

## Crystal data

[Re(C<sub>6</sub>H<sub>5</sub>N)Cl<sub>3</sub>·  
(C<sub>34</sub>H<sub>28</sub>FeP<sub>2</sub>)]·CH<sub>2</sub>Cl<sub>2</sub>*M<sub>r</sub>* = 1022.94

Monoclinic

*P*2<sub>1</sub>/*n**a* = 11.093 (2) Å*b* = 21.665 (4) Å*c* = 16.823 (5) Å

β = 104.404 (18)°

*V* = 3916.1 (16) Å<sup>3</sup>*Z* = 4*D<sub>x</sub>* = 1.735 Mg m<sup>-3</sup>*D<sub>m</sub>* not measured

## Data collection

Siemens P4 diffractometer

ω scans

Absorption correction:

ψ scan (North *et al.*,  
1968)*T<sub>min</sub>* = 0.632, *T<sub>max</sub>* = 0.731

7673 measured reflections

7284 independent reflections

5538 reflections with

*F* > 4σ(*F*)

## Refinement

Refinement on *F*<sup>2</sup>*R*(*F*) = 0.038*wR*(*F*<sup>2</sup>) = 0.077*S* = 1.030

7284 reflections

461 parameters

H atoms constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0263*P*)<sup>2</sup>  
+ 5.2393*P*]where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 38  
reflections

θ = 1.99–25.5°

μ = 3.913 mm<sup>-1</sup>*T* = 294 (2) K

Plate

0.60 × 0.10 × 0.08 mm

Green

*R<sub>int</sub>* = 0.033θ<sub>max</sub> = 25.50°*h* = 0 → 13*k* = -26 → 0*l* = -20 → 19

3 standard reflections

every 97 reflections

intensity decay: none

(Δ/σ)<sub>max</sub> = 0.001Δρ<sub>max</sub> = 0.789 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.762 e Å<sup>-3</sup>

Extinction correction:

*SHELXTL* (Bruker, 1997)

Extinction coefficient:

0.00025 (4)

Scattering factors from

*International Tables for  
Crystallography* (Vol. C)This paper was supported by the 63 Research Fund  
of Sung Kyun Kwan University (1997).Supplementary data for this paper are available from the IUCr  
electronic archives (Reference: DA1088). Services for accessing these  
data are described at the back of the journal.

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## Carbonyl-μ-dimethylacetylene dicarboxylate and carbonyl-μ-methyltetraolate manganesecobalt complexes

KARSTEN BECK,<sup>†</sup> JEANETTE A. KRAUSE BAUER AND  
JOHN J. ALEXANDERDepartment of Chemistry, University of Cincinnati,  
Cincinnati, OH 45221-0172, USA. E-mail:  
john.j.alexander@uc.edu

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## Abstract

The title complexes, heptacarbonyl-1κ<sup>4</sup>C,2κ<sup>3</sup>C-μ-(dimethyl but-2-ynedioato)-1:2κ<sup>2</sup>C<sup>2</sup>,1:2κ<sup>2</sup>C<sup>3</sup>-cobalt-manganese(*Mn—Co*), [MnCo(C<sub>6</sub>H<sub>6</sub>O<sub>4</sub>)(CO)<sub>7</sub>], (5), and heptacarbonyl-1κ<sup>4</sup>C,2κ<sup>3</sup>C-μ-(methyl but-2-ynoato)-1:2κ<sup>2</sup>C<sup>2</sup>,1:2κ<sup>2</sup>C<sup>3</sup>-cobaltmanganese(*Mn—Co*), [MnCo(C<sub>5</sub>H<sub>6</sub>O<sub>2</sub>)(CO)<sub>7</sub>], (6), were synthesized from MnCo(CO)<sub>9</sub> and the corresponding alkyne. The Mn—Co bond length is 2.5527 (5) Å for (5) and 2.5468 (4) Å for (6). The alkyne C—C bond is perpendicular to the Mn—Co bond.

<sup>†</sup> Current address: Department of Chemistry, University of Michigan, Ann Arbor, MI 48109-1055, USA.

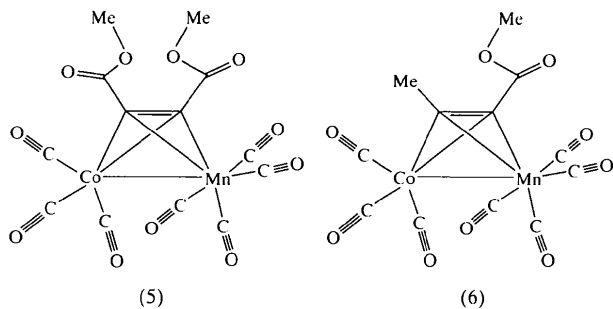
Table 1. Selected geometric parameters (Å, °)

Re—N1	1.717 (5)	Fe—C13	2.040 (6)
Re—Cl3	2.3961 (15)	Fe—C9	2.057 (6)
Re—Cl2	2.4193 (16)	Fe—C11	2.060 (6)
Re—Cl1	2.4392 (15)	Fe—C15	2.063 (6)
Re—P2	2.4620 (15)	Fe—C10	2.068 (6)
Re—P1	2.4674 (15)	Fe—C14	2.068 (6)
Fe—C12	2.020 (6)	P1—C7	1.812 (5)
Fe—C7	2.024 (5)	P2—C12	1.801 (6)
Fe—C8	2.028 (6)	N1—C1	1.395 (7)
Fe—C16	2.028 (6)		
N1—Re—Cl3	173.19 (16)	Cl2—Re—P2	165.07 (5)
N1—Re—Cl2	100.53 (16)	Cl1—Re—P2	86.24 (5)
Cl3—Re—Cl2	85.90 (6)	N1—Re—P1	91.58 (16)
N1—Re—Cl1	92.37 (16)	Cl3—Re—P1	86.69 (5)
Cl3—Re—Cl1	90.29 (5)	Cl2—Re—P1	85.40 (5)
Cl2—Re—Cl1	85.90 (6)	Cl1—Re—P1	170.98 (5)
N1—Re—P2	92.47 (16)	P2—Re—P1	101.69 (5)
Cl3—Re—P2	81.45 (5)	Cl1—N1—Re	170.7 (4)

Data collection: *XSCANS* (Siemens, 1995). Cell refinement: *XSCANS*. Data reduction: *SHELXTL* (Bruker, 1997). Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *ORTEP-3* (Farrugia, 1997). Software used to prepare material for publication: *SHELXTL*.

### Comment

Dicobalt-hexacarbonyl-alkyne complexes,  $[\text{Co}_2(\text{CO})_6(\mu\text{-alkyne})]$ , are important intermediates in the Pauson-Khand reaction which produces  $\alpha,\beta$ -unsaturated cyclopentenones by coupling an alkyne, an alkene and CO (Khand, Knox, Pauson & Watts, 1971; Khand, Knox, Pauson, Watts & Foremann, 1971). Over 70 structures of  $[\text{Co}_2(\text{CO})_6(\mu\text{-alkyne})]$  have been determined by X-ray crystallography (Went, 1997). Their structures contain a single Co—Co bond and the alkyne ligand is perpendicular to the Co—Co bond. Although the syntheses of complexes  $[\text{MnCo}(\text{CO})_7(\mu\text{-RC}_2\text{R}')] [ (1) R = R' = \text{Et}; (2) R = \text{Et}, R' = \text{Me}; (3) R = \text{Me}, R' = \text{Ph}; (4) R = R' = \text{Ph}]$  have been documented (Manning & Peterson, 1984), no X-ray data for these complexes have been reported. The structures of (1)–(4) are assumed to be similar to the structures of  $[\text{Co}_2(\text{CO})_6(\mu\text{-alkyne})]$  complexes based on  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{17}\text{O}$  NMR and IR spectral data. Isocyanide derivatives of  $[\text{MnCo}(\text{CO})_7(\mu\text{-alkyne})]$  are believed to be intermediates in Pauson-Khand-type reactions yielding unsaturated cyclic imines (Beck *et al.*, 1998). Therefore, it was important to obtain accurate structural information on Mn—Co—alkyne complexes. Since isocyanide derivatives of  $[\text{MnCo}(\text{CO})_7(\mu\text{-alkyne})]$  are too unstable to be isolated, an X-ray investigation of two  $[\text{MnCo}(\text{CO})_7(\mu\text{-alkyne})]$  complexes, namely  $[\text{MnCo}(\text{CO})_7(\text{MeO}_2\text{CC}_2\text{CO}_2\text{Me})]$ , (5), and  $[\text{MnCo}(\text{CO})_7(\text{MeO}_2\text{CC}_2\text{Me})]$ , (6), was undertaken.



The structures of (5) (Fig. 1) and (6) (Fig. 2) are similar to  $[\text{Co}_2(\text{CO})_6(\mu\text{-RC}_2\text{R})]$  [(7)  $R = \text{Ph}$ ; (8)  $R = \text{CO}_2\text{Me}$ ; (9)  $R = \textit{tert}$ -butyl; Gregson & Howard, 1983]. The bridging alkyne in (5) ( $88.0^\circ$ ) and (6) ( $92.1^\circ$ ) is perpendicular to the Mn—Co bond. This agrees well with the perpendicular bridging geometry reported for (7) ( $92^\circ$ ), (8) ( $89^\circ$ ) and (9) ( $90^\circ$ ). The Mn—Co bond distances of 2.5527 (5) Å in (5) and 2.5468 (4) Å in (6) indicate a single Mn—Co bond. This is longer than the Co—Co bond length in (7) [2.476 (2) Å], (8) [2.477 (3) Å] and (9) [2.460 (1) Å], but consistent with the Mn—Co bond length in  $[\text{MnCo}(\text{CO})_6(\text{Ph}_2\text{C}_4\text{Me}_2)]$  [2.5488 (8) Å; Einstein *et al.*, 1986].

The alkyne C—C bond distances in (5) and (6) [both 1.328 (3) Å] are close to those in (8) [1.33 (3) Å] and

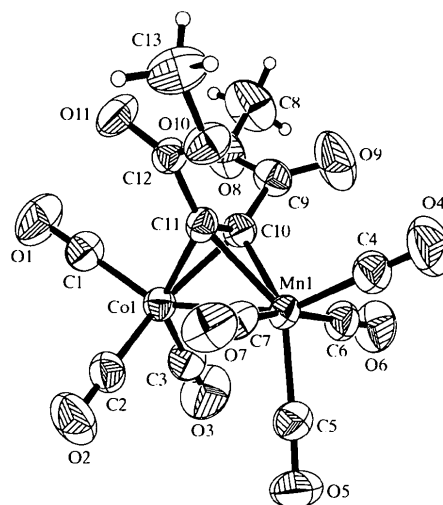


Fig. 1. The structure of (5) showing 50% probability displacement ellipsoids and the atomic labeling scheme. For clarity, only one conformation of the disordered methyl group is shown.

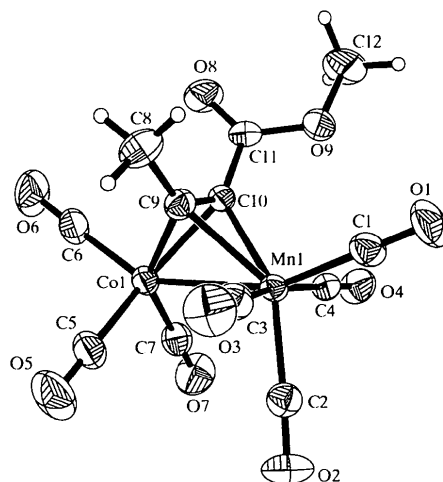


Fig. 2. The structure of (6) showing 50% probability displacement ellipsoids and the atomic labeling scheme. For clarity, only one conformation of the disordered methyl group is shown.

(9) [1.341 (3) Å]. In (7), however, possibly due to the bulky phenyl groups, the alkyne bond distance is slightly longer [1.36 (1) Å]. The average C=C bond length is 1.32 Å, compared with the average C≡C bond length of 1.21 Å (Orpen *et al.*, 1989). Hence, the alkyne C—C bond displays double-bond character upon coordination. This is also reflected in the bond angles between the alkyne substituents and the former C≡C bond, which are  $135.7 (2)$  and  $134.4 (2)^\circ$  for (5), and  $138.1 (2)$  and  $133.3 (2)^\circ$  for (6).

The average Co—C<sub>alkyne</sub> bond length in (5) and (6) (both 1.955 Å) is almost the same as in (7)–(9)

(1.959 Å). The average Mn—C<sub>alkyne</sub> bond distance in (5) and (6) (both 2.066 Å) is slightly longer than the Co—C<sub>alkyne</sub> bond, indicating a weaker metal–alkyne bond for manganese.

## Experimental

A threefold excess of the appropriate alkyne [MeO<sub>2</sub>CC<sub>2</sub>-CO<sub>2</sub>Me for (5) or MeO<sub>2</sub>CC<sub>2</sub>Me for (6)] was added to a hexane solution of MnCo(CO)<sub>9</sub> under argon. The mixture was exposed to UV irradiation at room temperature for 40 min. The reaction mixture was filtered and the filtrate cooled to 203 K to effect precipitation. Recrystallization from pentane at 203 K produced red solids in about 30% yield. Crystals of (5) and (6) suitable for X-ray analysis were obtained from pentane at 248 K. Data for (5): m.p. 340 K; IR (hexane): 2103 (*m*), 2080 (*w*), 2065 (*vs*), 2034 (*vs*), 2010 (*m*), 1988 (*s*), 1956 (*vw*), 1732 (*m*, ν<sub>C=O</sub>) cm<sup>-1</sup>; <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>): δ 3.39 p.p.m. Data for (6): m.p. 311 K; IR (hexane): 2194 (*m*), 2052 (*vs*), 2025 (*vs*), 2015 (*sh*), 1994 (*s*), 1976 (*s*), 1954 (*vw*), 1727 (*m*, ν<sub>C=O</sub>) cm<sup>-1</sup>; <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>): δ 3.32, 2.17 p.p.m.

### Compound (5)

#### Crystal data

[MnCo(C <sub>6</sub> H <sub>6</sub> O <sub>4</sub> )(CO) <sub>7</sub> ]	Mo Kα radiation
<i>M<sub>r</sub></i> = 452.05	λ = 0.71073 Å
Monoclinic	Cell parameters from 5693 reflections
<i>C</i> 2/ <i>c</i>	θ = 2.56–28.32°
<i>a</i> = 10.364 (1) Å	μ = 1.749 mm <sup>-1</sup>
<i>b</i> = 12.657 (1) Å	<i>T</i> = 273 (2) K
<i>c</i> = 26.590 (2) Å	Rod
β = 98.58 (1)°	0.30 × 0.20 × 0.20 mm
<i>V</i> = 3449.0 (5) Å <sup>3</sup>	Dark red
<i>Z</i> = 8	
<i>D<sub>x</sub></i> = 1.741 Mg m <sup>-3</sup>	
<i>D<sub>m</sub></i> not measured	

#### Data collection

SMART CCD diffractometer	3137 reflections with <i>I</i> > 2σ( <i>I</i> )
ω scans	<i>R</i> <sub>int</sub> = 0.035
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	θ <sub>max</sub> = 28.32°
<i>T</i> <sub>min</sub> = 0.738, <i>T</i> <sub>max</sub> = 0.928	<i>h</i> = -13 → 12
11 668 measured reflections	<i>k</i> = -16 → 13
4273 independent reflections	<i>l</i> = -35 → 35
	Intensity decay: none

#### Refinement

Refinement on <i>F</i> <sup>2</sup>	(Δ/σ) <sub>max</sub> = -0.001
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.038	Δρ <sub>max</sub> = 0.32 e Å <sup>-3</sup>
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.088	Δρ <sub>min</sub> = -0.39 e Å <sup>-3</sup>
<i>S</i> = 1.094	Extinction correction: none
4273 reflections	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
235 parameters	
H atoms constrained	
<i>w</i> = 1/[σ <sup>2</sup> ( <i>F</i> <sub>o</sub> <sup>2</sup> ) + (0.0388 <i>P</i> ) <sup>2</sup> + 0.4726 <i>P</i> ]	
where <i>P</i> = ( <i>F</i> <sub>o</sub> <sup>2</sup> + 2 <i>F</i> <sub>c</sub> <sup>2</sup> )/3	

Table 1. Selected geometric parameters (Å, °) for (5)

Co1—C2	1.807 (3)	Mn1—C10	2.056 (2)
Co1—C3	1.812 (3)	O8—C9	1.309 (3)
Co1—C1	1.820 (3)	O8—C8	1.454 (4)
Co1—C10	1.951 (2)	O9—C9	1.193 (3)
Co1—C11	1.958 (2)	O10—C12	1.325 (3)
Co1—Mn1	2.5527 (5)	O10—C13	1.452 (3)
Mn1—C4	1.818 (3)	O11—C12	1.196 (3)
Mn1—C5	1.836 (3)	C9—C10	1.476 (3)
Mn1—C6	1.857 (3)	C10—C11	1.328 (3)
Mn1—C7	1.857 (3)	C11—C12	1.484 (3)
Mn1—C11	2.052 (2)		
C10—Co1—Mn1	52.28 (7)	C9—C10—Co1	138.9 (2)
C11—Co1—Mn1	52.12 (7)	C11—C10—Mn1	70.99 (14)
C11—Mn1—C10	37.71 (9)	C9—C10—Mn1	133.6 (2)
C4—Mn1—Co1	155.64 (10)	Co1—C10—Mn1	79.09 (9)
C5—Mn1—Co1	97.86 (9)	C10—C11—C12	134.4 (2)
C11—Mn1—Co1	48.86 (7)	C10—C11—Co1	69.84 (15)
C10—Mn1—Co1	48.63 (7)	C12—C11—Co1	135.0 (2)
C9—O8—C8	116.2 (3)	C10—C11—Mn1	71.30 (15)
C12—O10—C13	115.7 (2)	C12—C11—Mn1	138.7 (2)
O9—C9—O8	123.5 (3)	Co1—C11—Mn1	79.02 (8)
O9—C9—C10	125.3 (3)	O11—C12—O10	125.2 (2)
O8—C9—C10	111.3 (2)	O11—C12—C11	122.9 (2)
C11—C10—C9	135.7 (2)	O10—C12—C11	111.9 (2)
C11—C10—Co1	70.44 (15)		

### Compound (6)

#### Crystal data

[MoCo(C <sub>5</sub> H <sub>6</sub> O <sub>2</sub> )(CO) <sub>7</sub> ]	Mo Kα radiation
<i>M<sub>r</sub></i> = 408.04	λ = 0.71073 Å
Triclinic	Cell parameters from 5750 reflections
<i>P</i> 1	θ = 2.55–28.28°
<i>a</i> = 8.0260 (1) Å	μ = 1.937 mm <sup>-1</sup>
<i>b</i> = 8.1299 (1) Å	<i>T</i> = 223 (2) K
<i>c</i> = 12.6612 (1) Å	Block
α = 87.343 (1)°	0.35 × 0.35 × 0.30 mm
β = 71.850 (3)°	Red
γ = 79.125 (1)°	
<i>V</i> = 770.879 (15) Å <sup>3</sup>	
<i>Z</i> = 2	
<i>D<sub>x</sub></i> = 1.758 Mg m <sup>-3</sup>	
<i>D<sub>m</sub></i> not measured	

#### Data collection

SMART CCD diffractometer	3048 reflections with <i>I</i> > 2σ( <i>I</i> )
ω scans	<i>R</i> <sub>int</sub> = 0.026
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	θ <sub>max</sub> = 28.28°
<i>T</i> <sub>min</sub> = 0.551, <i>T</i> <sub>max</sub> = 0.594	<i>h</i> = -10 → 10
8678 measured reflections	<i>k</i> = -10 → 10
3754 independent reflections	<i>l</i> = -16 → 16
	Intensity decay: none

#### Refinement

Refinement on <i>F</i> <sup>2</sup>	(Δ/σ) <sub>max</sub> = 0.001
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.029	Δρ <sub>max</sub> = 0.57 e Å <sup>-3</sup>
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.073	Δρ <sub>min</sub> = -0.30 e Å <sup>-3</sup>
<i>S</i> = 1.052	Extinction correction: none
3754 reflections	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
208 parameters	
H atoms constrained	
<i>w</i> = 1/[σ <sup>2</sup> ( <i>F</i> <sub>o</sub> <sup>2</sup> ) + (0.0388 <i>P</i> ) <sup>2</sup> + 0.4726 <i>P</i> ]	
where <i>P</i> = ( <i>F</i> <sub>o</sub> <sup>2</sup> + 2 <i>F</i> <sub>c</sub> <sup>2</sup> )/3	

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (6)

Co1—C5	1.808 (2)	Mn1—C4	1.861 (2)
Co1—C6	1.813 (2)	Mn1—C10	2.050 (2)
Co1—C7	1.822 (2)	Mn1—C9	2.107 (2)
Co1—C9	1.941 (2)	O8—C11	1.196 (2)
Co1—C10	1.970 (2)	O9—C11	1.331 (3)
Co1—Mn1	2.5468 (4)	O9—C12	1.456 (3)
Mn1—C1	1.814 (2)	C8—C9	1.494 (3)
Mn1—C3	1.838 (2)	C9—C10	1.328 (3)
Mn1—C2	1.844 (2)	C10—C11	1.474 (3)
C9—Co1—C10	39.68 (8)	C8—C9—Mn1	135.5 (2)
C9—Co1—Mn1	53.98 (6)	Co1—C9—Mn1	77.84 (7)
C10—Co1—Mn1	52.10 (5)	C9—C10—C11	133.3 (2)
C10—Mn1—C9	37.22 (7)	C9—C10—Co1	68.99 (12)
C10—Mn1—Co1	49.32 (5)	C11—C10—Co1	136.20 (14)
C9—Mn1—Co1	48.17 (5)	C9—C10—Mn1	73.73 (12)
C11—O9—C12	115.9 (2)	C11—C10—Mn1	137.82 (14)
C10—C9—C8	138.1 (2)	Co1—C10—Mn1	78.59 (7)
C10—C9—Co1	71.34 (11)	O8—C11—O9	124.5 (2)
C8—C9—Co1	136.5 (2)	O8—C11—C10	123.7 (2)
C10—C9—Mn1	69.05 (11)	O9—C11—C10	111.8 (2)

Methyl H-atom positions for both (5) and (6) were calculated ( $C-H = 0.96$  and  $0.97 \text{\AA}$ , respectively) after the location of at least one H atom from the electron-density map. Three of the four methyl groups have disordered H-atom positions and their occupancies were fixed at 0.5. All H atoms were treated with a riding model and the  $U_{iso}$  value was defined as  $1.5U_{eq}(C)$ .

For both compounds, data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structures: *SHELXTL* (Siemens, 1995); program(s) used to refine structures: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Data was collected through the Ohio Crystallographic Consortium, funded by the Ohio Board of Regents 1995 Investment Fund (CAP-075), located at the University of Toledo, Instrumentation Center in A&S, Toledo, Ohio. JAKB sincerely thanks Dr Ewa Skrzypczak-Jankun (University of Toledo) for assistance in using the CCD diffractometer system. KB gratefully acknowledges a Millennium Petrochemicals graduate fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1210). Services for accessing these data are described at the back of the journal.

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## [[ $\mu$ -Bis(salicylidene)-1,3-propanediaminato]-bis(4-methylpyridine)nickel(II)] dichloro-zinc(II)†

ORHAN ATAKOL,<sup>a</sup> FILIZ ERCAN,<sup>b</sup> CENGİZ ARICI,<sup>b</sup> HARTMUT FUESS<sup>c</sup> AND INGRID SVOBODA<sup>c</sup>

<sup>a</sup>Department of Chemistry, Ankara University, Tandogan 06100, Ankara, Turkey, <sup>b</sup>Department of Physics Engineering, Hacettepe University, Beytepe 06532, Ankara, Turkey, and <sup>c</sup>Strukturforschung, FB Materialwissenschaft, Technische Hochschule Darmstadt, Petersensstraße 23, D-64287 Darmstadt, Germany. E-mail: filiz@eti.cc.hun.edu.tr

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## Abstract

The title compound,  $[\text{Ni}(\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_2)(\text{C}_6\text{H}_7\text{N})_2\text{-}\{\text{ZnCl}_2\}]$ , is a heteronuclear dimeric complex. The Ni atom has an irregular octahedral environment involving two O atoms and two N atoms from the bis(salicylidene)-1,3-propanediaminate ligand in the equatorial plane, and two N atoms from the two 4-methylpyridine groups in the apical positions. The Ni—O—Zn, O—Zn—O, Zn—O—Ni and O—Ni—O angles in the bridged plane are  $99.58(9)$ ,  $80.59(8)$ ,  $100.57(9)$  and  $79.26(8)^\circ$ , respectively. The Ni···Zn distance is  $3.0896(9) \text{\AA}$ .

## Comment

Dinuclear homo- or heterometal complexes with double oxygen bridges have subnormal magnetic moments due to super-exchange interactions between the bridged metal ions. The magnetic interactions depend on the immediate environment of the bridge, as well as on the ligand arrangement about the metal ions. Structural details of doubly oxygen-bridged homonuclear dimeric copper(II) complexes have been reported previously (Kato *et al.*, 1964; Barclay & Hoskins, 1965; Butcher & Sinn, 1976; Kato & Muto, 1988). Dinuclear and trinuclear homo- or heterometal complexes based on Schiff base

† Systematic name: dichloro- $2\kappa^2\text{-Cl}$ -bis(4-methylpyridine- $1\kappa\text{N}$ )- $\mu$ - $\{2,2'\text{-}[1,3\text{-propanediy]bis(nitrilomethylidyne)diphenolato}\}$ - $1\kappa^4\text{O},\text{N},\text{N}',\text{O}'$ : $2\kappa^2\text{O},\text{O}'$ -nickel(II)zinc(II).