Barium Sulphate Precipitates using EDTA
An Electron Microscopic Study

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EDTA (disodium ethylenediamine tetraacetate) has been used to form complexes with many metallic ions. The authors have been studying the precipitation by instabilizing the metal complex with EDTA. The present note is a report on the sphere shaped barium sulphate precipitate by such a method.

The equivalent quantities of barium hydroxide and EDTA solutions were mixed and ammonia was added to make pH of the solution to 9 to 10. After addition of sulphuric acid, which was equivalent to barium hydroxide, the mixed solution was kept at 95°C. PH of the solution was decreased slowly, and the precipitate began to appear at pH 8. The precipitation was almost completed at pH 7.0 to 7.5. Barium sulphate precipitates were formed at the total concentration of 0.05 mole to 0.01 mole.

The precipitates formed at the initial stage of the reaction were sphere as shown in Fig. 1 and changed gradually to spindle shaped ones as shown in Fig. 2. The size of the precipitated particles increased with decreasing the total concentration of barium sulphate.

In the case that zinc ion was used to decompose barium complex instead of heating, the particles precipitated were also sphere as shown in Fig. 3.

In order to react barium ion with sulphate homogeneously, ammonium persulphate was used instead of sulphuric acid and decomposed by heating[1]. In this case the particles precipitated were rugged sphere as shown in Fig. 4. It seems that these rugged sphere particles are composed of small particles.

Generally speaking, barium sulphate precipitates formed by decomposition of barium-EDTA complex were of sphere or spindle shaped particles, which increased in size with decreasing the total concentration. The size of the precipitated particles was 0.1 to 1.0 μ. The formation of barium sulphate by decomposition of the complex is like to the precipitation from homogeneous solution, and the particles precipitated are somewhat larger than those formed by direct mixing of the reagents[2]. But these are not so large contrary to expectation, because EDTA would react partly as a protective colloid.

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REFERENCES

(1) H. A. Heyn and E. Schupak, *Anal. Chem.*, **26**, 1243 (1954).
(2) E. Suito and K. Takiyama, *Bull. Chem. Soc. Japan*, **27**, 121 (1954).

Electron micrographs of barium sulphate precipitates

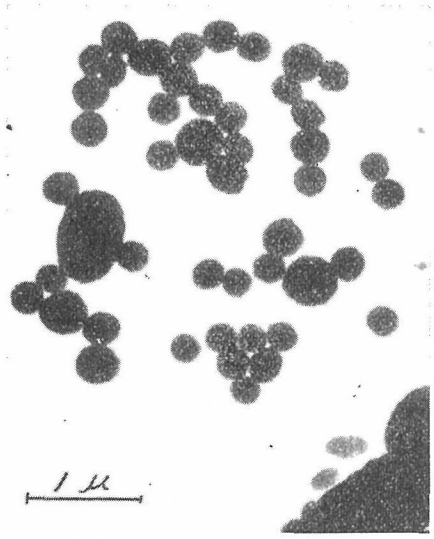

Fig. 1. $BaSO_4$ precipitate from Ba-EDTA and H_2SO_4 by heating. (a) Initial stage of precipitation. (×15,000)

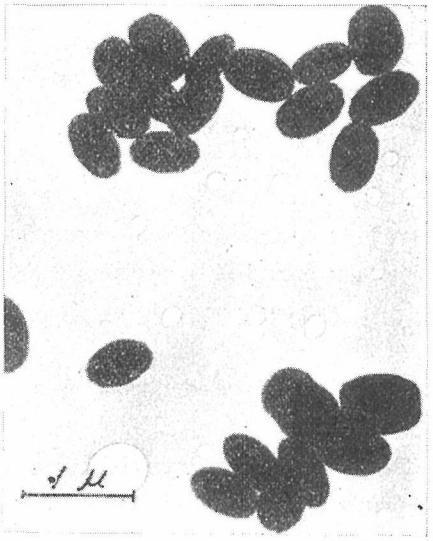

Fig. 2. $BaSO_4$ precipitate from Ba-EDTA and H_2SO_4 by heating. (b) After completion of precipitation. (×15,000)

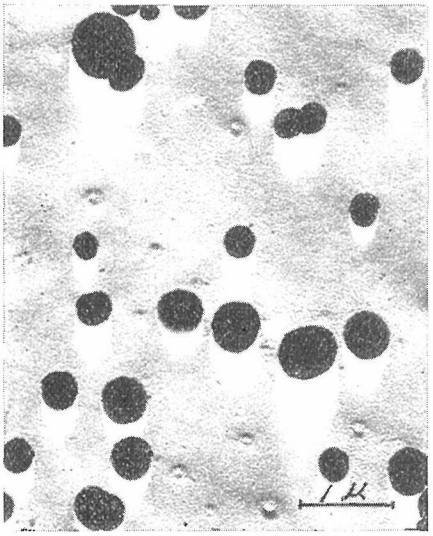

Fig. 3. $BaSO_4$ precipitate from Ba-EDTA and H_2SO_4 by addition of Zn ion. (×12,000)

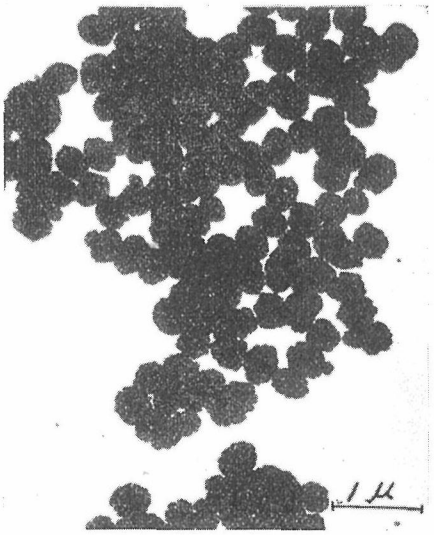

Fig. 4. $BaSO_4$ precipitate from Ba-EDTA and $(NH_4)_2S_2O_8$ by heating. (×12,000)