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# Dibromido(2,9-dimethyl-1,10-phenanthroline-$\kappa^2N,N'$)cobalt(II) acetonitrile monosolvate

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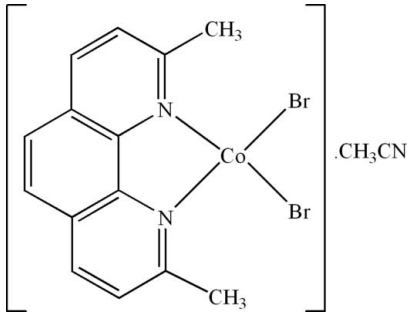
Key indicators: single-crystal X-ray study; $T$ = 120 K; mean $\sigma$(C–C) = 0.014 Å; $R$ factor = 0.075; $wR$ factor = 0.194; data-to-parameter ratio = 16.8.

In the title compound, $[CoBr_2(C_{14}H_{12}N_2)]\cdot CH_3CN$, the $Co^{II}$ atom is four-coordinated in a distorted tetrahedral geometry by two N atoms from a chelating 2,9-dimethyl-1,10-phenanthroline ligand and two terminal Br atoms. In the crystal, $\pi$–$\pi$ contacts between the pyridine and benzene rings [centroid–centroid distances = 3.828 (5), 3.782 (5), 3.880 (5) and 3.646 (5) Å] stabilize the structure.

## Related literature

For related structures, see: Akbarzadeh Torbati *et al.* (2010); Alizadeh *et al.* (2009); Ding *et al.* (2006); Fanizzi *et al.* (1991); Lemoine *et al.* (2003); Robinson & Sinn (1975).




## Experimental

### Crystal data

$[CoBr_2(C_{14}H_{12}N_2)]\cdot C_2H_3N$
$M_r$ = 468.04
Monoclinic, $P2_1/n$
$a$ = 7.6380 (5) Å
$b$ = 12.7943 (6) Å
$c$ = 17.9545 (11) Å
$\beta$ = 101.128 (5)°
$V$ = 1721.58 (18) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 5.64 mm$^{-1}$
$T$ = 120 K
0.35 × 0.20 × 0.15 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.259, $T_{max}$ = 0.459
8417 measured reflections
3366 independent reflections
2345 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.101

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.194
$S$ = 1.02
3366 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.11 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.03 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Co1—N1 | 2.051 (8) | Co1—Br1 | 2.3592 (14) |
| Co1—N2 | 2.036 (8) | Co1—Br2 | 2.3682 (14) |

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: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2596).

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# supporting information

*Acta Cryst.* (2012). E**68**, m1407 [doi:10.1107/S160053681204367X]

# Dibromido(2,9-dimethyl-1,10-phenanthroline-$\kappa^2N,N'$)cobalt(II) acetonitrile monosolvate

**Sadif A. Shirvan, Manouchehr Aghajeri, Sara Haydari Dezfuli, Fereydoon Khazali and Ali Borsalani**

## S1. Comment

2,9-Dimethyl-1,10-phenanthroline (dmphen) is a good bidentate ligand, and numerous complexes with dmphen have been prepared, such as that of mercury (Alizadeh *et al.*, 2009), copper (Lemoine *et al.*, 2003), nickel (Ding *et al.*, 2006), gold (Robinson & Sinn, 1975), platinum (Fanizzi *et al.*, 1991) and cobalt (Akbarzadeh Torbati *et al.*, 2010). Here, we report the synthesis and structure of the title compound.

In the title compound (Fig. 1), the $Co^{II}$ atom is four-coordinated in a distorted tetrahedral geometry by two N atoms from a chelating dmphen ligand and two terminal Br atoms. The Co—Br and Co—N bond lengths (Table 1) and angles are normal. In the crystal, $\pi$–$\pi$ contacts between the pyridine and benzene rings (Fig. 2), *Cg*3···*Cg*3$^{i}$, *Cg*3···*Cg*4$^{i}$, *Cg*3···*Cg*4$^{ii}$ and *Cg*4···*Cg*4$^{ii}$ [symmetry codes: (i) -x, 1-y, 1-z; (ii) 1-x, 1-y, 1-z, *Cg*3 and *Cg*4 are the centroids of the N2, C8–C11, C13 ring and C5–C8, C13, C14 ring, respectively], with centroid–centroid distances of 3.828 (5), 3.782 (5), 3.880 (5) and 3.646 (5) Å, stabilize the structure.

## S2. Experimental

For the preparation of the title compound, a solution of 2,9-dimethyl-1,10-phenanthroline (0.28 g, 1.33 mmol) in methanol (20 ml) was added to a solution of $CoBr_2$ (0.29 g, 1.33 mmol) in acetonitrile (15 ml) and the resulting blue solution was stirred for 15 min at 313 K. This solution was left to evaporate slowly at room temperature. After one week, blue block crystals of the title compound were isolated (yield: 0.46 g, 73.9%).

## S3. Refinement

All H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (aromatic) and 0.96 ($CH_3$) Å and with $U_{iso}$(H) = 1.2(1.5 for methyl)$U_{eq}$(C). The highest residual electron density was found 0.92 Å from Br2 the deepest hole 1.02 Å from Br1.

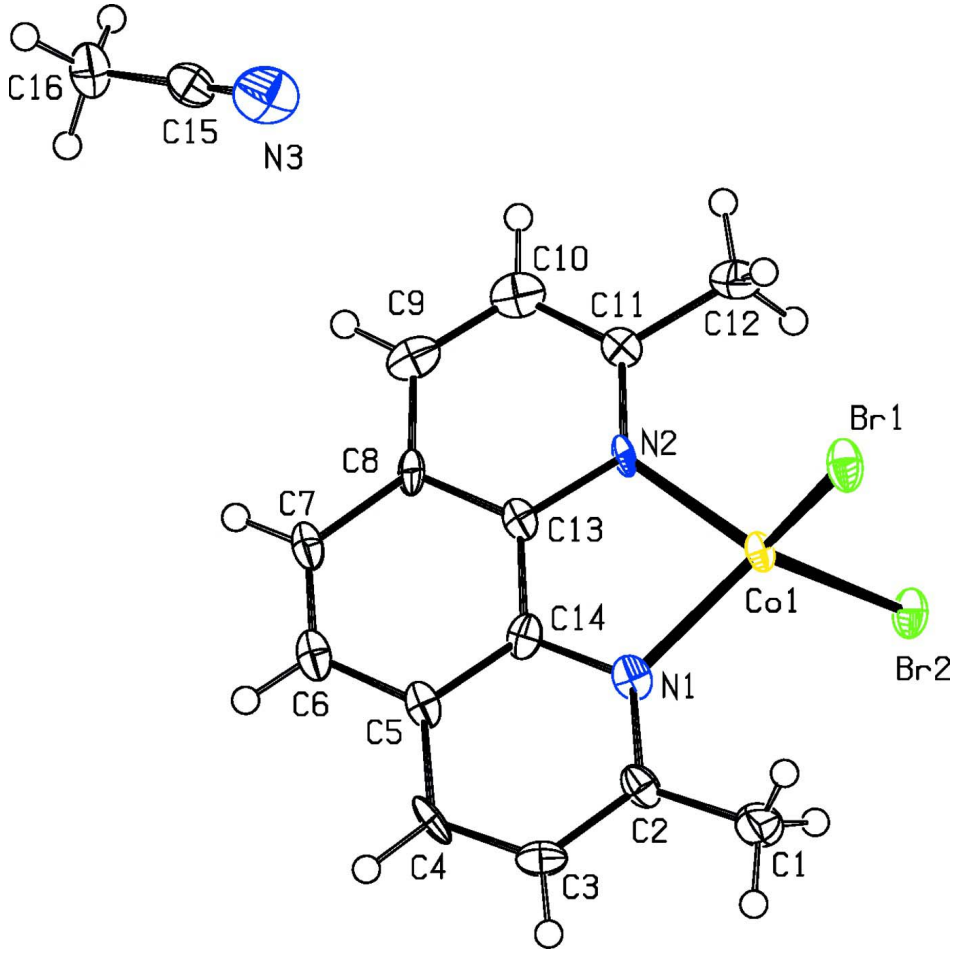


**Figure 1**

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

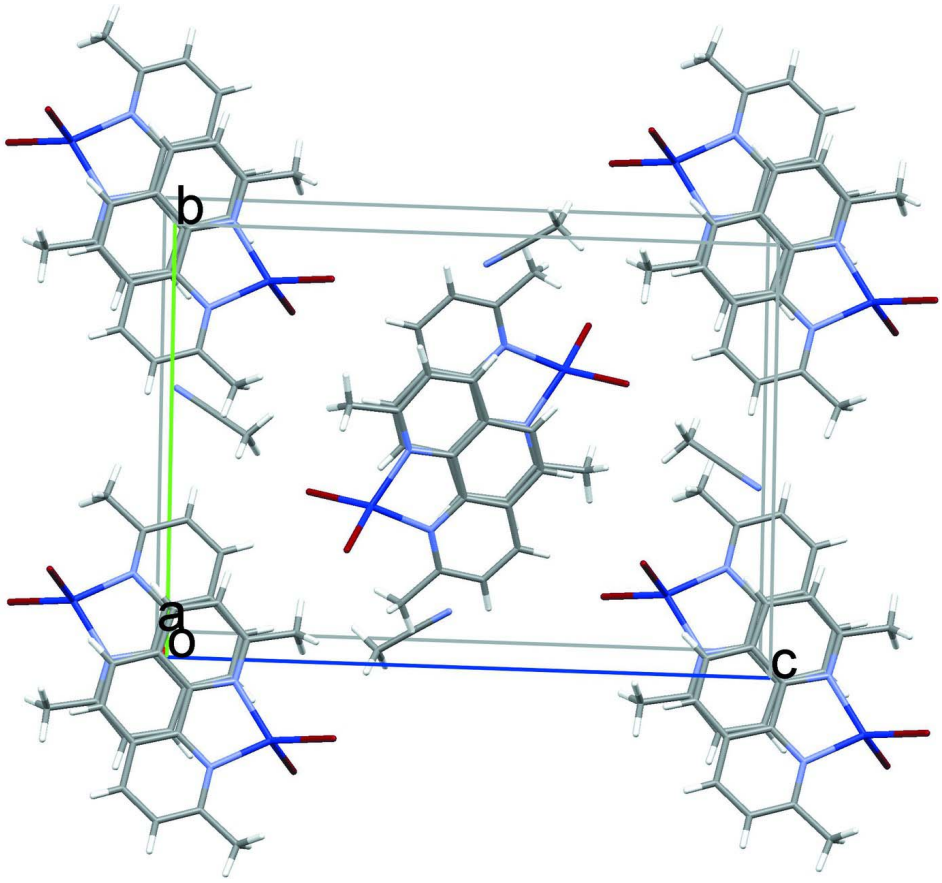


**Figure 2**

Crystal packing diagram for the title compound.

**Dibromido(2,9-dimethyl-1,10-phenanthroline-$\kappa^2N,N'$)cobalt(II) acetonitrile monosolvate**

*Crystal data*

$[CoBr_2(C_{14}H_{12}N_2)]\cdot C_2H_3N$
$M_r = 468.04$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.6380\ (5)$ Å
$b = 12.7943\ (6)$ Å
$c = 17.9545\ (11)$ Å
$\beta = 101.128\ (5)°$
$V = 1721.58\ (18)$ Å$^3$
$Z = 4$
$F(000) = 916$
$D_x = 1.806$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8417 reflections
$\theta = 2.0–26.0°$
$\mu = 5.64$ mm$^{-1}$
$T = 120$ K
Block, blue
0.35 × 0.20 × 0.15 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.259$, $T_{max} = 0.459$
8417 measured reflections
3366 independent reflections
2345 reflections with $I > 2\sigma(I)$
$R_{int} = 0.101$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.0°$
$h = -9\rightarrow9$
$k = -15\rightarrow13$
$l = -22\rightarrow20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.194$
$S = 1.02$
3366 reflections
200 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
$w = 1/[\sigma^2(F_o^2) + (0.1187P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 1.11$ e Å$^{-3}$
$\Delta\rho_{min} = -1.03$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Co1 | 0.15502 (14) | 0.35537 (10) | 0.33642 (7) | 0.0162 (3) |
| Br1 | 0.31107 (12) | 0.37421 (8) | 0.23591 (6) | 0.0249 (3) |
| Br2 | −0.12001 (11) | 0.26909 (8) | 0.29292 (6) | 0.0233 (3) |
| C1 | 0.3312 (15) | 0.1227 (8) | 0.3918 (6) | 0.032 (2) |
| H1A | 0.3906 | 0.1476 | 0.3528 | 0.038* |
| H1B | 0.2067 | 0.1129 | 0.3711 | 0.038* |
| H1C | 0.3828 | 0.0574 | 0.4112 | 0.038* |
| C2 | 0.3520 (11) | 0.2011 (8) | 0.4548 (5) | 0.020 (2) |
| C3 | 0.4380 (12) | 0.1773 (8) | 0.5287 (6) | 0.024 (2) |
| H3 | 0.4825 | 0.1103 | 0.5397 | 0.029* |
| C4 | 0.4587 (10) | 0.2517 (8) | 0.5861 (5) | 0.022 (2) |
| H4 | 0.5151 | 0.2350 | 0.6353 | 0.026* |
| C5 | 0.3920 (10) | 0.3531 (8) | 0.5678 (5) | 0.0176 (19) |
| C6 | 0.4112 (11) | 0.4362 (8) | 0.6219 (6) | 0.022 (2) |
| H6 | 0.4712 | 0.4237 | 0.6712 | 0.026* |
| C7 | 0.3452 (11) | 0.5316 (8) | 0.6035 (5) | 0.022 (2) |
| H7 | 0.3570 | 0.5835 | 0.6404 | 0.026* |
| C8 | 0.2557 (10) | 0.5549 (7) | 0.5268 (5) | 0.0178 (19) |
| C9 | 0.1829 (12) | 0.6513 (8) | 0.5024 (7) | 0.026 (2) |
| H9 | 0.1894 | 0.7065 | 0.5365 | 0.031* |
| C10 | 0.1008 (12) | 0.6654 (8) | 0.4275 (7) | 0.028 (2) |
| H10 | 0.0564 | 0.7308 | 0.4107 | 0.033* |
| C11 | 0.0845 (10) | 0.5820 (7) | 0.3772 (5) | 0.0166 (18) |
| C12 | −0.0028 (13) | 0.5960 (8) | 0.2951 (6) | 0.027 (2) |
| H12A | −0.1007 | 0.5481 | 0.2824 | 0.033* |

| | | | | |
|---|---|---|---|---|
| H12B | 0.0828 | 0.5825 | 0.2635 | 0.033* |
| H12C | −0.0460 | 0.6664 | 0.2870 | 0.033* |
| C13 | 0.2355 (10) | 0.4741 (7) | 0.4725 (5) | 0.0165 (19) |
| C14 | 0.3071 (10) | 0.3727 (7) | 0.4935 (6) | 0.0182 (19) |
| C15 | 0.1720 (12) | 0.9623 (8) | 0.5702 (6) | 0.027 (2) |
| C16 | 0.1350 (16) | 1.0197 (10) | 0.6364 (7) | 0.039 (3) |
| H16A | 0.2341 | 1.0645 | 0.6560 | 0.059* |
| H16B | 0.1176 | 0.9710 | 0.6750 | 0.059* |
| H16C | 0.0293 | 1.0612 | 0.6216 | 0.059* |
| N1 | 0.2885 (9) | 0.2967 (6) | 0.4379 (4) | 0.0182 (16) |
| N2 | 0.1506 (8) | 0.4887 (6) | 0.3979 (4) | 0.0138 (15) |
| N3 | 0.2013 (13) | 0.9203 (8) | 0.5206 (7) | 0.043 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Co1 | 0.0134 (5) | 0.0219 (7) | 0.0121 (6) | −0.0001 (5) | −0.0010 (4) | −0.0009 (5) |
| Br1 | 0.0201 (4) | 0.0384 (6) | 0.0167 (5) | −0.0082 (4) | 0.0050 (4) | −0.0041 (4) |
| Br2 | 0.0160 (4) | 0.0306 (6) | 0.0222 (5) | −0.0061 (4) | 0.0014 (3) | −0.0057 (4) |
| C1 | 0.042 (6) | 0.025 (6) | 0.027 (6) | 0.011 (5) | 0.005 (5) | 0.000 (5) |
| C2 | 0.016 (4) | 0.028 (5) | 0.017 (5) | 0.007 (4) | 0.002 (3) | 0.002 (4) |
| C3 | 0.022 (4) | 0.018 (5) | 0.032 (6) | 0.005 (4) | 0.008 (4) | 0.006 (4) |
| C4 | 0.011 (4) | 0.041 (6) | 0.011 (5) | 0.006 (4) | −0.004 (3) | 0.008 (4) |
| C5 | 0.006 (3) | 0.032 (5) | 0.015 (5) | −0.001 (3) | 0.003 (3) | 0.000 (4) |
| C6 | 0.013 (4) | 0.037 (6) | 0.015 (5) | −0.007 (4) | 0.004 (3) | −0.002 (4) |
| C7 | 0.019 (4) | 0.033 (6) | 0.012 (5) | −0.005 (4) | 0.002 (4) | −0.002 (4) |
| C8 | 0.011 (4) | 0.026 (5) | 0.015 (5) | −0.006 (3) | −0.001 (3) | −0.006 (4) |
| C9 | 0.016 (4) | 0.022 (5) | 0.041 (7) | −0.002 (4) | 0.013 (4) | 0.000 (5) |
| C10 | 0.018 (4) | 0.027 (6) | 0.040 (7) | 0.003 (4) | 0.010 (4) | 0.003 (5) |
| C11 | 0.009 (4) | 0.023 (5) | 0.019 (5) | 0.000 (3) | 0.007 (3) | 0.002 (4) |
| C12 | 0.025 (5) | 0.024 (5) | 0.034 (6) | 0.000 (4) | 0.006 (4) | 0.008 (5) |
| C13 | 0.007 (4) | 0.027 (5) | 0.015 (5) | −0.005 (3) | 0.003 (3) | 0.002 (4) |
| C14 | 0.008 (4) | 0.024 (5) | 0.023 (5) | −0.004 (3) | 0.002 (3) | −0.005 (4) |
| C15 | 0.023 (4) | 0.030 (6) | 0.027 (6) | 0.005 (4) | 0.002 (4) | 0.005 (5) |
| C16 | 0.045 (6) | 0.041 (7) | 0.026 (6) | −0.007 (5) | −0.005 (5) | −0.003 (5) |
| N1 | 0.008 (3) | 0.027 (4) | 0.020 (4) | 0.007 (3) | 0.004 (3) | −0.001 (3) |
| N2 | 0.006 (3) | 0.025 (4) | 0.009 (4) | −0.003 (3) | −0.003 (3) | −0.001 (3) |
| N3 | 0.043 (5) | 0.042 (6) | 0.050 (7) | 0.019 (5) | 0.018 (5) | 0.001 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Co1—N1 | 2.051 (8) | C8—C9 | 1.390 (14) |
| Co1—N2 | 2.036 (8) | C8—C13 | 1.409 (13) |
| Co1—Br1 | 2.3592 (14) | C9—C10 | 1.382 (16) |
| Co1—Br2 | 2.3682 (14) | C9—H9 | 0.9300 |
| C1—C2 | 1.497 (14) | C10—C11 | 1.387 (14) |
| C1—H1A | 0.9600 | C10—H10 | 0.9300 |
| C1—H1B | 0.9600 | C11—N2 | 1.321 (12) |

| | | | |
|---|---|---|---|
| C1—H1C | 0.9600 | C11—C12 | 1.507 (14) |
| C2—N1 | 1.329 (12) | C12—H12A | 0.9600 |
| C2—C3 | 1.395 (14) | C12—H12B | 0.9600 |
| C3—C4 | 1.389 (14) | C12—H12C | 0.9600 |
| C3—H3 | 0.9300 | C13—N2 | 1.383 (11) |
| C4—C5 | 1.409 (14) | C13—C14 | 1.429 (13) |
| C4—H4 | 0.9300 | C14—N1 | 1.382 (12) |
| C5—C14 | 1.389 (13) | C15—N3 | 1.099 (15) |
| C5—C6 | 1.428 (14) | C15—C16 | 1.471 (16) |
| C6—C7 | 1.337 (14) | C16—H16A | 0.9600 |
| C6—H6 | 0.9300 | C16—H16B | 0.9600 |
| C7—C8 | 1.444 (13) | C16—H16C | 0.9600 |
| C7—H7 | 0.9300 | | |

| | | | |
|---|---|---|---|
| N2—Co1—N1 | 83.3 (3) | C10—C9—H9 | 119.9 |
| N2—Co1—Br1 | 113.1 (2) | C8—C9—H9 | 119.9 |
| N1—Co1—Br1 | 118.62 (19) | C9—C10—C11 | 119.9 (9) |
| N2—Co1—Br2 | 117.58 (18) | C9—C10—H10 | 120.0 |
| N1—Co1—Br2 | 112.3 (2) | C11—C10—H10 | 120.0 |
| Br1—Co1—Br2 | 110.02 (6) | N2—C11—C10 | 122.1 (9) |
| C2—C1—H1A | 109.5 | N2—C11—C12 | 117.1 (8) |
| C2—C1—H1B | 109.5 | C10—C11—C12 | 120.8 (9) |
| H1A—C1—H1B | 109.5 | C11—C12—H12A | 109.5 |
| C2—C1—H1C | 109.5 | C11—C12—H12B | 109.5 |
| H1A—C1—H1C | 109.5 | H12A—C12—H12B | 109.5 |
| H1B—C1—H1C | 109.5 | C11—C12—H12C | 109.5 |
| N1—C2—C3 | 120.1 (9) | H12A—C12—H12C | 109.5 |
| N1—C2—C1 | 117.5 (8) | H12B—C12—H12C | 109.5 |
| C3—C2—C1 | 122.3 (9) | N2—C13—C8 | 122.6 (8) |
| C4—C3—C2 | 121.4 (9) | N2—C13—C14 | 117.6 (8) |
| C4—C3—H3 | 119.3 | C8—C13—C14 | 119.8 (8) |
| C2—C3—H3 | 119.3 | N1—C14—C5 | 122.0 (8) |
| C3—C4—C5 | 118.2 (9) | N1—C14—C13 | 117.8 (8) |
| C3—C4—H4 | 120.9 | C5—C14—C13 | 120.1 (8) |
| C5—C4—H4 | 120.9 | N3—C15—C16 | 179.1 (13) |
| C14—C5—C4 | 118.1 (9) | C15—C16—H16A | 109.5 |
| C14—C5—C6 | 119.0 (9) | C15—C16—H16B | 109.5 |
| C4—C5—C6 | 122.9 (9) | H16A—C16—H16B | 109.5 |
| C7—C6—C5 | 121.9 (9) | C15—C16—H16C | 109.5 |
| C7—C6—H6 | 119.1 | H16A—C16—H16C | 109.5 |
| C5—C6—H6 | 119.1 | H16B—C16—H16C | 109.5 |
| C6—C7—C8 | 120.7 (9) | C2—N1—C14 | 120.0 (8) |
| C6—C7—H7 | 119.7 | C2—N1—Co1 | 129.6 (7) |
| C8—C7—H7 | 119.7 | C14—N1—Co1 | 110.4 (6) |
| C9—C8—C13 | 116.7 (9) | C11—N2—C13 | 118.5 (8) |
| C9—C8—C7 | 124.8 (9) | C11—N2—Co1 | 130.6 (6) |
| C13—C8—C7 | 118.5 (9) | C13—N2—Co1 | 110.9 (6) |
| C10—C9—C8 | 120.1 (10) | | |

| | | | |
|---|---|---|---|
| N1—C2—C3—C4 | 0.4 (13) | C1—C2—N1—C14 | −178.8 (8) |
| C1—C2—C3—C4 | 178.9 (9) | C3—C2—N1—Co1 | −179.3 (6) |
| C2—C3—C4—C5 | −0.7 (13) | C1—C2—N1—Co1 | 2.1 (12) |
| C3—C4—C5—C14 | 0.8 (11) | C5—C14—N1—C2 | 0.4 (12) |
| C3—C4—C5—C6 | −177.9 (8) | C13—C14—N1—C2 | −179.6 (7) |
| C14—C5—C6—C7 | 1.9 (12) | C5—C14—N1—Co1 | 179.6 (6) |
| C4—C5—C6—C7 | −179.3 (8) | C13—C14—N1—Co1 | −0.4 (8) |
| C5—C6—C7—C8 | −1.8 (12) | N2—Co1—N1—C2 | 179.2 (7) |
| C6—C7—C8—C9 | 179.9 (8) | Br1—Co1—N1—C2 | −68.1 (8) |
| C6—C7—C8—C13 | 1.6 (12) | Br2—Co1—N1—C2 | 62.1 (7) |
| C13—C8—C9—C10 | −2.0 (12) | N2—Co1—N1—C14 | 0.0 (5) |
| C7—C8—C9—C10 | 179.6 (8) | Br1—Co1—N1—C14 | 112.7 (5) |
| C8—C9—C10—C11 | 2.6 (13) | Br2—Co1—N1—C14 | −117.1 (5) |
| C9—C10—C11—N2 | −2.2 (13) | C10—C11—N2—C13 | 1.2 (11) |
| C9—C10—C11—C12 | −179.3 (8) | C12—C11—N2—C13 | 178.4 (7) |
| C9—C8—C13—N2 | 1.1 (11) | C10—C11—N2—Co1 | −177.4 (6) |
| C7—C8—C13—N2 | 179.5 (7) | C12—C11—N2—Co1 | −0.2 (10) |
| C9—C8—C13—C14 | 180.0 (7) | C8—C13—N2—C11 | −0.7 (11) |
| C7—C8—C13—C14 | −1.6 (11) | C14—C13—N2—C11 | −179.6 (7) |
| C4—C5—C14—N1 | −0.7 (11) | C8—C13—N2—Co1 | 178.2 (6) |
| C6—C5—C14—N1 | 178.1 (7) | C14—C13—N2—Co1 | −0.7 (8) |
| C4—C5—C14—C13 | 179.3 (7) | N1—Co1—N2—C11 | 179.1 (7) |
| C6—C5—C14—C13 | −1.9 (11) | Br1—Co1—N2—C11 | 60.8 (7) |
| N2—C13—C14—N1 | 0.7 (10) | Br2—Co1—N2—C11 | −69.2 (7) |
| C8—C13—C14—N1 | −178.2 (7) | N1—Co1—N2—C13 | 0.4 (5) |
| N2—C13—C14—C5 | −179.3 (7) | Br1—Co1—N2—C13 | −117.9 (5) |
| C8—C13—C14—C5 | 1.8 (11) | Br2—Co1—N2—C13 | 112.1 (5) |
| C3—C2—N1—C14 | −0.2 (12) | | |