Gemology for Faceters #2

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In the March issue we discussed the importance of gem identification for faceters and provided information on the most significant common gemological instruments. In this, the second installment of a planned series of articles, we introduce the refractometer as arguably the most important single instrument in our quest for proper gem identification.

Mineral Definition by Chemistry and Structure

Every mineral is uniquely defined by two characteristics; chemistry and structure. The chemical composition of a mineral is provided by its chemical formula which typically omits minor and trace elements and those may be important in determining physical properties, including color. A full chemical analysis goes beyond a simple formula and lists all major, minor and sometimes trace elements. It used to be that chemical analysis was undertaken by complex wet chemistry methods that were time consuming and subject to error. Today, most chemical analysis of minerals is done by electron microprobe although a number of other analytical instruments and techniques are also used. The most extensive set of mineral formulae are published in the 9 volume set of *Rock Forming Minerals* by Deer, Howie & Zussman.

Minerals may have identical chemical compositions but completely different structures. The simplest and most elegant example is the difference between graphite and diamond. Exact same chemical composition - simply carbon, but different structures that provide for widely divergent physical properties.

While the chemical composition of a mineral is important to its definition, the second requirement for absolutely defining a mineral is the arrangement of the atoms in its crystal structure. The determination of crystal structure is done by x-ray diffraction, the main instruments used being the single crystal diffractometer and the powder diffractometer.

As an example of why structure is important let's take an aluminium silicate with the formula Al_2SiO_5. The formula defines three different minerals, kyanite, andalusite and sillimanite, and to know which one you have there has to be an understanding of the structure as well as the chemistry.

The combination of chemical composition and crystal structure is referred to as crystal chemistry and it is crystal chemistry that causes the complex physical, chemical and optical properties that characterize each mineral.

Fortunately we don't need electron microprobes and powder diffractometers to identify gemstones, but if you had this equipment and knew how to operate it you could unambiguously identify any of the known 4300 minerals. The fact that there are 14,000 historical mineral names is quite another issue.

As you start off, or continue, the quest to identify gem species it is important to understand that an absolutely definitive identification is not made by measuring physical and optical parameters. It always comes back to the basic two issues – chemistry and structure, or if you like, crystal chemistry.

Optical Mineralogy

Optical mineralogy, comprised of the disciplines of optical crystallography and optical crystal chemistry, is an enormously complex subject that is typically introduced to the geology major when they learn to use the polarizing petrographic microscope as a means of rock and mineral identification. Fortunately, we can skip most of the complexity and focus on the use of the critical angle refractometer.

Crystal chemistry causes light to behave in certain ways when it impacts a crystal and then passes through it. Practically speaking, the ratio of the speed of light in air to the speed of light in the crystal is known as the refractive index (R.I. for short) and for most minerals this is **the most important** of several measurable properties used in a gem's identification.

Optic Character

Several things can happen when a ray of light impinges upon a crystal. We know that the ray slows down, due to the optical density of the material, but beyond that we categorize the material as:

1. Isotropic
2. Uniaxial
3. Biaxial

Isotropic materials are those that form in the cubic crystal system (e.g. like diamond or garnet) or are amorphous, like glass, plastic and amber. Light travels in all directions at the same speed and as a result only one reading will be seen on the refractometer.

Uniaxial crystals have one optic axis which corresponds to the c-axis and where the light behaves as if isotropic. The light ray splits into two rays that travel at different speeds and vibrate in different directions, one in the horizontal plane, which is called the ordinary ray, and one in the vertical plane corresponding to the c-axis and this is called the extraordinary ray. In the refractometer you will see one fixed

reading and one variable reading as the stone is rotated. Uniaxial materials crystallize in the tetragonal, trigonal and hexagonal crystal systems and include such gems as corundum, quartz and beryl.

Biaxial crystals have two optic axes that are singly refractive and three refractive indices that vibrate in different directions designated alpha, beta and gamma. In the refractometer you will see two variable readings when the stone is rotated. Biaxial materials belong to the triclinic, monoclinic and orthorhombic crystal systems and include such stones as topaz, peridot and tanzanite.

Birefringence and Optic Sign

Looking at things in their simplest form, when you have a stone on the refractometer and you rotate it, three things can happen:

1. You get one reading that remains constant as the stone is rotated on the hemicylinder. In this case you have an isotropic (singly refractive) material.
2. You get one reading that remains constant and one reading that is variable and thus you have a uniaxial material.
3. You get two readings that are variable in which case you have a biaxial material.

Fortunately, the minimum and maximum readings are available to us on any single facet of the gemstone when it is rotated. The difference between the minimum and maximum readings is called the **birefringence**. Birefringence is an important quantity that is oftentimes more consistent than refractive indices which can be somewhat variable within gem species based on variations in crystal chemistry.

Uniaxial and biaxial materials have what is called an optic sign and can be either positive or negative (or rarely, in the case of biaxial materials, indeterminable). When the index of refraction of the extraordinary ray of a uniaxial material is greater than the ordinary ray the sign is positive, when the converse is true then the material is negative.

For biaxial crystals the sign is positive if beta is closer to alpha than gamma and negative if beta is closer to gamma than alpha.

In most cases you will not need to determine optic character and sign to make an identification. Typically, just determining the maximum and minimum R.I. and the birefringence will suffice. Note that it is not possible to determine optic character and sign on any facet of a doubly refractive stone.

Part of a refractometer scale with a monochromatic light source.

The Refractometer

So how good is your stop watch? It's clearly not practical to flip the light-switch on and off and record how much slower light is that passes through a crystal. Fortunately a refractometer can measure the critical angle of a gem allowing light to form a shadow line on the calibrated scale of the refractometer and so you can directly read the R.I. from the scale. More detail on how a refractometer works is available in many books on gemology and gem identification.

As indicated in the last article, a good refractometer is not inexpensive with new models ranging from $550-$950, yet with proper care they will last a lifetime. The best instruments are made by Gemological Products (Gem Pro refractometer), Rayner Instruments (Dialdex), GIA's Gem Instruments (Duplex II), System Eickhorst (SR 0.01) and Krüss (ER601 & ER604). Again, avoid the cheap Chinese eBay

Two U.S. made GemPro refractometers. On the right you can see the relative size of the hemicylinder and the magnifying eyepiece and polariod filter.

instruments that cost in the $100 range new, but by all means purchase a used high quality refractometer on eBay.

In addition you will need contact fluid with an R.I. of 1.80 +/- .1, usually included with the instrument, and a light source.

Dispersion & the Light Source

When light passes through an optically dense medium like a crystal its speed varies with its wavelength. Violet light with its shorter wavelength has the least velocity and is refracted most, while red light with its longer wavelength and greater velocity is refracted least. As a result, the refractive index of a gem will be slightly less for red light than it will be for violet light. The difference is known as dispersion and is commonly seen when a prism breaks light into its component colors or when a diamond flashes different colors of the spectrum as it is moved in relationship to the observer's eye. Different gem materials have different coefficients of dispersion, a measurable property.

The practical matter is that when you view a gem's refractive index on the refractometer scale the line that you see will not be as sharp in white light as it will be with monochromatic light (light of one-wavelength). By convention, sodium light is the standard, with a wavelength of 589 nm. As a result, many refractometers have built in yellow light sources that attempt to duplicate this wavelength by using bandpass filters or LEDs, thus providing a sharper shadow line on the refractometer scale and a more accurate reading.

Some refractometers come with built-in light sources (e.g. Krüss) but most use separate light sources that may have both white and yellow monochromatic light. GIA's older grey Utility Lamp is often seen on eBay for $100 or less and functions quite well. Fiber optic light sources and even a Maglite can also be used. The GemPro refractometer, at about $545, comes with a yellow monochromatic filter and represents the lowest-cost, high-quality instrument and is made in the U.S.. (http://www.gemproducts.com/products.html)

GIA makes the Utility Lamp. This is an older model with three ports. The yellow port is monochromatic and the top is useful for a dichroscope. Often found on eBay for $100.

Care and Feeding

Before we talk about the techniques of using the refractometer, there are a few things you should know.

1. Naturally occurring gem materials have refractive indexes that range from a low of about 1.37 in the case of opal to 2.87, in the case of hematite.

2. Most critical angle refractometers can measure from 1.30 to 1.80. The limitation is caused by the R.I. of the contact fluid between the gem and the hemicylinder (usually composed of leaded glass with an R.I. of 1.90 or so), with fluids over 1.81 being highly toxic and increasing in viscosity as R.I. gets higher. Fortunately, the R.I. of the most popular gemstones typically fall within the refractometer's range, the most common exception being diamond and many of its simulants along with some garnets and zircon.

3. Refractive index fluid is generally a mixture of diiodomethane (methylene iodide) and sulfur with added tetraiodoethlene, which is mildly toxic and corrosive. Don't let it get on your skin and wipe it off the hemicylinder surface with a tissue when you are finished. The liquid turns dark upon exposure to light so keep it in a dark place.

4. The glass of the hemicylinder is typically quite soft and can be scratched easily by a gem. Take care not to apply pressure to the gem when placing it on the hemicylinder, and don't use tweezers!

Using the Refractometer

On a faceted stone select the largest facet with the best polish, generally the table, and clean it quickly by rubbing it briskly back and forth on a piece of paper. Place a **very small** droplet of R.I. fluid on the center of the hemicylinder and place the stone, table facet down, on the drop. You've now made an optical connection between the hemicylinder and the stone.

Look through the eyepiece, moving your head up and down slightly until you see a shadow-line and note its position. It should be possible to interpolate to .001 if you are using a monochromatic yellow light source. If the only reading you are seeing is 1.80 or 1.81, then the material is over the limit of the refractometer.

Place the polarizing filter on the ocular and rotate it 90° back and forth (N-S to E-W). Does the shadow-line move? If it does not, rotate the stone 45° and rotate the polarizer back and forth again. Any movement? Repeat with the stone rotated to 90° and 135°. If the shadow-line stays the same then the stone is very likely singly refractive (isotropic).

If the shadow-line appears to move then record two readings, one with the polarizer at its initial position and one turned at 90°, and do that with the stone at the 45°, the 90° and the 135° position.

You will have eight readings in all. Note the high and low readings. These are the minimum and maximum R.I. values, subtract the lowest from the highest and you have the birefringence. Let's say your lowest reading is 1.624 and your highest is 1.644 with birefringence of .020. You have a tourmaline!

Plotting Optic Sign

For most cases the technique above will give you the numbers necessary to look up your unknown in a table of refractive indices and you'll be able to identify what you have, or more than likely, confirm your suspicions. If you can't manage a definitive answer with just R.I. you can revert to other gemological instruments to measure specific gravity, check the spectra or inspect internal characteristics and we'll discuss those techniques and more as the series of articles continues.

In very rare cases you might need to plot the indices of refraction and their variation to determine optic character and sign. Typical examples usually given are separating quartz from scapolite or peridot from sinhalite.

While with the standard procedure you might check a gem in four positions, when you are plotting I like to expand to eight sets of readings at 0°, 30°, 60°, 90°, 120°, 150° and 180°.

In the more complex case of a biaxial stone you will have two shadow lines that move. The higher R.I. is designated gamma, the lower, alpha. If gamma moves less than half way to the lowest alpha reading, the stone's sign is optically negative, if it moves more than half way, the stone is optically positive. If it moves exactly half way the stone is without sign as the 2V=90° (a 90° angle separates the two optic axes. The lowest R.I. reading of gamma is typically called beta. If the alpha and gamma actually meet, then that index is beta; if they don't, then it's a close approximation and you may want to try another facet. There is a technique to find true beta using the transmission angle of the polaroid filter of the refractometer but it is beyond the scope of this necessarily brief discussion.

You can easily graphically plot the eight upper readings as a line and the eight lower readings as a line to graphically see how things sort out.

A Few Caveats

Of course there are some exceptions that make this all a little more interesting.

- In some cases where birefringence is high one reading could be off the scale of the refractometer.
- If you have an R.I. reading on an isotropic stone between 1.50 and 1.70 the stone is most likely glass.
- The optic axis is the direction of single refraction in a doubly refractive gem and so along the optic axis there is only one constant R.I. reading. So, if the optic axis is perfectly aligned perpendicular with the facet upon which you are taking the reading it is possible for a uniaxial stone to act as if it were isotropic. This is, fortunately, unusual, and if you expect this is the case taking a reading on another facet, or using the polariscope, will solve the problem. This is why many formal gemological examination procedures start with the polariscope to determine single or double refraction and that test takes literally less than 10 seconds to accomplish. Some gemologists use the refractometer first and then quickly confirm whether or not the stone is isotropic with the polariscope. Some always take readings on two facets of every stone they test on the refractometer.
- If the facet on the refractometer is cut perfectly perpendicular to the optic axis of a uniaxial stone then the extraordinary ray, the one that usually moves, will remain stationary. Fortunately this is at it's full spacing from the ordinary ray so both R.I. indices and birefringence can be determined.
- Finally, in a biaxial crystal you may have a condition where the optic axis is perpendicular to the facet being tested and one shadow edge will remain stationary, so the stone appears uniaxial.
- In summary, you can find the minimum and maximum R.I.s, and thus the birefringence, on any facet, but you may not be able to determine optic character and sign on any facet of a doubly refractive stone.

Again, if more than two or three stones of every 100 you test require the determination of optic character and sign, I would be surprised. The high and low R.I.s and birefringence will almost always tell the tale.

The Distant Vision or Spot Technique

Occasionally you may want to determine a rough approximation of R.I. on a cabochon or a stone with no flat surfaces. In this case you can use the "distant vision" (U.K.) also called the "spot" (U.S.) technique.

Critical to the effort is a very tiny droplet of R.I. fluid, equivalent to no more than the distance between two of the closest divisions on your refractometer. Place the droplet on the hemicylinder and the apex of the cab on the drop thus making your optical connection.

Looking through the eyepiece of the refractometer from a distance of between 10" and 16" nod your head up and down a bit like you were nodding in agreement and look for the tiny dot that should turn

from dark to light. If you can move your head with precision you may actually see, under ideal conditions, a shadow line that bisects the spot and this is your R.I. approximation. If not, note where on the scale the spot turns from light to dark and read the scale. Spot readings are usually only given to .01 units and birefringence measurements are typically not possible.

Using a larger spot of fluid and rotating the polariod filter can induce "carbonate blink" if birefringence is very large and a very rough estimate of birefringence is sometimes possible. You might use this on a cab of rhodochrosite, for instance.

Some people have trouble getting the spot and the scale of the refractometer in simultaneous focus. This is where the Dialdex refractometer is superior because of its external scale.

In Closing

While optical mineralogy is a complex subject, the techniques necessary to effectively use a refractometer are easily learned and just require a precision instrument and a bit of practice. As a faceter, you are probably generally familiar with gem materials and the use of the refractometer is the quickest way to confirm your stone's identification. It will assist greatly in your confidence that what you are cutting and selling is what's been represented to you. And, who knows, you may discover that the 10 carat spinel you just cut is really a taaffeite!

Three contemporary refractometers. The one in back is the GemPro model available from Gemological Products in Oregon for $545 with a monochromatic filter that installs in the light port in the back. You could use a Maglite as a light source.

The center unit is made by GIA's Gem Instruments and is familiar to thousands of GIA trained gemologists. It comes with R.I. fluid but requires an external monochromatic light source. Cost is about $850.

The unit in the foreground is the Rayner Dialdex, made in the U.K. but hard to find. The Dialdex is unusual in that you match a black tape with the position of the shadow line and then read your R.I. directly from the dial mounted on the right; there is no internal scale. This is an excellent instrument and is available from Rubin & Son in Antwerp for about $760 and requires an external light source.

Editor's Note:

I'm happy to try to answer any technical gemological or gem instrument related questions for any USFG Member. I'm also quite willing to identify any gem material free of charge for USFG members. I can be reached at 208-712-0172 or by e-mail at bruce@gemscientist.com