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A redetermination of bis(5,5′-diethylbarbiturato)bis(imidazole)cobalt(II)

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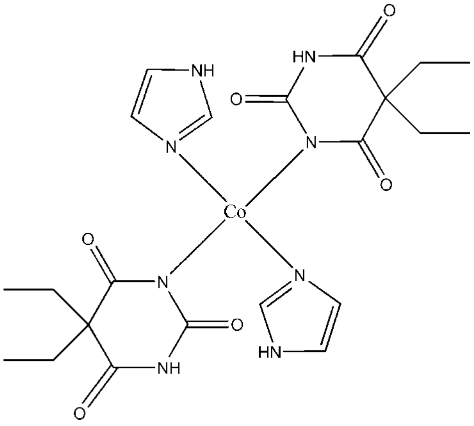
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.033; wR factor = 0.079; data-to-parameter ratio = 18.5.

The title complex, $[Co(C_8H_{12}N_2O_3)_2(C_3H_4N_2)_2]$, whose structure was first determined by Wang & Craven [(1971). *J. Chem. Soc. D*, pp. 290–291], has been redetermined with improved precision. A crystallographic twofold rotation axis passes through the Co atom, which is tetrahedrally coordinated by two N atoms from two barbital ligands and two N atoms from two imidazole ligands. The molecules are self-assembled *via* intermolecular N—H···O hydrogen-bonding interactions into a supramolecular network.

Related literature

For related literature, see: Wang & Craven (1971).



Experimental

Crystal data

$[Co(C_8H_{12}N_2O_3)_2(C_3H_4N_2)_2]$	V = 2744.27 (10) Å^3
M_r = 561.47	Z = 4
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 13.3750 (3) Å	μ = 0.68 mm^{-1}
b = 10.1371 (2) Å	T = 296 (2) K
c = 20.5791 (4) Å	0.32 × 0.26 × 0.25 mm
β = 100.409 (1)°	

Data collection

Bruker APEXII area-detector diffractometer	18228 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3141 independent reflections
T_{min} = 0.813, T_{max} = 0.849	2596 reflections with $I > 2\sigma(I)$
	R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032	170 parameters
$wR(F^2)$ = 0.079	H-atom parameters constrained
S = 0.98	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
3141 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.15	2.7932 (19)	131
N4—H4···O3^{ii}	0.86	2.12	2.9312 (18)	156

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 2004); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2187).

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S1. Comment

In the present paper we report the redetermination of the crystal structure of the title complex using CCD data at room temperature. The structure agrees with the results reported previously by Wang & Craven (1971; CCDC refcode BARICO) with lattice parameters a = 13.362 (2) Å, b = 10.133 (2) Å, c = 20.544 (4) Å β = 100.33 (3) °), but with improved precision.

As illustrated in Figure 1, the cobalt(II) atom, possesses a crystallogarphically imposed C_2 symmetry, and displays a tetrahedral coordination geometry provided by two N atoms from two barbital ligands and two N atoms from two imidazole ligands. Intermolecular N—H···O hydrogen bonding interactions (Table 1) govern the crystal packing (Fig. 2).

S2. Experimental

A mixture of cobalt nitrate (1 mmol), imidazole (1 mmol), barbital (1 mmol), NaOH (1.5 mmol) and H_2O (12 ml) was placed in a 23 ml Teflon reactor, which was heated to 433 K for three days and then cooled to room temperature at a rate of 10 K h^{-1}. The crystals obtained were washed with water and dryed in air.

S3. Refinement

All H atoms were placed at calculated positions and treated as riding on the parent atoms with C—H = 0.93–0.97 Å; N—H = 0.86 Å, and with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5 U_{eq}(C) for methyl H atoms.

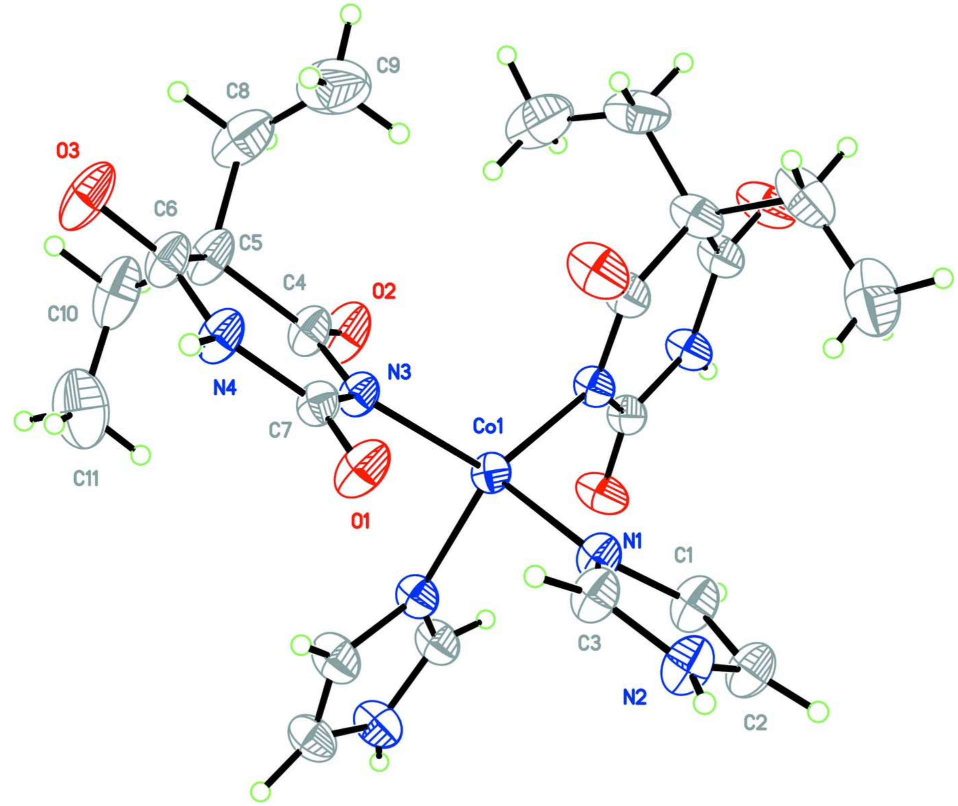

Figure 1

The molecular structure of the title compound, showing the atom numbering scheme and 30% probability displacement ellipsoids. Unlabelled atoms are related to the labelled atoms by the symmetry operator (-*x*, *y*, 0.5 - *z*).

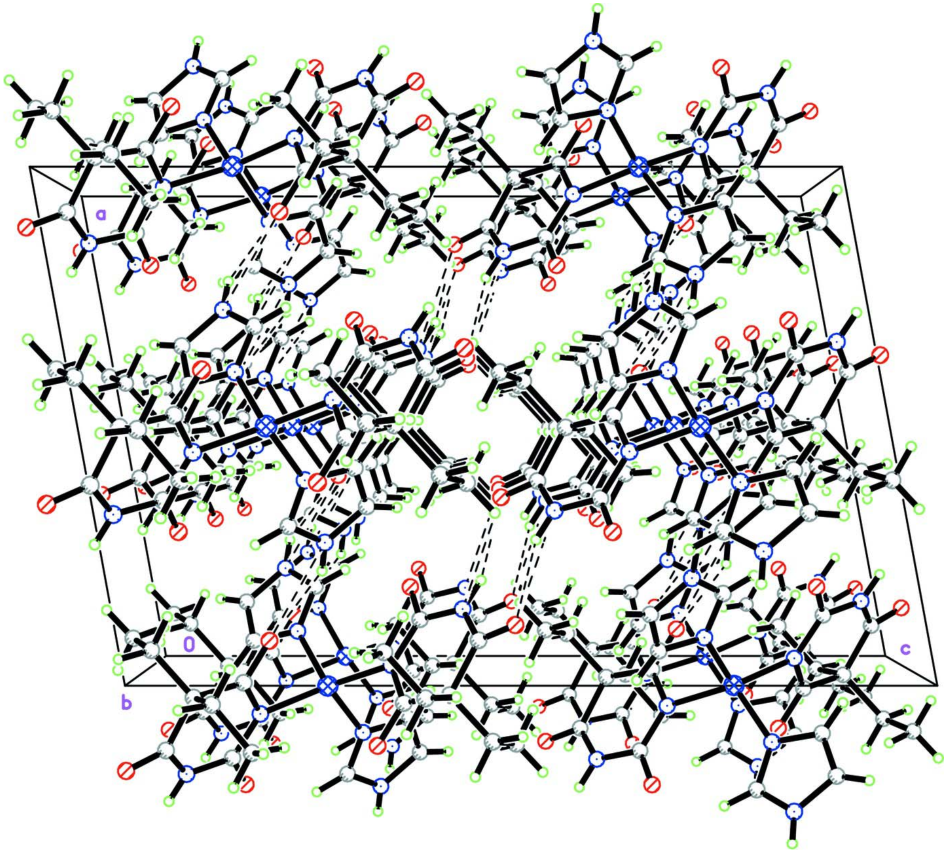

Figure 2

Packing diagram of the title compound viewed along the *b* axis. Intermolecluar hydrogen bonds are shown as dashed lines.

bis(5,5′-diethylbarbiturato)bis(imidazole)cobalt(II)

Crystal data

$[Co(C_8H_{12}N_2O_3)_2(C_3H_4N_2)_2]$
$M_r = 561.47$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 13.3750$ (3) Å
$b = 10.1371$ (2) Å
$c = 20.5791$ (4) Å
$\beta = 100.409$ (1)°
$V = 2744.27$ (10) Å^3
$Z = 4$

$F(000) = 1172$
$D_x = 1.359$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 3600 reflections
$\theta = 1.4$–28°
$\mu = 0.68$ mm^{-1}
$T = 296$ K
Block, pink
0.32 × 0.26 × 0.25 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.813$, $T_{max} = 0.849$

18228 measured reflections
3141 independent reflections
2596 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
$h = -17 \rightarrow 16$
$k = -12 \rightarrow 13$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.079$	$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 2.0613P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{max} < 0.001$
3141 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
170 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.07766 (14)	−0.1841 (2)	0.16104 (9)	0.0574 (5)
H1	0.0212	−0.1684	0.1284	0.069*
C2	0.14986 (15)	−0.2732 (2)	0.15704 (9)	0.0579 (5)
H2	0.1533	−0.3303	0.1221	0.069*
C3	0.18454 (13)	−0.17053 (19)	0.25137 (8)	0.0524 (4)
H3	0.2178	−0.1457	0.2932	0.063*
C4	−0.01891 (13)	0.20721 (19)	0.34360 (8)	0.0502 (4)
C5	0.00221 (14)	0.3032 (2)	0.40182 (9)	0.0581 (5)
C6	0.10237 (14)	0.27581 (19)	0.44751 (8)	0.0535 (5)
C7	0.13212 (12)	0.08668 (17)	0.37956 (7)	0.0418 (4)
C8	0.0058 (2)	0.4435 (2)	0.37466 (12)	0.0845 (8)
H8A	−0.0583	0.4615	0.3456	0.101*
H8B	0.0121	0.5050	0.4113	0.101*
C9	0.0911 (3)	0.4694 (3)	0.33716 (17)	0.1117 (11)
H9A	0.1553	0.4584	0.3663	0.168*
H9B	0.0858	0.5580	0.3203	0.168*
H9C	0.0865	0.4084	0.3011	0.168*
C10	−0.08480 (17)	0.2920 (3)	0.44236 (11)	0.0849 (8)
H10A	−0.0711	0.3533	0.4791	0.102*
H10B	−0.1479	0.3190	0.4145	0.102*
C11	−0.0995 (2)	0.1564 (4)	0.46924 (16)	0.1166 (11)
H11A	−0.1180	0.0958	0.4333	0.175*
H11B	−0.1526	0.1594	0.4950	0.175*
H11C	−0.0374	0.1279	0.4966	0.175*
Co1	0.0000	0.00895 (3)	0.2500	0.03642 (10)

N1	0.09912 (10)	−0.11875 (14)	0.22073 (6)	0.0452 (3)
N2	0.21731 (12)	−0.26297 (17)	0.21478 (8)	0.0589 (4)
H2A	0.2719	−0.3087	0.2257	0.071*
N3	0.04631 (10)	0.11057 (14)	0.33481 (6)	0.0408 (3)
N4	0.15545 (11)	0.16886 (15)	0.43447 (6)	0.0497 (4)
H4	0.2089	0.1495	0.4628	0.060*
O1	0.19031 (11)	−0.00247 (13)	0.37358 (7)	0.0620 (4)
O2	−0.09826 (10)	0.21962 (16)	0.30314 (6)	0.0724 (4)
O3	0.13421 (11)	0.34732 (15)	0.49451 (7)	0.0751 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0473 (10)	0.0736 (13)	0.0437 (9)	0.0084 (10)	−0.0125 (8)	−0.0131 (9)
C2	0.0524 (11)	0.0659 (12)	0.0504 (10)	0.0040 (10)	−0.0038 (8)	−0.0171 (9)
C3	0.0449 (10)	0.0648 (12)	0.0412 (9)	0.0095 (9)	−0.0089 (7)	−0.0080 (8)
C4	0.0446 (10)	0.0619 (11)	0.0368 (8)	0.0068 (9)	−0.0122 (7)	−0.0087 (8)
C5	0.0504 (11)	0.0683 (12)	0.0452 (9)	0.0222 (9)	−0.0187 (8)	−0.0180 (9)
C6	0.0497 (10)	0.0634 (12)	0.0394 (8)	0.0134 (9)	−0.0132 (7)	−0.0120 (8)
C7	0.0401 (9)	0.0465 (9)	0.0343 (7)	−0.0006 (8)	−0.0052 (6)	−0.0031 (7)
C8	0.0978 (19)	0.0642 (14)	0.0731 (14)	0.0269 (14)	−0.0336 (14)	−0.0164 (12)
C9	0.141 (3)	0.0743 (18)	0.105 (2)	−0.0077 (18)	−0.018 (2)	0.0143 (16)
C10	0.0596 (14)	0.127 (2)	0.0608 (13)	0.0337 (15)	−0.0081 (11)	−0.0310 (14)
C11	0.096 (2)	0.168 (4)	0.090 (2)	0.012 (2)	0.0298 (17)	0.004 (2)
Co1	0.03201 (16)	0.04302 (19)	0.02885 (14)	0.000	−0.00886 (11)	0.000
N1	0.0395 (8)	0.0538 (9)	0.0371 (7)	0.0054 (6)	−0.0067 (6)	−0.0036 (6)
N2	0.0468 (9)	0.0704 (11)	0.0539 (9)	0.0185 (8)	−0.0056 (7)	−0.0098 (8)
N3	0.0375 (7)	0.0476 (8)	0.0319 (6)	0.0007 (6)	−0.0080 (5)	−0.0059 (5)
N4	0.0439 (8)	0.0590 (9)	0.0370 (7)	0.0130 (7)	−0.0174 (6)	−0.0115 (6)
O1	0.0582 (8)	0.0623 (9)	0.0564 (7)	0.0201 (7)	−0.0142 (6)	−0.0180 (6)
O2	0.0549 (8)	0.0944 (11)	0.0536 (8)	0.0226 (8)	−0.0281 (6)	−0.0210 (7)
O3	0.0684 (9)	0.0848 (10)	0.0570 (8)	0.0268 (8)	−0.0291 (7)	−0.0361 (7)

Geometric parameters (Å, º)

C1—C2	1.336 (3)	C8—C9	1.512 (4)
C1—N1	1.379 (2)	C8—H8A	0.9700
C1—H1	0.9300	C8—H8B	0.9700
C2—N2	1.360 (2)	C9—H9A	0.9600
C2—H2	0.9300	C9—H9B	0.9600
C3—N1	1.311 (2)	C9—H9C	0.9600
C3—N2	1.325 (2)	C10—C11	1.507 (4)
C3—H3	0.9300	C10—H10A	0.9700
C4—O2	1.231 (2)	C10—H10B	0.9700
C4—N3	1.346 (2)	C11—H11A	0.9600
C4—C5	1.529 (2)	C11—H11B	0.9600
C5—C6	1.517 (2)	C11—H11C	0.9600
C5—C8	1.532 (3)	Co1—N1$^{\rm i}$	2.0210 (14)

C5—C10	1.554 (3)	Co1—N1	2.0210 (14)
C6—O3	1.222 (2)	Co1—N3	2.0249 (12)
C6—N4	1.349 (2)	Co1—N3^{i}	2.0249 (12)
C7—O1	1.213 (2)	N2—H2A	0.8600
C7—N3	1.3571 (19)	N4—H4	0.8600
C7—N4	1.393 (2)		

C2—C1—N1	110.08 (15)	C8—C9—H9C	109.5
C2—C1—H1	125.0	H9A—C9—H9C	109.5
N1—C1—H1	125.0	H9B—C9—H9C	109.5
C1—C2—N2	105.50 (16)	C11—C10—C5	115.1 (2)
C1—C2—H2	127.3	C11—C10—H10A	108.5
N2—C2—H2	127.3	C5—C10—H10A	108.5
N1—C3—N2	111.02 (15)	C11—C10—H10B	108.5
N1—C3—H3	124.5	C5—C10—H10B	108.5
N2—C3—H3	124.5	H10A—C10—H10B	107.5
O2—C4—N3	118.95 (15)	C10—C11—H11A	109.5
O2—C4—C5	118.64 (16)	C10—C11—H11B	109.5
N3—C4—C5	122.41 (14)	H11A—C11—H11B	109.5
C6—C5—C8	108.28 (18)	C10—C11—H11C	109.5
C6—C5—C4	112.75 (15)	H11A—C11—H11C	109.5
C8—C5—C4	108.56 (16)	H11B—C11—H11C	109.5
C6—C5—C10	108.49 (16)	N1^{i}—Co1—N1	100.33 (9)
C8—C5—C10	109.85 (19)	N1^{i}—Co1—N3	100.60 (5)
C4—C5—C10	108.90 (17)	N1—Co1—N3	117.89 (5)
O3—C6—N4	120.88 (15)	N1^{i}—Co1—N3^{i}	117.89 (5)
O3—C6—C5	121.64 (16)	N1—Co1—N3^{i}	100.60 (5)
N4—C6—C5	117.47 (14)	N3—Co1—N3^{i}	118.85 (8)
O1—C7—N3	122.91 (14)	C3—N1—C1	105.02 (15)
O1—C7—N4	118.29 (14)	C3—N1—Co1	132.44 (12)
N3—C7—N4	118.80 (15)	C1—N1—Co1	122.06 (11)
C9—C8—C5	115.1 (2)	C3—N2—C2	108.38 (15)
C9—C8—H8A	108.5	C3—N2—H2A	125.8
C5—C8—H8A	108.5	C2—N2—H2A	125.8
C9—C8—H8B	108.5	C4—N3—C7	121.87 (13)
C5—C8—H8B	108.5	C4—N3—Co1	112.43 (10)
H8A—C8—H8B	107.5	C7—N3—Co1	125.70 (11)
C8—C9—H9A	109.5	C6—N4—C7	126.37 (14)
C8—C9—H9B	109.5	C6—N4—H4	116.8
H9A—C9—H9B	109.5	C7—N4—H4	116.8

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2^{ii}	0.86	2.15	2.7932 (19)	131

N4—H4···O3^{iii}	0.86	2.12	2.9312 (18)	156

Symmetry codes: (ii) $x+1/2, y-1/2, z$; (iii) $-x+1/2, -y+1/2, -z+1$.