dation of Jiangsu Province and the Malaysian National Science Council for R&D (Grant No. 09-02-03-0004).

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: TA1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Boens, N., De Schryver, F. C. & Smets, G. (1975). *J. Polym. Sci. Polym. Chem. Ed.* **13**, 201–213.

Brockmann, T. W. & Tour, J. M. (1995). *J. Am. Chem. Soc.* **117**, 4437–4447.

Enraf–Nonius (1988). *CAD-4 Manual.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal3.0 Reference Manual.* Universities of Western Australia, Australia, and Maryland, USA.

Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Marsh, R. E., Ubell, E. & Wilcox, H. E. (1962). *Acta Cryst.* **15**, 35–41.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sheldrick, G. M. (1987). *SHELXTL-Plus.* PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Vrabel, V., Kello, E., Lokaj, J. & Konecny, V. (1989). *Collect. Czech. Chem. Commun.* **54**, 2408–2414.

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Hydronium 2-Carboxybenzenesulfonate

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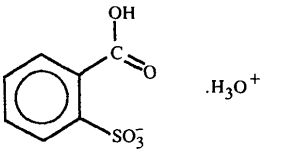
Abstract

2-Sulfobenzoic acid hydrate exists as the title salt, oxonium 2-carboxybenzenesulfonate, $H_3O^+.C_7H_5O_5S^-$, and is isomorphous with the ammonium derivative.

Comment

2-Sulfobenzoic acid as procured from commercial sources (Aldrich Chemical Company) has the formula $HO_3SC_6H_4CO_2H.xH_2O$, where x is probably equal to 3, as implied from the trihydrated product obtained by crystallizing ammonium hydrogen 2-sulfobenzoate (Okaya, 1967) from an aqueous acidic medium (Attig & Mootz, 1976). The commercial acid, when recrystallized from an ethanol solution containing excess dicyclohexylamine, afforded dicyclohexylammonium hydrogen 2-sulfobenzoate dihydrate (Ng, 1995). In the present study, the acid was recrystallized from ethanol to give 2-sulfobenzoic acid monohydrate, which formally exists as hydronium 2-carboxybenzenesulfonate, (I).



(I)

The hydrogen 2-sulfobenzoate anions are linked into a chain along the *b* axis by a strong hydrogen bond involving the carboxyl O5 and sulfonyl O3 atoms [O5···O3 2.661 (3) Å]. The H_3O^+ cations surround the chains and are hydrogen bonded to adjacent chains [O···O 2.890 (3)–3.060 (3) Å] resulting in a network structure. The title compound is isomorphous with the ammonium hydrogen analog.

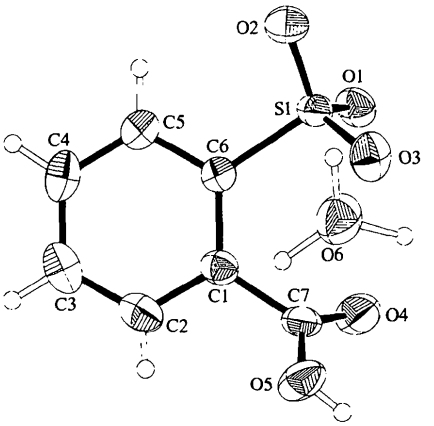


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of hydronium 2-carboxybenzenesulfonate at the 50% probability level. H atoms are drawn as small circles of arbitrary radii.

Experimental

2-Sulfobenzoic acid hydrate ($HO_3SC_6H_4$-2-$CO_2H.xH_2O$), purchased from the Aldrich Chemical Company, was recrystallized from ethanol.

Crystal data

$H_3O^+.C_7H_5O_5S^-$
M_r = 220.19

Mo $K\alpha$ radiation
λ = 0.71073 Å

Orthorhombic
Pbca
$a = 10.5774(4)$ Å
$b = 7.0688(3)$ Å
$c = 25.620(1)$ Å
$V = 1915.6(1)$ Å^3
$Z = 8$
$D_x = 1.527$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 12.0$–$13.0°$
$\mu = 0.339$ mm^{-1}
$T = 300$ K
Block
$0.43 \times 0.36 \times 0.29$ mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.874$, $T_{max} = 0.906$
1681 measured reflections
1681 independent reflections

1381 reflections with $I > 2\sigma(I)$
$\theta_{max} = 24.96°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 8$
$l = -30 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: 0.4%

Refinement

Refinement on F^2
$R(F) = 0.0393$
$wR(F^2) = 0.1121$
$S = 1.032$
1681 reflections
159 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.7926P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.225$ e Å^{-3}
$\Delta\rho_{min} = -0.320$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

S1—O1	1.448 (2)	C1—C7	1.502 (3)
S1—O2	1.441 (2)	O5···O3^{i}	2.661 (3)
S1—O3	1.456 (2)	O6···O1	2.900 (3)
S1—C6	1.779 (2)	O6···O2^{ii}	2.890 (3)
O4—C7	1.200 (3)	O6···O3^{iii}	3.060 (3)
O5—C7	1.312 (3)		
O1—S1—O2	112.4 (1)	C6—C1—C7	123.7 (2)
O1—S1—O3	110.8 (1)	C5—C6—S1	117.8 (2)
O2—S1—O3	113.9 (1)	C1—C6—S1	122.3 (2)
O1—S1—C6	107.0 (1)	O4—C7—O5	124.5 (2)
O2—S1—C6	105.7 (1)	O4—C7—C1	123.1 (2)
O3—S1—C6	106.6 (1)	O5—C7—C1	112.2 (2)
C2—C1—C7	117.6 (2)		

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

Data collection: *CAD-4 VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD-4 VAX/PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1996). Software used to prepare material for publication: *SHELXL93*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KH1131). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Attig, R. & Mootz, D. (1976). *Acta Cryst.* B**32**, 435–439.
Enraf–Nonius (1988). *CAD-4 VAX/PC Fortran System. Operator's Guide to the Enraf–Nonius CAD-4 Diffractometer Hardware, its Software and the Operating System.* Enraf–Nonius, Delft, The Netherlands.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Ng, S. W. (1995). *Acta Cryst.* C**51**, 1853–1855.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Okaya, Y. (1967). *Acta Cryst.* **22**, 104–110.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Zsolnai, L. & Pritzkow, H. (1996). *ZORTEP. Molecular Graphics Program.* University of Heidelberg, Germany.

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Heterocyclic *N*-Acetoxyarylamines, Models for the Putative Ultimate Carcinogens of Aromatic Amines: 2-Acetoxyamino-5-phenylpyridine and 2-Acetoxyaminopyridine

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Abstract

The structures of *O*-acetyl-*N*-(5-phenyl-2-pyridyl)-hydroxylamine, $C_{13}H_{12}N_2O_2$, (I), and *O*-acetyl-*N*-(2-pyridyl)hydroxylamine, $C_7H_8N_2O_2$, (II), have been determined in order to confirm earlier structure assignments based on spectroscopic information. Compound (I) is the probable mutagenic metabolite of the phenylalanine pyrolysis product 2-amino-5-phenylpyridine. The crystal structures of (I) and (II) are the first reported for heterocyclic *N*-acetoxyarylamines, the corresponding homocyclic arylamine derivatives being extremely unstable. In the solid state, both (I) and (II) exist as hydrogen-bonded dimers, with the arylamine N atom acting as donor and the pyridine N atom of