

Synthesis, structure, ESI mass spectrum and magnetic property of a monocationic cluster complex of chromium-sulfide with a hydrido ligand $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6](\text{BF}_4)$

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Received 30 January 2000; received in revised form 16 April 2000

Abstract

A monocationic cluster complex of chromium sulfide $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6](\text{BF}_4)$ (**2**) was prepared by the oxidation of the corresponding neutral complex $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]$ (**1**) with ferrocenium tetrafluoroborate and was characterized by the single-crystal X-ray structure analysis and ESI (electrospray-ionization) mass spectroscopy. Complex **2** has a Cr_6S_8 octahedral cluster core that holds an interstitial hydrogen atom. The Cr–Cr bond distances of **2** are ca. 0.2 Å longer than those of the starting neutral cluster **1**. Magnetic measurements showed that the elongation of the Cr–Cr bonds in the oxidized cluster **2** causes weakening of antiferromagnetic interaction through these bonds, as compared with the neutral complex **1**. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Chromium sulfide; Cluster complex; Monocationic; Octahedral core; Interstitial hydrido ligand; Antiferromagnetic interaction

1. Introduction

The hydride ligands in the octahedral metal cluster compounds are located in: (1) terminal [1]; (2) edge-bridging [1–6]; (3) face-capping [7–11]; or (4) interstitial positions [12–18]. Because the size of a hydrogen atom is far smaller than those of transition metal atoms, locating of hydride ligands in metal hydride compounds is not always easy [19]. We have reported the formation of $[\text{Cr}_6(\mu_3\text{-S})_8(\text{H})(\text{PEt}_3)_6]$ in which six triethylphosphine ligands coordinate to the terminal positions and eight sulfur atoms cap the triangular faces of the Cr_6 octahedral cluster core [20]. We have inferred the position of the hydride in the center of the Cr_6 core from FAB mass spectra, magnetism and reactivity. Single-crystal X-ray structural analysis also supported this conclusion. This chromium cluster complex undergoes one-electron oxidation and reduction. It is interesting to isolate these oxidized or reduced species to study the changes of the cluster core and the magnetic interaction between the paramagnetic chromium

atoms upon redox reactions. It is also important to know the effect of the redox reactions on the interstitial hydride. We have isolated an oxidized complex $[\text{Cr}_6(\mu_3\text{-S})_8(\text{H})(\text{PEt}_3)_6](\text{BF}_4)$ and described in this paper its structure, ESI mass spectrum, magnetism and reactivity.

2. Experimental

All of the manipulations were carried out under argon using Schlenk techniques. The solvents were dried according to established methods. Complex **1** was prepared by following the reported procedure [20]. Other reagents (Aldrich) were used as received.

2.1. Synthesis of $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6](\text{BF}_4)$ (**2**)

An acetone solution (25 cm³) of ferrocenium tetrafluoroborate (0.034 g, 0.12 mmol) was added to a suspension of **1** (0.18 g, 0.12 mmol) in toluene (100 cm³). The mixture was stirred for 5 h, and the solvent was removed in vacuo. The residue was extracted with

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acetone (45 cm³), and acetone was removed under reduced pressure. The resulting solid was extracted with dichloromethane (10 cm³), and hexane (70 cm³) was added to the extract. The solution was allowed to stand at –30°C for 2 days to form black plates of 2·2CH₂Cl₂, which were filtered off, washed with hexane (2 × 10 cm³), and dried in vacuo (0.12 g, 70%). The crystal solvent molecules were easily lost even at ambient pressure. **2**. ¹H-NMR (CD₂Cl₂, 23°C): δ = 1.85 (54H, PCH₂CH₃), δ = –11.77 (36H, PCH₂). The signal due to the interstitial hydride was not detected in the temperature range between 22°C and –80°C. Found: C, 31.54; H, 6.74; F, 5.89; S, 18.53%. Calc. for C₃₆H₉₁BCr₆F₄P₆S₈: C, 31.67; H, 6.72; F, 5.57; S, 18.79%.

2.2. ESI mass spectrum

The positive-ion ESI mass spectrum of **2** was obtained with a ThermoQuest LCQ mass spectrometer. The sample was dissolved in methanol at a concentration of 10 μM, and the solution was introduced into the electrospray source at 3.0 μl min^{–1} using the syringe pump. The ESI source was operated at 5.5 kV and the capillary heater was set to 200°C.

2.3. X-ray structure determination

A single crystal of 2·2CH₂Cl₂ suitable for X-ray analysis was obtained by allowing a dichloromethane–hexane solution to stand at –30°C. The crystal was sealed in a glass capillary for the X-ray measurement. Because the crystal contained dichloromethane molecules escaping from the crystal, the solvent was also sealed. The X-ray measurement of the crystal was performed on a Rigaku R-AXIS CS imaging plate diffractometer with graphite-monochromated Mo–K_α radiation at 21°C. The crystal showed no significant decay over the period of data collection. All calculations were performed using the TEXSAN crystallographic package [21]. The data were corrected for *Lp* factors. The primitive triclinic cell obtained through indexing was transformed to a *C*-centered monoclinic cell. Systematic absences indicated the possible space groups as *Cc* or *C2/c*, and the structure was successfully solved under *Cc*. The positions of the chromium atoms were determined by the direct method (SHELXS-97) [22], and the other non-hydrogen atoms were located using Fourier techniques (SHELXL-97) [22]. The chromium, sulfur, phosphorus and carbon atoms of the cluster unit were refined anisotropically, and all the non-hydrogen atoms of the solvent molecules and tetrafluoroborate anion were refined isotropically. Hydrogen atoms of the triethylphosphine ligands and the dichloromethane solvent molecules were placed in calculated positions by assuming idealized geometries and

setting the C–H distances to 0.95 Å. The interstitial hydrogen atom was not included in the refinement. The final cycle of full-matrix least-squares refinement was based on 5578 observed reflections (*I* > 5σ(*I*)).

Crystal data for [C₃₆H₉₁Cr₆P₆S₈][BF₄]·2CH₂Cl₂. MW = 1535.08, black crystal, 0.29 × 0.26 × 0.11 mm, monoclinic, *Cc* (no. 9), *a* = 16.948(7), *b* = 17.203(8), *c* = 24.15(2) Å, β = 97.42(6)°, *V* = 6980(7) Å³, *Z* = 4, *D*_{calc.} = 1.460 g cm^{–3}, μ = 14.68 cm^{–1}, *T* = 294 K, λ (Mo–K_α) = 0.7107 Å, *F*(000) = 1588. 12 099 reflections (11 196 independent, *R*_{int} = 0.055) were measured in the 2θ range 1.70–60.03° and 5578 data with *I* > 5σ(*I*) were used in refining 548 parameters to give *R* = 0.070 and *R*_w = 0.079 with *R* = Σ||*F*_o| – |*F*_c||/Σ|*F*_o| and *R*_w = {Σ[w(|*F*_o| – |*F*_c|)²]/Σ[w|*F*_o|²]}^{1/2} where *w* = 1. The final atomic parameters are deposited to CCDC.

2.4. Magnetic measurement

Magnetic susceptibility of **2** was measured in the temperature range 2–330 K with a Quantum Design MPMS2 SQUID. The absence of ferromagnetic impurities was checked by the magnetization measurement at 300 K. The magnetic field of the measurement was 5 kG in the temperature range 2–30 K and 10 kG in the range 30–330 K, where the magnetization versus magnetic field curve was linear. The crystalline sample (ca. 45 mg) was put into glass sample holder under a dinitrogen atmosphere. The magnetic susceptibility of the holder was determined separately. Diamagnetic correction was estimated from Pascal's constants [23].

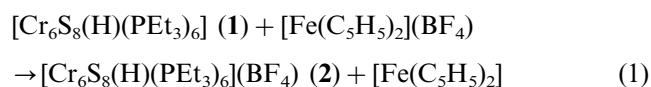
2.5. Other physical measurements

The ¹H-NMR (400 MHz) spectrum was measured in CD₂Cl₂ with a Jeol α-400 spectrometer using CH₂Cl₂ as the internal standard. The FT-IR spectrum was recorded on a Perkin–Elmer 1600 spectrophotometer using a KBr disk.

3. Results and discussion

3.1. Synthesis

The one-electron oxidized cluster [Cr₆S₈(H)(PEt₃)₆]⁺ yielded by the reaction of the neutral complex [Cr₆S₈(H)(PEt₃)₆] (**1**) with ferrocenium tetrafluoroborate, and was crystallized as a BF₄[–] salt **2** from a dichloromethane–hexane solution. The reaction scheme can be represented as follows (Eq. (1)).



The ESI mass spectrum and single-crystal X-ray structure analysis have shown that complex **2** has an interstitial hydrogen atom in the Cr_6S_8 cluster unit (vide infra). During the oxidation, the interstitial hydrogen atom in **1** is not eliminated.

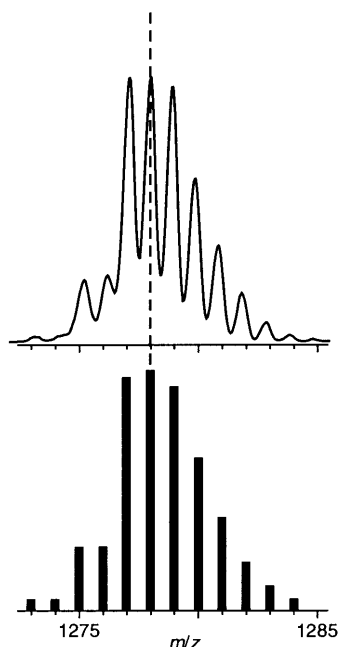


Fig. 1. Measured (top) and calculated isotope patterns (bottom) for $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^+$.

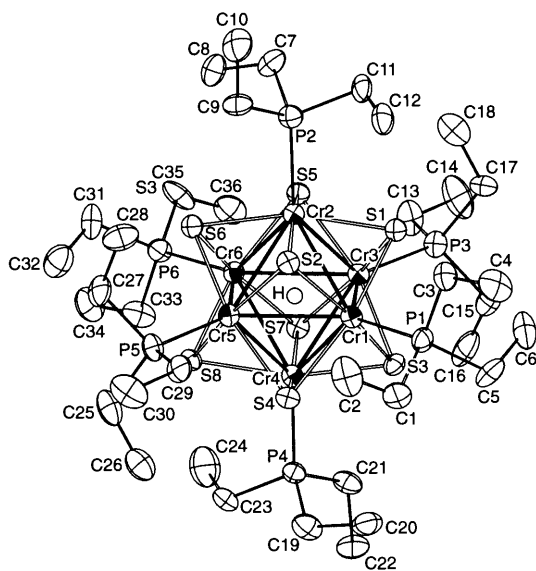


Fig. 2. ORTEP drawing of **2**, showing a $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^+$. The thermal ellipsoids are drawn at the 30% level. The proposed location of the interstitial hydride is shown in this drawing. Ethyl groups are omitted for clarity.

Table 1
Ranges and mean values of selected interatomic distances (Å) and angles (°) for **1** and **2**

	1	2
Cr–Cr	2.592(1)–2.596(1)	2.806(6)–2.854(4)
mean	2.59	2.83
Cr–S	2.327(1)–2.342(2)	2.348(7)–2.389(6)
mean	2.34	2.36
Cr–P	2.395(1)	2.377(9)–2.406(7)
mean		2.39
Cr–Cr–Cr ^a	59.94(1)–60.12(3)	59.3(1)–60.8(1)
mean	60.0	60.0
Cr–Cr–Cr ^b	90.0	89.1(2)–90.8(2)
mean		90.0
Cr–S–Cr	67.32(5)–67.66(4)	72.7(2)–74.6(2)
mean	67.5	73.5
S–Cr–S ^c	89.26(4)–90.00(4)	87.8(2)–90.0(3)
mean	89.6	88.8
S–Cr–S ^d	170.65(5)–170.89(5)	162.2(2)–163.9(3)
mean	170.8	163.1
S–Cr–P	92.23(4)–96.87(4)	94.3(3)–102.3(2)
mean	94.6	98.4

^a Within triangular faces.

^b Within equatorial squares.

^c Between *cis*-sulfides.

^d Between *trans*-sulfides.

3.2. ESI mass spectrum

We measured the positive-ion ESI mass spectrum of **2**. The comparison of the isotope pattern of the molecular ion peak with that calculated is shown in Fig. 1. The molecular ion peak for **2** is assignable to $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^+$ (Fig. 1), indicating that the cationic unit of **2** is $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^+$ having an extra hydrogen atom.

We have reported the positive-ion FAB (fast-atom-bombardment) mass spectrum of the starting neutral cluster **1** [20]. In this spectrum, the molecular ion peak corresponding to $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^+$ was observed. Since it is known that the positive-ion FAB mass spectra of neutral compounds show the molecular ion peak ascribable to either $[\text{Mol}]^{+\bullet}$ or protonated $[\text{Mol}-\text{H}]^+$ [24], the spectrum of **1** could be assigned to $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^{+\bullet}$ or $[\text{Cr}_6\text{S}_8(\text{PEt}_3)_6-\text{H}]^+$, and the confirmation of the existence of the extra hydrogen atom in **1** required the result of magnetic measurement. However, the molecular ion peak of **2** in the ESI mass spectrum is not ascribable to $[\text{Mol}-\text{H}]^+$. If the cationic unit of **2** is $[\text{Cr}_6\text{S}_8(\text{PEt}_3)_6]^{+\bullet}$, the protonated species $[\text{Cr}_6\text{S}_8(\text{PEt}_3)_6-\text{H}]^{2+}$ should be observed at the m/z value (ca. 639) which is a half that of $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^+$.

3.3. X-ray structure analysis

The structure of **2** was determined by single-crystal X-ray structure analysis. A drawing of the cationic unit $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]^+$ is shown in Fig. 2. Ranges and mean values of selected interatomic distances and angles are listed in Table 1. Table 1 also contains the corresponding geometric parameters for the neutral cluster **1**. The cluster comprises a slightly distorted Cr_6 octahedron and eight face-capping sulfur atoms. One triethylphosphine ligand coordinates to each chromium atom.

The ESI mass spectrum has indicated that cluster in **2** has an extra hydrogen atom. Since the terminal and face-capping positions are occupied by triethylphosphine ligands and sulfur atoms, respectively, there are only three possible bonding modes of the hydride: (1) interstitial; (2) bridging; or (3) sulfur-bonded. In the first case, the hydrogen atom is inside the octahedron, while in the second and third cases, the hydrogen atom is outside the octahedron. An absorption due to a chromium–hydrogen or a sulfur–hydrogen stretch was not observed in the FT-IR spectrum [25]. The $^1\text{H-NMR}$ spectra did not show any hydride protons even at -80°C probably due to the paramagnetism of the cluster complex. When the extra hydride was not included in the calculation of the X-ray structure determination, the difference-Fourier map showed a peak inside the Cr_6 octahedron ($0.6 \text{ e } \text{\AA}^{-3}$) while no peak was observed at the bridging or chalcogen-bonded positions in the map. The interstitial position of the hydride in **2** is also supported by its reactivity (*vide infra*).

Though Cr–S (2.34 versus 2.36 \AA) and Cr–P (2.40 versus 2.39 \AA) bond lengths of **1** and **2** are not very different, the 12 Cr–Cr distances (mean 2.83 \AA) of **2** are longer by 0.21–0.26 \AA than those of **1** (mean 2.59 \AA).

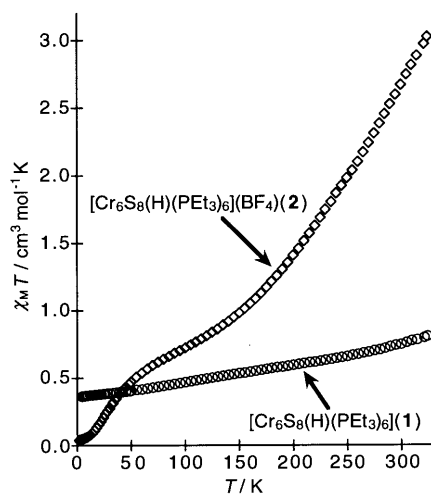


Fig. 3. Temperature dependence of the magnetic susceptibilities of $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6]$ (**1**) and $[\text{Cr}_6\text{S}_8(\text{H})(\text{PEt}_3)_6](\text{BF}_4)$ (**2**) in the form of $\chi_M T$ vs. T .

This elongation also causes the lengthening of Cr–H distances. It is known that the $[\text{M}_6\text{S}_8(\text{PET}_3)_6]$ type non-hydride derivatives of iron [26,27], cobalt [28,29] and molybdenum [30] exhibit several consecutive oxidation states. The complexes in each electron transfer series show shortening of the average metal–metal bond lengths as the cluster core is oxidized. The decrease of the lengths by one-electron oxidation is 0.02–0.04, 0.02 and 0.01 \AA for the iron, cobalt, and molybdenum series, respectively. These values are much smaller than the difference of the metal–metal distances for the chromium hydrides **1** and **2**.

3.4. The reactivity of the hydride

It is known that the positions of the hydrides in cluster compounds are sometimes responsible for their reactivity toward Lewis bases. The reaction of $[\text{Re}_6\text{S}_7(\text{SH})\text{Br}_6]^{3-}$ and $[\text{Re}_6\text{Se}_7(\text{SeH})\text{I}_6]^{3-}$ with primary amines eliminates the chalcogen-bonded hydrogen atom as a proton [31]. The edge-bridging hydrides in $[\text{Re}_7\text{H}_2\text{C}(\text{CO})_{21}]^-$ are also removed by methanol, acetone and THF, leading to the decomposition of the cluster framework [2]. On the other hand, the interstitial hydride in $[\text{HRu}_6(\text{CO})_{18}]^-$ is stable in a methanol solution of KOH and in a THF solution of KH [32].

The reactivity of **2** with ethanol, acetone or $n\text{BuNH}_2$ has been studied. If the deprotonated cluster $[\text{Cr}_6\text{S}_8(\text{PEt}_3)_6]^-$ forms, it should exhibit a different $^1\text{H-NMR}$ spectrum from that of **2**. However, the dissolution of **2** in ethanol or acetone gave only the product showing the same $^1\text{H-NMR}$ chemical shifts for PEt_3 ligands as that of **2**. $n\text{BuNH}_2$ reduced **2** to the starting neutral cluster **1** whose formation was confirmed by FAB mass spectrum, but the hydride was not eliminated. Therefore, it has been concluded that the hydrogen atom of **2** lies at the interstitial position.

In the study of the starting neutral cluster **1**, the inertness of the hydride has been related to both the strong Cr–H bonding and the steric protection by the Cr_6 octahedral skeleton [20]. However, the stability of the hydride in **2** to Lewis bases is considered to be due mainly to the steric hindrance, since the Cr–H bonds of **2** are longer and weaker than those of **1**.

3.5. Magnetic property

The measured temperature dependence of the $\chi_M T$ values (where χ_M represents the molar magnetic susceptibility) of **1** and **2** is shown in Fig. 3. These complexes are paramagnetic over the whole temperature range measured. The magnetic moments do not obey the Curie–Weiss law, and the $\chi_M T$ values decrease as the temperature is lowered. The $\chi_M T$ value for **2** at 2.0 K is $0.038 \text{ cm}^3 \text{ mol}^{-1} \text{ K}$, which is one-tenth the value ($0.375 \text{ cm}^3 \text{ mol}^{-1} \text{ K}$) expected for one unpaired electron. Thus

it is expected that the number of unpaired spins of **2** approaches zero near 0 K, and $S=0$ should be the ground state. This magnetic property is compatible with the even number of MCE (metal cluster electrons) (20 e) derived from the existence of an extra hydrogen atom.

The $\chi_{\text{M}}T$ values seem to be affected by the magnitude of antiferromagnetic interaction between the spins on six chromium atoms. When the interaction is weaker, the energy gaps between the ground and the excited states are smaller, leading to larger number of unpaired electrons and values of $\chi_{\text{M}}T$ for **2** above 50 K. The difference of antiferromagnetic couplings between the chromium atoms in **1** and **2** is due mainly to the interaction through the Cr–Cr bonds. Since the Cr–Cr bond distances of **2** (2.83 Å (av.)) are longer than those of **1** (2.59 Å (av.)), the interaction through Cr–Cr bonds is weaker and chromium atoms are more weakly coupled than those in **1**. This leads to more significant thermal variation of the $\chi_{\text{M}}T$ values for **2** than that for **1**.

4. Supplementary material

Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 139112 for **2**. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

We thank Dr K. Katsumata for use of the SQUID magnetometer.

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