

DYED PURPLE HYDROPHANE OPAL

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Opals with an unusual purple bodycolor and strong play-of-color have recently appeared in the market. Reportedly from a new deposit in Mexico, they have a vivid bodycolor unlike that of any natural play-of-color opal seen in the trade so far. This alone was enough to raise suspicion, and gemological and spectroscopic evidence indicates that the purple coloration is artificial. A comparison of this purple opal with numerous samples from Ethiopia's Wollo Province strongly suggests that it is actually dyed hydrophane opal from those deposits. Several previously undocumented characteristics of Wollo opals are described, including zeolite mineral inclusions.

Not often is a significant new deposit discovered of a well-known gem material. The 1994 discovery of play-of-color opal in Ethiopia's Shewa Province sparked the industry's attention. Unfortunately, much of this opal turned out to be unstable, and spontaneous fracturing rendered it largely unusable for jewelry (Johnson et al., 1996). In 2008, another large Ethiopian deposit was found in the province of Wollo. While similar in appearance to some of the Shewa material, this opal appears to be much more stable than its predecessor (Rondeau et al., 2010). The 2011 Tucson show saw an abundant influx of beautiful and relatively inexpensive opal from the Wollo deposit.

The new Ethiopian material displayed a property not often seen in opal. Much of the opal is hydrophane, meaning it is very porous and easily absorbs water (or other liquids), often turning translucent or transparent in the process. The degree to which these stones show this property varies, but some absorb water so readily that the tiny bubbles escaping from their surface give the impression of effervescence. This property, while interesting to watch, also

has an important implication: Any gem material that absorbs liquids so easily has the potential to be treated by methods such as dyeing or impregnation. Recognizing this possibility, we performed several experiments to determine the effect of such treatments on this opal, before they appeared in the market. These experiments—which were surprisingly successful—led us to believe that it would only be a matter of time until we encountered such treated material in the trade.

Indeed, in October 2011, we were presented with several samples of hydrophane opal that had a bright purple bodycolor (not known to occur naturally in play-of-color opal; e.g., figure 1), and our suspicions were immediately raised (Renfro and McClure, 2011). In addition, these opals were said to be from a new source in Mexico, but everything about them except the color reminded us of Ethiopian opal from Wollo.

Purple opal has been reported from several localities, including Mexico. However, all of the material examined to date was opaque (or at best translucent) and did not possess play-of-color. The purple in these common opals has been attributed to inclusions of fluorite (Fritsch et al., 2002).

While the color of the new play-of-color samples was said to be natural, the authenticity of any gem material can only be proven through scientific analysis and observation. Our goal in this study is to

See end of article for About the Authors and Acknowledgments.

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answer two fundamental questions: Is this purple opal naturally colored, and is it actually from a new deposit in Mexico? This article also reports the results of an experiment on the dyeing of hydrophane opal from Wollo Province.

MATERIALS AND METHODS

Nine purple opal cabochons ranging from 4.13 to 15.25 ct, and nine rough opal samples between 0.66 and 1.96 g, were submitted for testing at GIA's Carlsbad laboratory. Seven of the rough opals had a purple bodycolor of varying intensity, but one had a distinct green-blue bodycolor (0.76 g) and another was light blue (0.93 g). Client permission was obtained for potentially destructive testing on these stones, as hydrophane opal will occasionally crack when soaked in liquid (author NR's personal experience). To expand our sample base, we subsequently borrowed 22 rough opals (0.57–3.96 g) from the same source that submitted the purple cabochons. These ranged from light to very dark purple, except for three samples showing no visible purple color. Two of the 22 samples also showed an amber color zone. We also examined approximately 2 kg of rough natural hydrophane opal from Wollo (loaned from two reputable sources who obtained them in Ethiopia) for comparison with the purple opal.

We performed standard gemological characterization of all cut samples (both dyed and natural) with a Duplex II refractometer, a desk-model spectroscope, a long- and short-wave 4 watt UV lamp, and a gemological microscope. Inclusions were identified with a Renishaw InVia Raman microscope using a 514 nm argon-ion laser at a resolution of 1 cm^{-1} .

Visible spectroscopy measurements were made by soaking one purple opal cabochon and the green-blue piece of rough opal in acetone (with the client's permission) for 54 hours, and then placing the acetone in standard 1 cm glass cuvettes for analysis with a Perkin Elmer Lambda 950 spectrometer. We used a data interval and slit width of 1 nm, and baseline correction was accomplished using a 1 cm glass cuvette filled with pure acetone. Spectroscopy was not performed directly on the opals themselves, in order to eliminate interference from light scatter and the intrinsic opal spectrum that would prevent us from making baseline-corrected measurements of any dye present in the samples. Such measurements were necessary for color analysis, as described below.

From the absorption spectrum of the acetone solutions, we calculated color space coordinates to



Figure 1. The purple bodycolor of this new opal, reportedly from Mexico, raised concerns about the origin of the material. Shown here are three rough samples ranging from 1.28 to 3.09 g and five cabochons weighing 5.31–9.32 ct. Photo by Robert Weldon.

visually verify that the spectra collected were indeed responsible for the samples' coloration. Because of the very low absorption values of the solutions, they needed to be concentrated to visually resolve color. Since we could not physically concentrate our solutions and still have enough volume to fill the cuvettes, this was done artificially by multiplying the absorption values by a factor of 15. This method is equivalent to a solute (dye) concentration 15 times greater than that in the original solutions due to the linearity of absorption. The absorption values of the artificially "concentrated" acetone solutions were converted to transmission spectra using GRAMS spectroscopy software by Thermo; CIE $L^*a^*b^*$ color space coordinates were calculated using the GRAMS color analysis application. These coordinates were imported into Adobe Photoshop to produce color samples.

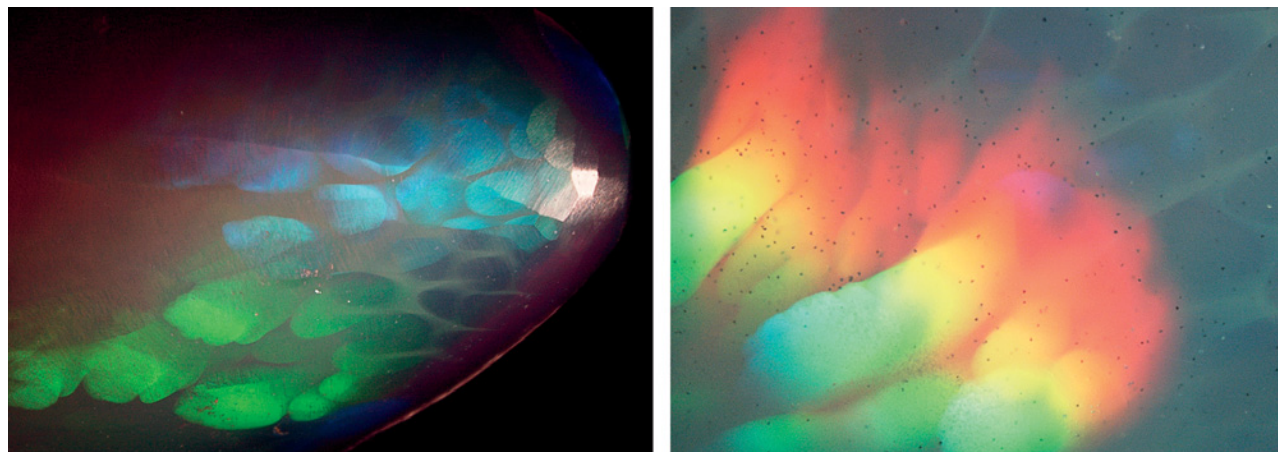


Figure 2. The purple opal (left, image width 7.8 mm) displayed a “digit” pattern of play-of-color and a cellular pattern of potch that had a greenish cast, much like natural Wollo opal (right, image width 4.3 mm). Photomicrographs by S. F. McClure.

Chemical analysis of 15 samples (seven untreated white Wollo opals and eight purple samples) was performed using a Thermo X Series II ICP-MS with a New Wave Research UP-213 laser ablation sampling system and a frequency-quintupled Nd:YAG laser (213 nm wavelength) with a 4 ns pulse width. We used 55- μm -diameter ablation spots, a fluence of around 10 J/cm², and a 7 Hz repetition rate. Qualitative chemical analysis of two rough samples (one treated and one untreated) showing black surface material was also performed with a Thermo ARL Quant-X EDXRF system in a vacuum, utilizing no filter at 4 kV and 1.98 mA, and a cellulose filter at 8 kV and 1.98 mA.

As noted above, given the hydrophane character of Wollo opal, it should be amenable to dyeing. To test this, eight rough (0.17–1.42 g) and three cabochons (1.65–3.55 ct) of hydrophane opal from the personal collection of author NR were immersed in variously colored solutions prepared from Sharpie permanent markers and acetone. The samples were soaked for anywhere from several minutes to several hours, depending on how rapidly the solution was

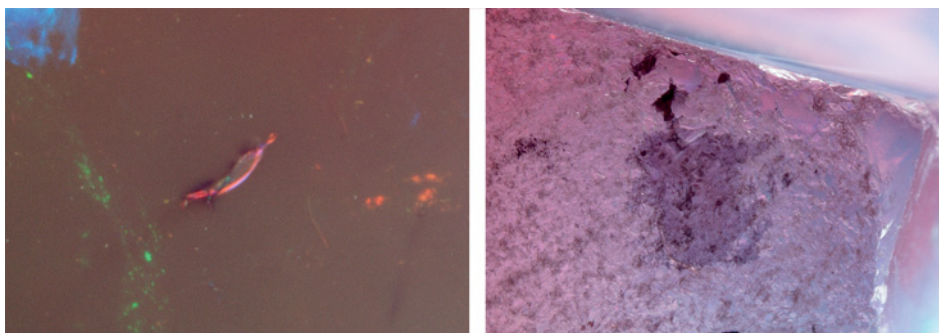
absorbed. After removal from the solution, the samples were dried under a tensor lamp for several hours until the acetone had completely evaporated.

RESULTS AND DISCUSSION

Gemological Properties. All the opals provided by our client were clearly hydrophane, as they tended to feel sticky when handled, a result of the opal trying to draw moisture from the skin. The spot RI measurements of the nine purple opal cabochons ranged from 1.37 to 1.41. The SG was between 1.70 and 1.77, as measured hydrostatically before allowing the stones to completely soak full of water. All samples showed a very weak blue reaction to long- and short-wave UV radiation. A broadband absorption was seen in the desk-model spectroscope from ~550 to 600 nm.

Magnification revealed octahedral to irregularly shaped dark crystals of pyrite, tube-like inclusions that resembled fossilized plant matter, and cellular play-of-color referred to as a “digit pattern” because of its resemblance to fingers (figure 2; Rondeau et al., 2010). The pattern consists of relatively large rounded cells separated by a thin network of potch (common

Figure 3. In many of the purple opals, color concentrations were evident in surface pits (left, image width 2.2 mm), and on unpolished areas (right, image width 2.4 mm). Photomicrographs by S. F. McClure.



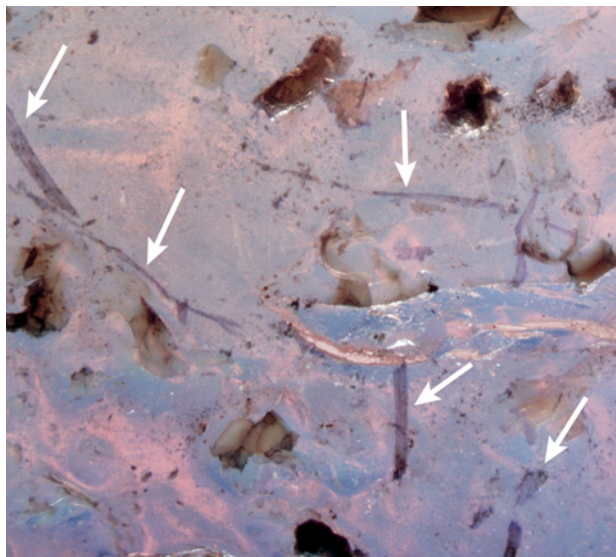
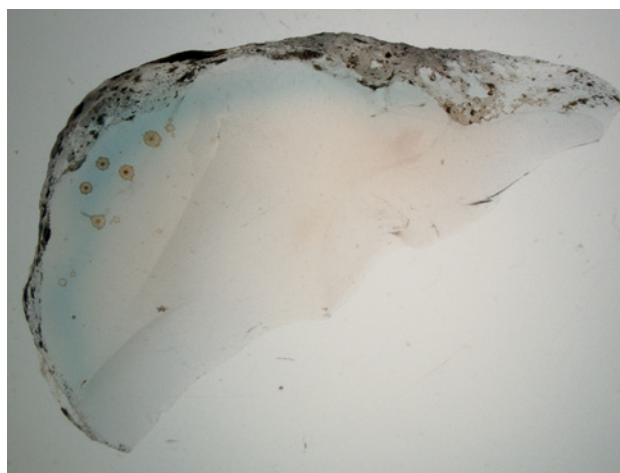


Figure 4. Surface-reaching inclusions in the purple opals sometimes had a purple color. Those shown here probably consist of fossilized plant matter. Photomicrograph by S. F. McClure; image width 3.1 mm.

opal without play-of-color) that has a slightly greenish appearance. Also seen in some samples were subtle purple color concentrations around pits, scratches, surface-reaching fractures, and sometimes on unpolished surfaces (figure 3). Some surface-reaching inclusions were also purple (figure 4). However, not all of the samples showed color concentrations.

Gem materials are often immersed in a liquid of similar refractive index to see subtle internal features such as color zoning. This is particularly help-

Figure 5. Immersion of this light blue rough opal in water showed a surface-conformal color layer, which is indicative of treatment. Photomicrograph by N. Renfro; image width 9.5 mm.



ful with rough material. With opal, water serves as an adequate immersion liquid. One of the rough stones in the initial group had a light blue bodycolor and did not appear to be treated. We were quite surprised when immersion revealed a blue zone along the surface of the stone (figure 5). This type of surface-conformal coloration is indicative of color treatment in many gem materials, including beryllium- and titanium-diffused corundum, smoke-treated opal, and dyed agate.

Spectroscopy of the Dye. No color was observed in the acetone after soaking the purple sample for up

NEED TO KNOW

- In late 2011, purple opal showing strong play-of-color appeared in the market, reportedly from a new deposit in Mexico.
- The presence of a dye was indicated by soaking the opal in acetone for an extended period (54 hours), followed by spectroscopic processing of the solution that yielded a purple color consistent with the opal's bodycolor.
- Physical, chemical, and microscopic properties of the purple opal overlap those of hydrophane opal from Ethiopia's Wollo Province, except for the purple color.
- Dye experiments on Wollo hydrophane opal produced vivid bodycolors.
- Multiple lines of evidence indicate that the purple opal is actually dyed Wollo hydrophane.

to 16 hours. After 54 hours, the acetone solution appeared very light purple. Spectroscopy of this sample revealed a broad asymmetrical feature with an apparent maximum located at about 594 nm and a shoulder close to 557 nm (figure 6, top). This feature was consistent with the broadband absorption observed in the desk-model spectroscope.

The CIE L*a*b* coordinates calculated using GRAMS and Adobe Photoshop software yielded a purple color (figure 6, center), consistent with the bodycolor of the opal. This proved that the coloring agent of the purple opal can be partially removed with acetone, and since we know of no naturally occurring coloring agent showing this behavior, we concluded that the opals are colored by an artificially introduced dye.

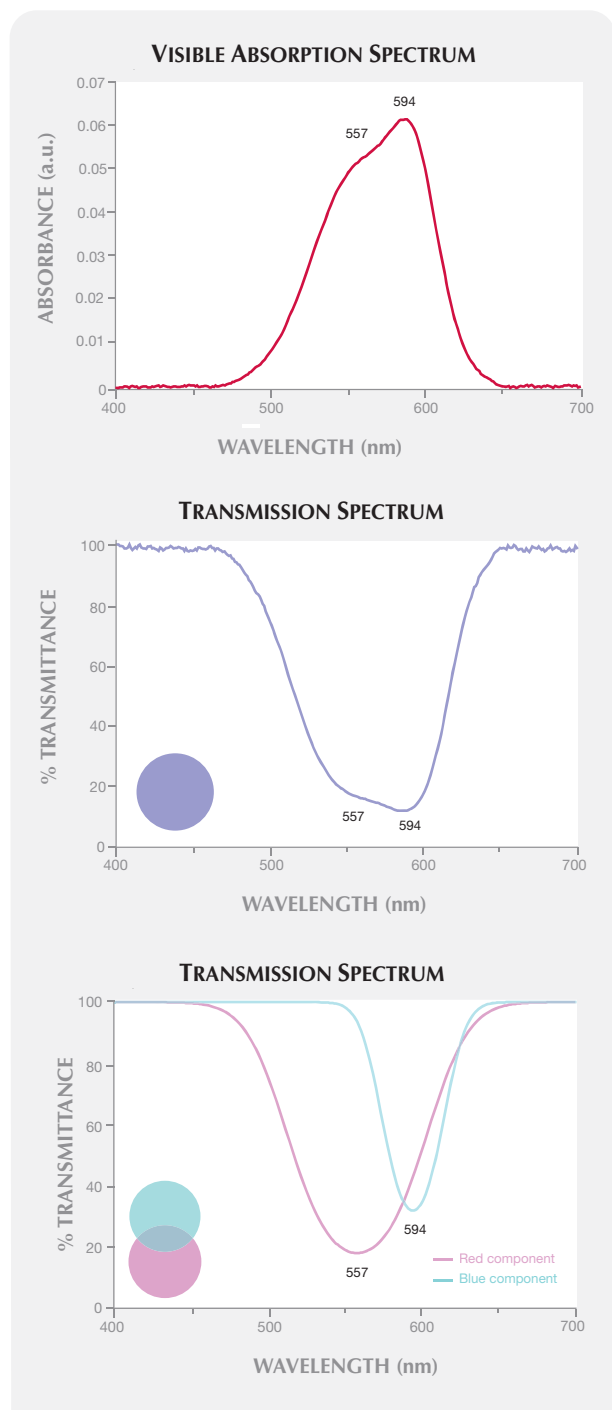


Figure 6. After a purple opal was soaked in acetone, the solution's visible-range spectrum revealed a broad asymmetrical absorption feature (top). The absorption from the artificially "concentrated" dye (center, transmission spectrum) generated a CIE $L^*a^*b^*$ color space coordinate consistent with the opal's purple bodycolor. Two color components of the purple dye were resolved from the transmission spectrum (bottom). The 557 nm feature produced the purplish pink component, and the 594 nm feature contributed the blue one.

To better understand the absorption spectrum of the purple dye, we used the peak fitting application of GRAMS to resolve the individual features from the asymmetric absorption (figure 6, bottom). The full width at half maximum (FWHM) of the 557 nm feature was 72 nm, and the FWHM of the 594 nm feature was 36 nm. Processing of these component spectra showed that the 557 nm feature was responsible for a purplish pink component, while the 594 nm band contributed a blue component. The combination of these purplish pink and blue features is responsible for the purple color.

This dye extraction procedure was also applied to the green-blue piece of rough (figure 7, inset). After the opal had soaked in acetone for several hours, we measured the solution's visible absorption. A broad band was recorded at 627 nm (figure 7); the calculated color was again consistent with the opal's bodycolor.

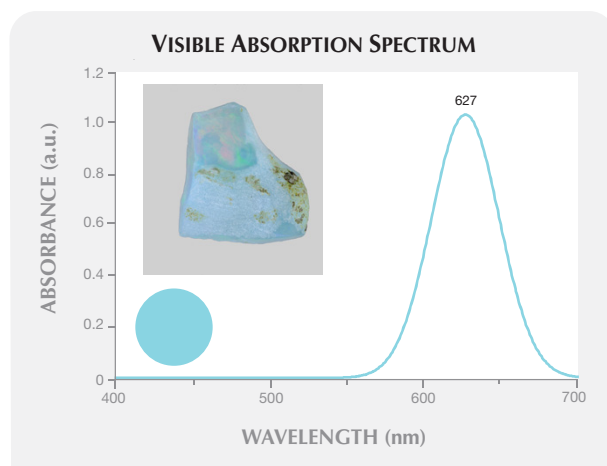


Figure 7. A green-blue opal was also soaked in acetone, and spectroscopy of the solution revealed a broad absorption feature centered at 627 nm. The corresponding color space coordinates yielded a color sample consistent with the bodycolor of the sample. Inset photo by Robison McMurtry.

Comparison of Dyed Purple Opal to Wollo Hydrophane. We compared the physical appearance, gemological properties, and other analytical results for the dyed material with opals from Wollo Province to assess the original source of the treated opal and help investigate claims of Mexican origin.

Gemological Properties. The RI and SG values of the dyed opal were virtually identical to those of Wollo opal. This is notable because both properties are par-

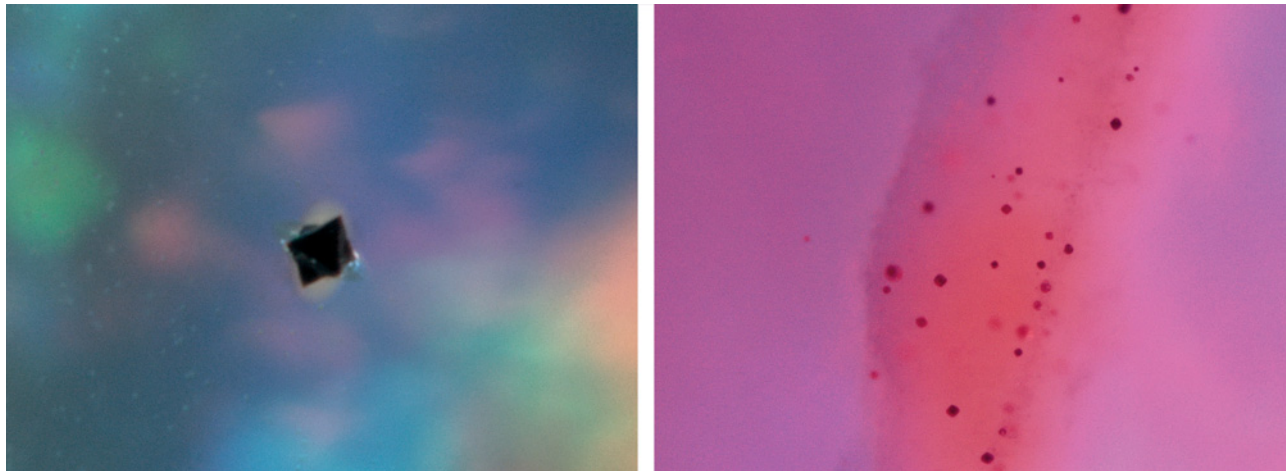


Figure 8. Both the Wollo (left) and dyed purple (right) opals showed irregular to octahedral black-appearing crystals of pyrite. Photomicrographs by N. Renfro (left, image width 0.8 mm) and S. F. McClure (right, image width 1.7 mm).

ticularly low for play-of-color opal (Webster, 1996). Ultraviolet fluorescence was also very similar between the two (see also Rondeau et al., 2010).

Most opal is porous to a minor degree, but it is quite unusual for it to be so porous as to qualify as hydrophane. Both Wollo opal and the purple opal display this property—sometimes it is so prominent that the transparency can be seen to improve as it soaks up water. Mexican hydrophane opal is known, but to our knowledge it is opaque and light pink or brown (with or without play-of-color).

The structure of the play-of-color is also noteworthy. Wollo opal sometimes displays an unusual “digit pattern” to its play-of-color that many consider unique to Ethiopian material (Rondeau et al., 2010). We observed this same pattern in some of the

purple opal. A similar pattern has been reported in some opal from Virgin Valley, Nevada, but it was smaller and had a slightly different appearance (Gübelin and Koivula, 2005).

Inclusions. Microscopic characteristics are essential to any comparison of gem materials. All of the features described in this section were seen in both the untreated Wollo samples and the purple opals. Among those reported previously in Ethiopian opal are small black octahedral crystals that have been suggested to be pyrite (Johnson et al., 1996; Rondeau et al., 2010; figure 8) and irregular tubular inclusions with a cellular structure that are probably fossilized plant matter (Rondeau et al., 2011; figure 9). Though neither can be considered unique to Ethiopia, pyrite

Figure 9. Irregular tube-like inclusions that appear to be fossilized plant matter occurred in both the Wollo opal (left, image width 4.4 mm) and the dyed opal (right, image width 5.0 mm) samples. Photomicrographs by S. F. McClure.



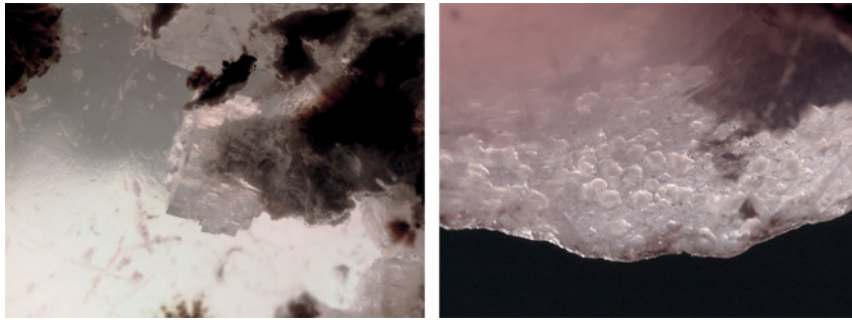


Figure 10. Pseudo-cubic crystals of a zeolite mineral (possibly chabazite) were found in both Wollo opal (left, image width 2.0 mm) and the dyed purple opal (right, image width 4.3 mm). We believe this is the first report of this mineral as an inclusion in opal. Photomicrographs by S. F. McClure.

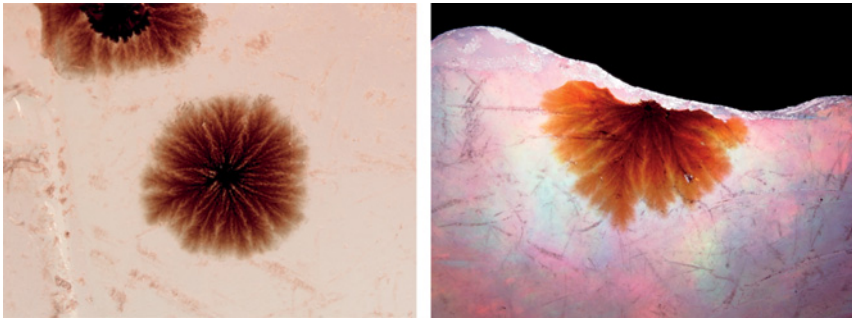


Figure 11. Surface to subsurface circular radiating brown inclusions were occasionally visible in both Wollo opal (left, image width 2.0 mm) and the dyed purple material (right, image width 6.5 mm). Photomicrographs by S. F. McClure.

octahedra are certainly rare in gem opal. Plant matter is found included in opal from a number of other deposits.

We discovered another type of inclusion that to the best of our knowledge has not been reported in opal: a zeolite mineral, possibly chabazite. It formed numerous small, transparent, colorless, euhedral pseudo-cubic crystals (figure 10). They were present only in rough material, at or near the surface, but always included within the opal, whether the natural Wollo or dyed purple material. The crystals appear to have been growing on the matrix before it was engulfed by the opal.

Also found in the rough opal samples, either at or just below the surface, was a flat round brown material with a radial structure (figure 11). These were typically seen along fracture surfaces. We were unable to match their Raman spectra to anything in our database.

Surface Characteristics. Because we examined numerous rough samples of both natural Wollo and dyed purple opal, we noted some surface features that would not be visible on cut stones.

Layers of a dark brown to black opaque material were present on many of the natural Wollo samples. The same material was found on the surface of the rough purple opal we examined—the only difference being that it was distinctly purplish (figure 12). We were unable to obtain a conclusive Raman spectrum from this material, but the spectra did indicate the presence of amorphous carbon. EDXRF analysis detected considerable Mn. Previous studies of Ethiopian opal have identified such material as a manganese oxide (Johnson et al., 1996; Rondeau et al., 2010).

The rough Ethiopian opal we examined sometimes had a brownish pink to pink opaque material on the surface that was very friable and had a matte

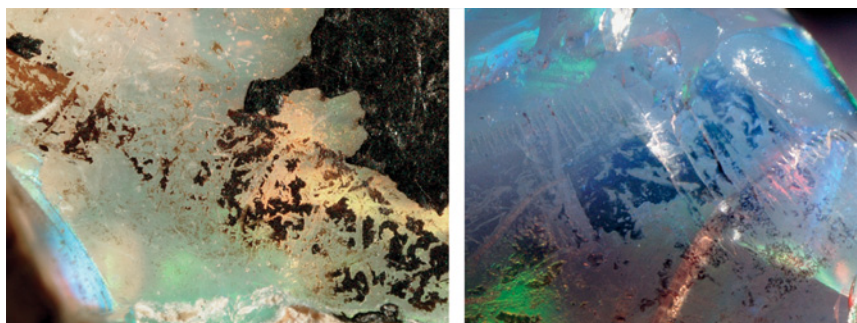


Figure 12. Black to brownish black surface coatings were present on rough pieces of both the Wollo (left) and purple (right) opals, the only difference being the purplish cast of the coating on the dyed material. Photomicrographs by S. F. McClure; image width 6.7 mm.

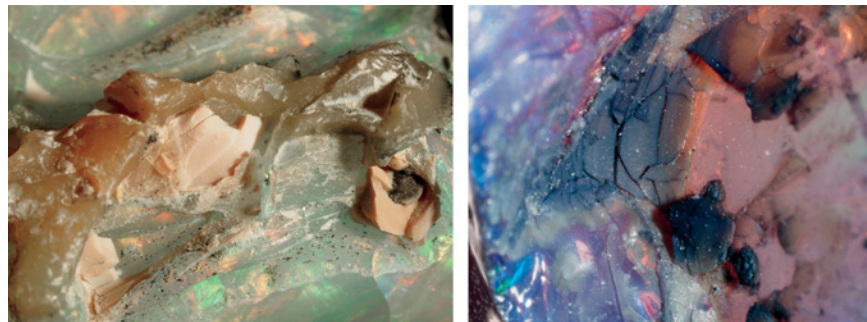


Figure 13. Some Wollo opals displayed opaque material on the surface that may be a type of common opal (left, image width 8.0 mm). Some of the purple samples showed similar material, except that it was purplish gray (right, image width 5.9 mm). Photomicrographs by S. F. McClure.



Figure 14. Well-formed quartz crystals with very short prism faces were visible in the matrix of both Wollo opal (left, image width 2.3 mm) and the dyed purple material (right, image width 1.4 mm). Photomicrographs by S. F. McClure.

or dull fracture luster (figure 13, left). Raman spectroscopy gave a poor unidentifiable signal, and LA-ICP-MS analysis showed it was composed primarily of Si. We suspect it is some kind of highly disordered common opal. This same material was found on the surface of some of the rough purple opal, the only difference being the color: It was mostly dark gray and uneven, with much darker fractures and an often purplish cast (figure 13, right).

Matrix. Many of the rough samples, both natural Wollo and dyed purple, still had matrix attached to them. The matrix was typically beige, though several pieces of the dyed opal had matrix that was very dark purplish gray. In all cases, it was a soft clay-like material that contained numerous mineral grains. These matrix-hosted mineral grains were found in both the Wollo and the purple opal, and consisted of:

(1) transparent light brown hexagonal crystals of quartz with very short prism faces (figure 14), some of which showed dissolution features and contained rounded light green and black inclusions (figure 15); (2) prismatic colorless to light yellow crystals of K-feldspar; and (3) dark green prismatic fractured crystals of aegirine, a pyroxene (figure 16). The quartz and feldspar were common, but we observed only one example of the aegirine in a Wollo opal and one in a purple sample.

One question that arises is how the purple opal can be dyed if the matrix of some pieces is still the natural beige color. Digging into an area of dark purplish gray matrix with a needle probe showed the normal beige color just below the surface (figure 17). This indicates that the dye did not penetrate the matrix very deeply. Therefore, one possible explanation for the natural-colored matrix on the purple

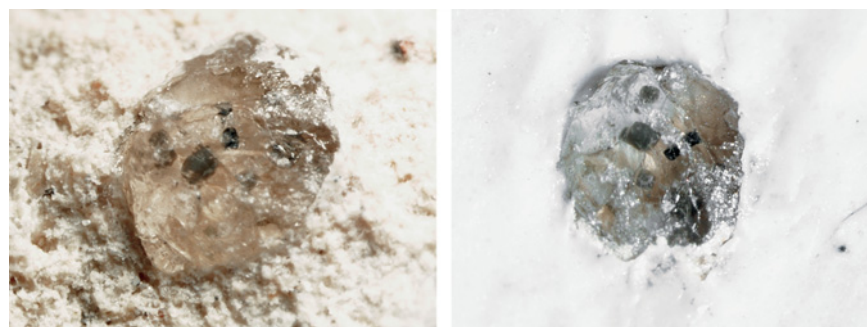


Figure 15. Some of the etched quartz attached to the matrix of both kinds of opals contained identical inclusions of a translucent green material and a black mineral (too small to identify with Raman analysis). Photomicrographs by S. F. McClure; image width 1.4 mm (left, Wollo opal; right, dyed purple opal).

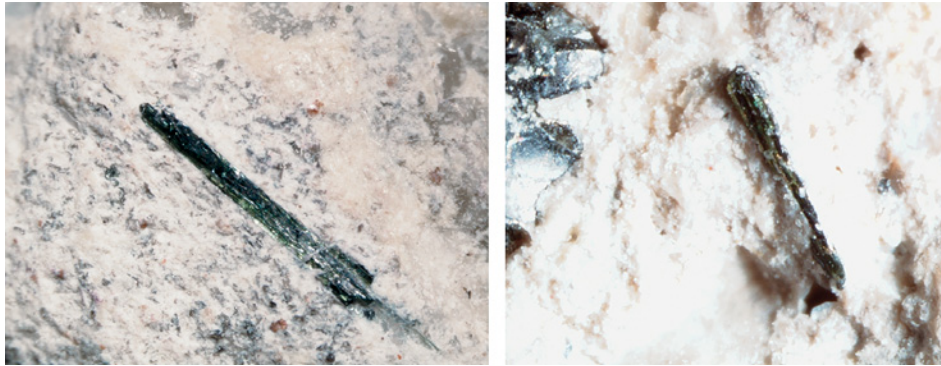
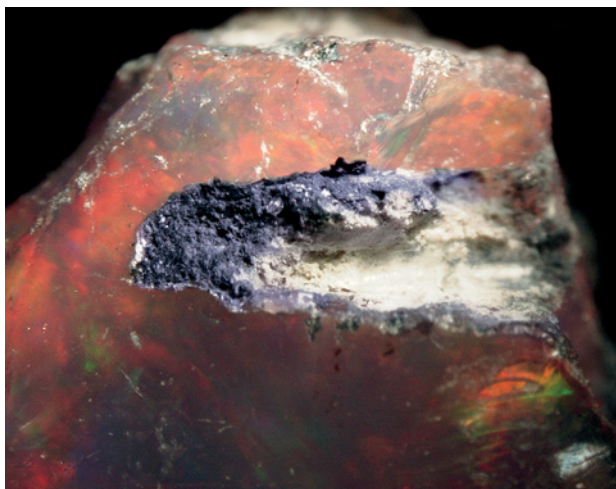


Figure 16. Aegirine crystals were found in the matrix of one Wollo (left, image width 2.2 mm) and one dyed purple opal (right, image width 1.9 mm). Photomicrographs by S. F. McClure.

opal is that the top layer of matrix was removed after the stones were dyed. The softness of the matrix would make this easy to do.

Chemical Composition. We suspected that chemical analysis would provide important clues to the origin of the purple opal. Gaillou et al. (2008) found that opal from the initial Ethiopian deposits at Shewa was easily distinguished by its high Ca content (>1000 ppmw), combined with the presence of Nb. That study found high Ca in orange to brown opals only—white Ethiopian opals were not discussed. Two years later, in a report on Wollo opal, Rondeau et al. (2010) noted a high Ba content (>100 ppmw) in white opals. This was based on a very small sample base, and the article acknowledged that further analyses were necessary.

Figure 17. In some instances, areas of matrix still attached to the rough purple opal had a dark purplish gray color. When scratched with a probe, the matrix under the surface displayed the normal beige color. Photomicrograph by S. F. McClure; image width 5.9 mm.



The small number of samples (15 total) analyzed for this study makes it difficult to detect meaningful chemical trends. Overall, the Ca content of all samples was much higher than the threshold reported by Gaillou et al. (2008): 8,000–10,000 ppmw or higher. Additionally, all the samples contained trace amounts of Nb. The Ba content of the purple opals tended to be lower (80–155 ppmw) but reached 475 ppmw. The Ba content of the untreated Wollo opal was higher overall (175–285 ppmw), but ranged from 65 to 1400 ppmw. There are several possible explanations for this, one of which is that we do not know the starting color of the dyed opal. It could have been yellowish or orangy, in which case it would be expected to have a lower Ba content, as reported by Rondeau et al. (2010). None of the other elements showed any meaningful trends. Clearly, further investigation is necessary to assess the compositional range of these opals.

Dyeing Ethiopian Hydrophane. Vivid bodycolors were produced in our dye experiments (figure 18), and they show how easy it is to artificially color Ethiopian hydrophane opal. We also noted dye concentrations around fine scratches and pits in the surface, implying that the dye transport mechanism is a function of surface area. As scratches represent localized zones of high surface area, it is reasonable to conclude that the transport of a mobile fluid in the opal is driven by capillarity.

To support this hypothesis, we scratched “GIA” on one white Ethiopian opal cabochon and immersed it in our blue dye solution for several seconds. The dye penetrated the stone much deeper in the area immediately surrounding the scratched letters (figure 19, left and center). Very fine scratches on the cabochon were also visibly colored by the blue dye. We then tested the stability of the dyed color by soaking the stone in pure acetone. After several hours, the acetone turned slightly blue and the stone became more uniformly colored and lighter (figure 19, right).

A second dyed blue opal was soaked in water for more than a week with no observable change in the color of the opal or the water. The stability of the dye is therefore largely dependent on the type of solvent to which it is exposed.

Interestingly, the client discovered that soaking the purple opals in hydrogen peroxide would decolorize them. With the client's permission, we demonstrated this on a 0.57 g piece of rough purple opal, soaking it in a 3% solution of hydrogen peroxide for 72 hours. A significant amount of color was lost from the stone, which had a light purplish gray appearance after soaking (figure 20). An absorption spectrum collected on the hydrogen peroxide after soaking the purple opal was featureless. This suggests that the dye was not necessarily removed from the opal or dissolved into the hydrogen peroxide solution; instead, the dye molecules were chemically altered into a compound that does not absorb visible light, or bleached. This technique may be effective in decolorizing some dyed opal, but it would be highly dependent on the type of dye used.

Identification and Origin of Dyed Purple Opal.

Purple color has never been reported for natural play-of-color opals. Microscopic examination strongly suggested the presence of a dyeing agent in the purple sample we examined: color concentrations in fractures and surface pits, patches of purple color on the surface, surface-reaching inclusions that were purple, and certain characteristics on the surface of the rough such as opaque material with dark purplish gray fractures and layers of a dark brown to black material with a purple cast. Proof of dye treatment was that the color could be partially

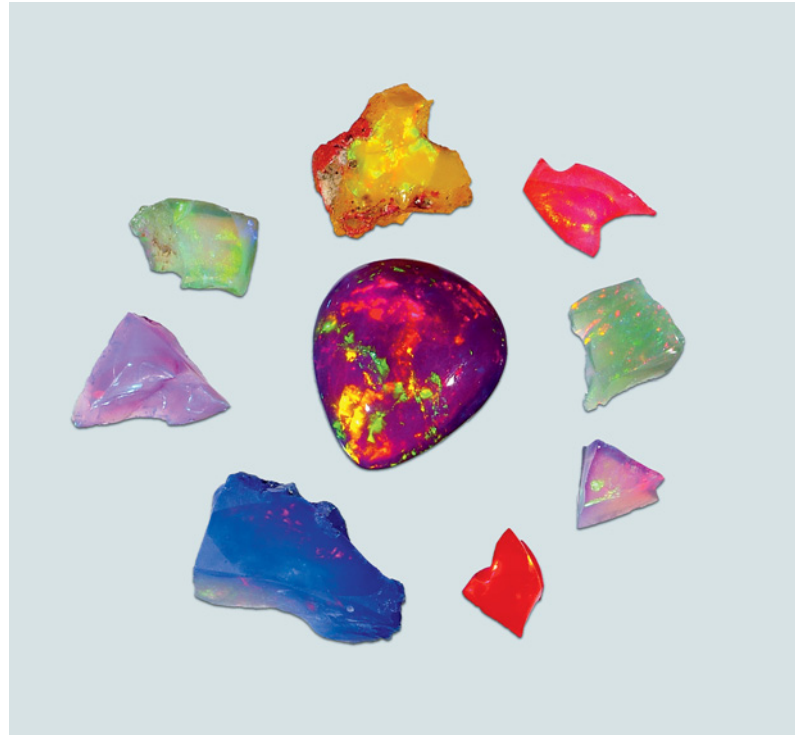
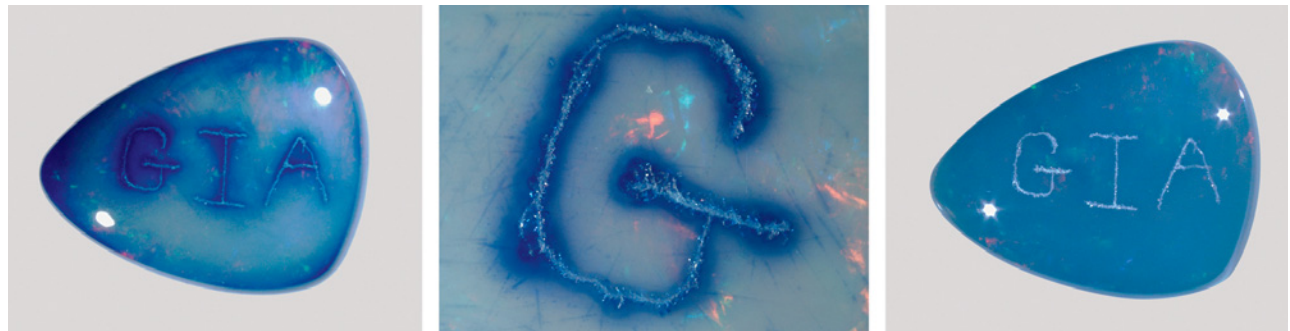


Figure 18. A variety of vivid colors were easily produced by dyeing white Wollo opals with solutions prepared from permanent marker ink and acetone. The cabochon in the center was submitted by a client and weighs 8.77 ct. The rough samples weigh 0.17–1.42 ct; photo by C. D. Mengason.

removed with acetone.

The purported Mexican origin of the purple opal can be dismissed. Instead, the purple material presents all the characteristics of opals from the Wollo deposit in Ethiopia. Both opals are hydrophane, sometimes showing a cellular play-of-color separated by greenish internal patch. Some contain tiny octa-

Figure 19. This 1.75 ct Wollo opal with “GIA” scratched onto the surface shows how dye transport is faster in localized regions with a high surface area, such as pits and scratches (left and center). After the opal was soaked in pure acetone for several hours, the blue dye was homogeneously distributed, eliminating visible dye concentrations (right). Photos by N. Renfro; center image width is 3.8 mm.



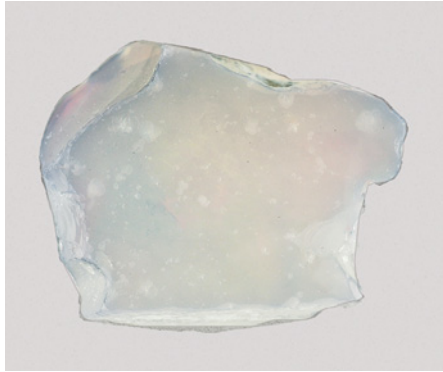


Figure 20. This 0.57 g rough piece of opal is shown before (left) and after (right) its purple color was removed by soaking in hydrogen peroxide for 72 hours. Photos by Robison McMurtry.

hedral inclusions of pyrite and rod- or tube-like inclusions that are probably fossilized plant matter. They have similar RI, SG, and UV fluorescence characteristics, as well as surface layers of manganese oxide. In addition to these properties, we observed some unusual inclusions in both Wollo and dyed purple samples that have not been reported previously, such as transparent pseudo-cubic crystals of a zeolite mineral (probably chabazite) and flat radial brown inclusions.

CONCLUSION

The evidence presented in this study indicates that

the purple samples examined by GIA were dyed opals from Ethiopia. The distinct hydrophane nature of Ethiopian opal makes it susceptible to many kinds of treatment—dyeing is just one. Smoke treatment of this opal to turn it black was recently described (Williams and Williams, 2011), as was sugar treatment (see the Gem News International entry on pp. 333–334 of this issue). Dyeing this material to more believable colors, such as the orange of Mexican fire opal, may pose new identification challenges, and we can expect to see more treatments applied to this hydrophane opal in the future.

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