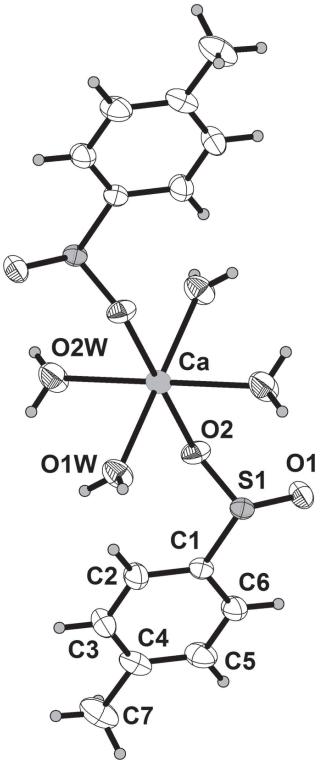
Tai Xi-Shi* and Li Peng-Fei

The crystal structure of *trans*-tetraaqua-bis(*p*-tolylsulfinato-κ*O*)calcium(II)), $C_{14}H_{22}O_8S_2Ca$



https://doi.org/10.1515/ncrs-2017-0206
Received July 16, 2017; accepted October 8, 2017; available online October 27, 2017

Abstract

$C_{14}H_{22}O_8S_2Ca$, monoclinic, $P2_1/c$ (no. 14), $a = 16.349(3)$ Å, $b = 5.0828(10)$ Å, $c = 12.183(2)$ Å, $\beta = 109.29(3)°$, $V = 955.5(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0376$, $wR_{ref}(F^2) = 0.1129$, $T = 293(2)$K.

CCDC no.: 1578699

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.18 × 0.17 × 0.17 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.58 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, ω-scans
$2\theta_{max}$, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6900, 1672, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1603
$N(param)_{refined}$:	116
Programs:	Bruker programs [1], SHELX [2, 3], DIAMOND [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.177639(19)	0.91649(7)	1.40501(3)	0.0260(2)
Ca	0.0000	1.0000	1.5000	0.0289(2)
O1W	0.11949(7)	1.1131(2)	1.66464(9)	0.0423(3)
H1WB	0.1382	1.2587	1.6977	0.063*
H1WA	0.1420	0.9853	1.7103	0.063*
O1	0.16399(7)	0.8873(2)	1.27650(8)	0.0361(3)
O2W	−0.00887(8)	0.6519(2)	1.62043(10)	0.0513(4)
H2WB	0.0365	0.6131	1.6773	0.077*
H2WA	−0.0424	0.5196	1.6101	0.077*
O2	0.10055(6)	0.7977(2)	1.43021(9)	0.0352(3)
C1	0.25877(8)	0.6708(3)	1.46800(11)	0.0274(3)
C2	0.26579(10)	0.5712(3)	1.57614(13)	0.0374(4)
H2	0.2279	0.6274	1.6139	0.045*
C3	0.32924(11)	0.3877(3)	1.62834(15)	0.0432(4)
H3	0.3335	0.3218	1.7012	0.052*
C4	0.38666(10)	0.3000(3)	1.57432(13)	0.0396(4)
C5	0.37915(10)	0.4050(4)	1.46657(14)	0.0433(4)
H5	0.4173	0.3504	1.4289	0.052*
C6	0.31589(10)	0.5904(3)	1.41331(13)	0.0368(4)
H6	0.3122	0.6596	1.3412	0.044*
C7	0.45396(14)	0.0953(3)	1.63094(17)	0.0578(5)
H7A	0.4396	−0.0665	1.5883	0.087*
H7B	0.4554	0.0672	1.7095	0.087*
H7C	0.5097	0.1549	1.6312	0.087*

Source of materials

1.0 mmol sodium *p*-tolylsulfinate (0.178 g) and 0.5 mmol calcium perchlorate tetrahydrate (0.311 g) were dissolved in an aqueous-ethanol (v:v = 1:2) solution. The above mixture was

***Corresponding author: Tai Xi-Shi,** College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong 261061, P.R. China, e-mail: taixs@wfu.edu.cn
Li Peng-Fei: College of Chemical Engineering, Qingdao University of Science and Technology, Qingdao,Shandong 266061, P.R. China

stirred for 5 h at 65 °C. The reactant was filtered. The yellow crystals of the title complex were obtained after 20 days.

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints. Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms.

Comment

Calcium is an important biological element. Relatively few studies have been done on synthesis, structure and property of Ca(II) complexes comparing to transition metal complexes [5–8]. In order to investigate the synthesis, structure and properties of a further Ca(II) complex, the title compound has been synthesized and structurally characterized. The title complex contains one Ca(II) ion, two *p*-tolylsulfinate ligands and four coordinated water molecules. The Ca(II) ion is six-coordinated with two O atoms from two *p*-tolylsulfinate ligands and four O atoms from four coordinated water molecules (*cf*. the figure). The Ca(II) ion adopts a distorted octahedral geometry with two O atoms (O2 and O2A) at the axial positions, and four O atoms(O1W, O1WA, O2W, O2WA) in the equatorial plane. Adjacent complexes are connected by intermolecular hydrogen bonds. Bond lengths and angles are in the expected ranges and fit with a structurally related compound [9] hydrogen bonds.

Acknowledgements: This project was supported by the National Natural Science Foundation of China (no. 21171132), the Natural Science Foundation of Shandong (ZR2014BL003), the project of Shandong Province Higher Educational Science and Technology Program (J14LC01) and Science Foundation of Weifang.

References

1. Bruker: SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2000).
2. Sheldrick, G. M.: A short history of SHELX. Acta. Crystallogr. **A64** (2008) 112–122.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXTL. Acta. Crystallogr. **C71** (2015) 3–8.
4. Brandenburg K.: DIAMOND. Visual Crystal Structure Information System. Ver. 3.2. Crystal Impact, Bonn, Germany, (2012).
5. Yang, Z.-Y.; Yang, R.-D.; Yu, K.-B.: The crystal structures and thermal stability of the complexes of 2- oxo-propionic acid (4-pyridinecarbonyl) hydrazone with alkaline earth metals. Acta. Chim. Sinica. **57** (1999) 236–243.
6. Wu, G.; Zhang, S.-Q.; Wang, X.-F.; Liu, G.-X.: Synthesis, structure, luminescence properties of alkaline earth metal calcium complex with maleic anhydride ligand and a new precursor to produce pure phase micro-sized calcite particles. Synth. React. Inorg. M. **44** (2014) 1429–1434.
7. Tai, X.-S.; Zhang, Y.-P.; Zhao, W.-H.: Synthesis, crystal structure and antitumor activity of a dinuclear calcium complex based on 1,5-naphthalenedisulfonate and 2,2′-bipyridine ligands. Res. Chem. Intermed. **41** (2015) 4339–4347.
8. Tai, X.-S.; Wang, X.: Synthesis and crystal structure of a 1D chained coordination polymer constructed from Ca^{2+} and 2-[(*E*)-(2-furoylhydrazono)methyl]benzenesulfonate. Crystals **5** (2015) 458–465.
9. Langs, D. A.; Hare, C. R.: The structure of bis(toluene-*p*-sulphinato)copper(II) tetrahydrate. Chem. Commun. (1967) 853–854.