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A Laboratory Overflow-Centrifuge for Heavy Liquid Mineral Separation

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Abstract

A laboratory overflow-centrifuge (the "LOC separator") has been constructed for rapid and efficient heavy liquid mineral separation in sands and ground rocks sized between 500 and 16 μm. A maximum volume of sink fraction of about 35 cm^3 can be extracted in a single operation. Using a set of heavy liquids with density intervals of 0.01 (g/ml), a procedure is described for the speedy isolation out of a sand or ground rock of any desired fraction with a range in specific gravity of 0.01. A virtually complete separation is possible of mineral particles with differences in density as little as about 0.005 (g/ml).

Introduction

Heavy liquid mineral separation by means of conventional glass funnels (for example, Milner, Ward, and Higman, 1962, p. 101 *ff*) can only efficiently be applied to granular material (closely sized ground rocks or free running sands) coarser than about 70 to 150 μm (200 to 100 mesh ASTM), the size limit depending upon the differences in density between the minerals to be separated. This limitation also holds for special devices such as the stopcock separator or the heavy liquid density gradient column (Muller and Burton, 1965), in which only small quantities can be handled in a single operation, and for various types of continuous density separators in which the heavy liquid is recirculated, allowing the handling of larger samples (for example, Fairbairn, 1955; Østergaard, 1968).

For the processing of material finer than about 70 μm, use of a centrifuge is necessary in order to increase the settling velocities and to prevent entrainment and/or agglomeration of the mineral particles. A difficulty when utilizing a centrifuge equipped with standard tubes is the separation of the light and heavy fractions without contaminating one with the other. In order to overcome this difficulty, several designs for special centrifuge tubes have been published, allowing a rapid and efficient separation of sink and float products. The most simple is the polythene sleeve technique described by Muller and Burton (1965), employing thin polythene sleeves inserted into a standard glass centrifuge tube. The same authors also developed an ingenious co-axial separator, again based upon the density gradient method. Other types of specially designed centrifuge tubes have been described, for example, by Nickel (1955), Cheeseman (1957) and Ludwig (1966). However, a drawback in all these techniques is that they allow only small samples to be handled in a single operation, making processing of larger samples very time-consuming. A centrifugal technique enabling the rapid processing of large samples (up to about 10 kg in a single operation) was developed by Verschure and IJlst (1966), who constructed an all-glass overflow-centrifuge for the continuous heavy liquid separation of heavy minerals out of sand or ground rock. This device was specially designed to extract heavy accessories with specific gravities differing substantially from those of the main constituents (for example, ore minerals, zircon, titanite, *etc* out of a ground granite).

The "LOC separator" described in this paper, on the other hand, is a high-speed (3000 rpm) all-metal laboratory overflow-centrifuge applicable as a sensitive tool for the speedy, easy, and efficient segregation of mineral particles differing only slightly (as little as about 0.005) in specific gravity. The amount of sample that can be processed in a single operation depends upon the proportion of the sink fraction; the maximum volume of heavy particles that can be collected in a single operation amounts to about 35 cm^3.

The LOC separator

This separator is shown in Figure 2 while its construction is diagrammatically explained in Fig-

ure 1. The apparatus is based upon a simple and cheap laboratory centrifuge (3000 rpm) of which the original rotating barrel is retained in order to maintain stability. The rotating holder of the tubes in the original centrifuge is replaced by the holder of the centrifuge vessel (e in Fig. 1); this holder and the inserted centrifuge vessel (c) are the rotating parts of the device. The normal diameter of the feeding funnel (a, Fig. 1) is 6 mm, but it is possible to insert accessory funnels with smaller diameters (4 or 2 mm, Fig. 3). With the exception of the polythene tube (f, Fig. 1) and the original components of the laboratory centrifuge, all parts of the overflow device are made of brass.

It is important to operate the LOC separator under conditions of constant temperature. Especially for the separation of fractions with small differences in specific gravity (in the order of 0.01 or less) the apparatus should be kept in a thermostatically controlled environment. In order to prevent the heat produced by the centrifuge motor from affecting the heavy liquid in the centrifuge vessel, slits are made in the upper side of the outer casing, allowing the circulation of air along the vessel.

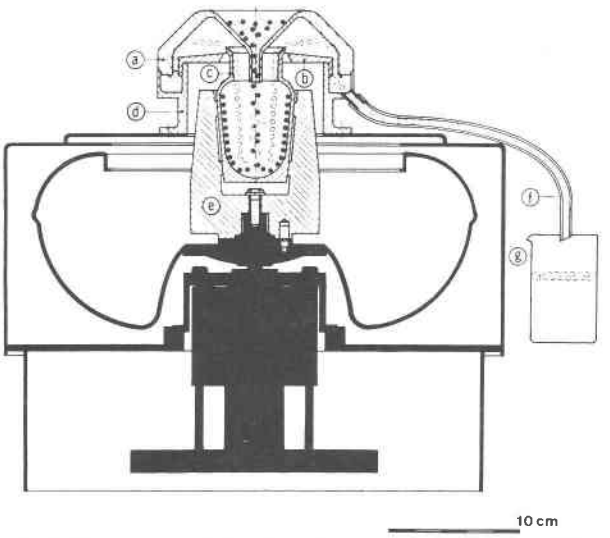

FIG. 1. Diagram of the LOC separator. (a) cover of the collecting vessel, also serving as feeding funnel with inside diameter of 6 mm; (b) protective ring, to prevent spilling into the centrifuge body; (c) centrifuge vessel; (d) bottom of the collecting vessel with overflow; (e) holder for the centrifuge vessel; (f) polythene tube; (g) overflow receiver (glass beaker). Parts belonging to the original commercial laboratory centrifuge (HOMEF LC-30) are shown in black.

Operation

Separation of float and sink fractions by means of the LOC separator can be achieved either continuously or discontinuously. The continuous procedure is applied to larger samples containing a high float/sink ratio. Moreover, the sink fraction should differ by more than 0.1 in specific gravity from the utilized heavy liquid; otherwise the settling time of the particles is too long to ensure efficient separation. On the other hand, the discontinuous procedure is used when the sample is small and/or when the difference in specific gravity between the sink fraction and the utilized heavy liquid is less than 0.1.

When applying the continuous procedure, the sample is mixed and thoroughly stirred with approximately double the volume of the heavy liquid in a glass beaker. The slurried sample is poured into the rotating centrifuge vessel (c, Fig. 1) via the feeding funnel (a). The sink fraction is collected in the centrifuge vessel, while the spilled heavy liquid containing the float fraction is received via the collecting vessel with overflow (a, b, d) and the tube (f) in a glass beaker (g). An extra amount of heavy liquid is then poured into the rotating centrifuge vessel in order to remove all remaining float particles. The maximum volume of heavy liquid in the full-speed rotating centrifuge vessel (shown as c in Fig. 1) amounts to about 48 cm^3, but in practice no more than approximately 35 cm^3 of sink fraction

FIG. 2. The LOC separator. The letters of the components correspond to those in the diagram of Figure 1. Two accessory funnels (Fig. 3) are also shown.

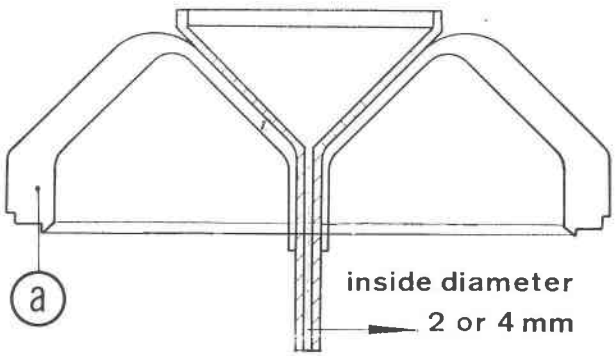

FIG. 3. Accessory funnels with different diameters to be inserted in the cover of the collecting vessel (Fig. 1, a), for the purpose of adjusting the feed rate of the slurried sample.

can be collected in a single operation. The collecting vessel (a, b, d) can be lifted together with the centrifuge lid to which it is fixed (Fig. 2), so that the centrifuge vessel (c) can easily be removed from the holder (e) and the sink poured out. The feeding funnel/cover assemblage (a) and the protective ring (b) can be taken from the bottom part (d); this facilitates cleaning of the collecting vessel after having completed the separation.

It was found that the feed rate of the slurried sample has to be adjusted to the particle size in order to obtain the optimum conditions for separation. The adjustment can be accomplished by inserting accessory funnels with different diameters (Fig. 3). An inside diameter of 6 mm (Fig. 1, a) gives the best results for size fractions between 500 and 250 μm (35 and 60 mesh ASTM). For size fractions between 250 and 63 μm (60 and 230 mesh ASTM) and for samples with particle size below 63 μm (230 mesh ASTM) accessory funnels have to be inserted with inside diameters of 4 mm and 2 mm, respectively.

When employing the discontinuous procedure, it has been found that for efficient separation in general not more than approximately 20 cm^3 of sample can be handled in a single operation. The sample is transferred into the centrifuge vessel (Fig. 1, c) together with the heavy liquid, with which it is thoroughly mixed into a slurry. Enough heavy liquid is added until the total volume of slurry amounts to about 48 cm^3. The centrifuge is then rotated for two to five minutes, depending upon the particle size of the sample as well as the difference in specific gravity between the sink fraction and the heavy liquid. Finally, about 100 ml of the utilized heavy liquid is poured into the rotating centrifuge vessel, allowing the spilled liquid to carry the float into the glass beaker. The sink fraction can then be easily collected from the centrifuge vessel.

Cleaning Assemblage

Cleaning of the sink and float fractions obtained from the heavy liquids is achieved under suction by means of a glass Witt's filter apparatus in which the normal funnel is replaced by a polythene Buchner funnel. A second assemblage of identical design is used for washing the drained fractions with 1.1.1.trichloroethane (IJlst, 1973). The liquid is collected in an Erlemeyer flask placed within the Witt's apparatus. A loose glass cylinder is inserted into the Buchner funnel to hold the filter paper tightly against the perforated polythene bottom. The lower side of this cylinder is polished in order to prevent mineral particles from bypassing the filter. It was found that Schleicher and Schüll 589 Black Ribbon filter paper (equivalent to Whatman No. 41) is most satisfactory in practice. The filtration assemblage is shown in Figure 4.

Procedure

A systematic and efficient plan of procedure for heavy liquid mineral separation by means of the LOC separator is shown in a "flow-sheet" (Fig. 5). Using a set of heavy liquids with density intervals of 0.01 (g/ml), any desired density fraction with a range in specific gravity of 0.01 can be isolated in at most seven operations. The first step consists in centrifuging the material with the 2.82 liquid. Depending upon the desired direction in the "flow-sheet," either the sink or the float fraction is selected for further treatment after cleaning and drying (s or f in the "flow-sheet," respectively). This fraction is then processed with a liquid differing by 0.32 (g/ml) in density from the 2.82 liquid (d = 3.14 or d = 2.50), and so on. As indicated in the "flow-sheet," the order of liquids employed in the succesive operations is such that the density interval between every liquid and the next is half of the interval between this liquid and the preceding one (0.32, 0.16, 0.08, 0.04, 0.02 and 0.01). After each separation either the sink or the float product is selected for further processing. In this way, any desired density fraction containing mineral particles with a range in specific gravity of 0.01 can be separated in at most seven operations out of ground rock or sand.

The whole procedure is monitored by microscopic examination of the float and sink fractions produced in each operation so as to determine the direction in the "flow-sheet" for the next separation. If a mineral is sought of which the specific gravity is approximately known, then, of course, not all seven steps are required, but the procedure need only be followed from a liquid with a density just above or below that of the desired mineral.

The set of heavy liquids used by the author is based upon bromoform-isoamylacetate solutions for the range 2.19 to 2.82, diiodomethane-orthodichlorobenzene solutions for the range 2.83 to 3.29, pure diiodomethane 3.30, and Clerici solutions for the range 3.31 to 3.45. If necessary this set can easily be extended above or below this range by means of Clerici solutions or bromoform-isoamylacetate solutions, respectively. None of these liquids is corrosive towards brass, which is an important property when working with the LOC separator. The advantages of isoamylacetate and orthodichlorobenzene as diluents for bromoform and diiodomethane, respectively, are discussed by IJlst (1973).

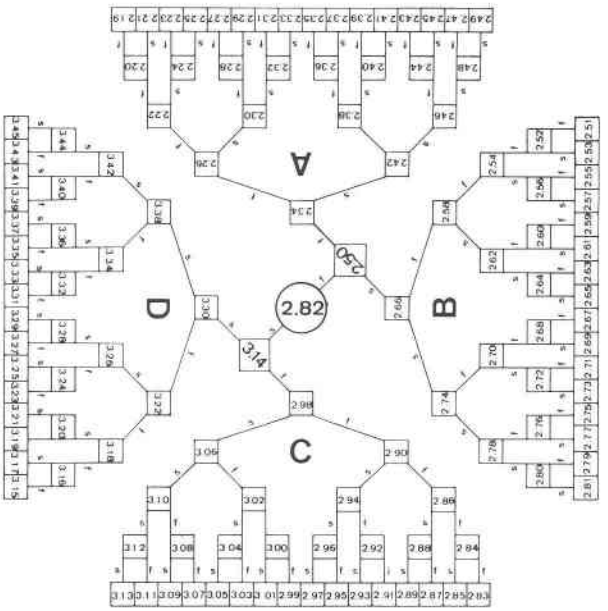

FIG. 5. Plan of procedure for heavy liquid mineral separation by means of the LOC separator, using a set of heavy liquids with density intervals of 0.01 g/ml. Starting with the bromoform-isoamylacetate solution $d = 2.82$, any desired density fraction with a range in specific gravity of 0.01 can be separated in at most seven operations. For further explanation see text.

Performance

Many tests with the LOC separator on comminuted rocks in the size range +16–500 μm have demonstrated that float and sink products can be obtained with an efficiency of over 99 percent in a single operation. The procedures and the sets of heavy liquids described in the foregoing paragraphs provide a rapid means of separating fractions with density differences as low as about 0.005 (g/ml). However, the recovery of a specific mineral from a heterogeneous mineral assemblage such as a rock powder depends strongly upon the degree of liberation of the mineral particles; in general the proportion of liberated constituents increases with reduction of the grain size. The LOC separator enables the processing of fine powders, so it is possible to achieve with this apparatus a high recovery of a specific mineral even when it has to be separated from a rock or sand in which it occurs intimately intergrown with other mineral phases. This may be illustrated by dressing experiments with commercial "pure quartz sand" (Merck), which were made for the purpose of obtaining quartz powder of extreme purity to be used as diluent in the preparation of powder pellets for X-ray spectrometry.

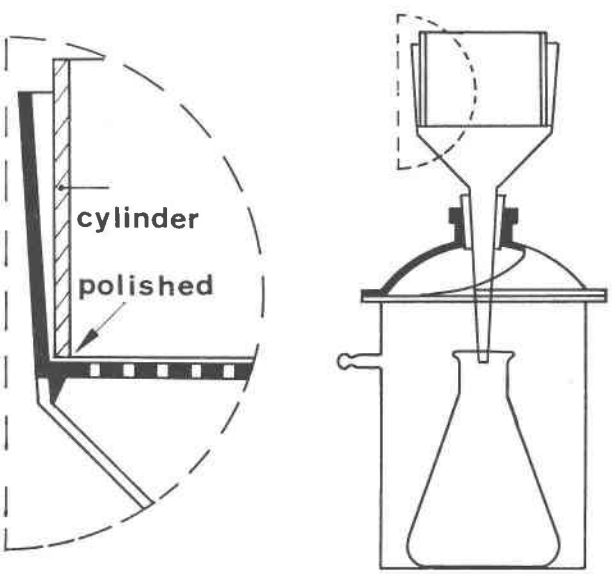

FIG. 4. Filtration assemblage for the cleaning of the sink and float fractions from the heavy liquid under suction.

The purchased quartz sand had a size between 500 and 1000 μm. Microscopic investigation revealed trace amounts of muscovite, while a minute proportion of potassium feldspar is intimately intergrown with quartz. The sand was ground and classified

into seven closely sized fractions, five above 36 μm (400 mesh ASTM) by means of sieving and two below 36 μm by settling in an Atterberg cylinder, applying the settling times for spherical quartz particles according to Stoke's law. Samples of 20 g were taken from all size fractions except the fine dust product (below 16 μm) which was discarded. Each of these six samples was processed individually in the LOC separator, using the discontinuous procedure. After having removed the traces of muscovite by processing the material in a liquid $d = 2.650$, the remaining float product was further separated into two fractions using a liquid $d = 2.640$. All fractions were weighed. The fraction lighter than 2.640 and that between $2.640 < d < 2.650$ were also analyzed for their potassium contents by flame photometry. Moreover, the specific gravities of the fraction $2.640 < d < 2.650$ were determined by means of a pycnometer for the four size fractions above 63 μm. The results (Table 1) are also shown graphically (Fig. 6). As is evidenced by the specific gravity and the very low potassium content (around the detection limit), the fractions $2.640 < d < 2.650$ are composed of pure quartz, while those lighter than 2.640 represent quartz intergrown with some potassium feldspar. From the data (Table 1) and graphs (Fig. 6), it is evident that the recovery of pure quartz increases rapidly with reduction in grain size, while even for the fine fractions there appears to be no contamination with lighter particles when processing with the 2.640 liquid. Concurrently with the increasing recovery of pure quartz, the proportion of potassium feldspar in the float fraction of the 2.640 liquid increases rapidly with reduction in grain size. From check examinations it was proven that virtually no particles of the sink product were retained in the float fractions. These dressing experiments demonstrate the efficiency of the LOC separator even for finely comminuted mixtures of minerals differing very little in specific gravity.

TABLE 1. Dressing Experiments with Commercial "Pure Quartz Sand"*

Size (μm)	Wt % Total Quantity	Sink 2.650 Wt %	2.640 < d < 2.650 Wt %	ppm K	Density	Float 2.640 Wt %	ppm K
<500>250	6.4	1.0	62.2	10	2.646	36.8	40
<250>125	33.8	1.0	72.1	10	2.646	26.9	60
<125 >88	11.1	1.4	76.5	11	2.646	22.1	90
<88 >63	13.1	1.1	83.7	11	2.647	15.2	140
<63 >36	10.2	1.1	86.6	13	--	12.3	212
<36 >16	10.8	1.1	90.8	10	--	8.1	260

* *Sink 2.650: muscovite; 2.640 < d < 2.650: pure quartz; float 2.640: quartz intergrown with some potassium feldspar.*

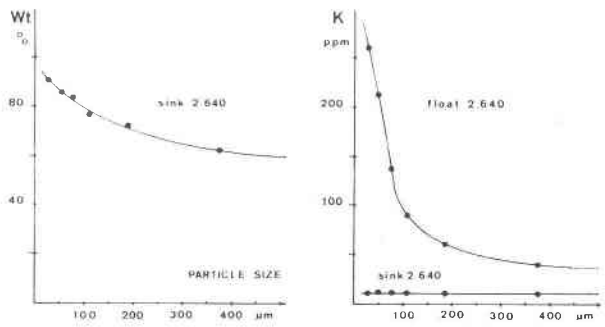

FIG. 6. Results of dressing experiments with the LOC separator on commercial "pure quartz sand." The left graph shows the increasing recovery of pure quartz (sink 2.640) with reduction of the particle size. The right graph demonstrates the increasing potassium content (proportion of potassium feldspar) in the remaining float fraction (float 2.640) with reduction of the particle size, while the potassium content of the quartz fraction (sink 2.640) remains constant and very low (around the detection limit).

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