Crystal structure of mono-aqua-bis((μ -homophthalato- O,O',O'')-(1,10-phenanthroline)cobalt(II)), $\text{Co}(\text{H}_2\text{O})(\text{C}_{12}\text{H}_8\text{N}_2)(\text{C}_9\text{H}_6\text{O}_4)$

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Abstract

$\text{C}_{42}\text{H}_{32}\text{Co}_2\text{N}_4\text{O}_{10}$, monoclinic, $P12_1/c1$ (no. 14), $a = 8.8186(6)$ Å, $b = 19.199(1)$ Å, $c = 13.0989(8)$ Å, $\beta = 123.070(4)^\circ$, $V = 1858.5$ Å³, $Z = 2$, $R_g(F) = 0.037$, $wR_{\text{ref}}(F^2) = 0.092$, $T = 173$ K.

Source of material

Homophthalic acid (H_2hpht , 0.0275 g, 0.15 mmol), 1,10-phenanthroline (phen, 0.030 g, 0.15 mmol), and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.045 g, 0.15 mmol) were put in 10 mL distilled H_2O and the pH was adjusted to about 4.2 by diluted aqueous solution of NaOH. Then the mixture was sealed in 25 mL Teflon-lined reactor and heated to 160 °C for 3 days, and then slowly cooled to room temperature. Red prismatic crystals were collected and washed with distilled water (yield, 47 %).

Experimental details

H atoms bonded to C atoms were placed in calculated positions ($d(\text{C}—\text{H}) = 0.95$ Å) and included into refinement in the riding model approximation. The H atoms on water molecules were located from difference Fourier maps.

Discussion

The title compound is isostructural with $\text{C}_{42}\text{H}_{32}\text{N}_4\text{O}_{10}\text{Zn}_2$ [1,2]. The asymmetric unit of the crystal structure consists of a $\text{Co}(\text{II})$ ion, a phen, a hpht^{2-} (hpht = homophthalate) species and a coordinated water molecule. The $\text{Co}(\text{II})$ is six-coordinated by two nitrogen atoms from phen ligand and four O atoms, three of which from two hpht^{2-} and one from coordinating H_2O in a distorted octahedral manner. The $\text{Co}—\text{O}(\text{N})$ bond distances are in the range 2.036–2.041 Å. In the hpht ligand, the carboxylate group monodentately

coordinates $\text{Co}(\text{II})$ and the ethylic carboxylate group chelates another $\text{Co}(\text{II})$. Two hpht link two $\text{Co}(\text{II})$ ions to form the dimeric basic building unit. Such units are linked together to form 1D double-chains by the hydrogen bonds via the coordinated water and one of ethyl carboxyl oxygen atoms. Another type of hydrogen bonding formed by coordinated water and uncoordinated oxygen atom of carboxylate is also observed in the chain. The double-chains are further stacked by π – π interactions of phen to form the final layer structure with the phenyl planes of hpht ligands from two layers meshed.

Table 1. Data collection and handling.

Crystal:	red prism, size $0.28 \times 0.40 \times 0.45$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	9.61 cm ^{−1}
Diffractometer, scan mode:	Bruker SMART CCD, ϕ/ω
$2\theta_{\text{max}}$:	54.96°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	14190, 4251
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3829
$N(\text{param})_{\text{refined}}$:	262
Programs:	SHELXS-97 [3], SHELXL-97 [4], SHELXTL [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.4057	0.2475	0.6649	0.052
H(4)	4e	0.6896	0.1983	0.7900	0.063
H(5)	4e	0.9225	0.2275	0.7638	0.068
H(6)	4e	0.8742	0.3107	0.6213	0.061
H(8A)	4e	0.6369	0.3706	0.4204	0.050
H(8B)	4e	0.4733	0.4056	0.4218	0.050
H(10)	4e	0.1246	0.3434	0.1732	0.053
H(11)	4e	0.0137	0.3201	−0.0306	0.063
H(12)	4e	−0.2088	0.3909	−0.1822	0.059
H(14)	4e	−0.4201	0.4958	−0.2383	0.058
H(15)	4e	−0.5266	0.5835	−0.1767	0.059
H(17)	4e	−0.5083	0.6525	−0.0018	0.060
H(18)	4e	−0.3757	0.6651	0.2063	0.065
H(1WA)	4e	−0.0286	0.3818	0.4255	0.065
H(1WB)	4e	−0.1357	0.4379	0.4167	0.065
H(19)	4e	−0.1535	0.5871	0.3402	0.058

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Co(1)	4e	0.05946(3)	0.45506(1)	0.32411(2)	0.0315(2)	0.0394(2)	0.0274(1)	0.0017(1)	0.0147(1)	0.0005(1)
O(1)	4e	0.2320(2)	0.37255(8)	0.3960(1)	0.0401(7)	0.0524(8)	0.0379(8)	0.0123(6)	0.0225(6)	0.0105(6)
N(1)	4e	−0.0276(2)	0.42536(9)	0.1458(1)	0.0347(8)	0.0425(9)	0.0300(8)	0.0008(7)	0.0164(7)	−0.0013(7)
C(1)	4e	0.2770(3)	0.33899(9)	0.4918(2)	0.040(1)	0.0303(9)	0.0331(9)	0.0006(7)	0.0194(8)	−0.0029(7)
O(2)	4e	0.1712(2)	0.32612(9)	0.5249(2)	0.0487(9)	0.0605(9)	0.058(1)	0.0067(7)	0.0356(8)	0.0155(8)
N(2)	4e	−0.1466(2)	0.53058(9)	0.2176(2)	0.0328(8)	0.0452(9)	0.0321(8)	0.0024(7)	0.0154(7)	−0.0025(7)
C(2)	4e	0.4681(3)	0.31115(9)	0.5686(2)	0.040(1)	0.0256(8)	0.0302(9)	0.0013(7)	0.0168(8)	−0.0024(7)
O(3)	4e	0.8051(2)	0.48155(7)	0.5062(1)	0.0441(8)	0.0479(8)	0.0438(8)	−0.0102(6)	0.0297(7)	−0.0074(6)
C(3)	4e	0.5014(3)	0.2612(1)	0.6560(2)	0.055(1)	0.032(1)	0.042(1)	0.0016(8)	0.026(1)	0.0046(8)
O(4)	4e	0.7358(2)	0.46980(7)	0.6415(1)	0.0501(8)	0.0477(8)	0.0331(7)	−0.0039(6)	0.0252(7)	−0.0024(6)
C(4)	4e	0.6693(3)	0.2312(1)	0.7296(2)	0.066(2)	0.032(1)	0.044(1)	0.009(1)	0.021(1)	0.0089(9)
C(5)	4e	0.8074(3)	0.2492(1)	0.7153(2)	0.050(1)	0.045(1)	0.052(1)	0.016(1)	0.013(1)	0.003(1)
C(6)	4e	0.7777(3)	0.2986(1)	0.6303(2)	0.039(1)	0.055(1)	0.052(1)	0.0046(9)	0.020(1)	−0.001(1)
C(7)	4e	0.6101(3)	0.3318(1)	0.5566(2)	0.037(1)	0.0355(9)	0.0310(9)	0.0006(7)	0.0157(8)	−0.0034(7)
C(8)	4e	0.6000(3)	0.3894(1)	0.4742(2)	0.038(1)	0.053(1)	0.032(1)	−0.0106(9)	0.0178(9)	−0.0020(9)
C(9)	4e	0.7199(2)	0.45035(9)	0.5452(2)	0.0263(9)	0.039(1)	0.0313(9)	0.0055(7)	0.0141(8)	0.0050(7)
C(10)	4e	0.0331(3)	0.3726(1)	0.1121(2)	0.046(1)	0.047(1)	0.040(1)	0.0057(9)	0.023(1)	−0.0009(9)
C(11)	4e	−0.0325(3)	0.3585(1)	−0.0098(2)	0.063(1)	0.053(1)	0.046(1)	0.002(1)	0.033(1)	−0.010(1)
C(12)	4e	−0.1635(3)	0.4001(1)	−0.0991(2)	0.059(1)	0.059(1)	0.033(1)	−0.006(1)	0.027(1)	−0.0081(9)
C(13)	4e	−0.2308(3)	0.4566(1)	−0.0671(2)	0.041(1)	0.048(1)	0.032(1)	−0.0085(8)	0.0194(9)	−0.0011(8)
C(14)	4e	−0.3704(3)	0.5023(1)	−0.1538(2)	0.051(1)	0.060(1)	0.027(1)	−0.001(1)	0.0165(9)	0.0064(9)
C(15)	4e	−0.4322(3)	0.5545(1)	−0.1172(2)	0.045(1)	0.058(1)	0.035(1)	0.006(1)	0.016(1)	0.0156(9)
C(16)	4e	−0.3595(3)	0.5673(1)	0.0088(2)	0.035(1)	0.045(1)	0.038(1)	0.0000(8)	0.0169(9)	0.0073(9)
C(17)	4e	−0.4162(3)	0.6212(1)	0.0533(2)	0.047(1)	0.048(1)	0.054(1)	0.0114(9)	0.026(1)	0.013(1)
C(18)	4e	−0.3388(3)	0.6285(1)	0.1756(2)	0.056(1)	0.051(1)	0.055(1)	0.014(1)	0.031(1)	0.000(1)
C(19)	4e	−0.2050(3)	0.5819(1)	0.2553(2)	0.046(1)	0.056(1)	0.040(1)	0.006(1)	0.022(1)	−0.006(1)
C(20)	4e	−0.1585(2)	0.4665(1)	0.0567(2)	0.0289(9)	0.040(1)	0.0293(9)	−0.0033(7)	0.0153(8)	0.0029(7)
C(21)	4e	−0.2223(2)	0.5233(1)	0.0966(2)	0.0295(9)	0.041(1)	0.0307(9)	−0.0034(7)	0.0152(8)	0.0013(8)
O(1W)	4e	−0.1044(2)	0.40843(7)	0.3724(1)	0.0352(7)	0.0443(7)	0.0423(8)	−0.0050(6)	0.0222(6)	−0.0076(6)

References

1. He, J. R.; Wang, Y. L.; Bi, W. H.; Zhu, X. D.: Syntheses and crystal structures of three new Zn(II) complexes constructed by homophthalate and diimine ligands. *J. Mol. Struct.* **787** (2006) 63–68.
2. Sun, J. H.: Di- μ -homophthalato- $\kappa^3O:O',O''\kappa^3O',O''$:O-bis[aqua(1,10-phenanthroline)zinc(II)]. *Acta Crystallogr.* **E62** (2006) m2799–m2801.
3. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 6.14. Bruker AXS, Madison, Wisconsin, USA 2000.