**Supporting Information for:**

Unprecedented Inequivalent Metal Coordination Environments in a Mixed-Ligand Dicobalt Complex

Giacomo Cioncoloni, Stephen Sproules, Claire Wilson and Mark D. Symes\*

WestCHEM, School of Chemistry, University of Glasgow, University Avenue, Glasgow, G12 8QQ, UK.

\*E-mail: mark.symes@glasgow.ac.uk

EXPERIMENTAL SECTION

**General Experimental Remarks:** All solvents were obtained from Sigma Aldrich and used as supplied. 5,5′-dimethyl-2,2′-bipyridine (98%), *o*-benzenedithiol (96%) and Co(NO3)2·6H2O (98%) and tetrabutylammonium hexafluorophosphate (TBA-PF6) (99%) were supplied by Sigma Aldrich.

All 1H and 13C NMR spectra were recorded on a Bruker AV 400 instrument, at a constant temperature of 300 K. Chemical shifts are reported in parts per million from low to high field. Coupling constants (*J*) are reported in hertz (Hz). Standard abbreviations indicating multiplicity were used as follows: m = multiplet, d = doublet, s = singlet. UV-Vis spectra were recorded on a JASCO V-670 spectrophotometer using 1 cm pathlength cuvettes. CHN analyses were collected by the services facility at the School of Chemistry, University of Glasgow, as were LM-MS mass spectra (ESI, positive mode, Bruker micrOTOF-Q machine). IR spectra were collected in the solid state on a Shimadzu IRAffinity-1S Fourier Transform Infrared Spectrophotometer. Melting points were gauged using an Electrothermal IA 9000 digital melting point machine. Experiments performed at “room temperature” were carried out at 20 °C. Electrochemical experiments were performed as below.

**Synthesis of [CoIII2(bdt)2(Me2bpy)3](NO3)2, [1](NO3)2:** To a solution of 5,5′-dimethyl-2,2′-bipyridine (0.640 g, 3.48 mmol, 2 eq.) in methanol (20 mL) under air was added a solution of Co(NO3)2·6H2O(0.508 g, 1.74 mmol, 1 eq.) in methanol (10 mL). To this was added a solution of *o*-benzenedithiol (0.248 g, 1.74 mmol, 1 eq.) in 10 mL methanol. The reaction mixture was observed to turn dark green upon addition of the dithiol. After stirring in air for 1 h, the solvent was removed under reduced pressure and the resulting solid re-dissolved in 10 mL MeOH. Around 25 mL of diethyl ether were then added, inducing the formation of a dark green precipitate and a dark brown supernatant solution. This dark green precipitate was isolated by filtration and then re-dissolved in 15 mL of MeOH. To this dark green solution was then added 50 mL of diethyl ether, yielding pure compound [**1**](NO3)2 as a green microcrystalline solid.This was carefully dried at 150 °C overnight to give a final anhydrous mass yield of 0.385 g (41%).Complex [**1**](NO3)2 is somewhat hygroscopic and hence absorbs water when exposed to laboratory air. Indeed, a sample of dry mass 364 mg was found to increase in mass by 25 mg after standing in air for 1 day (upon further standing the mass did not increase further). This corresponds to a mass-gain of around 6%, corresponding to around 3.5 molecules of H2O per formula unit of [**1**](NO3)2. CHN analysis was then performed on this hydrated sample: Anal. calcd. for C48H44Co2N8O6S4·(3.5H2O): C 50.66, H 4.52, N 9.85. Found: C 50.61, H 4.40, N 10.35. Yields are calculated from the dry (anhydrous) mass. 1H NMR (MeOD, 400 MHz), δ = 10.34-10.32 (m, 2H, Ha or Hi or Hp), 8.44-8.40 (m, 2H, Ha or Hi or Hp), 8.38 (d, 2H, *J =* 8.2, Hd or Hl or Hm), 8.29 (d, 2H, *J =* 8.3, Hd or Hl or Hm), 8.08-8.03 (m, 2H, Hc or Hk or Hn), 8.02-7.91 (m, 6H, Hd or Hl or Hm and Hc and/or Hk and/or Hn), 7.38-7.34 (m, 2H, Ha or Hi or Hp), 6.92-6.86 (m, 2H, He or Hf or Hg or Hh), 6.60 (dd, 2H, *J1 =* 8.0, *J2 =* 0.8, He or Hf or Hg or Hh), 6.56-6.50 (m, 2H, He or Hf or Hg or Hh), 6.03 (dd, 2H, *J1 =* 7.8, *J2 =* 0.9, He or Hf or Hg or Hh), 2.67 (s, 6H, Hb or Hj or Ho), 2.29 (s, 6H, Hb or Hj or Ho), 2.26 (s, 6H, Hb or Hj or Ho). Assignments of signals to groups of protons are based on 2D (COSY) spectra and the expected coupling patterns of the peaks, although the symmetry of the molecule prevents unambiguous assignments. Letter codes correspond to those shown in Scheme 1. The 1H NMR spectrum of this compound is shown in the Supporting Information (Figures S1 and S2). 13C NMR (MeOD, 400 MHz), δ = 159.5, 156.0, 155.9, 155.0, 153.1, 151.7, 149.2, 143.2, 142.8, 141.2, 140.7, 138.7, 138.5, 132.7, 132.4, 130.6, 129.8, 125.4, 125.1, 124.8, 123.2, 19.5, 18.9, 18.6.IR (solid state, cm−1) ν = 3041 (w), 1473 (w), 1432 (w), 1331 (s), 1236 (m), 825 (m), 734 (m). ESI-LMMS (methanol): *m/z* = 475.0508 [M]2+ (calcd. for C48H44Co2N6S4; 475.0587).

**Electrochemical Methods:** Electrochemical studies were performed in a single chamber cell in a three-electrode configuration using a CH Instruments CHI700 series potentiostat. The supporting electrolyte was 1 M TBA-PF6 in acetonitrile, unless otherwise noted. A Pt wire was used as the counter electrode, along with an Ag/AgNO3 pseudo reference electrode. Potentials are reported relative to the ferrocene/ferrocenium couple, the position of which was judged by adding ferrocene to the samples analyzed. Working electrodes were washed with acetone and deionized water prior to use. Cyclic voltammograms were collected at room temperature under an atmosphere of Ar at a scan rate of 100 mV s–1. A glassy carbon button electrode (area = 0.071 cm2, CH Instruments) was used as the working electrode for cyclic voltammetry. Measurements were conducted without stirring and with *i*R compensation enabled. Bulk electrolyses were carried out in 0.1 M TBA-PF6 in acetonitrile in a two-chamber cell, using an Ag/AgNO3 pseudo reference electrode, a large area carbon felt counter electrode and a large area carbon felt working electrode. Solutions were stirred during bulk electrolysis.

**Calculations:** The program package ORCA was used for all calculations.[[[1]](#endnote-2)] The geometries of all molecules were fully optimized by a spin-unrestricted DFT method employing the BP86 functional with acetonitrile as solvent.[[[2]](#endnote-3)] Triple-ξ-quality basis sets with one set of polarization functions (def2-TZVP) were used for all atoms.[[[3]](#endnote-4)] The single-point calculations were performed with PBE0[[[4]](#endnote-5)] functional on optimized and crystallographic coordinates using the same basis sets and enhanced integration accuracy for metal atoms (SPECIALGRIDINTACC 10). A scalar relativistic correction was applied using the zeroth-order regular approximation (ZORA) method.[[[5]](#endnote-6)] The RIJCOSX approximation[[[6]](#endnote-7)] combined with the appropriate Ahlrichs auxiliary basis set was used to speed up the calculations.[[[7]](#endnote-8)] The conductor-like screening model (COSMO) was used for all calculations.[[[8]](#endnote-9)] The geometry search for all complexes was carried out in redundant internal coordinates without imposing geometry constraints. The self-consistent field calculations were tightly converged (1 × 10–8 *E*h in energy, 1 × 10–7 *E*h in charge density, and 1 × 10–7 in the maximum element of the DIIS[[[9]](#endnote-10)] error vector). The geometry was converged with the following convergence criteria: change in energy <10–5 *E*h, average force <5 × 10–4 *E*h Bohr–1, and the maximum force 10–4 *E*h Bohr–1. The geometry search for all complexes was carried out in redundant internal coordinates without imposing geometry constraints. The stability of all solutions was checked by performing frequency calculations: No negative frequencies were observed. Molecular orbitals and spin density maps were visualized via the program Molekel.[[[10]](#endnote-11)]

**Crystallography:** Crystallographic data were collected at the EPSRC UK National Crystallography Service at the University of Southampton using a rotating anode radiation source.[[[11]](#endnote-12)] A dark blue/green opaque crystal of dimensions 0.10 × 0.09 × 0.02 mm was used for single crystal X-ray diffraction data collection. C48H44Co2N6S4·2(NO3)·1.25(H2O)·0.25(CH3OH) crystallised in the Monoclinic space group *P*21/*n* (space group No. 14), with unit cell dimensions *a* = 12.6021 (3), *b* = 22.7893 (5), *c* = 17.1872 (6) Å, β = 103.500 (3)° and *V* = 4799.7 (2) Å3, T = 100 K. 59216 reflections were measured by ω scans, 11002 independent reflections with *R*int = 0.053, θmax = 27.5°, θmin = 2.4° using Mo *Kα* radiation, λ = 0.71073 Å, on a Rigaku FRE+ equipped with VHF Varimax confocal mirrors and an AFC10 goniometer and HG Saturn 724+ detector diffractometer. Data were integrated using *CrysAlis PRO* 1.171.38.43 (Rigaku OD, 2015), with Lorentz and polarization corrections made. A multi-scan correction for absorption was applied *T*min = 0.538, *T*max = 1.000, μ = 0.93 mm−1, *Mr* = 1105.54, *F*(000) = 2284*ρcalcd* = 1.530 Mg m−3.

The structure was solved using ShelXT.[[[12]](#endnote-13)] All 11002 reflections were used in the refinement and positions and anisotropic atomic displacement parameters (adps) were refined for all fully occupied non-Hydrogen atoms using SHELXL within OLEX2.[[[13]](#endnote-14),[[14]](#endnote-15)] A region of lattice solvent was modelled as 0.75 H2O with two further 0.25 occupied H2O molecules and a 0.25 occupied molecule of MeOH. One nitrate anion was modelled with the oxygen atoms over 2 partially occupied (0.9/0.1) sites. The 0.75 occupied H2O hydrogen atoms were located in difference Fourier maps and refined with O-H distance restraints, hydrogen atoms for the 0.25 H2O and MeOH were not included in the model but were included in the unit cell contents and values derived from them, otherwise hydrogen atoms were placed in calculated positions and refined as part of a riding model or as a rigid rotor for Me hydrogens. Final *wR*(*F*2) = 0.097, *R*[*F*2 > 2σ(*F*2)] = 0.043 (8780 reflections with *I* > 2σ(*I*)) for 670 parameters, Δρmax = 0.50 e Å−3 and Δρmin = −0.39 e Å−3 maximum and minimum residual electron density. CCDC 1537318 contains the supplementary crystallographic data for this paper.

**Table S1.** Geometry Optimized Coordinates for [CoIII,III2(bdt)2(Me2bpy)3]2+

Co 3.61899346119338 15.90726059976303 8.31231950541295

Co 6.23341988657192 17.34313766711827 6.60197370641610

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H 6.72119893913484 17.17892478714695 0.38622474215283

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H 11.62864663850042 20.01828628486027 7.84798043076544

**Table S2.** Geometry Optimized Coordinates for [CoIII,II2(bdt)2(Me2bpy)3]1+

Co 3.52839390506793 15.80798856453735 8.22893019260935

Co 6.33770385871764 17.41413457052274 6.54888522320445

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S 2.14619784791926 17.54618665928048 7.68289181401158

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H 8.51516765915106 13.27656808716478 10.33032175798631

H 8.54242089042614 14.98212327773275 10.82217123373777

H 10.05487786639145 14.15154485302875 10.36759086864515

C 2.10185663815578 19.66745987278827 3.99593811612203

H 1.26120510348692 19.85323058210465 4.68309701867812

H 2.00686798052307 18.63899339922640 3.62587159710985

H 1.99300181611512 20.35890781937962 3.14942943689138

C 6.32505939589254 11.49086116252935 6.04524664611442

H 6.80300351478236 10.50966286073593 6.03263566481103

C 9.54420256618400 20.06259155192429 8.09338115177462

C 0.05943610373610 12.63549906946401 5.38220087059040

H 0.74600084863259 12.94695627039965 4.58491172683553

H -0.96146491979945 12.92473455442322 5.09248331571231

H 0.07931246832804 11.53665885667834 5.44459329142527

C 10.85977876800065 19.92747623301193 8.80650388829222

H 11.10067356069118 18.87602504358874 9.00773314158436

H 10.84301948318512 20.46710075350748 9.76478991671847

H 11.67556008002890 20.35998142422307 8.20818452901959

**Additional single crystal crystallographic data for complex [1]2+ (CCDC 1537318)**

**Refinement**

Crystal data, data collection and structure refinement details are summarized below.

**Computing details**

Data collection: *CrystalClear*-SM Expert 3.1 b27 (Rigaku, 2013); cell refinement: *CrysAlis PRO* 1.171.38.43 (Rigaku OD, 2015); data reduction: *CrysAlis PRO* 1.171.38.43 (Rigaku OD, 2015); program(s) used to solve structure: ShelXT (Sheldrick, 2015); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: Olex2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 (Dolomanov *et al.*, 2009).

**References**

[[15]](#endnote-16)Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

[[16]](#endnote-17)Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.

[[17]](#endnote-18)Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

*Crystal data*

|  |  |
| --- | --- |
| C48H44Co2N6S4·2(NO3)·1.25(H2O)·0.25(CH3OH) | *F*(000) = 2284 |
| *Mr* = 1105.54 | *D*x = 1.530 Mg m-3 |
| Monoclinic, *P*21/*n* | Mo *K* radiation,  = 0.71073 Å |
| *a* = 12.6021 (3) Å | Cell parameters from 24504 reflections |
| *b* = 22.7893 (5) Å |  = 1.9–27.5° |
| *c* = 17.1872 (6) Å |  = 0.93 mm-1 |
|  = 103.500 (3)° | *T* = 100 K |
| *V* = 4799.7 (2) Å3 | Cut Block, opaque dark blue |
| *Z* = 4 | 0.10 × 0.09 × 0.02 mm |

*Data collection*

|  |  |
| --- | --- |
| Rigaku FRE+ equipped with VHF Varimax confocal mirrors and an AFC10 goniometer and HG Saturn 724+ detector  diffractometer | 11002 independent reflections |
| Radiation source: Rotating Anode, Rigaku FRE+ | 8780 reflections with *I* > 2(*I*) |
| Confocal mirrors, VHF Varimax monochromator | *R*int = 0.053 |
| Detector resolution: 28.5714 pixels mm-1 | max = 27.5°, min = 2.4° |
|  scans | *h* = -1615 |
| Absorption correction: multi-scan  *CrysAlis PRO* 1.171.38.43 (Rigaku Oxford Diffraction, 2015) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. | *k* = -2829 |
| *T*min = 0.538, *T*max = 1.000 | *l* = -2220 |
| 59216 measured reflections |  |

*Refinement*

|  |  |
| --- | --- |
| Refinement on *F*2 | Primary atom site location: dual |
| Least-squares matrix: full | Hydrogen site location: mixed |
| *R*[*F*2 > 2(*F*2)] = 0.043 | H atoms treated by a mixture of independent and constrained refinement |
| *wR*(*F*2) = 0.097 | *w* = 1/[2(*F*o2) + (0.037*P*)2 + 6.005*P*]  where *P* = (*F*o2 + 2*F*c2)/3 |
| *S* = 1.06 | (/)max = 0.001 |
| 11002 reflections | max = 0.50 e Å-3 |
| 670 parameters | min = -0.39 e Å-3 |
| 3 restraints |  |

*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*. A region containing solvent was modelled as 0.75 H2O with hydrogens located in difference Fourier map, 0.25 MeOH and 0.25 H2O and a further separate 0.25 H2O all of which were modelled without H-atoms. The H were included in the unit cell contents and all values dervied from them. One of the NO3- anions was modelled with the oxygen atoms over 2 partially occupied sites with the occupancy refining to 0.9/0.1. |

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2)*

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | *x* | *y* | *z* | *U*iso\*/*U*eq | Occ. (<1) |
| Co1 | 0.54995 (3) | 0.70078 (2) | 0.50482 (2) | 0.01185 (8) |  |
| Co2 | 0.37780 (3) | 0.76246 (2) | 0.60399 (2) | 0.01115 (8) |  |
| S1 | 0.41693 (5) | 0.76699 (2) | 0.48192 (4) | 0.01213 (12) |  |
| S2 | 0.67458 (5) | 0.77413 (2) | 0.53444 (4) | 0.01385 (13) |  |
| S3 | 0.51514 (5) | 0.69616 (2) | 0.62584 (4) | 0.01206 (12) |  |
| S4 | 0.43999 (5) | 0.62172 (3) | 0.46839 (4) | 0.01580 (13) |  |
| N1 | 0.67428 (16) | 0.64808 (8) | 0.52709 (13) | 0.0141 (4) |  |
| N2 | 0.57015 (16) | 0.69755 (8) | 0.39575 (13) | 0.0154 (4) |  |
| N3 | 0.26599 (16) | 0.70239 (8) | 0.56724 (12) | 0.0125 (4) |  |
| N4 | 0.34346 (16) | 0.74519 (8) | 0.70875 (13) | 0.0132 (4) |  |
| N5 | 0.48038 (16) | 0.82532 (8) | 0.64835 (12) | 0.0121 (4) |  |
| N6 | 0.27371 (16) | 0.82901 (8) | 0.57922 (13) | 0.0138 (4) |  |
| C1 | 0.7278 (2) | 0.62953 (10) | 0.59915 (16) | 0.0175 (5) |  |
| H1 | 0.705975 | 0.643295 | 0.643916 | 0.021\* |  |
| C2 | 0.8147 (2) | 0.59054 (11) | 0.61024 (17) | 0.0199 (6) |  |
| C3 | 0.8445 (2) | 0.57065 (11) | 0.54205 (17) | 0.0209 (6) |  |
| H3 | 0.899972 | 0.543064 | 0.546652 | 0.025\* |  |
| C4 | 0.7925 (2) | 0.59136 (10) | 0.46759 (17) | 0.0178 (5) |  |
| H4 | 0.814743 | 0.579197 | 0.422219 | 0.021\* |  |
| C5 | 0.7065 (2) | 0.63069 (10) | 0.46111 (16) | 0.0151 (5) |  |
| C6 | 0.64727 (19) | 0.65830 (10) | 0.38622 (16) | 0.0151 (5) |  |
| C7 | 0.6659 (2) | 0.64753 (11) | 0.31154 (17) | 0.0190 (5) |  |
| H7 | 0.718005 | 0.620111 | 0.305352 | 0.023\* |  |
| C8 | 0.6066 (2) | 0.67784 (11) | 0.24596 (17) | 0.0206 (6) |  |
| H8 | 0.618845 | 0.670726 | 0.195490 | 0.025\* |  |
| C9 | 0.5287 (2) | 0.71896 (11) | 0.25514 (16) | 0.0182 (5) |  |
| C10 | 0.5134 (2) | 0.72674 (10) | 0.33182 (16) | 0.0165 (5) |  |
| H10 | 0.461008 | 0.753587 | 0.339224 | 0.020\* |  |
| C11 | 0.5981 (2) | 0.83620 (10) | 0.49671 (15) | 0.0147 (5) |  |
| C12 | 0.6492 (2) | 0.88993 (11) | 0.49032 (17) | 0.0198 (6) |  |
| H12 | 0.724804 | 0.892852 | 0.505596 | 0.024\* |  |
| C13 | 0.5872 (2) | 0.93861 (11) | 0.46131 (17) | 0.0239 (6) |  |
| H13 | 0.621806 | 0.974257 | 0.457975 | 0.029\* |  |
| C14 | 0.4745 (2) | 0.93533 (11) | 0.43706 (17) | 0.0219 (6) |  |
| H14 | 0.433882 | 0.968416 | 0.417207 | 0.026\* |  |
| C15 | 0.4228 (2) | 0.88253 (10) | 0.44262 (16) | 0.0170 (5) |  |
| H15 | 0.347312 | 0.879674 | 0.425812 | 0.020\* |  |
| C16 | 0.4846 (2) | 0.83374 (10) | 0.47351 (15) | 0.0141 (5) |  |
| C17 | 0.4308 (2) | 0.59203 (10) | 0.56019 (16) | 0.0172 (5) |  |
| C18 | 0.3898 (2) | 0.53558 (11) | 0.56629 (18) | 0.0255 (6) |  |
| H18 | 0.369445 | 0.512155 | 0.520950 | 0.031\* |  |
| C19 | 0.3796 (3) | 0.51461 (12) | 0.63966 (19) | 0.0313 (7) |  |
| H19 | 0.351272 | 0.477211 | 0.642642 | 0.038\* |  |
| C20 | 0.4103 (2) | 0.54764 (12) | 0.70874 (18) | 0.0254 (6) |  |
| H20 | 0.402000 | 0.532799 | 0.757371 | 0.030\* |  |
| C21 | 0.4536 (2) | 0.60310 (11) | 0.70463 (17) | 0.0185 (5) |  |
| H21 | 0.476832 | 0.625435 | 0.750757 | 0.022\* |  |
| C22 | 0.46196 (19) | 0.62514 (10) | 0.63040 (16) | 0.0151 (5) |  |
| C23 | 0.58775 (19) | 0.82023 (10) | 0.68042 (15) | 0.0138 (5) |  |
| H23 | 0.617659 | 0.782800 | 0.688807 | 0.017\* |  |
| C24 | 0.6565 (2) | 0.86836 (10) | 0.70174 (15) | 0.0149 (5) |  |
| C25 | 0.6096 (2) | 0.92350 (11) | 0.69214 (16) | 0.0174 (5) |  |
| H25 | 0.653222 | 0.956740 | 0.704031 | 0.021\* |  |
| C26 | 0.4975 (2) | 0.92923 (10) | 0.66477 (16) | 0.0157 (5) |  |
| H26 | 0.464910 | 0.966047 | 0.661645 | 0.019\* |  |
| C27 | 0.43487 (19) | 0.87975 (10) | 0.64225 (15) | 0.0139 (5) |  |
| C28 | 0.3173 (2) | 0.88103 (10) | 0.60833 (15) | 0.0147 (5) |  |
| C29 | 0.2533 (2) | 0.93085 (11) | 0.60350 (17) | 0.0199 (6) |  |
| H29 | 0.282924 | 0.965850 | 0.626709 | 0.024\* |  |
| C30 | 0.1446 (2) | 0.92771 (12) | 0.56356 (19) | 0.0274 (7) |  |
| H30 | 0.100549 | 0.960791 | 0.559966 | 0.033\* |  |
| C31 | 0.1013 (2) | 0.87555 (12) | 0.52896 (19) | 0.0255 (6) |  |
| C32 | 0.1690 (2) | 0.82697 (11) | 0.53962 (17) | 0.0206 (6) |  |
| H32 | 0.140404 | 0.791273 | 0.518266 | 0.025\* |  |
| C33 | 0.38776 (19) | 0.77026 (10) | 0.77916 (15) | 0.0145 (5) |  |
| H33 | 0.436908 | 0.800879 | 0.780352 | 0.017\* |  |
| C34 | 0.3639 (2) | 0.75276 (11) | 0.85075 (16) | 0.0168 (5) |  |
| C35 | 0.2887 (2) | 0.70755 (11) | 0.84741 (16) | 0.0182 (5) |  |
| H35 | 0.269152 | 0.695296 | 0.893741 | 0.022\* |  |
| C36 | 0.2433 (2) | 0.68089 (11) | 0.77507 (16) | 0.0172 (5) |  |
| H36 | 0.194057 | 0.650170 | 0.772605 | 0.021\* |  |
| C37 | 0.27176 (19) | 0.70036 (10) | 0.70607 (15) | 0.0135 (5) |  |
| C38 | 0.23024 (19) | 0.67529 (10) | 0.62701 (15) | 0.0135 (5) |  |
| C39 | 0.1608 (2) | 0.62748 (10) | 0.61195 (16) | 0.0169 (5) |  |
| H39 | 0.137227 | 0.609361 | 0.653389 | 0.020\* |  |
| C40 | 0.1270 (2) | 0.60705 (10) | 0.53431 (16) | 0.0181 (5) |  |
| H40 | 0.081923 | 0.574281 | 0.523625 | 0.022\* |  |
| C41 | 0.1598 (2) | 0.63514 (10) | 0.47245 (16) | 0.0164 (5) |  |
| C42 | 0.22858 (19) | 0.68338 (10) | 0.49189 (15) | 0.0144 (5) |  |
| H42 | 0.249636 | 0.703366 | 0.450773 | 0.017\* |  |
| C43 | 0.8719 (3) | 0.57173 (14) | 0.69321 (19) | 0.0339 (7) |  |
| H43A | 0.948297 | 0.580435 | 0.702127 | 0.051\* |  |
| H43B | 0.862211 | 0.530295 | 0.698904 | 0.051\* |  |
| H43C | 0.841619 | 0.592439 | 0.731632 | 0.051\* |  |
| C44 | 0.4642 (2) | 0.75424 (13) | 0.18627 (17) | 0.0247 (6) |  |
| H44A | 0.435897 | 0.728670 | 0.141884 | 0.037\* |  |
| H44B | 0.510683 | 0.783154 | 0.170736 | 0.037\* |  |
| H44C | 0.404823 | 0.773434 | 0.202184 | 0.037\* |  |
| C45 | 0.7764 (2) | 0.85857 (11) | 0.73320 (18) | 0.0225 (6) |  |
| H45A | 0.807587 | 0.891674 | 0.764953 | 0.034\* |  |
| H45B | 0.810648 | 0.853973 | 0.689215 | 0.034\* |  |
| H45C | 0.787678 | 0.823790 | 0.765594 | 0.034\* |  |
| C46 | -0.0149 (2) | 0.87065 (13) | 0.4802 (2) | 0.0421 (9) |  |
| H46A | -0.014920 | 0.855976 | 0.427847 | 0.063\* |  |
| H46B | -0.048752 | 0.908628 | 0.475571 | 0.063\* |  |
| H46C | -0.054846 | 0.844216 | 0.506254 | 0.063\* |  |
| C47 | 0.4207 (2) | 0.78189 (12) | 0.92722 (16) | 0.0212 (6) |  |
| H47A | 0.421650 | 0.823570 | 0.919294 | 0.032\* |  |
| H47B | 0.494271 | 0.767607 | 0.943237 | 0.032\* |  |
| H47C | 0.382706 | 0.773179 | 0.968165 | 0.032\* |  |
| C48 | 0.1282 (2) | 0.61436 (12) | 0.38797 (17) | 0.0231 (6) |  |
| H48A | 0.059866 | 0.593877 | 0.379190 | 0.035\* |  |
| H48B | 0.183412 | 0.588405 | 0.377795 | 0.035\* |  |
| H48C | 0.120978 | 0.647430 | 0.352515 | 0.035\* |  |
| O5 | 0.7153 (16) | 0.6497 (9) | 0.7930 (12) | 0.028 (6)\* | 0.101 (5) |
| O6 | 0.7516 (2) | 0.69832 (13) | 0.78350 (15) | 0.0418 (8) | 0.899 (5) |
| O7 | 0.712 (3) | 0.7421 (16) | 0.807 (2) | 0.075 (11)\* | 0.101 (5) |
| O8 | 0.6385 (2) | 0.74196 (12) | 0.83989 (16) | 0.0402 (8) | 0.899 (5) |
| O9 | 0.611 (2) | 0.7023 (12) | 0.8585 (16) | 0.047 (7)\* | 0.101 (5) |
| O10 | 0.6569 (2) | 0.64822 (11) | 0.85058 (17) | 0.0471 (9) | 0.899 (5) |
| O1W | 0.6996 (3) | 0.53314 (16) | 0.8143 (3) | 0.0542 (10) | 0.75 |
| H1WA | 0.662 (3) | 0.523 (2) | 0.7691 (17) | 0.036 (15)\* | 0.75 |
| H1WB | 0.683 (5) | 0.5676 (13) | 0.822 (4) | 0.08 (2)\* | 0.75 |
| N7 | 0.68209 (19) | 0.69516 (11) | 0.82444 (15) | 0.0258 (5) |  |
| O1 | 0.35197 (17) | 0.93560 (8) | 0.78680 (13) | 0.0306 (5) |  |
| O2 | 0.36252 (16) | 1.02680 (8) | 0.75128 (12) | 0.0264 (4) |  |
| O3 | 0.43001 (15) | 0.99950 (8) | 0.87446 (12) | 0.0244 (4) |  |
| N8 | 0.38117 (17) | 0.98773 (10) | 0.80437 (14) | 0.0207 (5) |  |
| O1M | 0.1509 (8) | 0.9946 (5) | 0.3668 (6) | 0.051 (3)\* | 0.25 |
| C1M | 0.0885 (11) | 1.0326 (6) | 0.4003 (9) | 0.040 (3)\* | 0.25 |
| O3W | 0.5864 (15) | 0.5406 (8) | 0.8878 (11) | 0.119 (6)\* | 0.25 |
| O2W | 0.6435 (9) | 0.4947 (4) | 0.6541 (6) | 0.052 (3) | 0.25 |

*Atomic displacement parameters (Å2)*

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | *U*11 | *U*22 | *U*33 | *U*12 | *U*13 | *U*23 |
| Co1 | 0.01222 (16) | 0.00785 (15) | 0.01577 (19) | 0.00024 (11) | 0.00386 (13) | 0.00025 (12) |
| Co2 | 0.01133 (16) | 0.00735 (14) | 0.01484 (18) | -0.00088 (11) | 0.00320 (13) | 0.00023 (12) |
| S1 | 0.0124 (3) | 0.0084 (3) | 0.0158 (3) | -0.0004 (2) | 0.0038 (2) | 0.0012 (2) |
| S2 | 0.0126 (3) | 0.0105 (3) | 0.0186 (3) | -0.0008 (2) | 0.0039 (2) | 0.0000 (2) |
| S3 | 0.0128 (3) | 0.0074 (3) | 0.0159 (3) | -0.0003 (2) | 0.0033 (2) | 0.0005 (2) |
| S4 | 0.0180 (3) | 0.0109 (3) | 0.0185 (3) | -0.0027 (2) | 0.0042 (3) | -0.0016 (2) |
| N1 | 0.0144 (10) | 0.0085 (9) | 0.0198 (12) | -0.0007 (7) | 0.0050 (9) | -0.0003 (8) |
| N2 | 0.0150 (11) | 0.0114 (10) | 0.0196 (12) | -0.0032 (8) | 0.0037 (9) | -0.0007 (8) |
| N3 | 0.0114 (10) | 0.0106 (9) | 0.0153 (11) | 0.0009 (7) | 0.0026 (8) | 0.0017 (8) |
| N4 | 0.0126 (10) | 0.0098 (9) | 0.0175 (12) | 0.0009 (7) | 0.0043 (8) | 0.0007 (8) |
| N5 | 0.0138 (10) | 0.0083 (9) | 0.0144 (11) | -0.0009 (7) | 0.0040 (8) | -0.0006 (8) |
| N6 | 0.0137 (10) | 0.0116 (9) | 0.0162 (12) | -0.0003 (8) | 0.0037 (8) | 0.0014 (8) |
| C1 | 0.0183 (13) | 0.0141 (11) | 0.0209 (15) | 0.0016 (9) | 0.0063 (11) | 0.0032 (10) |
| C2 | 0.0180 (13) | 0.0165 (12) | 0.0251 (15) | 0.0022 (10) | 0.0047 (11) | 0.0056 (11) |
| C3 | 0.0172 (13) | 0.0129 (12) | 0.0344 (17) | 0.0029 (9) | 0.0096 (12) | 0.0018 (11) |
| C4 | 0.0182 (13) | 0.0117 (11) | 0.0249 (15) | 0.0003 (9) | 0.0080 (11) | -0.0030 (10) |
| C5 | 0.0168 (12) | 0.0080 (10) | 0.0210 (14) | -0.0039 (9) | 0.0054 (10) | -0.0024 (9) |
| C6 | 0.0140 (12) | 0.0107 (11) | 0.0214 (14) | -0.0029 (9) | 0.0057 (10) | -0.0010 (10) |
| C7 | 0.0188 (13) | 0.0139 (12) | 0.0257 (16) | -0.0015 (9) | 0.0083 (11) | -0.0037 (10) |
| C8 | 0.0223 (14) | 0.0218 (13) | 0.0192 (15) | -0.0048 (10) | 0.0081 (11) | -0.0027 (11) |
| C9 | 0.0157 (13) | 0.0188 (12) | 0.0202 (15) | -0.0062 (10) | 0.0041 (11) | 0.0007 (10) |
| C10 | 0.0123 (12) | 0.0145 (11) | 0.0228 (15) | -0.0023 (9) | 0.0044 (10) | 0.0034 (10) |
| C11 | 0.0173 (13) | 0.0121 (11) | 0.0158 (14) | -0.0005 (9) | 0.0061 (10) | 0.0006 (9) |
| C12 | 0.0173 (13) | 0.0164 (12) | 0.0260 (16) | -0.0046 (10) | 0.0056 (11) | 0.0001 (11) |
| C13 | 0.0287 (15) | 0.0121 (12) | 0.0307 (17) | -0.0066 (10) | 0.0066 (13) | 0.0029 (11) |
| C14 | 0.0266 (15) | 0.0111 (12) | 0.0281 (16) | 0.0012 (10) | 0.0066 (12) | 0.0058 (11) |
| C15 | 0.0152 (12) | 0.0138 (12) | 0.0220 (15) | -0.0005 (9) | 0.0042 (10) | 0.0010 (10) |
| C16 | 0.0166 (12) | 0.0105 (11) | 0.0172 (14) | -0.0030 (9) | 0.0078 (10) | 0.0004 (9) |
| C17 | 0.0165 (13) | 0.0111 (11) | 0.0241 (15) | -0.0003 (9) | 0.0048 (11) | 0.0001 (10) |
| C18 | 0.0351 (16) | 0.0128 (12) | 0.0267 (17) | -0.0078 (11) | 0.0032 (13) | -0.0035 (11) |
| C19 | 0.0420 (18) | 0.0129 (13) | 0.0356 (19) | -0.0099 (12) | 0.0023 (14) | 0.0052 (12) |
| C20 | 0.0306 (16) | 0.0194 (13) | 0.0251 (17) | -0.0047 (11) | 0.0044 (12) | 0.0092 (11) |
| C21 | 0.0185 (13) | 0.0150 (12) | 0.0216 (15) | -0.0002 (9) | 0.0037 (11) | 0.0026 (10) |
| C22 | 0.0138 (12) | 0.0066 (10) | 0.0247 (15) | 0.0007 (8) | 0.0039 (10) | 0.0026 (9) |
| C23 | 0.0133 (12) | 0.0134 (11) | 0.0146 (13) | 0.0014 (9) | 0.0032 (10) | -0.0002 (9) |
| C24 | 0.0142 (12) | 0.0139 (11) | 0.0170 (14) | -0.0008 (9) | 0.0044 (10) | -0.0027 (9) |
| C25 | 0.0191 (13) | 0.0128 (11) | 0.0206 (14) | -0.0039 (9) | 0.0053 (11) | -0.0039 (10) |
| C26 | 0.0163 (12) | 0.0096 (11) | 0.0213 (14) | 0.0003 (9) | 0.0047 (10) | 0.0005 (10) |
| C27 | 0.0151 (12) | 0.0112 (11) | 0.0162 (13) | 0.0009 (9) | 0.0051 (10) | 0.0005 (9) |
| C28 | 0.0153 (12) | 0.0113 (11) | 0.0179 (14) | -0.0019 (9) | 0.0049 (10) | 0.0014 (9) |
| C29 | 0.0207 (14) | 0.0114 (11) | 0.0260 (16) | 0.0003 (9) | 0.0021 (11) | 0.0003 (10) |
| C30 | 0.0219 (15) | 0.0141 (12) | 0.0422 (19) | 0.0042 (10) | -0.0008 (13) | 0.0017 (12) |
| C31 | 0.0172 (14) | 0.0188 (13) | 0.0353 (18) | 0.0022 (10) | -0.0044 (12) | 0.0003 (12) |
| C32 | 0.0168 (13) | 0.0135 (12) | 0.0288 (16) | -0.0010 (9) | -0.0004 (11) | -0.0006 (11) |
| C33 | 0.0124 (12) | 0.0137 (11) | 0.0170 (14) | 0.0013 (9) | 0.0024 (10) | -0.0007 (9) |
| C34 | 0.0159 (12) | 0.0163 (12) | 0.0178 (14) | 0.0050 (9) | 0.0029 (10) | 0.0004 (10) |
| C35 | 0.0171 (13) | 0.0208 (13) | 0.0176 (14) | 0.0042 (10) | 0.0060 (10) | 0.0052 (10) |
| C36 | 0.0141 (12) | 0.0159 (12) | 0.0218 (15) | -0.0009 (9) | 0.0044 (10) | 0.0041 (10) |
| C37 | 0.0125 (12) | 0.0104 (11) | 0.0180 (14) | 0.0026 (9) | 0.0044 (10) | 0.0006 (9) |
| C38 | 0.0119 (12) | 0.0122 (11) | 0.0163 (13) | 0.0007 (9) | 0.0031 (10) | 0.0001 (9) |
| C39 | 0.0169 (13) | 0.0139 (12) | 0.0212 (15) | -0.0015 (9) | 0.0073 (11) | 0.0025 (10) |
| C40 | 0.0150 (12) | 0.0126 (11) | 0.0260 (15) | -0.0041 (9) | 0.0034 (11) | -0.0021 (10) |
| C41 | 0.0149 (12) | 0.0137 (11) | 0.0203 (14) | -0.0015 (9) | 0.0035 (10) | -0.0024 (10) |
| C42 | 0.0138 (12) | 0.0124 (11) | 0.0173 (14) | 0.0001 (9) | 0.0041 (10) | 0.0009 (9) |
| C43 | 0.0308 (17) | 0.0374 (17) | 0.0339 (19) | 0.0183 (13) | 0.0084 (14) | 0.0117 (14) |
| C44 | 0.0234 (15) | 0.0289 (15) | 0.0209 (16) | -0.0001 (11) | 0.0035 (12) | 0.0050 (12) |
| C45 | 0.0172 (13) | 0.0169 (12) | 0.0325 (17) | -0.0028 (10) | 0.0039 (12) | -0.0054 (11) |
| C46 | 0.0244 (16) | 0.0229 (15) | 0.066 (3) | 0.0031 (12) | -0.0152 (16) | -0.0028 (15) |
| C47 | 0.0239 (14) | 0.0231 (13) | 0.0174 (14) | 0.0023 (10) | 0.0060 (11) | -0.0003 (11) |
| C48 | 0.0237 (14) | 0.0223 (13) | 0.0230 (16) | -0.0083 (11) | 0.0049 (12) | -0.0059 (11) |
| O6 | 0.0293 (14) | 0.070 (2) | 0.0274 (15) | 0.0030 (12) | 0.0101 (11) | -0.0012 (13) |
| O8 | 0.0452 (17) | 0.0358 (15) | 0.0332 (16) | 0.0181 (12) | -0.0035 (12) | -0.0092 (12) |
| O10 | 0.0465 (17) | 0.0364 (15) | 0.0481 (19) | -0.0161 (12) | -0.0098 (13) | 0.0154 (13) |
| O1W | 0.048 (2) | 0.035 (2) | 0.084 (3) | 0.0054 (17) | 0.024 (2) | -0.004 (2) |
| N7 | 0.0233 (13) | 0.0278 (13) | 0.0220 (14) | 0.0057 (10) | -0.0038 (10) | -0.0054 (10) |
| O1 | 0.0408 (12) | 0.0223 (10) | 0.0328 (13) | -0.0170 (9) | 0.0172 (10) | -0.0099 (9) |
| O2 | 0.0304 (11) | 0.0211 (10) | 0.0286 (12) | 0.0009 (8) | 0.0083 (9) | -0.0011 (8) |
| O3 | 0.0231 (10) | 0.0223 (10) | 0.0267 (12) | -0.0026 (8) | 0.0034 (8) | -0.0075 (8) |
| N8 | 0.0173 (11) | 0.0199 (11) | 0.0273 (14) | -0.0032 (9) | 0.0099 (10) | -0.0059 (10) |
| O2W | 0.078 (8) | 0.032 (5) | 0.049 (7) | -0.009 (5) | 0.020 (6) | -0.018 (5) |

*Geometric parameters (Å, º) for (2016ncs0400)*

|  |  |  |  |
| --- | --- | --- | --- |
| Co1—S1 | 2.2212 (6) | C23—H23 | 0.9300 |
| Co1—S2 | 2.2688 (6) | C23—C24 | 1.393 (3) |
| Co1—S3 | 2.2261 (7) | C24—C25 | 1.382 (3) |
| Co1—S4 | 2.2702 (7) | C24—C45 | 1.497 (3) |
| Co1—N1 | 1.940 (2) | C25—H25 | 0.9300 |
| Co1—N2 | 1.951 (2) | C25—C26 | 1.388 (4) |
| Co2—S1 | 2.2668 (7) | C26—H26 | 0.9300 |
| Co2—S3 | 2.2620 (6) | C26—C27 | 1.379 (3) |
| Co2—N3 | 1.960 (2) | C27—C28 | 1.460 (3) |
| Co2—N4 | 1.987 (2) | C28—C29 | 1.384 (3) |
| Co2—N5 | 1.9608 (19) | C29—H29 | 0.9300 |
| Co2—N6 | 1.985 (2) | C29—C30 | 1.383 (4) |
| S1—C16 | 1.766 (2) | C30—H30 | 0.9300 |
| S2—C11 | 1.749 (2) | C30—C31 | 1.383 (4) |
| S3—C22 | 1.760 (2) | C31—C32 | 1.384 (4) |
| S4—C17 | 1.745 (3) | C31—C46 | 1.512 (4) |
| N1—C1 | 1.333 (3) | C32—H32 | 0.9300 |
| N1—C5 | 1.350 (3) | C33—H33 | 0.9300 |
| N2—C6 | 1.359 (3) | C33—C34 | 1.392 (4) |
| N2—C10 | 1.340 (3) | C34—C35 | 1.391 (4) |
| N3—C38 | 1.362 (3) | C34—C47 | 1.497 (4) |
| N3—C42 | 1.342 (3) | C35—H35 | 0.9300 |
| N4—C33 | 1.337 (3) | C35—C36 | 1.382 (4) |
| N4—C37 | 1.358 (3) | C36—H36 | 0.9300 |
| N5—C23 | 1.342 (3) | C36—C37 | 1.389 (3) |
| N5—C27 | 1.360 (3) | C37—C38 | 1.454 (3) |
| N6—C28 | 1.352 (3) | C38—C39 | 1.383 (3) |
| N6—C32 | 1.337 (3) | C39—H39 | 0.9300 |
| C1—H1 | 0.9300 | C39—C40 | 1.383 (4) |
| C1—C2 | 1.389 (3) | C40—H40 | 0.9300 |
| C2—C3 | 1.387 (4) | C40—C41 | 1.384 (4) |
| C2—C43 | 1.502 (4) | C41—C42 | 1.392 (3) |
| C3—H3 | 0.9300 | C41—C48 | 1.490 (4) |
| C3—C4 | 1.378 (4) | C42—H42 | 0.9300 |
| C4—H4 | 0.9300 | C43—H43A | 0.9600 |
| C4—C5 | 1.391 (3) | C43—H43B | 0.9600 |
| C5—C6 | 1.470 (4) | C43—H43C | 0.9600 |
| C6—C7 | 1.380 (4) | C44—H44A | 0.9600 |
| C7—H7 | 0.9300 | C44—H44B | 0.9600 |
| C7—C8 | 1.383 (4) | C44—H44C | 0.9600 |
| C8—H8 | 0.9300 | C45—H45A | 0.9600 |
| C8—C9 | 1.393 (4) | C45—H45B | 0.9600 |
| C9—C10 | 1.387 (4) | C45—H45C | 0.9600 |
| C9—C44 | 1.503 (4) | C46—H46A | 0.9600 |
| C10—H10 | 0.9300 | C46—H46B | 0.9600 |
| C11—C12 | 1.400 (3) | C46—H46C | 0.9600 |
| C11—C16 | 1.394 (3) | C47—H47A | 0.9600 |
| C12—H12 | 0.9300 | C47—H47B | 0.9600 |
| C12—C13 | 1.381 (4) | C47—H47C | 0.9600 |
| C13—H13 | 0.9300 | C48—H48A | 0.9600 |
| C13—C14 | 1.387 (4) | C48—H48B | 0.9600 |
| C14—H14 | 0.9300 | C48—H48C | 0.9600 |
| C14—C15 | 1.382 (3) | O5—N7 | 1.28 (2) |
| C15—H15 | 0.9300 | O6—N7 | 1.247 (3) |
| C15—C16 | 1.390 (3) | O7—N7 | 1.19 (4) |
| C17—C18 | 1.399 (3) | O8—N7 | 1.256 (3) |
| C17—C22 | 1.399 (4) | O9—N7 | 1.19 (3) |
| C18—H18 | 0.9300 | O10—N7 | 1.230 (3) |
| C18—C19 | 1.382 (4) | O1W—H1WA | 0.839 (19) |
| C19—H19 | 0.9300 | O1W—H1WB | 0.83 (2) |
| C19—C20 | 1.383 (4) | O1—N8 | 1.259 (3) |
| C20—H20 | 0.9300 | O2—N8 | 1.257 (3) |
| C20—C21 | 1.385 (3) | O3—N8 | 1.247 (3) |
| C21—H21 | 0.9300 | O1M—C1M | 1.384 (13) |
| C21—C22 | 1.398 (4) |  |  |
|  |  |  |  |
| S1—Co1—S2 | 89.63 (2) | C19—C20—C21 | 119.2 (3) |
| S1—Co1—S3 | 83.83 (2) | C21—C20—H20 | 120.4 |
| S1—Co1—S4 | 95.94 (2) | C20—C21—H21 | 120.4 |
| S2—Co1—S4 | 173.08 (3) | C20—C21—C22 | 119.3 (3) |
| S3—Co1—S2 | 95.87 (3) | C22—C21—H21 | 120.4 |
| S3—Co1—S4 | 88.82 (2) | C17—C22—S3 | 119.36 (19) |
| N1—Co1—S1 | 175.44 (6) | C21—C22—S3 | 118.8 (2) |
| N1—Co1—S2 | 85.81 (6) | C21—C22—C17 | 121.8 (2) |
| N1—Co1—S3 | 96.47 (7) | N5—C23—H23 | 118.5 |
| N1—Co1—S4 | 88.62 (6) | N5—C23—C24 | 123.1 (2) |
| N1—Co1—N2 | 83.43 (9) | C24—C23—H23 | 118.5 |
| N2—Co1—S1 | 96.76 (6) | C23—C24—C45 | 119.4 (2) |
| N2—Co1—S2 | 90.27 (6) | C25—C24—C23 | 117.5 (2) |
| N2—Co1—S3 | 173.84 (6) | C25—C24—C45 | 123.1 (2) |
| N2—Co1—S4 | 85.02 (6) | C24—C25—H25 | 120.0 |
| S3—Co2—S1 | 81.99 (2) | C24—C25—C26 | 120.0 (2) |
| N3—Co2—S1 | 91.66 (6) | C26—C25—H25 | 120.0 |
| N3—Co2—S3 | 92.96 (6) | C25—C26—H26 | 120.4 |
| N3—Co2—N4 | 82.03 (8) | C27—C26—C25 | 119.3 (2) |
| N3—Co2—N5 | 174.02 (8) | C27—C26—H26 | 120.4 |
| N3—Co2—N6 | 94.31 (8) | N5—C27—C26 | 121.4 (2) |
| N4—Co2—S1 | 171.18 (6) | N5—C27—C28 | 114.8 (2) |
| N4—Co2—S3 | 92.13 (6) | C26—C27—C28 | 123.8 (2) |
| N5—Co2—S1 | 93.47 (6) | N6—C28—C27 | 114.7 (2) |
| N5—Co2—S3 | 90.80 (6) | N6—C28—C29 | 121.1 (2) |
| N5—Co2—N4 | 93.19 (8) | C29—C28—C27 | 124.3 (2) |
| N5—Co2—N6 | 82.41 (8) | C28—C29—H29 | 120.6 |
| N6—Co2—S1 | 92.43 (6) | C30—C29—C28 | 118.9 (2) |
| N6—Co2—S3 | 170.96 (6) | C30—C29—H29 | 120.6 |
| N6—Co2—N4 | 94.18 (8) | C29—C30—H30 | 119.9 |
| Co1—S1—Co2 | 97.08 (3) | C31—C30—C29 | 120.2 (2) |
| C16—S1—Co1 | 103.94 (8) | C31—C30—H30 | 119.9 |
| C16—S1—Co2 | 109.18 (9) | C30—C31—C32 | 117.5 (2) |
| C11—S2—Co1 | 102.71 (8) | C30—C31—C46 | 122.4 (2) |
| Co1—S3—Co2 | 97.08 (3) | C32—C31—C46 | 120.1 (2) |
| C22—S3—Co1 | 104.19 (9) | N6—C32—C31 | 123.0 (2) |
| C22—S3—Co2 | 109.70 (8) | N6—C32—H32 | 118.5 |
| C17—S4—Co1 | 102.90 (9) | C31—C32—H32 | 118.5 |
| C1—N1—Co1 | 126.13 (17) | N4—C33—H33 | 118.4 |
| C1—N1—C5 | 120.1 (2) | N4—C33—C34 | 123.2 (2) |
| C5—N1—Co1 | 113.81 (17) | C34—C33—H33 | 118.4 |
| C6—N2—Co1 | 113.47 (17) | C33—C34—C47 | 119.4 (2) |
| C10—N2—Co1 | 127.20 (18) | C35—C34—C33 | 117.5 (2) |
| C10—N2—C6 | 119.2 (2) | C35—C34—C47 | 123.1 (2) |
| C38—N3—Co2 | 114.39 (16) | C34—C35—H35 | 120.1 |
| C42—N3—Co2 | 126.90 (16) | C36—C35—C34 | 119.8 (2) |
| C42—N3—C38 | 118.5 (2) | C36—C35—H35 | 120.1 |
| C33—N4—Co2 | 127.04 (16) | C35—C36—H36 | 120.3 |
| C33—N4—C37 | 119.1 (2) | C35—C36—C37 | 119.5 (2) |
| C37—N4—Co2 | 113.72 (17) | C37—C36—H36 | 120.3 |
| C23—N5—Co2 | 127.35 (16) | N4—C37—C36 | 120.9 (2) |
| C23—N5—C27 | 118.5 (2) | N4—C37—C38 | 114.8 (2) |
| C27—N5—Co2 | 114.10 (16) | C36—C37—C38 | 124.3 (2) |
| C28—N6—Co2 | 113.52 (16) | N3—C38—C37 | 114.6 (2) |
| C32—N6—Co2 | 127.36 (17) | N3—C38—C39 | 121.5 (2) |
| C32—N6—C28 | 119.1 (2) | C39—C38—C37 | 123.9 (2) |
| N1—C1—H1 | 118.6 | C38—C39—H39 | 120.5 |
| N1—C1—C2 | 122.8 (2) | C40—C39—C38 | 119.0 (2) |
| C2—C1—H1 | 118.6 | C40—C39—H39 | 120.5 |
| C1—C2—C43 | 120.1 (3) | C39—C40—H40 | 119.8 |
| C3—C2—C1 | 117.0 (2) | C39—C40—C41 | 120.3 (2) |
| C3—C2—C43 | 123.0 (2) | C41—C40—H40 | 119.8 |
| C2—C3—H3 | 119.7 | C40—C41—C42 | 117.5 (2) |
| C4—C3—C2 | 120.6 (2) | C40—C41—C48 | 122.8 (2) |
| C4—C3—H3 | 119.7 | C42—C41—C48 | 119.6 (2) |
| C3—C4—H4 | 120.4 | N3—C42—C41 | 123.1 (2) |
| C3—C4—C5 | 119.2 (2) | N3—C42—H42 | 118.5 |
| C5—C4—H4 | 120.4 | C41—C42—H42 | 118.5 |
| N1—C5—C4 | 120.2 (2) | C2—C43—H43A | 109.5 |
| N1—C5—C6 | 114.7 (2) | C2—C43—H43B | 109.5 |
| C4—C5—C6 | 125.0 (2) | C2—C43—H43C | 109.5 |
| N2—C6—C5 | 114.1 (2) | H43A—C43—H43B | 109.5 |
| N2—C6—C7 | 120.8 (2) | H43A—C43—H43C | 109.5 |
| C7—C6—C5 | 125.2 (2) | H43B—C43—H43C | 109.5 |
| C6—C7—H7 | 120.3 | C9—C44—H44A | 109.5 |
| C6—C7—C8 | 119.5 (2) | C9—C44—H44B | 109.5 |
| C8—C7—H7 | 120.3 | C9—C44—H44C | 109.5 |
| C7—C8—H8 | 119.8 | H44A—C44—H44B | 109.5 |
| C7—C8—C9 | 120.3 (3) | H44A—C44—H44C | 109.5 |
| C9—C8—H8 | 119.8 | H44B—C44—H44C | 109.5 |
| C8—C9—C44 | 122.5 (3) | C24—C45—H45A | 109.5 |
| C10—C9—C8 | 116.9 (2) | C24—C45—H45B | 109.5 |
| C10—C9—C44 | 120.7 (2) | C24—C45—H45C | 109.5 |
| N2—C10—C9 | 123.3 (2) | H45A—C45—H45B | 109.5 |
| N2—C10—H10 | 118.3 | H45A—C45—H45C | 109.5 |
| C9—C10—H10 | 118.3 | H45B—C45—H45C | 109.5 |
| C12—C11—S2 | 120.86 (19) | C31—C46—H46A | 109.5 |
| C16—C11—S2 | 120.90 (18) | C31—C46—H46B | 109.5 |
| C16—C11—C12 | 118.2 (2) | C31—C46—H46C | 109.5 |
| C11—C12—H12 | 120.0 | H46A—C46—H46B | 109.5 |
| C13—C12—C11 | 120.0 (2) | H46A—C46—H46C | 109.5 |
| C13—C12—H12 | 120.0 | H46B—C46—H46C | 109.5 |
| C12—C13—H13 | 119.4 | C34—C47—H47A | 109.5 |
| C12—C13—C14 | 121.2 (2) | C34—C47—H47B | 109.5 |
| C14—C13—H13 | 119.4 | C34—C47—H47C | 109.5 |
| C13—C14—H14 | 120.3 | H47A—C47—H47B | 109.5 |
| C15—C14—C13 | 119.5 (2) | H47A—C47—H47C | 109.5 |
| C15—C14—H14 | 120.3 | H47B—C47—H47C | 109.5 |
| C14—C15—H15 | 120.2 | C41—C48—H48A | 109.5 |
| C14—C15—C16 | 119.5 (2) | C41—C48—H48B | 109.5 |
| C16—C15—H15 | 120.2 | C41—C48—H48C | 109.5 |
| C11—C16—S1 | 119.61 (18) | H48A—C48—H48B | 109.5 |
| C15—C16—S1 | 118.86 (18) | H48A—C48—H48C | 109.5 |
| C15—C16—C11 | 121.5 (2) | H48B—C48—H48C | 109.5 |
| C18—C17—S4 | 121.8 (2) | H1WA—O1W—H1WB | 107 (6) |
| C18—C17—C22 | 117.7 (2) | O6—N7—O8 | 118.0 (3) |
| C22—C17—S4 | 120.42 (18) | O7—N7—O5 | 118 (2) |
| C17—C18—H18 | 120.0 | O9—N7—O5 | 132.4 (17) |
| C19—C18—C17 | 120.1 (3) | O9—N7—O7 | 108 (2) |
| C19—C18—H18 | 120.0 | O10—N7—O6 | 122.1 (3) |
| C18—C19—H19 | 119.1 | O10—N7—O8 | 119.9 (3) |
| C18—C19—C20 | 121.9 (2) | O2—N8—O1 | 120.0 (2) |
| C20—C19—H19 | 119.1 | O3—N8—O1 | 119.0 (2) |
| C19—C20—H20 | 120.4 | O3—N8—O2 | 121.0 (2) |

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