CONCENTRATION OF HEAVY ACCESSORIES FROM LARGE ROCK SAMPLES

H. W. FAIRBAIRN, *Department of Geology and Geophysics, Massachusetts Institute of Technology, Cambridge, Massachusetts.*

ABSTRACT

Concentration of heavy accessories from large rock samples (circa 50 lb.) requires, in the interest of efficiency, some modification of standard techniques currently used for small samples. To this end a continuous flow heavy-liquid separator has been designed which reduces handling and economizes on time. An improved method is described for recovery of heavy liquids by adding water directly to the filtered, but unwashed, sand. A number of additional points in technique are also included as well as an appendix listing the major items of equipment needed for work with large samples.

INTRODUCTION

Recent developments in geochronology, using zircon concentrates from igneous rocks (Larsen et al., 1952; Hurley and Fairbairn, 1953) require in many cases the processing of samples of fifty pounds or more. The concentration of these heavy accessories by crushing, grinding, sizing, heavy-liquid separation, and magnetic separation is a well known technique, but many details of the operation, as currently described (see texts listed), are suitable for small samples only. The present paper reconsiders the whole matter and reports on certain labor-saving modifications which, by laboratory standards, make processing of large samples reasonably efficient. Although developed for a special purpose, they have general application and will be useful for concentrating accessories from a variety of rocks.

PREPARATION OF THE SAND

Quantitative separation of the components of a rock, either by heavy-liquid or magnetic means, depends to a large degree on close sizing of the rock particles, or sand. In a poorly-sized sand smaller, heavy grains tend to be rafted up by larger, lighter, grains and conversely, lighter grains may be dragged down to contaminate the sink fraction.* Preparation of an adequately sized sand is therefore essential and, for large samples, consists of the steps outlined below.

The field sample must first be reduced to fragments small enough to be fed into a laboratory jaw breaker. This can be done by hand or, more rapidly, with a hydraulic-type chisel. The material is then passed through the breaker twice, setting the jaws to a smaller opening for the second pass. The product from the breaker, now a coarse sand, goes through a

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* See p. 466 for details of an experiment bearing on this.

disc grinder, two or three times depending on the rock, with closer settings of the discs between each run. To avoid an undue proportion of very fine dust (as well as a lowering of grinding efficiency) the sand is screened between each grinding operation. A continuous-discharge vibrating screen with two $12'' \times 30''$ screen beds gives (1) a $+40$ oversize product which is returned to the disc grinder, (2) a $+200$ product which is set aside, (3) a -200 product which is usually discarded. When grinding is completed, the stock of $+200$ sand is passed through the screen at least twice to remove remaining fines. From a 50-lb. sample, if all the oversize is reduced, up to 30 lbs. of $-40 +200$ sand may thus be obtained.

Magnetic Separation

To reduce as far as possible the volume of sink resulting from heavy-liquid separation, preliminary magnetic separation of major constituents such as biotite and hornblende is always advisable. A continuous-flow induced-roll separator, fitted with an auxiliary device for removing magnetite, accomplishes this most efficiently (Fig. 1). On a platform just above the roll separator a vibrating feed, equipped with a large hopper, passes a thin layer of the $+200$ sand forward to the triangular receptacle on the front of the roll separator. Before leaving the conveyor, however, this thin layer of grains passes under a slowly moving transverse belt, above which is suspended a row of Alnico horseshoe magnets. High-magnetic material (magnetite, iron filings, etc.) is lifted to the belt, passes over an aluminum pulley and is dropped into a receptacle beneath. Low-magnetic minerals are next separated from non-magnetic by the strong field of the roll separator, and the products are collected in two pails attached to the frame. An air cleaner (not shown) removes the inevitable dust.

Lacking this equipment, a Frantz separator, set up for vertical feed, will remove biotite, but not ordinary hornblende or pyroxene. The removal of these latter two minerals with the Frantz inclined feed is possible, but hardly practicable for a large sample of mafic rock. The equipment described above provides an adequate solution.

Heavy-Liquid Separation

Since the major part of most common rocks is non-magnetic quartz and feldspar, removal of this fraction from its associated heavy accessories is at best a time-consuming process. If a sand much finer than -200 must be processed, a large centrifuge will be necessary equipment. For $+200$ sand, with which this paper is concerned, gravity settling is adequate. To eliminate some of the handling, however, as well as to
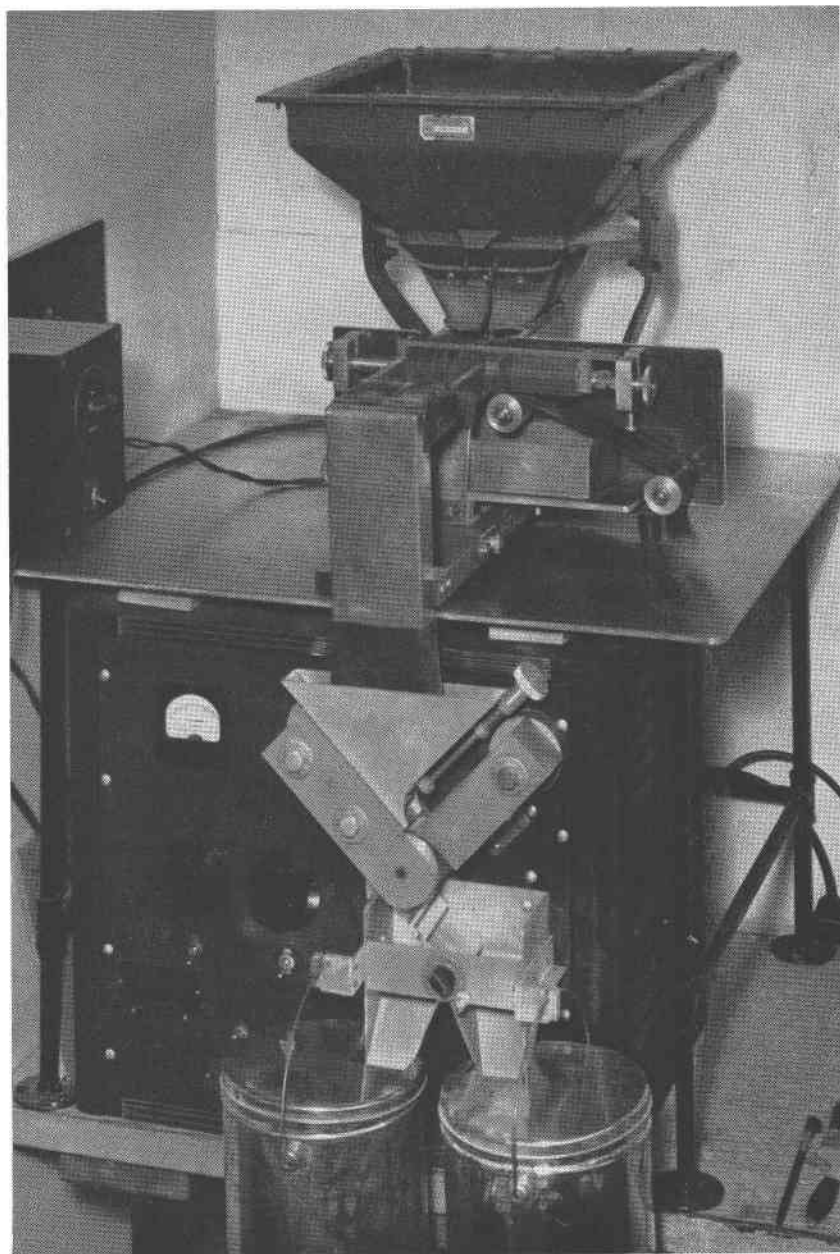
FIG. 1. Induced-roll magnetic separator (Carpco) with auxiliary vibrating feed
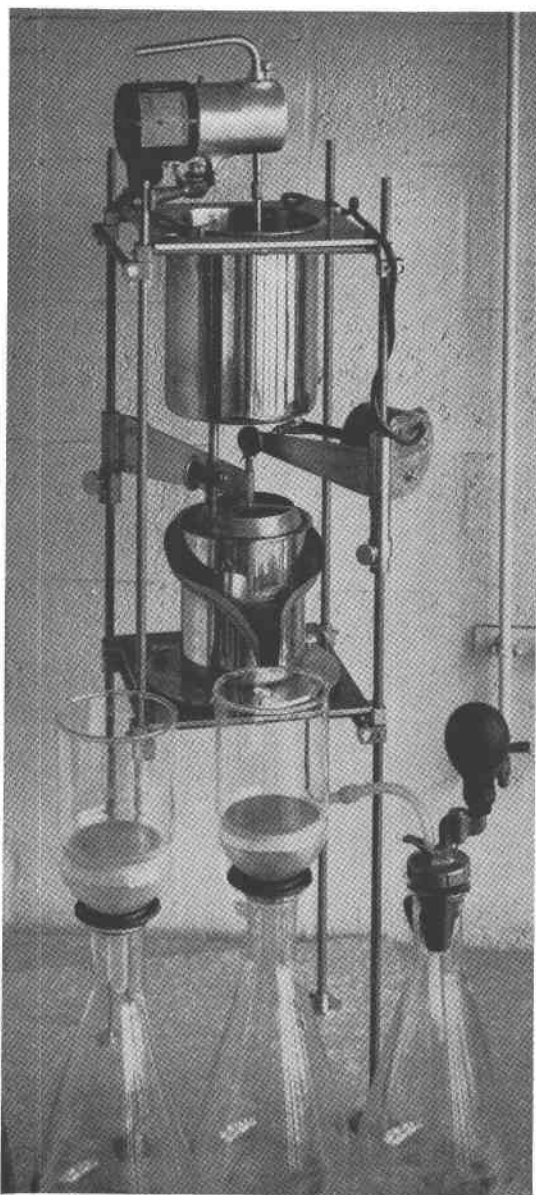and device for removing magnetite.

Fig. 2. Heavy-liquid separator for large samples. See text for description.

economize somewhat on time, a heavy-liquid separator, designed for use with large samples, has proved its worth in recent tests.

A 9-quart stainless steel beaker at the top of the assembly shown in Fig. 2 is filled with a 2:1 mixture of tetrabromoethane (acetylene tetrabromide) and sand. The mixture is kept in constant motion by a 4-propeller stirrer (with a lid on the beaker to prevent splashing), and leaks slowly through a conical valve in the bottom of the beaker. This valve has a screw adjustment outside the beaker so that, as the head of fluid falls, the opening may be increased to maintain uniform flow. The liquid-sand mixture leaves the beaker through a wide-diameter brass* tube (to
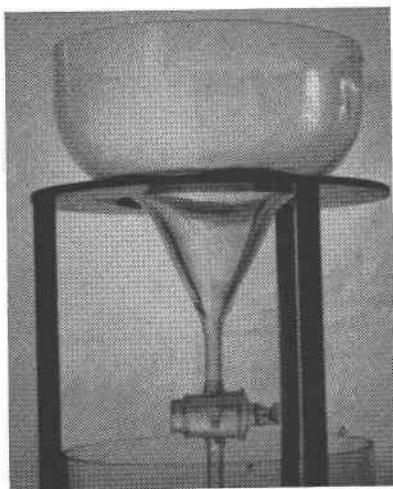

Fig. 3. Separatory funnel used either (1) for concentration of heavy accessories from small samples, or (2) for recovery of tetrabromoethane from acetone washings. Constructed from a 5-gal. Pyrex carboy, as described in text.

prevent clogging) and is fed into a 4-quart stainless steel beaker below, near the wall and at a point about one inch from the bottom. This beaker is previously filled with tetrabromoethane. The rising quartz-feldspar-muscovite fraction is removed from the beaker by a jig. This consists of a horizontal collar with inclined sides which is given an up-and-down motion into the liquid by a cam directly above. This action moves the floating sand over the edge into an inclined drain which encircles the beaker. Vacuum filtering is carried out in 2-liter fritted-disc Büchner funnels. When the run is completed, the heavy accessories zircon, mona-

* Aluminum, iron, and rubber are unsatisfactory materials for use with tetrabromoethane.

zite, apatite, sphene, etc. are collected from the bottom of the separation beaker and fractionated further by magnetic and heavy-liquid treatment. The filter flasks are emptied by means of a rubber bulb pump (Fig. 2, lower right).

For methylene iodide separations, or for tetrabromoethane separations with only a few pounds of sand, separatory funnels of the design shown in Fig. 3 have been found very satisfactory. The one illustrated is made by cutting off the bottom of a 5-gal. Pyrex carboy, drawing out the neck, and fitting to this a stopcock. This funnel design has the advantage over conventional funnels that, for equivalent volumes of sand, the thickness of the layer is much reduced and rapid collection of the heavy accessories is thus facilitated. The disadvantage of a fairly flat shoulder, which catches much of the sink, is not a serious handicap as rotary stirring will displace the sink towards the center.

Smaller models of this funnel have been found to be exceedingly useful.

RECOVERY OF LIQUIDS

With large samples a considerable volume (about 3 gallons) of heavy liquid is required for the initial separation. Although the cost of the tetrabromoethane used by the writer is only a small fraction of the cost of bromoform used in many laboratories,* nevertheless it is uneconomical not to recover most of it. Furthermore, if unwashed sand is discarded, disposal becomes a problem as the liquid is toxic.†

As a result of some experimentation, the following procedure for recovery of tetrabromoethane has been found incomparably more efficient than any other known to the writer. The method applies equally well to bromoform.

The funnel which receives float from the separation beaker (Fig. 2) should be filled with sand to not more than half capacity. After the liquid has been pumped through to the flask the vacuum is disconnected and about one-half liter water is added to the sand.‡ This is stirred in thoroughly and thus displaces an additional amount of heavy liquid to the bottom which had not been removed by the initial pumping. This liquid and the overlying water are now pumped through to the flask.

* $0.55 per pound as against $2.00 a pound or, by volume $3.50 per liter and $14.00 per liter (approx.). These are commercial grades, obtainable from Dow Chemical Co., Midland, Mich.

† Although the catalog of harmful effects attributed to the liquid, as compiled by Sax in "Handbook of Dangerous Materials," makes alarming reading, in an adequately ventilated laboratory the amount of the vapor in the air can easily be kept below the critical concentration of 5 parts per million.

‡ This seems to have been first suggested by Smithson (1934).

The flask can later be emptied with a convenient hand pump (Fig. 2) and the undiluted heavy liquid then separated from the water.

The funnel is then transferred to another filter flask and, without vacuum, about one-half liter acetone is stirred into the sand. This is now pumped out and the sand is then discarded. Although additional washing of this type with acetone would be necessary to remove the heavy liquid quantitatively, this should not be done unless the quartz-feldspar sand is required for some other investigation.*

Since the sand before washing with acetone contains water as well as tetrabromoethane, the acetone will dissolve preferentially in the water and excess will go with the heavy liquid. The filter flask will thus have a layer of partly concentrated tetrabromoethane overlain by an acetone-water solution. This lighter fraction may be discarded. The heavy layer is transferred to a large beaker and a stream of water passed through it. A mineral fragment placed in the liquid will serve as density control and will float after a few minutes' washing.†

Following are some figures relevant to the procedure just outlined.

	Tetrabromoethane	Bromoform
Vol. of liquid required to saturate given vol. of sand	100 cc.	100 cc.
Vol. of liquid recovered in first filtration	48 cc.	79 cc.
Vol. of liquid recovered after addition of water.	33 cc.	8
Vol. of liquid recovered from heavy layer in filtrate after one acetone wash	11 cc.	n.d.
Vol. liquid recovered from upper layer in filtrate after one acetone wash	1 cc.	n.d.
Total recovery (disregarding final 1 cc. from upper layer)	92 cc.	n.d.

The greater viscosity of tetrabromoethane over bromoform shows clearly in the above tests and, if the water stage of Smithson were omitted, use of tetrabromoethane would involve a bigger recovery problem than would bromoform (only 48 cc. recovered directly compared with 79 cc.). However, use of water as described above neutralizes this advantage and, as tetrabromoethane is much the cheaper liquid, a loss of 8% can be tolerated.‡

* A test made on a *second* wash showed that, disregarding the time required, the cost of the acetone used was about three times the cost of the tetrabromoethane which could be recovered.

† The writer uses lepidolite (2.80) in order to hasten fractionation of heavy accessories. If the liquid is required to be at maximum density a piece of aragonite (2.94) should be used.

‡ This is not of course 8% of the liquid used for the separation, but only 8% of the liquid needed to saturate the sand.

For general acetone washings, where Smithson's method is not used, the usual laborious flask-shaking method of recovery may be avoided if 400 cc. aliquots of the wash material are fed slowly with gentle stirring into a large separatory funnel (Fig. 3) containing at least 6 liters of water. The recovered heavy liquid is withdrawn through the bottom, the funnel is drained, re-filled with fresh water, and another 400 cc. of acetone wash is added. The recovered heavy liquid can then be brought to full strength by passing water through it, as already noted.

Bromoform is soluble in water at 30° to the extent of 1 cc./liter H_2O. The writer has not found any similar data for tetrabromoethane, but from tests made the solubility would appear to be much less than for bromoform. Losses from this source using the recovery method above are therefore small. On the other hand, methylene iodide is moderately soluble (4cc./liter H_2O) and, in view of the cost of the liquid,* recovery of its acetone washings with water is not recommended. Evaporation at room temperature, using an electric fan and with a fragment of sillimanite ($D = 3.20$) for density control, is preferable. Losses can be kept under 5%.

There are no shortcuts for recovery of diluted Clerici solution. Since it decomposes all too readily and is an expensive mixture,* evaporation at room temperature with the aid of an electric fan is the safest procedure. Fortunately it is needed in small quantities only. Willemite ($D = 4.2$) may be used as density indicator.

DECOLORIZATION

Heavy liquids (except Clerici solution) tend to discolor with use and in contact with iron-rich minerals, particularly magnetite, will decompose as well. The latter can be prevented by immediate washing of such mixtures. Ordinary discoloration can be removed in a number of ways, one method being as follows. The liquid, diluted well with acetone (at least 5:1), is gently heated and a small amount of finely ground bone charcoal is stirred into it. When most of the acetone is gone, the liquid is filtered with a double thickness of paper and the decolorized liquid collected for final recovery with water. Decoloration need seldom be carried out for tetrabromoethane; for methylene iodide on the other hand deep color handicaps observation of the sink and the above decolorization method provides a remedy.

EFFICIENCY OF SEPARATIONS

No comprehensive experiments have been carried out to test efficiency of separation, as the goal of the writer's investigation did not re-

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* About $0.20 per cc. for methylene iodide; $0.50 per cc. for Clerici solution.

quire strictly quantitative separation of heavy accessories. However, a few tests have been made as recorded below.

The need for closely sized sand in heavy liquid separations has already been pointed out. As an example of this, 100 mg. zircon was added to a prepared mixture of about 3 lbs. of quartz, feldspar, biotite, the entire sample having been sized to −40 +200. The mixture was placed in tetrabromoethane in a large separatory funnel of the kind shown in Fig. 2. After an initial stirring of a few seconds the sink contained 53 mg. zircon; after a second, 15 mg.; after a third, 8 mg.; and after a fourth, 5 mg., totalling 80 mg. recovery. The later sinks contained an increasing proportion of smaller grains. For quantitative separation, therefore, it is apparent that much closer sizing than −40 +200 would be necessary. For ordinary work, however, semi-quantitative fractionation is usually adequate.

The efficiency of fractionation of the new heavy-liquid separator (Fig. 2) is unknown but is believed to be very high if the flow rate is kept low. A sample of float, concentrated at a rate of flow of about 5–6 liters per hour, was transferred to a large Pyrex separator (Fig. 3). After repeated stirrings, and finally standing overnight, no trace of a sink fraction could be found.

No tests have been made with methylene iodide, but since it has lower viscosity than other heavy liquids in common use, separations are believed to be fairly complete. Clerici solution on the other hand is relatively viscous and small-diameter heavy accessories which should normally sink are commonly rafted up by large-diameter fragments belonging to the float. Close sizing is therefore highly important here, as well as patience while particles settle slowly through the column, if losses of hardly won heavy accessories are to be avoided.

From a few tests made with the Frantz magnetic separator it would appear that recovery of non-magnetics is about 100%. Sizing is important here also and the magnetic and non-magnetic products obtained at given settings should be rerun at least once for quantitative recovery.

Concluding Remarks

The foregoing account of concentration procedure for large samples is intended to amplify the established procedures used for small samples. Since the general method is used in a great many laboratories it may well be that apparatus and procedures for processing of large samples have been developed but not reported in the literature. If such is the case, and also if any pertinent published information has been missed, the writer would like to be informed.

APPENDIX

As an aid to readers who may wish to work with large samples, the following details concerning equipment are appended. Many of the units may be purchased through local supply houses.

1. Hydraulic Rock Chisel, Ward's Nat. Science Establishment, Rochester, N. Y.
2. Laboratory Jaw Breaker, Denver Fire Clay Co., Denver, Colo.
3. Laboratory Disc Grinder, Denver Fire Clay Co., Denver, Colo.
4. Laboratory model Low-Head Vibrating Screen, Allis-Chalmers Co., Milwaukee, Wis. A second screen can be fitted on the apparatus so as to increase its utility. This equipment, together with the breaker and grinder, should be in a separate room with adequate dust removal facilities.
5. Induced Roll Magnetic Separator (Carpco), Separations Engineering Corp., 110 E. 42nd St., New York 17, N. Y.
6. Isodynamic Magnetic Separator. S. G. Frantz Co., N. J. If a choice must be made between this separator and the one above, the Frantz equipment should be selected because of its greater versatility.
7. Heavy-Liquid Separator (described in text)
   (a) 4 qt. and 9 qt. Stainless Steel Beakers, The Vollrath Co., Sheboygan, Wis. It is recommended that beakers of this type be used instead of glass beakers for the entire laboratory operation.
   (b) "Lightnin'" Portable Mixer, Model F, with Ring Stand Clamp, and 3 extra Propellers, Mixing Equipment Co. Inc., 135 Mt. Read Boulevard, Rochester 11, N. Y.
   (c) "Slo-Speed" Motor (to operate cam) Serial E-6, Cat. 75765, 85 RPM at full load, Precision Scientific Co., Chicago, Ill.
   (d) Pneumatic Acid Pump, with plastic tubing, Thompson Mfg. Co., Erie, Penn.
   (e) 2-liter Funnels, Büchner type, with fritted glass disc (coarse), Corning Glass works, Corning, N. Y.
   (f) 4-liter Pyrex Filter Flasks, Corning Glass Works, Corning, N. Y.
   (g) Flexaframe Rod, Connectors, and Feet, Fisher Scientific Co., Pittsburgh 19, Penn.
8. Vibrating Feed, Model 1-B, Jeffrey Mfg. Co., Columbus 16, Ohio

The services of a competent shop man for about 10 days will be needed

for the various units described in this paper. There are no very critical dimensions, but the writer will be glad to furnish further details on request.

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