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- [3] [4]Radialene (**1**) was isolated as yellow crystals in 11% yield by the intramolecular reaction of hexadecamethyl-3,5,8,10,13,15,18,20-octasilacycloicosa-1,6,11,16-tetrayne with $[(C_5H_4Me)Mn(CO)_3]$ (3 molar equiv) by irradiation ($\lambda > 300$ nm) under reflux in THF. However, when one molar equivalent of the manganese complex was used, the octasilyl-substituted trimethylenecyclopentene derivative was obtained in 17% yield. For π -electron systems, such as **1**, derived from silicon-containing macrocyclic polyacetylene, see H. Sakurai, *Pure Appl. Chem.* **1996**, 68, 327. For the trimethylenecyclopentene and its tetraanion tetralithium, see: A. Sekiguchi, T. Matsuo, C. Kabuto, *Angew. Chem.* **1997**, 109, 2572; *Angew. Chem. Int. Ed. Engl.* **1997**, 36, 2462.
- [4] Spectral data for **1**: m.p. 219–220 °C; 1H NMR ($[D_1]chloroform$, TMS): $\delta = 0.05$ (s, 8H, CH_2), 0.14 (s, 48H, CH_3); $^{13}C\{^1H\}$ NMR ($[D_1]chloroform$, TMS): $\delta = 2.1$ (CH_3), 10.8 (CH_2), 134.7 (C), 167.5 (C); $^{29}Si\{^1H\}$ NMR ($[D_1]chloroform$, TMS): $\delta = -7.8$.
- [5] The [4]radialene dianion, which was obtained by metalating 1,2-dimethyl-3,4-bismethylenecyclobutene with $nBuLi/KOtBu$ or $nBuLi/N,N,N',N'$ -tetramethylethylenediamine, has been characterized by the quenching experiments, see: W. T. Thorstad, N. S. Mills, D. Q. Buckelew, L. S. Govea, *J. Org. Chem.* **1989**, 54, 773.
- [6] Crystal structure analysis of **2**: A single crystal ($0.35 \times 0.30 \times 0.25$ mm) was sealed in a capillary glass tube for the data collection. Diffraction data were collected at 150 K on a Mac Science DIP2020 Image Plate Diffractometer employing graphite-monochromated MoK_{α} radiation ($\lambda = 0.71073$ Å); $C_{36}H_{72}Li_2O_8Si_8$, $M_r = 767.47$, $a = 9.506(1)$, $b = 22.105(1)$, $c = 11.869(1)$ Å, $\beta = 112.197(1)^\circ$, $V = 2309.20(1)$ Å³, monoclinic, space group = $P2_1/c$, $Z = 2$, $\rho_{calcd} = 1.164$ g cm⁻³. The final R factor was 0.031 ($R_w = 0.038$) for 4208 reflections with $I_o > 2\sigma(I_o)$. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-101 602. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).
- [7] The variable-temperature NMR experiment cannot be conducted in $[D_8]toluene$. When the temperature is lowered, the crystals of the bis-contact ion pair precipitate which hampers the dynamic NMR study.
- [8] Spectral data for **2** in $[D_8]THF$: 1H NMR ($[D_8]THF$, 298 K, TMS): $\delta = -0.23$ (d, $J = 12.6$ Hz, 4H, CH_2), -0.17 (d, $J = 12.6$ Hz, 4H, CH_2), 0.04 (s, 48H, CH_3); $^{13}C\{^1H\}$ NMR ($[D_8]THF$, 298 K, TMS): $\delta = 6.4$ (CH_3), 6.9 (CH_3), 11.4 (CH_2), 66.8 (C), 180.7 (C); $^{29}Si\{^1H\}$ NMR ($[D_8]THF$, 298 K, TMS): $\delta = -13.5$; $^6Li\{^1H\}$ NMR ($[D_8]THF$, 298 K, LiCl in MeOH, external standard): $\delta = -0.38$, -0.66 .
- [9] The signal at $\delta = -0.38$, which is assigned to the solvated Li^+ ion, becomes more intense on addition of 6LiBr due to the rapid exchange. However, the intensity of the signal at $\delta = -0.66$ for the Li^+ ion of the CIP remains unchanged because this ion is bound to the π -electron system of the framework.
- [10] The Li^+ ion walk of the CIP is suppressed at 173 K so that Li^+ is fixed at one site of the framework. This gives rise to eight ^{13}C NMR signals for $SiMe_2$ and three methylene groups ($\delta = 9.7$, 10.4, and 11.4), two endocyclic carbons ($\delta = 185.9$ and 177.8), and two exocyclic carbons ($\delta = 83.5$ and 50.2). In the ^{29}Si NMR spectrum, four signals were observed at $\delta = -15.7$, -14.3 , -12.6 , and -11.6 .

$[(CO)_5Cr]_6Ge_6]^{2-}$, A Molecular Organometallic Derivative of the Unknown Zintl Ion $[Ge_6]^{2-}$

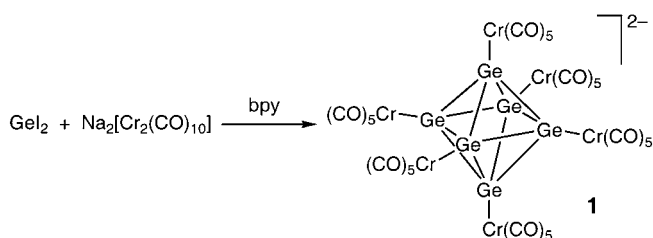
Peter Kircher, Gottfried Huttner,* Katja Heinze, and Gerd Renner

Dedicated to Professor Manfred Weidenbruch on the occasion of his 60th birthday

The synthesis of $[(CO)_5Cr]_6Sn_6]^{2-}$ ^[1] illustrated that clusters composed exclusively of main group elements could be prepared from organometallic precursors; the resulting cluster is embedded in a matrix of organometallic protective groups. At the same time it was shown that octahedral clusters, which obey the usual counting rules^[2] in terms of the structure and the electron count, were not restricted to boron^[3] in main group chemistry. Whereas the $[Sn_6]^{2-}$ ion^[1] protected by organometallic groups was initially the only example for this, in the meantime octahedral clusters that obey the counting rules^[2] have been established as building blocks of solids. Both $[Ga_6]^{8-}$ and $[Ti_6]^{8-}$ exhibit octahedral structures with 14 framework electrons.^[4] Two-dimensional corner-sharing indium octahedra are present in Rb_2In_3 .^[4] The phase KTI contains tetragonally compressed thallium octahedra with 12 framework electrons.^[4] All these octahedral clusters are built from elements of Group 13.^[5, 6] For elements of other groups the only example to date was the $[Sn_6]^{2-}$ ion, which was protected by organometallic groups.^[1]

The reaction principle based on the treatment of pentacarbonylchromate with element halides had with $K_2[Cr(CO)_5]$ and $SnCl_2$ led to the octahedral cluster $[(CO)_5Cr]_6Sn_6]^{2-}$.^[1] The transfer of this principle to other pentacarbonylmetalates and other element halides appeared straightforward; however, numerous attempts proved unsuccessful. We have now found that the reaction of GeI_2 with $Na_2[Cr_2(CO)_{10}]$ leads to the desired anionic cluster $[(CO)_5Cr]_6Ge_6]^{2-}$ (**1**) provided that 2,2'-bipyridine is added to the reaction mixture (Scheme 1).

After the reaction 2,2'-bipyridine (bpy) is mainly contained as ligand in the main product $[(CO)_5Cr]_2Ge_2(bpy)]$.^[7] The role of the 2,2'-bipyridine in the formation of **1** is not clear; however, without it compound **1** is not obtained. Since the



Scheme 1. Synthesis of **1**. bpy = 2,2'-bipyridine.

[*] Prof. Dr. G. Huttner, Dipl.-Chem. P. Kircher, Dipl.-Chem. K. Heinze, G. Renner
Anorganisch-chemisches Institut der Universität
Im Neuenheimer Feld 270, D-69120 Heidelberg (Germany)
Fax: (+49) 6221-545707

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reaction of the starting materials to give **1** must involve electron transfer steps, it seems plausible to assume that the ligand functions as a redox mediator. However, 2,2'-bipyridine could also coordinate intermediary precursors as a chelate ligand and thus stabilize those that are required for the construction of the cluster.^[8] The red Ph_4P salt of **1** can be obtained in analytically pure form as single crystals. The positions and the intensities of the CO bands in the IR spectrum resemble those of the analogous $[\text{Sn}_6]^{2-}$ derivative, which indicates that the two compounds have a similar structure; this was subsequently confirmed by an X-ray crystal structure analysis (Figure 1).^[9, 10]

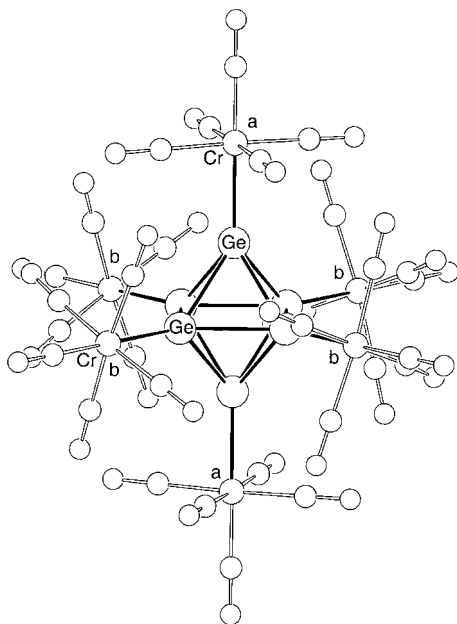


Figure 1. View of the structure of **1** in the crystal. The labeling of the germanium atoms follows the symbolic designation (a, b) of the chromium atoms. The anion has crystallographic D_4 symmetry. The apical building blocks Ge_a and Cr_a lie on the C_4 axis, the equatorial building blocks Ge_b and Cr_b on two of the four C_2 axes in the equatorial plane. Selected distances [pm] and angles [$^\circ$]: $\text{Ge}_a\text{--Cr}_a$ 242.6(2), $\text{Ge}_b\text{--Cr}_b$ 239.5(2), $\text{Ge}_a\text{--Ge}_b$ 254.1(1), $\text{Ge}_b\text{--Ge}_b$ 252.5(2); $\text{Ge}_b\text{--Ge}_a\text{--Cr}_a$ 134.64(3), $\text{Ge}_a\text{--Ge}_b\text{--Cr}_b$ 135.36(3), $\text{Ge}_b\text{--Ge}_b\text{--Cr}_b$ 135.00.

The cluster **1** has crystallographic D_4 symmetry (Figure 1), the $[\text{Ph}_4\text{P}]^+$ ions crystallographic S_4 symmetry. The distances between equatorial and axial germanium atoms are practically equal to the ones between the equatorial germanium atoms (Figure 1). The Ge–Cr distances also do not differ significantly. The Ge–Ge–Cr angles are 135° ; the value that is expected for an ideal octahedral structure with ideal radial orientation of the Ge–Cr bond. In contrast to $[\{(\text{CO})_5\text{Cr}\}_6\text{Sn}_6]^{2-}$ in which the crystallographic C_i symmetry enforces an eclipsed orientation of the equatorial carbonyl groups of the three pairs of $[(\text{CO})_5\text{Cr}]$ entities which are *trans* to each other, these groups adopt a staggered arrangement in **1**. The Ge–Cr distances correspond to those determined for pentacarbonylchromium germylene complexes $[\{(\text{CO})_5\text{Cr}\}\text{--GeR}_2]$ and their base adducts.^[11] The Ge–Ge distances are

similar to those in organogermanium cage compounds (Figure 1): In the prismatic cage of R_6Ge_6 ($\text{R} = \text{CH}(\text{SiMe}_3)_2$), the Ge–Ge distances range between 252 and 258 pm.^[12] Compared with $[\text{Ge}_9]^{4-}$ (Ge–Ge 252–300 pm^[4, 13]), a Zintl ion, which, like the $[\text{Ge}_6]^{2-}$ ion in **1**, obeys the cluster counting rules in terms of its structure and electron count, the Ge–Ge distances in **1** are short. Also in anionic germanium clusters whose electron count deviates from the ideal value, the average Ge–Ge distances are longer than in **1** ($[\text{Ge}_4]^{2-}$: 277–279, $[\text{Ge}_9]^{2-}$: 254–286, $[\text{Ge}_9]^{3-}$: 253–327 and 249–332, $[\text{Ge}_{10}]^{2-}$: 251–295 pm).^[4, 14] Similarly in the heterometallic cluster $[\text{Ge}_9(\mu_{10}\text{--Ge})\text{Ni}(\text{PPh}_3)]^{2-}$, which contains a germanium atom in the center, the distances between the germanium atoms at the surface are longer on average (Ge–Ge 274 pm).^[15]

In accord with the normal to short bond lengths in **1** and the remarkable thermal stability of $[\text{Ph}_4\text{P}]_2[\{(\text{CO})_5\text{Cr}\}_6\text{Ge}_6]$ (decomposition at 105°C), clusters of this kind are intrinsically stable. However, since **1** only forms if 2,2'-bipyridine is present in the reaction mixture, the route according to which such clusters are formed from building blocks protected by organotransition metal groups is so complex that a generalization of this synthetic method is not possible without further detailed investigation.

Experimental Section

All experiments were carried out with Schlenk techniques under an argon atmosphere with exclusion of moisture and oxygen. All solvents were used freshly distilled. NMR: Bruker-Avance-DPX-200, $T = 298\text{ K}$, ^1H NMR (200.13 MHz), $^{13}\text{C}\{^1\text{H}\}$ NMR (50.323 MHz), the solvent was used as internal standard, TMS as external standard: $[\text{D}_6]\text{DMSO}$ ($\delta(^1\text{H}) = 2.49$, $\delta(^{13}\text{C}) = 39.7$), $^{31}\text{P}\{^1\text{H}\}$ NMR (81.015 MHz), external standard 85% H_3PO_4 . IR: Bruker-FT-IR-IFS-66, CaF_2 cells. C,H analysis: Mikroanalytisches Labor, Organisch-chemisches Institut der Universität Heidelberg.

1: GeI_2 (326 mg, 1 mmol) was added in one portion to a stirred solution of $\text{Na}_2[\text{Cr}_2(\text{CO})_{10}]$ (430 mg, 1 mmol) in THF (50 mL). After 5 min, the red solution was treated with 2,2'-bipyridine (156 mg, 1 mmol), and immediately thereafter it was filtered through Kieselgur and concentrated to 3 mL under high vacuum. The dark red residue was purified by chromatography on silica gel (20 cm, $\varnothing = 3\text{ cm}$, diethyl ether). $[\text{Cr}(\text{CO})_6]$, $[(\text{CO})_4\text{Cr}(\text{bpy})]$, and $[\{(\text{CO})_5\text{Cr}\}_2\text{Ge}(\text{bpy})]$ were separated by using diethyl ether and THF as eluent.^[7] The Na salt **1** remained as a dark red band on the column and was subsequently eluted with ethanol. The ethanol solution was treated with solid $[\text{Ph}_4\text{P}]\text{Cl}$ (750 mg, 2 mmol). The Ph_4P salt of **1** precipitated immediately as an orange powder, and was separated from the reaction solution by using a G4 frit. The orange solid was washed with ethanol ($2 \times 5\text{ mL}$) and diethyl ether ($2 \times 5\text{ mL}$), and then dried under high vacuum. The Ph_4P salt of **1** was dissolved on the frit in dimethyl acetamide (DMA; 3 mL) and layered with ethanol (50 mL). This gave analytically pure red single crystals that were suitable for X-ray crystal structure analysis. Yield of crystalline material 250 mg (11%). ^1H NMR: $\delta = 8.01\text{--}7.67$ (m, H_{arom}); ^{13}C NMR: $\delta = 222.2$ (s, CO_{ax}), 217.0 (s, CO_{eq}), 136.2 (s, C_{para}), 135.4 (d, C_{ortho} , $^2J_{\text{C,P}} = 10\text{ Hz}$), 131.3 (d, C_{meta} , $^3J_{\text{C,P}} = 13\text{ Hz}$); ^{31}P NMR: $\delta = 23.5$ (s); IR (DMA): $\tilde{\nu}_{\text{CO}} = 2043$ vs, 2010 w, 1951 vs, 1910 s cm^{-1} ; C,H analysis calcd: C 41.33, H 1.78; found: C 41.27, H 2.01.

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Partially Oxidized Zintl Ions?

The Characterization of



Peter Kircher, Gottfried Huttner,* Laszlo Zsolnai, and Alexander Driess

Dedicated to Professor Heribert Offermanns on the occasion of his 60th birthday

The characterization of mixed-valent and subvalent compounds is an important key principle in the search for solid phases with unusual material properties.^[1] Molecular analogues of such subvalent phases are only found in rare cases in the chemistry of main group elements in the homoatomic aggregates $[\text{E}_x]^{m-}$ ^[2] denoted as Zintl ions and in the isopoly-anions $[\text{E}_y]^{n+}$.^[3] Subvalent compounds are also known in the form of partially oxidized derivatives of molecular element modifications (e.g. $\text{P}_4/\text{P}_4\text{S}_3$). A new class of compounds of this type would be generated if Zintl ions $[\text{E}_x]^{m-}$ could be partially oxidized, in which case—as in P_4S_3 —some of the element–element bonds would be retained. However, a direct transformation of Zintl ions in this sense has not been achieved so far.

The characterization of $[(\text{CO})_5\text{Cr}]_6\text{Sn}_6]^{2-}$ had shown that a de novo synthesis of Zintl ions from mononuclear precursors may be achieved by using organometallic protective groups.^[4] Furthermore, the preparation of $[(\mu_3\text{-OH})_4(\mu_3\text{-O})_4\{(\text{CO})_5\text{Cr}\}_6\text{Sn}_6]^{5-}$ showed that by analogous syntheses compounds are accessible that can be considered formally as completely oxidized derivatives of Zintl ions. Therefore it seemed appropriate to attempt, under modified conditions, to construct cage compounds that contain within the same molecule building blocks in which the main group elements, as in Zintl ions, are linked directly and building blocks in which the main group elements are bridged by oxygen.

In analogy to the preparation of $[(\mu_3\text{-OH})_4(\mu_3\text{-O})_4\{(\text{CO})_5\text{Cr}\}_6\text{Sn}_6]$ from $\text{K}_2[\text{Cr}(\text{CO})_5]$, SnCl_2 , and H_2O in THF,^[5] SnCl_2 was treated with $\text{Na}_2[\text{W}_2(\text{CO})_{10}]$ in the hope that the $[(\text{CO})_5\text{W}]$ derivatives of the desired cage compounds and clusters would be more stable than their $[(\text{CO})_5\text{Cr}]$ analogues. We finally obtained a few crystals of the compound $[(\mu_3\text{-OH})_4(\mu_3\text{-O})_4\{(\text{CO})_5\text{W}\}_6\text{Sn}_6]$ (**1**), the $[(\text{CO})_5\text{Cr}]$ analogue of which is already known (Scheme 1).^[5]

An aggregate, which, unlike the tin(II) compound **1**, also contains tin in a lower oxidation state, was first obtained when the orange solution of $\text{Na}_2[\text{W}_2(\text{CO})_{10}]$ in ethanol was treated with SnCl_2 . This led spontaneously to a red solution, from which, after the addition of a solution of $[\text{Ph}_4\text{P}]\text{Cl}$ in ethanol, the phosphonium salt of $[(\mu_3\text{-OH})(\mu_3\text{-O})_3(\text{OEt})_3\{(\text{CO})_5\text{W}\}_7\text{Sn}_7]^{2-}$ (**2**) precipitated as an orange solid (Scheme 2).

[*] Prof. Dr. G. Huttner, Dipl.-Chem. P. Kircher, Dr. L. Zsolnai, Dipl.-Chem. A. Driess
Anorganisch-chemisches Institut der Universität
Im Neuenheimer Feld 270, D-69120 Heidelberg (Germany)
Fax: (+49) 6221-545707

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