Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tris(*tert*-butylisonitrile)hexacarbonyl-μ_3-ethylidyne-*triangulo*-tricobalt(I)-(3 *Co*—*Co*)

Jolene M. Brown and Brian K. Nicholson*

Chemistry Department, University of Waikato, Private Bag 3105, Hamilton 3240, New Zealand
Correspondence e-mail: B.Nicholson@waikato.ac.nz

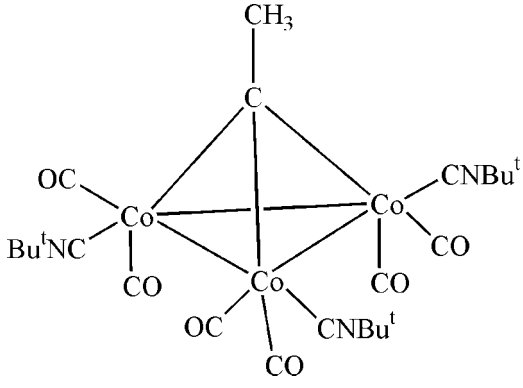
Received 10 September 2007; accepted 11 September 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.025; wR factor = 0.059; data-to-parameter ratio = 14.9.

The title molecule, $[Co_3(C_2H_3)(C_5H_9N)_3(CO)_6]$ or $[Co_3(\mu_3\text{-}CCH_3)(CN^tBu)_3(CO)_6]$, lies on a threefold rotation axis. The three isonitrile ligands each occupy an equatorial site on each of the three Co atoms. The average Co—Co bond length is 2.4769 (6) Å. The *tert*-butyl groups are disordered over two orientations, with site occupancies of *ca* 0.6:0.4.

Related literature

For details of the synthesis, see Newman & Manning (1974). For the structure of the parent nonacarbonyl cluster, see Sutton & Dahl (1967). Other examples of equatorially tri-substituted derivatives of $[Co_3(\mu_3\text{-}CR)(CO)_9]$ include the $(MeO)_3P$ derivative (Dawson *et al.*, 1979). Axial substitution appears to be favoured only by very bulky or chelating ligands (D'Agostino *et al.*, 1991; Renouard *et al.*, 1996).



Experimental

Crystal data

$[Co_3(C_2H_3)(C_5H_9N)_3(CO)_6]$
M_r = 621.29
Trigonal, $R3c$
a = 16.9804 (6) Å
c = 17.4605 (11) Å
V = 4360.0 (4) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 1.73 mm^{-1}
T = 293 (2) K
0.54 × 0.13 × 0.11 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.672, T_{max} = 0.830
10767 measured reflections
1991 independent reflections
1757 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.059
S = 1.00
1991 reflections
134 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), with 989 Friedel pairs
Flack parameter: 0.01 (2)

Data collection: *SMART* (Bruker 2001); cell refinement: *SAINT* (Bruker 2001); data reduction: *SAINT* (Bruker 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Version 1.70.01; Farrugia, 1999).

The authors thank Dr Jan Wikaira, University of Canterbury, New Zealand, for the collection of the X-ray intensity data.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2355).

References

Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
D'Agostino, M. F., Frampton, L. S. & McGlinchey, M. J. (1991). *Organometallics*, **10**, 1383–1390.
Dawson, P. A., Robinson, B. H. & Simpson, J. (1979). *J. Chem. Soc. Dalton Trans.* pp. 1762–1768.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Newman, J. & Manning, A. R. (1974). *J. Chem. Soc. Dalton Trans.* pp. 2549–2553.
Renouard, C., Rheinwald, G., Stoeckli-Evans, H., Süss-Fink, G., Braga, D. & Grepioni, F. (1996). *J. Chem. Soc. Dalton Trans.* pp. 1875–1883.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sutton, P. W. & Dahl, L. F. (1967). *J. Am. Chem. Soc.* **89**, 261–268.

  doi:10.1107/S1600536807044261

supplementary materials

Acta Cryst. (2007). E**63**, m2542 [**doi:10.1107/S1600536807044261**]

Tris(*tert*-butylisonitrile)hexacarbonyl-μ_3-ethylidyne-*triangulo*-tricobalt(I)(3 *Co-Co*)

J. M. Brown and B. K. Nicholson

Comment

The title compound is the first structurally characterized isonitrile derivative of a $[Co_3(\mu_3\text{-CR})(CO)_9]$ cluster. The three $CNBu^t$ ligands have displaced three equatorial CO ligands in the parent molecule, to give a molecule with C_3 symmetry. The substitution has had little effect on the parameters of the cluster core with average Co—Co and Co—C distances (2.4769 (6) and 1.908 (3) Å respectively) that do not differ significantly from those of parent (2.467 (7) and 1.90 (2) Å, (Sutton & Dahl, 1967) though the low precision of the earlier study would mask any small changes.

Experimental

The compound was prepared by thermal reaction between $[Co_3(\mu_3\text{-CR})(CO)_9]$ and $CNBu^t$ (Newman & Manning, 1974). X-ray crystals were grown from pentane.

Refinement

The *tert*-butyl groups are disordered over two orientations which refined to a 0.64:0.36 occupancy ratio; this accounts for the large differences between the displacement parameters of the C4 carbon atom and the attached CH_3 carbon atoms. All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.96 Å, $U_{iso} = 1.5U_{eq}$ (C).

Figures

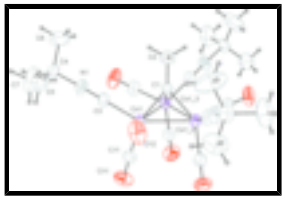


Fig. 1. Structure of $[Co_3(\mu_3\text{-}CCH_3)(CO)_6(CNBu^t)_3]$ with diplacement parameters drawn at the 30% probability level. Only the major disorder component of the *t*-butyl group is shown.

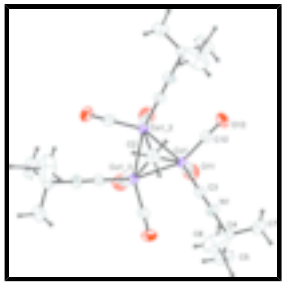


Fig. 2. A view down the threefold axis. Only the major disorder component of the *t*-butyl group is shown.

tris(*tert*-butylisonitrile)hexacarbonyl-μ$_3$-ethylidyne- *triangulo*-tricobalt(I)(3 Co—Co)

Crystal data

$[Co_3(C_2H_3)(C_5H_9N)_3(CO)_6]$	$Z = 6$
$M_r = 621.29$	$F_{000} = 1908$
Trigonal, $R3c$	$D_x = 1.420$ Mg m^{-3}
Hall symbol: R 3 -2"c	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 16.9804$ (6) Å	Cell parameters from 5750 reflections
$b = 16.9804$ (6) Å	$\theta = 2.4$–26.4º
$c = 17.4605$ (11) Å	$\mu = 1.73$ mm^{-1}
$\alpha = 90$º	$T = 293$ (2) K
$\beta = 90$º	Hexagonal rod, black
$\gamma = 120$º	0.54 × 0.13 × 0.11 mm
$V = 4360.0$ (4) Å^3	

Data collection

Siemens SMART CCD diffractometer	1991 independent reflections
Radiation source: fine-focus sealed tube	1757 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.036$
$T = 293$(2) K	$\theta_{max} = 26.4$º
multi–scan	$\theta_{min} = 2.4$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -21 \rightarrow 20$
$T_{min} = 0.672$, $T_{max} = 0.830$	$k = -21 \rightarrow 21$
10767 measured reflections	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.025$	$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.059$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.00$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
1991 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}
134 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 989 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.01 (2)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Co1	0.92643 (2)	0.01828 (2)	0.164850 (14)	0.04289 (10)	
N1	0.91952 (19)	0.17320 (19)	0.24181 (17)	0.0676 (7)	
C1	1.0000	0.0000	0.2372 (3)	0.0441 (9)	
C2	1.0000	0.0000	0.3218 (3)	0.0698 (14)	
H2A	1.0116	0.0581	0.3401	0.105*	0.333
H2B	1.0466	−0.0116	0.3401	0.105*	0.333
H2C	0.9419	−0.0466	0.3401	0.105*	0.333
C3	0.92327 (19)	0.1150 (2)	0.21234 (18)	0.0557 (7)	
C4	0.9179 (2)	0.2509 (2)	0.27631 (19)	0.0684 (9)	
C11	0.9168 (2)	0.0388 (2)	0.06419 (19)	0.0593 (7)	
O11	0.9109 (2)	0.0514 (2)	0.00155 (15)	0.0992 (9)	
C12	0.8144 (2)	−0.0659 (2)	0.18837 (19)	0.0618 (8)	
O12	0.74179 (17)	−0.1194 (2)	0.2037 (2)	0.1022 (10)	
C5	0.9649 (10)	0.3290 (5)	0.2265 (6)	0.120 (5)	0.640 (16)
H51	0.9328	0.3171	0.1788	0.180*	0.640 (16)
H52	1.0255	0.3405	0.2172	0.180*	0.640 (16)
H53	0.9676	0.3812	0.2506	0.180*	0.640 (16)
C6	0.9631 (13)	0.2680 (9)	0.3533 (7)	0.166 (8)	0.640 (16)
H61	0.9298	0.2155	0.3853	0.249*	0.640 (16)
H62	0.9643	0.3197	0.3768	0.249*	0.640 (16)
H63	1.0243	0.2797	0.3470	0.249*	0.640 (16)
C7	0.8180 (6)	0.2243 (6)	0.2791 (9)	0.135 (6)	0.640 (16)
H71	0.7858	0.1729	0.3124	0.202*	0.640 (16)
H72	0.7926	0.2086	0.2285	0.202*	0.640 (16)
H73	0.8126	0.2745	0.2980	0.202*	0.640 (16)
C5A	1.0165 (10)	0.3310 (10)	0.2776 (15)	0.126 (10)	0.360 (16)
H51A	1.0400	0.3441	0.2263	0.188*	0.360 (16)
H52A	1.0533	0.3150	0.3083	0.188*	0.360 (16)
H53A	1.0176	0.3836	0.2989	0.188*	0.360 (16)
C6A	0.8664 (11)	0.2787 (10)	0.2206 (10)	0.100 (7)	0.360 (16)
H61A	0.8968	0.2940	0.1719	0.150*	0.360 (16)
H62A	0.8646	0.3304	0.2407	0.150*	0.360 (16)
H63A	0.8054	0.2290	0.2142	0.150*	0.360 (16)
C7A	0.875 (2)	0.2261 (16)	0.3488 (9)	0.154 (13)	0.360 (16)
H71A	0.9097	0.2094	0.3820	0.231*	0.360 (16)
H72A	0.8149	0.1754	0.3432	0.231*	0.360 (16)
H73A	0.8727	0.2766	0.3706	0.231*	0.360 (16)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.03864 (19)	0.04002 (19)	0.05232 (16)	0.02140 (15)	−0.00286 (15)	−0.00329 (17)
N1	0.0639 (16)	0.0525 (15)	0.0928 (18)	0.0338 (14)	−0.0040 (15)	−0.0173 (14)
C1	0.0416 (13)	0.0416 (13)	0.049 (2)	0.0208 (7)	0.000	0.000

C2	0.077 (2)	0.077 (2)	0.055 (3)	0.0386 (11)	0.000	0.000
C3	0.0456 (15)	0.0494 (16)	0.0751 (18)	0.0259 (14)	−0.0041 (13)	−0.0059 (14)
C4	0.078 (2)	0.0545 (18)	0.081 (2)	0.0398 (18)	−0.0010 (18)	−0.0181 (16)
C11	0.0538 (18)	0.0548 (17)	0.067 (2)	0.0257 (14)	−0.0064 (14)	0.0037 (14)
O11	0.105 (2)	0.111 (2)	0.0676 (16)	0.0438 (19)	−0.0143 (14)	0.0177 (15)
C12	0.0504 (19)	0.0576 (18)	0.083 (2)	0.0312 (16)	0.0002 (15)	0.0059 (14)
O12	0.0457 (14)	0.0793 (18)	0.171 (3)	0.0234 (13)	0.0190 (16)	0.0303 (18)
C5	0.169 (15)	0.064 (5)	0.121 (6)	0.054 (7)	0.021 (7)	0.002 (4)
C6	0.29 (2)	0.143 (12)	0.095 (7)	0.132 (15)	−0.076 (12)	−0.049 (8)
C7	0.096 (6)	0.085 (6)	0.237 (18)	0.056 (5)	0.031 (7)	−0.024 (8)
C5A	0.087 (9)	0.075 (9)	0.21 (3)	0.038 (7)	−0.026 (10)	−0.075 (13)
C6A	0.104 (14)	0.079 (11)	0.141 (12)	0.063 (12)	−0.024 (10)	−0.024 (9)
C7A	0.31 (4)	0.132 (16)	0.074 (13)	0.15 (2)	0.067 (19)	0.015 (11)

Geometric parameters (Å, °)

Co1—C12	1.764 (3)	C5—H51	0.9600
Co1—C11	1.815 (3)	C5—H52	0.9600
Co1—C3	1.865 (3)	C5—H53	0.9600
Co1—C1	1.908 (3)	C6—H61	0.9600
Co1—Co1^{i}	2.4769 (6)	C6—H62	0.9600
N1—C3	1.143 (4)	C6—H63	0.9600
N1—C4	1.464 (4)	C7—H71	0.9600
C1—C2	1.477 (8)	C7—H72	0.9600
C2—H2A	0.9600	C7—H73	0.9600
C2—H2B	0.9600	C5A—H51A	0.9600
C2—H2C	0.9600	C5A—H52A	0.9600
C4—C7A	1.413 (16)	C5A—H53A	0.9600
C4—C5	1.446 (8)	C6A—H61A	0.9600
C4—C6	1.503 (10)	C6A—H62A	0.9600
C4—C7	1.522 (9)	C6A—H63A	0.9600
C4—C6A	1.532 (14)	C7A—H71A	0.9600
C4—C5A	1.541 (14)	C7A—H72A	0.9600
C11—O11	1.129 (4)	C7A—H73A	0.9600
C12—O12	1.139 (4)		
C12—Co1—C11	102.54 (15)	O12—C12—Co1	179.1 (3)
C12—Co1—C3	96.66 (13)	C4—C5—H51	109.5
C11—Co1—C3	102.54 (14)	C4—C5—H52	109.5
C12—Co1—C1	104.04 (11)	H51—C5—H52	109.5
C11—Co1—C1	143.23 (15)	C4—C5—H53	109.5
C3—Co1—C1	99.12 (12)	H51—C5—H53	109.5
C12—Co1—Co1^{i}	150.03 (10)	H52—C5—H53	109.5
C11—Co1—Co1^{i}	96.93 (10)	C4—C6—H61	109.5
C3—Co1—Co1^{i}	101.12 (9)	C4—C6—H62	109.5
C1—Co1—Co1^{i}	49.53 (8)	H61—C6—H62	109.5
C12—Co1—Co1^{ii}	92.89 (10)	C4—C6—H63	109.5
C11—Co1—Co1^{ii}	104.41 (11)	H61—C6—H63	109.5

C3—Co1—Co1^{ii}	148.64 (9)	H62—C6—H63	109.5
Co1^{i}—Co1—Co1^{ii}	60.0	C4—C7—H71	109.5
C3—N1—C4	176.9 (4)	C4—C7—H72	109.5
C2—C1—Co1	131.45 (10)	H71—C7—H72	109.5
Co1^{ii}—C1—Co1	80.95 (16)	C4—C7—H73	109.5
C1—C2—H2A	109.5	H71—C7—H73	109.5
C1—C2—H2B	109.5	H72—C7—H73	109.5
H2A—C2—H2B	109.5	C4—C5A—H51A	109.5
C1—C2—H2C	109.5	C4—C5A—H52A	109.5
H2A—C2—H2C	109.5	H51A—C5A—H52A	109.5
H2B—C2—H2C	109.5	C4—C5A—H53A	109.5
N1—C3—Co1	178.6 (3)	H51A—C5A—H53A	109.5
C7A—C4—N1	109.7 (8)	H52A—C5A—H53A	109.5
C5—C4—N1	109.2 (4)	C4—C6A—H61A	109.5
C5—C4—C6	111.5 (7)	C4—C6A—H62A	109.5
N1—C4—C6	107.7 (6)	H61A—C6A—H62A	109.5
C5—C4—C7	108.3 (8)	C4—C6A—H63A	109.5
N1—C4—C7	105.4 (4)	H61A—C6A—H63A	109.5
C6—C4—C7	114.5 (9)	H62A—C6A—H63A	109.5
C7A—C4—C6A	112.1 (14)	C4—C7A—H71A	109.5
N1—C4—C6A	106.7 (5)	C4—C7A—H72A	109.5
C7A—C4—C5A	114.7 (15)	H71A—C7A—H72A	109.5
N1—C4—C5A	107.4 (5)	C4—C7A—H73A	109.5
C6A—C4—C5A	105.9 (11)	H71A—C7A—H73A	109.5
O11—C11—Co1	179.9 (4)	H72A—C7A—H73A	109.5

Symmetry codes: (i) −*x*+*y*+2, −*x*+1, *z*; (ii) −*y*+1, *x*−*y*−1, *z*.

Fig. 1

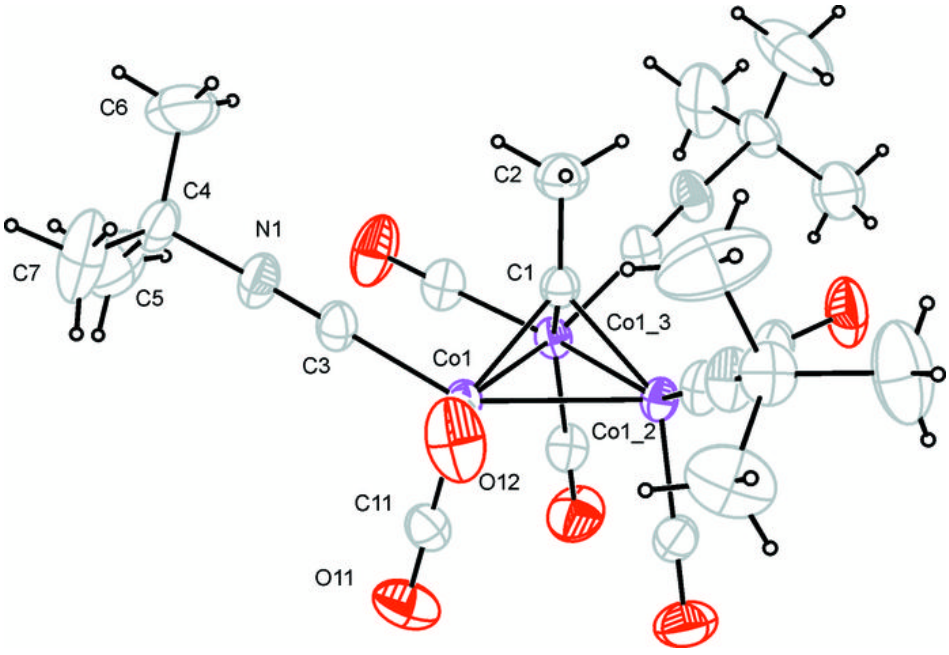


Fig. 2