

## Lab Information Circular

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### Fracture Filling - Revisited

The fracture-filling of emeralds is causing sleepless nights to everyone associated with the emerald trade - be it a buyer, a seller, consumer or a lab technician. This issue first became the cause of concern when fracture filling of Colombian emeralds with 'Opticon', an epoxy resin was introduced in mid 1980s. These opticon-filled emeralds took more than 15 years in settling down in the trade and then was widely accepted that "Colombian emeralds are resin-filled". However, for so many years resin filling was associated with colombian stones and not with other localities. Some Brazilian goods were also being filled with resins.

Other localities like Zambia, Madagascar, Russia, etc have been commonly known for oiling. It was the beginning of December 2011, when we started to see resin-filling in emeralds of Zambian origin.

This was followed by a continuous sighting of such filled emeralds in January and as well as in February. Few initial reports of these resin filled emeralds triggered a concern amongst the trade which resulted in flow of high numbers of emeralds to the laboratory. As a result, the ratio of emeralds tested at the Gem Testing Laboratory Jaipur increased amazingly high to more than 70%. Interestingly, the percentage of resin-filled emeralds tested in January was around 23.83, while in February 2012, the figure was around 17.24 percent. Another fact about these filled emeralds is that most of such filled emeralds are high quality, in sizes more than 5 carats.

Due to this rising concern of the trade, we present an overview of fracture filling on emeralds in this issue of Lab Information Circular (LIC). As we all know, the vast majority of emeralds on the market today have undergone fracture filling or clarity enhancement. For many centuries, fissures or fractures in emeralds have been filled with oils or other natural substances to

enhance their clarity. For approximately 30 years, the substances used also comprise various artificial resins, mainly epoxy resins. And in the recent times, several other new types of resins are being used as fillers.

There is an exhaustive list of filler substances available in the market which can be used in emeralds. These have been broadly classified as, oils, resins (hardened & unhardened) and waxes (e.g. paraffin). There could be several types of oils like cedar wood oil, olive oil, sesame oil, paraffin oil, etc while resins include Opticon (epoxy),

Palm oil, Palma (epoxy), Permasafe, ExCel, etc.

The clarity of emerald can be improved by impregnating the above mentioned materials in the open fractures or fissures. Air is present in fissures and due to the

difference in the refractive index of the air and the stone, light gets scattered or reflected and the fissures are easily visible. By filling the substances that almost match the refractive indices of emerald make the fissures less visible, especially to the unaided eyes. Since, light transmits properly through the stone, the colour also enhances. Although, coloured oils and resins are also used to fill emeralds, we are not focusing these here.

Techniques used for filling emeralds involve placing them in bowls / jars of oil under a lamp or leaving the jars near the window or use of vacuum pressure to force the oil / resin into the fissures. This is however, preceded by cleaning process, sometimes multistep, using chemicals or solvents of variable harshness. Fillers, whether oil or resin are usually heated before filling to reduce their viscosity. In majority of emerald producing countries, treatment is performed on rough itself; and irrespective of the type and nature of filler, the term 'oiling' is used.



*Figure 1: Range of emeralds in various transparencies with no filler substance (top row), with oil (middle row) and with resin (bottom row)*

*continued to page 4...*

After cutting and polishing, fissures become evident on the surface and hence require re-filling. Now, the manufacturer applies oil to get rid of surface roughness, but the stone may turn out to be resin-filled. This creates confusion as, he has used only oil, but in fact, the stones were previously filled with resin.

**Oil** originally meant olive oil, which is not generally used in emeralds today. Its common meaning is one of "numerous unctuous combustible substances that are liquid or at least easily liquefiable on warming, are soluble in ether but not in water" (Johnson et al, 1999). Oils are simple to introduce into fissures and comparatively easy to clean out, and stones can be readily re-oiled. The improvement in appearance on application of oil is less than resins; the state of emerald is quite close to the real state as compared to the use of resins.

**Resin** originally meant pine sap. Today, resin can mean at least three different things: natural plant exudates (i.e., saps) hardened or unhardened; hardened manufactured polymers; or the unhardened prepolymer "building blocks" that can be used to make manufactured polymers (Johnson et al, 1999). Compared to oils, resins are more viscous and may possess solidifying property. When fresh, resins give much better appearance to a stone. They adhere well in the fissures, and hence are difficult to dissolve or remove once they change their appearance or decompose.

Currently, emerald buyers are losing confidence because of the non-disclosure or incomplete disclosure. The same was witnessed in the past also when 'opticon treated' emeralds entered the marketplace. Most of the trade associations advocate for complete disclosure of the product under sale. But in fact, complete disclosures are not always made and in many cases, the owner of the stone is unaware about the presence of treatment/s on his stone/s. And, there are also the cases, when a stone undergoes various types of treatments with different substances at various stages of trade; this makes the situation much more complicated. For example, an emerald manufacturer gets rough and cuts them himself and then places the fashioned emeralds in oil. But, he is not sure whether the rough was previously filled with oil or resin.

When talking about emeralds which are filled with oil and resin, both are required to be mentioned separately because

resin is accepted only by few traders while oils have universal acceptance. One of the major reasons for this is the stability of the filler. No doubt, resins are much more durable than emeralds, but the stability of the treatments with organic substances is difficult to define. However, this depends on time and the temperature. Further, when a filler substance is said to be permanent as well as removable, it casts uncertainty on its stability. Experiments have shown that resins (hard fillers) have also deteriorated and changed their appearance with time and under normal usage conditions, damaging the host emerald by expanding their cracks.

In general, resins have refractive index closer to emerald as compared to oils and hence the effect of filling is much better. This may also lead to a problem for a jeweller or setter as he may not evaluate the extent and position of the fractures. This may damage the emerald during setting. In addition, when compared to durability of an emerald which can be worn for many centuries and lifetimes without damage, none of the treatments (fillers) should be considered as permanent or stable.

The identification of the filler substance is possible by a step by step process. The first step is careful observation of the fracture under a microscope, followed by under long wave ultra violet light. The fractures once located are then analyzed using Infrared or Raman spectrometers to judge the chemistry of fillers, which helps to differentiate oil from resin. This is to be noted, that various types of oils and resins may not be identified conclusively, especially when present in emerald or any other stone. There can also be cases, when presence of one filler masks the features of other filler, and hence some filler present in minor quantities may not be detected. There can also be some emeralds, where filler may not be identified conclusively, due to decomposition of filler.

*On identification of fracture filling in emeralds, please see supplementary chart given as annexure 1.*

#### *Selected References:*

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Written and Edited by: [Gagan Choudhary](#), Deputy Director (Tech. & Training)  
Contact for further details:

[Meenu Brijesh Vyas](#), Asst. Director (Tech. & Training)  
[Radhamani Amma](#), Sr. Executive (Coordination & Info.)  
[Niranjan K. Srinivas](#), Executive (Tech. & Training)  
[Kailash Chand Daurata](#), Jr. Executive

#### **GEM TESTING LABORATORY**

Rajasthan Chamber Bhawan  
M.I. Road, Jaipur 302003, INDIA  
Phone: 91-141-2568221, 2573565  
Fax: 91-141-2567921  
Email: [gtl@gjepcindia.com](mailto:gtl@gjepcindia.com)  
Web: [www.gtljaipur.info](http://www.gtljaipur.info)