

EDITORS

Thomas M. Moses and
Shane F. McClure
GIA Laboratory

DIAMOND

With Color Contributed by the 594 nm Center?

The 594 nm center is commonly observed in the absorption spectra of irradiated and annealed diamonds and also is detected occasionally in some naturally colored diamonds (see, e.g., T. W. Overton and J. E. Shigley, "A history of diamond treatments," Spring 2008 *Gems & Gemology*, pp. 32–55). Its occurrence is widely used as an identification feature for colored

diamonds; however, its contribution to the bodycolor of a diamond is not well recognized. Recently, in the New York laboratory, we examined a diamond with a strong orange color that was believed to be partially caused by the 594 nm absorption.

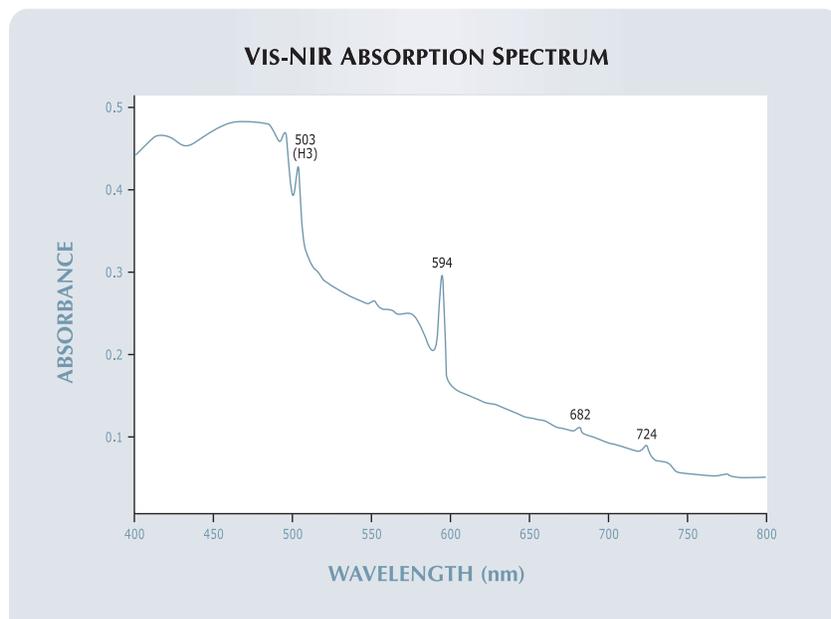
This 0.90 ct marquise-cut stone ($9.12 \times 5.19 \times 3.43$ mm) was color graded Fancy Deep brownish yellowish orange (figure 1). With magnification,

the color distribution appeared very slightly patchy, but we saw no other internal features (the clarity grade was VVS₂). When exposed to long-wave ultraviolet (UV) radiation, it displayed a moderately strong green fluorescence, with a weak yellow reaction to short-wave UV; no phosphorescence was observed. The DiamondView instrument revealed blue and greenish yellow fluorescing growth zones.

Figure 1. The deep orange color of this 0.90 ct treated diamond was likely influenced by a very strong 594 nm absorption.



Figure 2. The 594 nm absorption center, seen here in the Vis-NIR spectrum of the diamond in figure 1, is a common result of irradiation and annealing treatment. The very strong absorption in this diamond is likely a significant contributor to the orange bodycolor.



The absorption spectrum in the mid-infrared (IR) region revealed that this diamond contained a high concentration of nitrogen, as is typical of a type Ia stone. Moderately strong H1a, H1b, and H1c absorptions were also observed. The Vis-NIR spectrum showed a gradual increase in absorption toward the blue end of the spectrum (figure 2). In addition to weak N3 (415 nm) and very strong H3 (503 nm) absorptions, moderately strong absorptions were also observed at 682 and 724 nm. An outstanding feature was the extremely strong absorption at 594 nm and its possible side band at ~575 nm. All of these spectral features established that the color of this diamond was the result of irradiation and subsequent annealing.

The intensity of the 594 nm absorption is normally far less than that of the N3 or H3. This stone's strong selective absorption in the 550–600 nm region efficiently blocked yellow and some green light, making it very likely that the 594 nm absorption contributed significantly to the observed orange color. It is very rare to encounter a colored diamond in which the bodycolor has been influenced by this defect.

Wuyi Wang

Subtle Flash-Effect Colors in a Filled Diamond

Glass filling is often easily observed due to obvious flash-effect colors in a large feather or feathers. In some diamonds, though, the effect is not so easy to detect.

Earlier this year, a 5.01 ct pear-shaped brilliant was submitted to the New York laboratory for a full grading report. A few large feathers were noted, but none exhibited any flash-

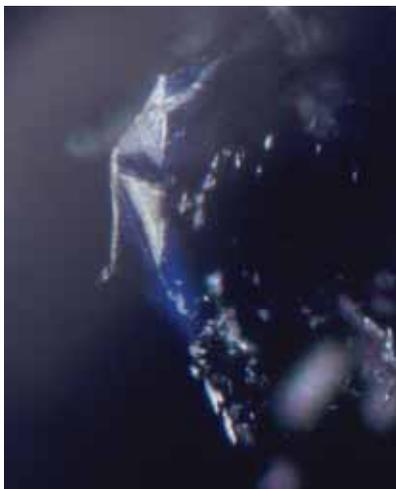


Figure 3. These subtle blue and pale purple flash-effect colors are indications that this diamond has been glass filled. Magnified 60 \times .

effect colors. However, closer scrutiny through the pavilion of the diamond revealed a small feather that had very subtle blue and pale purple colors (figure 3). Only one other small feather exhibited similar colors.

When a grader observes what is thought to be potential glass filling, the diamond is sent to the Gem Identification Department for further testing. Fractures that exhibit flash-effect colors are tested for the presence of lead, a major component of the filling material. This stone was tested by energy-dispersive X-ray fluorescence (EDXRF) spectroscopy, which confirmed the presence of lead.

It is GIA's policy not to grade diamonds treated by glass filling, even when only a small amount is noted.

Vincent Cracco and Paul Johnson

"Unidentified Clarity Characteristic" in Diamond

Professional diamond graders are taught to classify internal characteristics in diamonds using such dry descriptive terms as *crystal*, *pinpoint*, and *feather*. Due to time constraints, solid inclusions are rarely fully identified unless it is necessary for scientific reasons. Depending on their size, solids are gen-

erally described as either *pinpoints* or *crystals* for the purpose of assigning clarity grades. That said, from time to time we have reported on features in diamonds that captured our imaginations because they resembled familiar objects from our daily lives (e.g., Winter 2007 Lab Notes, pp. 363–364).

Occasionally, brightly colored mineral inclusions such as red and orange garnets and green diopside crystals will catch a diamond grader's attention simply because of their intense hues. More commonly, however, solid inclusions are essentially transparent and colorless, or opaque and black, and thus command little interest beyond their impact on the clarity grade.

Since diamond is composed of carbon, it stands to reason that one of the most commonly encountered dark inclusions in diamond is graphite, the hexagonal polymorph of carbon. True "carbon spots," most graphite inclusions appear as nondescript smudges in cleavage cracks or within the gaps between solid inclusions and the host diamond. Typically, these are considered undesirable features; occasionally, however, even opaque black graphite can create an interesting internal scene that captures the viewer's imagination.

One example of this was a tiny hexagonal platelet that appeared to be graphite, although it could not be analyzed because of its depth in the diamond. The platelet had formed aligned along an octahedral plane as graphite hexagons in diamonds commonly do, and it was skirted by a small cleavage disk caused by the expansion strain that occurred when the diamond in this spot was converted to graphite. Normally, graphite platelets are so thin as to be virtually invisible when viewed edge-on, but this particular inclusion had a very distinct depth to it, as it was made up of a stack of graphite plates of different diameters (figure 4). This assemblage caused it to resemble a tiny "flying saucer" of the type seen in 1950s science fiction movies. The fact that it was so small it appeared grainy even with the sharpest possible focus also enhanced the imagery. While diamond grading can

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Figure 4. Aligned along an octahedral plane in its diamond host, and measuring only 0.36 mm across, this minute crystal formation (probably graphite) is reminiscent of the “flying saucers” often featured in early science fiction movies and grainy photos purported to be of such phenomena.

become routine, inclusions are occasionally fun, and this “unidentified clarity characteristic” provides an excellent example of an inclusion that stimulates the imagination.

John I. Koivula and Karla F. Onstott

Gem-quality CVD SYNTHETIC DIAMOND with Traces of Boron

Several years after it was first announced, gem-quality chemical vapor deposition (CVD)-grown synthetic diamond is still encountered only rarely among the stones submitted for a GIA diamond report (e.g., Spring 2008 Lab Notes, pp. 67–69). Recently, a brown round cut submitted to the New York laboratory was identified as a CVD synthetic diamond with some unusual spectroscopic features.

This 1.25 ct sample ($6.82 \times 6.97 \times 4.20$ mm) was color graded Fancy brown (figure 5). It showed very slightly patchy color distribution and strong graining with magnification. Several fractures were present along the girdle region. It fluoresced very weak yellow to long-wave UV radiation and weak orange to short-wave UV; no phosphorescence was observed. The DiamondView reaction was dominated by mod-

erately strong orangy red fluorescence with a notable irregular blue region (figure 6); a moderately strong blue phosphorescence was also observed. These luminescence features are typical for CVD-grown diamonds (see Wang et al., “Latest-generation CVD-grown synthetic diamonds from Apollo Diamond Inc.,” Winter 2007 *Gems & Gemology*, pp. 294–312).

The infrared absorption spectrum revealed that this diamond contained no detectable nitrogen, which is not unexpected for CVD synthetic diamonds (most of which are nominally type IIa), but we did observe weak absorptions at 2925 and 2800 cm^{-1} from trace amounts of boron. This is the first type IIb CVD diamond we have identified in the lab. Unlike the latest CVD-grown diamonds from Apollo Diamond with strong brown color (again, see Wang et al., 2007), this synthetic diamond showed no absorption in the 1500–1300 cm^{-1} region. We did observe weak absorptions at 7353, 6856, 6425, and 5564 cm^{-1} in the near-infrared region. The Vis-NIR spectrum, collected at liquid-nitrogen temperature, showed a gradual increase in absorption toward the low-wavelength side (figure 7). In addition to a very strong absorption from the Si-V defect (737.0 nm)—an outstanding feature of this diamond—a weak peak at 596.5 nm and a

Figure 5. This 1.25 ct Fancy brown synthetic diamond proved to be CVD grown, but it had some unusual spectroscopic features.

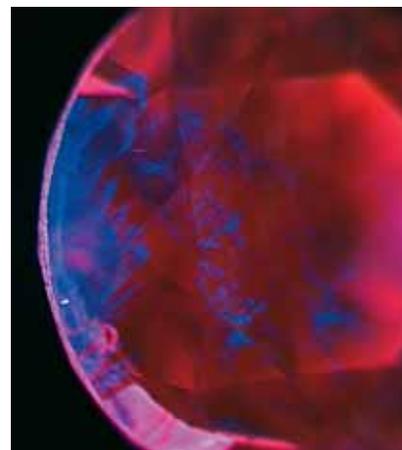


Figure 6. In the DiamondView, the synthetic diamond in figure 5 is dominated by moderately strong orangy red fluorescence with a large irregular blue region. This luminescence is similar to that seen in products from Apollo Diamond Inc.

broad band centered at ~ 520 nm were also observed. The assignment of these peaks is not clear, but it is obvious that the trace boron in this diamond does not contribute much to the bodycolor of this stone. Si-V is a common defect in CVD synthetic diamond; however, its concentration is usually extremely low and only rarely can it be detected with absorption spectroscopy. The photoluminescence spectrum (514 nm excitation), collected at liquid-nitrogen temperature, displayed strong emissions from N-V centers (zero-phonon lines [ZPL] at 575 and 637 nm) and from the Si-V defect (doublet at 736.6 and 736.9 nm). Weak but clear emissions at 596.5 and 597.1 nm were also recorded.

P. M. Martineau et al. (“Identification of synthetic diamond grown using chemical vapor deposition [CVD],” Spring 2004 *Gems & Gemology*, pp. 2–25) described gem-quality boron-doped type IIb CVD synthetic diamonds, but the gemological and spectroscopic features of their (blue) samples were very different from those of the 1.25 ct brown CVD synthetic we examined. Whether the boron in this diamond was doped intentionally or

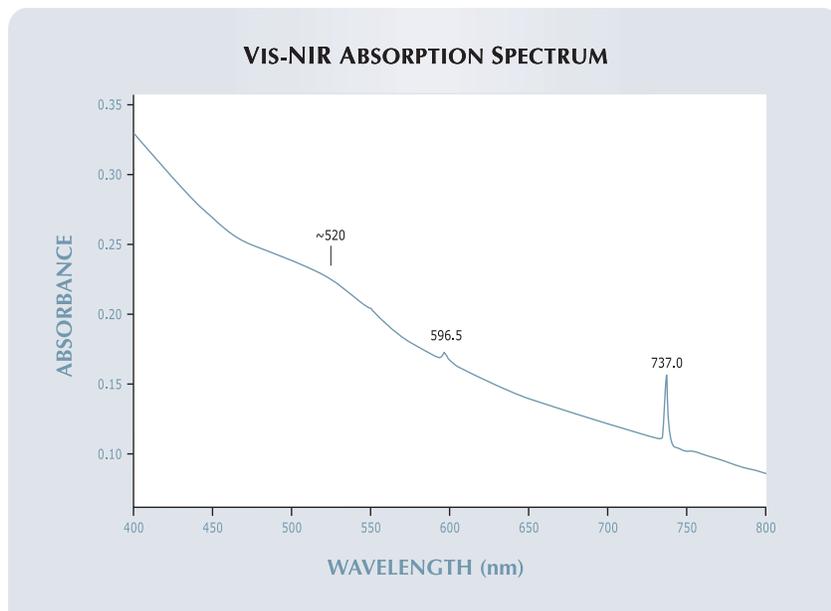


Figure 7. An outstanding feature of the brown CVD synthetic diamond is a very strong absorption from the Si-V defect (ZPL at 737.0 nm) in the Vis-NIR spectrum. This defect has only rarely been detected in the absorption spectra of CVD-grown diamonds.

incorporated accidentally is unclear.

All these absorption and luminescence features established that this was an as-grown CVD synthetic diamond, though with some unusual features compared to previously reported material. These include its relatively large size (1.25 ct), its normal cutting proportions, the fact that it contained trace boron, and its extremely high concentration of Si-V defects.

Wuyi Wang and Thomas M. Moses

Bleached *Pinctada Margaritifera* CULTURED PEARLS in Non-“Chocolate” Colors

The bleaching of *P. margaritifera* cultured pearls to achieve a brown color has become a popular and widely disclosed treatment (see W. Wang et al., “Identification of ‘chocolate pearls’ treated by Ballerina Pearl Co.,” Winter 2006 *Gems & Gemology*, pp. 222–235). Over the last year, however, the New York laboratory received for identification two strands of cul-

tured pearls (which proved to be from the *P. margaritifera* mollusk) that featured colors other than brown that showed evidence of bleaching.

The cultured pearls in the first

Figure 8. The cultured pearls (10.50–13.50 mm) in this variously colored strand are from the *Pinctada margaritifera* mollusk. All except the greenish ones showed evidence of bleaching.



strand (figure 8) had good luster, moderately spotted surfaces, and good matching. Those in the second strand (figure 9) had very good luster, lightly spotted surfaces, and very good matching. The color variation in the second strand was broader and more pronounced. In addition to “chocolate pearls,” both strands contained cultured pearls in an assortment of atypical colors. Many of the grays and greens were uncharacteristically muted in tone and saturation, and there were a number of unusual light-toned yellow-browns or brown-yellows. Overall, these cultured pearls were lighter in tone and warmer in hue than most *P. margaritifera* pearls, but darker and cooler than most of those from the *Pinctada maxima* mollusk. In most of the cultured pearls, magnification revealed evidence of bleaching, including slightly desiccated patches and color distribution (color concentrations and streaks) typical of Tahitian cultured pearls that have been bleached brown, with similar though subtler color variegation in some colors.

Exposure of the second strand to long-wave UV radiation also produced some unusual results. The tone and intensity of the reaction was directly related to the tone and saturation of the color in the cultured pearls:



Figure 9. All but two of the cultured pearls (11.00–13.50 mm) in this second strand from *P. margaritifera* also showed evidence of bleaching.

Lighter samples fluoresced weak-to-moderate yellow, and dark neutrals produced weak dark or inert reactions. Additionally, the reactions varied in hue throughout the strand, and they differed significantly from the inert to weak reddish brown reaction normally produced by naturally colored *P. margaritifera* pearls. A number of samples showed yellow, orange, or reddish orange reactions, and one fluoresced a distinct green-yellow.

EDXRF analysis of all the cultured pearls in the second strand detected calcium and strontium but no silver, which indicated that they were not dyed. UV-Vis reflectance spectroscopy revealed evidence of bleaching in the browns and all but two of the other colors. The background tilted toward the high-energy side, and the organic peaks were weak and broad (see, e.g., figure 9 of Wang et al., 2006, p. 228). While the level of treatment applied to the other colors visually appeared less pronounced than what is often found in “chocolate pearls,” UV-Vis reflectance spectroscopy did not produce any results to corroborate this.

The popularity of “chocolate pearls” seems to have led manufacturers to extend a similar bleaching treatment to cultured pearls of other colors.

Akira Hyatt

Blue-Green PLAGIOCLASE FELDSPAR, Possibly Colored by Copper

Blue-to-green potassium feldspar, known in the trade as *amazonite*, owes its color to a combination of natural irradiation and trace concentrations of lead and water (A. M. Hofmeister, and

G. R. Rossman “A spectroscopic study of irradiation coloring of amazonite: Structurally hydrous, Pb-bearing feldspar,” *American Mineralogist*, Vol. 70, 1985, pp. 794–804). The Carlsbad lab recently received for identification two opaque bluish green rocks (figure 10) measuring 46.90 × 35.18 × 21.38 mm and 45.38 × 24.05 × 21.69 mm that, on initial observation, appeared to be amazonite. On closer inspection, however, the rocks lacked the mottled grid-like color patterns (exsolution lamellae, caused by the intergrowth of microcline and albite) that characterize amazonite. Instead, the structure was massive and fairly uniform.

On the exterior of the rocks was a brownish crust, beneath which were layers and veins of a darker bluish green material. A rough RI measurement of 1.54 suggested a plagioclase feldspar (oligoclase). Laser ablation–inductively coupled plasma–mass spectrometry (LA-ICP-MS) and electron microprobe analyses confirmed that the composition fell within the albite-oligoclase range. Very little potassium was detected; nor was lead present in significant concentrations (<4 ppm). Instead, the

Figure 10. Chemical analysis of these two samples of bluish green plagioclase feldspar (45.38 and 46.90 mm in longest dimension) suggested that they were colored by copper.



analyses showed high levels of copper (up to ~12,000 ppm). Great care was taken during analysis to make sure that none of the surface minerals were mixed with the feldspar.

The client, Daniel Anderson, was kind enough to loan us several additional pieces of rough for testing, and these samples reinforced our earlier observations and analyses. Mr. Anderson plans to polish this plagioclase feldspar and introduce it to the market in the near future. He reports that it occurs in altered pegmatites of Jurassic age (~150–200 million years) in northern Nevada. Further testing is currently being performed to determine the extent to which copper might be responsible for the color.

It is quite unusual to come across feldspar with such a distinct bluish green color that is not an alkali (potassium) feldspar colored by lead.

*Alethea Inns and
Christopher M. Breeding*

Unusual Green Fluid Inclusions in QUARTZ

Petroleum is one of the most important substances on Earth. Civilization as we know it would cease to exist without natural petroleum and the products derived from it. Because of this, fluid inclusions containing natural petroleum have fascinated geologists, gemologists, and other earth scientists for decades.

Hydrocarbons usually form primary fluid inclusions composed of liquid natural petroleum and methane gas, with water sometimes also present as an immiscible secondary liquid phase (see, e.g., Spring 2004 Gem News International, pp. 79–81). Opaque black to dark brown translucent bituminous material, identified under the general heading *asphaltite*, may also be present as a solid daughter phase. The liquid petroleum portion is generally yellow in color, but it may range from nearly colorless to deep golden brown to pinkish orange (similar to the color of gasoline). If present, water will be forced to the outermost edges of the void, and the

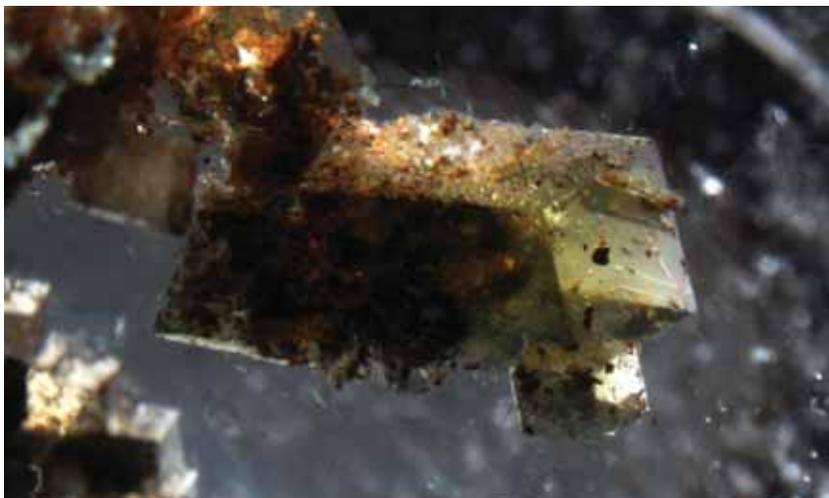


Figure 11. The primary petroleum fluid inclusions in the trigonal cavities of this quartz had an unusual green color that was not the result of either visible-light luminescence or UV fluorescence. Field of view 4.4 mm.

vapor bubble (again, usually methane) will always be in the liquid petroleum portion and not in the water.

An interesting identifying characteristic of petroleum in fluid inclusions is that when it is illuminated from the side with an incandescent fiber-optic light source, it displays a slight bluish green to green luminescence. This reaction is different from the long-wave UV fluorescence it also displays—commonly a chalky blue to yellow, or very rarely orange.

Recently, Kevin Lane Smith, a jeweler and lapidary from Tucson, Arizona, showed this contributor a polished quartz specimen that, without magnification, appeared to contain a few small light green angular crystals. On closer inspection, however, it was determined that the green material was actually a liquid filling small trigonal cavities (figure 11). Detailed micro-examination showed that the inclusions were intact with no surface-reaching cracks. Exposure to UV radiation caused the liquid to fluoresce a chalky blue, and light transmitted through the side of the stone caused the fluid inclusions to luminesce a chalky bluish green, both as would be expected of natural petroleum (again, see Spring 2004 GNI entry). This

behavior strongly suggested that these fluid inclusions were composed of natural petroleum. The unusual green color did not appear to be caused by luminescence or UV radiation, however, since it was clearly visible in dark-field illumination without any overshadowing chalkiness.

John I. Koivula

Inclusions within Inclusions in RUBY

The New York laboratory recently had the opportunity to examine an attractive 2.06 ct red oval mixed-cut ruby (figure 12) with a highly interesting inclusion scene. Standard gemological testing identified the stone as a natural ruby. With magnification, we saw numerous transparent near-colorless crystals. The pristine state of the inclusions indicated that the stone had not been heat treated. What was most interesting was that several of the crystals clearly had inclusions of their own.

The crystal in figure 13 contained a rod-shaped inclusion that appeared to break through another crystal, both showing a multistep surface. All the elements in this inclusion exhibited birefringence between crossed



Figure 12. This untreated natural ruby (7.76 × 6.32 × 4.94 mm) contained some unusual crystal inclusions.



Figure 13. Hosted within this pristine crystal in the ruby in figure 12 were rod-shaped crystals that still retained their original crystal faces. Field of view 0.76 mm.



Figure 14. Another included crystal (identified as corundum) in the ruby contained a fan-shaped spray of needles. Field of view 0.63 mm.

polarizers. Unfortunately, we were not able to obtain sufficiently clear Raman spectra to identify them.

Other attractive inclusions were discovered within a crystal that was visible through the pavilion. Located between two cavities in the host ruby, the crystal displayed a nicely formed fan-shaped spray of needles (figure 14). The transparent-to-translucent needles appeared unaltered and also exhibited birefringence. We were able to identify the host crystal as corundum, but the Raman laser could not reach the needles. According to GIA chief gemologist John Koivula, these needles were probably boehmite or diaspore, both of which are alteration products of corundum.

Their complete enclosure in the crystal inclusions indicates that the unaltered rod-shaped crystals and the fan-shaped spray of needles could have formed before the growth of the host ruby. Thus, these inclusions could be defined as both protogenetic (pre-existing inclusions) and idiomorphic (still retaining their original crystal faces).

Even though mineral inclusions in corundum have been studied extensively, none of us at the New York lab had previously seen such well-formed “inclusions within inclusions.”

Wai L. Win and Riccardo Befi

Blue SAPPHIRE, With Unusually High Concentration of Rare-Earth Elements

Common chemical impurities in natural corundum include Mg, Ti, V, Cr, Fe, and Ga. A number of colors can be induced by these elements through various mechanisms as they integrate into the corundum lattice (see, e.g., E. Fritsch and G. R. Rossman, “An update on color in gems, part 2: Colors

involving multiple atoms and color centers,” Spring 1988 *Gems & Gemology*, pp. 3–15). Be has also been found in natural corundum from several geographic sources in trace amounts ranging from less than 1 ppm to over 10 ppm; other trace elements (e.g., Nb, Ta, U, Pb, Th, Zr, or W) are almost always present with naturally occur-

Figure 15. This blue sapphire (5.95 × 5.91 × 3.52 mm) has an unusual trace-element composition: relatively high concentrations of light rare-earth elements.

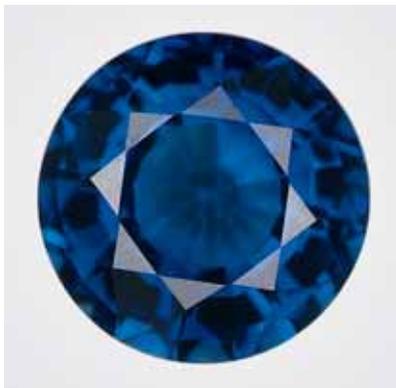


Figure 16. Three spots on both blue and near-colorless zones in the girdle region of the blue sapphire in figure 15 were analyzed using LA-ICP-MS, but no substantial variation in trace-element composition was detected. Field of view 3.5 mm.



TABLE 1. Trace-element composition of the unusual 1.02 ct blue sapphire.^a

Element	Mg	Ti	V	Cr	Fe	Ga	Zr	Nb	La	Ce	Nd	Sm	Ta	W	Pb	Th	U
Concentration (ppm)	19.11	195.4	3.15	2.87	3887	59.95	0.05	0.11	0.66	1.55	0.29	0.02	0.26	0.12	0.37	4.96	0.01

^aAverage of three spots, as recorded with LA-ICP-MS. Rare-earth elements are highlighted in blue.

ring Be (A. H. Shen et al., “From the GIA Laboratory: Beryllium in corundum—The consequences for blue sapphire,” *GIA Insider*, Vol. 9, No. 2, January 26, 2007). However, these elements—in contrast to the substitution impurities noted earlier—are believed to occur within clouds of submicroscopic particulate inclusions, not as part of the crystal lattice. In the New York laboratory, we recently examined a sapphire that displayed atypical trace-element composition—specifically, relatively high concentrations of other rare-earth elements (REEs) that appear to be independent of submicroscopic “clouds” or inclusions.

This intense blue 1.02 ct round-cut stone (figure 15) was identified as natural sapphire by standard geological testing. With magnification, it exhibited sharply defined blue and near-colorless parallel bands (figure 16). Small melted inclusions with altered or melted radial expansion fractures were

also observed, indicating the stone was heat treated. No clouds or bands of particles were visible at 80× magnification, and there was no detectable chalky yellow fluorescence to short-wave UV radiation, as is sometimes seen in heated blue sapphire.

Three spots along the girdle region (again, see figure 16) were analyzed using LA-ICP-MS, calibrated against corundum standards for Be, Mg, Ti, V, Cr, Fe, and Ga, and against National Institute of Standards and Technology (NIST) glasses for other elements. The blue and near-colorless zones showed similar trace-element composition. Consistent with blue sapphire from many different sources, this stone had a high concentration of Fe, moderate amounts of Ga and Ti, and very low concentrations of Mg, V, and Cr (table 1). An outstanding feature was the relatively high concentration of light REEs (La, Ce, Nd, Sm), which we have observed only rarely in the thousands

of natural corundum samples we have examined from various geographic sources. In addition, despite significant amounts of Zr, Nb, Ta, W, Pb, Th, and U, no Be was detected (our instrument detection limit for Be is 0.01 ppm).

Observations from this unusual stone strongly indicate that microinclusions in corundum may have large variations in composition, including (to the best of our knowledge) previously undocumented rare-earth elements. This stone also confirms that Nb and Ta may occur independently of Be in sapphire.

Donna Beaton and Wuyi Wang

PHOTO CREDITS

Jian Xin (Jae) Liao—1, 5, 8, 12, and 15;
Vincent Cracco—3; John I. Koivula—4 and 11;
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