NOTES AND NEWS

QUALITATIVE DETERMINATION OF FLUORINE IN MINERALS

FRANK C. FOLEY AND PHILIP W. WEST,
University of North Dakota, Grand Forks, North Dakota

A method for the quantitative determination of fluorine in samples of ground water from North Dakota,[1] which proved reliable for amounts of less than one part per million of fluorine, has also been used successfully for the qualitative determination of fluorine in minerals. In most cases the method is rapid and has proved effective when used by elementary students in mineral identification.

The procedure is a modification of the zirconium-alizarin colorimetric determination of fluorine in water[2,3] and depends on the fading of a zirconium-alizarin lake in the presence of fluorine. The pink color fades to yellow with the amount and speed of fading in direct proportion to the fluorine present.

INDICATOR

The zirconium-alizarin indicator is prepared as follows: (a) dissolve 0.17 gm. alizarin sodium sulphonate in 100 ml. distilled water; (b) dissolve 0.87 gm. zirconium nitrate in 100 ml. distilled water; add (a) to (b) slowly and with constant stirring. If kept in a cool, dark place the indicator is sensitive for about six months.

PROCEDURE

Prepare a sodium carbonate bead in a platinum wire loop and fuse into it as much of the powdered mineral as is used in a normal bead test. Dissolve the bead in 1 ml. 1:1 hydrochloric acid in a test tube, then dilute to 5 ml. with distilled water. Add two drops of the indicator and mix. If fluorine is present the original pink color fades to yellow. As an aid in observing fading, particularly where fluorine is present in small quantity, prepare a blank of 1 ml. 1:1 hydrochloric acid diluted to 5 ml. as before and with two drops of indicator. By comparison with the blank, fading is usually seen in the test in two to three minutes though very small quantities may require as long as twenty minutes to show fading. In cases where fading is slight, it is more readily observed by looking directly into the test tube held above a white background.

[1] Abbott, G. A., The fluoride content of North Dakota ground waters as related to the occurrence and distribution of mottled enamel: *N. Dak. Geol. Survey. Bull.* **9**, 1937.

[2] Thompson, T. G., and Taylor, H. J., *Ind. Eng. Chem., Anal. Ed.*, **5**, 8709 (1933).

[3] Sanchis, J. M., *ibid.*, **6**, 134–135 (1934).

Interfering Substances

It was found that large amounts of phosphates, sulphates, arsenates and sulphides cause some fading of the lake. The latter two may be removed by roasting and sulphates by precipitation with barium chloride and filtering through fluorine-free filter paper. No satisfactory method of removing phosphate is known except distillation.

In cases where phosphates are present, or where traces of fluorine must be detected in the presence of large amounts of interfering substances, a Willard-Winter distillation[4] is recommended with a fluorine test made on the distillate.

Fuse 2 or 3 gm. of finely powdered material to be tested with sodium carbonate. Dissolve the fused material in 50 ml. 1:1 sulphuric acid in a 125 ml. distilling flask set up as shown in Fig. 1. Heat until the temperature reaches 140°C., then hold at that temperature by admitting

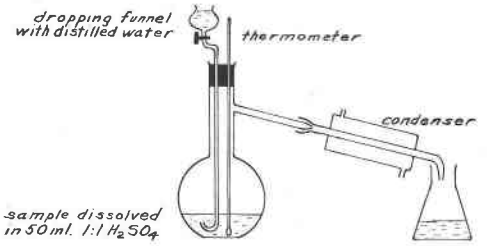

Fig. 1

small amounts of distilled water from the dropping funnel. When 50 ml. of distillate have been caught, remove any sulphate that may have come over during distillation, make alkaline with ammonium hydroxide and concentrate to about 4 ml. Neutralize with 1:1 hydrochloric acid, then add 1 ml. excess. Add two drops of indicator and compare after fifteen minutes with a blank. By this procedure 0.05 mg. per liter may be detected easily. If the amount of fluorine is small, a quantitative estimation may be made by a comparison of the test with standards made from a standard sodium fluoride solution.

[4] Willard, H. H., and Winter, O. B., *Ind. Eng. Chem., Anal. Ed.*, **5**, 7 (1933).