Large CORAL Bead Necklace

Sought after as a gem material for more than 2,000 years, today coral is one of the most popular organic gems in the marketplace, next to pearls ("Coral: More than seasonal," *The Guide*, May/June 2003, pp. 7–8, 13). Coral's enduring appeal may be due to its broad range of color saturation, from vivid "ox-blood" red, through the softer pinkish orange of "salmon," further to the pale pink of "angel skin," and beyond even that to white. Combined with a Moh's hardness of 3.5, it is not surprising that coral is a favorite carving material for use as *objets d'art* and in jewelry, as well as fashioned into cabochons and beads.

The coral used as a gem material is actually the accumulated calcium carbonate secreted by colonies of tiny anemone-like sea animals. Coral polyps are fairly delicate sea creatures, sensitive to changes in water depth, temperature, and clarity, as well as to modern hazards such as pollution and overfishing (see, e.g., R. Webster, *Gems*, 5th ed., rev. by P. G. Read, Butterworth-Heinemann, Oxford, 1994, pp. 559–564). These modern hazards and quotas imposed to protect endangered corals have led to shortages, particularly of the finer material.

The East Coast laboratory was therefore very fortunate to have the opportunity to identify a necklace containing 11 large round pinkish orange beads that were graduated in size from 34.45 to 24.45 mm (figure 1). All had a high polish, and only a few contained minor blemishes, which were almost hidden close to their drill holes. Each bead displayed coral's classic wavy parallel fibrous structure. Standard gemological testing identified the beads as coral, and swabbing with acetone in tiny inconspicuous places did not reveal any dye. Fourier-transform infrared (FTIR)

Figure 1. The 11 coral beads in this necklace, which graduated in size from 34.45 to 24.45 mm, were found to be untreated.

spectroscopy found no evidence of polymer impregnation. Thus, we concluded that these beads were, in fact, of natural color and not impregnated—an impressive result for such a well-matched, highly polished set of beads this size. Alternating with yellow metal spheres pavé set with numerous transparent yellow round brilliants, these coral beads were the highlight of a stunning necklace.

Wendi M. Mayerson

DIAMOND

Four Blue Diamonds from a Historic Necklace

The Cullinan blue diamond necklace (figure 2) holds a special place in the history of African diamond mining. While its classic style, workmanship, and rare blue diamonds have made it famous, it started as a personal memento—a gift to celebrate a special event. This Edwardian gold back, silver top, festoon necklace was presented by Thomas Cullinan, then chairman of the Premier mine, to his wife Annie in 1905 to commemorate the gift of the 3,106 ct Cullinan diamond to England's King Edward VII and Cullinan's subsequent knighthood. Mr. Cullinan could not have known at the time that blue diamonds such as those in the necklace would one day become as synonymous with the Premier (recently renamed Cullinan) mine as the famous large colorless crystals it has produced. Nor would he have known that blue diamonds would remain rare and among the most highly valued of all diamonds.

The exhibition of the Cullinan blue diamond necklace at GIA's museum in Carlsbad this fall presented a welcome opportunity to examine the necklace for the first time in many years, and to do so in detail for the first time ever. For this occasion, the current owner requested that detailed gemological information accompany the exhibition of the piece so as to better inform the public about this unique necklace. For our examination,

Figure 2. The Cullinan blue diamond necklace dates back to 1905 and contains several rare type IIb blue diamonds, the largest of which is 2.60 ct. Courtesy of S. H. Silver Co., Menlo Park, California.

the four principal blue diamonds (an oval shape weighing 2.60 ct—referred to as the Cullinan blue diamond—and three Old European cut brilliants weighing 0.75 ct, 0.73 ct, and 0.42 ct) were removed from the mounting for grading (figure 3), and the necklace itself was given a thorough inspection. Three of the four blue diamonds (all except the 0.42 ct) had been previously graded by GIA, most recently in 1993. That grading took place prior to the 1995 enhancements to GIA's colored diamond color grading system. Additionally, the 0.75 ct and 0.73 ct Old European brilliants had been graded in the mounting, which did not allow a precise assessment. Thus, this was an important opportunity to review the grading of these diamonds.

The 2.60 ct oval-shaped center stone was described as Fancy Intense blue. The three Old European brilliants were each color graded Fancy grayish blue. The range of color in which blue diamonds occur is rela-

tively compressed in saturation, and varies more widely in tone. Therefore, appearance differences are often differences in lightness to darkness rather than the strength or purity of the color. The four blue diamonds are relatively similar in tone, which explains their selection for the necklace. It is the stronger saturation of the 2.60 ct oval that accounts for its Fancy Intense grade.

Other gemological properties were consistent with those of typical type IIb natural-color blue diamonds (see J. M. King et al., "Characterizing natural-color type IIb blue diamonds," Winter 1998 *Gems & Gemology*, pp. 246–268). None of the diamonds showed a noticeable reaction to long- or short-wave ultraviolet radiation, and all of them were electrically conductive. Electrical conductivity is measured by placing the stone on a metal base plate and touching a probe carrying an electrical current to various surfaces of the

Figure 3. The four main blue diamonds are shown here removed from the necklace. The Fancy Intense blue oval at top weighs 2.60 ct, while the three Fancy grayish blue Old European brilliants weigh, from left to right, 0.75 ct, 0.42 ct, and 0.73 ct.

diamond. The conductivity value can vary with direction, so a number of measurements are made and the highest value is recorded. As noted in previous studies (again, see King et al., 1998), the highest electrical conductivity value can vary widely within and between color grades. We found this to be the case with these diamonds, as each showed a range of values. Interestingly, the Fancy Intense blue oval—the most strongly colored—displayed the lowest value.

It is common for type IIb blue diamonds to phosphoresce after exposure to short-wave UV radiation, and this characteristic was noted for all four stones. Although the best-known phosphorescent reaction in blue diamonds is that of the famous Hope diamond, the Hope's long-lasting strong red phosphorescence is actually quite rare. It is much more common for phosphorescence to be very weak to weak blue or yellow and of short duration; this more common reaction was exhibited by the diamonds from the necklace.

Microscopic examination revealed characteristics consistent with other type IIb blues. Such diamonds frequently have an uneven color distribution, which was observed here. It is also typical for them to have relatively few solid inclusions, with fractures and indented naturals being more common. This was also consistent with our findings. As would be expected, the blue diamonds, as well as other diamonds in the necklace, exhibited minor chips and abrasions consistent with the necklace's nearly 100-year history (figure 4).

Examination of these historic stones also gave us an important opportunity to study spectroscopic features of known natural IIb diamonds. Infrared spectroscopy showed absorptions in all four stones at 2801 and 2454 cm^{-1}, which are typical features of type IIb diamonds. Photoluminescence spectra collected using an argon laser (at 488 nm excitation) revealed emission features characteristic of natural diamonds. A relatively strong 3H emission (503.5 nm) was detected in all four stones.

Luminescence imaging is a useful way to study diamond growth, and was performed using the De Beers DiamondView. As shown in figure 5, blue luminescence and networks of polygonized dislocations are evident. Similar dislocation features were observed in all four stones. This type of dislocation network is a specific feature of natural diamond, and has never been observed in synthetic diamond.

John M. King, TMM, and Wuyi Wang

Figure 4. Minor chips and abrasions such as those seen here on the 0.42 ct Old European brilliant are often encountered on diamonds that have been worn over a long period of time.

Figure 5. This luminescence image of the 2.60 ct oval-shaped diamond was collected using the De Beers DiamondView. A blue luminescence and networks of polygonized dislocations are evident; such dislocation networks are characteristic of natural diamond.

Irradiated Blue Diamond Crystal

Irradiation with or without annealing is a common technique used to enhance the color of diamonds, and those suspected of being treated in this manner are routinely submitted to the laboratory for origin-of-color testing. Most such stones are treated after faceting, and depending on the type of treatment, diagnostic color distribution features are sometimes seen. Among the few laboratory-irradiated rough diamonds we have examined was a 2.95 ct well-formed bluish green octahedron (Winter 1989 Lab Notes, pp. 238–239).

Recently, a large crystal that resembled the sample in the 1989 Lab Note was submitted to the East Coast laboratory for origin-of-color determination. The experienced client who brought the stone to our attention was suspicious of its origin even though it was represented as coming directly from the mine in central Africa.

The 11.60 ct crystal (figure 6), which measured 13.11 × 12.85 × 9.09 mm, exhibited typical octahedral crystal morphology and showed obvious resorption features on its surface. It also showed a distinct greenish blue coloration. In contrast to similarly colored natural diamonds, no green or brown radiation stains were observed with magnification. However, a slight color concentration was evident at the edges of the crystal faces (again, see figure 6). We observed a strong blue fluorescence to long-wave UV radiation and a weak green-yellow reaction to short-wave UV.

Infrared spectroscopy revealed features typical of a type Ia diamond with very high nitrogen content and a weak absorption due to hydrogen impurities. In rare cases, a high concentration of structurally bonded hydrogen in diamond could produce blue coloration, but that definitely was not the case for this crystal. A weak H1a absorption was present (at 1450 cm^{-1}), but there were no H1b, H1c, or H2 absorptions. In the UV-visible spectrum collected when the diamond was cooled by liquid nitrogen (figure 7), strong N3 (415

Figure 6. The strong blue coloration in this 11.60 ct diamond octahedron proved to be the result of laboratory irradiation.

nm), moderate N2 (478 nm), weak H3 (503 nm), and weak 595 nm absorptions were detected; a strong and broad GR1 (741 nm) absorption also was apparent.

These gemological and spectroscopic features led to the conclusion that this crystal was artificially irradi-

ated without subsequent annealing. Considering the large size of the crystal, it is possible that the radiation-related color does not penetrate evenly throughout, as is suggested by the concentration of color along the edges.

Wuyi Wang and TMM

Irradiated Type IIb Diamond

Type IIb diamonds are often blue, due to small amounts of boron impurities. Depending on the occurrence of other defects (e.g., plastic deformation), some type IIb diamonds exhibit a gray or, more rarely, a brown color (see, e.g., King et al., 1998, cited in earlier entry). Blue also can be produced in an otherwise colorless diamond by exposure to radiation (either naturally or in a laboratory) to create a vacancy defect. Although it is technically possible to enhance the blue color of a type IIb diamond by irradiation, mixing of two different color-causing mechanisms in the same diamond may not necessarily produce an attractive color. Brown natural

Figure 7. A strong and broad GR1 absorption (741 nm), together with a weak absorption at 595 nm, indicated that the 11.60 ct greenish blue diamond was irradiated in a laboratory. The pre-existence of relatively strong Cape absorptions (e.g., N3 and N2) resulted in the green modifier.
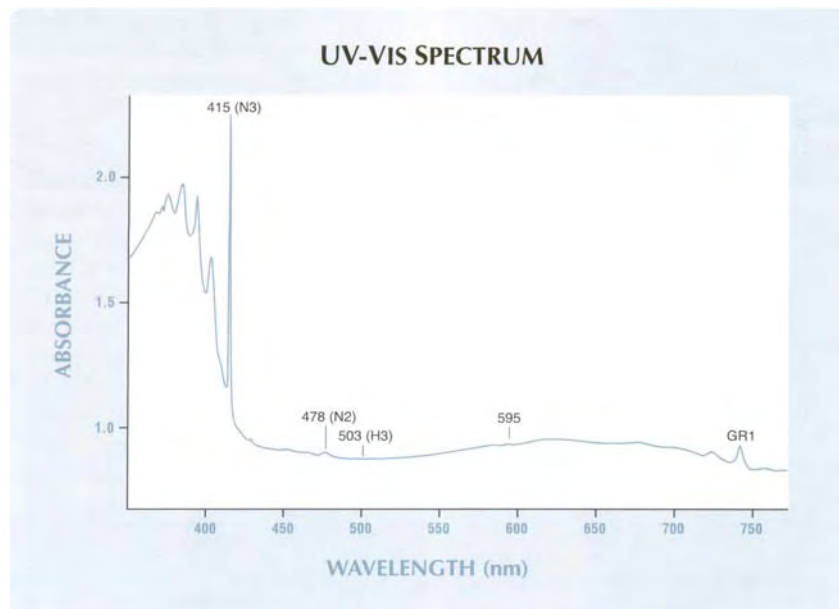
Figure 8. This 2.00 ct Fancy Dark green-gray type IIb diamond was found to have been laboratory irradiated.

radiation stains were reported on an unusual type IIb blue diamond (Fall 1991 Lab Note, pp. 174–175), but the radiation did not appear to have had any effect on the color of this faceted stone. An artificially irradiated type IIb diamond recently submitted to

Figure 9. When examined with a microscope and diffused light, the diamond in figure 8 revealed a strong concentration of blue near the culet, which proved that it had been artificially irradiated. Field of view is 6.0 mm high.
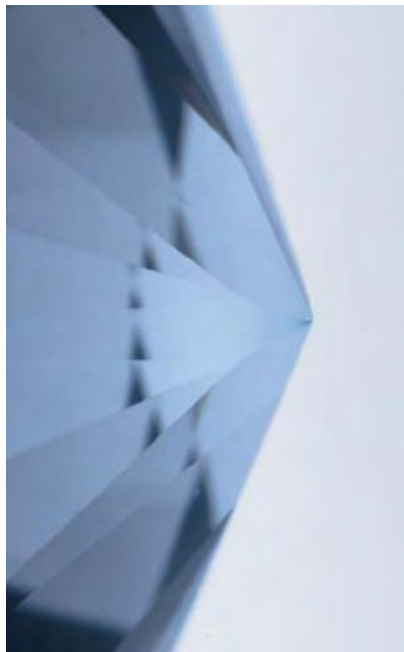
the East Coast laboratory gave us an extremely rare opportunity to examine a sample of this nature.

The 2.00 ct oval brilliant cut (10.52 × 7.14 × 4.21 mm) in figure 8 was color graded Fancy Dark green-gray. Although this hue was not outside the range of hues we have seen in natural-color type IIb diamonds, the cause of the color was not immediately obvious. No green or brown natural radiation stains were observed on the surface with magnification and dark-field illumination. Nor did the diamond have any notable internal characteristics such as solid inclusions or fractures, or any distinct colored graining. We did not see fluorescence to either long- or short-wave UV radiation, but very weak yellow phosphorescence was detected after exposure to short-wave UV. Infrared absorption spectroscopy only showed features typical of a type IIb diamond (e.g., strong and clear absorptions at 2801 and 2454 cm^{-1} due to substitutional boron impurities). However, when the

diamond was examined more carefully with low-power binocular magnification and diffused light, a distinct blue color concentration was noted near the culet (figure 9). This type of color zoning is typical for diamonds that have been artificially irradiated with a low-energy source, as is usually done today with an electron beam.

Absorption spectroscopy collected at liquid nitrogen temperature in the ultraviolet-visible range (figure 10) showed a strong GR1 band (vacancy, 741 nm) and several other lines of the GR series (GR2 through GR8). A clear TR12 absorption at 469.9 nm also was detected. It is very rare for a natural-color type IIb diamond to show any detectable GR1 or TR12 absorption with UV-Vis absorption spectroscopy. In addition, in the photoluminescence spectrum seen with a laser Raman microspectrometer, the intensity of the 3H defect with a zerophonon line at 503.5 nm (another typical radiation-related defect in diamond) was significantly stronger than

Figure 10. This UV-Vis absorption spectrum of the green-gray diamond shows a strong GR1 band and some additional lines from the GR series. A clear TR12 band also was detected. A gradual increase in absorption at higher wavelengths is attributed to the presence of boron impurities.
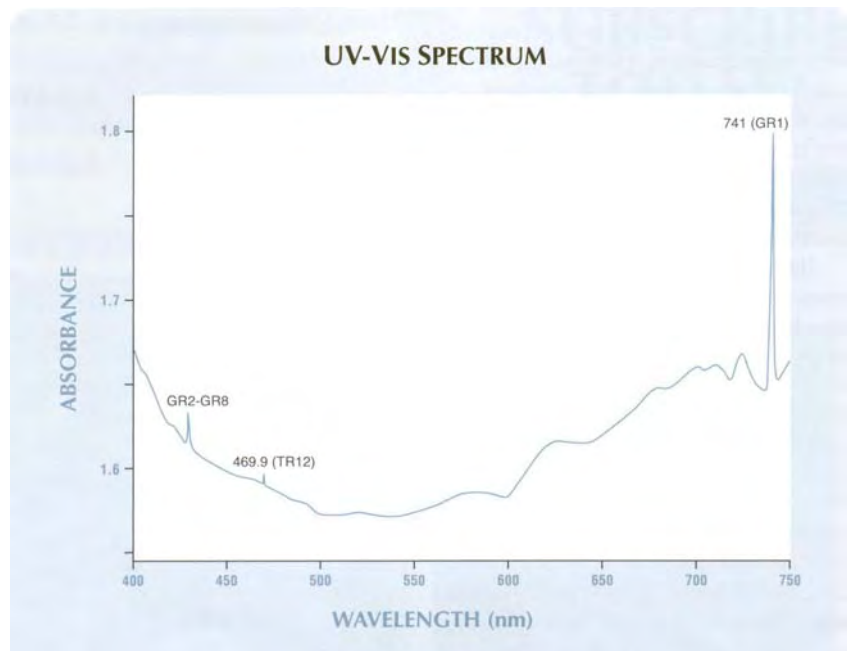
Figure 11. The color appearance of the 12+ ct Very Light blue type IaB oval modified brilliant in the center is due to scattering of light and not to any of the typical causes of natural blue color. The 6+ ct pear-shaped diamond on the left is colorless (D-color); the 5.5+ ct type IIb on the right is Fancy Light blue.

that in any natural-color type IIb diamonds we have examined. All these observations led to the conclusion that this type IIb diamond had been treated by irradiation in a laboratory.

Little is known about the interaction between vacancies and boron in diamond and the possible impact on color. The color of this specific sample is not very attractive. A possible reason is that prior to irradiation the stone may have had a strong brown component. This also would explain the green coloration after treatment.

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Unusual Cause of
### Blue Color in a Diamond

It is unusual for the laboratory to encounter natural blue diamonds with a cause of color other than boron impurities, natural irradiation, or the presence of hydrogen (see, e.g., E. Fritsch and K. Scarratt, "Natural-color nonconductive gray-to-blue diamonds," Spring 1992 *Gems & Gemology*, pp. 35–42; and King et al., 1998, cited above). Thus, it was quite a surprise when the East Coast lab recently tested an oval diamond over 12 ct (figure 11, center) with a blue color typical of that produced by boron but none of the other properties one would expect for such diamonds.

When color graded, the oval was classified as Very Light blue. On testing for electrical conductivity, the

diamond was found to be nonconductive. We observed a moderate blue reaction to both long- and short-wave UV radiation. Infrared spectroscopy proved that the diamond was type IaB and did not show any elevated levels of hydrogen. Spectroscopic analysis further ruled out any possibility that this stone was irradiated in a laboratory. However, microscopic examination revealed clouds of pinpoints and internal whitish graining.

In this case, we believe the color was caused by a scattering of light in the diamond from the clouds of pinpoint inclusions. This effect is similar to the one that causes cigarette smoke to appear blue, a phenomenon often referred to as the Tyndall effect. Named after its discoverer, 19th-century British physicist John Tyndall,

Figure 12. More typically, the scattering of light from micro inclusions produces a predominantly white (as shown in this 7.00 ct marquise) or gray color in diamond.
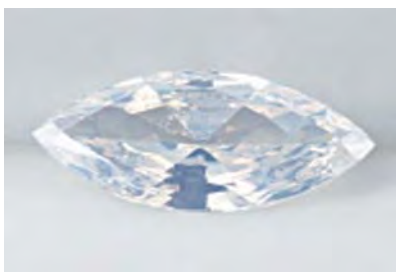
this effect is caused by reflection and/or scattering of light by very small particles in suspension in a transparent medium. It is often seen from the dust in the air when sunlight enters a room through a window or comes down through holes in clouds. Most diamonds that have dense clouds and also show scattering have a predominantly white or gray appearance (e.g., figure 12). The difference in color is probably related to the size of the particles and the density of the cloud.

We have documented diamonds with these properties from a few different geographic locations, including India and Australia. This unusual stone indicates that micro-inclusions in diamond can generate colors other than black, gray, and white. The nature of the micro-inclusions (chemistry, particle size, density, and distribution) and the size of the stone, along with the light source and its distribution of output, are all factors in determining the final color appearance of the diamond.

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NEPHRITE that Mimics Serpentine

Because of their similar appearances, some nephrite and serpentine can be impossible to distinguish by visual observation alone. Although the two mineral species usually can be readily separated on the basis of their refractive index and specific gravity values, in some cases even these physical properties can be misleading. The West Coast laboratory recently received a 142.27 ct translucent mottled green-gray carving of a water buffalo that exhibited both an artificial water-soluble reddish stain outlining details of the carving, and a light brownish yellow coating that partially concealed the underlying host material (figure 13).

Microscopic examination revealed that the host was a fine-grained aggregate. When tested in an inconspicuous area, the coating dented and scratched easily with a metal probe, similar to the response of a wax.

Figure 13. This nephrite carving (43.95 × 30.35 × 9.98 mm) could be confused with serpentine due to the effects of an unidentified coating.

istry, S.G., and FTIR and Raman spectra were all consistent with nephrite.

In an attempt to confirm or explain the uncharacteristically low R.I. values, the thin film that coated the specimen was polished off in a small inconspicuous area to better expose the host material. A higher vague R.I. reading between 1.60 and 1.61 was then obtained, indicating that the coating was responsible for lowering the apparent R.I. This piece was a valuable reminder of the care and diligence that should be exercised when obtaining physical properties for gem identification.

CYW

However, it did not melt readily when exposed to a thermal reaction tester; instead it merely yielded more easily and turned brown under the microscope with prolonged contact. It also did not react to a dilute HCl solution. This coating may have been an attempt at "antiquing" the carving; however, it is not known whether the coating was natural or an artificially applied substance. In addition, a very thin coating, which may have been separate from—or an extension of—the built-up areas, created an invisible film over the host material. It too could be scratched off, possibly deceiving an unwary observer into believing that the host material was soft. However, only the thin film yielded to the tip of the probe, whereas the material beneath was harder than the metal point.

The R.I. of the greenish areas was approximately 1.58, varying from 1.57 to 1.59. The higher value was obtained on a small area where the thin coating had been removed with solvent; areas with the thicker brownish yellow coating had lower R.I. values than areas where the green-gray host material was clearly visible.

The reaction to long-wave UV radiation was a combination of an inert background mottled with medium chalky green-yellow fluorescence that appeared weak to medium yellow on the areas with the thicker brownish yellow coating. The reaction to short-wave UV was also a combination of an inert background with a mottled very weak to weak yellow fluorescence on the coating. There was no significant visible-light spectrum.

With only this initial examination—the appearance of the carving, the illusory "soft" nature of the material, and the low R.I. values rarely attained by nephrite—a hasty evaluation could lead a gemologist to misidentify this material as serpentine. However, further testing revealed its true composition. The specific gravity, measured hydrostatically, was approximately 2.96; the FTIR spectrum was consistent with those of other nephrites; and the Raman spectrum of the greenish areas was consistent with the nephrite reference in the database. To characterize the material further, EDXRF analysis was performed by GIA senior research associate Sam Muhlmeister. The major elements present were Si, Ca, and Mg, with a trace of Mn and Fe. The presence of Ca in the structure excluded serpentine, while the chem-

Pink OPAL

This past summer, the East Coast laboratory received for identification two pairs of partially drilled translucent to semi-translucent variegated pink drops (figure 14). The larger pair measured 35.90 × 13.95 × 13.90 mm and 35.60 × 13.95 × 13.90 mm and had a total weight of 64.73 ct; the smaller pair had a total weight of 29.28 carats.

At first glance, the drops resembled conch pearls in their coloration. However, they did not exhibit any flame-like structure; nor were they the standard shape of conch pearls. In fact, their symmetrical shape indicated they were not pearls at all. They also resembled massive rhodochrosite, but they lacked the botryoidal structure and distinctive color zoning commonly associated with that mineral. Standard gemological testing revealed spot refractive indices of 1.45, weak-to-medium whitish fluorescence to long-wave UV, very weak yellow to no reaction to short-wave UV, and specific gravities ranging from 2.16 to 2.23. With a desk-model spectroscope, all four drops showed a weak band at approximately 490 nm, a weak line at approximately 550 nm, and a cutoff from 430 nm. These properties suggested opal, but glass could not be ruled out.

A Fall 1982 Lab Note (pp. 172–173) described a variegated light pink and

Figure 14. These four variegated pink drops, which range from 14.01 to 32.68 ct, were identified as pink opal.

gray carving of a bird that was identified as opal by the West Coast laboratory. Not until the Winter 1991 issue (pp. 259–260) did information become available in *G&G* regarding the location of the mines that reportedly were producing this translucent-to-opaque pink material, along with blue opal: the Acari copper mining area near Arequipa, Peru. It was said that some of the pink material even "exhibits a color reminiscent of rhodochrosite" (p. 259).

Our next step was to use energy-dispersive X-ray fluorescence (EDXRF) to investigate the chemistry of the four drops and compare the findings to a sample of known pink opal from Peru. EDXRF revealed the presence of Mg, Al, Si, K, Ca, Mn, and Fe. As opal is amorphous silica, the other elements can be attributed to mineral impurities, such as palygorskite, that are commonly associated with this type of opal (see J. Hyrsl, "Gemstones of Peru," *Journal of Gemmology*, Vol. 27, No. 6, 2001, pp. 328–334, and the Fall 1982 Lab Note). The spectra were nearly identical, clearly identifying these drops as pink opal, possibly from Peru.

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RUBIES, Clarity Enhanced with a Lead Glass Filler

Heat treatment of natural rubies frequently leaves a glassy residue within surface-reaching fractures and cavities. This filling is produced during the heating process and can facilitate the partial healing of fractures within the rubies. It is often detectable with magnification by observing the difference in luster compared to the surrounding corundum (see, e.g., Fall 2000 Lab Notes, pp. 257–259). This type of glass residue has a relatively low R.I. compared to corundum and is usually not considered to

be a clarity enhancement on its own.

Earlier this year, however, the Gemmological Association of All Japan (GAAJ) issued a lab alert describing rubies that had not been heat treated, but that showed a flash effect in their fractures caused by a clarity enhancement similar to that traditionally used in diamonds ("Lead glass impregnated ruby," *GAAJ Lab Alert*, 2004, GAAJ Research Laboratory, http://www.gaaj-zenhokyo.co.jp/researchroom/kanbetu/2004/gaaj_alert-040315en.html). Their EDXRF chemical analyses revealed the presence of elevated levels of lead (Pb) in the material filling the fractures in these stones. The results were later confirmed by the AGTA ("New ruby treatment arrives in the United States," AGTA Gemstone Update, http://www.agta.org/consumer/news/20040702rubytreatment.htm).

Recently, the West Coast laboratory had the opportunity to examine two purplish red mixed-cut oval stones (3.19 and 2.76 ct) that contained numerous large surface-reaching fractures and cavities filled with a glassy substance (figure 15). Both gems were identified as natural ruby by their inclusions, R.I., and visible absorption spectra in a desk-model spectroscope. Unlike the stones described by GAAJ, the presence of thermally altered inclusions showed that these rubies had been heated. The glassy fillings contained numerous flattened gas bubbles or voids (figure 16), and showed a

Figure 15. These two rubies contain numerous fractures filled with a high-lead-content glass that undoubtedly significantly improved their apparent clarity.
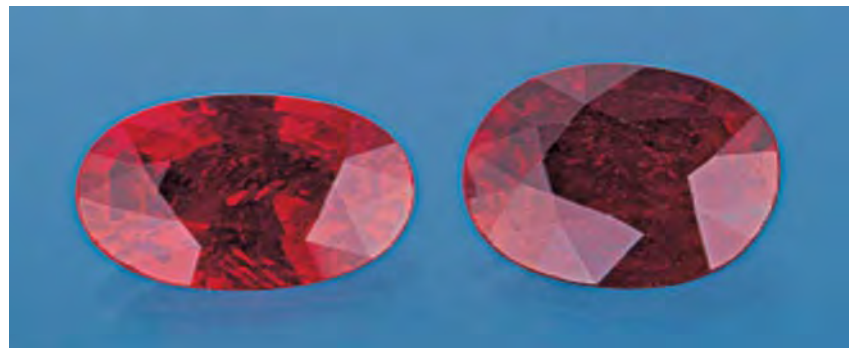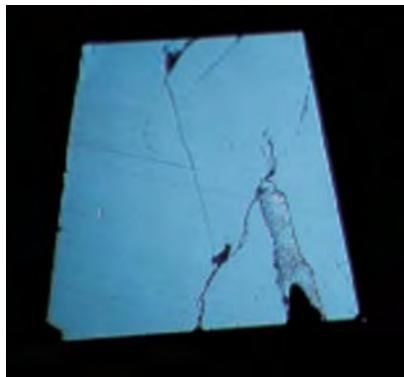
Figure 16. The glassy fillings in these clarity-enhanced rubies all contained numerous flattened gas bubbles (left) and elongated and irregular voids (right). Magnified 27×.

distinctly higher surface luster than the corundum (figure 17). The filled fractures were very low relief in all viewing directions and displayed weak to moderate blue-to-violet and orange flash effects. In some directions, the fractures had a slightly hazy appearance (figure 18). The 3.19 ct ruby, in particular, had several large filled cavities.

The filling material in the largest cavity was translucent, yellow in color, and contained many spherical gas bubbles (figure 19). In another large filled cavity, the filling material was also yellow, but transparent with no visible gas bubbles. The filling material in the fractures did not have a visible body color, but this was undoubtedly because of the small amount of material present. All the filled cavities showed poor polish at the surface, indicating a much lower hardness than the surrounding corundum (again, see figure 17). Unlike the residues we have seen previously in heat-treated rubies, the nature and abundance of the fillings in these two stones suggested an intent to hide the fractures and improve the apparent clarity of the gems.

To learn more about the filling material, we employed EDXRF analysis, Raman spectroscopy, and fluorescence imaging. EDXRF, performed by GIA senior research associate Sam Muhlmeister, showed elements typical of ruby (Al, Cr, and Fe) and elevated levels of Pb similar to those reported by GAAJ. None of the other elements that have been reported in glass-like ruby fillings (Si, P, Ca, and Ti) were detected (again, see the Fall 2000 Lab Note). It is important to note that lighter elements such as boron cannot be measured using EDXRF.

Raman spectroscopy was performed to compare the glass filler in these two rubies with five different glass samples known to contain significant Pb. Although the spectrum for this filler was not a match for any of the glasses in our collection, it had many of the same luminescence features as a sample of lead borate glass.

Last, we observed reactions to high-energy short-wave UV radiation using the De Beers DiamondView instrument. The filling material responded very strongly, fluorescing bright blue in contrast to the red reaction (caused by chromium) of the surrounding corundum. For comparison, traditional glass-filled cavities in rubies show an inert to dull gray reaction (figure 20, left and right). None of the glass samples from our collection showed any reaction in the DiamondView.

Key gemological identification features of this new filler include a distinct luster, flash effects, haziness, gas bubbles and voids, together with very low relief of the fractures in all viewing directions. In addition, elevated Pb and the absence of other significant ele-
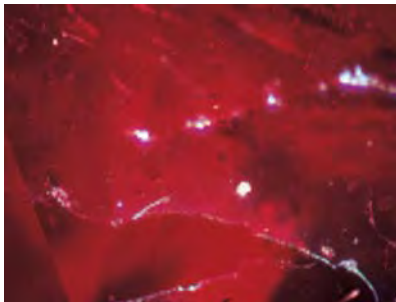
Figure 17. The luster of this filler material was actually higher than the ruby, which is the opposite of what is typically seen with glassy residues. Surface cavities filled with this lead glass also showed a poor polish compared to the surrounding corundum, indicating a much lower hardness. Magnified 40×.

Figure 18. In some directions, the filled fractures showed a slightly hazy appearance. Magnified 36×.

Figure 19. In one large filled cavity, the filler was translucent and yellow in color. One of the many spherical gas bubbles that were present can be seen in the center of this image. Magnified 27×.
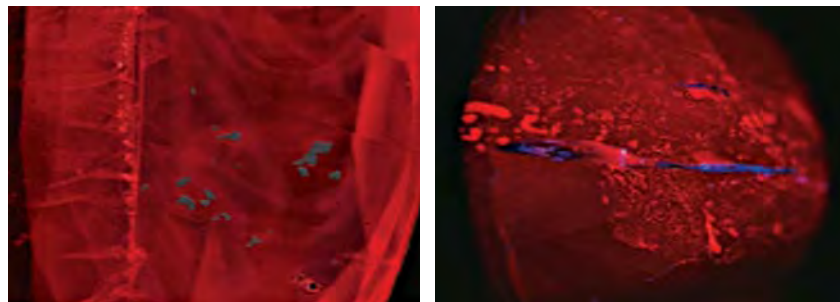
Figure 20. These DiamondView images show the inert to dull gray reaction of a traditional glass filler (left) compared to the bright blue reaction of the new lead glass filler (right).

ments attributable to the filler in the EDXRF analysis are characteristic of the samples we have seen thus far. Blue fluorescence in the DiamondView may also be useful for identification.

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Unusual SYNTHETIC RUBY Triplet Displaying Asterism

The identification of loose assembled stones is relatively straightforward. However, when they are mounted in jewelry (often bezel set), almost all

Figure 21. The red asteriated cabochon in this ring (13.00 × 11.80 × 10.55 mm) was identified as a triplet consisting of a synthetic ruby top and bottom joined by a near-colorless glue layer that surrounded an unidentified center section.

signs of assembly can be hidden. Frequently only the top portion of the assemblage can be identified, with few indications as to the identity of the bottom part possible because of the restrictions the mounting places on testing procedures (as was the case with the assembled stone reported in the Fall 1993 Lab Notes, p. 205).

Recently, the New York laboratory was asked to identify a large red double cabochon that displayed a weak six-rayed star; it was set in a yellow metal ring with 22 transparent near-colorless brilliants (figure 21). A 1.76 spot R.I. reading, characteristic absorption spectrum, and the presence of curved striae and gas bubbles seen with magnification identified the top of the cabochon

Figure 22. When the cabochon in figure 21 is viewed in profile, unmounted and immersed in methylene iodide, the distinctive layers that make up this assemblage are clearly visible, as are numerous gas bubbles in the near-colorless cement.

as synthetic ruby manufactured by a melt process. Further examination through the top of this mounted cabochon revealed a deeper layer of colorless cement with numerous oval gas bubbles, followed by another layer that exhibited the hexagonal growth structure typical of natural corundum.

Although at this point the cabochon appeared to be a simple assembled stone consisting of a synthetic ruby top and a natural ruby bottom, held together by a near-colorless cement layer, closer inspection of the convex bottom with a fiber-optic light and magnification revealed the presence of curved striae. This indicated that the bottom piece was *also* a melt-grown synthetic ruby. This was very unusual, but to fully identify the parts of this assemblage it would have to be removed from the mounting. Fortunately, the cabochon was held in place by only four small prongs, so with the client's permission we had it unmounted for further analysis.

The bottom piece exhibited diagnostic properties identical to those of the synthetic ruby top. Immersion of the cabochon in methylene iodide revealed a thin grayish blue layer completely encapsulated within a thick layer of colorless cement (figure 22). Due to its location within the glue, it was not possible to definitively identify this layer. However, the hexagonal growth structure seen in figure 23

Figure 23. Seen through the bottom of the cabochon while it was immersed in methylene iodide, the hexagonal growth structure of the unidentified section is apparent within the cement.
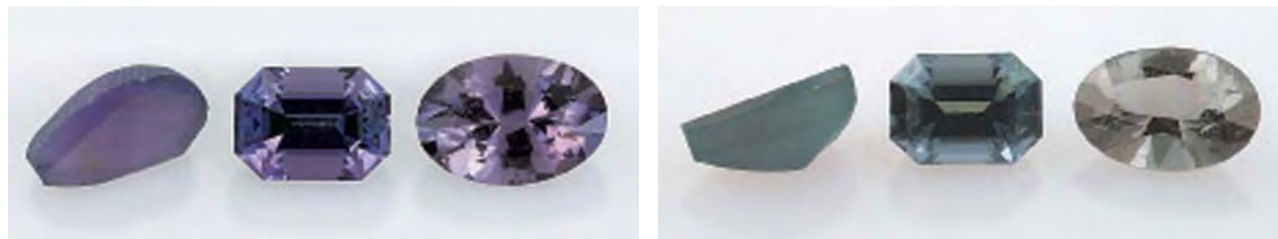
Figure 24. These three cuprian tourmalines from Mozambique (5.47, 5.68, and 5.37 ct) exhibit a strong "reverse" color change from fluorescent light (left) to incandescent light (right).

indicated that it most likely was a thin slice of natural corundum that contained the three directions of rutile needles, or "silk," necessary to create asterism. The conclusion that appeared on the gem identification report read as follows: "Triplet consisting of a synthetic ruby top and a synthetic ruby bottom, joined together with a near colorless cement containing an unidentified center section."

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Copper-bearing Color-Change TOURMALINE from Mozambique

Color-change tourmaline has been reported in *G&G* on only three prior occasions: dravite from East Africa (Fall 1991 Gem News, pp. 184–185), uvite possibly from East Africa (Fall 2000 Gem News, pp. 270–271), and copper-manganese-bearing elbaite from Nigeria (Fall 2001 Gem News International, pp. 239–240).

Elsewhere in the literature, there are limited reports of color-change tourmalines, which typically proved to be chromium- and vanadium-bearing specimens in the dravite-uvite series (H. Bank and U. Henn, "Colour-changing chromiferous tourmalines from East Africa," *Journal of Gemmology*, Vol. 21, No. 2, 1988, pp. 102–103; A. Halvorsen and B. B. Jensen, "A new colour-change effect," *Journal of Gemmology*, Vol. 25, No. 5, 1997, pp. 325–330).

Noel Rowe of Rough to Cut in San Jose, California, recently submitted to the West Coast laboratory a color-change tourmaline that differed

significantly from those previously described. Viewed table-up, this 5.68 ct transparent emerald-cut stone (11.63 × 8.76 × 7.48 mm) exhibited a distinct color change from purple in fluorescent light to gray–bluish green in incandescent light (figure 24). Particularly notable is the fact that the colors exhibited were the reverse of the usual alexandrite effect: cool colors such as green, blue, or gray in day or fluorescent light and warm colors such as purple, red, or brown in incandescent light. All other previously reported color-change tourmalines followed the usual convention.

This stone was eye-clean, and even with magnification it was exceptionally free of inclusions except for a tiny crystal near the surface of the pavilion and three miniscule needles near the girdle. Although optical properties and Raman analysis proved that this stone was indeed tourmaline (the Raman spectrum was consistent with rubellite in the database), the lack of inclusions and the very unusual phenomenon warranted further investigation.

To facilitate the investigation of this intriguing material, Mr. Rowe and the owner of the stone, Bruce Fry of Mars, Pennsylvania, supplied two additional tourmalines that exhibited similar properties. One was a 5.47 ct pear shape pre-form that had roughly the same color change as the emerald-cut stone. The other was a 5.37 ct oval that had a weaker color change from grayish purple in fluorescent light to gray in incandescent light (again, see figure 24). According to Mr. Fry, these three stones are the only tourmalines of this type that he or his South African suppliers have

ever seen. He stated that the emerald-cut and pear pre-form stones were from Moiane in northern Mozambique, approximately 200 km north of Nampula. The origin of the oval is only known as northern Mozambique. The specimens were alluvial in origin, and at least one of the stones was reportedly mistaken for a low-value iolite after being discovered by a local gold miner. Both the emerald cut and the pear shape pre-form were thought to be from the same lot; although cut recently, they were probably recovered several years ago. The oval was cut from a crystal with a color distribution that included a thin "rind" of pink skin and other small pink zones. Mr. Fry retained a flame-shaped zone of pink when he faceted the stone approximately four years ago.

The pear pre-form had a frosted surface with small areas of the original skin. Although the view into the stone was obscured by the frosted surface, magnification did reveal some sparse "trichites" (interconnected secondary two-phase inclusions) and a couple of fractures extending in from the surface that contained an orangy yellow residue, possibly iron-oxide staining. The stone was vaguely bicolored with a pink zone encompassing approximately half of it toward the tip. The oval also contained trichites, in addition to the pink flame-shaped zone that extended inward from the side. All the stones displayed the strong doubling at facet junctions typical of tourmaline.

The R.I. values of the emerald-cut and oval stones were ω = 1.639–1.640

and $\epsilon = 1.621$ (values for the pear preform were unattainable due to the rough surface). The hydrostatic specific gravities ranged from approximately 3.05 to 3.06 for the three samples. All three stones were inert to both long- and short-wave UV radiation. Dichroism was similar in all three—grayish violet to violet and grayish green to pale green—varying mostly in intensity.

To characterize these stones further, GIA senior research associate Sam Muhlmeister performed EDXRF qualitative chemical analysis. Al and Si were the major elements detected; the oval had a minor amount of Mn, while traces of Mn were seen in the other two samples. All the stones also showed traces of Ca, Cu, Ga, and Bi, with the oval showing Zn and Sr, and both the emerald cut and the oval showing some Pb (the presence of which was inconclusive in the pre-form).

The copper content is especially noteworthy. The only other commercially available tourmalines that contain copper are the copper-manganese-bearing elbaites, well-known for their exquisite blue colors, from São José da Batalha, Paraíba, Brazil (and surrounds), and western Nigeria (see, e.g., E. Fritsch et al., "Gem-quality cuprian-elbaite tourmalines from São José da Batalha, Paraíba, Brazil," Fall 1990 *Gems & Gemology*, pp. 189–205; and B. Laurs et al., "More on cuprian elbaite tourmaline from Nigeria," Spring 2002 Gem News International, pp. 99–100). One Nigerian cuprian elbaite that was examined by the Gübelin Gem Lab—a 22.98 ct violet gemstone—also exhibited a color change (C. P. Smith et al., "Nigeria as a new source of copper-manganese-bearing tourmaline," Fall 2001 Gem

News International, pp. 239–240). However, both the dichroism (purple-violet and slightly grayish violet-blue) and the color change (violet to purple) were different from what was seen in the three stones from Mozambique.

It is likely that Cu and Mn, at least in part, are the cause of color in these three specimens (Fritsch et al., 1990). Trace amounts of Pb in these samples could possibly be the result of residue from the polishing process and may not be reliable (D. Dirlam et al., "Liddicoatite tourmaline from Anjanabonoina, Madagascar," Spring 2002 *Gems & Gemology*, pp. 28–53).

The EDXRF analyses helped narrow the list of possible tourmaline species to which these stones belong. The lack of significant amounts of Ca, Fe, and Mg eliminates the calcic tourmalines and several other end members (e.g., dravite). Solely based on the elements detected, the remaining possibilities include the alkali tourmalines elbaite and olenite and the X-site-vacant tourmaline, rossmanite (see, e.g., F. C. Hawthorne and D. J. Henry, "Classification of the minerals of the tourmaline group," *European Journal of Mineralogy*, 1999, Vol. 11, pp. 201–215). However, quantitative chemistry, using a method that would detect lighter elements such as Na and Li, would be necessary to fully characterize these stones and the tourmaline species to which they belong.

Polarized UV-visible spectroscopy (also performed by Sam Muhlmeister) and oriented FTIR spectroscopy were conducted for comparison to other tourmalines. The UV-Vis spectra were very similar to those reported for the cuprian elbaites from the Paraíba mines in Brazil, with broad absorption peaks centered around

485–520 nm (which was attributed to Mn^{3+} in unheated Paraíba tourmalines) and Cu^{2+} absorptions centered around 690 and 895–905 nm in the $E \perp c$ direction and 710–720 and 900–920 nm in the $E \parallel c$ direction (again, see Fritsch et al., 1990). Although the FTIR spectra were consistent with other tourmalines, determining whether or not subtle differences exist to distinguish these stones would require further study.

Several aspects of these three tourmaline specimens are very intriguing: their strong and distinct color change, the fact that the color change relative to the type of light source is opposite the normal alexandrite effect, the copper content, and the source location in East Africa.

We have no current explanation for the reverse nature of the color change. We are continuing research into this seemingly new variety of tourmaline, with a focus on obtaining quantitative chemistry to more fully characterize this material and determine the species to which it belongs. Quantitative chemical analysis will also provide greater insight into the cause of color in these stones and possibly even clues to the unusual nature of this phenomenon. Perhaps East Africa will become a new source of copper-manganese-bearing change-of-color tourmalines.

CYW