

## Ethylenediammonium thiophene-2,5-dicarboxylate dihydrate, an acid–base complex with a three-dimensional hydrogen-bonding system

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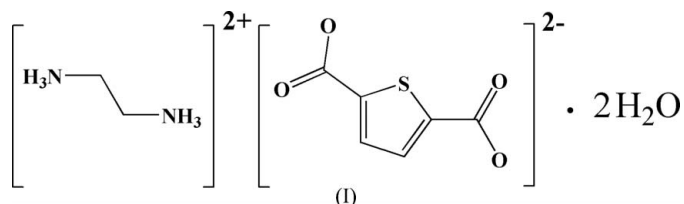
## Key indicators

Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.029  
 $wR$  factor = 0.084  
Data-to-parameter ratio = 12.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound,  $\text{C}_2\text{H}_{10}\text{N}_2^{2+} \cdot \text{C}_6\text{H}_2\text{O}_4\text{S}^{2-} \cdot 2\text{H}_2\text{O}$ , is built of ethylenediammonium dications, occupying a special position on an inversion center, thiophene-2,5-dicarboxylate dianions, in a special position on the twofold axis, and water molecules in general positions. All residues are involved in an extensive hydrogen-bonding system, which links them into a three-dimensional supramolecular arrangement.

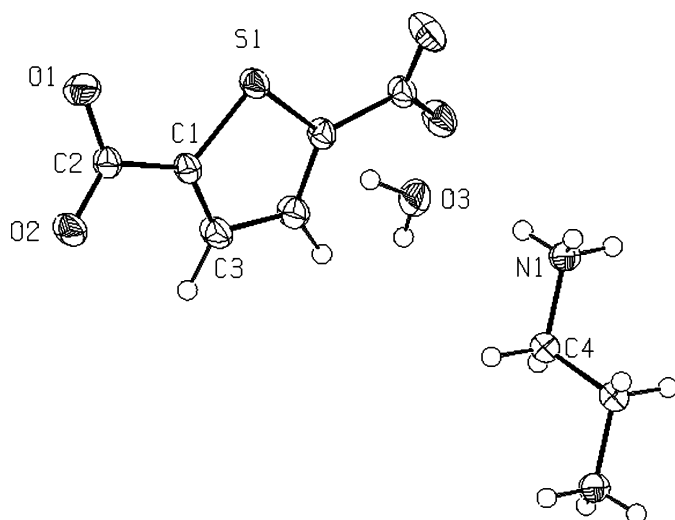
## Comment

Supramolecular chemistry based on metal-ion-directed, hydrogen-bond or  $\pi$ - $\pi$  interaction assembly of organic molecular building blocks is receiving increasing attention owing to potential applications in the fields of catalysis, nonlinear optics, sensors, magnetism and molecular recognition (Lehn, 1995; Yaghi, *et al.*, 1998). Thiophene-2,5-dicarboxylic acid (TDA) and its derivatives represent a convenient substrate for construction of supramolecular assemblies of various kinds. Chen *et al.* (1999) have reported the structural characterization of a coordination polymer involving thiophene-2,5-dicarboxylate (TDC), which can potentially serve as a monodentate, bidentate or tridentate ligand; it can also have a bridging or chelating ligand function (Chen *et al.*, 1998). Particular emphasis has been placed on the hydrogen-bonded systems, and strong  $\text{N}-\text{H} \cdots \text{O}$  bonds are most frequently responsible for the formation of supramolecular arrangements in the crystals of adducts formed by organic bases and carboxylic acids. In this paper, we report the structure of the ethylenediammonium salt of TDC, with a three-dimensional supramolecular arrangement held together by an extensive system of strong hydrogen bonds.



The crystal structure of the title compound is built of ethylenediammonium dications, which occupy a special position on an inversion center, TDC dianions on the twofold axis and water molecules in general positions (Fig. 1).

All five symmetry-independent 'active' H atoms (three ammonium H atoms of the cation and two H atoms of the water molecule) participate in an extensive system of hydrogen bonds, which link cations, anions and water mol-


**Figure 1**

View of the title compound, showing the atom labeling and displacement ellipsoids drawn at the 50% probability level. The unlabeled atoms of the ethylenediammonium dication are related to the corresponding labeled atoms through the symmetry transformation  $(1 - x, 1 - y, 1 - z)$ . The unlabeled atoms of the TDA ion are derived by the transformation  $(-x, y, \frac{3}{2} - z)$ .

ecules into an infinite three-dimensional framework. The packing is shown in Fig. 2.

## Experimental

A mixture of TDA (0.0127 g, 0.125 mmol), ethylenediamine (0.04 ml, 0.45 mmol) and water (0.6 ml) was kept in a Teflon-lined autoclave at 353 K for 3 d; it was then cooled to room temperature. The precipitated colorless crystals were filtered off, washed with distilled water and dried in air.

### Crystal data

$C_2H_{10}N_2^{2+} \cdot C_6H_2O_4S^{2-} \cdot 2H_2O$   
 $M_r = 268.29$   
 Monoclinic,  $C2/c$   
 $a = 11.466$  (1) Å  
 $b = 11.840$  (1) Å  
 $c = 10.2335$  (8) Å  
 $\beta = 121.865$  (1)°  
 $V = 1179.9$  (2) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.510$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.29$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 Block, colorless  
 $0.43 \times 0.41 \times 0.37$  mm

### Data collection

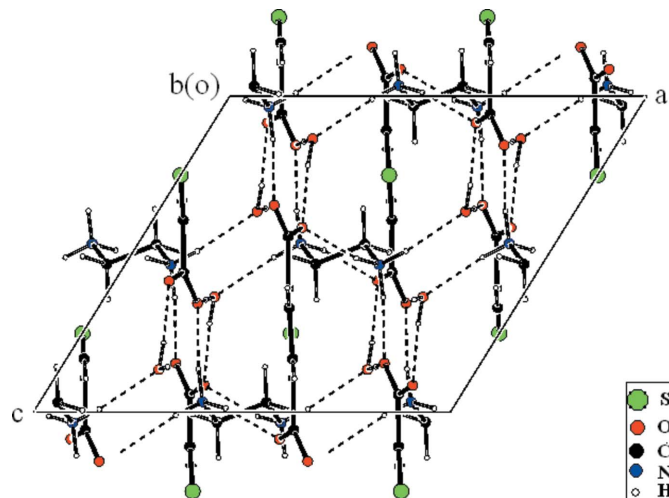
Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.884$ ,  $T_{\max} = 0.899$

3847 measured reflections  
 1414 independent reflections  
 1337 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.012$   
 $\theta_{\text{max}} = 28.0^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.084$   
 $S = 1.09$   
 1414 reflections  
 111 parameters  
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.5722P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.35$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97  
 Extinction coefficient: 0.0088 (15)


**Figure 2**

Packing diagram for the title compound, viewed down the  $b$  axis. Hydrogen bonds are shown as dashed lines.

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O3-H3B \cdots O1^i$	0.85 (2)	1.87 (2)	2.7223 (13)	173 (2)
$O3-H3A \cdots O2^{ii}$	0.82 (2)	1.97 (2)	2.7818 (14)	170 (2)
$N1-H1C \cdots O2^i$	0.909 (19)	1.887 (19)	2.7788 (13)	166.6 (16)
$N1-H1B \cdots O1^{iii}$	0.912 (17)	1.921 (17)	2.7970 (14)	160.6 (15)
$N1-H1A \cdots O3^{iv}$	0.853 (18)	1.973 (18)	2.8092 (15)	166.3 (16)

Symmetry codes: (i)  $x, y, z - 1$ ; (ii)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (iii)  $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$ ; (iv)  $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$ .

All H atoms were located in difference Fourier maps and refined isotropically [ $C-H = 0.926$  (16)– $1.033$  (16) Å].

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 1995) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXTL.

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