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# Octaaquaytterbium(III) tris(3-carboxy-4-hydroxybenzenesulfonate) monohydrate

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The title compound, $[Yb(H_2O)_8](C_7H_5O_6S)_3\cdot H_2O$, was synthesized by the reaction of $Yb_2O_3$ with 5-sulfosalicylic acid in water. The compound contains $Yb(H_2O)_8^{3+}$ monomers; the 3-carboxy-4-hydroxybenzenesulfonate anions do not coordinate to ytterbium, but act as counter-anions.

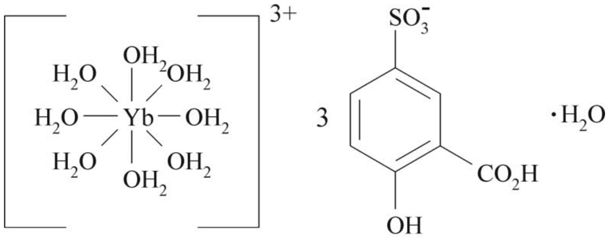
**Key indicators**

Single-crystal X-ray study
$T$ = 298 K
Mean $\sigma$(C–C) = 0.005 Å
Disorder in main residue
$R$ factor = 0.025
$wR$ factor = 0.052
Data-to-parameter ratio = 11.5

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

## Comment

In connection with our investigations of the structrural chemistry of lanthanide (Ln) carboxylates, we examined the $Ln^{3+}$ complexes with 5-sulfosalicylic acid where until now only the neodymium (Wang *et al.*, 2004), samarium (Aliev *et al.*, 1994), and europium (Starynowicz, 2000) compounds are known. In contrast with these compounds where the anion is coordinated to the metal ion, the $Yb^{3+}$ ion in the title compound, (I), is surrounded only by water molecules and the anion acts as counter-ion. These structural characteristics are known from lanthanide compounds with ethyl sulfates (Gerkin & Reppart, 1984) and trifluoromethanesulfonates (Chatterjee *et al.*, 1988).



**(I)**

The structure of $[Yb(H_2O)_8](H_2L)_3\cdot H_2O$ ($H_3L$ = 3-carboxy-4-hydroxybenzenesulfonic acid (5-sulfosalicylic acid; $C_7H_6O_6S$)) is shown in Fig. 1. The $Yb^{3+}$ ion is coordinated by eight water molecules, giving a square antiprismatic coordination. The Yb—O distances are between 2.278 (3) and 2.372 (4) Å (*cf.* Table 1) which is similar to other compounds with $[Yb(H_2O)_8]^{3+}$ cations (Makha *et al.*, 2005). There is one additional uncoordinated water molecule in the asymmetric unit. The organic anions are non-coordinating. The $[Yb(H_2O)_8]^{3+}$ cations, water molecules and $H_2L^-$ anions are connected through a complex pattern of hydrogen-bonding interactions. Selected hydrogen-bond parameters are listed in Table 2. The crystal structure shows large Yb···Yb separations, the shortest internuclear distance being 6.792 (2) Å.

The extended structure is formed by $\pi$–$\pi$ stacking of the organic anions (Janiak, 2000). The shortest orthogonal distance between two aromatic fragments is 3.422 (1) Å.

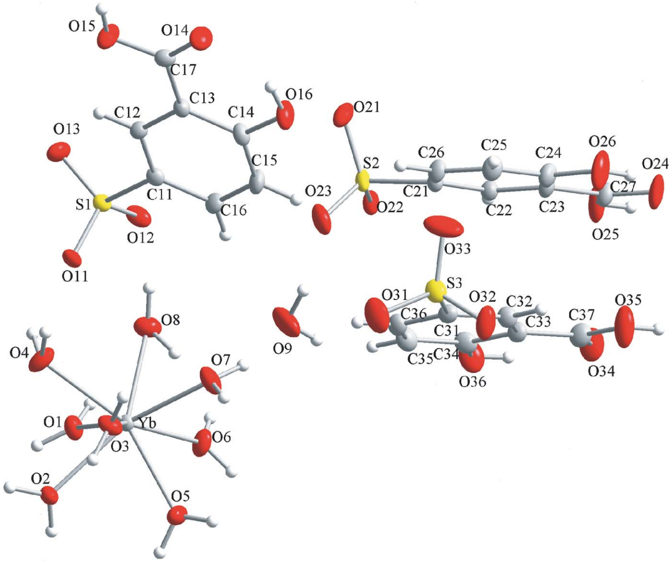


**Figure 1**
View of the asymmetric unit of (I), with the labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

## Experimental

$[Yb(H_2O)_8](H_2L)_3 \cdot H_2O$ was prepared by the reaction of 0.20 g $Yb_2O_3$ (1 mmol $Yb^{3+}$) and 0.76 g $H_3L$ (3.5 mmol) in $H_2O$ (10 ml) at about 323 K in a glass container. After 1 d, the remaining solid was filtered off. After a few weeks, colourless crystals were formed.

### *Crystal data*

$[Yb(H_2O)_8](C_7H_5O_6S)_3 \cdot H_2O$
$M_r = 986.69$
Triclinic, $P\overline{1}$
$a = 7.523$ (2) Å
$b = 12.445$ (4) Å
$c = 19.475$ (6) Å
$\alpha = 74.74$ (3)°
$\beta = 82.28$ (3)°
$\gamma = 77.68$ (3)°
$V = 1712.6$ (8) $Å^3$
$Z = 2$
$D_x = 1.913$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 3.02$ mm$^{-1}$
$T = 298$ (2) K
Needle, colourless
0.59 × 0.07 × 0.07 mm

### *Data collection*

Stoe IPDS diffractometer
$\varphi$ scans
Absorption correction: none
24267 measured reflections
6202 independent reflections
5073 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 25.9°$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.052$
$S = 0.91$
6202 reflections
541 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0265P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.87$ e Å$^{-3}$
$\Delta\rho_{min} = -0.84$ e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Yb1—O7 | 2.278 (3) | Yb1—O6 | 2.338 (3) |
| Yb1—O2 | 2.305 (3) | Yb1—O5 | 2.339 (3) |
| Yb1—O4 | 2.321 (3) | Yb1—O3 | 2.339 (3) |
| Yb1—O1 | 2.328 (3) | Yb1—O8 | 2.372 (4) |

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

| $D—H\cdots A$ | $D—H$ | $H\cdots A$ | $D\cdots A$ | $D—H\cdots A$ |
|---|---|---|---|---|
| O1—H11*O*···O16$^{i}$ | 0.89 (4) | 2.04 (4) | 2.904 (4) | 165 (6) |
| O1—H12*O*···O31$^{ii}$ | 0.94 (4) | 1.78 (4) | 2.707 (5) | 171 (7) |
| O2—H21*O*···O11$^{iii}$ | 0.90 (4) | 1.88 (4) | 2.778 (4) | 176 (7) |
| O2—H22*O*···O33*A*$^{i}$ | 0.93 (4) | 1.73 (5) | 2.63 (2) | 163 (7) |
| O2—H22*O*···O33$^{i}$ | 0.93 (4) | 1.88 (5) | 2.690 (10) | 145 (6) |
| O3—H31*O*···O12$^{iv}$ | 0.95 (4) | 1.81 (4) | 2.757 (4) | 171 (6) |
| O3—H32*O*···O14$^{v}$ | 0.95 (4) | 1.88 (4) | 2.825 (4) | 172 (6) |
| O4—H41*O*···O11 | 0.96 (4) | 1.97 (5) | 2.880 (4) | 158 (6) |
| O4—H42*O*···O13$^{vi}$ | 0.95 (4) | 1.75 (4) | 2.701 (4) | 176 (7) |
| O5—H51*O*···O32*A*$^{i}$ | 0.91 (4) | 2.08 (6) | 2.95 (4) | 158 (6) |
| O5—H51*O*···O33$^{i}$ | 0.91 (4) | 2.07 (6) | 2.777 (15) | 133 (6) |
| O5—H52*O*···O22$^{iv}$ | 0.93 (4) | 1.97 (4) | 2.862 (4) | 161 (6) |
| O6—H61*O*···O24$^{vii}$ | 0.91 (4) | 2.03 (5) | 2.866 (4) | 152 (6) |
| O6—H62*O*···O32$^{ii}$ | 0.92 (4) | 1.77 (4) | 2.688 (11) | 175 (7) |
| O6—H62*O*···O32*A*$^{ii}$ | 0.92 (4) | 2.42 (7) | 3.33 (5) | 169 (6) |
| O7—H71*O*···O9 | 0.94 (4) | 1.68 (4) | 2.602 (5) | 164 (7) |
| O7—H72*O*···O21$^{iv}$ | 0.90 (4) | 1.87 (4) | 2.752 (4) | 167 (7) |
| O8—H81*O*···O12 | 0.91 (4) | 1.96 (5) | 2.845 (4) | 163 (7) |
| O8—H82*O*···O7 | 0.97 (4) | 1.85 (5) | 2.762 (5) | 155 (6) |
| O8—H82*O*···O9 | 0.97 (4) | 2.50 (7) | 3.038 (5) | 115 (5) |
| O9—H91*O*···O34$^{vii}$ | 0.92 (4) | 1.88 (5) | 2.776 (4) | 164 (7) |
| O9—H92*O*···O23 | 0.96 (4) | 1.78 (4) | 2.720 (5) | 165 (7) |
| O15—H15*O*···O21$^{viii}$ | 0.82 | 1.84 | 2.640 (4) | 165 |
| O16—H16*O*···O14 | 0.82 | 1.88 | 2.600 (4) | 146 |
| O25—H25*O*···O22$^{ix}$ | 0.82 | 1.94 | 2.689 (4) | 151 |
| O26—H26*O*···O24 | 0.82 | 1.94 | 2.654 (4) | 146 |
| O35—H35*O*···O32$^{x}$ | 0.82 | 1.93 | 2.700 (6) | 156 |
| O35—H35*O*···O32*A*$^{x}$ | 0.82 | 1.98 | 2.720 (16) | 150 |
| O36—H36*O*···O34 | 0.82 | 1.93 | 2.644 (5) | 145 |

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

All H atoms on C atoms were positioned geometrically and refined as riding, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The H atoms of the carboxyl and hydroxyl groups were also positioned geometrically and refined as riding, with O—H = 0.82 Å and $U_{iso}(H) = 1.5U_{eq}(O)$. The positions of the water H atoms were not observed directly in Fourier maps. To include them and construct the hydrogen-bond network, the method of Nardelli (1999), as implemented in the *WinGX* software (Farrugia, 1999), has been adopted. The full structure was then refined using restraints [O—H = 0.99 Å, H···H = 1.55 Å and $U_{iso}(H) = 1.5U_{eq}(O)$]. Atoms O32 and O33 are disordered over two positions, with site occupancies of 0.74 (4) and 0.26 (4).

Data collection: *IPDS Software* (Stoe & Cie, 1998); cell refinement: *IPDS Software*; data reduction: *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

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