metal-organic papers

(η^6-*N,N*-Diethylaniline)(η^4-1,2,3,4-tetramethyl-cyclobutadiene)cobalt(I) hexafluorophosphate

Alan R. Kennedy* and Peter L. Pauson

Department of Pure and Applied Chemistry, University of Strathclyde, Glasgow G1 1XL, Scotland

Correspondence e-mail:
a.r.kennedy@strath.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.052
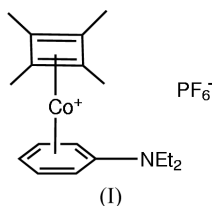wR factor = 0.141
Data-to-parameter ratio = 16.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title isocobaltocenium salt, [Co(C$_8$H$_{12}$)(C$_{10}$H$_{15}$N)]PF$_6$, is shown to exist as discrete monomers with near equivalent C—C bonds in the cyclobutadiene ring.

Comment

Isocobaltocenes are the series of compounds in which, in place of two cyclopentadienyl residues, the cobalt is π-bonded to one cyclobutadiene and one benzene ring. Species with bulky cyclobutadiene ring substituents have been known since the work of Maitlis & Efraty (1965) and have been widely crystallographically characterized. However, despite synthetic protocols being well known (Cook *et al.*, 1987), the only known structures of tetramethylcyclobutadiene-based isocobaltocenes are two species with unusual substituted boratabenzenes (Herberich *et al.*, 2002).

(I)

Reported here is the structure of (I), a tetramethylcyclobutadiene isocobaltocenium salt with a simple *N,N*-diethyl-
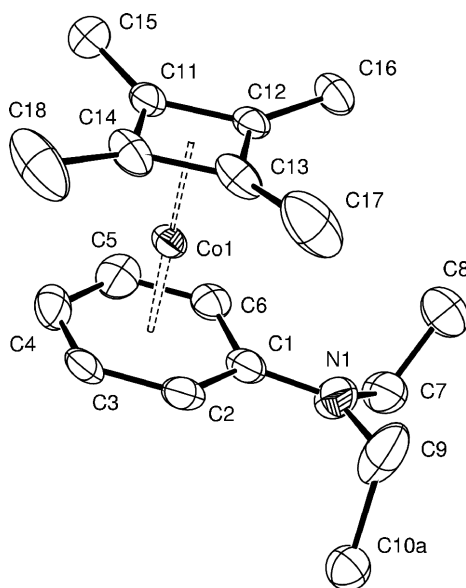

Figure 1
The molecular structure of the cation of (I), shown with 50% probability displacement ellipsoids. H atoms and disordered fragments have been omitted for clarity.

aniline group as its arene component (Fig. 1). The centroid of the cyclobutadiene group lies further from Co1 than the centroid of the benzene ring (1.705 *versus* 1.592 Å) and this distance is also slightly longer than those found for the boratabenzene species (range 1.686–1.702 Å; Herberich *et al.*, 2002). However, it falls into the middle of the range found for nine general Co–tetramethylcyclobutadiene complexes found in a search of the Cambridge Structural Database (Version 5, with updates to December 2004; Allen, 2002). The cyclobutadiene ring C—C distances show only slight signs of alternate single- and double-bond nature (Table 1), indicating a high degree of aromaticity. The Co atom is placed over the centre of the cyclobutadiene ring but is displaced away from the amine-substituted C1 atom of the benzene ring. No strong intermolecular contacts were observed.

Experimental

The title compound was prepared according to the method of Cook *et al.* (1987).

Crystal data

$[Co(C_8H_{12})(C_{10}H_{15}N)]PF_6$	$D_x = 1.519$ Mg m^{-3}
$M_r = 461.31$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 21 381
$a = 8.4201$ (2) Å	reflections
$b = 16.2404$ (3) Å	$\theta = 3.0–27.5°$
$c = 15.1960$ (4) Å	$\mu = 0.99$ mm^{-1}
$\beta = 103.844$ (1)°	$T = 120$ (2) K
$V = 2017.63$ (8) Å^3	Cut block, yellow
$Z = 4$	$0.12 \times 0.10 \times 0.10$ mm

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.078$
φ and ω scans	$\theta_{max} = 27.5°$
21 381 measured reflections	$h = -10 \rightarrow 10$
4587 independent reflections	$k = -21 \rightarrow 21$
3068 reflections with $I > 2\sigma(I)$	$l = -17 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.052$	$+ 0.6904P]$
$wR(F^2) = 0.141$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
4587 reflections	$\Delta\rho_{max} = 0.49$ e Å^{-3}
273 parameters	$\Delta\rho_{min} = -0.78$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Co1—C14	1.973 (3)	Co1—C2	2.109 (3)
Co1—C11	1.987 (3)	Co1—C6	2.126 (3)
Co1—C13	1.992 (3)	Co1—C1	2.241 (3)
Co1—C12	2.004 (3)	C11—C14	1.447 (5)
Co1—C3	2.077 (3)	C11—C12	1.450 (4)
Co1—C5	2.088 (4)	C12—C13	1.442 (4)
Co1—C4	2.101 (3)	C13—C14	1.452 (5)
C14—C11—C12	90.0 (3)	C12—C13—C14	90.1 (3)
C13—C12—C11	90.1 (3)	C11—C14—C13	89.8 (3)

H atoms were included in the riding-model approximation with C—H distances: CH$_3$ 0.98, CH$_2$ 0.99 and CH 0.95 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(C_{methyl})$. The PF$_6^-$ anion is rotationally disordered about the F3—P1—F4 axis. After several trial calculations, the remaining four F atoms were refined over 12 sites with set occupancies. The largest remaining electron-density peaks all occur near to the PF$_6^-$ anion. The C10 methyl group was refined as disordered over two sites with occupancies refined [0.52 (2):0.48 (2)]. The occupancies of atoms F1, F2, F5 and F6 were set at 0.5 and the occupancies of atoms F7–F12 were set at 0.25.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1988); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Cook, M. R., Härter, P., Pauson, P. L. & Šraga, J. (1987). *J. Chem. Soc. Dalton Trans.* pp. 2757–2760.
Herberich, G. E., Basu Baul, T. S. & Englert, U. (2002). *Eur. J. Inorg. Chem.* pp. 43–48.
Hooft, R. (1988). *COLLECT.* Nonius BV, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Maitlis, P. M. & Efraty, A. (1965). *J. Organomet. Chem.* **4**, 175–176.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97.* University of Göttingen, Germany.

supporting information

(η⁶-*N*,*N*-Diethylaniline)(η⁴-1,2,3,4-tetramethylcyclobutadiene)cobalt(I) hexafluorophosphate

Alan R. Kennedy and Peter L. Pauson

(η⁶-*N*,*N*-Diethylaniline)(η⁴-1,2,3,4-tetramethylcyclobutadiene)cobalt(I) hexafluorophosphate

Crystal data

$[\text{Co}(\text{C}_8\text{H}_{12})(\text{C}_{10}\text{H}_{15}\text{N})]\text{PF}_6$	$F(000) = 952$
$M_r = 461.31$	$D_x = 1.519$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.4201$ (2) Å	Cell parameters from 21381 reflections
$b = 16.2404$ (3) Å	$\theta = 3.0$–$27.5°$
$c = 15.1960$ (4) Å	$\mu = 0.99$ mm^{-1}
$\beta = 103.844$ (1)°	$T = 120$ K
$V = 2017.63$ (8) Å^3	Cut block, yellow
$Z = 4$	$0.12 \times 0.10 \times 0.10$ mm

Data collection

Nonius KappaCCD diffractometer	3068 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.078$
Graphite monochromator	$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
φ and ω scans	$h = -10 \rightarrow 10$
21381 measured reflections	$k = -21 \rightarrow 21$
4587 independent reflections	$l = -17 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H-atom parameters constrained
$wR(F^2) = 0.141$	$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2 + 0.6904P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	
4587 reflections	$(\Delta/\sigma)_{max} = 0.001$
273 parameters	$\Delta\rho_{max} = 0.49$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.78$ e Å^{-3}
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}	Occ. (<1)
Co1	0.44977 (5)	0.14989 (2)	0.68037 (3)	0.02448 (16)	
P1	0.07791 (11)	−0.09763 (5)	0.76435 (7)	0.0360 (2)	
F1	0.0804 (8)	−0.1934 (4)	0.7872 (5)	0.065 (2)	0.50
F2	0.0287 (7)	−0.0740 (4)	0.8545 (4)	0.0581 (16)*	0.50
F3	0.2675 (3)	−0.10062 (16)	0.81208 (16)	0.0621 (7)	
F4	−0.1109 (3)	−0.09398 (17)	0.7176 (2)	0.0812 (9)	
F5	0.1390 (9)	−0.1072 (5)	0.6680 (5)	0.057 (2)	0.50
F6	0.0897 (8)	−0.0010 (4)	0.7584 (5)	0.071 (2)*	0.50
F7	0.0865 (7)	−0.0158 (4)	0.8326 (4)	0.0219 (14)*	0.25
F8	0.1053 (8)	−0.0390 (5)	0.6877 (5)	0.0245 (15)*	0.25
F9	0.0087 (12)	−0.1080 (8)	0.8508 (6)	0.044 (2)*	0.25
F10	0.0699 (10)	−0.1755 (6)	0.7049 (7)	0.036 (2)*	0.25
F11	0.0515 (16)	−0.1946 (9)	0.7480 (10)	0.040 (3)*	0.25
F12	0.0552 (10)	−0.1542 (6)	0.8531 (6)	0.0355 (18)*	0.25
F13	0.123 (2)	−0.1303 (11)	0.6776 (13)	0.058 (5)*	0.25
F14	0.0764 (12)	−0.0062 (6)	0.7203 (8)	0.042 (2)*	0.25
N1	0.7040 (4)	0.11021 (18)	0.8825 (2)	0.0429 (8)	
C1	0.5745 (4)	0.14937 (18)	0.8282 (2)	0.0329 (8)	
C2	0.5938 (5)	0.22397 (19)	0.7831 (2)	0.0374 (8)	
H2	0.7007	0.2445	0.7865	0.045*	
C3	0.4575 (5)	0.2681 (2)	0.7334 (2)	0.0412 (9)	
H3	0.4735	0.3195	0.7070	0.049*	
C4	0.2990 (5)	0.2371 (2)	0.7224 (2)	0.0431 (9)	
H4	0.2069	0.2676	0.6904	0.052*	
C5	0.2796 (5)	0.1602 (2)	0.7596 (2)	0.0425 (9)	
H5	0.1730	0.1375	0.7512	0.051*	
C6	0.4145 (4)	0.1157 (2)	0.8094 (2)	0.0322 (7)	
H6	0.3983	0.0622	0.8308	0.039*	
C7	0.6824 (5)	0.0478 (2)	0.9488 (2)	0.0472 (10)	
H7A	0.5771	0.0578	0.9652	0.057*	
H7B	0.7710	0.0540	1.0044	0.057*	
C8	0.6839 (5)	−0.0384 (2)	0.9146 (3)	0.0526 (10)	
H8A	0.5876	−0.0473	0.8646	0.079*	
H8B	0.6819	−0.0771	0.9637	0.079*	
H8C	0.7833	−0.0473	0.8929	0.079*	
C9	0.8713 (5)	0.1362 (3)	0.8877 (3)	0.0701 (15)	
H9A	0.8831	0.1581	0.8289	0.084*	
H9B	0.9489	0.0900	0.9063	0.084*	
C10A	0.9020 (11)	0.2107 (5)	0.9677 (6)	0.040 (3)*	0.518 (17)
H10A	1.0149	0.2306	0.9787	0.060*	0.518 (17)

H10B	0.8823	0.1883	1.0241	0.060*	0.518 (17)
H10C	0.8264	0.2564	0.9468	0.060*	0.518 (17)
C10B	0.9617 (14)	0.1965 (6)	0.9370 (7)	0.052 (3)*	0.482 (17)
H10D	0.9046	0.2492	0.9233	0.078*	0.482 (17)
H10E	1.0682	0.1997	0.9216	0.078*	0.482 (17)
H10F	0.9780	0.1842	1.0017	0.078*	0.482 (17)
C11	0.3432 (4)	0.09746 (18)	0.5630 (2)	0.0323 (8)	
C12	0.4799 (4)	0.04821 (17)	0.61148 (19)	0.0253 (7)	
C13	0.5916 (4)	0.1125 (2)	0.6003 (2)	0.0353 (8)	
C14	0.4550 (5)	0.16211 (19)	0.5519 (2)	0.0359 (8)	
C15	0.1647 (5)	0.0810 (3)	0.5277 (3)	0.0552 (11)	
H15A	0.1036	0.1326	0.5260	0.083*	
H15B	0.1281	0.0417	0.5676	0.083*	
H15C	0.1453	0.0580	0.4665	0.083*	
C16	0.4987 (4)	−0.03750 (19)	0.6468 (2)	0.0348 (8)	
H16A	0.5008	−0.0756	0.5971	0.052*	
H16B	0.4064	−0.0512	0.6731	0.052*	
H16C	0.6011	−0.0422	0.6935	0.052*	
C17	0.7718 (5)	0.1187 (3)	0.6168 (4)	0.0657 (13)	
H17A	0.8231	0.0851	0.6695	0.099*	
H17B	0.8051	0.1763	0.6284	0.099*	
H17C	0.8065	0.0989	0.5634	0.099*	
C18	0.4395 (7)	0.2401 (2)	0.4985 (3)	0.0679 (14)	
H18A	0.4331	0.2272	0.4347	0.102*	
H18B	0.5351	0.2750	0.5222	0.102*	
H18C	0.3401	0.2692	0.5035	0.102*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0316 (3)	0.0195 (2)	0.0230 (2)	0.00180 (17)	0.00793 (18)	−0.00229 (16)
P1	0.0291 (5)	0.0295 (5)	0.0451 (6)	−0.0006 (4)	0.0001 (4)	0.0083 (4)
F1	0.060 (4)	0.038 (3)	0.093 (6)	0.003 (2)	0.007 (4)	0.042 (4)
F3	0.0297 (12)	0.0898 (19)	0.0587 (15)	0.0015 (11)	−0.0057 (11)	0.0010 (13)
F4	0.0309 (13)	0.0776 (19)	0.118 (2)	−0.0047 (12)	−0.0167 (14)	0.0332 (16)
F5	0.063 (4)	0.065 (5)	0.043 (3)	−0.024 (3)	0.012 (2)	0.013 (3)
N1	0.0461 (19)	0.0327 (16)	0.0421 (18)	−0.0054 (14)	−0.0047 (14)	0.0082 (13)
C1	0.048 (2)	0.0233 (16)	0.0258 (17)	0.0008 (14)	0.0063 (16)	−0.0028 (13)
C2	0.050 (2)	0.0251 (17)	0.0321 (19)	−0.0023 (15)	0.0005 (16)	−0.0072 (14)
C3	0.065 (3)	0.0208 (17)	0.0325 (19)	0.0105 (16)	0.0012 (18)	−0.0089 (14)
C4	0.050 (2)	0.046 (2)	0.0326 (19)	0.0235 (18)	0.0097 (17)	−0.0055 (16)
C5	0.042 (2)	0.060 (3)	0.0295 (18)	0.0087 (18)	0.0166 (17)	−0.0041 (17)
C6	0.041 (2)	0.0339 (18)	0.0238 (16)	0.0020 (15)	0.0119 (15)	−0.0035 (14)
C7	0.063 (3)	0.039 (2)	0.034 (2)	0.0015 (18)	0.0016 (19)	0.0028 (16)
C8	0.063 (3)	0.037 (2)	0.061 (3)	0.0005 (19)	0.023 (2)	0.0026 (19)
C9	0.045 (2)	0.058 (3)	0.089 (4)	−0.014 (2)	−0.021 (2)	0.036 (2)
C11	0.049 (2)	0.0237 (16)	0.0220 (16)	0.0082 (14)	0.0035 (15)	−0.0035 (12)
C12	0.0363 (18)	0.0211 (15)	0.0195 (15)	0.0015 (13)	0.0085 (13)	−0.0038 (12)

C13	0.048 (2)	0.0247 (17)	0.041 (2)	0.0015 (15)	0.0252 (17)	−0.0052 (14)
C14	0.060 (2)	0.0248 (17)	0.0295 (18)	0.0075 (15)	0.0231 (17)	0.0033 (13)
C15	0.052 (3)	0.050 (2)	0.052 (2)	0.0150 (19)	−0.012 (2)	−0.0141 (19)
C16	0.050 (2)	0.0230 (16)	0.0336 (18)	0.0058 (15)	0.0149 (16)	0.0013 (14)
C17	0.052 (3)	0.045 (2)	0.114 (4)	−0.005 (2)	0.047 (3)	−0.018 (3)
C18	0.131 (4)	0.033 (2)	0.058 (3)	0.019 (2)	0.059 (3)	0.0158 (19)

Geometric parameters (Å, º)

Co1—C14	1.973 (3)	C7—C8	1.495 (5)
Co1—C11	1.987 (3)	C7—H7A	0.9900
Co1—C13	1.992 (3)	C7—H7B	0.9900
Co1—C12	2.004 (3)	C8—H8A	0.9800
Co1—C3	2.077 (3)	C8—H8B	0.9800
Co1—C5	2.088 (4)	C8—H8C	0.9800
Co1—C4	2.101 (3)	C9—C10B	1.352 (9)
Co1—C2	2.109 (3)	C9—C10A	1.691 (10)
Co1—C6	2.126 (3)	C9—H9A	0.9900
Co1—C1	2.241 (3)	C9—H9B	0.9900
P1—F10	1.546 (8)	C10A—H10A	0.9800
P1—F13	1.548 (19)	C10A—H10B	0.9800
P1—F8	1.564 (7)	C10A—H10C	0.9800
P1—F9	1.568 (9)	C10B—H10D	0.9800
P1—F2	1.571 (6)	C10B—H10E	0.9800
P1—F6	1.576 (6)	C10B—H10F	0.9800
P1—F4	1.579 (2)	C11—C14	1.447 (5)
P1—F3	1.588 (2)	C11—C12	1.450 (4)
P1—F1	1.592 (5)	C11—C15	1.494 (5)
P1—F11	1.602 (14)	C12—C13	1.442 (4)
P1—F14	1.627 (9)	C12—C16	1.487 (4)
P1—F5	1.671 (7)	C13—C14	1.452 (5)
N1—C1	1.358 (4)	C13—C17	1.481 (5)
N1—C9	1.454 (5)	C14—C18	1.493 (4)
N1—C7	1.470 (4)	C15—H15A	0.9800
C1—C6	1.417 (5)	C15—H15B	0.9800
C1—C2	1.420 (4)	C15—H15C	0.9800
C2—C3	1.409 (5)	C16—H16A	0.9800
C2—H2	0.9500	C16—H16B	0.9800
C3—C4	1.399 (5)	C16—H16C	0.9800
C3—H3	0.9500	C17—H17A	0.9800
C4—C5	1.397 (5)	C17—H17B	0.9800
C4—H4	0.9500	C17—H17C	0.9800
C5—C6	1.405 (5)	C18—H18A	0.9800
C5—H5	0.9500	C18—H18B	0.9800
C6—H6	0.9500	C18—H18C	0.9800
C14—Co1—C11	42.86 (14)	C1—C6—H6	119.4
C14—Co1—C13	42.95 (14)	Co1—C6—H6	128.2

C11—Co1—C13	61.91 (15)	N1—C7—C8	113.3 (3)
C14—Co1—C12	61.99 (12)	N1—C7—H7A	108.9
C11—Co1—C12	42.62 (12)	C8—C7—H7A	108.9
C13—Co1—C12	42.29 (13)	N1—C7—H7B	108.9
C14—Co1—C3	106.54 (13)	C8—C7—H7B	108.9
C11—Co1—C3	135.23 (13)	H7A—C7—H7B	107.7
C13—Co1—C3	123.42 (15)	C7—C8—H8A	109.5
C12—Co1—C3	165.54 (14)	C7—C8—H8B	109.5
C14—Co1—C5	138.04 (16)	H8A—C8—H8B	109.5
C11—Co1—C5	109.11 (15)	C7—C8—H8C	109.5
C13—Co1—C5	166.19 (14)	H8A—C8—H8C	109.5
C12—Co1—C5	123.91 (14)	H8B—C8—H8C	109.5
C3—Co1—C5	70.38 (15)	C10B—C9—N1	130.6 (8)
C14—Co1—C4	113.03 (14)	C10B—C9—C10A	27.7 (5)
C11—Co1—C4	112.70 (14)	N1—C9—C10A	103.1 (5)
C13—Co1—C4	152.28 (15)	C10B—C9—H9A	94.3
C12—Co1—C4	151.15 (15)	N1—C9—H9A	111.1
C3—Co1—C4	39.11 (15)	C10A—C9—H9A	111.1
C5—Co1—C4	38.95 (14)	C10B—C9—H9B	98.3
C14—Co1—C2	122.01 (15)	N1—C9—H9B	111.1
C11—Co1—C2	164.86 (14)	C10A—C9—H9B	111.1
C13—Co1—C2	107.67 (15)	H9A—C9—H9B	109.1
C12—Co1—C2	137.26 (13)	C9—C10A—H10A	109.5
C3—Co1—C2	39.33 (13)	C9—C10A—H10B	109.5
C5—Co1—C2	83.08 (15)	C9—C10A—H10C	109.5
C4—Co1—C2	70.89 (15)	C9—C10B—H10D	109.5
C14—Co1—C6	168.49 (14)	C9—C10B—H10E	109.5
C11—Co1—C6	125.70 (14)	H10D—C10B—H10E	109.5
C13—Co1—C6	136.05 (14)	C9—C10B—H10F	109.5
C12—Co1—C6	109.27 (12)	H10D—C10B—H10F	109.5
C3—Co1—C6	83.21 (14)	H10E—C10B—H10F	109.5
C5—Co1—C6	38.94 (13)	C14—C11—C12	90.0 (3)
C4—Co1—C6	70.59 (14)	C14—C11—C15	135.6 (3)
C2—Co1—C6	69.42 (13)	C12—C11—C15	133.8 (3)
C14—Co1—C1	151.21 (15)	C14—C11—Co1	68.08 (18)
C11—Co1—C1	154.34 (12)	C12—C11—Co1	69.32 (17)
C13—Co1—C1	113.93 (14)	C15—C11—Co1	126.7 (3)
C12—Co1—C1	115.67 (12)	C13—C12—C11	90.1 (3)
C3—Co1—C1	69.56 (12)	C13—C12—C16	134.8 (3)
C5—Co1—C1	69.11 (14)	C11—C12—C16	134.6 (3)
C4—Co1—C1	82.52 (13)	C13—C12—Co1	68.44 (16)
C2—Co1—C1	37.96 (12)	C11—C12—Co1	68.06 (16)
C6—Co1—C1	37.76 (12)	C16—C12—Co1	126.8 (2)
C1—N1—C9	121.6 (3)	C12—C13—C14	90.1 (3)
C1—N1—C7	121.7 (3)	C12—C13—C17	134.4 (3)
C9—N1—C7	116.1 (3)	C14—C13—C17	134.7 (3)
N1—C1—C6	121.9 (3)	C12—C13—Co1	69.27 (17)
N1—C1—C2	121.6 (3)	C14—C13—Co1	67.82 (18)

C6—C1—C2	116.4 (3)	C17—C13—Co1	128.0 (3)
N1—C1—Co1	136.6 (2)	C11—C14—C13	89.8 (3)
C6—C1—Co1	66.71 (18)	C11—C14—C18	134.8 (4)
C2—C1—Co1	65.99 (18)	C13—C14—C18	134.6 (4)
C3—C2—C1	121.3 (3)	C11—C14—Co1	69.06 (17)
C3—C2—Co1	69.09 (18)	C13—C14—Co1	69.23 (18)
C1—C2—Co1	76.05 (19)	C18—C14—Co1	127.1 (2)
C3—C2—H2	119.3	C11—C15—H15A	109.5
C1—C2—H2	119.3	C11—C15—H15B	109.5
Co1—C2—H2	127.6	H15A—C15—H15B	109.5
C4—C3—C2	120.8 (3)	C11—C15—H15C	109.5
C4—C3—Co1	71.3 (2)	H15A—C15—H15C	109.5
C2—C3—Co1	71.57 (18)	H15B—C15—H15C	109.5
C4—C3—H3	119.6	C12—C16—H16A	109.5
C2—C3—H3	119.6	C12—C16—H16B	109.5
Co1—C3—H3	130.1	H16A—C16—H16B	109.5
C5—C4—C3	118.4 (3)	C12—C16—H16C	109.5
C5—C4—Co1	70.0 (2)	H16A—C16—H16C	109.5
C3—C4—Co1	69.54 (19)	H16B—C16—H16C	109.5
C5—C4—H4	120.8	C13—C17—H17A	109.5
C3—C4—H4	120.8	C13—C17—H17B	109.5
Co1—C4—H4	132.4	H17A—C17—H17B	109.5
C4—C5—C6	121.3 (4)	C13—C17—H17C	109.5
C4—C5—Co1	71.0 (2)	H17A—C17—H17C	109.5
C6—C5—Co1	72.0 (2)	H17B—C17—H17C	109.5
C4—C5—H5	119.4	C14—C18—H18A	109.5
C6—C5—H5	119.4	C14—C18—H18B	109.5
Co1—C5—H5	130.3	H18A—C18—H18B	109.5
C5—C6—C1	121.2 (3)	C14—C18—H18C	109.5
C5—C6—Co1	69.07 (19)	H18A—C18—H18C	109.5
C1—C6—Co1	75.53 (19)	H18B—C18—H18C	109.5
C5—C6—H6	119.4		
C9—N1—C1—C6	167.8 (4)	C7—N1—C9—C10B	−88.3 (7)
C7—N1—C1—C6	−21.4 (5)	C1—N1—C9—C10A	86.2 (5)
C9—N1—C1—C2	−8.5 (5)	C7—N1—C9—C10A	−85.1 (4)
C7—N1—C1—C2	162.3 (3)	C13—Co1—C11—C14	−49.9 (2)
C9—N1—C1—Co1	79.0 (5)	C12—Co1—C11—C14	−98.7 (3)
C7—N1—C1—Co1	−110.2 (4)	C3—Co1—C11—C14	60.5 (3)
C14—Co1—C1—N1	−54.3 (5)	C5—Co1—C11—C14	141.6 (2)
C11—Co1—C1—N1	51.5 (5)	C4—Co1—C11—C14	99.9 (2)
C13—Co1—C1—N1	−23.0 (4)	C2—Co1—C11—C14	−0.8 (6)
C12—Co1—C1—N1	23.7 (4)	C6—Co1—C11—C14	−178.28 (18)
C3—Co1—C1—N1	−141.6 (4)	C1—Co1—C11—C14	−137.1 (3)
C5—Co1—C1—N1	142.4 (4)	C14—Co1—C11—C12	98.7 (3)
C4—Co1—C1—N1	−179.8 (4)	C13—Co1—C11—C12	48.83 (19)
C2—Co1—C1—N1	−111.4 (4)	C3—Co1—C11—C12	159.2 (2)
C6—Co1—C1—N1	112.4 (4)	C5—Co1—C11—C12	−119.7 (2)

C14—Co1—C1—C6	−166.7 (2)	C4—Co1—C11—C12	−161.35 (19)
C11—Co1—C1—C6	−60.9 (4)	C2—Co1—C11—C12	97.9 (5)
C13—Co1—C1—C6	−135.5 (2)	C6—Co1—C11—C12	−79.6 (2)
C12—Co1—C1—C6	−88.7 (2)	C1—Co1—C11—C12	−38.3 (4)
C3—Co1—C1—C6	106.0 (2)	C14—Co1—C11—C15	−131.4 (4)
C5—Co1—C1—C6	29.9 (2)	C13—Co1—C11—C15	178.7 (3)
C4—Co1—C1—C6	67.8 (2)	C12—Co1—C11—C15	129.9 (4)
C2—Co1—C1—C6	136.2 (3)	C3—Co1—C11—C15	−70.9 (4)
C14—Co1—C1—C2	57.1 (3)	C5—Co1—C11—C15	10.2 (3)
C11—Co1—C1—C2	162.9 (3)	C4—Co1—C11—C15	−31.5 (3)
C13—Co1—C1—C2	88.3 (2)	C2—Co1—C11—C15	−132.3 (5)
C12—Co1—C1—C2	135.1 (2)	C6—Co1—C11—C15	50.3 (3)
C3—Co1—C1—C2	−30.2 (2)	C1—Co1—C11—C15	91.5 (4)
C5—Co1—C1—C2	−106.2 (2)	C14—C11—C12—C13	−0.1 (2)
C4—Co1—C1—C2	−68.4 (2)	C15—C11—C12—C13	171.8 (4)
C6—Co1—C1—C2	−136.2 (3)	Co1—C11—C12—C13	−66.62 (17)
N1—C1—C2—C3	−174.3 (3)	C14—C11—C12—C16	−172.9 (3)
C6—C1—C2—C3	9.2 (4)	C15—C11—C12—C16	−1.0 (6)
Co1—C1—C2—C3	54.4 (3)	Co1—C11—C12—C16	120.6 (4)
N1—C1—C2—Co1	131.3 (3)	C14—C11—C12—Co1	66.49 (17)
C6—C1—C2—Co1	−45.2 (3)	C15—C11—C12—Co1	−121.6 (4)
C14—Co1—C2—C3	76.5 (3)	C14—Co1—C12—C13	49.7 (2)
C11—Co1—C2—C3	77.2 (6)	C11—Co1—C12—C13	99.3 (3)
C13—Co1—C2—C3	121.6 (2)	C3—Co1—C12—C13	10.1 (6)
C12—Co1—C2—C3	158.5 (2)	C5—Co1—C12—C13	−179.2 (2)
C5—Co1—C2—C3	−67.3 (2)	C4—Co1—C12—C13	137.0 (3)
C4—Co1—C2—C3	−29.2 (2)	C2—Co1—C12—C13	−58.3 (3)
C6—Co1—C2—C3	−105.0 (2)	C6—Co1—C12—C13	−138.5 (2)
C1—Co1—C2—C3	−132.0 (3)	C1—Co1—C12—C13	−98.1 (2)
C14—Co1—C2—C1	−151.5 (2)	C14—Co1—C12—C11	−49.6 (2)
C11—Co1—C2—C1	−150.8 (5)	C13—Co1—C12—C11	−99.3 (3)
C13—Co1—C2—C1	−106.5 (2)	C3—Co1—C12—C11	−89.1 (6)
C12—Co1—C2—C1	−69.5 (3)	C5—Co1—C12—C11	81.5 (2)
C3—Co1—C2—C1	132.0 (3)	C4—Co1—C12—C11	37.7 (3)
C5—Co1—C2—C1	64.6 (2)	C2—Co1—C12—C11	−157.6 (2)
C4—Co1—C2—C1	102.7 (2)	C6—Co1—C12—C11	122.2 (2)
C6—Co1—C2—C1	26.9 (2)	C1—Co1—C12—C11	162.65 (19)
C1—C2—C3—C4	−3.9 (5)	C14—Co1—C12—C16	−179.7 (3)
Co1—C2—C3—C4	53.8 (3)	C11—Co1—C12—C16	−130.1 (4)
C1—C2—C3—Co1	−57.7 (3)	C13—Co1—C12—C16	130.6 (4)
C14—Co1—C3—C4	106.4 (2)	C3—Co1—C12—C16	140.7 (5)
C11—Co1—C3—C4	68.2 (3)	C5—Co1—C12—C16	−48.6 (3)
C13—Co1—C3—C4	150.4 (2)	C4—Co1—C12—C16	−92.5 (4)
C12—Co1—C3—C4	142.2 (5)	C2—Co1—C12—C16	72.3 (3)
C5—Co1—C3—C4	−29.5 (2)	C6—Co1—C12—C16	−7.9 (3)
C2—Co1—C3—C4	−133.0 (3)	C1—Co1—C12—C16	32.5 (3)
C6—Co1—C3—C4	−67.4 (2)	C11—C12—C13—C14	0.1 (2)
C1—Co1—C3—C4	−103.8 (2)	C16—C12—C13—C14	172.9 (3)

C14—Co1—C3—C2	−120.7 (2)	Co1—C12—C13—C14	−66.15 (17)
C11—Co1—C3—C2	−158.8 (2)	C11—C12—C13—C17	−170.4 (4)
C13—Co1—C3—C2	−76.6 (3)	C16—C12—C13—C17	2.4 (7)
C12—Co1—C3—C2	−84.8 (6)	Co1—C12—C13—C17	123.4 (4)
C5—Co1—C3—C2	103.5 (2)	C11—C12—C13—Co1	66.27 (17)
C4—Co1—C3—C2	133.0 (3)	C16—C12—C13—Co1	−121.0 (4)
C6—Co1—C3—C2	65.6 (2)	C14—Co1—C13—C12	−99.0 (3)
C1—Co1—C3—C2	29.2 (2)	C11—Co1—C13—C12	−49.25 (18)
C2—C3—C4—C5	−1.9 (5)	C3—Co1—C13—C12	−176.98 (18)
Co1—C3—C4—C5	52.0 (3)	C5—Co1—C13—C12	2.7 (7)
C2—C3—C4—Co1	−53.9 (3)	C4—Co1—C13—C12	−134.9 (3)
C14—Co1—C4—C5	139.4 (2)	C2—Co1—C13—C12	142.69 (18)
C11—Co1—C4—C5	92.7 (2)	C6—Co1—C13—C12	64.3 (3)
C13—Co1—C4—C5	165.2 (3)	C1—Co1—C13—C12	102.51 (19)
C12—Co1—C4—C5	66.0 (3)	C11—Co1—C13—C14	49.8 (2)
C3—Co1—C4—C5	−132.4 (3)	C12—Co1—C13—C14	99.0 (3)
C2—Co1—C4—C5	−103.1 (2)	C3—Co1—C13—C14	−78.0 (2)
C6—Co1—C4—C5	−28.9 (2)	C5—Co1—C13—C14	101.7 (6)
C1—Co1—C4—C5	−65.8 (2)	C4—Co1—C13—C14	−35.9 (4)
C14—Co1—C4—C3	−88.1 (2)	C2—Co1—C13—C14	−118.3 (2)
C11—Co1—C4—C3	−134.9 (2)	C6—Co1—C13—C14	163.3 (2)
C13—Co1—C4—C3	−62.4 (4)	C1—Co1—C13—C14	−158.49 (19)
C12—Co1—C4—C3	−161.5 (2)	C14—Co1—C13—C17	130.1 (4)
C5—Co1—C4—C3	132.4 (3)	C11—Co1—C13—C17	179.9 (4)
C2—Co1—C4—C3	29.4 (2)	C12—Co1—C13—C17	−130.9 (4)
C6—Co1—C4—C3	103.6 (2)	C3—Co1—C13—C17	52.2 (4)
C1—Co1—C4—C3	66.6 (2)	C5—Co1—C13—C17	−128.2 (6)
C3—C4—C5—C6	1.9 (5)	C4—Co1—C13—C17	94.2 (5)
Co1—C4—C5—C6	53.7 (3)	C2—Co1—C13—C17	11.8 (4)
C3—C4—C5—Co1	−51.8 (3)	C6—Co1—C13—C17	−66.6 (4)
C14—Co1—C5—C4	−63.6 (3)	C1—Co1—C13—C17	−28.4 (4)
C11—Co1—C5—C4	−102.8 (2)	C12—C11—C14—C13	0.1 (2)
C13—Co1—C5—C4	−150.1 (6)	C15—C11—C14—C13	−171.5 (4)
C12—Co1—C5—C4	−147.9 (2)	Co1—C11—C14—C13	67.76 (18)
C3—Co1—C5—C4	29.6 (2)	C12—C11—C14—C18	170.3 (4)
C2—Co1—C5—C4	68.0 (2)	C15—C11—C14—C18	−1.3 (7)
C6—Co1—C5—C4	133.6 (3)	Co1—C11—C14—C18	−122.0 (4)
C1—Co1—C5—C4	104.5 (2)	C12—C11—C14—Co1	−67.63 (17)
C14—Co1—C5—C6	162.8 (2)	C15—C11—C14—Co1	120.7 (4)
C11—Co1—C5—C6	123.6 (2)	C12—C13—C14—C11	−0.1 (2)
C13—Co1—C5—C6	76.3 (6)	C17—C13—C14—C11	170.3 (4)
C12—Co1—C5—C6	78.5 (2)	Co1—C13—C14—C11	−67.60 (18)
C3—Co1—C5—C6	−104.0 (2)	C12—C13—C14—C18	−170.4 (4)
C4—Co1—C5—C6	−133.6 (3)	C17—C13—C14—C18	0.1 (7)
C2—Co1—C5—C6	−65.6 (2)	Co1—C13—C14—C18	122.1 (4)
C1—Co1—C5—C6	−29.1 (2)	C12—C13—C14—Co1	67.48 (17)
C4—C5—C6—C1	3.8 (5)	C17—C13—C14—Co1	−122.1 (4)
Co1—C5—C6—C1	57.0 (3)	C13—Co1—C14—C11	98.1 (2)

C4—C5—C6—Co1	−53.3 (3)	C12—Co1—C14—C11	49.30 (19)
N1—C1—C6—C5	174.4 (3)	C3—Co1—C14—C11	−140.2 (2)
C2—C1—C6—C5	−9.1 (4)	C5—Co1—C14—C11	−61.4 (3)
Co1—C1—C6—C5	−54.0 (3)	C4—Co1—C14—C11	−99.1 (2)
N1—C1—C6—Co1	−131.6 (3)	C2—Co1—C14—C11	179.74 (18)
C2—C1—C6—Co1	44.9 (3)	C6—Co1—C14—C11	7.0 (8)
C14—Co1—C6—C5	−81.5 (7)	C1—Co1—C14—C11	142.2 (2)
C11—Co1—C6—C5	−75.6 (3)	C11—Co1—C14—C13	−98.1 (2)
C13—Co1—C6—C5	−160.5 (2)	C12—Co1—C14—C13	−48.84 (19)
C12—Co1—C6—C5	−120.5 (2)	C3—Co1—C14—C13	121.6 (2)
C3—Co1—C6—C5	67.0 (2)	C5—Co1—C14—C13	−159.5 (2)
C4—Co1—C6—C5	28.9 (2)	C4—Co1—C14—C13	162.7 (2)
C2—Co1—C6—C5	105.1 (2)	C2—Co1—C14—C13	81.6 (2)
C1—Co1—C6—C5	132.1 (3)	C6—Co1—C14—C13	−91.1 (7)
C14—Co1—C6—C1	146.4 (6)	C1—Co1—C14—C13	44.1 (3)
C11—Co1—C6—C1	152.22 (19)	C11—Co1—C14—C18	131.0 (5)
C13—Co1—C6—C1	67.4 (3)	C13—Co1—C14—C18	−130.9 (5)
C12—Co1—C6—C1	107.4 (2)	C12—Co1—C14—C18	−179.7 (5)
C3—Co1—C6—C1	−65.1 (2)	C3—Co1—C14—C18	−9.2 (4)
C5—Co1—C6—C1	−132.1 (3)	C5—Co1—C14—C18	69.6 (5)
C4—Co1—C6—C1	−103.3 (2)	C4—Co1—C14—C18	31.9 (4)
C2—Co1—C6—C1	−27.07 (19)	C2—Co1—C14—C18	−49.3 (4)
C1—N1—C7—C8	96.8 (4)	C6—Co1—C14—C18	138.0 (6)
C9—N1—C7—C8	−92.0 (4)	C1—Co1—C14—C18	−86.8 (5)
C1—N1—C9—C10B	82.9 (7)		