

LABORATORY REPORT

Novi Sad Report no. 167/17

Report of laboratory tests

Testing performed by : *The Laboratory for testing materijals in Cultural Heritage Department of Materijals Engineering*

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X-ray Pyorescent Spectroetry (XRF)

Infrared spectrometry with Fourier Transform (FTIR) Description and identification Sample M1 samples

Date of test *03.10-17.10.2017*

The report is certified by : Prof.Dr Jonjaua Ranogajec , head of laboratory.

Note 1 : The results of this laboratory test report refer only to the submitted samples.

Note 2 : 1. Description of samples

- **A sample marked M1 was brought for analysis in a piece of rock material of approx.. 3x2 irregular in shape, dark brown to black in color and uneven surface. By crushing the , it was noticed that its interior is darker in color compared to the exterior.**

Note : Fayalite is an iron-silicate mineral from the olivine subgroup that belongs to the group of non-silicate / orthosixicate.

Olives are minerals that can be founded on Earth in volcanic regions (the melting point of fayalite is above 1200 C), while they can also be founded in the city of meteorites², on the Moon³, on Mars ^{4,5} , or on asteroids.

Reference : 1. International Mineralogical Association, <http://rruff.info/Fayalite/RO70293> ; database of Mineral properties, 2. Fukang and other Pallasites.

Farlan.com (2008-04-30) ; Z.Meyer, C. (2003) ; „Mare Basalt Volcanism``.

NASA Lunar Petrographic Educational Section Set. OUR. 4. Pretty Green Mineral ... Mission update 2006...

UMD Deep Impact Website, University of Maryland Ball Aerospace & Technology Corp. S.Hoefen, T.M. , et. al. 2003.

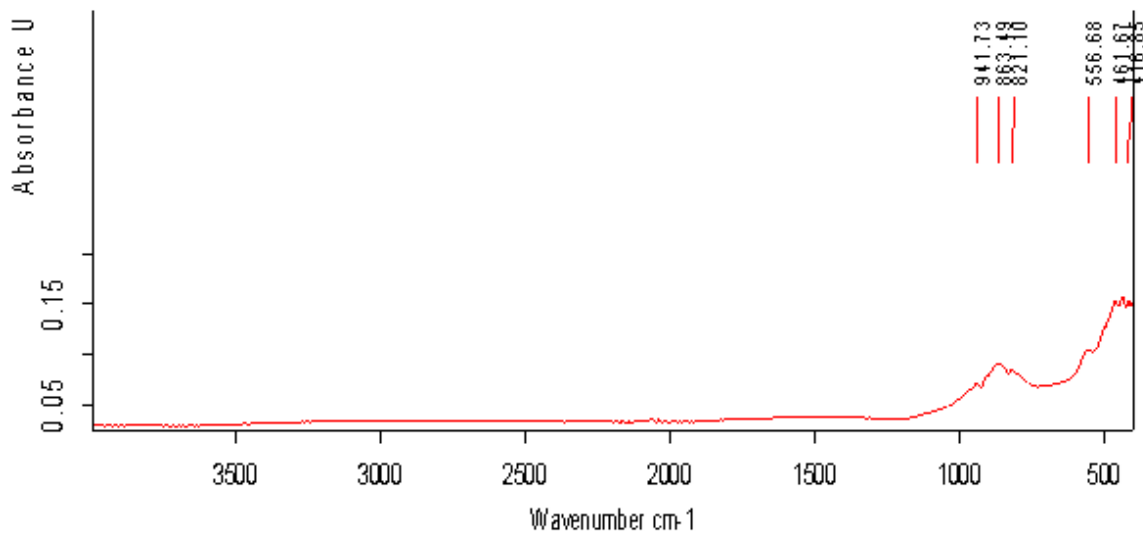
``Discovery of Olivine in the Nill Fossae Region of Mars``. Science 302, 627-30.

`` <http://www.sciencemag.org/content/302/5645/627> ``

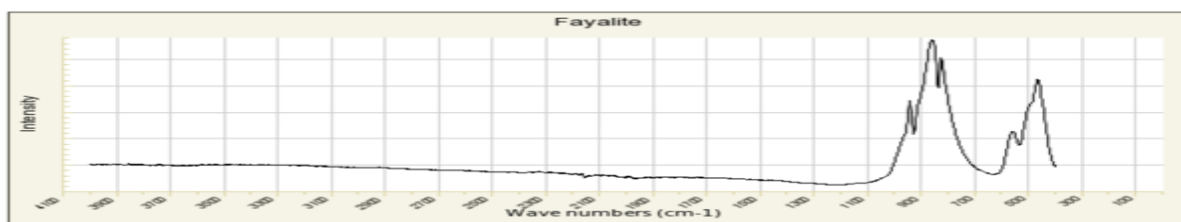
3.1.1. Comment and opinions – FTIR analysis

Comparing the FTIR spectrum of the sample (Figure 3.1) with the spectrum of fayalite minerals (Figure 3.2) of the database shows a complete match of the characteristic maxima of the samples with the characteristic maxima of the fayalite, and based on the FTIR analysis it can be concluded that the sample is M1 mineral fayalite.

3.2. XRF analysis – X – Ray fluorescence spectrometry



Due to the impossibility of comparasion for cobalt with a standard, the estimated concentration of that chemical element is about 0.5%



Slika 3.2. FTIR spektar minerala fajalita iz baze podataka Internacionalne mineraloške asocijacije¹.

Display of the XRF recording site on the M1 sample (left) and the results of mass concentrations of chemical elements from the composition of the M1 sample at the recording point (right).

3.2.1. COMMENTS AND OPINIONS – XRF ANALYSIS THE RESULTS OF XRF ANALYSIS

(Image 3.3) indicate the presence of a large proportion of iron (Fe) of 46.84% . The next most present chemical element is silicon (Si) with a concentration of 4.53% , followed by magnesium (Mg), aluminum(Al) and calcium (Ca) with proportions higher than 1% . Other chemical elements identified by the XRF method are found in traces with concentrations less than 0.5%



| Element | Net | Conc. % |
|---------|--------|---------|
| Mg | 47 | 1,197 |
| Al | 365 | 1,381 |
| Si | 2051 | 4,534 |
| P | 230 | 0,207 |
| S | 119 | 0,068 |
| K | 533 | 0,214 |
| Ca | 4600 | 1,192 |
| Ti | 1049 | 0,133 |
| Cr | 315 | 0,018 |
| Mn | 21297 | 0,428 |
| Fe | 917736 | 46,837 |
| Co | 10869 | * |
| Zn | 208 | 0,005 |

* Usled nemogućnosti upoređivanja rezultata za kobalt sa standardom, procenjena koncentracija tog hemijskog elementa iznosi oko 0,5%.

2.1. FTIR analysis – Infrared spectrometry with Fourier transform Sample preparation was performed by grinding in an agate mortar to a powder, and this the homogenized sample was recorded with an FTIR device.

FTIR analysis was performed by Alpha – BRUKER optics (Germany) FTIR camera in ATR (Attenuated Total Reflectance) recording, with a diamond crystal, in the range of wave numbers from 400 to 4000 cm⁻¹ and a resolution of 4 cm⁻¹, the FTIR spectrum of each sample is the average spectrum 24 recording.

The obtained FTIR spectra were analyzed using OPUS software (**BRUKER, Germany**).

2.2. XRF analysis – X – ray fluorescence spectrometry XRF analysis (X-Ray Fluorescence Spectroscopy) was performed using a mobile ARTAX 200 i-XRF spectrometer (BROKER Nano, Germany) equipped with an Rh cathode ray tube and an integrated camera.

The spectra were obtained under the following experimental conditions : voltage 25kV, current 1500MA, recording time of 100 s under helium atmosphere. The obtained spectra were analyzed using integrated **ARTAX SPECTRA 7** software developed by **BRUKER**.

The described portable device works in non-contact mode at a distance of 2 st with the inclination of the emitted X-ray is 45` in relation to the examined surface. 2.2. XRD analysis – X – ray diffraction analysis X-ray structural analysis (XRD) is a diffractometric mehod used to determine the crystalline phases present in the material using X-rays. The method is non-destructive and is suitable for testing multicomponent systems, which include tested rock materials.

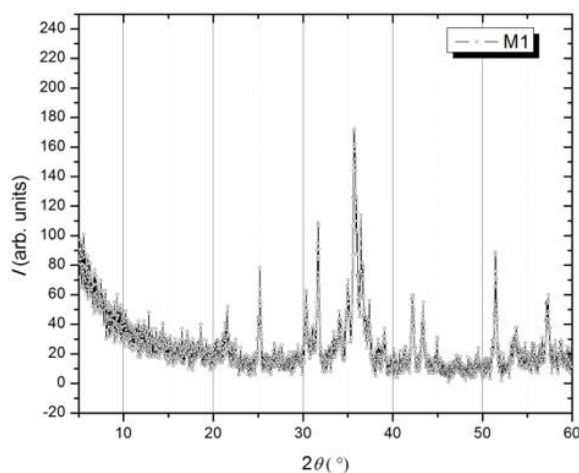
The method is based on the repulsion of the X-pin from the base levels of the crystal. Since each mineral has a different arrangement of crystal planes and a different distance between them, with a help of an appropriate database (ASTM card) it is possible to identify a certain mineral phase present in the tested materijals.

In this study, all tests were performed on previously sprayed equipment on the device XRD PW 1710, PHILIPS (Netherlands).

The characteristic irradiation of the copper anode (Cu Ka) in the range of 20% 3D (5-60)* with a step of 0.02* and an exposure time of 2s was used. 2

3.1. FTIR TEST – INFRARED SPECTROMETRY WITH FURIE TRANSFORMATION

The results of FTIR with a testing device are showed on the picture below 3.1.



Slika 3.4. Rezultat XRD analize (difraktogram) snimljen na uzorku M1.

Display of FTIR spectrum of sample M1

Figure 3.2. shows the spectrum of fayalite minerals from the database of the international Mineralogical Association.

3.2. FTIR spectrum of fayalite minerals from the database of the International Mineralogical Association

Conclusion based on laboratory tests that are based on analyzes (report number D20-1457) performed on the M1 sample using the FTIR, XRF, XRD method, we come to the following opinion and conclusion : The subject from which the M1 sample was taken with a high possibility is METEORITE, rock-metal (46.8% Fe, other metals and minerals). Based on analyzes performed by various methods, the age of the object from which the M1 sample was taken can be estimated at more than 700 000 000 years.

Date of examination 30.10.2017.

The report is certified by : prof.dr. Jonjaua Ranogajec.

**The result of XRD analysis (diffractogram) recorded on sample M1. XRD analysis of sample M1 (Figure 3.4) identified the following mineral phases, ranked by presence : Fayalite (Fe_2SiO_4), Forsterite (Mg_2SiO_4), Galaxite ($\text{Mn}_{0.17}\text{Fe}_{0.83}$) ($\text{Al}_{1.96}\text{Fe}_{0.04}$) Donatite (Fe, Mg) (Cr, Fe) 20,4 .
Conclusion of the test Considering the test results of the M1 sample by the FTIR, XRF and XRD methods, it can be concluded with certainty that it is a mineral of chemical composition (Fe_2SiO_4).**

This is supported by the results of the FTIR analysis (Figure 3.1.) which completely coincide with the reference spectrum of fayalite (Figure 3.2) . The chemical composition of the sample M1 was determined by XRF analysis (Fe-46,84% , Si 4,53% , while oxygen (O) could not be identified by XRF method). In the end, X-ray structural analysis confirmed that it was fayalite with the presence of impurity , or some other mineral phases, companions (forsterite, galaxite and donatite)