Gem Trade LAB NOTES

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Dyed Natural BERYL

The trade has given much attention recently to the "oiling" of emeralds, that is, the filling of surface-reaching fractures with balsam oil, Opticon, or another substance to lessen their visibility. In most such procedures, the filling material is colorless.

The dyeing of green beryl is an ancient process whereby a green oil is introduced into the natural fractures to imitate emerald. From time to time, we see dyed green beryl or "emerald" melee lots in the laboratory, but only rarely do we see green dye in larger stones. Recently, however, the East Coast laboratory identified green dye in a fracture in an 8.38-ct natural "emerald" (figure 1). Figure 2 shows a patch of waxy green material (probably dop wax) left on the girdle of this relatively large "emerald." Both of these materials would artificially enhance the color of the stone.

Although we observed weak chromium lines in the Beck hand spectroscope and a weak red reaction to the color filter, we could not determine the true body color of the stone because of the artificial coloring agents present. Consequently, the stone could only be identified as dyed natural beryl. *DH*

Figure 1. Green dye is evident in this fracture in an 8.38-ct beryl. Magnified 12×.

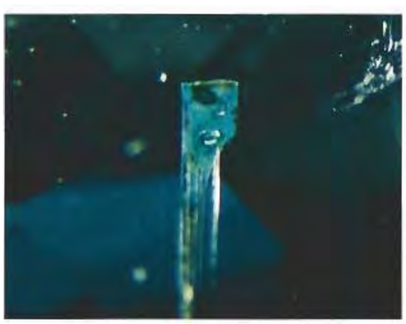
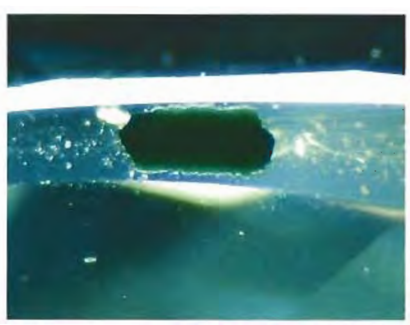


Figure 2. Green wax (probably dop wax) was also observed in a cavity on the girdle of the 8.38-ct beryl. Magnified 25×.



Carved CHRYSOBERYL

On occasion we see a well-known gem material used in an uncommon way. The West Coast laboratory recently saw a fine example of a carved horse's head made out of a piece of chrysoberyl (figure 3) that would have produced a cat's-eye if it had been cabochon cut. The carving measured approximately 20.3 × 28.7 × 6.2 mm and weighed 31.04 ct.

A spot reading of 1.75 on the refractometer and a strong 445-nm absorption band observed in the hand-held spectroscope confirmed the identity of the material. The stone was translucent to semitranslucent and an uneven yellow-brown (with some greenish component).

While no one area had a surface with enough curvature to display an eye, there was a strong sheen over the entire stone that gave rise to shimmering bands of light along the contours of the carving. According to our client, the piece was carved in Idar-Oberstein, Germany.

Shane F. McClure

DIAMOND

Electron Irradiated

Electron irradiation of diamonds ordinarily produces a blue or green color. When a 1.19-ct light bluish green round brilliant was recently submitted to the East Coast laboratory, it appeared to be a stone that could owe its color either to natural irradiation – that is, alpha or beta and attendant gamma rays – or to laboratory irradiation by neutrons. It is very often not possible to determine whether a green diamond has been artificially or naturally irradiated.

It is known, however, that electron irradiation of diamonds may produce characteristic zoning, such as around the culet or girdle edge, or along keel lines or facet junctions. In the case of this 1.19-ct light bluish green diamond, immersion in methylene io-

Editor's Note: The initials at the end of each item identify the contributing editor who provided that item.

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Figure 3. The chrysoberyl from which this 31.04-ct carving was fashioned is chatoyant.

dide and examination over diffused illumination readily revealed the blue coloration around the culet (figure 4). The original body color of the diamond was probably a light yellow before treatment. The combination of the yellow body color and the blue treated zone produces the appearance of a bluish green diamond.

For further information, see "Contribution to the Identification of Treated Colored Diamonds: Diamonds with Peculiar Color-Zoned Pavilions," by E. Fritsch and J. Shigley, *Gems & Gemology,* Summer 1989, pp. 95–101.

Figure 4. The zone of blue color around the culet of this diamond, seen here immersed in methylene iodide, shows that this bluish green stone had been subjected to electron irradiation. Magnified 6×.

The penetration of electrons into the diamond and the resultant coloration is very shallow, only about 0.5 mm. If a diamond treated in this manner were to be recut, the thin surface layer might be removed, thus altering the color. *DH*

Fancy Black

A 4.26-ct fancy black round brilliant–cut diamond (figure 5) was examined in the East Coast laboratory and found to be natural color and electroconductive in certain areas. Ordinarily, such conductivity would indicate a rare type IIb diamond. Although gray diamonds are often IIb's, we have never encountered a type IIb black diamond.

Exposure of the stone to long-wave ultraviolet radiation produced a distinct patchy blue fluorescence (figure 6) that was confined to clear areas of the diamond. The fluorescence changed to a chalky greenish yellow with exposure to short-wave U.V. These reactions indicate that the stone must be a highly included type I diamond. We subsequently confirmed this conclusion with infrared spectroscopy.

We concur with Kenneth Scarratt, head of the Gem Testing Laboratory of Great Britain, who speculated in a letter to the editor of the *Journal of Gemmology* (April 1990, p. 120) that the conductive property of such diamonds is due to the black inclusions, which are probably graphite. *GRC*

Laser Drill Hole Anomaly

In the 20 years since the application of laser drilling to improve the clarity appearance of diamonds was first reported in this column (Fall 1970, p. 224), we have noted numerous variations in the use and appearance of laser drill holes. For instance, in one case obvious cleavages radiating out

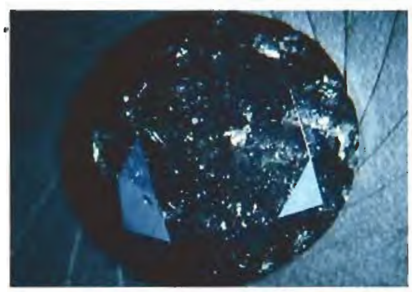
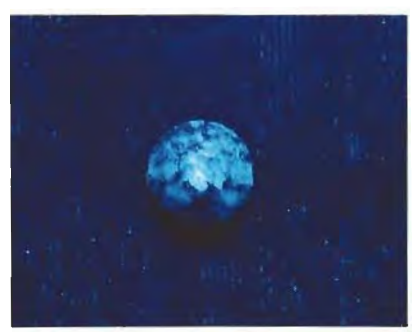


Figure 5. This 4.26-ct natural-color black diamond was found to be electrically conductive in selected areas. Magnified 10×.

Figure 6. The patchy blue fluorescence to long-wave U.V. radiation of the stone in figure 5 indicated that it was a highly included type I diamond.



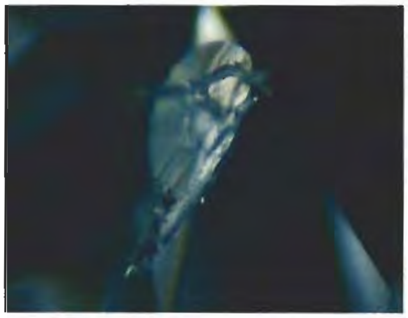


Figure 7. An unusual tubular channel pattern can be seen branching from the laser drill hole (here, slightly out of focus to the left of the inclusion) in this diamond. Magnified 63×.

from a long laser drill hole resulted in a poorer clarity appearance than expected (Fall 1976, p. 218). One laser hole reached two rather widely separated crystals, allowing them both to be bleached. We have also noted stones with natural tubular channels that individually resembled laser drill holes, but in aggregate appearance looked like branching "roots" within the stone (Spring 1973, p. 138; Winter 1973–74, p. 250; Fall 1979, p. 199).

Our latest observation of a laser drill hole anomaly is shown in figure 7. The diamond, a 1.55-ct pale green marquise, had natural brown irradiation stains on the girdle. The laser drill hole either has reached an internal cleavage or fracture and continued on erratically in several directions, causing a root-like pattern along the fracture, or has reached preexisting branching tubules that happen to have the same diameter as the drill hole. If the latter is the case, then the fracture may have originally been a black-appearing "light trap" that has been made white by laser drilling and bleaching. *GRC*

Laser Drilled to Reach an Included Diamond Crystal

Although we occasionally encounter diamond crystals as inclusions in a host diamond, the West Coast laboratory recently examined a 0.50-ct round brilliant-cut diamond that was both unusual and somewhat puzzling. A well-formed modified octahedral diamond crystal, measuring approximately 0.35 mm × 0.40 mm, was observed as an inclusion in this stone (figure 8).

Ordinarily, an included diamond crystal would have low relief and be barely discernable, since it has the same refractive index as its host. The unusually high relief exhibited by this diamond inclusion is probably due to a very minute separation, or interface, between the inclusion and the host. As light passing through the diamond goes from diamond to air at the point of the interface and then back to diamond, it "frames" the included crystal.

The most puzzling aspect of this stone, however, was the presence of a laser drill hole extending from the table down to the surface of the included diamond crystal. Laser drilling is used to remove or bleach dark inclusions from gem diamonds or to introduce a filling into fractures and cleavages that will make them less visible. It is not clear why it was attempted in this instance, because the included diamond crystal is impervious to the agents used to remove inclusions after laser drilling and thus could not be altered in size or appearance.

Mark Kazibutowski

Figure 8. This diamond crystal included in a cut diamond shows unusually high relief. Note the laser drill hole extending from the surface of the faceted stone to the included crystal. Magnified 40×.

OPAL, with an Unusual Inclusion

Recently, an unusual ring was brought to the West Coast laboratory for examination (figure 9). The ring contained a large (25.6 × 16.5 × 14.5 mm) Mexican opal that was quite transparent and had a nice play-of-color, displaying prominent flashes of green, orange, and red with lesser flashes of yellow and blue. The opal gave a spot refractive index reading of 1.43, and was inert to both long- and short-wave ultraviolet radiation. The hand-held spectroscope revealed a general absorption area up to approximately 440 nm, with a weak absorption band from approximately 500 to 540 nm. These properties are typical of many Mexican opals.

This opal is unusual because of the presence of a large (9 × 12.5 mm) centrally located egg-shaped inclusion that is best seen with transmitted light (figure 10). The inclusion was made up of an opaque white material that had a slightly "fuzzy" appearance at its surface. Because of the location of the inclusion in the middle of the stone, we could not perform any tests to determine its true identity. Although similar inclusions have been reported in opals before, it is rare to find such a large and dramatic example.

Christopher P. Smith

PEARLS

Cultured, with Colored Bead Nuclei

The laboratories on both coasts were given the opportunity to study a new type of bead nucleus used in pearl culturing in Japan. According to Yoshiko Doi, president of GIA's affiliate in Japan, AGTA, these beads are composed of powdered oyster shell that has been bonded with a type of cement and then dyed and fashioned

Figure 9. The 25.6 × 16.5 × 14.5 mm Mexican opal in this ring contains an unusually large oval inclusion, the outline of which is barely visible with reflected light.

Figure 10. When the stone in figure 9 is illuminated by transmitted light, the opaque, whitish inclusion is much more obvious.

into spheres. The sample beads we examined were dark grayish green and measured approximately 8 mm in diameter. An aggregate surface structure was observed with magnification. Transparent near-colorless and opaque white rounded grains were embedded in the dense green matrix material.

Specific gravity was determined by the hydrostatic method to be 2.74 ± 0.02. The refractive index was determined by the spot method to be on the low side of 1.50. The strong birefringence and the effervescence that occurred when a small droplet of a 10% hydrochloric acid solution was applied to the bead indicate that it is a carbonate material. When a cotton swab soaked with acetone was applied to the surface, a small amount of green dye was removed.

X-ray diffraction analysis showed that the material is calcite. An EDXRF chemical analysis performed by GIA's Dr. Emmanuel Fritsch revealed calcium plus titanium, cobalt, vanadium, and strontium, with smaller amounts of chromium and iron. Permission was granted to prepare a thin section from one of the beads. Dr. Ilene Reinitz, of GIA New York, obtained an infrared spectrum that showed distinct absorption peaks which were identified as calcite and the polymer or plastic that was used as the bonding agent in the material.

Subsequently, we received a few cultured pearls, averaging approximately 8.5 mm in diameter, that reportedly contained this new type of bead nucleus. Two of these pearls are shown in figure 11 with a section of a green bead nucleus. Magnification revealed that the nacreous layer was remarkably transparent. The drill hole of one of the pearls (figure 12) shows the thin nacre over a green bead nucleus, as well as some of the plastic bonding agent that melted from the heat of drilling. Because the nuclei were not freshwater shell, we did not expect these pearls to fluoresce to X-radiation and they did not. An X-radiograph showed a definite conchiolin layer, as well as the differences in the X-ray transparencies of the nucleus material and the nacreous layer.

KH, Ilene Reinitz, and Tom Moses

Freshwater Natural

The West Coast laboratory recently received for examination the interesting strand of freshwater natural pearls illustrated in figure 13. These pearls possessed a variable fair to very good luster and ranged in color from brownish to purplish pink, with a few being pinkish orange. They ranged in diameter from approximately 3.70 mm to 8.15 mm. Although some were slightly "off

Figure 11. These two cultured pearls, 8.5 mm in diameter, were reportedly grown using as nuclei green beads similar to that shown here.

Figure 12. The drill hole in one of the cultured pearls in figure 11 shows the thin nacre and the plastic binding agent which apparently melted from the heat of drilling. Magnified 15×.

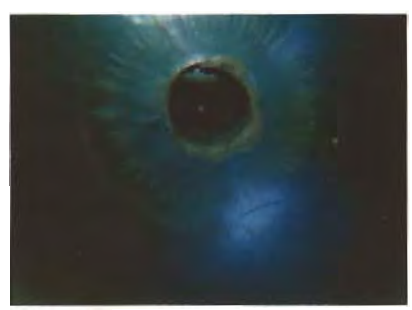


Figure 13. The natural freshwater pearls in this strand were found in the San Angelo area of West Texas over the course of 15 years. They range in diameter from approximately 3.70 to 8.15 mm.

round," most were spherical. They were identified as natural pearls on the basis of X-radiography performed by Robert Crowningshield, which revealed the very compact structure and extremely thin layers of conchiolin that are typical of some freshwater natural pearls.

Freshwater pearls generally show X-ray luminescence, caused by the presence of trace amounts of manganese. With this type of pearl, however, the staffs of both the East and West Coast laboratories have noted that the greater the intensity of the pink "body" color is, the weaker the fluorescence appears. The X-ray fluorescence reaction of the pearls in this strand, which ranged from inert to a weak slightly orangy yellow, supports this observation.

Our client reported that these pearls were recovered from lakes and rivers in the San Angelo area of West Texas. Over a period of 15 years, more than 30,000 pearls of variable quality were collected, ranging in diameter from 2 mm to 13 mm, with an average of 3.5 to 4 mm. Rarely are the pearls from this area spherical. Thus, it took nearly 15 years to accumulate the round pearls assembled in this strand. *RK*

Gray Baroque Cultured

Most of the natural-color gray to black cultured pearls from Polynesia that have been examined at the GIA Gem Trade Laboratory have been larger than Japanese pearls and have been round, oval, or ringed, but symmetrical in shape. Recently, however, we examined some attractive baroque Polynesian pearls (figure 14). An X-radiograph of this necklace (figure 15) shows that the nuclei vary from approximately 7.50 to 9.00 mm. The latter is larger than most Japanese mollusks can accommodate.

We were told by the client that baroques from Tahiti are more valuable than those from Japan. This prompted us to determine whether gray baroque cultured pearls from Japan were available and if they could be confused with natural-color gray cultured pearls from the South Seas. Figure 16 shows the result of our investigation. The nine baroques arranged in a square were loaned to us by a dealer in Japanese cultured pearls. The 10th pearl, shown by itself in the bottom row, is very similar in appearance and came from a parcel of Polynesian baroques. The X-radiograph (figure 17) shows that the Tahitian pearl has a very large nucleus (9.5 mm), contrasted with the 6.50 to 7.00 mm nuclei in the Japanese products. The X-radiograph also suggests that the gray color of the Japanese pearls is probably due to the appearance of the dark conchiolin through the nacre, the fragility of which may explain the dealer's remark about relative values. *GRC*

SAPPHIRE

Diffusion-Treated Montana Rough

During the last several months, the East Coast laboratory has heard rumors about heat-treatable sapphires from Montana being offered for sale. We were shown several lots of reportedly heatable pale greenish rough reputedly from mines along the Missouri River. Currently, lab staff members are conducting investigations both here and in Thailand to determine what type of Montana rough will respond to heat treatment.

One dealer contacted us after he was offered several hundred carats of

Figure 14. Tahiti has produced some fine gray baroque cultured pearls as well as rounds. The Tahitian pearls in this necklace are approximately 11.5 to 14 mm in diameter.

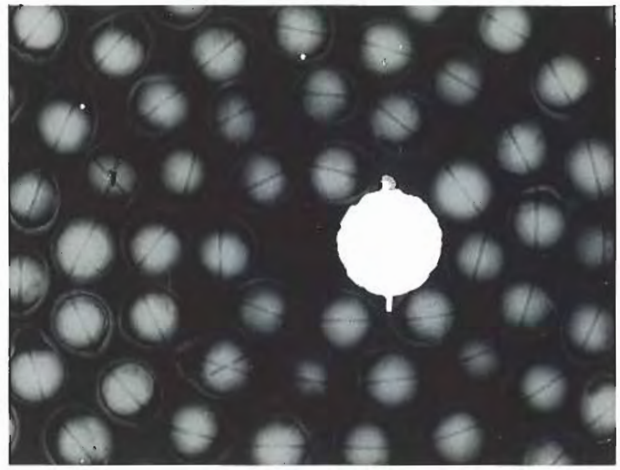


Figure 15. An X-radiograph of the necklace in figure 14 shows that the nuclei range from 7.5 to 9 mm in diameter.

Montana rough by a supplier who claimed the stones could be heated to improve the color. The dealer showed us several blue rough specimens that he was told were examples of this material after heat treatment. The seven palest of the 10 crystals were clearly heat treated, but the color was splotchy. The three most attractive darker blue stones had areas of dark blue color bleeding from included crystals, a characteristic of heat-treated stones, but closer examination revealed that there was also clear evidence of surface diffusion treatment (see, e.g., Kane et al., "The Identification of Blue Diffusion-Treated Sapphires," *Gems & Gemology,* Summer 1990, pp. 115–133).

Figure 18 shows a 2.73-ct rough specimen with lighter areas in the center and the ends that were not affected by the diffusion process. When the crystal was immersed in methylene iodide, we readily determined that the color was the result of a surface diffusion treatment. Clearly, use of such material to influence a potential investor is dishonest, since the layer of diffused color would be removed when the rough was faceted. *DH and GRC*

Large Colorless

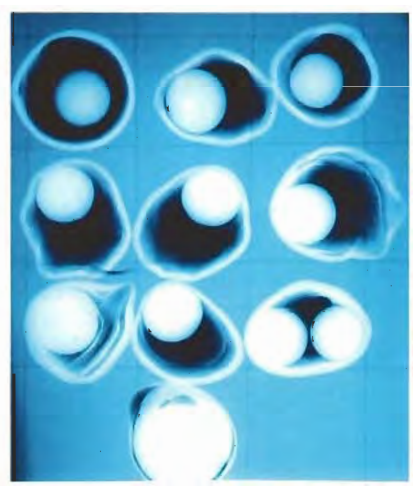
Although colorless sapphires are not extremely rare, large ones are seen only infrequently. A 63.65-ct colorless emerald-cut sapphire (figure 19), the largest yet encountered by our laboratory, was recently submitted to the West Coast laboratory for an identification report. Our client stated that he purchased this stone nearly 40 years ago and that it reportedly was found in Sri Lanka.

Refractive indices of 1.760–1.768 were as expected for this material. Using polarized light and a glass sphere as a condensing lens, we observed the uniaxial optic figure through the table in a direction nearly perpendicular to it. A very weak 450-nm absorption band was resolved with a hand-held type spectroscope, indicating the presence of at least some iron.

Some of the gemological properties are consistent with those reported in the literature for Sri Lankan sapphires. Specifically, a strong yellow-

Figure 16. The nine cultured baroque pearls in the square are from Japan, while the single cultured baroque at the bottom is from Polynesia (11.5 to 14 mm). All are similar in appearance.

Figure 17. An X-radiograph of the cultured pearls in figure 16 reveals both the larger nucleus (approximately 9.5 mm in diameter) of the Polynesian sample at the bottom and the large areas of dark conchiolin in the Japanese samples.



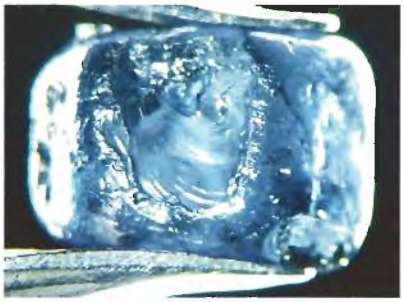


Figure 18. Immersion in methylene iodide revealed areas of diffusion treatment in this approximately 6.89 × 6.07 × 4.90 mm piece of sapphire rough from Montana. The thin layer of blue color would undoubtedly be removed during cutting.

ish orange fluorescence was observed when the stone was exposed to long-wave U.V. radiation. The same color fluorescence, but of very weak intensity, was seen with short-wave U.V. No colored transmission luminescence was seen. In addition, a dozen or more crystals with white-appearing strain halos were apparent with the unaided eye under the step facets at one end.

Magnification revealed that most of these inclusions appeared to be small to large opaque black uraninite crystals with characteristic tension halos. As is generally observed with this type of inclusion, when the gemstone is tilted to allow the background to become bright, the small tension fractures take on a distinct brown appearance. A number of straight and irregular, randomly oriented, "stringers" of minute white particles were also visible throughout the stone, as were several randomly oriented flat "flakes" and short needles. Although they were subtle in appearance, these features were easily seen with darkfield illumination in the gemological microscope. *RK*

Figure 19. This unusually large, 63.65-ct, colorless natural sapphire is reportedly from Sri Lanka.

Cobalt Colored Color-Change SPINEL

The West Coast laboratory had an opportunity to examine a cobalt spinel that exhibited a very unusual change of color: a dark, richly saturated, slightly violetish blue in fluorescent light, and a dark purple in incandescent light (figure 20). The color change was nicely enhanced by the contemporary design of the bicolored white and yellow metal man's ring in which the stone was set.

Figure 20. The 2.24-ct cobalt-colored spinel in this ring appears blue in fluorescent light and, as seen here, purple in incandescent light.

We found the stone's properties to be consistent with natural cobalt-blue spinels the lab has examined in the past (which reportedly originate in Sri Lanka; see *Gems & Gemology,* Summer 1986). A single refractive index reading at 1.720 was obtained using a Duplex II refractometer. Examination with the polariscope showed a singly refractive reaction, with very slight ADR. The stone exhibited a weak red transmission and a moderate red color when viewed through a Chelsea filter. The diagnostic absorption band of a natural cobalt spinel was present at 460 nm in the hand-held spectroscope, as were the other bands at approximately 480, 552, 559, 575, and 622 nm, with a broad general absorption from 660 nm up. (For more information on cobalt-blue spinels, see J. Shigley and C. Stockton, " 'Cobalt-Blue' Gem Spinels," *Gems & Gemology,* Spring 1984, pp. 34–41).

With magnification, under one end of the crown we observed a cluster of moderate-size inclusions that consisted of a well-formed octahedral crystal and several whitish irregularly shaped crystals. Radiating from this crystal group and throughout the stone were rain-like pinpoint "stringers" and numerous small fine needles. *Patricia Maddison*

SYNTHETICS and SIMULANTS in Period Jewelry

One of our Austrian readers, Karl H. Heldwein of Vienna, saw the earring mentioned under this heading in the Summer 1990 issue of *Gems & Gemology* and was kind enough to send a letter with his comments on similar items seen in his country. He states, "We in Vienna, Austria, frequently run into pieces like that. The stamp is the current Russian hallmark which, to my knowledge, has been used since the mid-1950s." He enclosed a photo of an item very similar to figure 16 in the Summer 1990 issue. However, it had a blue cabochon for the center stone instead of a faceted piece. In his letter, he says that the cabochon was glass and the near-colorless brilliant-cut diamonds were VS to I_1 in clarity. The piece would obviously have to be considered a reproduction.

Charles C. Cage, of New Orleans, Louisiana, sent us a very comprehensive review of Russian hallmarking in the 19th and 20th centuries. He confirmed that the "hammer and sickle" mark is the current one used in Russia, and that it has been in use since 1958.

We thank Messrs. Heldwein and Cage for sharing this information with us. Comments from our readers are always welcome. *CF*

FIGURE CREDITS

Photos in figures 1–2, 5–6, 14, 16, and 17 are by Nicholas DelRe. Shane F. McClure took the photographs in figures 3, 9, 10, 13, and 19. Holly Baxter provided the photo in figure 8. Vinnie Cracco is responsible for figure 7. The photomicrograph in figure 4 is by John I. Koivula. Robert Crowningshield did the X-radiography in figure 15. Robert Weldon shot the photos in figures 11, 12, and 20. Dave Hargett furnished figure 18.

A HISTORICAL NOTE

Highlights from the Gem Trade Lab 25, 15, and five years ago

FALL 1965

The New York laboratory described and illustrated a 10+-ct Montana sapphire crystal, the largest yet encountered. Also discussed were abalone pearls, the danger of leaving cultured pearls in jewelry cleaner, and the confusion arising from calling maw-sit-sit jade albite, rather than chrome albite. X-ray diffraction analysis established that this material actually contains ureyite, now renamed kosmochlor, rather than jadeite.

FALL 1975

The Los Angeles lab had the opportunity to examine a very unusual cyclotron-treated diamond. The fact that this stone was treated from one side of the girdle, rather than from the culet or the table as is usually done, made it unique in our experience. In this stone, the color zoning associated with cyclotron treatment was apparent only on the side of the keel line and that side of the girdle that was treated. In addition, the lab identified as marble a ceremonial sword that showed, under 50× magnification, some tiny fossil foraminifera.

Several imitations and simulants were seen in the New York lab, including coated garnet to imitate cuprite, prosopite to imitate turquoise, coated green beryl to simulate emerald, and a parcel of Thai-cut synthetic rubies that to the inexperienced eye were very natural looking. These proved to be flux-grown stones, many with the bluish bands usually associated with Chatham synthetic rubies.

FALL 1985

Our East Coast lab had occasion to examine a star stone that was in fact a flux-grown synthetic sapphire. The star was surface induced by diffusion treatment. A hydrothermally grown synthetic emerald, heavily included with spicules, was also illustrated.

Unusual items encountered in the West Coast lab were an 11.73-ct gambling die that was cut from an industrial-quality diamond, a black star sapphire doublet, and a carved material that somewhat resembled jade (see figure). X-ray diffraction analysis revealed that the carving was a rock consisting of plagioclase feldspar and green muscovite mica.