## **Interaction of Acyclic Tetraenes and Trienes with Transition Metals: Organonickel Compounds**

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Received December 13, 1994<sup>®</sup>

The tetraene 3,6-dimethylene-1,7-octadiene (DMOD) reacts with zerovalent [NiPR<sub>3</sub>] species to give  $(\eta^2, \eta^2$ -DMOD)NiPR<sub>3</sub> in which the methylene group of one 1,3-diene fragment and the vinyl group of the second interact with the metal atom. This unsymmetrical complexation of the tetraene has been confirmed by X-ray diffraction for the  $P(OC_6H_4$ -o-Ph)<sub>3</sub>-stabilized derivative (space group  $P2_1/c$ , a = 10.057(1) Å, b = 36.196(1) Å, c = 10.944(1) Å,  $\alpha = \gamma =$ 90°,  $\beta = 105.76(1)^\circ$ , Z = 4, R = 0.056,  $R_w = 0.071$ ) and is the result of the preferred coordination of a 1,6-heptadiene chain to the metal atom. The NMR spectra indicate that the molecule is fluxional with a pairwise exchange of the methylene groups and of the vinyl groups. The structure of the product formed in the presence of the bidentate ligands  $Pr_{i_2}^{i_2}P(CH_2)_n PPr_{i_2}^{i_2}$  (n = 1, 2, 3) is dependent upon the length of the chain bridging the two P-atoms, and compounds have been isolated having the composition  $(\eta^2, \eta^2, \eta^2, \eta^2, DMOD)Ni_2(Pri_2 PCH_2PPr_{2}^{i}$ ,  $(\eta^4, \eta^4-DMOD)[Ni(Pr_{2}^{i}PC_2H_4PPr_{2}^{i})]_2$ , and  $(\eta^2, \eta^2-DMOD)[Ni(Pr_{2}^{i}PC_3H_6PPr_{2}^{i})]_2$ . The molecular structure of the bis(diisopropylphosphino)ethane-containing compound has been established by X-ray diffraction (space group C2/c, a = 17.535 (2) Å, b = 24.152 (1) Å, c =11.767(1) Å,  $\alpha = \gamma = 90^{\circ}$ ,  $\beta = 120.65(1)^{\circ}$ , Z = 4, R = 0.034,  $R_{\rm w} = 0.035$ ) and confirms that the two 1,3-diene fragments of the tetraene interact with  $[Ni(Pr_{2}PC_{2}H_{4}PPr_{2})]$  moieties. The trienes 3-methylene-1,6-heptadiene and 3-methylene-1,7-octadiene react with  $[NiPPr_{3}]$ species to give  $(\eta^2, \eta^2$ -triene)NiPPr<sup>i</sup><sub>3</sub> compounds in which a 1,6-heptadiene chain is complexed to the metal atom.

## Introduction

In the preceding publication,<sup>1</sup> we have shown that the tetraene 3.6-dimethylene-1.7-octadiene (DMOD) interacts with zerovalent [FePR<sub>3</sub>] species in an  $\eta^4, \eta^4$ -manner whereas 3-methylene-1,6-heptadiene (MHD) and 3methylene-1,7-octadiene (MOD) form compounds containing an  $\eta^2$ ,  $\eta^4$ -bonded triene molecule. Interesting as these compounds may be, their predictive value as model intermediates for potential zerovalent ironcatalyzed reactions is limited by the paucity of reactions involving this metal. The situation in the case of nickel is, however, completely different, and a gamut of catalytic reactions involving 1,3-dienes have been reported<sup>2</sup> suggesting that a study of the stoichiometric behavior of DMOD would be of considerable interest.

Although the nickel-catalyzed cyclooligomerization and cyclo-cooligomerization of 1,3-butadiene has been extended to methyl-substituted 1,3-dienes such as isoprene and piperylene,<sup>2</sup> the investigation of substrates containing two 1,3-diene fragments is limited to the 1.1'- and 1.2'-bridged tetraenes discussed in the Introduction of the preceding paper<sup>1</sup> and, as far as we are

aware, reactions involving 2,2'-bridged species such as DMOD have not been reported. Here we describe the stoichiometric reactions between DMOD and ligandmodified zerovalent nickel species and the extension to the trienes MHD and MOD.

## **Results and Discussion**

Reactions of 3,6-Dimethylene-1,7-octadiene (D-MOD). Zerovalent nickel complexes such as (cdt)Ni or  $(cod)_2Ni$  (cdt = 1,5,9-cyclododecatriene, cod = 1,5cyclooctadiene) react with DMOD at -10 °C in diethyl ether with decomposition and formation of a nickel mirror. In contrast, in the presence of a monodentate P-donor ligand, (cod)<sub>2</sub>Ni reacts readily to give the yellow compounds 1-3 in high yield (eq 1). The phosphitestabilized derivative is stable at room temperature whereas the trialkylphosphine derivatives should be stored at -30 °C.

$$(\text{cod})_{2}\text{Ni} + \text{PR}_{3} + \text{DMOD} \xrightarrow{-2\text{cod}} (\eta^{2}, \eta^{2} - \text{DMOD})\text{NiPR}_{3} \quad (1)$$

$$\mathbf{1}, \mathbf{R} = \text{Pr}^{i}; \mathbf{2}, \mathbf{R} = \text{Cy};$$

$$\mathbf{3}, \mathbf{R} = \text{OC}_{6}\text{H}_{4}\text{-}o\text{-Ph}$$

The IR spectra of 1-3 indicate the presence of uncomplexed double bonds ( $\nu(C:C)$  ca. 1600 cm<sup>-1</sup>), and this is confirmed by the NMR spectroscopic results discussed below. The DMOD molecule could complex to the [NiPR<sub>3</sub>] fragment in an  $\eta^2, \eta^4$ -manner to give an 18-electron system or in an  $\eta^4$ - or  $\eta^2, \eta^2$ -manner to give a 16-electron system. Both electronic configurations

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 <sup>a</sup> Abstract published in Advance ACS Abstracts, March 15, 1995.
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Figure 1. Molecular structures of (top)  $(\eta^2, \eta^2$ -DMOD)NiP(OC<sub>6</sub>H<sub>4</sub>-o-Ph)<sub>3</sub> (3) and (bottom)  $(\eta^4, \eta^4$ -DMOD)[Ni(Pri<sub>2</sub>PC<sub>2</sub>H<sub>4</sub>-PPri<sub>2</sub>)]<sub>2</sub> (4).

have been observed in relevant examples, e.g. (cdt)-NiPR<sub>3</sub> and (1,5-hexadiene)NiPR<sub>3</sub>.<sup>4,5</sup> The crystal structure of the tris(o-phenylphenyl)phosphite derivative **3** has been determined by X-ray diffraction and establishes the  $\eta^2, \eta^2$ -complexation of the DMOD molecule.

The molecular structure of 3 is shown in Figure 1, and selected structural parameters are listed in Table 1. The nickel atom lies in a trigonal environment and is bonded to the P-atom of the phosphite as well as to the methylene group (C9-C10) of one 1,3-diene fragment and to the vinyl group (C1-C2) of the second. The maximum deviation of these atoms from the plane formed by these atoms and the Ni-atom is 0.075 Å. The

Table 1. Selected Structural Parameters for  $(\eta^2, \eta^2$ -DMOD)NiP(OC<sub>6</sub>H<sub>4</sub>-o-Ph)<sub>3</sub> (3)

bond lengths (Å)		bond angles (deg)	
Ni-P	2.090(1)	C1-Ni-C2	39.9(1)
Ni-C1	2.015(3)	C9-Ni-C10	40.5(1)
Ni-C2	2.017(3)	C1-C2-C3	124.7(3)
Ni-C9	2.060(3)	C2-C3-C5	116.8(3)
Ni-C10	1.997(3)	C2-C3-C4	122.2(4)
C1-C2	1.376(5)	C3-C5-C6	113.3(3)
C9-C10	1.406(5)	C4-C3-C5	120.9(4)
C2-C3	1.464(5)	C5-C6-C9	116.5(3)
C3-C4	1.335(6)	C6-C9-C8	118.8(3)
C3-C5	1.485(6)	C6-C9-C10	119.6(3)
C5-C6	1.517(5)	C9-C8-C7	128.5(3)
C6-C9	1.509(5)	Ni-P-O1	121.5(1)
C9-C8	1.456(5)	Ni-P-O2	116.5(1)
C7-C8	1.317(5)	Ni-P-O3	118.1(1)
Ni-D1	1.902	P-Ni-D1	116.3
Ni-D2	1.894	P-Ni-D2	115.1
		D1-Ni-D2	128.2

<sup>(4)</sup> Henc, B.; Jolly, P. W.; Salz, R.; Stobbe, S.; Wilke, G.; Benn, R.; Mynott, R.; Seevogel, R.; Goddard, R.; Krüger, C. J. Organomet. Chem. **1980**, 191, 449.

<sup>(5)</sup> Michaelis, S. Doctoral Thesis, Ruhr-Universität Bochum, 1991.

Ni-bonded 1,6-heptadiene chain adopts a chair conformation. The uncomplexed methylene group (C3-C4)is cis to the complexed vinyl group (C1-C2) with a dihedral angle between C1/C2/C3/C4 of 27° whereas the uncomplexed vinyl group (C7-C8) is trans to the methylene group (C9-C10) with a dihedral angle between C7/C8/C9/C10 of 102°. The encapsulation of the Ni-atom by the biphenyl groups can be clearly seen in Figure 1 and will account in part for the stability of the compound in comparison with the trialkylphosphine derivatives 1 and 2. The distance between the Ni-atom and the center of the closest phenyl ring, however, is 4.16 Å, so that it is unlikely that a direct interaction occurs between the metal atom and  $\pi$ -electrons of the ring. The preferred unsymmetrical complexation of the tetraene to the Ni-atom has its origin in the observation by Pörschke et al. that the  $\eta^2, \eta^2$ -complexation of 1,6heptadiene to zerovalent nickel is more favorable than that of 1.5-hexadiene<sup>5,6</sup> or of 1.7-octadiene which fails to form complexes.<sup>7</sup> This stability is associated with the strain-free, chair conformation adopted by the 1,5heptadiene fragment and the resulting planar arrangement of the Ni-atom and alkene groups. Moreover, the angle of 128.2° between the midpoints of the double bonds and the nickel atom (D1-Ni-D2) is similar to that observed in (CH<sub>2</sub>:CH<sub>2</sub>)<sub>2</sub>NiPCy<sub>3</sub>.<sup>8</sup>

The NMR spectroscopic data for 1-3 (Tables 2 and 3) indicate that the complexes have the same structure in solution as in the crystal. For example, the <sup>13</sup>C-NMR data for 1 and 2 at -70 °C show the expected magnetic inequivalence of the ten C-atoms of the tetraene and confirm that one methylene group and one vinyl group interact with the metal atom. At higher temperatures, the molecules are fluxional and a simplification of the spectra occurs. For example, the <sup>1</sup>H-NMR spectrum of **3** at room temperature contains only 6 signals for the 14 H-atoms of the tetraene, none of which have chemical shifts typical for an uncomplexed double bond: H-2 which is found at 6.3 ppm in the free tetraene is shifted to 4.2 ppm, while H-1 and H-4 which are found at ca. 5 ppm in the free tetraene are shifted to 3.5-3.1 ppm (Table 2). Extensive correlation and NOESY experiments show that the fluxionality arises from the pairwise exchange of the two methylene groups and of the two vinyl groups. This could proceed by a stepwise, intramolecular exchange of the alkene groups, and a simplified sequence involving the intermediate formation of 18-electron species is shown as Scheme 1.

An indication that relatively stable intermediates are involved in this process is obtained from the lowtemperature <sup>31</sup>P-NMR spectra of **3**: at -80 °C two signals are observed at 165.1 and 152.5 ppm (ratio 1:5). Unfortunately, the <sup>1</sup>H- and <sup>13</sup>C-NMR spectra of **3** are relatively uniformative and are broad even at -100 °C.

The reaction of DMOD with zerovalent nickel in the presence of a bidentate phosphine takes a completely different course. Initially, the behavior of bis(diisopropylphosphino)ethane was investigated and since (cod)<sub>2</sub>Ni forms a stable complex with this ligand, use was made of the more reactive (cdt)Ni. The reaction with DMOD (eq 2) led to the formation of a red compound 4 in high yield. The compound is surprisingly stable and melts

		-	Table 2. H-NMR Spectrosco	ppic Data for the (DMOD)Ni Co	mpounds <sup>a</sup>	
				$\delta(\mathbf{H}_n m) (J, \mathbf{H}_Z)$		i
	$(\eta^2, \eta^2$ -DMOD)NiPPr <sup>1</sup> <sub>3</sub> (1) <sup>b</sup>	$(\eta^2, \eta^2$ -DMOD)NiPCy <sub>3</sub> (2) <sup>b</sup>	$(\eta^2, \eta^2$ -DMOD)NiP(OC <sub>6</sub> H <sub>4</sub> -o-Ph) <sub>3</sub> (3) <sup>c1</sup>	$(\eta^4, \eta^4$ -DMOD)[Ni(Pr <sup>1</sup> <sub>2</sub> PC <sub>2</sub> H <sub>4</sub> PPr <sup>1</sup> <sub>2</sub> )] <sub>2</sub> (4) <sup>d</sup>	$(\eta^2,\eta^2$ -DMOD)[Ni(Pri <sub>2</sub> PC <sub>3</sub> H <sub>6</sub> PPri <sub>2</sub> )] <sub>2</sub> (5) <sup>c</sup>	(η <sup>2</sup> ,η <sup>2</sup> ,η <sup>2</sup> ,η <sup>2</sup> -DMOD)Ni <sub>2</sub> (Pr <sup>i</sup> <sub>2</sub> PCH <sub>2</sub> PPr <sup>i</sup> <sub>2</sub> (6) <sup>e</sup>
IZ IE	2:49 dd (1Z,2 12.9; 1Z,P 9.2) 1.95 dd (1E.2 8.8: 1E.P 2)	~1.90 00.1~	3.46 dd (1Z,2 15.3; 1Z,P 3.9) 3.09 dd (1E.2 9.9; 1E.P –)	2.23 dq (1Z,2 12.0; 1Z,1E 1.7) 2.48 dq (1E.2 8.5; 1E.P 1.7)	2.35 dbr (1Z,2 12.0) 2.05 dbr (1E.2 9.7)	ų
5	3.39 ddd (2,P 5.0)	~3.36	4.24 ddd (2,P 1.7)	4.63 dd	3.81 dd	5.27 ddbr
4Z	4.21 s	4.21 s	3.04 d (4Z,P 5.9)	3.30 s	$4.73 \text{ sbr}^{\kappa}$	
4E	4.44 s	4.42 s	3.34 s (4E,P 2.2)	3.61 sbr	4.63 sbr <sup>s</sup>	
5a/5b	3.02 dt/2.15 dd (5a,5b 13.0)	3.02 dt/2.14	2.05 br	2.70 s	2.59 s	
6a/6h	2.83 dt/1.22 (6a,6b 14.2; 6a,P 5.0)	2.84/1.24				
ZL	4.94 d (7Z,8 17.5)	4.93 d (7Z,8 17.3)				
ТE	4.73 d (7E,8 10.9)	4.72 d (7E,8 10.9)				
×	5.53 dd	5.51 dd				
10Z	2.13 d (10Z,P 4.8) 2.26 d (10E,P 9.5)	2.05 2.05				
Misc.	2.33/1.20 (Pr)	2.14/1.80-1.29 (Cy)	7.41–6.74 (Ar)	2.70-0.86 (Pr <sup>1</sup> <sub>2</sub> PC <sub>2</sub> H4PPr <sup>1</sup> <sub>2</sub> )	1.95-0.80 (Pri <sub>2</sub> PC <sub>3</sub> H <sub>6</sub> PPri <sub>2</sub> )	1.57 t/1.35-1.00 m (Pr <sup>1</sup> 2PCH <sub>2</sub> PPr <sup>1</sup> 2)
n	$^{h} d_{8}^{-}$ THF, $-70$	<sup>2</sup> C. <sup>c</sup> d <sub>8</sub> -toluene, 27 <sup>o</sup> C. <sup>c</sup>	$^{1}$ $d_{8}$ -toluene, 60 °C. ' $d_{8}$ -THF, 27 °C	C. <sup>J</sup> J(5a, 6a) 5.0, J(5a, 6b) 12.8, J(5b	, 6a) 0, J(5b, 6b) 4.2. <sup>s</sup> Assignment co	ould be reversed. <sup>4</sup> Unassigned abs at d
ш						

3.45-3.20 m, 2.83 d, 2.68 m, 2.45 s, 2.27 m, 2.05-1.80 m.<sup>1</sup> Molecule fluxional

<sup>(6)</sup> Proft, B.; Pörschke, K.-R.; Lutz, F.; Krüger, C. Chem. Ber. 1991, 124, 2667.

<sup>(7)</sup> Pörschke, K.-R. Private communication, 1994.

<sup>(8)</sup> Krüger, C.; Tsay, Y.-H. J. Organomet. Chem. 1972, 34, 387.

			$\delta(\mathbf{C}_n) (J(\mathbf{C},\mathbf{P}) \mathbf{H}\mathbf{z})$	
	$\overline{(\eta^2,\eta^2-\text{DMOD})\text{NiPPr}^{i_3}}_{(1)^b}$	$(\eta^2, \eta^2$ -DMOD)NiPCy <sub>3</sub> (2) <sup><i>i</i></sup>	$(\eta^4.\eta^4\text{-}DMOD)[\text{Ni}(\text{Pr}^i_2\text{PC}_2\text{H}_4\text{PPr}^i_2)]_2$ (4) <sup>c</sup>	$(\eta^2, \eta^2, \eta^2, \eta^2 \text{-DMOD})\text{Ni}_2(\text{Pr}^{i_2}\text{PCH}_2\text{PPr}^{i_2})$ (6) <sup>c</sup>
1	44.4	44.3	41.8 (6.2)	d
2	76.4 (7.9)	76.5 (6.7)	74.5	80.9 (10.5)
3	152.2 (3.7)	152.2 (3.6)		100.0 (10.5/2.5)
4	100.3	100.0	66.7	d
5	37.9 (4.2)	37.8	43.2	d
6	39.4 (2.2)	39.3		
7	104.8 (2.2)	104.6		
8	144.8 (4.0)	144.2 (3.7)		
9	77.7 (9.6)	77.9		
10	58.0	58.0		
Misc.	26.2/20.2 (Pr <sup>i</sup> )	37.6-27.6 (Cy)	$26.3 - 18.2 (Pr_{2}PC_{2}H_{4}PPr_{2}^{i})$	27.4-14.2 (Pr <sup>i</sup> <sub>2</sub> PCH <sub>2</sub> PPr <sup>i</sup> <sub>2</sub> )
a	$^{h} d_{8}$ -THF, -7	0 °C. <sup>c</sup> d <sub>8</sub> -THF, 27 °C. <sup>d</sup> Un	assigned abs for $C_1/C_4/C_5$ at 43.3(3.0)/44.1	(2.0)/54.5.

Scheme 1



$$2(cdt)Ni + DMOD + 2Pr_{2}^{i}PC_{2}H_{4}PPr_{2}^{i} \xrightarrow{-2cdt} (\eta^{4}, \eta^{4}-DMOD)[Ni(Pr_{2}^{i}PC_{2}H_{4}PPr_{2}^{i})]_{2} (2)$$

$$4$$

at 140 °C without decomposition. The elemental analysis and mass spectrum indicated that a binuclear species has been formed. The IR and NMR spectra suggest that all four double bonds of the tetraene interact with the metal atoms, while the NMR spectra suggest that the molecule is symmetrical. Repeated recrystallization from toluene led to the isolation of red needles (in addition to an amorphous powder) suitable for an X-ray diffraction study, and the crystal structure determination established the  $\mu$ - $\eta^4$ , $\eta^4$ -complexation of the tetraene to the two nickel atoms.

The molecular structure of **4** is shown in Figure 1, and selected structural parameters are listed in Table 4. The nickel atom lies at the center of a distorted tetrahedron and is bonded to the P-atoms of the bidentate ligand and to one of the 1,3-diene fragments. The molecule has a center of inversion at the middle point of  $C5-C5^*$ .

Table 4. Selected Structural Parameters for  $(\eta^4, \eta^4$ -DMOD)[Ni(Pri<sub>2</sub>PC<sub>2</sub>H<sub>4</sub>PPri<sub>2</sub>)]<sub>2</sub> (4)

bond lengths (Å)		bond angles (deg)		
Ni-P1	2.128(1)	C1-C2-C3	122.0(3)	
Ni-P2	2.156(1)	C2-C3-C4	118.7(3)	
Ni-C1	2.155(3)	C2-C3-C5	119.6(2)	
Ni-C2	2.025(2)	C4-C3-C5	121.8(3)	
Ni-C3	2.068(2)	C3-C5-C5*	112.2(2)	
Ni-C4	2.183(3)	P1-Ni-P2	92.8(1)	
C1-C2	1.379(5)	D1-Ni-D2	64.6	
C2-C3	1.434(4)	D1-Ni-P1	126.2	
C3-C4	1.377(4)	D1-Ni-P2	125.8	
C3-C5	1.503(4)	D2-Ni-Pl	126.8	
C5-C5*	1.531(3)	D2-Ni-P2	123.8	
Ni-D1	1.974			
Ni-D2	2.012			

Although the presence of an inversion center can be expected to lead to a simplification of the NMR spectra, that for the <sup>31</sup>P nuclei is unexpectedly complex and consists at -80 °C of an apparent triplet at ca. 98.0 ppm and a double doublet at ca. 54.8 ppm. This spectrum arises from the overlap of two AB-spin systems having very similar chemical shifts and identical coupling constants and intensities and suggests that the molecule is formed as diastereomers. These could be associated either with the arrangement of the ethano bridge in the bidentate ligand with respect to the 1,3-diene fragment or alternatively be the result of the presence of two chiral centers  $(C3/C3^*)$  in the molecule. The first possibility has been discussed for  $(\eta^4-1,3$ -butadiene)Ni- $(Cy_2PC_2H_4PCy_2)^9$  and has been shown to exist in the crystal but is not maintained in solution. The crystal structure of 4 is of the meso-form (4a; RS/SR), but in addition a racemic form is possible (4b; RR/SS; only the SS-form is shown) which differs only in the mode of complexation of a 1,3-diene fragment. It is possible that the choice of a crystal having the meso structure was accidental or that the racemic form did not form suitable crystals and precipitated as an amorphous powder (see above).

The <sup>31</sup>P NMR spectra of **4** are temperature dependent: the two overlapping AB-systems observed at -80°C coalesce at room temperature to become two broad signals at room temperature and one sharp signal at +90 °C. This process, which is reversible, suggests that two consecutive processes occur: initially the P-atoms

 <sup>(9)</sup> Benn, R.; Betz, P.; Goddard, R.; Jolly, P. W.; Kokel, N.; Krüger,
 C.; Topalović, I. Z. Naturforsch. 1991, 46b, 1395.

Scheme 2





exchange and this is followed by diastereomer exchange. Any mechanism explaining these effects must involve side exchange of the metal atom at the 1,3-diene fragment. A possibility which accounts for both effects is shown as Scheme 2 and involves the intermediate formation of an  $\eta^2$ -bonded diene fragment followed by isomerization from the *cis* to a *trans* configuration. This mechanism, which has precedence in the literature,<sup>6</sup> avoids the previously favored<sup>10</sup> rotation of the  $\eta^2$ -bonded

diene fragment about the Ni-alkene axis which is now believed to be energetically unlikely in these systems. An alternative mechanism involving the isomerization of the  $(\eta^4$ -1,3-diene)Ni fragment to a nickel cyclopentene ring followed by ring inversion can be envisaged and has been invoked for Zr-containing species<sup>11</sup> but is without precedence in organonickel chemistry. Unfortunately, further information could not be obtained from the <sup>1</sup>H- and <sup>13</sup>C-NMR spectra since the relative insolubility of the compound precluded the measurement of low-temperature spectra.

Changing the length of the chain between the two phosphorus atoms in the bidentate ligand has a remarkable effect upon the course of the reaction between DMOD and zerovalent nickel.

The product of the reaction in the presence of bis-(diisopropylphosphino)propane is a yellow compound (5) whose mass spectrum indicates the composition (DMOD)-

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 $[Ni(Pr_{2}PC_{3}H_{6}PPr_{2})]_{2}$ . However, in contrast to 4 the IR spectrum suggests the presence of uncomplexed double bonds ( $\nu$ (C:C) 1590, 885 cm<sup>-1</sup>), and this is supported by the <sup>1</sup>H-NMR spectrum in which signals at 4.6 and 4.7 ppm are assigned to the protons of the uncomplexed methylene groups (these are observed at 5.25 and 5.04 ppm in DMOD). On the basis of this evidence, a possible structure for 5 is shown in which the Ni-atoms are complexed to the vinyl groups and lie in a 16electron, trigonal planar environment:



In the presence of bis(diisopropylphosphino)methane, a completely different reaction occurs: the product of the reaction between (cdt)Ni, the bidentate ligand, and DMOD is a dark violet compound (6) which is stable at room temperature and whose mass spectrum and elemental analysis indicate the composition (DMOD)- $Ni_2(Pr_2PCH_2PPr_2)$ . The <sup>31</sup>P-NMR spectrum is that of an AB-spin system suggesting that the two P-atoms lie in different environments. The <sup>13</sup>C-NMR spectrum contains 5 signals attributable to the 10 C-atoms of the DMOD molecule indicating that the tetraene is bonded in a symmetrical manner to the two metal atoms. In addition, the <sup>13</sup>C-NMR spectrum contains a signal at 14.2 ppm which is assigned to the methylene C-atom of the bidentate ligand and is typical for systems in which the ligand bridges two metal atoms. This signal is shifted to lower field in species in which the ligand is acting in a chelating manner and, for example, appears at 33.5 ppm in  $(\eta^3$ -C<sub>3</sub>H<sub>5</sub>)<sub>2</sub>Fe(Pr<sup>i</sup><sub>2</sub>PCH<sub>2</sub>PPr<sup>i</sup><sub>2</sub>),<sup>12</sup> while it absorbs at 14.7 pppm in the free ligand. A structure which unites all these observations is shown below:



Popular prejudice would attribute the dark color of 6 to a metal-metal interaction similar to that observed for  $[(cyclooctatetraene)Ni]_2^{13}$  or  $[Pd(Pr^i_2PCH_2PPr^i_2)]_2$ ;<sup>14</sup> however, this must remain speculation since we were unable to grow crystals of a quality sufficient for an X-ray diffraction study.

The difference in behavior between the ethano- and propano-bridged bidentate ligands is presumably associated with the difference in their space-filling properties: models suggest that the Ni-atom in 3 is less shielded by the P-bonded isopropyl groups than in 5. The behavior of the methano-bridged bidentate ligand is certainly a result of the unfavorable bite angle in this ligand which leads to a destabilization of a chelating arrangement.

Reaction of 3-Methylene-1,6-heptadiene and 3-Methylene-1,7-octadiene with Zerovalent Nickel. In the hope of obtaining additional information on the mechanism of the rearrangement of the (DMOD)NiPR<sub>3</sub> species shown in Scheme 1, we prepared the complexes 7 and 8 by reacting  $(cod)_2Ni$  with PPr<sup>i</sup><sub>3</sub> and the trienes



3-methylene-1,6-heptadiene and 3-methylene-1,7-octadiene. In both cases, the NMR spectroscopic results (see Experimental Section) indicate that a 1,6-heptadiene chain is complexed in an  $\eta^2, \eta^2$ -manner to the metal atom. This is attained by the complexation of the terminal olefin and the vinyl group, in the case of 7, or the methylene group, in the case of 8, and underlines once again the special stability associated with the 1,6heptadiene chain.

Both 7 and 8 are more stable than the corresponding DMOD-containing species, but unfortunately, the NMR spectra of neither compound show any temperaturedependent effect between -80 and +60 °C.

## **Experimental Section**

The general experimental conditions and instrumentation have been described in the preceding publication.<sup>1</sup> (Cod)<sub>2</sub>Ni and (cdt)Ni were prepared according to published procedures.<sup>3</sup>

 $(\eta^2, \eta^2$ -DMOD)NiPPr<sup>i</sup><sub>3</sub> (1). DMOD (0.99 mL, 5.83 mmol) and PPri3 (1.12 mL, 5.80 mmol) was added to a suspension of (cod)<sub>2</sub>Ni (1.60 g, 5.82 mmol) in diethyl ether (200 mL) cooled to -78 °C. The mixture was warmed to -30 °C and stirred for 48 h. The resulting dark yellow solution was filtered through a pad of avicel at -30 °C in order to remove traces of precipitated nickel and concentrated to 50 mL. The solution was held at -78 °C for 1 week to give the compound as a bright yellow crystalline solid which was washed at -78 °C with precooled pentane  $(2 \times 10 \text{ mL})$ . The compound decomposes at room temperature and should be stored at -30 °C. Yield: 1.46 g (4.13 mmol, 71%). Anal. Calcd for  $C_{19}H_{35}NiP:\ C,\,64.6;$ H, 10.0; Ni, 16.6; P, 8.8. Found: C, 64.7; H, 10.1; Ni, 16.5; P, 8.7. IR (KBr): 2930 (s), 1610 (s), 1460 (s), 880 (s), 860 (s), 650 (s) cm<sup>-1</sup>. MS (90 °C): m/e 352 (M<sup>+</sup>, 66%), 218 (M<sup>+</sup> -DMOD, 18%), 192 ( $M^+ - PPr^i_3$ , 59%), 176 (100%). <sup>31</sup>P-NMR  $(d_8$ -THF, -70 °C):  $\delta$  52.7. <sup>1</sup>H- and <sup>13</sup>C-NMR spectroscopic data: see Tables 2 and 3.

 $(\eta^2, \eta^2$ -DMOD)NiPCy<sub>3</sub> (2) was prepared as described above by reacting PCy<sub>3</sub> as a yellow solid in 66% yield. Anal. Calcd for C<sub>28</sub>H<sub>47</sub>NiP: C, 71.1; H, 10.0; Ni 12.4; P, 6.5. Found: C, 71.0; H, 9.9; Ni, 12.3; P, 6.6. IR (KBr): 2920 (s), 1610 (s), 1490 (s) cm<sup>-1</sup>. MS (80 °C): m/e 472 (M<sup>+</sup>, 0.4%) (the compound decomposed during measurement). <sup>31</sup>P-NMR ( $d_8$ -THF, -70 °C):  $\delta$  42.0. <sup>1</sup>H- and <sup>13</sup>C-NMR spectroscopic data: see Tables 2 and 3.

 $(\eta^2, \eta^2$ -DMOD)NiP(OC<sub>6</sub>H<sub>4</sub>-o-Ph)<sub>3</sub> (3) was prepared as described above by reacting  $P(OC_6H_4-o-Ph)_3$  as a pale yellow crystalline solid in 41% yield. Anal. Calcd for C<sub>46</sub>H<sub>41</sub>O<sub>3</sub>NiP: C, 75.5; H, 5.7; Ni, 8.0; P, 4.2. Found: C, 75.7; H, 5.8; Ni, 7.9; P, 4.2. IR (KBr): 1475 (s), 1430 (s), 1190 (s), 1110 (s), 910 (s), 880 (s), 755 (s), 700 (s). MS (180 °C): m/e 730 (M<sup>+</sup>, 0.2%)

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Krüger, C. Inorg. Chim. Acta 1994, 222, 179.

Table 5. Crystallographic Data for 3 and 4

	3	4
formula	C <sub>46</sub> H <sub>41</sub> NiO <sub>3</sub> P	C <sub>38</sub> H <sub>78</sub> Ni <sub>2</sub> P <sub>4</sub>
mol wt	731.5	776.4
cryst size, mm	$0.39 \times 0.35 \times 0.42$	$0.53 \times 0.35 \times 0.25$
V, Å <sup>3</sup>	3834.1	4287.2
$D_c$ , g cm <sup>-3</sup>	1.27	1.20
T, °Č	20	20
λ(Cu), Å	1.541 78	1.541 78
Ζ	4	4
$\mu_{abs}$ , cm <sup>-1</sup>	5.86	26.75
refins measd	9483 $(\pm h, \pm k, \pm l)$	13 927 $(\pm h, \pm k, +l)$
ind reflns	7600	4410
obsd reflns	6325	3972
no. of variables	460	355
space group (No.)	$P2_1/c$ (14)	<i>C</i> 2/ <i>c</i> (15)
a, b, c, Å	10.057(1), 36.196(1),	17.535(2), 24.152(1),
	10.944(1)	11.767(1)
$\alpha, \beta, \gamma, \deg$	90.0, 105.76(1), 90.0	90.0, 120.65(1), 90.0
R	0.056	0.034
Rw	0.071	0.035
res electron	0.24	0.30
density, e Å <sup>-3</sup>		

(the compound decomposed during the measurement). <sup>31</sup>P-NMR ( $d_8$ -toluene): 27 °C,  $\delta$  152.0; -80 °C, 152.5, 165.1 (ratio 5:1). <sup>1</sup>H-NMR spectroscopic data: see Table 2. Crystal structure: see Figure 1 and Tables 1 and 5.

 $(\eta^4, \eta^4 - DMOD)[Ni(Pr_{2}^iPC_2H_4PPr_{2}^i)]_2$  (4). DMOD (0.56) mL, 3.30 mmol) and Pr<sup>i</sup><sub>2</sub>PC<sub>2</sub>H<sub>4</sub>PPr<sup>i</sup><sub>2</sub> (1.03 mL, 3.30 mmol) were added to a suspension of (cdt)Ni (0.83 g, 3.31 mmol) in diethyl ether (200 mL) cooled to -78 °C. The reaction mixture was allowed to warm to room temperature over 5 h. A yellow solid precipitated and then dissolved to give a red solution. The reaction mixture was stirred for 2 d, filtered through a pad of avicel, and evaporated to dryness. The residue was dissolved in toluene (50 mL) at 50 °C and filtered. Cooling the filtrate to -78 °C gave the compound as red needles which were washed with precooled pentane (10 mL) at -78 °C and dried in high vacuum. Yield: 1.09 g (1.40 mmol, 85%). Anal. Calcd for C<sub>38</sub>H<sub>78</sub>Ni<sub>2</sub>P<sub>4</sub>: Ni, 15.1; P, 16.0. Found: Ni, 14.7; P, 15.4 (satisfactory analytical data could not be obtained despite repeated recrystallization, however, in all cases the values correspond to a Ni:chelate phosphine:olefin ratio of 2:2:1). IR (KBr): 2980-2860 (s), 2460 (s), 1025 (s), 880 (s), 680 (s), 670 (s), 645 (s), 620 (s). MS (130 °C): m/e 774 (M<sup>+</sup>, 0.1%). <sup>31</sup>P-NMR (*d*<sub>8</sub>-toluene): 90 °C, δ 73.0; -80 °C, δ 98.3 (*J*(P,P) ca. 40 Hz), 97.8 (J(P,P) ca. 40 Hz), 55.0 (J(P,P) 41.5 Hz), 54.8 (J(P,P) 40.5 Hz) (the spectrum consists of two AB-spin systems in the ratio 1:1 which could not be assigned). <sup>1</sup>H- and <sup>13</sup>C-NMR spectroscopic data: see Tables 2 and 3. Crystal structure: see Figure 1 and Tables 4 and 5.

 $(\eta^2, \eta^2$ -DMOD)[Ni(Pri<sub>2</sub>PC<sub>3</sub>H<sub>6</sub>PPri<sub>2</sub>)]<sub>2</sub> (5) was prepared as described above by reacting Pri<sub>2</sub>PC<sub>3</sub>H<sub>6</sub>PPri<sub>2</sub> as a yellow powder in 53% yield. The substance could not be obtained analytically pure. IR (KBr): 2980-2860 (s), 1460 (s), 1025 (s), 885 (s). MS (180 °C): m/e 802 (M<sup>+</sup>, 5.8%). <sup>31</sup>P-NMR ( $d_8$ -toluene): 27 °C,  $\delta$  30.7; -80 °C, 30.5, 24.6 both as broad doublets. <sup>1</sup>H-NMR spectroscopic data: see Table 2.

 $(\eta^2, \eta^2, \eta^2, \eta^2$ -DMOD)Ni<sub>2</sub>(Pri<sub>2</sub>PCH<sub>2</sub>PPri<sub>2</sub>) (6). DMOD (0.17 mL, 1.00 mmol) and Pri<sub>2</sub>PCH<sub>2</sub>PPri<sub>2</sub> (0.55 mL, 1.84 mmol) were added to a suspension of (cdt)Ni (0.46 g, 1.83 mmol) in diethyl ether (200 mL) cooled to -78 °C. The reaction mixture was allowed to warm to room temperature over 5 h and the resulting dark red solution stirred for a further 2 d. The solution was filtered through a pad of avicel and evaporated to dryness, and the residue was dissolved in pentane (20 mL). The pentane solution was filtered and held at -78 °C for 5 d to give the compound as a dark-violet to black solid which was dried under high vacuum. Yield: 0.34 g (0.68 mmol, 74%). Anal. Calcd for C<sub>23</sub>H<sub>44</sub>Ni<sub>2</sub>P<sub>2</sub>: C, 55.3; H, 8.9; Ni, 23.5; P, 12.4. Found: C, 54.3; H, 8.6; Ni, 24.1; P, 12.9 (although the analytical results are not completely satisfactory, the Ni:P ratio is 1:1). IR (KBr): 2980-2840 (s), 1465 (s), 1385 (s), 1360 (s)

Table 6. Final Coordinates and Equivalent IsotropicThermal Parameters ( $Å^2$ ) of the Non-Hydrogen Atoms for  $3^a$ 

			1 0	
atom	x	у	z	$U_{ m eq}$
Ni1	0.0386(1)	0.1525(1)	0.4529(1)	0.048(1)
Р	-0.0719(1)	0.1086(1)	0.3486(1)	0.045(1)
O1	-0.0880(2)	0.0696(1)	0.4149(2)	0.053(1)
02	-0.0122(2)	0.0917(1)	0.2367(2)	0.055(1)
O3	-0.2317(2)	0.1152(1)	0.2746(2)	0.050(1)
<b>C</b> 1	-0.1275(3)	0.1853(1)	0.4368(3)	0.066(2)
C2	-0.0083(3)	0.2048(1)	0.4907(3)	0.060(2)
C3	0.0607(4)	0.2292(1)	0.4207(3)	0.076(2)
C4	-0.0074(5)	0.2463(1)	0.3126(4)	0.115(4)
C5	0.2130(4)	0.2331(1)	0.4710(4)	0.083(3)
C6	0.2883(3)	0.1964(1)	0.4696(3)	0.070(2)
C7	0.3345(3)	0.1919(1)	0.7521(3)	0.071(2)
C8	0.2696(3)	0.1659(1)	0.6737(3)	0.059(2)
C9	0.2433(3)	0.1644(1)	0.5368(3)	0.052(2)
C10	0.2254(3)	0.1294(1)	0.4798(3)	0.068(2)
C20	0.0048(3)	0.0602(1)	0.5311(3)	0.057(2)
C21	-0.0136(3)	0.0741(1)	0.6437(3)	0.062(2)
C22	0.0831(4)	0.0634(1)	0.7541(4)	0.090(3)
C23	0.1914(5)	0.0407(1)	0.7524(6)	0.120(4)
C24	0.2069(4)	0.0277(1)	0.6434(7)	0.111(4)
C25	0.1128(4)	0.0371(1)	0.5278(4)	0.081(3)
C26	-0.1294(3)	0.0988(1)	0.6487(3)	0.066(2)
C27	-0.1057(5)	0.1296(1)	0.7257(4)	0.095(3)
C28	-0.2126(7)	0.1527(1)	0.7329(6)	0.125(5)
C29	-0.3431(7)	0.1447(2)	0.6677(6)	0.123(5)
C30	-0.3697(4)	0.1148(2)	0.5898(5)	0.103(4)
C31	-0.2632(3)	0.0916(1)	0.5804(4)	0.076(2)
C40	0.0503(3)	0.1144(1)	0.1655(3)	0.056(2)
C41	0.1678(3)	0.1007(1)	0.1369(3)	0.066(2)
C42	0.2307(4)	0.1240(1)	0.0689(4)	0.086(3)
C43	0.1810(5)	0.1586(1)	0.0317(4)	0.101(4)
C44	0.0646(5)	0.1715(1)	0.0609(4)	0.091(3)
C45	-0.0015(3)	0.1489(1)	0.1277(3)	0.070(2)
C46	0.2290(4)	0.0648(1)	0.1857(4)	0.078(3)
C47	0.1550(5)	0.0323(1)	0.1699(5)	0.103(4)
C48	0.2156(7)	-0.0008(1)	0.2166(6)	0.126(5)
C49	0.3536(8)	-0.0008(2)	0.2838(7)	0.149(7)
C50	0.4278(6)	0.0304(2)	0.3014(7)	0.138(5)
C51	0.3680(4)	0.0628(1)	0.2529(5)	0.104(4)
C60	-0.3050(3)	0.0996(1)	0.1590(3)	0.050(2)
C61	-0.3813(3)	0.1239(1)	0.0683(3)	0.057(2)
C62	-0.4495(3)	0.1091(1)	-0.0492(3)	0.072(2)
C63	-0.4440(4)	0.0718(1)	-0.0729(4)	0.087(3)
C64	-0.3696(4)	0.0485(1)	0.0181(4)	0.086(3)
C65	-0.2993(3)	0.0623(1)	0.1363(3)	0.065(2)
C66	-0.3894(3)	0.1641(1)	0.0934(3)	0.061(2)
C67	-0.3455(4)	0.1902(1)	0.0212(4)	0.083(3)
C68	-0.3533(5)	0.2276(1)	0.0474(6)	0.108(4)
C69	-0.4056(5)	0.2387(1)	0.1396(7)	0.117(4)
C70	-0.4536(4)	0.2139(1)	0.2120(5)	0.097(3)
C71	-0.4436(3)	0.1767(1)	0.1902(4)	0.074(2)
		,		

 ${}^{a} U_{eq} = {}^{1}/_{3} \sum_{i} \sum_{j} U_{ij} a_{i} * a_{j} * \bar{a}_{i} \cdot \bar{a}_{j}.$ 

cm<sup>-1</sup>. MS (120 °C): m/e 498 (M<sup>+</sup>, 0.6%), 364 (M<sup>+</sup> – DMOD, 0.1%) (the sample decomposed during the measurement). <sup>31</sup>P-NMR ( $d_8$ -THF, 27 °C):  $\delta$  67.9, 72.0, J(P,P) 176.5. <sup>1</sup>H- and <sup>13</sup>C-NMR spectroscopic data: see Tables 2 and 3.

 $(\eta^2, \eta^2$ -MHD)NiPPr<sup>i</sup><sub>3</sub> (7). MHD (0.47 mL, 2.95 mmol) and PPri<sub>3</sub> (0.56 mL, 2.90 mmol) were added to a suspension of  $(cod)_2Ni$  (0.80 g, 2.91 mmol) in diethyl ether (80 mL) cooled to -78 °C. The reaction mixture was stirred at -30 °C for 4 d to give a red solution which was filtered through a pad of avicel at -30 °C and concentrated to ca. 20 mL. The solution was cooled to -78 °C for 14 d to give the compound as a yellow crystalline solid which was washed with precooled pentane (2  $\times$  10 mL) at -78 °C and dried under high vacuum. The compound is stable at room temperature. Yield: 0.64 g (1.96 mmol, 67%). Anal. Calcd for C<sub>17</sub>H<sub>33</sub>NiP: C, 62.4; H, 10.2; Ni, 17.9; P, 9.5. Found: C, 62.3; H, 10.2; Ni, 18.0; P, 9.4. IR (KBr): 3005-2870 (s), 1465 (s), 1180 (s), 885 (s), 655 (s). MS (80 °C): m/e 326 (M<sup>+</sup>, 45%), 218 (M<sup>+</sup> – MHD, 20%), 176 (60%), 134 (100%). <sup>31</sup>P-NMR (*d*<sub>8</sub>-toluene, 60 °C): 56.7. <sup>1</sup>H-NMR (*d*<sub>8</sub>toluene, -30 °C): δ 4.95 (s, H-4E), 4.63 (s, H-4Z), 4.15 (ddd,

H-2, J(2, 1Z) ca. 13.2 Hz, J(2, 1E) ca. 8.7 Hz, J(2, P) ca. 5.1 Hz, 3.56 (m, H-7), 2.90–2.30/2.05–1.95 (m, H-1/5/6/8), 1.90/0.94 (Pr<sup>i</sup>). <sup>13</sup>C-NMR ( $d_8$ -THF, 27 °C):  $\delta$  153.2 (C-3), 100.9 (C-4, J(C,P) 2.8), 70.6/66.9 (C-2/7, J(C,P) 9.4/8.5 Hz), 51.6 (C-1, J(C, P) 4.7 Hz), 41.0 (C-8, J(C, P) 5.1 Hz), 39.0/38.6 (C-5/6) 6, J(C, P) 5.3/2.8 Hz), 26.4/20.5 (Pr<sup>i</sup>). The numbering scheme is shown below:



 $(\eta^2, \eta^2$ -MOD)NiPPr<sup>i</sup><sub>3</sub> (8) was prepared in 64% yield as described above as a yellow-brown solid by reacting MOD. Anal. Calcd for C<sub>18</sub>H<sub>35</sub>NiP: C, 63.4; H, 10.3; Ni 17.2; P, 9.1. Found: C, 63.3; H, 10.4; Ni, 17.1; P, 9.2. IR (KBr): 3000-2800 (s), 1610 (s), 1455 (s), 650 (s), 530 (s). MS (80 °C): m/e $340\,(M^+,\,87\%),\,218\,(M^+-MOD,\,29\%),\,180\,(M^+-PPr^i{}_3,\,30\%),$ 176 (100%). <sup>31</sup>P-NMR ( $d_8$ -toluene, -30%):  $\delta$  53.1. <sup>1</sup>H-NMR (d<sub>8</sub>-toluene, 60 °C):  $\delta$  5.77 (dd, H-2), 4.96 (dt, H-1Z), 4.77 (dt, H-1E), 1.99/1.01/1.02 (Pri), J(1Z, 2) 17.4 Hz, J(1Z, 5) 1.8 Hz, J(1E, 2) 10.9 Hz; additional unassigned absorptions at  $\delta$  3.01/ 2.74/2.51-2.32/1.71/0.95-0.55. <sup>13</sup>C-NMR (d<sub>8</sub>-toluene, -30 °C):  $\delta$  144.1 (C-2, J(C, P) 3.6 Hz), 104.8 (C-1, J(C, P) ca. 2.7 Hz), 77.5 (C-8, J(C, P) 9.0 Hz), 73.9 (C-3, J(C, P) 10.2 Hz), 52.3/51.4 (C-4/9, J(C, P) 5.7/4.6 Hz), 34.1/32.5/28.8 (C-5/6/7, J(C, P) ca. 0/ca. 2.2/6.1 Hz), 25.5/10.9 (Pr<sup>i</sup>). The numbering scheme is shown below:



Table 7. Final Coordinates and Equivalent Isotropic Thermal Parameters ( $Å^2$ ) of the Non-Hydrogen Atoms for  $4^a$ 

		( ,		
atom	x	у	Z.	$U_{eq}$
Ni	0.2853(1)	0.4203(1)	0.2728(1)	0.032(1)
P1	0.1972(1)	0.3760(1)	0.0977(1)	0.038(1)
P2	0.3143(1)	0.3480(1)	0.3955(1)	0.036(1)
Cl	0.2256(2)	0.4993(1)	0.2646(4)	0.061(3)
C2	0.3098(2)	0.4927(1)	0.3720(2)	0.045(2)
C3	0.3841(2)	0.4794(1)	0.3576(2)	0.039(2)
C4	0.3703(2)	0.4725(1)	0.2323(3)	0.053(2)
C5	0.4743(2)	0.4727(1)	0.4786(3)	0.044(2)
C6	0.2060(2)	0.3019(1)	0.1427(3)	0.052(2)
C7	0.2373(2)	0.2930(1)	0.2872(3)	0.055(2)
C8	0.2153(2)	0.3748(1)	-0.0450(3)	0.053(2)
C9	0.3099(2)	0.3590(2)	-0.0020(4)	0.068(3)
C10	0.1506(3)	0.3393(2)	-0.1615(4)	0.083(3)
C11	0.0759(2)	0.3898(1)	0.0136(3)	0.063(2)
C12	0.0446(2)	0.3881(2)	0.1141(5)	0.085(4)
C13	0.0532(3)	0.4447(2)	-0.0591(4)	0.085(3)
C14	0.2929(2)	0.3456(1)	0.5359(3)	0.052(2)
C15	0.3665(3)	0.3723(2)	0.6590(3)	0.072(3)
C16	0.2051(3)	0.3735(2)	0.4957(4)	0.073(3)
C17	0.4263(2)	0.3151(1)	0.4707(3)	0.050(2)
C18	0.4531(2)	0.3091(2)	0.3664(4)	0.069(3)
C19	0.4379(3)	0.2603(2)	0.5413(5)	0.083(4)

"  $U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \overline{a}_i \cdot \overline{a}_j$ .

**Single-Crystal X-ray Diffraction Studies of 3 and 4.** The crystal structure analyses were carried out using an Enraf-Nonius CAD-4 diffractometer. Crystallographic data and details of the refinements are listed in Table 5, and final coordinates and equivalent isotropic thermal parameters are in Tables 6 and 7. Further details are to be found in the supplementary material.

**Supplementary Material Available:** Detailed information on the crystal structure determination of **3** and **4** including tables of data collection parameters, final atomic positional parameters, final thermal parameters, and interatomic distances and angles and ORTEP diagrams (19 pages). Ordering information is given on any current masthead page. OM9409480