

Gem and mineral identification using GL Gem Raman and comparison with other portable instruments

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Abstract Several mainly silicate minerals in their gemstone varieties have been analysed by the Gem Raman portable system by Gemlab R&T, Vancouver, Canada, in order to ascertain the general performance of this relatively non-expensive tool developed exactly for the purpose of gemstone identification. The Raman spectra of gemstones acquired by this system have been subsequently critically compared with the data obtained by several other portable or handheld Raman instruments. The Raman spectra acquired with the Gem Raman instrument were typically of lesser quality when compared with the spectra taken by other instruments. Characteristic features such as steep baseline probably due to the fluorescence of the minerals, Raman bands much broader and therefore less resolved closely located Raman bands, and generally greater shifts of the band positions from the reference values were encountered. Some gemstone groups such as rubies did not provide useful Raman spectra at all. Nevertheless, general identification of gemstones was possible for a selection of gemstones.

1 Introduction

Precious stones belong to a few mineral groups in the mineralogical system which are highly valued and sought after and occur more or less rarely in rocks or secondary deposits. Their identification is traditionally based on

 \boxtimes Adam Culka culka@natur.cuni.cz observation of their physical properties. Common gemmological techniques are mainly optical, and the instruments used include: the refractometer (for determination of the refractive index), polariscope (optical characteristics), dichroscope (pleochroism), spectroscope (visible spectrum), and microscope (characteristic inclusions). Proper use of such instruments, along with the correct interpretation of the results, usually enables one to determine the entire range of gems, but sometimes there are cases where the results are ambiguous.

In the last few decades, Raman spectroscopy has been confirmed as an excellent tool for identification and discrimination of minerals. The classical advantages of Raman microspectrometry are of importance also in gemmology and cultural heritage areas: a possibility to obtain spectra non-destructively, a potential of discriminating very small parts of artefacts. Today, Raman spectra are frequently recorded and interpreted at the same time that new minerals are described and accepted by the International Mineralogical Association (IMA). With this approach, further use of this technique for discrimination and identification purposes is facilitated and welcomed by mineralogists s.l. Raman spectroscopy is also widely used for a non-destructive unambiguous identification of pigments, biomaterials, binders, ceramic and glass in the areas of the arts and cultural heritage $[1-3]$, and reviewed $[4, 5]$ $[4, 5]$ $[4, 5]$ $[4, 5]$.

During the last decade, increasingly smaller, lightweight Raman spectrometers have appeared on the market and are increasingly used in such diverse areas as forensic science, geoscience, art, and archaeology. An ease of acquisition of analytical data, a non-destructive mode of obtaining Raman spectra and a portability and enhanced mobility of the instrumentation are welcomed in all these three disciplines and in very diverse situations. This is not the goal of

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this short paper to highlight all these projects and new achievements.

Very light and portable, battery-operated Raman instruments have recently been applied to discriminate minerals from several groups $[6–8]$, some of which are considered as gemstones. Very good results and an excellent fast identification of the gemstones directly mounted in the frame of religious and secular jewels were obtained using small Raman instruments mainly with near-infrared excitation [\[9](#page-8-0)].

An overview of the characteristics and possibilities of portable Raman spectrometers is presented in the survey work of Vandenabeele et al. [\[10](#page-8-0)]. Here, instruments are divided among transportable, mobile, portable, handheld categories, and the newest and smallest that can fit into the palm of one's hand. Among the transportable spectrometers are listed devices that can be carried, but are not designed for frequent movement from place to place, and also are not very light. The term mobile device is used for all the devices which are intended for frequent movement and relocation. Vandenabeele et al. defined portable devices as those that can be easily brought to a new place, for example in a suitcase or a backpack. Handheld Raman spectrometers can be held by the operator in one hand and at the same time can analyse the object to be inspected.

The Gem Raman instrument probably fits into the mobile category, and it is relatively light (10 kg) but is intended to be operating on a bench in a gemmological laboratory and probably not to be moved frequently. Unfortunately, to our best knowledge, no peer-reviewed studies involving this instrument have been published. A few information sources are available, conference abstracts [\[11](#page-8-0), [12](#page-8-0)], and a report about using this instrument for a mineralogical database expansion (Raman and photoluminescence) [\[13](#page-8-0)], but since this instrument is typically used in private gemmological laboratories, the lack of published data is understandable. Therefore, the aim of this study was to test the general performance of the Gem Raman system for a gem and mineral identification. Especially interesting was to ascertain the quality of Raman spectra of gems obtained with this cheap instrument in a direct comparison to the more advanced and rugged multi-purpose handheld Raman instruments produced by well-established manufacturers at a premium price.

2 Experimental

2.1 Portable Raman spectrometers

The main Raman instrument used in this study was Gem Raman (Fig. 1) from GemLab Research and Technology, Vancouver, Canada. It is a portable system

Fig. 1 A detail of the sampling stage of the Gem Raman instrument

intended to be positioned on a bench and powered from the power grid. It has a moderate weight of 10 kg, and it is equipped with a 532-nm laser (this line is a result of frequency doubling of the Nd-YAG 1064-nm laser line). The Raman uses a high-resolution spectrometer with a Toshiba TCD1304AP CCD (3648 pixel, 8 μ m \times 200 μ m). Spectral range is 200–2500 cm⁻¹ with a resolution of 10 cm^{-1} and the laser spot size in the range of 10–15 micrometres. Inside the instrument is a sampling stage $(90 \times 90 \times 50 \text{ mm})$ for sample placement. A photoluminescence analysis is also possible using this instrument in the range of 530–750 nm which can provide additional information about the sample chemistry. This instrument was operated by a skilled gemmologist, but not a Raman spectroscopist, as is the primary purpose of this relatively simple and not very polished instrument. The spectra of minerals have been acquired using the approach where the settings of the instrument for each sample were tried and changed to obtain 'the best results'. However, the usual settings used were as follows: accumulation time 50–200 ms (occasionally 10–1000 ms) and usually 3–5 accumulations. A boxcar parameter (pinhole width) was set to 5–10. The actual power of the laser at the sample was not measured; however, the manufacturer stated that a 300-mW laser was used. The instrument contains an analogue regulator of the output laser power of a kind, with a maximum at 1.7 units, and this was typically set in the interval of 1–1.7 for the mineral analysis. Instrument is operated using a laptop computer, and the spectra can be easily compared to the linked RRUFF mineralogical database for an easier identification. This instrument is available since 2014 and was purchased by a private gemmological laboratory in Prague in 2015.

The handheld Inspector Raman from Delta Nu weighs 1.9 kg. It is equipped with a 785-nm diode laser, with a maximum output power of 120 mW declared by the

manufacturer, and a thermoelectrically cooled CCD detector. This instrument is powered by batteries but can be connected to electrical grid as well. It provides Raman data over the $200-2000$ cm⁻¹ wavenumber range, with a spectral resolution of 8 cm^{-1} . This instrument is remotely controlled via a USB cable-connected laptop. It allows the operator to select from a wide range of settings for measurements, including: more detailed power output selection, the number of accumulations, and the duration of accumulation times. The spectra were typically recorded with this setting: 1–10 s accumulation time, and usually 1–10 accumulation for a relatively quick identification depending on the analysed gemstone. Laser power was set to 'high' for most measurements (a real output laser power was measured at ca. 45 mW using a LaserCheck energy meter); in some cases, the laser power was set to lower settings to avoid detector saturation. This instrument was purchased in 2008.

The Bravo handheld Raman spectrometer from Bruker is a newly available system (end of 2015). This instrument was developed for an easy chemical identification and features two improvements: it uses a combination of two excitation wavelengths and a sequentially shifted excitation by design in order to suppress the fluorescence. It weighs 1.5 kg and is battery-powered. The excitation wavelengths are probably 785- and 1064-nm lasers (obscured by the manufacturer) with output power \100 mW. Spectral range is 300–3200 cm^{-1} at 10–12 cm^{-1} resolution. The instrument is operated using the buttons and a touchscreen. The spectra were typically recorded using the automated settings (less than 1 min for the analysis) usually rather more quickly.

The experimental settings of the different instruments was not the same, and also each instrument uses a different excitation line. The approach was to use each instrument in a best possible way to obtain the good quality of the resulting spectra in a reasonable time for each analysis. Spectra obtained with all instruments were exported and then viewed and processed in GRAMS/AI 9.0 and OPUS 7.7 spectroscopic software. They are presented in this study both as unmodified raw spectra and as baseline-corrected spectra for a detailed comparison.

2.2 Samples of gemstones

A selection of different gemstones from several groups of the mineralogical system has been analysed using the Gem Raman instrument. Of the gemstones that provided spectra with identifiable Raman bands, examples are summarized in Table [1](#page-3-0), and the positions of the Raman bands of these gemstones are given for each instrument.

3 Results and discussion

The analysed gemstones or minerals belong mainly to the silicates group of mineralogical system with some representatives of rather interesting and complex silicates such as for instance iolite and scapolite. Large selection of beryls has been analysed both in beryl variety and in emerald variety. Other types of gemstones that have been analysed were spinels (spinel, chrysoberyl) or aluminium oxide minerals (ruby). Single stones from common groups of minerals less frequently used in gemmology have been also tested (celestine). The Raman data are presented here in the form of a table where the basic spectroscopic parameters such as the number and positions of the detected Raman bands for each mineral are given. Strongest or signature bands are given in bold. The wavenumber positions are then compared with the reference values.

3.1 Ruby

The Gem Raman instrument was unable to acquire any usable Raman spectra of rubies, of which a moderate selection has been analysed. The Raman bands were masked by a great amount of other signal probably due to the fluorescence/luminescence or instrument related factors. The other two instruments were able to record goodquality spectra of the rubies analysed in this study. In a different study of our group [\[9](#page-8-0)], it has been established that the small handheld Raman instruments with a 785-nm excitation are feasible for a quick identification of rubies on artefacts.

3.2 Emerald

Minerals and gemstones from the beryl group such as an orange beryl from Tanzania and several emeralds of different hues of green from several localities have been analysed. The Gem Raman instrument acquired goodquality Raman spectra of colourless and orange beryls (Fig. [2\)](#page-4-0). Emeralds from localities such as Ural, Australia and Brazil (Goias) provided spectra of various qualities with a moderately sloped baseline probably due to the presence of Cr^{3+} Cr^{3+} Cr^{3+} (Fig. 3). Nevertheless, usually the two signature Raman bands were identified in their spectra: bands around 1070 and 680 cm^{-1} , these are attributed to the Si–O symmetric stretching vibration, and the Be–O symmetric ring deformation, respectively [[14\]](#page-8-0). On the same samples, the two other instruments with the 785-nm excitation provided better Raman spectra. However, several emeralds that have been analysed did not provide usable spectra at all (even with the 785-nm instruments), and these were typically the gemstones of darker green Table 1 Raman bands positions of analysed gemstone varieties of minerals

The numbers in bold represent strong Raman bands

FWHM full width at half maximum

hues, with the highest amount of chromium. This is in accordance to our results obtained on another series of emeralds from different sites (data not given here).

3.3 Chrysoberyl

Gem Raman instrument was unable to record a useful spectrum of chrysoberyl, and the two other instruments provided good spectra for chrysoberyl identification. Therefore, synthetic alexandrite, a colourless variety of chrysoberyl, was analysed additionally to have more data for comparison purposes. Four distinct and relatively broad bands were detected in the spectrum of alexandrite (Fig. [4\)](#page-5-0) obtained by the Gem Raman instrument: 510, 632, 774, and 925 cm^{-1} . While the 'signature' is generally correct, there appears to be a shift of about $8-10$ cm⁻¹ towards lower wavelengths when compared to the spectra from other instruments or the reference [\[6](#page-8-0)]. This fact and also the fact that the full width at half maximum (FWHM of a given Raman band) values were highest in this sample could be explained by the existence of other close Raman bands that the Gem Raman was unable to resolve and they contribute to the bands described here.

3.4 Spinel

Spinel ($MgAl₂O₄$) that belongs to the same group of aluminium spinels as chrysoberyl was also analysed by the two portable instruments. The Raman bands are listed in Table 1 and are generally in a good agreement and also with the reference values [[22\]](#page-8-0). The most intense Raman band located at 404 and 408 cm⁻¹ is attributed to the E_g mode. The band located at 514 cm^{-1} in the spectra taken by the Gem Raman instrument is probably and artefact, since it was not found in the other spectra or in the reference.

3.5 Garnet

Another gem that was analysed belongs to the group of garnets. The sample comes from Tanzania and is presumably of pyrope/spessartine composition. The most diagnostic band was shifted from 918 cm^{-1} in the spectra obtained by Inspector Raman instrument to 912 cm^{-1} in the spectra of Gem Raman, and other important garnet bands were shifted from 358 and 860 to 348 and 850 cm^{-1} . Due to the composition of this gem, it was useless to

compare the wavenumber positions with the spectra of end members of the garnet group that are nicely presented and commented in Kolesov and Geiger [[15\]](#page-8-0); nevertheless, the garnet Raman bands were sufficient for the identification.

3.6 Celestine

For mineral celestine, which belongs to the group of sulphates, Gem Raman was able to record several bands (451, 621, 996, 1093, 1154 cm^{-1}) which are generally in relatively good agreement with reference values [\[16](#page-8-0)], with the exception of the main SO_4^{2-} symmetric stretching band

which is shifted from 1003 to 996 cm^{-1} , which is a major shift for the spectra of minerals from the sulphate group. Therefore, unambiguous identification of this sulphate is not possible using the Gem Raman. On the other hand, the sulphate minerals have a rare occurrence among the gem quality stones, and the instrument was able to identify this stone as a sulphate quite easily.

3.7 Iolite

Iolite a gemstone variety of a complex magnesium iron aluminum silicate mineral cordierite provided Raman Fig. 4 Raman spectrum of alexandrite (synt.) as acquired with Gem Raman (top) and with Bravo (bottom)

Fig. 5 Raman spectrum of iolite as acquired with Gem Raman (top) and with Inspector Raman (bottom)

spectra completely without the usual high background (see Fig. 5). Also the wavenumbers positions of the signature Raman bands from spectrum obtained using Gem Raman were in a quite good agreement with the reference (about 3 cm^{-1} difference) [[17\]](#page-8-0). The limitation of the instrument became clear, for example, in the case of the strong band and a shoulder located at 575 and 555 cm^{-1} , respectively, in the spectrum taken by Inspector Raman instrument, while only one band can be observed (564 cm^{-1}) in the spectrum taken with the Gem Raman. Additionally, the bands due to the $CO₂$ vibrations in channel cavities in iolite [[18\]](#page-8-0) were also observed located at 1184 and 1390 cm^{-1} .

3.8 Scapolite

Raman shift (cm-1)

Gem scapolite is a solid-solution series consisting of two main end members, marialite $(Na_4[A]_3Si_9O_{24}]Cl$ and meionite $(Ca_4[A_6Si_6O_{24}]CO_3)$, and a third end member, silvialite $(Ca_4[Al_6Si_6O_{24}]SO_4)$ [\[17](#page-8-0)]. Raman bands located at \sim 1095 and \sim 990 cm⁻¹ in the spectra of both the Gem Raman and Bravo instruments (see Fig. [6\)](#page-6-0) were attributed to the symmetrical stretching vibration of carbonate and sulphate groups, respectively. Other important bands located at around 455 and 535 cm⁻¹ are probably due to vibration modes in the Si, Al, O framework. Positions of Raman bands are shifted

Fig. 6 Raman spectrum of scapolite as acquired with Gem Raman (top) and with Bravo (bottom)

significantly to lower wavenumbers in the Gem Raman spectra (see Table [1](#page-3-0)).

3.9 Kyanite

For a gem variety of the mineral kyanite, a good agreement in the positions of the significant Raman bands was achieved (see Table [1](#page-3-0)) among the values obtained by the portable instruments and the reference [\[24](#page-8-0)], although only the strongest Raman bands located at 303, 384, 481 and 948 cm $^{-1}$ were found in the spectra taken with the Gem Raman instrument.

3.10 Adularia

Adularia, a gem variety of microcline, a potassium-rich alkali feldspar provided good-quality spectra using both the Gem Raman and Bravo instruments. While the positions of the diagnostic Raman bands in the spectrum from Bravo instrument are in excellent agreement with the reference values [[20\]](#page-8-0), the positions of the corresponding bands in the spectrum from the Gem Raman are again shifted to the lower wavenumbers (see Table [1\)](#page-3-0).

3.11 Forsterite

Synthetic forsterite, a magnesium end member of the olivine group, provided the Raman spectra that were in an excellent agreement between the two instruments as well as with the reference values [\[21](#page-8-0)]. The missing bands in the Raman spectrum taken by the Gem Raman (586 and 881 cm^{-1}) are actually just included in the broadened adjacent bands of higher intensity (602 and 856 cm^{-1}), and

this is reflected by the FWHM parameter of the 856 cm^{-1} Raman band which increases from 16 (Bravo) to 47 (Gem Raman), almost three times.

3.12 Sodalite

The greatest difference in the wavenumbers position of the Raman bands in the spectra recorded by the Gem Raman and Bravo instruments was encountered when analysing the gem variety of the tectosilicate of a complex composition $\text{Na}_8(\text{Al}_6\text{Si}_6\text{O}_{24})\text{Cl}_2$ —sodalite. The shifts to the lower wavelength of up to 13 cm^{-1} for the Gem Raman instrument are reported in Table [1](#page-3-0). Additionally, a strong feature located at 914 cm^{-1} is completely missing in the spectra taken by the Bravo instrument and reference [[23\]](#page-8-0), and thus it is very likely an instrument artefact.

3.13 Fluorescence of $Cr³⁺$ in rubies and emeralds

Fluorescence of several elements can complicate or even disallow the acquisition of Raman spectrum of a mineral. However, the actual impact of the fluorescence on the Raman spectrum can vary significantly, based on the number of fluorescent elements and their concentration as well as laser power used for excitation for example. For some minerals, the fluorescence can be quite specific and even have diagnostic use.

Fluorescence is encountered very often in rubies and emeralds. The red colour of rubies and green colour of emeralds are also caused by this phenomenon. A colourless corundum crystal is built up from the octahedra, where each aluminium atom is surrounded by six oxygen atoms. In this setting, all electrons are paired and

are used up in chemical bonds, and the mineral is colourless. When a minor part (cca 1%) of Al atoms is replaced by Cr atoms, each of the chromium atoms must provide three electrons to bond with surrounding oxygens and thus become Cr^{3+} . Unlike Al^{3+} , the Cr^{3+} still has partially filled 3d orbital with unpaired electrons. These non-bonding electrons can be excited (absorption of violet and green-yellow light) by the incident light and exhibit fluorescence (red light) and thus are responsible for the red colour of rubies together with the small transmission of light in the blue region [[25\]](#page-8-0).

Analogically, in the case of emeralds, the Al^{3+} ions are also replaced by Cr^{3+} ions in the crystal structure, and the colourless beryl becomes a green emerald. The difference in colour, green instead of red, is caused by a different symmetry in the emerald crystals and a weaker interaction of Cr^{3+} with the surrounding ions. This weaker interaction means that less energy of the incident light is needed to excite the unpaired electrons (absorption of violet and yellow-red light) generating fluorescence (red light). Together with a significant transmission of light in the green-blue region, the resulting colour is shifted merely to a green region of the spectrum in the case of emeralds, as opposed to a greater shift to the red region of the spectrum in the case of rubies [\[25](#page-8-0)].

Generally speaking, with the increasing concentration of the Cr^{3+} ions the resulting red and green colours are of a deeper hue and consequently exhibit much higher amount of fluorescence, especially when excited by the high-intensity light source such as lasers.

3.14 GL Gem Raman performance discussion

Table [1](#page-3-0) gives an overall idea of how this instruments stand in a direct comparison with two handheld Raman spectrometers. Several parameters need to be assessed when scrutinizing the quality of resulting Raman spectra and to what extent they allow or complicate the identification of the minerals or gemstones. Two fundamental conditions such as the amount of background and the level of noise probably come first. Sometimes the fluorescence or other phenomena mask almost all usable Raman information; in some cases, the detector can be even saturated. In the case of GL Gem Raman instrument and the tested samples, the background was significant and very steep (see the figures). The other two instruments provided spectra with much 'flatter' baselines and therefore performed much better in this context. Factors (instrumental related) that influence the level of fluorescence the most can be excitation wavelength and laser power.

The level of noise, or signal-to-noise ratio, was generally sufficiently low in the spectra taken by all spectrometers, and this is consistent with the fact that the studied samples had high quality. All the samples were cut and polished gemstones and minerals of moderate sizes.

When Raman bands are positively located in the spectrum, the two parameters of these bands can be discussed to evaluate the performance of an individual instrument and its ability to identify minerals. The most crucial is the position of the Raman band and the shift of this value from the reference value from the literature. The shift of $0-3$ cm⁻¹ from the reference value is generally regarded as a very good agreement. This was the case for most Raman bands in the spectra obtained by the Bravo and Inspector Raman instruments (see Table [1\)](#page-3-0). However, the shifts in the spectra obtained by the Gem Raman spectrometer were much more variable and generally greater with a tendency of shifting towards lower wavenumbers. This could be explained by generally lower quality of the Gem Raman instrument's components and design.

This general lower quality is reflected in a substantial increase of the full width at half maximum (FWHM) parameter of the Raman bands that was observed and recorded for the strongest Raman bands of each mineral (see Table [1\)](#page-3-0). This increase of 2–3 times is very significant and can only be explained by merging of close Raman bands a few times. These unusually broad Raman bands in the spectra of minerals taken by the Gem Raman instrument is a characteristic of this instrument that further complicates the mineral identification and must be taken into account.

4 Conclusions

The GL Gem Raman system permits a relatively easy analysis of gemstone quality minerals of several groups in the mineralogical system. However, the character of resulting Raman spectra is generally of inferior quality when compared to the data from other portable instruments (Raman bands are generally much broader and have greater shifts in wavenumber position). Furthermore, the instrument was not able to record usable Raman spectra of gemstones from important groups for gemmologists such as rubies, which generally provide good spectra when analysed by other portable instruments. The Gem Raman instrument was able to identify some of the emerald gemstones, which are generally a challenge for a Raman spectroscopy due to the high amount of fluorescence background. The need for placement the analysed sample inside the instrument's sampling stage is also a limitation, when compared with other portable instruments with optical head or fibre optics. While this setup is generally suitable for analyses of relatively small and loose gemstones, it may prohibit the proper study of more sizeable works of arts or artefacts adorned with gemstones. Main

(strong) Raman bands are frequently recorded on the minerals investigated in this study (with exception of rubies and emeralds with high content of Cr^{3+}). Their positions roughly correspond to the correct values. Identification of minerals using the new tested gemmological tool is allowed on this base. Such a performance can be seen as sufficient for rough determination of a given mineral. However, unambiguous identification can sometimes be tricky, and additionally, deeper spectroscopic detail is difficult to obtain, i.e. structural differences between varieties, minor modifications related to the composition and reflecting provenance.

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