FILLERS AND DISCLOSURE: At the Heart of the Emerald Debate

As the controversy over emerald fillers continues, this issue of the Gazette *endeavours to throw some light on this matter with the following articles:*

1. A report from Prof. Dr. H.A. Hänni from the Swiss Gemmological Institute, on "Fracture Filling in Emeralds and Its Detection."
2. Address by Mr. Israel Z. Eliezri, Colgem Ltd., at the First World Emerald Congress, Bogota, Colombia, February 23-25, 1998.
3. "Letter from Bogota" by Mr. Robert Linder of Lindeau Gems.
4. A Paper by Mr. Harry Levy, president of CIBJO Coloured Stone Commission on the subject of Disclosure in the Gem Trade.

1 Fracture Filling In Emeralds And Its Detection With Laboratory Methods

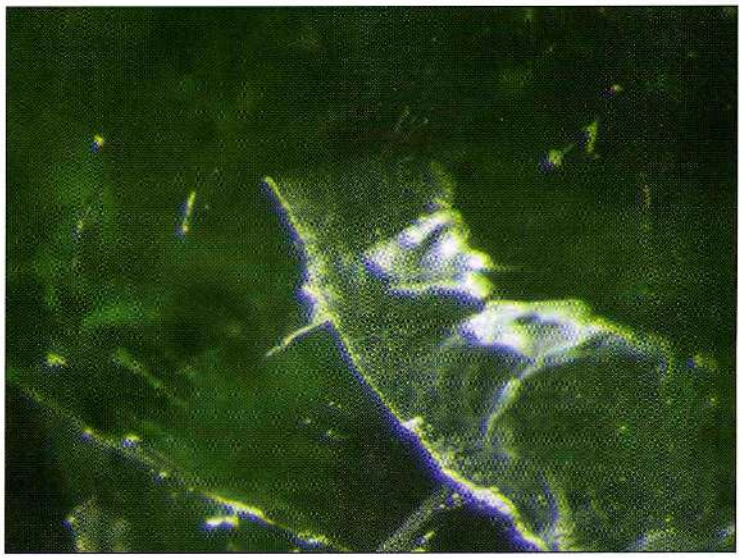
BY PROF. DR. H.A. HÄNNI, SSEF SWISS GEMMOLOGICAL INSTITUTE, BASEL, SWITZERLAND

INTRODUCTION

This articles takes up a subject which was introduced some years ago *(see March ICA Gazette 1992 "New fracture filling material have replaced oil")* to draw the attention of the readers to the subject, and give a more detailed view on what was done with emeralds.

During the past years the topic was discussed among traders, laboratory people and even consumers. In the USA recently an important law case brought to our memory that "oiling" of emeralds has to be understood and treated with prudence and delicacy.

Painful experience demonstrated that "disclosure" is requested on every level of the trade. Currently some filling substances are not at all in favour, and a special attention may be given to the analytical possibilities which allow the identification of filler substances. A general information such as "clarity enhanced" or "colourless foreign substance present" does not satisfy the needs of a great deal of emerald dealers or consumers. Some are scared by the possibility that artificial resins might decompose or polymerize, and become evident and spoil the good aspect they previously lended the stone. Also, it was always argued that epoxies were difficult if ever to remove from the depth of the fissures.



Emerald with a decomposed whitish filling, giving epoxy spectral features when analyzed with FTIR or Raman spectroscopy.

Therefore many of those who were in favour of the more permanent artificial resins prefer now again the easier soluble oils or resins,

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Fracture Filling In Emeralds And Its Detection With Laboratory Methods

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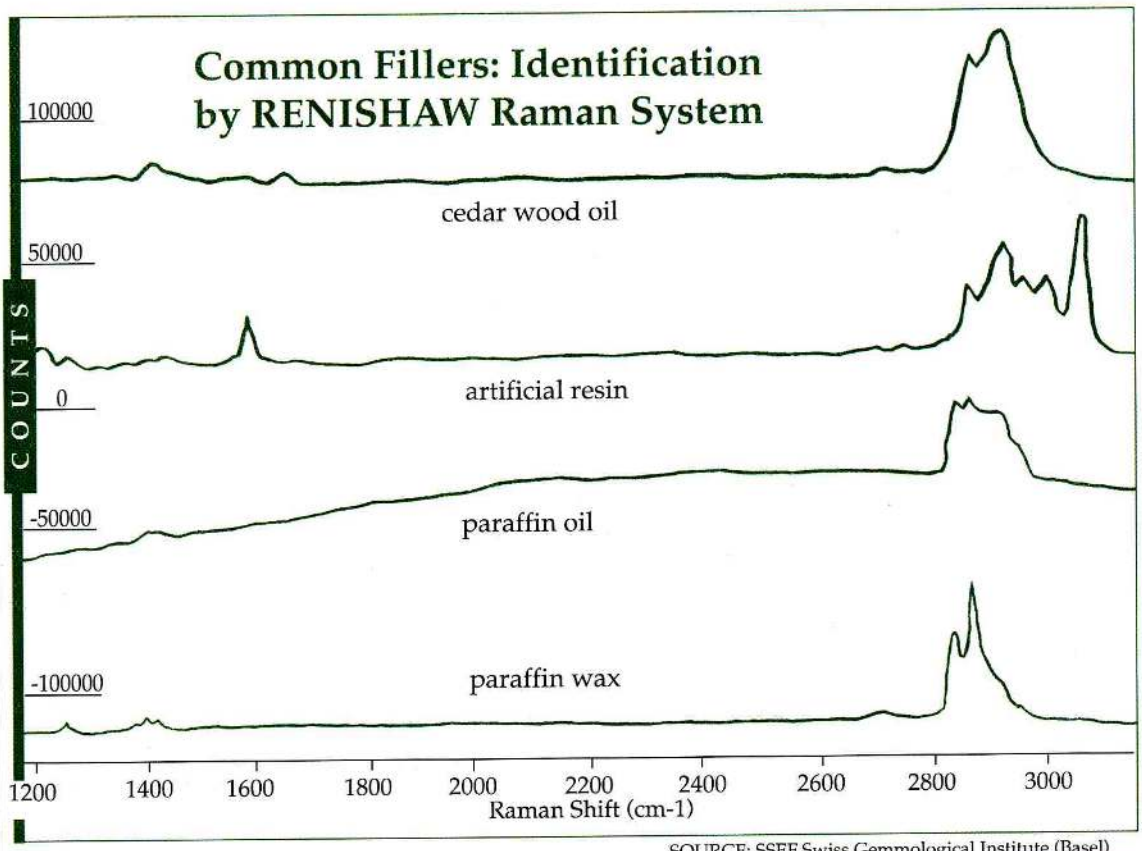
e.g. cedar wood oil.

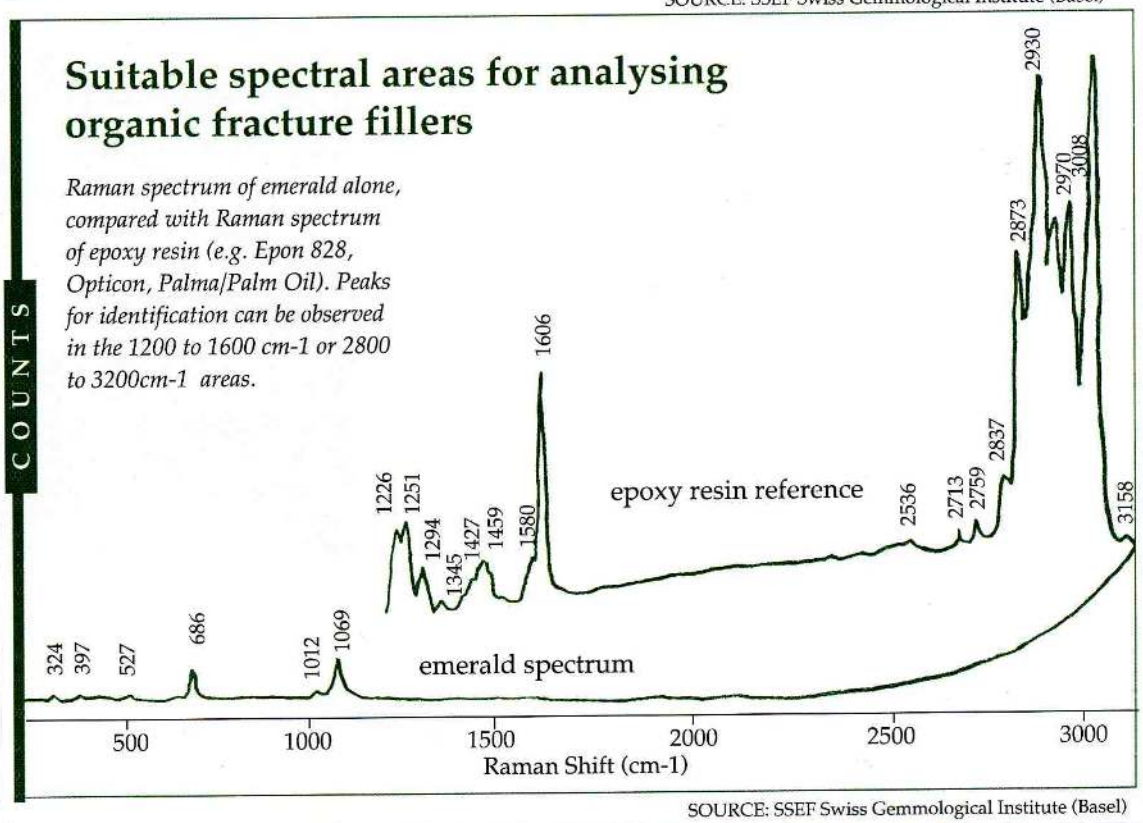
During the discussions at the latest Tucson ICA meeting and the following World Emerald Congress in Bogota it became evident that the emerald trade in Colombia is suffering due to the fact that an important number of dealers do not want to buy Palma or Opticon treated emeralds. The situation is particularly difficult because fillers cannot be identified on the spot, and the stocks seem to be already treated with epoxies. The situation in Bogota reminds one very much of the situation in Hong Kong some years ago when the epoxy-treated B-jades came up. The Chinese jade dealers, in contrast to their Colombian counterparts, quickly found ways to analyze their stones, and to identify and disclose the treated merchandise.

Observations of Fillings in Emeralds.

Fillings in fractures are usually visible during a microscopic inspection. A loupe is usually not sufficient to get a reliable estimation of the treatment when stones of higher quality are involved. In various gemmological articles the characteristics of treated fissures, e.g. air bubbles, dendritic patterns, purple or orange flashes have been described. Under UV-radiation the extent of treatment can be observed due to a whitish to yellow fluorescence of most organic fillers.



SOURCE: SSEF Swiss Gemmological Institute (Basel)



SOURCE: SSEF Swiss Gemmological Institute (Basel)

Identification of Fillings in Emeralds.

However, such observations need confirmation with analytical instruments.

An identification is usually possible with infrared spectrometry or Raman spectrometry. Where relevant molecular signals appear, an attribution to reference spectra taken from various filler substances is possible. In the SSEF Swiss Gemmological Institute fracture treatment analyses have been offered as a gemmological service since two years. The identifications are carried out in three steps. After an optical inspection (1) the stone is tested by FTIR (2) and with the Raman Microscope (3). The combination of the two methods guarantees a high degree of identification.

Fourier Transform Infrared spectrometry (FTIR) is a more versatile method of conventional IR spectrometry. A major problem is that fillers cannot be analyzed in their most characteristic fingerprint area due to the absorption behaviour of the emerald. Fortunately emeralds are transparent to infrared radiation in the 2800 - 3200 cm-1 spectral area. The analytical procedure records an average spectrum of a larger volume, since the transmitted or diffused reflectance mode is applied. This means that eventually various fillers from different fractures are cumulated in the same spectrum (macro-analysis). Especially lower grade emeralds with significant treatments are easily analyzed with FTIR.

With Raman micro-spectrometry, the detection of organic fillers in emeralds proves to be very successful. Waxes, oils and resins show distinct spectra which make it possible to identify the substance in the fissures. The characteristic peak areas are between 1300 and 1700 cm-1 and 2800 and 3200 cm-1. The analytical procedure records a micro-spectrum of a small volume, since the focused laser beam is narrow and directed into an individual fissure. This means that eventually various fillers in different fractures have to be recorded in numerous individual spectra (micro-analysis).

The situation is complicated by the fact that a stone may contain different substances, or mixtures of pure organic fillers. It is therefore necessary to compare not only peak positions, but also peak intensity ratios. This is important in order to trace admixtures or residual fillers from earlier treatments.

Because the successful identification of an organic filler by Raman or FTIR spectroscopy is confined to a reference data file, the identification depends on a comprehensive reference spectrum collection, which is maintained and constantly enlarged in SSEF laboratory since 1992.

Additionally, several users of the Raman microscope as well as the manufacturer are working on compiling a database. In our reports we differentiate between oils (e.g. cedar wood oil), waxes (e.g. paraffin), and artificial resins (e.g. Epon 828, Palma, Opticon). The latter substances are artificial, i.e. designed products without natural counterparts. Natural substances like cedar wood oil may be produced more or less from natural sources. A fully synthetic production is also possible and duplicates chemistry and structure of natural cedar wood oil.

Outlook

For those who care what filler is used for enhancement of an emerald, some specialized gemmological laboratories can identify the substances with FTIR and/or Raman spectrometry . Even combinations of substances are identified when stones were repeatedly treated with different fillers. A new service — washing out of unpleasant fillers — is offered by a few specialized companies, such as Raymond Naftule, Arthur Groom and Ted Themelis. We hope that instrumental identification of fissure fillers by comparison with reference substances will become as usual a procedure as the identification of epoxy-treated B-jade. SSEF Swiss Gemmological Institute is in due course releasing its treatment information with every single emerald, ruby, sapphire, as full disclosure, on the test reports. It is interesting to note that since we have been releasing emerald treatment slips, the amount of cedar wood oil enhanced stones increased and even a few virtually untreated emeralds have been seen in the SSEF laboratory.

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2 Address By Israel Z. Eliezri At First World Emerald Congress, Bogota, Colombia

As we are standing at the beginning of the 21st century, we look back to a century that has been most revolutionary in technological developments. We began the 20th century by riding donkeys and horses and we are about to close it while flying into space and sending space crafts to other planets. Scientists started the century using abacus calculators, but now they have incredibly powerful computers at their disposal. And a hundred years ago, my trip to Colombia from the Holy Land would have taken me close to a month leaving my home only yesterday, it took me less than a day to arrive here. Naturally, we can think of countless other examples in almost all fields of modern life today.

Looking at our professional field, things developed differently. Here, we are observing the exploration, mining, processing and mar-

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