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Two three-dimensional networks in two polymorphs of biphenyl-4,4′-diaminium bis(3-carboxy-4-hydroxybenzenesulfonate) dihydrate

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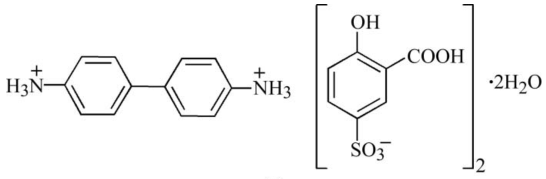
Two polymorphs of biphenyl-4,4′-diaminium bis(3-carboxy-4-hydroxybenzenesulfonate) dihydrate, $C_{12}H_{14}N_2^{2+}\cdot 2C_7H_5O_6S^-\cdot 2H_2O$, have been obtained and crystallographically characterized. Polymorph (I) crystallizes in the space group $P2_1/c$ with $Z' = 2$ and polymorph (II) in the space group $P\overline{1}$ with $Z' = 0.5$. The benzidinium cation in (II) is located on a crystallographic inversion centre. In both (I) and (II), the sulfonic acid H atoms are transferred to the benzidine N atoms, forming dihydrated 1:2 molecular adducts (base–acid). In the crystal packings of (I) and (II), the component ions are linked into three-dimensional networks by combinations of $X—H\cdots O$ (X = O, N and C) hydrogen bonds. In addition, π–π interactions are observed in (I) between inversion-related benzene rings [centroid–centroid distances = 3.632 (2) and 3.627 (2) Å]. In order to simplify the complex three-dimensional networks in (I) and (II), we also give their rationalized topological analyses.

Comment

Polymorphism, the existence of more than one crystalline form of a compound, is a well known and widely studied phenomenon in the fields of materials (Girlando *et al.*, 2010) and active pharmaceutical ingredients (APIs) (Robinson, 2010; Arenas-García *et al.*, 2010). Benzidine (BZD) is capable of crystallizing in at least four crystalline forms, owing to rotation about its central C—C single bond (Rafilovich & Bernstein, 2006; Rafilovich *et al.*, 2007; Dobrzycki & Wozniak, 2006), thus providing a key material for crystallographers to investigate the polymorph phenomenon. However, to date, many studies of polymorphism have focused on single-component compounds (Probert *et al.*, 2010), and research into the polymorphism of multi-component compounds is comparatively rare (Stahly, 2007; Budzianowski *et al.*, 2010). In this paper, we report two polymorphic forms of a hydrated binary organic adduct, benzidinium bis(5-sulfosalicylate) dihydrate [systematic name: biphenyl-4,4′-diaminium bis(3-carboxy-4-hydroxybenzenesulfonate) dihydrate], (I) and (II).



(I)

The two polymorphs of the title compound were unexpectedly obtained simultaneously by mixing BZD and 5-sulfosalicylic acid (5-H_2SSA) in a 1:2 molar ratio. Crystals of two shapes, plates and blocks, were formed over a period of two weeks. X-ray diffraction showed the plate crystals, (I), to be monoclinic (space group $P2_1/c$, $Z' = 2$), with the asymmetric unit consisting of two benzidinium divalent cations, four 5-sulfonate monoanions (5-HSSA$^-$) and four solvent water molecules (Fig. 1). For all 5-HSSA$^-$ anions in (I), common intramolecular $S(6)$ hydrogen bonds were formed [see Bernstein *et al.* (1995) for a definition of hydrogen-bond motifs]. The sulfonate O atoms are positioned slightly differently relative to their respective benzene rings. For instance, the oxygen-to-benzene plane distances are 0.215 (6)/1.038 (6)/1.326 (6), 0.354 (6)/0.917 (6)/1.383 (6), 0.304 (6)/0.948 (6)/1.373 (6) and 0.112 (7)/1.003 (6)/1.355 (6) Å for the S1, S2, S3 and S4 sulfonate anions, respectively, and these values are comparable with those in some analogues (Meng *et al.*, 2007, 2008). For the two BZD^{2+} cations in the asymmetric unit of (I), the dihedral angles between two benzene planes are 29.5 (2) and 31.0 (2)° for the N1/N2- and N3/N4-containing cations, respectively.

In comparison, the block-shaped crystals of (II) crystallize in the triclinic space group $P\overline{1}$ with $Z' = 0.5$, *i.e.* its asymmetric unit is composed of half a benzidinium cation, one 5-HSSA$^-$ anion and one water molecule (Fig. 2). An inversion centre is located at the mid-point of the central C—C single bond of the BZD^{2+} cation and a dihedral angle of exactly 0° is formed between the two benzene rings. The three sulfonate O atoms, O4, O5 and O6, are 1.073 (4), 1.276 (4) and 0.277 (4) Å, respectively, away from the benzene plane. These conformational diversities of both cations and anions may favour the formation of even more polymorphs.

In the packing structures of both (I) and (II), the component ions are linked into complex three-dimensional frameworks by a combination of $X—H\cdots O$ (X = O, N and C) hydrogen bonds. In (I), the supramolecular structure can be relatively easily analysed in terms of the three aspects mentioned below.

Firstly, ammonium atoms N1 act as hydrogen-bond donors *via* atoms H1*A*, H1*B* and H1*C*, and are hydrogen bonded to three independent sulfonate O atoms, *viz.* O5, O24($-x$, $1 - y$, $1 - z$) and O12, respectively. In the same way, atom N2 is also

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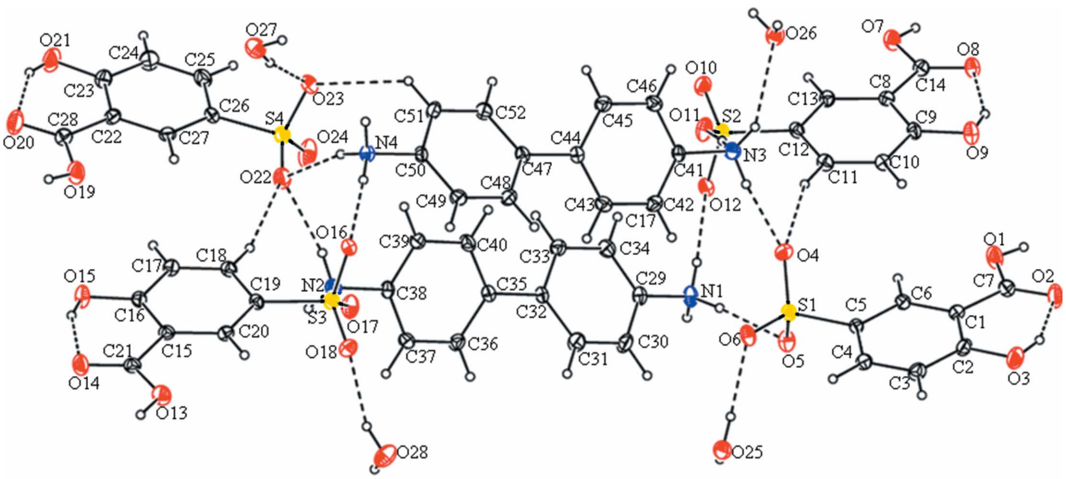

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

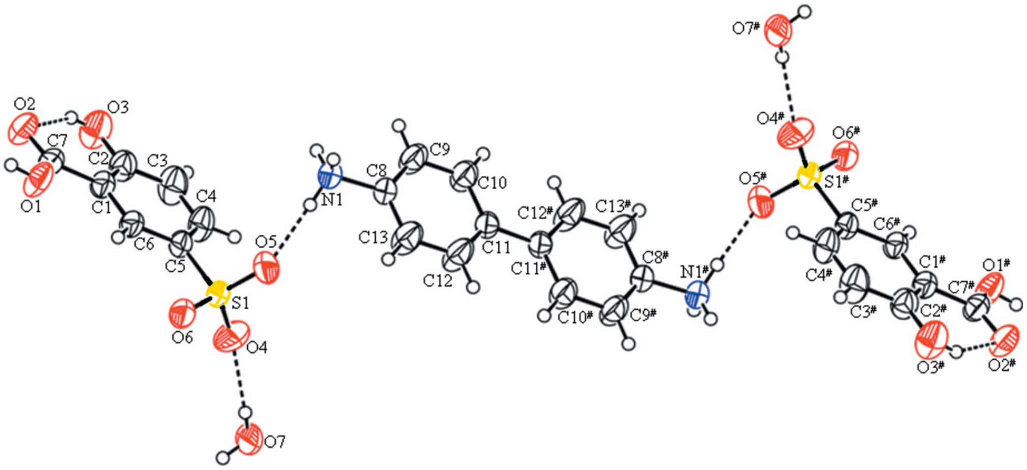

Figure 2
The molecular structure of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines. Atoms marked with a hash symbol (#) are at the symmetry position $(-x, -y, 2 - z)$.

hydrogen bonded to three other independent sulfonate O atoms, *viz.* O17, O22 and O12$(-x, 1 - y, 1 - z)$. In addition, atom N2 is also hydrogen bonded to water molecule O25 at $(x, \frac{1}{2} - y, \frac{1}{2} + z)$. Thus, the BZD^{2+} cation, as a whole, is linked to six symmetry-independent 5-$HSSA^-$ anions and one water molecule. Similarly, the other benzidinium N atoms, N3 and N4, acting as hydrogen-bond donors *via* six H atoms (H3*A*–H3*C* and H4*A*–H4*C*) are linked to seven independent O atoms, of which five are from sulfonate anions and the remaining two from water O atoms.

Secondly, carboxyl atoms O7, O1, O13 and O19 in the four sulfonate anions act as hydrogen-bond donors and link to sulfonate atom O5 at $(-x, -y + 1, -z)$ and three water atoms, *viz.* O25 at $(x, -y + \frac{1}{2}, z - \frac{1}{2})$, O26 at $(x, y, 1 + z)$ and O27 at $(1 - x, 1 - y, 2 - z)$, respectively.

Thirdly, as far as the four solvent water molecules are concerned, atom O27 acts as a hydrogen-bond donor to sulfonate atom O23 and water atom O28 at $(-x + 1, y + \frac{1}{2}, -z + \frac{3}{2})$. The other three water molecules are all hydrogen bonded to two sulfonate O atoms, *i.e.* atoms O6 and O10$(-x, y - \frac{1}{2}, \frac{1}{2} - z)$ for water O25, atoms O23$(x, \frac{3}{2} - y, z - \frac{1}{2})$ and O18$(1 - x, 1 - y, 1 - z)$ for water O26, and atoms O6$(x, \frac{1}{2} - y, \frac{1}{2} + z)$ and O18 for water O28. These extensive hydrogen bonds are sufficient to link the BZD^{2+} cations, 5-$HSSA^-$ anions and H_2O molecules into a complex three-dimensional network in (I) (Table 1 and Fig. 3).

Aiming to understand the network better, we attempted to give it a standard topological simplification (Baburin & Blatov, 2007), *i.e.* all the ion centroids were regarded as different nodes in a new network. According to the numbers of intermolecular hydrogen bonds originating from one individual ion as both hydrogen-bond donor and acceptor, the two benzidinium cations can be regarded as seven-connected nodes, the S1-, S3- and S4-containing 5-$HSSA^-$ anions as six-connected nodes, the S2-containing 5-$HSSA^-$ anion as a five-connected node, water molecules O25, O26 and O28 as four-connected nodes and water molecule O27 as a three-connected node. The final 3,4,5,6,7-connected three-dimensionally topological network with stoichiometry $(3\text{-}c):(4\text{-}c)_3:(5\text{-}c):(6\text{-}c)_3:(7\text{-}c)_2$ was then rationalized with a Schlafli symbol of $(3.4.5^4)$-

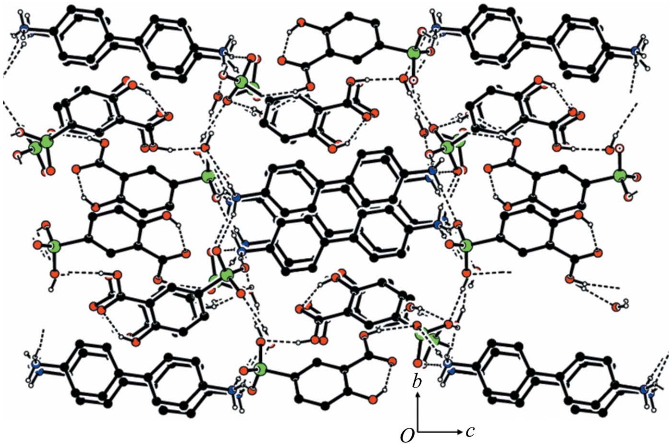

Figure 3
Part of the crystal structure of (I), showing the formation of the three-dimensional network, viewed perpendicular to the (100) plane. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif have been omitted.

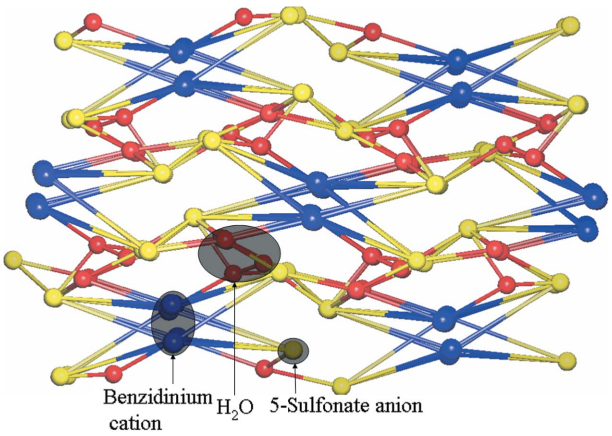

Figure 4
A schematic view of the formation of the 3,4,5,6,7-connected topological network in (I) when the cations, anions and water molecules are regarded as nodes. (In the electronic version of the paper, the blue, yellow and red spheres represent benzidinium cations, 5-sulfosalicylate anions and water molecules, respectively.)

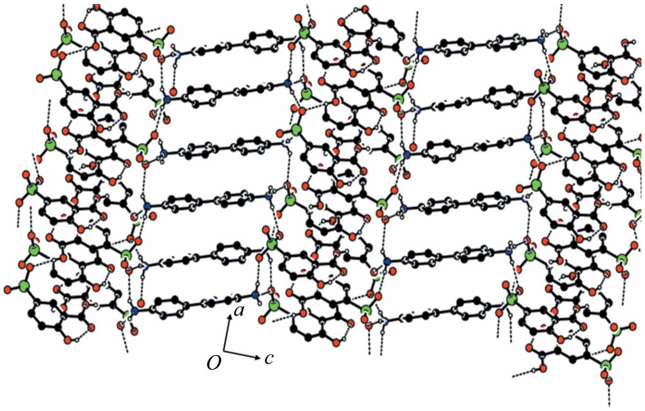

Figure 5
Part of the crystal structure of (I), showing the formation of the two-dimensional (010) layer built from hydrogen bonds (dashed lines), without considering water molecules O25–O28. For the sake of clarity, H atoms not involved in the motif have been omitted.

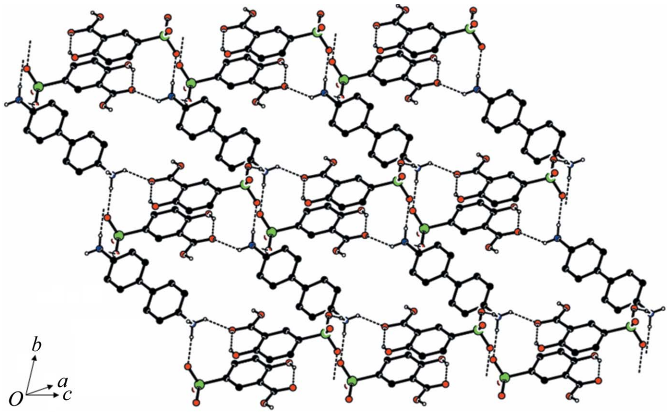

Figure 6
Part of the crystal structure of (II), showing the formation of the two-dimensional ($1\overline{1}0$) layer built from hydrogen bonds (dashed lines), without considering water molecule O7. For the sake of clarity, H atoms not involved in the motif have been omitted.

$(3.4.5^5.6^7.7)(3.4^{11}.5^5.6^4)(3.4^3.5^5.6^5.7)(3.4^8.5^5.6^7)(3^2.4.5^2.6)$-$(3^2.4^4.5^2.6^2)(4.5^4.6)(4^2.6)(4^5.5^3.6^5.7^2)$ (Fig. 4).

Although it may seem meaningless to simplify the three-dimensional hydrogen-bonding network in (I), it can help us to analyse the topology of the hydrogen-bond nets and molecular packing conveniently. For instance, from a topological viewpoint it can be easily seen that the BZD^{2+} cations and 5-$HSSA^-$ anions in (I) are linked together into a two-dimensional layer running parallel to the (010) plane (Fig. 5). These adjacent (010) layers are further joined together by water molecules, resulting in the final three-dimensional network with hydrogen bonds as the edges.

In addition to the above-mentioned hydrogen bonds, four $C-H\cdots O$ hydrogen bonds (Table 1) and two π–π stacking interactions are observed in (I). For the two π–π interactions, one exists between inversion-related C8–C13 benzene rings [$Cg1\cdots Cg1(-x, 1-y, z) = 3.632$ (2) Å; $Cg1$ is the centroid of the C8–C13 ring] and the other between inversion-related C15–C20 benzene rings [$Cg2\cdots Cg2(1-x, 1-y, 2-z) = 3.627$ (2) Å; $Cg2$ is the centroid of the C15–C20 ring].

In (II), benzidinium atom N1 in the asymmetric unit acts as a hydrogen-bond donor, *via* atoms H1*A*, H1*B* and H1*C*, to carboxyl atom O2 at $(1-x, 1-y, -z)$ and sulfonate atoms O5 and O6 at $(1-x, 1-y, 1-z)$, respectively (Table 2). Thus, each BZD^{2+} cation should be linked to six independent 5-$HSSA^-$ anions, of which four are $N-H\cdots O_{\rm sulfonate}$ and two are $N-H\cdots O_{\rm carboxylate}$ hydrogen bonds, forming a two-dimensional layer structure running parallel to the ($1\overline{1}0$) plane (Fig. 6). In one layer, three types of cyclic hydrogen-bond motifs [$R_4^4(12)$, $R_4^4(20)$ and $R_4^4(38)$] are observed. Carboxyl atom O1 is hydrogen bonded to water atom O7 at $(1+x, y, z-1)$, which thus acts as a hydrogen-bond acceptor. Water atom O7 is hydrogen bonded to sulfonate atoms O4 and O6 at $(-x, 2-y, 1-z)$ through atoms H7*A* and H7*B*, forming an $R_4^4(12)$ hydrogen-bond motif. With the aid of water molecules, these adjacent ($1\overline{1}0$) layers are linked together, forming the final complex three-dimensional network (Fig. 7).

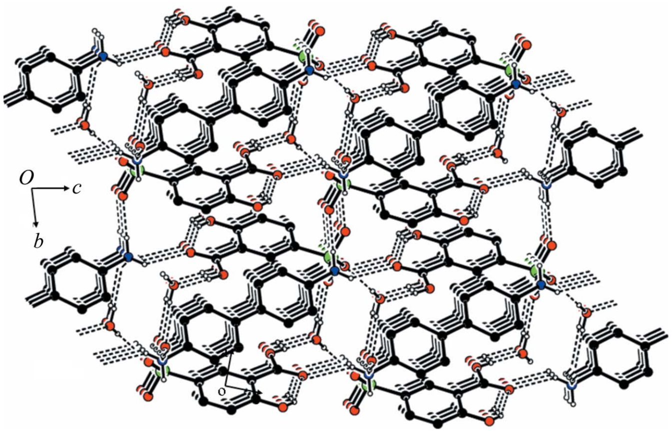

Figure 7
Part of the crystal structure of (II), showing the formation of the three-dimensional network, viewed perpendicular to the (100) plane. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif have been omitted.

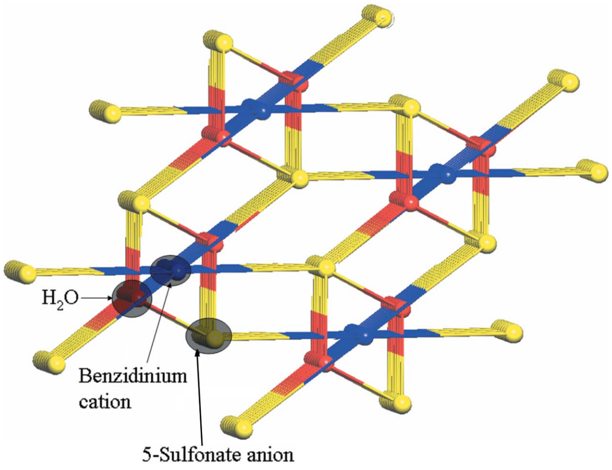

Figure 8
A schematic view of the formation of the 3,6-connected topological network in (II) when the cations, anions and water molecules are regarded as nodes. (In the electronic version of the paper, the blue, yellow and red spheres represent benzidinium cations, 5-sulfosalicylate anions and water molecules, respectively.)

In order to simplify the network in (II), the BZD^{2+} cation and 5-HSSA^{-} anion can both be regarded as six-connected nodes and water atom O7 as a three-connected node. The whole structure can be rationalized as a 3,6-connected topological three-dimensional network with stoichiometry $(3\text{-}c)_2$:$(6\text{-}c)_3$ and a Schlafli symbol of $(4^2.6)_2(4^5.6^5.8^5)_2(4^6.6^6.8^3)$ (Fig. 8).

Further analysis with *PLATON* (Spek, 2009) shows that no $C—H\cdots\pi$ or $\pi–\pi$ interactions exist in (II). In comparison, two-dimensional layers are formed in both (I) and (II) by hydrogen bonds between BZD^{2+} cations and 5-HSSA^{-} anions, but their packing patterns are different. This may, to a large extent, be attributed to the differently oriented $N—H\cdots O$ hydrogen bonds, because of their different BZD^{2+} and 5-HSSA^{-} configurations.

In short, two polymorphs of benzidinium bis(5-sulfosalicylate) dihydrate were obtained simultaneously at room temperature, and this is largely attributed to the conformational diversities of the BZD^{2+} and 5-HSSA^{-} ions. In order to simplify the hydrogen-bonded network, we have given their standard topological analyses. If the crystallization conditions were to be varied, more polymorphs might be obtained. Further research on this is ongoing in our laboratory.

Experimental

All reagents and solvents were used as obtained without further purification. Benzidine (0.1 mmol, 18.4 mg) and 5-sulfosalicylic acid dihydrate (0.2 mmol, 50.8 g), in a 1:2 molar ratio, were dissolved in 95% methanol (10 ml). The mixture was stirred for half an hour at ambient temperature and then filtered. The resulting pale-yellow solution was kept in air for two weeks. Colourless plate- and block-shaped crystals of (I) and (II), respectively, suitable for single-crystal X-ray diffraction analysis, grew at the bottom of the vessel by slow evaporation of the solution. The crystals were separated manually.

Compound (I)

Crystal data

$C_{12}H_{14}N_2^{2+}\cdot 2C_7H_5O_6S^{-}\cdot 2H_2O$
$M_r = 656.62$
Monoclinic, $P2_1/c$
$a = 15.1851\ (3)$ Å
$b = 18.6584\ (6)$ Å
$c = 19.9114\ (6)$ Å
$\beta = 91.300\ (2)^\circ$
$V = 5640.0\ (3)$ Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.27$ mm^{-1}
$T = 298$ K
0.40 × 0.10 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.901$, $T_{\max} = 0.989$
48494 measured reflections
12875 independent reflections
6411 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.100$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.165$
$S = 0.95$
12875 reflections
877 parameters
28 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

Compound (II)

Crystal data

$C_{12}H_{14}N_2^{2+}\cdot 2C_7H_5O_6S^{-}\cdot 2H_2O$
$M_r = 656.62$
Triclinic, $P\overline{1}$
$a = 8.6625\ (14)$ Å
$b = 9.2851\ (15)$ Å
$c = 9.7249\ (16)$ Å
$\alpha = 76.514\ (2)^\circ$
$\beta = 72.834\ (3)^\circ$
$\gamma = 77.947\ (3)^\circ$
$V = 718.4\ (2)$ Å^3
$Z = 1$
Mo $K\alpha$ radiation
$\mu = 0.26$ mm^{-1}
$T = 298$ K
0.23 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.932$, $T_{\max} = 0.950$
7548 measured reflections
3068 independent reflections
2615 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.094$

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O5	0.87 (2)	2.05 (2)	2.917 (4)	175 (4)
N1—H1*B*···O24^{i}	0.85 (2)	1.92 (2)	2.756 (4)	168 (4)
N1—H1*C*···O12	0.87 (2)	2.06 (2)	2.927 (4)	170 (3)
N2—H2*A*···O17	0.87 (2)	2.12 (2)	2.901 (4)	150 (3)
N2—H2*B*···O22	0.87 (2)	2.17 (2)	3.010 (5)	162 (3)
N2—H2*C*···O12^{i}	0.86 (2)	2.18 (3)	2.918 (4)	143 (3)
N2—H2*C*···O25^{ii}	0.86 (2)	2.37 (4)	2.868 (4)	117 (3)
N3—H3*A*···O4	0.85 (2)	1.94 (2)	2.775 (4)	166 (4)
N3—H3*B*···O11	0.87 (2)	1.94 (2)	2.796 (4)	171 (4)
N3—H3*C*···O16^{iii}	0.87 (2)	2.29 (3)	3.005 (4)	139 (3)
N3—H3*C*···O26	0.87 (2)	2.38 (3)	2.954 (5)	124 (3)
N4—H4*A*···O16	0.88 (2)	1.98 (2)	2.858 (4)	170 (3)
N4—H4*B*···O28^{iv}	0.88 (2)	1.89 (2)	2.755 (4)	167 (3)
N4—H4*C*···O22	0.88 (2)	2.11 (2)	2.964 (4)	165 (3)
O1—H1*D*···O25^{v}	0.83 (2)	1.77 (2)	2.585 (4)	165 (5)
O3—H3*D*···O2	0.83 (2)	1.87 (3)	2.601 (4)	147 (5)
O7—H7*A*···O5^{vi}	0.81 (2)	2.04 (2)	2.846 (3)	169 (4)
O9—H9*A*···O8	0.83 (4)	1.84 (3)	2.580 (4)	148 (4)
O13—H13*A*···O27^{vii}	0.83 (2)	1.82 (2)	2.639 (4)	170 (5)
O15—H15*A*···O14	0.82 (4)	1.81 (3)	2.582 (4)	155 (4)
O19—H19*A*···O26^{viii}	0.84 (2)	1.80 (2)	2.631 (4)	171 (5)
O21—H21*A*···O20	0.81 (4)	1.84 (3)	2.572 (4)	148 (5)
O25—H25*A*···O6	0.82 (2)	1.97 (2)	2.781 (4)	177 (5)
O25—H25*B*···O10^{ix}	0.82 (2)	1.91 (2)	2.715 (4)	168 (5)
O26—H26*A*···O23^{x}	0.82 (2)	2.08 (3)	2.822 (4)	150 (4)
O26—H26*B*···O18^{iii}	0.81 (2)	1.97 (2)	2.782 (4)	175 (5)
O27—H27*A*···O23	0.84 (2)	2.01 (2)	2.838 (4)	170 (5)
O27—H27*B*···O28^{iv}	0.82 (2)	2.33 (3)	3.089 (5)	156 (4)
O28—H28*A*···O6^{ii}	0.84 (2)	2.05 (3)	2.847 (4)	159 (5)
O28—H28*B*···O18	0.84 (2)	2.00 (2)	2.805 (4)	160 (5)
C11—H11···O4	0.93	2.45	3.341 (4)	160
C18—H18···O22	0.93	2.47	3.381 (4)	165
C48—H48···O3^{ii}	0.93	2.56	3.475 (5)	168
C51—H51···O23	0.93	2.52	3.177 (4)	128

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

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.154$
$S = 1.07$
3068 reflections
220 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.42$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

H atoms bonded to C atoms were positioned geometrically, with C—H = 0.93 Å (aromatic), and refined in a riding mode, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(aromatic C). H atoms bonded to N and O atoms were found in difference Fourier maps and their N—H and O—H distances were refined with restraints of N—H = 0.86 (2) Å and O—H = 0.82 (2) Å, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$ or $1.5U_{\rm eq}({\rm O})$.

For both compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86 (2)	2.33 (2)	2.884 (2)	123 (2)
N1—H1*B*···O5	0.88 (2)	1.91 (2)	2.774 (3)	171 (2)
N1—H1*C*···O6^{ii}	0.88 (2)	1.91 (2)	2.779 (3)	172 (2)
O1—H1···O7^{iii}	0.82 (2)	1.75 (2)	2.567 (2)	177 (3)
O3—H3*A*···O2	0.81 (2)	1.88 (3)	2.601 (2)	148 (4)
O7—H7*A*···O4	0.79 (2)	1.94 (2)	2.691 (2)	159 (4)
O7—H7*B*···O6^{iv}	0.80 (2)	2.00 (2)	2.760 (2)	158 (4)
C4—H4···O4^{v}	0.93	2.56	3.343 (3)	142
C13—H13···O3^{vi}	0.93	2.43	3.289 (3)	155

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, -y+1, -z+1$; (iii) $x+1, y, z-1$; (iv) $-x, -y+2, -z+1$; (v) $-x, -y+1, -z+1$; (vi) $x, y, z+1$.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3381). Services for accessing these data are described at the back of the journal.

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