

The pair of ions $\text{IF}_6^\ominus/\text{XeF}_8^{2\ominus}$ provide the second example demonstrating that a structural principle remains unchanged on introducing a non-bonding electron pair, but that merely all the bond lengths are enlarged. The first example is the pair $\text{BrF}_6^\ominus/\text{BrF}_8^\ominus$.^[4] The likely explanation for the failure of the electron-pair repulsion model^[5] is that the bromine atom with six fluorine atoms and the iodine or xenon atom with eight fluorine atoms as ligands are sterically already so crowded that the non-bonding electron pair must reside in a centrosymmetric orbital, which, however, leads to a bond lengthening.

Experimental

1: In a metal vacuum apparatus NOF and IF₇ were condensed in the molar ratio 3:1 into a Teflon FEP tube, which was then sealed. Upon storage at -20°C, colorless crystals separated out. A suitable crystal (0.6 × 0.6 × 0.2 mm³) was transferred with a special apparatus [6] to a CAD4 diffractometer (MoK_α, -163°C, ω-θ scan). *a* = 618.92(7), *b* = 1039.43(17), *c* = 2842.14(15) pm, *P*₂,*P*₂,*P*₂ (No. 19). 4475 measured reflections in the range 2° < θ < 35°, +*h*, +*k*, +*l*, 4445 unique reflections, 4070 reflections with *I* ≥ 3σ(*I*), μ = 19.5 cm⁻¹, full matrix. SHELXTL plus, all atoms anisotropic, *R* = 0.038, *R*_w = 0.032.

2: Anhydrous (CH₃)₄N[⊕]F[⊖] [7] was suspended in CH₃CN in a Teflon FEP tube; a slight excess of IF₇ was then condensed into the suspension. The fluoride dissolved with formation of a complex, which upon cooling crystallized out in the form of needle shaped crystals. Diffraction photographs showed that all reflections are slightly split. *a* = 866.9(3), *b* = 866.9(2), *c* = 683.6(3) pm, α = β = γ = 90° (-153°C). 927 measured reflections in the range 2° < θ < 30°, ±*h*, +*k*, +*l*, 460 unique reflections, 431 reflections with *I* ≥ 3σ(*I*), μ = 31.8 cm⁻¹, full matrix. Calculation with SHELXTL plus in space group *P*₄/*m**m**m* (No. 129); C, N and I anisotropic, H in isotropically fixed calculated positions, F fourfold disordered, isotropic: *R* = 0.049, *R*_w = 0.043. Calculation with the program SFLS [8] in the space group *P**m**m**n* (No. 59) according to the twinning law (010)(-100)(001) gave *R* = 0.051, *R*_w = 0.045 for C, N, I anisotropic, F isotropic, twofold disordered, without H. For a non-disordered anion the space group *P*₂,*m**n* (No. 31) would suffice. Refinement in this space group however generates four positions again for each fluorine atom.

Further details of the crystal structure determinations are available on request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, W-7514 Eggenstein-Leopoldshafen (FRG), on quoting the depository number CSD-55274, the names of the authors, and the journal citation.

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IF₆[⊖], 53568-65-3; (NOF)₂ · NO[⊕]IF₈[⊖], 134152-23-1; [(CH₃)₄N]IF₈, 134131-34-3.

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[3] S. W. Peterson, J. H. Holloway, B. A. Coyle, J. M. Williams, *Science* 173 (1971) 1238.
[4] A. R. Mahjoub, A. Hoser, J. Fuchs, K. Seppelt, *Angew. Chem.* 101 (1989) 1528; *Angew. Chem. Int. Ed. Engl.* 28 (1989) 1526; K. O. Christe, W. W. Wilson, *Inorg. Chem.* 28 (1989) 3275; W. W. Wilson, K. O. Christe, *ibid.* 28 (1989) 4172. In this sense, even the anions of the type [TeX₆]^{2⊖} (X = Cl, Br, I) and [SeX₆]^{2⊖} (X = Cl, Br) and similar antimony and bismuth anions represent no exception from the electron-pair repulsion model. High crystal symmetry (at least the presence of one symmetry center for the central atom) generates dynamically distorted structures. With lower symmetry the structures are even statically distorted: W. Abriel, C. Friedrich, *Z. Naturforsch.* B40 (1985) 1691; W. Abriel, *ibid.* B42 (1987) 415; W. Abriel, E.-J. Zehnder, *ibid.* B42 (1987) 1273; *Acta Crystallogr. Sect. B* 42 (1986) 449; W. Abriel, H. Ehrhardt, *Angew. Chem.* 96 (1984) 965; *Angew. Chem. Int. Ed. Engl.* 23 (1984) 963.
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[6] M. Veith, H. Bärninghausen, *Acta Crystallogr. Sect. B* 30 (1974) 1806; H. Schumann, W. Genthe, E. Hahn, M. B. Hossein, D. von der Helm, *J. Organomet. Chem.* 299 (1986) 67.
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Different Reaction Behavior of Lead Bis(tri-*tert*-butoxygermanate) and Lead Bis(tri-*tert*-butoxystannate) towards Nonacarbonyldiiron**

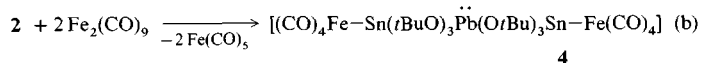
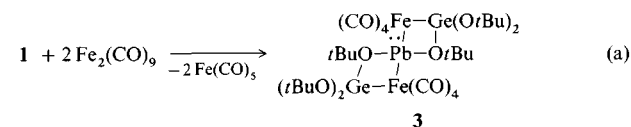
By Michael Veith* and Johannes Hans

Dedicated to Professor Hans Georg von Schnering on the occasion of his 60th birthday

In the course of our comparative studies on molecular compounds of elements of the fourth main group in their lower oxidation state II (cf. Refs. [1-3]) we have now carried out reactions of the homologues lead bis(tri-*tert*-butoxygermanate) **1** and the corresponding stannate **2** with nonacar-



bonyldiiron in order to investigate the stereochemical activity of the lone pair of electrons on the metallic element. Whereas **1** at room temperature contains a linear arrangement of the metallic elements (Ge, Pb and Ge are connected to each other via *tert*-butoxy groups, whereby the lead atom forms the center of the $\bar{3}(S_6)$ symmetric arrangement and thus possesses no stereochemically effective electron pair), the Sn...Pb...Sn array in **2** is bent.^[3] We had expected that it would be possible to influence the coordination sphere of the central lead atom by coupling Fe(CO)₄ units to the Lewis basic germanium and tin atoms in **1** and **2**, respectively, and by the concomitant withdrawal of electron density. As can be seen in Equations (a) and (b) the products **3** and **4** are



formed, which are similar in their composition, behave similarly spectroscopically, and differ noticeably only in their color.^[4]

Single-crystal X-ray structure analyses of **3** and **4** (Fig. 1 and 2) show that **3** has a pseudocrystallographic twofold axis, **4** a crystallographic twofold axis, in each case passing through the lead atom. In **4** the metal atoms are, as expected, coupled one-dimensionally, whereas **3** is spirocyclic, with the lead atom as spirocenter of two four-membered PbFeGeO rings. In the slightly folded, trapezoidal oxametallacycles the lead atom (smallest angle), as expected, takes over the main burden of the ring strain. Whereas all five metal atoms in **3** are coupled via σ bonds, in **4** only the tin atoms are bound directly to the iron atom.

Due to replacement of four *tert*-butoxy groups by two Fe(CO)₄ moieties the environment of the lead atom in **3** is completely different from that in the starting compound **1**.

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**] Chemistry of Alkoxyannates, Part 7. This work was supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.—Part 6: M. Veith, K. Kunze, *Angew. Chem.* 103 (1991) 92; *Angew. Chem. Int. Ed. Engl.* 30 (1991) 95.

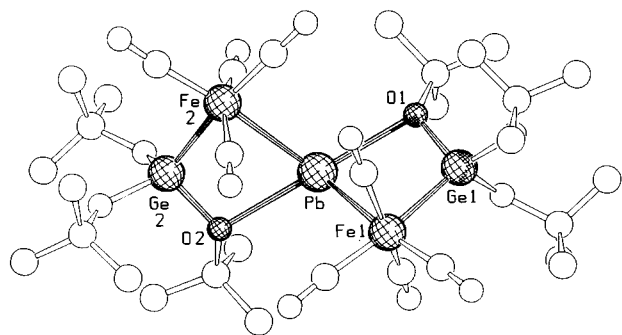


Fig. 1. Molecular structure of **3** [4, 5]. The unmarked spheres are carbon atoms or oxygen atoms. Hydrogen atoms have been omitted. Important bond lengths [Å] and angles [°] (see also text): Ge1–Fe1 2.395(3), Ge2–Fe2 2.396(3), Ge1–O1 1.80(1), Ge1–O5 1.75(1), Ge1–O6 1.782(8), Ge2–O3 1.78(1), Ge2–O4 1.789(9); Fe1–Pb–Fe2 112.9(1), O1–Pb–Fe2 108.5(2), O2–Pb–Fe1 109.1(2), Pb–Fe1–Ge1 80.0(1), Pb–Fe2–Ge2 80.4(1), O1–Ge1–Fe1 102.9(3), O2–Ge2–Fe2 103.0(3), Ge1–O1–Pb 100.5(4), Ge2–O2–Pb 100.6(4).

The lead atom is ψ -trigonal-pyramidally coordinated with two long^[3] Pb–O bonds (2.64(1) Å), which run axially (O1–Pb–O2 = 177.7(2)°) and two likewise long Fe–Pb distances (Pb–Fe1 = 2.939(2), Pb–Fe2 = 2.934(2) Å) in the equatorial plane, which is inclined to the O1–Pb–O2 axis (O1–Pb–Fe = 72.5(2)°, O2–Pb–Fe2 = 72.6(2)°). It can be concluded from the observed geometry and the long distances that the central lead atom is, furthermore, divalent, but in contrast to

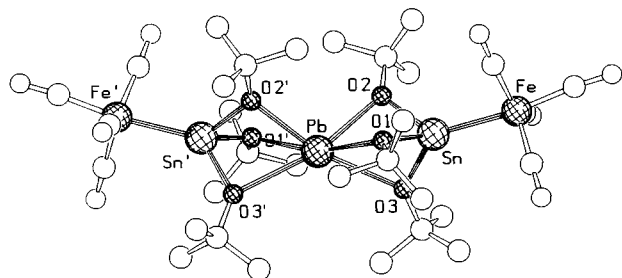
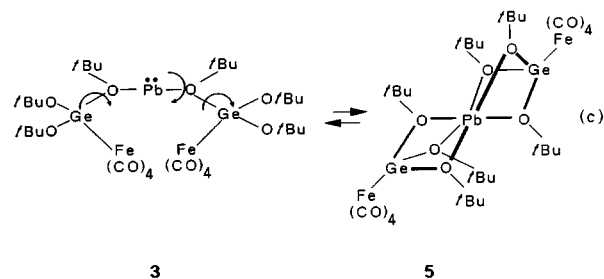


Fig. 2. Molecular structure of **4** [4, 5] (see also caption to Fig. 1). Selected bond lengths [Å] and angles [°]: Pb–O1 2.566(7), Pb–O2 2.482(7), Pb–O3 2.730(7), Fe–Sn 2.476(1), Sn–O1 2.027(6), Sn–O2 2.031(6), Sn–O3 1.992(7), Sn···Pb 3.327(1); C(axial)–Fe–Sn 171.6(5), Fe···Sn···Pb 172.6(1), mean values: O–Pb–O 63.2(1), 113.5 (6.0), 164.6(8), O–Sn–O 84.9(2.0), O–Sn–Fe 128.6(2.0).

that in **1** has a stereochemically effective electron pair (for related and similar coordination spheres of Si^{II}, Ge^{II} and Sn^{II} with ligands such as C(PMe₂)₃⁺ cf. Ref. [6]). The unusually long Pb–Fe contacts in **3** are unmistakable when compared with the Pb–Fe distances in [Pb{Fe(CO)₄}]₄ (2.62(1) Å)^[7] or in the dianion [Pb{Fe(CO)₄}]₂Fe₂(CO)₈²⁻ (2.62(1)–2.83(1) Å).^[8] The bonds from the distorted tetrahedrally coordinated germanium atoms can be regarded as simple σ bonds (in [Ge{Fe(CO)₄}]₄: Ge–Fe = 2.41(2) Å^[9] and in the [GeFe₄(CO)₁₄]²⁻ dianion 2.38(2) Å^[7]), the bonds to the two-coordinate oxygen atoms being expectedly shorter than those to the three-coordinate atoms.^[3] Accordingly, on the basis of the structure and simple bonding models, **3** can be formally described in terms of a Pb²⁺ ion and two [Ge(OtBu)₃Fe(CO)₄]⁻ ions which coordinate the lead atom chelate-like via an oxygen atom and a “nucleophilic” iron atom. This description is naturally to be regarded as only *one* of many possibilities, since other resonance structures can be arrived at with other electron assignments. Also worth men-

tioning is that **3** can be converted, at least purely formally (but reasonably from a structural point of view), into a structure **5** which is analogous to **4**, by rotation about the Ge–O axes of the four-membered rings **3** remaining after opening of the Fe–Pb bonds [Eq. (c)].



How far such a rearrangement in the reverse direction plays a role in the formation of **3** is still unclear. Remarkably, however, only one ¹H-NMR signal is observed for the *tert*-butyl groups of **3** in solution,^[4] which at least indicates intramolecular mobility (also the CO vibration frequencies of **3** hardly differ from those of **4**).

The most important difference between **2**^[3] and **4** is the enhanced distortion of the coordination sphere of the lead atom; thus, for example, the Sn···Pb···Sn angle decreases from 173.5° to 165.0(0)°, and the angle of intersection of the O₃-planes of the tris(*tert*-butoxy) moieties increases from 10.8° to 24.3°. The overall striking feature is that the whole molecule is *curved*, with the curvature extending to the axial carbonyl groups (concerning curved structures in chemistry see Ref. [10]).

It follows from these results that a withdrawal of electron density at the germanium or tin atom in compounds such as **1** and **2** not only has a local effect, but affects the whole molecule, and the stereochemical activity of the lone electron pair at the central lead atom is manifested more strongly. In analogy to the definition presented in Ref. [3], the ligands at the lead atom (tri(*tert*-butoxy)germanate and -stannate) appear to become inflexible in the transition from **1** and **2** to **3** and **4**, respectively. However, as the different structures of **3** and **4** show, the withdrawal of electron density can be so strong that rearrangements occur. Referred to the metal atoms, we have here the “transition” from a structure with bonding oxygen atoms between the metals (**4**) to a metal cluster (**3**).

Experimental

A solution of 0.7 mmol of **1** [3] or **2** [3] in 25 mL of toluene was added dropwise to a suspension of 0.51 g (1.40 mmol) of nonacarbonyldiiron in 15 mL of toluene. After 2 h and 12 h stirring, respectively, the reaction is complete; the solution of **1** turned ruby red, that of **2** yellow. After filtration and removal of solvent in a vacuum there remained a solid residue, which was recrystallized from 5 mL of cyclohexane (**3**) or toluene (**4**); yields: **3**, 0.72 g (84%); **4**, 0.79 g (95%) [4].

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1, 133778-93-5; **2**, 133778-94-6; **3**, 133798-04-6; **4**, 133798-05-7; Fe₂(CO)₉, 15321-51-4.

- [1] M. Veith, *Angew. Chem.* 99 (1987) 1; *Angew. Chem. Int. Ed. Engl.* 26 (1987) 1.
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- [4] **3**: m.p. 180 °C (decomp.); ruby-red to black crystals; ¹H NMR (80 MHz, benzene, 25 °C, TMS): δ = 1.49; IR (THF): ν(CO) = 2048 (s), 1965 (vs), 1925 (vs) cm⁻¹; MS (70 eV): *m/z* 1114 (*M*⁺ - CH₃ with ⁵⁶Fe, ⁷⁴Ge, ²⁰⁷Pb, 60%), 668 ([Pb(O*t*Bu)₃GeFe(CO)₄]⁺, 100). Monoclinic, space group: *C2/c*, *a* = 33.57(2), *b* = 9.329(9), *c* = 29.19(2) Å, β = 103.1(1)°; *Z* = 8; 3478 reflections with 548 *F*_o < 2σ, 281 parameters, *R* = 0.051, *R*_w = 0.049 [5].—**4**: m.p. 190 °C (decomp.); golden yellow crystals; ¹H NMR (80 MHz, benzene, 25 °C, TMS): δ = 1.42; IR (THF): ν(CO) = 2040 (m), 1962 (vs), 1936 (vs) cm⁻¹; MS (70 eV): *m/z* 1217 (*M*⁺ with ⁵⁶Fe, ¹¹⁶Sn, ²⁰⁷Pb, 10%), 712 ([Pb(O*t*Bu)₃SnFe(CO)₄]⁺, 100). Monoclinic, space group *C2/c*, *a* = 14.273(8), *b* = 16.278(9), *c* = 20.158(9) Å, β = 102.1(1)°; *Z* = 4; 3789 reflections with 553 *F*_o < 2σ, 259 parameters, empirical absorption correction, *R* = 0.044, *R*_w = 0.053 [5].
- [5] Further details of the crystal structure investigations are available on request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, W-7514 Eggenstein-Leopoldshafen 2 (FRG), on quoting the depository number CSD-55254, the names of the authors, and the journal citation.
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Ring Opening of a Cyclobutabenzene Complex under Very Mild Conditions**

By Hans G. Wey and Holger Butenschön*

Dedicated to Professor Paul Binger on the occasion of his 60th birthday

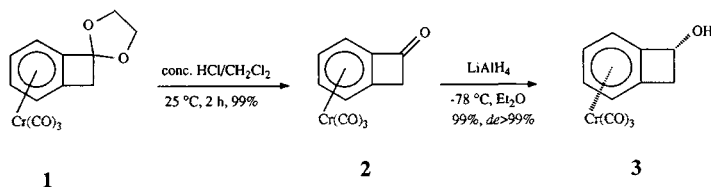
Cyclopentadienyl and arene complexes with annelated four-membered ring moieties are of special interest because of their ring-opening reactions leading to non-planar di-*exo*-methylene compounds and successive stereoselective cycloadditions with dienophiles. So far only two such reaction sequences are known: an η⁵-(bicyclo[3.2.0]hepta-1,3-dienyl)-cobalt(I) complex reacts at 200 °C in 13 h with several dienophiles to give the corresponding [4 + 2] cycloadducts.^[1] Reaction of η⁶-(1-ethoxycyclobutabenzene)tricarbonylchromium(0) with the unusual dienophile *trans*-bis(trimethylsilyl)ethene at 160 ° leads in 6 h to formation of the corresponding tetralin complex.^[2] We report here on the

The reaction of 1-oxocyclobutabenzene with [Cr(CO)₆] affords **2** in only 4% yield. In contrast, **2** is readily accessible in 99% yield as an orange-red powder (m.p. 70.9 °C) by hydrolysis of **1**^[3] in a two-phase mixture of dichloromethane and concentrated hydrochloric acid. The identity of **2**, with its three sp²-C centers in the four-membered ring one of the most strained arene complexes known to date, follows unequivocally from the spectroscopic data (IR, ¹³C NMR; ¹H NMR see Table 1), the mass spectrum, and the correct elemental analysis. Like **2**, the tricarbonylchromium(0) com-

Table 1. ¹H-NMR data of **2–4** and **6**.

2 : (200 MHz, [D ₈]THF): δ = 3.81 (d, 1 H, <i>exo</i> -2-H or <i>endo</i> -2-H, ² <i>J</i> (<i>exo</i> -2, <i>endo</i> -2) = -16.4 Hz), 4.02 (d, 1 H, <i>exo</i> -2-H or <i>endo</i> -2-H), 5.27 (dd, 1 H, 4-H, ³ <i>J</i> (4,5) = 6.2 Hz, ³ <i>J</i> (4,3) = 6.4 Hz), 5.82 (dd, 1 H, 5-H, ³ <i>J</i> (5,6) = 6.2 Hz), 5.89 (d, 1 H, 6-H), 6.02 (d, 1 H, 3-H)
3 : (200 MHz, [D ₈]THF): δ = 2.87 (dd, 1 H, <i>endo</i> -2-H, ² <i>J</i> (<i>endo</i> -2, <i>exo</i> -2) = -13.9 Hz, ³ <i>J</i> (<i>endo</i> -2, <i>exo</i> -1) = 2.2 Hz), 3.35 (dd, 1 H, <i>exo</i> -2-H, ³ <i>J</i> (<i>exo</i> -2, <i>exo</i> -1) = 5.2 Hz), 4.72 (br, 1 H, OH), 5.04 (ddd, 1 H, 4-H, ³ <i>J</i> (3,4) = 6.2 Hz, ³ <i>J</i> (4,5) = 5.9 Hz, ⁴ <i>J</i> (4,6) = 1.3 Hz), 5.06 (dd, 1 H, 1-H), 5.39 (ddd, 1 H, 5-H, ³ <i>J</i> (5,6) = 6.2 Hz, ⁴ <i>J</i> (3,5) = 0.7 Hz), 5.45 (dd, 1 H, 6-H, ⁵ <i>J</i> (1,6) = 0.9 Hz), 5.68 (dd, 1 H, 3-H)
4 : (200 MHz, [D ₈]THF): δ = 3.04 (dd, 1 H, <i>endo</i> -2-H, ² <i>J</i> (<i>exo</i> -2, <i>endo</i> -2) = -13.5 Hz, ³ <i>J</i> (<i>endo</i> -2, <i>exo</i> -1) = 2.3 Hz), 3.34 (dd, 1 H, <i>exo</i> -2-H, ³ <i>J</i> (<i>exo</i> -2, <i>exo</i> -1) = 5.4 Hz), 3.56 (d, 1 H, OH), ³ <i>J</i> (OH, <i>exo</i> -1) = 9.4 Hz), 3.87 (m, 1 H, 5-H, ³ <i>J</i> (5,6) = 6.5 Hz, ³ <i>J</i> (4,5) = 5.9 Hz, <i>J</i> (3,P) = 6.0 Hz), 4.33 (ddd, 1 H, 4-H, ³ <i>J</i> (3,4) = 6.1 Hz, <i>J</i> (4,P) = 3.4 Hz), 4.76 (d, 1 H, 3-H), 5.05 (m, 1 H, <i>exo</i> -1-H), 5.25 (d, 1 H, 6-H), 7.3 (m, 9 H, 8-H, 9-H), 7.52 (m, 6 H, 7-H)
6 : (400 MHz, [D ₈]THF): δ = 2.73 (dd, 1 H, 4-H, ² <i>J</i> (4α, 4β) = -17.0 Hz, ³ <i>J</i> (4β,3) = 11.9 Hz), 3.04 (dd, 1 H, 4-H, ³ <i>J</i> (4α,3) = 6.3 Hz), 3.12 (dd, 1 H, 2-H, ³ <i>J</i> (3,2) = 12.0 Hz, ³ <i>J</i> (2,1) = 3.4 Hz), 3.24 (ddd, 1 H, 3-H), 3.65 (s, 3 H, 12-H), 3.68 (s, 3 H, 11-H), 4.79 (dd, 1 H, ³ <i>J</i> (1,OH) = 3.4 Hz), 5.16 (d, OH), 5.36 (dd, 1 H, 6-H or 7-H), 5.43 (d, 1 H, 8-H or 5-H), 5.58 (dd, 1 H, 7-H or 6-H), 5.63 (d, 1 H, 5-H or 8-H)

plex **3** of the 1-hydroxycyclobutabenzene is also not obtainable by direct reaction of the ligand with [Cr(CO)₆] or [Cr(CO)₃(NH₃)₃].^[3b] Therefore **2** was reduced with lithium aluminum hydride to **3**. The withdrawal of electrons by the tricarbonylchromium(0) moiety leads to a noticeable increase in reactivity of the keto group: whereas 1-oxocyclobutabenzene is reduced to the extent of 83% in 30 min in boiling diethyl ether,^[4] the reduction of the corresponding tricarbonylchromium(0) complex **2** proceeds immediately at -78 °C, as can be seen by the immediate color change from orange-red to pale yellow upon dropwise addition of **2** to the reducing agent. The yield of **3** of 99%—under considerably milder conditions and shorter reaction time—is also significantly higher than that in the reduction of the tricarbonylchromium(0) complexes of indan-1-one (36 °C, 8 h, 84%) and tetralin-1-one (36 °C, 8 h, 72%).^[5] Moreover, the reduction of **2** to **3** proceeds diastereoselectively to the *endo*-1-hydroxy compound: the ¹H- and ¹³C-NMR spectra show only the signals of one diastereomer. Also only one diastereomer is detectable by analytical HPLC (*de* > 99%). That **3** is, in analogy to the products of the reductions of the higher homologues,^[5] in fact the *endo*-1-hydroxy compound could be demonstrated by an NOE experiment. A CO ligand in **3** was replaced by a PPh₃ ligand by irradiation in the presence of PPh₃; this led to formation of **4** in 49% yield.



preparation of the tricarbonylchromium(0) complexes **2** and **3** of the 1-oxo and the 1-hydroxycyclobutabenzene, respectively, as well as on the ready ring-opening of the alkoxide **5** obtainable from **3** and the trapping of the intermediate with conventional dienophiles.

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