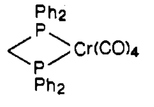
plexes are in fact the algebraic sums of "through-the-metal" and "backbone" contributions, which may be of opposite sign. This additive approach to coupling in ring compounds has subsequently been applied to other types of compounds, for example cyclic stanna-[8] and platinaalkanes.[9] In transition-metal phosphine complexes, recent work using multiple-resonance techniques[2] has shown that the through-the-metal contribution is negative in carbonyl compounds of chromium(0), molybdenum(0), and tungsten(0) and that the backbone contribution can be of either sign depending on the length of the backbone and the type and hybridization of the atoms in it. This leads to dramatic variations in the magnitudes *and the signs* of $^2J(\mathrm{PMP})_{\mathrm{cis}}$ in such complexes. As examples we cite values of −13.0, +47.0, 0 ±0.5, and +21 Hz in **1**, **2**, and **3**,[2] and in **4**,[6] respectively. More strikingly, in **5** the three values of $^2J(\mathrm{PMP})_{\mathrm{cis}}$ are −18.3, +17.6, and −17.2 Hz for P_AP_B, P_AP_C, and P_BP_C, respectively.[3] Similarly, in **6** they are +25 and +87 Hz for P_AP_B and P_CP_D, respectively, whereas the couplings for P_AP_C, P_BP_C, and P_BP_D, which do not have backbone contributions, are −20, −19, and −20 Hz, respectively, the trans coupling for P_AP_D being +87 Hz.[4]



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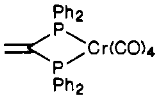

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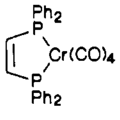

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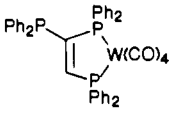

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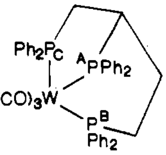

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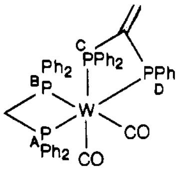

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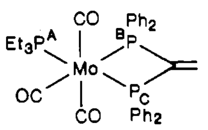

7

Although much of this variation is due to changes in the ligand backbone, there is also a dependence on the characteristics of the metal center, and work on carbonyl complexes of the tridentate ligand $(Ph_2P)_2CHCH_2PPh_2$ has confirmed values of ca. −41, −30, and −22 Hz for the through-the-metal contributions for chromium(0), molybdenum(0), and tungsten(0), respectively.[5] For square-planar palladium(II) and platinum(II) the corresponding figures are ca. −15 and −32 Hz.[6] These ranges are large enough to suggest that when certain substituents are present or when other factors such as bond-angle constraints are operative, through-the-metal contributions might well in some cases become positive. An additional important consequence is that large backbone contributions can lead to an inversion of the general rule[10,11] that states that trans $^2J(\mathrm{PMP})$ couplings are larger than their cis counterparts. For example, in **7** the cis coupling for P_BP_C is +79 Hz, whereas the trans one for P_AP_C is smaller at +69 Hz, the coupling for the nonchelated P_AP_B pair being "normal" at −24 Hz.[4]

It is thus clear that while the signs given for the geminal interphosphorus couplings in the particular ruthenium complexes cited by Baker and Field[1] are correct, it is not generally true to claim that they will always or even normally be negative in all transition-metal complexes, and it is essential to undertake appropriate experimental sign determinations. In this context it should be noted that while spectral analysis can often give the relative signs of certain coupling constants or combinations of them, it cannot always lead to a complete set. However, selective multiple-resonance[2,5,6] or two-dimensional[4] experiments will usually provide an unambiguous solution.

Acknowledgment. We thank the SERC for support.

Registry No. P, 7723-14-0.

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Preparation and Crystal Structure of a New Type of Triply Bridged Pyrazabole[1]

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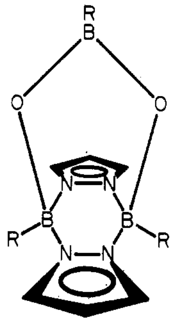
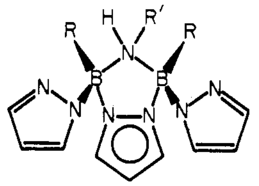
Received October 14, 1987

The reaction of *B*-triorganylboroxins, (−BRO−)$_3$, with pyrazole, Hpz, has recently been found to yield a novel type of pyrazabole in which the two boron atoms of a pyrazabole skeleton, $B(\mu\text{-pz})_2B$, are linked by a third bridging unit, i.e., O−BR−O, as shown in **1**.[2] Subsequently, such species have been observed as products in various other reactions, suggesting that the framework of **1** is chemically quite stable. Since part of the original boroxin framework is preserved in **1**, it was assumed that other boron heterocycles should react with pyrazole in analogous fashion. However, the reaction of borazines, (−BRNR′−)$_3$, with pyrazole unexpectedly led to (μ-amido)(μ-pyrazolato)diboron species, **2**, which are relatives of the pyrazaboles but do not feature the B_2N_4 skeleton of a pyrazabole.[3] In the search for true analogues of **1**, i.e., triply bridged pyrazaboles but with a unit other than O−BR−O as the third bridge between the two pyrazabole boron atoms, the reaction of a 1,2,4,3,5-dithiaazadiborolidine with pyrazole has been studied.



1



2

Experimental Section

Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, NY. Melting points (uncorrected) were determined on a Mel-Temp block.

NMR spectra were recorded on solutions in $CDCl_3$ on a Varian XL-200 instrument. Chemical shift data are given in ppm with positive values indicating downfield shift from the reference (internal Me_4Si for ^1H NMR, external $Et_2O{\cdot}BF_3$ for ^{11}B NMR); s = singlet, d = doublet,

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[§] University of Munich.

Table I. Atomic Coordinates (×10^4) and Equivalent Isotropic Thermal Parameters (Å^2 × 10^3) for Non-Hydrogen Atoms (Isotropic Thermal Parameters for Hydrogen Atoms) for $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ (**4**)[a]

atom	*x*	*y*	*z*	*U*[b]
S	525 (1)	−2054 (1)	3037 (1)	45 (1)
B	709 (2)	1076 (4)	3220 (2)	35 (1)
N1	980 (1)	2091 (3)	2245 (1)	35 (1)
N2	304 (1)	2095 (3)	1557 (1)	35 (1)
C3	699 (2)	2914 (4)	774 (2)	47 (1)
C4	1647 (2)	3442 (5)	958 (2)	55 (1)
C5	1800 (2)	2920 (4)	1896 (2)	48 (1)
C6	1484 (2)	1552 (6)	4021 (20)	55 (1)
H3	391 (18)	2984 (47)	251 (20)	43 (8)
H4	1991 (22)	4046 (5)	614 (20)	65 (9)
H5	2352 (24)	2993 (44)	2238 (18)	65 (9)
H6a	2094 (23)	1125 (53)	3806 (20)	76 (10)
H6b	1240 (23)	1015 (54)	4670 (22)	73 (9)
H6c	1610 (23)	3071 (56)	4110 (21)	67 (9)

[a] In this and all subsequent tables esd's are given in parentheses. [b] Equivalent isotropic *U* for non-hydrogen atoms defined as one-third of the trace of the orthogonalized U_{ij} tensor.

t = triplet, and an asterisk denotes a broad signal. Coupling constants *J* are given in hertz. Mass spectral data were obtained on a VG ZAB-2F spectrometer.

$CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ (4). A mixture of 3.60 g (17 mmol) of $CH_3B(\mu\text{-NC}_6H_5)(\mu\text{-SS})BCH_3$ (**3**), 2.40 g (35 mmol) of Hpz, 30 mL of ether, and 30 mL of benzene was heated to reflux with stirring for 8–10 h. All volatile material was removed at room temperature under high vacuum. The pasty residue was dissolved in hot benzene and filtered. On cooling of the clear filtrate to room temperature, a pale yellow crystalline precipitate appeared, and a second crop of product was obtained on concentration of the benzene solution. The solid material (consisting primarily of the desired compound) was dried in air, and a colorless impurity (which was identified as $CH_3B(\mu\text{-pz})_2(\mu\text{-OBCH}_3O)BCH_3$; see below) was sublimed off under high vacuum (95–100 °C bath temperature). The remaining yellow material was again recrystallized from benzene to give a pure product, mp 268–270 °C. Anal. Calcd for $C_8H_{12}B_2N_4S_2$ (M_r = 249.96): C, 38.44; H, 4.84; B, 8.65; N, 22.42; S, 25.65. Found: C, 38.39; H, 4.87; B, 8.27; N, 22.70; S, 25.62.

NMR data: $\delta(^1H)$ 7.74 (2 H, d, *J* = 2.4), 6.46 (1 H, t, *J* = 2.4), 0.86 (3 H, s); $\delta(^{11}B)$ −0.9 (s, $h_{1/2}$ = 60 Hz). Major ion clusters were observed in the 70-eV mass spectrum in the *m/z* 250, 203, 186, and 171 regions.

Crystal data on $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ were obtained on a Syntex R3 automated diffractometer by using graphite-monochromated Mo Kα radiation and standard operating techniques. Unit cell parameters were obtained from the least-squares refinement of the diffracting positions of 20 high-angle (18° < 2θ < 30°) reflections. The pale yellow compound crystallizes in the orthorhombic system in space group *Pbcn* (No. 60) with a = 6.089 (2) Å, b = 13.852 (2) Å, c = 14.179 (3) Å, V = 1195 (4) Å^3, Z = 4, and d_{calcd} = 1.389 g/cm^3; μ = 4.03 cm^{-1}, $\bar{U}$ = 0.039, and $F(000)$ = 519.87. Intensity data were collected (crystal size 0.2 × 0.3 × 0.3 mm) by the ω-scan technique in the range 2° < 2θ < 50° in $h,k,\pm l$. A total of 2440 reflections were measured (scan speed 2–29.3°/min, scan width 0.9°). After intensity correction (2 control measurements after every 48 measurements; variation 1.000–1.063) and adjustment of symmetry-equivalent reflections (R_{merge} = 0.022), there were a total of 1014 independent reflections, 865 of which had $I > 3\sigma(I)$, which were converted to structure factors after applying Lp correction. The structure was solved (SHELXTL, Version 4.1 structure solution package) by direct methods, and the best *E* map revealed the positions of all non-hydrogen atoms. After convergence was achieved in further refinement with anisotropic temperature factors, all hydrogen positions were revealed from a difference Fourier map. Refinement of their positions was included with use of isotropic temperature factors in the final refinement (full-matrix least-squares method). The final refinement converged at R = 0.042, R_w = 0.049 for 97 refined parameters (largest Δ/σ = 0.08; highest residual electron density = 0.31 e/Å^3 between S and C6). Definitions are $R = \sum||F_o| - |F_c||/\sum|F_o|$ and $R_w = \sum(w^{1/2}||F_o| - |F_c||)/\sum(w^{1/2}|F_o|)$, where $w^{-1} = \sigma^2(F_o) - g(F_o)^2$. Atomic coordinates and isotropic thermal parameters are listed in Table I.

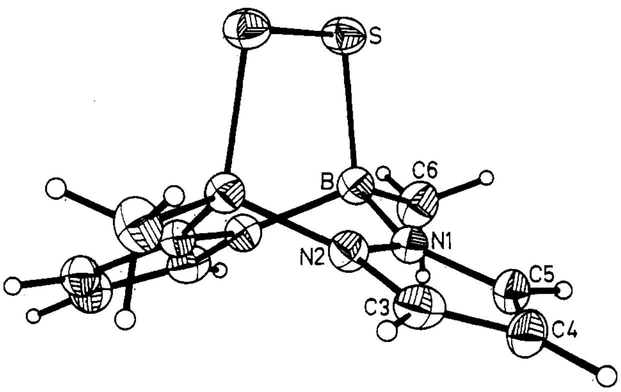


Figure 1. ORTEP plot of $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ (**4**).

Table II. Bond Distances (Å) for $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ (**4**)

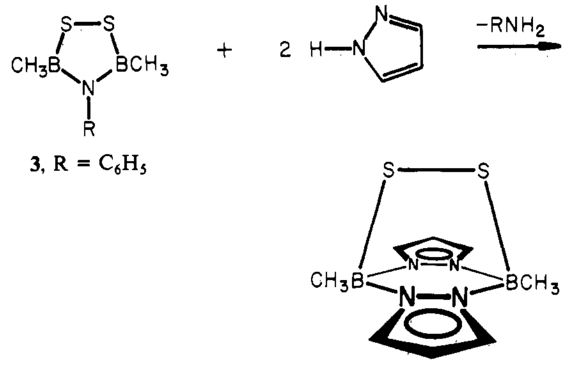
B–S	1.940 (3)	N1–N2	1.352 (3)
B–N1	1.560 (3)	N1–C5	1.338 (3)
B–N1*	1.566 (3)	C5–C4	1.383 (4)
B–C6	1.589 (4)	C4–C3	1.377 (4)
S–S*	2.105 (1)	C3–N2	1.334 (3)
C6–H6a	0.935 (32)	C5–H5	0.907 (31)
C6–H6b	1.033 (32)	C4–H4	0.775 (21)
C6–H6c	0.950 (34)	C3–H3	0.855 (28)

$CH_3B(\mu\text{-pz})_2(\mu\text{-OBCH}_3O)BCH_3$ was prepared by hydrolysis of $Br(CH_3)B(\mu\text{-pz})_2BBr(CH_3)$ (obtained from equimolar amounts of CH_3BBr_2 and $(CH_3)_3Si(pz)$[4]) or by reaction of $(\text{-BCH}_3O\text{-})_3$ with Hpz[2] as a colorless crystalline solid. It was readily purified by sublimation under vacuum (bath temperature 90–100 °C); mp near 290 °C (with massive sublimation). Anal. Calcd for $C_9H_{15}B_3N_4O_2$ (M_r = 243.68): C, 44.36; H, 6.20; B, 13.31; N, 22.99. Found: C, 44.19; H, 6.10; B, 12.96; N, 22.77.

NMR data: $\delta(^1H)$ 7.71 (4 H, d, *J* = 2.4), 6.30 (2 H, t, *J* = 2.4), 0.58 (6 H, s), 0.10 (3 H, s); $\delta(^{11}B)$ 32.7* (1 B, s), 1.3 (2 B, s, $h_{1/2}$ = 90 Hz). The mass spectrum exhibited no parent ion peak, but major ion clusters were observed near *m/z* 229, 171, 159, 135, 128, 93, and 67.

Results and Discussion

The reaction of 4-phenyl-3,5-dimethyl-1,2,4,3,5-dithiaazadiborolidine, $CH_3B(\mu\text{-NC}_6H_5)(\mu\text{-SS})BCH_3$ (**3**), with pyrazole proceeds quite readily with the elimination of aniline and formation of the triply bridged pyrazabole $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ (**4**), as shown in eq 1. This latter compound is the first example of a triply bridged neutral pyrazabole where the third bridge between the two boron atoms of the pyrazabole skeleton is not an OBRO unit.



3, R = C_6H_5

4

(1)

The yield of **4** is extremely sensitive to the purity of the reagents. With pure reagents and completely dry solvents, yields range from 75 to 90%. However, even small amounts of moisture lead to a

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(1) Boron–Nitrogen Compounds. 116 (K.N.). Part 115: Brock, C. P.; Das, M. K.; Minton, R. P.; Niedenzu, K. *J. Am. Chem. Soc.*, in press. Part 114: Das, M. K.; Mariategui, J. F.; Niedenzu, K. *Inorg. Chem.* **1987**, *26*, 3114–3116.

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Table III. Bond Angles (deg) for $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ (**4**)

B–S–S*	100.8 (1)	B–C6–H6a	109.1 (18)
S–B–C6	111.3 (2)	B–C6–H6b	110.9 (18)
S–B–N1*	107.4 (2)	B–C6–H6c	113.4 (19)
S–B–N1	107.6 (2)	H6a–C6–H6b	119.9 (26)
N1–B–N1*	103.7 (2)	H6a–C6–H6c	98 (27)
B–N1–N2	118.2 (2)	H6b–C6–H6c	104.5 (26)
B*–N2–N1	118.3 (2)	C3–C4–C5	105.9 (3)
N1–B–C6	113.4 (2)	N2–C3–C4	108.7 (2)
C6–B–N1*	112.9 (2)	N1–C5–H5	122.5 (18)
B–N1–C5	133.0 (2)	N2–C3–H3	122.3 (18)
B*–N2–C3	133.1 (2)	C5–C4–H4	128.3 (22)
N2–N1–C5	108.7 (2)	C4–C5–H5	129.1 (18)
N1–N2–C3	108.4 (2)	C4–C3–H3	128.9 (18)
N1–C5–C4	108.2 (2)	C3–C4–H4	125.4 (22)

lower yield and formation of the triply bridged pyrazabole $CH_3B(\mu\text{-pz})_2(\mu\text{-OBCH}_3\text{eo})BCH_3$ as well as hydrogen sulfide and elemental sulfur as major byproducts.

The $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ molecule has crystallographic C_2 symmetry, but even in the solid state it is rather close to point group C_{2v}, which corresponds to the (solution) NMR data. An ORTEP plot of the species is given in Figure 1, bond distances are given in Table II, and bond angles are listed in Table III.

The bridging S_2 group imposes the boat conformation for the B_2N_4 ring of the molecule, which is common for most pyrazaboles.[5] The bond distances within the pyrazabole skeleton parallel those found for a series of other pyrazaboles, but the N1–N2 distance is one of the shortest measured in pyrazaboles. The two B–N distances as well as the C–N and C–C distances are essentially equal. The S–S bond (2.105 (1) Å) is a typical single bond but is noticeably longer than in S_8 or disulfanes; it is also longer than that found in $CH_3B(\mu\text{-NCH}_3)(\mu\text{-SS})BCH_3$, containing trigonal boron.[6] The B–S bond distances in trigonal boron derivatives range from 1.79 to 1.84 Å,[7] but longer distances have been observed for the 1:1 molar adduct of 4,6-dibromo-1,2,3,5,4,6-tetrathiadiborolane with 1,3,2-trimethyl-1,3,2-diazaborolidine, in which all boron atoms are four-coordinate.[8] The B–S distances for the B–S–S–S–B unit of the latter adduct are 1.910 (12) and 1.930 (12) Å, respectively, comparable to that found in **4** (1.940 (3) Å).

The N–B–N angle is, however, smaller by about 2° as compared to those of other pyrazaboles; only that of $C_2H_5B(\mu\text{-pz})_2(\mu\text{-OBC}_2H_5O)BC_2H_5$ is even smaller.[6] This is undoubtedly the consequence of the third bridge between the two boron atoms, especially since the N–B–S angle (and the N–B–O angle in $C_2H_5B(\mu\text{-pz})_2B(\mu\text{-OBC}_2H_5O)BC_2H_5$) is also smaller than comparable data in normal pyrazaboles, i.e., those where the two B atoms are linked only by the two pz groups. It is noteworthy that the dihedral angle B–S–S–B is 0°. The folding angle θ_1 (between a BN_2 plane and the N_4 plane) is 39.9°, the largest one measured for any pyrazabole, and the roof angle θ_2 formed between the two pz rings in a butterfly arrangement is also extremely high (46.2°).[5] Thus, the additional S–S bridge between the two B atoms enhances the boat conformation of the B_2N_4 ring.

The formation of $CH_3B(\mu\text{-pz})_2(\mu\text{-OBCH}_3O)BCH_3$ as byproduct in the reaction according to eq 1 in the presence of moisture is probably due to the hydrolytic sensitivity of the starting dithiaazadiborolidine, since $CH_3B(\mu\text{-pz})_2(\mu\text{-SS})BCH_3$ is not moisture-sensitive. The former triply bridged pyrazabole is more readily accessible by the conventional[2] interaction of pyrazole with $(-BCH_3O-)_3$ and also by the hydrolysis of 4,8-dibromo-4,8-dimethylpyrazabole.[4]

The present work shows that additional triply bridged neutral pyrazaboles should be available by reaction of various boron heterocycles with pyrazole. It remains to be seen whether or not the third bridge can also be a single atom, but species containing a N–N unit as the third bridge between the two boron atoms of a pyrazabole skeleton are likely to exist.

Acknowledgment. We gratefully acknowledge a gift of 2-phenyl-3,5-dimethyl-1,2,4,3,5-dithiaazadiborolidine from Professor A. Meller, Universität Göttingen, West Germany. This work was supported by the Office of Naval Research (K.N.) and the Fonds der Chemischen Industrie (H.N.).

Registry No. **1** (coordination compound entry), 112840-86-5; **1** (borane entry), 112863-45-3; **3**, 63261-96-1; **4** (coordination compound entry), 112840-87-6; **4** (borane entry), 112840-89-8; Hpz, 288-13-1; $Br(CH_3)B(\mu\text{-Pz})_2BBr(CH_3)$, 112840-88-7.

Supplementary Material Available: A listing of anisotropic thermal parameters for non-hydrogen atoms for **4** (1 page); a structure factor table (5 pages) for **4**. Ordering information is given on any current masthead page.

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Mechanism of Catalytic Isotope Exchange in Alcohols Promoted by Early-Transition-Metal Alkoxides. Comment on the Paper by Nugent and Zubyk[1]

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A recent paper by Nugent and Zubyk[1] concerning the isotopic H–D exchange in alcohols catalyzed by early-transition-metal alkoxides appears relevant to the mechanism of higher alcohol synthesis from syngas under metal oxide catalysis.[2] Nugent and Zubyk[1] reported separate α- and β-exchange processes for scrambling H–D in alcohols in the presence of various $M_x(OEt)_y$ ($M = Zr, x = 1, y = 4$; $M = Nb, Ta, x = 2, y = 10$) complexes at temperatures from 180 to 220 °C. The β-hydrogens exchanged rapidly into the hydroxyl position, while the α-hydrogens underwent a separate scrambling process; the two processes are summarized schematically as

α-exchange

$$CH_3CH_2OH + CH_3CD_2OH \leftrightarrow CH_3CHDOH$$

β-exchange

$$CH_3CH_2OD \leftrightarrow CH_{3-n}D_nCH_2OH(D)$$

Similar rates were observed for the two processes and "the same alcohol which has undergone α-exchange has also undergone (multiple) β-exchange". Racemization of 2-butanol and epimerization of *tert*-butylcyclohexanol accompanied their exchange reactions. This evidence was interpreted to indicate a common intermediate, proposed to be an organic carbonyl compound, was responsible for both exchange processes. In a previous paper,[3] amines were shown to catalyze the β-exchange process, although the authors did not elaborate on this point.

The mechanism proposed for the conversion of syngas into higher alcohols over metal oxide catalysts involves a stepwise reduction of coordinated CO to formaldehyde, which is reduced further to methanol or undergoes CO-insertion to form higher products.[2] An intermediate in the reaction path leading to higher alcohols is an η^3-enolate. Stability differences of various η^3-enolate species are thought to contribute to the branching observed in the higher alcohol products. An important competing reaction is

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[1] Nugent, W. A.; Zubyk, R. M. *Inorg. Chem.* **1986**, *25*, 4604–4606.

[2] Mazanec, T. J. *J. Catal.* **1986**, *98*, 115–125.

[3] Nugent, W. A.; Ovenall, D. W.; Holmes, S. J. *Organometallics* **1983**, *2*, 161–162.