Pei-xuan Li* and Wei Hui

Crystal structure of poly[triaqua-bis(μ_3 -3,3'-((5-carboxylato-1,3-phenylene)bis(oxy))dibenzoato)-tris(1,10-phenanthroline)cobalt(II)], $C_{78}H_{46}N_6O_{20}Co_3$

Table 1: Data collection and handling.

Crystal:	Pink block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.83 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	33479, 11606, 0.051
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 8450
$N(param)_{refined}$:	964
Programs:	SHELX [1], Bruker [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
Co1	0.29610(6)	0.13132(2)	0.51774(4)	0.03142(14)
Co2	−0.25365(6)	0.00405(2)	0.86193(4)	0.03301(14)
Co3	−0.09238(6)	0.19125(2)	1.16675(4)	0.03625(15)
O1	−0.1057(3)	0.03522(5)	0.9002(2)	0.0355(6)
O1W	0.3092(3)	0.13568(6)	0.3870(2)	0.0396(7)
H1WB	0.2720(4)	0.1511(4)	0.3580(7)	0.048*
H1WC	0.2554(4)	0.1233(4)	0.3414(7)	0.048*
O2	0.0623(3)	0.02581(5)	1.07235(19)	0.0356(6)
O2W	−0.2046(3)	0.00334(6)	1.01586(19)	0.0400(7)
H2WB	−0.2678(2)	0.0137(5)	1.0158(5)	0.048*
H2WC	−0.1183(2)	0.0105(5)	1.0572(5)	0.048*
O3	0.0519(3)	0.06144(6)	0.8301(2)	0.0426(7)
O3W	−0.1093(3)	0.18379(6)	1.2898(2)	0.0439(7)
H3WB	−0.0643(8)	0.1964(1)	1.3360(2)	0.053*
H3WC	−0.0656(2)	0.1687(5)	1.3191(7)	0.053*
O4	0.0441(3)	0.07409(6)	0.5201(2)	0.0398(7)
O4W	0.8921(5)	0.24621(9)	0.4900(3)	0.0962(14)
H4WC	0.9292(5)	0.2616(2)	0.5186(2)	0.115*
H4WD	0.9276(4)	0.2414(2)	0.4548(1)	0.115*
O5	0.1543(3)	0.09004(6)	0.3103(2)	0.0435(7)
O6	0.2474(3)	0.09151(5)	0.48322(19)	0.0348(6)
O7	0.5686(3)	0.03645(7)	0.9453(2)	0.0571(9)
O8	0.5574(3)	0.03054(6)	0.7961(2)	0.0440(7)
O9	0.0297(3)	0.19677(6)	1.0949(2)	0.0418(7)
O10	0.1445(3)	0.17952(6)	1.2540(2)	0.0425(7)
O11	0.2002(3)	0.17607(6)	1.0206(2)	0.0467(8)
O12	0.0104(4)	0.26928(6)	0.8735(2)	0.0606(9)
O13	−0.0484(3)	0.26812(6)	0.7085(2)	0.0485(8)

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Abstract

$C_{78}H_{46}N_6O_{20}Co_3$, monoclinic, $P2_1/c$ (no. 14), $a = 10.789(4)$ Å, $b = 48.885(18)$ Å, $c = 15.205(4)$ Å, $\beta = 124.604(19)^\circ$, $V = 6601(4)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0539$, $wR_{ref}(F^2) = 0.1205$, $T = 293(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
O14	0.0528(3)	0.16991(6)	0.6654(2)	0.0483(8)
O15	0.1803(3)	0.13622(6)	0.5916(2)	0.0419(7)
O16	0.0538(3)	0.14296(6)	0.4209(2)	0.0406(7)
N1	-0.2986(4)	-0.00518(7)	0.7117(2)	0.0395(8)
N2	-0.4116(4)	-0.02759(7)	0.8106(3)	0.0423(9)
N3	-0.1738(4)	0.15118(7)	1.1102(3)	0.0410(8)
N4	-0.3064(4)	0.19901(7)	1.0288(3)	0.0396(8)
N5	0.3873(4)	0.17139(7)	0.5671(3)	0.0375(8)
N6	0.5107(3)	0.12354(7)	0.6556(2)	0.0333(8)
C1	-0.2440(5)	0.00679(11)	0.6637(4)	0.0554(13)
H1A	-0.179168	0.021608	0.696064	0.066*
C2	-0.2826(7)	-0.00255(13)	0.5628(4)	0.0701(17)
H2A	-0.244055	0.006123	0.528780	0.084*
C3	-0.3760(7)	-0.02421(14)	0.5168(4)	0.0744(18)
H3A	-0.401409	-0.030465	0.450705	0.089*
C4	-0.4338(6)	-0.03706(11)	0.5660(4)	0.0596(14)
C5	-0.3928(5)	-0.02680(9)	0.6641(3)	0.0453(11)
C6	-0.4552(5)	-0.03843(9)	0.7165(3)	0.0433(11)
C7	-0.5574(6)	-0.06038(10)	0.6699(4)	0.0627(14)
C8	-0.5934(7)	-0.07000(13)	0.5711(5)	0.0855(19)
H8A	-0.660795	-0.084484	0.539074	0.103*
C9	-0.5370(7)	-0.05960(13)	0.5214(5)	0.0791(19)
H9A	-0.564416	-0.066962	0.456286	0.095*
C10	-0.6159(6)	-0.06989(12)	0.7253(6)	0.0812(19)
H10A	-0.685125	-0.084156	0.697103	0.097*
C11	-0.5736(6)	-0.05873(13)	0.8188(5)	0.0778(17)
H11A	-0.612349	-0.065126	0.856121	0.093*
C12	-0.4719(5)	-0.03768(10)	0.8582(4)	0.0580(13)
H12A	-0.443585	-0.030007	0.923119	0.070*
C13	0.0123(4)	0.04023(8)	0.9896(3)	0.0294(9)
C14	0.0999(4)	0.06513(8)	1.0029(3)	0.0299(9)
C15	0.1633(5)	0.07982(9)	1.0967(3)	0.0408(10)
H15A	0.150131	0.073674	1.148783	0.049*
C16	0.2451(5)	0.10317(9)	1.1154(4)	0.0529(12)
H16A	0.284491	0.113041	1.178182	0.063*
C17	0.2676(6)	0.11164(9)	1.0400(4)	0.0536(12)
H17A	0.326518	0.127015	1.053005	0.064*
C18	0.2049(5)	0.09789(9)	0.9456(4)	0.0445(11)
H18A	0.219497	0.104112	0.894268	0.053*
C19	0.1201(4)	0.07483(8)	0.9266(3)	0.0335(9)
C20	0.1298(4)	0.06067(8)	0.7823(3)	0.0349(9)
C21	0.2736(4)	0.05104(8)	0.8360(3)	0.0348(9)
H21A	0.324499	0.046246	0.907733	0.042*
C22	0.3426(4)	0.04845(8)	0.7836(3)	0.0317(9)
C23	0.2663(4)	0.05527(8)	0.6769(3)	0.0330(9)
H23A	0.311622	0.053006	0.640555	0.040*
C24	0.1217(4)	0.06552(8)	0.6249(3)	0.0326(9)
C25	0.0515(4)	0.06827(9)	0.6766(3)	0.0374(10)
H25A	-0.046066	0.075077	0.641267	0.045*
C26	0.5022(5)	0.03776(8)	0.8463(3)	0.0363(10)
C27	0.0134(4)	0.05489(8)	0.4435(3)	0.0359(10)
C28	-0.0683(5)	0.03230(10)	0.4326(3)	0.0518(12)
H28A	-0.089876	0.028624	0.482597	0.062*
C29	-0.1180(6)	0.01517(10)	0.3485(4)	0.0619(14)
H29A	-0.174455	-0.000131	0.341035	0.074*
C30	-0.0867(5)	0.02004(9)	0.2750(3)	0.0533(13)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H30A	-0.123111	0.008478	0.216622	0.064*
C31	-0.0008(5)	0.04221(8)	0.2886(3)	0.0411(10)
H31A	0.021323	0.045542	0.238748	0.049*
C32	0.0546(4)	0.05994(8)	0.3741(3)	0.0306(9)
C33	0.1585(4)	0.08231(8)	0.3896(3)	0.0314(9)
C34	0.0606(4)	0.14196(8)	0.5064(3)	0.0327(9)
C35	-0.0800(4)	0.14716(8)	0.5009(3)	0.0355(9)
C36	-0.2150(5)	0.13846(9)	0.4119(4)	0.0472(11)
H36A	-0.217321	0.130673	0.355181	0.057*
C37	-0.3469(5)	0.14116(10)	0.4058(4)	0.0549(13)
H37A	-0.437454	0.135130	0.345469	0.066*
C38	-0.3439(5)	0.15257(11)	0.4876(4)	0.0570(13)
H38A	-0.432520	0.153761	0.484352	0.068*
C39	-0.2124(5)	0.16246(10)	0.5758(4)	0.0472(11)
H39A	-0.212404	0.170786	0.630791	0.057*
C40	-0.0808(4)	0.15997(8)	0.5820(3)	0.0351(9)
C41	0.0574(4)	0.18532(9)	0.7441(3)	0.0372(10)
C42	0.0181(4)	0.21233(9)	0.7265(3)	0.0393(10)
H42A	-0.024930	0.220038	0.659194	0.047*
C43	0.0440(4)	0.22802(8)	0.8118(3)	0.0343(9)
C44	0.1094(4)	0.21643(8)	0.9107(3)	0.0373(10)
H44A	0.129380	0.227004	0.968242	0.045*
C45	0.1454(5)	0.18915(8)	0.9244(3)	0.0366(10)
C46	0.1191(4)	0.17322(9)	0.8414(3)	0.0381(10)
H46A	0.142602	0.154684	0.851035	0.046*
C47	0.3137(5)	0.18880(8)	1.1135(3)	0.0393(10)
C48	0.4480(6)	0.19495(11)	1.1267(4)	0.0598(14)
H48A	0.458847	0.192547	1.070773	0.072*
C49	0.5629(6)	0.20447(12)	1.2217(5)	0.0661(15)
H49A	0.653850	0.208468	1.230866	0.079*
C50	0.5503(5)	0.20845(11)	1.3054(4)	0.0610(14)
H50A	0.630735	0.215238	1.370312	0.073*
C51	0.4162(5)	0.20217(10)	1.2910(3)	0.0526(12)
H51A	0.406339	0.204384	1.347565	0.063*
C52	0.2944(4)	0.19256(8)	1.1934(3)	0.0346(9)
C53	0.1475(5)	0.18911(8)	1.1786(3)	0.0340(9)
C54	0.3250(5)	0.19484(10)	0.5228(4)	0.0529(12)
H54A	0.230079	0.194876	0.458733	0.063*
C55	0.3941(6)	0.21948(10)	0.5670(5)	0.0669(15)
H55A	0.346622	0.235735	0.532399	0.080*
C56	0.5300(6)	0.22005(11)	0.6602(4)	0.0647(14)
H56A	0.576254	0.236740	0.690398	0.078*
C57	0.6013(5)	0.19605(9)	0.7113(4)	0.0461(11)
C58	0.5236(5)	0.17167(9)	0.6595(3)	0.0375(10)
C59	0.5908(4)	0.14617(8)	0.7072(3)	0.0318(9)
C60	0.7333(4)	0.14525(10)	0.8040(3)	0.0413(11)
C61	0.8060(5)	0.16988(12)	0.8551(4)	0.0537(13)
H61A	0.898816	0.169355	0.920792	0.064*
C62	0.7439(5)	0.19402(11)	0.8109(4)	0.0538(13)
H62A	0.795241	0.209960	0.846159	0.065*
C63	0.7924(5)	0.11932(11)	0.8443(4)	0.0539(13)
H63A	0.887973	0.117604	0.907131	0.065*
C64	0.7116(5)	0.09665(10)	0.7925(4)	0.0518(12)
H64A	0.750664	0.079335	0.819270	0.062*
C65	0.5697(5)	0.09966(9)	0.6988(3)	0.0418(10)
H65A	0.513392	0.084007	0.664881	0.050*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C66	−0.3828(4)	0.17628(9)	0.9740(3)	0.0364(10)
C67	−0.5237(5)	0.17724(10)	0.8771(3)	0.0453(11)
C68	−0.5840(5)	0.20280(13)	0.8371(4)	0.0615(15)
H68A	−0.676883	0.204399	0.771927	0.074*
C69	−0.5086(6)	0.22540(13)	0.8921(4)	0.0685(15)
H69A	−0.550289	0.242602	0.865777	0.082*
C70	−0.3686(5)	0.22300(10)	0.9884(4)	0.0515(12)
H70A	−0.317184	0.238784	1.025353	0.062*
C71	−0.5960(5)	0.15238(11)	0.8260(3)	0.0507(13)
H71A	−0.688971	0.152894	0.760459	0.061*
C72	−0.5342(5)	0.12823(11)	0.8697(4)	0.0508(12)
H72A	−0.585364	0.112220	0.834948	0.061*
C73	−0.3902(5)	0.12670(9)	0.9692(3)	0.0418(11)
C74	−0.3140(4)	0.15090(9)	1.0185(3)	0.0360(10)
C75	−0.3184(6)	0.10230(10)	1.0168(4)	0.0561(13)
H75A	−0.365733	0.085707	0.986190	0.067*
C76	−0.1778(6)	0.10281(10)	1.1088(4)	0.0627(14)
H76A	−0.127944	0.086558	1.141727	0.075*
C77	−0.1097(5)	0.12768(10)	1.1531(4)	0.0581(13)
H77A	−0.013782	0.127717	1.216374	0.070*
C78	−0.0026(5)	0.25731(9)	0.7965(3)	0.0384(10)

Source of material

The 3,5-bis(3-carboxyphenoxy)benzoic acid 39.4 mg, (0.1 mmol), 1,10-phenanthroline hydrate 19.8 mg (0.1 mmol) and cobalt (II) acetate 17.7 mg (0.1 mmol) were dissolved in 5 mL methanol and acetonitrile and stirred for 15 minutes. The mixture was transferred to the autoclave lined with PTFE and heated at 200 °C for 12 hours. Then it was reduced to 120 °C and heated for 4 hours, and finally slowly reduced to room temperature.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [1]. The hydrogen atoms were placed at calculated positions and refined as riding atoms.

Comment

Functional complexes are widely used in gas storage and separation, catalysis, drug delivery, chemical sensing and so on. Three benzene rings can support the space well and form functional complexes more powerfully, and the existence of oxygen ion give them amount of space to rotate and twist, to provide different coordinations. In this project we used 1,10-phenanthroline as auxiliary ligands [3, 4] to synthesize the title complexes.

The asymmetric unit of the title structure contains three Co metal atoms, three phenanthroline molecules, two anionic 3,3'-((5-carboxylato-1,3-phenylene)bis(oxy)) dibenzoates and four water molecules (three of them are Co-coordinated). The anionic ligands connect three Co(II) nodes to a 3D network, whereas the 1,10-phenanthroline and water ligands are not in a bridging mode. Geometric parameters are all in the expected ranges [5].

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References

- Sheldrick, G. M.: SHELXTL, Version 5.1, Software Reference Manual, Madison, WI, USA (1997).
- BRUKER. SAINT, APEX2 and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
- Ramadan, A. M.; Shaban, S. Y.; Ibrahim, M. M.; El-Shami, F. I.; Al-Harbi, S. A.: Ternary complexes containing copper(II), *L*-valinate and α,α' -bipyridyl or 1,10-phenanthroline: synthesis, characterization, ligand substitution and oxidase biomimetic catalytic activity studies. *J. Mol. Struct.* **1189** (2019) 360–376.
- Lu, J. F.; Jin, L. X.; Song, J.; Zhao, C. B.; Yue, S. Y.; Li, L.; Yang, H. T.; Cao, X. Y.; Ge, H. G.: A novel 3D metal coordination polymer based on tetranuclear cobalt cluster building blocks: synthesis, crystal structure and photocatalytic property. *Chin. J. Struct. Chem.* **37** (2018) 1814–1820.
- Fan, L.; Gao, L.; Wang, X.; Wang, J.; Zhao, L.; Fang, K.; Hu, T.: Structural diversity and magnetic properties of four Cu(II)/Co(II) coordination complexes based on 3,5-bis(2-carboxylphenoxy) benzoic acid. *Polyhedron* **141** (2018) 133–139.