# *Direct measurement of standard reflectances with the microphotometer.*

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*Summary.* A visual microphotometer depending on three polarizing screens is used to provide the primary scale for measuring the ratio of intensity between the incident and reflected beams for a prepared standard surface, which is intended for subsequent use in the ordinary procedure with the ore-microscope. A reversing mirror on the microscope stage gives reflection at quasi-normal incidence. Direct measurements on a surface of diamond confirm the validity of the Fresnel formula for a polished surface of this substance, and there is general agreement for a series of transparent bodies ranging up to 17 % reflectance. The standard is a glass-covered metallic surface with a reflectance near 50 %. For pyrite $R_D = 54{\cdot}5$ %.

REFLECTANCE for small and irregular grains under the microscope is conveniently measured visually by the microphotometer; the measurement being made by comparison with a standard surface of known reflectance. The standard itself should as far as possible be immune from tarnish or other changes, and its reflectance at normal incidence must be ascertained by an experimental determination in which the intensities of incident and reflected light are directly compared by means of a photometer operating according to a known intensity law (Walsh, p. 190).[1] At present the basic determination can be carried out on one of three adequate types of photometer: the inverse square apparatus so widely used in optical bench methods, the calibrated photo-cell, and the polarization photometer in its recent accurate form with three polarizing filters. The latter is here employed.

*Photometer.* A Cooke, Troughton, and Simms visual microphotometer, of the type designed by the present writer (1953, p. 131), replaces the usual eyepiece in an ore-microscope of which the stage can be lowered to accommodate a special apparatus with reversing mirror. This photometer has two fixed parallel discs of polaroid with high polarizing ratio, between which a third disc with a graduated circle rotates giving an intensity closely in accordance with the law $I \propto \sin^4\theta$ (W. H. Steel, 1951). For the comparison, which is visual, the Lummer-Brodhun cube is abandoned in favour of a small inclined metallized reflecting surface. Comparison depends upon the matching of this area with the adjoining

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[1] For full discussion of this and similar details references have been given to the textbook Photometry by J. W. T. Walsh (2nd edn, 1953).

microscope field, and does not rely upon the disappearance of the boundary, a method liable to inaccuracy (Walsh, p. 202). The photometer lamp transformer was supplied in parallel with the second lamp from a common voltage regulator (230 volts) and the lighting was adjusted so that the middle range of the divided circle (70° to 35°) was in use. All readings were taken on the same side of the zero, but a zero correction (0·6°) was separately determined and applied throughout.

*Monochromatism.* All readings were made in monochromatic light obtained by supporting a colour screen over the eyepiece. The screens were Ilford 'spectrum' gelatine filters, having effective band widths not exceeding 250 Å. This is the widest band suitable for accurate measurements on the dispersion of reflectance in most metals and minerals; since the standard here prepared has nearly linear dispersion this band width will not be a source of error in its determination.

Matching is easiest for a yellow colour, and for this and other obvious reasons the use of the yellow (sodium) screen might well be given priority in general determinative work. When the field intensity is too low to permit the easy focusing of scratches, &c., the facility of matching diminishes, but the average value is still useful. Too bright a field causes retinal fatigue.

*Lamp.* The lamp was an ordinary 150-watt bulb in a cylindrical lamp house with a single condensing lens and ground glass. It provided an illuminated disc 3¼ inches in diameter (surrounded by a black screen) and was supported at a distance of about 2 feet from, and in line with, the inclined spindle of the reversible mirror. Thus the beam was incident on the mirror at the same angle in the direct and reversed positions. This lamp was supplied from the same voltage regulator as the photometer. With the 33-mm. objective this diameter and distance provided an aperture cone about equal to that of the photometer.

### *Reversing mirror for measurement at quasi-normal incidence.*

If a mirror is inclined so as to reflect a uniform beam coming from a distant lamp at right angles to the line of sight, its surface will appear to be uniformly illuminated, an effect known as the 'Maxwell view' (Walsh, p. 152). If it is then turned through 180° about an axis in the direction of the lamp, the light from it can be reflected back upon the surface under test and an image of the mirror will be formed within the surface.

When the inclination of the mirror to the rotation axis is exactly 45° the edge of the mirror will overlap and obscure the image reflected from the test surface at normal incidence; but by altering this angle by a few degrees a sufficient area of the reflected image can be made visible beyond the edge of the mirror. The intensities of the direct image and of that reflected in the surface can then be measured with the photometer, their ratio being the reflectance for quasi-normal incidence.

The mirror is a small polished surface of stainless steel inclined at $48\frac{1}{2}°$ to the spindle. It is useful to leave a few coarse scratches or particles on the surface both for focusing the surface and to identify the same area when the mirror is reversed (fig. 1*c*).

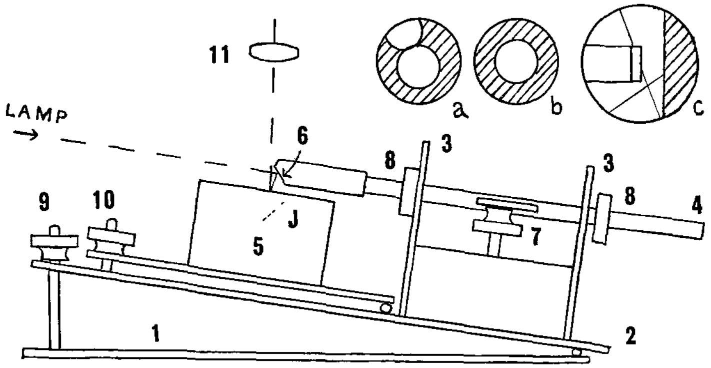

FIG. 1. Inclined reversing mirror for measuring reflectance.

The arrangement for use on the microscope stage is shown in fig. 1. The base plate 1 can be fixed to the stage by two screws as usual so that the rotation axis intersects the axis of the microscope. The rest of the instrument is supported on the plate 2 which is inclined at 7°. This is equal to the angle of incidence for the axial ray of the beam that will be used. Two bearings 3 support the spindle 4 which is parallel with 2. The surface to be tested is set up in a mount 5; it stands on the inclined plate and is parallel with the spindle. At the end of the spindle is the mirror 6. By means of two adjustable stops 7 the rotation of the spindle and mirror can be arrested at two positions separated by 180°. The spindle is free to move for about $\frac{1}{8}$ inch axially so as to allow the image of the desired area to be brought under the comparison area of the photometer in the field of the microscope. Since this movement is often repeated it is convenient to have adjustable stops 8 on the spindle.

*Adjustment.* The mirror is directed upward with the spindle advanced toward the lamp. A suitable area is selected and focused (fig. 1*c*). When the mirror is reversed and withdrawn as in fig. 1 the same area must be seen under the photometer when the microscope objective 11 has been lowered so as to bring the reflected image *J* into focus. At the same time the aperture relations of the beam are checked by inspecting the Ramsden circle with a Becke lens. The required geometric conditions should be sufficiently realized in the construction of the apparatus, so that the image of the lamp is already visible at the back of the objective (fig. 1*a*) in both the direct and reversed settings of the mirror, but it is convenient to provide two adjustment heads 9 and 10 for small final adjustments of this image in the direction of the spindle, while the stops 7 control the position at right angles. Heads 10 and 7 also serve to correct the levelling of the mount. These adjustments must not involve a departure of more than, say, one degree from the strict geometric conditions. By this means the image of the lamp is adjusted to coincide with the aperture disc of the photometer, for both the direct and the reversed settings of the mirror (fig. 1*b*). The disc occupies about one-half of the diameter of the 33-mm. objective (N.A. 0·1) and represents an incident cone of about $\pm 3^\circ$ surrounding the axial ray, which is incident on the object at 7°. Careful checking is essential, otherwise errors may arise from the Stiles–Crawford effect (Walsh, p. 59); for the present photometer the artificial pupil is about $1\frac{1}{2}$ mm. in diameter. Readings are then taken for the intensity of the focused image of the mirror, alternately in the direct and reversed settings. For the present purpose ten pairs of readings were averaged for each determination. There is no correction for reflection in the objective; the value of $R$ is simply the ratio of the two intensities obtained from the table of $\sin^4\theta$ (Manual, p. 194).

### *A standard reflecting surface.*

A standard for general use is best chosen with a reflectance of about 50 %. Cleavage planes cannot be relied on to provide a sufficient area; it was therefore made from a polished metal. An open metal surface requires to be repolished at intervals, and this leads to some uncertainty due to different methods of preparation. The covered standard here used was prepared by Mr. B. O. Payne; it consists of a surface plated with the alloy ‘inconel’, polished and covered with a thin coverglass cemented with balsam. Although in the end some change is to be expected, this standard remains constant over a considerable period and only requires to be cleaned like a glass lens before use. It can be redetermined when required. The thin glass is uniform in effect and does not interfere with the use of the standard at low magnification. Average values were, for mean wave-lengths in m$\mu$: $\lambda$ 670, 54·6;

λ 650, 54·1; λ 580, 51·8; λ 520, 49·0; λ 490, 47·3 %. Duplicates can be checked by the photocell.

*Verification of the Fresnel formula for transparent mineral standards.*

Transparent minerals are subject to errors from back reflections; these can be eliminated by using a shallow prism, such as a 12° prism of sapphire (A. F. Hallimond, 1956, p. 135). Diamond seems an ideally suitable material, though its reflectance (17·2) is still rather low for general use.

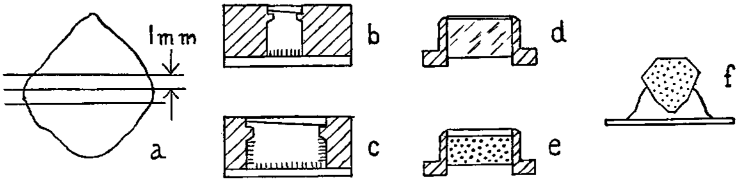

FIG. 2. Photometric standard surfaces. *a*, method of cutting the diamond octahedron; *b*, diamond prism mounted in ebonite with a black velvet surface underneath; *c*, sapphire prism over a box lined with black velvet; *d*, surface plated with 'inconel' metal under a coverglass; *e*, basal section of quartz mounted on clear 'black' glass; *f*, a cut garnet.

The prism (see below) has an angle of 5°. This value was chosen as the minimum that would throw the back reflection clear of the objective, while using the least possible thickness of diamond. It was mounted with the top surface level, over a small cavity lined with black velvet (fig. 2*b*). It is not necessary to varnish the back of these prisms.

*The Fresnel formula.* Although the reflectance can be calculated by the formula $(n-1)^2/(n+1)^2$, it is not *a priori* certain that the surface as prepared will give exactly this value. Direct measurements were accordingly made by the present method, on diamond, garnet, and sapphire. The direct beam is here so much brighter than the reflected that it was found useful to lay a neutral glass screen (transmitting about $\frac{1}{5}$) over the eyepiece while the direct measurement was made. Any change in the Fechner ratio is more than compensated by the avoidance of the bright alternate readings, which cause retinal fatigue.

Reflectance for Ilford screen 606, wave-length 580 m$\mu$.

| Mineral | Observed values (each 10/10 pairs) | | | | | Mean | Calc. | Refr. ind. |
|---|---|---|---|---|---|---|---|---|
| Diamond | 17·3 | 17·6 | 17·4 | 17·6 | 17·2 | 17·4 | 17·23 | 2·419 |
| Sapphire | 7·6 | 7·7 | — | — | — | 7·6 | 7·7 | 1·77 |
| Garnet | 8·2 | 8·2 | — | — | — | 8·2 | 8·3 | 1·809 |

The sapphire and garnet were polished for one or two minutes with $\frac{1}{4}$ $\mu$ diamond paste on selvyt, and cleaned with $CCl_4$. In garnet the internal reflection is absorbed by the strongly coloured mineral.

*Minor errors.* The distance between the lamp and the objective is the same in the direct and reversed settings apart from the small increase due to the axial withdrawal of the mirror by $\frac{1}{8}$ inch. In 24 inches this causes a change in intensity equal to 1 per cent., the observed value of $R$ being too low by that amount. The loss is, however, compensated by the fact that the stainless steel mirror caused a weak polarization, the component perpendicular to the plane of incidence being brighter in the ratio (measured) 6:4. Although the mean value of the two components after quasi-normal reflection in the object should still be very close to the true normal value for $R$, the predominance of the higher component will cause a slight increase in the observed value.

After allowing for these details, the above results seem sufficiently in agreement with the Fresnel formula to justify the calculated values for the three minerals. With quartz (fig. 2*e*, $R = 4{\cdot}58$) they form a useful series when surfaces of lower reflectance are required.

## *Reflectance of Pyrite.*

Pyrite has sometimes been used as a standard, the reflectance determined by Orcel being 54 % at $\lambda$ 590 m$\mu$. Since the screens commonly used transmit rather wide bands, it may be of interest to record a series of direct measurements by the present method with 'spectrum' screens.

An irregular crystal of pyrite from Rio Marina, Elba, containing plates of hematite, was polished as usual. Before each set of readings the surface was polished for 1 minute with $\gamma$-alumina in 'DC 703' silicone on selvyt, and cleaned with $CCl_4$. After one or two days the reflectance of pyrite diminishes, but there seemed little difference in the present results taken within a few hours of polishing.

Each value is the mean of ten pairs of readings, reflected/direct, and is represented by a circular point in fig. 3. As an independent test, the reflectance was also measured in the ordinary way, by substitution with the standard 'inconel' surface prepared above; these results are shown as open circles. For each indirect value six settings were averaged for pyrite, then for the inconel standard. The results, which may be regarded as typical of those to be expected in ordinary determinations, seem in sufficient agreement with the direct series, thus confirming the use of this standard.

The mean values in the direct series for pyrite are:

| $\lambda$ | 470 | 490 | 520 | 550 | 580 | 610 | 650 | 670 | m$\mu$. |
|---|---|---|---|---|---|---|---|---|---|
| $R$ | 49·1 | 50·9 | 52·6 | 52·7 | 54·3 | 55·1 | 56·4 | 55·0 | % |

The reflectance falls off rather steeply towards the blue (Orcel records 43 at 460 m$\mu$). There is a maximum in the red followed by a small reduction at the last, deep red, wave-length. This seems to agree with the values recorded for a 'red' screen (Ramdohr, 1950, p. 572) though at a somewhat longer wave-length.

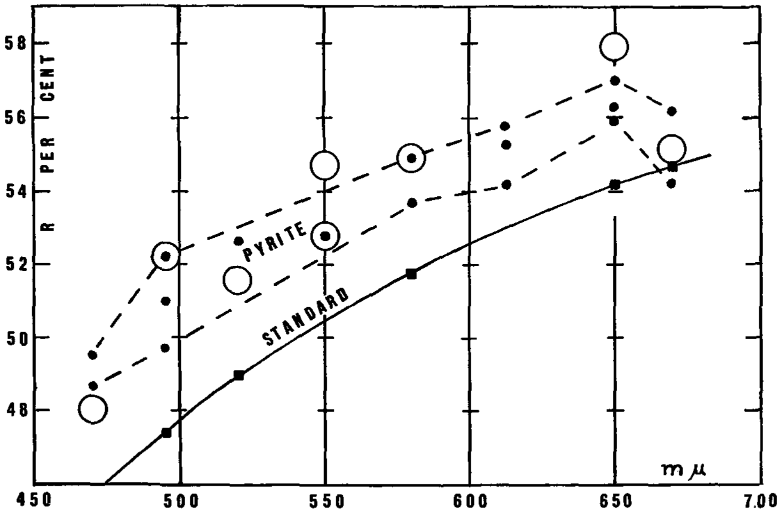

Fig. 3. Dispersion of reflectance. Values for 'inconel' standard (■), and for pyrite (direct, ●; by comparison with 'inconel' standard, ○).

In sodium light the value $54\frac{1}{2}\pm\frac{1}{2}$ is indicated for pyrite.

### *Preparation of the standard diamond prism.*

The late Dr. P. Grodzinsky (Industrial Distributors Ltd., Diamond Research Department, Holborn), who very kindly provided the diamond prism here used, supplied the following details of the preparation of four prisms.

The raw material was two octahedral diamonds each of about 0·6 carat. These diamonds were sawn by Mr. Lever, Woodbridge House, in the way indicated in fig. 2*a*; i.e. the diamond was sawn through the girdle, and two additional saw cuts were made, one on each of the remaining half pyramids. The thickness of the plate was about 1 mm. Subsequently the side faces were finely polished and the angle of 5° taken off one side by Messrs. A. and P. Rotenberg. The result was a plate approximately $4\frac{1}{2}$ mm. square. This emits very little diffuse light when under the full intensity of the ore-microscope. There are still some variations in level, but these do not affect the readings since they will only cause a slight spreading of the aperture disc. The surface finish of diamonds has recently been investigated by A. L. Bailey and M. Seal (1956) and by E. M. Wilks (1953). Polish scratches are everywhere present; they may average 0·01 mm. apart and up to 1,000 Å. in depth, but the depth is reduced by finer polishing. In the present case the aperture disc is well defined and there seems no appreciable loss of light by diffusion at the surface, the reflection being essentially specular.

*Acknowledgements.* The author is indebted to Mr. B. W. Anderson for kindly providing the prism of synthetic sapphire and for the loan of specimens, to the late Dr. P. Grodzinski for the diamond prism described above, and to Mr. B. O. Payne, of Messrs. Cooke, Troughton, and Simms, Ltd., for the preparation of experimental standard surfaces, including that described.

### *References.*

Bailey (A. I.) and Seal (M.), Industr. Diamond Rev., 1956, vol. 16, p. 145.
Hallimond (A. F.), Manual of the Polarizing Microscope, 2nd edn, 1953 (reprinted 1956). York; Cooke, Troughton, and Simms.
Ramdohr (P.), Die Erzmineralien..., Berlin, 2nd edn, 1955.
Steel (W. H.), Journ. Opt. Soc. Amer., 1951, vol. 41, p. 223.
Walsh (J. W. T.), Photometry, 2nd edn, 1953. London, Constable.
Wilks (E. M.), Journ. Opt. Soc. Amer., 1953, vol. 43, p. 84.