**Zeitschrift:** Schweizerische mineralogische und petrographische Mitteilungen = Bulletin suisse de minéralogie et pétrographie

**Band:** 64 (1984)

**Heft:** 3

**Artikel:** Mineral separation by centrifugation with heavy liquids - improvement of a method

**Autor:** Gautier, André M. / Pechmann, Edmund von

**DOI:** https://doi.org/10.5169/seals-49557

*Schweiz. mineral. petrogr. Mitt. 64, 459–464, 1984*

# Mineral separation by centrifugation with heavy liquids – improvement of a method

by *André M. Gautier** and *Edmund von Pechmann***

**Abstract**

The centrifugation increases the speed gravity separation of finely ground samples with heavy liquids. But contamination of the sink fraction by particles of the float fraction might occur when emptying the centrifugation tubes. A very simple, efficient and cheap device, preventing this disadvantage, is described here. In addition, it reduces appreciably the handling of the highly toxic heavy liquids. This device consists of a tube-funnel which is placed into the centrifugation tube. During the centrifugation the sink fraction falls to the bottom of the centrifugation tube through the funnel; this latter, containing the float fraction, is then closed with a PVC rod and is taken out of the centrifugation tube.

This device has been used successfully for samples with a strong interlocking of opaque minerals and matrix or with thin coatings of opaque minerals on quartz grains, which made very fine grinding necessary.

*Keywords:* mineral separation, heavy liquids.

**Résumé**

Le recours à la centrifugation, pour accélérer la séparation gravifique de minéraux, finement broyés, au moyen de liqueurs lourdes, se heurte à des problèmes de contamination de la fraction lourde par des particules de la fraction légère lorsque l'on vide des éprouvettes. Il est décrit ici un dispositif simple, efficace, et bon marché, qui supprime cet inconvénient. Il réduit de plus au maximum les manutentions de liqueurs lourdes, hautement toxiques. Ce dispositif consiste en un tube-entonnoir placé à l'intérieur d'une éprouvette. Pendant la centrifugation, la fraction lourde se concentre au fond de l'éprouvette après avoir traversé l'entonnoir; par la suite, ce dernier, trappant la fraction légère, est fermé au moyen d'une tige de PVC et est retiré du tube de la centrifugeuse.

Ce dispositif a été utilisé avec succès pour des échantillons où le minerai et la gangue étaient finement enchevêtrés, ou où de fines pellicules de minerai étaient plaquées sur des grains de quartz, ce qui avait nécessité un très fin broyage.

* Uranerzbergbau-GmbH, Kölnstrasse 367, D-5300 Bonn 1, Western Germany. Present address: Lindenweg 22, D-5305 Alfter-Oedekoven, Western Germany.

** Uranerzbergbau-GmbH, Kölnstrasse 367, D-5300 Bonn 1, Western Germany.

**Kurzfassung**

Zentrifugieren beschleunigt zwar die Schwermineralabtrennung aus fein gemahlenen Proben in Schwereflüssigkeiten, birgt jedoch die Gefahr, dass bei der Leerung der Zentrifugengläser die Schwermineralfraktion durch die Leichtmineralfraktion verunreinigt wird. Dieser Nachteil wird durch das hier beschriebene einfache, wirkungsvolle und billige Hilfsmittel vermieden, wodurch zugleich der Umgang mit den hochgiftigen Schwereflüssigkeiten auf einem Mindestmass gehalten werden kann. Ein im unteren Teil trichterförmiger Glaszylinder wird in ein Zentrifugenglas gesetzt; während des Schleudervorganges fällt die Schwermineralfraktion durch die Trichteröffnung auf den Boden des Zentrifugenglases. Der Trichtereinsatz wird nach Verschliessen seiner Öffnung mit einem PVC-Stäbchen mitsamt der leichteren Fraktion aus dem Zentrifugenglas herausgehoben.

Das beschriebene Hilfsmittel wurde sehr erfolgreich bei Proben angewandt, die zur Mineraltrennung sehr fein gemahlen werden mussten, z. B. wenn die Grundmasse sehr fein mit den Erzmineralien verwachsen war oder wenn in einem Sandstein die Opakbestandteile feinste Häutchen auf den Quarzkörnern bildeten.

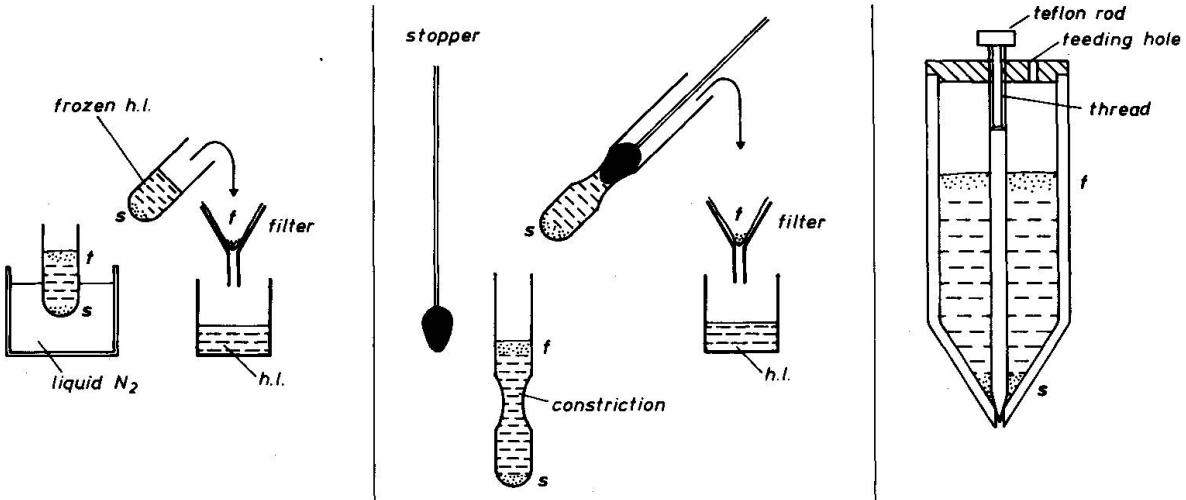
## 1. Introduction

The density separation of very fine fractions (i.e. with a grain size between 0.010 and 0.040 mm) of mineral phases in heavy liquids is a standard procedure in the mineralogical laboratory.

The several hours' need to complete a gravity separation in this granulometry range can be drastically reduced with the use of a centrifuge. However, mineral grains of the float fraction commonly stick to the centrifuge tube walls and contaminate the sink fraction during subsequent recovery.

## 2. Recovery methods

The conventional recovery methods of the sink and float fractions from the centrifugation tubes are (fig. 1):




1a: Cryogenization

1b: Constricted tubes after R.SCHOEN and D.E. LEE (1964)

1c: Separating vessel principle, here after E.RÖDEL (1981)

*Fig. 1* The conventional methods of recovery of the sink (s) and float (f) fractions from the centrifugation tubes (h.l. = heavy liquid).

*a) The cryogenization* (Fig. 1 a)

The tube with the heavy liquid is dipped into liquid nitrogen after the centrifugation, in such a way that the lowest part of the heavy liquid and the sink will freeze at the bottom of the tube. The float is then poured out and the upper part of the tube cleaned with a jet of heavy liquid. The sink is recovered after melting of the frozen heavy liquid.

The disadvantages to this technique are that liquid nitrogen may be difficult to obtain outside large research facilities and cannot be stored over long periods of time as would be necessary in a field laboratory, and that a jet of hazardous heavy liquid is required to clean the tube.

*b) The constricted tubes* (Fig. 1 b) (after SCHOEN & LEE, 1964, in HUTCHISON, 1974)

The centrifugation tubes have been blown in such a way that they have a constriction at their lower third. Specially shaped stoppers are pushed through the "float" cakes, to close the constriction after the centrifugation. The float and the sink fractions are recovered separately. The tube is cleaned with a jet of heavy liquid between the two operations.

The disadvantage to this technique is the use of a jet of hazardous heavy liquid for cleaning.

*c) Centrifugation glass tubes built like a separating vessel*

This method requires tubes that have a funnel shaped bottom with a stopcock that allows the recovery of the sink. Such centrifugation tubes are used routinely for biological work. However, they are not strong enough for use with heavy liquids. Another development of tubes built on the separating vessel principle is proposed by RÖDEL (1981) (Fig. 1 c): The lowest point of the funnel shaped tube bottom has a small orifice closed by a Teflon rod in this technique. The Teflon rod is screwed through the special stopper closing the upper end of the tube. The sink can be recovered by unscrewing the Teflon rod, as with a standard separating vessel. This method is very convenient, apart from the very high price of the tubes[1].

Any convenient recovery method of the sink and float fractions should:

- prevent the float from going into suspension again and mixing with the sink, and
- reduce to a minimum the handling of hazardous heavy liquids (HAUFF &

[1] manufactured by Heraeus-Christ GmbH, D-3360 Osterode, FRG.

AIREY, 1980). The use of a jet of heavy liquid from a squeeze bottle, as it is the case in several laboratory procedures, is of particular concern.

### 3. An improvement of the centrifugation glass tubes (Fig. 2)

The equipment needed includes a second tube, funnel shaped at the bottom, with an outward lip at its top, that allows it to hang inside the standard 100 ml centrifuge tube. The bottom of the second tube has an aperture approximately

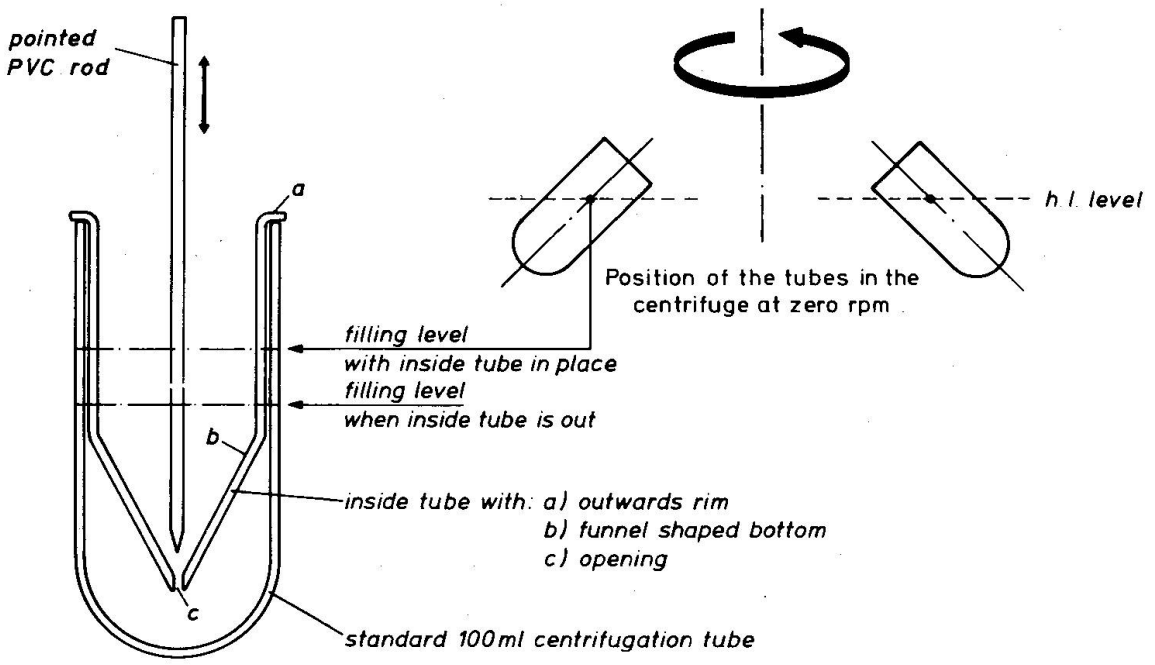



*Fig. 2* Description of the improved equipment for the recovery of the sink and float fractions. The respective filling levels have been marked inside the centrifugation tube with HF acid.

2 mm in diameter. The aperture is closed by a pointed PVC, polyethylene or Teflon rod, which extends above the top of the tubes. The filling level of the tubes has been marked with hydrofluoric acid.

The separation procedure using this design is as follows (Fig. 3):

a) The centrifugation tubes with their inner tubes set inside are filled up to the marks with the appropriate heavy liquid, and the apertures of the inner tubes are then closed.
b) The mineral sample, which has been crushed, washed, sieved, eventually separated into fractions of similar grain size by decantation, and dried, is poured into the inner tubes. All the tube weights are equalized, a standard procedure with centrifuge use.

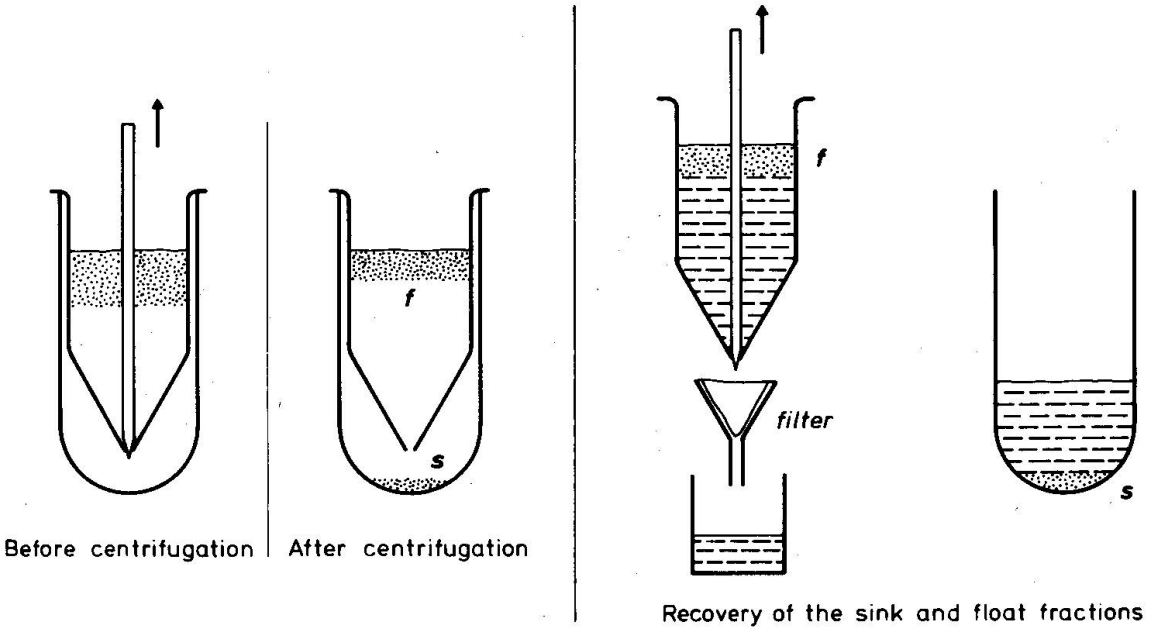



*Fig. 3* Separation principle and procedure.

c) The sample is stirred with a glass rod until the mineral powder is well suspended in the heavy liquid. The rods closing the inner tubes are pulled out.
d) The tubes are accelerated in the centrifuge (we use approx. 3000–3500 rpm for an outer diameter of 28 cm, with methylene iodide), causing the heavy grains to go through the opening of the funnel shaped inner tube into the centrifuge tube.
e) After centrifugation, the rods are carefully set again into the apertures. The inside tubes containing the float fractions are removed and emptied over a filter. The centrifuge tubes with the sink are emptied over a separate filter. After recollection of the heavy liquids, the tubes can be cleaned over the respective filters with a jet of alcohol (or acetone).

Remarks:
- The filling level of the heavy liquid should be low enough to prevent hydrostatic pressure from lifting the inside tube.
- The slope of the funnel shaped inner tube should be steep enough to prevent the sink particles from adhering to its surface.
- Once filled with the sample, the inside tube should never be lifted when the rod is not closing the aperture. A slight lift will cause a strong succion through the aperture, and light mineral grains may be sucked into the lower part of the tube, causing a contamination.

We have used this method very successfully in our laboratory for the separation of very finely dispersed grain coatings of pitchblende and coffinite from a

sandstone host. The separation could be done fast, without mineral grains from the float fraction contaminating the sink fraction, and without using jets of heavy liquids. The cost of the described inner tubes is very low, and they can be prepared easily by any laboratory glass blower.

**Acknowledgement**

The authors are grateful to the management of Uranerzbergbau-GmbH, Bonn, for permission of publication of this paper. They are indebted to Dr. F. Bianconi for critically reading the manuscript. The authors are very thankful to Mr. James D. Rasmussen for correcting the English text. The authors politely thank Mrs. M. Abu Salah, Mrs. E. Gruno, Mr. G. Möller and Mr. H. Fahnenstich for their help during preparation of the manuscript.

**References**

HAUFF, P. L. & AIREY, J. (1980): The handling, hazards, and maintenance of heavy liquids in the geologic laboratory. U.S. Geological Survey Circular 827, 24 p.

HUTCHISON, C. E. (1974): Laboratory handbook of petrographic techniques. Wiley, New York - London - Sydney - Toronto, 527 p.

RÖDEL, E. (1981): Schweretrennung von Mineralgemischen in Laborzentrifugen. Erzmetall, 34, 6, 349-354.

SCHOEN, R. & LEE, D. E. (1964): Successful separation of siltsize minerals in heavy liquids. U.S. Geol. Survey, Profess. Paper 501-B, B 154–B 157.

Manuscript received 10 August, 1984.