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A stain test for fluorite

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Abstract

Fluorite, as loose sand grains or in sawed slabs, can be stained using a simple procedure. The two basic steps consist of: (1) coating the grains with calcium sulfate by reacting them for 10 minutes in hot 9 M (18N) sulfuric acid; then, (2) the sulfate coating is stained pink or light purple by immersion for 5 minutes in a cold solution of 0.5M $BaCl_2$ saturated with $KMnO_4$.

Introduction

While investigating the reaction times for the chromate staining of barite (Carlson *et al.*, 1973), it came to our attention that no stain test was known for fluorite (Hosking, 1957, p. 13). After a number of experimental trials, it was discovered that fluorite developed a white chalky coating when it reacted with hot concentrated sulfuric acid. This suggested that a stain test could be developed making use of the known procedure for giving anhydrite (Hosking, 1957, p. 13; Reid, 1969, p. 15) a pink coating.

Procedure

Reagents

(1) Hydrochloric acid solution; 10 per cent HCl; (2) sulfuric acid solution; 9M H_2SO_4 (addition of 0.5 mole sodium borate/liter to the acid enhances the final stain); (3) purple dye (Reid, 1969, p. 15); a solution containing 104.2 g $BaCl_2$ per liter, and 30 g $KMnO_4$ per liter. The latter dissolves slowly, and this solution should be prepared well in advance of its use; (4) basic solution: 50 g NaOH per liter.

Pretreatment

Crushed material or sediment samples should be subjected to thermal shock by boiling the sample in water for a few minutes, rinsing with cold water, and then sieving; this eliminates grains which will crumble during the later steps. Next, the sample is immersed in a warm solution of dilute HCl for 5 minutes. For sawed slabs this increases the relief of the fluorite relative to any carbonate matrix and in loose grains dissolves away or reduces the size of accompanying carbonate grains.

Stain

The sample to be stained is heated for 10 minutes over a boiling water bath in a sulfuric acid solution. The degree of final stain increases with the time the sample is heated. After the required time has elapsed, the acid solution is decanted and the sample rinsed with water.

Next, the sample is immersed for 5 minutes in cold, purple dye. After the required time has elapsed, the dye solution is decanted and the sample washed thoroughly in running water.

Post-treatment

Loose grains are resieved to eliminate partially digested carbonate minerals, and stained slabs are placed in a basic solution and heated until the solution becomes a light to medium shade of blue (purple if the last rinse was incomplete). This step removes any stain absorbed by the matrix. The solution is decanted, and the sample is rinsed and dried.

Result

Fluorite is stained pink or light purple. Dolomite is stained a dark purple. Both gypsum and anhydrite are also stained, but these are not commonly associated with fluorite. The interference by dolomite and calcite can be serious but is made recognizable by the relief differences created by pretreatment with HCl.

Nature of the sulfate coating

Experimentation proved that fluorite can stain only when it has been converted to sulfate using H_2SO_4 having concentrations between 30 and 70 percent. This was surprising, as it had been expected that a coating of anhydrite formed in concentrated H_2SO_4

by the reaction: $CaF_2 + H_2SO_4 = CaSO_4 + 2HF$ should stain.

To understand this, powdered fluorite was attacked using 10 percent, 50 percent, and concentrated H_2SO_4. After blotting the samples dry, the reacted powder was examined on an X-ray diffractometer, and this showed the sulfate coatings to be $CaSO_4$ from the 50 percent and concentrated acid and $CaSO_4 \cdot 2H_2O$ from the 10 percent acid. If instead of blotting the samples dry, the reacted powders were rinsed with water and then air-dried, the coatings consisted of $CaSO_4$ from the concentrated acid, a mixture of $CaSO_4$ and $CaSO_4 \frac{1}{2} H_2O$ from the 50 percent solution, and no coating from the 10 percent solution. From these observations, it is inferred that at 10 percent H_2SO_4, the $CaSO_4 \cdot 2H_2O$ coating washes or dissolves away during the rinsing, leaving no coating to be stained. For the $CaSO_4$ formed from concentrated acid, the washing leaves the coating unchanged, and this unreactive coating does not stain in a cold solution. For unknown reasons, the $CaSO_4$ coating formed at 50 percent H_2SO_4 is still reactive and forms $CaSO_4 \frac{1}{2} H_2O$ with the rinse water. The presence of reactive $CaSO_4$ and $CaSO_4 \cdot \frac{1}{2} H_2O$ on the fluorite grains determines whether or not the grains are stained.

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