

HEAVY MEDIA COLUMN SEPARATION: A NEW TECHNIQUE FOR PETROGRAPHIC ANALYSIS

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ABSTRACT

A heavy media column is used for the separation of the mineral fractions of a sample. Thus the directly weighed per cent of the mineral composition of the rock or ore sample is accomplished. The technique may be applied to the concentration of a particular species, even in trace amounts, for further mineralogical studies or for practical commercial metallurgical mineral beneficiation purposes.

INTRODUCTION

In 1954, the writer separated a hundred per cent extraction of pure mineral fractions from a heavy mineral suite. The weight per cents of the mineral fractions obtained were used as a standard to evaluate the accuracy of the point-count petrographic method, and the efficiency of the laboratory metallurgical heavy mineral separation results.

The heavy mineral sample, which was either a Wilfley table concentrate or heavy mineral fraction of a tetrabromoethane heavy media separation, was immersed in a thallos formate or Clerici solution and water mixture in a small 50-ml beaker. The beaker was placed under a heat lamp for slow evaporation of the water content of the mixture. As the water content diminished, the liquid media became heavier and the heavy mineral particles floated in order of increasing specific gravity. The mineral particles which floated were removed by a small silk screen net and an ordinary water color paint brush into a series of beakers containing warm distilled water. When no more solids were floated, the beaker was removed from the heat. The thallos salt or salts froze at the room temperature. The top of the frozen thallos cake was then washed out carefully with boiling water. The solids at the bottom were the mineral particles with a specific gravity greater than 4.95, the specific gravity of the thallos formate.

The disadvantages of the above method are:

(1) temperature control of the liquid is critical as the specific gravity is affected by any temperature change, (2) convection currents caused by the heat lamp may carry mineral particles of higher specific gravity than the median to the surface, and (3) there is no control on the completeness of removing all the floated particles before floating the next successive rising of higher specific gravity particles.

To avoid the above disadvantages, a double layered conical tube apparatus² was constructed (Fig. 1). The bottom of the inner tube has a 3-ml

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² To use this apparatus as a centrifuge tube, one has to add a rubber ring between the necks of the tubings to balance the strain of the inner tube against the outer. The writer, in

diameter opening to allow the heavy minerals to pass into the bottom of the outer tube. The light fractions and the liquid can then be transferred from the inner tube into another outer tube. The addition of the light members of the heavy medium would lower the specific gravity of the mixture and the mineral particles would sink to the bottom of the outer tube at each lowering of the specific gravity of the liquid median. After each sink of the heavy mineral fraction of a particular specific gravity, the light minerals and the major portion of the liquid median are transferred to another outer tube to make separation. Repetition of this operation with the same inner tube and a series of outer tubes, the complete mineral suite can be separated into mineral fractions according to the decreasing order of specific gravity.

The mixing of the water and the thallos formate not as easy as expected, and the resulting liquid mixture became heterogeneous and formed stratified layers. The different mineral fractions were separated into stratified suspensions according to their different specific gravities.

This phenomenon forms the basis for the technique of the heavy media column separation as a petrographic method. The column, once set up, becomes stable at room temperature and is able to separate the mineral constituents of rocks and ores into fractions according to their individual specific gravity. If liberation is complete, pure or relatively pure mineral fractions are produced. The column can be repeatedly used and may have metallurgical possibilities in the field of ore separation.

EQUIPMENT AND HEAVY LIQUIDS REQUIRED

Column containers. Two or more 25-mm O.D., 1.45-ml thick glass tubes four feet long are used as column containers. The upper end of the tubes should be flared and wired to hang. The wire should be stiff enough to form a loop of at least 20 cm in diameter above the flared neck of the tubings to facilitate pouring liquids and solids from the upper end. The 25-ml O.D. tubing will handle 30 to 70 gram samples, depending on their grain size. For 15-ml O.D. four-foot tubing, 10 to 30 g samples are preferred. About 8 to 10 inches of $\frac{3}{4}$ -inch diameter rubber hose is attached to the lower ends of the tubing and fastened with adequate hose clamps. For the small diameter tubing, rubber tubing of $\frac{3}{8}$ inch O.D. is used without clamping. Tygon tubing is semi-transparent, but tends to soak up the tetrabromoethane heavy media, first turning transparent, and then

1956, showed Dr. E. H. Nickel the advantages of this system in comparison with the one he had just described (1955). It had also been demonstrated, with the heavy media column separator, to Professor R. L. Miller and his sedimentation class at the Petrographical Laboratory of the Engineering Division of Crane Co. at Chicago in 1955. But in 1957, the double-tube apparatus was published in the *Canadian Mineralogist*, Cheeseman (1957).

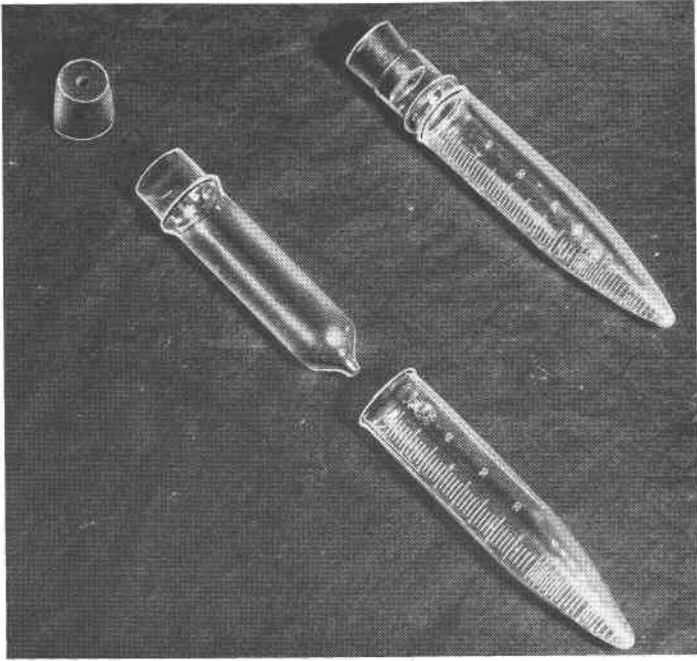


FIG. 1. Double tubes heavy media separation assemblage. Also, illustrates view of the separated components.

sweating the heavy media out of the tubing. For short periods it is better, but rubber tubes are preferred because they are more elastic and last longer.

Roller pinch valves. In order to move the liquid column up and down inside the glass tubing, a roller pinch valve was designed (Fig. 2). It consists of two metal rods and two washers. The rods can be made from welding rods, but the writer also used 6-inch common nails with a diameter of $\frac{1}{4}$ inch and $11/16$ inch I.D. washers.

Filtration assemblages. The heavy media used are expensive and poisonous. A filtration assemblage was constructed to recover the heavy liquids and to eliminate the use of filter paper (Fig. 2). With a $1\frac{1}{2}$ -inch rubber hose and two hose clamps, a filtration disc is fastened between two segments of glass tubing, the same diameter as the column glass container. The upper segment of the glass tubing is about three inches, and the lower one is about two inches long. The disc is made of one and one-half inch diameter circular filter paper sandwiched between two nylon, silk

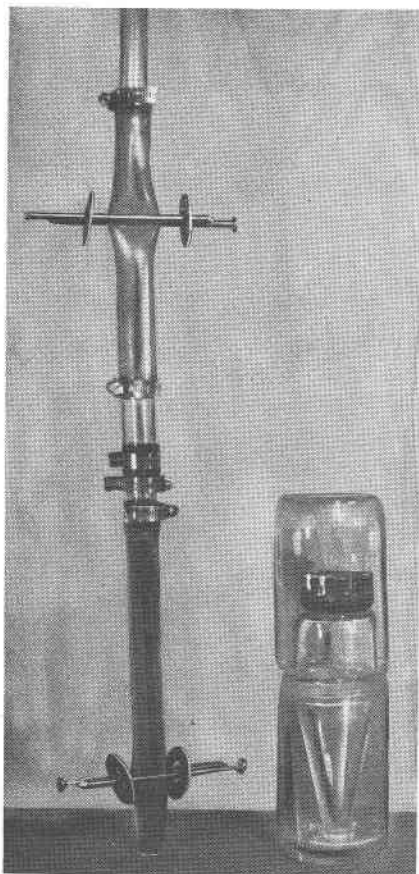


FIG. 2. The filtration assemblage (lower left) with roller pinch valve attached and (right) the heavy media recovery assemblage.

screens or linen discs of the same diameter. In case the samples to be filtered contain no very fine particles, 200 mesh silk screen alone is used as the filtering disc. The disc is first wrapped around the lower end of the glass segment and then fastened by the rubber hose and clamp. The lower tubing segment will secure the disc further. Another 8-inch rubber hose is clamped on the lower end of the lower glass segment. This assemblage, so constructed, can be used repeatedly and has many advantages over the use of filtering paper.

The heavy media recovery assemblage. When tetrabromoethane or other volatile heavy liquids are used, the mineral particles soak up the heavy media and the large size fractions are hard to clean by washing. In this

case, a sweat box type heavy media recovery assemblage (Fig. 2) is used to drive the heavy liquid out of the solids with a heat lamp. The assemblage is made out of two large wide-mouth instant coffee jars and one beer bottle. The narrow neck collar of one jar is cut out so that the other one can be fit upside down to cover it in the manner of a box. The beer bottle is placed upside down in the glass box assemblage to form an elevated platform for the drying tray. The drying trays are cut out of the bottom one and one quarter inch section of a beer bottle.

Collections of mineral or solid particles of known specific gravity. Two series of solids or mineral particles are desirable, but not absolutely necessary. One series is in the size range of about $\frac{3}{4}$ - to $\frac{1}{2}$ -inch pieces to be used for the preparation of liquids of the desired specific gravities; and the other series consists of uniform $\frac{1}{4}$ -inch pieces to be put into the column to aid the identification of the specific gravity range of each mineral fraction. The collection should consist of the common rock-forming minerals, heavy minerals, or ore minerals which one would expect to be separated with the heavy media column. For very low specific gravity solids, glass fragments, and plastics which resist the heavy liquids used, aid in the separation of zeolites and clay minerals.

Stirrer. A half blade of a double-edged razor or a paper clip wire, pre-coated with plastic, and a strong hand magnet, are used for stirring purposes. The stirrer is necessary only when very fine-grained samples are separated.

Heavy Media. All the heavy media listed on page 329 of Krumbein and Pettijohn (1938) can be used to establish a heavy media column for the present purpose. The writer uses thallos formate (s.g. 4.95) for the heavy minerals (s.g. higher than 2.95) and tetrabromoethane for the light minerals (s.g. lower than 2.95). Theoretically, one can use thallos formate and water combination to make a column to separate the minerals having a specific gravity range from 1 to 4.95. For routine column separation of rocks or detrital grains, the use of thallos formate is highly impractical because of its extremely poisonous property and high cost. A heat lamp is required, for the thallos salt heavy specific gravity ends when the mixture has a specific gravity of 3.5 and up (Krumbein and Pettijohn, 1938, p. 327).

PREPARATION AND PROCEDURE

Preparation of heavy liquids. Because of the convenience in capacity (300 milliliters) and the ease in pouring, the writer used beer bottles as con-

tainers for the different specific gravity liquids to be used in the column. The following formula was used in arriving at the desired specific gravities:

$$V_1 = V_h \frac{G_h - G_x}{G_x - G}$$

Where

- G_x = the specific gravity of the mixture desired,
- G_h = the specific gravity of the heavy end member of the mixture,
- G_l = the specific gravity of the light end member of the mixture,
- V_h = the volume of the heavy member of the mixture,
- V_l = the volume of the light member of the mixture.

In the above formula the volume of V_1 is set to find how much volume V_h is necessary to make a liquid of desired specific gravity G_x .

A second method of obtaining the volume proportion of the two end members in making a liquid of desired specific gravity is to make a mixing curve (a straight line) for the two end members. This is similar to the phase diagram of a binary system. The liquid is thoroughly mixed and tested for specific gravity by: (1) directly measuring the weight of 100 milliliters of the mixture in a graduate cylinder, (2), placing two mineral fragments of known specific gravity into the liquid mixture. If necessary, the specific gravity is adjusted by adding small amounts of either the heavy or the light end member into the mixture by a dropper or a syringe, until one of the two mineral fragments floats and the other sinks. A suction shock on the mixture with the two fragments is sometimes necessary to avoid air bubbles trapped within the cracks of the solids. When both solid fragments sink, suction can also be used to boil off the alcohol or other easily volatile light specific gravity end member until the lighter one of the two floats.

With the above procedure, a series of liquids can be prepared with specific gravities equal to the middle points of the specific gravity of each successive pair of the minerals to be separated.

Procedure

1. To establish a heavy media column

The sample, if it is a rock, is crushed to pass a 16 mesh or finer screen to liberate the mineral particles. The crushed sample is then dried and split to a 30- to 70-g. sample. At least two of the column containers (A and B) are hung with pulleys and nylon cords in groups. The heavy liquids are poured into one of the containers in the order of heavier one first. The glass tubing should be slanted, so that the liquid can be poured trickling down the side without causing churning when reaching the bottom. Each liquid column should be about four inches in length (about 30 to 40 ml). Between each pouring a fraction of the sample should be also poured into the column. The solids which float would form a barrier and would prevent the mixing of two different specific gravity liquids. As soon as the solids

are wetted completely, a thin layer of the light liquid end member is poured on top of the column to prevent evaporation. Several suction shocks are applied to the column, so that all the trapped air can be released.

The column is allowed to stand with occasional stirring, if necessary. The stirring can be done with a half razor blade and hand magnet or by slowly pinching the rubber tubing above the roller pinch valve at the bottom of the column. The action would allow the stratified solid grains to form passageways for the sinking and floating particles and also to mix the liquids of the two different specific gravities to form a layer of intermediate or diffused specific gravity and consequently broaden the band of the solid particles. The broadened band indicates that there is a very small variation of specific gravity between particles even of the same mineral species. The stirring is necessary when the population of certain mineral varieties is large or when the sample contains mostly the clay size particles. The column is established and ready for separation when no more particles travel between stratifications. The time for the complete establishment of the column is between 15 minutes to overnight, dependent on the particle size of the sample.

At this stage or earlier the $\frac{1}{4}$ -inch diameter mineral collection particles of known specific gravity can be dropped into the column to observe the specific gravity range of the liquid segments and the stratified solid layerings or to mark the mineral fractions after they have been separated and dried.

2. Fractionation and filtration

To make a separation, the filtration assemblage is attached at the lower end of the column. After maneuvering the first roller pinch valve to lower the liquid column to a convenient position, another roller pinch valve can be used to separate two individual solid strata. When using Tygon tubing instead of rubber hose, you can see through the tubing and find the favorable position to put the second valve. With the rubber hose, a three to four inch long liquid segment is broad enough to be estimated where the valve should be placed to make separation. This position can also be checked by squeezing the upper solid into the glass tubing.

As one lowers the lower pinch valve, a third pinch valve can be inserted close to the upper valve, and with two lower valves the separated bottom portion of the column can be carried down to the top of the filtration assemblage.

Before releasing the lower column segment into the filtration assemblage, the other empty glass column container (column container B) should be lowered down and connected to the lower end of the filtration assemblage by simply inserting the rubber hose into the flared upper opening. Both the assemblage and the tubing B should be slanted. The separated column segment is then released into the empty glass tubing through the filtration assemblage. The filtration process can be speeded up by rolling down the roller pinch valve. In this operation no air bubbles should be allowed to enter the upper section of the column and disturb the stratification.

After the transfer of all the liquid to column B, it may be used to separate the next sample.

3. Washing of the fractions

After the liquid is completely drained, the filtration assemblage is removed from the column containers. The solids can be easily transferred into a drying dish. The washing is simplified by closing the lower end with a roller pinch valve and turning the assemblage upside down. The rubber hose is used as a rubber nipple, as it was a dropper to suck the washing liquid (alcohol or water in case of thallos formate), allowing the solids to drop into the drying dish. The washing can be repeated by decanting the washing solution from the dish until it is ready for drying. After drying, the filtration assemblage is ready to be used again.

4. *Drying and recovery of the heavy liquid*

For the thallos salt, the fractions should be repeatedly washed with distilled water and then dried directly under the heat lamp. To test the thoroughness of the washing, a drop of potassium dichromate is added to a drop of the washing on top of a glass slide. If thallos ions are present, insoluble red crystals are produced.

For tetrabromoethane, or other relatively volatile heavy liquids, the drying tray is then put on an elevated platform of the drying assemblage or sweat box. A group of these sweat boxes can be set under one heat lamp to dry. A temperature of 60° to 75° C. is produced at a distance of four to six inches from the lamp. Intense heat would dissociate this compound to give a brown-colored bromine gas. Slow and low heat drying is advised. The drying time is between 15 minutes to overnight, depending on the grain size and the thoroughness of the washing. When the sample is very fine-grained, the liquid washings cannot be drained out easily and a cover glass is needed over the drying dish to prevent spattering.

All the mineral fractions can be separated in the above manner and are ready to be weighed and examined.

MAINTENANCE OF THE COLUMN

Both the tetrabromoethane and the thallos salt column were found to be stable for at least three months. But above room temperature, at the summer heat temperature of 80° to 90° F., the column will give broad band solid suspensions due to diffusions, and is stable for only a month. The column in this condition can be used for a period of a month. When solid suspensions were stored with the liquid column, the life was prolonged.

With the repeated transfer between the two column containers, the liquid sections became shortened, but the particular specific gravity liquids can be added or subtracted at will.

The rubber hose has a more superior resistance than the Tygon hose to soaking up tetrabromoethane, but also becomes swollen and cracks after aging. It is not advisable to use Tygon tubing in the storing of an established column.

The thallos formate can be cleaned by bone charcoal (Rankama, 1936). It can easily be recovered by evaporation of the water solutions under a heat lamp. A very small amount of formic acid should be added to the water solution during the process of slow evaporation to prevent the formation of thallos carbonate precipitation.

The tetrabromoethane can be cleaned by Fuller's earth (Venuto, 1958) (Griffitts and Marranzino, 1960). Wyoming bentonite drilling mud was found very effective in cleaning the tetrabromoethane.

The condensate collected in the bottom section of the sweat box is the recovered tetrabromoethane. The recovery of the tetrabromoethane from the tap water washing of the alcohol or acetone—heavy liquid mixture is usually not necessary, as they can be used for intermediate specific gravity liquids or can be converted to a desired specific gravity.

Rubber gloves and apron do not have to be used in handling tetrabromoethane, but are required in the handling of the thallos compounds. Tetrabromoethane can be washed off with alcohol or other light diluents. The cleansers with strong bleaching agents can also be effective in removing the heavy organic compounds from hands.

EXAMPLES APPLIED

Figure 3 illustrates the first heavy media column established with thallos formate for the heavy mineral concentrate from the Southeastern States. With a better control on the specific gravity of the liquids, the column recovered a hundred per cent extraction of the pure mineral frac-

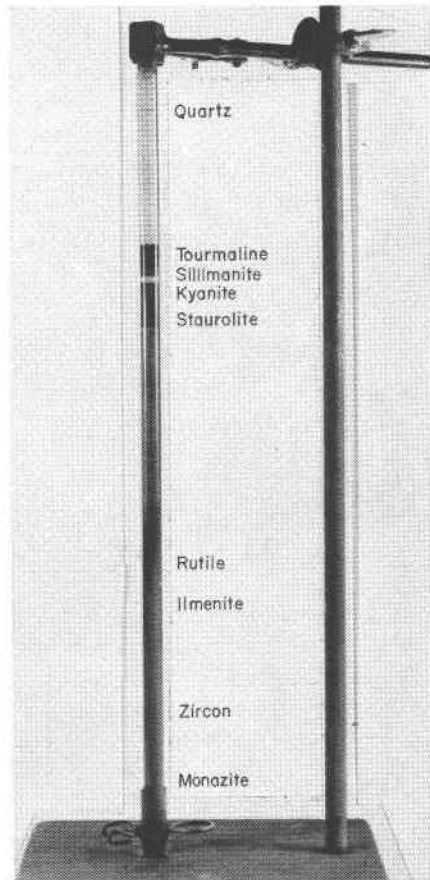


FIG. 3. Heavy media column with thallos formate and water to show the stratified heavy minerals.

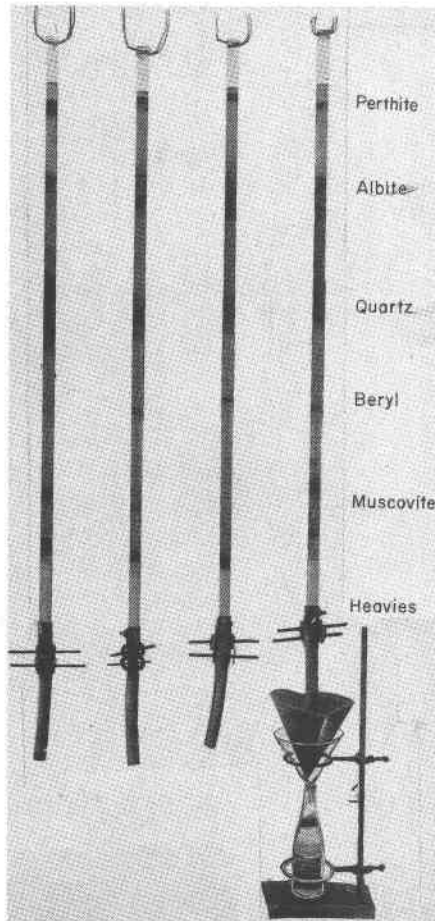


FIG. 4. Heavy media columns of pegmatite minerals from drill cores.

tions of the particular mineral suite. Figure 4 shows the application of the separation on pegmatite minerals. With the column, clean mineral fractions of perthite, albite, quartz, and muscovite were separated out. The column was also used successfully to separate the mineral fractions of beryllium-bearing tuffaceous beds in Utah. The column method has been used in making petrographic analyses of various rocks and ore samples. Because of the use of a relatively large amount of the sample, the heavy liquid column separation also aids in the detection of mineral even in a trace amount. The presence of trace amounts of cassiterite and columbite contained in heavy mineral deposits of the southeastern United States was found by the column method. In the volcanic tuff sample, montmoril-

lonite was separated with the opal, due to their similarity in specific gravity and the composite character of the two minerals. For the separation of clay minerals with heavy liquids and centrifuge, see references by Loughnan (1957) and Kittrick (1961). The column separation may probably be applied to the separation of the different clay minerals equally well.

Since tetrabromoethane is recoverable, the column method has the advantage of making a clean separation of pegmatite minerals in just one operation with almost a hundred per cent recovery of the fractions. The use of this technique would be very attractive in commercial scale mineral beneficiation of pegmatite minerals.

ACKNOWLEDGMENTS

The above technique was developed while with the Engineering Division of the Crane Company, and further refined with the Heavy Minerals Company, and Vitro Minerals Corporation. The writer is grateful for the encouragement freely delivered from all his associates, and he would also like to thank Mr. Eugene B. Hotchkiss, Executive Vice President, and Mr. Cecil H. Smith, Exploration Manager, of Vitro Minerals Corporation for their special interest in the technique and the permission in the publishing of this paper.

Figures 1 and 3 were taken by the Photography Section of the Engineering Division of the Crane Company, Chicago, Illinois.

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Manuscript received, May 3, 1963; accepted for publication, November 27, 1963.