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TSAVORITE AND OTHER GROSSULAR GARNETS FROM ITRAFO, MADAGASCAR

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Grossular [Ca₃Al₂(SiO₄)₃] belongs to the garnet group and exhibits various colors, ranging from colorless, pink, brown, yellow, orange, and green, this latter known with the varietal name "tsavorite" [1].

A new deposit, producing fine gem-quality grossular, including tsavorite, was discovered in 2002 in central Madagascar, at Itrafo, Andrembesoa, Antananarivo region. The deposit is composed by a subvertical vein, characterized by a fine-grained massive, graphite-rich, rock, crosscut by calcite veinlets, rich in grossular crystals.

A full gemological and mineralogical characterization by means of standard gemological methods, combined with EMPA and LA-ICP-MS chemical analyses, and UV-Vis-NIR and mid-IR spectroscopy, performed on various samples representative of the gem material produced in this deposit, show the typical properties of grossular worldwide [1]. In particular, the chemical composition is close to almost pure grossular (Grs>92 mol.%), in agreement with the gemological properties (RI=1.740-1.742; SG=3.58-3.62). The observed inclusions (i.e. fluid inclusions and crystals, mainly graphite) are consistent with the geological environment of graphite-bearing metamorphic rocks [2]. The chemical and spectroscopic data show that iron and vanadium (Fe₂O₃: 1.08-1.85 wt%, V₂O₃: 0.19-0.83), in various ratios, but always higher than 1, are responsible for the various colorations, ranging from brown green, brownish and yellowish green to green. In particular, iron has a significant effect of stones with evident brown and yellow hues, whereas vanadium causes the purer green colors. The low chromium content (<71 ppm) suggests that its contribution to the color can be considered negligible. Comparing the gemological, chemical, and spectroscopic features of these tsavorite gems with those from other classic localities, i.e. Tanzania, Kenya, and Gogogogo in Madagascar [3, 4, 5, 6], the main difference concerns their higher iron content (Fe₂O₃>1 wt% and Adr>3 mol.%), as well as the Fe₂O₃/V₂O₃ ratio (>1), consistently with the brown and yellow hues, rather common in these stones. Although the deposit is relatively large and new veins could be discovered, future production will be limited due to the access difficulties and the safety problems.

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AN ITALIAN JADE: THE NEPHRITE FROM VAL MALENCO (SONDRIO)

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Nephrite jade is an almost monomineralic rock, mainly a tremolite $[\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2]$ -actinolite $[\text{Ca}_2(\text{Mg,Fe})_5\text{Si}_8\text{O}_{22}(\text{OH})_2]$ composite, valued for ornamental carvings and gems. Major nephrite sources include the Kunlun Mountains of Xinjiang, China; the East Sayan Mountains of Siberia, Russia; Chuncheon in South Korea; South Westland in the South Island of New Zealand; and Cowell, Australia.

A deposit of gem-quality nephrite jade has been discovered some years ago at Mastabia, in Val Malenco, some 20 km N-NW of the town of Sondrio, northern Italy. The discovery of the nephrite jade is attributed to Mr. Pietro Nana (Sondrio) who first noticed an attractive green stone in the discarded waste materials of an abandoned talc mine situated at an altitude of 2077 m. Nephrite is hosted in a talc-tremolitic orebody, associated with dolomitic marble and calc-silicate rocks [1]

This study aims to provide a review and an update of nephrite jade from Val Malenco investigating a suite of gem-quality samples from this locality by means of standard gemmological methods, X-ray powder diffraction, combined with quantitative full-phase analysis using the Rietveld method, EMP and LA-ICP-MS chemical analyses, and mid-IR spectroscopic measurements.

The nephrite jade from Val Malenco is mainly composed of pure tremolite, with an almost stoichiometric values of Si, Ca and Mg a.p.f.u.

The XRPD data and the bulk mid-IR spectra are consistent with pure tremolite, although show, in some cases, the occurrence of minor amount of calcite, quantified by means of Rietveld analysis as minor of 5 wt%. An exceptional value of 30 wt% was measured only in one sample. The specific gravity varies with the calcite content ($G=2.74-2.96$). The nephrite jade shows a micro- to crypto-crystalline texture that consists of a fibrous intergrowth of about 10-20 μm long tremolite crystals, that occur together with other accessory constituents including calcite, talc, diopside, apatite and opaque iron minerals. Colour ranges from white to white-green, with the intensity of coloration inversely related to the calcite content, rather than to the concentration of Fe, Mn, and Cr, which are the major colouring elements of nephrite[2]. Indeed, they are present at very low trace levels (<0.1 wt%) as are the other elements of the first transition series (Sc, Ti, V, Co, Ni, Cu, Zn), the alkaline earth metals (Sr, Ba), the alkaline metals (K, Rb, Cs). The only exception is sodium which ranges from 0.12 to 0.22 wt% as Na_2O . All the nephrite samples have low total rare earth elements (ΣREE) ranging from 0.15 to 0.80 ppm, with the major contribution to the total budget given by light and middle REE (i.e. La, Ce and Nd).

On the basis of the low Fe/(Fe+Mg) ratio (<0.002) and the low content of Co (0.07-0.27 ppm), Cr (1.86-4.36 ppm), and Ni (0.21-4.36 ppm), the nephrite from Val Malenco can be classified as dolomite-related nephrite [3].

The compact and fine-grained microstructure, as well as the low content of colouring agents, confer to nephrite from Val Malenco an agreeable aspect, making it worthy of attention as gemmological material. Sondrio provides the principal outlet for finished articles but the quantity of nephrite jade available remains uncertain. Local mineral collectors continue to work the deposits and it is likely that some amounts of gem-quality pieces will continue to be produced in the future.

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Figure 1. Bowl and necklaces fashioned from Val Malenco nephrite. Photo by, and specimens courtesy of, Mr. Pietro Nana (Sondrio).

Figura 1. Coppa e collane lavorate in nefrite della Val Malenco. La foto e i campioni si devono alla cortesia del Sig. Pietro Nana (Sondrio).

BLUE AND PINK OPALS FROM ACARI, PERU

Their optical, structural and spectroscopic features

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ABSTRACT

Some blue, greenish blue, and pink Andean opals that were collected from the copper mining area of Acari, near Nazca in the Arequipa Department of Peru, have been examined for their physical properties, macroscopic and microscopic characteristics, X-ray powder diffraction patterns, EDS-MP chemical analyses, and Raman, FT-IR and ESR spectra.

All samples showed no *play-of-colour*. Determined refractive index and specific gravity were within the range of opal, except for the most transparent green samples that had higher values for both properties that were probably influenced by the presence of small percentages of chalcedony. X-ray diffraction patterns detected variable amounts of cristobalite and tridymite in both the blue and pink opal varieties, allowing them to be classified as opal-CT. The cristobalite/tridymite (C/T) ratio was generally higher in blue than pink opals.

Both palygorskite and sepiolite mineral phases were identified in yellowish opaque areas of the blue opals, whereas only variable amounts (25 to 46 wt%) of palygorskite were always found in pink opals. Small amounts of quartz were detected in both types of opal. The FT-IR spectra of both blue and pink opals highlighted the typical features of four-coordinated silica, water in opal and water of crystallisation in phyllosilicates.

EDS-MP chemical analyses revealed that SiO₂ is actually the major component in both blue and pink opals. Black dendrites in these opals proved to be a complex Ba and Mn oxide of romanechite and/or hollandite composition. Phyllosilicates, in variable amounts, were always present in both types of opal. Small amounts of CuO, below the sensitivity limit of the instrument, were detected in blue opal. Nevertheless, the presence of copper was confirmed by ESR spectra. Therefore the Cu²⁺ ion can be considered the colouring agent of blue opal. Chromophoric contributions to the colour of pink opals were detected by Raman and ESR spectroscopy and tentatively ascribed to biogenic compounds, such as carotenoids and quinones (which can be fixed by phyllosilicates), and/or to Fe(III) centres.

Based on local geology, and the determined structure and composition of the samples, the pink and blue opals-CT from Acari can be associated with volcanism and genetically related to chemical and volcanic events.

Key-words: opal, gemmological properties, X-ray diffraction, chemical analyses, chromophores, spectroscopy



Fig.1



Fig. 5



Fig. 7

A NEW THEORY FOR THE GENESIS OF JASPER AND CHALCEDONY

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Although they are very familiar materials, Jasper, Chalcedony and the numerous variety of amorphous or microcrystalline silica have maintained until today an aura of mystery and gave rise to a huge amount of little in-depth theories and suppositions. One of the problems of the study of these materials is that within the same specimen may have evidences of a plastic behavior and evidences of a brittle behavior and this has prompted the creation of theories which gave more importance to one or the other phenomena.

This study is based on the physics of colloidal silica. There are two processes that modify its properties, the polymerization which causes a hardening of the solution and the alkali-silica reaction which causes dilution. These two processes are strongly controlled by the presence of water and are shuttled between the extremes of a hard rock and a dilute solution. In the middle of the two extremes, there are two physical conditions of colloidal solutions with different properties, the sol, with the appearance of a bechamel and the gel with a gelatin appearance. The sol is subject to internal gravitative movements and has ductile behavior, while the gel is a solid and then can suffer fracture and inside there is no movement and it is like frozen.

The type of polymerization of the silica is a phenomenon that depends on pH. At high pH, the OH- groups join around the Si atom with the negative charge outward. In this case the molecules are negatively charged and may grow, but do not join together and remain separate. This is the case of Opal. In a Jasper, the polymerization is base-catalyzed and the molecules aggregate in highly branched clusters. In chalcedony instead the polymerization is acid-catalyzed and molecules have a linear growth.

To understand the genesis of siliceous materials, we must highlight the seasonal variations due to the presence or absence of water in the system. The geological environment is pedogenetic. During the wet season the alkali-silica reaction dissolves siliceous rocks such as volcanic glass, quartz sand or organic Opal of the siliceous plancton (radiolarians and diatoms) and enters the silica in solution. During the dry season, the polymerization accumulates this silica in a sol or gel state. After numerous wet-dry cycles, a large quantity of colloidal silica can be accumulated inside cavities that may be vesicles of a lava, fractures of the host rock or void inside fossils.

After the accumulation of the colloidal silica, the continuous alternation of wet and dry seasons causes the formation of numerous structures, both in jasper and in chalcedonies. With this type of analysis, you can easily explain the genesis of Thunder eggs, Orbicular Jaspers, Orbed Jaspers, Brecciated Jaspers, Jasp-agates, moss agates, agate plumes, banded agates, chalcedonies without bands and numerous other materials with intermediate characters.

The study was conducted with a pure morphological analysis of samples that allowed us to reconstruct the physics of the various stages of the formation of numerous variety of silica. A new genetic classification of Jaspers and of all silica variety have been also suggested [1].

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Fe and Mn speciation in multicoloured tourmalines

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Four multicoloured tourmalines, where the variation of colours occurs in concentric bands, have been analysed by X-ray Absorption Spectroscopy (XAS) at the Fe and Mn K-edge, in order to investigate the oxidation state and the site location of Fe and Mn, with the final purpose of understanding the physico-chemical factors affecting their colour. The natural multicoloured crystals present only Fe and Mn as chromophore elements, but despite the quite simple chemistry, they show a variety of colours including hues of blue, pink, red and green. In zones where Mn is predominant, a pink colour is observed, whereas Fe predominance induces green or blue colours. Interestingly, the presence of Fe and Mn has been recognized also in the colourless areas.

XAS measurements have been collected at beamline GILDA-BM08 of the ESRF. The beam size at the sample was 0.15×1 mm in order to be able to selectively measure the different coloured zones of the crystals. A 13-elements high-purity Ge detector allowed to collect the fluorescence spectra through a cross section of each of the samples. Detection of the Mn K α and Fe K β X-ray emission lines allowed to selectively collect profiles of Mn and Fe abundances across the studied crystals. Pre-edge peak (PE) analysis shows that Mn and Fe are dominantly divalent in all the measured samples. PE integrated intensities are compatible with the location of Mn and Fe in the octahedral sites. Preliminary theoretical XANES calculations also agree with the octahedral coordination of Mn and Fe. For these samples, the change in colour appears related to the variation in the relative abundance of Mn and Fe rather than to variations of their oxidation states.

OLD AND NEW TECHNIQUES FOR IDENTIFICATION AND STUDY OF PEARLS AND CORALS

E.Gambini¹, M.Superchi²

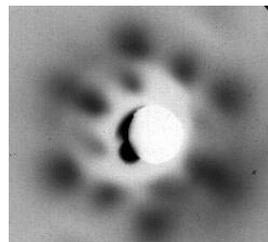
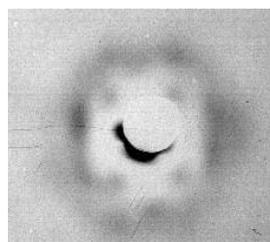
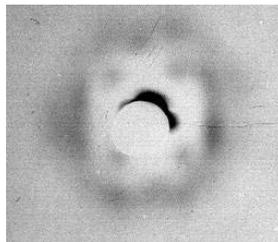
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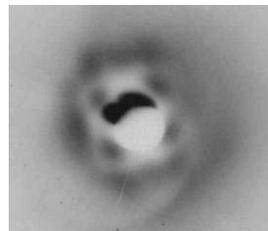
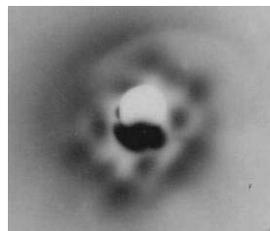
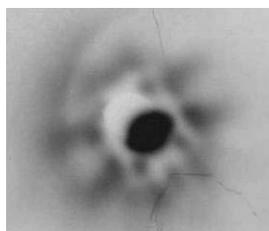
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Discussions on the identification of the quality of nucleated cultured pearls are generally related to the surface, thickness of the nacre-layer and treatments. This refers to the idea that the nuclei used for cultured pearls are made of mother-of-pearl obtained from freshwater molluscs of the Mississippi Basin (USA). Many innovations have taken place in recent years in the cultured pearl industry. In the case of nucleated cultured pearls, one of the new aspects is the use of other materials for the nuclei. This happens commonly without the correct declaration, which is required and necessary, not only because nuclei of different materials give to the product different durability and characteristics, but also because the production of those cultured pearls have different costs.

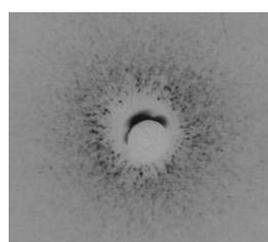
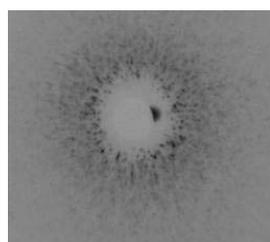
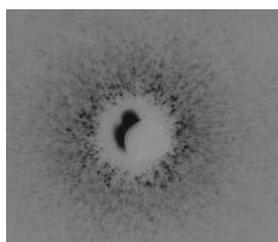
This issue involves unfair competition among Traders, lack of transparency to Consumers and matters related to the protection of certain species, as parts of animals protected by CITES are used as well. Hence the interest in finding a non-destructive method to identify the nature of the nuclei. The use of the old Laue diffraction (single crystal) method is useful and convenient because of the presence in laboratories of X-ray generators, used also for radiographs and luminescence (not exclusively on pearls). Different types of recurrent Laue-grams, distinctive for some materials used for the nuclei, have been collected. These results are useful, but not necessarily sufficient to represent a complete and final solution of the issue, even for the news that turn up daily on the gem market. Examples of the results, in orthogonal directions, will be presented.



3 Laue on a
“classical” nucleus
(layered mother-of-
pearl)



3 Laue on a crossed
lamellar shell nucleus
(Tridacna type)



3 Laue on a
“Bironite” nucleus

Regarding corals, a common and widespread issue is to determine the origin of colour (natural or due, in whole or in part, to treatment). In addition, in some cases it could be requested (or needed) to establish the type of the coral, not only related to market issues, but to possible protected species as well. This is not always easy, given that the materials to be analysed are cut, polished and sometimes treated. Of course, the careful observation of the sample is always the first thing to do (and also the last, final one), together with the detection of standard data, but the results obtained with the microprobe laser Raman at CISGEM gave good guidelines on this front and are used since then for daily analysis. This method, besides determining whether the calcium carbonate is present as calcite or aragonite, allows to detect the presence, type and consistency of biogenic pigments. A survey of results obtained on some Corals and other materials will be presented.

Genus and species - Material	Frequency range of spectral lines (Raman Shift cm-1)		
	C=C	CH3	C-C
Corallium rubrum	1517-1520	1020-1023	1129-1131
Corallium japonicum			
Corallium elatius			
Melithaea ochracea			
“Stylaster”	1522	1025	1132
“Allopora” (pink)	1515-1521	1010	1157
“Allopora” (purple)			
“Allopora” (orange)	1527	1006	1159
Tubipora musica	1510	1014	1124
<i>Heliopora coerulea</i>	1600 band	1467	1263
Beta carotene	1517	1009	1156
Carrot	1519	1007	1156

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Yellow sapphires: XAS determination of the iron oxidation state and structural environment.

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The Fe oxidation state and structural environment in two yellow sapphires has been investigated by Extended X-ray Absorption Fine Structure Spectroscopy (EXAFS) and X-ray Absorption Near Edge Structure (XANES) at the Fe K-edge. Preliminary SEM study of two yellow sapphires (one faceted, and one cabochon) allowed to ascertain that, within error, Fe is the only transition element present in the two studied gems.

XAS measurements have been performed at the GILDA beamline (ESRF, Grenoble, F) at the Fe K-edge by using a Si (311) monochromator which allowed to collect spectra with an energy resolution close to 0.2 eV. Fluorescence signal has been detected by means of a high purity Ge detector.

Pre-edge peak analysis allowed to determine the $\text{Fe}^{3+}/(\text{Fe}^{3+}+\text{Fe}^{2+})$ ratio which, within an error of 0.05, is close to unity. Detection of a component at the high energy side of the pre-edge peak is interpreted as an iron-iron excitation (Glatzel et al., 2008) giving rise to the possibility that minor Fe clustering (i.e. two Fe octahedral sharing a face) exist in these samples.

EXAFS data analysis allowed to determine a $\langle\text{Fe-O}\rangle=1.95 \pm 0.01 \text{ \AA}$. This distance, despite clearly higher than the average M-O distance in pure corundum ($\langle\text{Al-O}\rangle=1.912 \text{ \AA}$), is still much smaller than the $\langle\text{Fe-O}\rangle$ distance in pure Fe_2O_3 ($\langle\text{Fe-O}\rangle=2.031 \text{ \AA}$). The structural control on the Fe-O distances and Cr-O distances gives hints to explain the solubility of these elements in the corundum lattice.

Despite the excellent fit of the EXAFS data, the Fe-M signal across the shared octahedral face is too small to allow detection, by EXAFS analysis, of Fe clustering in these samples.

A review of treatments, synthetics, and other important gemological developments in the last 10 years

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It is important to state from the outset in this abstract that general reviews, such as this broad-based overview of treatments and synthetics over the previous decade can serve only as guides to the available gemological literature on those subjects. Anyone seeking in-depth information regarding specific treatments or synthetics and their gemological identification should follow up by seeking out more detailed references in order to gain the knowledge required to recognize and correctly identify the various treated and synthetic materials of concern.

Over the past decade treatment methods utilized on commercially important natural gems and also on some synthetics, as well as ongoing developments and improvements in the synthesis of a variety of gem materials have caused a certain amount of turmoil within the international gem community. Various treatment processes such as beryllium infusion of corundum, HPHT treatment of diamonds, and solder-glass filling of cracks in gems were by far the big news items in the previous decade pushing synthetics from the front page and also from the memories of many gemologists and jewelers. Advances in gemstone treatment, from more sophisticated coatings, to high temperature and pressure alterations and elemental diffusion, all the way to glass in-fillings, were once again the main topics of discussion in gemological circles. The past decade focused on the development of these new treatment processes and the detection of enhanced natural gem materials, rather than on laboratory grown synthetics. In spite of the gem trade's focus on gemstone treatment processes and their detection, a few new synthetics were developed, and their commercial introduction had its own impact.

Where synthetic colored stones were concerned, the past decade brought a constant trickle of previously known synthetics through the worldwide network of gem identification laboratories and into the gem trade. This was generally not "new" technology impacting the trade however, but merely the expected flow of previously manufactured synthetic products, and in some instances modifications of those materials.

Anything that affects the gem diamond industry however, will always have an effect on the entire gemological community and the jewelry industry as a whole. Such is the case with the development and wide-spread publicity surrounding the introduction of faceted single-crystal gem-quality CVD synthetic diamonds, particularly those which are essentially colorless. These joined with the already existing HPHT metal-flux synthetic diamonds to create a great deal of debate within a worried trade over how to identify and deal with these diamonds as compared to natural gems.

In reviewing the volume of published technical gemological information and research, as well as the continuous attention in the trade press and in certain popular mainstream magazines, the development of gem-quality synthetic diamonds as a

commercially viable product for the jewelry industry was most certainly the primary focus on new synthetics. And now with the addition of transparent gem-quality synthetic nano polycrystalline diamonds it seems that this focus will continue as NPD develops.

By comparison, news pertaining to advancements regarding the synthesis of colored stones such as beryl, chrysoberyl, corundum, and opal was virtually non-existent. In the past decade only a few interesting and noteworthy synthetics such as Malossi hydrothermal synthetic emeralds (and other colors of beryl), and “MexiFire” iron-colored and copper-colored “Peruvian” blue synthetic opals were produced and introduced into the marketplace.

Even though synthetics have been somewhat downplayed over the past decade as compared to the growing concern over gem treatments, for good reason synthetic gem materials such as synthetic rubies, sapphires, and alexandrites should be of considerable concern to the gem and jewelry industry. In one form or another, true synthetics have been with us for over a century now and as technology, being what it is, advances new and interesting products of this genre with continue to be produced. Gemologists must therefore be competent in the identification of all synthetics regardless of when they were produced. In this regard the recognition of these materials by gemologists continues to remain important. So it doesn't really matter if a synthetic gem was grown in the 2012 or more than 100 years ago in the 1880s...when the first true synthetics, “Geneva Rubies” were sold as natural rubies within the gem and jewelry trade...gemologists must still be prepared to deal with any and all of these synthetics.

There is no question that the development of synthetics and treatment processes and the detection methodology that results from their development is of great importance to the international gem community as a whole, and this will not change. Any and all major commercial gems have synthetic counterparts, and those synthetics continue to appear in the trade, sometimes being represented as natural gemstones. Hundreds of thousands of carats of true synthetics, if not millions of carats, have been produced by many different manufacturers throughout the years using several different processes, chief among those being melt-growth, flux-growth, and hydrothermal growth. There will always be unusual materials synthesized like apatite and topaz with what appears to be no real commercial potential. Such stones will present their own unique brand of gemological challenges. However, advancements in the synthesis and treatment of gems will always be focused on the most commercially important gems, such as diamonds, emeralds, rubies, and sapphires.

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AVASPEC 2048: AN INNOVATIVE SPECTROSCOPIC METHODOLOGY TO DICRIMINATE THE QUALITY GEM MINERALS.

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The AvaSpec 2048 is a spectrometer that allows to obtain the electromagnetic spectra in the λ range between 400 and 1000 nm, i.e. in the wave band of the visible and close to the infrared. The spectrometer is equipped with an Avasoft software that convert the AvaSpec signals into absorption spectra. The absorption mode (or transmission mode) wavenumbers can be read by means of a USB port, reproduced on a spreadsheet and processed.

At lower wavenumber the spectra are characterized by a strong background noise (from 350 to 430 nm) produced by the weak light source power. To solve this problem, the spectra were reproduced and cleaned of the noise, with the statistical software IGOR Pro.

The analyses have been carried out on a large number of green minerals that also represent the most important natural and synthetic gemstones present today on the market: emeralds, tourmalines, garnets, opals, quartz, corundum.

The aim of this work is to provide an original database for the gem-quality green minerals.

All the samples have been previously investigated to determine the common gemmological properties (size, gravity measurement, colour, luster, cut, fluorescence) and subsequently analyzed through the AvaSpec 2048 equipment.

We collected spectra of gemstones characterised by the same chromophore chemical elements in different legand field [2]: for example Cr, Fe, V responsible of the different shades of colour.

We also collected several spectra on minerals like beryl aquamarine and zoisite tanzanite [1], that are precious gemstones frequently submitted to thermal treatment aimed to the improvement of the colour. These ameliorative treatments are very commonly carried out on the gems before the marketing. The spectra obtained on the minerals before and after the thermal treatment show several and relevant differences, and therefore represent a useful and non destructive method to recognize the colour change that take place through the heating.

We also investigated some synthetic Beryl Emeralds (AGEE, Biron) and artificial material: it resulted that the spectra are very different and easily recognizable from the natural ones.

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LAPIS LAZULI: IDENTIFICATION OF PROVENANCE MARKERS BY MEANS OF CONVENTIONAL ANALYSIS TECHNIQUES AND PROTON MICROSCOPY

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Lapis lazuli is a blue semi-precious stone that can be found in few places in the world, due to the low probability of geological conditions in which it can form. Because of its peculiar color and for its rareness lapis lazuli have been used for more than 7000 years for the manufacturing of precious objects and jewels (beads, gems, seals, small decorative artworks, etc). The possibility to associate the raw material to man-made objects is plausible and for these reasons a provenance study of lapis lazuli could provide answers to some important issues; for example the use and the dissemination of this rock through historic commercial routes, answering some open-questions and clarifying some obscure points especially for ancient time when written testimonies are scanty or absent at all.

The main quarries for this stone are in Sar-e-Sang, Badakhshan (Afghanistan), although other known quarries could have been exploited since antiquity: Pamir Mountains (Tajikistan), Chagai Hills (Pakistan) and Lake Baikal (Siberia) in Asia and Ovalle (Chile) for the American continent. Moreover, archeologists debated on the presence of other small ancient local quarries in Sinai and in Iran, but at now they are not geologically confirmed [1].

Lapis lazuli is generically classified as a metamorphic rock, even if this definition could not be considered exhaustive, due to the complexity of mechanism involved in its genesis. It is characterized by the presence of the mineral lazurite (giving the blue color to the rock),

combined with other types of minerals whose presence and relative amount varies from and within quarries; among these: calcite, wollastonite, phlogopite, feldspars, diopside, pyrite and others feldspathoids of the same lazurite family such as hauyne, sodalite and nosean [2]. For example, in Fig. 1 are shown optical and cathodoluminescence images of a sample from Pamir where it is evident the heterogeneity of lapis lazuli.

Ongoing research is divided into two phases partially advancing in parallel: the characterization of rocks from certain provenances to identify markers that distinguish among the various quarries of

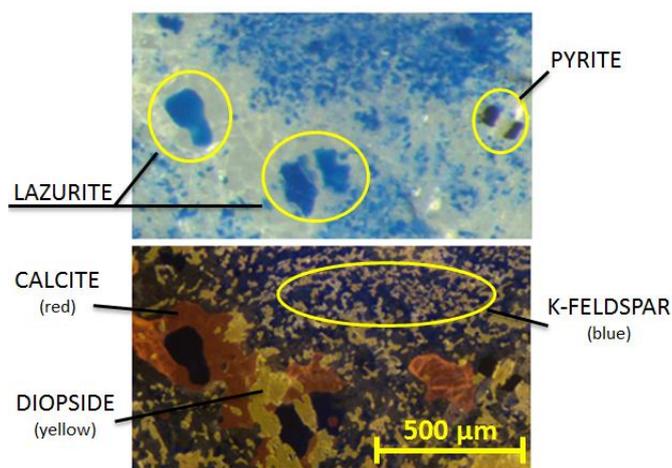


Fig. 1: Optical Microscope (OM) in visible light and cathodoluminescence (CL) images of a sample from Pamir.

lapis lazuli and the analysis of artworks made of lapis lazuli looking for these markers. We performed a systematic characterization of rocks of certain provenance through a multi-technique approach [1,3,4]. Since the crystals dimensions can vary from fraction of a micron to hundreds of microns we decided to use mainly microscopic techniques allowing us to analyze the single crystals. In particular we looked for the presence or the absence of a phase, or one or more elements present in a peculiar mineral, or the different luminescence of the same mineral.

At now, the rocks analyzed in this work (37 samples) came from four different sources: Afghanistan (19 samples), Tajikistan (4 samples), Siberia (5 samples) and Chile (9 samples). To speed up the characterization and perform also in-vacuum analysis we prepared semi-thin sections, but we demonstrated that the measurements able to distinguish the provenance can be carried out in air and on rocks or artworks with different shapes. In fact, the ultimate goal is the analysis of archaeological or artistic objects using only completely non-invasive techniques. An initial characterization of the rock samples permitted to identify the distribution of the main mineral phases present in the rocks, their composition and their luminescence spectra. These measurements have been carried out by means of optical microscope, cold-cathodoluminescence (cold-CL) and Scanning Electron Microscopy (SEM-EDX and SEM-CL). Further investigations have been carried out using proton microscopy, mainly micro-PIXE (Proton Induced X-ray Emission) and micro-IL (IonoLuminescence), that can be performed also in air and without sample preparation, allowing to analyze artworks and whole stones without cutting. Other in air microscopy techniques used are micro-XRF (X-Ray fluorescence) and micro-Raman. It should be noted that all of the markers found are detectable by proton microscopy which makes this technique suitable to the aims pursued.

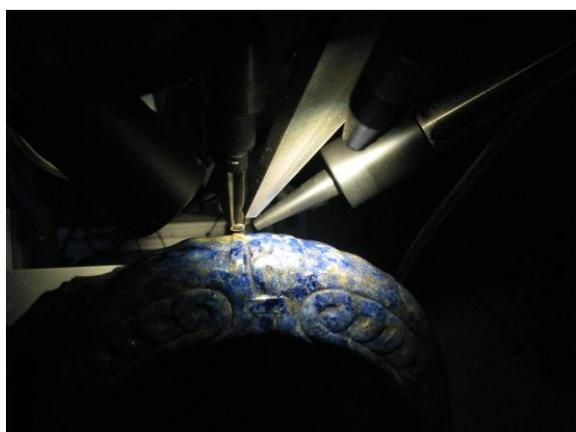


Fig. 2: Proton microscopy of an object belonging to the "Collezione Medicea di pietre lavorate".

Chilean rocks seem to be the easier to be distinguished from others rocks. As reported in previous studies [for example 2 and 5], we confirmed the high contents of wollastonite in all the analyzed Chilean samples (absent in all other analyzed samples). About the three Asian provenances we studied the composition analysis of pyrite seems to be the most suitable to discriminate samples from Tajikistan due to the contemporary presence of copper (up to 400 ppm) and absence of nickel (below 250 ppm). Moreover, in all samples from this provenance we also observed a 700 nm luminescence emission band in diopside not observed in all the Afghan samples and a high quantity of a peculiar cancrinite mineral. In many Afghan samples diopside contains a high quantity of Ti (up to 5000 ppm), V (up to 1000 ppm) and Cr (up to 1000 ppm) not observed in the other Asian samples while Siberian samples can have a high Sr contents (up to 1000 ppm). At last, the Siberian samples seem to have a high quantity of barium distributed in the rock. It should be noted that due to the low number of analyzed samples, all the results on Siberian and Tajikistan samples must be confirmed by means of a larger statistics.

At now, other than increasing statistic analyzing new rocks from certain provenances, we started to analyze artworks. Some preliminary measurements have been carried out on precious objects belonging to the "Collezione Medicea di pietre lavorate" of the Museum of Natural History of Firenze (Fig. 2). These measurements demonstrate the applicability of the method, allowing to suggest the origin of the raw material used for precious objects or archaeological findings in an absolutely non-invasive way.

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NEW GEMOLOGICAL FINDINGS AND OTHER BRAZILIAN CURIOSITIES

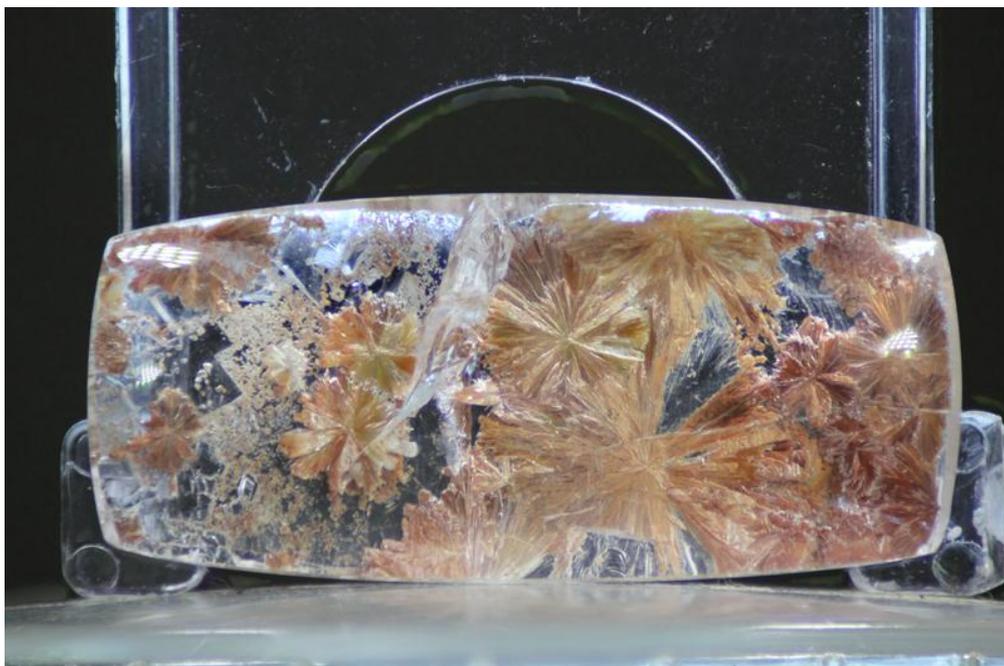
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While the possibility of new, mineralogical interesting finds is somewhat limited, every year gemologically discoveries are encountered. One of the most important source of gems is Brazil, a country well-known especially for the huge variety and the production of precious and semi-precious colored gemstones. Due to intense mining and field research activity, many new gemstones have been extracted in Brazil in recent years. "Medusa" quartz (or "Paraiba" quartz), "Sunset" quartz, "Pink Fire" quartz, topaz with fluorite, cat's eye emerald, quartz with celsian and ankaingite, "Churrasco" quartz [1, 2, 3, 4, 5] and "Flower" quartz (Figure 1), new gemstones that appeared on the Brazilian market in the last five years, are described in details. The increased relevance of semi-precious colored gemstones is due to the vast success obtained among gem buyers and jewellers. Just as for other precious gemstones an elevated marketability often results in increased falsification rate (e.g. synthetic stones, imitations, doublets, laboratory treatments), so, also Brazil, one of the most important semi-precious gemstones producer, is now prone to a number of potential falsifications. Some successful recent gem falsifications (rutilated quartz doublets, a new imitation of imperial topaz, quartz with fake inclusions, "Safirita" blue-quartz [6, 7, 8, 9] and "Ametista opalizada") are presented in this work.

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STUDIO DI DIAMANTI DOPO TRATTAMENTO IN BAGNO DI RODIO

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In the present work, four brilliant-cut diamonds (diameter 1.35 and 1.65 mm) have been studied because after a rhodium treatment of the jewel in which the gems were mounted, these diamonds, originally uncolored, displayed a gray color, lowering significantly their commercial value. It must be specified that rhodium treatment is normally used in jewelry to give a shining appearance to the alloys of white gold and that other diamond gems, treated with the same rhodium bath, did not exhibit change in color or brilliance. Several washing attempts (ultrasonic soap solution, degreasing in sulfuric acid and rinsing with deionized water, finally nitric acid) could not restore the original appearance and value of the diamonds. The fact that after the same treatment other gems did not exhibit the same effects proves that the phenomenon cannot be ascribed to a rhodium solution contamination [1].

We studied therefore the gems by different and complementary analytical techniques, such as optical gemology microscopy, micro-Raman Spectroscopy and Environmental Scanning Electron Microscopy with Energy Dispersion Spectrometer (ESEM/EDS). All these techniques allow studying the samples without processing; therefore the diamonds could be analyzed without any manipulation.

Preliminary data revealed significant differences in rhodium grain distribution covering the surface of the gems on the different samples and presence of Raman bands not ascribable to diamond on the recorded spectra. Surface irregularities of the gems, caused by small amounts of grease, abrasive polishing powders, burns may be suggested as adhesion sites for rhodium particles.

Further studies are foreseen to study deeply the cause of the observed phenomenon and to investigate why rhodium is detected only on a few samples; by this way useful information for the prevention of this regrettable and costly problem may be attained.

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IVORY, IVORIES AND IVORY-LIKE MATERIALS

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Ivory has always been considered one of the most attractive and valuable ornamental materials. It is a product of the Animal Kingdom and its gemmological classification is therefore based on genetical criteria, as it is considered a gemmological material from “parts of animals”. Ivory in fact comes from the tusks, or prominent teeth, of some mammalian species (both actual and extinct), which practically are the source of all the ivory on the trade. They are: African and Asian elephant, mammoth, hippopotamus, warthog, sus babiroussa, wild boar, tiger, black bear, cave bear, American elk, Siberian husky, walrus, narwhal, sperm whale, dugong.

From a compositional standpoint ivory is dentine, an organic, calcified and extremely elastic tissue that forms the teeth of all mammalian species. It is made up by an organic matrix and by an inorganic phase. The organic component is principally a protein: the type I collagen. The inorganic phase is constituted by nanocrystals of a biological (non-stoichiometric) carbonated hydroxyapatite.

The structural features of ivory vary from species to species. The density also varies, as it increases with decreasing protein content. In contrast, the refractive index of ivory, irrespective of source, ranges in the same average values. To genetically identify different types of ivory requires the examination of the surface texture in all possible directions. A pattern of rhomb shaped lozenges, best known as “engine-turned” texture, even if characterized by different features, identifies elephant (Fig. 1) and mammoth (Fig. 2) ivory. As no “engine-turned” pattern can be observed in all the other kinds of ivory, such texture is of fundamental importance for the identification.



Fig. 1 – Elephant ivory

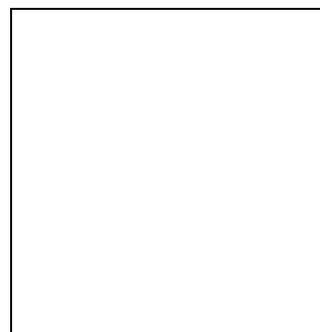


Fig. 2 – Mammoth ivory

Several different ivory-like materials can be found on the trade. They can be of natural origin or of artificial origin. Among the first ones, bone and the so-called “vegetable ivory” may be mentioned, whereas different kind of plastics belong to the last ones. Apart bone, which from a compositional and structural standpoint can be look-alike ivory, all other ivory-like materials can be identified by typical structure and physical properties.

Owing to its complex composition, due to the biological origin, and because of several imitations on the trade, a successful identification of ivory includes more steps, starting from a gemmological standard examination up to supplemental advanced techniques, such as spectrophotometry, nuclear magnetic resonance and biochemical analyses.

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Geology and gemology of the Ethiopian opals

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Since 2008, the Wegel Tena area in the Wollo Province, Ethiopia, is becoming a major producer of play-of-color opal [1, 2]. A recent investigation of this deposit proved that opal formation is mainly due to pedogenetic weathering of a volcano-sedimentary layer [3, 4]: the host rock is composed mainly of smectites and remaining volcanic glass and feldspars, rare quartz plus accessory minerals. Abundant pedogenetic features are observed: mud platelets, concentric cracks around clay gravels, and plant fossils (mostly rootlets) inside opal (Figure 1). The opal-bearing layer is covered, about 3 meters above, by a conglomerate level that probably marks the surface level at the time of opal formation (Figure 2).

The geochemistry of opal shows two clear sources of silica for their formation: those rich in uranium (U) and calcium (Ca), and poor in strontium (Sr) and barium (Ba) are typical of volcanic glass weathering into smectites, when those poor in U and Ca, and rich in Sr and Ba are typical of feldspar weathering into smectites. The fact that these two distinct signatures are not mixed inside the opal indicates that underground waters did not move at all during opal formation in this soil.

Some other opals worldwide show similar features and host-rock, such as those from Java, Indonesia [5], and from Virgin Valley, Nevada, USA [6]. A detailed investigation of these deposits may provide tracks on a more general issue: in which specific context does the weathering of volcanics produce gem opal, and what is necessary for its preservation?



Figure 1: plant rootlets fossils are locally abundant in opals from Wollo and prove their grew together. Picture width: 1.5 cm. Photo by F. Mazzero.



Figure 2 : The horizontal opal-bearing level occurs in weathered volcanics few meters below the paleosurface. Photo by B. Rondeau.

Wollo opals often show optical features that are otherwise very rare in other deposits. Some show spectacular digits (rounded columns of play-of-color embedded in common opals; Figure 3); these are due to a succession of growth, dissolution and re-precipitation processes [7]. Some others show diffraction patches displaying the whole rainbow (Figure 4), instead of a vivid patch of a unique wavelength as classically observed; from now, we found no satisfying optical explanation for this behavior.



Figure 3: Many opals from Wollo show “digits”, rounded columns embedded in a matrix of common opal. Picture width: 2 cm. Photo by F. Mazzero.

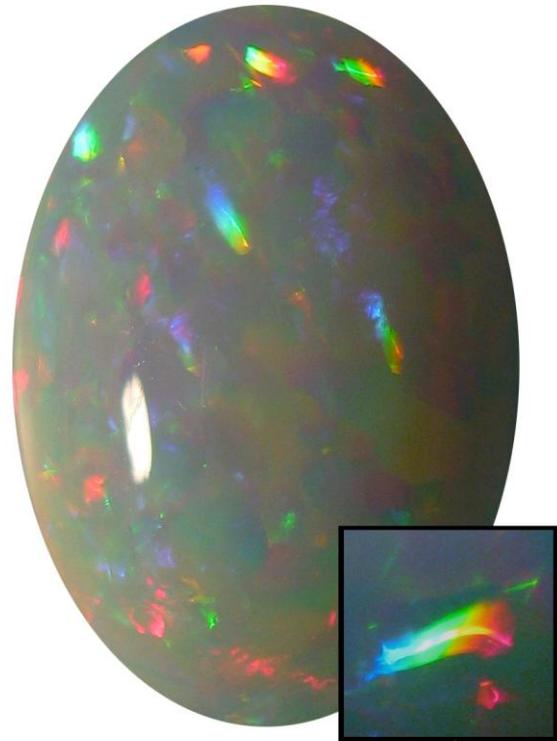


Figure 2 : Some Wollo opals display unusual patches of diffraction covering the whole rainbow of visible light. Picture width: 2 cm. Inset: 2 mm. Photo by F. Mazzero.

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TREATMENTS OF CORUNDUMS

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Untreated rubies and sapphires have become so increasingly rare, they are no longer affordable to the average consumer. The demand for precious fine gems has increased, therefore, our industry is forced to accept many treatments as standard in order to meet the demand for jewelry grade gemstones. This has not come to pass without bitter disputes, but in the end, many treatments are currently regarded as expected and tolerated. Until a few years ago one of the main concerns of the gemologist was simply to positively identify a gemstone and then determine whether or not it was treated. Today, a gemologist must be able to positively identify the nature and extent of treatment a gemstone has routinely undergone. This is especially true regarding corundum, as it is estimated that greater than 95% of the material available for the consumer has been subjected to some kind of “improvement process”. It is clear, that in order to properly value a ruby or sapphire it is essential to be able to determine the extent of modification the gem has undergone to achieve its current appearance. Therefore, the question of value is a function of the “rarity” and appearance of the rough corundum prior to any form of modification or treatment. The relentless race in recent years, for producing valuable appearing gems at low cost has resulted in treatments which can transform the lowest quality rough corundum, previously only suitable for use as industrial abrasives, into affordable “gems”. Enhancement by impregnation with lead glass is one of these treatments which pose serious problems for our industry. The final product is essentially unsuitable for use in jewelry because the glass filling is unstable with normal wear. Exposure to acidic substances as common as lemon juice and vinegar can produce irreparable damage. With the express intention of providing better protection to consumers through mandatory disclosure and identification of treatments, a bill has been approved by the House of Representatives¹ and is currently awaiting final approval in the Senate². Disclosure is one of the keywords of the bill. It would mandate that correct and full descriptions of treatment and modification a gemstone has undergone be fully and accurately disclosed at the time of sale³. Full disclosure would be obligatory.



Lead glass filled ruby, before (top) and after (bottom) immersion in pickling solution. (Photo Scarani)

-In our discussion, enhancement methods of corundum will be discussed together with available techniques for proper identification.

-We will also address correct nomenclature to describe these treatments as developed by LMHC⁴ to be officially established as law:

[1] Chamber of Deputies, Act No. 225-2274-A, XVI Legislature, [2] Senate, Senate document no. 3048, XVI Legislature; [3] LMHC information sheets # 1, 2, 3, 7 – <http://www.lmhc-gemology.org>

1 November 30, 2011 (Unified Mazzocchi, 225 and Mattesini, 2274 [1]

2 s.3048 [2]

3 Chapter I, definitions, Art. 1 to 7

4 Laboratory Manual Harmonization Committee) [3]

MINERALOGICAL AND GEMMOLOGICAL PROPERTIES OF ALEXANDRITES FROM URALIAN EMERALD MINES AND SELECTED OTHER SOURCES

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Alexandrite, a variety of chrysoberyl, and one of the finest colour-change natural gemstones, may almost be called “the national gemstone of Russia”. This great prestige is based on two facts: its noble name given in 1842 in honour of the Tsarevich Alexander Nikolaevich (the future Tsar Alexander II) and its dazzling colours, green in daylight and red in incandescent light, the military colours of Imperial Russia. Although quantities of facetable quality alexandrite are considerably less than those of emerald, alexandrite is counted among and compared to the “big four” of the gem business: diamond – ruby – sapphire – emerald.

In several natural emerald-alexandrite deposits, alexandrites are found in phlogopite lenses or massive phlogopite bodies at or close to the contact between pegmatitic intrusions and mafic to ultramafic metamorphic rocks. This is the typical contact metamorphic-metasomatic paragenetic association of alexandrite best known from the “classical” deposits of the Ural Mountains in Russia. Similar alexandrite occurrences are the Novello deposit, Masvingo District, Zimbabwe, and the Mayoka deposit, Lake Manyara mining area, Tanzania. Although details of the emerald-alexandrite formation within these schist-type deposits are still under discussion, a multistage metamorphic genesis in so-called ‘blackwall’ contact zones is generally accepted for this type of deposit.

A comparison of alexandrites originating from such massive phlogopite host rocks indicates that the samples from Novello are most similar to some of the Russian material and that an equivalent to the Lake Manyara material is also found in Russia. However, it must be emphasised that the Russian samples originate from numerous deposits exploited by surface and underground mining over a considerably larger area than the Lake Manyara and Novello sources. Consequently, the Russian samples show a larger variability of characteristic mineralogical and gemmological features.

Single crystals, contact twins, and cyclic twins (trillings) are present in all three areas, penetration twins are mainly found in the Lake Manyara deposit. The most impressive rough crystals from all three mining areas are cyclic twins (trillings), which show a habit variation from dipyramidal to equidimensional and columnar. The growth sequence of alexandrite trillings is given as a schematic overview in Figure 1. The first stage of crystal growth (A) is characterized by a simple dipyramidal habit with the dipyramid **o** and the pinacoid **a** as dominant crystal forms. In the last step of crystal growth (H), the columnar trillings are characterized by the prism **i** and the pinacoid **a**. Intermediate steps (B to G) are described as a step by step increase in size of **i** and a corresponding decrease in size of **o**. During this sequence, the dipyramid **w** also grows but only temporarily (E). The general crystal morphology of twinned and untwinned chrysoberyls from all three deposits is reflected in their internal growth patterns as demonstrated in Figure 1 by some examples. Alexandrites from other sources and similar metamorphic genesis, e.g. from Brazil or Madagascar, fit within this general scheme.

Inclusions or platelets of phlogopite mica are present in the chrysoberyls from all three areas. Other mineral inclusions which are common in material from Lake Manyara are apatite and zircon; channels parallel to the *a*-axis are also common.

References

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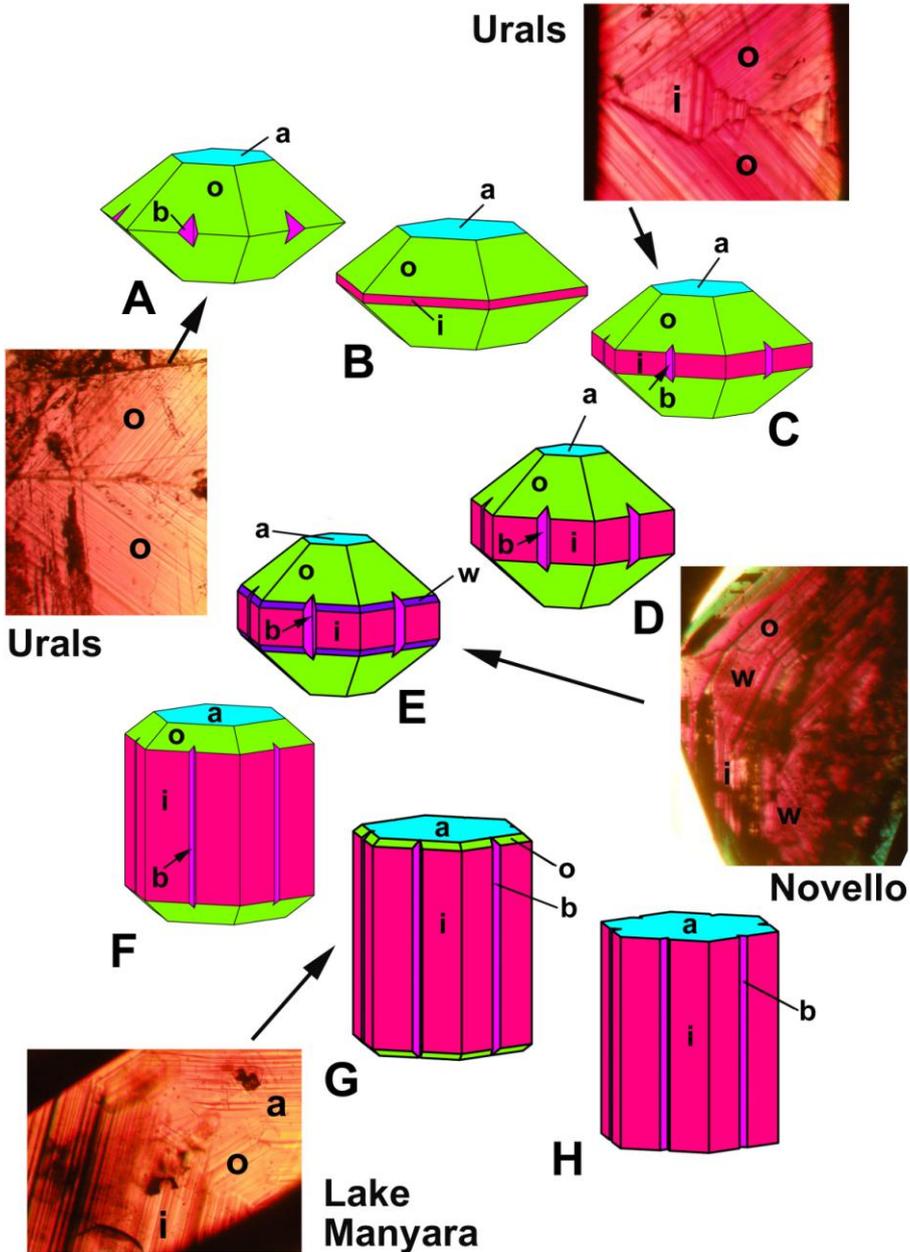


Figure 1: Growth sequence of alexandrite and chrysoberyl trillings with dipyramidal to columnar habit originating from phlogopite schist-related deposits. Examples of the complete growth sequence from A to H can be obtained from stones mined from the Uralian emerald-alexandrite belt, Russia, crystals A to C represent typical habits which were unearthed in the nineteenth century, samples D and E represent crystals mined in the twentieth century in the famous Malysheva Mine, and habits F to H represent mostly smaller crystals from the more recent production from Malysheva. Crystals D and E are typical examples of alexandrite trillings from the Novello deposit, Zimbabwe. Also E represents a morphology developed at the beginning of crystal growth in the Lake Manyara deposit, Tanzania, but not seen in well developed crystals from this deposit. Crystals F to H represent later stages of Lake Manyara material and these habits are common in trillings from this locality. Examples of characteristic internal growth patterns corresponding to the external morphologies are also shown.