Sapphire Origin Determination – Past and Present

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In the world of gems, "origin" can refer to the source country, mode of formation, or cause of color for a particular gemstone. "Geographic origin" is the location from which the gem originates. "Mode of formation" refers to the geologic conditions under which the gem was formed. "Cause of color" refers to the color-causing trace elements and the oxidation state of those chromophores under natural conditions or after heat treatment. For example, a sapphire may originate from Sri Lanka (Figure 1), have been formed under metamorphic conditions, and been heat treated to improve its color. To better understand how these terms evolved, it is important to understand the gemological history that brought us to this point.

Geologists and gemologists have studied the origin of sapphires for hundreds of years. Early gemologists focused their attention on the physical and optical properties of corundum. Prior to the turn of the 19th century they were more concerned about

Facing page: These sapphires, ranging from 3 to 50 ct, are from Sri Lanka and Madagascar. Courtesy of RareSource. Photo by Dirk Bakker.

Figure 1 (above): Sri Lankan sapphires: 20.26 ct yellow round, 12.04 ct pink cushion, 15.11 ct padparadscha oval, 15.14 ct blue oval. Courtesy of RareSource. Photo by Mark Mauthner.

Figure 2: Dr. Edward Gübelin using the darkfield Gemmoscope, which he developed in 1942 using the latest in Zeiss optics.

the many simulants that were used to mimic ruby and sapphire. The first serious identification challenges came when synthetics and cultured pearls entered the scene at the end of the 19th century. By 1913, it was estimated that over 10 million carats of flame-fusion synthetic rubies were being produced annually. Chromium was used to create ruby, but efforts to create blue sapphire were hindered by early attempts to use cobalt. Once iron and titanium were introduced, the color was greatly improved.

The gemological microscope was the primary instrument used for distinguishing between the new flame-fusion synthetics and their natural counterparts. The process resulted in curved growth layers known as "curved striae" observed under the microscope. In 1926 Dr. Herman Michel from Vienna published *Die Kunstliche Edelsteine* (*Artificial Gems*), one of the first comprehensive books on imitation and synthetic gemstones featuring early inclusion photographs comparing natural with imitation and synthetic gems. In 1923, Edward J. Gübelin's father established a small but well-equipped gem-testing laboratory at the Gübelin company headquarters in Lucerne, Switzerland. Edward Senior sent Charles Salquin and later his son Edward Joseph to study under Professor Michel in Vienna to become more aware of various gems and their new synthetic counterparts.

After completing his university studies in mineralogy, Edward J. decided to enroll in correspondence classes with the Gemological Institute of America (GIA). He traveled by steamship to New York and completed his coursework in Los Angeles in 1939, becoming the first European to receive GIA's Certified Gemologist diploma, the equivalent of today's Graduate Gemologist. He returned to Lucerne and rejoined Salquin in the lab, which served the Gübelin stores and their customers. As word got out that Gübelin was issuing letters of authenticity for the gems sold in their jewelry, other stores wanted to know if the lab could test their gemstones (Figure 2). In 1940 the firm began issuing diamond and colored stone certificates signed by Edward J. Gübelin. Initially there were only a few requests, but as more and more synthetics entered the industry, more gems were submitted. Throughout his career, Gübelin traveled extensively to gemstone sources and published numerous books and hundreds of articles in magazines and journals and was a prolific contributor to *Gems & Gemology*, *The Journal of Gemmology*, and many others. In 1953, GIA published Gübelin's *Inclusions as a Means of Gemstone Identification*, the first book dedicated to the study of gemstone inclusions. Gübelin's 1974 *Internal World of Gemstones* captivated a young John Koivula, who ultimately became the global expert on inclusions in gems. Geographic origin classification was not as important as it is today, so most inclusion study focused on differentiating between natural and synthetic or imitation gem materials.

In 1986, Gübelin and Koivula coauthored the first in what would become the *Photoatlas of Inclusions in Gemstones* series. These three volumes represent the most comprehensive study of gemstone inclusions ever published. Meanwhile, *The Journal of Gemmology* and *Gems & Gemology* produced many articles on natural gemstone sources as well as imitations and synthetics.

The Gemmological Association of Great Britain, known today as Gem-A, was founded in 1908 by the National Association of Goldsmiths to provide the UK with reliable gem experts and a gemological testing facility to restore confidence to the jewelry sector in the face of synthetics and cultured pearls. Interestingly, its first American graduate was GIA founder Robert M. Shipley. Basil W. Anderson, a pioneer in the gemological application of spec-

Figure 3: Laser ablation–inductively coupled plasma–mass spectrometer (LA-ICP-MS) system used for the quantitative chemical analysis of trace elements in gem materials. When the laser is focused on the surface of the gem sample, it removes very tiny particles of material for analysis in the mass spectrometer. Courtesy of GIA. Photo by Kevin Schumacher.

troscopy, wrote the first gemology diploma course for the Gemmological Association in 1910. He later became the first director of the Gem Testing Laboratory, which was established in 1925. In those days, the gemological spectroscope was used to identify gems based on their absorption spectrum within the visible range. The Gem Testing Laboratory was also renowned for cultured pearl testing using a pearl endoscope to detect the mussel shell nucleus. Anderson first published *Gem Testing* in 1942. *The Journal of Gemmology* was established in 1947, becoming the first European journal dedicated to the subject. Though it was a significant contributor to gemological testing and laboratory developments in the 20th century, Gem-A today is dedicated primarily to gemological education, gem instruments, and publishing *The Journal of Gemmology* and *Gems & Jewellery*.

In 1931, Robert M. Shipley founded GIA as a school for gemology. His goal was to restore the public's trust in the jewelry trade by training "certified" jewelers who would eventually be united in a national guild. This guild was founded in 1934 and became known as The American Gem Society. This was also the same year *Gems & Gemology* was established, becoming the first publication dedicated solely to gemology. Under the guidance of Richard T. Liddicoat, GIA devised the famous Four C's of color, cut, clarity, and carat weight and created the International Diamond Grading System in 1953. GIA began issuing its first diamond grading reports in 1955. The lab also accepted colored gems and quickly became world renowned for its expertise in identifying gemstones, synthetics, simulants, and treatments.

Robert Crowningshield became the laboratory director in New York in 1950 and is credited with many gemological achievements, including the use of the spectroscope to identify irradiated yellow diamonds. He also used the spectroscope to identify almost every colored gemstone he tested. Crowningshield is credited with the first comprehensive study on lotus-colored *padparadscha* sapphire, which has posed other origin challenges over the decades. There is considerable debate over the true color of a genuine *padparadscha*, and some maintain that only those from Sri Lanka are deserving of this regal name. The major labs have conducted extensive research into what historically qualifies as a *padparadscha*, and each has its own color criteria. Most have decided that it should be a color call and not dependent on origin. Heated examples that are not heavily zoned may also qualify.

Since most synthetic sapphires were grown using the flame-fusion process, identifying them was quite easy for the trained gemologist. Advanced heat treatment techniques to improve color and clarity in sapphires, introduced in the early 1970s, dramatically increased the supply of sapphires and rubies. This created concern about being able to differentiate between heated and non-heated corundum. To keep up with the effects of heat treatment on inclusions and absorption spectra, GIA and European labs initiated before-and-after treatment experiments. The U.S. investment boom of the late 1970s and early 1980s contributed to higher and higher prices, which resulted in more demand for gems with accompanying lab documents. It quickly became evident that most of the rubies and sapphires being sold as investment gems had been heat treated. The public was initially unaware and did not seem to care much about the process until their investments collapsed in the early '80s. The greater demand for lab reports overwhelmed existing labs and provided an opportunity for other competing labs to emerge.

Geographic origin and cause of color did not really become a concern until the 1970s, when auction houses made a few major mistakes and buyers began insisting on third-party laboratory opinions for added security. At the time, the Gübelin Laboratory was the only one providing geographic origin reports. The Swiss Foundation for Gemmological Research (SSEF) was founded in 1974 by a group of dealers in Basel as a European alternative to the Gübelin Gemmological Laboratory. The SSEF's first director, George Bosshart, became an expert on advanced spectroscopy, particularly in diamonds.

Meanwhile, there was no laboratory offering geographic origin reports in the United States. Then, in 1977, Cap Beasley founded American Gemological Laboratories and decided two years later to focus solely on colored stones. He was also a pioneer in color description and grading using a numerical system. By the early 1980s, sophisticated buyers were asking for untreated rubies and sapphires with geographic origin determination. Along with inclusion analysis, the Gübelin Gem Lab and SSEF drew upon technology from outside the gemological field and began using a spectrophotometer. This machine was capable of measuring the UV/visible/near-infrared absorption spectrum one nanometer at a time. It provided a graphic printout of the measured spectrum, which could then be compared with known origin spectra. Along with the observation of microscopic inclusions, this was the main tool for origin determination in the 1980s and 1990s.

Energy-dispersive X-ray fluorescence analysis (EDXRF) was another technique used to identify trace elements in gems. For example, sapphires of basaltic origin have higher trace amounts of iron than those of metamorphic or pegmatitic origin. In the late '80s and early '90s, Professor Stern from the University of Basel was one of the pioneers in this arena. Both Gübelin and SSEF used the university's XRF machine until they were each able to purchase their own many years later. This machine was also essential in identifying the synthetics that flowed into the global market after the collapse of the Soviet Union. For decades the Soviet government had funded the creation of almost any synthetic gem imaginable for scientific and strategic applications. Fourier-transform infrared (FTIR) spectroscopy also became more useful, particularly for identifying treatments and fillers. Bosshart went to the Gübelin Lab in the early 1990s and Henry Hänni became the new director of SSEF. Under Hänni's supervision, SSEF was one of the first labs to employ Raman spectroscopy.

Although GIA didn't provide geographic origin reports until the early 2000s, its lab used the same advanced instruments for gem identification and gem treatment detection. Through the years they were able to develop a large reference database. In 2005, GIA acquired the Edward J. Gübelin gem collection comprising over 2,800 gems. The collection serves as the foundation for their online Gem Database Project, which provides gemstone images, inclusion photomicrographs, and chemical, spectrometric and X-ray data that anyone can access online.

Japan was the largest colored gemstone consumer in the 1980s and 90s. The country's enormous demand coupled with limited consumer knowledge created a perfect environment for gemological laboratories to thrive there. Today there are approximately 30 independent gemological labs in Japan. The top international labs also benefited from the tremendous demand generated by Japan during that time. One of the major sapphire developments in the 1990s was the surface diffusion technique to improve the color of a blue sapphire. The process would alter the surface by diffusing iron with titanium to improve blue sapphire or by diffusing iron with chromium to create orange sapphire. At first this process was easily identified by color concentrations on the facet junctions and in fractures when the sapphire was viewed under diffuse transmitted illumination while immersed in methylene iodide or other liquid with a similar refractive index.

In the early 2000s, a more advanced diffusion technique using beryllium allowed for deeper diffusion, which required more advanced testing techniques to identify the treatment. This "lattice diffusion" process was identified in pink orange sapphires made to look like fine *padparadscha* sapphire. Unfortunately, thousands of these diffusion-treated *padparadscha* sapphires reached the Japanese market before some of the labs there realized what was happening. Because this process requires higher temperatures, sapphire with inclusions showed obvious alteration. But cleaner examples required instruments that could detect light elements, which most labs did not use unless special testing was required. In those days, analytical techniques that could accurately detect light elements such as beryllium were only available at universities. These techniques included secondary ion mass spectrometry (SIMS), laser ablation–inductively coupled plasma–mass spectrometry (LA-ICP-MS), and laser-induced breakdown spectroscopy (LIBS).

SIMS can be used to analyze the composition of solid surfaces and thin films by hitting the surface of the material in question with a focused ion beam measuring parts per billion. SIMS is commonly used in geology to identify rock composition. This method of testing is primarily used on research material due to small cratering it causes on the surface being tested and is therefore rarely used on client gems. Before the advent of Raman, this was the only way to accurately identify small surface reaching inclusions in gemstones.

LA-ICP-MS (figure 3) is a newer technique, borrowed from the medical, forensic, and pharmaceutical fields, that allows for advanced quantitative chemical analysis. A laser is used to ablate the surface of the material by creating a plasma gas that allows for measurements of parts per quadrillion. The laser damage, which can be focused on the girdle of the stone, is so minute that the test can easily be applied to client gems with the owner's permission. This method can also be used for radiometric age-dating of isotopes such as uranium and lead. In 2016, the Gübelin Gem Lab began using this technique to age-date sapphires by measuring zircon inclusions. If the age of the inclusion can be determined, one can assume that the host formed around that same time period. Since we know the general time periods in which sapphires formed in different parts of the world, it is possible to narrow down the geographic origin based on inclusion age. For example: Kashmir sapphires from the younger Himalayas were formed approximately 15 million years ago, while sapphires from the older Mozambique belt region in Africa are approximately 500 million years old.

LIBS allows a short laser pulse beam to be focused onto the sample surface, creating a high-temperature plasma gas of free electrons, atoms, and ions. When the plasma cools it emits light with spectral peaks that can be associated with specific elements. This quantitative technique measures in the parts per billion and is extremely fast and accurate. It has also become much more affordable in recent years and thus more widely used among gemological laboratories. Trace elements and their relative ratios have become extremely important in distinguishing a gem's possible source country, its mode of formation, and in some cases even the exact mine it came from.

The latest and most exciting technological development is inductively coupled plasma–time of flight–mass spectrometry (ICP-TOF-MS). This technique allows for the spectral measurement of the lightest to the heaviest isotopes with greater speed and accuracy. It essentially takes a snapshot of the entire mass spectrum, providing a fuller elemental composition of the gem material in question. The limitations are that the user must provide a preselected list of isotopes and that its sensitivity to lighter elements is only in the parts per million. Future developments will likely rectify these current limitations.

While these incredible advancements in gem testing techniques have helped labs gain more accurate data, new gemstone sources with overlapping characteristics continue to pose challenges to accurate geographic origin determination. The 2008 U.S. ban on Burmese jade and ruby spurred demand for geographic origin designation. The sociopolitical issues driving demand for origin reports stem from sustainability, transparency, and traceability concerns that consumers are expressing through their buying choices. In the future, this may become as important a driver for origin demand as the rarity of an exotic locale is today.

The challenges facing gemstone origin will likely continue, but technological advancements will hopefully narrow the inconsistencies among gemological laboratories. As long as research scientists continue to share their expertise and collaborate with the incredibly talented laboratory gemologists around the globe who are dedicated to providing the best service possible, the future of origin reporting will be secure and reliable.