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Bis(pentane-2,4-dionato- κ^2O,O')(1,10-phenanthroline- κ^2N,N')cobalt(II)

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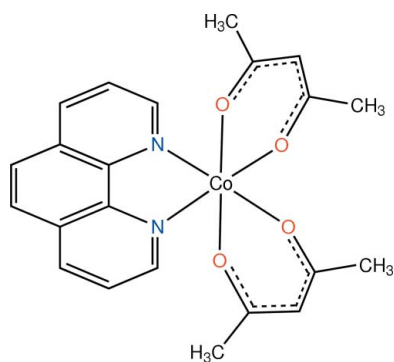
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.002$ Å; R factor = 0.032; wR factor = 0.089; data-to-parameter ratio = 17.1.

In the title compound, $[Co(C_5H_7O_2)_2(C_{12}H_8N_2)]$, the Co^{II} cation lies on a twofold rotation axis and is coordinated by four O atoms from two acetylacetonate (acac) ligands and two N atoms from a 1,10-phenanthroline (phen) ligand in a slightly distorted octahedral environment, with Co–O bond lengths of 2.0565 (11) and 2.0641 (11) Å and Co–N bond lengths of 2.1630 (12) Å. In the crystal, there are no significant hydrogen-bonding or π – π interactions.

Related literature

For applications of metal complexes containing β -diketones, see: Garibay *et al.* (2009); Kaitner *et al.* (2008). For related cobalt(II) structures, see: Meštrović & Kaitner (2006); Riblet *et al.* (2010). For the synthetic procedure, see: Ellern & Ragsdale (1968).



Experimental

Crystal data

$[Co(C_5H_7O_2)_2(C_{12}H_8N_2)]$
 $M_r = 437.35$
 Orthorhombic, $Pbna$
 $a = 10.2660$ (2) Å
 $b = 12.6981$ (3) Å
 $c = 15.5885$ (3) Å

$V = 2032.10$ (7) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.88$ mm⁻¹
 $T = 293$ K
 $0.60 \times 0.30 \times 0.13$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
 Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
 $T_{min} = 0.622$, $T_{max} = 0.895$
 4293 measured reflections
 2294 independent reflections
 2022 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.089$
 $S = 1.08$
 2294 reflections
 134 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 0.28$ e Å⁻³
 $\Delta\rho_{min} = -0.36$ e Å⁻³

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2467).

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Bis(pentane-2,4-dionato- $\kappa^2 O, O'$)(1,10-phenanthroline- $\kappa^2 N, N'$)cobalt(II)**Franc Perdih****S1. Comment**

Metal β -diketonate compounds attracted great interest, because metal complexes of β -diketonate derivatives are useful building blocks for design of porous and supramolecular materials and can be good precursors in metal-organic chemical vapour deposition (MOCVD) (Garibay *et al.*, 2009; Kaitner *et al.* 2008).

In the title molecule (Fig. 1), the cobalt(II) cation lies on a twofold axis, and is surrounded by six donor atoms arranged at the vertices of a distorted octahedron. The coordination polyhedron of Co^{II} is formed by four oxygen atoms of two symmetry related 2,4-pentanedionato ligands and two nitrogen atoms of 1,10-phenanthroline. Consequently, the octahedron is defined by two symmetry-independent cobalt-oxygen bonds Co–O1 2.0565 (11) Å (in *trans* position to N1 of the phen ligand) and Co–O2 2.0641 (11) Å (in *cis* position to N1 of the phen ligand) and a cobalt-nitrogen bond Co–N1 2.1630 (12) Å. These bond lengths are similar with the ones observed in adducts (1,10-phenanthroline)bis(1,3-diphenyl-1,3-propanedionato)cobalt(II) (Meštrović & Kaitner 2006) and (2,2'-bipyridine)bis(4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato)cobalt(II) (Riblet *et al.*, 2010). The discrepancy of the coordination polyhedron of Co^{II} from the ideal octahedral arrangement is well illustrated by the angles O1–Co–O2 and N1–Co–N1 of 87.62 (4)° and 77.20 (6)°, respectively. In the crystal structure, there are no hydrogen-bonding or π - π interactions.

S2. Experimental

(1,10-Phenanthroline- $\kappa^2 N, N'$)bis(2,4-pentanedionato- $\kappa^2 O, O'$)cobalt(II) was prepared according to the published procedure (Ellern & Ragsdale, 1968). 0.25 mmol (0.064 mg) of bis(2,4-pentanedionato- $\kappa^2 O, O'$)cobalt(II) was dissolved in 5 ml of warm chloroform and than 0.25 mmol (0.045 mg) of 1,10-phenanthroline was added. Orange crystals suitable for single-crystal X-ray diffraction were obtained after slow evaporation.

S3. Refinement

Although H atoms were visible in a difference Fourier map they were treated in riding mode in geometrically idealized positions, with C–H = 0.93 (aromatic and alkenyl) or 0.96 Å (CH₃), and with $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$, where $k = 1.5$ for methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. To improve the refinement results, one reflection with too high value of $\delta(F^2)/\text{e.s.d.}$ and with $F_o^2 < F_c^2$ was excluded from the refinement.

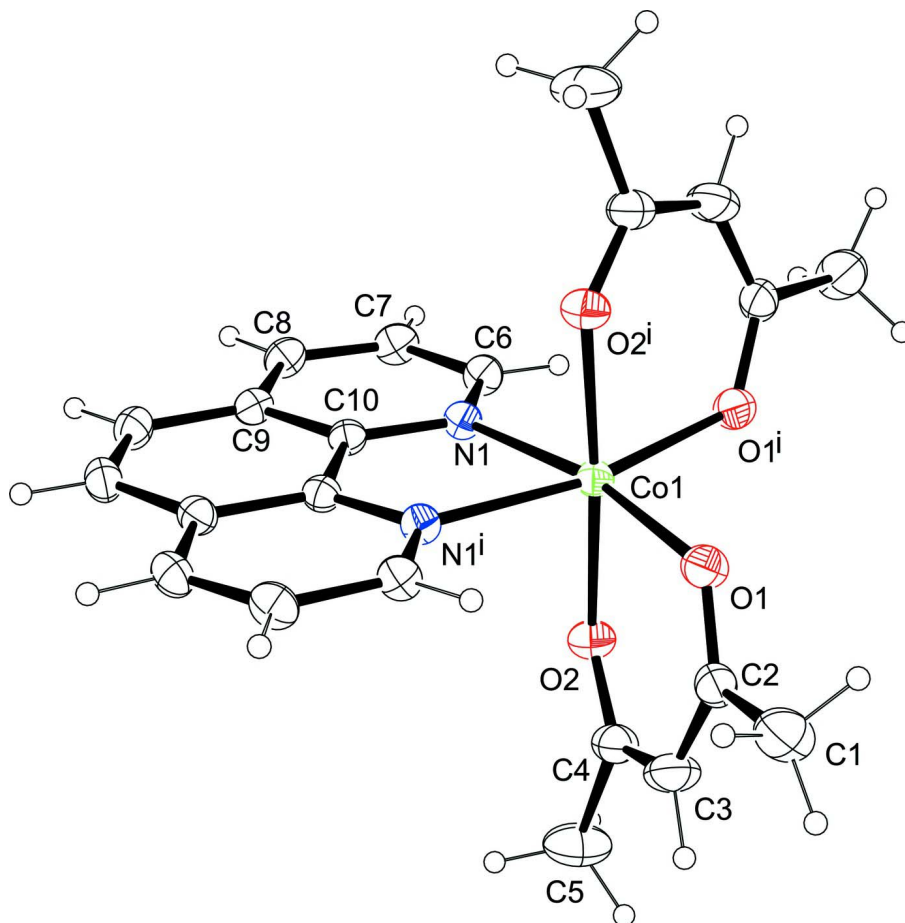


Figure 1

The molecular structure of the title complex showing the numbering scheme and displacement ellipsoids drawn at the 30% probability level. Symmetry code: $i = x, -y+1/2, -z+1$.

Bis(pentane-2,4-dionato- κ^2O,O')(1,10-phenanthroline- κ^2N,N')cobalt(II)

Crystal data

$[\text{Co}(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_{12}\text{H}_8\text{N}_2)]$

$M_r = 437.35$

Orthorhombic, $Pbna$

Hall symbol: $-P\ 2ac\ 2b$

$a = 10.2660\ (2)\ \text{\AA}$

$b = 12.6981\ (3)\ \text{\AA}$

$c = 15.5885\ (3)\ \text{\AA}$

$V = 2032.10\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 908$

$D_x = 1.43\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2617 reflections

$\theta = 0.4\text{--}27.5^\circ$

$\mu = 0.88\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, orange

$0.6 \times 0.3 \times 0.13\ \text{mm}$

Data collection

Nonius KappaCCD area-detector
diffractometer

Graphite monochromator

Detector resolution: $0.055\ \text{pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SCALEPACK*; Otwinowski & Minor, 1997)

$T_{\min} = 0.622$, $T_{\max} = 0.895$

4293 measured reflections

2294 independent reflections

2022 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.5^\circ$
 $h = -13 \rightarrow 13$

$k = -16 \rightarrow 16$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.089$
 $S = 1.08$
 2294 reflections
 134 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
 $w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.6276P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.36 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. 346 frames in 4 sets of φ scans + ω scans. Rotation/frame = 2.0° . Crystal-detector distance = 34.7 mm. Measuring time = 20 s/ $^\circ$.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	-0.01460 (3)	0.25	0.5	0.03014 (12)
N1	0.15007 (12)	0.34681 (9)	0.46431 (8)	0.0314 (3)
O1	-0.14810 (11)	0.14898 (9)	0.55387 (7)	0.0427 (3)
O2	-0.00799 (11)	0.32708 (9)	0.61650 (7)	0.0401 (3)
C1	-0.2481 (2)	0.03967 (18)	0.65656 (14)	0.0662 (6)
H1A	-0.3184	0.0319	0.6165	0.099*
H1B	-0.2827	0.0488	0.7133	0.099*
H1C	-0.1943	-0.0222	0.6551	0.099*
C2	-0.16730 (15)	0.13500 (14)	0.63257 (10)	0.0417 (4)
C3	-0.1192 (2)	0.19883 (16)	0.69833 (11)	0.0536 (5)
H3	-0.1388	0.1791	0.7543	0.064*
C4	-0.04462 (18)	0.28934 (14)	0.68732 (10)	0.0423 (4)
C5	-0.0002 (2)	0.34879 (19)	0.76625 (12)	0.0711 (7)
H5A	0.0868	0.3276	0.7808	0.107*
H5B	-0.0576	0.3334	0.8132	0.107*
H5C	-0.0016	0.4231	0.7547	0.107*
C6	0.14845 (15)	0.44271 (11)	0.43038 (10)	0.0360 (3)
H6	0.0684	0.4745	0.4198	0.043*
C7	0.26196 (16)	0.49794 (11)	0.40985 (11)	0.0402 (4)
H7	0.257	0.5652	0.3864	0.048*

C8	0.38011 (15)	0.45233 (12)	0.42452 (10)	0.0383 (3)
H8	0.4563	0.4877	0.4099	0.046*
C9	0.38654 (14)	0.35112 (11)	0.46198 (9)	0.0329 (3)
C10	0.26736 (14)	0.30172 (10)	0.48049 (9)	0.0295 (3)
C11	0.50593 (15)	0.29880 (15)	0.48177 (11)	0.0384 (4)
H11	0.5847	0.3317	0.4696	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.03095 (18)	0.03099 (18)	0.02847 (17)	0	0	0.00224 (10)
N1	0.0328 (6)	0.0283 (6)	0.0331 (6)	0.0006 (5)	0.0004 (5)	0.0019 (5)
O1	0.0419 (6)	0.0490 (7)	0.0372 (6)	-0.0132 (5)	-0.0024 (5)	0.0051 (5)
O2	0.0501 (6)	0.0370 (6)	0.0331 (5)	-0.0006 (4)	0.0016 (5)	-0.0016 (4)
C1	0.0646 (12)	0.0776 (14)	0.0563 (12)	-0.0301 (11)	-0.0085 (9)	0.0266 (11)
C2	0.0338 (7)	0.0511 (9)	0.0401 (8)	-0.0038 (7)	-0.0014 (6)	0.0124 (7)
C3	0.0636 (12)	0.0658 (12)	0.0315 (8)	-0.0112 (9)	0.0035 (8)	0.0083 (8)
C4	0.0484 (9)	0.0463 (9)	0.0320 (8)	0.0059 (8)	-0.0009 (7)	-0.0019 (7)
C5	0.1033 (19)	0.0719 (15)	0.0381 (10)	-0.0125 (12)	-0.0019 (10)	-0.0115 (10)
C6	0.0378 (7)	0.0312 (7)	0.0389 (8)	0.0028 (6)	0.0019 (6)	0.0039 (6)
C7	0.0483 (9)	0.0304 (7)	0.0420 (9)	-0.0016 (6)	0.0046 (7)	0.0064 (6)
C8	0.0391 (8)	0.0354 (8)	0.0404 (8)	-0.0062 (6)	0.0074 (6)	0.0032 (6)
C9	0.0344 (7)	0.0332 (7)	0.0311 (7)	-0.0022 (6)	0.0030 (6)	-0.0014 (6)
C10	0.0318 (7)	0.0283 (7)	0.0285 (6)	-0.0001 (5)	0.0009 (5)	-0.0011 (5)
C11	0.0315 (7)	0.0413 (10)	0.0424 (8)	-0.0040 (6)	0.0037 (6)	-0.0001 (7)

Geometric parameters (Å, °)

Co1—O1	2.0565 (11)	C3—H3	0.93
Co1—O1 ⁱ	2.0565 (11)	C4—C5	1.514 (2)
Co1—O2	2.0641 (11)	C5—H5A	0.96
Co1—O2 ⁱ	2.0641 (11)	C5—H5B	0.96
Co1—N1 ⁱ	2.1630 (12)	C5—H5C	0.96
Co1—N1	2.1630 (12)	C6—C7	1.397 (2)
N1—C6	1.3277 (18)	C6—H6	0.93
N1—C10	1.3570 (18)	C7—C8	1.363 (2)
O1—C2	1.2551 (19)	C7—H7	0.93
O2—C4	1.261 (2)	C8—C9	1.413 (2)
C1—C2	1.514 (2)	C8—H8	0.93
C1—H1A	0.96	C9—C10	1.4049 (19)
C1—H1B	0.96	C9—C11	1.428 (2)
C1—H1C	0.96	C10—C10 ⁱ	1.448 (3)
C2—C3	1.397 (3)	C11—C11 ⁱ	1.363 (4)
C3—C4	1.392 (3)	C11—H11	0.93
O1—Co1—O1 ⁱ	96.41 (7)	C4—C3—H3	117.2
O1—Co1—O2	87.62 (4)	C2—C3—H3	117.2
O1 ⁱ —Co1—O2	94.90 (4)	O2—C4—C3	125.87 (15)

O1—Co1—O2 ⁱ	94.90 (4)	O2—C4—C5	115.61 (17)
O1 ⁱ —Co1—O2 ⁱ	87.62 (4)	C3—C4—C5	118.51 (16)
O2—Co1—O2 ⁱ	176.23 (6)	C4—C5—H5A	109.5
O1—Co1—N1 ⁱ	93.51 (5)	C4—C5—H5B	109.5
O1 ⁱ —Co1—N1 ⁱ	168.64 (4)	H5A—C5—H5B	109.5
O2—Co1—N1 ⁱ	91.00 (4)	C4—C5—H5C	109.5
O2 ⁱ —Co1—N1 ⁱ	86.05 (4)	H5A—C5—H5C	109.5
O1—Co1—N1	168.64 (4)	H5B—C5—H5C	109.5
O1 ⁱ —Co1—N1	93.51 (5)	N1—C6—C7	122.76 (14)
O2—Co1—N1	86.05 (4)	N1—C6—H6	118.6
O2 ⁱ —Co1—N1	91.00 (4)	C7—C6—H6	118.6
N1 ⁱ —Co1—N1	77.20 (6)	C8—C7—C6	119.36 (13)
C6—N1—C10	118.17 (12)	C8—C7—H7	120.3
C6—N1—Co1	127.88 (10)	C6—C7—H7	120.3
C10—N1—Co1	113.95 (9)	C7—C8—C9	119.81 (14)
C2—O1—Co1	126.30 (10)	C7—C8—H8	120.1
C4—O2—Co1	125.47 (11)	C9—C8—H8	120.1
C2—C1—H1A	109.5	C10—C9—C8	116.76 (13)
C2—C1—H1B	109.5	C10—C9—C11	119.70 (13)
H1A—C1—H1B	109.5	C8—C9—C11	123.54 (14)
C2—C1—H1C	109.5	N1—C10—C9	123.11 (12)
H1A—C1—H1C	109.5	N1—C10—C10 ⁱ	117.45 (7)
H1B—C1—H1C	109.5	C9—C10—C10 ⁱ	119.44 (8)
O1—C2—C3	125.43 (15)	C11 ⁱ —C11—C9	120.87 (9)
O1—C2—C1	116.13 (16)	C11 ⁱ —C11—H11	119.6
C3—C2—C1	118.43 (16)	C9—C11—H11	119.6
C4—C3—C2	125.66 (15)		
O1—Co1—N1—C6	-143.2 (2)	C1—C2—C3—C4	-179.5 (2)
O1 ⁱ —Co1—N1—C6	7.72 (13)	Co1—O2—C4—C3	-13.7 (3)
O2—Co1—N1—C6	-86.96 (13)	Co1—O2—C4—C5	165.38 (13)
O2 ⁱ —Co1—N1—C6	95.39 (13)	C2—C3—C4—O2	-0.7 (3)
N1 ⁱ —Co1—N1—C6	-178.89 (15)	C2—C3—C4—C5	-179.73 (19)
O1—Co1—N1—C10	36.0 (3)	C10—N1—C6—C7	0.8 (2)
O1 ⁱ —Co1—N1—C10	-173.10 (10)	Co1—N1—C6—C7	180.00 (11)
O2—Co1—N1—C10	92.22 (10)	N1—C6—C7—C8	0.3 (2)
O2 ⁱ —Co1—N1—C10	-85.43 (10)	C6—C7—C8—C9	-1.4 (2)
N1 ⁱ —Co1—N1—C10	0.29 (7)	C7—C8—C9—C10	1.4 (2)
O1 ⁱ —Co1—O1—C2	-113.29 (15)	C7—C8—C9—C11	-178.19 (15)
O2—Co1—O1—C2	-18.62 (14)	C6—N1—C10—C9	-0.9 (2)
O2 ⁱ —Co1—O1—C2	158.57 (14)	Co1—N1—C10—C9	179.85 (11)
N1 ⁱ —Co1—O1—C2	72.24 (14)	C6—N1—C10—C10 ⁱ	178.45 (15)
N1—Co1—O1—C2	37.5 (3)	Co1—N1—C10—C10 ⁱ	-0.82 (19)
O1—Co1—O2—C4	19.03 (14)	C8—C9—C10—N1	-0.2 (2)
O1 ⁱ —Co1—O2—C4	115.26 (13)	C11—C9—C10—N1	179.37 (14)
N1 ⁱ —Co1—O2—C4	-74.45 (13)	C8—C9—C10—C10 ⁱ	-179.54 (15)
N1—Co1—O2—C4	-151.54 (14)	C11—C9—C10—C10 ⁱ	0.1 (2)
Co1—O1—C2—C3	12.6 (3)	C10—C9—C11—C11 ⁱ	0.2 (3)

Co1—O1—C2—C1	-166.56 (13)	C8—C9—C11—C11 ⁱ	179.75 (19)
O1—C2—C3—C4	1.4 (3)		

Symmetry code: (i) $x, -y+1/2, -z+1$.