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Key indicators

Single-crystal X-ray study T = 292 KMean σ (C–C) = 0.004 Å R factor = 0.045 wR factor = 0.122 Data-to-parameter ratio = 14.2

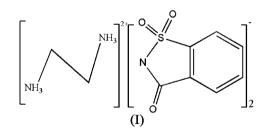
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Ethylenediammonium disaccharinate

The asymmetric unit of the title compound, $C_2H_{10}N_2^{2^+}\cdot 2C_7H_4NO_3S^-$, is composed of two saccharinate anions and one doubly protonated ethylenediamine cation. These are linked by N-H···O and N-H···N hydrogen bonds and aromatic π - π stacking interactions, leading to a two-dimensional framework structure.

Comment

Saccharin is a versatile polyfunctional ligand which has been used to build novel complexes with transition metals and some ancillary ligands (Falvello *et al.*, 2001; Yilmaz *et al.*, 2002). However, as far as the authors are aware, there are no structures reported in the literature where saccharin interacts with organic bases through hydrogen bonds to form supramolecular assemblies. Our research groups are currently investigating supramolecular structures of co-crystals containing saccharin and a series of organic bases. Here, we report the title co-crystal of saccharin, (I), incorporating the organic base ethylenediamine.



The structure of (I) is illustrated in Fig. 1. The asymmetric unit consists of two saccharinate anions and one doubly protonated ethylenediamine cation. These ions are linked into a two-dimensional framework structure by a combination of N-H···O and N-H···N hydrogen bonds (Fig. 2, Table 1). Moreover, π - π stacking interactions are observed between the C1-C6 benzene ring at (x, y, z) and the C8-C13 benzene rings of centrosymmetrically related molecules at (-x, 1 - y, 1 - z) and (1 - x, 1 - y, 1 - z), with centroid-centroid distances of 3.749 (4) and 3.726 (5) Å, respectively.

Experimental

All reagents were commercially available and of analytical grade. Saccharin (2.0 mmol, 0.376 g) and ethylenediamine (1.0 mmol, 0.06 g) were dissolved in water (20 ml). The mixture was stirred for 10 min at 353 K. The solution was then filtered and the filtrate was kept at room temperature. Colourless crystals of (I) were obtained from the filtrate after 3 d.

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3612 independent reflections 2944 reflections with $I > 2\sigma(I)$

 $R_{\rm int} = 0.020$ $\theta_{\rm max} = 26.0^{\circ}$ $h = -8 \rightarrow 9$ $k = -12 \rightarrow 12$ $l = -16 \rightarrow 16$

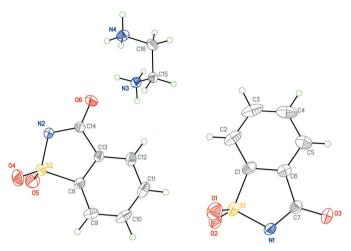


Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

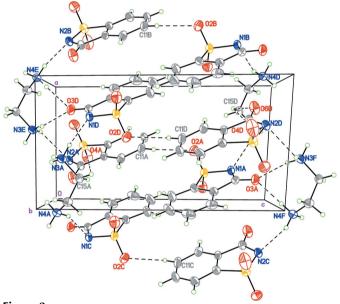


Figure 2

A view of the crystal packing of (I) along the b axis, showing the linkage of the ions by hydrogen-bonding interactions (dashed lines). [Symmetry codes: (A) x, y, z; (B) 1 + x, y, z; (C) -x, 1 - y, 1 - z; (D) 1 - x, 1 - y, 1 - zz; (E) 1 - x, 1 - y, -z; (F) x, y, 1 + z.]

from 2723

Crystal data

$C_2H_{10}N_2^{2+} \cdot 2C_7H_4NO_3S^-$	Z = 2
$M_r = 426.46$	$D_x = 1.522 \text{ Mg m}^{-3}$
Triclinic, P1	Mo $K\alpha$ radiation
a = 7.3344 (9) Å	Cell parameters from 27
b = 10.0494 (12) Å	reflections
c = 13.2454 (16) Å	$\theta = 2.5 - 28.2^{\circ}$
$\alpha = 84.741 \ (2)^{\circ}$	$\mu = 0.33 \text{ mm}^{-1}$
$\beta = 86.943 \ (2)^{\circ}$	T = 292 (2) K
$\gamma = 73.305 (2)^{\circ}$	Needle, colourless
$V = 930.8 (2) \text{ Å}^3$	$0.40 \times 0.20 \times 0.10 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-			
detector diffractometer			
Thin-slice ω scans			
Absorption correction: multi-scan			
(SADABS; Bruker, 2001)			
$T_{\min} = 0.880, \ T_{\max} = 0.968$			
7288 measured reflections			

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	+ 0.2579P]
$wR(F^2) = 0.122$	where $P = (F_0^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{\rm max} < 0.001$
3612 reflections	$\Delta \rho_{\rm max} = 0.37 \ {\rm e} \ {\rm \AA}^{-3}$
255 parameters	$\Delta \rho_{\rm min} = -0.28 \text{ e } \text{\AA}^{-3}$
H-atom parameters constrained	

Table I			
Hydrogen-bond	geometry	(Å,	°).

$\overline{D - \mathbf{H} \cdots A}$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdot \cdot \cdot A$
N3-H3A···O3 ⁱ	0.89	2.05	2.850 (2)	149
$N3-H3B \cdot \cdot \cdot N1^{ii}$	0.89	2.00	2.883 (3)	173
N3-H3C···N2 ⁱⁱⁱ	0.89	2.49	3.085 (3)	125
$N4-H4A\cdots O3^{i}$	0.89	1.97	2.816 (2)	158
$N4-H4B\cdots N2^{iv}$	0.89	2.09	2.867 (2)	146
$N4-H4C\cdots N1^{v}$	0.89	2.28	3.124 (3)	158
N3-H3 C ···O4 ^{vi}	0.89	2.31	2.886 (3)	123
C11-H11···O2	0.93	2.42	3.280 (3)	154
C15-H15B···O6	0.97	2.37	3.083 (3)	130
$C15-H15A\cdots O4^{vi}$	0.97	2.41	2.934 (3)	113

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

All H atoms were placed in calculated positions, with C-H = 0.93-0.97 Å and N-H = 0.89 Å, and refined as riding, with $U_{iso}(H) =$ $1.2U_{eq}(C)$ or $1.5U_{eq}(N)$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: PLATON.

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References

Bruker (2001). SAINT (Version 6.45), SMART (Version 5.628) and SADABS (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Falvello, L. R., Gomez, J., Pascual, I., Tomas, M., Urriolabeitia, E. P. & Schultz, A. J. (2001). Inorg. Chem. 40, 4455-4463.

Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.

Spek, A. L. (2003). J. Appl. Cryst. 36, 7-13.

Yilmaz, V. T., Guney, S., Andac, O. & Harrison, W. T. A. (2002). Polyhedron, 21, 2393-2402.