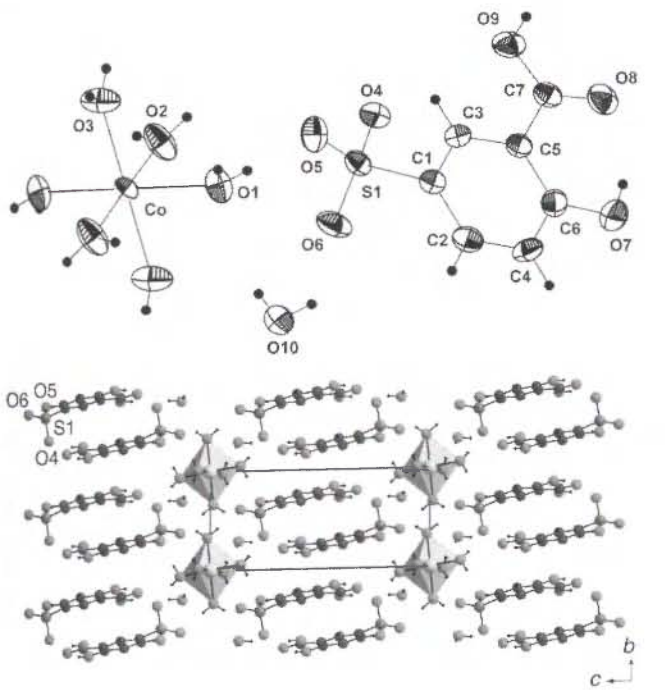
Crystal structure of hexaaquacobalt(II) bis(3-carboxy-4-hydroxybenzenesulfonate) dihydrate, $[Co(H_2O)_6][C_7H_5O_3SO_3]_2 \cdot 2H_2O$

J. Abdelhak, S. Namouchi Cherni and T. Jouini*

Faculté des Sciences, Département de Chimie, 2092 Campus Universitaire, Tunis, Tunisia

Received February 10, 2005, accepted and available on-line March 8, 2005; CCDC no. 1267/1482



Abstract

$C_{14}H_{26}CoO_{20}S_2$, triclinic, $P\bar{1}$ (no. 2), $a = 7.048(1)$ Å, $b = 7.149(1)$ Å, $c = 13.733(2)$ Å, $\alpha = 90.73(1)°$, $\beta = 90.55(1)°$, $\gamma = 118.79(1)°$, $V = 606.3$ Å^3, $Z = 1$, $R_{gt}(F) = 0.025$, $wR_{ref}(F^2) = 0.070$, $T = 293$ K.

Source of material

A mixture of 5-sulfosalicylic acid dihydrate and $CoSO_4 \cdot 7H_2O$ (2:1 molar ratio) in 50 ml ethanol/water (1:1 *v/v*) was heated to boiling point and stirred for two hours. A pink phase being microcrystalline precipitates immediately. After two days, single pink crystals were grown by slow evaporation from aqueous solution at room temperature.

Discussion

The isolated compound, hexaaquacobalt(II) 3-carboxy-4-hydroxybenzensulfonate dihydrate, consists of a hexaaquacobalt(II) cation, the 3-carboxy-4-hydroxybenzensulfonate anion and water molecules (figure, top). Its main features are similar to those of the isostructural compound of $[Cu(H_2O)_6](C_7O_6H_5S)_2 \cdot 2H_2O$ [1], but there are some slight differences which will be discussed. The Co^{II} atom is located on an inversion center and is octahedrally coordinated by six water molecules, while all other atoms are in general positions. The $(C_7O_6H_5S)^-$ is not coordinated to cobalt, but there are important hydrogen bonds joining cation and anion. The Co^{II} ionic radius of 0.72 Å is slightly shorter than that of Cu^{II}, so we notice a light octahedral distortion with regard to the isostructural copper compound [1]. Due to the Jahn-Teller effect, the Co1—O3 distance of 2.062(2) Å is much shorter than those for Co1—O1 and Co1—O2 (2.101(1) Å, 2.112(1) Å, respectively). The Co—O distances and O–Co–O angles are comparable with those found in $[Co(H_2O)_6](C_7H_5O_3SO_3)_2 \cdot 4H_2O$ [2] or $Na_2[Co(H_2O)_6](C_{10}H_4O_8)_2 \cdot 4H_2O$ [3].

The title structure is an alternate stacking of layers of hexaaquacobalt(II) cations and organosulfonate anions. The phenyl rings of the anions are approximately perpendicular to the *b,c* plane. Within the sulfonate layer, the sulfite groups of every two closest organic molecules pointing in opposite direction along the S1–O4 connection (figure, bottom). This packing affords the formation of a short almost linear O–H···$O_{sulfonate}$ hydrogen bond involving each water H atom. The O···O distances range from 2.624(3) Å to 2.856(2) Å, the O–H···O angles from 148(3)° to 179(3)°. The conformation of the sulfonate anion is similar to that of the 2-aminotoluene-4-sulfonate ion in [4], the only difference is due to the substitution of the phenyl ring.

Table 1. Data collection and handling.

Crystal:	pink parallelepiped, size 0.6 × 0.8 × 0.8 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.73 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	53.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3273, 2633
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1939
$N(param)_{refined}$:	222
Programs:	SHELXS-97 [5], SHELXL-97 [6], DIAMOND [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	0.338(4)	0.311(4)	0.258(2)	0.050(6)
H(2)	2*i*	0.828(3)	0.334(3)	0.420(1)	0.032(5)
H(3)	2*i*	0.127(4)	0.212(3)	0.392(2)	0.042(6)
H(4)	2*i*	0.363(4)	−0.280(4)	0.186(2)	0.064(8)
H(5)	2*i*	0.279(4)	0.166(4)	0.610(2)	0.051(7)
H(6)	2*i*	0.479(5)	−0.245(5)	0.118(2)	0.07(1)
H(7)	2*i*	0.897(4)	0.279(4)	0.643(2)	0.064(8)
H(8)	2*i*	0.128(4)	−0.052(4)	−0.167(2)	0.054(7)
H(9)	2*i*	0.113(4)	0.133(5)	−0.175(2)	0.065(8)
H(10)	2*i*	0.294(5)	−0.080(5)	0.074(2)	0.071(9)
H(11)	2*i*	0.199(4)	0.422(4)	−0.033(2)	0.064(8)
H(12)	2*i*	0.168(5)	0.398(5)	0.060(2)	0.070(9)
H(13)	2*i*	0.376(5)	0.123(5)	0.077(2)	0.055(7)

* Correspondence author (e-mail: tahar.jouini@fst.rnu.tn)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co(1)	1*a*	0	0	0	0.0279(2)	0.0235(2)	0.0220(2)	0.0119(1)	–0.0017(1)	0.0002(1)
O(1)	2*i*	0.1429(2)	0.0550(2)	–0.1386(1)	0.0542(8)	0.0360(7)	0.0272(6)	0.0285(6)	0.0037(5)	0.0030(5)
O(2)	2*i*	0.1287(3)	0.3324(2)	0.0140(1)	0.069(1)	0.0255(6)	0.0298(7)	0.0153(6)	0.0004(6)	–0.0012(5)
O(3)	2*i*	0.2812(2)	0.0216(3)	0.0578(1)	0.0372(7)	0.0346(7)	0.0619(9)	0.0146(6)	–0.0202(7)	–0.0019(7)
S(1)	2*i*	0.7855(1)	0.4234(1)	0.2254(1)	0.0316(2)	0.0256(2)	0.0207(2)	0.0108(2)	–0.0011(1)	0.0005(1)
O(4)	2*i*	0.9270(2)	0.6545(2)	0.2425(1)	0.0338(6)	0.0265(6)	0.0284(6)	0.0094(5)	–0.0037(5)	0.0038(4)
O(5)	2*i*	0.9113(2)	0.3113(2)	0.2281(1)	0.0518(8)	0.0409(7)	0.0412(7)	0.0286(6)	0.0094(6)	0.0005(6)
O(6)	2*i*	0.6533(2)	0.3840(2)	0.1373(1)	0.0418(7)	0.0445(7)	0.0240(6)	0.0048(6)	–0.0077(5)	0.0018(5)
C(1)	2*i*	0.6067(3)	0.3323(2)	0.3245(1)	0.0292(8)	0.0215(7)	0.0251(7)	0.0102(6)	0.0002(6)	0.0019(5)
C(2)	2*i*	0.3938(3)	0.2971(3)	0.3164(1)	0.0331(8)	0.0307(8)	0.0308(8)	0.0145(7)	–0.0070(6)	0.0011(6)
C(3)	2*i*	0.6870(3)	0.3096(2)	0.4132(1)	0.0251(7)	0.0240(7)	0.0279(7)	0.0103(6)	–0.0012(6)	0.0016(6)
C(4)	2*i*	0.2638(3)	0.2393(3)	0.3970(1)	0.0260(8)	0.0366(9)	0.0413(9)	0.0149(7)	–0.0035(7)	0.0000(7)
C(5)	2*i*	0.5588(3)	0.2554(2)	0.4957(1)	0.0292(8)	0.0200(7)	0.0253(7)	0.0097(6)	–0.0016(6)	0.0002(5)
C(6)	2*i*	0.3443(3)	0.2193(3)	0.4876(1)	0.0286(8)	0.0257(7)	0.0317(8)	0.0100(6)	0.0035(6)	–0.0005(6)
C(7)	2*i*	0.6487(3)	0.2440(2)	0.5918(1)	0.0335(8)	0.0207(7)	0.0270(8)	0.0083(6)	–0.0024(6)	0.0006(6)
O(7)	2*i*	0.2126(2)	0.1671(3)	0.5644(1)	0.0343(7)	0.0602(9)	0.0366(7)	0.0192(7)	0.0079(6)	0.0008(6)
O(8)	2*i*	0.5466(2)	0.2079(2)	0.6673(1)	0.0456(7)	0.0463(7)	0.0250(6)	0.0159(6)	0.0017(5)	0.0034(5)
O(9)	2*i*	0.8492(2)	0.2780(2)	0.5882(1)	0.0352(7)	0.0480(7)	0.0296(6)	0.0172(6)	–0.0067(5)	0.0052(5)
O(10)	2*i*	0.3686(3)	–0.2900(3)	0.1285(1)	0.0401(8)	0.0539(9)	0.0311(7)	0.0213(7)	–0.0024(6)	–0.0046(6)

References

1. Ma, J.-F.; Yang, J.; Liu, J.-F.: Hexaaquacopper(II) 3-carboxy-4-hydroxybenzenesulfonate dihydrate. Acta Crystallogr. E59 (2003) m485-m486.
2. Ma, J.-F.; Yang, J.; Liu, J.-F.: Hexaaquacobalt(II) 3-carboxy-4-hydroxybenzenesulfonate tetrahydrate. Acta Crystallogr. E59 (2003) m481-m482.
3. Karanović, L.; Poleti, D.; Bogdanović, G. A.; Spasojević-de Biré, A.: Disodium hexaaquacobalt(II) bis[dihydrogen 1,2,4,5-benzenetetracarboxylate(2–)] tetrahydrate. Acta Crystallogr. C55 (1999) 911-913.
4. Gunderman, B. J.; Dubey, S. N.; Squattrito; P. J.: Hexaaquacobalt(II) Bis(2-aminotoluene-4-sulfonate). Acta Crystallogr. C53 (1997) 17-19.
5. Sheldrick, G. M.: SHELXS-86. In: *Crystallographic Computing 3* (Eds. G. M. Sheldrick, C. Krüger, R. Goddard), pp. 175-189. Oxford University Press 1985.
6. Sheldrick; G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
7. Brandenburg. K.: DIAMOND. Visual Crystal Structure Information System. Version 2.0. Crystal Impact GbR, Bonn, Germany 1998.