

TECHNIQUE IN MINERALOGRAPHY AT HARVARD

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INTRODUCTION

Charles Palache's impress on the science of mineralogy, as other contributors to this Special Number doubtless will have emphasized, has come not alone because he is a lover of minerals and a master of their secrets; he has made mineralogy a living subject to the many his influence has reached, and this is largely because of his own keen interest in the *geological* aspects of mineral occurrence. In years of roaming through Western mining districts before I knew Palache, in repeated instances it was possible to recognize young mining engineers as Palache-trained because there would early emerge in one fashion or another their genuine feeling for minerals.

When the writer came to Harvard in 1909 to assist with the instruction in mining geology and began to pursue the microscopical study of ores in polished section by methods just then coming into use, Palache

from the very first showed the most generous and sympathetic interest in this means of strengthening the mineralogy of the opaque minerals. And it is in large measure due to his unfailing encouragement, his wise counsel and the aid he has given in countless ways that a reasonably systematic attack has been maintained in the Laboratory of Mining Geology toward improved identification and a better understanding of the sulphides and related minerals.

It seems appropriate, therefore, that this Special Number intended to record examples of Palache's varied influences should contain a summary of the techniques and methods that have been developed with the aid of men who were his students and in an atmosphere pervaded by his inspiration. At the same time, opportunity is here afforded to respond to many requests that have come for description or discussion of mineralographic technique as employed at Harvard. In the following pages, attention is restricted to instrumental and manipulative procedure with the opaque minerals; no consideration is given to those operations chiefly dependent on transmitted light, nor to those more interpretative and philosophical phases of mineralography, such as textures, sequence, inversion, exsolution, genesis, deformation, alteration, etc., which more or less directly depend on the determinative steps here discussed.

PREPARATION OF THE SPECIMEN

Polishing

In the paper by the late William Campbell (1906), which virtually ushered in the specialized microscopical study of opaque minerals by methods developed in metallography, relatively little emphasis was placed on the quality of polish to be given the materials for study. It became evident, however, that polishing methods suitable for such homogeneous and coherent substances as metals gave far less satisfactory results when applied to the varied mineral aggregations found in natural ores. It was finally concluded in this Laboratory that avoidance of the difficulties encountered, of which residual pits and scratches on the hard minerals and excessive relief between hard and soft minerals were dominant, could not be hoped for except by much lower pressures and slower speeds between specimen and lap, especially in early stages. Trial supported this idea, but indicated that the time required was thereby so greatly increased as to make hand-polishing impossible economically.

By 1920, therefore, a mechanical polisher had been designed and built to rotate ten appropriately mounted specimens under adjustable spring pressure against a counter-revolving lap, of cast iron for the grinding stage, and fabric-covered for the polishing stage. After several years of

tantalizing encouragements that never reached satisfaction, during which N. Sabsay resourcefully tested one modification after another, it was decided to start anew.

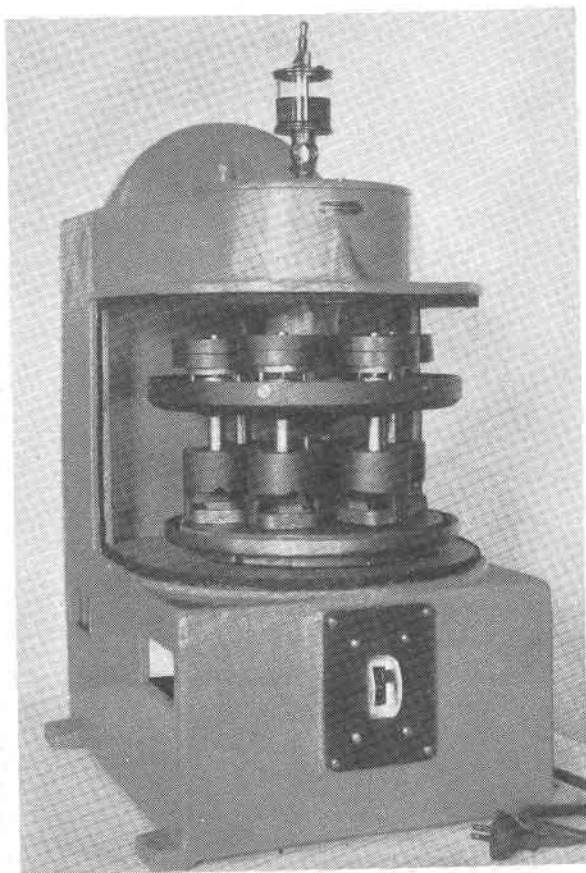


FIG. 1. Latest Model of Polishing Machine shown with transparent dust-proof doors removed.
(Courtesy of Mico Instrument Co.)

After an exhaustive review by J. W. Vanderwilt¹ of the literature on polishing and buffing in all sorts of industries, followed by trials of many methods and materials, it was decided that there could be no mastery of relief so long as fabric-covered laps should be used; and eventually the experimental work pointed definitely to laps of essentially non-yielding metal (soft lead) even for the final stages of polish. The

¹ Now Chief Geol., Climax Molybdenum Co., Colorado.

resulting machine and method described by Vanderwilt (1928) have since been substantially perfected, the machine more than the method. In these improvements, as well as in many other of our instrumental problems, we have had the effective cooperation of D. W. Mann, instrument maker for the Department of Physics at Harvard, and the Mann Instrument Company² of Cambridge. The fifth and latest model of the polishing machine is shown in Fig. 1.

The operation of polishing mechanically on metal laps is an art, not a fool-proof process. Its present state results from investigations by Drs. A. B. Yates,³ H. J. Fraser,⁴ and E. B. Dane, Jr.,⁵ and from the accumulated experience and steady interest of C. S. Fletcher.⁶ Adequate description of the process would exceed the space appropriate here. Suffice it to say that this method, while not yet perfect, produces a quality of polished surface that far excels anything yet produced on heterogeneous ores by hand polishing. None of us familiar with both types of polishing would now willingly work with fabric-polished preparations. The difference is not so much one of mere "shininess"; far more important is the greatly improved seeing permitted by the absence of the dark bands due to relief. It has been found that these previously-obscured contacts between hard and soft minerals are favored places for minute components of prime significance. For example, it is now possible to see plainly, as to color and shape, grains of gold embedded in pyrite even though these be so tiny that 3×10^{10} such particles would be required to make one cent's worth. Since gold grains of much larger size would look only like black pits if the polishing were done on fabric-covered laps, it is not surprising that many have been disappointed in studying gold ores of moderate tenor under the microscope or have proposed weird explanations of the manner of occurrence of the gold shown by assay. Production of polished surfaces that are virtually optically plane, by reduction of maximum relief to about one half of a wavelength, has allowed the attainment of a precision and reliability in several of the procedures described hereafter that would have been utterly impossible on surfaces polished by the old methods. This is especially true for the determination of hardness, reflectivity, and color as well as for microphotography.

Our efforts to produce sections for simultaneous examination by both transmitted and reflected light have not been attended with the success

² Now Mico Instrument Company.

³ Now Chief Geol., International Nickel Co., Ltd., Ontario.

⁴ Now Ass't Prof. of Mineralogy and Mineragraphy, Calif. Inst. Tech.

⁵ Research Assoc. in Geol., Harvard.

⁶ Preparator, this Laboratory.

which descriptions by others had led us to expect. J. H. Moses,⁷ who handled these trials, resorted to innumerable devices to put a polish on a thin section or to thin a section already polished; but, despite use of a wide range of cementing media, he was unable to subdue with consistent regularity the tendency of the polished slice to peel away from the glass mounting slide. We await better instruction on this important accomplishment.

Abrasives

It need only be mentioned at this time that the use of metal laps for final polishing requires abrasives far finer and more accurately sized than will suffice for polishing on fabric laps. Special means must therefore be employed to secure the suitable powders. The procedure outlined by Vanderwilt (1928) is substantially the same as now followed. The metal laps are vastly more economical of abrasive than is the older method; therefore, all the surroundings are cleaner and danger of injuring the specimen through accidental contamination by coarser particles is minimized.

Since the fundamental principle of our polishing method is gentlest possible treatment of the specimen from the very beginning, the preliminary flat surface cannot be secured, as previously, by grinding down a lump of the ore against a coarse wheel. Instead, the specimen is obtained by cutting a slice from the hand specimen by a special diamond saw, procured from the Mico Instrument Co.; this yields an almost ground-glass-smooth surface which is entirely acceptable for the succeeding stages of fine-grinding and polishing. For the examination of some given portion lying near the center of a large specimen, the part in question is removed by a special diamond bit, having a thin wall; it is rotated against the specimen by being clamped in an ordinary drill press; from the short section of resulting core a slice is sawn for mounting.

Mounting

Polishing by machine virtually requires that the specimens be of uniform shape and size. For use with the first machine we secured from the American Brass Company a specially drawn rectangular brass tubing $1\frac{1}{2} \times 1\frac{1}{8}$ " O.D., from which were cut sections $\frac{7}{8}$ " long. The specimen was cemented in one of these boxes by means of a special sealing wax, and backed with plaster of Paris. After several thousand specimens had been so mounted, we found that every kind of wax we tried eventually warped or shrank or otherwise became useless.

In the search for a substitute, molded bakelite suggested itself; and

⁷ Now Geologist, Cerro de Pasco Copper Corp., Peru.

after a trial specimen mounted in bakelite and polished was found at the end of three years to be in as satisfactory condition as at the beginning, we abandoned the mounting in brass tubes in 1927, disposing of our large unused supply of tubes to those who had adopted that scheme. Since that time, all our specimens have been mounted in rectangular blocks of molded bakelite of the same dimensions as before. See Fig. 2. The underside is recessed centrally to prevent rocking on the microscope stage.

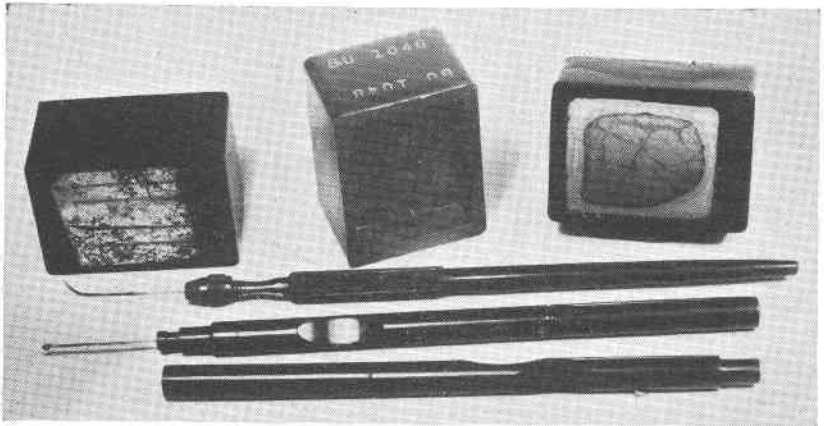


FIG. 2. A. The bakelite mounts, method of numbering, and, at right, use of Wood's metal inset for mounting heat-sensitive specimen.

- B. Standard scratching needle in holder.
- C. Platinum loop; holder in open position.
- D. Platinum loop; holder in closed position.

Note reflection of platinum wire and of needle, also of their shadows, in mirror surface of left-hand specimen.

This method of mounting, suitable not only for solid specimens but also for single crystals, grains, powders and concentrates as fine as 400 mesh, has now been rather widely adopted. We strongly favor mounts of rectangular section instead of the circular section which some have been led to adopt on account of lower initial cost of the molding die. Besides the fact that, when accidentally dropped, the rectangular mount will not roll away, collar-button fashion, there are the great advantages that the area of surface is greater, that the numbering can be easily impressed on the side of the mount where it is in view during study, and also that any given feature on the specimen can be precisely and instantly located by the mechanical stage or other simple coordinate system.

By an appropriate holder on the stage it is possible to remove a speci-

men for washing or for some special test, then return it so that the exact spot initially centered is restored automatically to that same position. Since all our microscopes, including the microphotographic equipment, are calibrated to the same coordinates, it follows that any given feature, such as a certain tiny grain, can be recorded as to coordinates and specimen number, and then can be instantly found by any other worker at any later time on his own instrument. This is an enormous saver of time, above all in microphotography at high powers.

Each specimen mount is clearly and permanently numbered on one end by impressing the district symbol and the appropriate number (e.g., BIS 1361-H; BVD 14869—Bisbee and Bushveld, respectively) into the bakelite by means of a hardened steel numbering machine set in a small press. The number is impressed twice on each specimen so that by no chance shall there be obliteration. The old scheme of painting the numbers on was ever an abomination.

Prior to mounting, the specimen is given impregnation by bakelite resinoid baked for 4 hours at 90°C.; this is especially necessary for porous or friable materials.

With a pressure of about 5000 lbs. per square inch we mount our specimens at a molding temperature of about 125°C. With a very few exceptions, involving some pyrrhotite ores and certain specimens containing the richest silver minerals, we have detected no sign of decomposition or modification by the mounting operation, such as described by Bateman (1929). We have tried a considerable number of other plastics, certain of which mold at somewhat lower temperatures than bakelite, also a variety of cements that do not require any heat; but thus far we have found nothing so generally satisfactory as bakelite. When dealing with specimens which we think the molding temperature might affect, we "solder" the specimen in a *hollow* bakelite mount (which we mold specially for that purpose) by running in molten Wood's metal (M.P. 70°C.) under pressure, then letting it set. This works satisfactorily.

Our bakelite-mounted specimens now number over 6000, including a considerable number remounted from the old brass tubes. An occasional molding (4 at a time) goes wrong; but the great majority, including specimens over 9 years old, are entirely stable and in every way satisfactory.

MICROSAMPLING

The experience of this Laboratory has led with increasing emphasis to the conclusion that determinative procedure must be suited to dealing with the smallest possible grains. A great expanse of homogeneous ma-

terial more often than not involves little obstacle to identification. There is room to allow all the necessary tests to be readily applied, and repeated when necessary; interference by other minerals as to chemistry and color is minimized; all conditions are at their best. But determinative difficulties multiply as the grains grow smaller. Moreover, naturally enough, the less common minerals and the rarer elements are most likely to occur in tiny grains. It is these in particular that need identification, not only for the mere satisfaction of completeness but also for the significant genetic clues that such constituents so commonly afford. All our technique has therefore been designed to go as far as possible, first in disclosing the tiny grains by the best attainable polishing, and then in mastering the identification of these tiny constituents.

For microchemical, spectrographic and x -ray determinations, it is essential to remove from the polished surface an uncontaminated sample of the mineral in question. For this purpose the microdrill described by Haycock (1931) was further perfected at the hands of G. A. Harcourt by improvement of bearings and guide so that it turns at high speed with beautiful steadiness, by an arrangement whereby it may at will be swung either to a position where the point of the drill is exactly in the optical axis of the microscope or to a position entirely out of the way, and in connection with a microscope having a mechanical stage that can be raised and lowered. Dr. Harcourt's description of the drill appears on page 517 of this Special Number.

The drilling can now be held within the limits of a grain having a surface exposure as little as 0.02 mm. in diameter. Thus may be secured a sample for spectrographic determination without any danger whatever of contamination of this sample from adjoining grains, unless the contamination is accomplished by drilling *through* the grain into another mineral that lies below the polished surface. In case the texture of the ore suggests that there may be danger of this latter outcome, or in case the amount safely obtainable from any single grain is insufficient, an adequate sample that is probably pure can be usually secured by drilling tiny amounts from a number of different grains of the same mineral, taking care to drill only very shallow depressions in any one grain; the aggregate of the drillings from the several grains constitutes an adequate sample.

Reference should here be made to the aids for quantitative estimation of the components of an ore in the form of special micrometer eyepiece grids reading directly in percentages which we had made in connection with the quantitative microscopical study by Dr. E. Thomson (1930).⁸

⁸ Now Assoc. Prof. of Mineralogy, Univ. of Toronto.

MINERAL IDENTIFICATION

General Treatises

The first study at Harvard of polished sections of ores was made in 1909–10 by the late C. T. Brodrick⁹ and the writer. We found all too frequently grains of material that we were unable to identify either by our own efforts or from what was then available in the literature. This was evidently a universal difficulty at that time, so that it was either necessary to make a guess at mineral identification or to designate many species as “unknown.”

Therefore, it was decided to undertake a systematic determination of the opaque minerals. The major execution of this task was undertaken in 1911 by Joseph Murdoch,¹⁰ who brought to it a noteworthy devotion and resourcefulness. At the time Murdoch's book “Microscopical Investigation of the Opaque Minerals” (1916) went to press, the most important existing paper on opaque mineral identification gave partial tests for 37 varieties. Murdoch's treatise included systematized identification data for 186 definite varieties besides disposing of many other names that had been applied to materials which his study showed to be identical with better established varieties or else obvious mixtures. The new science was christened *mineralography*, since it bears the same kind of relation to the broader field of mineralogy that petrography bears to petrology and metallography to metallurgy. This was the first systematic and comprehensive work on opaque mineral identification. The diagnostic criteria, the arrangement of data and the general conception of the tabular presentation have set the style for nearly all of the several treatises that have since appeared.

One of Murdoch's important accomplishments had been the assembling of a suite of all the well-authenticated varieties of opaque minerals which he had been able to secure during five years of quest. This became known as the standard Murdoch Suite; with its progressive refinement, and its gradual enrichment chiefly from the great mineral collection administered by Palache, it has remained the indispensable basis of all our determinative work of the last twenty years.

The next important improvement of opaque mineral identification was started by C. M. Farnham,¹¹ who after several years in the mines had returned for graduate study. Farnham recognized some of the shortcomings of Murdoch's work and aimed to correct these and also to increase the number of diagnostic tests. With the Murdoch Suite as a

⁹ Later Chief Geol., Russo-Asiatic Corpn., Urals and Siberia.

¹⁰ Now Ass't Prof. of Geol., Univ. of Calif. at Los Angeles.

¹¹ Now Consulting Geol., Barre Plains, Massachusetts.

basis, he was able to advance toward these objectives. As W. M. Davy at the Massachusetts Institute of Technology was then engaged in a similar study, these two wisely decided to pool their results, which in due time were published (Davy and Farnham—1920).

E. E. Fairbanks,¹² after some study as a special student at the Massachusetts Institute of Technology, came to our Laboratory to work with the Murdoch Suite and familiarize himself with the technique then employed. Shortly afterward he brought out as editor (Fairbanks—1928) an interesting book on laboratory procedure to which chapters were contributed by various specialists, whose willingness to do this resulted from his own enthusiasm.

After several years as Instructor in this Laboratory, where he contributed to every phase of our mineralographic technique as then in use, M. N. Short¹³ went to the U. S. Geological Survey, where he had unusual opportunity to assemble a second standard suite of minerals analogous to the Murdoch Suite. Interchange of specimens between the two suites, which were studied at both ends of the line, helped more than anything previously had done to discover errors of identification. Short became a recognized authority in this field, bringing out (1931) what we at Harvard regard as the most reliable of the treatises that have thus far appeared on identification of the opaque minerals. As Short had been recalled to Harvard as lecturer for 1927–28, when the technique of this Laboratory had been further advanced, it will suffice to refer to details in his treatise for several of the topics considered later. Dr. Short is now engaged in a thorough-going revision of his book.

Farnham (1931) published a revision of the earlier work by Davy and himself, incorporating a number of improvements and corrections. This later work, though done elsewhere, is looked on at Harvard with the interest that comes from early association.

Physical Properties

Hardness. While the old method of polish by cloth laps was in use, much could be determined as to relative hardness of the minerals by observing their relief against some known species present in the section, such as quartz, pyrite or sphalerite. But since the relief was a consequence both of the actual hardness-difference between adjacent minerals and of the method and duration of polishing that had been applied to the particular specimen, hardness distinctions made in this way were only very roughly quantitative. With the perfecting of polish so that

¹² Now Representative, Science Service, New York City.

¹³ Now Prof. of Petrography, Univ. of Ariz.

now the surface is almost optically flat regardless of the varying hardnesses of the minerals involved, that former rough and ready test of relative hardness is practically eliminated.

We employ two methods for ascertaining hardness. The approximate method uses a bent needle mounted in a holder of standard length and weight so that the observer becomes accustomed to a standard leverage as he attempts to scratch the polished surface and thus to a standard range of pressures which he applies. Sharps' No. 8 needles are used; and whenever a needle shows under the microscope the first signs of wear it is discarded for a new one. About three millimeters from the point, the tip portion is bent about 50° out of line. This bending is easily accomplished in a wholesale way by gripping the very tips of an entire packet (25) of needles in a heavy steel vise which prevents the temper being drawn from the points when a narrow, intense blow-pipe flame is directed along the row of needles just above the vise until they redden and can be simultaneously bent over to the proper angle. This bending of the point enables the needle to be brought readily between the specimen and the objective and yet allows the actual scratching point to be drawn across the mineral at the fairly steep optimum angle for producing a scratch with least pressure. The standard holder adopted is a broach holder such as used by dentists. These can be purchased at insignificant cost from S. H. Reynolds Sons Company, Boston. The hard-rubber handle is ribbed to prevent turning while held gently in the fingers, and the neat little steel compression chuck grips the needle firmly, yet allows instantaneous replacement. See Fig. 2.

The precision method of determining hardness is accomplished by an instrument built for the writer in 1914 in the shops of the Geophysical Laboratory in Washington. When first used, a sharp needle was employed as the scratching point. S. B. Talmage¹⁴ later experimented with the instrument and after various substitutes for steel had been tried, including agate and sapphire, we finally decided to standardize on the diamond. Dr. Talmage (1925-B) described the instrument and published a table of hardnesses as measured by its means. Since that time, with the cooperation of Dr. E. B. Dane, Jr., the precision of the instrument has been materially increased in a variety of ways, including the substitution of sapphire cone bearings for the previous hardened steel cups; the mechanical suspension of the balance beam and the micrometer adjustment for balancing it at zero are now essentially perfect. The precision of the instrument therefore depends upon the constancy of the diamond point, that is, upon its retention of sharpness and on its dupli-

¹⁴ Now Prof. of Geol., New Mexico School of Mines.

cability if replacement is necessary. Our diamond point (or blade, really) was made by Arthur A. Crafts & Co., of Boston. After several years of use no blunting by actual wear has been detected. Before certain guards were introduced, however, an occasional accident caused a chipping of the sharp edge; this was immediately disclosed on the first ensuing trial with the instrument, whereupon the diamond point was sent back to the makers for resharpening. When they first made the point, they took a highly enlarged photographic profile of its edge; when resharpening is necessary, they merely duplicate this profile. Each time the point has been resharpened it has exactly duplicated the readings it previously gave. Some years ago while our equipment was being moved to present quarters, the diamond point was so badly damaged that it had to be discarded and a new one made. We were fearful lest the entirely new piece of diamond used might have actually different hardness characteristics from the one we had first employed, since, although the mineralogist looks upon the diamond as of constant hardness, the diamond driller knows that different diamonds vary greatly in resistance to wear as well as in brittleness. When the new point was tried out on a considerable number of the minerals whose hardness had been established by the old point, we found the hardness readings the same as before.

It therefore appears that here is an instrument which is duplicable within the necessary limits of accuracy. So far as we are aware nothing of corresponding precision is in use elsewhere, except in Mass. Institute of Technology. This seems unfortunate, since hardness is an essential and dependable characteristic of a mineral and this instrument will test hardness with beautiful precision; moreover, it would be regrettable to have differing arbitrary data on hardness arise from the use of instruments that have not been designed to give standardized results. Advantages possessed by this instrument over the microcharacter of Bierbaum (1923) were listed by Talmage (1925 B, pages 541-42). Further experience adds the following advantages: (1) The dulling of the diamond point is detected more readily and, therefore, earlier; (2) dependable tests can be made on much smaller grains, and there is no danger of the scratch reading through to an unconcealed different mineral that lies below; (3) recognition of the thin, clean "standard" scratch is more sensitive and reliable than the actual measurement of width of a deeper scratch—this is especially true on brittle minerals, N.B. the microcharacter was designed to test soft-bearing metals which tend to give smooth, ductile scratches whereas the ragged and variable scratches likely to be produced on minerals are pictured by Hodge and McKay (1934) together with the highly variable measurements of width that inevitably result.

Inasmuch as the hardness is determined by the production of a faint

but definite "standard scratch" on the surface of the mineral, it is essential that the area to be tested be entirely smooth with respect not only to absence of other scratches but also freedom from pits and undulations. The relief-free mirror polish on even the hardest minerals by the lead-lap method affords an ideal surface for the determination of hardness by this instrument. Its precision and control are such that four parallel but perfectly distinct scratches can be made, at successively different settings of the balance weight, on a mineral grain not more than one fiftieth of a millimeter in diameter. As four successive tries ordinarily suffice to arrive at a scratch of the intensity defined as "standard," it follows that the hardness of grains even down to this minute size can ordinarily be ascertained reliably. By drawing a scratch across adjacent minerals their relative hardness is beautifully disclosed even though no relief whatever from the polishing is to be seen at their boundary.

Cleavage. D. H. McLaughlin¹⁵ (1917) noted that certain relatively soft minerals, such as bornite, which do not ordinarily exhibit cleavage either in the hand specimen or in polished section, can be made to disclose this property, in a way analogous to production of the "percussion figure" of mica, by pressing the point of the scratching needle vigorously into the polished surface. Orientation of grains may thus be determined even if the mineral is optically isotropic.

Specific Gravity. G. V. Douglas,¹⁶ A. W. Waldo,¹⁷ H. Berman¹⁸ and G. A. Harcourt¹⁹ have in turn sought to develop a reliable method for determining specific gravity of a minute particle of a given uncontaminated mineral grain removed from the polished surface. None of their ingenious schemes, however, has yet overcome the difficulty occasioned by surface tension of the immersion liquid, which becomes aggravated in inverse proportion to the weight of the particle to be tested. Since each tested grain weighs a small fraction of a milligram, the disturbing effect of surface tension has thus far prevented the attainment of reliable results. The subject deserves further effort.

Magnetism. This useful determinative test encounters serious limitation when applied to polished sections in which the magnetic grains are small and scattered. A first trial involved bringing a magnetized needle in the field of the microscope in a balanced position and passing it closely above the polished surface. If one end of the needle came near enough to a magnetic grain, the needle would be drawn or repelled and balance

¹⁵ Prof. of Mining Geol., Harvard.

¹⁶ Now Prof. of Geol., Dalhousie Univ.

¹⁷ Now Research Asst. in Mineralogy, Penn. State Coll.

¹⁸ Research Assoc. in Mineralogy, Harvard.

¹⁹ Instructor in Mining Geol., Harvard.

upset. But this was an extremely precarious scheme. H. E. McKinstry²⁰ devised a very satisfactory substitute. After a needle has been magnetized it is cut in two right through the eye. The smaller part, a tiny curved piece of steel, is neatly cemented at its middle to the end of a 3-inch length of horsehair, and by fixing the opposite end of the horsehair appropriately, one has in effect a miniature "horseshoe magnet" supported by a "spring" just above the surface of the polished section and in the axis of the microscope. By then drawing the specimen systematically under the magnet by means of a mechanical motion of the stage, each grain suspected of being magnetic can be tested; if the grain proves to be magnetic, the observer at the microscope sees the little magnet drop sharply against the grain and hold there.

W. D. de Laguna²¹ has recently essayed some tests whereby minerals of lower magnetic permeability than magnetite and pyrrhotite might be made to disclose the magnetic property by examining them when in the influence of a strong magnetic field. Not enough progress has yet been made with such tests to insure their success, but in any case their promise has not yet been killed off. Some of the newer alloys of extremely high magnetic permeability can be had in the form of relatively fine powder and when such powder is dusted over the inclined surface of a polished section, particles of it tend to adhere to such grains in the specimen as are magnetic. There is still possibility of developing something useful in this direction, but thus far practical difficulties have been encountered through insufficient fineness of the magnetized powder and too brief retention of its full magnetic power so that fresh supplies must frequently be secured.

Electrical Conductivity. C. M. Farnham sought to measure the electrical conductivity of sulphides by bringing into contact with a given grain on the polished surface two needle electrodes in a circuit that included a voltmeter. Promising results were secured. Three lists of minerals, those which were virtually perfect conductors, those which were approximate non-conductors and those which stood in an intermediate position, were included by Farnham in a report submitted in connection with his studies for the Master's degree. This method was carried further and the lists extended in the book by Davy & Farnham (1920). Later workers in our Laboratory, however, found great difficulty in securing consistent measurements on minerals of the intermediate list.

The late R. D. Harvey²² in time undertook to apply the conductivity test under rigid control. Dr. Harvey's paper (1928) records the variety

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²¹ Graduate student, Harvard.

²² Later, Petrographer, Cerro de Pasco Copper Corpn., Peru.

of efforts that he made toward standardization and the results he secured. His apparatus involves terminals of standard size and shape applied to the specimen at a standard distance apart and at a standard pressure; for it was found that if any of these factors were varied, unreliable results were inescapable. The use of his apparatus and methods gives the best results that we have been able to secure; but the chance of erratic readings is still so high that we have virtually abandoned this approach toward mineral identification.

X-Ray Analysis. Through cooperative arrangements, first with the Department of Metallurgy and later with the Department of Mineralogy, it has been possible to study the atomic structure of the opaque minerals by means of *x*-ray diffraction photographs. A. W. Waldo (1935) was the first in our Laboratory to attack this subject systematically. He studied the copper-bearing minerals and in his publication of the resulting data he has performed a distinct service in devising a scheme of indexing whereby the diffraction lines furnished by any material can instantly be compared with those known minerals which give closely similar patterns; thus identification is greatly facilitated.

Application of the *x*-ray method to the whole range of ore minerals is now being carried forward by G. A. Harcourt with improved tubes and cassettes, and under conditions that lead to more than mere determination of spacing of the atomic planes and empirical diffraction patterns. He has so perfected the technique that satisfactory powder photographs can be secured from as little as 0.02 mg. of material. By means of the microdrill, pure material in adequate amount for identification can thus be secured of a mineral even if a member of an extremely fine intergrowth. The details of this work are described by Dr. Harcourt in this Special Number.

Optical Methods

Color. On a well polished specimen, color is the first quality of the mineral that impresses the observer at the microscope. Many of the colors are so distinctive as to afford instant identification of the mineral, but since the vast majority of the opaque minerals fall in the "grays" and "whites," the color differences are too small to be identified by reliance on one's color memory. Moreover, the "color interference" caused by adjacent grains of different color is surprisingly serious. Therefore, if color is to be used as a diagnostic property for more than a relatively small number of the more intensely colored minerals, some rigorous scheme of color measurement or color comparison is necessary.

Murdoch met this situation by so shaping his specimens that there could be brought in juxtaposition under the field of the microscope the

mineral whose color was to be determined and a known mineral of approximately similar color used as the standard of comparison. By this means, combined with his unusually accurate color sense, Murdoch was able, I am convinced, to make useful color distinctions which succeeding workers have failed to duplicate, partly because they have been unwilling to spend the patient care he used. For a time, therefore, color as a diagnostic character fell into disuse and even disrepute; this is certainly regrettable.

With the cooperation of A. W. Pinger²³ in 1921, color was again attacked by the comparison method, using two microscopes connected by a comparison eyepiece instead of the more laborious and less satisfactory method used by Murdoch. S. B. Talmage (1925-A) inherited this equipment and carried forward a range of experiments which finally resulted in the production for us by the Eastman Kodak Co. of three colored wedges and a neutral wedge, similar to those used in their standard optical colorimeter. The principle is that by appropriate position of the three standard color wedges in the beam that enters the vertical illuminator it is possible to induce upon a standard (colorless) reflector a synthetic color which exactly matches the hue of the color reflected by the mineral being tested, and by appropriate placing of the neutral wedge the intensity of this synthetic hue is likewise matched with the unknown mineral. By recording the position (in distance from the thin or zero end) of each of the four wedges as they stand when this match is secured, one attains a four-number quantitative specification of the color of the mineral tested. The scheme is correct in principle but has drawbacks in practice, partly because the considerable thickness of glass interposed in the form of wedges "muddies" the color, and partly because, with four independent variables, a good deal of fitting and trying has to be done before perfect match is secured; the task is, therefore, extravagant of time. For the present we have returned to the comparison of the unknown against a succession of known specimens until approximate match is attained.

Dr. Dane and the writer are now engaged in the design of a new color comparator which we hope will incorporate the advantages of the wedge colorimeter method without its disadvantages. If these hopes shall be realized, it should be possible to use color as one of the most simple and reliable steps in mineral identification.

Internal Reflection. The color of a mineral, as shown by such light as happens to be reflected through it from fractures and from the boundaries of other minerals, deserves better use than that which can be

²³ Now Chief Geologist, New Jersey Zinc Co., Franklin Furnace.

made by light that happens to fall obliquely on the specimen. The annular lamp known as the Silverman Illuminator was, therefore, adopted by us in 1924 and so arranged that by the throwing of a switch the specimen could be examined at will by vertically incident light or by the oblique beam from the Silverman Illuminator; this gave, respectively, the color reflected from the surface and the color, if any, reflected from within the grain. It was later found that the internal reflection shows up as well or better when an intense beam of light is passed through a nicol prism and thence to the specimen by a vertical illuminator. Best of all is the type of oblique illuminator and attendant special objectives marketed by Leitz under the term "Ultrapaque" and by Zeiss and others under the name "Epi-Condenser Illuminator." By this means internal reflection even of grains so tiny as to require oil immersion objectives can be studied satisfactorily. Our microphotographic equipment includes the ultrapaque scheme of illumination.

Reflectivity. Attempts by the writer in 1927 to measure the intensity of reflection from the polished surfaces by means of a cuprous-oxide cell were quite without success. By 1934 photoelectric equipment had been so greatly improved and reflectivity measurements by others seemed so promising that it appeared advisable to renew our efforts, especially as the quality of polish then available to us was not only far better but also much more uniform than before. J. H. Moses undertook this task. He assembled a combination of equipment somewhat different from that used by preceding workers, the central item being a high-sensitivity "Photox" cell made by Westinghouse. He worked with the Murdoch Suite, which by that time had been rigidly checked spectrographically by Dr. Harcourt. Since the investigation was primarily for the purpose of simplifying and confirming mineral identification *in polished section*, in which the crystal orientation is usually not known, Moses concluded that it was useless to employ plane polarized light. Moreover, his experimental work showed that although reflectivity does vary with orientation, in only a relatively small proportion of the total number of opaque varieties does orientation affect the reflectivity by more than 1%, which was the net limit of error accepted by Moses as about the best that can be attained under present conditions. He found that different specimens of the same mineral, when equally well polished and when shown spectrographically to be similar, will ordinarily give reflectivity measurements that check to within about 1%; but if the composition is variable, either because of isomorphism or through sub-microscopically disperse "impurities," greater variations are to be expected. He showed, too, as we had suspected, that quality of polish has a pronounced effect on reflectivity; for example, on various samples of pure copper

well polished, readings checked at 32.4 (arbitrary galvanometer units); but the same copper with a polish described by our standards as "fair" gave a reading of 31.1; and a "poor" polish, 29.8. It is partly on this account that the results secured by Moses show numerous disagreements with data of other investigators whose method of polish, even though high "burnish" is secured, does not give the optically plane surface like that yielded by metal laps.

Moses (1936) tabulated the opaque minerals in order of their reflectivity. Over a total range of 50 galvanometer units (which could be read usually to tenths), he found that the minerals are pretty well scattered; also that most minerals which come close together in his reflectivity table can be readily distinguished by some simple test or combination of tests, such as hardness, polarization, or etch behavior. Dr. Moses expects soon to publish the description of his equipment and methods and his reflectivity data for the Murdoch Suite.

The color comparator now being designed will, in reality, achieve its object by measuring the reflectivity of the minerals for each of several wave lengths.

It may be concluded that reflectivity is a useful help in mineral identification, but in its present state of application it is neither fool-proof nor all-sufficient. The precautions that have constantly to be observed against disturbing influences indicate that it is not a method to be used by a relative novice like a penny-in-the-slot machine. It is also clearly indicated that if various investigators are to reach approximate agreement on reflectivity data, there must be better standardization on polish than now exists.

Polarization. The interesting phenomena disclosed by opaque minerals in polarized light were long used by us only in an empirical way; that is to say, with one nicol color changes were recognized for a few minerals, and with crossed nicols four classes were established, namely, those in which polarization effects can not be detected and those which show respectively feeble, moderate and strong anisotropism. The desirability became evident of confirming and clarifying the conclusions regarding these phenomena that had been published by others and of proceeding as far as might be possible toward duplicating for the opaque minerals the marvelous results achieved by applications of polarized light in thin-section mineralogy.

E. A. Goranson,²⁴ well trained in physics and in mathematics, undertook this study. As his interesting and important results (1933) are soon to be printed, only the briefest reference to the most vital conclusions

²⁴ Now Geologist, East Malartic Mines, Ltd., P.Q.

will here be made. From a welter of scattered and more or less abstruse and theoretical writings, Goranson has assembled a straightforward and useful formulation of the relations between refractive index, absorption index and reflecting power, of the role of each in opaque-mineral optics, and of the deductions as affecting instrumental procedure and the phenomena of surface color and reflected dichroism. The distinction is explained between true interference colors due to phase difference in thin sections and the polarization colors due to difference in amplitude for reflecting minerals, and the great limitations thereby imposed when dealing with the latter are listed and clarified.

The phenomena that can be utilized for the reflecting minerals and the optimum conditions for observing them are set forth. The very practical conclusions are drawn that deviations from optically plane polish introduce erratic and disturbing anomalies, that all adjustments of the microscope and the illumination must be made with extreme precision, that anisotropic effects are most pronounced and therefore for feebly anisotropic species are most readily detected when the analyzer is slightly (i.e., 1° – 5°) off the 90° position with respect to the polarizer, and that accessories useful in transmitted light, such as gypsum plate, etc., have little or practically no influence on anisotropic effects in reflected light.

Chemical Properties

Etch Tests. Mineralography inherited from metallography the scheme of etching with various reagents, primarily for the purpose of establishing identification, secondarily for the purpose of revealing structure. It had been and still is the common practice in metallography to etch the entire surface of the specimen, since the ordinary metallurgical specimen is uniform throughout. This procedure was followed in our early work with ores; but we found it had two drawbacks; first, the normal behavior of a given mineral to a given reagent might be interfered with when the reagent was in contact also with one or more additional minerals; second, after each etching it was necessary to repolish the entire surface; this was extravagant of time. We discovered that quite satisfactory results could be secured by etching a tiny area of a given grain while in processes of observation under the microscope, and that practice has now become general. The reagent was first applied by a micro-pipette made by drawing a glass tube to a fine capillary. But difficulty was commonly experienced in getting the reagent to "feed" in right amount onto the specimen, and also the specimen was often scratched by the sharp edge of the pipette. We finally came to a scheme which has now become our standard—that of a platinum wire loop mounted in an appropriate handle. See Fig. 2. The handles that we use are turned from hard rubber

and are rather similar to, but smaller than, a fountain pen; like the pen point, the platinum loop is protected when not in use by a removable cover which, when in use, is slipped on the opposite end of the holder to give it extra length. The wire is readily cleaned and, therefore, can be used for one reagent after another without any danger of contamination. The loop picks up invariably almost the same identical quantity of reagent; the area of the etch reaction is, therefore, practically constant. The little lens-like drop of reagent held in the loop constitutes, as a matter of fact, a powerful magnifying lens. The result is that when the loop with its reagent is brought into the field of view of the microscope and is in the act of being lowered to contact with the polished surface, this little liquid lens serves as an auxiliary to the microscope system and just before the polished surface is touched the area immediately underneath the drop springs into focus more highly magnified than before, so that the exact spot where the reagent is to be applied is seen with special clarity.

For many of the minerals, the simple etch reactions with one or more of the reagents now generally accepted as standard are highly useful for purposes of identification, but they are becoming supplemented more and more by microchemical reactions in the narrower and more precise sense.

Microchemical Reactions. Although the writer was one of the early students of Chamot, American leader in microchemistry, Harvard was late in applying these methods to identification of the opaque minerals. After some pioneering efforts by K. K. Landes²⁵ and R. W. Goranson,²⁶ two men worked in our Laboratory who received instruction in this phase of the subject from Lindgren, and they, H. E. McKinstry (1927) and M. N. Short (1931), have helped to carry it to its present highly useful state. We have come to put increasing dependence on the methods and data assembled by Short, whose kindness in supplying us with advance editions of his tables has kept us in touch with the latest data.

Spectrographic Analysis. Through the enthusiasm of G. V. Douglas, a Hilger Quartz Spectrograph was purchased for our Laboratory in 1925, but before systematic efforts at its use for mineral identification had advanced very far he was enticed away to Rio Tinto. H. H. Chen (1927) established spectrographically the presence of molybdenum and vanadium in microscopically homogeneous galena from several Western mining districts where the oxidized ores are relatively enriched in those rare elements.

After a considerable amount of experience with the spectrograph in

²⁵ Now Assoc. Prof. of Geology, University of Kansas.

²⁶ Now Physicist, Geophysical Laboratory, Carnegie Institution of Washington.

connection with geological problems at Queen's University, G. A. Harcourt came to this Laboratory and undertook first the systematic testing of the minerals of the Murdoch Suite and then the general problem of mineral identification by spectrographic methods. Approximate quantitative data are secured by various means, including rotating sectors of both the "wedge" and the "step" types. The sampling and testing processes have been so perfected that an uncontaminated component of an extremely fine-grained intimate intergrowth may now be removed, studied by *x*-ray and then, on the same sample, analyzed spectrographically. This exceedingly valuable technique is described by Dr. Harcourt in this Special Number.

MICROPHOTOGRAPHY

Ever-widening extension of mineralographic study whereby are encountered new and interesting relationships of texture, sequence and alteration, coupled with better polishing, could not fail to increase the desirability of good microphotographs. Growing recognition of the tremendous importance of the super-minute and improved technique for revealing it have imposed new burdens on the photography. The attempt to record important facts by sketches or camera lucida drawings is ordinarily to be discouraged because of likelihood of unintentional or subconscious distortion by either commission or omission.

Our own early experience in microphotography was gained chiefly at the hands of Murdoch who, in turn, was helped greatly by Prof. Albert Sauveur and Dr. H. M. Boylston, then of the Department of Metallurgy, from their rich background of metallographic technique. Procedure was standardized and various improvements incorporated through the skillful work of Short, Yates and Dane, each of whom has carried us successively farther toward satisfactory accomplishment.

After years of partly-satisfactory use of one of the well-known elaborate microphotographic units for opaque specimens and another for thin-section photography, and a growing realization of what seemed to us shortcomings in these as well as all the other models on the market, a new microcamera was projected. The design was improved in many ways through the mechanical ingenuity and sagacity in optics possessed by Dr. Dane. The instrument, built by Mann Instrument Company of Cambridge, was finished at the end of 1933 and has realized most of the high hopes that led to its inception. Although embodying many of the features of the better instruments now available, it differs in numerous important respects. Inasmuch as a complete description will soon appear (Graton and Dane, 1937), only the more outstanding features need here be listed.

The instrument accommodates either transparent or opaque specimens. It is, moreover, so designed that both those kinds of components in an appropriately prepared section may be simultaneously illuminated, each in its special way, studied and photographed; so that the varied relations of both ore and gangue minerals to one another and to each other can be recorded in a single photograph. Ahrens prisms of unusually large cross section afford polarized light for both transmitted and reflected work.

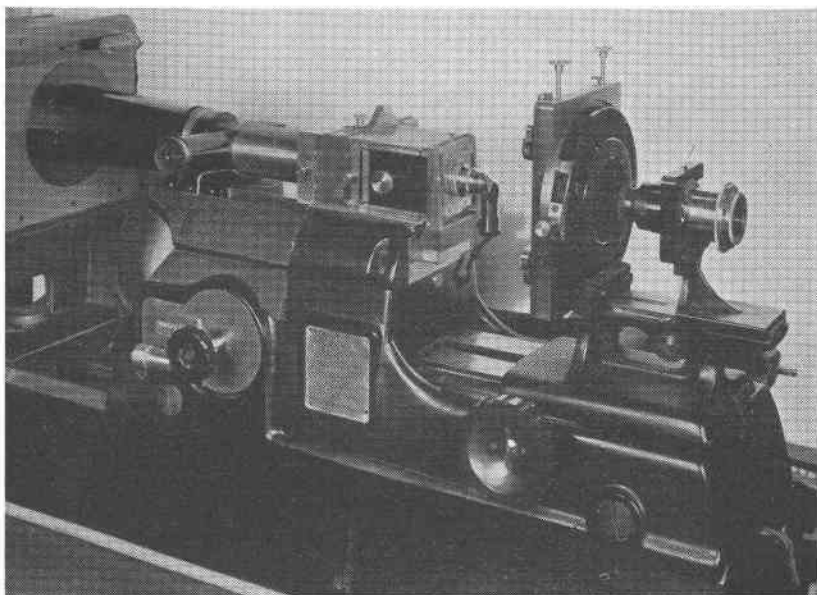


FIG. 3. Microscope Portion of New Precision Microcamera.

Perhaps the greatest departure from conventional design is in the fine-focussing mechanism, where back-lash is eliminated and the focussing attained by a motion of such precision that one division on the focussing scale equals 0.00001 mm., or about $1/54$ of the wave-length of the mercury line by means of which this interval and the accuracy of the entire focussing mechanism have been checked by the interferometer. With so slow a fine-focussing motion, hand-focussing becomes extravagant of time and therefore is replaced by motor-driven focussing. These refinements in focussing are justified for the highest powers by the essentially optically plane polish now available, and are believed to put the precise control of focussing on a level not previously attained. It is

found that when best possible focus is obtained, visibly evident impairment of that focus is produced when the focussing is changed by 3 to 10 scale-divisions (i.e., 0.00003–0.0001 mm. or 300–1000Å). Since no such exacting control over the focussing mechanism has heretofore been possible, it seems inescapable that much photography has been done at something less than the best focus that might have been secured from the optics employed.

Massive construction and best precision-instrument design help to afford accuracy, constancy, absence of vibration, etc. With our highest-powered combination of objective and ocular and maximum bellows draw, direct magnifications of well over 40,000 diameters can be secured. Such fantastic figures are, of course, in that range known as “empty magnification” and represent merely enlargement rather than improved resolution. But the instrument has a useful range up to 4–6000 diameters. Fig. 3 gives an idea of the microscope portion of the instrument.

CONCLUSION

When writing the preface to Murdoch's book 22 years ago, I gave particular emphasis to the inexpensive equipment and the simple technique required for mineralographic investigation. Everyone who has followed the subsequent evolution of microscopy as applied to the study of ores will realize how fallacious that emphasis has proved to be. At long last, geology is becoming in respectable degree a quantitative science; this means a science attacked by instruments, and instruments of ever-increasing precision and scope. This is plainly true in crystallography and in petrography, as it is also in mineral synthesis and in geophysics. It is no less true in mineralography. Indeed, it seems fair to go far toward the conclusion that, just as in physics, chemistry and biology, those branches of geological science that are becoming most *instrumental* are the branches that are advancing most rapidly and soundly.

The task of correct and complete identification of the opaque minerals has proved surprisingly difficult. No longer can accurate results or even results sufficiently accurate for most practical purposes be gained by the simple means of so-called determinative mineralogy; nor by those rough-and-ready steps of polishing and testing which constituted mineralographic technique a quarter-century ago—they are as inadequate and outmoded as an automobile of corresponding vintage. New means of approach, new instrumental appliances, each usually more intricate and exacting than before, have had to be sought, devised and put to use. This trend will undoubtedly continue.

Unfortunately, and contrary to the hope I long held, most of those methods of quantitative precision, refined especially in recent years,

which have contributed so splendidly to improved mineralogy of the transparent varieties have not yet been found of anything like corresponding usefulness when applied to the opaque minerals. For although it is now established that all the "opaque" minerals have some degree of transparency, the magnitude and perhaps even to some extent the nature of this transparency cannot yet be effectively put into harness, as it were, and made to pull out for direct diagnostic use such highly significant and beautifully quantitative data as indices of refraction, birefringence, extinction angle, optical character, optic angle, etc., which now constitute the modern stock-in-trade of the worker with transparent varieties. For the opaque minerals, only a few qualitative or semi-quantitative equivalents have thus far been developed. Toward the filling of these great present gaps, effort in the future must plainly be directed, both through more resourceful instrumental assault and through ever deeper probing into the nature and significance of the crystalline state.

In the meantime, the problem of opaque mineral identification seems to be finding increasingly effective solution through the application of *x*-ray and spectrographic methods, combined with those techniques which insure uncontaminated samples of even the tiniest particles or the most intimate intergrowths (since this obstacle of fine grain and intermixture is especially serious among the opaque species). By the conjunction of these methods in skilled hands it is now possible to secure pure material from a particle not more than 0.02 mm. in diameter. And upon so minute a quantity of material as 0.02–0.002 mg., which is an amount just barely visible to the unaided eye when viewed on a glazed paper in intense light, it is now possible to determine such fundamental facts as the kinds, the approximate proportions and the space arrangement of the component atoms. This is, indeed, a long step toward the ultimate. When *x*-ray, under the extremely exacting conditions imposed by the infinitesimal quantities of sample, can be made to go still further in disclosing full details of the space lattice, and when spectrographic analysis shall have become more precisely quantitative, the task of mineral identification will presumably have been brought to approximate perfection. With the number and enthusiasm of the workers now engaged in this field, it is altogether probable that such a level of attainment is not very far off. Even then, the other properties, such as color, specific gravity, polarization, etc., when mastered, together with those already under control, will still afford useful diagnostic short-cuts. Moreover, unmeasured opportunities lie ahead for further discovery of the interrelations between the physical, optical and chemical properties, and for

thus resolving these now diverse data into a compact and all-inclusive philosophy of the nature of crystals and of matter in general.

Except in a narrow sense, identification of the opaque minerals, to which this paper is so fully devoted, is not an end in itself. Like the determination of the kinds of bacteria, it has a broader and deeper use. There must be recognized the same kind of dangers in turning loose a microscopical specialist (who is nothing more) upon the problem of finding ore in a region of complex geological controls that there would be in turning over the stemming of a serious epidemic to a narrow specialist in microscopical bacteriology. But the time has definitely passed when the mining geologist is safe in relying solely on what some used to single out for worship as "the broad field relations." And the former pastime of looking condescendingly on the microscopical investigation of ores as high-brow and supertheoretical is no longer good sport, but tends immediately to challenge either the progressiveness or the mental integrity of whoever might still be tempted to indulge in it. Medicine has demonstrated not only the value but the absolute necessity of combining clinical and laboratory investigations. The profession of ore hunting will prosper and grow in merit in proportion as it learns to do the same.

The accomplishments summarized in this paper have been contributed by those responsible for them as increments toward that rapidly growing and imposing accumulation by many investigators in all parts of the world. I am confident that I speak for all those who have had a hand in these efforts at Harvard, including my colleague, Prof. Russell Gibson, who has shared in the supervision of these efforts, as well as for those associates who have aided in advancing other aspects of the science of mineralography, in recording our exhilaration at participating in the exploration and development of a new, specialized field. In this we have ever felt the contagious enthusiasm of that true scientist and delightful gentleman to whom this Special Number is dedicated.

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