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Improved heavy-liquid separation at fine particle sizes

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Abstract

A method is described for effective heavy-liquid separation of material in the particle size range 50 to 10 μm. The method involves centrifugal separation of the material in a necked glass tube, in which a density gradient column spanning the specific gravity range of the light minerals is positioned above the constriction, the specific gravity of the heavy liquid from the base of the density gradient column to the bottom of the tube being the desired specific gravity of separation. When the sample is stirred into the gradient and centrifuged, the light mineral particles spread out loosely through the density gradient and allow the heavy particles to pass through into the lower compartment of the tube. This method has been tested with various heavy liquids and mineral mixtures, and has been shown to be consistently more effective than the normal method using a liquid of uniform specific gravity, in which heavy particles tend to be entrained in the compact mat of light particles which rises to the top of the liquid during centrifugation.

Introduction

Heavy-liquid separation is an important tool of the mineralogist in the concentration of specific minerals or groups of minerals, and facilities for heavy-liquid separation are almost ubiquitous in geological and mineralogical laboratories. The most commonly used liquids are bromoform (sp.gr. 2.89), tetrabromoethane (sp.gr. 2.96), di-iodomethane (sp.gr. 3.32) and Clerici solution (sp.gr. up to 4.3 at room temperature). The first three (organic) liquids can be diluted with acetone, alcohol, or NN-dimethylformamide, among others, whereas Clerici solution is diluted with distilled water. Separation of grains coarser than about 50 μm is normally done statically in a separation funnel or similar device, whereas finer-sized material is normally separated centrifugally, using one of a number of separation tubes or sleeves.

With the advent of the electron-probe microanalyzer the need for separating pure mineral samples for chemical analysis has decreased considerably. However, such samples are still required for geochronology studies, and in beach-sand exploration the proportion of 'heavy minerals' is the first parameter determined on drill-core material. In ore-dressing mineralogical studies, heavy-liquid separation still forms an integral part of the assessment procedure, both with regard to concentrating small proportions of valuable heavy minerals for microscopic study, and for determining the liberation characteristics of the valuable minerals (Muller *et al.*, 1969; Henley *et al.*, 1973). In addition, pure samples of specific minerals may need to be prepared for analysis to determine how closely they meet commercial specifications. In contrast to much of the normal geological and mineralogical heavy-liquid separation work, ore-dressing mineralogical evaluation commonly requires separation of finely-ground material, much of which may lie within the size range 50 to 10 μm. Heavy-liquid separation within this size range requires the use of a centrifuge, and it is the purpose of this note to describe an improved separation procedure for this type of material.

The improved method

The improved method was developed as a result of problems encountered in the centrifugal separation of particles in the size range 50 to 10 μm using a heavy liquid of uniform density. Our normal centrifugal heavy-liquid separation procedure involves the use of a simple necked glass tube (Fig. 1) of about 130 ml capacity in which the angle of necking is very shallow. After centrifugation a conical plunger is inserted gently through the 'floats' to seal off the neck, at which stage 'floats' and 'sinks' can be recovered sepa-

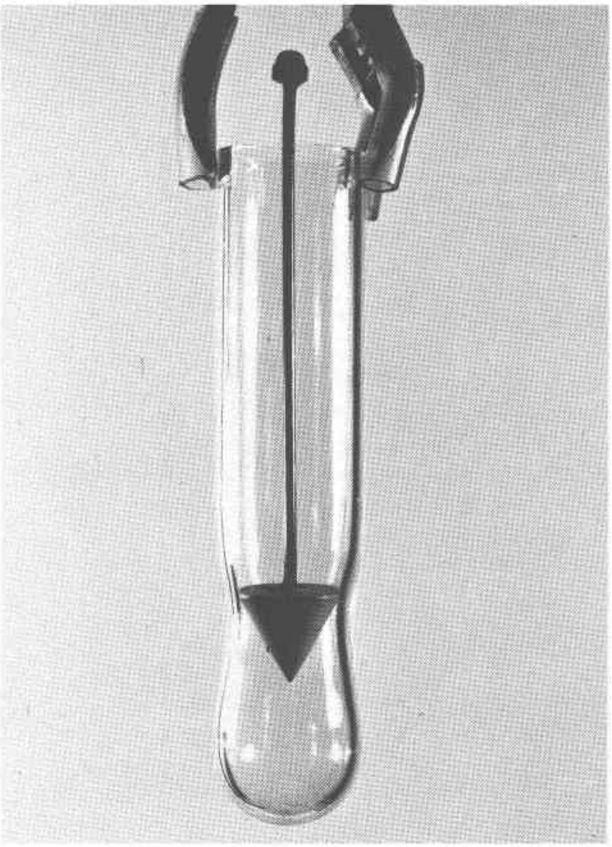

Fig. 1. Necked glass tube and plunger for centrifugal heavy liquid separation.

rately. This procedure has been found to be simpler to use than any of the multiple-tube centrifuge systems, and can be used easily with all heavy liquids, although care has to be taken with high-density Clerici solution because of the danger of shattering the glass if the centrifuge is run at high speed. However, despite its simplicity, repeated separations are required to give good recovery of heavy minerals into 'sinks,' because of entrainment of a significant proportion of the heavy minerals in the predominant light fraction. The entrainment problem increases as the particle size decreases (and the number of particles per unit weight increases), and is particularly acute in the −20+10 μm size range (10 μm is the approximate practical lower size limit of heavy liquid separation; below this size entrainment and flocculation prevent effective separation).

The new method involves incorporating a suitable density-gradient column in the centrifuge tube above the neck, to spread out the light minerals and prevent the formation of a compact mat of light mineral particles such as is obtained when using a heavy liquid of uniform density. The gradient is chosen to range in specific gravity from that at which the actual separation is required (*e.g.*, 3.3) to a value which is slightly less than the majority of the light minerals present (*e.g.*, 2.4, if quartz, feldspar, and biotite are the major minerals present). On centrifugation, each of the light minerals positions itself at a level corresponding to its density in the gradient, and the mass of light minerals is thus distributed through a significant proportion of the volume of the gradient (Fig. 2). Where a mineral of uniform density (*e.g.*, quartz) is present, it will form a discrete, compact layer which will tend to entrain heavy particles, but for many minerals slight variations in density will tend to spread out the layers vertically and allow the heavy mineral grains to pass through into the 'sinks.'

Incorporation of a density gradient column into the separation tube is a relatively simple matter. The tube is half-filled with dense liquid and the light liq-

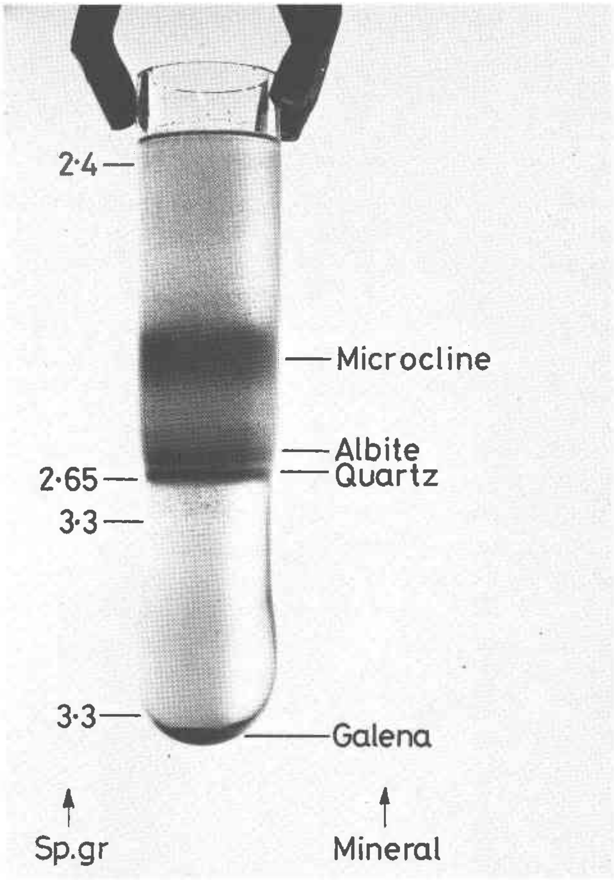

Fig. 2. Density gradient separation of C3 fraction of granite/galena mixture. Note the spreading of the light minerals through the gradient, thereby reducing entrainment of the dense galena.

uid is carefully poured in on top. Rotation of the tube with a swinging motion of the hand will cause swirling in the tube which will tend to homogenize the two liquids at their junction and thereby create a density gradient. This procedure is continued until the desired gradient is obtained, as indicated by the vertical separation in the tube of two glass marker beads of known intermediate densities. The precise profile of the gradient is generally not critical, the essential requirement being the spreading apart of the various light minerals to prevent entrainment of heavy minerals. The use of the gradient also facilitates insertion of the conical plunger to seal off the neck of the tube, as the mineral grains are loosely in suspension and not present as a compacted mat. The density gradient is remarkably stable, as once formed and left undisturbed, molecular diffusion is the only mechanism tending to cause homogenization. As pointed out by Muller and Burton (1965), density gradients may remain stable from several days to several weeks.

Comparison of methods

A comparative study was made of the two separation methods (*i.e.*, uniform-density liquid and density-gradient column), using a mixture of pulverized granite (composed of quartz, albite, microcline, and biotite and containing 20 ppm Pb and 5 ppm Cu) and pure galena; subsequently a briefer series of tests was carried out using a mixture of the pulverized granite and pure chalcopyrite. The heavy liquid used for the main tests was di-iodomethane (sp.gr. 3.3); dilution for the density gradients was with NN-dimethylformamide (Meyrowitz *et al.*, 1960). In addition, some tests were carried out using tetrabromoethane as the heavy liquid. Tests were also carried out on different amounts of sample to determine the effect of this variable on separation efficiency.

The galena and chalcopyrite were hand-picked from massive ore, crushed, separated at specific gravity 3.3 to remove any possible intergrown silicates, and then pulverized to -53 μm. The granite was separately pulverized to -53 μm. Separate mixtures of granite with about 2 percent galena and 2 percent chalcopyrite respectively were prepared and sized in a Warman Cyclosizer to give size fractions in the range 53 to 10 μm (equivalent quartz sphere), the -10 μm fraction not being retained. A number of tests were carried out using the combined $-53+10$ μm fractions and also individual size fractions within this range.

For the tests with uniform-density liquid, a known weight of sample was stirred into the liquid and any entrained air removed by vacuum treatment; the sample was then centrifuged, and the 'floats' removed, washed, dried, and weighed. One-eighth of the 'floats' was riffled out for analysis for Pb or Cu, the remainder was returned to a separate tube, and the whole procedure repeated. In this way representative 'floats' material at each stage was obtained for analysis, and by calculating the distribution of Pb or Cu between 'floats' and 'sinks' at each separation stage, it was possible to monitor the progressive change in separation efficiency.

A similar procedure was adopted for the density-gradient tests, except that a measured weight of material was stirred into a density gradient of known characteristics (determined using glass beads of known specific gravity) rather than a liquid of uniform density. The results of the initial comparison test using 10g of $-53+10$ μm sample showed that, whereas the uniform-density liquid gave 'floats' products containing 27.1 percent of the galena after one stage of separation and 6.8 percent after four stages, the corresponding 'floats' products from the density-gradient separation contained 7.7 and 0.6 percent of the galena respectively.

Table 1. Percentage of galena in total $-53+10$ μm size fraction of granite–galena mixture reporting into 'floats' for various initial sample weights (density-gradient separation method)

Sample weight (g)	Number of separation stages	
	1	2
5.0	8.0	3.7
2.5	4.9	2.1
1.25	2.8	1.2

Table 2. Percentage of galena in size fraction reporting into 'floats'*

(a) Separation in liquid of uniform density

Size fraction	Number of separation stages			
	1	2	3	4
C1	0.6	0.2	0.2	0.2
C2	5.0	1.3	1.0	0.6
C3	5.2	2.4	1.9	1.7
C4	42.6	10.1	3.5	2.3
C5	28.3	23.0	18.4	18.4

(b) Separation in density gradient column

Size fraction	Number of separation stages			
	1	2	3	4
C1	0.2	0.2	0.1	0.1
C2	1.3	1.0	0.7	0.3
C3	2.8	1.4	0.9	0.9
C4	30.2	4.7	1.6	0.8
C5	44.1	12.5	3.3	1.3

**3g initial weight of granite-galena mixture.*

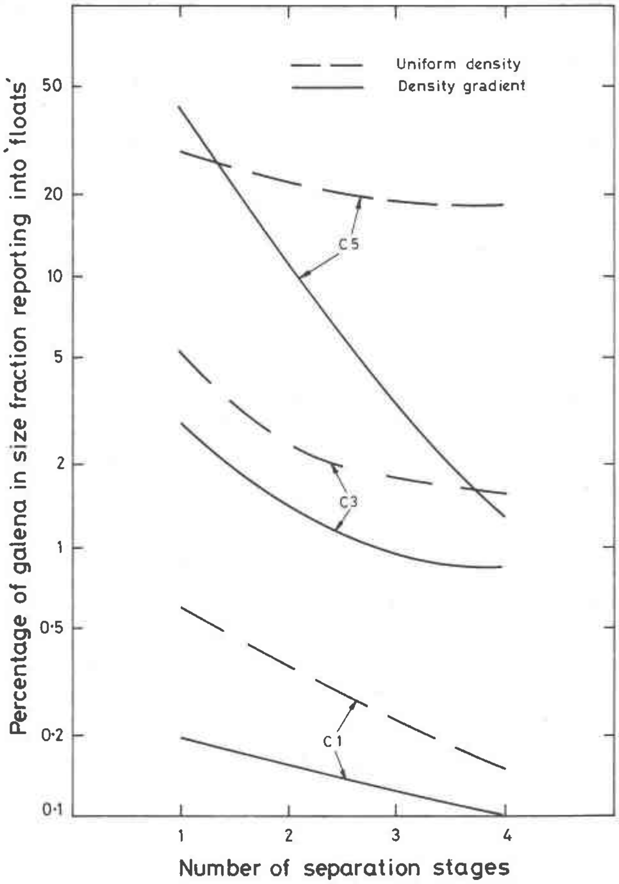

Fig. 3. Diagram illustrating the relationship between particle size, separation efficiency, and number of separation stages for the uniform-density and density-gradient centrifugal separation methods. Note the improved separation efficiency of the density-gradient method as compared to the uniform-density method, and the increasing efficiency of both methods with increasing particle size and number of stages of separation. [Increased efficiency is indicated by a decrease in the proportion of galena present in (*i.e.* entrained in) 'floats.']

Density-gradient separation tests on the −53+10 μm sample using progressively decreasing sample weight gave the results shown in Table 1, clearly indicating, as would be expected, that decreasing the sample weight increased separation efficiency.

Comparative separation tests on individual Cyclosizer size fractions (C1 to C5[1]) of the granite–galena mixture gave the results shown in Table 2, which indicate that for all size fractions the density-gradient separation method is superior to the uniform-density separation method and that there is a progressive decrease in efficiency with decreasing particle size for both separation methods. The results for the C5 fraction are particularly interesting; with the uniform-density separation method, 18.4 percent of the galena still remains in 'floats' after four stages of separation, whereas the corresponding figure for the density-gradient separation method is only 1.3 percent. The data for the C1, C3, and C5 size fractions are illustrated diagrammatically in Figure 3.

Table 3 summarizes the data for the tests on the granite–chalcopyrite mixture. It is interesting to note that there is generally less difference between the two separation methods and that at most particle sizes the separation has been more efficient than that for the corresponding granite–galena mixture. I suggest that these effects are due to the difference in average par-

Table 3. Percentage of chalcopyrite in size fraction reporting into 'floats'*

Size fraction	Uniform-density liquid Number of separation stages 1	 4	Density gradient column Number of separation stages 1	 4
C1	1.0	0.1	1.3	<0.1
C2	1.6	0.1	1.8	0.1
C3	2.3	0.1	1.3	0.1
C4	5.3	0.7	3.2	0.5
C5	20.9	1.6	5.7	0.9

**3g initial weight of granite-chalcopyrite mixture*

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[1] The particle sizes corresponding to these Cyclosizer fractions are approximately as follows:

Cyclosizer Fraction	Granite (μm)	Galena (μm)	Chalcopyrite (μm)
C1	−53+43	−53+22	−53+31
C2	−43+32	−22+16	−31+23
C3	−32+23	−16+12	−23+17
C4	−23+15	−12+8	−17+11
C5	−15+11	− 8+6	−11+8

ticle size of the heavy mineral in the two sets of experiments; in any Cyclosizer fraction the grains of galena are significantly smaller than chalcopyrite because of the difference in density between the two minerals, and the efficiency of separation is greater the less the difference in size between heavy and light minerals.

Tests using tetrabromoethane (sp.gr. 2.96) gave similar results to those obtained using di-iodomethane.

Tests on the $-10+5$ μm (equivalent quartz sphere) fraction of the granite–chalcopyrite mixture showed that both methods of separation were equally ineffective—even after five stages of separation the 'floats' still contained about 25 percent of the chalcopyrite in the size fraction.

Conclusions

Overall, the results of the tests indicate that: (a) the density-gradient separation method is more efficient than the uniform-density separation method at all particle sizes down to 10 μm; (b) the difference in efficiency between the two methods increases with increasing density of the heavy fraction; (c) the efficiency of both methods decreases with decreasing particle size and with increasing sample weight; and (d) both methods are relatively ineffective at particle sizes below 10 μm.

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