
NOTES • AND • NEW TECHNIQUES

REFLECTANCE INFRARED SPECTROSCOPY IN GEMOLOGY

By F. Martin, H. Mérioux, and P. Zecchini

External reflectance infrared spectroscopy permits a very rapid and nondestructive determination of the species of a mineral. It is particularly useful with polished gems in that it requires no more than placing the stone, loose or mounted, on a reflection device in the infrared spectrometer and observing the recorded spectrum. Therefore, compared to the classical methods of physical measurement such as refractive index, specific gravity, and the like, reflectance spectroscopy provides a much faster means of establishing the species of the gem and, in some cases, whether it is natural or synthetic.

Gem identification requires broad knowledge built on a number of observations. Generally, the identity of a gem material can be easily determined on the basis of physical and optical properties such as specific gravity, refractive index, and visible-light absorption spectrum. In some cases, however, such as the separation of scapolite from quartz (figure 1), more sophisticated methods may be required. X-ray diffraction and microprobe analyses have been used for this purpose, but they require very special equipment and sample preparation (which may be destructive).

External reflection infrared spectroscopy (commonly referred to as reflectance spectroscopy) provides an intermediate solution for these problem cases. It has been used in mineralogy for many years to measure optical constants such as refractive index (Simon, 1951) and to determine fundamental vibrations of crystals (Simon, 1953). Ba-

sically, reflectance spectroscopy measures the vibration energy of the atoms inside the crystals. Inasmuch as these energies vary from one gem material to another, the reflectance spectrum serves as a "fingerprint" of the stone. This allows identification of the mineral species and, in some cases, determination as to whether the stone is natural or synthetic.

The measurements for reflectance spectroscopy are much easier than for most other sophisticated techniques. Because a gem—whether mounted or loose—generally has at least one polished face, it need only be placed on the reflectance device in the spectrometer to obtain the spectrum. No elaborate sample preparation is required, and the method is completely non-destructive. Results can be obtained in a few minutes.*

This article presents a general application of reflectance infrared spectroscopy to identify a cut stone, with sample spectra provided for more than 60 natural gemstones, synthetics, and simulants

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(some of which were first reported by Leung et al., 1982; Ramirez et al., 1983, 1985; Zecchini, 1986; Zecchini et al., 1986, 1987).

INSTRUMENTATION AND EXPERIMENTAL PROCEDURES

For this study, we used Perkin-Elmer instrumentation: two conventional grating infrared spectrometers, PE 580 B and PE 983, connected to PE 3600 data stations (which enabled us to record the spectra and provide graphic representation of the data) and the reflectance accessory illustrated in figure 2. This accessory is readily available from most infrared spectrometer accessory manufacturers.

For a given crystal, two types of infrared spectra can be recorded: a transmission spectrum (from which, if the sample is "transparent" to the infrared beam, an absorption spectrum can be derived) and a "mirror-like" (or specular) reflection spectrum correlated to the lattice vibrations of the crystal. According to observations made on the infrared spectra of gem crystals, the fundamental vibration energies of atoms in crystals are situated in the infrared domain, typically between 1500 cm^{-1} (6600 nm) and 100 cm^{-1} ($100,000\text{ nm}$), so reflection spectra can be observed only in this region.

The transmission method would be very powerful if it were feasible to cut the stone; observations through a thin (few microns thick) slab give, directly, very accurate results on real lattice vibration absorption positions. Such exact positions, which are not necessary for our purpose, can be calculated from the reflection spectrum (Laroussi, 1984). Although direct observation through a faceted stone is sometimes possible, the transmission spectrum from such thicker materials does not show the fundamental crystal lattice vibrations (i.e., there is almost total absorption in that range), but rather only reveals the typical impurities such as water, carbon dioxide, nitrogen, hydroxide, and the like. Moreover, the stone must be placed in front of the incident beam in such a way that light-

**Time varies according to the spectrometer used and the observable surface of the sample. With a conventional grating spectrometer and an observable surface larger than about 2 mm^2 , it takes no longer than five minutes. If an FTIR spectrometer is used, even less time should be needed. For smaller samples, additional time is often required for placement of the sample on the reflection device in the right position to obtain sufficient intensity.*



Figure 1. The separation of scapolite (right and bottom) and quartz (top and left), may be difficult to perform using standard gemological testing but is accomplished in a matter of minutes with reflectance infrared spectroscopy. Photo © Tino Hammid.

scattering dispersion is minimized, which is very difficult with most dispersive infrared spectrometers.

Reflectance is much easier to perform: Simply place a facet of the polished gem on the reflection device. Because the observed bands are very specific to a given crystal, the identification is easily made by comparing the spectrum obtained with prerecorded spectra for various gem materials.

TESTING AND RESULTS

To determine the usefulness of this technique for gem identification, we obtained infrared spectra, using reflectance spectroscopy, on more than 60 natural and synthetic gem materials. We studied at least five samples of each gem, and found that within the same mineral species, the variations between spectra for different varieties or from different localities are very small.

The reflectance spectrum of a pyrope garnet is shown in figure 3 both in a conventional manner, a continuous line that rises and falls with the reflectance of the sample, and converted to a set of parallel bars with height and thickness correlated to the reflective power. This latter format facili-

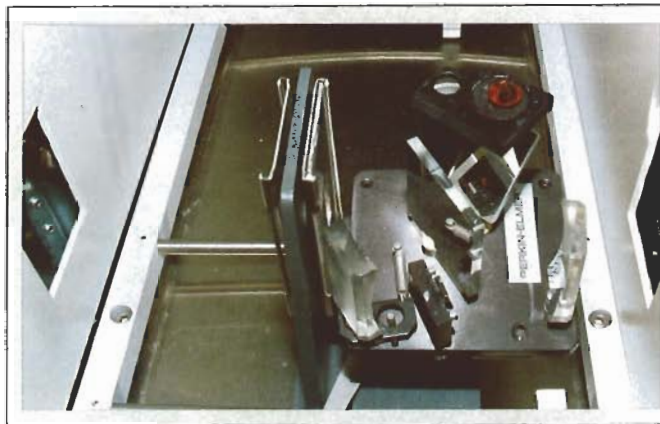
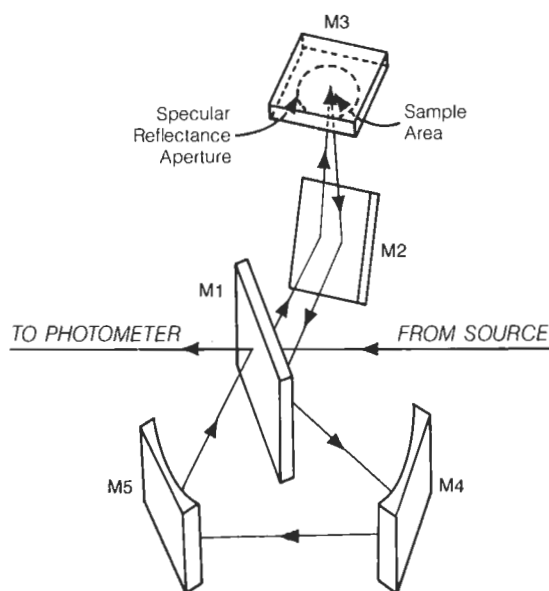


Figure 2. This optical scheme shows how the sample interacts with the source of infrared light and the photometer through a series of mirrors (M1–M5) in the reflection accessory to produce the reflectance spectrum.



tates presentation of the data and comparison from one gem material to another. The ability to present the results for many stones on a single piece of paper (figure 4) allows for a very rapid and effective identification of, for example, corundum from spinel, calcite from aragonite, cubic zirconia (djevalite) from strontium titanate (fabulite), and grossular garnet from pyrope garnet. Only type IIa diamond, which is transparent to the infrared beam in the $100\text{--}1500\text{ cm}^{-1}$ range, cannot be determined, although all of its common simulants can be readily characterized by this method.

This technique is particularly useful for the separation of some synthetics from their natural counterparts. This is the case for alexandrite (figure 5) and for spinel (again, see figure 4). The different sites occupied by the atoms in natural as compared to synthetic stones can be observed as a difference in the lattice vibration spectra.

As part of our study, we also investigated the effects of polarization. That is, on crystals that have one or two optic axes, the incident beam is partially polarized and the spectrum observed depends on the orientation of the facet used and its

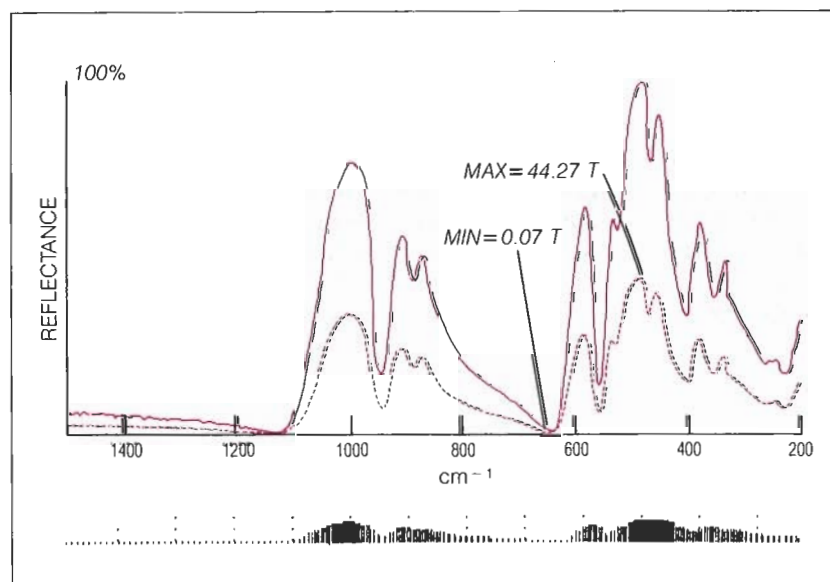


Figure 3. The reflectance spectrum of a pyrope garnet as it normally appears (upper part) and as converted (lower part) for this study. For the conversion, the recorded infrared spectrum (broken line) is expanded between 0 and 100% (continuous line). In this expanded spectrum, a point represents each 10 cm^{-1} . The height of the bar at each point represents variations in reflection intensity. That is, the bar is counted as nine units if the reflection intensity varies between 90% and 100%, as eight units if between 80% and 89%, and so on. The black regions show where the largest reflective power is located; to achieve this, one correlates the thickness of the bar to its height.

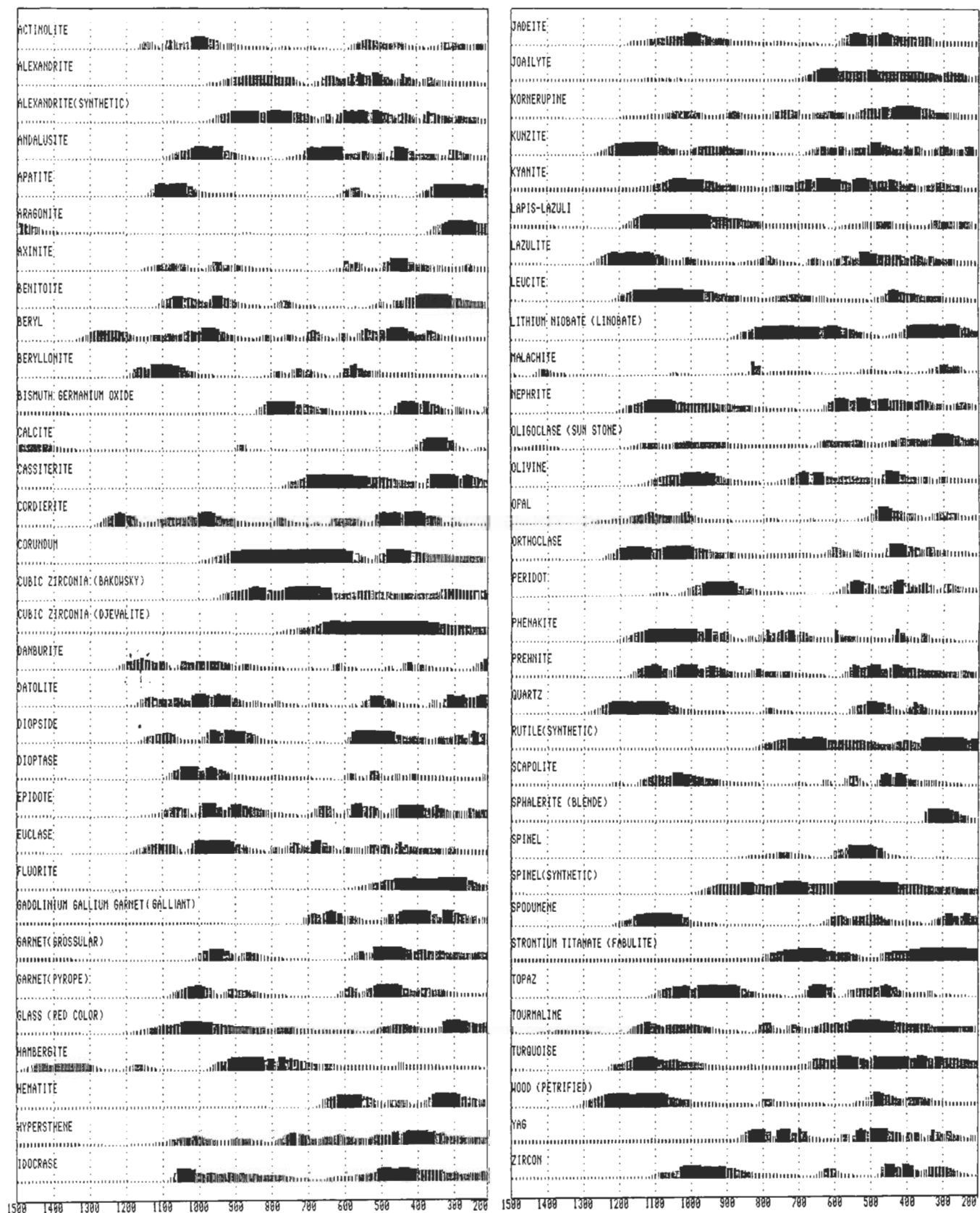


Figure 4. The authors recorded the reflectance spectra of at least five samples each of more than 60 gems, synthetic gems, and simulants, and then converted their results to these simple bar graphs for easy comparison. The authors found no significant variability within a species for the materials they tested.

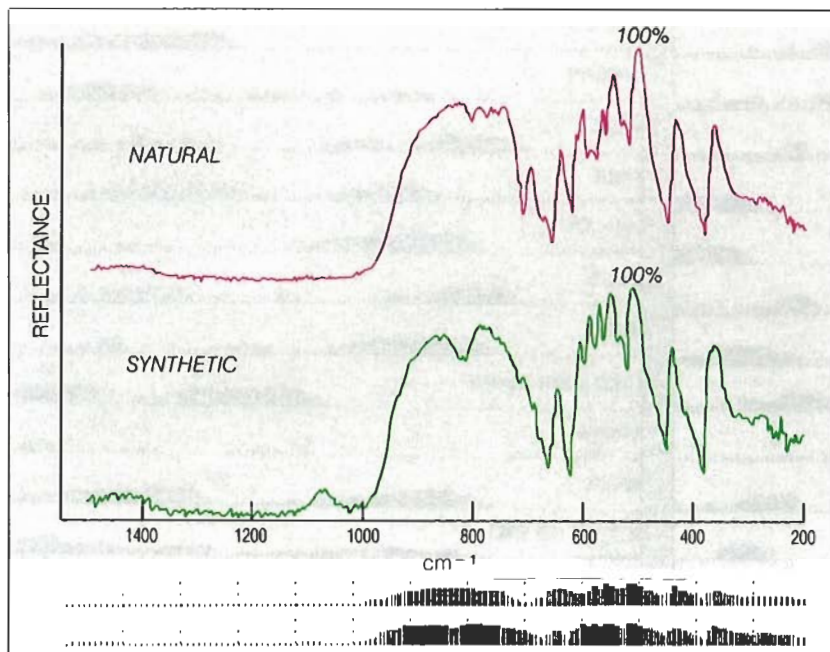


Figure 5. Natural and synthetic alexandrite (here manufactured by Created Crystals) are easily separated by the reflectance method. Note, however, that because the bargraphs are simplified representations, the difference between the two spectra—that is, an additional weak reflection observed between 1050 and 1100 cm^{-1} in the man-made stone—is better seen with the conventional spectra.

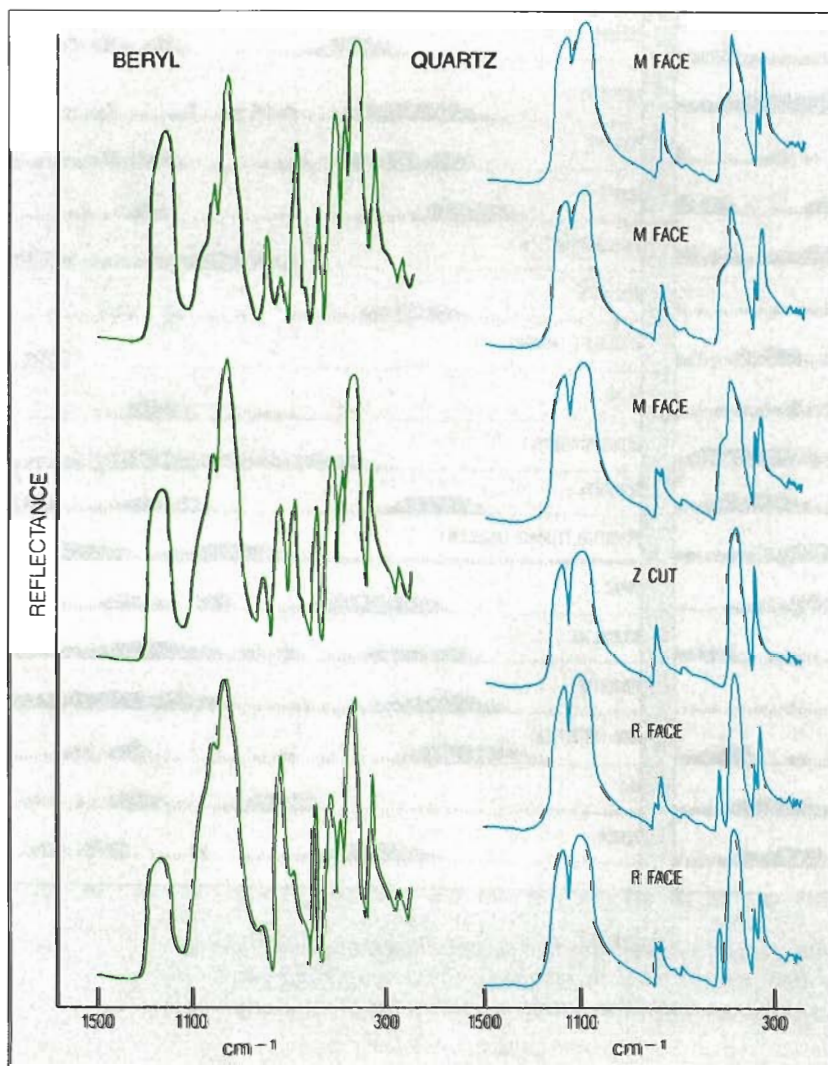


Figure 6. Although the position in which the sample is run does have some effect on the reflectance infrared spectrum, all of the spectra are within the range for that gem material, thus allowing its identification. In the left-hand column, rotations (at 30° steps) about the axis normal to the table of this faceted beryl induce these spectra, all of which are characteristic of this gem species. In the right-hand column, rotations (again, at 30° steps) about the axis normal to the M (minor rhombohedron) and R (major rhombohedron) faces of a natural quartz show only small differences from one spectrum to another. For a Z cut—the observed plate was cut perpendicular to the Z (or optic axis) of the crystal; the same spectrum is obtained for any rotation about this axis (isotropic direction).

position on the reflectance accessory. We found that while the spectra do differ slightly according to the position of the sample, overall they are still within the spectral range of that particular species (figure 6).

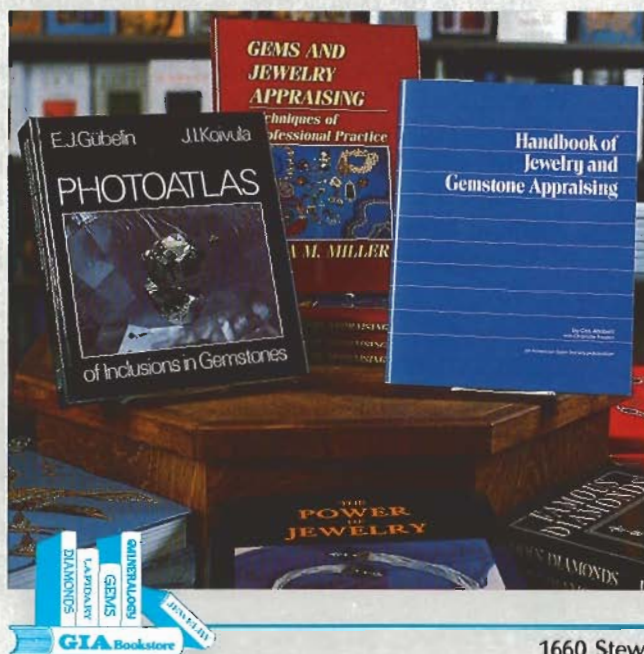
CONCLUSION

External reflection infrared spectroscopy provides a nondestructive method for identifying the mineral species of most gem materials, whether loose or mounted in jewelry. Our results were obtained with faceted stones, but they may also be obtained with uncut crystals that have at least one polished face. In some cases, such as chrysoberyl (including alexandrite) and spinel, this technique can be used to determine the natural or synthetic origin of the stone as well.

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