metal-organic compounds

[Bis(3-phenylprop-2-enylidene)propane-1,3-diamine-κ²*N*,*N'*]dibromidocobalt(II)

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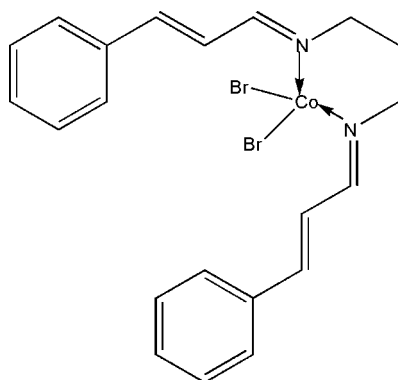Correspondence e-mail: hapipah@um.edu.my

In the crystal structure of the title compound, $[CoBr_2(C_{21}H_{22}N_2)]$, the Co^{II} atom is four-coordinated by two bromide anions and two N atoms from the bidentate Schiff base ligand in a distorted tetrahedral geometry.

Related literature

For a related compound, see: Srivastava *et al.* (1990).

Experimental

Crystal data

$[CoBr_2(C_{21}H_{22}N_2)]$	$V = 2157.5$ (5) Å³
$M_r = 521.16$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 14.0306$ (19) Å	$\mu = 4.51$ mm⁻¹
$b = 11.9962$ (16) Å	$T = 100$ (2) K
$c = 13.6738$ (19) Å	$0.32 \times 0.12 \times 0.10$ mm
$\beta = 110.375$ (2)°	

Data collection

Bruker APEXII area-detector diffractometer	9901 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3802 independent reflections
$T_{min} = 0.323$, $T_{max} = 0.659$	3275 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$	235 parameters
$wR(F^2) = 0.080$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{max} = 0.96$ e Å⁻³
3802 reflections	$\Delta\rho_{min} = -0.34$ e Å⁻³

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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supporting information

[Bis(3-phenylprop-2-enylidene)propane-1,3-diamine-κ^2N,N']dibromidocobalt(II)

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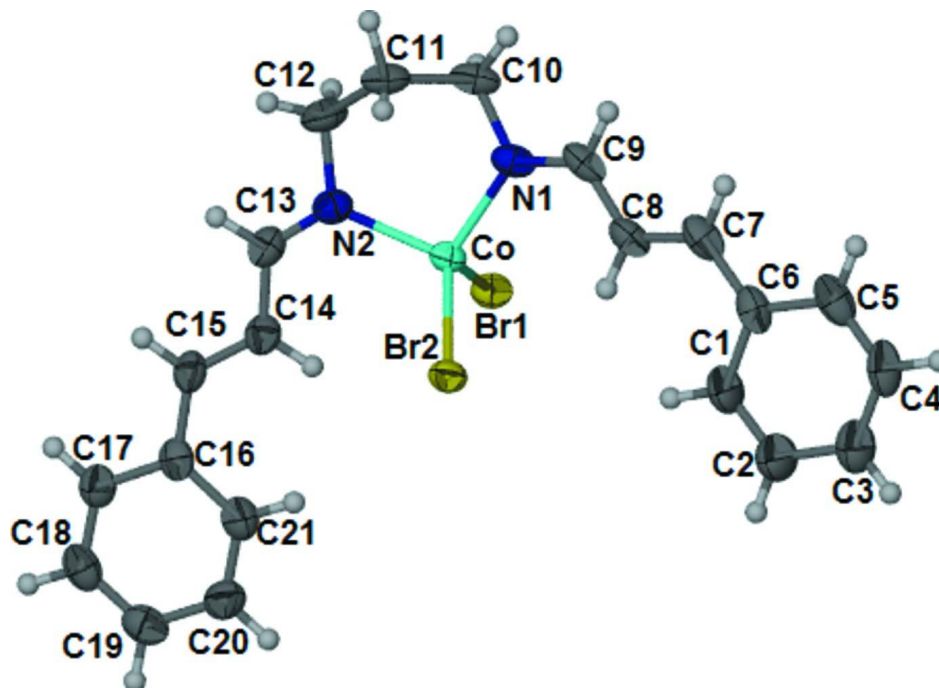
S1. Comment

Synthesis of a new four-coordinated complex of Co(II) is to identify the steric arrangement with this type of bidentate ligand and could lead to either pseudo-tetrahedral or square planar geometry. Reaction of anhydrous $CoBr_2$ with the ligand, N,*N*-bis(3-phenyl-propenylidene)-1,3-propanediamine has yielded the title compound with distorted tetrahedral geometry.

S2. Experimental

The ligand of N,*N*-bis(3-phenyl-propenylidene)-1,3-propanediamine was prepared by condensation of 2?mmol of cinnamaldehyde and 1 mmol ethylenediamine in 10 ml dichloromethane. The mixture was cooled in an ice bath and then was added dropwise to a solution of 1 mmol anhydrous $CoBr_2$ in 10 ml dichloromethane under nitrogen atmosphere. The mixture was stirred for 3 h and then filtered. To filtrate, 20 ml chloroform was added and kept overnight. The crystals suitable for X-ray were filtered off and washed with chloroform (64% yield). Elemental analysis for $C_{21}H_{22}Br_2CoN_2$ Calcd. C, 48.40; H, 4.25; N, 5.38%; Found: C, 48.32; H,4.21; N, 5.32%.

S3. Refinement

All H atoms were placed at calculated positions (C—H O.95 - 0.99Å) with $U_{iso}(H)$ set to 1.2 times $U_{eq}(C)$.

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of the title complex at 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

[Bis(3-phenylprop-2-enylidene)propane-1,3-diamine- $\kappa^2 N,N'$]dibromidocobalt(II)

Crystal data

[CoBr$_2$(C$_{21}$H$_{22}$N$_2$)]	$F(000) = 1036$
$M_r = 521.16$	$D_x = 1.604$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 4346 reflections
$a = 14.0306$ (19) Å	$\theta = 2.3–29.6°$
$b = 11.9962$ (16) Å	$\mu = 4.51$ mm^{-1}
$c = 13.6738$ (19) Å	$T = 100$ K
$\beta = 110.375$ (2)°	Block, green
$V = 2157.5$ (5) Å^3	$0.32 \times 0.12 \times 0.10$ mm
$Z = 4$	

Data collection

Bruker APEXII area-detector diffractometer	9901 measured reflections
Radiation source: fine-focus sealed tube	3802 independent reflections
Graphite monochromator	3275 reflections with $I > 2\sigma(I)$
ω scan	$R_{int} = 0.023$
Absorption correction: multi-scan ($SADABS$; Sheldrick, 1996)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$T_{min} = 0.323$, $T_{max} = 0.659$	$h = -16 \rightarrow 16$
	$k = -14 \rightarrow 14$
	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.031$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.080$	
$S = 1.01$	H-atom parameters constrained
3802 reflections	$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 2.376P]$
235 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.96$ e Å^{-3}
	$\Delta\rho_{min} = -0.34$ e Å^{-3}

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}
Br1	0.43506 (3)	0.59326 (3)	0.13742 (3)	0.02898 (11)
Br2	0.17275 (3)	0.75199 (3)	0.04140 (3)	0.02782 (11)
Co	0.28730 (4)	0.63613 (4)	−0.00789 (3)	0.02382 (13)
N1	0.3244 (2)	0.6980 (3)	−0.1274 (2)	0.0278 (7)
N2	0.2176 (2)	0.4912 (2)	−0.0717 (2)	0.0286 (7)
C1	0.3361 (4)	1.0847 (3)	0.0581 (3)	0.0456 (11)
H1A	0.3094	1.0167	0.0732	0.055*
C2	0.3418 (4)	1.1776 (4)	0.1210 (4)	0.0498 (11)
H2A	0.3188	1.1725	0.1785	0.060*
C3	0.3802 (3)	1.2761 (3)	0.1007 (4)	0.0433 (10)
H3A	0.3846	1.3389	0.1444	0.052*
C4	0.4120 (3)	1.2839 (3)	0.0176 (3)	0.0397 (10)
H4A	0.4379	1.3527	0.0031	0.048*
C5	0.4070 (3)	1.1931 (3)	−0.0459 (3)	0.0385 (10)
H5A	0.4295	1.2000	−0.1035	0.046*
C6	0.3690 (3)	1.0907 (3)	−0.0263 (3)	0.0327 (9)
C7	0.3671 (3)	0.9943 (3)	−0.0910 (3)	0.0354 (9)
H7A	0.3832	1.0071	−0.1521	0.042*
C8	0.3451 (3)	0.8893 (3)	−0.0736 (3)	0.0304 (8)
H8A	0.3283	0.8738	−0.0134	0.037*
C9	0.3458 (3)	0.7994 (3)	−0.1420 (3)	0.0311 (8)
H9A	0.3631	0.8156	−0.2017	0.037*
C10	0.3299 (3)	0.6147 (3)	−0.2044 (3)	0.0329 (9)
H10A	0.3895	0.5657	−0.1725	0.039*
H10B	0.3389	0.6531	−0.2647	0.039*

C11	0.2336 (3)	0.5444 (3)	−0.2418 (3)	0.0340 (9)
H11A	0.1745	0.5939	−0.2520	0.041*
H11B	0.2267	0.5121	−0.3106	0.041*
C12	0.2293 (3)	0.4507 (3)	−0.1697 (3)	0.0375 (9)
H12A	0.1714	0.4012	−0.2066	0.045*
H12B	0.2924	0.4061	−0.1520	0.045*
C13	0.1673 (3)	0.4295 (3)	−0.0322 (3)	0.0318 (8)
H13A	0.1370	0.3643	−0.0696	0.038*
C14	0.1526 (3)	0.4507 (3)	0.0653 (3)	0.0297 (8)
H14A	0.1831	0.5143	0.1055	0.036*
C15	0.0965 (3)	0.3817 (3)	0.0998 (3)	0.0313 (8)
H15A	0.0666	0.3201	0.0563	0.038*
C16	0.0764 (3)	0.3912 (3)	0.1974 (3)	0.0293 (8)
C17	0.0274 (3)	0.3040 (3)	0.2283 (3)	0.0322 (8)
H17A	0.0061	0.2406	0.1844	0.039*
C18	0.0091 (3)	0.3078 (3)	0.3206 (3)	0.0354 (9)
H18A	−0.0238	0.2471	0.3402	0.043*
C19	0.0385 (3)	0.3995 (3)	0.3848 (3)	0.0364 (9)
H19A	0.0262	0.4021	0.4488	0.044*
C20	0.0866 (3)	0.4888 (4)	0.3552 (3)	0.0373 (9)
H20A	0.1071	0.5521	0.3993	0.045*
C21	0.1046 (3)	0.4853 (3)	0.2625 (3)	0.0347 (9)
H21A	0.1362	0.5469	0.2424	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0299 (2)	0.0322 (2)	0.02463 (19)	0.00440 (15)	0.00925 (15)	0.00136 (15)
Br2	0.0296 (2)	0.02873 (19)	0.0292 (2)	0.00210 (15)	0.01535 (15)	−0.00126 (15)
Co	0.0262 (3)	0.0260 (3)	0.0212 (2)	0.0021 (2)	0.0107 (2)	0.00126 (19)
N1	0.0265 (16)	0.0358 (18)	0.0228 (15)	0.0092 (14)	0.0106 (13)	0.0055 (13)
N2	0.0319 (16)	0.0307 (16)	0.0220 (15)	0.0019 (14)	0.0078 (13)	0.0000 (13)
C1	0.060 (3)	0.034 (2)	0.050 (3)	−0.014 (2)	0.028 (2)	0.004 (2)
C2	0.068 (3)	0.037 (2)	0.052 (3)	−0.012 (2)	0.030 (2)	−0.001 (2)
C3	0.041 (2)	0.032 (2)	0.054 (3)	−0.0029 (19)	0.013 (2)	0.005 (2)
C4	0.029 (2)	0.027 (2)	0.058 (3)	−0.0019 (17)	0.0083 (19)	0.012 (2)
C5	0.027 (2)	0.041 (2)	0.048 (2)	0.0038 (18)	0.0133 (18)	0.020 (2)
C6	0.0232 (19)	0.033 (2)	0.040 (2)	−0.0012 (16)	0.0086 (17)	0.0112 (17)
C7	0.030 (2)	0.041 (2)	0.038 (2)	0.0031 (18)	0.0145 (17)	0.0157 (18)
C8	0.0246 (18)	0.038 (2)	0.032 (2)	0.0037 (16)	0.0143 (16)	0.0099 (17)
C9	0.0229 (18)	0.043 (2)	0.030 (2)	0.0056 (17)	0.0127 (16)	0.0143 (18)
C10	0.034 (2)	0.043 (2)	0.0262 (19)	0.0143 (18)	0.0161 (17)	0.0061 (17)
C11	0.042 (2)	0.039 (2)	0.0235 (19)	0.0099 (19)	0.0144 (17)	−0.0010 (17)
C12	0.053 (3)	0.035 (2)	0.027 (2)	0.0027 (19)	0.0169 (19)	−0.0055 (17)
C13	0.034 (2)	0.0276 (19)	0.031 (2)	−0.0017 (17)	0.0072 (17)	−0.0023 (16)
C14	0.0291 (19)	0.0301 (19)	0.0258 (18)	−0.0031 (16)	0.0044 (15)	0.0005 (16)
C15	0.028 (2)	0.0303 (19)	0.032 (2)	−0.0077 (16)	0.0060 (16)	−0.0031 (16)
C16	0.0198 (18)	0.033 (2)	0.032 (2)	−0.0022 (16)	0.0039 (15)	0.0016 (16)

C17	0.029 (2)	0.028 (2)	0.038 (2)	−0.0001 (16)	0.0100 (17)	0.0014 (17)
C18	0.0249 (19)	0.036 (2)	0.046 (2)	0.0023 (17)	0.0140 (18)	0.0096 (19)
C19	0.030 (2)	0.047 (2)	0.035 (2)	0.0060 (19)	0.0141 (17)	0.0045 (19)
C20	0.032 (2)	0.041 (2)	0.040 (2)	−0.0026 (18)	0.0141 (18)	−0.0102 (18)
C21	0.030 (2)	0.035 (2)	0.039 (2)	−0.0036 (17)	0.0121 (17)	0.0011 (18)

Geometric parameters (Å, º)

Br1—Co	2.3753 (6)	C10—C11	1.523 (5)
Br2—Co	2.3924 (6)	C10—H10A	0.9900
Co—N1	2.021 (3)	C10—H10B	0.9900
Co—N2	2.037 (3)	C11—C12	1.510 (5)
N1—C9	1.286 (5)	C11—H11A	0.9900
N1—C10	1.474 (5)	C11—H11B	0.9900
N2—C13	1.266 (5)	C12—H12A	0.9900
N2—C12	1.487 (4)	C12—H12B	0.9900
C1—C6	1.386 (5)	C13—C14	1.441 (5)
C1—C2	1.393 (6)	C13—H13A	0.9500
C1—H1A	0.9500	C14—C15	1.336 (5)
C2—C3	1.366 (6)	C14—H14A	0.9500
C2—H2A	0.9500	C15—C16	1.461 (5)
C3—C4	1.361 (6)	C15—H15A	0.9500
C3—H3A	0.9500	C16—C17	1.396 (5)
C4—C5	1.379 (6)	C16—C21	1.406 (5)
C4—H4A	0.9500	C17—C18	1.373 (5)
C5—C6	1.402 (5)	C17—H17A	0.9500
C5—H5A	0.9500	C18—C19	1.379 (6)
C6—C7	1.451 (6)	C18—H18A	0.9500
C7—C8	1.338 (5)	C19—C20	1.399 (6)
C7—H7A	0.9500	C19—H19A	0.9500
C8—C9	1.429 (5)	C20—C21	1.376 (5)
C8—H8A	0.9500	C20—H20A	0.9500
C9—H9A	0.9500	C21—H21A	0.9500
N1—Co—N2	100.80 (12)	N1—C10—H10B	109.5
N1—Co—Br1	111.12 (8)	C11—C10—H10B	109.5
N2—Co—Br1	108.83 (9)	H10A—C10—H10B	108.1
N1—Co—Br2	113.70 (8)	C12—C11—C10	115.2 (3)
N2—Co—Br2	110.32 (8)	C12—C11—H11A	108.5
Br1—Co—Br2	111.49 (2)	C10—C11—H11A	108.5
C9—N1—C10	117.3 (3)	C12—C11—H11B	108.5
C9—N1—Co	127.8 (3)	C10—C11—H11B	108.5
C10—N1—Co	114.9 (2)	H11A—C11—H11B	107.5
C13—N2—C12	116.6 (3)	N2—C12—C11	112.8 (3)
C13—N2—Co	124.9 (2)	N2—C12—H12A	109.0
C12—N2—Co	118.5 (2)	C11—C12—H12A	109.0
C6—C1—C2	120.6 (4)	N2—C12—H12B	109.0
C6—C1—H1A	119.7	C11—C12—H12B	109.0

C2—C1—H1A	119.7	H12A—C12—H12B	107.8
C3—C2—C1	120.6 (4)	N2—C13—C14	124.9 (3)
C3—C2—H2A	119.7	N2—C13—H13A	117.5
C1—C2—H2A	119.7	C14—C13—H13A	117.5
C4—C3—C2	119.7 (4)	C15—C14—C13	120.6 (3)
C4—C3—H3A	120.1	C15—C14—H14A	119.7
C2—C3—H3A	120.1	C13—C14—H14A	119.7
C3—C4—C5	120.7 (4)	C14—C15—C16	126.8 (3)
C3—C4—H4A	119.6	C14—C15—H15A	116.6
C5—C4—H4A	119.6	C16—C15—H15A	116.6
C4—C5—C6	120.8 (4)	C17—C16—C21	118.0 (3)
C4—C5—H5A	119.6	C17—C16—C15	119.3 (3)
C6—C5—H5A	119.6	C21—C16—C15	122.7 (3)
C1—C6—C5	117.6 (4)	C18—C17—C16	121.5 (4)
C1—C6—C7	121.8 (3)	C18—C17—H17A	119.3
C5—C6—C7	120.6 (3)	C16—C17—H17A	119.3
C8—C7—C6	126.7 (3)	C17—C18—C19	120.2 (4)
C8—C7—H7A	116.6	C17—C18—H18A	119.9
C6—C7—H7A	116.6	C19—C18—H18A	119.9
C7—C8—C9	122.6 (3)	C18—C19—C20	119.6 (4)
C7—C8—H8A	118.7	C18—C19—H19A	120.2
C9—C8—H8A	118.7	C20—C19—H19A	120.2
N1—C9—C8	123.9 (3)	C21—C20—C19	120.3 (4)
N1—C9—H9A	118.1	C21—C20—H20A	119.8
C8—C9—H9A	118.1	C19—C20—H20A	119.8
N1—C10—C11	110.7 (3)	C20—C21—C16	120.4 (4)
N1—C10—H10A	109.5	C20—C21—H21A	119.8
C11—C10—H10A	109.5	C16—C21—H21A	119.8
N2—Co—N1—C9	160.1 (3)	C10—N1—C9—C8	−179.2 (3)
Br1—Co—N1—C9	−84.6 (3)	Co—N1—C9—C8	−1.1 (5)
Br2—Co—N1—C9	42.1 (3)	C7—C8—C9—N1	−179.6 (4)
N2—Co—N1—C10	−21.7 (3)	C9—N1—C10—C11	−130.3 (3)
Br1—Co—N1—C10	93.5 (2)	Co—N1—C10—C11	51.3 (3)
Br2—Co—N1—C10	−139.7 (2)	N1—C10—C11—C12	−80.7 (4)
N1—Co—N2—C13	−169.1 (3)	C13—N2—C12—C11	147.5 (4)
Br1—Co—N2—C13	74.0 (3)	Co—N2—C12—C11	−35.0 (4)
Br2—Co—N2—C13	−48.7 (3)	C10—C11—C12—N2	70.0 (4)
N1—Co—N2—C12	13.6 (3)	C12—N2—C13—C14	176.2 (3)
Br1—Co—N2—C12	−103.3 (3)	Co—N2—C13—C14	−1.1 (5)
Br2—Co—N2—C12	134.1 (2)	N2—C13—C14—C15	178.7 (4)
C6—C1—C2—C3	−0.2 (7)	C13—C14—C15—C16	178.8 (4)
C1—C2—C3—C4	0.8 (7)	C14—C15—C16—C17	−171.5 (4)
C2—C3—C4—C5	−0.7 (6)	C14—C15—C16—C21	8.5 (6)
C3—C4—C5—C6	0.0 (6)	C21—C16—C17—C18	−1.7 (5)
C2—C1—C6—C5	−0.5 (6)	C15—C16—C17—C18	178.3 (3)
C2—C1—C6—C7	177.9 (4)	C16—C17—C18—C19	0.6 (6)
C4—C5—C6—C1	0.6 (6)	C17—C18—C19—C20	0.2 (6)

C4—C5—C6—C7	−177.8 (3)	C18—C19—C20—C21	0.0 (6)
C1—C6—C7—C8	−7.3 (6)	C19—C20—C21—C16	−1.2 (6)
C5—C6—C7—C8	171.1 (4)	C17—C16—C21—C20	1.9 (5)
C6—C7—C8—C9	−179.6 (4)	C15—C16—C21—C20	−178.0 (4)