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"Piranha" AGATE

The West Coast laboratory recently examined a well-polished oval cabochon of attractively patterned agate. This 17.02 ct cabochon showed an unusual "eye" structure that was equally interesting in appearance on both the dome and base (figure 1), although it differed in color between the two sides. While most so-called eye agates display a single circular-to-semicircular multi-layered struc-

tural pattern around a central acicular inclusion, in this particular piece a well-matched pair of "eyes" had been more-or-less centered in the host during fashioning.

The agate from which this 31-mm-long cabochon was fashioned was recovered from the Piranha River in Ecuador, hence the name "Piranha" agate. Agates from this locality are commonly colored red-brown, orange, yellow, and black by iron compounds, with the patterns set off against a

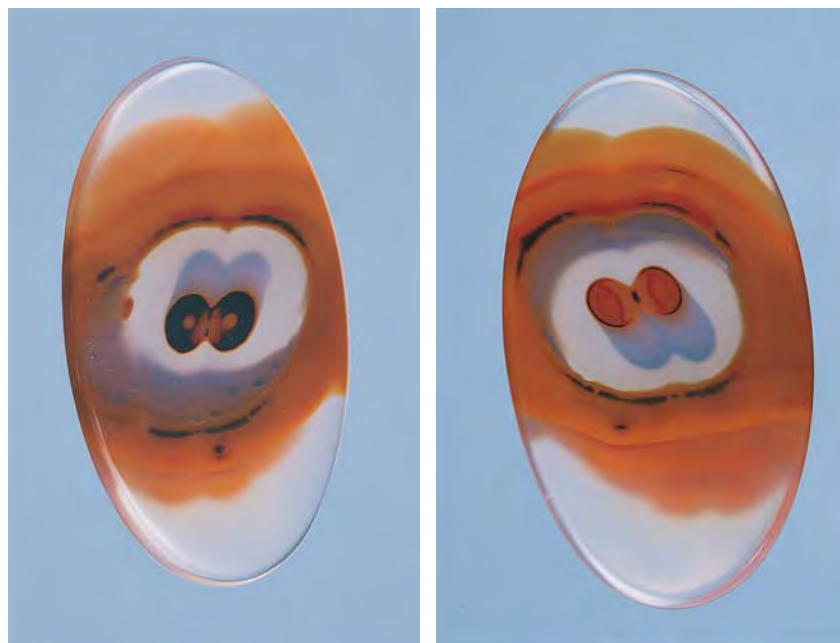
background of translucent bluish white to white chalcedony. A display of these agates at the Tucson gem show in February 2002 showed similar colors and patterns. Raman analysis of the agate examined in our laboratory identified hematite and goethite as possible mineral colorants in both the black and orange areas, although the characteristic peaks were weak and poorly defined.

Another interesting feature of this agate was the way it was cut. Even though the cabochon was only 3.69 mm thick, the steep angle of the "eye" tube structure created an illusion of much greater depth (again, see figure 1). We do not know if this was intentional. None of the Piranha agates displayed in Tucson showed this pseudo-depth illusion.

While agates are one of the most common gem materials, beautifully patterned examples are unusual, and those suitable for jewelry applications are much rarer. Since no two patterns are ever the same, agates such as this one could be used by an imaginative designer to create a very distinctive piece of jewelry.

JK and Maha Tannous

Figure 1. This colorful 31-mm-long "Piranha" agate from Ecuador shows an interesting double-eye pattern on both its dome (left) and base (right), although the "eyes" are dramatically different.



Editor's note: The initials at the end of each item identify the editor(s) or contributing editor(s) who provided that item. Full names are given for other GIA Gem Trade Laboratory contributors.

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Surface-Treated AMBER

In the Winter 2000 issue of *Gems & Gemology* ("Gemstone enhancement and detection in the '90s," pp. 336–359), S. F. McClure and C. P. Smith reported on surface-enhanced amber in which a shallow dark brown surface layer is produced by controlled heating (figure 2). We have found that this treatment actually results in a wide range of colors, from brownish yellow to dark red-brown (see, e.g., figure 3, left). In numerous samples that have come through the lab, we have seen that the heating physically alters the surface of the amber and, thus, some of its gemological properties. In fact, the properties of some samples of this treated amber are closer to those reported for plastic than amber, which makes the identification a challenge.

We have noted that the refractive index of the surface-enhanced amber increases with the depth of color: that is, the darker the color of the surface layer, the higher the R.I. We have recorded a range of spot R.I.'s for this treated amber, with the highest being 1.60 for the darkest red-brown material. This is a significant deviation from the 1.54 R.I. characteristic of untreated amber.

We have also noticed an inverse relationship between the darkness of the enhanced color and the material's reaction to ultraviolet (UV) radiation: the darker the surface color, the lower the intensity of fluorescence. The surface enhancement tends to quench the strong chalky yellow and/or blue fluorescence characteristic of amber in long- and (weaker) short-wave UV (figure 3, right). The darkest treated material we have tested typically was inert or had only very weak brownish orange fluorescence to both wavelengths.

An amber bead necklace recently submitted to the West Coast laboratory for identification provided additional information. The beads showed a range of colors, including yellow, yellow-orange, orange-brown, and dark red-brown. The client was



Figure 2. The thin dark brown surface layer that originally covered this cabochon (39.25 × 25.70 × 9.05 mm) has been polished off the top, so it occurs only on the back (see inset). This allowed the carver to cut flowers into the pale yellow body of the piece so they would stand out against the remaining dark brown surface layer that was created by controlled heating.

particularly interested in the darkest beads, since their properties were quite different from those that have been reported for amber. We noted in all but the yellow beads that the varying depths of color correlated to the variations in refractive index and

fluorescence that we had noted in other samples of treated amber, as described above.

Since the properties at the surface of this treated material are not consistent with those reported for amber, how do we identify it? Specific gravity

Figure 3. Some surface-enhanced amber appears orangy brown in standard illumination, as evident in this 25-mm-diameter hololith. With exposure to long-wave UV radiation (right), a weak brownish orange fluorescence, atypical of amber, is seen on most of the hololith; the strong chalky blue fluorescence commonly seen in amber is visible only in the small area where the surface layer has been polished off. The R.I. of the dark surface was 1.59, and that of the underlying yellow material was 1.54.



is useful for loose samples, as we have observed no difference in the S.G. recorded for loose treated and untreated material. In both cases it is around 1.08, which is significantly lower than the S.G. of most plastics used as amber imitations. Another consistent property is amber's odor when touched with a thermal reaction tester. Treated or untreated, amber will emit a resinous odor, as opposed to the typical acrid odor of most plastics. Nevertheless, this identification can be especially tricky when the material is mounted in jewelry.

To learn more about this surface-treated amber, we acquired five samples for research purposes and polished through their dark surface layers. In all cases, this layer was extremely thin, and the underlying material was very pale yellow—much paler than the typical color of untreated amber. Changes in R.I. and fluorescence occurred only at the darker, heat-induced surface; the underlying amber retained the usual properties. Removal of the surface layer is obviously destructive and therefore not feasible in most cases. However, the difference in fluorescence may sometimes be viewed at small chips around drill holes or on girdle edges. Observation of this difference between surface and internal fluorescence can be a useful aid to identification, since plastic imitations would not react in this way.

We previously reported on amber with similar surface-related coloration that faded on exposure to light (see Summer 1993 Lab Notes, pp. 122–123). That report did not note any variances in refractive index, although it did mention an unusual orange fluorescence. We did not test the samples mentioned above for color stability.

The conclusion on GIA Gem Trade Laboratory reports for this material is accompanied by the following comment: "The color of this amber is primarily confined to a shallow surface layer that has been artificially induced by heat treatment. Such color may fade."

Elizabeth Quinn and SFM

Unusual CERAMIC Gem Simulant

Laboratory gemology is not always concerned with the identification of treatments and natural-versus-synthetic determinations. Occasionally, we have the distinct privilege of examining items that are downright baffling. A good example from the recent past was an iridescent piece of hard plastic that its owner believed was an opal. The micro- and macro-structure, texture on the broken surface, and curvature of this specimen made us suspect that it was a fragment from a bowling ball. Recently, the West Coast laboratory examined an unpolished hemispherical "cabochon" of dense white material that a pearl distributor had submitted for identification.

This hemisphere was opaque (figure 4) with a sawn back; closer examination revealed two parallel mold marks extending diagonally across the dome. It weighed 125.39 ct and measured approximately 20 mm in diameter. We could not take a refractive index reading because of the lack of a polished surface.

No visible spectrum was observed in surface-reflected light through a prism spectroscope. When exposed to long-wave UV radiation, the item showed a moderate, somewhat mottled brownish red luminescence, while the reaction to short-wave UV was pink rather than red and much

Figure 4. Represented as a pearl, this 20 mm opaque white "cabochon" was identified as a molded form of ceramic.



weaker. Hydrostatic specific gravity was calculated, from an average of three readings, as 3.57.

With magnification, the two parallel mold seams were clearly visible, as were saw marks on the base. In addition, small, dull, conchoidal fractures were evident around the edge of the base, and the material itself revealed a very fine granular texture, reminiscent of a ceramic, such as porcelain that would be used in various common bathroom fixtures. This was clearly a manufactured product and not a pearl.

Since we are not often called on to identify ceramic materials, we performed energy-dispersive X-ray fluorescence analysis to complete the examination. This showed the presence of aluminum, barium, calcium, gallium, iron, strontium, and titanium, with Al, Ga, and Sr being dominant. Laser Raman microspectrometry showed a broad curve, as would be expected for an amorphous substance such as a glass, but no sharp peaks.

JIK, KNH, and Sam Muhlmeister

CORUNDUM

More Bulk Diffusion—Rubies and Orange Sapphire

In the Fall 2002 Lab Notes (pp. 254–255), we described an orangy yellow sapphire that had been subjected to bulk (also called lattice) diffusion with beryllium. *Bulk* or *lattice diffusion* is defined as "a diffusion process which takes place through the bulk lattice of the crystal and excludes such mechanisms as short circuit diffusion along dislocations, grain boundary diffusion, and surface diffusion" (Kizilyalli et al., "Definitions of terms for diffusion in the solid state," *Pure and Applied Chemistry*, Vol. 71, No. 7, 1999, pp. 1307–1325). In terms of this corundum treatment, the orange component of the color is a result of beryllium diffusion into the bulk lattice of the crystal, which creates a Be concentration gradient, as opposed to Be diffusion within grain boundaries or on the surface. We continue to investigate the range



Figure 5. The color in this 0.63 ct oval mixed-cut ruby was produced by bulk diffusion treatment with beryllium.

of corundum that has been subjected to this diffusion treatment and the color changes that result.

Figure 5 shows an attractive oval mixed-cut ruby with clear evidence of heat treatment that was submitted to the East Coast laboratory for routine testing. Although we were not able to identify the exact nature of the inclusions, the stress fractures emanating from them—as well as the partial healing of these and other fractures—indicated that the heating conditions were extreme (see figure 6). Examination of the stone while it was immersed in methylene iodide revealed what appeared to be a very shallow orange rim or other form of color concentration near the ends of the sample, but we could not conclusively determine if this was the case. Immersion observations also revealed a few small areas on the surface that showed differences in luster, which indicated that a glass-like residue had filled shallow cavities during the heating process.

Suspicious that this ruby might have been subjected to a diffusion treatment comparable to that more commonly seen in orange-pink and yellow sapphires, we performed chemical analysis using secondary ion mass spectrometry (SIMS). As previously described (see, e.g., Spring 2002 Gem News International, pp. 86–90), this method allows for the detection of trace amounts of light elements to

the level of parts per million (ppm). The SIMS analysis confirmed our initial concern: The concentration of Be at the surface of this stone was about 11 ppm. Based on our previous analyses, this is much higher than the Be concentrations in either natural, untreated sapphires (typically less than 1 ppm) or those heated by traditional methods, and it is within the concentration range that would produce an orange rim and alter the intrinsic color of the ruby. We are not certain what the starting material looked like, but we suspect that it was purplish and much darker.

About a week after the previous stone was submitted for testing, we received a second natural ruby of approximately the same color. As with the previous stone, this ruby displayed evidence of heating. In this case, the evidence consisted of dust-like rutile surrounded by color concentrations indicative of internal diffusion, which had been dissolved, and also healed “fingerprints.” Exami-

Figure 6. Stress fractures and partially healed “halos” surrounding included crystals in the ruby shown in figure 5 provide clear evidence that the stone was subjected to extremely high temperatures. Magnified 63×.



Figure 7. This 2.78 ct oval mixed-cut ruby appears to have been treated by the same method as the ruby shown in figure 5.

nation in immersion clearly revealed a rim of orange color that followed the facet outline. Such color zoning proved that this stone also had been subjected to lattice diffusion treatment. The ruby illustrated in figure 7 was treated in a similar manner. These examples of orangy red ruby are typical of what we have begun to associate with this type of Be diffusion.

Most recently, Yoshiko Doi, president of GIA Japan, sent us an orange sapphire that was clearly heated and in fact had not been repolished after its removal from the crucible (figure 8). Microscopic examination showed the corroded surface one would expect for a heated sapphire, but it also revealed numerous areas of new crystal growth. Such recrystallization is often seen in rubies and sapphires that are subjected to high temperatures with fluxing agents (see, e.g., Fall 2002 Lab Notes, pp. 255–256), but we were surprised at the extent of the new crystal growth. Although most of the new crystals were similar in habit to tabular corundum (figure 9), we turned to chemical analysis to positively identify them.

One of the contributors (MH) collaborated with researchers at the Laboratory for Mineral Deposits Research at the University of Maryland, where the stone was examined with a JEOL JXA-8900R electron microprobe with imaging capabilities (figure 10). Chemical analysis of the



Figure 8. The unpolished surface of this 0.48 ct orange sapphire displayed obvious signs of heat treatment.

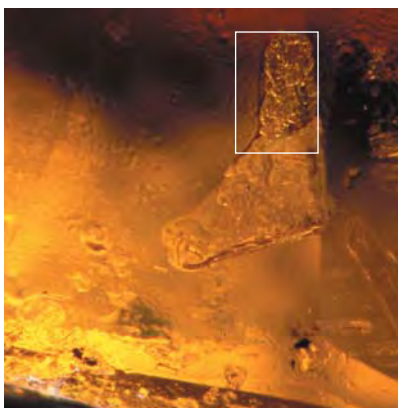


Figure 9. Microscopic examination (at 45×) of the sapphire in figure 8 revealed tabular crystal regrowth after extreme heating.

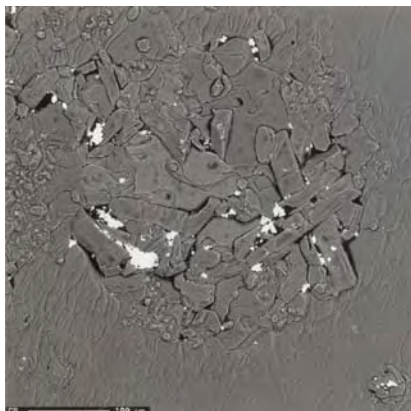
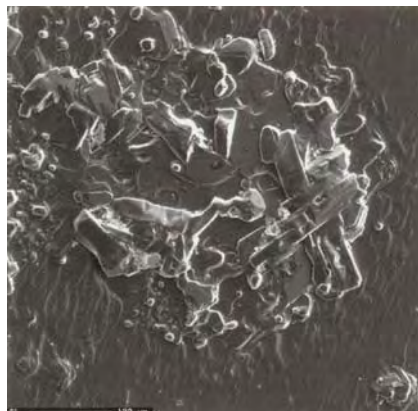
tabular regrown crystals by energy-dispersive spectrometry (EDS) indicated that they were corundum. It appears that the extremely high temperatures being used, combined with fluxes, essentially dissolve part of the surface of the corundum; this serves as a feed source for new corundum growth that develops during cooling. SIMS analysis of the unpolished surface revealed a Be concentration of 99 ppm, far higher than that seen in repolished sapphires that were apparently treated

by the same method. This high Be concentration confirms that Be was diffused into the sample during the annealing process.

Thus far, we have confirmed Be lattice diffusion as the cause of color in pinkish orange, orange, orangy red, and yellow sapphires, as well as in ruby. This treatment is indeed being used to produce a very wide range of colors in corundum.

TMM, Matthew Hall,
and Wuyi Wang

Figure 10. These secondary electron (left) and backscattered electron (right) images of the treated sapphire in figure 8 clearly show the tabular habit of the newly grown crystals. EDS chemical analysis indicated that these crystals are corundum. The bright spots in the image on the right are remnants of silver powder used for SIMS analysis. The scale bar is 100 μm .



DIAMOND

With Internal Inscriptions

Diamond inscriptions are typically created by lasering characters on the girdle surface. Recently, though, the East Coast lab has seen three inscriptions that were totally within the interior of the diamond. Two different methods appear to be responsible.

The first two diamonds had characteristics similar to those caused by internal laser drilling, as documented by S. McClure et al. ("A new lasering technique for diamond," Summer 2000 *Gems & Gemology*, pp. 138–146). It is possible that the technology described in that article was a collateral effect of early experiments to place internal inscriptions in diamonds. The inscription in figure 11 was placed on an internal fracture. The black letters "NE" are clearly visible. The other small internal fractures in the same area have black lines extending along their length. These black lines are typical of those associated with fractures created during the internal laser drilling process. It appears that the black lines on this fracture were controlled to create the letters.

Figure 12 shows a similar internal inscription. In this case, the number "2" is inscribed on a small fracture present in this 2.14 ct oval brilliant.

The inscription "31107345" in the third stone, a 1.01 ct rectangular modified brilliant, was also totally internal, but had a much different appearance (figure 13). We believe ion implantation is responsible for this second type of inscription, because it is not associated with any inclusions. This is a method (also known as doping) whereby ions (charged atoms) are introduced into a solid through their high kinetic energy (see R. Kalish and S. Prawer, "Ion implantation of diamond and diamond films," in M. A. Prelas, G. Popovici, and L. K. Bigelow, Eds., *Handbook of Industrial Diamonds and Films*, Marcel Dekker Inc., New York, 1998, pp. 945–982). By bombarding the diamond with atoms of either argon or boron, one can make "marks" inside the stone.

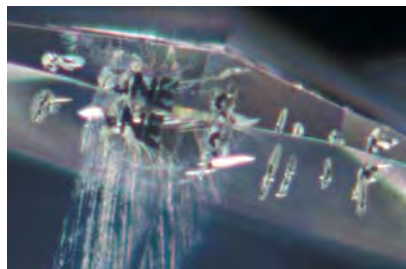


Figure 11. This 0.57 ct diamond contains a series of internal fractures with black lines, typical of those created by a recently documented internal laser drilling process. One of these fractures, located just behind the prominent, surface-reaching feather, has the letters "NE" inscribed on it. Magnified 63 \times .



Figure 12. This internal fracture has the number "2" inscribed on it. Magnified 63 \times .



Figure 13. A mirror reflection is proof that this inscription in a 1.01 ct diamond is totally internal. Ion implantation is thought to be responsible for the inscription. Magnified 63 \times .

The marks are actually slight deformations in the crystal lattice that scatter light, and thus they tend to show up as thin white lines. Note that the depth of the inscription is uniform, and that it is suspended a uniform distance below the surface (i.e., a few tenths of a millimeter). This can be achieved by controlling the energy of a beam of ions of a particular size.

It is possible that these three internal inscriptions were done for trial purposes, since we have seen only a limited number of them to date. However, given the apparent high degree of control in the internal laser process, we believe such inscriptions may become more common. Internal inscriptions would have one benefit over the typical surface inscription in that they would be extremely difficult to remove. However, creating fractures in a diamond or printing letters below the surface would amount to adding inclusions to a stone, which could have a direct effect on the clarity grade.

Joshua Sheby and Vincent Cracco

Two Large GLASS Imitation Jade Carvings

It has been said that imitation is the sincerest form of flattery, and given

the high value of jade, it is not surprising that it is so frequently imitated. Over the years, many jade imitations have been reported in both Lab Notes (see, e.g., Fall 1983, pp. 173–174; Summer 1996, p. 123; Summer 2001, p. 133) and Gem News (Winter 1990, p. 309; Summer 1995, p. 137). Most of these items were small—loose cabochons or carvings set in jewelry.

This past fall, however, one client sent the East Coast laboratory two large carvings for identification: one approximately 35.00 \times 14.00 \times 9.00 cm (see figure 14), and the other approximately 50.00 \times 16.00 \times 8.00 cm. Each was packed in a padded box that was ornately decorated. The client told us the two carvings recently had been purchased together in Asia as jadeite jade for a large amount of money.

Both items were a semi-translucent to opaque very light grayish green, and both appeared to be carved from the same material (the results of their testing were identical). The polish was poor, and the refractive indices were in the low 1.50s. The objects fluoresced very weak yellow to long-wave UV radiation, and weak to medium yellow to short-wave UV. Close examination revealed no crystalline structure and a few tiny conchoidal chipped areas that displayed vitreous luster. Horizontal illumination with a pinpoint fiberoptic light showed small but eye-visible round and elongated gas bubbles throughout both items, along with the swirled flow lines that are commonly seen in glass. This narrowed the identification to glass or plastic. Testing

with hardness points on inconspicuous areas demonstrated that the material had a Mohs hardness greater than 5, thereby eliminating plastic. Both were

Figure 14. This large figurine (35 \times 14 \times 9 cm), purchased in Asia as jadeite jade, was identified as manufactured glass.



identified as “Glass, a manufactured product.”

In this era of new treatments and ever-increasing synthetics, these carved items should serve as a reminder that some of the oldest and least expensive imitations are still being offered, and gemologists should not dismiss such possibilities because of the apparent age or simplicity of the piece.

Wendi M. Mayerson and KNH

JADEITE

Bleached, Impregnated, and Dyed, with Unusual Inclusions

A translucent, variegated green bangle bracelet was recently submitted to the East Coast lab for identification. Standard gemological testing proved the bangle to be dyed jadeite jade: an R.I. of 1.66, a 437 nm line and 650 nm “dye-band” visible with the desk-model spectroscope, an aggregate structure, and color concentrations along grain boundaries. Reflected light in combination with magnification revealed an unusual surface texture, the result of preferential erosion of certain grains during the “bleaching” process commonly associated with “B jade” (see, e.g., Winter 1994 Lab Notes, pp. 266–267). The randomly oriented interlocking grains of jadeite become visible as the acid-soluble

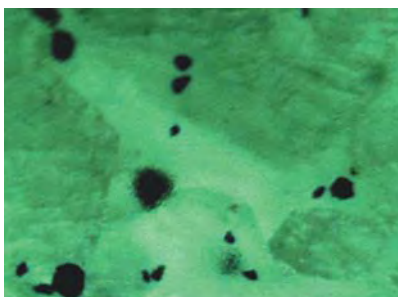
Figure 15. With reflected light and magnification (here, 50×), the differences in luster between the jadeite grains and the filled channels and small cavities surrounding them can be seen.



Figure 17. Several of the “golden” yellow cultured pearls in this graduated strand (12–15 mm) proved to be enhanced.

minerals that lay between them are removed, creating narrow outlines around the grains and small cavities between them. These channels and cavities are then filled with either a polymer or a wax. Reflected light and magnification also revealed the differences in luster between the jadeite grains and the filled channels and small cavities (figure 15), which indicates that this bracelet was indeed impregnated. The fact that the bangle fluoresced a medium mottled greenish yellow to long-wave UV radiation, and a very weak mottled yellow to short-

Figure 16. The dark violetish blue inclusions seen “floating” in the filler are visible indications that this jadeite bangle bracelet has been treated. Here, they appear black due to the lighting conditions. Magnified 300×.



wave UV, further indicated impregnation (see Spring 1995 Lab Notes, p. 55).

All jadeite submitted to the GIA Gem Trade Laboratory is checked for impregnation via infrared spectroscopy. Such testing confirmed that this bracelet was impregnated, and it was officially identified as “dyed and impregnated jadeite jade.” Although infrared spectroscopy is currently the most efficient method to prove impregnation (see E. Fritsch et al., “Identification of bleached and polymer-impregnated jadeite,” Fall 1992 *Gems & Gemology*, pp. 176–187), its use is generally restricted to laboratories or large companies due to the high cost of the instrument. Therefore, when visible indications are present, they are extremely helpful for the practicing gemologist.

Such was the case with our bangle bracelet. Not only did the visual clues mentioned above serve as useful indicators of treatment, but when the bangle was viewed in combined transmitted and reflected light with the added strength of a fiber-optic light, several dark inclusions could also be seen “floating” in a lower-luster (filled) area (figure 16). These were reminiscent of the tiny green spherules and gas bubbles discovered in the polymer of a bangle bracelet examined earlier (Spring 1999 Lab Notes, pp. 44–45). What

made these new inclusions so unusual was the fact that, although the bracelet was dyed green, these inclusions were dark violetish blue and found in only two small areas of the filler. Even though we could not determine their identity, they are another good visible indication that the bracelet was treated.

Siau Fung Yeung and
Wendi M. Mayerson

CULTURED PEARLS

With Treated Yellow Color

For the past several months, the East and West Coast laboratories have seen an increasing number of strands of "golden" yellow pearls submitted for identification. These graduated strands all featured fairly large (12–15 mm) yellow pearls that were well matched in color and luster. X-radiography, reaction to long-wave UV radiation, and visual inspection readily identified the majority as cultured pearls of natural color. However, a few cultured pearls in one necklace (figure 17) had characteristics that ultimately proved their color was enhanced.

Routine visual inspection revealed that some of the cultured pearls had peculiar opaque white non-nacreous areas on the surface. A few of these growth features actually represented depressions in the nacre, some with a



Figure 18. The red spots in these different types of growth marks seen on "golden" cultured pearls indicate treated color. Although here, because of the lighting conditions used, the growth marks appear yellow, they were actually white. Magnified 15×.

central opening. While examining these cultured pearls with long-wave UV to determine the origin of color, we noticed that the greater part of the surface area fluoresced yellow-green, but the nonnacreous areas fluoresced yellowish white and their centers were inert. Examination of these areas with strong overhead illumination at 15× magnification revealed isolated bright red spots (see figure 18). One such growth mark also showed a small opening in the nacre that had a bright red rim. Since this type of spotty red coloration does not occur naturally in yellow pearls, we believe it must have been the result of treatment.

This is not the first time we have seen such evidence of color enhance-

ment in yellow cultured pearls. This contributor recalls having noticed similar features in known treated yellow cultured pearls at least five years ago, just as "golden" pearls were gaining in popularity. The presence of such characteristics allows the practicing gemologist to conclusively identify treated color in yellow cultured pearls.

KNH

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Vincent Cracco—figures 6, 8, 9, 11, 12, and 13;
Wendi Mayerson—figures 15 and 16;
John I. Koivula—figure 18.

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