

LAB 10: OPTICAL MATERIALS AND DISPERSION II

The Abbe refractometer is a classic optical instrument used to measure the refractive index of liquids or solids. Abbe refractometers are routinely used in modern chemistry labs for identifying chemical compounds. The instrument is based on the principle of total internal reflection (TIR). Experimentally, a 'shadow boundary' created at the sample's critical angle is used to determine the index. A filtered white light source is used to measure the index, n_d , at the yellow sodium D line of 589 nm.

ABBE REFRACTOMETER

Unlike a spectrometer with its difficult alignment, the refractometer is easy to use. Various types exist, but all function on the concept of the critical angle. The basic principle is shown in Fig. 10.1. Light from a source is incident on a sample of index n , smaller than the prism index n' . The source is diffused so rays are incident on the sample at all angles, up to grazing incidence. The ray at grazing incidence is refracted into the prism at the critical angle, and has the smallest angle of refraction of all other rays at the second face. The result of this is that the ray at the critical angle defines a shadow boundary when looking through a telescope at the light leaving the prism. By measuring the angle between the shadow boundary and the normal to the second face of the prism, the index of the sample may be calculated.

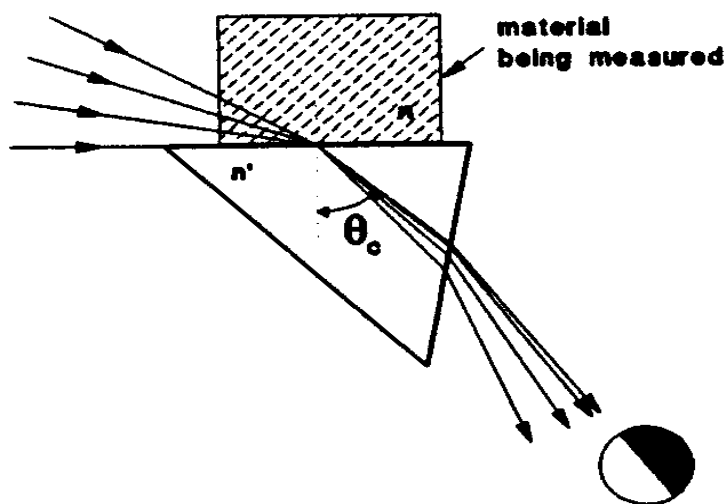


Figure 10.1. Refraction by the prism in a refractometer.

Of the critical-angle refractometers in common use, the Abbe refractometer is by far the most important. In the classical form of the Abbe refractometer the essential components are a white light source, a pair of prisms (one being a measuring prism, the second being used for illumination), and a simple inverting telescope in front of which is placed a pair of counter-rotating prisms. These prisms are rotated (and then fixed in place) in order that they may impose a dispersion equal and opposite to the dispersion of the measuring prism plus the sample system. When this condition is reached the shadow boundary becomes almost achromatic without color; it can then be set accurately on the telescope cross hairs. Without these prisms, a broad rainbow would appear due to the fact that the refractive indices and therefore the critical angle are wavelength-dependent.

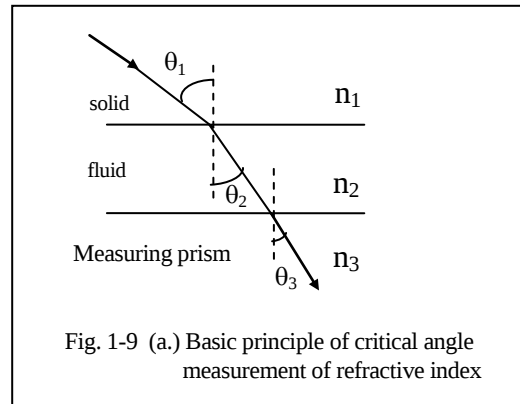
"White light" Abbe refractometers are normally calibrated to read n_D , the sample refractive index for sodium yellow light (589 nm). The rotating prisms used to annul the dispersion of the sample-plus-prism combination are designed to allow sodium D light to pass undeviated; hence, the direction of the observed achromatic boundary coincides with that due to sodium D light conditions, and the instrument will read directly in n_D .

The measuring and illuminating prisms are usually of 30° - 60° - 90° construction, made from glass, the refractive index of which is higher than the upper limit of the instrument range. The prisms are housed, one in each half of a hinged water jacket (used for making index vs. temperature measurements). The two halves of the water jacket, when brought together, enclose a thin film of the liquid being measured. When a solid is being measured the water jacket is left open and the solid is placed on the measuring prism with a contact liquid of high refractive index placed between.

In practice, modern instruments have a stationary measuring prism and a stationary telescope. The sample surface of the measuring prism is usually horizontal and is sealed from the interior of the instrument. The light is directed into the measuring prism by the illuminating prism, and then into the telescope by a rotating mirror, to which is affixed the refractive index scale. Normally, laboratory forms of Abbe refractometers can give an accuracy of from one to two units in the fourth decimal place.

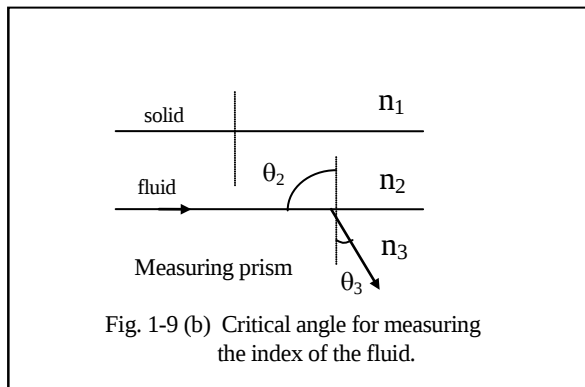
The Abbe refractometer is easily implemented in a compact design. Since the refractive index of a material varies with its composition, it is useful in measuring the concentration of liquids (i.e. percent alcohol, concentration of sugar in serum, etc.). The device is relatively inexpensive, accurate, requires little maintenance or calibration, uses small sample volumes, is easy to use, and lasts indefinitely with reasonable care. It is a workhorse of analytical chemistry labs.

To understand the optical principles behind the refractometer, consider Fig. 10.2. Part (a) shows a general ray passing through the system, along with the angles and indices. The ray shown in (b) is at the critical angle for measuring the index of the fluid. The ray shown in (c) is at the critical angle for measuring the index of the solid.



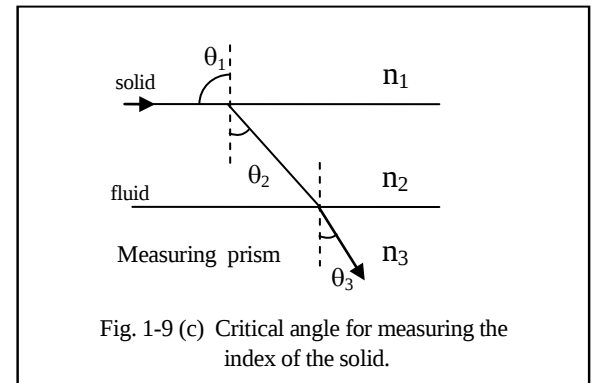
$$n_1 \sin \theta_1 = n_2 \sin \theta_2 = n_3 \sin \theta_3$$

(a) Basic principle.



$$n_2 = n_3 \sin \theta_3$$

(b) Measuring the index of a fluid.



$$n_1 = n_2 \sin \theta_2 = n_3 \sin \theta_3$$

(c) Measuring the index of a solid.

Figure 10.2. Critical angle.

- To measure the index of the liquid, part (b), which condition must hold: $n_2 > n_3$ or $n_3 > n_2$? Explain.
- To measure the index of the solid, part (c), which condition must hold: $n_1 > n_2$ or $n_2 > n_1$? Explain.

The general setup is shown in Fig. 10.3. Using the following relationships, an equation relating the index of the sample to the refraction angle D may be derived:

10-4

$$C = A - B \quad (10.1)$$

Snell's Law tells us

$$\sin D = n_g \sin (A-B) \quad (10.2)$$

For the limiting ray,

$$\sin B = \frac{n_s}{n_g} \quad (10.3)$$

where n_s = Sample (liquid) refractive index

n_g = Glass prism refractive index

$$\cos B = \frac{\sqrt{n_g^2 - n_s^2}}{n_g} \quad (10.4)$$

From which it follows that:

$$n_s = \sin A \sqrt{n_g^2 - \sin^2 D} - (\cos A)(\sin D) \quad (10.5)$$

The instrument operates according to this equation, and is designed and calibrated to read n_d directly at the sodium D wavelength.

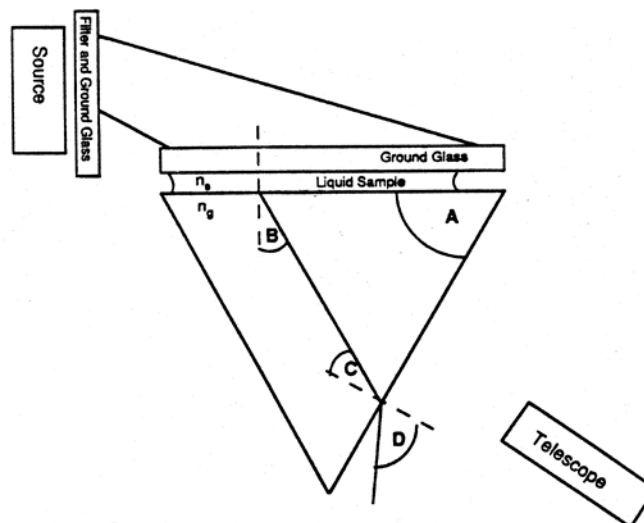


Figure 10.3. Experimental setup for a refractometer.

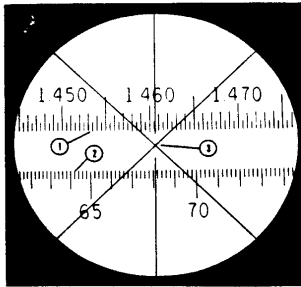
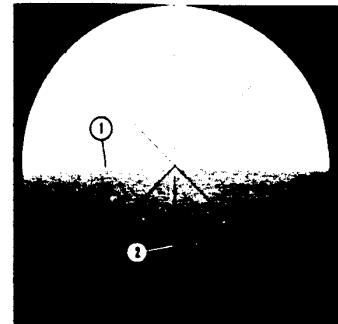


Figure 10.4. View of internal scales in the ABBE 3-L Refractometer. (Courtesy of Spectronic Instruments, Inc. Rochester NY, a Life Sciences International Company).

- 1 Refractive index (n_d) Scale
- 2 Total Dissolved Solids Scale
- 3 Dual Reticle



- 1 Total Reflection Borderline
- 2 Dual Reticle

- 1 Prism Field Lamp
- 2 Upper Prism Case
- 3 Measuring Prism
- 4 Lower Prism Case
- 5 Hinged Light shield
- 6 Compensator Dial
- 7 Thermometer Assembly
- 8 Water Inlet Fitting

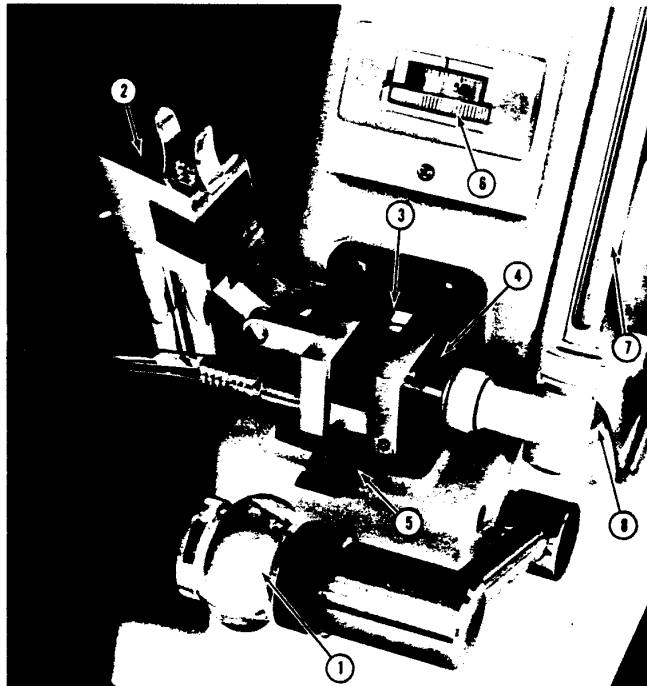


Figure 10.5. ABBE-3L Refractometer. (Courtesy of Spectronic Instruments, Inc., Rochester NY, a Life Sciences International Company).

EXPERIMENT

Familiarize yourself with the operation of the Abbe refractometer before making index measurements:

1. Locate the power switch on the left side of the instrument. It has these positions:

UP:	off
CENTER:	illuminates the prism/sample
DOWN:	illuminates the scale

2. Locate the source and note how it moves on its arm. Also note how the inside of the plastic diffuser is constructed.

WARNING: The next step involves the use of 1-Bromonaphthalene as an index matching fluid. This fluid acts as a skin and eye irritant, and is **POISONOUS** if ingested. Avoid inhalation of the vapors. Be extremely careful not to spill the bottle and keep it tightly closed when not in use. Wash your hands after this part of the lab if you've handled the fluid or samples.

3. Open the top (illuminating) prism. Apply one or two drops of distilled water to the bottom (measuring) prism. Use care not to scratch the prism surface with the dropper.
4. Gently close the top prism, until it snaps shut. This forms a thin, uniform layer of water between the two prisms.
5. Refer to Fig. 10.4 to learn how to read the Refractive Index Scale. The index is read where the vertical cross hair intersects the top (n_d) scale.
6. Hold the power switch in the down position. Use the hand wheel on the right side of the case to set the index scale (visible through the eyepiece) to read 1.3333, the refractive index of water.

7. Release the switch to the center position. Look through the eyepiece and note the shadow boundary. Adjust the source arm and rotate the shield for best contrast and definition of the boundary. Refer to Fig. 10.5 to confirm what you should be seeing.
8. The compensator (rotating prism) corrects for the dispersion inherent in the refractometer optics and the sample when using white light. The compensator is adjusted using the large dial on the front of the instrument. When properly set, the shadow boundary seen through the eyepiece will be achromatic (without color) in the center (at the cross hair), with a faint red color at one end, and a faint blue color at the other end. Remove the plastic cover and rotate the compensator dial until the center of the boundary becomes achromatic.
9. Use the handwheel and center the shadow boundary exactly on the reticle "X" cross hairs.
10. Hold the switch in the down position and note the index reading. It may (or may not read 1.3333). To adjust the scale so that the reading is exactly 1.3333, use the allen wrench to rotate the setscrew (located in the middle of the right-hand side of the housing). Rotate the screw and adjust the scale reading to be exactly 1.3333. The instrument is now calibrated!
11. Open the top prism. Use lens tissue to dry off the water from both prisms.
12. Put on a pair of gloves, and place one drop of index matching fluid (**1-Bromonaphthalene**) on the bottom prism. Place one of the two glass-disk samples on top. **DO NOT** close the top prism!!! Measure the refractive index of this sample. Repeat for the other glass disk.
13. Use a clean piece of lens tissue and acetone to wipe the fluid off of the bottom prism. The prism may be gently wiped with the tissue. Dragging the tissue is not necessary.
14. Throw away the lens tissue, take off the gloves, and go wash your hands to make sure that you have not contacted any of the 1-Bromonaphthalene.

- **IDENTIFY THE GLASS TYPES OF THE UNKNOWN SAMPLE DISKS.**

ABBE-3L REFRACTOMETER

OPTICAL LAYOUT

For purposes of this part of the lab, the metal side panel of the Spectronic Instruments ABBE-3L refractometer has been replaced with a plexiglass panel. Look through the clear panel and study the optical path of the refractometer. With the aid of Fig. 10.6, a full-scale labeled drawing of the refractometer, answer the following questions in your write-up.

- Describe, in words, the light path when the sample is illuminated.
- Describe, in words, the light path when the scale is illuminated.
- Through what angle (approximately) does prism P1 deviate the beam?
- Does prism P1 act to change the parity of the image of the scale as seen through the telescope? Why or why not?
- How is the calibration of the instrument changed? (In other words, how is the scale setting adjusted to match the refractive index of the standard sample of glass)? Explain optically how this is made to happen.
- Explain how the scale gets illuminated.

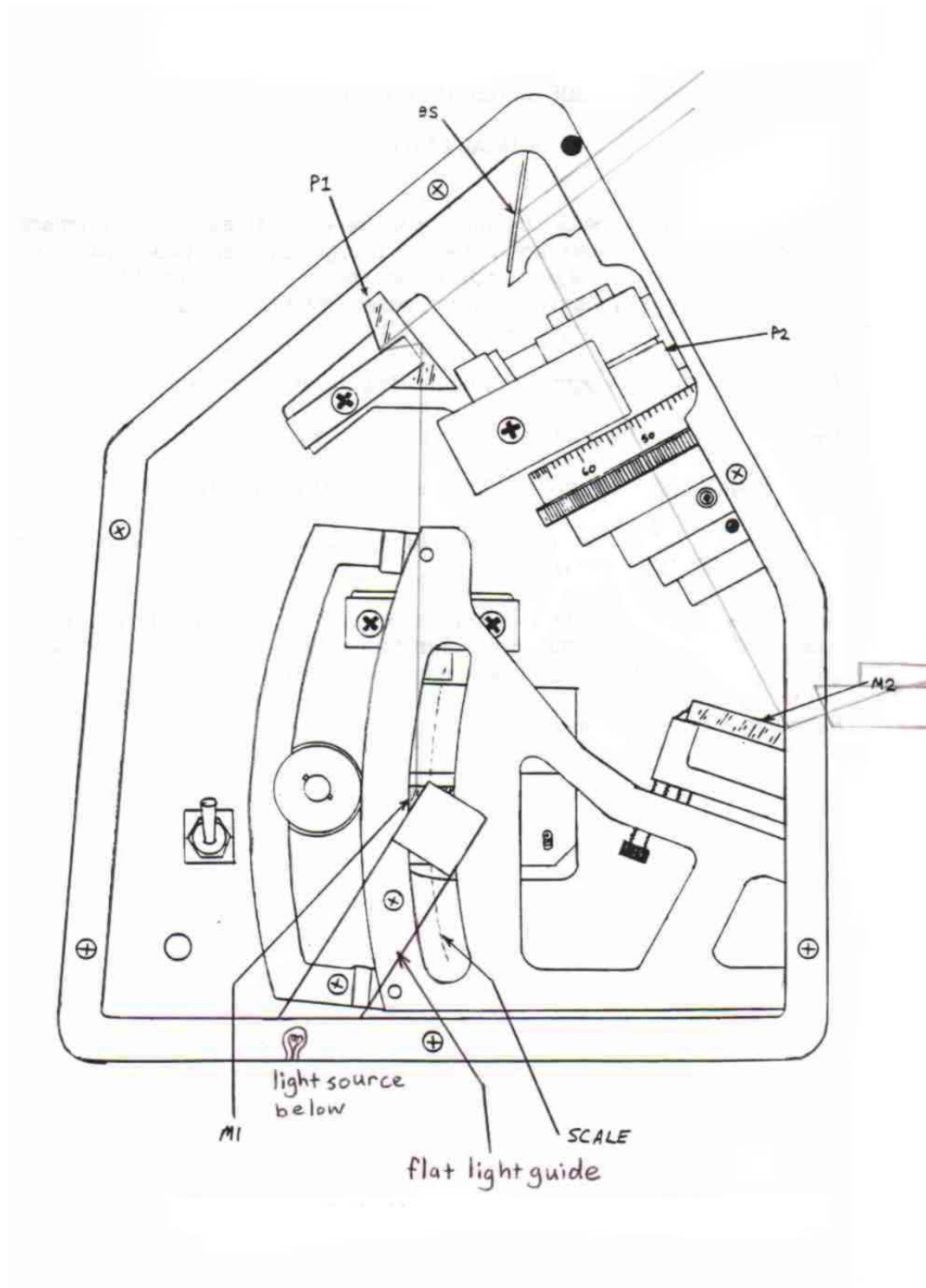


Figure 10.6. Optical layout of the ABBE-3L refractometer.