GIA GEM TRADE LAB NOTES

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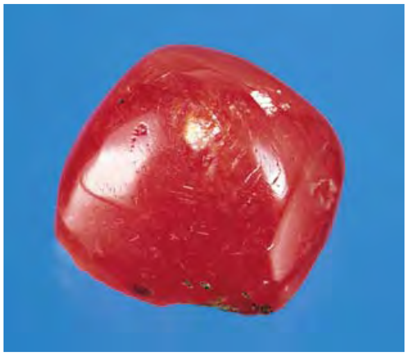
CALCITE, Colored by Inclusions

A rare-mineral dealer from Colorado sent the 10.45 ct orange-red cabochon shown in figure 1 to the West Coast laboratory for identification last summer. He stated that the material was found at the site of an old copper mine in his local area. The dealer knew that the material was a carbonate, but he was unsure whether it was an unusual color of rhodochrosite or another carbonate mineral colored by inclusions.

Refractive indices of 1.49 and about 1.65 were obtained by the spot method, with a weak but clear birefringence blink. When we examined the cabochon with magnification, we saw strong doubling and pock marks on the surface, with frequent undercutting, which implies a low hardness. A small drop of dilute hydrochloric acid caused the material to effervesce strongly, thus confirming that it was a carbonate. Two hydrostatic determinations of the specific gravity yielded a value of 2.73. Although rhodochrosite is also soft and effervesces to HCl, we identified the bulk material as calcite on the basis of specific gravity together with the refractive indices.

Figure 1. This 10.45 ct orange-red calcite is colored by inclusions of chalcotrichite, a fibrous variety of cuprite.



Magnification also revealed numerous orange and red reflective needles, as well as clusters of transparent-to-translucent purplish red crystals. Using diffused light, we observed that the body of the cabochon was quite pale, and the overall color was due to these inclusions. The cabochon fluoresced weak red to long-wave UV radiation and weak, moderately chalky reddish orange to short-wave UV. Lines at 500, 590, and 610 nm were visible in a desk-model spectroscope. From their habit and color, as well as from the reported provenance of the cabochon, we believe that the inclusions are chalcotrichite, a fibrous variety of cuprite.

The appearance of these inclusions and the overall color of the material reminded us of some bright red quartz from Zacatecas, Mexico, reported in the Spring 1993 Gem News section (pp. 59–60). That material was also found in an old copper mine, and EDXRF of those inclusions showed them to be rich in copper and almost free of iron. The color is quite distinct from the more brownish red of the iron-rich inclusions, such as goethite and lepidocrocite, that are found in "strawberry" quartz from Russia (Gem News, Spring 1995, p. 63). *IR and CYW*

CORUNDUM, Diffusion Treated

For some time now, laboratories on both coasts have been receiving large lots of rubies and sapphires for identification, which includes a determination of whether the stones have been subjected to any type of enhancement. One such parcel recently submitted to the West Coast lab contained small, attractive red stones, with one oval mixed cut that stood out visually from the rest because it was much darker in tone. (A closer inspection also revealed that this stone had a poor polish, unlike the rest of the parcel.) The client believed that they were all rubies and sent

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Editor's note: The initials at the end of each item identify the editor(s) or contributing editor(s) who provided that item.

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them to the lab out of concern about heat treatment.

Ruby has a characteristic spectrum—with chromium lines at about 694 nm, 668 nm, and 659 nm, and another pair of lines at 476 nm and 468 nm—that conclusively distinguishes it from other red gems such as garnet and spinel, or from imitations such as glass. Consequently, we began our examination with a desk-model spectroscope to verify quickly whether all the stones in the parcel were ruby. We found this spectrum for all the stones except the darker-toned oval, which showed only a single strong absorption line in the red end of the visible spectrum.

Next we turned to the refractometer, but we experienced some difficulties obtaining the R.I. for this stone. The table gave an indistinct reading over the limits of the refractometer, whereas one small area of the pavilion yielded a vague reading of 1.7. The polariscope, however, revealed that the stone was doubly refractive, with a clear uniaxial figure. The specific gravity was measured hydrostatically at 4.00. This combination of properties proved that the stone was indeed corundum.

We turned next to observation with the microscope, and saw clearly why the refractive index reading of this oval was difficult to obtain and rather vague. The stone was heavily scratched on all sides and showed a very thin, crazed surface layer similar in appearance to that seen in Lechleitner synthetic emerald overgrowth on beryl. However, this oval mixed cut did not have any inclusions except for a tiny "fingerprint," which was too small to indicate whether the stone was of natural or synthetic origin. (Magnification revealed evidence of heat treatment in the rest of the stones in the parcel.)

Examination in diffused light showed that the purplish red color of this stone was irregularly distributed and concentrated at the facet junctions. These features indicate diffusion treatment. When we immersed the stone in methylene iodide, the diffusion layer became quite prominent, as illustrated in figure 2. This figure also reveals some areas that did not have the red diffusion layer, probably due to recutting or polishing. On the basis of these results, the laboratory identified the stone as a diffusion-treated corundum and included a comment stating that its natural or synthetic origin is currently undeterminable. *KH*

Figure 2. When immersed in methylene iodide and placed over diffused transmitted light, this 0.69 ct oval mixed-cut corundum showed both the color concentrations on facet junctions and the spots devoid of color that indicate that it was colored by diffusion treatment.

DIAMOND

Colored by Pink Coating

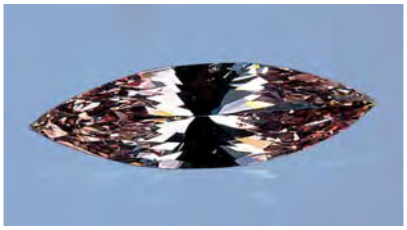
In the second edition of his book *Gemstone Enhancement* (Butterworth-Heinemann, 1994), Dr. Kurt Nassau references Benvenuto Cellini's *Treatise on Goldsmithing*, published in Venice in 1568, about gemstone treatment. Specifically, he cites Cellini's discussion of a variety of coatings and elaborate backings that were used to enhance diamonds primarily, as it was against the law to coat emerald, ruby, and sapphire. Not only does Cellini mention the blue dye—indigo—that was used to coat yellow diamonds, but he also states that "smoky colors" were the most desirable and describes the several steps required to achieve them.

Over the years, most coated diamonds that we have encountered in the laboratories have been light yellow to near colorless, with the net result of the treatment being that the stones appear less colored, or "whiter." In some instances, we have seen diamonds that were "painted" to imitate fancy colors. The most notable in recent memory was a 10.88 ct light yellow emerald cut that had been coated pink with fingernail polish (reported in Gem Trade Lab Notes, Summer 1983, pp. 112–113).

The 5.75 ct brownish purple-pink marquise brilliant shown in figure 3 is the most recent example of a coated diamond that simulates a color that rarely occurs naturally in diamonds. In fact, the color reminded us of some of the first diamond crystals we saw from Russia in the early 1970s, which were given to one of the editors by Robert Webster. At the time, we were under the impression that this color would be representative of the production from Russia, but since then we have seen very few diamonds (from Russia or elsewhere) in this color range.

The first thing we saw with magnification was that the stone did not have the characteristic colored glide planes that are usually present in diamonds in this hue range. Identification was rather straightforward when we viewed the stone with a microscope using darkfield illumination and a white diffuser plate between the light source and the diamond: The speckled surface color typical of a coating was readily visible (figure 4). Although we were unable to identify the exact nature of the coating sub-

Figure 3. The brownish purple-pink color of this 5.75 ct marquise diamond was the result of a surface coating.



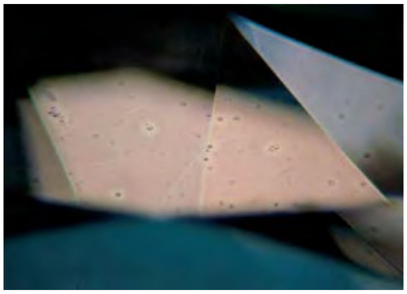


Figure 4. The spotty surface coating on the diamond shown in figure 3 was revealed with magnification (here, 63×) and a diffuse light source.

stance, it appeared to have been sputtered onto the entire stone and was most visible as small concentrated spots on the pavilion.

GRC and TM

With Different Color Appearances Depending on Viewing Position

From our discussions with members of the diamond trade, we are aware that they move colored diamonds through a number of positions when assessing the color. While this can be helpful in making manufacturing decisions, it has been our experience that consistent, repeatable results for color grading are best obtained by limiting the color appearance variables and, especially, controlling the viewing environment. With colored diamonds, the face-up position best accounts for the influence of cutting style on color appearance and is also the angle from which the diamonds will be viewed when worn in jewelry (see, e.g., King et al., *Gems & Gemology,* Winter 1994, p. 225). For these reasons, the laboratory color grades *colored* diamonds in the face-up position only.

A 2.67 ct emerald-cut diamond that was recently submitted to the East Coast laboratory provided an interesting example of the potential confusion that can arise if the stone is placed in more than one position for grading. Following GIA GTL's standard color-grading methodology, the diamond was described as Fancy Deep Brown-Yellow (figure 5). Later, during the diamond's examination for origin determination, it was viewed through the pavilion and a noticeably different brown-orange bodycolor was observed (figure 6). A grader attempting to reconcile these two appearances might reach a color description that was not directly observed from either viewing angle. Examination of the stone face-up, though, results in the most consistent description of the diamond's characteristic color.

John King

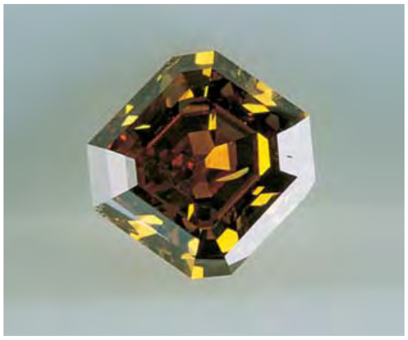


Figure 5. The face-up color of this 2.67 ct emerald-cut diamond received a grade of Fancy Deep Brown-Yellow.

Figure 6. The bodycolor of the diamond in figure 5 appears to be much more orange when it is viewed through the pavilion.

Manufactured GLASS, Represented as "Green Obsidian"

It is usually a straightforward exercise to distinguish between glass imitations and the natural materials that are being imitated (see, for instance, "Glass Imitations of Various Gems," by Nicholas DelRe, in the Summer 1992 Lab Notes, pp. 125–126). Among the features that can be diagnostic for glass are its singly refractive optic character, low refractive index, and low specific gravity; also, swirled growth bands and (spherical or stretched) gas bubbles may be visible with magnification. Of course, the presence of any "mold marks" on the surface of a suspect gem or object is another factor that should always be taken into consideration. However, it is much more difficult to distinguish between a natural glass, such as obsidian or moldavite, and a manufactured glass.

The six modified triangular brilliants shown in figure 7 were represented as natural green obsidian from Tunduru, Tanzania. They ranged in size from 9.06 × 9.46 × 6.11 mm (2.05 ct) to 12.03 × 12.21 × 8.18 mm (4.93 ct), and in color from yellowish green to green. They had the following gemological properties: color distribution—even; diaphaneity—transparent; optic character—singly refractive with weak anomalous double refraction; R.I.—1.518 to 1.530; S.G.—2.50 to 2.52; inert, faint yellow, or weak-to-medium green to short-wave ultraviolet radiation, and inert to long-wave UV; no obvious lines in the spectrum seen with a handheld spectroscope, but dark in the blue and in the red (above 650 nm) regions. This was enough information to confirm that these samples were glass, but not to identify whether they were of natural or manufactured origin.

With magnification, we saw a few perfectly spherical gas bubbles and swirl lines with weak-to-moderate curvature. These inclusions did not resemble those in natural glasses with which we are familiar: Obsidian, for example, usually contains tiny crystalline inclusions, and the swirls and bubbles in moldavite are often quite contorted (see, for instance, A. de Goutière, "Photogenic Inclusions in

Figure 7. These six modified triangular brilliants (2.05–4.93 ct) were represented as natural "green obsidian," but they proved to be manufactured glass.

Moldavite," *Journal of Gemmology*, Vol. 24, No. 6, pp. 415–419). The inclusions in these six triangular brilliants were strong—but not conclusive—indications that the glass was manufactured rather than natural. To be certain of the identification, we went to advanced testing procedures.

Three samples—one of each intensity of green—were examined by Fourier-transform infrared (FTIR) spectroscopy. We have analyzed several natural and manufactured glasses over the past few years; most samples, from both categories, are opaque below about 2100 cm^{-1} (this edge shifts depending on the nature and size of the sample). Most obsidians show a saturated "hump" that rises sharply at about 3700 cm^{-1} and tails off to lower wavenumbers (to about 3200 cm^{-1}, but this is quite variable). There are sometimes two weak, broad features centered around 3900 and 4500 cm^{-1} as well. Moldavite and Libyan Desert glass also have distinctive spectra.

Although not all manufactured glasses have the same infrared spectra, many show a common pattern—which we have not seen in natural glasses—that consists of a broad plateau from about 3600 cm^{-1} to the 2100 cm^{-1} (or so) absorption edge, with superimposed broad peaks at 2830 cm^{-1} and about 3520 cm^{-1}. (For example, brown glass beer bottles have this spectrum.) The three samples we examined also had this "manufactured glass" infrared spectrum. EDXRF analysis on one sample found major Si; minor Ca, Al, and Na; and trace amounts of K, Ti, Cr, Fe, Cu, Pb, Sr, and Zr. We concluded that this material was manufactured glass; in fact, we cannot recall seeing an example of transparent "green obsidian" that has ever proved to be a natural glass.

MLJ, IR, and Philip Owens

PEARLS

Cultured, with Dolomite Beads

Although commercial pearl culturing is nearly a century old, from time to time we still see examples of innovation in this field. For example, we reported on pearls cultured with dyed green beads made from powdered oyster shell and a polymer in the Fall 1990 Lab Notes section (pp. 222–223). Earlier this year, the West Coast laboratory received nine loose, undrilled pearls for identification, along with some beads used in their culturing that were represented to be dolomite (figure 8).

The beads, about 8 mm in diameter, were white and translucent. They had an aggregate structure, but the surfaces were poorly polished and the refractometer showed only a birefringence blink. They fluoresced a weak white to both long- and short-wave UV radiation, and a weak pinkish orange to X-rays. The specific gravity was measured hydrostatically at 2.84. These properties are consistent with dolomite, but without a refractive index reading they do not conclusively identify the material.

We next turned to several methods of advanced testing to gain additional information on the beads. EDXRF analysis showed the presence of both calcium and magnesium, which is also consistent with dolomite. The Raman spectrum matched our reference spectrum for dolomite, but the Raman spectra for dolomite and magnesite are very similar. An X-ray diffraction pattern confirmed the identification as dolomite.

The client was able to share some information regarding the dolomite beads and the pearls cultured on them. The beads are fashioned in South Korea, but our client did not know the source of the dolomite rock itself. These nine pearls were cultured in Japan to test whether dolomite works well as a bead nucleus for cultured pearls; during this test, it was found that the overall yield was about the same as for pearl culturing using freshwater shell beads. Dolomite is of interest for pearl nuclei because it is quite easy to obtain in sizes large enough to fashion beads up to 20 mm in diameter, whereas the traditional freshwater shell used for culturing rarely grows to such a thickness.

The nine samples ranged in size from 10.75 × 10.30 mm to 7.90 × 7.35 mm, and in color from white to gray to light yellow. On exposure to X-rays, they fluoresced a very weak orange; as is the case with other cul-

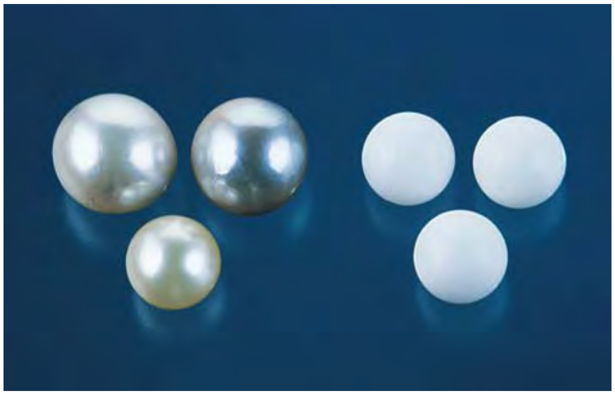


Figure 8. The pearls on the left, which range from 10.75 × 10.30 mm to 7.90 × 7.35 mm, were cultured on white dolomite beads similar to the ones shown on the right.

Figure 9. In this X-radiograph, the pearls cultured on dolomite beads have a very different appearance from the Tahitian black cultured pearl placed in the center as a reference.

tured pearls, this X-ray luminescence comes from the bead nucleus. The X-radiograph showed all nine to be cultured pearls, but the nuclei in these pearls looked distinctly different from the shell beads typically used to culture pearls, as illustrated in figure 9. The dolomite nuclei appeared darker (more transparent to the X-rays), and some of them had a mottled appearance. One sample showed a thick dark layer of conchiolin at the surface of the bead, which gave a silvery appearance to the pearl overall.

CYW and IR

Cultured, with Treated Black Color

The strand of fairly large black pearls shown in figure 10 was brought to the West Coast lab for identification. The strand consisted of 34 "circled" drop-shaped and oval pearls, ranging from approximately 13 × 10 mm to 10 × 9 mm, which were predominantly bluish black. However, some of the pearls also showed distinct green, and even violet, overtones. Because the pearls had the "metallic" appearance that is usually associated with irradiated freshwater tissue-nucleated cultured pearls (see Winter 1988 Lab Notes, p. 244), the client suspected that the color had been enhanced through irradiation.

X-radiography confirmed that the pearls were cultured. Visual examination of the pearl surface with 20× binocular magnification revealed that the color was not uniform or evenly distributed, but rather it was concentrated in dark reddish brown "spots" that gave the cultured pearls a peculiar speckled appearance (figure 11). This type of color distribution typically results not from irradiation, but from dyeing, which is commonly done with a silver nitrate solution. EDXRF analysis of the pearls revealed silver, which proved that these cultured pearls owed their black color to dye, not radiation.

KH

Figure 10. The client suspected that this strand of black cultured pearls was colored by irradiation.

QUARTZITE, Dyed to Imitate Sugilite

The 1.12 ct purple cabochon shown in figure 12 was one of a group of five submitted to the West Coast lab for identification. The purple color was variegated and within the range seen commonly for sugilite (see J. Shigley et al., "The Occurrence and Gemological Properties of Wessels Mine Sugilite," *Gems & Gemology*, Summer 1987, pp. 78–89). However, this cabochon served as a reminder that the gemologist must examine all the properties and make sure they form a consistent picture to arrive at a correct result.

The material appeared translucent and showed an aggregate structure. We obtained a spot refractive index reading of 1.55. While the R.I. reported for sugilite is 1.607–1.610, the aforementioned article and the *Gem Reference Guide* (GIA, 1990, p. 235) warn that because gem-quality sugilite may contain quartz impurities, the refractive index reading can be around 1.54. The desk-model spectroscope showed a broad band from 540 to 580 nm. Purple sugilite has a characteristic spectrum—with lines at 411, 420, 437, and 445 nm, and a band at 550 nm—but Webster's *Gems* (4th ed., Butterworths, 1983, p. 359) states that these lines in the violet end are difficult to see without using a very bright light and a blue filter. However, the specific gravity, measured hydrostatically, was 2.64. This value is identical to that expected for quartz, but is quite low for sugilite, which is normally 2.74 even if it contains some quartz.

The combination of refractive index and specific gravity indicated a quartz aggregate. Examination with magnification revealed that this color was concentrated around and between the grains of the aggregate, which is diagnostic for dyed material (figure 13). Quartz (like sugilite) is usually inert to both long- and short-wave UV, but this cabochon fluoresced a weak greenish yellow, with a somewhat stronger reaction to long-wave UV. This fluorescence may well have been due to the purple dye.

Figure 11. Examination with magnification of the cultured pearls in figure 10 revealed the spotty color distribution that is characteristic of dyed black pearls. Magnified 20×.

Figure 12. This 1.12 ct purple cabochon looks very much like sugilite.

Figure 13. Closer examination of the cabochon shown in figure 12 revealed the characteristic appearance of a dyed aggregate material, in this case quartzite. Magnified 30×.

This material looks like "purple onyx," also a dyed purple quartzite, which we discussed in the Winter 1990 Gem News section (p. 309). However, the particular shade of purple is different (this one is more reddish), as are the fluorescence and spectrum. Also in Gem News (Summer 1991, pp. 122–123), we described dyed purple quartzite that imitated dyed lavender jadeite.

IR and MLJ

An Unusual SAPPHIRINE

Not long ago, a client told us about an 8 ct "idocrase" from the new alluvial gem deposits in Tunduru, Tanzania. We could not recall seeing idocrase from that area, so we readily accepted the opportunity to examine the stone shown in figure 14.

The 8.64 ct cushion mixed cut was a moderately dark brownish orangy red. Microscopic examination revealed several long, thin needles scattered throughout the stone and a plane of crystals at one end. We measured refractive indices of 1.700–1.707, giving a birefringence of 0.007, and we saw a biaxial figure in the polariscope. The specific gravity was 3.43, measured by the hydrostatic method. The stone was inert to long-wave UV and weakly fluoresced a chalky green color to short-wave UV. The refractive index and specific gravity were consistent with idocrase, but idocrase is uniaxial (although rarely it is anomalously biaxial).

However, the stone also displayed strong trichroism: brownish red, light orange, and colorless. Idocrase usually displays only weak pleochroism (and only two colors would be expected). Examination with a desk-model spectroscope did not reveal the band at 461 nm typical

Figure 14. This 8.64 ct brownish orangy red cushion mixed cut from Tunduru, Tanzania, turned out to be the largest sapphirine ever seen at the GIA Gem Trade Laboratory.

for idocrase (described in Webster's *Gems*, 4th ed., Butterworths, 1983, pp. 329–330), but instead it showed sharp lines in the red portion of the spectrum. The spectroscope lamp also revealed moderate red transmission (red fluorescence to visible light), a property shown by many gems that are colored by chromium.

It was apparent that further investigation was necessary, so we turned to the Raman microspectrometer. We were quite surprised when the spectrum obtained perfectly matched our reference spectrum for sapphirine. We had fallen into one of the oldest traps in gem identification: We had not considered this possibility because the stone was almost three times the size of the largest faceted sapphirine we had ever heard of. We were also not aware of any chrome-bearing sapphirine, or one of this color. An X-ray diffraction analysis confirmed the stone's identity. Since it was such an unusual stone, we also examined the chemistry by EDXRF. As expected from the formula $[(Mg,Al)_8(Al,SiO)_6O_{20}]$ for sapphirine, Mg, Al, and Si were present as major elements; minor amounts of Cr, Ca, Ti, Fe, Ni, and Ga were found as well. Because idocrase is a calcium-magnesium aluminosilicate, the EDXRF result alone would not have identified this gem.

Thirteen years ago, we encountered another sapphirine that was similar in some respects to the current stone (Fall 1985 Lab Notes, pp. 176–177). That stone was described as purplish pink and weighed 1.54 ct. It had nearly identical optical properties, which are at the low end of the reported ranges for this mineral. To the best of our knowledge, this 8.64 ct sapphirine is the largest sapphirine that has been reported to date. It is also the only one we know of that has been reported from Tunduru. *SFM*

PHOTO CREDITS

Nicholas DelRe photographed figures 2, 3, 4, and 6. Maha DeMaggio took photos 1, 5, 7, 8, 10, 12, and 14. The X-radiograph in figure 9 was produced by Karin Hurwit. John Koivula provided figures 11 and 13.