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An Improved Method for Rapid, Low Loss Density Separations with Heavy Liquids

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Abstract

A rapid, clean, and low-loss approach to heavy-liquid density separations has been developed. The technique utilizes freezing in liquid nitrogen to immobilize particle distributions and therefore allows the investigator to work with stationary density gradients. Procedurally, the material to be examined, along with the appropriate heavy liquid, is introduced into a specially constructed centrifuge tube. After either gravitational or centrifugal separation the tube is frozen and cut to separate the density fractions of interests.

Introduction

In making density and grain-size separations of lunar soils, constraints of small sample size and the need for cleanliness made the standard density techniques unacceptable from the point of view of time, losses, and handling. The technique we developed reduced all these problems and, in addition to increased cleanliness, had the advantage of creating an immobilized density continuum.

The basis of our method is solidification of the heavy liquid, through freezing, to preserve the density stratifications of the sample. The frozen liquid and sample can then be separated without disruption of the layering, loss of sample, or contamination. With the exception of an article by Cadogan *et al* (1973), we have not come across a similar process.

The method discussed below has been used successfully with undiluted and acetone diluted bromoform, methylene iodide, and 1,1,2,2 tetrabromoethane. We see no reason why it shouldn't work with other heavy liquids.

Experimental

Construction of the Centrifuge Tube

Specifically, a 3.5 cm length of 5/16″ O.D. Teflon rod is partially inserted into a 10 cm length of translucent, 5/16″ I.D. thinwall Teflon (FEP) tubing. If gravitational density separations were done, this press-fit seal between the rod and the tube proved sufficient to prevent any leakage. However, if the separation was done with a centrifuge, additional tightening of the tube-rod junction was necessary. The simplest solution was to adapt a common laboratory plastic tube union (*e.g.*, Nalgene polypropylene 3/8″ Tube-to-Tube-Union) by cutting the union into two parts (Fig. 1). The remaining coupling and nut served as a compressive fitting when placed around the tube and rod junction and tightened firmly.

Density Separation

The heavy liquid and sample were next introduced into the tube and the desired amount of settling allowed to occur. When a centrifuge was used, it was also necessary that the Teflon plug be supported from below. If this was not done, the plug at times would shift and cause loss of fluid and sample. The standard 40 ml conical-bottom centrifuge tube served as an ideal holder because it prevented both the plug and the tube union from shifting downward during centrifuging.

When the settling of the sample was complete, the union was carefully removed from the tube and rod combination. The tube and rod were then inserted in liquid nitrogen for one or two minutes until the mixture of liquid and sample solidified. The tube was then removed from the liquid nitrogen, cut at the desired levels, and the density fractions separated and allowed to melt into separate containers.

Another method proved practical but less convenient. Density separation was done in the normal way with heavy liquids in a pyrex centrifuge tube. After the separation was complete, a small thin rod of low thermal conductivity was inserted part way into the tube and the combination placed in liquid nitrogen. After freezing, the tube and rod were removed from the nitrogen and the tube was dipped into warm water. From the moment that the tube was immersed in the water, steady light tension was maintained on the rod until a core of the heavy liquid and sample mixture could be removed. This core was cut and apportioned in the same manner as was done with a separation in the Teflon tubing. The disadvantages of this method stem from the fact that, in the absence of the Teflon sleeve, the core melts faster and all procedures have to be done quickly before it begins to soften.

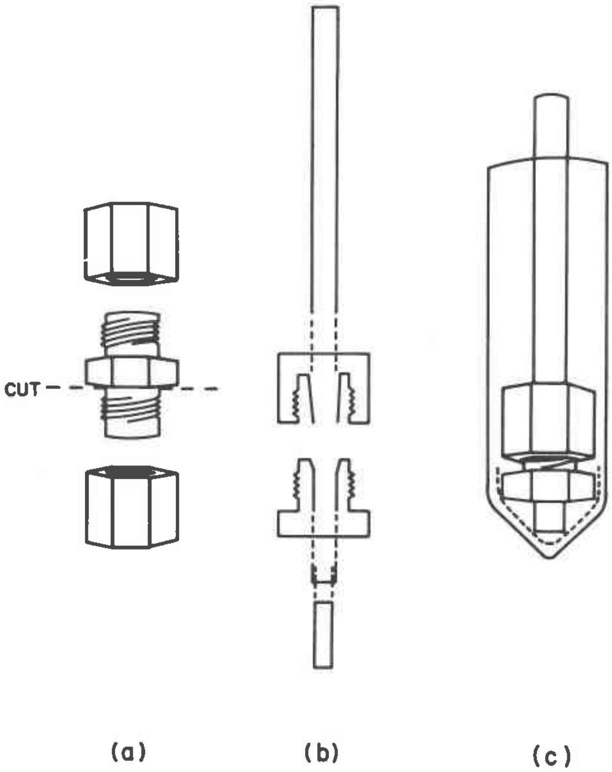

FIG. 1. (a) Tube union, (b) exploded view of assembly, and (c) assembled unit in supporting 40 ml centrifuge tube. Note that the tube union is cut directly below the central nut and that both the shoulders of the union and the end plug of the Teflon tube in (c) are shown supported by the 40 ml centrifuge tube.

Results and Conclusion

The benefits of this method are the immobilization of the density graduations in the tube, low losses, and reduced sample handling. So far we have used mainly the standard "sink-float" approach with the separations continued until nearly complete, at which point the tube is frozen and cut in the middle. We are also experimenting with a soil fraction whose particle sizes have been fixed within a narrow range with successive sievings. This soil is then introduced into the top layer of a column of heavy liquid whose density is low enough that all mineral components of interest sink. Then, after waiting until there is a distribution of sample up and down the tube, we freeze the mixture and cut cross-sections at the strata where, from Stokes Law calculations, we expect the various minerals to be.

Acknowledgments

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References

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