0.9999y' - 0.0330z' - 0.4185$, $z =$

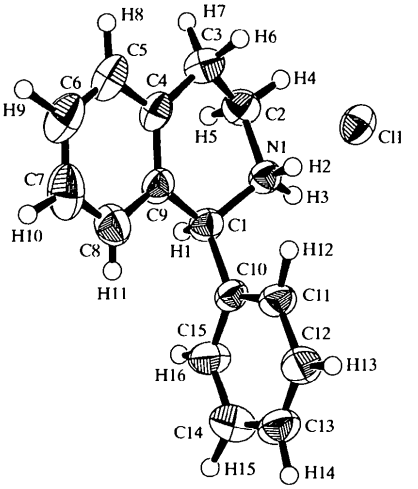


Fig. 1. *ORTEP*II drawing (Johnson, 1976) of molecule *A* of (I) with the atomic numbering. Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as spheres of arbitrary size.