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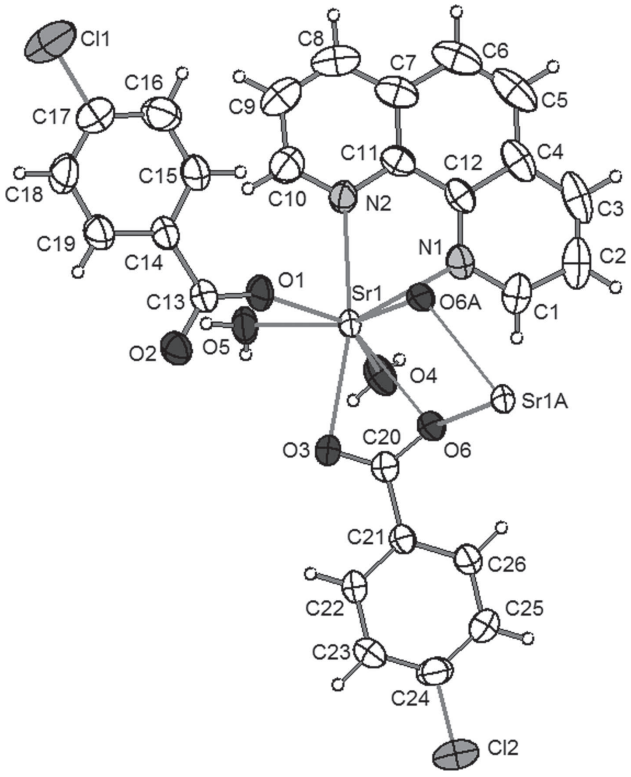
Ling-Xia Hu, Bi-Song Zhang*, Jing-Ping Qiu, Chang-Sheng Wu and Zhen-Xiang Liu

Crystal structure of tetraaquabis(μ$_2$-4-chlorobenzoato-κ^2*O*:*O*,*O*′)bis(4-chlorobenzoato-κ*O*)bis(1,10-phenanthroline-κ^2*N*,*N*′) distrontium(II), $C_{52}H_{40}Cl_4N_4O_{12}Sr_2$



DOI 10.1515/ncrs-2015-0243
Received March 2, 2016; accepted March 21, 2016; available online April 18, 2016

Abstract

$C_{52}H_{40}Cl_4N_4O_{12}Sr_2$, triclinic, $P\bar{1}$ (no. 2), $a = 8.9531(18)$ Å, $b = 9.0657(18)$ Å, $c = 16.911(3)$ Å, $\alpha = 75.56(3)°$, $\beta = 81.96(3)°$, $\gamma = 73.84(3)°$, $V = 1273.0(4)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.0347$, $wR_{ref}(F^2) = 0.0760$, $T = 293(2)$ K.

CCDC no.: 1469747

Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.12×0.19×0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	23.73 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10084, 4461
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3783
$N(param)_{refined}$:	334
Programs:	ABSCOR [6], RAPID-AUTO [7], SADABS [8], SHELX [9]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
HO(4A)	2*i*	0.0685	0.8507	0.4775	0.075
HO(4B)	2*i*	0.1394	0.9052	0.5217	0.075
HO(5A)	2*i*	0.3034	1.0804	0.4509	0.073
HO(5B)	2*i*	0.4328	1.1026	0.4108	0.073
H(1)	2*i*	0.1888	0.4889	0.4766	0.064
H(2)	2*i*	0.0501	0.3563	0.4253	0.082
H(3)	2*i*	0.0347	0.4037	0.2877	0.085
H(5)	2*i*	0.1105	0.5486	0.1434	0.092
H(6)	2*i*	0.2287	0.7264	0.0635	0.088
H(8)	2*i*	0.3674	0.9403	0.0496	0.086
H(9)	2*i*	0.4727	1.0799	0.1123	0.085
H(10)	2*i*	0.4770	1.0163	0.2528	0.068
H(15)	2*i*	0.7596	0.8558	0.2405	0.062
H(16)	2*i*	0.8414	0.9783	0.1122	0.075
H(18)	2*i*	1.0701	1.1948	0.2142	0.071
H(19)	2*i*	0.9858	1.0725	0.3428	0.062
H(22)	2*i*	0.4766	0.7278	0.7397	0.053
H(23)	2*i*	0.4465	0.6424	0.8802	0.064
H(25)	2*i*	0.2317	0.3291	0.8513	0.057
H(26)	2*i*	0.2708	0.4085	0.7106	0.047

***Corresponding author: Bi-Song Zhang,** College of Pharmaceutics and Material Engineering, Jinhua Polytechnic, Jinhua, Zhejiang 321007, People's Republic of China, e-mail: zbs_jy@163.com
Ling-Xia Hu, Jing-Ping Qiu, Chang-Sheng Wu and Zhen-Xiang Liu: College of Pharmaceutics and Material Engineering, Jinhua Polytechnic, Jinhua, Zhejiang 321007, People's Republic of China

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sr(1)	2*i*	0.43522(3)	0.74564(3)	0.43691(2)	0.0325(2)	0.0289(2)	0.0329(2)	−0.0136(1)	−0.0032(1)	−0.0070(1)
N(1)	2*i*	0.2485(3)	0.6168(3)	0.3728(2)	0.040(2)	0.038(2)	0.049(2)	−0.014(1)	−0.008(1)	−0.010(1)
N(2)	2*i*	0.3747(3)	0.8464(3)	0.2756(2)	0.046(2)	0.037(2)	0.042(1)	−0.015(1)	−0.010(1)	−0.003(1)
O(1)	2*i*	0.6938(2)	0.8088(3)	0.3914(1)	0.042(1)	0.060(2)	0.058(1)	−0.033(1)	0.003(1)	−0.017(1)
O(2)	2*i*	0.8517(2)	0.8912(3)	0.4511(1)	0.037(1)	0.054(2)	0.048(1)	−0.018(1)	−0.002(1)	−0.018(1)
O(3)	2*i*	0.4432(3)	0.7512(3)	0.5893(1)	0.063(2)	0.041(1)	0.038(1)	−0.030(1)	−0.003(1)	−0.006(1)
O(4)	2*i*	0.1529(2)	0.8371(3)	0.4955(1)	0.031(1)	0.054(2)	0.073(2)	−0.009(1)	−0.005(1)	−0.032(1)
O(5)	2*i*	0.3717(3)	1.0470(2)	0.4174(1)	0.054(1)	0.039(1)	0.062(1)	−0.023(1)	0.006(1)	−0.019(1)
O(6)	2*i*	0.5977(2)	0.4711(2)	0.4229(1)	0.044(1)	0.031(1)	0.036(1)	−0.010(1)	−0.0023(9)	−0.0116(9)
Cl(1)	2*i*	1.0192(2)	1.1977(2)	0.05503(7)	0.0872(8)	0.116(1)	0.0624(6)	−0.0352(8)	0.0013(6)	0.0168(6)
Cl(2)	2*i*	0.3183(2)	0.4099(2)	0.98378(5)	0.120(1)	0.0993(9)	0.0350(5)	−0.0438(8)	0.0014(5)	−0.0045(5)
C(1)	2*i*	0.1810(4)	0.5106(4)	0.4204(2)	0.050(2)	0.043(2)	0.069(2)	−0.021(2)	−0.007(2)	−0.004(2)
C(2)	2*i*	0.0980(5)	0.4289(5)	0.3899(3)	0.054(2)	0.047(2)	0.110(4)	−0.025(2)	−0.008(2)	−0.014(2)
C(3)	2*i*	0.0889(4)	0.4575(5)	0.3087(3)	0.052(2)	0.060(3)	0.120(4)	−0.020(2)	−0.022(2)	−0.042(3)
C(4)	2*i*	0.1600(4)	0.5670(4)	0.2553(2)	0.038(2)	0.051(2)	0.080(3)	−0.004(2)	−0.017(2)	−0.034(2)
C(5)	2*i*	0.1598(5)	0.6022(6)	0.1675(3)	0.063(3)	0.097(4)	0.090(3)	−0.009(3)	−0.028(2)	−0.057(3)
C(6)	2*i*	0.2283(5)	0.7092(6)	0.1200(3)	0.070(3)	0.102(4)	0.055(2)	−0.008(3)	−0.022(2)	−0.035(2)
C(7)	2*i*	0.3010(4)	0.7981(5)	0.1539(2)	0.052(2)	0.068(3)	0.046(2)	−0.001(2)	−0.013(2)	−0.020(2)
C(8)	2*i*	0.3672(5)	0.9172(5)	0.1064(2)	0.075(3)	0.091(3)	0.037(2)	−0.009(3)	−0.004(2)	−0.006(2)
C(9)	2*i*	0.4307(5)	0.9986(5)	0.1432(2)	0.084(3)	0.063(3)	0.054(2)	−0.023(2)	−0.003(2)	0.010(2)
C(10)	2*i*	0.4325(4)	0.9594(4)	0.2282(2)	0.064(2)	0.055(2)	0.053(2)	−0.022(2)	−0.007(2)	−0.004(2)
C(11)	2*i*	0.3071(4)	0.7666(4)	0.2391(2)	0.036(2)	0.045(2)	0.041(2)	−0.003(2)	−0.009(1)	−0.015(2)
C(12)	2*i*	0.2376(3)	0.6474(4)	0.2907(2)	0.031(2)	0.037(2)	0.051(2)	−0.003(1)	−0.011(1)	−0.018(2)
C(13)	2*i*	0.7963(3)	0.8776(4)	0.3890(2)	0.026(2)	0.034(2)	0.049(2)	−0.007(1)	0.000(1)	−0.016(1)
C(14)	2*i*	0.8601(3)	0.9535(4)	0.3059(2)	0.032(2)	0.037(2)	0.048(2)	−0.011(1)	0.001(1)	−0.018(1)
C(15)	2*i*	0.8208(4)	0.9258(4)	0.2356(2)	0.055(2)	0.059(2)	0.055(2)	−0.032(2)	−0.001(2)	−0.019(2)
C(16)	2*i*	0.8694(4)	0.9985(5)	0.1587(2)	0.065(3)	0.080(3)	0.049(2)	−0.024(2)	−0.007(2)	−0.019(2)
C(17)	2*i*	0.9594(4)	1.1005(5)	0.1519(2)	0.053(2)	0.059(2)	0.051(2)	−0.014(2)	0.002(2)	−0.007(2)
C(18)	2*i*	1.0056(4)	1.1277(4)	0.2198(2)	0.060(2)	0.058(2)	0.067(2)	−0.035(2)	0.008(2)	−0.014(2)
C(19)	2*i*	0.9552(4)	1.0540(4)	0.2966(2)	0.053(2)	0.062(2)	0.054(2)	−0.030(2)	0.004(2)	−0.023(2)
C(20)	2*i*	0.4101(3)	0.6232(3)	0.6192(2)	0.026(2)	0.032(2)	0.038(2)	−0.006(1)	−0.005(1)	−0.008(1)
C(21)	2*i*	0.3803(3)	0.5747(3)	0.7106(2)	0.032(2)	0.029(2)	0.036(2)	−0.007(1)	−0.003(1)	−0.010(1)
C(22)	2*i*	0.4297(4)	0.6455(4)	0.7620(2)	0.059(2)	0.038(2)	0.043(2)	−0.024(2)	−0.001(2)	−0.011(2)
C(23)	2*i*	0.4105(4)	0.5961(4)	0.8460(2)	0.077(3)	0.055(2)	0.039(2)	−0.028(2)	−0.009(2)	−0.016(2)
C(24)	2*i*	0.3377(4)	0.4779(4)	0.8780(2)	0.059(2)	0.054(2)	0.032(2)	−0.015(2)	0.002(2)	−0.007(2)
C(25)	2*i*	0.2831(4)	0.4078(4)	0.8286(2)	0.052(2)	0.049(2)	0.044(2)	−0.025(2)	0.003(2)	−0.005(2)
C(26)	2*i*	0.3059(4)	0.4561(4)	0.7446(2)	0.043(2)	0.043(2)	0.039(2)	−0.019(2)	−0.001(1)	−0.013(1)

Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

$SrCl_2{\cdot}6H_2O$ (0.1334 g, 0.50 mmol) was dissolved in 5 mL distilled water, and then 8 mL 1M Na_2CO_3 solution was added. $SrCO_3$ was obtained by filtration, which was then washed with 10 mL distilled water for 5 times. The freshly prepared $SrCO_3$, 4-chlorobenzoic acid (0.0404 g, 0.26 mmol), 1,10-phenanthrine monohydrate (0.0509 g, 0.26 mmol), CH_3OH (5 mL), and distilled water (10 mL) were mixed and stirred for 2.0 h. Subsequently, the resulting cream suspension was heated in a 23 mL Teflon-lined stainless steel autoclave at 433 K for 97 h. After the autoclave was cooled to room temperature after 43 h, the solid was filtered off. The resulting filtrate allowed to evaporate at room temperature for 4 weeks afforded colorless block crystals.

Discussion

Strontium(II) complexes are part of a study on the metal ions with halogen benzoic acid complexes. As part of this study we have already reported a chain-type strontium(II) complex [1]. In this paper we report synthesis and crystal structure of a dinuclear 4-chlorobenzoato strontium(II) complex. The crystal structure of title compound is similar to the structures $[Pb(FC_6H_4COO)_2(C_{12}H_8N_2)]$ [2], $[Cd(FC_7H_4COO)_2(C_{12}H_8N_2)]$ [3], $La_2(C_{12}H_8N_2)_2$ $(ClC_7H_4O_2)_6$ [4] and $Mn_2(C_7H_4O_2I)_2(C_{12}H_8N_2)_4[I]_2{\cdot}2H_2O$ [5]. Within the title compound, each Sr(II) atom is coordinated by two nitrogen

atoms from one chelating 1,10-phenanthroline (phen) ligands and six oxygen atoms from two water molecules and three 4-chlorobenzoato ligands to complete a significantly distorted SrN_2O_6 square antiprismatic coordination geometry. The μ_2 carboxylate group of two 4-chlorobenzoic anions connect Sr1 and $Sr1^{i}$ (i: $-x+1,-y+1,-z+1$) to complete a dinuclear complex (figure). The Sr—O bond lengths are in the range of 2.510(2) to 2.715(2) Å, the Sr—N bond lengths are 2.733(3) and 2.750(3) Å, and the Sr(1) and $Sr(1)^{i}$ distance is 4.337(2) Å. Two symmetry related dinuclear molecules giving rise to a one-dimensional supramolecular chain structure extending along the [001] direction by O4—HO4$\cdots$$O2^{ii}$ (ii: $x-1$, y, z) hydrogen bonds. The one-dimensional chains are connected via O4(O5)—HO4(HO5)$\cdots$$O2^{iii}$ ($O3^{iii}$) (iii: $-x+1$, $-y+2$, $-z+1$) hydrogen bonds to a two-dimensional layer parallel to the (001) plane, with O—H$\cdots$O bond lengths in the range of 2.763 to 2.831 Å and the O—H$\cdots$O bond angles in the range of 159 to 171°.

Acknowledgements: Authors gratefully acknowledge the financial support of the National Natural Science Foundation of China (grovint no:51343003). The authors thank the responsible editor for supplying the figure.

References

1. Zhang, B.-S.: *catena*-poly[[diaquastrontium]-bis(μ-2-bromobenzoato)-κ²*O*,*O*′]. Acta Crystallogr. **E65** (2009) m1500.
2. Zhang, B.-S.: Crystal structure of (2,2′-bipyridine-κ*N*,*N*′)bis(2-fluorobenzoato)lead(II), $Pb(C_7H_4O_2F)_2(C_{10}H_8N_2)$. Z. Kristallogr. NCS **221** (2006) 355–356.
3. Lou, Q.-Z.: Crystal structure of (1,10-phenanthroline-*N*,*N*′)bis(2-fluorobenzoato)cadmium(II), $Cd(C_7H_4O_2F)_2(C_{12}H_8N_2)$. Z. Kristallogr. NCS **222** (2007) 105–106.
4. Zhang, B.-S.: Crystal structure of bis-(1,10-phenanthroline-*N*,*N*′)bis(2-chlorobenzoato)tetrakis(μ₂-chlorobenzoato) dilanthanum(III), $La_2(C_{12}H_8N_2)_2(ClC_7H_4O_2)_6$. Z. Kristallogr. NCS **221** (2006) 509–510.
5. Zhang, B.-S.: Crystal structure of bis(bis(1,10-phenanthroline-*N*,*N*′)(μ₂-iodobenzoato)manganese(II)) diiodide dihydrate, $[Mn_2(C_7H_4O_2I)_2(C_{12}H_8N_2)_4][I]_2 \cdot 2H_2O$. New Cryst. Struct. NCS **222** (2007) 274–276.
6. Higashi, T.: ABSCOR. Rigaku Corporation, Tokyo, Japan, 1995.
7. Rigaku: RAPID-AUTO and Crystal Structure. Rigaku Corporation, 1998.
8. Sheldrick, G. M.: SADABS. University of Göttingen, Germany, 1996.
9. Sheldrick, G. M.: A short history of SHELX. Acta Cryst. **A64** (2008) 112–122.