

SIMPLE FIELD METHOD FOR THE DETERMINATION OF PHOSPHATE  
IN PHOSPHATE ROCKSLEONARD SHAPIRO, *U. S. Geological Survey, Washington, D.C.*

A convenient field test has been developed in the Geological Survey for the detection and semi-quantitative estimation of phosphate in phosphate rocks. The basis of the test is the yellow phosphovanadomolybdate color which has been used for the determination of phosphorus in a variety of materials (Mission 1908). Materials containing wavellite or pseudowavellite must first be decomposed in boiling sulfuric and nitric acids to make the test, but tests for apatite may be made by merely shaking the finely ground sample with the nitric acid solution of the reagent. Although the test was devised to distinguish between samples containing 5, 15 and 30 per cent  $P_2O_5$ , it was found capable of giving results to the nearest 5 per cent. Interfering ions such as arsenic are not normally present in sufficient quantity to need consideration. Occasionally the samples must be ignited to remove organic matter.

*Apparatus*

- 1 small porcelain mortar and pestle.
- 1 alcohol lamp.
- 1 measuring spoon—a small metal rod with a 1/16 inch hole drilled to a depth of 1/16 inch. Do not use a rod of plastic or other material which might become charged with static electricity.
- 3 reference solutions described below.
- 1 small dropper with rubber bulb.
- 3 test tubes marked at 15 ml., of the same size as that containing the reference solutions.
- 1 test tube clamp.

*Reagents*

Mixed acid; 15 ml. of concentrated nitric acid plus 30 ml. of concentrated sulfuric acid.  
Vanadomolybdate reagent: Dissolve .300 g. of ammonium metavanadate in 200 ml. of water, and add 50 ml. of concentrated nitric acid. Dissolve 12.5 g. ammonium molybdate in 100 ml. of water. Add this to the vanadate solution and make to 500 ml.

*Preparation of Reference Solutions*

A known phosphate rock sample containing phosphate as apatite and a rock sample known to contain a negligible amount of phosphate are mixed in the proper proportion to give 5, 15 and 30 percent  $P_2O_5$ . For example, using Bureau of Standards Sample #120 (Tennessee phosphate 35.3%  $P_2O_5$  and Sample #1A (limestone) take the following:

- .3 g. #120 plus 1.7 g. #1A  $\approx$  5 per cent  $P_2O_5$
- .9 g. #120 plus 1.2 g. #1A  $\approx$  15 per cent  $P_2O_5$
- 2.0 g. #120 plus .3 g. #1A  $\approx$  30 per cent  $P_2O_5$

Grind each mixture together with the mortar and pestle, and carefully mix again. Then transfer a sample of each of these to test tubes, measuring the samples by volume by dipping the measuring spoon into the powder and sweep away the excess with a finger. Pour vanadomolybdate reagent into each tube to the 15 ml. mark. Stopper the tube with a cork and invert two or three times. After five minutes, the color is at a maximum and remains stable for several days. Permanent standards may then be made by diluting an aqueous solution of potassium dichromate in test tubes until there is a match between each dichromate solution and previously prepared standards. Fifteen ml. of each of the dichromate solutions is then sealed permanently in the test tubes, these will last indefinitely.

#### *Test for Apatite*

Crush a small quantity of the rock to an impalpable powder. Then shake a measured volume of the powder in a test tube with 15 ml. of vanadomolybdate reagent and compare with the permanent standards representing 5, 15 and 30 per cent  $P_2O_5$ .

#### *Testing Samples Containing Wavellite and Pseudowavellite*

Wavellite and pseudowavellite do not dissolve readily in cold dilute nitric acid and must first be boiled with concentrated sulfuric and nitric acids before testing. Apatite may thus easily be distinguished from wavellite and pseudowavellite.

Dip the measuring spoon into the sample, ground to an impalpable powder, to fill the hole in the measuring spoon; sweep away the excess with a finger, and transfer the contents of the spoon to a test tube. Add 10 drops of the mixed acid and heat over the alcohol burner for about a minute. Stop heating before the contents of the test tube go to dryness. Cool and add the vanadomolybdate reagent to the 15 ml. mark. Stopper the tube with a cork, shake, and compare with standards after 5 minutes.

#### REFERENCE

MISSION, G., Colorimetric estimation of phosphorus in steels: *Chem. Ztg.*, **32**, 633 (1908).

#### NEW LOCALITY OF SHORTITE

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#### *Introduction*

The mineral shortite has been identified in the drill core of the Sun Oil Company's Ouray Well located in Uintah County, Utah. It is believed