A METHOD OF REDUCING SOME METALS IN CRYSTALLIZED FORM ON GLASS SLIPS AS PERMANENT MICROSCOPE MOUNTS.

By Jas. H. Bowman.

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The process applies to such metals as are reducible from solution by some other metal.

For some years I had intermittingly attempted the deposition of gold and silver in the crystallized forms known as fern and moss figures on glass slips for use as microscopic objects and had been partially successful. Finally I happened upon a method that gave quite satisfactory results in respect to both the perfection of the figures and the permanency of the mounts.

The metal with which I first obtained success was silver and the method used will indicate a general process which may be applied with variations in manipulation to gold, antimony, copper, bismuth, cadmium, tin, lead, etc., each metal requiring some modification of the treatment peculiar to itself.

Silver Crystals.—A solution is prepared by dissolving silver nitrate in distilled water—1 to 10—and adding an equal quantity of a concentrated solution of zinc nitrate. A small drop of the mixed solution is placed on a chemically clean glass slip and spread by drawing the end of another slip backwards and forwards over it until a thin film covers the desired portion of the slip. Then on filing a piece of metallic zinc in such manner that the filings fall thinly and somewhat evenly over the moist surface, reduction of the silver begins at once, each particle of the zinc becoming a center of crystallization as shown in Plate 1, Fig. 1. The zinc nitrate was introduced to prevent the drying of the solution by evaporation before the reduction of the silver becomes complete. The slip is placed, moist side upward, on a piece of damp filter paper and covered with a bell jar. Under the jar the moist atmosphere retards drying until crystallization is completed, which usually takes from 15 to 20 minutes. The slip is removed and allowed to dry in the air, not completely, however, as incrustations will form on the crystals—but until nearly dry. At this stage a few drops of a thin solution of Canada balsam in xylol, introduced at the side, is run over the preparation, the slide being slanted to allow the balsam to reach every part of the surface. It is then set aside to dry in a dust proof atmosphere.

In an hour or two it may be covered in the usual way practised with balsam mounts. The thin solution of balsam holds the delicate crystals of silver against disruption by the final covering and mounting process.

From the zinc centers radiating, fern-like, crystals spring. If the solution be too deep on the slip the reduced silver will remain an amorphous mass but with a filmy solution the fern-like crystals will be very beautiful, and have a bright metallic luster. If the zinc particles are too sparcely spread, silver nitrate will remain in solution and will, by reduction, blacken the balsam. This does not in any way impair the beauty of the slide as the black gives a good background for the crystalline centers of metals.

Gold Crystals.—Both fern-like and filamentous crystals of this metal are obtained by reducing gold chloride by zinc. A saturated solution of gold chloride is made and slightly acidulated with hydrochloric acid mixed with an equal quantity of concentrated solution of zinc chloride. A very small drop is placed on a cleaned slip and then treated in the manner described for silver. The areas of crystal growth should be close together, otherwise in the spaces amorphous gold will be reduced by the balsam and will give the mount a blemished appearance. The crystals form in about the same time as in the case of silver. If wire gold crystals are desired a very thin film of solution only must be used; by deepening the film, fern-like forms are obtained.

Copper Crystals.—The solution should be composed of a cold saturated solution of copper sulfate mixed with an equal part of concentrated solution of zinc chloride and acidulated slightly with hydrochloric acid. Treat as for silver above. In drying, there is a strong tendency to the formation of a green incrustation which ruins the mount, hence it is advisable to add the thin balsam solution while the mount is still quite moist. If time be not allowed for complete reaction, bubbles of hydrogen are formed and interfere with the perfection of the mount.

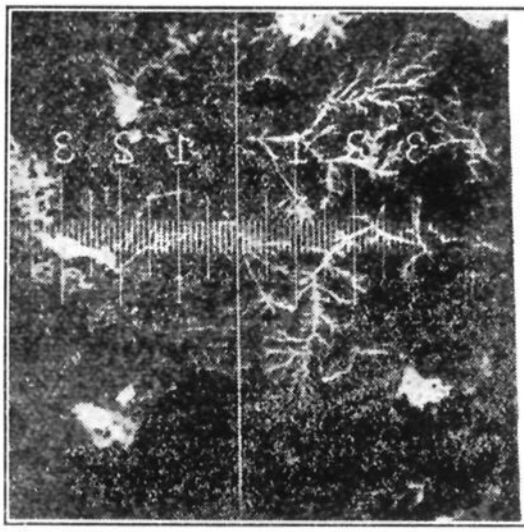
Lead Crystals.—A concentrated cold solution of lead acetate is mixed with an equal quantity of a 25% solution of glycerin in water. The subsequent procedure is similar to that for the other metals. There are two reasons for using glycerin. It prevents too rapid drying and the incrustation of the crystals and further it promotes the solubility of the difficultly soluble lead compound which is precipitated during the process. I do not know of any other agent than glycerin that will prevent the incrustation referred to. It should be noted that unless the crystal centers are close together resolution of the crystals is liable to take place.

Bismuth Crystals.—A concentrated solution of bismuth chloride is mixed with 25% of glycerin, treated as in the other cases. There is a tendency in this case for the reduced metal to be black and amorphous. It is a question of proper acidity which must be determined by trial.

Tin Crystals.—A concentrated solution of tin chloride mixed with an equal quantity of solution of zinc chloride, treated as in the case of silver.

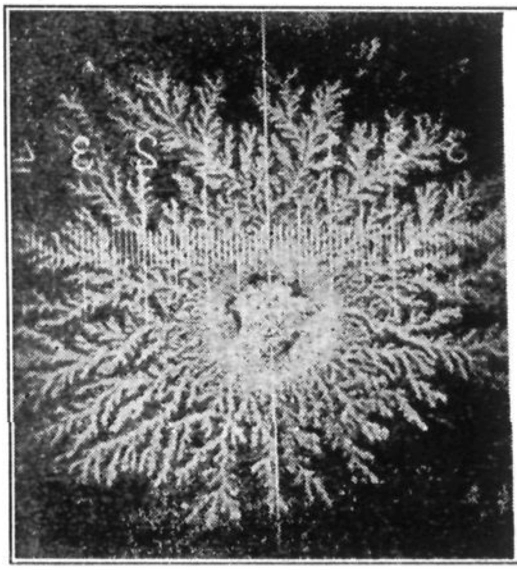
Cadmium Crystals.—Proceed as in the case of tin.

The crystal clusters formed by this method have the reducing particle of zinc left in the center. This objection cannot be very well avoided.

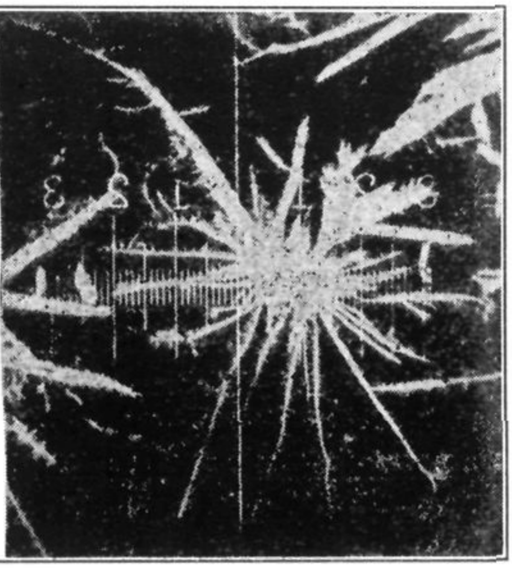


Silver.

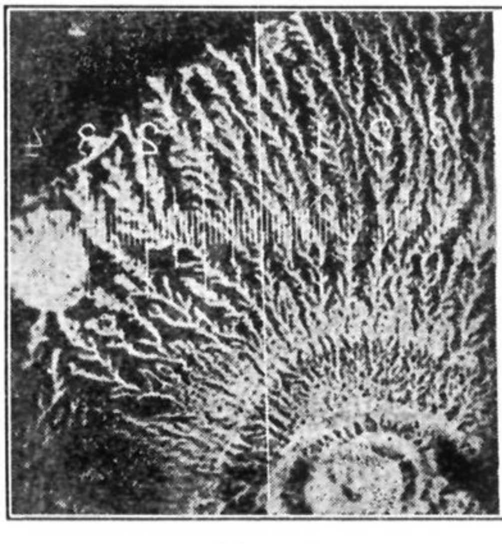
Gold.



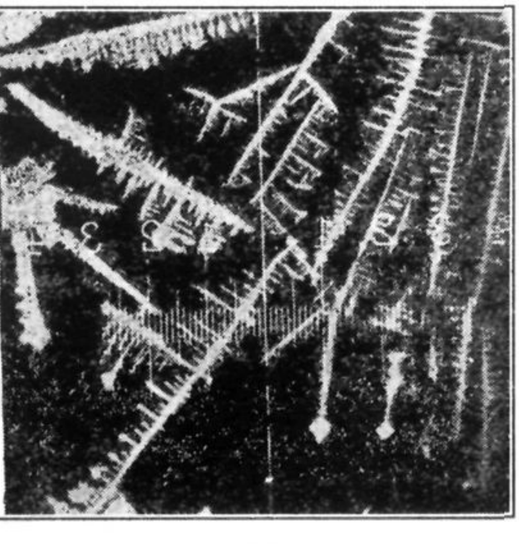
Copper.



Lead.



Bismuth.



Tin.

During the process some of the zinc seems to be carried into the crystals, suggesting that the purity of the metal of the crystals increases as it gets further from the center. This is very noticeable in the examples of both gold and copper.

The crystals from these metals differ much in their branching and curving tendencies. In tin, the crystals are straight and the side growths are at right angles. Lead crystals show less tendency to side branching and curve but little. In gold, as well as in some of the other metals, the branching is quite arborescent. Are the crystals of tin an illustration of compounding by relay?

The crystals formed by this method are well represented in the accompanying plates—they are of some scientific interest and form brilliant microscopic objects when viewed by reflected light.

I owe the microphotographs to the kindness of Mr. J. T. Burt-Gerrans, of the University of Toronto. Each small division of the scale corresponds to 0.015 mm. on the slide.

LONDON, ONT.

[CONTRIBUTION FROM THE HARRIMAN RESEARCH LABORATORY, ROOSEVELT HOSPITAL, NEW YORK CITY.]

A NEW TEST FOR COPPER.[1]

BY W. G. LYLE, L. J. CURTMAN AND J. T. W. MARSHALL.

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Introduction.

The test to be described in this paper is based on the insolubility of the copper salt of α-amino-*n*-caproic acid ($CH_3(CH_2)_3CHNH_2COOH$). Kudielka,[2] in 1908, prepared this acid and its copper, nickel and cobalt salts and determined their solubilities in water. In the course of their work on the quantitative determination of amino acids, Kober and Sugiura[3] state that the insoluble complex which *n*-aminocaproic acid forms with copper may be useful in analytical work.

Some preliminary work on the part of the authors indicated that an aqueous solution of *n*-aminocaproic acid was an exceedingly valuable reagent for the detection of small amounts of copper. It was the object of this investigation to ascertain the most favorable conditions for the use of this reagent and to determine its applicability to solutions of salts of foreign metals containing small amounts of copper.

The reagent was prepared from commercial *n*-caproic acid by the method proposed by Abderhalden, Fuchs and Froehlich[4] and further purified

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[1] Read before the New York Academy of Sciences, March 15, 1915, and the New York Section of the American Chemical Society, April, 1915.

[2] *Monatsh.*, **29**, 351 (1908).

[3] THIS JOURNAL, **35**, 1584 (1913).

[4] *Z. physik. Chem.*, **86**, 455 (1913).