

## LAB NOTES

## EDITOR

C.W. Fryer  
Gem Trade Laboratory, West Coast

## CONTRIBUTING EDITORS

Robert Crowningshield • David Hargett • Thomas Moses  
Gem Trade Laboratory, East Coast

Karin Hurwit • Robert E. Kane  
Gem Trade Laboratory, West Coast

**A Large CHLORITE Carving**

Recently submitted to the West Coast laboratory for identification was the opaque, mottled, yellowish brown to brownish yellow oriental carving illustrated in figure 1. Our client reported that this interesting piece—possibly a scepter—was believed to be nearly 1200 to 1300 years old, from the T'ang dynasty. Examination with the unaided eye revealed an overall pearly luster, and one small area showed a dull, pearly, granular fracture. The lighter, brownish yellow layer of material on the top and bottom of the carving also displayed a weak sheen.

A precise refractive index proved difficult to obtain due to the curved surfaces and the condition of the polish; using the shadow method, however, we determined an approximate value of 1.57.

Because of the difficulty of identifying such a carving with standard gemological tests, we obtained the client's permission to scrape a minute amount of powder from an inconspicuous recessed area for X-ray powder diffraction analysis. The



Figure 1. This interesting carving (13.3 × 3.6 × 2.5 cm) is made from clinochlore IIb, a member of the chlorite group.

results matched our standard pattern for clinochlore IIb, which is a member of the chlorite group.

As Klein and Hurlbut pointed out in the *Manual of Mineralogy*, 20th ed. (1977), it is extremely difficult to distinguish between the members of the chlorite group without detailed study of the X-ray and optical properties or quantitative chemical analyses. Although previous researchers have designated many varietal and species names to this extensive solid-solution group, research published in *Reviews in*

*Mineralogy*, Volume 19, *Hydrous Phyllosilicates*, edited by S. W. Bailey (1988), recommends that only the species names—clinochlore, chamosite, pennantite, nimite, and bailey-chlore—be used for members of the chlorite group.

Therefore, on the GIA Gem Trade Laboratory identification report for the carving shown in figure 1, we stated: "The X-ray diffraction pattern of this material matches our standard pattern of CLINOCHLORE IIb, a member of the CHLORITE GROUP."

RK and Christopher P. Smith

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

© 1991 Gemological Institute of America

### Treated-Color DIAMOND with Natural Radiation Stains

When green diamonds are submitted to the Gem Trade Laboratory, we look for, among many other things, physical characteristics that indicate whether the color is natural or produced by treatment. One indication that a polished diamond had been exposed to radiation in nature is the presence of green or brown radiation stains on a natural surface left by the cutter. It is known that these stains develop when the diamond crystal is exposed to alpha-particle radiation within the earth. However, they cannot be considered proof that the green body color is of natural origin, because alpha rays penetrate only a few tenths of a millimeter in diamonds. The stains will remain green unless the diamond is exposed to heat, either naturally or artificially, and will change to brown at about 500°C.

During routine examination of a 0.82-ct dark green marquise brilliant, the East Coast laboratory observed dark green natural radiation stains on the girdle at each point (figure 2). However, the uniform dark green color of the diamond, possibly the result of neutron irradiation, caused us to suspect that it might have been treated. In fact, we concluded—on the basis of its characteristic spectrum and uniform coloration as seen in

*Figure 2. Natural dark green radiation stains were seen on the girdle at each point of this 0.82-ct treated green diamond. Magnified 30×.*



*Figure 3. This vase, which measures 25.0 × 16.4 × 9.0 cm, was carved from an unusual color of massive grossularite garnet.*

immersion—that this diamond was treated. This stone may have been carefully selected for treatment because it had natural radiation stains; perhaps it was a pale green diamond that the treaters wanted to “enhance.” It is also possible that the treaters were not aware of the stains.

If this diamond had been annealed following irradiation, the radiation stains would have turned brown and the body color would have become orange to yellow. DH

### Massive GROSSULARITE GARNET Carving

An odd-color vase, carved in an oriental flora-and-fauna motif, was recently submitted to the West Coast laboratory for identification (figure 3). It was a translucent mottled brownish yellow to brown with grayish white areas and some whitish yellow veins; the moderate polish and sub-vitreous luster suggested a rather hard material.

Spot refractive index readings taken from three different locations gave values of approximately 1.74. For the most part, the vase was inert to both long- and short-wave ultraviolet radiation, although some of the whitish yellow veins and grayish white areas fluoresced moderate to strong yellow to both wavelengths. A diagnostic spectrum could not be obtained with the hand-held spectroscope. When the vase was examined with the polariscope, we noted an aggregate reaction.

Because the standard gemological tests that could be conducted on this material were inconclusive, we decided to scrape a minute amount of powder from an inconspicuous area for X-ray powder diffraction analysis. The resulting pattern matches the standard pattern for grossularite garnet. Massive grossularite garnet is not commonly seen in the lab as caryings. A carving of the same material, although green in color, was reported in the Spring 1985 Lab Notes section.

Patricia Maddison

### Heat-Damaged JADEITE

A  $14.85 \times 11.75 \times 4.20$  mm jadeite jade cabochon set in a woman's ring was submitted to the East Coast laboratory for a damage report. The client was concerned about a change in appearance that was noticed after the ring had been repaired. The discolored area around the girdle was most apparent with transmitted light (figure 4). In addition, when the stone was exposed to long-wave ultraviolet radiation, the discolored area fluoresced pale blue (figure 5).

We have encountered heat-damaged jadeite before (see the Summer 1982 Lab Notes), but in that case the discoloration was fairly random. We nevertheless suspect that the change in appearance of the present stone is also related to heat. Heat alone may have been the culprit, or the stone may have received the customary paraffin bath during manufacture and the heat partially removed the

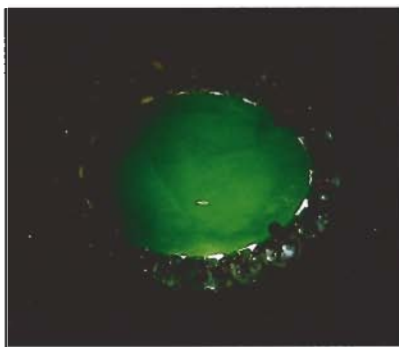


Figure 4. In transmitted light, discoloration apparently caused by exposure to excess heat is evident around the girdle of this  $14.85 \times 11.75 \times 4.20$  mm jadeite jade cabochon.

paraffin. It is possible that the original appearance could be restored by another paraffin bath, although there was no evidence of paraffin present at the time the stone was tested. In any case, the person who performed the repair could have avoided the damage by simply removing the stone before heat was applied to the setting.

GRC

### Banded LAPIS LAZULI

One of our *Gems & Gemology* subscribers recently gave the West Coast laboratory the opportunity to examine and photograph the lapis lazuli cabochon ring shown in figure 6. Note the very prominent curved banding, easily seen with the unaided eye, that runs across the entire curved surface of the  $19.5 \times 14.7 \times 8.25$  mm stone. The subscriber had read with interest the entries in the Spring 1988 and Summer 1990 Lab Notes sections, which described similar, smaller cabochons of banded lapis. This stone had a spot R.I. of 1.51 and a vague reading (from calcite) at 1.48 to 1.65, was inert to long-wave U.V. radiation, and had a somewhat patchy, moderate chalk whitish green reaction to short-wave U.V.—all of which is consistent with lapis lazuli. Testing with an acetone-

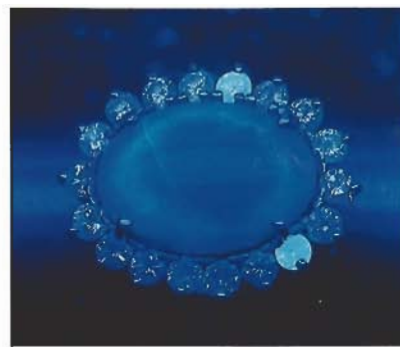


Figure 5. When the stone shown in figure 4 was exposed to long-wave U.V. radiation, the discolored area fluoresced pale blue.

soaked cotton swab established that this piece was not dyed.

With low magnification ( $10\times$ ) and oblique fiber-optic illumination, we determined that the curved bands in this stone—as with the two previously examined by the laboratory—were comprised of layers of densely packed dark blue grains alternating with layers of light blue, near-colorless, and dark blue grains. The space between the alternating bands ranged from approximately 0.3 to 0.6 mm.

RK

Figure 6. Parallel curved banding is readily apparent in this  $19.5 \times 14.7 \times 8.25$  mm lapis lazuli cabochon.



### Unusually Large Worked and Plugged Cultured PEARL

An unusually large ( $32.20 \times 25.68 \times 21.10$  mm; 96.00 ct) baroque pearl was submitted to the East Coast laboratory for identification (figure 7). Because of its size, we originally speculated that it might have been natural, as cultured pearls this large are very rare.

However, an X-radiograph revealed a relatively small shell bead, about 9.5 mm in diameter, as the nucleus (figure 8). In addition, we observed that what was apparently a groove or opening in the original cultured pearl had been filled with a cement-like material. The area around the "plugged" spot appeared to have been buffed.

If this cultured pearl were to be drilled, care would have to be taken to drill into the shell nucleus. If the drilling were to extend into a hollow space, the pearl could fracture.

DH

### QUARTZITE and DOLOMITE Bead

Several colored stone dealers have remarked that black is currently fashionable and, thus, that there is a demand for jewelry with black gems. Some dealers have told us that the dyed black chalcedony traditionally used is sometimes difficult to find, so that substitutes may be called for.

Figure 7. At  $32.20 \times 25.68 \times 21.1$  mm, this is an unusually large cultured pearl.



Such was the case with the black pear-shaped bead shown in figure 9.

Initial testing at the East Coast lab indicated that the material was a carbonate; with the spot method, the bead displayed the typical red-to-green blink in the refractometer, and gave a spot reading of 1.66. A tiny drop of dilute hydrochloric acid applied to an inconspicuous area of the bead produced an effervescent reaction. The specific gravity, obtained hydrostatically, was 2.74. Because these results could apply to several carbonates and various rock mixtures, we performed an X-ray powder diffraction analysis. This test proved that the material was a rock consisting principally of dolomite with some quartzite.

Usually, dolomite will not effervesce to room-temperature hydrochloric acid, but it may do so if the dolomite consists of fine-grained crystals, as was the case with this bead.

DH

### Coated SAPPHIRE

With the proliferation of diffusion treatment, heat treatment, and the filling of cavities in corundum, the laboratory is particularly diligent when examining rubies and sapphires for enhancement.

Recently, a client submitted to the East Coast lab a medium-tone yellowish orange sapphire with the comment that "something did not

Figure 8. Note the small shell bead in the large cultured pearl shown in figure 7.

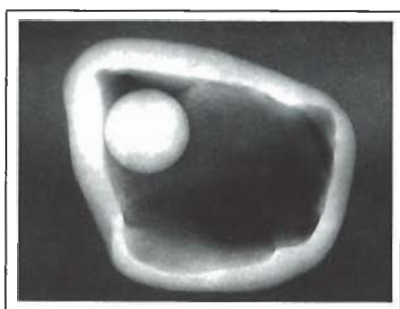


Figure 9. Testing proved that this 15.2 mm (diameter)  $\times$  25.1 mm (long) bead was a rock consisting mainly of dolomite with some quartzite.

look right about the color." The oval mixed cut measured approximately  $6.80 \times 4.90 \times 3.36$  mm and weighed 0.98 ct.

The R.I. of 1.762–1.770 identified the stone as corundum, and the presence of fluid-filled healing fractures confirmed it to be natural sapphire. However, no absorption features were visible with a desk-model spectroscope, and there was no reaction to long-wave ultraviolet radiation, indicating a treated color. Further, this stone showed no pleochroism, which is inconsistent with this depth of color even in a heat-treated sapphire. Subsequent examination of the stone in reflected light revealed purple iridescence on the pavilion facets (figure 10), which is unknown in natural sapphire, while a diffused darkfield environment revealed color concentrated along the edge of the pavilion surface. We concluded that the stone had been coated.

In an attempt to analyze the surface color concentration on the pavilion, GIA Research performed a qualitative chemical analysis by



Figure 10. Purple iridescence on the pavilion of this medium-dark 0.98-ct yellowish orange sapphire suggests that it has been coated.



Figure 11. After the sapphire shown in figure 10 had been soaked at room temperature for five hours in concentrated HCl, the depth of color was reduced considerably.



Figure 12. With additional soaking in HCl heated to a low temperature, the "yellowish orange" sapphire became completely colorless.

EDXRF. The analysis revealed Fe as the most abundant trace element, with small amounts of K, Ca, Ti, and Ga. A visible-range absorption spectrum of the stone, taken with a Pye-Unicam Model 8800 spectrophotometer, showed a sharp feature at 387 nm that is related to  $\text{Fe}^{3+}$ , and increasing absorption toward the ultraviolet starting about 500 nm. These results are consistent with natural-color orange sapphire and so did not, by themselves, help characterize the coating.

With the client's permission, we soaked the stone in concentrated hydrochloric acid at room temperature for five hours (figure 11). The depth of color was greatly reduced, although some of the coating remained on the pavilion. These more resistant areas were on facets with deep polishing lines or scratches. To complete the cleansing, we soaked the stone in more HCl at about 50°C for another two hours, at which point it was entirely colorless (figure 12). The beginning of increasing absorption in the visible spectrum shifted down to about 400 nm, but the sharp  $\text{Fe}^{3+}$  feature was unchanged; this suggests that the orange color of the coating resulted from broad absorption in the green and blue portions of the spectrum.

Although surface coatings such as the "Aqua Aura" process reported on topaz and quartz (see, e.g., *Gems & Gemology*, Fall 1990, p. 234, and *Journal of Gemmology*, April 1989, pp. 364–367) are com-

mercially available, this is our first encounter with such a coating on corundum.

Recently, there has been a great deal of research and development in dry thin film technology and its application in jewelry manufacturing (e.g., *American Jewelry Manufacturers*, May 1991). We would not be surprised to see a variety of coatings on more types of gems in the coming years.

TM and Ilene Reinitz

### Twinned SYNTHETIC SAPPHIRES

Within a short period of time, the East Coast laboratory encountered two synthetic sapphires that the clients had assumed were natural because of the presence of repeated

twinning (figure 13) and what appeared to be boehmite needles contained within the planes (figure 14).

Straight twinning lamellae were considered proof of natural origin until 1920, when this feature was noted as an anomaly in synthetic sapphire by Sandmeier, and corroborated by W. Plato. For a detailed description of this phenomenon, see the article "Polysynthetic Twinning in Synthetic Corundum," by W. F. Eppler (*Gems & Gemology*, Summer

Figure 13. The repeated twinning evident in this 17.95 × 13.90 × 6.80 mm synthetic blue sapphire had led the client to believe it was natural. Magnified 10×.



Figure 14. The presence of what appeared to be boehmite needles together with repeated twinning also created problems in the client's identification of this 26.00 × 13.12 × 9.20 mm reddish orange sapphire. Magnified 23×.



1964). More recently, we reported on the presence of these phenomena in the Summer 1984 (p. 111) and Spring 1989 (p. 38) Lab Notes sections.

We conclusively identified the two recent examples described above as synthetic sapphires by the presence of small gas bubbles and curved growth bands, which we observed with magnification and diffused

darkfield illumination. The appearance of repeated twinning in a synthetic sapphire can prove to be an identification challenge, especially when the stone in question may have been heat treated. Thermal treatment can diminish curved color banding, so that it is only evident when the stone is examined with immersion. TM

#### FIGURE CREDITS

Figure 1 is by Robert E. Kane. Figures 2, 4, 5, 7, and 10-14 are by Nick DellRe. Maha Smith took figure 3. Figures 6 and 9 are © GIA and Tino Hammid. The X-radiograph in figure 8 is by Robert Crowningshield. The Historical Note photo of the heart-shaped inclusion in diamond is by A. de Goutière.

## A HISTORICAL NOTE

### HIGHLIGHTS FROM THE GEM TRADE LAB 25, 15 AND FIVE YEARS AGO

#### WINTER 1966

The New York lab reported on some of Chatham's new synthetic flux-grown ruby. All four of the crystals examined contained natural sapphire seeds. The natural-seed centers were much more opaque to short-wave ultraviolet radiation than was the surrounding synthetic material.

Another interesting item concerned Mexican topaz that had been treated in the atomic pile at Brookhaven, New York, and altered to a very dark brown. This material may have been the predecessor of the treated blue topaz that is so much in evidence today. It would have been interesting to see if these stones turned blue with heat treatment, as does the topaz irradiated by today's methods.

Although the Los Angeles lab reported on different types of synthetic sapphires and on a one-plus-carat uvarovite crystal from Quebec, the most interesting item was a parcel of stones that arrived at the lab with a most fascinating letter. The client wrote that he had located a single deposit in California that produced blue and pink sapphires, green and brown peridot, zircon, almandite, diopside, enstatite, spinel, and even tiny diamond crystals.

The parcel of stones he submitted did, in fact, contain these materials, but only the small diamond

could be considered gem quality. He claimed that the deposit was a pipe with a quartz-feldspar dike intruding into it. Naturally, he was unwilling to be more specific as to location.

#### WINTER 1976

The Santa Monica laboratory had occasion to examine and photograph a very unusual laser-drilled diamond. Usually, when a diamond is laser drilled in an attempt to enhance its clarity, the drilling is done from the pavilion. This stone had between 25 and 30 drill holes through the table! Although not as readily apparent as one might expect when viewed perpendicular to the table, they were quite evident when viewed from any other angle.

The New York laboratory reported on various colored diamonds, both treated and natural. In addition, one fascinating photograph showed how the location of an inclusion can seriously affect the apparent clarity of a stone. In this instance, the single small inclusion was reflected in virtually every facet of the host diamond. If this inclusion had not been in this exact position, the stone would have looked like a VS<sub>2</sub> rather than an SI<sub>2</sub>.

#### WINTER 1986

The East Coast laboratory illustrat-

ed and discussed the separation of synthetic from natural amethyst by the presence of the twinning that occurs in natural amethyst and/or the spicules that are sometimes seen in hydrothermally grown synthetic amethyst

GIA's permanent collection, in Santa Monica, received a gift of two (rough and cut) rare clinohumites. The crystal weighs 1.72 ct and the cut stone, 0.39 ct.

Since this is the Winter issue, with St. Valentine's day just around the corner, it seems appropriate to show again the very rare heart-shape inclusion in a small (0.015 ct) diamond that was brought to our attention by Mr. A. de Goutière of Victoria, B.C., Canada.

*Note the heart-shaped cloud in this 0.015-ct brilliant-cut diamond. Magnified 12×.*

