THE USE OF THOULET'S SOLUTION FOR HEAVY MINERAL SEPARATION

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PREPARATION AND SEPARATION

(a) Preparation of the Rock Specimen.

The rock was broken into bits about $\frac{1}{4}'' \times \frac{1}{4}'' \times \frac{1}{4}''$ in size, and crushed in a porcelain mortar. The grains were run though a series of sieves so that the final grain size was between .125 and .250 mm. This size was chosen because it was about that of the smaller grains in the thin sections of the same rocks.

(b) Preparation of the Solution.

The solution was prepared by adding distilled water to mercuric iodide powder and potassium iodide crystals in a porcelain evaporating dish in the following proportions:

mercuric iodide	seven parts
potassium iodide	six parts
water	two parts

Eighty-seven grams of mercuric iodide, seventy-five grams of potassium iodide and twenty-seven ml. of water made about fifty ml. of solution. The resulting solution was evaporated over a steam bath until a thin crystalline film began to form over the surface of the liquid, or until a small fragment of pure fluorite (specific gravity = 3.18) just began to rise from the bottom of the evaporating dish. The solution was filtered and its specific gravity checked with a pycknometer. If the specific gravity was too low, a little more mercuric iodide was added to the solution, together with a little hot water, and a re-evaporation was carried out as described above. About two ml. of mercury were kept in the evaporating dish during the process, in order to prevent the separation of free iodine. The maximum specific gravity for the solution (and this should be checked on cold solution, because the specific gravity of the warm solution is somewhat lower) is 3.19, but it was found desirable to use the solution a little below its saturation point, with the specific gravity about 3.15 or 3.16. The solution is a clear, light yellow color.

(c) Technique in Separation

About thirty-five ml. of solution were placed in a separatory funnel and a one gram sample of ground rock was added. The mixture was stirred three or four times, at one minute intervals. When the separation was distinctly clean the heavy portion was drawn off into a funnel containing rapid filtering paper. The filtered solution was caught in a beaker and returned to the bottle of fresh solution. (The solution must be kept in a brown glass bottle with a glass stopper and a few ml. of mercury should be kept in the bottle to prevent the separation of free iodine.) The mineral grains in the filter paper were washed with hot ten per cent potassium iodide solution, these washings being saved for later recovery of the solution. The mineral grains were then washed with hot, distilled water until no yellow color was left on the filter paper. A final washing was made with acetone to aid in drying the grains. The light fraction was drawn off into a funnel large enough to receive all of the solution. This eliminated the necessity for opening and closing the stop-cock of the separatory funnel several times, and thus prevented the scratching or clogging of the stop-cock by the mineral grains. The filtering of this second fraction took about ten to fifteen minutes and it was found helpful to use a hot-water funnel. This inhibited somewhat the crystallization of the solution on the filter paper. The separatory funnel was washed down with hot potassium iodide solution, and the grains were collected with the rest of the light fraction. Further washing and drying were carried out as described above.

Advantages of the Solution

The specific gravity of from 3.12 to 3.16 will keep biotite in mica-rich rocks in the light fraction of the separation. The fairly low viscosity of the fresh solution and its lack of convection currents make a rapid and clean separation possible. It is not volatile and, therefore, gives off no unpleasant fumes. If the rocks are crushed and ready for sampling before the preparation of the solution, about twenty to twenty-five separations may be run through with fifty ml. of solution. Only about one-half to one ml. of solution is lost, but collected in the washings, with each separation.

Disadvantages of the Solution

After continued use the solution becomes more viscous and crystallizes on the filter paper during filtering, thus slowing up the process considerably and causing greater loss of fresh solution with each separation. The use of a hot-water funnel will help some, but after several weeks of use, or even after standing in the bottle for several weeks, the solution begins to deteriorate. The apparatus must be all glass or porcelain since the solution is very corrosive and will attack rubber or metal. It causes bad burns if it touches the skin. Considering the solution expendable, a separation costs about five or six cents. A pound of mercuric iodide plus a pound of potassium iodide costs about $7.50 and yields about three hundred ml. of solution, from which one hundred and twenty-five to one hundred and fifty separations can be made.

Method of Recovery*

The cost of separation may be reduced somewhat by partial recovery of the solution. The following method was tried and found to recover about one-half of the solution.

The used solution is added to the washings. An excess of potassium iodide is thus introduced from the washings. This is desirable since the mercuric iodide will crystallize first out of solution unless the potassium iodide is present in slight excess of the stoichiometric proportions of the double salt. With evaporation, this double salt will be the first to crystallize out. When the solution is evaporated about half-way to dryness and permitted to cool, these crystals may be collected, the liquid being drawn off by suction. The dry crystals are re-dissolved with hot distilled water and the liquid is evaporated as in the original preparation. The resulting solution will have the correct specific gravity since it is made of the pure double salt. The purpose for evaporating only half-way to dryness is to keep the excess potassium iodide in solution. If this should crystallize out and be dissolved along wih the double salt, then the resulting solution will have too low a specific gravity.

* Procedure suggested by A. Rosenzweig, Department of Geology, Bryn Mawr College, Bryn Mawr, Pennsylvania.

THE SUPERABUNDANT INDEX IN THE HEXAGONAL BRAVAIS SYMBOL

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Every crystallographer knows that, in the hexagonal four-index symbol referred to the Bravais axes, the sum of the first three indices is zero. This is why one of these indices, usually the third one, is often omitted altogether and replaced by a period. The general form is then symbolized $\{hk.l\}$. The period stands for $-(h+k)$.

Example: $\{21.4\}=\{21\bar{3}4\}$.

The purpose of the four-index symbol, however, is to bring out the symmetry of the form, by the permutations of the first three indices. Leaving out one of the three defeats the very purpose of the notation. The third index should, therefore, be explicitly written. Some authors represent it by the letter i, others by the same letter i surmounted by a negative sign ($\bar{i}$). This has been a source of misunderstanding.[1]

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[1] Pabst, A., *Am. Mineral.*, **32**, 16–30 (1947). Professor Pabst now states (priv. comm.) that he would like to amend his text by deleting lines 15–16 and footnote 5, on page 21. His criteria are written for the general form $\{hkil\}$.