GIA GEM TRADE LAB NOTES

EMERALD, An Interesting "Daughter" Crystal

Emeralds from Colombia are known to gemologists for their three-phase inclusions, which typically contain a brine solution, a gas bubble, and a cube of halite (a "daughter mineral," sodium chloride). For decades, these distinctive inclusions have served as a valuable means of separating such emeralds from their numerous synthetic counterparts, since such inclusions have never been observed in a laboratory-grown emerald.

Although gemologists have become accustomed to the cube form for the daughter crystals in the three-phase inclusions in Colombian emeralds, other isometric forms for these salt crystals have also been seen. For example, in plate 2 of his monograph "Composition of Fluid Inclusions" (U.S. Geological Survey Professional Paper 440-JJ, 1972), Dr. Edwin Roedder showed a photomicrograph depicting octahedral halite crystals in a three-phase inclusion in an emerald from the Muzo mine.

Consequently, the triangular crystal observed in the three-phase inclusion shown in figure 1, in an emerald sent to the West Coast GIA Gem Trade Laboratory for identification, was unusual but not totally unexpected. At first glance, one might think that this crystal was from the trigonal crystal system. But polarized light did not show any evidence that this material was doubly refractive. It behaved as one would expect of an isometric, unstrained crystal such as halite. We conclude that its triangular form was due to space restrictions, because the crystal was actually a highly modified octahedron showing growth preference to one octahedral face. Examining the photomicrograph by Roedder, one can easily see the source of the triangular form: A single octahedral face is a perfect triangle.

The world of synthetic gemstones also provides us with an example of such modification in the isometric system. Although the element platinum crystallizes in the isometric system, it forms distinct triangles and hexagons in manufactured gems such as synthetic rubies and synthetic alexandrites. This is no accident. In the case of a triangular platelet, the crucible-derived platinum has shown preference to one octahedral face over all other faces in the presence of the host material. In the case of a hexagon, a combination of an octahedral face with its edges modified by cube faces is the dominant form.

The cube is the most common isometric form shown by salt crystals in the fluid inclusions in Colombian emeralds. However, any of the other isometric forms cannot be discounted, and the gemologist should remember that modifications of these forms are a distinct possibility.

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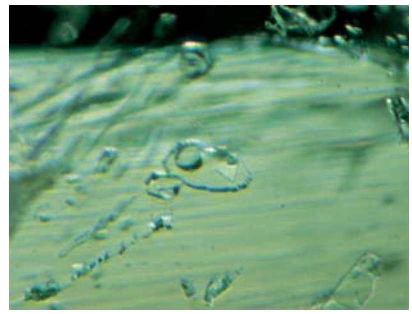

Figure 1. The 0.7-mm-long three-phase inclusion in the center of this photomicrograph of a Colombian emerald contains a triangular daughter crystal of salt.

HEMIMORPHITE, Rough and Fashioned

Many of the gem materials listed on GIA's "B Gem Property Chart" are seldom seen in either the East or West Coast labs. Thus, it was quite a coincidence last winter when a 234 ct piece of greenish blue fibrous rough received for identification in the West Coast lab, and an 8 x 6 mm greenish blue cabochon sent to the East Coast lab a few weeks later, turned out to be the same "B Chart" material.

The cabochon, which was mounted in a white metal ring (from which the client later removed it for advanced testing), was translucent (figure 2); magnification revealed a fibrous, banded structure. We obtained a spot refractive index of 1.62. The stone was inert to ultraviolet radiation, and showed no lines in the hand spectroscope. In the polariscope, it gave an aggregate reaction.

The piece of rough showed similar properties, with the same fibrous, banded structure that was evident in the cabochon (figure 3). A discreet

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hardness test revealed that the material was rather soft, with a Mohs value less than 5. Although the refractive index, aggregate nature, and greenish blue color were not sufficient to identify the cabochon, the additional property of hardness narrowed the choices down to two likely possibilities: smithsonite, a zinc carbonate; or hemimorphite, a hydrated zinc silicate.

Chemical analysis by EDXRF showed the same results for both the rough and the cabochon: large quantities of Si and Zn, with small amounts of Fe, Cu, and Pb. The presence of silicon as a major element indicated that both samples were hemimorphite. The last time either lab recalls receiving a sample of hemimorphite for identification was in 1971 (Winter 1971–1972 Lab Notes, pp. 383–384), when the properties offered the same two choices and we were able to make the distinction on the basis of specific gravity. This test should still provide the key discrimination in cases where it is practical.

IR and MLJ

Figure 2. This 8 × 6 mm cabochon proved to be the rare gem mineral hemimorphite.

Figure 3. A fibrous, banded structure is typical of hemimorphite. Magnified 30×.

JADEITE, With Inclusions of Zircon

As the practice of bleaching and impregnating jadeite has become more common over the last few years, the amount of jadeite we test in the laboratory has increased significantly. During the microscopic examination of one variegated green jadeite cabochon for possible evidence of treatment, we noted that the stone contained a number of small crystal inclusions, some of which reached the surface (figure 4). These surface-reaching crystals appeared to be somewhat harder than the surrounding jadeite, and in reflected light they had a higher surface luster. With the recent acquisition of GIA's Renishaw Raman Imaging Microscope System, we were able to examine two of these crystals nondestructively.

By focusing the argon laser through the 50× objective of the Leica targeting microscope and running several continuous scans, we obtained clear spectra of the two "inclusions." A computer search of our data base revealed that these spectra matched the spectrum for zircon. Visible characteristics of the inclusions (e.g., crystal form, color, transparency, and relative hardness) were consistent with this identification. This is the first time that we have encountered zircon crystals as inclusions in jadeite. Coincidentally, about a month prior to our discovery, a zircon inclusion in a jadeite cabochon was also identified by means of the same technique at the Center for Gemstone Testing in Bangkok (Kenneth Scarratt, pers. comm.).

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MOTHER-OF-PEARL Doublet

Pearl and shell are often used to make various assemblages, the most common of which are mabe pearls. The relatively subtle differences in physical and gemological properties between a pearl, mother-of-pearl, and the shell from pearl-forming mollusks can make the complete identification of some assemblages from these materials especially challenging. The animals that make pearls secrete calcium carbonate (as either aragonite or calcite) and conchiolin (a brown to black substance composed of various proteins) to create their shells. Control over whether calcite or aragonite is produced has been attributed to a variety of factors (see, e.g., A. M. Belcher et al., "Control of Crystal Phase Switching and Orientation by Soluble Mollusc-Shell Proteins," *Nature*, Vol. 381, 1996, pp. 56–58).

For the most part, the differences in properties between pearl, mother-of-pearl, and shell are caused by differences in the arrangement of the calcium carbonate and the conchiolin. The bulk of a shell is composed of columns of calcium carbonate, with small areas of conchiolin between them; mother-of-pearl is composed of long, thin sheets of aragonite alternating with thin layers of conchiolin; and a pearl is composed of overlapping thin curved layers of aragonite and conchiolin (see J. Taburiaux's *Pearls: Their Origin, Treatment, and Identification*, Chilton Book Co., Radnor PA, 1985, pp. 107–113, for more details and some excellent sketches). The thin, alternating layers of conchiolin and aragonite produce a phenomenon known as orient. The curved geometry of these layers causes a pearl to have orient from all

Figure 4. Raman microanalysis of the two largest included crystals in this jadeite cabochon showed that they were zircon. Magnified 15×.

viewing angles, while the flat arrangement found in mother-of-pearl allows orient to be seen from only one direction. The underlying shell does not show orient.

The 0.55 ct orangy pink cabochon shown in figure 5 presented just such an identification challenge to the West Coast laboratory. The assembled nature of the piece was apparent with magnification. The top was transparent and primarily colorless, although magnification revealed scattered red spots; it gave a range of spot R.I. values, from 1.50 to 1.65. This material also melted readily when the thermal tester was applied to an inconspicuous spot, emitting a strong odor of burning plastic.

The translucent white base of the assemblage showed a parallel, undulating, fibrous structure when viewed with magnification. It yielded a refractive index of 1.53 with a weak birefringence blink, and it effervesced to dilute HCl. The blink and effervescence pointed to a carbonate, and the refractive index indicated aragonite, rather than calcite. The piece showed an aggregate reaction between crossed polarizers, consistent with either shell or mother-of-pearl. The determination finally rested on the appearance of the base to the naked eye: Orient was seen from one angle in this piece of mother-of-pearl.

The position of the pink color just under the plastic top reminded us of the "treated" mabe pearls reported in the Fall 1991 Lab Notes section (p. 177). In that case, a mother-of-pearl base was covered with a plastic dome and a highly reflective coating, then placed in an oyster to receive a thin layer of nacre. All of these components were visible in a sample that our client cut open. We were not permitted to destroy the present cabochon, so we could not determine exactly where and how the pink color had been applied. *IR and MLJ*

Figure 5. Magnification revealed that this 0.55 ct cabochon was assembled from a mother-of-pearl base and a plastic top.

Cat's-Eye OPAL

In spring 1997, the West Coast lab received for identification the brownish yellow cat's-eye shown in figure 6. The chatoyancy in this 9.00 ct oval double cabochon resulted from numerous parallel "needles"—both fine and coarse—which were oriented across the width of the stone. A spot R.I. of 1.45 and a specific gravity of 2.08, obtained hydrostatically, pointed to opal as the bulk material, notwithstanding that brown is an unusual color for opal. Opal is singly refractive, but phenomenal opal often exhibits anomalous double refraction between crossed polarizers. In this case, observation down the length of the cabochon revealed an indistinct uniaxial optic figure. The gem also displayed distinct dichroism, with one color more yellow and the other more brown. The cabochon was inert to long-wave ultraviolet radiation and fluoresced weak red to short-wave UV. It had a slightly resinous luster.

These properties closely match those of a chatoyant opal described in the Fall 1990 Gem News (pp. 232–233). In that report, the authors suggested that the opal could be a pseudomorph after a fibrous iron-bearing mineral, and that the inclusions were most likely remnants of goethite. GIA Research analyzed the present cabochon using EDXRF and found silicon, iron, potassium, calcium, and zinc. The presence of iron supports the idea that the inclusions could be goethite.

The brownish yellow color of this cabochon (which appears to be directly related to the inclusions) is similar to the dark brown color described for the treated-color cat's-eye chrysoberyls that made news last fall because of their residual radioactivity (see, e.g., Fall 1997 Gem News, pp. 221–222). However, the properties of opal are very different from those of chrysoberyl. In particular, the differences in luster (vitreous to resinous for opal, subadamantine to vitreous for chrysoberyl), refractive index (1.45 versus 1.74), and specific gravity (2.15 as compared to 3.73) are so large that the two minerals can be readily distinguished. Cat's-eye opal is not known to be irradiated, or to show any residual radioactivity.

CYW and IR

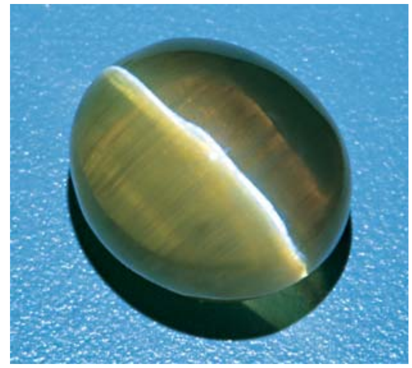

Figure 6. The unusual color and rare chatoyancy of this 9.00 ct opal are caused by inclusions of fine and coarse "needles," possibly goethite.

RUBY, with Surface Evidence of Treatment

Rubies and sapphires often show obvious signs of high-temperature heat treatment after being subjected to temperatures of 1500°C or more during the treatment process. Much of this evidence is internal and cannot be removed through normal lapidary techniques. In certain cases, however, the surface of the treated gemstone retains at least some of the visible signs of heat treatment, such as surface-sintered residue, glass fillings, near-surface solid and fluid inclusions that have ruptured through—and spread out onto—the surface, or partial melting of the surface itself if the treatment temperature is high enough (this last indication, "fireskin," is described on p. 140 of the Summer 1997 Lab Notes). Although usually the surface features can be completely removed by repolishing, it is also true that any cavities, pits, and other depressions in a surface that might contain evidence of heat treatment are likely to be missed during the repolishing process.

Recently, in the West Coast lab, we encountered two instances of repolishing after heat treatment that were unique to our experience. Both of the gems were mixed-cut rubies with brilliant-cut crowns and step-cut pavilions, one over 8 ct and the other just over 1 ct. They were sent in by different clients at different times.

Not only did both of these rubies contain what we have come to consider obvious internal signs of heat treatment (such as melted and exploded crystals surrounded by glassy "fingerprint" inclusions, discoid fractures, etc.), but both also had surface pits (up to 2.6 mm long on the 8 ct stone) that had been partially repolished (figure 7). When the stones were viewed with magnification in surface-reflected light, semi-circular grooves were readily apparent near the edges of these pits. The only way such marks could have been made on the surface of a gemstone as hard as ruby would have been by the use of a rotary tool and, probably, a diamond compound as the abrasive polishing agent. These attempts to remove the surface evidence of treatment in the tell-tale pits were not particularly successful, as other evidence of heat treatment was still visible. In addition, the presence of the curved grooves provided evidence that the indented surfaces in the cavities had been polished in an attempt to conceal the treatment. It is possible that the repolishing was intended to remove the glass-like fillings deposited in surface depressions that are often a by-product of the heat treatment of rubies.

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Figure 7. A rotary tool was used to partially repolish this 2.6-mm-long pit in the surface of a ruby, probably to remove evidence of heat treatment. Notice the curved grooves made by the tool in the faceted surfaces near the edge of the pit.

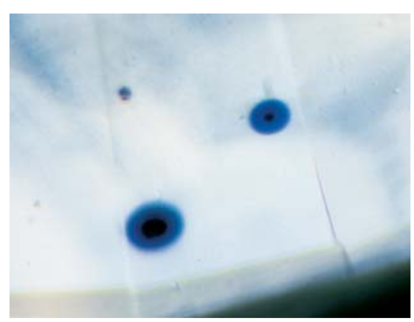
SAPPHIRE, Internal Diffusion Revisited

From time to time during the mid-1980s to early 1990s, we encountered transparent spherical clouds of intense blue color surrounding dark crystal inclusions in heat-treated sapphires. These stones were all from alluvial deposits in Montana, such as Rock Creek and Eldorado Bar, and the spherical blue clouds were important evidence of heat treatment. (In our experience, sapphires from the primary deposit at Yogo Gulch are not heat treated, and hence do not show such clouds.) For the last several years, however, we have not seen any sapphires with these vivid blue spheres. We were therefore surprised to observe such inclusions in a round-brilliant-cut sapphire submitted for identification last fall to the East Coast laboratory (figure 8).

These spherical blue clouds are caused by internal diffusion of color-causing elements from solid inclusions, which happens during heat treatment at temperatures that approach the melting point of sapphire. In effect, the host corundum cannibalizes the inclusions for their chromophores, diffusing those elements away from the inclusions, and creating more-or-less spherical clouds of color. Internal diffusion was first described in a 1987 report in the *Journal of Gemmology* (J. I. Koivula, "Internal Diffusion," Vol. 20, No. 7–8, pp. 474–477). The smaller the inclusions were, the smaller—and generally less intense—the blue spherical clouds appeared. This makes such spheres more difficult to recognize.

An excellent example of this more subtle form of internal diffusion is found surrounding tiny, dust-like needles of rutile and ilmenite in heat-treated sapphires from many different localities. The clouds of color produced in this manner consist of very small pale yellow or blue spots, often

Figure 8. These blue spherical clouds surrounding dark crystal inclusions in a heat-treated sapphire are caused by internal diffusion. Magnified 20×.



causing a subtle mottled appearance in the sapphire. This form of internal diffusion is best observed in diffused transmitted white light, and is a useful indication of heat treatment.

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SCAPOLITE, Showing Asterism

Scapolite is an unusual gem, more common in collections than in jewelry. Found in a variety of colors in Tanzania, Myanmar, Madagascar, Brazil, and Sri Lanka, scapolite often contains interesting inclusions. In some stones, masses of long, slender inclusions lead to chatoyancy or, more rarely, to asterism.

Recently, a dealer shared two large reddish brown scapolite cabochons from Tanzania with the West Coast laboratory. Both were mined in the mid-1980s, the 30 ct cabochon from the Dodoma area in central Tanzania and the 17.04 ct cabochon from Umba, in northern Tanzania. The Dodoma scapolite showed strong chatoyancy, and the Umba cabochon (figure 9) displayed a strong chatoyant band that provides the central arms of an otherwise weak, but well-formed, eight-ray star. Asterism in scapolite is quite rare, especially in such a distinct star with so many rays; we have previously reported on a weak four-ray star in a gray scapolite from Sri Lanka (Fall 1990 Gem News, p. 233) and a distorted, six-ray star in reddish-brown scapolite from Kenya (Spring 1984 Lab Notes, pp. 49–50).

Both cabochons showed properties typical for scapolite: a spot refractive index of 1.55 and a specific gravity of 2.66, measured hydrostatically. They were inert to long-wave ultraviolet radiation, and fluoresced weak red to short-wave UV. The chatoyancy in the 30 ct Dodoma scapolite was caused by long, thin, reddish brown inclusions (figure 10), similar to the goethite in scapolite illustrated in the *Photoatlas of Inclusions in Gemstones* (E. J. Gübelin and J. I. Koivula, 1986, ABC Edition, Zurich, p. 370). The inclusions in the asteriated stone were similar in color, but much shorter and more densely concentrated (figure 11). Although scapolite is uniaxial, the inclusions in both specimens created a pseudobiaxial figure in the polariscope. These inclusions were also the cause of the face-up color in these stones. Because of the relatively precise parallel alignment of the brown needle-like inclusions in the larger cabochon, it was easy to see between them when looking at the piece in profile view. This allowed us to see the true body color of the stone, which was light yellow, a common color for scapolite.

Figure 9. Note the strong chatoyant band and six additional weak, but distinct, rays in this 17.04 ct star scapolite from Tanzania's Umba Valley.

In the early 1980s, Graziani and others investigated a small number of specimens of reddish brown scapolite from Tanzania to determine the exact identity of the phenomenon-causing inclusions (G. Graziani and E. Gübelin, "Observations on Some Scapolites of Central Tanzania," *Journal of Gemmology*, 1981, Vol. 17, No. 6, pp. 395–405; and G. Graziani, E. Gübelin, and S. Lucchesi, "Observations on Some Scapolites of Central Tanzania: Further Investigations," *Journal of Gemmology*, 1983, Vol. 18, No. 5, pp. 379–381). They found long, thin growth tubes filled with both small scapolite crystals and crystals of an iron oxide or hydroxide. By bracketing the temperature history of their samples, these authors concluded that—along with the small crystals of scapolite—lepidocrocite [γ-FeO(OH)] had originally grown in these tubes, rather than goethite [α-FeO(OH)], and that these inclusions had dehydrated at modest temperatures (~350°C) to the magnetic mineral maghemite (γ-Fe_2O_3).

SFM and IR

Figure 10. These long, slender, reddish brown inclusions are responsible for both the chatoyancy and the overall color of this 30 ct scapolite cabochon. Magnified 20×.

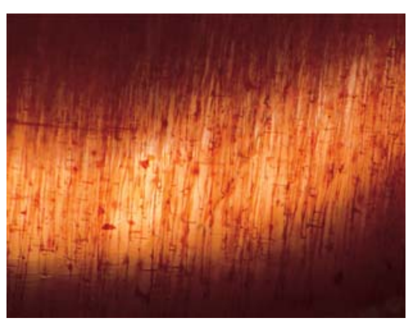

Figure 11. The reddish brown inclusions in the star scapolite shown in figure 9 are shorter and more densely concentrated than those in the 30 ct cat's-eye stone, and they are oriented in four different directions, rather than just one. The more intense central arms result from even denser concentrations of the inclusions. Magnified 20×.

TOPAZ, Natural-Color Green

The unusual 2.59 ct light bluish green oval brilliant shown in figure 12 was submitted to the West Coast laborato-

Figure 12. A natural-color green topaz, such as this oval brilliant, is quite rare. This color usually arises from irradiation treatment.

ry for identification. The measured refractive indices (1.611–1.620) and the biaxial optic character indicated topaz. Specific gravity obtained by the hydrostatic method and with the DiaMension noncontact measuring device resulted in values of 3.55 and 3.56, respectively. The stone fluoresced green, weakly to long-wave UV and with medium intensity to short-wave UV. These properties confirmed the identification. Two distinctive features were seen with magnification: (1) a cluster of parallel, small, thread-like inclusions; and (2) straight and angular growth lines that delineated a zone of more saturated color deep in the pavilion.

Green is an unusual color for topaz. Like the saturated blue colors most commonly seen on the market today, green typically results from irradiation in a nuclear reactor, although at higher temperatures than those that produce blue (see C. E. Ashbaugh III and J. E. Shigley, "Reactor-Irradiated Green Topaz," *Gems & Gemology*, Summer 1993, pp. 116–121). Such irradiation causes residual radioactivity, which decays over time. The presence of radioactivity is proof of treated color, but the absence of it is ambiguous; this gemstone displayed no radioactivity above normal background to a handheld Geiger counter. However, the neutrons from a reactor completely penetrate a gemstone such as topaz, resulting in even coloration, whereas this stone showed marked color zoning with magnification. Thus, we concluded that this stone was a rare natural green topaz. *IR and CYW*

PHOTO CREDITS

John Koivula photographed figures 1, 4, 7, and 8. Maha DeMaggio took photos 2, 5, 6, and 12. Shane F. McClure provided figures 3 and 9–11.