

NOTES AND NEWS

USE OF AMMONIUM HYPOPHOSPHITE IN DETERMINATIVE MINERALOGY

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The attention of mineralogists and chemists should be directed to the ammonium hypophosphite test for certain metals as recently described by Van Valkenburgh and Crawford (1). The test, which was developed and perfected in the chemical and mineralogical laboratories of the University of Colorado, has been used sufficiently to demonstrate its superiority over many of the well established procedures of determinative mineralogy. The present writer has utilized the test successfully with many simple and complex mineral aggregates. Students quickly master the details of the technique and are enthusiastic in their support of the merits of the test.

Ammonium hypophosphite ($\text{NH}_4\text{H}_2\text{PO}_2$) is cheap and easily procured. The writer uses hypophosphite manufactured by the Mallinkrodt Chemical Works and labelled "for manufacturing use only." There is no great advantage in using an expensive, chemically pure reagent.

The procedure for making a test is as follows: about 0.1 gram of finely powdered mineral is mixed with 2 grams of ammonium hypophosphite and heated over a Bunsen burner flame in a porcelain evaporating dish. Decomposition of the hypophosphite produces various gases which ignite and yield thick white clouds of smoke. Heating is continued until a clear fusion is obtained. The fusion is water soluble.

Cobalt, titanium, and tungsten all produce blue melts when hot, but cobalt causes the fusion to turn pink upon cooling. To test for tungsten, add a shallow layer of water to the cold melt and allow it to stand for a few minutes. If tungsten is present the melt will gradually turn deep violet. The speed of this reaction depends on the rate at which the water penetrates the fused mass and on the concentration of tungsten. A shallow water layer above a fusion containing titanium assumes a delicate rose color. To confirm the presence of titanium add hydrogen peroxide, which produces a strong orange-red color in the solution.

Vanadium, chromium, and uranium all color the cold fusion green. The vanadium fusion is distinctive in that it is reddish when hot and changes through yellow to green as it cools. A water layer over the vanadium melt becomes pale green on standing and turns pink upon addition of hydrogen peroxide. To prove the presence of uranium, add sufficient ammonium carbonate to a water layer over the melt to make a distinctly basic solu-

tion. Then add hydrogen peroxide, when, if uranium is present, the solution will turn yellowish orange. If the green color of the melt is caused by chromium, hydrogen peroxide will not cause any color change.

Molybdenum in all minerals, except molybdenite, gives a reddish-brown fusion. With molybdenite the melt is covered with concentrated nitric acid, the acid is boiled off, and the mixture is fused again. With such treatment the fusion assumes a blue or blue-green color. Addition of water causes the melt to turn yellow.

Manganese produces a colorless melt. When concentrated nitric acid is added and the mixture is boiled to remove the excess acid, the melt assumes the familiar permanganate purple.

Tellurium in minerals is freed to produce small metallic globules which float in the melt. Strong heating for two or three minutes causes wine-colored aureoles to form around the globules. Water changes the wine color to black.

Columbium ordinarily is associated with tantalum. Accordingly, a positive test for columbium indicates the presence of tantalum. Fusions of columbium minerals in ammonium hypophosphite are colorless. The mineral powder rarely completely disintegrates. Concentrated hydrochloric acid is added to the fusion, the mixture brought to a boil and a small piece of mossy tin added. If columbium is present, a brilliant blue color results.

Conceivably, mineral mixtures may occur in nature that will not yield satisfactory tests, but in ordinary laboratory procedure involving the examination of pure minerals or simple aggregates, no difficulty will be encountered. The tests are sufficiently sensitive for most purposes. Depth of color in the melt and rate of reaction upon addition of water or acid permit rough quantitative estimates of metallic content.

The writer is indebted to Dr. Van Valkenburgh and Mr. Crawford for permission to make the results of their work available to mineralogists.

REFERENCES

- (1) VAN VALKENBURGH, H. B. AND CRAWFORD, T. C., Detection of certain metals in minerals and ores: *Industrial and Engineering Chemistry*, **13**, 459 (1941).