

(Busz, 1893), we recommended that kamarezite be removed from the list of accepted mineral species; this recommendation has been accepted by a majority vote of the Commission on New Minerals and Mineral Names, I.M.A. (Chairman of the Commission, written comm., 1964).

ACKNOWLEDGMENTS

The authors are grateful to Prof. Clifford Frondel of Harvard University, Dr. Brian Mason¹ of The American Museum of Natural History, Dr. George Switzer of the U. S. National Museum, and Dr. Helmut Winkler² of the University of Marburg, who made available to us specimens labeled "kamarezite." Appreciation is also expressed to the following: Prof. Neuhaus, for his assistance in attempting to locate the type specimen of kamarezite; Dr. Max Hey, for his helpful comments and suggestions relating to possible interpretations of discrepancies between the analyses of kamarezite and brochantite; Dr. Alfred A. Moss, for the zinc analysis on the British Museum's specimen of kamarezite; Mr. Owen Ramsburg, private mineral collector of Washington, D. C., for the loan of his specimen of brochantite; and Harry J. Rose, Jr., our colleague at the U. S. Geological Survey, for x -ray fluorescence analyses.

REFERENCES

- BUSZ, K. (1893) ["Kamarezit," ein neues Mineral.] *Sitzber. niederrhein. Ges. Bonn*, **50**, 83-84.
- (1895) Mittheilungen über Caledonit, Kamarezit, Breithauptit und Magnetkies. *Neues Jahrb. Mineral.* **1**, 111-127.
- FORD, W. E. (1910) On some remarkable twins of atacamite and some other copper minerals from Collahurasi, Tarapaca, Chile. *Am. Jour. Sci.* **30**, 16-24.
- LEVY, A. (1824) On the new mineral substance (brochantite). *Ann. Phil.* **8**, 439-441.
- PALACHE, C. (1939) Brochantite. *Am. Mineral.* **24**, 463-481.
- , H. BERMAN AND C. FRONDEL (1951) *Dana's System of Mineralogy*. 7th ed., Vol. II. John Wiley & Sons, New York.

¹ Present address: U. S. National Museum, Washington, D.C.

² Present address: Göttingen Universität, Göttingen, Germany.

THE AMERICAN MINERALOGIST, VOL. 50, SEPTEMBER, 1965

MELANTERITE-ROZENITE EQUILIBRIUM

ERNEST G. EHLERS AND DAVID V. STILES, *Dept. of Mineralogy,
The Ohio State University, Columbus, Ohio.*

INTRODUCTION

Samples of altered pyrite were collected from an abandoned mine dump in the upper reaches of Sandy Run in Brown Township, Vinton County, Ohio, as part of a study dealing with the oxidation of iron sulfides. The

pyrite is from the Middle Kittanning (No. 6) Coal, which comprises part of the Allegheny Formation of the Pennsylvanian System.

The pyrite samples were encrusted with either masses of pale green fibers, extremely fine white powder, or a mixture of both. The fibrous material was determined optically to be melanterite, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. The remaining material, as determined by standard *x*-ray powder diffractometer techniques, proved to be mixtures of melanterite, rozenite, $\text{FeSO}_4 \cdot 4\text{H}_2\text{O}$, and halotrichite $(\text{Mg,Fe})\text{Al}_2(\text{SO}_4)_4 \cdot 22\text{H}_2\text{O}$. The *x*-ray spacings and intensities of melanterite and rozenite agreed almost completely with those given in a comprehensive study by Jambor and Traill (1963). Magnesium halotrichite, obtained from an adjacent locality, has recently been described by Brant and Foster (1959). Rozenite, which is monoclinic, has been clearly distinguished from triclinic siderotil $(\text{Fe, Cu})\text{SO}_4 \cdot 5\text{H}_2\text{O}$ by Jambor and Traill. Their nomenclature has been approved by the Commission of Mineral Names, IMA.

X-ray diffraction samples were prepared by grinding with an agate pestle and mortar. The fine powder was mixed to a slurry with acetone. A few drops of the slurry was removed with an eye dropper, and allowed to evaporate on a glass slide. The samples were run on a Norelco high angle diffractometer using Cu radiation with a Ni filter at 35 KV and 15 MA. Several of these smear mounts were re-examined by *x*-ray techniques several days after being collected, and it was discovered that the relative proportions of the melanterite and rozenite showed considerable change; the proportion of rozenite had increased; a few days later it was found that the proportion of melanterite had increased. Midgley (1962), during a discussion of an occurrence of rozenite (referred to as siderotil) stated that melanterite is the stable hydrate at room temperatures. Jambor and Traill (1963) state that, "Contrary to the statement of Midgley (1962, p. 408), the tetrahydrate (and not melanterite) is the stable phase at normal temperatures and humidities." In view of the apparent nonstability of both melanterite and rozenite in our samples, it was decided to examine the equilibrium relations between these minerals under controlled conditions.

The pentahydrate of iron sulfate, siderotil, was synthesized by Eckel (1933) from cuprian melanterite (pisanite) in an environment with less than 38% relative humidity at 22° C. Jambor and Traill (1963) who experimentally examined the stability of this mineral, concluded that the pentahydrate will only form in an environment in which at least 1.2% copper is present. Thus it was not expected to enter into the present study.

EXPERIMENTAL PROCEDURE

A device was constructed which allowed examination of a powdered sample by *x*-ray diffraction at fixed relative humidity values. Air, ob-

tained from a standard line, was brought to a tee connection. From the tee, the air could be brought through either a two-foot tube filled with either Drierite or Linde Molecular Sieve for drying purposes, or bubbled through two 1000 cc cylinders of water for achieving maximum saturation. The relative humidity in the system was controlled by the proportion of air allowed to flow through the wetting or drying sides of the system. The wet and dry air flows were recombined in a large bell jar. Wet and dry bulb thermometers were placed within the jar to measure the relative humidity. A rotating fan blade within the jar aided in the mixing of the wet and dry air, as well as encouraging evaporation on the wet bulb thermometer. The accuracy of the humidity readings was checked against a Serdex humidity gauge, as well as completely saturated air, and found to be $\pm 2\%$ above approximately 60% relative humidity, grading down to $\pm 5\%$ at the minimum obtainable humidity of 36%. The 36% minimum value appeared to be caused by the constant presence of moisture from the wet bulb. A few runs were made using almost completely dry air (which had been run through the Molecular Sieve) without the presence of the wet bulb.

The air at known relative humidity was then brought to a Norelco high angle x -ray diffractometer; a pair of holes were drilled in the center of the flat side of the radiation shield, and the air was constrained to flow through one of these, directly over the smear slide sample. The narrow x -ray entrance port was covered with Scotch tape, which caused no noticeable decrease in peak intensities. The air was allowed to escape through the second hole.

The samples examined by this technique were Fischer C. P. iron sulfate, which initially consisted of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ as determined by x -ray diffraction.

EXPERIMENTAL RESULTS

The $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, which was the starting compound used in this experiment, was completely converted to rozenite when subjected to humidities, less than 70–80%. The converted phases were in every case too fine-grained for observation with the polarizing microscope.

The powder diffraction pattern of rozenite presented is virtually identical to the data presented by Jambor and Traill (1963) on rozenite from Manitoba. The stability fields as seen in Figure 1 are a function not only of humidity but also of temperature. As would be expected, the field of stability of rozenite increases at the expense of melanterite with increasing temperature.

Conversion of melanterite to rozenite, or rozenite to melanterite was usually accomplished within an hour, although in a few cases three to

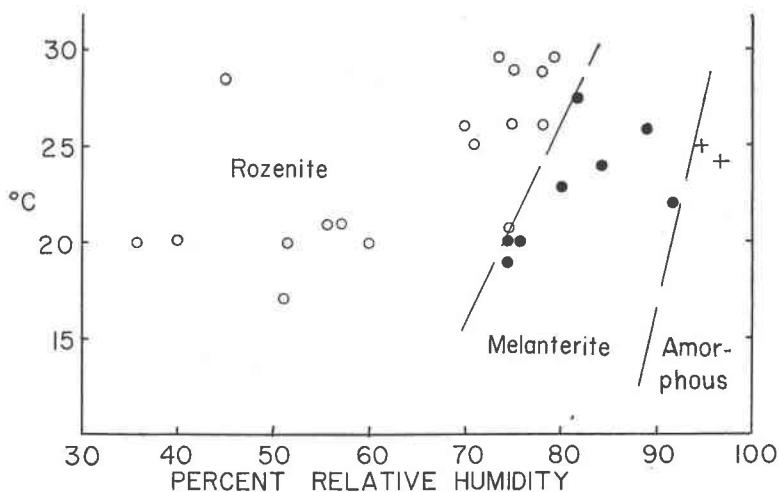


FIG. 1. Stability fields of melanterite and rozenite at room temperatures as a function of relative humidity. The phases indicated have in all cases formed from conversion of other phases of different hydration states. The open circles indicate rozenite, filled circles melanterite, and crosses amorphous material.

four hours were required; this did not seem to be a function of the state of the particular sample, as duplicate runs were often performed on the same sample. Above approximately 95% relative humidity, no diffraction pattern was obtained, as a result of deliquescence of the melanterite.

An attempt was made to determine the equilibrium boundary between rozenite and the mono-hydrate szomolnokite, by running completely dry air over a rozenite sample. A run of approximately eight hours did not cause any detectable conversion.

DISCUSSION

The non appearance of the pentahydrate siderotil in this study agrees with the conclusion of Jambor and Traill (1963) that at least a small amount of copper is necessary for its synthesis. It has been clearly shown by reversible reactions that either melanterite and rozenite are stable at room temperatures as a function of the relative humidity.

The diverse statements of Midgley, and Jambor and Traill on the stability of melanterite and rozenite under "normal" conditions can now be viewed in proper perspective. Rozenite has been demonstrated to be the stable phase for humidity conditions less than 70–80%, whereas melanterite is stable at higher values. Midgley's work was done in Great Britain, which is known for its high relative humidity. Hence under normal British conditions melanterite is probably the stable phase for much

of the year. Jambor and Traill conducted their investigations in Ottawa, Ontario, a mid-continental region of moderate humidity, in which rozenite is probably the stable phase for much of the year. Hence both sets of investigators reached correct conclusions for their particular localities.

The problem is to a great extent caused by the normal reluctance of melanterite to dehydrate under conditions of low humidity. This metastable persistence is probably due to large grain size and lack of appreciable air movement over the sample.

ACKNOWLEDGMENTS

The authors wish to thank the National Institute of Health for financial support of this project under grant WP-00340, as well as Michael Fleischer of the U. S. Geological Survey for information on the proper nomenclature of the minerals examined.

REFERENCES

- BRANT, R. A. AND W. R. FOSTER (1959) Magnesium halotrichite from Vinton County, Ohio. *Ohio Jour. Sci.* **59**, 187-192.
- ECKEL, EDWIN B. (1933) Stability relations of a Colorado pisanite (cuprian melanterite) *Am. Mineral.* **18**, 449-454.
- JAMBOR, J. L. AND R. J. TRAILL (1963) On rozenite and siderotil. *Canad. Mineral.* **7**, 751-763.
- MIDGLEY, H. G. (1962) On the occurrence of siderotil in Thames River gravel. *Am. Mineral.* **47**, 404-409.

THE AMERICAN MINERALOGIST, VOL. 50, SEPTEMBER, 1965

SULFATE MINERALS: THEIR ORIGIN IN THE CENTRAL KENTUCKY KARST¹

E. R. POHL, *Cave Research Foundation, Yellow Springs, Ohio*

AND

WILLIAM B. WHITE, *Materials Research Laboratory and
Department of Geochemistry and Mineralogy, The
Pennsylvania State University, University
Park, Pennsylvania.*

Sulfate minerals, especially gypsum, are present in large quantities in most of the extensive limestone caves of the Mississippian plateaus and outliers of Central Kentucky. Many descriptions of these deposits have been published (Locke, 1842; Weller, 1927; Huff, 1940); however, the origin of the deposits has remained obscure.

¹ Contribution No. 8 of the Cave Research Foundation and Contribution No. 63-78 of the College of Mineral Industries, The Pennsylvania State University.