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# Investigation on the gemological, physical and compositional properties of some opals from Slovakia ("Hungarian" opals)

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## Abstract

The "Hungarian" opal deposits, now in Slovakia, represented the largest and most significant gem opal deposit in Europe from the Roman Times to XIX century. The physical and compositional properties of some "Hungarian" opals have been investigated through several methodologies such as optical analysis, specific gravity, refractive indices, XRPD, IR and LA-ICP-MS. The investigated opals are white, show appreciable play of color with indigo or orange-blue flashes and are surely relevant for gemological purposes. XRD and IR analyses showed the samples are made up by amorphous opal (A). The host rocks are mostly made up by an assemblage of feldspars (oligoclase and sanidine) with lower cristobalite, augite, quartz and kaolinite. The trace element contents are around 200-300 ppm; the most abundant element are Ca, Al, Na, K with lower amounts of Fe, Mg, Sc, Cr. Cromophore elements like V, Cr, Cu, Co, Ni are low or absent. The homogeneous and low trace element composition and the amorphous structure can represent an identification marker. The low trace element contents, especially for elements like Ca, Al, Na, Ba, and Sr would indicate these opals formed through precipitation from residual silica-rich hydrothermal fluids in closed systems, in condition of low temperature.

*Key words*: Hungarian opals; IR spectroscopy; trace elements; XRPD; gemstone.

## Introduction

The "Hungarian" opal deposits, now in Slovakia, represented the largest and most significant gem opal deposits in Europe from the Roman Times to XIXth century, and dominated the world market until the discovery of the rich opal mines in Australia. The mines are located in

South Eastern Slovakia in the Libanka-Simonka mounts, in the area of Dubnik near Kosice, between Zlatá Baňa and Červenica (Leechman, 1961; Webster, 1975; Eckert, 1997; Rondeau et al., 2004; Caucia et al., 2012b). However, these opals are still called "Hungarian" as this region until the end of the World War I, was part of Hungary. The maximum of the mining activity occurred in the mid-XIXth century, when the mines were conducted by the jeweller from Vienna - Solomon Goldschmidt and his heirs, but the activity stopped in 1922 (Dud'a and Molnár, 1992; Caucia et al., 2012b). The cessation of the opal mining at Dubnik was not due to the exhaustion of the deposits but rather to the strong competition of overseas countries (especially Australia), which made the mining activity not profitable. "Hungarian" opals are generally represented by milky terms, harlequin, fire and black opals, as well as hydrophane and hyalite, and are highly appreciated in the world markets for their unique characteristics, in particular the play-of-color. A notable example of "Hungarian" gem opal is represented by the "Harlequin": with a weight of 594 g (2.970 ct) and a length of 13 cm is the biggest Dubník opal in the world. It was found in 1775 in the bed of the brook Oľšavka, and now is hosted in the Museum of Natural History in Vienna. Another notable sample was represented by an opal christened "Burning of Troy" for its flaming lights, that was presented to Joséphine de Beauharnais by Napoleon I of France. Unfortunately, at the end of the World War II this opal disappeared definitively and was probably cut in more little gems. Particularly important is also the famous necklace of Queen Isabella, wife of the "Hungarian" King and Duke of Siebenbürgen, Johann Zapolsky, of 1540. This jewel contains 16 cut "Hungarian" opals of different size and is hosted in the National Museum of Budapest (Eckert, 1997; Caucia et al., 2012b).

Despite their importance, the physical and chemical characteristics of the "Hungarian" opals have been little investigated, if compared to other opals like those from Australia or Mexico; this is mainly due to the fact that Slovakian mines closed prior to the development of many modern techniques of chemical and physical analyses (Kaliciak et al., 1976; Rondeau et al., 2004). The physical and chemical features can be related to the gemological properties of the opals, like the body color or the luminescence, and also to the process of formation and the area of provenance. As described in Gaillou et al. (2008), the identification of the chemical and physical properties of opals from a particular geographic area is important for several reasons. For example, for the sellers is very useful to know the provenance, as the gems of some localities are more valuable than others. In addition, in the archaeometrical investigation, knowledge of the geographical origin is crucial to reconstruct the ancient trade routes of gem (Giuliani et al., 2000; Gaillou et al., 2008).

In this work, the gemological, geochemical and mineralogical properties of some samples of "Hungarian" opals are discussed and also compared with those in literature (Rondeau et al., 2004; Gaillou et al., 2008).

## Geological setting and origin of the "hungarian" opals

Slovakian opals have been considered as formed in a volcanic environment (Webster, 1975; Rondeau et al., 2004), in particular in the volcanic complex of Prešov-Tokaj Mountains, also named Zlatá Baňa layered volcanic complex. The volcanic formations are around 10 to 15 million year old (Zelenka, 1994), of andesitic composition and exhibit minor mineralogical variations. The opal mineralization is described as the last hydrothermal stage of this volcanism (Harman and Chovanec, 1981). In particular, two hydrothermal stages are identified from the mineral association (Kaliciak et al., 1976): pyrite, antimonite, marcasite have crystallized at high temperature while marcasite, chalcedony and

different opal varieties formed at lower temperature. Lastly, limonite, white hydrophane and secondary metallic minerals crystallized during a last supergene stage. The opal host rock is a coarse conglomerate that includes andesitic blocks or gravels of various sizes, developed by tectonic constraints: opal occurrence is disseminated along a major tectonic fault which is responsible for a small horizontal displacement.

According with Rondeau et al. (2004), the origin of most "Hungarian" opals is not related to volcanism but to a precipitation process during a tectonically-controlled low temperature stage. "Hungarian" opals are of type A (amorphous), show the main Raman peak at 437 $cm^{-1}$ and their microstructure consists of large silica spherules with diameters of 125 to 270 nm. The properties of "Hungarian" opals are surprisingly very similar to those of opals in sedimentary deposits and differ from those of volcanic ones. The physical properties seem more determined by the temperature of formation and less by the composition of the host rocks (Rondeau et al., 2004). Based on isotopic data (Rondeau et al., 2004), the temperature of formation of Slovakian opals would be relatively low, lower than 45 °C, similar to that of Australian opals of sedimentary origin (Jones et al., 1964; Keller, 1990), but very different from that of volcanic opals from Mexico (Koivula et al., 1983).

## Materials and methods

The opals investigated in this work derive from rough samples of the Museum of Mineralogy of the "Dipartimento di Scienze della Terra e dell'Ambiente" (University of Pavia), and from the personal collection of the first Author.

The analysed opals can be described as follows (see also Figure 1):

- Samples 1 - 6 are white, translucent and vitreous opals with play of color; sample 4 shows some blue veins;

- Samples 7, 8 and 9 are white, translucent and porcelanaceous opal (dull); sample 7 also shows a weak play of color;

Three gemstones from rough samples were fashioned as cabochon of various shapes (gems 1, 4, 7, Figure 1).

For the analyses, we selected only opal samples that appeared pure and not affected by the presence of contaminations, that are also the most relevant for gemological purposes.

The gems were examined by standard gemological methods to determine their optical properties, hydrostatic SG, UV fluorescence (366-254 nm) and microscopic features. Specific gravity and refractive index measures have been carried out using a Presidium PCS100 Sensible Balance and a Kruss Rifractometer ER6040 equipment, respectively. Detection limits of the refractive index were $1.30 < n < 1.80$.

X-ray powder diffraction data (XRPD) have been collected with a Philips PW1800 powder diffractometer, with CuKα radiation ($\lambda = 1.5418$ Å) and a scan speed of 1°/min, in the range between 2-65° 2θ. Qualitative and semi-quantitative analyses of the mineral phases in the opals and host rocks have been evaluated through the program "PANalytical X'Pert HighScore".

Mid-infrared spectra (FT-IR; 4000-400 $cm^{-1}$) have been recorded in transmission mode using a Nicolet Nexus FT-IR spectrometer, equipped with a 4x beam condenser collector, accumulating 200 scans at a resolution of 4 $cm^{-1}$. We operated by means of KBr compressed pellets, after a pre-treatment of 150 °C and fluxing the sample compartment with gaseous nitrogen.

Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) microanalyses were performed with a double-focusing sector - field ICP-SFMS model Element I, Thermo Finnigan Mat at IGG-CNR of Pavia. Quantification was performed using $SiO_2$ (stoichiometric value) as internal standard and NIST SRM 610 synthetic glass as external standard. Precision and accuracy were estimated

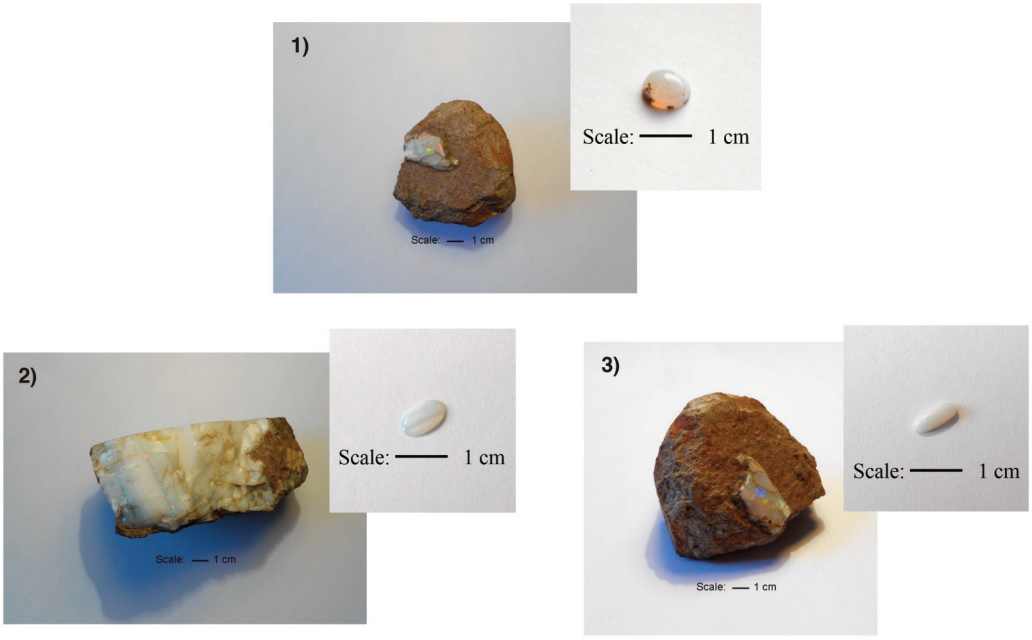


Figure 1. Photo n. 1, sample n. 1 (rough and gemstone):white opal with evident play of color; photo n. 2, sample n. 4 (rough and gemstone): white and massive opal that shows the conchoidal fracture, the vitreous luster and the play of color; the microtexture is columnar; photo n. 3, sample n. 7 (rough and gemstone): beautiful gem-quality sample of white opal into a volcanic rocks, clearly showing the play of color with blue shadow.

by the analysis of a BCR-2 standard and resulted better than 5 and 10%, respectively, for concentration at ppm level. Opal fragments were mounted on epoxy resin and polished before analyses.

## Gemological properties

The appearance and gemological properties of some investigated "Hungarian" opals are reported in Table 1. Concerning color, degree of transparency and luster, the opals are translucent with a vitreous or dull luster and show play of color (samples 1, 4 and 7). Play of color exhibits a variable intensity, from weak to medium, with indigo or orange-blue flashes.

Luminescence, as revealed by UV lamp analysis, is quite variable: the sample n. 1 is inert to the short wavelength radiation and shows a weak lilac fluorescence when exposed to the long ones, while the sample n. 4 shows medium blue fluorescence when exposed to the short wavelength; sample 7 is inert to both short and long wavelength.

The specific gravity values are between 2.01-2.10 i.e., in the range of literature data.

The cut gems were enough transparent to evaluate the refraction indices. The values vary between 1.439 and 1.442 and, similarly with specific gravity, are comparable with the literature data (O'Donoghue, 2006; Caucia et al., 2009, 2012a; Simoni et al., 2010), also with those reported for "Hungarian" opals (Rondeau et al., 2004). None of the samples observed through the polariscope showed anomalous birefringence, when rotated a full 360°.

Table 1. Gemmological properties of the studied samples.

| Sample | Color | Weight (carat) | Trasparency | Luster | Gravity | Refraction Index | Optic effects | UV (short) | UV (long) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | white | 2.19 | Tl | V | 2.01 | 1.440 | play of color | inert | weak lilac |
| 4 | white | 0.37 | T/Tl | V | 2.09 | 1.442 | play of color | medium blue | inert |
| 7 | white | 0.41 | Tl | Dull | 2.10 | 1.439 | weak play of color | inert | inert |

The observations with the light microscopy did not reveal the presence of inclusions, with the sole exception of the gem n. 1 that showed small black inclusions of dendritic shape.

## XRPD Data

XRPD investigations allow distinguishing the opals into three general groups (Jones and Segnit, 1971; Ghisoli et al., 2010): opal C (relatively well ordered α-cristobalite), opal CT (disordered α-cristobalite with α-tridymite-type stacking) and opal A (amorphous). The investigated opals are made up by amorphous opal (opal A; Figure 2a); other mineralogical phases were not detected, at least in sensible amounts. The XRD pattern exhibits a shoulder centered around 4.08 angstroms. We also analysed the mineralogical composition of host rocks that included the opal, that resulted made up by an assemblage of feldspar (oligoclase and sanidine, around 70%), cristobalite (around 20%) and minor amounts of augite, quartz and kaolinite (Figure 2b).

## FT-IR Data

The infrared spectra of the samples n. 1 and 4 (Figure 3a, b) are characterized by a multi-component broad absorption band centered at about 3400 $cm^{-1}$, due to the OH stretching vibration of water molecules, as well as the water bending vibration at ~ 1640-1648 $cm^{-1}$. The other three strong bands at ~ 1100, 790 and 470 $cm^{-1}$ are common to all silicates with tetrahedrally coordinated silicon and are related to the fundamental Si-O stretching vibration. In particular, the 1100 and 790 $cm^{-1}$ bands are generally assigned, respectively, to antisymmetric and symmetric Si-O-Si stretching, whereas the 470 $cm^{-1}$ band is related to O-Si-O bending vibration (Jones and Segnit, 1971; Farmer, 1974; Zarubin, 2001 and references therein; Fritsch et al., 2004; Brajkovic et al., 2007; Caucia et al., 2008; Adamo et al., 2010).

## LA-ICP-MS Data

LA-ICP-MS analyses have been conducted on different spots of 9 opal samples, to determine the trace element composition (Table 2; see detection limits from Miller et al., 2012).

The trace element contents in the investigated opals are around 200-300 ppm: these values are low if compared with those of opals worldwide (Gaillou et al., 2008; Caucia et al., 2009, 2012a; Simoni et al., 2010; Rondeau et al., 2010, 2012). The most abundant elements are Ca (90-178 ppm), Al (67-125 ppm), Na (22-95 ppm), K (15-

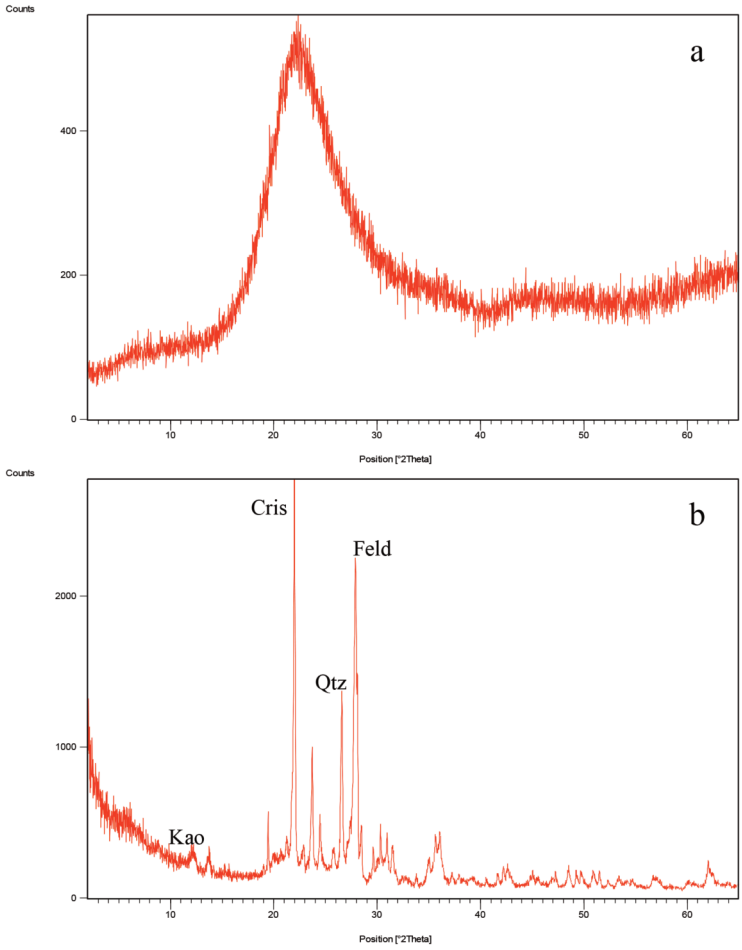


Figure 2. XRPD patterns of the sample n. 1: a) body of the opal showing the amorphous structure; b) host rock of the opal showing high contents of feldspars. Kao = kaolinite; Cris = cristobalite; Qtz = quartz; Feld = feldspars.

44 ppm), with lower amounts of Fe (4-14 ppm), Mg (3-10 ppm), Sc (2-3 ppm), Cr (1,4-2.5 ppm). Cromophore elements like Fe, V, Cr, Cu, Co, Ni are low or absent and, therefore, the investigated samples appear white.

## Discussion

Usually CT opals are found in volcanic environment, while A opals are typical of both sedimentary (the most widespread) or hydrothermal environments (Gaillou et al., 2008; Ghisoli et al., 2010). Our XRD investigations highlighted the "Hungarian" opals are all of A type, as already pointed out by previous research (Rondeau et al. 2004 and references therein). The results through FT-IR Spectroscopy are in agreement with the XRPD data, allowing us to determine the composition of the opals through the combination of these two analytical

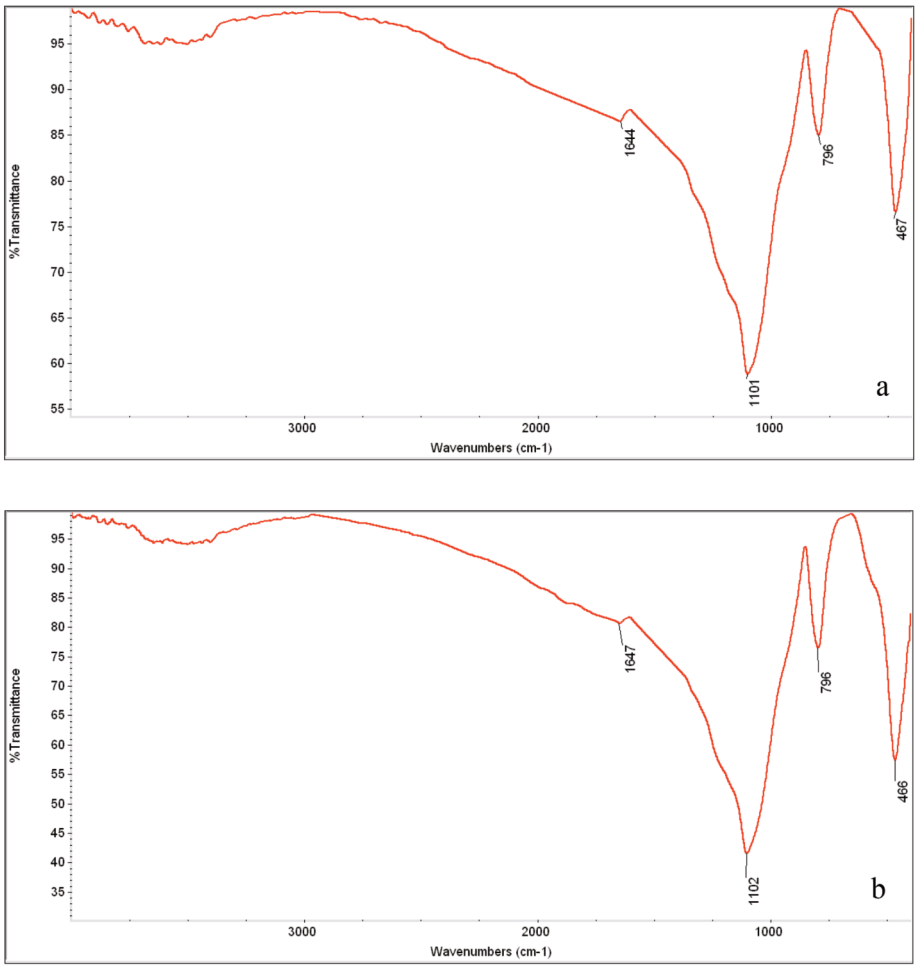


Figure 3. IR spectra of the analyzed opals: a) sample n. 1 (white opal A); b) sample n. 4 (white opal A).

techniques. In fact the frequencies of the bands at 1101-1004, 796, and 466-467 $cm^{-1}$ in samples n. 1 (Figure 3a) and 4 (Figure 3b) are compatible with the A typology (Adamo et al., 2010).

The mineralogical assemblage of the host rocks determined by XRD investigation is mostly made up by feldspars and is coherent with the description of the outcropping lithologies reported in literature (relatively altered andesite; Webster, 1975; Rondeau et al., 2004).

In general, the distribution of trace elements in opals may be due to substitutions in the pseudo-crystallographic structure or to the presence of inclusions of other minerals. Al is frequently the most abundant impurity in opals, and substitutes locally for silicon; the imbalance of charge is compensated by the ingress of monovalent or divalent cations such as $Na^{+}$, $K^{+}$, $Ba^{2+}$, $Mg^{2+}$, $Ca^{2+}$ (Gaillou et al., 2008 and references therein). In our opals, mineral inclusions have not been

Table 2. Trace elements abundances (ppm) for the opals analysed in this work.

| | Sample | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Elements | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| Li | 0.2 | 0.4 | 0.4 | 0.5 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 |
| Be | 0.8 | 0.3 | 0.4 | 0.8 | 1.1 | 0.0 | 0.5 | 0.5 | 0.4 |
| B | 0.5 | 0.8 | 0.4 | 0.8 | 1.1 | 1.7 | 0.6 | 0.7 | 0.5 |
| Na | 25.0 | 95.0 | 27.0 | 17.0 | 22.0 | 35.0 | 32.0 | 38.0 | 39.0 |
| Mg | 2.6 | 10.1 | 7.0 | 3.0 | 4.5 | 3.6 | 4.7 | 5.8 | 7.2 |
| Al | 77.0 | 77.0 | 75.0 | 67.0 | 77.0 | 71.0 | 75.0 | 112.0 | 125.0 |
| K | 37.0 | 44.0 | 25.0 | 18.0 | 19.0 | 15.0 | 23.0 | 35.0 | 40.0 |
| Ca | 117.0 | 145.0 | 114.0 | 90.0 | 178.0 | 96.0 | 113.0 | 111.0 | 122.0 |
| Sc | 2.9 | 2.7 | 3.3 | 2.5 | 2.1 | 2.4 | 3.0 | 2.2 | 2.8 |
| Ti | 2.1 | 1.9 | 1.1 | 0.7 | 0.6 | 0.7 | 1.0 | 0.9 | 1.1 |
| V | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 |
| Cr | 2.1 | 2.4 | 1.8 | 1.8 | 1.5 | 2.0 | 1.9 | 1.9 | 2.0 |
| Mn | 0.5 | 0.9 | 0.3 | 0.3 | 0.5 | 0.3 | 0.2 | 0.5 | 0.4 |
| Fe | 4.1 | 8.4 | 5.1 | 7.0 | 10.3 | 11.2 | 8.2 | 13.7 | 10.9 |
| Ni | 0.9 | 1.1 | 0.6 | 1v0 | 0.8 | 1.0 | 1.2 | 1.6 | 1.0 |
| Cu | 0.1 | 0.2 | 0.3 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Zn | 0.5 | 0.7 | 1.3 | 0.4 | 0.7 | 1.0 | 0.5 | 0.8 | 0.6 |
| As | 0.3 | 0.3 | 0.3 | 0.3 | 0.3 | 0.2 | 0.3 | 0.2 | 0.3 |
| Rb | 0.1 | 0.1 | 0.1 | 0.0 | 0.1 | 0.1 | 0.2 | 0.1 | 0.2 |
| Sr | 0.2 | 0.4 | 0.2 | 0.2 | 0.3 | 0.3 | 0.3 | 0.4 | 0.4 |
| Cd | 0.3 | 0.4 | 0.2 | 0.2 | 0.3 | 0.3 | 0.4 | 0.3 | 0.3 |
| Ba | 0.0 | 0.1 | 0.0 | 0.2 | 0.1 | 0.2 | 0.2 | 0.1 | 0.3 |
| Total | 276.0 | 392.0 | 266.0 | 212.0 | 321.0 | 242.0 | 267.0 | 327.0 | 354.0 |

identified, neither by optical observations nor by XRD investigations: the opals appear to be quite pure. Therefore, the distribution of trace elements appears especially determined by substitutions in the structure.

The “Hungarian” opals analyzed in this work are characterized by very low amounts of Ba (< 1 ppm); according with Gaillou et al. (2008), Ba concentrations allow to differentiate sedimentary opals from volcanic ones, as the first feature Ba contents > 120 ppm, while the volcanic opals show lower contents. Anyway, there are also some exceptions: for instance, the opals from Wollo Province in Ethiopia formed through weathering of ignimbrites with consequent liberation of silica, and contain very variable Ba contents (Rondeau et al., 2010, 2012). The low Ba contents in our opals would be typical of volcanic terms, but a high temperature magmatic origin is discounted by

stable isotope analyses performed by Rondeau et al. (2004). The host rocks of our "Hungarian" opals are largely made up by feldspars, that contain high contents of elements like Ca, Al, K, Na, Ba and Sr while all these elements are low, or absent, in the opals. The geochemical composition of host rocks does not, apparently, contribute to that of the opals; this means that the origin of the opals is not related to the direct weathering of feldspars. A reasonable hypothesis is that our opals formed through precipitation from residual silica-rich fluids formed during the last hydrothermal stages, in closed systems. The trace elements were extracted from the fluids during the previous formation of feldspars and other minerals that make up the host rocks. The hydrothermal genesis took place at low temperature, as also confirmed by the amorphous structure and stable isotope composition.

Ca in the opals worldwide is the only element that varies significantly with the geographic origin of the samples (Gaillou et al., 2008). Ca contents in our "Hungarian" opals are quite low (around 100 ppm) when compared to opals from other occurrences and show a homogeneous distribution: Ca contents can therefore be used as marker for these opals, if they are not contaminated. Usually, the color of the opal is related to inclusions of colored minerals (Fritsch et al., 1999, 2004), and/or to the abundance of some cromophore elements (McOrist and Smallwood, 1997; Fritsch et al., 1999; Gaillou et al., 2008 and references therein; Caucia et al., 2009, 2012a; Simoni et al., 2010). The low content of trace element especially of the chromophore, together with the relatively scarcity of inclusions determine the absence of color in our opals, that appear white.

## Conclusions

"Hungarian" opals are of great historical importance and samples from Dubnik or from the area of Tokaj Mountains are still sold as raw materials or as cabochon cut gemstones in the tourist areas near the Balaton Lake. The samples of white opal analysed in this work can be considered as precious as they show, albeit with different intensities, the phenomenon of play-of-color, with flashes on the blue-indigo shades. Also the blue veins are related to the play-of-color, do not depend on the chemical composition but to the diffraction of visible light.

Gemological properties and mineralogical composition of our "Hungarian" opals are in agreement with data reported in literature (Rondeau et al., 2004 and references therein). The investigated samples are made up by amorphous opal (opal A) and show low contents of trace elements, as they are pure phases. The samples exhibit a quite homogeneous elemental composition that, together with the typical amorphous structure, can represent valuable geographical markers. On the base of isotopic investigation, Rondeau et al. (2004) hypothesize the "Hungarian" opals more likely formed during a low temperature tectonic event, rather than during a high temperature volcanic one. We think that the low trace element contents, especially for elements like Ca, Al, K, Na, Ba, Sr that commonly occur in feldspars, also support an origin through precipitation from residual silica-rich hydrothermal fluids in a closed system, at low temperature.

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