

LAB NOTES

Editors

Thomas Moses, Ilene Reinitz, and
Shane F. McClure
GIA Gem Trade Laboratory

Contributing Editors

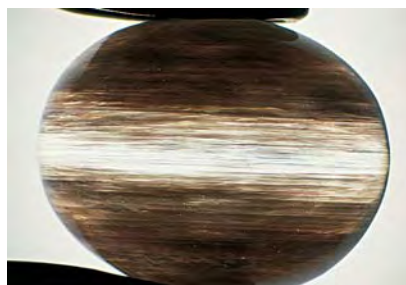
G. Robert Crowningshield
GIA Gem Trade Laboratory, East Coast
Karin Hurwit, Mary L. Johnson,
and Cheryl Y. Wentzell
GIA Gem Trade Laboratory, West Coast

BERYL with Unusual Eye

The West Coast lab recently examined what at first appeared to be a brown chatoyant cabochon. The refractive indices of 1.577–1.583, uniaxial negative optic character, and specific gravity of 2.72 (measured hydrostatically) identified the material as beryl. In transmitted light, the overall brown color of the 8.80 ct cabochon was clearly banded with thin brown layers oriented parallel to the length of the cabochon (figure 1). The stone showed no spectrum (when viewed with a desk-model spectroscope) or fluorescence; its pleochroic colors were brown and black.

Observation with magnification revealed finely laminated twinning planes throughout the stone (figure 2).

Figure 1. This 8.80 ct brown cabochon is natural beryl. Vertically oriented, densely included twinning planes with colorless zones in between form the apparent "eye." Transmitted light, magnified 10x.



Because of dense layers of brown inclusions along these planes, they appeared dark and translucent; however, the areas between the planes were colorless and transparent. These growth structures and inclusions not only proved that the beryl cabochon was natural, but they also were responsible for its unusual appearance.

When viewed through the flat back of the cabochon, the stone looked almost completely colorless; the refraction caused by the curved surface of the cabochon created the brown face-up appearance. The only area where the colorless layers could be seen through the top was at the apex of the cabochon. At this point, the vertical orientation of the twinning planes created a transmission "window" that allowed the viewer to see through the stone (again see figure 1). The colorless band along the top of the brown stone

gave the appearance of an eye. However, this "eye" moved across the cabochon only to a limited degree as the stone was rocked. This phenomenon would have to be referred to as a "pseudo-eye," since *chatoyancy* is defined as a product of reflection from oriented inclusions (see the *GIA Jeweler's Manual*, 3rd ed., 1989).

Because the "eye" was actually a zone where one could see through the stone, it assumed the color of the background over which it was placed. When the cabochon was viewed from the side, perpendicular to the twin planes, it showed a very weak diffused star. *SFM*

DIAMOND**Exhibiting Much Fire**

A friend of the East Coast lab recently showed us a 2.31 ct round brilliant (figure 3), which this diamantaire described as a "fireball," for possible use in our ongoing research into how cutting proportions affect the face-up appearance of a round brilliant cut diamond. The diamond exhibited a great deal of fire (the face-up expression of how a diamond disperses white light into its component colors) and showed strong contrasts of bright and dark

Figure 2. Dense layers of brown inclusions along lamellar twinning planes give this beryl its brown color. Magnified 15x.



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

Gems & Gemology, Vol. 35, No. 4, pp. 202–207

©1999 Gemological Institute of America



Figure 3. This 2.31 ct round brilliant diamond appeared very fiery when rocked in ordinary room light, or, as here, when illuminated with a narrow incandescent light source.

areas, especially when it was rocked from side to side. We recorded the following proportions: 34.1° crown angle, 42.4° pavilion angle (45.6% pavilion depth), 55% table size, 59.7% star-facet length, 85.3% lower-girdle-facet length, a fully faceted girdle varying from medium to slightly thick, and a small culet. The symmetry was very good; the primary faults were two flat spots in an otherwise round outline, and a slightly uneven crown height (i.e., the table was not parallel to the girdle). These proportions would receive a cut grade of 5 from AGS, midway between their best grade of 0 and their worst one of 10, and this combination produces a "typical" WLR value of 0.277. (For an explanation of WLR, see T. S. Hemphill et al., "Modeling the appearance of the round brilliant cut diamond: An analysis of brilliance," *Gems & Gemology*, Fall 1998, pp. 158–183.) IR

Identifying Treated-Color Rough with the Spectroscope

The GIA Gem Trade Laboratory has issued reports distinguishing between natural-color and treated-color yellow diamonds since the 1950s. The spectroscope is a necessary tool for this identification, as discussed in two landmark 1957 *Gems & Gemology* articles by G. R. Crowningshield ("An introduction to spectroscopy in gem testing," Summer 1957, pp. 46–55;

and "Spectroscopic recognition of yellow bombarded diamonds and bibliography of diamond treatment," Winter 1957, pp. 99–104). Despite technological advances in treatment methods, and the common use of spectrophotometers in today's gem testing labs, the spectroscope remains invaluable. This was clearly demonstrated by the case of the yellow octahedron shown in figure 4, which was submitted to the East Coast lab for color-origin determination.

The 13.12 ct crystal showed a variety of etched features, striations, trigons, channels, and cavities (see Yu. L. Orlov, *The Mineralogy of the Diamond*, John Wiley & Sons, New York, 1973, for illustrations), which were shiny and transparent, indicating that this was a natural diamond crystal. When the crystal was examined over diffused light, the bodycolor looked quite pale, and careful comparison of the appearance viewed from all sides showed one face to be somewhat more yellow than the other seven. A desk-model spectroscope showed a strong line at 415 nm, but no band at 478 nm; the latter is the most easily observed part of the "Cape" series spectrum that produces yellow in strongly colored yellow diamonds. Instead, there were strong lines at 496 and 503 nm, and a weak line at 595 nm. The diamond fluoresced a moderate chalky green-yellow to long-wave UV radiation; this was localized at the face that appeared darker in color. Weak orange fluorescence was observed with short-wave UV. This diamond crystal did not show any green luminescence to strong transmitted visible light.

A pair of lines in the hand spectroscope at 495 and 503 in a yellow diamond can indicate natural color, when the pair is associated with green "transmission" luminescence, but in the absence of this property they suggest laboratory treatment. Such treated-color diamonds usually also show a line at 595 nm and/or a pair of lines in the near-infrared region (A. T. Collins et al., "Spectroscopic studies of the H1b and H1c absorption lines



Figure 4. Examination with a desk-model spectroscope proved that this 13.12 ct diamond crystal was treated to enhance its yellow color.

in irradiated, annealed type-Ia diamonds," *Journal of Physics C: Solid State Physics*, Vol. 19, 1986, pp. 3933–3944). We checked the visible-range spectrum with a Hitachi U-4001 UV-visible spectrophotometer, and verified that the pair of lines seen in the hand spectroscope corresponded to the well-known bands associated with the H3 (503 nm) and H4 (496 nm) color centers. We also obtained both mid- and near-infrared spectra, which revealed a weak H1b line (4935 cm^{-1}) and a very weak H1c line (5164 cm^{-1}), and confirmed what the desk-model spectroscope had indicated (and the color and fluorescence zoning had suggested): The yellow color of this diamond crystal was due to laboratory irradiation and annealing.

IR

Yellow to Yellow-Green Diamonds Treated by HPHT, from GE and Others

Researchers from the Superabrasives division of General Electric Company (GE) recently announced a new product they have developed: strongly luminescent greenish yellow to yellow-green diamonds, or saturated yellow diamonds, that have been transformed from natural type I brown diamonds (J. Casey, pers. comm., 1999).

GE has indicated that, unlike most earlier treated-color diamonds in this hue range, the stones are not exposed to laboratory radiation; rather, GE uses



Figure 5. These representative greenish yellow to yellow-green diamonds, each approximately half a carat, were treated by a high-temperature, high-pressure process at GE Superabrasives.

high-pressure, high-temperature (HPHT) treatment. These are the same general conditions that GE researchers described in the Fall 1999 issue of *Gems & Gemology* (see the box titled "Research on diamonds at the General Electric Company," by T. Anthony and J. Casey, on page 15 of the article "Observations on GE-Processed diamonds: A photographic record," by T. M. Moses et al.). Although the exact conditions to which these colored diamonds are exposed have not been divulged, we believe that they are roughly similar to those used to remove color from type IIa diamonds.

Figure 5 shows the color range of this new product. These diamonds are quite similar in appearance and gemological properties to the luminescent "green transmitters" that we observed in the fall of 1996 and reported in the Summer 1997 Lab Notes section (p. 136). We thought at that time that the treatment was probably being done in Russia with the presses that are used

to grow synthetic diamonds, a suspicion that was later confirmed through conversations with a client.

Over the last several months, GIA researchers have also examined similar colored diamonds, also with strong green luminescence, that were loaned to us by Novatek Company (Provo, Utah). Novatek has recently begun to market this product under the name Novadiamond and plans a formal announcement to the trade at the February Tucson show. Novatek representatives have told us that these diamonds, too, are subjected to an HPHT process, for which a patent is pending. More recently, a New York client brought us yet another selection of similar treated-color diamonds from the Far East, reportedly treated in China (figure 6).

We have had the opportunity to test samples from each group—General Electric, Novatek, and reported Chinese—and our preliminary results for 47 of these diamonds show that their properties are similar to those described in the 1997 Lab Note mentioned above. The treated diamonds fluoresce a strong greenish yellow to long-wave UV, and generally a weaker greenish yellow to short-wave UV, with some localized chalkiness to both. For all three groups, the mid-infrared spectra showed that these were type I diamonds, with moderate to high amounts of nitrogen, and typically with more B aggregates than A aggregates (see, e.g., E. Fritsch and K. Scarratt, "Natural-color nonconductive gray-to-blue diamonds," *Gems & Gemology*, Spring 1992, especially pp.

38–39). Many of these diamonds showed well-defined color zoning, with brown to yellow octahedral graining observed when examined with diffused light. Excitation with a strong light source such as a fiber-optic illuminator causes these colored graining planes to luminesce bright green; this luminescence strongly contributes to their characteristic overall face-up greenish color.

Typical features observed with a handheld spectroscope in these HPHT treated-color diamonds are a weak absorption line at 415 nm, a pair of lines at 495 nm and 503 nm (which vary in strength from stone to stone), and emission lines at about 513 and 518 nm. This spectrum is not indicative of treatment for this color range, but it implies that the diamond requires further inspection. UV-Vis-NIR spectroscopy corroborated these absorption features, and showed the H2 peak (at 985 nm) in all of the samples that had a green component to the color. Magnification revealed that, like the treated-color diamonds described in 1997, a number of the samples from each source showed evidence of high-temperature exposure, such as frosted naturals, frosted feathers, or faceted girdles with bearding.

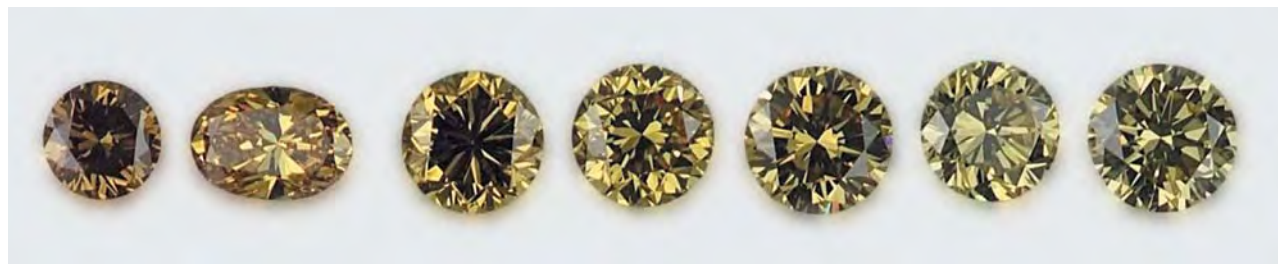
TM and IR

PEARLS

Freshwater Natural

The West Coast lab received two pearls, each approximately 13 mm in diameter, that a client had purchased some time ago in Hawaii. The client

Figure 6. This group of brownish yellow to greenish yellow diamonds, ranging from 0.22 to 0.39 ct, were reportedly treated by an HPHT process in China.



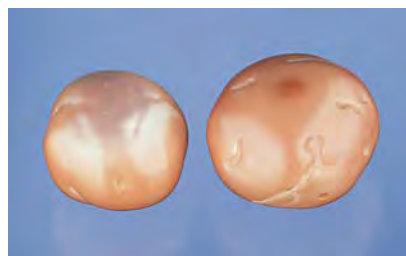


Figure 7. These brownish pink baroque pearls, each approximately 13 mm, proved to be of freshwater origin.



Figure 8. The fine suture lines on the side with high luster of each of the pearls in figure 7 indicated natural pearl formation. Magnified 20x.



Figure 9. The honeycomb structure of the dull side on each of the pearls in figure 7 is typical of the prismatic calcite layer seen in shells. Magnified 30x.

had been told that the pearls originated from the South Seas. A preliminary examination showed an appearance unlike the lustrous pearls that are commonly found in South Sea waters, but rather like that of calcareous concretions, or nonnacreous pearls. As can be seen in figure 7, they were uneven brownish pink in color and slightly baroque in shape.

It also was obvious to the unaided eye that each showed quite a range of luster: One side had a fairly high luster, and the opposite side was rather dull. Examination with magnification in reflected light revealed the fine suture lines (mottled surface texture) that are indicative of pearl formation (figure 8), whereas the duller area had the honeycomb structure usually seen in the prismatic calcite layer of shells (figure 9). The X-radiograph also showed structural features that indicate natural pearl formation. Much to our surprise, the pearls luminesced a weak yellowish white to X-rays. This confirmed that these natural pearls were of freshwater rather than saltwater origin. Even though we cannot prove their provenance, it is interesting to note that contributing editor G. Robert Crowningshield recalled having examined similar brownish pink natural pearls that had been brought from Pakistan for the New York World's Fair in 1964 [B. Anton, "Pink pearls of Pakistan," *Gems & Gemology*, Vol. 11, No. 6, 1964, pp. 175–179, 191].

KH

Notable Strand of Large Black Cultured Pearls

The East Coast lab recently received a strand of 23 round cultured pearls that graduated in size from 19.18 mm to 15.68 mm (figure 10). While we have tested larger individual black cultured pearls (Gem Trade Lab Notes, Winter 1987, pp. 234–235), these cultured pearls are notable for their consistently large size throughout the strand. The X-radiograph showed that they were bead nucleated, with nacre of varying thickness. Examination with long-wave UV radi-

ation revealed a weak reddish brown fluorescence in all of the cultured pearls, which proved that they were of natural color.

When we observed the strand in standardized lighting and viewing conditions, we described all of the cultured pearls as black using the Pearl Description System in GIA's 1999 *Pearls* course. Note that the GIA color description system represents a range of color sensations. Therefore, even though some of the cultured pearls in the strand displayed a subtle increase in saturation from neutral

Figure 10. Pictured next to a strand of typical-size 6.5–7.0 mm pearls, this strand of black cultured pearls, which graduate from 19.18 to 15.68 mm, is one of the largest we have encountered.



black toward greenish black, all of them still corresponded to the range described as black. The most noticeable variation in appearance within the strand was due to the presence of a pink overtone on some of the cultured pearls.

Akira Hyatt

SAPPHIRE

With a Valuable Inclusion

Many jewelers and gem dealers still tend to regard inclusions as mistakes of nature that diminish the quality of what otherwise might be a perfect gem. Now that the vast majority of rubies and sapphires are heat treated, a little inclusion can go a long way toward insuring that a fine gemstone is of natural color. Such was the case with a transparent 3.02 ct oval mixed-cut blue sapphire that was submitted to the East Coast lab for identification.

With near-monochromatic sodium-equivalent light, the stone showed refractive indices of 1.761–1.769. No fluorescence reaction was observed to either long- or short-wave UV radiation. A typical iron absorption spectrum was noted through a desk-model spectroscope. The presence of angular growth and color zoning, seen with a microscope, made it easy to determine that this sapphire was natural. The more difficult part of the identification was establishing whether the blue color

was of natural origin or the result of heat treatment. There was no evidence of heat treatment present, but the color zoning alone was not enough to prove natural color either.

A careful facet-by-facet search finally resulted in the discovery of a tiny (only 0.09 mm) transparent crystal. It was situated near the girdle edge on the pavilion side. When examined with polarized light, the inclusion exhibited birefringence (figure 11). There were no signs of any heat damage associated with this inclusion. It was so small, however, that its reaction to polarized light might have been anomalous. Only by identifying this inclusion could we determine conclusively if the host sapphire had been heat treated (from the expected reaction of the mineral to heat treatment).

Because the crystal was very close to the surface, we used laser Raman microspectrometry. We were able to obtain a clear Raman spectrum, which most closely matched the pattern shown by the mineral calcite, although other carbonates such as dolomite could not be ruled out. The identification of this transparent mineral inclusion as a carbonate proved that the host sapphire had not been heat treated to improve its color, because a carbonate such as calcite or dolomite would not have survived the high temperatures ($\geq 1500^{\circ}\text{C}$) commonly used to effect a color change in corundum.

In the case of this sapphire, an inclusion was an extremely valuable asset, because it enabled us to state on the report that the stone was of natural color.

John I. Koivula

With High Refractive Indices

A semi-transparent, brownish orange, rectangular cushion-shaped mixed-cut stone set in a yellow metal ring was recently submitted to the East Coast lab for identification (figure 12). The stone measured approximately 8x12 mm. With the microscope, we observed abundant “rust” red to dark brown and black tiny, skeletal crystal inclusions. These inclusions (figure 13) con-



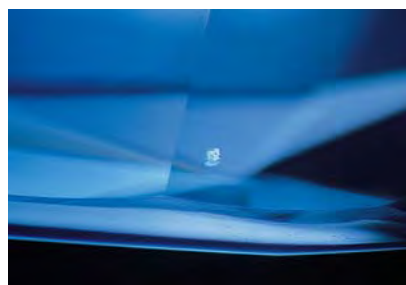
Figure 12. This 8 x 12 mm brownish orange stone proved to be a natural sapphire, but with unusually high refractive indices.

tributed to the bodycolor of their host as well as to its slight turbidity; they looked very much like the exsolution crystals of ilmenite and hematite often seen in similarly colored sapphires from the Umba River region in Tanzania. The simultaneous reflectance and iridescence of the inclusions showed that they were oriented in distinct

Figure 13. The numerous skeletal inclusions in this sapphire appeared to be responsible for at least part of the bodycolor of their host. Magnified 40x.



Figure 11. Measuring only 0.09 mm in largest dimension, this tiny carbonate inclusion proved that the host, a natural sapphire, had not been heat treated.



planes, which supported the possibility that these inclusions had exsolved from their host. After this initial phase of the examination, it seemed that the identification would be relatively straightforward, and we moved on to the next step, obtaining a refractive index reading.

The table facet was well polished, and raised above the mounting, so we anticipated no difficulty in measuring the R.I.'s accurately. Using a near-monochromatic sodium-equivalent light source, we obtained a reading of 1.770–1.779—a complete surprise. This is higher than any reading previously obtained in the Gem Trade Laboratory from any faceted sapphire of any color, with the exception of the diffusion-treated red corundum reported by S. McClure et al. ("Update on diffusion-treated corundum: Red and other colors," *Gems & Gemology*, Spring 1993, pp. 16–28). First we checked the refractometer carefully for damage to the hemicylinder contact surface, and then we inspected the table of the sapphire for cleanliness. Nevertheless, even with fresh R.I. contact liquid, the reading was the same.

We next tested two faceted flame-fusion synthetic sapphires from our reference collection, one blue and one orange, on the same instrument as controls. These samples showed refractive indices in the expected range of 1.762–1.770, confirming that the refractometer readings were accurate.

The mounted sapphire was inert to both long- and short-wave UV radiation. Using a Beck desk-top prism spectroscope, we saw an absorption band between 520 and 580 nm, a cut-off in the blue from 490 nm down, and another cutoff in the red from 660 nm up. While this spectrum is consistent with the brownish red color of the gemstone, it is not diagnostic. Laser Raman microspectrometry confirmed the identification of this stone as sapphire; no unusual features were noted in the Raman spectrum.

We next turned to energy-dispersive X-ray fluorescence (EDXRF) analysis to see if the chemistry offered any clues as to why the R.I. readings were unusually high. We analyzed the sapphire for both major and trace elements using an EDXRF Spectrace 5000 instrument. In addition to the

expected aluminum, the analysis showed iron, chromium, titanium, gallium, and vanadium. The strength of the iron peak as compared to the others suggested as much as 2 wt.% FeO. This amount of iron could account for the lack of fluorescence even with the presence of chromium and vanadium, as iron tends to quench fluorescence. The iron might also be responsible for the elevated R.I.'s. In *Gems* (4th ed., Butterworths, London, 1983, especially p. 91), Webster reports that some iron-rich green sapphires show elevated refractive indices of 1.77 and 1.78. While we have seen similar-appearing sapphires in the past with slightly elevated refractive indices, this is the first one we have encountered with this extreme reading.

John I. Koivula and Shane Elen

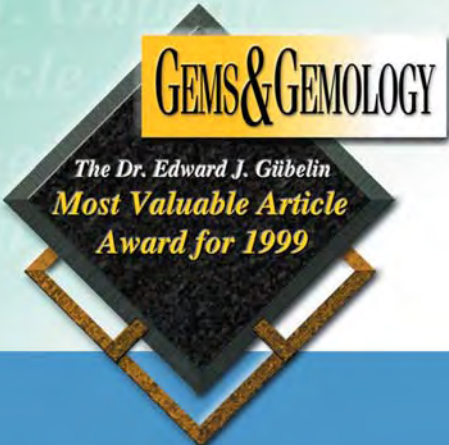
PHOTO CREDITS

Shane McClure took figures 1 and 2. Vincent Cracco and Elizabeth Schrader took figure 3.

Elizabeth Schrader was the photographer for figures 4 and 6. Maha Tannous provided figures 5, 7, and 12. John Koivula photographed figures 8, 9, 11, and 13. John M. King supplied figure 10.

Vote now and win!

Please see card between
pages 218 & 219 for ballot



Simply tell us which three 1999 articles you found most valuable, and you could win a FREE leather portfolio PLUS a five-year subscription to GEMS & GEMOLOGY. Mark the articles in order of preference on the enclosed ballot card. Then mail the card to arrive no later than March 15, 2000 and it will be entered in a drawing for the grand prize—

**A FREE leather portfolio PLUS
a five-year subscription to**

GEMS & GEMOLOGY

 **Send in your ballot today!**