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REFRACTOMETER PERILS

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The determination of indices of refraction by the immersion technique is very common in mineralogical work. As long as one is satisfied with results to .002 or .003, slapstick methods are in order. But if the limit is to be .001 or better, considerable care is required, in spite of what one might gather from the mineralogical literature. This note is concerned with the ordinary Abbe prism refractometer which is so commonly used for standardizing the immersion media in the range below 1.84. This instrument may be read directly to .001 and by estimation to .0001, but Emmons as well as Fairbairn and Sheppard state that it is correct to .0002 in its lower range, but is less accurate in its upper range.

This refractometer is not given suitable description in any mineralogical text book in English with which the writer is familiar. A fairly good brief résumé appears in Gibb; a more complete and generally satisfactory treatment is by Bauer. Using this instrument (without Amici prisms) with a mercury-vapor lamp having suitable filters to effectively isolate the light of the 4358, 5461, and "5780" lines should yield quite satisfactory results. But the writer failed to get consistent figures, and so a more careful examination was carried out.

First the setting of the instrument was checked. This is normally done by comparing the reading given by a test glass plate against its n_D value (supplied with the instrument). The writer was unable to get satisfactory checks of his readings on subsequent days, in spite of the fact that he was very careful with the illumination. That is, the light was so directed against the non-polished (i.e., diffusing) end of the test plate that true grazing incidence was bound to occur between it and the contact oil (of higher n than that of the test plate). Checks of this nature were attempted on two different instruments (a standard one with range 1.30–1.70, and a high-index one of range 1.45–1.84) with five different test plates (see Table 1), using four different contact oils.

After consulting Tilton's excellent papers, it was concluded that the major trouble lay in the shape of the contact oil film. Thereafter the writer was very careful to use a minimum amount of oil, wiping off any excess at the base with lens tissue, and pressing the test plate tightly against the refractometer glass. Obviously this last must be done with considerable care, but it seems to be essential to avoid a wedge-shaped oil film instead of a parallel-surfaced film. The best way to make sure that one does not have a wedge-shaped oil film is to take readings from a

TABLE 1. INDICES OF REFRACTION MEASURED FOR THE FIVE GLASS TEST PLATES AT FOUR DIFFERENT WAVELENGTHS

Test plate → Instrument → n of oil → Light	1.5174				1.6107				1.6495				1.7205		1.7898		
	1.45-1.84		1.30-1.70		1.45-1.84		1.30-1.70		1.45-1.84		1.30-1.70		1.45-1.84		1.7205		1.7898
	1.66	1.74	1.66	1.74	1.66	1.74	1.66	1.74	1.66	1.74	1.66	1.74	1.66	1.74	1.82	1.775	1.82
5893	1.5177	1.5177	1.5176	1.5176	1.6109	1.6109	1.6107	1.6107	1.6109	1.6109	1.6497	1.6495	1.6495	1.6495	1.7203	—	1.7896
5780 ± 0010	1.5182	1.5182	1.5182	1.5182	1.6114	1.6114	1.6114	1.6113	1.6114	1.6113	1.6507	1.6506	1.6504	1.6504	1.7220	—	1.7913
5461	1.5194	1.5193 +	1.5195	1.5195	1.6132	1.6130 -	1.6134	1.6133	1.6134	1.6133	1.6541	1.6540	1.6541	1.6542 -	1.7262	—	1.7969
4358	1.5269	1.5269	1.5273	1.5272	1.6235	1.6236	1.6238	1.6238	1.6235	1.6236	1.6737	1.6736	1.6741	1.6741	1.7521	1.8289	—

The corrections added to the actual readings to yield the above results were as follows:

5780 ± 0010	.0022	.0015	.0022	.0015	.0022	.0015	.0022	.0015	.0022	.0015	.0022	.0015	.0022	.0015	.0022	.0015	.0022
5461	.0093	.0065	.0092	.0065	.0092	.0065	.0092	.0065	.0092	.0065	.0092	.0065	.0092	.0065	.0092	.0065	.0092
4358	.0524	.0348	.0518	.0347	.0518	.0347	.0518	.0347	.0518	.0347	.0518	.0348	.0515	.0348	.0515	.0348	.0519

TABLE 2. ASSUMED CORRECT n-VALUES OF THE GLASS TEST PLATES

λ	1.5174	1.6107	1.6495	1.7205	1.7898
578	1.5178	1.6113	1.6505	1.7219	1.7917
546	1.5191	1.6132	1.6541	1.7266	1.7975
436	1.5268	1.6236	1.6738	1.7523	1.8291

TABLE 3. DIFFERENCE BETWEEN MEASURED VALUES AND ASSUMED TRUE VALUES

Plate → R → λ	1.5174		1.6107		1.6495		1.7205		1.7898
	High-n	Standard	High-n	Standard	High-n	Standard	High-n	Standard	High-n
589	+ .0003	+ .0002	+ .0002	—	+ .0001	—	—	—	—
578	+ .0004	+ .0004	+ .0001	+ .0001 -	+ .0001 +	—	—	—	—
546	+ .0003	+ .0004	- .0001	+ .0001 +	—	—	—	—	—

single test plate using oils of two different n -values. If these readings do not check each other, and the error is due to the presence of a wedge-shaped film, it is obvious that other things being equal the result obtained using the film of lower n will be the smaller, and nearer to the true value. Unless the image of the total reflection boundary line was perfectly straight and sharp, no readings were taken. Thus any oil or dirt on the non-polished end of the test plate or on the refractometer glass surface just below it might cast a shadow that would break the regularity of this line. It should be unnecessary to emphasize that when one is looking at the true total reflection boundary line between light and dark, small movements of the mirror or lamp will not cause it to change position, as will occur if one is dealing with a false line. If the observed boundary line is not straight and sharp, this may be due to an unsatisfactory oil contact film or light source, or the latter or/and the mirror may not be properly adjusted. It is very important to focus the ocular so that the cross-lines are sharp; the shorter the wavelength, the lower the eye-lens. A pin-hole cap for the eye-lens should improve the results.

Having observed all the precautions cited above, the writer obtained data as shown in Table 1. Each value given represents a checked result of two or more readings. The contact oils used were as follows: 1.66-alpha bromonaphthalene; 1.74-methylene iodide; 1.775-sulphur in methylene iodide; 1.82-methylene iodide in phenyldiiodoarsine. The true indices of refraction of the test plates were assumed to be those values given in Table 2. These were obtained by taking the figures measured for $\lambda = 436$ (corrected for a true zero-setting) to be correct, and drawing straight lines on Hartmann dispersion paper through these and the (assumed true) given n_D values, as is shown in Fig. 1. Even if the $\lambda = 436$ figures are in error by a small amount, this will have no appreciable effect on the other numbers shown in Table 2.

In effect Table 3 gives the differences between corresponding values in Tables 1 and 2. From Table 3 the following conclusions may be drawn. The standard refractometer reads slightly high in the low- n range, and substantially correct elsewhere. The high- n instrument is substantially correct in its middle range, but reads too high in its low range and too low in its high range.

The above tests were made at room temperature (*ca.* 20° C.). Bauer (p. 1208) notes that the value obtained for a liquid is usually too low (by up to .0003 for low n liquids) if the instrument is adjusted by using a test glass plate. In any case for accurate work with liquids, the refractometer should be calibrated with these; certified samples can be obtained from the Bureau of Standards.

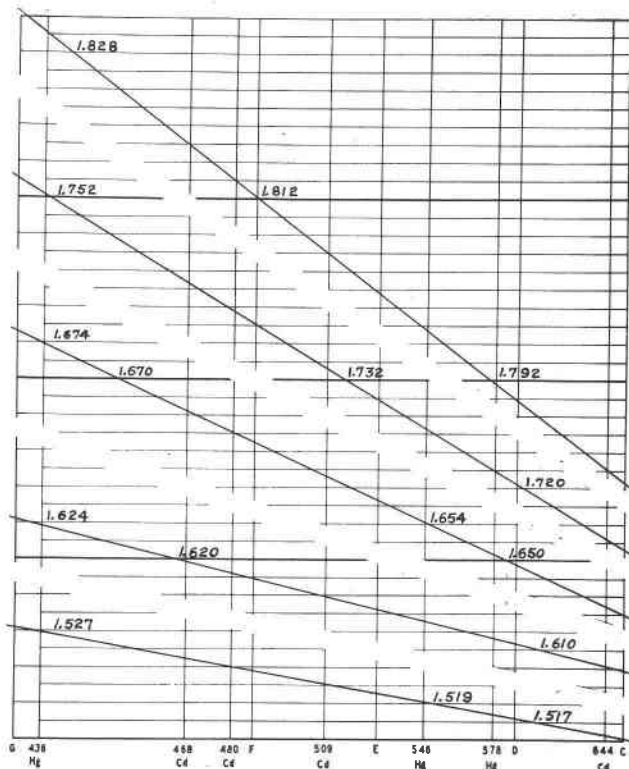


FIG. 1. Dispersion curves for the five glass test plates (see Table 2). The n values assigned to certain horizontal lines are given by the numbers shown above three (four in the central part) lines in each of the five parts of the graph. The spacing between adjacent horizontal lines represents an n -difference of .002 in each case.

REFERENCES

- BAUER, N.; see Weissberger.
 EMMONS, R. C., The universal stage. *Geol. Soc. Amer., Mem.* **8**, 94, 98 (1943).
 FAIRBAIRN, H. W. AND SHEPPARD, C. W., Maximum error in some mineralogic computations. *Am. Mineral.*, **30**, 683 (1945).
 GIBB, T. R. P., Optical methods of chemical analysis, 326-330 (1942).
 RATH, R., Dispersionsbestimmung mit Zeisschen Abbe Refraktometern. *Neues Jb. Min. Abh.*, **87**, 163-184 (1954) and **90**, 1-6, (1957).
 TILTON, L. W., Testing and accurate use of Abbe-type refractometers. *Journ. Opt. Soc. Amer.*, **32**, 371-381 (1942).
 TILTON, L. W., Sources of error in precise commercial refractometry. *J. Research Natl. Bur. Standards*, **30**, 311-328 (1943).
 VON WOLFF, T. F., Methodisches zur quantitativen Gesteins-und Mineral-Untersuchung mit Hilfe der Phasenanalyse. *Mineral. und Petrogr. Mitt.*, **54**, 1-122 (1942). See especially pp. 45-60.
 WEISSBERGER, A. (ed.), Physical methods of organic chemistry. Vol. 1, Pt. 2, Chap. XX on refractometry by N. Bauer. 1203-1214 (1949).