

GRAVITY-SEPARATION IN POWDER MOUNTS AS AN AID TO THE PETROGRAPHER

WILFRID R. FOSTER, *Champion Spark Plug Company, Ceramic Division, Detroit, Michigan.*

ABSTRACT

Attention is called to the tendency on the part of refractive index liquids to cause gravity-separation in immersion mounts of mineral mixtures. By the proper choice of liquids, this tendency can be accentuated and used to advantage in the routine petrographic examination of a number of raw and processed minerals. A satisfactory procedure for this purpose is outlined, and suitable media are described. The value of the method is illustrated by a number of a typical examples of its application.

INTRODUCTION

The use of refractive index liquids for the determination of the specific gravities of minerals under the microscope has been advocated by Rogers (7), Meen (5) and Jahns (2). The phenomenon of gravity settling in index oils can also be used with considerable profit in the microscopic analysis of mineral mixtures. Many petrographers probably do not realize the tendency of index oils to effect segregations in mounts of polymineralic powders, and, therefore, sometimes fail to detect certain constituents present in small amounts. Once recognized, however, this behavior proves to be an advantage rather than a disadvantage. There are numerous instances in which a pair of minerals that are practically indistinguishable through their principal optical properties can thus be differentiated on the basis of specific gravity differences.

PROCEDURE

The technique involved need be no more elaborate than the preparation of an ordinary powder mount. The use of a naked mount without benefit of cover-slip, as practiced by Rogers (7), is not recommended. Meen (5) employed a covered mount and observed the behavior of the smallest of the particles. This method is quite satisfactory. It may be found preferable, however, to insure that the depth of the film of immersion oil be several times greater than the diameter of even the larger grains. This may be accomplished by adding to the fine-grained powder a few coarser sand grains in order to prop up the cover-slip. Satisfactory results may also be obtained by the use of "hanging-drop" or "deep-well" slides, but the depressions in these are unnecessarily large and deep and require rather large amounts of liquid.

A method which has been found very convenient involves the use of a simple and easily constructed slide. Two strips about three-eighths

inches in width are cut from the ends of a 26×46 millimeter slide. These are cemented along the sides of the upper surface of a second slide by means of "water-glass." They are then ground down to about one-fourth or one-fifth of their original thickness. This provides a shallow trough down the center of the slide about one inch in length, one-quarter inch in width, and about 200 to 250 microns in depth. Cover-slips obtained by quartering five-eighths inch square cover-glasses will just bridge the trough (Fig. 1).

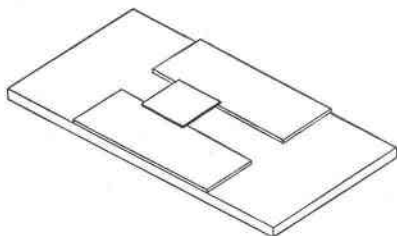


FIG. 1

A drop of the desired immersion liquid is placed in the trough, a pinch of the mineral powder added and stirred, a cover-slip dropped on, and, if necessary, a second drop of liquid placed so as to creep under the cover-slip and expel the remaining air. By a mere change of focus of the microscope objective, one can readily differentiate between grains which lie on the bottom of the trough and those which float just beneath the cover-slip. If the "floats" should be found to mask the underlying "sinks" too completely, the slide should be slightly tilted on end and the cover-slip carefully moved a short distance towards the raised end of the trough. The lighter minerals will tend to follow this movement, whereas the heavier constituents will lag behind.

SUITABLE MEDIA

In general, liquids of low indices of refraction also have low specific gravities, and liquids of high indices have high specific gravities. Consequently, any blended series of index media covering a considerable range of indices will serve also as a ready-made series of specific gravity standards. The more satisfactory liquids for this purpose exhibit a small change of specific gravity for the smallest measurable increment of refractive index. This feature permits close calibration of specific gravity through measurement of the refractive index, as practiced by Jahns (2), Meen (5) and Merwin (6). Curves showing the relationship between refractive indices and specific gravities for a number of commonly used liquid series are presented in Fig. 2. It will be seen that mixtures of the

halogenated naphthalenes and methylene iodide are less suitable than Clerici, Klein, Thoulet, or Rohrbach solutions. The former, however, are available in many petrographic laboratories and can be used to effect many desired segregations in mounts of powdered mineral mixtures.

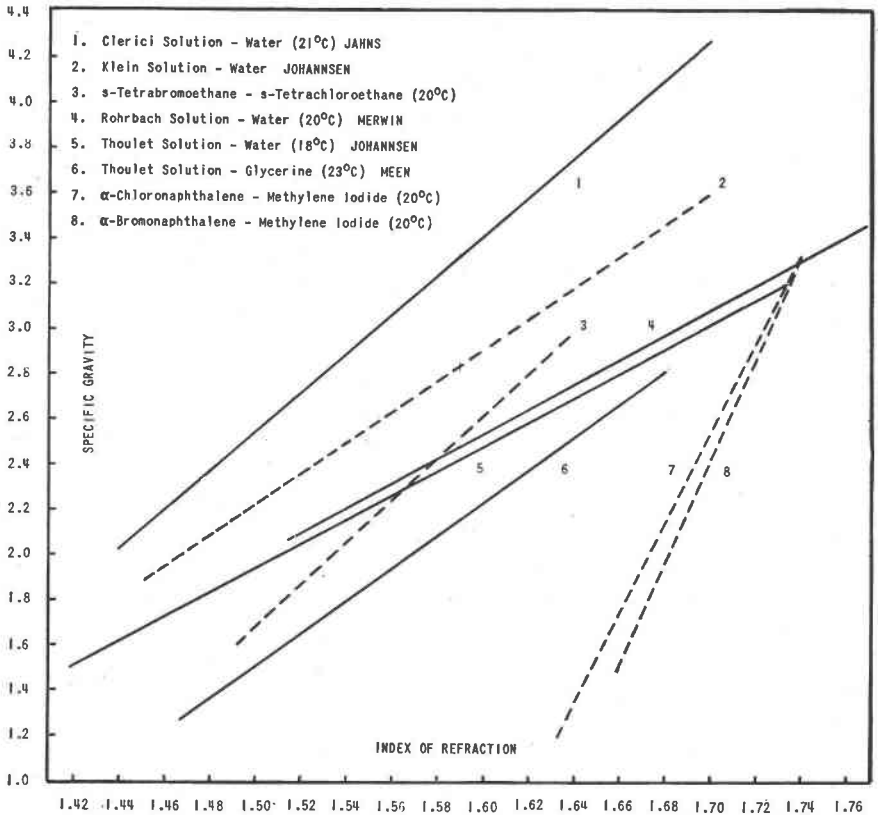


FIG. 2. Relation between specific gravity and refractive indices for various liquid-mixtures. Solid Lines: Plotted from data of previous workers as indicated. Broken Lines: Plotted by interpolation of data for pure end-members, taken largely from Lange (4).

There are instances in which it is profitable to employ a liquid combining a certain specific gravity with a certain refractive index. Thus, in complex mineral mixtures it may be desirable approximately to match the indices of a particular constituent while at the same time bringing about gravity separation. It has been found possible by judicious choice of liquids, either singly or in appropriate mixtures, to obtain almost any

desired combination of refractive index and specific gravity. For such purposes, the extensive data given by Lange (4) will prove helpful.

TYPICAL APPLICATIONS

In the routine examination of refractories by the immersion method, alpha-alumina (corundum) in small amounts can easily be overlooked if one fails to take cognizance of the gravity-separating action of methylene iodide-rich immersion liquids. Even in the shallow depth of the usual microscope mount the tiny alumina grains, resting directly on the slide, will be out of focus with the bulk of the powder and thus escape detection. Careful focussing on the lowermost level of the mount in effect screens out the predominant lighter constituents so that the alumina can be readily recognized. The presence of small quantities of corundum in rock-powders is also more readily confirmed by the use of this procedure.

In special porcelains containing beryllia (bromellite) and magnesia (periclase) or magnesium aluminate (spinel) small amounts of the latter two constituents are difficult to detect. The refractive indices of all three of these crystalline phases are quite close and in aggregate-grains the trace of magnesia or spinel may go unnoticed. However, in a liquid (S.G. = 3.2) which approximately matches the indices, any free grains of magnesia (S.G. = 3.6) or spinel (S.G. = 3.6) will assume a lower level in the mount than will the beryllia (S.G. = 3.0). Careful search of the bottom level of the immersion mount will reveal the isotropic magnesia or spinel. In like manner the presence of chrysoberyl (S.G. = 3.7) in beryllia bodies can be established, notwithstanding the fact that the two compounds are very similar in refractive indices and birefringence.

During an investigation of the impurities in a grab sample of brucite from Luning, Nevada, powder from a supposed crust of hydromagnesite (S.G. = 2.18) was being checked microscopically in a certain index liquid (S.G. = 2.15). As was anticipated, the irregular fragments of hydromagnesite rested on the bottom of the mount. Unexpectedly, however, there also appeared some floating laths and needles, suggesting a different and lighter mineral. Further work led to its identification as the rare mineral artinite (S.G. = 2.03), hitherto unreported from any American locality except Hoboken, New Jersey. It is interesting to note that the mean indices of hydromagnesite and artinite are very close. Had there been no gravity separation, the artinite needles would undoubtedly have been dismissed as fibrous grains of hydromagnesite. It had been intended to report this occurrence in a separate paper until inquiries revealed that the recent paper of Hurlbut (1) was already in press.

The advantages of this technique in more successfully studying certain of the impurities in commercial brucite have been suggested in the preceding paragraph. Thus hydromagnesite (S.G. = 2.18), artinite (S.G. = 2.03), and hydrotalcite (S.G. = 2.06) have decidedly lower specific gravities than brucite (S.G. = 2.4). Similar benefit may be derived in the examination of samples of some of the common hydrated magnesium silicates. The presence of brucite in chlorite (S.G. = 2.7), talc (S.G. = 2.7) or serpentine (S.G. = 2.6) might be easily overlooked because of the nearness of its indices to those of these three common associates. But the use of methylene bromide (S.G. = 2.5) as an immersion liquid will cause the lighter brucite to segregate in the upper level of the mount.

An andalusite-diaspore "ore" employed in the manufacture of spark plug insulators is checked petrographically to insure uniformity in the composition of successive shipments. A preliminary estimate of the subordinate pyrophyllite and muscovite is obtained from an ordinary powder mount. If these lighter constituents are then floated by using *s*-tetrabromoethane (S.G. = 2.96) as the immersion medium, the determination of the relative proportions of the two heavier and predominant constituents, andalusite and diaspore, is considerably facilitated. It so happens that the immersion oil almost matches the andalusite grains, but is distinctly lower in indices than the diaspore.

The minerals andalusite (S.G. = 3.15) and barite (S.G. = 4.5) are particularly difficult to distinguish optically when both are present in the same mixture. Examples of natural occurrences in which these two minerals occur together are an andalusite-corundum "ore" from Mono County, California, and a dumortierite-andalusite "ore" from Oreana, Nevada. The usual microscopic examination of these materials invariably failed to reveal the presence of the less abundant barite, although subsequent chemical analyses indicated an appreciable barium oxide content. The use of methylene iodide (S.G. = 3.3) solved the difficulty by floating the andalusite and leaving the heavier barite at the bottom of the mount. Low-fluorine topaz (S.G. = 3.5) is another mineral that is very easily confused optically with andalusite. Topaz is a fairly common associate of the sillimanite minerals and thus may accompany andalusite, yet stand little chance of detection in the mixture. Gravity separation in methylene iodide will aid in its recognition.

The presence of black opaque flakes in a rock-powder often poses the problem as to whether these represent carbonaceous material or a metallic mineral. A simple gravity-settling test will indicate whether it is the relatively light carbonaceous matter or a heavy metallic compound. Disseminated graphite may be distinguished from molybdenite in this manner. The special slide, previously described, is also suitable for test-

ing the possible magnetism of black opaque grains. A strong horse-shoe magnet lying flat on the stage of the microscope is moved to and fro alongside the slide. Any magnetic grains are free to rotate, unhampered by the overlying cover-slip, as the magnet is moved.

That the specific gravity of a mineral can be accurately determined on the stage of the microscope by the use of index liquids has been ably demonstrated by Jahns (2) and Meen (5). It should be pointed out, however, that prior isolation of the mineral is not at all necessary. The specific gravity of a mineral can be just as accurately measured in a mixture, providing that its optical properties are sufficiently characteristic to distinguish it from its associates. Should the sample at hand be that of a pure mineral, it may be found helpful to add a tiny bit of fine-grained corundum (S.G. = 4.0) or other heavy mineral to the powder mount during the preparation of the latter. It is then an easy matter to determine whether the mineral under investigation is at the same level as the corundum or at the higher level just beneath the cover-slip.

No attempt has been made to consider all of the possible applications of gravity settling in the microscopic examination of powdered mineral mixtures. Only those cases encountered in the routine analysis of certain ceramic raw materials and products have been discussed. If one may fairly judge from the data given in the mineralogical literature, the following pairs of minerals having closely similar optical properties should also lend themselves readily to differentiation by this procedure: cremoreite (S.G. = 2.2) and riversideite (S.G. = 2.64); fluorite (S.G. = 3.18) and yttrocerite (S.G. = 3.36); dolomite (S.G. = 2.87) and aragonite (S.G. = 2.94); colemanite (S.G. = 2.42) and howlite (S.G. = 2.58); ulexite (S.G. = 1.65) and inyoite (S.G. = 1.87); kurnakovite (S.G. = 1.85) and inderborite (S.G. = 1.93). Doubtless many other instances could be found among both minerals and inorganic chemicals.

REFERENCES

- (1) HURLBUT, C. S. (1946), Artinite from Luning, Nevada: *Am. Mineral.*, **31**, 365-369.
- (2) JAHNS, R. H. (1939), Clerici solution for the specific gravity determination of small mineral grains: *Am. Mineral.*, **24**, 116-122.
- (3) JOHANNSEN, A. (1918), *Manual of Petrographic Methods* (McGraw-Hill Book Co., Inc. New York, N. Y.), pp. 250, 255.
- (4) LANGE, N. A. (1944), Index of refraction-liquids: *Handbook of Chemistry*, 5th Edition (Handbook Publishers, Inc., Sandusky, Ohio), pp. 932-1026.
- (5) MEEN, V. B. (1933), Determination of specific gravity of minerals by use of index liquids: *Univ. Toronto Stud.*, Geol. Series, No. **35**, 47-50.
- (6) MERWIN, H. E. (1911), A method of determining the density of minerals by means of Rohrbach's solution having a standard refractive index: *Am. Jour. Sci.*, 4th ser., **32**, 425-428.
- (7) ROGERS, A. F. (1906), The determination of minerals in crushed fragments by means of the polarizing microscope: *School of Mines Quarterly*, **25**, 340-359.