

LAB NOTES

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AMBER, Damaged in Cleaning

Recently the East Coast laboratory received a telephone call from an acquaintance who was quite agitated as she described the appearance of a necklace of amber beads that she had attempted to clean by immersion in denatured alcohol. Some of the beads had turned chalky white and others had lost all luster. She explained that she often "washed" natural and imitation stone bead necklaces in this fashion, with great results. If it worked with those materials, why not amber?

While she was still on the telephone, we selected a piece of amber and dipped it into the alcohol we use to clean diamonds while grading. In an instant, our sample was "ruined." Figure 1 shows the 30-in. (75 cm) necklace that she subsequently sent to us for examination.

A check of the literature for information about the reaction of amber to alcohol turned up nothing. In fact, one source implies that amber is attacked only by acids and strong solvents. (One test for amber is its resistance to ether—surely a strong solvent.) Evidently, the alcohol in perfumes does not affect amber, or surely it would have come to our attention by now.

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

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Figure 1. When these amber beads (10.25 × 7.34 mm to 25.30 × 16.75 mm) were "washed" by immersion in alcohol, many of them acquired a white film that could only be removed by repolishing.

Our friend's necklace had been assembled to illustrate the broad range of color and quality of amber from Santo Domingo. Resistance to alcohol appears to be another variable. Several of the lighter, more transparent beads showed little damage, while most of the beads that were darker and more translucent developed an opaque white layer. Repolishing the beads removed the coating. The reaction of these beads makes it evident that alcohol should

be included among the list of solvents that may attack organic materials such as amber. GRC

DIAMOND

Electron-Treated, Large

The East Coast laboratory has seen a marked increase in diamonds that have been color treated by electrons. With magnification and diffused illumination, electron treatment can be

recognized in some diamonds by the presence of a zone of color at the culet or along facet junctions (as discussed in the article by Fritsch and Shigley, *Gems & Gemology*, Summer 1989, pp. 95–101).

Recently, we examined the largest electron-treated diamond we have seen to date in this lab. The 37.43-ct radiant-cut yellow diamond measured approximately 22.14 × 16.98 × 11.48 mm. A yellow color zone, typical of that produced by electron treatment, was observed in the culet area. DH

Electron Treated, in Period Jewelry

At about the same time, the East Coast lab examined a yellow old-European-cut diamond, set in a white metal Art Deco brooch, that also revealed the tell-tale yellow “cone” around the culet that proves electron treatment. The stone measured approximately 7.00 × 5.00 mm.

The curious thing was that the brooch was probably manufactured before electron treatment was available. Although the cutting style of the treated stone matched that of the other diamonds in the pin, careful inspection revealed that the bezel setting encasing that stone had been replaced. This suggests that the original stone was either (1) unmounted, treated, and reset or (2) replaced with a treated diamond that was carefully selected to match the other stones in cutting style. TM

Fracture Filled

Over the last few years, the filling of fractures in diamonds has been the subject of great concern and study. A comprehensive article published in the Summer 1989 issue of *Gems & Gemology* (Koivula et al., “The Characteristics and Identification of Filled Diamonds,” pp. 68–83) discussed the orange/blue flash effect that is a major identifying characteristic of a filled stone. In that article, however, near-colorless diamonds were used to illustrate this feature with the exception of one yellowish brown dia-

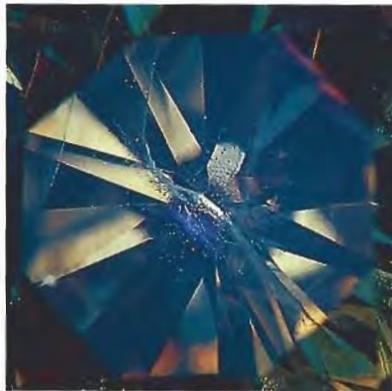


Figure 2. A purple flash is readily visible in this filled yellowish brown diamond when viewed with darkfield illumination and 20× magnification.

mond. This exception pointed out the fact that unless the stone is oriented properly, the flash effect might be more difficult to detect with darkfield illumination in colored than “colorless” diamonds.

A subsequent update (*Gem News*, *Gems & Gemology*, Spring 1990, pp. 103–105) illustrated a near-colorless diamond with new flash-effect colors: pinkish purple and yellowish green.

Recently, the East Coast laboratory examined an obviously filled yellowish brown diamond with the “new” flash-effect colors (figure 2). Unlike the “old” (orange/blue) flash-effect colors in the fracture-filled yellowish brown diamond shown in the 1989 article, the “new” (purple/green) flash-effect colors are readily visible with darkfield illumination and magnification. Figure 3 shows both orange and green flashes in this same stone. Nicholas DelRe

Green Surface-Colored Rough Fashioned to a Fancy Light Yellow

A client recently allowed the East Coast laboratory to follow the progress of a 22.94-ct light green rough octahedral diamond through sawing, cutting, and polishing and record its gemological and spectral characteristics at each stage.



Figure 3. In the same stone as figure 2, these filled fractures reveal both the “original” orange flash-effect color and its newer green counterpart. Magnified 15×.

Examination of the rough diamond with both the unaided eye and a gemological microscope, using diffused illumination, led us to speculate that the green color was confined to the surface. Once the crystal was cut and polished, we believed, the green color would disappear and the resulting faceted stones would have a yellow body color.

Infrared spectroscopy of the rough crystal revealed that it was a type IaA + B, with the body color due to absorption caused by the presence of nitrogen. The crystal displayed the classic Cape absorption lines in the Beck hand-held spectroscope.

We then examined the rough crystal with a Pye Unicam Model 8400 UV-VIS spectrophotometer to look for absorption lines associated with the radiation damage to the lattice structure that contributes to a green color. The crystal did reveal this type of absorption, known as the GR1, with its characteristic sharp band at 741 nm.

Although we saw no natural brown or green radiation stains on the rough crystal, we believe that the green coloration was natural. The stone was reported to be from Angola, and Central Africa is known to be uranium rich; natural radiation from uranium can affect the coloration of diamond crystals. Further-



Figure 4. Even after a diamond with what appeared to be a green "skin" was sawed, the green surface color was still prominent. The 6.56-ct piece is shown here.

more, there was no artificial color zoning such as is induced by electron or cyclotron treatment.

The crystal was then sawed into two pieces, 15.93 and 6.56 ct. Although these two pieces retained the original greenish coloration (figure 4), we were surprised that both showed a very weak absorption line at 503 nm that was not present in the original piece of rough. Perhaps this was caused by the heat of sawing, which required three eight-hour days to complete.

The larger piece of rough yielded an 11.01-ct radiant cut with no trace of green color visible; this stone was graded "fancy light yellow." Thus, our original speculation about the final color proved to be correct. The U.V.-visible spectrum of the fashioned stone also showed that the GRI radiation absorption band was no longer present, undoubtedly because the green surface had been completely removed in fashioning. *DH*

Green, with Radiation Stains in Etch Channels

We have often noted a relationship between a face-up green color in diamonds and the presence of brown or green radiation stains. In many polished stones where the surface is entirely removed during cutting,

these stains are confined to etch channels, with figures 5 and 6 being two good examples. The striated or sculpted surfaces of these epigenetic features show specific preferential attack at defects in the crystal structure caused by the etching fluid. Etch channels are usually V shaped and narrow as they penetrate the crystal. Thus, the narrow portion of the channel remaining on a polished diamond may appear at first glance to be a mechanical fracture or cleavage. However, the absence of a fracture or cleavage surface suggests that it is actually the result of etching.

A possible explanation for this association of radiation stains and etch channels is that the same fluid causes both. Diamonds have been etched in the laboratory (producing etch channels, trigons, and other features observed in natural diamonds)

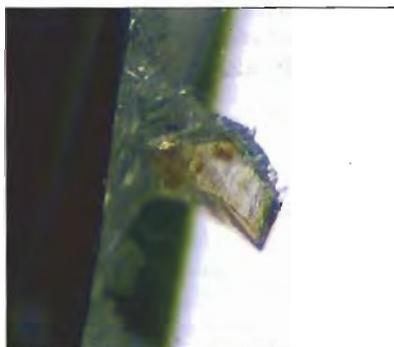


Figure 5. Brown irradiation stains are evident in this etch channel at the girdle of a very light green 2.49-ct old-mine-cut diamond. Magnified 45 \times .

with potassium chlorate and sodium perchlorate, potassium and sodium nitrate and hydroxide, and silicate melts including kimberlite (as summarized in Orlov's *Mineralogy of the Diamond*, English edition published by John Wiley & Sons, Toronto, 1977, pp. 85–103). Any fluid that contains potassium will include some of the long-lived radioactive isotope K-40, which makes up 0.01% of all potassium in nature. In addition, two



Figure 6. Green stains line the etch cavities that extend into this 0.60-ct marquise-cut diamond and may contribute to its overall color. Magnified 50 \times .

other long-lived radioactive nuclides, thorium-232 and uranium-238, are frequently found in natural fluids rich in potassium. Whatever the precise composition of the etching fluids that attack natural diamonds, irradiation could easily occur at the same time.

Such radiation stains, which line the channels and penetrate into the diamond approximately 1 or 2 mm, may be the cause of the apparent color in the stone shown in figure 6. The "stained" channel illustrated in that figure is just visible along the lower edge of the diamond at its belly. There is another, similar stain in the pavilion at the left point of the marquise-cut stone. The presence of these stains is a strong indicator of natural color, although further corroboration is usually necessary.

TM and Ilene Reinitz

PEARL

Cultured "Demi-Pearl"

Staff members at the West Coast lab had the opportunity to examine a unique pearl formation. At first glance, the 6-mm round drilled bead (figure 7) resembled another unusual pearl formation that we had described as a "demi-pearl" in the Fall 1989 Gem Trade Lab Notes section.

Like the earlier sample, the two halves of this bead also were different

colors: The white part showed the pearly luster and orient commonly associated with fine pearls, while the medium dark pinkish brown part showed some areas of darker color concentration, but no other structural characteristics. Visually, the darker portion resembled the calcareous concretions produced by clams or oysters. Since this pearl was drilled, we were able to see through the drill hole a distinct demarcation between the bead nucleus and the fairly thin overlying partly nacreous, partly concretionary layer. The X-radiograph clearly showed the difference in transparency between the bead nucleus and the top layer, thus proving that this "demi-pearl," unlike the one previously described, was the result of a culturing process. As would be expected in a cultured pearl, the fluorescence of the shell nucleus to X-radiation was visible through the lighter-colored nacreous layer, but it was masked by the darker pinkish brown layer. Again, we do not know under what circumstances this unusual "demi-pearl" was formed in the mollusk. KH



Figure 7. Half of this 6-mm cultured "demi-pearl" is nacreous and half resembles a calcareous concretion.

Freshwater "Rosebud" Pearls

The necklace of graduated freshwater pearls shown in figure 8 generated great interest in the East Coast laboratory. It consisted of natural freshwater "rosebud" pearls ranging from



Figure 8. The bumps, ridges, and high luster of the graduated pearls in this necklace strongly suggest that they are freshwater "rosebud" pearls, which are characteristically found only in American waters.

approximately $6.70 \times 4.80 \times 3.20$ mm to $15.50 \times 13.80 \times 5.50$ mm. Although a few pearls appeared to be slightly out of place, the overall match of color, overtone, and luster was consistent.

This necklace resembled those illustrated and described in the Spring 1989 (p. 35) and Fall 1990 (p. 220) Lab Notes sections. It appears to be an American product from the *Unio* mussel and probably was assembled in the early 1900s, when American pearl production peaked.

"Rosebud" pearls have been described by Sweaney and Latendresse (*Gems & Gemology*, Fall 1984, pp. 125-140): "This characteristically American freshwater pearl is typically high domed, with a flat back and roughly roundish outline." The pearls in this necklace also had the prerequisite bumps and ridges as well as a high luster. As expected, the pearls luminesced strongly to X-rays, and the X-radiograph showed only a few features in the structure.

TM

Rare Assembled Cultured Blister Pearls

At first glance, the beads shown in the ring in figure 9 appeared to be $3/4$ cultured blister pearls, which were first described in *Gems & Gemology*

in the Summer 1981 issue. However, the X-radiograph (figure 10) taken in our East Coast laboratory indicates that these are "plugged" cultured blister pearls with relatively thick nacre. That they were grown on an Australian *Pinctada maxima* yellow-lip shell is shown by the yellow worked area of shell evident around their backs. One might be tempted to call them mabe pearls, but that would be misleading since almost all mabe pearls are grown in the black-winged pearl oyster, *Pteria penguin*, which is called mabe by the Japanese.

Characteristically, mabe pearls have a very thin nacreous layer that is grown around (but does not adhere to) steatite (soapstone) hemispheres, which are discarded when the blister is sawed from the shell. After it is cleaned thoroughly, the inside of the thin nacre may be lacquered to enhance the color; then a hemisphere of mother-of-pearl is cemented into the cavity and covered with a low cabochon shell base. This contrasts with the $3/4$ cultured blister pearls, which retain their large saltwater shell nuclei and require no assemblage. Because of the setting, we could not determine how these "plugged" cultured blister pearls were finished on the base.

GRC



Figure 9. Unlike mabe pearls, these "plugged" cultured blister pearls (approximately 17 mm in diameter) have a relatively thick nacre and were grown in an Australian *Pinctada maxima* oyster.

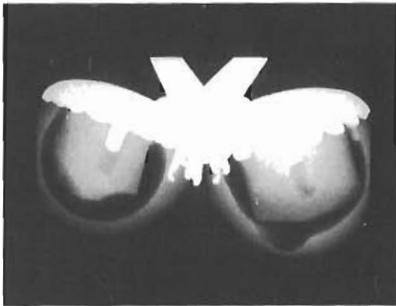


Figure 10. The X-radiograph of the cultured blister pearls in figure 9 clearly shows the "plugs."

An Early(?) Flux-Grown SYNTHETIC RUBY

When the staff of the New York laboratory first reported their examination of flux-grown synthetic rubies in the Spring 1965 issue of *Gems & Gemology*, they undoubtedly referred to something similar to the synthetic ruby shown in figure 11, which was recently submitted to us. However, at that time the journal was not published in color, and the fact that the seed crystal used to initiate crystal growth was outlined in blue was not noted. Later, in the Winter 1969–1970 issue, they did note the blue outline, but could give no explanation for it; again, the ma-

terial was not illustrated in color.

We can now speculate that the presence of iron and titanium in the natural corundum seed crystal provides the mechanism for the blue color. It is probable that the heat from the synthesis process draws the Fe and Ti to the surface of the crystal and, when they come into contact with the flux solution, creates the blue outline.

By the mid-1970s, the manufacturers were evidently growing crystals large enough to eliminate all evidence of the seed crystal in faceting. We have not seen a blue-zoned flux-grown synthetic ruby such as the one pictured here for many years. However, we have noted in some of the rubies from Vietnam blue zones (rather than outlines) that remind us of some of the early faceted flux-grown synthetic rubies we examined so long ago. GRC

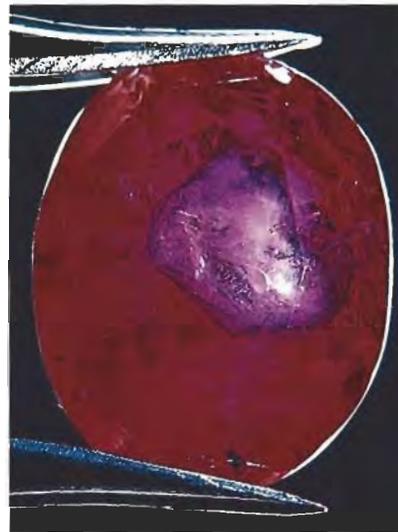


Figure 11. This early flux-grown synthetic ruby reveals a blue outline around the natural corundum seed. Magnified 10×.

SPINEL, with Unusual Green Fluorescence

The attractive 9.78-ct oval mixed cut shown in figure 12 in incandescent



Figure 12. This 9.78-ct spinel showed an attractive color change from bluish violet in fluorescent light to slightly pinkish purple in incandescent light.

light was recently submitted to our West Coast laboratory for identification. A medium dark, slightly pinkish purple in incandescent light, this stone exhibited a medium dark bluish violet when viewed in fluorescent illumination. Routine gemological testing established its identity as a natural spinel.

Specifically, although the single refractive index reading of 1.714 is slightly lower than is normally expected, it is the same as that recorded for a 16.39-ct color-change cobalt spinel described in the Fall 1990 Gem News section of *Gems & Gemology*. When we viewed the stone with a Chelsea filter, we observed a weak dull brownish pink throughout most of it, with flashes of dull brownish red in the crown facets. Moderate anomalous double refraction was seen in the polariscope. A specific gravity of 3.60 was determined by the hydrostatic method. Microscopic examination revealed an interesting display of inclusions: a complex interweaving of "needles" (figure 13) and stringers of fine particles intersecting in three directions, as well as a few small crystals and fractures (several with brownish orange iron-oxide stains).

When exposed to long-wave ultraviolet radiation, this color-change spinel exhibited a striking moderate to strong chalky, slightly yellowish

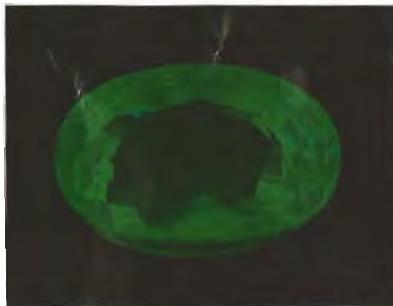


Figure 13. These needle-like inclusions were seen in the spinel shown in figure 12. Magnified 25×.

green fluorescence (figure 14). A similar, but much weaker, reaction was observed with short-wave U.V. radiation. Green fluorescence is quite unusual for a natural spinel. We recall having seen it only once before, in a natural spinel that exhibited a color change from pinkish purple to purplish pink (see the Winter 1984 Lab Notes section).

Figure 15 shows the absorption curve as recorded with a Pye Unicam Model 8800 UV-VIS spectrophotometer in GIA Research. The spectrum shows features related to

Figure 14. The spinel in figure 12 fluoresced a striking chalky, slightly yellowish green to long-wave U.V. radiation.



Fe^{2+} , with trace amounts of Co^{2+} , both in tetrahedral coordination. These two elements are responsible for the bluish violet coloration and the moderate color change. The very sharp peak at about 428 nm is attributed to Mn^{2+} in tetrahedral coordination (see Schmetzer et al., 1989, *Neues Jahrbuch für Mineralogie Abhandlungen*, Vol. 160, No. 2, pp. 159–180). Energy-dispersive X-ray fluorescence confirmed those assignments by showing the presence of Mn, Fe, Zn, and Ga in addition to the major constituents Mg and Al. Trace amounts of Co are below the detection limits of our EDXRF system. The green fluorescence is due to the tetrahedral Mn^{2+} , which is known to give rise to a green fluorescence in other materials, such as feldspars and some glasses (see Waychunas, 1988, *Reviews in Mineralogy*, Vol. 18: *Spectroscopic Methods in Mineralogy and Geology*, Mineralogical Society of America, Washington, DC, pp. 639–664). Also, flame-fusion synthetic spinel doped with manganese shows a very strong green fluorescence.

RK and Emmanuel Fritsch

Cobalt-Colored SYNTHETIC SPINEL, with Unusual Inclusions

An attractive, 2.51-ct oval blue stone was submitted to the East Coast laboratory by a dealer who thought it was a rare cobalt-colored blue spinel. However, its optical characteristics—R.I. of 1.728, red fluorescence to long-wave ultraviolet radiation, and chalky yellow fluorescence to short-wave U.V.—matched those of cobalt-bearing synthetic spinel. The fact that natural cobalt-colored blue spinels are usually inert to both long- and short-wave U.V. (see the Kane entry in the Summer 1986 Lab Notes section) strongly indicated that our stone was indeed a synthetic.

When we examined the stone with magnification, we were surprised to see misty stringers and veils unlike anything we had ever seen in synthetic spinel (figure 16). To find a single known synthetic spinel with the same wispy inclusions—and thus confirm that this was indeed a synthetic—we had to examine well over 100 similar-colored synthetic spinels with binocular magnification. The known synthetic spinel we found

Figure 15. The U.V.- visible absorption spectrum of the spinel in figure 12 reveals features related to Fe^{2+} and Co^{2+} that are responsible for the bluish violet coloration and moderate color change.

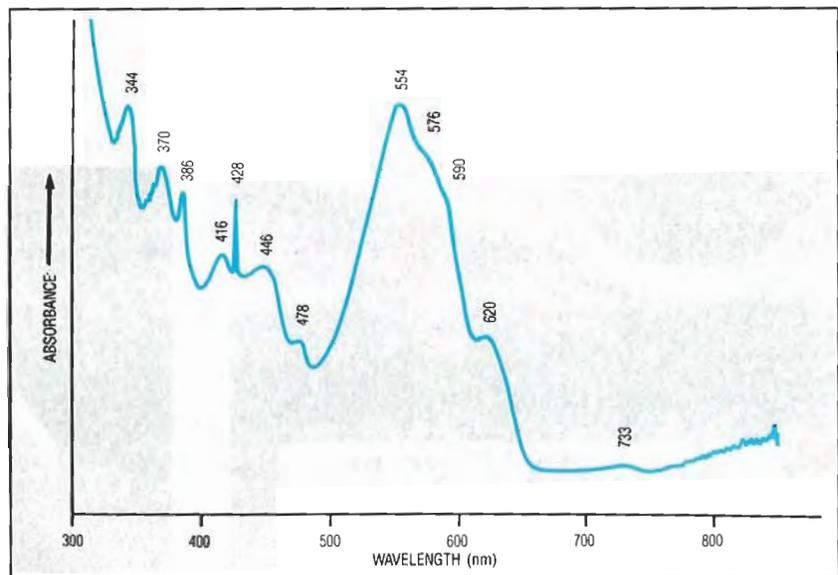




Figure 16. The unusual wisps and veils in this cobalt-colored synthetic spinel appeared to be associated with strain patterns. Magnified 50 \times .

looked like our sample. When both synthetic spinels were examined with crossed polarizers, the wisps and veils appeared to be associated with strain patterns. GRC

Dark Blue TOPAZ Damaged by Treatment

Blue topaz is one of the most common gems in the trade today. It is widely known that most of the various shades of blue marketed do not occur naturally, but are usually produced by treatment with irradiation followed by heat. Presently, there is no routine gemological test to separate natural- from treated-color blue topaz.

Some stones, however, provide indicators of treatment. The pres-

ence of residual radiation, noted in rare instances, conclusively proves that the topaz has been treated. The Geiger counter we use in the East Coast lab registered only background levels when the 20.88-ct topaz illustrated in figure 17 was tested. Proof of electron treatment was provided by the internal damage, referred to as "internal lightning," that was evident. As reported by Nassau in the Spring 1985 issue of *Gems & Gemology* (p. 30), the halos around the individual crystals most likely are also the result of the electrical charge.

Even though topaz samples are cooled during treatment, the buildup of a negative charge from the accelerator can damage the stones. To avoid damage, higher energy



Figure 17. "Internal lightning" reveals damage in a topaz treated by electron irradiation. Magnified 20 \times .

levels are usually used so that most of the electron beam passes through the sample. TM

Radioactive ZIRCON

We recently received for testing an 11.56-ct stone that the dealer believed was zircon and suspected was radioactive.

Routine gemological testing proved that the stone was zircon, and the Geiger counter showed that it was in fact radioactive, emitting over 0.4 milliroentgens (mR) per hour (figure 18). As normal background level is about 0.02 mR/hour, it would probably be unsafe to wear this stone in jewelry.

Ordinary "high zircon" is zirconium silicate crystallized in the tetragonal system. If the crystal contains radioactive uranium or thorium, alpha particles will destroy the crystal structure over time, forming what is called "low zircon," or amorphous silica and zirconium. As expected for a "low zircon," no dichroism or doubling of facet junctions was observed in the stone we tested. Nor could we locate a uniaxial figure in the polariscope, since the crystal structure had deteriorated. Instead, we saw the bright circular bands of color (figure 19) typical of a metamict zircon. DH

Figure 18. This green zircon sitting on top of a Geiger counter probe shows a radiation reading of over 0.4 mR/hour.

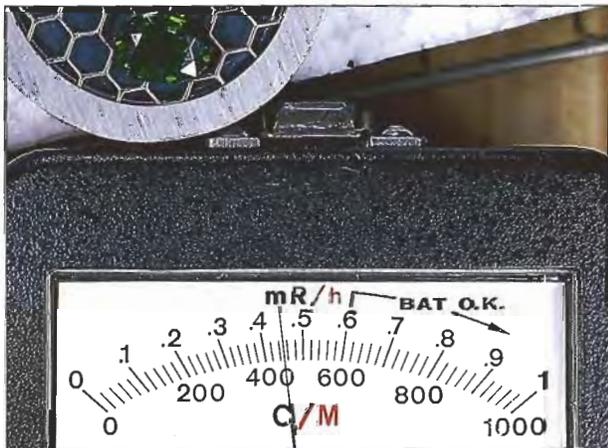
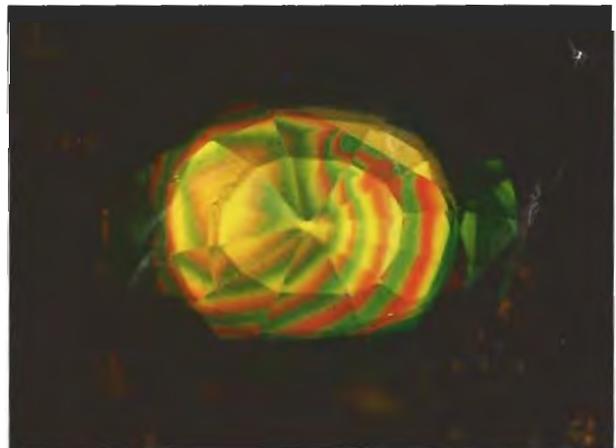


Figure 19. Only interference colors, but no optic figure, could be resolved in the metamict zircon shown in figure 18.



A HISTORICAL NOTE

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

SUMMER 1966

The New York lab described and illustrated a remarkably thin—0.6 mm—rose-cut diamond that was 14.5 mm in diameter. This thin crown was used in conjunction with a cone-shaped metallic backing, also shown, to simulate a much larger diamond when set. Treated red-brown and red-orange diamonds were discussed and their absorption spectra illustrated.

SUMMER 1976

Chrysoberyl is well known for cat's-eye material, but it rarely shows asterism. A chrysoberyl showing a very distinct four-rayed star was seen in the Santa Monica lab. They also encountered another imitation amber with insect inclusions, in this case a large cicada in what turned out to be plastic.

The New York lab discussed diamonds and inclusions in diamonds. A cutter decided to eliminate a cloudy

area in the center of a 50-ct piece of rough by cutting an 8.55-ct cube containing the cloud. The cloud shows a dark cross at its center. Other unusual cuts and shapes of diamond were shown.

SUMMER 1986

The Los Angeles lab reported on some unusual items, including a nearly black devitrified glass that was being sold in Hong Kong as black "onyx." An unusual way to use thin slices of opal was illustrated in an assembled bead necklace where the opal had been applied in sections to a round bead backing material and then shaped and polished. A most unusual cat's-eye rutile was seen in Santa Monica. Although rutile does not normally have a structure that would produce chatoyancy, this material was apparently fibrous enough to produce a cat's-eye effect when fashioned.



This unusual cat's-eye rutile weighs 1.43 ct. Magnified 3×.

FIGURE CREDITS

The photos for figures 1–3, 5, 6, 8, 9, 11, and 16–18 were supplied by Nicholas DelRe. Figures 4 and 19 were taken by Dave Hargett. John I. Koivula shot the photomicrograph in figure 7 and the Historical Note. Bob Crowningshield took the X-radiograph in figure 10. Figure 12 is by Robert Weldon, figure 13 is by Robert E. Kane, and figure 14 is by Shane F. McClure. Mike Moon ran the UV-VIS spectrum illustrated in figure 15.

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