

Measuring Refractive Index Using the GPR 11-37

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The refractive index (RI) of a liquid can be measured using the automatic refractometer (Index Instruments, model GPR 11-37). The refractometer finds the critical angle of an incident beam of monochromatic light (in this case, the sodium-D line with a wavelength of 589nm) passing through the synthetic sapphire prism into the liquid sample as illustrated in Fig. 1. At the critical angle, no transmitted light enters the liquid so this refractometer can be used for both transparent and opaque liquids.

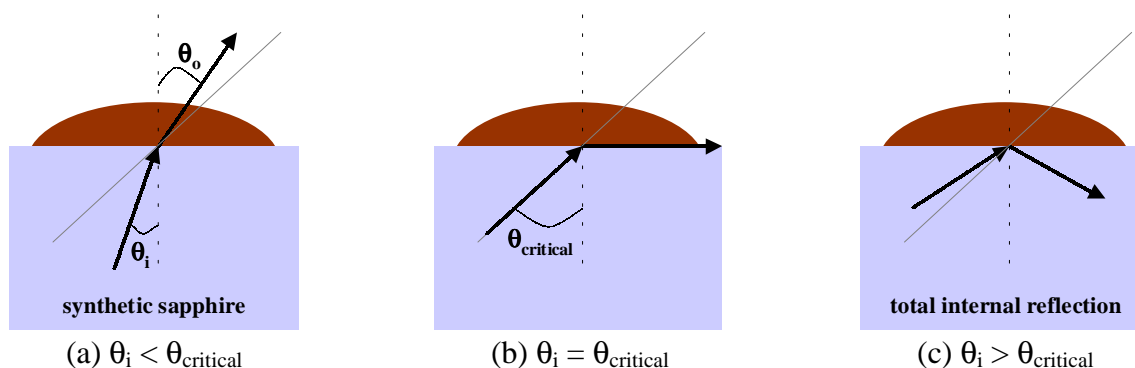


Figure 1. Refraction and reflection of a light wave as it passes from a denser material (the synthetic sapphire crystal) into one that is less dense (the liquid sample). In this simplified diagram, refraction at the outer boundary of the liquid sample is ignored.

The range of RI measurable with this instrument is 1.32-1.70. The accuracy is ± 0.00005 RI unit for $RI < 1.45$ and ± 0.0001 for $RI \geq 1.45$. Temperature can be controlled to an accuracy of $\pm 0.1^\circ\text{C}$ by connecting the refractometer to the Fisher constant temperature circulating water bath, enabling measurements over the range 10-70 $^\circ\text{C}$.

The following steps are recommended for routine RI measurement. (For detailed operating procedures, please refer to the Operator's Handbook for the Index Instruments GPR 11-37 Refractometer).

1. Warm up

- (a) Turn on the water bath circulator. Set the temperature to 20 $^\circ\text{C}$ by adjusting the coarse-tune knob. Then turn on the refractometer and let the whole system to warm up for about 30 minutes. Avoid turning the refractometer on prior to the water bath as the voltage surge that occurs when the water bath is switched on might damage the refractometer.

- (b) Clean the surface of refractometer prism using ethanol and lens wiper. Do not use regular Kimwipes or other coarse wipers as they may permanently damage the prism surface. Dry the surface with a dry wiper. Make sure no stains are left on prism surface.
- (c) Put several drops of distilled water onto the prism surface using a plastic syringe with no needle attached. If air bubbles are trapped at the edge of prism, displace them by touching the bubbles gently with syringe. If a needle is used with syringe, never touch the needle tip to the prism surface since it may scratch and permanently damage the surface. Cover the cell with SGP0 standard cap. To avoid trapping any air bubbles between cap and prism, wet the cap bottom first and lower the cap slowly to the cell at an angle. Trapped air can be detected by the error signal “Unable to read sample.”
- (d) In “Automatic Print Mode,” the RI of water and temperature of the prism surface will display on the screen. This temperature reading may differ slightly from that indicated by the thermometer in the water bath due to heat loss (or gain) along the circulating tube from water bath to refractometer, especially when the preset temperature is significantly different from ambient temperature. Use the refractometer temperature reading, adjusting the fine-tune knob on water bath until the desired temperature is displayed ($\pm 0.1^\circ\text{C}$).

2. Zero the calibration

A full-scale calibration of the refractometer is very time consuming and is only required annually. However, a simple zeroing calibration should be performed at the beginning of every series of measurements. Cover the surface of the prism with distilled water and cover with SGP0 cap, then press the “zero” button on the GPR 11-37. The screen will show “Zeroing...” After several seconds, the RI of water will be displayed. At 20°C , this value must be 1.33300 ± 0.00005 . If there is a small discrepancy, repeat the zeroing procedure until the correct value is obtained. The instrument is now ready for use.

3. Measure RI of liquid sample

For routine RI measurements, the GPR 11-37 refractometer can be set to the following default settings: “ONE SHOT AND LOCK,” “ENTER DELAY TIME 000 sec,” “SAMPLE TYPE: NORMAL,” and “AUTOMATIC PRINT MODE”. In some cases, the sample type may be changed to “EMULSION”. Do not change the other settings without consulting the operating manual.

The GPR 11-37 refractometer has two caps: the SGP0 Standard Cap and the SGP5 Process Flow Cell. The SGP0 cap can be used for non-volatile liquids and mixtures for which evaporation is not a problem. For pure compounds, such as water, n-alkanes higher than n-C7, toluene, 1-methylnaphthalene, etc., SGP0 cap can be used if the temperature is low (*e.g.*, 20°C) and the duration of the experiment is short. In most cases, especially when volatile compounds such as pentane or hexane or crude oils are involved, it is strongly recommended to use SGP5

cell to measure RI as samples inside this cell can be sealed satisfactorily with the aid of an O-ring. Specific steps include the following:

- (a) Clean the prism surface.
- (b) Center an O-ring around the prism.
- (c) Mount SGP5 cap on top of the centered O-ring with the inlet and outlet tubes oriented roughly perpendicular to the plane formed by two alignment stems. Use the “fixing bar” and knurled nuts to press the cap against the O-ring just until it begins to feel tight. Do not overtighten the nuts, as this might cause deformation to the metal plate around prism.
- (d) Use a 1-cc plastic syringe (with needle) to inject liquid into the cell via one of the inlet/outlet tubes. Inject the first 0.1 cc slowly to ensure that the prism surface is completely covered with liquid and no air is trapped. The total amount of liquid needed depends on the specific measurement:
 - If a single measurement is taken at one temperature, about 0.2-0.5 cc of liquid should be enough. For liquids that contain very volatile components, use a larger volume (*e.g.*, 0.5-1.0 cc) to minimize compositional changes near the prism surface during measurement.
 - If measurements are to be made at a series of temperatures, fill the SGP5 cell and almost fill the inlet/outlet tubes (about 2 cc), leaving room for expansion as temperature is increased.
- (e) Seal the inlet and outlet tubes with rubber stoppers.
- (f) In “Automatic Print Mode”, an RI value will be displayed immediately after the sample contacts the prism surface; an updated value will appear every 3 seconds. The reading may shift slightly as the sample temperature equilibrates to that of the prism surface. It can increase or decrease, depending on whether the prism temperature is lower or higher than the sample temperature. **Record the reading as soon as it stabilizes.** If the sample is allowed to remain on the prism, RI may continue to change slowly, especially if the sample contains particles that settle on the prism, components that can adsorb on the prism, or volatile species that evaporate. If RI at multiple temperatures is to be measured, it is a good practice to use a fresh sample for each temperature.

If the sample is a viscous crude oil (especially if it also contains emulsified water) the GPR 11-37 may display “Unable to read sample.” Change “SAMPLE TYPE” from “NORMAL” to “EMULSION.” RI measured in “EMULSION” mode is less accurate, therefore it is recommended to use the “NORMAL” mode whenever possible.

After finishing RI measurements, turn off refractometer first, then set the water bath control to 20°C and turn off the water bath. Use ethanol (and toluene if crude oil has been used) and lens wipers to completely clean the prism surface. Always cover the prism with SGP0 cap after use.