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VOLUME 80 | JANUARY 2024


## IIGJ - RESEARCH \& LABORATORIES CENTRE

DELHI $\downarrow$ JAIPUR

## MARKING 52 YEARS OF GTL: A LOOK AT THE HISTORY

It all began in the late 1960's. Many new gemstones, synthetics and treated gemstones were circulating in the trading circles, and the Indian jewellers were in general ignorant of the technological advancements outside the country. In this scenario, Padmashri Khailshankar Durlabhji, also the founder chairman of the Gem \& Jewellery Export Promotion Council, initiated the entire concept of opening a laboratory in Jaipur. There was considerable opposition to this! However, with the support of few farsighted and dedicated people, in the interest of upliftment of the Indian Gem \& Jewellery Industry, a dialogue was started with the Gem Testing Laboratory of Great Britain (now Gem-A), and Mr. Basil W. Anderson, one of the world's leading gemmologists, was the main advisor who worked out the basic requirements for a laboratory at Jaipur. Hence, with the arduous efforts of a few honourable members of this trade, Gem Testing Laboratory (GTL) was born in Jaipur on $12^{\text {th }}$ August 1972.

For a city which prided itself on its 'eye of experience', this was a source of amusement and disdain. There were strong questions raised regarding the necessity of a laboratory in Jaipur. Vinod Gupta, the first gemmologist at GTL had a tough task ahead of him - doing his job and convincing the traders of his genuineness. Over the next seven years, there was a gradual change in the attitude of local traders and with this came a reluctant, though partial acceptance of the laboratory. However, GTL faced many challenges, hiccups, ups and downs during the next decade due to the unavailability of qualified gemmologist to run the operations.

In January 1990, with the appointment of Shyamala Fernandes as chief gemmologist, there was no looking back! Revamp of testing systems and protocols to match with international standards helped GTL to make steady progress in establishing its acceptance and niche in the industry.

Education being the need of the day, Diploma Course in gemmology were formally commenced in October 1990, although, gemmology courses were also being conducted in 1970s. Various types of gemmology courses were being offered by GTL until 2010, when Indian Institute of Gems \& Jewellery (IIGJ) was established in Jaipur. Since 2010 all gemmology courses designed by GTL are being offered by IIGJ, Jaipur.

The year 2012...a leap for GTL
GTL took a big leap in the year 2012 with the introduction of new and advanced spectrometers in its armoury. The laboratory procured some of the most advanced and sophisticated equipment available, such as Laser Raman and UV-Vis-NIR spectrometers, DiamondView imaging system, Imaging FTIR (i.e., FTIR with a microscope), etc. making GTL as advanced as any other gemmological laboratory worldwide.

## And then, the year 2020

Standing on the threshold of completing 50 years of existence, GTL was relocated to a sprawling $10,000 \mathrm{sq} \mathrm{ft}$ premises in Sitapura, Jaipur. Laboratory infrastructure now includes few highly sophisticated, sensitive, and state-of-the-art instruments, such as Laser Ablation- In Coupled Plasma - Mass Spectrometer (LA-ICPMS) for chemical fingerprinting, xray microCT for 2D/3D x-ray imaging, automated screening machines for loose and mounted diamonds, in addition to other spectrometers such as Laser Raman, Infra-red, X-ray fluorescence, etc.

Taking this legacy into future, GTL was merged with the Indian Gemmological Institute - Gem Testing Laboratory, Delhi - another laboratory set-up by the GJEPC in the year 1978, to form IIGJResearch \& Laboratories Centre (IIGJ-RLC).

There are many firsts with GTL, some of them are as follows:

* GTL was the first Indian gemmological laboratory to use Fourier Transform Infrared (FTIR) spectrometer for gem testing in the year 1999.

* GTL was the first Indian gemmological laboratory to start separating nature of fillers in emerald i.e., oil vs resin, in the year 2000.
* GTL was the first institute worldwide to introduce Master's Diploma in Gemmology, duration of which was eight months. The programme was aimed to train and polish the candidates to take up a role as gemmologist in any gemmological laboratory.
* GTL was the first Indian gemmological laboratory to use Energy Dispersive X-ray Fluorescence (EDXRF) spectrometer for gemstone analyses.
* GTL's current director, Gagan Choudhary is the first and only Indian gemmologist to be on editorial board of GIA's Gems \& Gemology journal.
* GTL is the first and only Indian gemmological laboratory to use Laser Ablation - Inductively Coupled Plasma Mass Spectrometer (LA-ICPMS) for gemstone analyses, especially, while issuing reports on origin of gemstones.

Publications and conferences are the lifeblood for any academic institution. GTL has not lagged in this too! Shyamala Fernandes in 1990's started publishing papers and articles in various gemmological and trade journals, along with participation in international conferences. This was carried on by the current director and chief gemmologist, Gagan Choudhary, who till date, has over 150 articles in various international and domestic journals, in addition to three books on gemmology under his name.

With the struggle to create its acceptance in Jaipur trade until early 1990's, GTL (now, IIGJ-Research \& Laboratories Centre) has now grown into one of the most serious and reputed gemmological laboratories worldwide after 52 years of hard work, sincerity, dedication, and integrity.

## LA-ICP-MS: A POWERFUL ANALYTICAL TOOL

Gem testing has undergone a remarkable transformation in recent years with the integration of advanced analytical techniques in all major gem labs. One such groundbreaking technology is Laser Ablation - Inductively Coupled Plasma - Mass Spectrometry (LA-ICP-MS).

LA-ICP-MS is a cutting-edge analytical technique that combines laser ablation with mass spectrometry to analyze the elemental composition of materials. In gemmological context, this technique allows scientists and gemmologists to precisely determine the concentrations of various elements within gemstones up to ppb (parts per billion) levels. This powerful analytical method has revolutionized the field of gemmology, offering unprecedented insights into the composition, origin, and authenticity of gemstones.


1. LA-ICP-MS installed at the IIGJ-RLC is extensively used for detailed chemical fingerprinting, before issuing reports on origin.

IIGJ-RLC is the first and only gemmological laboratory in the country to house LA-ICP-MS for its routine certification, thereby making the issued reports more authentic.

In addition to detailed inclusion study, which forms the basis of origin determination by most gemmological laboratories, comprehensive chemical fingerprinting, using LA-ICP-MS, is routinely performed at IIGJ-RLC, before issuing a report on origin. Detailed chemical fingerprinting becomes more important when two stones are formed in similar geological environments but belong to different locations. For example, emeralds from Colombia and Afghanistan (Panjsher), both forming in similar geological environment, display similar inclusion scene, but have different chemical ratios.
2. Inclusion scene in emeralds from Colombia (top) and Afghanistan (bottom) are very similar, such as these jagged 3-phase inclusions. However, chemical


Similarly, emeralds from Russia and Colombia appear similar, but have different chemical ratios. The three commercially important sources of emerald, namely, Zambia, Ethiopia and Brazil - all associated with mica schists have very similar inclusion scene, and hence chemical fingerprinting helps separating these emerald sources.


3. Binary plot of Cs-Li can conclusively separate emeralds from Colombia and Afghanistan (Panjsher), even though their inclusions are similar.
4. Ternary plot, such as this comparing ratio of Cs-Li-Rb is useful in separating schist-hosted emeralds from Zambia, Ethiopia and Brazil.

Another example is that of rubies originating from Myanmar (Burma) and Mozambique; although rubies in both these deposits have formed in different geological environments, but their appearance tend to overlap in many cases.

Myanmar rubies are known for their bright and fluorescent red colour, mainly due to high chromium and low iron contents, whereas Mozambique rubies exhibit a darker red, primarily due to their higher iron content and lower chromium. Therefore, measurement of iron content in ppm can help separate the rubies in low iron or high iron types, followed by analyses of

5. Ternary plot, such as this comparing ratio of $\mathrm{Fe}-\mathrm{V}-\mathrm{Cr}$ is useful in separating rubies from Myanmar (low iron) and Mozambique (high iron). other elements, such as vanadium or gallium.

Hemwant Yadav \& Pushpendra Solanki

## COBALT BEARING GAHNITE

Gahnite $\left(\mathrm{ZnAl}_{2} \mathrm{O}_{4}\right)$, a zinc-rich member of the spinel group has gained some attention in the gem industry in recent years due to its discovery in Nigeria's Kaduna state. The IIGJ-Research \& Laboratories Centre (IIGJ-RLC), Jaipur also received few faceted specimens of gahnite for identification, although in small sizes.

The unknown submitted transparent samples were rich blue in colour and weighed in the range 0.27 - 0.30ct. Refractive index was 'over the limit' of gemmological refractometer, while hydrostatic specific gravity was measured at $4.38-4.46$; these specimens appeared pink to red under Chelsea filter and displayed strong cobalt-related features in desk-model spectroscope. Reaction under Chelsea filter and absorption pattern suggested presence of cobalt.

Raman spectroscopy revealed major peaks at $\sim 420$, 510 and $660 \mathrm{~cm}^{-1}$, which matched with Raman spectra of gahnite as per RRUFF database. EDXRF analyses revealed high zinc content, associated with gahnite. To confirm cobalt-related features observed in desk-model spectroscope, UV-Vis-NIR spectroscopy was further performed to establish the cause of colour. Absorption spectra displayed strong cobalt-related features between $\sim 500$ to 650 nm , although associated with weaker iron-related features; additional iron-related features were visible at $\sim 460$ and 470 nm .

6. Representative sample of rich blue cobalt-bearing gahnite submitted at the IIGJ-RLC, Jaipur for identification.

7. UV-Vis-NIR spectrum of blue gahnite characterized by dominant cobalt-related features in the region 500-650nm. Features at 460 and 470 nm are related to iron.

Detailed and more accurate chemical analysis was performed using LA-ICP-MS, results of which showed zinc $(\mathrm{Zn})$ or gahnite $\left(\mathrm{ZnAl}_{2} \mathrm{O}_{4}\right)$ component to be more than $95 \%$, with other members of Alspinel in minor concentrations.

Under magnification, major inclusions present were scattered transparent and anhedral colourless crystals, which were identified as quartz and zircon by Raman spectroscopy. In addition, zones of fine discs were also present, which appeared to follow the cubic symmetry of spinel.

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This is to reiterate that spinel is a group of minerals with four major species of gem interest: spinel $\left(\mathrm{MgAl}_{2} \mathrm{O}_{4}\right)$, galaxite $\left(\mathrm{MnAl}_{2} \mathrm{O}_{4}\right)$, hercynite $\left(\mathrm{FeAl}_{2} \mathrm{O}_{4}\right)$ and gahnite $\left(\mathrm{ZnAl}_{2} \mathrm{O}_{4}\right)$. And, based on the composition, each species is classified. The samples described above contain high percentage of Zn , and hence belong to the gahnite end member. Although, these gahnite samples are coloured blue by cobalt, they cannot be classified as 'cobalt spinel', which is Mg-rich species. However, IIGJ-RLC adds a comment to its identification reports, stating, "This gahnite is coloured by traces of cobalt".

Gagan Choudhary

## CHAROITE WITH UNUSUAL BANDING

Charoite is an ornamental gem known for its purple colour with wavy to irregular bands and silky lustre. However, recently we received few purpleviolet beads of charoite which displayed bands with a prominent and angular orientation, rather than irregular patterns commonly seen. Further, these beads also lacked the typical silky sheen or lustre, associated with charoite.

Due to lack of typical visual features, Raman spectroscopy was performed on all beads. Raman spectra had a good match with those of charoite, present in laboratory's and RRUFF databases, based on which identity of these beads as charoite was established.

9. A classic example of charoite displaying irregular wavy bands and silky sheen / Iustre.

10. These beads of charoite lacked typical wavy bands and silky lustre but displayed bands with prominent and angular orientation.

11. Charoite beads displayed prominent and angular orientation of bands / planes, instead of irregular wavy bands.

Further microscopic study of these beads suggested that the bands are associated with planes oriented in angular patterns, commonly seen in minerals with easy cleavages or flaky structures. The presence of these planes also suggested low compactness of these charoite samples, and hence the absence of silky sheen or lustre. In addition, due to the weakness in structure and presence of planes, these beads were impregnated with a colourless resin.

## TRANSPARENT SCHEELITE

IIGJ-RLC, Jaipur received a transparent yellow coloured faceted gemstone for identification, which unusually turned out to be scheelite. The submitted 3.86ct sample displayed a bright vitreous to sub-adamantine lustre with eye visible inclusions, making the sample slightly hazy. The sample showed anisotropic optic character, refractive index was 'over the limit' of standard refractometer, hydrostatic SG was measured at 5.86, and under UV light, it appeared blue in shortwave and orange in longwave.

12. This 3.86ct yellow scheelite was submitted at IIGJ-RLC for identification.

Under magnification, fluid inclusions such as fingerprints and 2-phase were seen, along with weak to moderate doubling of facet edges.

Identity of this sample was established by Raman spectroscopy, which displayed features typically associated with scheelite, and EDXRF which revealed tungsten (82.08\%) and calcium (16.62\%) as major components.

Scheelite is a calcium tungstate mineral $\left(\mathrm{CaWO}_{4}\right)$ and is a major ore for tungsten. Although, scheelite is found at many places worldwide, gem-quality crystals are rare, and receiving such gems for identification is unusual for gem labs.

Gagan Choudhary

## DYED SAPPHIRE WITH ‘CHECKERBOARD’ PATTERN

A 4.27ct pink stone was submitted for identification at the IIGJ-RLC, Jaipur which caught attention due to its unusual checkerboard pattern visible face up. The submitted sample displayed criss-cross pattern of planes with pink-red colour, against a colourless body. Because of this unusual visual appearance, stone was directly observed under the microscope; presence of pink-red dye along the planes and other surface reaching fissures was much obvious. Such pattern of intersecting planes referred as 'twinning planes' are commonly seen in rubies and sapphires from many locations. These twinning planes because of micro-spaces also allow other natural substances or minerals such as boehmite or diaspore to deposit along them.

13. This 4.27ct dyed pink sapphire was unusual for its checkerboard pattern.

In many cases, such as low-quality translucent to opaque corundum with such twinning planes are dyed into several colours like red, green, blue, etc. However, transparency, colourless body, and seepage of dye along twinning planes made this specimen interesting.

Further gemmological and spectroscopic tests confirmed the specimen as sapphire.

Gagan Choudhary

14. Pink to red colour concentration along twinning planes was obvious in this 4.27 ct dyed pink sapphire.

## HARDENED PAINT ‘FORDITE’

Although fordite is not a gemstone but has gained a lot of popularity as a gem, for use in fashion jewellery. Fordite is a paint slag or say, a byproduct of automotive paint industry. It is basically a hardened paint, which is formed as a result of continuous deposition of paint layers over a period of time. During the era of hand spray-painting of cars, oversprayed paint accumulated in layers, which gradually hardened up during the curing process of paint in ovens; some of these paint layers have gone through the baking process numerous times.

Eventually, the hardened paint layer became so thick and heavy that it turned out to be an obstacle in the assembly line and had to be removed. The resultant multicoloured attractive material caught eyes of factory workers, to be used for adornment, and further use in jewellery.

15. Representative sample of hardened paint ' fordite' displaying striking colour layers / bands.

The name 'fordite' is inspired by the Ford Motor Company, where it was first extracted in 1940s, but today the name is applied to any hardened paint (slag), from various automotive plants. Formation of fordite is now ceased due to technological improvement in automotive paint industry too; cars are now painted with electrostatic process which leaves no or little oversprayed paint. $\uparrow$ Gagan Choudhary

## BROMINE GLASS-FILLED SAPPHIRE

Recently, we received a transparent 12.08ct pink stone (said to be ruby / sapphire) for origin identification, which based on the gemmological properties was identified as sapphire. Under magnification, the stone displayed clusters of colourless crystals which appeared to have slightly corroded surface, scattered brown crystals, liquid films associated with whitish crystals, planes of negative crystals (fingerprints) and few surface reaching fissures. Overall inclusion scene did not reveal obvious signs of heating, since corroded surfaces of crystals may also be seen in few mineral phases, such as boehmite or diaspore.

Careful observation of surface reaching fissures revealed blue and golden (yellow) colour flashes; such flash effects are commonly seen in rubies and sapphires filled with high RI glass, such as lead or bismuth. Therefore, based on colour flashes, presence of filling was established. However, to determine exact nature of filler, EDXRF analysis was performed, which revealed presence of bromine ( Br ) and calcium $(\mathrm{Ca})$, while the expected lead $(\mathrm{Pb})$ or bismuth (Bi) were not detected.

Presence of bromine in this sapphire was unusual and interesting for us, as bromine was detected as a filler substance in corundum for the first time at the laboratory since the commercial introduction of glass-filled rubies in gem trade in the year 2003. However, bromine is regularly detected in glassfilled diamonds along with lead. Furthermore, no references of bromine being present as a filler in corundum could be found but has been reported as a filler in diamonds.

Further chemical analysis suggested Myanmar (Burma) to be the origin of this sapphire.

Gagan Choudhary

> 16. This 12.08 ct pink sapphire originating from Myanmar was filled with bromine-based glass.

17. The 12.08ct pink sapphire displayed blue and yellow colour flashes along fissures suggesting presence of high RI glass. Also note the cluster of crystals with slightly corroded surface.

18. Presence of bromine (Br) in this filled sapphire was confirmed with EDXRF spectroscopy.

## COATED SILLIMANITE CAT'S EYE, IMITATING CHRYSOBERYL

IIGJ-RLC, Jaipur received three transparent yellowish green cabochons displaying sharp cat's eye effect for identification. All the three samples were impressive because of their even colouration, transparency, sharp cat's eye and sizes; they weighed 16.13, 9.49 and 7.67 ct , respectively. Their visual appearance such as sharpness of eye and body colour suggested these cabochons to be chrysoberyl cat's eye, however, gemmological testing proved otherwise.

Spot refractive index at $\sim 1.66$ was measured for all three cabochons, with a distinct birefringence blink; this straightaway ruled out the possibility of chrysoberyl, and suggested sillimanite. Under magnification, fine parallel fibres were present in one direction causing chatoyancy or cat's eye effect; in addition, base of the cabochons displayed fine step-like patterns, indicating presence of cleavage. Such orientation of fibrous inclusions and cleavage direction is associated with sillimanite. Furthermore, a layer of yellowish green colour restricted to base of these cabochons was also seen, suggesting that the yellowish green face up appearance of these cabochons is not the inherent body colour, but due to the coating layer. However, from some areas, layer of paint was chipped off.

Under UV lamp (both longwave and shortwave), these cabochons displayed a strong green fluorescence, typically seen in coated (painted) stones. Identity of these cabochons as sillimanite was further confirmed by infrared and Raman spectroscopies, which displayed characteristic spectra. $\downarrow$ Gagan Choudhary

19. These impressive cabochons with high transparency, sharp eye and even colour, imitating chrysoberyl were identified as coated sillimanite.

20. Such fine parallel needles or fibres causing chatoyancy is commonly seen in sillimanite.

21. Coloured layer with chipped off areas was readily visible at the base of these sillimanite cabochons.

22. All the three coated sillimanite cabochons displayed strong fluorescence under longwave and shortwave UV.

## FANCY COLOURED LAB-GROWN DIAMONDS ON THE RISE

With the increasing scale of production of laboratory grown diamonds along with the technological modifications in growth process, these grown diamonds now appear in much larger sizes, but mainly in the colour range of colourless to near colourless (with brown tints).

In addition to the sizes, introduction of nitrogen during the growth process and post-growth treatments have also resulted in fancy colours of lab-grown diamonds, which otherwise were restricted to pale browns to near colourless and colourless.


In past few months, IIGJ-Research \& Laboratories Centre, Jaipur has witnessed CVD grown diamonds in fancy colours on a regular basis. Some of these colours include blue, orangish brown and pink, and in sizes up to 2.01 ct.

During routine identification process, all these diamonds were identified as type Ila in infrared spectroscopy, suggesting that they typically lacked detectable nitrogen, which qualified them for further analyses in UV imaging (such as DiamondView) and photoluminescence spectroscopy. DiamondView imaging of orangish brown and pink samples displayed strong orange fluorescence and phosphorescence, while blue sample displayed weak fluorescence; in addition, all samples revealed striated growth pattern, typically associated with CVD grown diamonds.
23. Representative colours of fancy coloured CVD grown diamonds seen at IIGJ-RLC in past few months.

24. Typical example of striated growth pattern in CVD grown diamonds, along with orange fluorescence.

Photoluminescence spectroscopy further confirmed these diamonds as CVD grown; the characteristic feature at $\sim 737 \mathrm{~nm}$, associated with Si-V- defect was present in all samples, and could be detected even at room temperature.

Gagan Choudhary

25. Photoluminescence spectrum displaying feature at $\sim 737 n m$, characteristic for CVD grown diamonds.

## CVD GROWN DIAMOND ROUGH - A PHOTO STORY



Recently, IIGJ-RLC, Delhi received a parcel of synthetic rough diamonds grown by CVD process, in the colour range from near colourless to pale brown and gray, and in sizes from $\sim 2$ to 13ct. These diamonds were readily identified as CVD grown synthetic by their square profile - characteristic of the crystals grown by this process. Majority of the crystals had a gray-brown skin, while many crystals were associated with rim of aggregation of fine cubic crystals of diamond with a dark skin. Such rims are usually formed towards the end stages of growth process due to disturbance in temperature - pressure conditions, along with the supply of feed gases.

These crystals displayed various types of growth patterns on their surfaces, ranging from wavy layered to cubic growth hillocks and planes; side view of these crystals displayed parallel to sub-parallel striations. Many of these crystals also displayed incipient cleavage planes. Presented here are some examples of growth features on present these crystals. $\downarrow$ Gagan Choudhary \& Meenakshi Chauhan


## MAN-MADE GLASS WITH NATURAL-LIKE FEATURES, IMITATING EMERALD

Use of man-made glass as imitation of several popular stones is well-known in the gem trade. Several examples of fake encounters have been shared in numerous issues of this newsletter as well as at other platforms. Recently, a green transparent 16.03ct octagon step cut sample was submitted for identification, which on initial observation with unaided eyes appeared emerald, due to its typical colour shade and presence of eye-visible feather like inclusions.

However, standard gemmological testing ruled out the possibility of emerald. The sample displayed isotropic optic character with strain and single refractive index at 1.61 ; these properties suggested the sample to be a man-made glass.

At higher magnifications, the sample displayed irregular, randomly oriented feather-like features, which also contained minute whitish droplets - such patterns are commonly seen as fingerprints in emerald. In addition to these feathers, the sample also displayed randomly oriented fractures displaying slight orange to golden colour flashes- the effect commonly seen in resin-filled emeralds. The overall inclusion scene of this sample was convincing enough to misidentify it as emerald with fissure filling.

27. This 16.03 ct glass sample was interesting for its natural inc/usion scene like these feathers; also note golden coloured fissures (left) similar to those seen resin-filled emeralds.

Conclusive identity of this specimen as glass was established through infrared spectroscopy, however, infrared spectra did not display any feature associated with resin, even though orange to golden flash effect was visible. Such examples pose threat as well as challenge for a gem dealer, who is usually equipped only with a hand loupe; observation of visual appearance and inclusion scene alone may lead to misidentification even by an experienced observer.

## FAKE RUBY ROUGH

Man-made and synthetic gemstones, both rough and cut, are a regular feature at IIGJ-RLC. We, also receive fake gemstones, especially in rough form, derived and modified from man-made glass or synthetic gemstones, and presented as natural expensive gemstones, like ruby, sapphire or emerald. Recently, we received 65.14ct rough specimen which appeared to be ruby. The sample was red in colour with distinct surface markings in multiple directions and associated white and brown substances commonly seen in natural ruby rough, especially those originating from marble-hosted deposits, such as Myanmar (Burma).

Careful observation of surface features immediately raised doubts on this specimen due to absence of typical and symmetrical growth striations found in natural ruby crystals. Furthermore, white mineral associated with ruby crystals are calcite / dolomite in marble hosted rubies, which typically display a granular texture with cleavages of individual grains. However, in the specimen the surface of white substance was smooth, and no reflective surface of individual grains was visible. Further microscopic observations revealed curved zonal clouds of minute gas bubbles or unmelted feed powder, confirming its synthetic origin, grown by flame-fusion (Verneuil) process. Elongated 'bomb-shaped' gas bubbles in one direction were also present- another feature associated synthetic corundum grown by Verneuil process. In addition to this, the specimen was not simply a single modified piece of synthetic ruby, but a composite made from several pieces of synthetic ruby, to give appearance of a twinned crystal.

## A project of





[^0]:    8. Clusters of transparent colourless crystals in tested gahnite were identified as quartz and zircon. Also note small crystals with stress cracks.
