Synthesis of a Metallamacrocyclic Re(CO)$_3$ Complex Using a Tridentate Bridging Ligand

Thomas Brasey,[a] Andrey Buryak,[a] Rosario Scopelliti,[a] and Kay Severin*[a]

Keywords: Host−guest chemistry / Metallamacrocycles / Rhenium / Silver

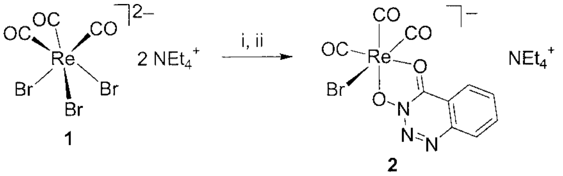
The reaction of [ReBr$_3$(CO)$_3$][NEt$_4$]$_2$ with 3-hydroxy-1,2,3-benzotriazine-4(3*H*)-one in the presence of base gave the ionic rhenium complex [ReBr(C$_7$H$_4$N$_3$O$_2$)(CO)$_3$][NEt$_4$] (**2**) which was crystallographically characterized. A neutral metallamacrocycle **3** was obtained upon abstraction of [NEt$_4$]Br from complex **2**. This complex cocrystallizes with an unusual C_3-symmetric [Ag(η^2-benzene)$_3$(OH$_2$)][BF$_4$] complex as evidenced by single-crystal X-ray diffraction.

Introduction

The *fac*-Re^{I}(CO)$_3$ fragment has frequently been used as a building block for the construction of self-assembled metallomacrocycles,[1] some of which have found applications in the field of sensing[2] or catalysis.[3] For the synthesis of these macrocycles, bidentate ligands with N-heterocycles as donor groups were employed almost exclusively. We have recently shown that simple tridentate ligands with N,O,O′-donor atoms such 2,3-dihydroxypyridine[4] or 3,4-dihydroxy-2-methyl-pyridine[5] are well suited for the assembly of organometallic half-sandwich complexes of Ru^{II}, Rh^{III} and Ir^{III}.[6] In continuation of these studies, we have investigated the reaction of [ReBr$_3$(CO)$_3$][NEt$_4$]$_2$[7] (**1**) with 3-hydroxy-1,2,3-benzotriazine-4(3*H*)-one. Complex **1** was chosen as the starting material because the three bromo ligands can easily be replaced. When **1** is dissolved in water, for example, the thermodynamically stable[8] but kinetically labile[9] aqua complex [Re(CO)$_3$(OH$_2$)$_3$]$^+$ is formed. The benzotriazine derivative appeared to be a suitable reaction partner for the construction of a neutral metallamacrocycle because it can occupy the three available coordination sites by acting as a monoanionic, bridging chelate ligand.

Results and Discussion

A mixture of complex **1**, the ligand 3-hydroxy-1,2,3-benzotriazine-4(3*H*)-one and one equivalent of KOH was dissolved in methanol. After 12 h, the solvent was evaporated and the product was separated from the bromide salts by Soxhlet extraction with benzene. In this way we were able to obtain the adduct **2** in 72 % yield (Scheme 1). The infrared spectrum of **2** was characteristic of a Re(CO)$_3$ complex with strong bands in the carbonyl region at ν_{CO} = 2007 and 1855 cm^{-1}. The NMR spectra of **2** confirmed the presence of the benzotriazine ligand and the tetraethylammonium cation.



Scheme 1. Synthesis of complex **2**; (i) 3-hydroxy-1,2,3-benzotriazine-4(3*H*)-one, KOH (1 equiv.), MeOH, 12 h, r.t.; (ii) evaporation of MeOH and extraction with benzene

The structure of **2** was established by single-crystal X-ray analysis. The deprotonated benzotriazine derivative is coordinated by the two oxygen atoms to the Re atom (Figure 1). This is different from what has been observed for Ru- and Os-carbonyl complexes with this ligand; here a N−N′- or a N−O-coordination mode was found.[10] The third coordination site in **2** is still occupied by one bromo ligand, which is positioned *trans* to a carbonyl ligand, resulting in a negatively charged Re(CO)$_3$ complex. Due to the small bite angle of the chelate ligand, the octahedral geometry around the Re atom is slightly distorted.

In order to induce macrocyclization, the remaining bromo ligand of complex **2** was abstracted with AgBF$_4$ in methanol (Scheme 2). After crystallization from benzene/pentane, a product of the empirical formula [Re(CO)$_3$(C$_7$H$_4$N$_3$O$_2$)]$_n$ (**3**) was obtained. An alternative and simpler way to synthesize this complex is by suspending **2** in water followed by extraction of the product with diethyl ether (NEt$_4$Br remains in the aqueous phase).

The presence of the Re(CO)$_3$ fragment was confirmed by IR spectroscopy (ν_{CO} = 2026 and 1882 cm^{-1}). ^{1}H NMR

---

[a] Institut des Sciences et Ingénierie Chimiques, École Polytechnique Fédérale de Lausanne,
1015 Lausanne, Switzerland
Fax: (internat.) + 41-(0)21-693-9305
E-mail: kay.severin@epfl.ch

DOI: 10.1002/ejic.200300880

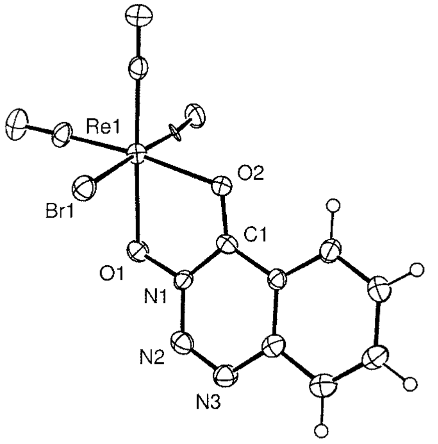
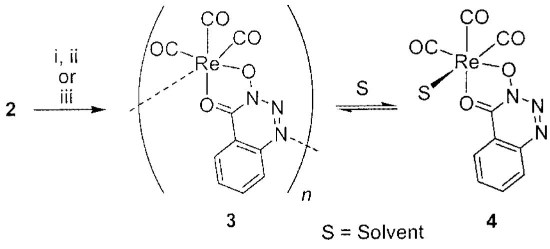


Figure 1. Molecular structure of complex **2** in the crystal; the $[NEt_4]^+$ cation and the CH_2Cl_2 molecule are not shown for clarity; selected bond lengths [Å] and angles [°]: Re1−O1 2.168(10), Re1−O2 2.163(9), Re1−Br1 2.6544(15), N1−O1 1.359(14), C1−O2 1.251(16); O1−Re1−O2 75.2(4), Br1−Re1−O1 83.9(3), Br1−Re1−O2 83.6(3)



Scheme 2. Synthesis of the metallamacrocycle **3**; (i) $AgBF_4$ (1.1 equiv.), MeOH, 1 h, r.t.; (ii) crystallization from benzene/pentane; alternatively: (iii) separation with Et_2O/H_2O (yield: 80 %); in polar solvents such as methanol, the monomeric adducts **4** are formed

spectroscopic investigations in C_6D_6 were indicative of a highly symmetrical structure due to the presence of only one set of signals for the ligand. Unfortunately, we were not able to determine unequivocally the aggregation number of **3** with either mass spectrometry or single-crystal X-ray analysis, but based on the coordinate vectors of the benzotriazine ligand, a trimeric (see below) or tetrameric macrocycle seemed to be the most likely geometry.

When complex **3** is dissolved in coordinating polar solvents, both the NMR spectra as well as the UV-Vis spectroscopic data change substantially. In benzene, the macrocyclic complex **3** displays a maximum at $\lambda = 426$ nm, whereas in methanol the value is shifted to $\lambda = 373$ nm. A similar value is found in acetonitrile ($\lambda = 376$ nm). In C_6D_6, the signals for the benzotriazine ligand range from $\delta = 6.62-8.68$ ppm, whereas in CD_3OD the signals are confined to a region between $\delta = 7.92$ and 8.31 ppm (CD_3CN: 7.88−8.25 ppm). A plausible explanation of this data is that in coordinating solvents, we generate the monomeric solvent adducts **4** (Scheme 2). Such behaviour is not unprecedented: a solvent-dependent equilibrium between a dimer and a monomer has been described for $Re(CO)_3$ complexes with chelating $[NO]^-$ ligands.[11]

As a side product of the reaction between **2** and $AgBF_4$ (2 equiv.), we were able to isolate crystals of the macrocyclic compound **5** (ca. 5 % yield). As evidenced by X-ray crystallography, complex **5** contains an unusual C_3-symmetric $[Ag(\eta^2\text{-benzene})_3(OH_2)][BF_4]$ guest. A graphical representation of the structure is shown in Figure 2. The Re complex forms a trimeric metallamacrocycle. The $Re(CO)_3$ fragments are connected by the two O-atoms and the N1-atom of the benzotriazine ligand with the metal atoms being 7.066(2) Å apart from each other. To the best of our knowledge, this is the first structurally characterized macrocyclic $Re(CO)_3$ complex built with a tridentate chelate ligand. The noncovalent interactions with the guest molecule are of special interest. The metallamacrocycle forms a slightly concave cavity which has exactly the right size and symmetry to encapsulate the $[BF_4]^-$ anion.[12] On the other side of the macrocycle — and well shielded from the $[BF_4]^-$ anion — a $[Ag(\eta^2\text{-}C_6H_6)_3(OH_2)]^+$ complex is located. This cation represents one of the rare cases in which a $[Ag]^+$ ion is bound to three free arene ligands.[13] There are several examples of complexes containing the $[Ag(\eta^{1/2}\text{-arene})_3]^+$ structural motif, but here, chelating arene ligands such as cyclophane-type macrocycles were used to increase the stability of the complex.[14] The compound $[Ag(C_6H_6)_3][BF_4]$ on the other hand, is highly unstable: out of solution, it decomposes instantaneously even in an inert atmosphere (Figure 2).[13a]

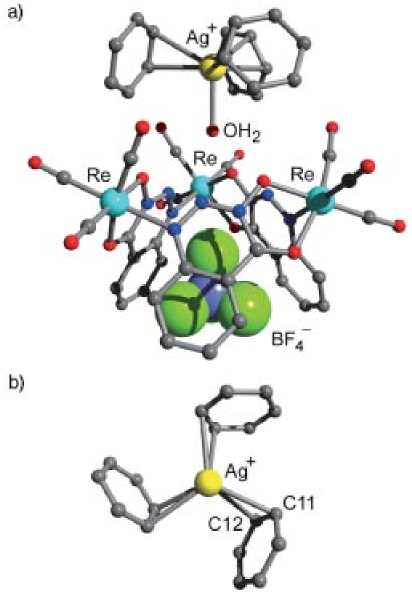


Figure 2. a) Graphic representation of the molecular structure of **5** in the crystal; the hydrogen atoms and a cocrystallized benzene molecule are not shown for clarity; b) view along the C_3-symmetry axis on the $[Ag(\eta^2\text{-benzene})_3]^+$ complex

The $[Ag]^+$ ion in complex **5** is situated on the symmetry axis and is thus coordinated to three benzene solvent molecules in a highly symmetrical fashion (Figure 2 b). In this context, it is interesting to note that the threefold symmetry was claimed to be unfavorable for $Ag(arene)_3$ coordination.[15] In view of our results, this statement appears to be questionable. At least the energy difference between a symmetrical and a distorted geometry must be small since there are only weak packing effects, which can influence the geometry of the $[Ag]^+$ complex in **5**. The Ag−C distances found for **5** are within the expected range [Ag−C11: 2.623(18); Ag−C12: 2.612(17) Å]. A water molecule is bound to the fourth coordination site of the silver ion [Ag−O: 2.297(3) Å]. It is within expected hydrogen-bond lengths to the oxo-groups of the bridging benzotriazine ligand [O···O′: 3.112(18) Å] thereby connecting the silver complex with the $Re(CO)_3$-macrocycle.

Conclusions

In summary, we have described the synthesis and the structure of a new $Re(CO)_3$-containing metallamacrocycle which is able to act as a host for the unusual silver complex $[Ag(\eta^2\text{-}C_6H_6)_3(OH_2)][BF_4]$. In elaboration of previous approaches to construct $Re(CO)_3$ macrocycles, an anionic, tridentate chelate ligand was used to bridge the rhenium centers. It seems likely that this synthetic concept can be expanded by employing other organometallic fragments such as *fac*-$[M^{II}(allyl)(CO)_2]$ (M = Mo, W) or *fac*-$[Pt^{IV}(CH_3)_3]$ and/or by using structurally related anionic ligands. The synthesis and the host-guest chemistry of such complexes is currently being investigated in our laboratory.

Experimental Section

General: All reactions were performed under dry dinitrogen, using standard Schlenk techniques. Complex $[ReBr_3(CO)_3][NEt_4]_2$ (**1**) was prepared according to a literature procedure.[7] The ^1H and ^{13}C spectra were recorded on a Bruker Advance DPX 400 or a Bruker Advance 200 spectrometer with the residual protonated solvents (^1H, ^{13}C) as internal standards. All spectra were recorded at room temperature.

Synthesis of Complex 2: Complex $[ReBr_3(CO)_3][NEt_4]_2$ (770 mg, 1.0 mmol) was added to a solution of the ligand 3-hydroxy-1,2,3-benzotriazine-4(3*H*)-one (163 mg, 1.0 mmol) and KOH (2.0 mL of 0.496 M, 1.0 mmol) in methanol (25 mL). After 12 h, the solvent was removed under reduced pressure and the product was separated by Soxhlet extraction with benzene. Evaporation of the solvent gave 465 mg of complex **2** (72 %). IR: $\tilde{\nu}$ = 2007 (CO), 1855 (CO) cm^{-1}. ^1H NMR ($CDCl_3$): δ = 1.30 (t, ^3J = 7 Hz, 12 H, CH_3, NEt_4), 3.32 (q, ^3J = 7 Hz, 8 H, CH_2, NEt_4), 7.68 (t, ^3J = 8 Hz, 1 H, aromatic CH), 7.78 (t, ^3J = 8 Hz, 1 H, aromatic CH), 8.08 (d, ^3J = 8 Hz, 1 H, aromatic CH), 8.24 (d, ^3J = 8 Hz, 1 H, aromatic CH) ppm. ^{13}C NMR ($CDCl_3$): δ = 10.7 (CH_3), 55.7 (CH_2), 121.4, 126.0, 130.8, 135.4, 137.0, 146.5, 157.8 (aromatic carbon atoms), 194.2, 198.6, 198.8 (CO) ppm. Crystals were obtained by slow diffusion of pentane into a solution of **2** in CH_2Cl_2. $C_{18}H_{24}BrN_4O_5Re\cdot 1/3\ C_6H_6$ (668.56 $g\cdot mol^{-1}$): calcd. C 35.93, H 3.92, N 8.38; found C 35.69, H 4.69, N 7.91.

Synthesis of Complex 3. Method A: A mixture of complex **2** (50 mg, 78 μmol) and $AgBF_4$ (17 mg, 88 μmol) was stirred in methanol (10 mL). After 1 h, the precipitate was filtered off and the solvent was evaporated. The product was then extracted with benzene. After evaporation of the solvent, the product was dried in vacuo (21 mg, 63 %). Crystals of complex **5** were obtained as a side product from benzene/pentane in reactions using a twofold excess of $AgBF_4$ with respect to complex **2**.

Method B: Complex **2** (50 mg, 78 μmol) is dissolved in a mixture of diethyl ether (10 mL) and H_2O (10 mL). The Et_2O phase was separated and washed two more times with H_2O (10 mL). After evaporation of the organic phase, the product was dried in vacuo (27 mg, 80 %). IR: $\tilde{\nu}$ = 2026 (CO) cm^{-1}, 1882 (CO). ^1H NMR (C_6D_6): δ = 6.62 (t, ^3J = 8 Hz, 1 H, aromatic CH), 6.82 (t, ^3J = 8 Hz, 1 H, aromatic CH), 7.63 (d, ^3J = 8 Hz, 1 H, aromatic CH), 8.68 (d, ^3J = 8 Hz, 1 H, aromatic CH) ppm. ^{13}C NMR ($C_6D_5CD_3$): δ = 122.12, 129.07, 130.03, 135.12, 138.09, 148.65 (aromatic carbon atoms; CO: n.o.) ppm. $C_{30}H_{12}N_9O_{15}Re_3\cdot H_2O\cdot Et_2O$ (1389.23 $g\cdot mol^{-1}$): calcd. C 29.40, H 1.74, N 9.07; found C 29.13, H 2.04, N 9.14.

Crystallographic Data: Complex **2** × 0.5 CH_2Cl_2: $C_{18.5}H_{25}BrClN_4O_5Re$, M_r = 684.98 $g\cdot mol^{-1}$, monoclinic, space group $P2_1/c$, a = 12.2500(8), b = 10.3793(5), c = 18.3764(14) Å; β = 96.137(6)°, V = 2323.1(3) $Å^3$, T = 140(2) K, Z = 4, μ = 7.102 mm^{-1}, λ = 0.71073 Å, 12362 measured reflections, 3865 independent reflections, R_{int} = 0.0499, R_1 [$I > 2\sigma(I)$] = 0.0697, wR_2 (all data) = 0.1662. Complex **5** × 3 C_6H_6: $C_{66}H_{50}AgBF_4N_9O_{16}Re_3$, M_r = 1978.43 $g\cdot mol^{-1}$, rhombohedral, space group $R3c$, a = 20.8533(16), c = 26.492(3) Å, V = 9976.8(15) $Å^3$, T = 140(2) K, Z = 6, μ = 5.819 mm^{-1}, λ = 0.71073 Å, 19101 measured reflections, 3875 independent reflections, R_{int} = 0.1648, R_1 [$I > 2\sigma(I)$] = 0.0662, wR_2 (all data) = 0.1542. The benzene molecules have been constrained to regular hexagons with d(C−C) = 1.39 Å. Diffraction data were collected at 140 K, using Mo-K_α radiation and a Oxford Diffraction diffractometer with a kappa geometry and equipped with a Sapphire CCD detector. Data reduction was performed with CrysAlis RED 1.6.9.[16] Absorption correction was applied to all data sets using an empirical method (DIFABS).[17] Structure solutions were determined with ab initio direct methods.[18] All structures were refined using the full-matrix least-squares on F^2 with all non-H atoms anisotropically defined. The hydrogen atoms were placed in calculated positions using the "riding model" with $U_{iso} = a\cdot U_{eq}(C)$ (where a is 1.5 for methyl hydrogen atoms and 1.2 for others, C is the parent carbon atom). Space group determination, structure refinement and geometrical calculations were carried out on all structures with the SHELXTL software package, release 5.1.[19] The graphical representation of the molecular structures of **2** was generated with the program ORTEP.[20] CCDC-216353 (for **2**) and -216354 (for **5**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/const/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; Fax: (internat.) + 44-1223-336-033; or deposit@ccdc.cam.ac.uk).

Acknowledgments

We gratefully acknowledge support of this work by the Swiss National Science Foundation.

---

[1] For reviews see: [1a] S.-S. Sun, A. J. Lees, *Coord. Chem. Rev.* **2002**, *230*, 171−192. [1b] P. H. Dinolfo, J. T. Hupp, *Chem. Mat.* **2001**, *13*, 3113−3125.

[2] [2a] M. H. Keefe, J. L. O'Donnell, R. C. Bailey, S. T. Nguyen, J. T. Hupp, *Adv. Mat.* **2003**, *15*, 1936−1939. [2b] P. de Wolf, S. L. Heath, J. A. Thomas, *Chem. Commun.* **2002**, 2540−2541. [2c] G. A. Mines, B.-C. Tzeng, K. G. Stevenson, J. Li, J. T. Hupp,

Angew. Chem. **2002**, *114*, 162−165; *Angew. Chem. Int. Ed.* **2002**, *41*, 154−157. [2d] S. J. Lee, W. Lin, *J. Am. Chem. Soc.* **2002**, *124*, 4554−4555. [2e] D. Xu, K. T. Khin, W. E. van der Veer, J. W. Ziller, B. Hong, *Chem. Eur. J.* **2001**, *7*, 2425−2434. [2f] S. Bélanger, J. T. Hupp, *Angew. Chem.* **1999**, *111*, 2360−2362; *Angew. Chem. Int. Ed.* **1999**, *38*, 2222−2224. [2g] S. Bélanger, J. T. Hupp, C. L. Stern, R. V. Slone, D. F. Watson, T. G. Carrell, *J. Am. Chem. Soc.* **1999**, *121*, 557−563.

[3] M. L. Merlau, M. del Pilar Mejia, S. T. Nguyen, J. T. Hupp, *Angew. Chem.* **2001**, *113*, 4369−4372; *Angew. Chem. Int. Ed.* **2001**, *40*, 4239−4242.

[4] [4a] Z. Grote, R. Scopelliti, K. Severin, *Angew. Chem.* **2003**, *115*, 3951−3955; *Angew. Chem. Int. Ed.* **2003**, *42*, 3821−3825. [4b] M.-L. Lehaire, R. Scopelliti, H. Piotrowski, K. Severin, *Angew. Chem.* **2002**, *114*, 1477−1480; *Angew. Chem. Int. Ed.* **2002**, *41*, 1419−1422. [4c] M.-L. Lehaire, R. Scopelliti, K. Severin, *Chem. Commun.* **2002**, 2766−2767. [4d] M.-L. Lehaire, R. Scopelliti, K. Severin, *Inorg. Chem.* **2002**, *41*, 5466−5474. [4e] H. Piotrowski, K. Severin, *Proc. Natl. Acad. Sci. USA* **2002**, *99*, 4997−5000. [4f] H. Piotrowski, K. Polborn, G. Hilt, K. Severin, *J. Am. Chem. Soc.* **2001**, *123*, 2699−2700. [4g] H. Piotrowski, G. Hilt, A. Schulz, P. Mayer, K. Polborn, K. Severin, *Chem. Eur. J.* **2001**, *7*, 3196−3208.

[5] T. Habereder, M. Warchhold, H. Nöth, K. Severin, *Angew. Chem.* **1999**, *111*, 3422−3425; *Angew. Chem. Int. Ed.* **1999**, *38*, 3225−3228.

[6] For a review see: K. Severin, *Coord. Chem. Rev.* **2003**, *245*, 3−10.

[7] R. Alberto, A. Egli, U. Abram, K. Hegetschweiler, V. Gramlich, P. A. Schubinger, *J. Chem. Soc., Dalton Trans.* **1994**, 2815−2820.

[8] A. Egli, K. Hegetschweiler, R. Alberto, U. Abram, R. Schibli, R. Hedinger, V. Gramlich, R. Kissner, P. A. Schubinger, *Organometallics* **1997**, *16*, 1833−1840.

[9] B. Salignac, P. V. Grundler, S. Cayemittes, U. Frey, R. Scopelliti, A. E. Merbach, R. Hedinger, K. Hegetschweiler, R. Alberto, U. Prinz, G. Raabe, U. Kölle, S. Hall, *Inorg. Chem.* **2003**, *42*, 3516−3526.

[10] S. Kumaresan, K.-L. Lu, J.-T. Hung, F.-Y. Lee, Y.-S. Wen, J. R. Hwu, *J. Organomet. Chem.* **1997**, *549*, 155−161.

[11] [11a] R. Czerwieniec, A. Kapturkiewicz, R. Anulewicz-Ostrowska, J. Nowacki, *J. Chem. Soc., Dalton Trans.* **2002**, 3434−3441. [11b] R. Czerwieniec, A. Kapturkiewicz, R. Anulewicz-Ostrowska, J. Nowacki, *J. Chem. Soc., Dalton Trans.* **2001**, 2756−2761.

[12] For supramolecular coordination compounds which act as hosts for the [BF$_4$]$^-$ anion see: [12a] L. Raehm, L. Mimassi, C. Guyard-Duhayon, H. Amouri, *Inorg. Chem.* **2003**, *42*, 5654−5659. [12b] O. V. Dolomanov, A. J. Blake, N. R. Champness, M. Schröder, C. Wilson, *Chem. Commun.* **2003**, 682−683. [12c] C.-Y. Su, Y.-P. Cai, C.-L. Chen, F. Lissner, B.-S. Kang, W. Kaim, *Angew. Chem.* **2002**, *114*, 3519−3523; *Angew. Chem. Int. Ed.* **2002**, *41*, 3371−3375. [12d] H. Amouri, M. N. Rager, F. Cagnol, J. Vaissermann, *Angew. Chem.* **2001**, *113*, 3748−3750; *Angew. Chem. Int. Ed.* **2001**, *40*, 3636−3638. [12e] F. Fochi, P. Jacopozzi, E. Wegelius, K. Rissanen, P. Cozzini, E. Marastoni, E. Fisicaro, P. Manini, R. Fokkens, E. Dalcanale, *J. Am. Chem. Soc.* **2001**, *123*, 7539−7552. [12f] K. M. Kim, J. S. Park, Y.-S. Kim, Y. J. Jun, T. Y. Kang, Y. S. Sohn, M.-J. Jun, *Angew. Chem.* **2001**, *113*, 2524−2526; *Angew. Chem. Int. Ed.* **2001**, *40*, 2458−2460.

[13] [13a] A. S. Batsanov, S. P. Crabtree, J. A. K. Howard, C. W. Lehmann, M. Kilner, *J. Organomet. Chem.* **1998**, *550*, 59−61. [13b] M. Munakata, L. P. Wu, T. Kuroda-Sowa, M. Maekawa, Y. Suenaga, G. L. Ning, T. Kojima, *J. Am. Chem. Soc.* **1998**, *120*, 8610−8618.

[14] For some selected examples see: [14a] T. Lahtines, E. Wegelius, K. Rissanen, *New. J. Chem.* **2001**, *25*, 905−911. [14b] T. Seppälä, E. Wegelius, K. Rissanen, *New J. Chem.* **1998**, 789−791. [14c] F. R. Heirtzler, H. Hopf, P. G. Jones, P. Bubenitschek, *Chem. Ber.* **1995**, *128*, 1079−1082. [14d] F. R. Heirtzler, H. Hopf, P. G. Gones, P. Bubenitschek, *Tetrahedron Lett.* **1995**, *36*, 1239−1242. [14e] F. R. Heirtzler, H. Hopf, P. G. Gones, P. Bubenitschek, V. Lehne, *J. Org. Chem.* **1993**, *58*, 2781−2784. [14f] H. C. Kang, A. W. Hanson, B. Eaton, V. Boekelheide, *J. Am. Chem. Soc.* **1985**, *107*, 1979−1985.

[15] S. V. Lindeman, R. Rathore, J. K. Kochi, *Inorg. Chem.* **2000**, *39*, 5707−5716.

[16] Oxford Diffraction Ltd, Abingdon, Oxfordshire, UK, **2001**.

[17] N. Walker, D. Stuart, *Acta Crystallogr., Sect. A* **1983**, *39*, 158−166.

[18] G. M. Sheldrick, *Acta Crystallogr., Sect. A* **1990**, *46*, 467−473.

[19] G. M. Sheldrick, *University of Göttingen*, **1997**; Bruker AXS, Inc., Madison, Wisconsin, 53719, USA, **1997**.

[20] L. J. Farrugia, *J. Appl. Crystallogr.* **1997**, *30*, 565.

Received November 27, 2003