

Poly[[triquabis( $\mu_4$ -3-carboxy-4-hydroxybenzene-sulfonato)disilver(I)] monohydrate]Shan Gao,<sup>a</sup> Zhi-Bao Zhu,<sup>a</sup> Li-Hua Huo<sup>a</sup> and Seik Weng Ng<sup>b\*</sup><sup>a</sup>College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China, and <sup>b</sup>Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

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## Key indicators

Single-crystal X-ray study

T = 295 K

Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$ 

R factor = 0.028

wR factor = 0.074

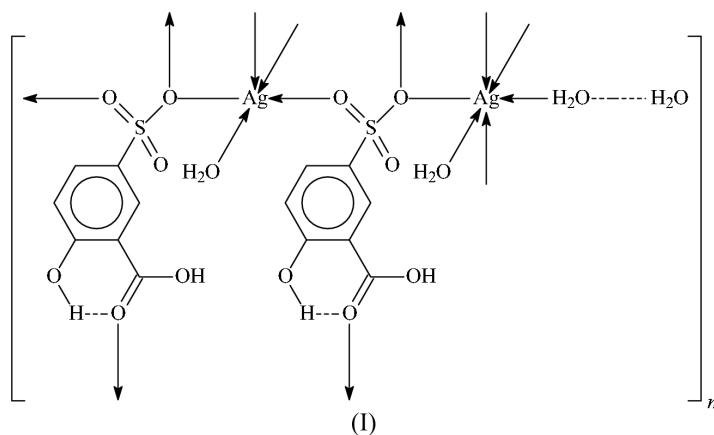
Data-to-parameter ratio = 13.6

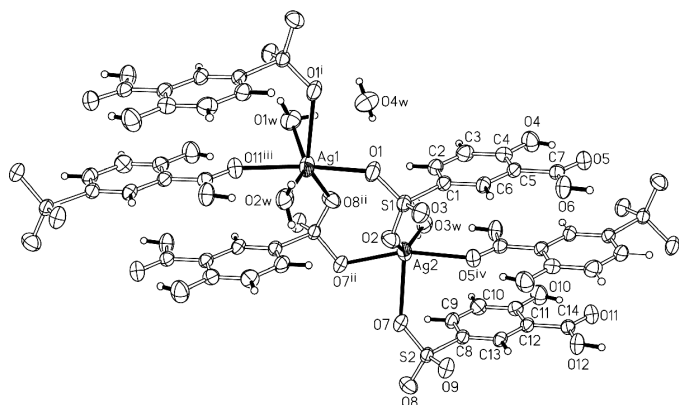
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the layer structure of the title polymeric complex,  $\{[\text{Ag}_2(\text{C}_7\text{H}_5\text{O}_6\text{S})_2(\text{H}_2\text{O})_3] \cdot \text{H}_2\text{O}\}_n$ , both substituted benzenesulfonate anions use two of their sulfonyl O atoms to link to three Ag atoms and their double-bond carbonyl O atom to another Ag atom in a  $\mu_4$ -bonding mode. The two symmetry-independent Ag atoms are coordinated by water molecules, one by one water molecule and the other by two water molecules so that one Ag atom is five- and the other six-coordinate.

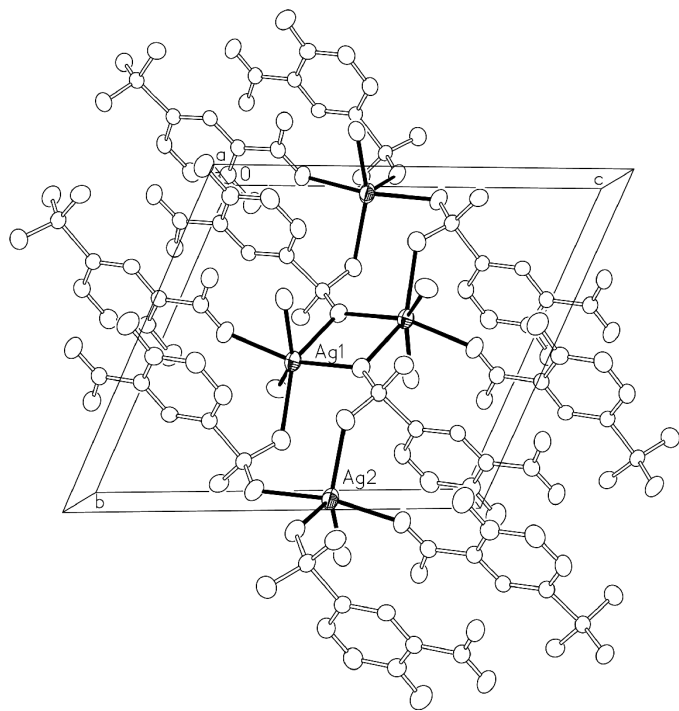
## Comment

The number of metal derivatives of sulfosalicylic (3-carboxy-4-hydroxybenzenesulfonic) acid, as noted from a search of the Cambridge Structural Database (Version 5.25; Allen, 2002), is rather small, the examples then (November 2003) being limited to the europium(II) bis[3-carboxy-4-hydroxybenzenesulfonate(−)] (Starynowicz, 2000), copper(II) sodium(I) 3-carboxy-4-hydroxybenzenesulfonate(3−) (Marzotto *et al.*, 2001, 2003), barium(II) bis[3-carboxy-4-hydroxybenzenesulfonate(−)] (Ma *et al.*, 2003), samarium(III) 3-carboxy-4-hydroxybenzenesulfonate(−) 3-carboxy-4-hydroxybenzenesulfonate(2−) (Aliev *et al.*, 1994) and trisodium(I) dilanthanum(III) terbium(III) tetrakis[3-carboxy-4-hydroxybenzenesulfonate(3−)] (Sun *et al.*, 1995) hydrates only. Among more recent examples are the main group rubidium (Hu *et al.*, 2005) and hydrogen sodium (Zhu *et al.*, 2005) derivatives; transition metal derivatives do not involve the anion in direct coordination as the metal atom is surrounded instead by six water molecules (Ma *et al.*, 2003*a,b,c,d*). Among the recent examples are complexes with *N*-heterocyclic ligands (Icbudak *et al.*, 2003; Li *et al.*, 2004; Wang *et al.*, 2004).





**Figure 1**  
ORTEP plot (Johnson, 1976) of a portion of the layer structure. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. The symmetry codes are those used in Table 1.



**Figure 2**  
ORTEP plot (Johnson, 1976) of the layer structure projected along the *a* axis. The uncoordinated water molecules are not shown.

amines (Zheng *et al.*, 2001, 2003), as well as from the fact that silver benzenesulfonate has its metal atom in an unusual nine-coordinate environment (Shimizu *et al.*, 1999). In the title compound, (I), having an empirical silver(I) 3-carboxy-4-hydroxybenzenesulfonate dihydrate formulation, the asymmetric unit has two Ag atoms as well as two sulfonate monoanions. One of the metal atoms exists in a five- and the other in a six-coordinate environment (Fig. 1). Both monoanions use two of their sulfonyl O atoms to link to three Ag atoms; their double-bond carbonyl O atoms also interact with other Ag atoms to give a  $\mu_4$ -bonding mode for the monoanion. The bonding interactions lead to the formation of layers (Fig. 2); one of the Ag atoms is coordinated by one water

molecule whereas the other is coordinated by two water molecules.

**Experimental**

Silver nitrate (0.17 g, 1 mmol), sulfosalicylic acid (0.22 g, 1 mmol) and sodium hydroxide (0.04 g, 1 mmol) were placed in ethanol (20 ml) and the mixture vigorously stirred to dissolve the reagents. The solution was filtered; colorless crystals separated from the solution after several days. Elemental analysis found (calculated): C 23.11 (23.29), H 2.42% (2.51%).

**Crystal data**

[Ag<sub>2</sub>(C<sub>7</sub>H<sub>5</sub>O<sub>6</sub>S)<sub>2</sub>(H<sub>2</sub>O)<sub>3</sub>]<sub>2</sub>·H<sub>2</sub>O  
*M<sub>r</sub>* = 722.14  
 Triclinic, P1  
*a* = 8.309 (1) Å  
*b* = 11.264 (2) Å  
*c* = 12.611 (2) Å  
 $\alpha$  = 112.743 (6)°  
 $\beta$  = 97.608 (7)°  
 $\gamma$  = 95.516 (7)°  
*V* = 1064.9 (3) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 2.252 Mg m<sup>-3</sup>  
 Mo K $\alpha$  radiation  
 Cell parameters from 8670 reflections  
 $\theta$  = 3.2–27.4°  
 $\mu$  = 2.12 mm<sup>-1</sup>  
*T* = 295 (2) K  
 Block, colorless  
 0.38 × 0.26 × 0.19 mm

**Data collection**

Rigaku R-AXIS RAPID diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)  
*T<sub>min</sub>* = 0.316, *T<sub>max</sub>* = 0.668  
 10379 measured reflections

4829 independent reflections  
 4464 reflections with *I* > 2 $\sigma$ (*I*)  
*R<sub>int</sub>* = 0.024  
 $\theta_{max}$  = 27.4°  
*h* = -10 → 10  
*k* = -14 → 14  
*l* = -16 → 16

**Refinement**

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.028  
*wR*(*F*<sup>2</sup>) = 0.074  
*S* = 1.00  
 4829 reflections  
 356 parameters  
 H atoms treated by a mixture of independent and constrained refinement

*w* = 1/[ $\sigma^2(F_o^2) + (0.0411P)^2 + 1.1907P$ ]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 $(\Delta/\sigma)_{max}$  = 0.001  
 $\Delta\rho_{max}$  = 1.11 e Å<sup>-3</sup>  
 $\Delta\rho_{min}$  = -1.04 e Å<sup>-3</sup>  
 Extinction correction: SHELXL97  
 Extinction coefficient: 0.070 (2)

**Table 1**

Selected geometric parameters (Å, °).

Ag1—O1	2.374 (2)	Ag2—O2	2.517 (2)
Ag1—O1 <sup>i</sup>	2.695 (2)	Ag2—O5 <sup>iv</sup>	2.420 (2)
Ag1—O8 <sup>ii</sup>	2.520 (2)	Ag2—O7	2.505 (2)
Ag1—O11 <sup>iii</sup>	2.411 (2)	Ag2—O7 <sup>ii</sup>	2.373 (2)
Ag1—O1 <sub>w</sub>	2.629 (3)	Ag2—O3 <sub>w</sub>	2.467 (2)
Ag1—O2 <sub>w</sub>	2.651 (2)		
O1—Ag1—O1 <sup>i</sup>	80.07 (7)	O11 <sup>iii</sup> —Ag1—O2 <sub>w</sub>	77.42 (7)
O1—Ag1—O8 <sup>ii</sup>	90.15 (7)	O1 <sub>w</sub> —Ag1—O2 <sub>w</sub>	148.95 (7)
O1—Ag1—O11 <sup>iii</sup>	158.51 (8)	O2—Ag2—O5 <sup>iv</sup>	91.56 (7)
O1—Ag1—O1 <sub>w</sub>	123.46 (8)	O2—Ag2—O7	102.94 (7)
O1—Ag1—O2 <sub>w</sub>	81.31 (7)	O2—Ag2—O7 <sup>ii</sup>	96.13 (7)
O1 <sup>i</sup> —Ag1—O8 <sup>ii</sup>	135.29 (7)	O2—Ag2—O3 <sub>w</sub>	146.10 (7)
O1 <sup>i</sup> —Ag1—O11 <sup>iii</sup>	97.81 (7)	O5 <sup>iv</sup> —Ag2—O7	88.40 (7)
O1 <sup>i</sup> —Ag1—O1 <sub>w</sub>	75.70 (7)	O5 <sup>iv</sup> —Ag2—O7 <sup>ii</sup>	162.89 (7)
O1 <sup>i</sup> —Ag1—O2 <sub>w</sub>	92.68 (7)	O5 <sup>iv</sup> —Ag2—O3 <sub>w</sub>	83.95 (7)
O8 <sup>ii</sup> —Ag1—O11 <sup>iii</sup>	105.60 (7)	O7—Ag2—O7 <sup>ii</sup>	75.01 (7)
O8 <sup>ii</sup> —Ag1—O1 <sub>w</sub>	73.95 (7)	O7—Ag2—O3 <sub>w</sub>	110.47 (7)
O8 <sup>ii</sup> —Ag1—O2 <sub>w</sub>	129.05 (7)	O7 <sup>ii</sup> —Ag2—O3 <sub>w</sub>	97.79 (7)
O11 <sup>iii</sup> —Ag1—O1 <sub>w</sub>	75.84 (8)		

Symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z*; (ii) 2 - *x*, 2 - *y*, 1 - *z*; (iii) *x* - 1, *y* - 1, *z* - 1; (iv) 2 - *x*, 2 - *y*, 2 - *z*.

**Table 2**  
Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4 $o$ ···O5	0.85 (1)	1.81 (2)	2.612 (3)	156 (4)
O6—H6 $o$ ···O3 $w^{iv}$	0.85 (1)	1.77 (1)	2.621 (3)	177 (4)
O10—H10 $o$ ···O11	0.85 (1)	1.77 (2)	2.587 (3)	159 (4)
O12—H12 $o$ ···O2 $w^v$	0.85 (1)	1.76 (1)	2.599 (3)	174 (4)
O1 $w$ —H1 $w1$ ···O4 $w$	0.85 (1)	1.94 (1)	2.787 (4)	177 (4)
O1 $w$ —H1 $w2$ ···O3 $i$	0.85 (1)	1.99 (2)	2.815 (3)	164 (5)
O2 $w$ —H2 $w1$ ···O3 $vi$	0.85 (1)	2.03 (2)	2.847 (3)	162 (3)
O2 $w$ —H2 $w2$ ···O4 $w^j$	0.85 (1)	1.91 (2)	2.734 (3)	164 (3)
O3 $w$ —H3 $w1$ ···O9 $vii$	0.85 (1)	1.91 (1)	2.757 (3)	174 (3)
O3 $w$ —H3 $w2$ ···O1 $w^{viii}$	0.85 (1)	1.89 (1)	2.722 (3)	169 (3)
O4 $w$ —H4 $w2$ ···O2 $vii$	0.85 (1)	2.03 (1)	2.858 (3)	165 (4)
O4 $w$ —H4 $w1$ ···O9 $vii$	0.85 (1)	2.00 (1)	2.847 (3)	170 (5)

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

The water and hydroxyl H atoms were found in a difference Fourier map and were refined with distance restraints of O—H = 0.85 (1) Å and H···H = 1.39 (1) Å. The carbon-bound H atoms were placed at calculated positions (C—H = 0.93 Å) and were included in the refinement in the riding-model approximation with their displacement parameters tied to those of their parent atoms [ $U_{iso}(H) = 1.2U_{eq}(C)$ ]. In the final difference Fourier map, the largest peak was 0.7 Å from atom Ag2 and the deepest hole 0.7 Å from Ag1.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976)..

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