

<b>ARTIST:</b>	Josip Diminić
<b>TITLE OF THE WORK and YEAR OF EXECUTION:</b>	Objekt I / Object I (1979)
<b>MATERIALS:</b>	Painted steel

	Name and description of the sample	Analytical methods	Notes
1	<b>5/1 – coatings (cross section)</b>	Micro FTIR, Optical microscopy , SEM/EDS	Samples 5/4, 5/5 and 5/6 are taken from the same position as samples 5/1, 5/2 and 5/3. In order to get the insight of the coatings, optical microscopy, SEM/EDS and micro FTIR analysis were performed on prepared crossed sections of samples 5/1, 5/2 and 5/3.
2	<b>5/2 – coatings (cross section)</b>	Micro FTIR, Optical microscopy , SEM/EDS	
3	<b>5/3 – coatings (cross section)</b>	Micro FTIR, Optical microscopy , SEM/EDS	
4	<b>5/7 – corrosion products</b>	SEM/EDS	
5	<b>5/8 – corrosion products</b>	FTIR	

#### Description of the analytical methods, equipment and procedures:

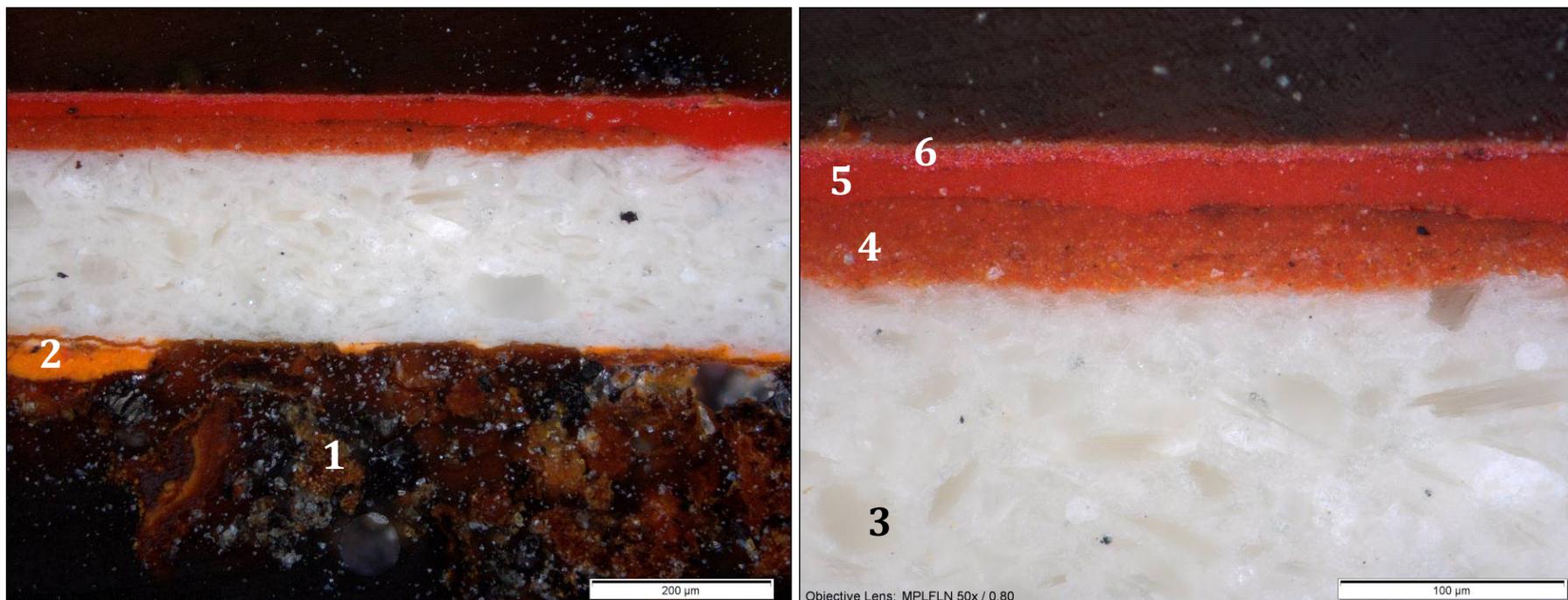
**-Optical microscopy:** analysis performed on sample or cross section using visible (VIS), ultraviolet (UV), polarized (POL) or infrared (IR) light depending on the characteristic of the observed sample. Observation and images taken from 50X to 1000X magnification. **Equipment used:** Optical microscopy Olympus BX51 and optical microscopy Carl Zeiss Image m2M.

**-Fourier Transform Infrared Spectroscopy (FTIR):** analysis performed using KBr pellets preparation (2 mg sample + 120 mg KBr). Each spectrum is a result of 64 scans taken at resolution of 4 cm<sup>-1</sup> in the range from 4000 to 400 cm<sup>-1</sup>. Collected spectra were baseline corrected and when necessary smoothed according to Savitzky/Golay algorithm. **Equipment used:** FTIR spectrometer Tensor 27 Bruker.

**-Micro Fourier Transform Infrared Spectroscopy (μFTIR):** analysis performed on prepared cross section using Attenuated Total reflection objective (ATR) suitable of analysis on area of approximately 50 x 50 μm. The spectra are the results of 32 scans taken at resolution of 4 cm<sup>-1</sup> in the range from 4000 to 600 cm<sup>-1</sup>. **Equipment used:** FTIR microscope Hyperion 1000 Bruker and as source FTIR spectrometer Tensor 27 Bruker.

**-Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)- SEM/EDS:** analysis performed operating under low vacuum conditions for non-conductive samples (80 Pa) and under high vacuum for conductive samples. Images were recorded with Backscattered electrons detector (BSED) with spot from 3 to 5, working distance 10 mm, acceleration voltage from 20 to 30 kV. **Equipment used:** FEG Quanta 250 FEI. EDS microanalysis were performed on observed samples at acceleration voltage of 30 kV and working distance 10 mm. **Equipment used:** Penta FET X-act detector Oxford Instruments. NOTE: The EDS microanalysis of the chemical composition by SEM is performed by analysing the chemical composition in a small sample segment and under a certain magnification, whereby the results are not quantitatively comparable, i.e. the measurements vary considerably from one point to another due to inhomogeneity of the tested samples, surface contamination, segregation of the elements and sensitivity of the method. The results of EDS analysis do not represent the chemical composition of the whole sample but the chemical composition of the examined point/field on the sample's surface.

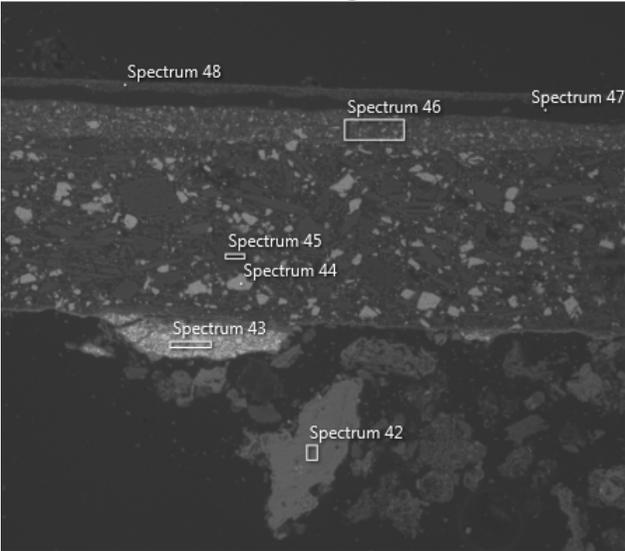
**Results:**  
**Sample 5/1**



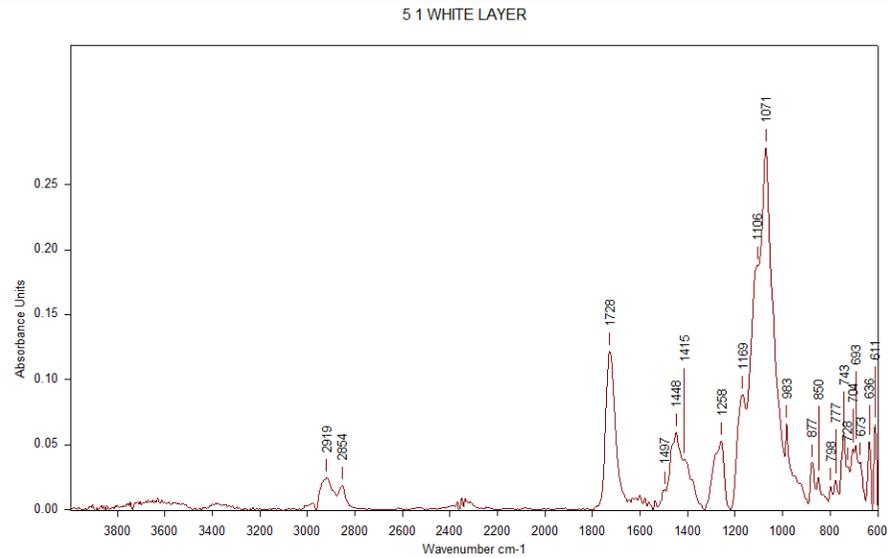
**Sample 5/1** – optical microscopy (magnification 50X and 500X), SEM/EDS and micro FTIR analysis has shown the following structure and composition

- 1- Support (steel)
- 2- Orange base coat of regular thickness about 100  $\mu\text{m}$ , containing minium and an organic binder.
- 3- White ground layer of regular thickness of about 200  $\mu\text{m}$ , containing barite, chalk and alkyd binder.
- 4- Red paint layer, average thickness 50  $\mu\text{m}$ , containing iron red, barium sulphate, silicates, alkyd binder possibly minium and chrome red.
- 5- Red paint layer, average thickness 30  $\mu\text{m}$ , containing organic pigment and binder
- 6- Red paint layer, average thickness 20  $\mu\text{m}$ , containing minium, chrome red, titanium white, most probably barite and alkyd binder.

**Sample 5/1 - SEM/EDS analysis**

<p style="text-align: center;"><b>Electron Image 17</b></p>  <p style="text-align: center;">250µm</p>	<p>Spectrum 42</p> <table border="1"> <thead> <tr> <th>Wt%</th> <th>Wt% Sigma</th> </tr> </thead> <tbody> <tr><td>Fe</td><td>42.89 0.97</td></tr> <tr><td>O</td><td>34.92 1.02</td></tr> <tr><td>C</td><td>19.12 1.37</td></tr> <tr><td>Pb</td><td>2.46 0.61</td></tr> <tr><td>Si</td><td>0.61 0.12</td></tr> <tr><td>Total</td><td>100.00</td></tr> </tbody> </table>	Wt%	Wt% Sigma	Fe	42.89 0.97	O	34.92 1.02	C	19.12 1.37	Pb	2.46 0.61	Si	0.61 0.12	Total	100.00	<p>Spectrum 43</p> <table border="1"> <thead> <tr> <th>Wt%</th> <th>Wt% Sigma</th> </tr> </thead> <tbody> <tr><td>Pb</td><td>50.65 1.14</td></tr> <tr><td>C</td><td>32.19 1.18</td></tr> <tr><td>O</td><td>14.21 0.87</td></tr> <tr><td>Fe</td><td>2.94 0.19</td></tr> <tr><td>Total</td><td>100.00</td></tr> </tbody> </table>	Wt%	Wt% Sigma	Pb	50.65 1.14	C	32.19 1.18	O	14.21 0.87	Fe	2.94 0.19	Total	100.00	<p>Spectrum 44</p> <table border="1"> <thead> <tr> <th>Wt%</th> <th>Wt% Sigma</th> </tr> </thead> <tbody> <tr><td>Ba</td><td>38.57 1.23</td></tr> <tr><td>O</td><td>25.88 1.35</td></tr> <tr><td>C</td><td>25.04 1.84</td></tr> <tr><td>S</td><td>8.13 0.38</td></tr> <tr><td>Mg</td><td>1.06 0.23</td></tr> <tr><td>Fe</td><td>0.68 0.18</td></tr> <tr><td>Si</td><td>0.62 0.18</td></tr> <tr><td>Total</td><td>100.00</td></tr> </tbody> </table>	Wt%	Wt% Sigma	Ba	38.57 1.23	O	25.88 1.35	C	25.04 1.84	S	8.13 0.38	Mg	1.06 0.23	Fe	0.68 0.18	Si	0.62 0.18	Total	100.00																																								
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## Sample 5/1 - Micro FTIR analysis



Experiment HYPERION1000\_ATR.xpm

Operator Name Administrator

Instrument Type TENSOR 27

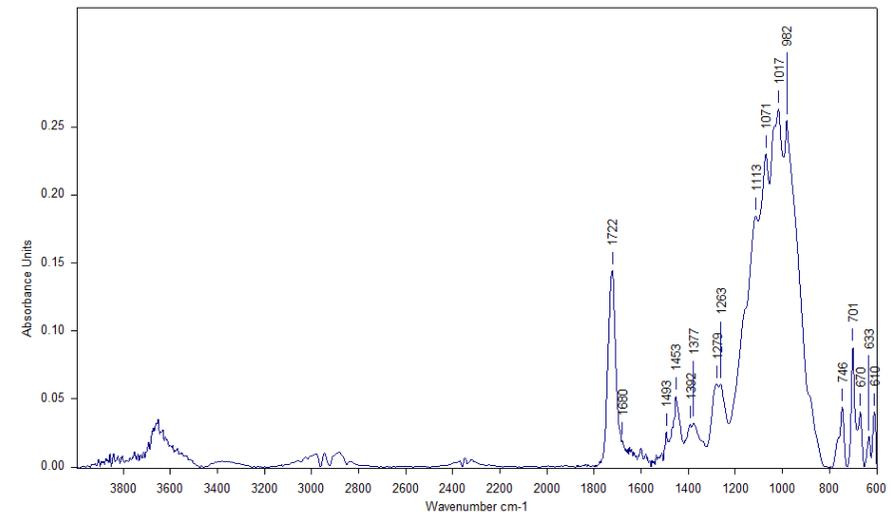
Resolution 4

Path of File C:\CAPUS\MICRO FTIR\MEAS

Date of Measurement 23.7.2019.

Sample Form HYPERION 1000\_15x objective

Sample Scans 32



Experiment HYPERION1000\_ATR.xpm

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Resolution 4

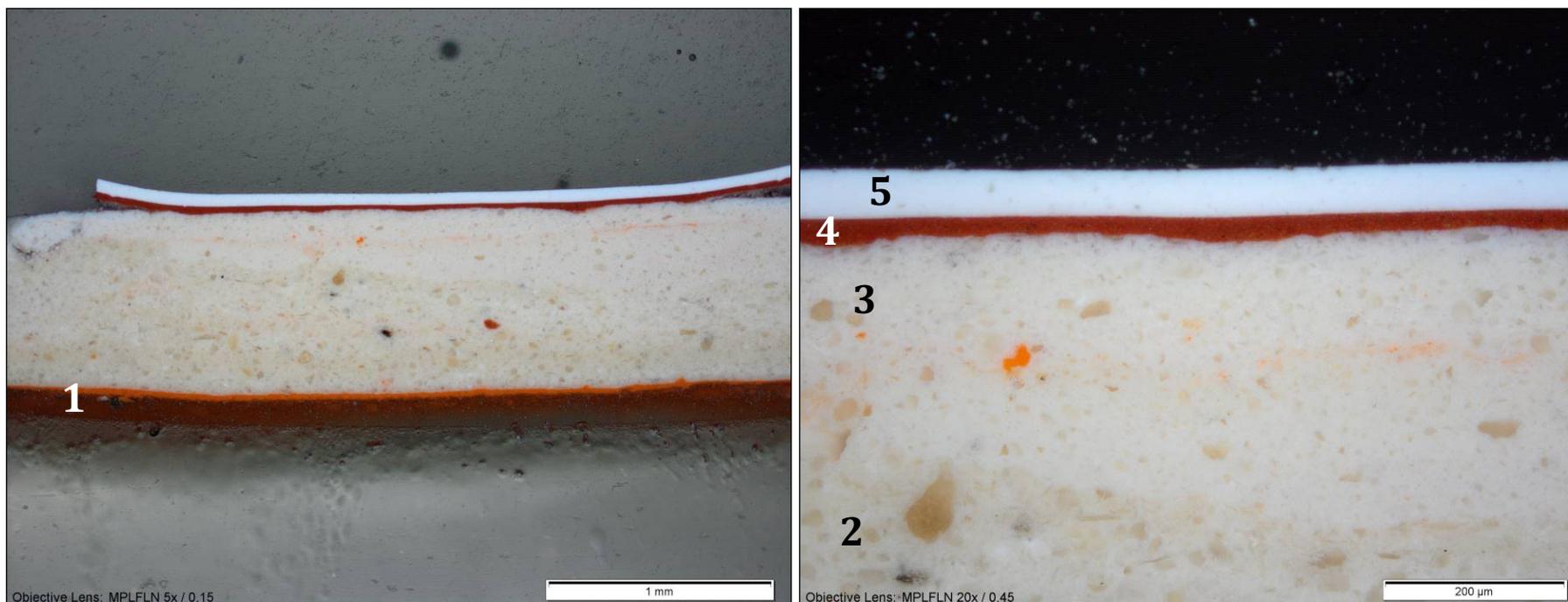
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Date of Measurement 23.7.2019.

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## Sample 5/2

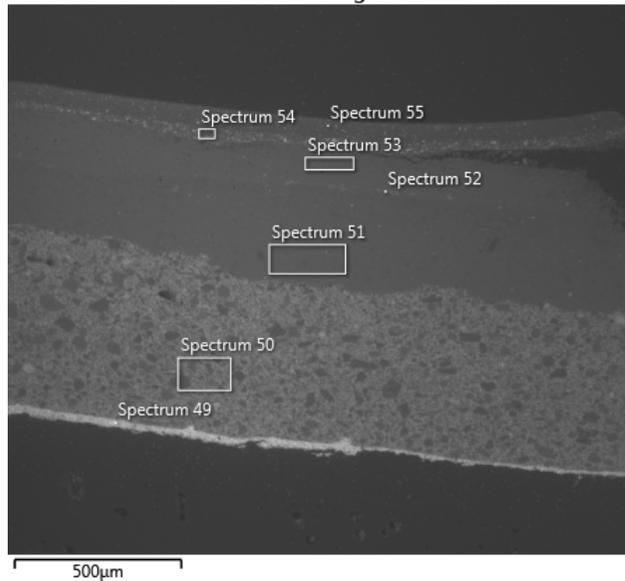


**Sample 5/2** – optical microscopy (magnification 50X and 200X), SEM/EDS and micro FTIR analysis has shown the following structure and composition

- 1- Base coat of regular thickness about 20  $\mu\text{m}$ , containing minium and an organic binder.
- 2- White layer of regular thickness about 250  $\mu\text{m}$  (probably mastic) mainly made of barium sulphate, chalk, zinc white, silicates and alkyd binder
- 3- Second white ground of irregular thickness (probably mastic) consisting of chalk, titanium white and zinc white and alkyd binder. Between layer 2 and 3 it is visible a thin irregular layer consisting of minium.
- 4- Red paint layer of regular thickness about 20  $\mu\text{m}$  containing alkyd binder, barite, chalk, titanium white and iron leading to the conclusion that red ochre is also present.
- 5- White paint layer consisting mainly of titanium white and alkyd binder.

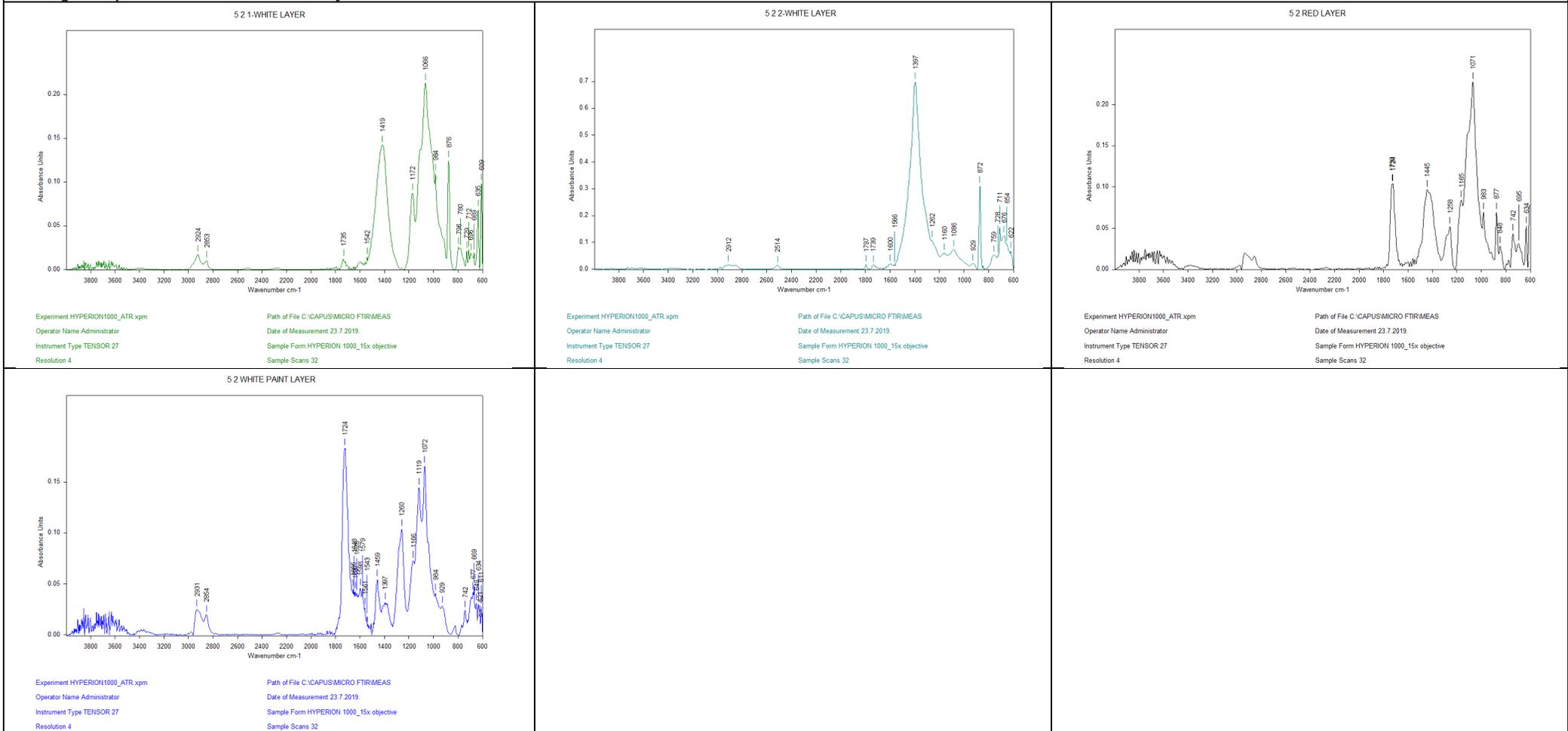
### Sample 5/2 - SEM/EDS analysis

Electron Image 18

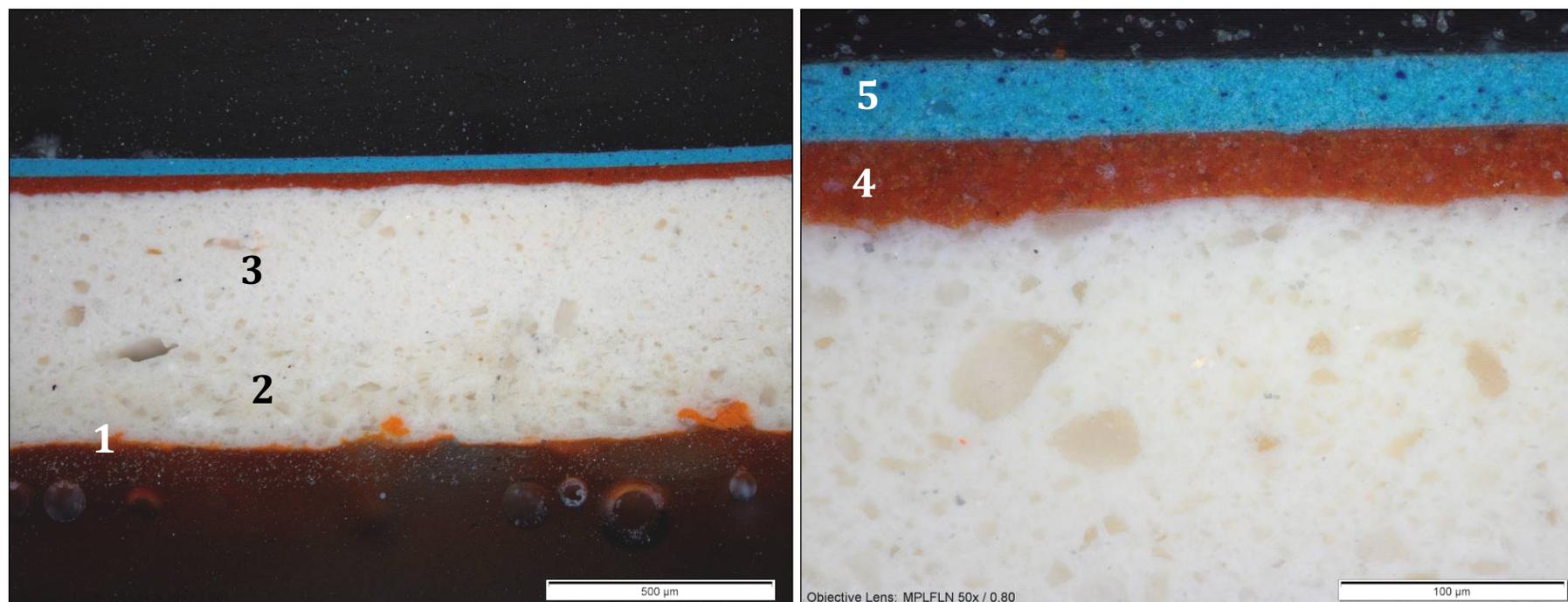


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## Sample 5/2 - Micro FTIR analysis



## Sample 5/3

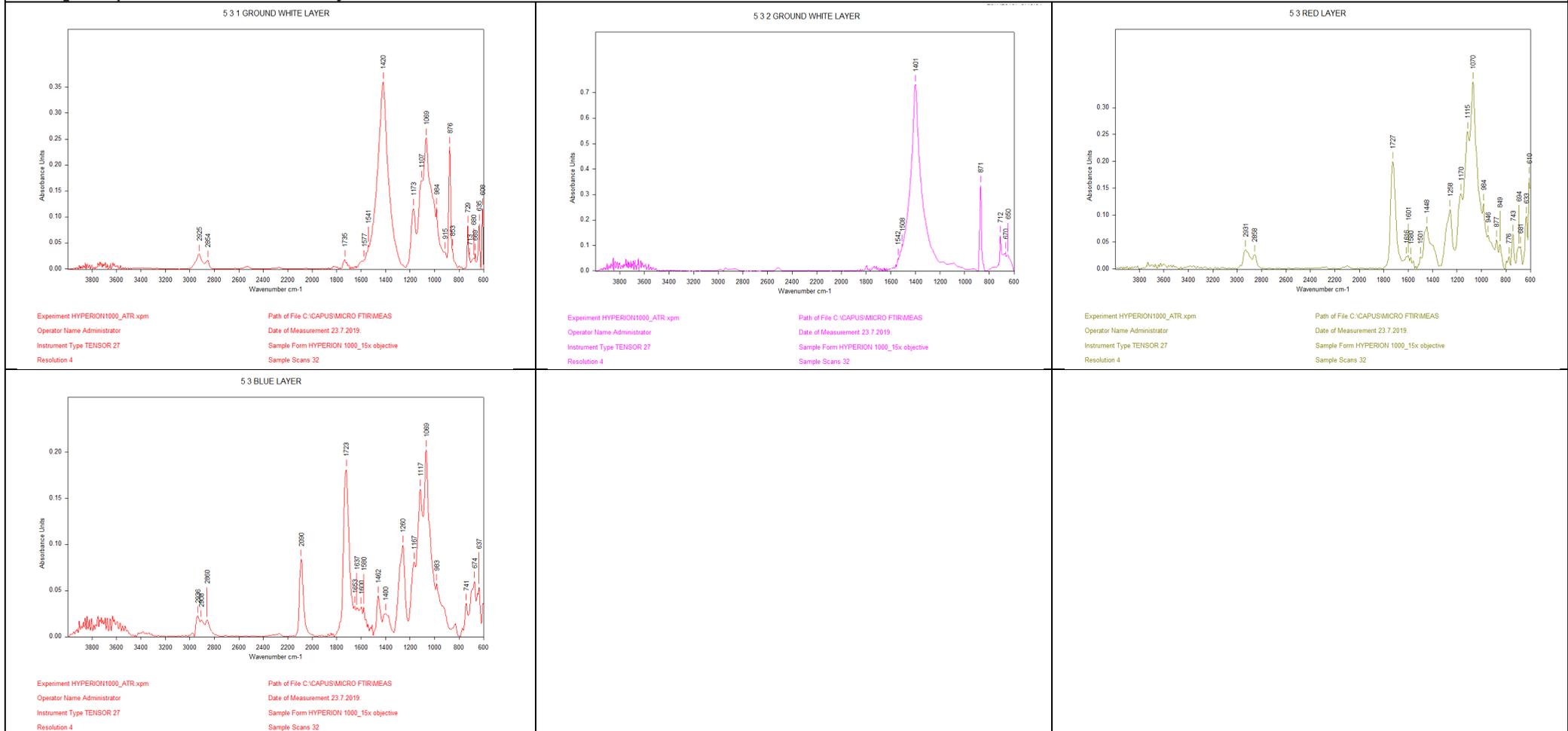


**Sample 5/3** – optical microscopy (magnification 50X and 500X), SEM/EDS and micro FTIR analysis has shown the following structure and composition

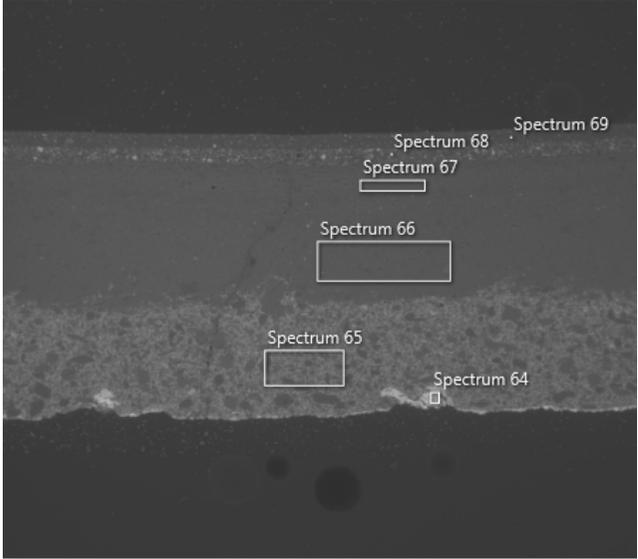
- 1- Base coat of regular thickness about 20 µm, containing minium and an organic binder.
- 2- White layer of regular thickness (probably mastic) about 250 µm mainly made of barite, chalk, zinc white, silicates, and alkyd binder.
- 3- Second white ground of irregular thickness (probably mastic) consisting of chalk, titanium white and zinc white. No peaks assignable to organic binders has been detected most probably because one particle of chalk has been analysed. According to the all the previous analysis, it is assumable that the layer contains some organic binder.
- 4- Red base coat of regular thickness about 50 µm containing alkyd binder, barium sulphate, most probably chalk, titanium white and iron leading to the conclusion that red ochre is present and that unidentified organic red pigment could also be present.

- 5-
- 6- Blue paint layer consisting mainly of titanium white, alkyd binder and Prussian blue.

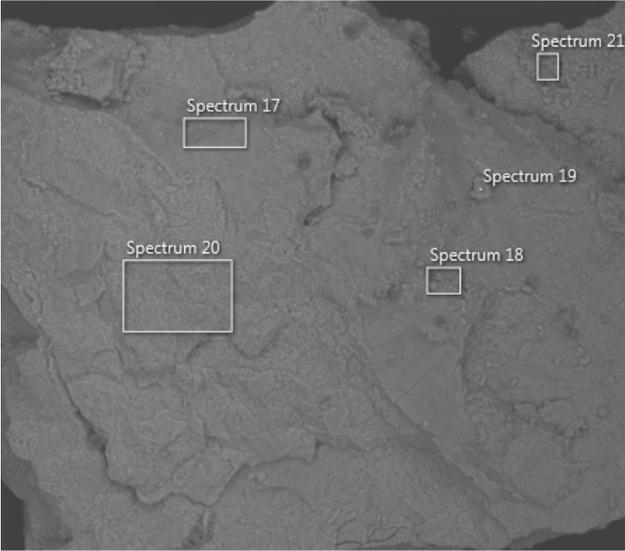
### Sample 5/3 - Micro FTIR analysis



### Sample 5/3 - SEM/EDS analysis

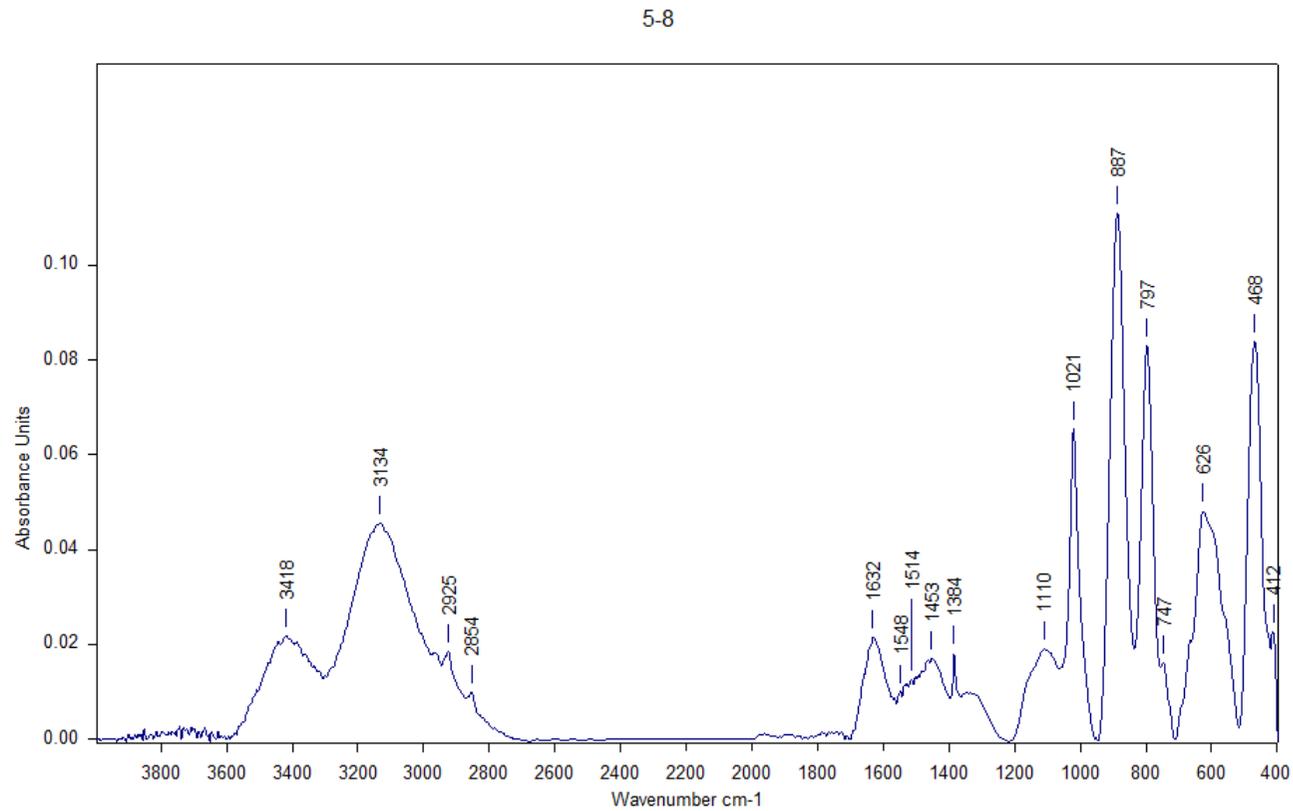
<p style="text-align: center;">Electron Image 24</p> 	<p>Spectrum 64</p> <table border="1"> <thead> <tr> <th>Wt%</th> <th>Wt% Sigma</th> </tr> </thead> <tbody> <tr><td>Pb</td><td>48.57 0.79</td></tr> <tr><td>C</td><td>30.40 0.82</td></tr> <tr><td>O</td><td>16.12 0.70</td></tr> <tr><td>Fe</td><td>2.07 0.11</td></tr> <tr><td>Ca</td><td>1.09 0.08</td></tr> <tr><td>Zn</td><td>1.05 0.12</td></tr> <tr><td>Ba</td><td>0.70 0.17</td></tr> <tr><td>Total</td><td>100.00</td></tr> </tbody> </table>	Wt%	Wt% Sigma	Pb	48.57 0.79	C	30.40 0.82	O	16.12 0.70	Fe	2.07 0.11	Ca	1.09 0.08	Zn	1.05 0.12	Ba	0.70 0.17	Total	100.00	<p>Spectrum 65</p> <table border="1"> <thead> <tr> <th>Wt%</th> <th>Wt% Sigma</th> </tr> </thead> <tbody> <tr><td>C</td><td>33.48 0.94</td></tr> <tr><td>O</td><td>32.29 0.71</td></tr> <tr><td>Ba</td><td>9.49 0.22</td></tr> <tr><td>Ca</td><td>7.07 0.14</td></tr> <tr><td>Zn</td><td>5.49 0.17</td></tr> <tr><td>S</td><td>3.90 0.10</td></tr> <tr><td>Si</td><td>3.01 0.09</td></tr> <tr><td>Mg</td><td>2.16 0.11</td></tr> <tr><td>Al</td><td>0.92 0.07</td></tr> <tr><td>Pb</td><td>0.88 0.24</td></tr> <tr><td>Na</td><td>0.76 0.18</td></tr> <tr><td>Fe</td><td>0.28 0.05</td></tr> <tr><td>K</td><td>0.26 0.03</td></tr> <tr><td>Total</td><td>100.00</td></tr> </tbody> </table>	Wt%	Wt% Sigma	C	33.48 0.94	O	32.29 0.71	Ba	9.49 0.22	Ca	7.07 0.14	Zn	5.49 0.17	S	3.90 0.10	Si	3.01 0.09	Mg	2.16 0.11	Al	0.92 0.07	Pb	0.88 0.24	Na	0.76 0.18	Fe	0.28 0.05	K	0.26 0.03	Total	100.00	<p>Spectrum 66</p> <table border="1"> <thead> <tr> <th>Wt%</th> <th>Wt% Sigma</th> </tr> </thead> <tbody> <tr><td>O</td><td>45.04 1.10</td></tr> <tr><td>Ca</td><td>21.82 0.49</td></tr> <tr><td>C</td><td>21.04 0.95</td></tr> <tr><td>Ti</td><td>8.53 0.25</td></tr> <tr><td>Zn</td><td>1.95 0.17</td></tr> <tr><td>Mg</td><td>0.89 0.13</td></tr> <tr><td>Si</td><td>0.50 0.07</td></tr> <tr><td>S</td><td>0.23 0.06</td></tr> <tr><td>Total</td><td>100.00</td></tr> </tbody> </table>	Wt%	Wt% Sigma	O	45.04 1.10	Ca	21.82 0.49	C	21.04 0.95	Ti	8.53 0.25	Zn	1.95 0.17	Mg	0.89 0.13	Si	0.50 0.07	S	0.23 0.06	Total	100.00									
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**Sample 5/7 - SEM/EDS analysis**

<p style="text-align: center;"><b>Electron Image 9</b></p>  <p style="text-align: left; margin-left: 40px;">250µm</p>	<p>Spectrum 17 Wt% Wt% Sigma</p> <p>O 46.89 1.01</p> <p>Fe 37.11 0.80</p> <p>C 13.98 1.39</p> <p>Si 0.80 0.12</p> <p>Al 0.45 0.13</p> <p>Zn 0.44 0.14</p> <p>Ca 0.32 0.07</p> <p>Total 100.00</p>	<p>Spectrum 18 Wt% Wt% Sigma</p> <p>O 45.49 1.16</p> <p>Fe 35.17 0.88</p> <p>C 16.87 1.56</p> <p>Si 1.40 0.15</p> <p>Al 0.78 0.16</p> <p>Ca 0.29 0.08</p> <p>Total 100.00</p>	<p>Spectrum 19 Wt% Wt% Sigma</p> <p>Fe 52.79 0.81</p> <p>O 47.21 0.81</p> <p>Total 100.00</p>
	<p>Spectrum 20 Wt% Wt% Sigma</p> <p>Fe 70.85 0.93</p> <p>O 29.15 0.93</p> <p>Total 100.00</p>	<p>Spectrum 21 Wt% Wt% Sigma</p> <p>Fe 45.89 0.98</p> <p>O 42.51 1.01</p> <p>C 11.08 1.43</p> <p>Si 0.52 0.13</p> <p>Total 100.00</p>	

The corrosion products are consisted mainly in iron oxides. Silicates are also present. The relatively high concentration of carbon in several points of the sample surface suggests that coatings might be also represented.

### Sample 5/8 - FTIR analysis



The spectrum shows that the main component of the corrosion is iron oxide. Weak peaks attributable to organic compound are also visible



This document was produced within the project ***Conservation of Art in Public Spaces (CAPuS)***.

Author:

Tea Zubin Ferri – Institute Materials Research Centre of Region of Istria METRIS (CROATIA)



**Education, Audiovisual and  
Culture Executive Agency**  
Erasmus+: Higher Education-Knowledge  
Alliances, Bologna Support, Jean Monnet

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Knowledge Alliances 2017, Project N°  
588082-EPP-A-2017-1-IT-EPPKA2-KA

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