

When a simple approximation is adopted to Eq. (1), it becomes as follows :

$$h_n' = d_N \cdot \Delta\theta_n / \lambda \sin\theta_1 \dots\dots\dots (3)$$

The D' and M' evaluated by this equation, are also shown in Table 1. They are somewhat smaller than those obtained by the Eq. (1). Whichever method may be adopted, the results show that the thickness of the flaky single microcrystals of metallic gold is very thin, and that its distribution extends over a range of about 30–120 Å. The average value of the thickness is about 80 Å in D and about 30 in M values. (see *Proc. Japan Academy*, 29, 331 (1953)).

3. Formation and Aging of Precipitates. (IV)

Observation of the Gel and the Gelatinous Precipitate by Electron Microscope

Eiji SUITO and Kazuyoshi TAKIYAMA

(Suito Laboratory)

I. Sol and gel having fiber structure. Some inorganic sols or gels have the fiber structure and a few authors have reported on these sols or gels. The authors have also observed vanadium pentoxide sol and mercury sulfosalicylic acid gel by electron microscope.

Vanadium pentoxide sol : 2 g of NH_4VO_3 was mixed with 6 ml. of 3 *N* HCl and precipitated V_2O_5 was washed, then it was dispersed in 200 ml. of water. After one night the sol was composed of short fibrils a few $\text{m}\mu$ wide and about 1 μ long. After one month the fibers became long and its length was a few μ , but the width was only 10 $\text{m}\mu$ as shown in Photo 1. The fibers have probably grown along a particular axis on standing. (Electron diffraction pattern of the fiber crystal was obtained.)

Mercury sulfosalicylic acid gel : 2 g of sulfosalicylic acid was dissolved in 40 ml. of water and some yellow mercury oxide was added to the solution at 80°C. The solution was heated to boil and stored at room temperature. The solution became gelatinous after one night. The electron microscopic observation showed that the gel had the net work or fiber structure (Photo 2). This structure did not change during a few months.

II. Gelatinous precipitates. $\text{Fe}(\text{OH})_3$ and $\text{Al}(\text{OH})_3$ precipitates are gelatinous and hard to filter and wash. The authors observed these hydroxides in order to know the general shape of the gelatinous precipitate.

Ferric hydroxide precipitate : 5 ml. of 1.5 M $FeCl_3$ was diluted with water to 100 ml. and added 6 N NH_4OH , then gelatinous $Fe(OH)_3$ precipitated. The particle of this gelatinous precipitate was sphere, 10 $m\mu$ in diameter, as shown in Photo 3. Sometimes the particles made a special coagulation during the drying of the specimen as shown in Photo 4.

Ferric hydroxide sol : 0.5 ml. of 1.5 M $FeCl_3$ was added to 100 ml. of boiling water. The reddish brown sol was formed. This sol was very stable. The particle of this sol was below 10 $m\mu$. The sol was dialysed by collodion membrane for 2 hours. The color of it became deeper than the original sol. The particle of this sol was also below 10 $m\mu$. The original sol was dialysed for 5 hours, then the gelatinous precipitate, which was similar to the precipitate formed from $FeCl_3$ and NH_4OH , was obtained. The particle was sphere about 10 $m\mu$ in diameter. After 3 months some undispersed portion was contained in the precipitate and this portion was sampled out. Its shape was like string and the another portion was sphere of 10 $m\mu$ as shown in Photo 5. The string was probably by the coagulation of the gelatinous precipitate, but the detail was unknown.

Aluminum hydroxide precipitate : $AlCl_3$ solution was mixed with NH_4OH . The particle of the gelatinous $Al(OH)_3$ precipitate was spherical, and the diameter was 10 $m\mu$ as shown in Photo 6. But $Al(OH)_3$, precipitated from $Al_2(SO_4)_3$, was very smaller than that, formed from $AlCl_3$.

$Fe(OH)_3$ and $Al(OH)_3$ precipitates are gelatinous and hydrated very strongly. These precipitates are composed of very small particles which are about 10 $m\mu$. But the specimen have to be dried to observe by electron microscope, and therefore the particles mentioned above, are the dehydrated parts.

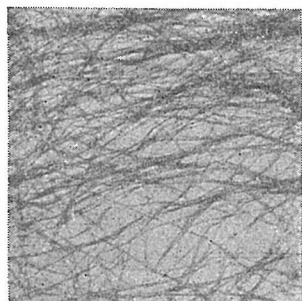


Photo 1. V_2O_5 Sol.
 $\times 10,000$

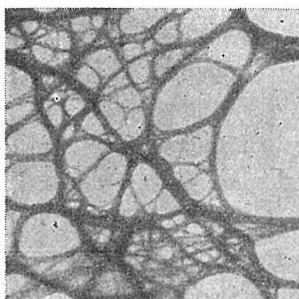


Photo 2. Mercury sulfosalicylic
acid gel. $\times 10,000$

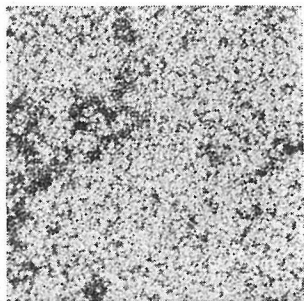


Photo 3. $\text{Fe}(\text{OH})_3$.
 $\times 15,000$

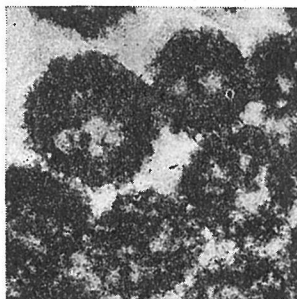


Photo 4. $\text{Fe}(\text{OH})_3$.
 $\times 8,000$

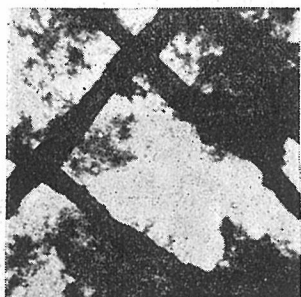


Photo 5. $\text{Fe}(\text{OH})_3$.
 $\times 5,000$

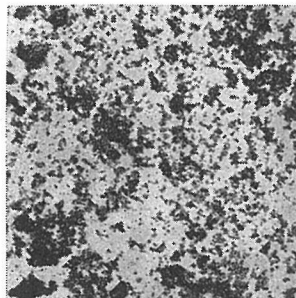


Photo 6. $\text{Al}(\text{OH})_3$.
 $\times 15,000$

4. Morphology of Organometallic Precipitates by Electron Microscope

Masayoshi ISHIBASHI, Eiji SUITO,
Kazuyoshi TAKIYAMA and Eiichi SEKIDO

(Ishibashi and Suito Laboratories)

The shape and size of the precipitate are important for the analytical chemistry. Recently the organometallic compounds have attracted attention for quantitative analysis. And the authors studied organometallic compounds the precipitates of Ni-, Pb-dimethylglyoxime, Cu-, Al-, Pb-, Mg- oxinate and Cu-, Al-cupferrate were observed by electron microscope.

Precipitates were prepared by mixing of necessary reagents for each sample. Ni-dimethylglyoximes were precipitated from NiSO_4 solution of 2.0, 1.0, 0.5, 10^{-1} , 10^{-2} , 10^{-3} , 10^{-4} and 10^{-5} M. But the other precipitates were prepared at the usual condition for the quantitative analysis.