

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Poly[tri- μ_4 -formato-cobalt(II)potassium]Susanne Wöhlert,^{a*} Mario Wriedt,^b Inke Jess^a and Christian Näther^a^aInstitut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Max-Eyth-Strasse 2, 24118 Kiel, Germany, and ^bDepartment of Chemistry, Texas A&M University, College Station, Texas 77843, USA

Correspondence e-mail: swoehlert@ac.uni-kiel.de

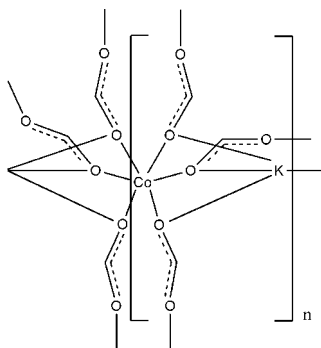
Received 18 February 2011; accepted 7 March 2011

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{O}-\text{C}) = 0.002$ Å; R factor = 0.020; wR factor = 0.046; data-to-parameter ratio = 16.5.

In the crystal structure of the title compound, $[\text{CoK}(\text{CHO}_2)_3]_n$, the Co^{2+} cations are coordinated by six O-bonded formate anions in an octahedral coordination mode and the K^+ cations are eightfold coordinated by seven O-bonded formate anions within irregular polyhedra. The Co^{2+} cations are connected by bridging formate anions into a three-dimensional coordination network in which the K^+ cations are embedded. The asymmetric unit consists of one Co^{2+} cation located on a center of inversion, one K^+ cation located on a twofold axis and two crystallographically independent formate anions, of which one is located on a twofold axis and the other occupies a general position.

Related literature

For background to this work see: Boeckmann *et al.* (2010); Wriedt & Näther (2010); Wriedt *et al.* (2009). For structures of bimetallic compounds based on potassium formate, see: Antsyshkina *et al.* (1983); Leontiev *et al.* (1988). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$[\text{CoK}(\text{CHO}_2)_3]$
 $M_r = 233.08$
 Monoclinic, $C2/c$
 $a = 10.7244$ (8) Å
 $b = 8.9653$ (6) Å
 $c = 6.8742$ (5) Å
 $\beta = 95.539$ (6)°
 $V = 657.85$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.22$ mm⁻¹
 $T = 293$ K
 $0.16 \times 0.09 \times 0.06$ mm

Data collection

Stoe IPDS-2 diffractometer
 Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
 $T_{\min} = 0.711$, $T_{\max} = 0.817$
 6120 measured reflections
 892 independent reflections
 853 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.046$
 $S = 1.15$
 892 reflections
 54 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.25$ e Å⁻³
 $\Delta\rho_{\min} = -0.57$ e Å⁻³

Table 1

Selected bond lengths (Å).

K1—O1	2.7371 (10)	Co1—O1	2.0943 (10)
K1—O2 ⁱ	2.8193 (10)	Co1—O2 ⁱⁱ	2.1015 (10)
K1—O11 ⁱ	2.8507 (11)	Co1—O11 ⁱⁱⁱ	2.1026 (9)

Symmetry codes: (i) $x - \frac{1}{2}, y - \frac{1}{2}, z$; (ii) $x, -y + 1, z - \frac{1}{2}$; (iii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$.

Data collection: *X-AREA* (Stoe & Cie, 2008); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *XCIF* in *SHELXTL*.

We gratefully acknowledge financial support by the DFG (project number NA 720/3-1) and the State of Schleswig-Holstein. We thank Professor Dr Bensch for access to his experimental facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2172).

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Antsyshkina, A. S., Porai-Koshits, M. A., Ostrikova, V. N. & Sadikov, G. G. (1983). *Koord. Khim.* **9**, 855–864.
 Boeckmann, J., Wriedt, M. & Näther, C. (2010). *Eur. J. Inorg. Chem.* pp. 1820–1828.
 Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
 Leontiev, A. Yu., Arion, M. D., Razdobreev, I. M., Kiosse, G. A., Yablokov, Yu. V., Malinovskii, T. I. & Popvich, G. A. (1988). *Dokl. Akad. Nauk SSSR*, **300**, 1129–1140.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Stoe & Cie (2008). *X-AREA*, *X-RED32* and *X-SHAPE*. Stoe & Cie, Darmstadt, Germany.
 Wriedt, M. & Näther, C. (2010). *Z. Anorg. Allg. Chem.* **636**, 569–575.
 Wriedt, M., Sellmer, S. & Näther, C. (2009). *Inorg. Chem.* **48**, 6896–6903.

supporting information

Acta Cryst. (2011). E67, m422 [doi:10.1107/S1600536811008737]

Polymeric potassium triformatocobalt(II)

Susanne Wöhlert, Mario Wriedt, Inke Jess and Christian Näther

S1. Comment

In our current investigation on the synthesis, structures and properties of new coordination polymers based on paramagnetic transition metal, small-sized anions and N-donor ligands, we have shown that thermal decomposition reactions are an elegant route for the discovery and preparation of new ligand-deficient coordination polymers (Boeckmann *et al.*, 2010; Wriedt & Näther, 2010; Wriedt *et al.*, 2009). Within this project we tried to prepare new ligand-rich precursor compounds based on cobalt formate and pyrazine as coligand. However, reaction of cobalt(II) chloride, potassium formate and pyrazine in acetonitrile unexpectedly resulted in single crystals of the title compound.

In the crystal structure of the title compound, each cobalt(II) cation is coordinated by six bridging formate anions with Co—OCHO distances between 2.0943 (10) Å and 2.1026 (9) Å. The CoO₆ octahedron is slightly distorted with angles ranging from 82.66 (4) ° to 97.34 (4) ° and 180° (Fig. 1 and Tab. 1). The K⁺ cations are coordinated by eight oxygen atoms belonging to seven formate anions within irregular polyhedra. The K—O distances range from 2.7371 (10) Å to 2.8507 (11) Å and the O—K—O angles are between 59.81 (3) ° and 147.11 (3) °. The cobalt cations are connected via μ -1,3 bridging formate anions into a three dimensional coordination network (Fig. 2). Within this network's cavities are formed in which the K⁺ cations are embedded (Fig. 3). The shortest Co...Co distances amount to 5.6487 (3) Å and the shortest K...K distances are 3.9067 (4) Å.

According to a search in the CCDC database (ConQuest Ver.1.12.2010) (Allen, 2002) mixed cobalt and potassium formates are unknown but bimetallic compounds based on potassium formate are known with different metals (Antsyshkina *et al.*, 1983 and Leontiev *et al.*, 1988).

S2. Experimental

Potassium formate (KCHOO) and pyrazine were obtained from Alfa Aesar and cobalt(II) chloride was obtained from Acros Organics. All chemicals were used without further purification. 0.25 mmol (32.5 mg) CoCl₂, 0.5 mmol (42.1 mg) KCHOO and 0.5 mmol (40 mg) pyrazine were reacted with 1 ml acetonitrile in a closed test-tube at 120°C for three days. On cooling block-shaped single crystals of the title compound were obtained in a mixture with an unknown phase. It must be noted, that the reaction under similar conditions without pyrazine does not lead to the formation of the title compound.

S3. Refinement

The H atoms were positioned with idealized geometry and were refined isotropically with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and C—H distances of 0.93 Å using a riding model.

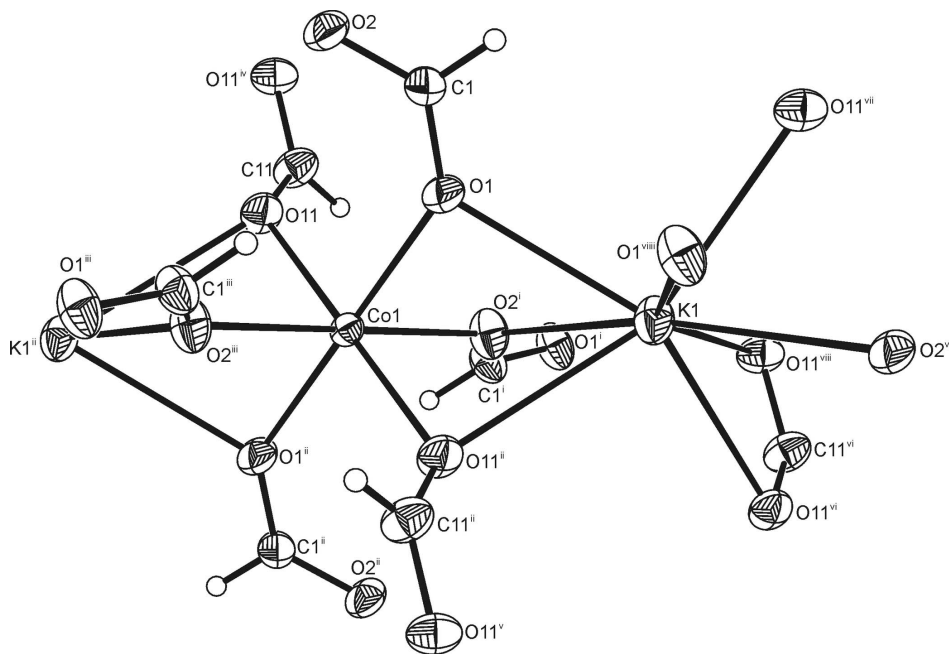


Figure 1

Crystal structure of the title compound with labelling and displacement ellipsoids drawn at the 50 % probability level. Symmetry codes: i = $-x+1/2, y-1/2, -z+1/2$; ii = $-x+1/2, -y+1/2, -z$; iii = $+x, -y+1, +z-1/2$; iv = $-x+1, +y, -z+1/2$; v = $+x-1/2, -y+1/2, +z-1/2$.

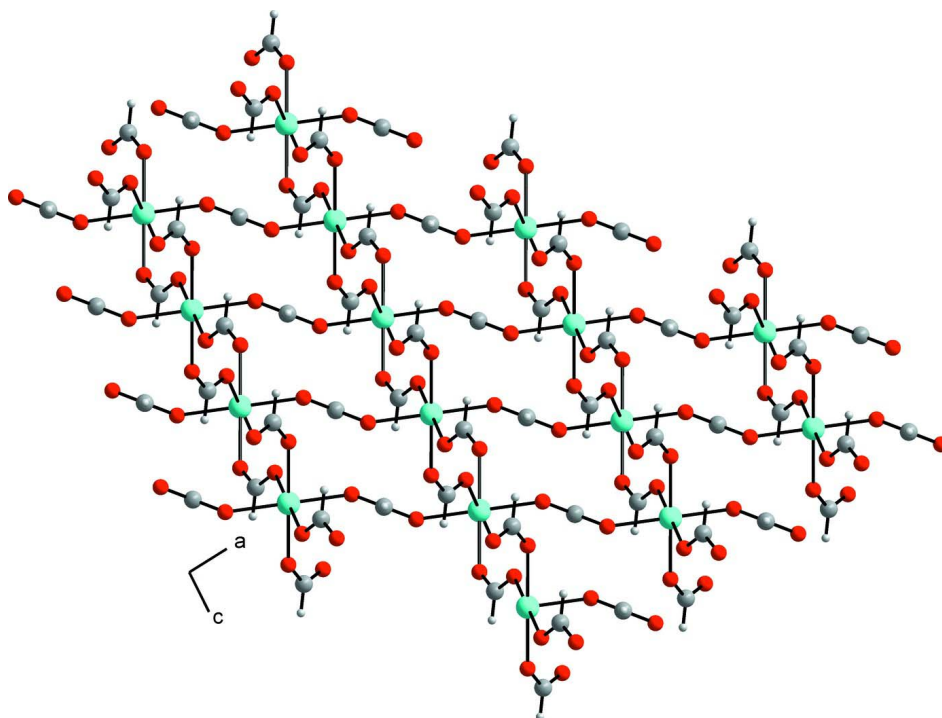
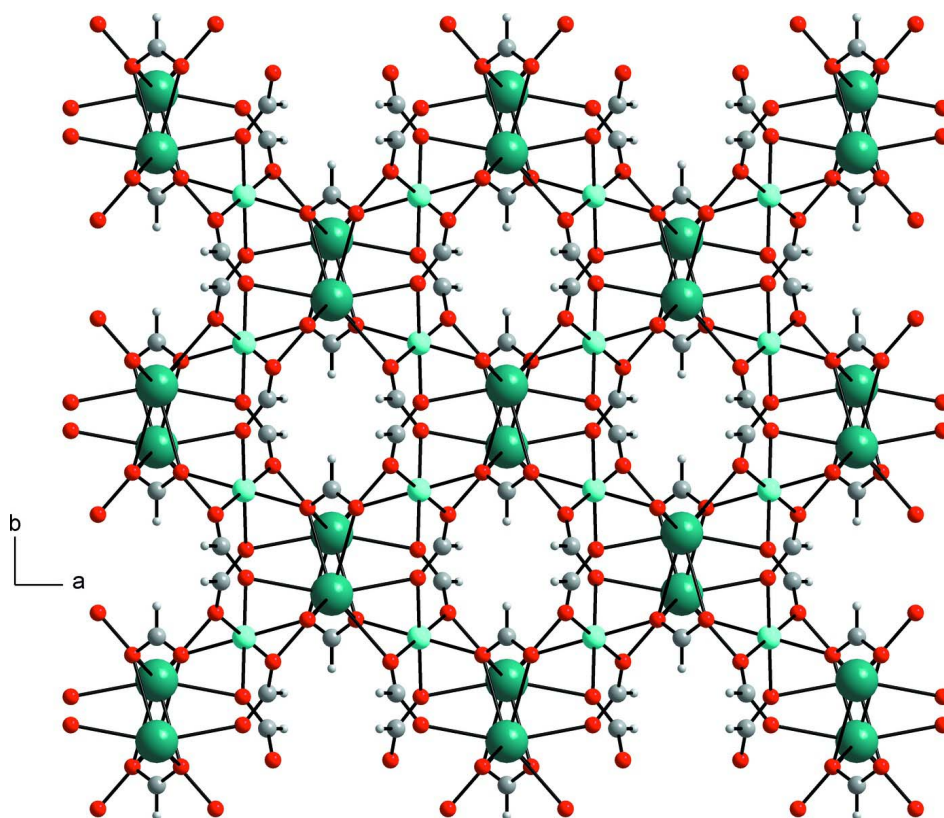


Figure 2

Crystal structure of the title compound with view along the crystallographic b axis. The K^+ cations are omitted for clarity.

**Figure 3**

Crystal structure of the title compound with view along the crystallographic *c* axis.

Poly[tri- μ -formato-cobalt(II)potassium]

Crystal data

[CoK(CHO₂)₃]
 $M_r = 233.08$
 Monoclinic, *C2/c*
 Hall symbol: -C 2yc
 $a = 10.7244$ (8) Å
 $b = 8.9653$ (6) Å
 $c = 6.8742$ (5) Å
 $\beta = 95.539$ (6)°
 $V = 657.85$ (8) Å³
 $Z = 4$

$F(000) = 460$
 $D_x = 2.353$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 6120 reflections
 $\theta = 3.0\text{--}29.2^\circ$
 $\mu = 3.22$ mm⁻¹
 $T = 293$ K
 Block, light blue
 $0.16 \times 0.09 \times 0.06$ mm

Data collection

Stoe IPDS-2
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: numerical
 (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
 $T_{\min} = 0.711$, $T_{\max} = 0.817$

6120 measured reflections
 892 independent reflections
 853 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$
 $\theta_{\text{max}} = 29.2^\circ$, $\theta_{\text{min}} = 3.0^\circ$
 $h = -14 \rightarrow 14$
 $k = -12 \rightarrow 12$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.046$
 $S = 1.15$
 892 reflections
 54 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.1263P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.57 \text{ e } \text{Å}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0126 (12)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	0.0000	0.10357 (5)	0.2500	0.02582 (12)
Co1	0.2500	0.2500	0.0000	0.01561 (10)
O1	0.16793 (10)	0.33291 (11)	0.24238 (15)	0.0274 (2)
O2	0.25445 (9)	0.54804 (11)	0.34563 (15)	0.0271 (2)
C1	0.18397 (13)	0.44031 (15)	0.3568 (2)	0.0244 (3)
H1	0.1371	0.4392	0.4638	0.029*
O11	0.43048 (9)	0.31029 (12)	0.12114 (14)	0.0260 (2)
C11	0.5000	0.2478 (2)	0.2500	0.0271 (4)
H11	0.5000	0.1441	0.2500	0.033*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0278 (2)	0.01932 (19)	0.0318 (2)	0.000	0.01004 (16)	0.000
Co1	0.01611 (14)	0.01374 (14)	0.01648 (14)	-0.00041 (8)	-0.00102 (8)	0.00122 (8)
O1	0.0332 (5)	0.0223 (5)	0.0277 (5)	-0.0087 (4)	0.0091 (4)	-0.0093 (4)
O2	0.0300 (5)	0.0204 (4)	0.0318 (5)	-0.0063 (4)	0.0076 (4)	-0.0089 (4)
C1	0.0312 (6)	0.0213 (6)	0.0215 (6)	-0.0042 (5)	0.0069 (5)	-0.0037 (5)
O11	0.0215 (4)	0.0303 (5)	0.0245 (5)	-0.0036 (4)	-0.0063 (4)	0.0033 (4)
C11	0.0270 (9)	0.0217 (9)	0.0310 (10)	0.000	-0.0057 (8)	0.000

Geometric parameters (Å, °)

K1—O1	2.7371 (10)	Co1—O11 ^{iv}	2.1026 (9)
K1—O2 ⁱ	2.8193 (10)	O1—C1	1.2448 (17)
K1—O11 ⁱⁱ	2.8335 (10)	O2—C1	1.2335 (17)
K1—O11 ⁱ	2.8507 (11)	C1—H1	0.9300
K1—C11 ⁱ	3.189 (2)	O11—C11	1.2356 (13)
Co1—O1	2.0943 (10)	C11—H11	0.9300
Co1—O2 ⁱⁱⁱ	2.1015 (10)		
O1—K1—O1 ^v	82.62 (5)	O1—Co1—O11 ^{iv}	87.99 (4)
O1—K1—O2 ⁱ	140.19 (3)	O1 ^{iv} —Co1—O11 ^{iv}	92.01 (4)
O1 ^v —K1—O2 ⁱ	59.81 (3)	O2 ⁱⁱⁱ —Co1—O11 ^{iv}	94.96 (4)
O2 ⁱ —K1—O2 ^{vi}	159.66 (4)	O2 ^{vi} —Co1—O11 ^{iv}	85.04 (4)
O1—K1—O11 ⁱⁱ	92.48 (3)	O11 ^{iv} —Co1—O11	180.00 (6)
O1 ^v —K1—O11 ⁱⁱ	63.08 (3)	C1—O1—Co1	137.25 (9)
O2 ⁱ —K1—O11 ⁱⁱ	60.35 (3)	C1—O1—K1	128.31 (9)
O2 ^{vi} —K1—O11 ⁱⁱ	126.22 (3)	Co1—O1—K1	94.38 (3)
O11 ⁱⁱ —K1—O11 ^{iv}	148.37 (5)	C1—O2—Co1 ^{vii}	126.81 (9)
O1—K1—O11 ⁱ	147.11 (3)	C1—O2—K1 ^{viii}	138.13 (9)
O1 ^v —K1—O11 ⁱ	123.09 (3)	Co1 ^{vii} —O2—K1 ^{viii}	91.88 (3)
O2 ⁱ —K1—O11 ⁱ	71.79 (3)	O2—C1—O1	127.90 (13)
O2 ^{vi} —K1—O11 ⁱ	89.25 (3)	O2—C1—H1	116.0
O11 ⁱⁱ —K1—O11 ⁱ	116.58 (3)	O1—C1—H1	116.0
O11 ^{iv} —K1—O11 ⁱ	93.17 (3)	C11—O11—Co1	129.50 (9)
O11 ⁱ —K1—O11 ^{vi}	45.45 (4)	C11—O11—K1 ^{iv}	125.17 (6)
O1—K1—C11 ⁱ	138.69 (2)	Co1—O11—K1 ^{iv}	91.47 (3)
O2 ⁱ —K1—C11 ⁱ	79.83 (2)	C11—O11—K1 ^{viii}	94.23 (9)
O11 ⁱⁱ —K1—C11 ⁱ	105.81 (2)	Co1—O11—K1 ^{viii}	124.29 (4)
O11 ⁱ —K1—C11 ⁱ	22.727 (19)	K1 ^{iv} —O11—K1 ^{viii}	86.83 (3)
O1—Co1—O1 ^{iv}	180.0	O11 ^{ix} —C11—O11	126.09 (18)
O1—Co1—O2 ⁱⁱⁱ	97.34 (4)	O11 ^{ix} —C11—K1 ^{viii}	63.04 (9)
O1 ^{iv} —Co1—O2 ⁱⁱⁱ	82.66 (4)	O11—C11—H11	117.0
O2 ⁱⁱⁱ —Co1—O2 ^{vi}	180.00 (3)	K1 ^{viii} —C11—H11	180.0

Symmetry codes: (i) $x-1/2, y-1/2, z$; (ii) $x-1/2, -y+1/2, z+1/2$; (iii) $x, -y+1, z-1/2$; (iv) $-x+1/2, -y+1/2, -z$; (v) $-x, y, -z+1/2$; (vi) $-x+1/2, y-1/2, -z+1/2$; (vii) $-x+1/2, y+1/2, -z+1/2$; (viii) $x+1/2, y+1/2, z$; (ix) $-x+1, y, -z+1/2$.