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Sulfur-Bridged Sandwich Cubane-Type Molybdenum−Antimony Cluster. Synthesis, X-ray Structure, and ^{121}Sb Mössbauer Spectra of $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](CH_3C_6H_4SO_3)_8 \cdot 24H_2O$

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A sulfur-bridged sandwich cubane-type molybdenum−antimony cluster $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9]^{8+}$ (**2**) has been synthesized through the reaction of incomplete cubane-type molybdenum cluster $[Mo_3S_4(H_2O)_9]^{4+}$ (**1**) with antimony metal, and has been isolated as **2**$(pts)_8 \cdot 24H_2O$ (**2·pts**) (Hpts = *p*-toluenesulfonic acid), whose structure has been characterized by X-ray crystallography. Crystal data of **2·pts**: triclinic, space group $P\bar{1}$, $a = 14.468(3)$ Å, $b = 18.531(5)$ Å, $c = 13.713(6)$ Å, $\alpha = 105.26(2)°$, $\beta = 119.71(1)°$, $\gamma = 72.48(3)°$, $V = 3016(1)$ Å^3, $Z = 1$, $D_{calcd} = 1.696$ g cm^{-3}, $D_{obsd} = 1.72$ g cm^{-3}, R (R_w) = 4.7 (7.5) for 6593 reflections ($I > 2.0\sigma(I)$). The Mo−Sb distances (3.68[2]) Å) are much longer than the Mo−Mo (2.717[7] Å) distances. Peak positions and ϵ values (λ_{max}, nm (ϵ, M^{-1} cm^{-1})) of the electronic spectrum of **2·pts** in 2 M HCl are 384 (13 220), 625 (10 440), and 700 (10 080). The ^{121}Sb Mössbauer parameters ($\delta = -2.64$ mm s^{-1}, $e^2qQ = 18.1$ mm s^{-1}, $\eta = 0.29$), and the binding energies (eV) and full widths at half-maximum (in parentheses, eV) obtained from XPS spectrum (Sb$_{3d3/2}$, 540.3(2.3); Mo$_{3d3/2}$, 233.2(2.1); Mo$_{3d5/2}$, 230.2(1.9); (C$_{1s}$ = 285.0)) of **2·pts** indicate that the oxidation number of antimony is III, and the mean oxidation number of molybdenum is +3.5.

Introduction

As the extension of the metal incorporation reaction,[1] we report here the reaction of the sulfur-bridged incomplete cubane-type molybdenum cluster $[Mo_3S_4(H_2O)_9]^{4+}$ (**1**) with antimony metal to give a sulfur-bridged sandwich cubane-type molybdenum−antimony cluster $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9]^{8+}$ (**2**). Antimony has high affinity toward sulfur, Sb_2S_3 and Sb_2S_5 being known as sulfides, and progress of the metal incorporation reaction has been anticipated.[2] So far, many antimony halides have been reported, however, reports on mixed-metal clusters containing antimony are very rare,[3] and as for cubane-type molybdenum−antimony clusters, only $[Mo_3SbS_4Cl_3(S_2P(OEt)_2)_4(EtOH)] \cdot EtOH$ (**3**) and $[Mo_3SbS_4Cl_3(SXP(OEt)_2)_4(C_3H_3ON)]$ (**4**, where X = 0.5S + 0.5O) have been reported so far,[4] the starting materials for **3** and **4** being $[Mo_3S_4(S_2P(OEt)_2)_4(L)]$ (L = H_2O, C_3H_3ON) and $SbCl_3$.

We have successfully isolated **2** as a solid sample, **2**$(pts)_8 \cdot 24H_2O$ (**2·pts**) (Hpts = *p*-toluenesulfonic acid), determined the X-ray structure, and characterized the cluster by means of electronic spectroscopy, X-ray photoelectron spectroscopy, and ^{121}Sb Mössbauer spectroscopy. A preliminary report of portions of this work has appeared.[5]

Experimental Section

Materials. *p*-Toluenesulfonic acid (Hpts) was used after recrystallization from water. Antimony (powder) was purchased from Nakalai Tesque Co. Ltd. The molybdenum cluster **1** in dilute HCl and the solid sample $[Mo_3S_4(H_2O)_9](pts)_4 \cdot 9H_2O$ (**1·pts**) were obtained according to published procedures.[6]

Mössbauer Measurements. ^{121}Sb Mössbauer spectra were measured with an Austin Science S-600 Mössbauer spectrometer using a Ca^{121m}SnO$_3$ source (16 MBq) and a germanium detector.[7,8] Both the source and the samples containing 15 mg Sb cm^{-2} were kept at 20 K in a cryostat incorporating a closed-cycle refrigerator.[9] The Doppler

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(1) For example, see: (a) Shibahara, T.; Akashi, H.; Kuroya, H. *J. Am. Chem. Soc.* **1986**, *108*, 1342−1343. (b) Shibahara, T.; Kobayashi, S.; Tsuji, N.; Sakane, G.; Fukuhara, M. *Inorg. Chem.* **1997**, *36*, 1702−1706 and references therein.

(2) Although antimony is sometimes classified as a nonmetal, the element is called a metal here.

(3) For example, see: (a) (Pb−Sb) Beswick, M. A.; Cromhout, N. L.; Harmer, C. N.; Paver, M. A.; Raithby, P. R.; Rennie, M.-A.; Steiner, A.; Wright, D. S. *Inorg. Chem.* **1997**, *36*, 1740−1744. (b) (Co−Sb) Leigh, J. S.; Whitmire, K. H.; Yee, K. A.; Albright, T. A. *J. Am. Chem. Soc.* **1989**, *111*, 2726−2727. (c) (Mo−Sb) Baker, P. K.; Fraser, S. G. *Inorg. Chim. Acta* **1986**, *116*, L1−L2. (d) (Mo−Sb) Baker, P. K.; Bury, A. *Polyhedron* **1988**, *8*, 917−920. (e) (Co−Sb) Foust, A. S.; Dahl, L. F. *J. Am. Chem. Soc.* **1970**, *92*, 7337−7341. (f) (W−Sb) Huttner, G.; Weber, U.; Sigwarth, B.; Scheidsteger, O. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 215−216. (g) (Fe−Sb) Drake, G. W.; Schimek, G. L.; Kolis, J. W. *Inorg. Chem.* **1996**, *35*, 4534−4535.

(4) (a) Lu, S.-F.; Huang, J.-Q.; Lin, Y.-H.; Huang, J.-L. *Huaxue Xuebao* **1987**, *45*, 666−675. (b) Lin, X.; Shang M.; Huang J.-L. *Jiegou Huaxue* **1993**, *12*, 155−159.

(5) Shibahara, T.; Hashimoto, K.; Sakane, G. *J. Inorg. Biochem.* **1991**, *43*, 280−280.

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velocity was measured with an Austin Science LC-9 laser interferometer and calibrated by measuring the ^{57}Fe spectrum of an iron foil at 20 K using a ^{57}Co(Rh) source. The spectra were computer-fitted with twelve quadrupole-split lines using a transmission integral method developed by Shenoy et al.[10] The isomer shifts are given relative to InSb at 20 K.

Other Measurements. XPS data were obtained on a Shimadzu ESCA 850 spectrometer. ICP spectrometry was used for the analysis of molybdenum and antimony using a Shimadzu ICPS-500 analyzer. Electronic absorption spectra were obtained on a Hitachi U-2000 double-beam spectrophotometer. The ϵ values of **2** in 4 M Hpts were calculated based on the concentration of molybdenum and antimony determined by ICP spectroscopy. The ϵ values of **2** in 2 M HCl were calculated based on the concentration of **1** (620 nm, $\epsilon = 315$ M^{-1} cm^{-1}) formed through air-oxidation of **2** in the solvent.

Synthesis of Compounds. All experiments were carried out under a dinitrogen atmosphere. Trace amount of dioxygen in the dinitrogen gas for use was removed by passing the gas into an alkaline pyrogaloll solution.

$[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9]^{8+}$ (2) and $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](CH_3C_6H_4SO_3)_8 \cdot 24H_2O$ (2·pts). A mixture of antimony (2 g, powder) and **1** (0.1 M in 6 M HCl, 20 mL) was stirred for 7 days at room temperature (ca. 20−25 °C), during which the color of the solution changed from green to bluish-green. The resultant solution was filtered and the unreacted powder was removed, then, the filtrate was diluted to 30 times its original volume with water as soon as possible. Chromatography was then performed on a Dowex 50W-X2 column (length 20 cm, diameter 2.2 cm). The resin was washed with 0.5 M HCl to remove any antimony species (colorless, detected by the addition of 1% Na_2S aqueous solution to give red-orange turbidity or precipitate), unreacted **1** (ca. 54%) were eluted with 1 M HCl, and elution with 2 M HCl yielded a green solution (ca. 150 mL) containing **2** (yield 40% based on **1** used). The solution was analyzed to give Mo/Sb = 5.70.

To obtain crystals of **2·pts**, the solution of **2** in 2 M HCl was absorbed onto a short-column cation exchanger, Dowex 50W-X2 (length 1 cm, diameter 2.2 cm). The resin was washed with 0.1 M Hpts to remove chloride ion, and slow elution with 4 M Hpts gave a bluish green solution. The concentrated part of the solution was stored in a freezer. After several days, deep bluish green crystals deposited, which were collected by filtration and washed with ethyl acetate; yield 30% based on the solution **2** in 2 M HCl. Anal. Found (calcd): C, 22.64 (21.84); H, 4.52 (4.58)%.

Structural Determination of $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](CH_3C_6H_4SO_3)_8 \cdot 24H_2O$ (2·pts). A deep bluish green crystal of **2·pts** was mounted in a glass capillary. Crystal data and collection and refinement parameters are summarized in Table 1 (and also in Table S1, Supporting Information). The atomic coordinates, thermal parameters, and $F_o - F_c$ Tables are listed in the Supporting Information (Tables S2−S4, respectively). A large decay (25.6% decline) was observed, and correction was applied. The structure was solved by SHELXS-86,[11] and the remaining non-hydrogen atoms were located from difference maps. All calculations for the refinement were performed using the teXsan crystallographic software package.[12] An empirical absorption correction program was applied.[13] All the carbon atoms of the phenyl groups (except positions 1 and 4) of the pts$^-$ anions are disordered and isotropic temperature factors are applied. No attempt was made to locate hydrogen atoms.

Table 1. Crystal Data for $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](CH_3C_6H_4SO_3)_8 \cdot 24H_2O$ (**2·pts**)

empirical formula	$C_{56}H_{140}Mo_6O_{66}S_{16}Sb_1$
fw	3080.14
space group	$P\bar{1}$
a (Å)	14.468(3)
b (Å)	18.531(5)
c (Å)	13.713(6)
α (deg)	105.26(2)
β (deg)	119.71(1)
γ (deg)	72.48(3)
V (Å^3)	3016(1)
Z	1
D_{calc}	1.696
D_{obs}	1.72
μ (cm^{-1})	11.92
radiation, λ (Å)	Mo Kα, 0.710 69
R^a	0.047
$R_w{}^b$	0.075

^{a} $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. ^{b} $R_w = [\ \Sigma w(|F_o| - |F_c|)^2/\Sigma wF_o{}^2\ ]^{1/2}$; $w = 1/\sigma^2(F_o) = [\sigma_c{}^2(F_o) + {}^1/_4 p^2(F_o)^2]^{-1}$.

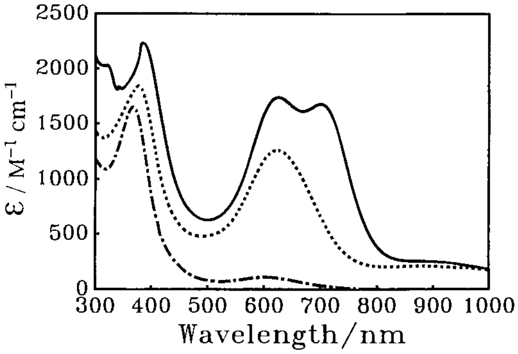

Figure 1. Electronic spectra of clusters: (—) **2** in 2 M HCl; (···) **2** in 4 M Hpts; (—·—) **1** in 2 M Hpts. (For comparison the ϵ values of **2** and **1** are multiplied by $^1/_6$ and $^1/_3$, respectively).

Results and Discussion

Synthesis and Properties of Sandwich Cubane-Type Molybdenum−Antimony Cluster. Through the reaction of **1** with antimony in hydrochloric acid under a dinitrogen atmosphere, we have obtained a sandwich cubane-type cluster **2** in a moderate yield (40%). The use of a higher HCl concentration (6 M HCl) than that of 2 M HCl accelerates the reaction. Electronic spectra of **2** in 2 M HCl and **2** in 4 M Hpts are shown in Figure 1 together with that of **1** in 2 M Hpts. The spectrum of **2** in 2 M HCl has two peaks in 500−800 nm region, while the spectrum of **2** in 4 M Hpts has only peak in the region. Peak positions and ϵ values ($\lambda_{max,}$ nm (ϵ, M^{-1} cm^{-1})) are as follows: 384 (13 220), 625 (10 440), and 700 (10 080) for **2** in 2 M HCl; 377 (11 040) and 623 (7560) for **2** in 4 M Hpts. The spectra of **2** in 0.1 M HCl (peak at 616 nm), in 1 M H_2SO_4 (peak at 592 nm), and in 2 M $HClO_4$ (peak at 615 nm) also have peaks only in the visible region, respectively. Addition of NaCl to the solution of **2** in 0.1 M HCl to give 0.1 M HCl + 1.9 M NaCl solution gives two peaks at 625 and 700 nm in the visible region. We do not have a definite answer to explain these phenomena: coordination of Cl$^-$ ion to the central Sb atom may be one of the causes. The cluster **2** is extraordinarily air sensitive compared with other mixed-metal clusters: for example, $[Mo_3NiS_4(H_2O)_{10}]^{4+}$ in 2 M Hpts is air-oxidized by only ca. 15% in a month,[14] and $[Mo_3FeS_4(H_2O)_{10}]^{4+}$ in diluted HCl is air-oxidized to give $[Mo_3S_4(H_2O)_{10}]^{4+}$ and Fe^{2+} in several hours,[15] and both the solid samples $[Mo_3NiS_4(H_2O)_{10}](pts)_4 \cdot 7H_2O$ and $[Mo_3FeS_4(H_2O)_{10}](pts)_4 \cdot 7H_2O$ can be handled in the air.

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Table 2. Selected Interatomic Distances (Å) and Angles (deg) for $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](CH_3C_6H_4SO_3)_8{\cdot}24H_2O$ (**2·pts**)

distances				angles			
Sb1-Mo1	3.7025(8)	Mo1-S2	2.350(2)	S2-Sb1-S3	77.83(6)	S2-Mo1-S4	88.90(7)
Sb1-Mo2	3.6961(7)	Mo1-S4	2.342(2)	S2–Sb1-S4	77.79(6)	S2-Mo2-S3	89.18(7)
Sb1-Mo3	3.642(1)	Mo2-S2	2.346(2)	S3-Sb1-S4	79.64(6)	S4-Mo3-S3	90.23(7)
mean	3.68[2]	Mo2-S3	2.339(2)	mean	78.4[6]	mean	89.4[4]
Sb1-S2	2.643(2)	Mo3-S3	2.342(2)	S2-Sb1-S3′	102.17(6)	Mo1-S1-Mo2	70.69(5)
Sb1-S3	2.593(2)	Mo3-S4	2.343(2)	S2-Sb1-S4′	102.21(6)	Mo1-S1-Mo3	71.49(5)
Sb1-S4	2.590(2)	mean	2.344[2]	S3-Sb1-S4′	100.36(6)	Mo2-S1-Mo3	71.13(5)
mean	2.61[2]	Mo1-O11	2.175(5)	mean	101.6[6]	mean	71.1[2]
Mo1-Mo2	2.7035(8)	Mo2-O21	2.203(5)	Mo2-Mo1-Mo3	60.10(2)	Sb1-S2-Mo1	95.56(6)
Mo1-Mo3	2.7280(9)	Mo3-O31	2.176(5)	Mo1-Mo2-Mo3	60.39(2)	Sb1-S2-Mo2	95.43(6)
Mo2-Mo3	2.7201(8)	mean	2.18[1]	Mo1-Mo3-Mo2	59.50(2)	Sb1-S3-Mo2	96.95(6)
mean	2.717[7]	Mo1-O12	2.145(5)	mean	60.0[3]	Sb1-S3-Mo3	94.98(6)
Mo1-S1	2.333(2)	Mo1-O13	2.185(5)	S1-Mo1-S2	108.93(7)	Sb1-S4-Mo1	97.17(6)
Mo2-S1	2.340(2)	Mo2-O22	2.146(5)	S1-Mo1-S4	107.80(6)	Sb1-S4-Mo3	95.04(6)
Mo3-S1	2.337(2)	Mo2-O23	2.168(5)	S1-Mo2-S2	108.83(7)	mean	95.9[4]
mean	2.337[2]	Mo3-O32	2.174(5)	S1-Mo2-S3	108.01(6)	Mo2-S2-Mo1	70.29(5)
		Mo3-O33	2.169(5)	S1-Mo3-S3	108.01(6)	Mo2-S3-Mo3	71.06(5)
		mean	2.165[6]	S1-Mo3-S4	107.64(7)	Mo1-S4-Mo3	71.22(5)
				mean	108.2[2]	mean	70.9[3]

Table 3. Selected Interatomic Distances (Å) in Cubane-Type Molybdenum–Antimony Clusters[a] $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](pts)_8{\cdot}24H_2O$ (**2·pts**), $[Mo_3SbS_4Cl_3(S_2P(OEt)_2)_4(EtOH)]{\cdot}EtOH$ (**3**), $[Mo_3SbS_4Cl_3(SXP(OEt)_2)_4(C_3H_3ON)]$ (**4**, X = 0.5S + 0.5O), and $[Mo_3S_4(H_2O)_9](pts)_4{\cdot}9H_2O$ (**1·pts**)[b,c]

cluster	core/charge	Mo–Mo	Mo–M	Mo–μ_3-S	Mo–μ_3-S′	Sb–μ_3-S′	ref
2·pts	$Mo_3S_4SbS_4Mo_3$/+8	2.717[7]	3.680[19]	2.337[2]	2.344[2]	2.609[17]	this work
3	Mo_3SbS_4/+7	2.731[7]	3.822[5]	2.337[3]	2.308[8]	2.775[21]	4
4	Mo_3SbS_4/+7	2.741[7]	3.825[11]	2.339[5]	2.314[7]	2.780[15]	4
1·pts	Mo_3S_4/+4	2.735[8]	–	2.337[5]	2.283[4][d]	–	6c

[a] Hpts = *p*-toluenesulfonic acid, $CH_3C_6H_4SO_3H$. [b] The incomplete cubane-type molybdenum cluster **1·pts** is included. [c] The symbol μ_3-S′ is used for the bridging sulfur atoms connecting two Mo atoms and an incorporated Sb atom, while the symbol μ_3-S is used for the bridging sulfur atoms connecting three Mo atoms. [d] An averaged Mo-μ_2-S distance.

Even in vigorous air-free condition, the peak height at 625 nm decreases and that at 700 nm increases slowly. The peak at 700 nm shifts to longer wavelength (to ca. 715 nm) with increasing peak height, then the peak decreases, while the peak at 625 nm decreases constantly. Introduction of air to the solution accelerate the decrease of the peak heights to give finally the spectrum of **1**. Air-oxidation of **2** is one reason for the spectral change, however, H^+ ion oxidation cannot be negligible: if solid sample **2·pts** was dissolved in 0.1 M HCl + 1.9 M NaCl, similar spectrum to that in 2 M HCl was obtained, and the spectral change is much slower than that in 2 M HCl.

Lu et al. and Lin et al. (see ref 4) have reported the synthesis of clusters **3** and **4** with $Mo_3SbS_4{}^{7+}$ cores by the reaction of $[Mo_3S_4(S_2P(OEt)_2)_4(L)]$ (L = H_2O, C_3H_3ON) with $SbCl_3$, the solvent being the mixture of acetone and ethanol,[4] where, we suppose, no formal redox reaction between $(Mo^{IV})_3$ in **3** (and **4**) and Sb^{III} in $SbCl_3$ occurred. To the contrary, reductive addition of Sb to **1** occurred in the formation of **2** through the reaction of **1** with Sb metal, which is verified by X-ray photoelectron spectroscopy and Mössbauer spectroscopy (see below). We have tried to isolate an intermediate having an $Mo_3SbS_4{}^{7+}$ core from **2** in vain. Either the organic solvents used or the ligand $S_2P(OEt)_2$ employed in **3** and **4**, or both of them, may be operative for the stabilization of the $Mo_3SbS_4{}^{7+}$ core.

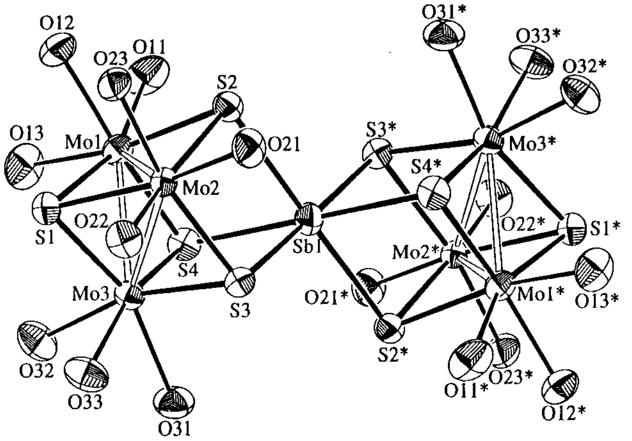

Figure 2. Perspective view of $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9]^{8+}$. The asterisked atoms are related to the unasterisked ones by a center of symmetry.

X-ray Structure of $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](CH_3C_6H_4SO_3)_8{\cdot}24H_2O$ (2·pts). The structure of the cation of **2·pts** is shown in Figure 2. Two incomplete cubane-type cores, Mo_3S_4's, are bridged by an antimony atom which lies on a center of symmetry. Interatomic distances and angles are collected in Table 2 (and in Table S5). Some of the distances are extracted from the Table 2 and are tabulated in Table 3 together with those of the relevant clusters **3**, **4**, and **1**. Although the Mo–Sb distances in **2** are shorter than those in **3** and **4**, no direct bonds exist between the metal atoms. Hydrogen bonds prevail among coordinated water molecules, water molecules of crystallization, and oxygen atoms of pts^- anions (Table S6).

X-ray Photoelectron Spectroscopy. The following binding energies (eV) and full widths at half-maximum (fwhm; in parentheses, eV) are obtained from XPS spectrum of **2·pts**: $Sb_{3d3/2}$, 540.3 (2.3); $Mo_{3d3/2}$, 233.2 (2.1); $Mo_{3d5/2}$, 230.2 (1.9); (C_{1s} = 285.0). Compared with the binding energies of antimony metal ($Sb_{3d3/2}$, 537.50),[16] and molybdenum ($Mo_{3d3/2}$, 233.6 (2.1);

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(16) *Practical Surface Analysis, Vol. 1, Auger and X-ray Photoelectron Spectroscopy*, 2nd ed.; Brigs, D., Seah, M. P., Eds.; John Wiley & Sons: New York, 1988.

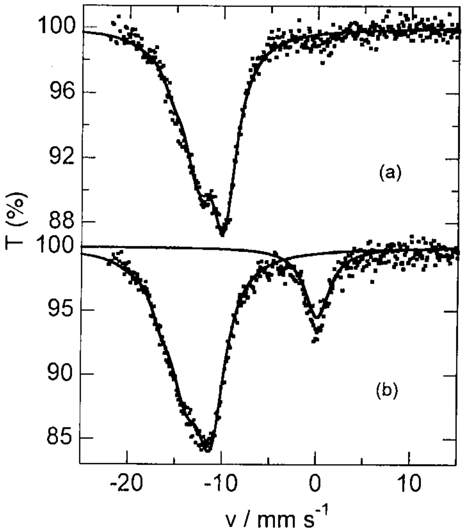

Figure 3. ^{121}Sb Mössbauer spectra: (a) **2·pts**, (b) partially air-oxidized sample of **2·pts**.

$Mo_{3d5/2}$, 230.7 (1.9)) in **1·pts**,[17] those of antimony and molybdenum in **2** are higher and lower, respectively, and it is clear that reductive addition of antimony has occurred. However, the oxidation number of antimony in **2·pts**, that is, either Sb(III) or Sb(V), could not be determined from the XPS experiment.

Mössbauer Spectroscopy. The ^{121}Sb Mössbauer spectroscopy is very useful for the determination of the oxidation number of antimony. The ^{121}Sb Mössbauer spectrum of **2·pts** is shown in Figure 3a, and that of **2·pts** taken after exposure to the air half a day is also shown in Figure 3b. The parameters such as isomer shift (δ), quadrupole coupling constant (e^2qQ), and asymmetry parameter (η) are listed in Table 4. As shown in Figure 2, the X-ray structural study has revealed quasi-octahedral coordination about Sb by six ligating-sulfur atoms. The parameters indicate that the oxidation number of antimony in **2·pts** is III, though both the δ and e^2qQ values seem slightly large for the sulfur ligation.[8] The oxidation numbers of molybdenum atoms in **2·pts** are then calculated to be either 3Mo(III) + 3Mo(IV) or 6Mo(+3.5). The XPS peaks of molybdenum atoms ($Mo_{3d5/2}$ and $Mo_{3d3/2}$) in **2·pts** show no splitting, and the fwhm of the peaks are not large compared with those in **1·pts** (see the section X-ray Photoelectron Spectroscopy). Therefore, the mean oxidation number +3.5 was assigned to the molybdenum atoms in **2·pts**. Partial air-oxidation of **2·pts** gives mixture of Sb(V) and Sb(III). This indicates that the air-oxidation of **2·pts** causes oxidation of antimony at first then oxidation of molybdenum.

Table 4. ^{121}Sb Mössbauer Parameters of $[(H_2O)_9Mo_3S_4SbS_4Mo_3(H_2O)_9](CH_3C_6H_4SO_3)_8 \cdot 24H_2O$ (**2·pts**)

compound	site	δ,[a] mm s^{-1}	e^2qQ, mm s^{-1}	η	relative area (%)
2·pts	Sb(III)	−2.64	18.1	0.29	100
2·pts[b]	Sb(V)	8.67	0	0	17
	Sb(III)	−3.87	18.0	0.12	83

[a] Relative to InSb. [b] Partially oxidized (see text).

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Supporting Information Available: X-ray crystallographic files, in CIF format, for compound **2·pts** are available on the Internet only. Ordering and access information is given on any current masthead page.

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