Crystal structure of *catena*-(μ_4-5-bromoisophthalato)-(μ_2-1,6-bis(imidazol-1-yl)hexane)cobalt(II), $Co(C_8H_3O_4Br)(C_{12}H_{18}N_4)$

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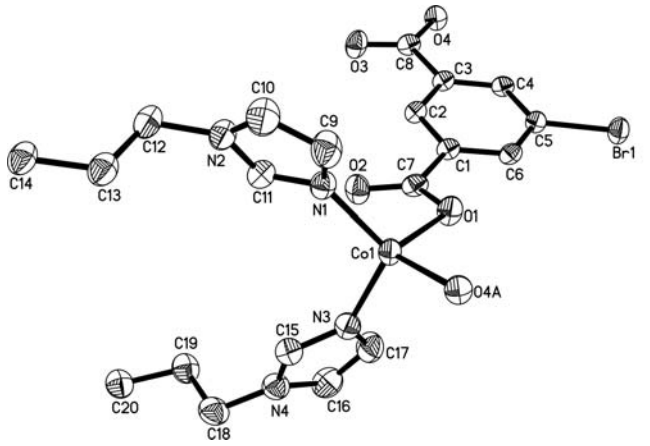

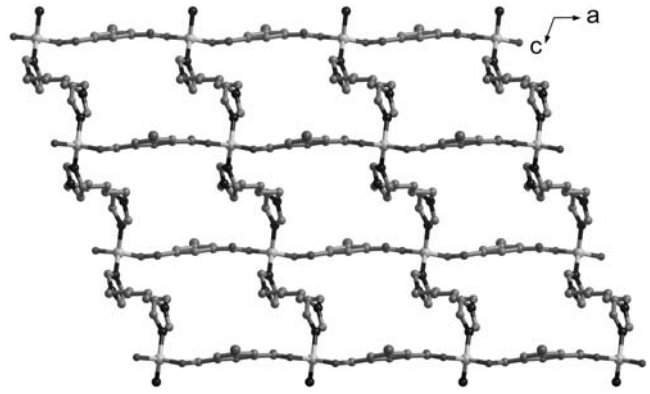

Abstract

$C_{20}H_{21}BrCoN_4O_4$, monoclinic, $P2_1/n$ (no. 14), a = 10.095(1) Å, b = 15.355(2) Å, c = 14.853(2) Å, β = 108.439(1)°, V = 2184.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.071, T = 296 K.

Source of material

1,6-bis(imidazol-1-yl)hexane (bih, 22 mg, 0.1 mmol), 5-bromoisophthalic acid (H_2BIPA, 25 mg, 0.05 mmol), $Co(OAc)_2 \cdot 4H_2O$ (24 mg, 0.1 mmol) and KOH (5.6 mg, 0.1 mmol) were added to water (12 ml) in a Teflon-lined stainless steel vessel. The mixture was heated at 433 K for 3 d, and then slowly cooled down to room temperature. Red block-shaped crystals of the title complex were obtained.

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Discussion

The design and construction of metal-organic coordination polymers are of current interest in the fields of supramolecular chemistry and crystal engineering. Major reasons for this interest stem from their intriguing variety of topologies and structural diversity, such as helixes and diamondoid nets, and because of their potential applications as functional materials, in catalysis, luminescenece, magnetism, sorption, ion exchange and nonlinear optics [1-5]. It is well known that rigid benzene polycarboxylate acids and their derivatives, such as 1,4-benzenedicarboxylic acid [6], 1,2,3-benzenetricarboxylic acid [7,8], 5-nitro-1,2,3-benzenetricarboxylic acid [7], and 5-bromoisophthalic acid [8], are widely used as building blocks to link metal ions to produce metal-organic frameworks (MOFs).

The crystal structure of the title complex comprises a 5-bromoisophthalate anion (BIPA), a Co(II) ion and a 1,6-bis(imidazol-1-yl)hexane molecule (bih) per asymmetric unit (figure, top). Both of the carboxylate groups of the acid are deprotonated. The Co(II) ion has the distorted tetrahedral coordination which is composed of two nitrogen atoms from the bih ligands and two oxygen atoms from the carboxyl groups of BIPA. The bond angles around central Co(II) ion range from 99.50(7)° to 113.9(1)°. Four Co(II) ions are interlinked by the two bih and the two BIPA ligands, thus affording a square grid with a large window of 10.09 × 15.42 Å^2, forming a puckered 4^4 net (figure, bottom). The identical 2D single nets are interlocked with each other and form an interesting 3-fold parallel interpenetrated 2D / 3D architecture. This structure of the title complex is similar to Zn(BIPA)(bix) [8].

Table 1. Data collection and handling.

Crystal:	red block, size 0.14 × 0.21 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	27.97 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51.0°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16458, 4062
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3058
$N(param)_{refined}$:	271
Programs:	SHELXS-97, SHELXL-97 [9]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.2737	0.6719	0.5334	0.044
H(4)	4*e*	0.0668	0.8966	0.5214	0.045
H(6)	4*e*	0.4521	0.8970	0.4980	0.046
H(9)	4*e*	0.9496	0.6567	0.7524	0.086
H(10)	4*e*	0.9624	0.5178	0.8341	0.091

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11)	4*e*	0.6852	0.4997	0.5800	0.059
H(12A)	4*e*	0.7169	0.3611	0.6775	0.078
H(12B)	4*e*	0.7931	0.3713	0.7870	0.078
H(13A)	4*e*	0.9352	0.3487	0.6515	0.076
H(13B)	4*e*	1.0116	0.3595	0.7606	0.076
H(14A)	4*e*	0.9105	0.2270	0.7872	0.073
H(14B)	4*e*	0.8447	0.2156	0.6772	0.073
H(15)	4*e*	0.8304	0.5186	0.4238	0.060

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(16)	4*e*	0.5490	0.6091	0.1934	0.085
H(17)	4*e*	0.5945	0.7181	0.3177	0.080
H(18A)	4*e*	0.7696	0.4139	0.2871	0.082
H(18B)	4*e*	0.6930	0.4529	0.1863	0.082
H(19A)	4*e*	0.4779	0.4305	0.2043	0.076
H(19B)	4*e*	0.5453	0.4066	0.3117	0.076
H(20A)	4*e*	0.5619	0.3012	0.1598	0.074
H(20B)	4*e*	0.6357	0.2776	0.2668	0.074

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)	4*e*	0.77312(3)	0.68793(2)	0.53256(3)	0.0294(2)	0.0357(2)	0.0628(3)	0.0002(2)	0.0188(2)	0.0015(2)
N(1)	4*e*	0.8021(2)	0.6020(1)	0.6389(2)	0.040(1)	0.044(1)	0.057(2)	–0.004(1)	0.015(1)	0.002(1)
N(2)	4*e*	0.8123(3)	0.4768(2)	0.7120(2)	0.060(2)	0.048(2)	0.054(2)	–0.002(1)	0.014(1)	0.006(1)
N(3)	4*e*	0.7327(2)	0.6328(2)	0.4042(2)	0.040(1)	0.050(2)	0.057(2)	0.004(1)	0.015(1)	0.003(1)
N(4)	4*e*	0.6954(3)	0.5343(2)	0.2925(2)	0.059(2)	0.057(2)	0.053(2)	–0.004(1)	0.019(1)	–0.004(1)
O(1)	4*e*	0.6199(2)	0.7710(1)	0.5234(1)	0.029(1)	0.048(1)	0.086(2)	0.0017(8)	0.027(1)	0.005(1)
O(2)	4*e*	0.5024(2)	0.6464(1)	0.5042(1)	0.042(1)	0.047(1)	0.090(2)	0.0076(9)	0.028(1)	0.001(1)
O(3)	4*e*	0.0368(2)	0.6459(1)	0.5475(2)	0.043(1)	0.046(1)	0.094(2)	–0.0047(9)	0.031(1)	0.008(1)
O(4)	4*e*	–0.0706(2)	0.7710(1)	0.5519(1)	0.0267(9)	0.050(1)	0.076(1)	–0.0029(8)	0.0238(9)	–0.004(1)
Br(1)	4*e*	0.24751(3)	1.03320(2)	0.48932(2)	0.0525(2)	0.0391(2)	0.0746(2)	0.0018(1)	0.0251(2)	0.0048(1)
C(1)	4*e*	0.3827(2)	0.7767(2)	0.5155(2)	0.023(1)	0.045(2)	0.040(2)	0.001(1)	0.009(1)	0.003(1)
C(2)	4*e*	0.2703(2)	0.7323(2)	0.5278(2)	0.028(1)	0.041(1)	0.040(2)	0.001(1)	0.008(1)	0.006(1)
C(3)	4*e*	0.1528(2)	0.7764(2)	0.5319(2)	0.022(1)	0.044(2)	0.039(2)	–0.000(1)	0.009(1)	0.004(1)
C(4)	4*e*	0.1460(2)	0.8662(2)	0.5203(2)	0.024(1)	0.047(2)	0.044(2)	0.004(1)	0.014(1)	0.002(1)
C(5)	4*e*	0.2577(2)	0.9097(2)	0.5073(2)	0.033(1)	0.038(1)	0.045(2)	0.001(1)	0.012(1)	0.002(1)
C(6)	4*e*	0.3768(2)	0.8664(2)	0.5057(2)	0.027(1)	0.045(2)	0.046(2)	–0.004(1)	0.015(1)	0.004(1)
C(7)	4*e*	0.5093(3)	0.7263(2)	0.5132(2)	0.029(1)	0.055(2)	0.050(2)	0.005(1)	0.017(1)	0.008(1)
C(8)	4*e*	0.0329(2)	0.7265(2)	0.5455(2)	0.025(1)	0.053(2)	0.047(2)	–0.002(1)	0.011(1)	0.002(1)
C(9)	4*e*	0.8974(4)	0.6074(2)	0.7273(2)	0.079(2)	0.057(2)	0.067(2)	–0.017(2)	0.006(2)	–0.010(2)
C(10)	4*e*	0.9052(4)	0.5310(2)	0.7730(2)	0.091(3)	0.068(2)	0.054(2)	–0.007(2)	0.000(2)	–0.002(2)
C(11)	4*e*	0.7530(3)	0.5220(2)	0.6330(2)	0.043(2)	0.053(2)	0.049(2)	–0.003(1)	0.010(1)	0.005(1)
C(12)	4*e*	0.8006(3)	0.3829(2)	0.7246(2)	0.075(2)	0.053(2)	0.067(2)	–0.004(2)	0.021(2)	0.013(2)
C(13)	4*e*	0.9285(3)	0.3365(2)	0.7140(2)	0.075(2)	0.056(2)	0.053(2)	–0.006(2)	0.013(2)	0.005(2)
C(14)	4*e*	0.9241(3)	0.2391(2)	0.7267(2)	0.071(2)	0.054(2)	0.049(2)	–0.002(2)	0.008(2)	0.009(2)
C(15)	4*e*	0.7658(3)	0.5550(2)	0.3821(2)	0.042(2)	0.055(2)	0.050(2)	0.001(1)	0.011(1)	0.001(1)
C(16)	4*e*	0.6111(4)	0.6042(2)	0.2546(2)	0.074(2)	0.078(2)	0.053(2)	0.005(2)	0.009(2)	0.015(2)
C(17)	4*e*	0.6364(3)	0.6636(2)	0.3237(2)	0.075(2)	0.058(2)	0.064(2)	0.011(2)	0.017(2)	0.010(2)
C(18)	4*e*	0.6895(4)	0.4477(2)	0.2506(2)	0.077(2)	0.071(2)	0.059(2)	–0.008(2)	0.024(2)	–0.015(2)
C(19)	4*e*	0.5567(3)	0.4017(2)	0.2494(2)	0.065(2)	0.061(2)	0.056(2)	–0.003(2)	0.008(2)	–0.009(2)
C(20)	4*e*	0.5550(3)	0.3062(2)	0.2233(2)	0.067(2)	0.064(2)	0.046(2)	0.001(2)	0.006(2)	–0.007(2)

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