MINERAL SEPARATION AND PROVENANCE LAB EXERCISE

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INTRODUCTION

This lab exercise has three main objectives: 1) to help students learn to identify mineral grains under the binocular microscope; 2) to demonstrate differences in density and magnetic properties among minerals; 3) to see how a river's sand-sized sediment fraction reflects its source.

PROBLEM

Students will examine mineral separates from sand samples collected from a number of rivers each of which drains a slightly different geologic terrane. Differences in mineralogic composition and mineral abundance will be used to suggest different provenance for each of the sand samples.
These sand samples may be treated as unknowns or the students may be informed of where the sand was sampled. At the end of the mineral separations and binocular microscope identification, the students may be allowed to consult geologic maps of the regions sampled to aid in their provenance determination.

Sand samples from a minimum of the three rivers will be provided. The number of samples examined should be proportional to class size with the students divided into groups of three to four.

PROCEDURE

Sieving

All pebbles and grains >20 mesh must be sieved out. If the sand sample contains significant clay, this must also be removed either by using a flotation table such as a Wilfley or a Gemini gold table or by "gold-panning" the sample. "Gold-panning" involves using either a gold pan or an aluminum baking dish. The sample is placed in the container and wet thoroughly with running water. The wet sample is then agitated to suspend the clay fraction which is decanted into some kind of sediment trap. The clay fraction must be removed because it will make the heavy liquid separation extremely difficult and unsuccessful.

Separations

The sieved sand samples will be subjected to a series of mineral separation techniques which will separate the less dense (<2.9 g/cc) from the more dense (>2.9 g/cc) minerals (Klein

and Hurlbut, 1993) and the magnetic from non-magnetic minerals (Rosenblum, 1953). This will permit easier identification and estimation of percentages.

The samples will be weighed at the start of the exercise and at various points as indicated during the separation. This helps determine how much sample is lost during processing. The final report will include the sample weights, estimation of percentages of minerals at different stages during the process, and a provenance determination for the sand samples which may be verified by examination of a geologic map.

MINERAL SEPARATIONS

Before beginning mineral separations, sand samples must be weighed. All data can be recorded on the attached worksheet.

Heavy Liquids

We will be using sodium polytungsate (SPT) which has a specific gravity of 2.89. It is non-toxic for inhalation and skin contact so it can be used without a fume hood and protective clothing. It is water-soluble. Please wash your hands after use to remove any residue. SPT is **VERY EXPENSIVE** (~$90/lb.) so be careful to recover all of the solution possible and avoid spilling it.

Sodium polytungstate (SPT) evaporates easily so do not leave the bottle uncovered. The heavy liquid separations should be done quickly and efficiently to avoid excessive evaporation of the SPT. All separates need to be rinsed thoroughly, several times with water, preferably distilled water. If rinsing is not carefully done, the sodium polytungstate will dry into a hard mass on the sand grains and prohibit further processing.

Directions.

1. **After making sure that the stopcock is closed,** pour SPT into a large separatory funnel in an amount proportional to sample size. Mark the funnel with the sample number.

2. Add a sand sample and stir with stirring rod until all sand is wet thoroughly.

3. Allow the heavy minerals (>2.9 g/cc) to sink. This should take **no longer** than 10 minutes.

4. With help, draw off the heavy minerals into a funnel lined with a filter paper and collect SPT in an Erlenmeyer flask. Because of the high viscosity of SPT, a vacuum pump can be used to help speed up the draining of the filter paper. Transfer the funnel containing the heavy mineral separate to a second Erlenmeyer flask and rinse several times with water, preferably distilled, from a squeeze bottle.

5. Remove the heavy fraction on the filter paper after rinsing and dry on a watch glass. An acetone rinse will facilitate quicker drying.

6. Repeat the drawing-off, rinsing and drying procedures for the light mineral fraction (<2.9 g/cc).

7. Examine the two fractions (light and heavy) under a microscope and determine the mineral composition and percentages for each fraction.

8. Weigh each fraction to determine amount of sample lost in processing.

9. Repeat the heavy liquid separation on the other sand samples.

Magnetic Separation

This mineral separation step will use just the heavy mineral fraction. Here, separation of the magnetic from non-magnetic minerals is accomplished using a Frantz isodynamic separator.

1. First, to remove the extremely magnetic minerals, such as magnetite, from the heavy mineral fraction we will use a free-fall magnetic separation. Attach a paper cone to the front of the Frantz which has had its magnet rotated into a vertical position. Place a beaker or pan underneath the Frantz and turn magnet current to the maximium (~1.6A).

2. Pour sample through the paper cone. The magnetic minerals will stick to it. Non-magnetic minerals will "fall" through into the beaker. Remove the beaker of non-magnetic minerals when all of the sample has gone through the funnel.
3. Place another beaker underneath the funnel and turn off magnet current. The magnetic fraction will fall into beaker. Save and examine under binocular microscope. Note the mineral compositions and percentages.

4. Examine the non-magnetic minerals under a binocular microscope. Note the mineral compositions and estimate percentages.

Now, the Frantz is changed in orientation so that it is tilted 10° to the back (dial viewed when looking at front of Frantz) and 25° down toward the front (dial viewed when looking at magnet end on). The current will be increased incrementally and fractions collected at different current settings representing differences in magnetic susceptibility. Place all magnetic fractions and the final non-magnetic fraction in separate vials labeled according to amp setting. Examine all fractions, magnetic and non-magnetic under binocular microscope and determine mineral composition and estimate percentages.

5. First, place the non-magnetic free-fall sample in the Frantz cup making sure it is closed, turn up current to 0.3A; and turn on vibrating mechanism. Open the cup enough to allow grains to vibrate freely down the trough through the magnet and into the collection cups.

6. Collect magnetic and non-magnetic fractions. Remember the magnetic fraction is always the one pulled uphill, i.e., the one in the cup at the highest angle, closest to the analyst.

7. Always place the non-magnetic fraction back in the Frantz cup. Repeat the non-magnetic and magnetic separation at 0.5A, 0.8A, and 1.2A.

8. Weigh all fractions, magnetic and non-magnetic, for each sand sample to determine total loss during separation.

9. Use Table 1 to identify the minerals present in each magnetic fraction.

Table 1. Magnetic Susceptibility for Common Heavy Minerals

0.3 A Magnetic	0.5 A Magnetic	0.8 A Magnetic	1.2 A Magnetic	1.2 A Non-Magnetic
magnetite	ilmenite garnet olivine chromite chloritoid	hornblende biotite hypersthene augite actinolite epidote monazite staurolite chlorite tourmaline (dark)	diopside enstatite spinel tremolite muscovite zoisite clinozoisite tourmaline (light) sphene andalusite	zircon apatite pyrite corundum topaz fluorite kyanite sillimanite anhydrite beryl rutile barite

REPORT

Your written report, parts of which may be recorded on the worksheet, should include the following:

1. Sample fraction weights at indicated points during the separation and a total percentage of loss during separation.

2. Estimated mineral percentages and compositional differences between separation fractions.

3. A summary of the total mineral composition for each sand sample.

4. A reasonable guess for the source rock or rocks of the sand sample based on mineralogy and mineral percentages.

5. Verification of provenance determination by examination of a regional geologic map.

REFERENCES

Klein, Cornelius and Hurlbut, Cornelius S., Jr. (1993) *Manual of Mineralogy*. 21st edition, John Wiley and Sons, Inc., New York, 681 p.

Mack, Walter N. and Leistikow, Elizabeth A. (1996) Sands of the World. Scientific American, 275, 62-67.

Rosenblum, Sam (1953) Magnetic susceptibilities of minerals in the Franz isodynamic magnetic separator. American Mineralogist, 43, 170-173.

ADVICE TO THE INSTRUCTOR

This lab is designed for small mineralogy lab sections with 15 students being a reasonable upper limit. I have the students work in groups of three or four so that they can all have a task in each step of the separation process. With larger groups, some students cannot actively participate in all separation steps and may lose interest.

It is also necessary to have some mineral separation equipment available. My research specialty is fission-track dating so I have state-of-the-art facilities. Most large research universities may be similarly equipped. Smaller colleges may not have either a Franz magnetic separator or appropriate large separatory funnels. In this case, it is probably not wise to do this lab.

Separatory funnels - Remember sand-sized grains need to pass through the stopcock so use a large (1000 ml) separatory funnel. Do a trial run with the separatory funnels before you choose to do the lab with students. Be sure to take the stopcocks out of the funnels and rinse well immediately after doing the separation otherwise, the stopcock may freeze shut with SPT.

Sodium Polytungstate (STP) - This product, although non-toxic, is more difficult to use than the toxic heavy liquids. As indicated previously, it evaporates extremely quickly and is very viscous. Thorough rinsing of samples and separatory funnels is absolutely necessary! Using a small vacuum pump to help drain the liquid is highly recommended. This will cut the heavy liquid separation time in half and reduce student restlessness.

Sodium Polytungstate (STP), as well as information on recovering STP from its solid form in case of evaporation, can be obtained from the following vendor:

POLY-GEE Sodium Polytungstate (density 2.8 liquid or powder)
Geoliquids, Inc.
15 E. Palatine Rd. Suite 109
Prospect Heights, Illinois 60070
800-827-2411
847-215-0938
847-215-9821 (fax)

Franz Isodynamic Separator - If your department does not have one of these, a separation of the very magnetic minerals (magnetite) can be accomplished with an hand magnet held behind a sheet of paper. If you do have access to a Franz, Table 1 gives the magnetic susceptibilities for common heavy minerals. This will give you an idea of what minerals appear in different fractions with increasing current.

WORKSHEET FOR MINERAL SEPARATION LAB

Initial Sample Weight (after sieving) _____g **Sample Number: _____**

STP Separation:

Light (<2.9 g/cc) Mineral Weight _____g Mineral Composition (%)

Heavy (>2.9 g/cc) Mineral Weight _____g Mineral Composition (%)

Loss of sample _____g

Magnetic Separation:

Sample Weight Before Separation _____g

Free-Fall Magnetic Fraction Weight _____g Mineral Composition (%)

Non-Magnetic Sample _____g Mineral Composition (%)

0.3 A Magnetic Fraction Weight _____g Mineral Composition (%)

0.5 A Magnetic Fraction Weight _____g Mineral Composition (%)

0.8 A Magnetic Fraction Weight _____g Mineral Composition (%)

1.2 A Magnetic Fraction Weight _____g Mineral Composition (%)

1.2 A Non-magnetic Fraction Weight _____g Mineral Composition (%)

Total Magnetic and Non-Magnetic Fractions_____g Mineral Composition of Entire Sample (%)

Total Sample Loss _____g