

| | |
|---|-------------------|
| ARTIST: | Dora Kovačević |
| TITLE OF THE WORK and YEAR OF EXECUTION: | Zid / Wall (1985) |
| MATERIALS: | Painted steel |

| | Name and description of the sample | Analytical methods | Notes |
|---|--|---|-------|
| 1 | 22/1 - coatings (cross section) | Micro FTIR, Optical microscopy, SEM/EDS | |
| 2 | 22/2 - coatings | FTIR, Optical microscopy, SEM/EDS | |
| 3 | 22/3 - coatings | FTIR | |
| 4 | 22/4 - coatings | FTIR | |
| 5 | 22/5 - corrosion products | SEM/EDS | |
| 6 | 22/5 - corrosion products | FTIR | |

Description of the analytical methods, equipment and procedures:

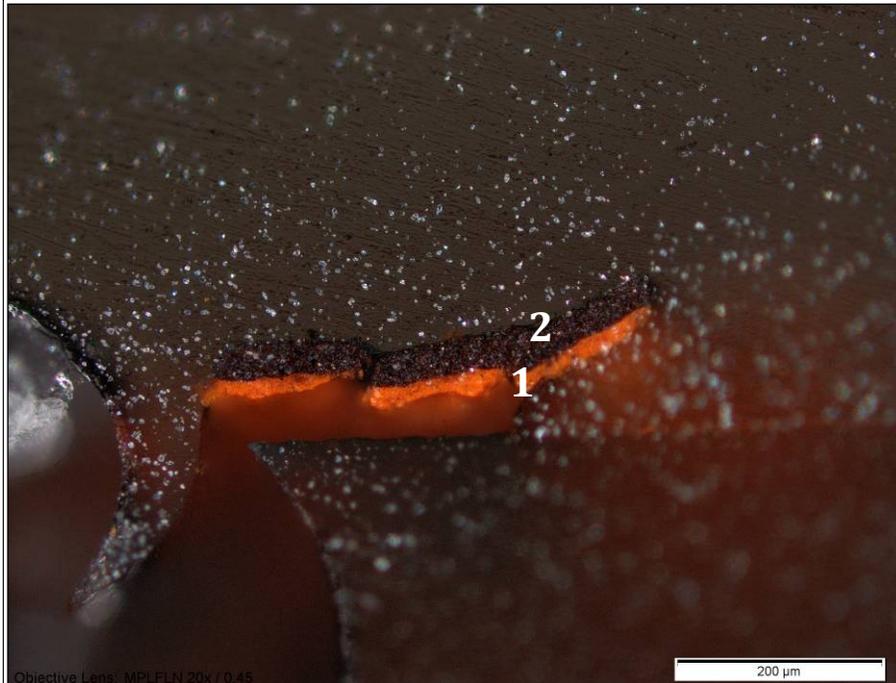
-Optical microscopy: analysis performed on sample or cross section using visible (VIS), ultraviolet (UV), polarized (POL) or infrared (IR) light depending on the characteristic of the observed sample. Observation and images taken from 50X to 1000X magnification. **Equipment used:** Optical microscopy 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⁻¹ in the range from 4000 to 400 cm⁻¹. 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⁻¹ in the range from 4000 to 600 cm⁻¹. **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.

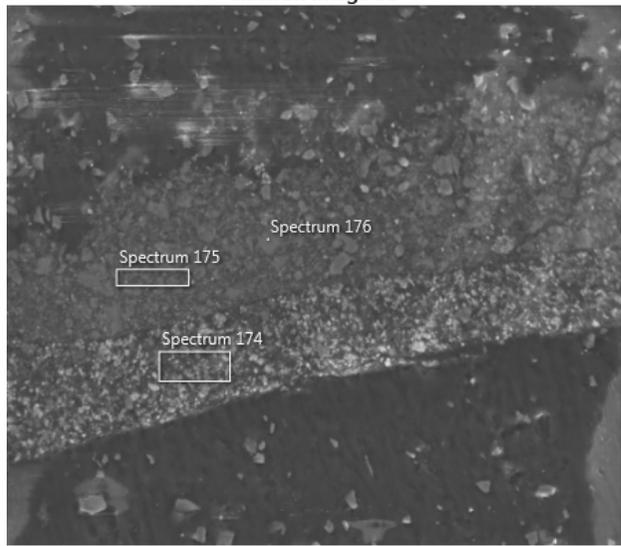
Results:
Sample 22/1



Sample 22/1 – optical microscopy (magnification 200X), SEM/EDS and micro FTIR analysis has shown the following structure and composition

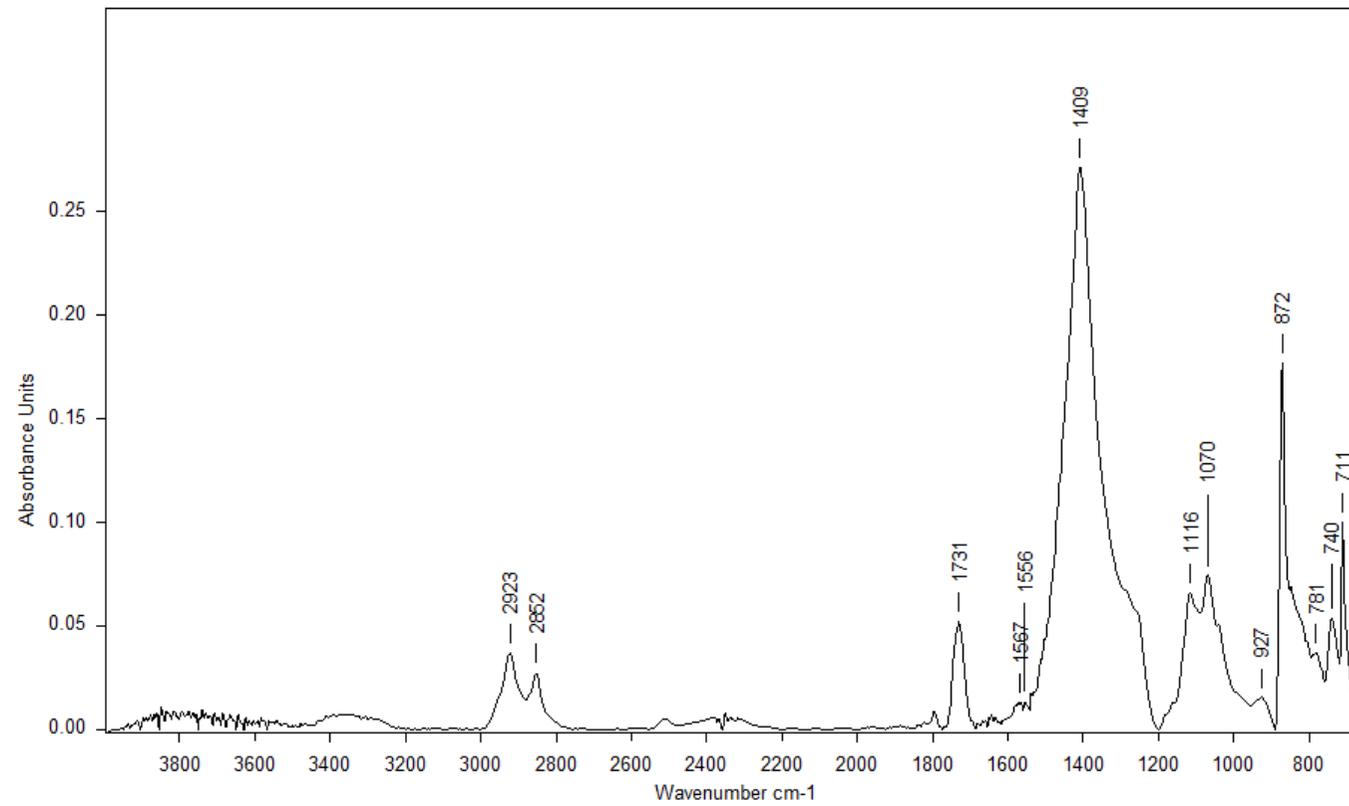
- 1- Red base coat, irregular thickness about 50 μm, containing alkyds, chalk and red ochre most probably. (Original base coat possibly missing?)
- 2- Black top coat of regular thickness about 50 μm, containing barite, chalk and alkyd binder. It was not possible to achieve a good micro FTIR spectrum of the layer.

Sample 22/1 - SEM/EDS analysis

| Electron Image 55 | | | | Spectrum 174 | | | Spectrum 175 | | | Spectrum 176 | | |
|---|--------|-----------|-------|--------------|-----------|-------|--------------|------|-----------|--------------|--|--|
| | Wt% | Wt% Sigma | | Wt% | Wt% Sigma | Wt% | Wt% Sigma | Wt% | Wt% Sigma | | | |
|  | | | | | | | | | | | | |
| C | 57.95 | 1.15 | O | 43.63 | 1.01 | O | 48.48 | 1.08 | | | | |
| O | 29.05 | 1.09 | C | 42.41 | 0.96 | C | 32.59 | 0.97 | | | | |
| Ba | 5.69 | 0.37 | Ca | 9.67 | 0.30 | Ca | 18.32 | 0.49 | | | | |
| S | 2.23 | 0.13 | Fe | 3.43 | 0.27 | Fe | 0.62 | 0.19 | | | | |
| Fe | 2.23 | 0.21 | Si | 0.86 | 0.11 | Total | 100.00 | | | | | |
| Cl | 2.05 | 0.13 | Total | 100.00 | | | | | | | | |
| Ca | 0.57 | 0.09 | | | | | | | | | | |
| K | 0.22 | 0.07 | | | | | | | | | | |
| Total | 100.00 | | | | | | | | | | | |

Sample 22/1- Micro FTIR analysis

22-1 RED LAYER



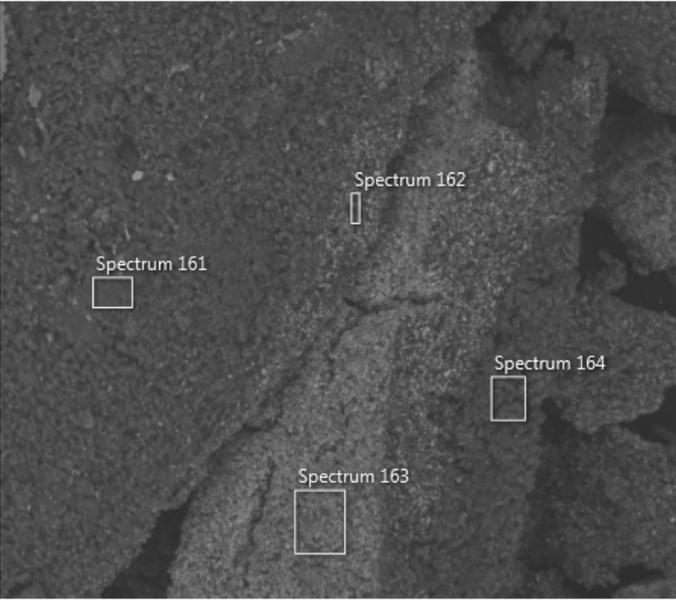
Results:
Sample 22/2



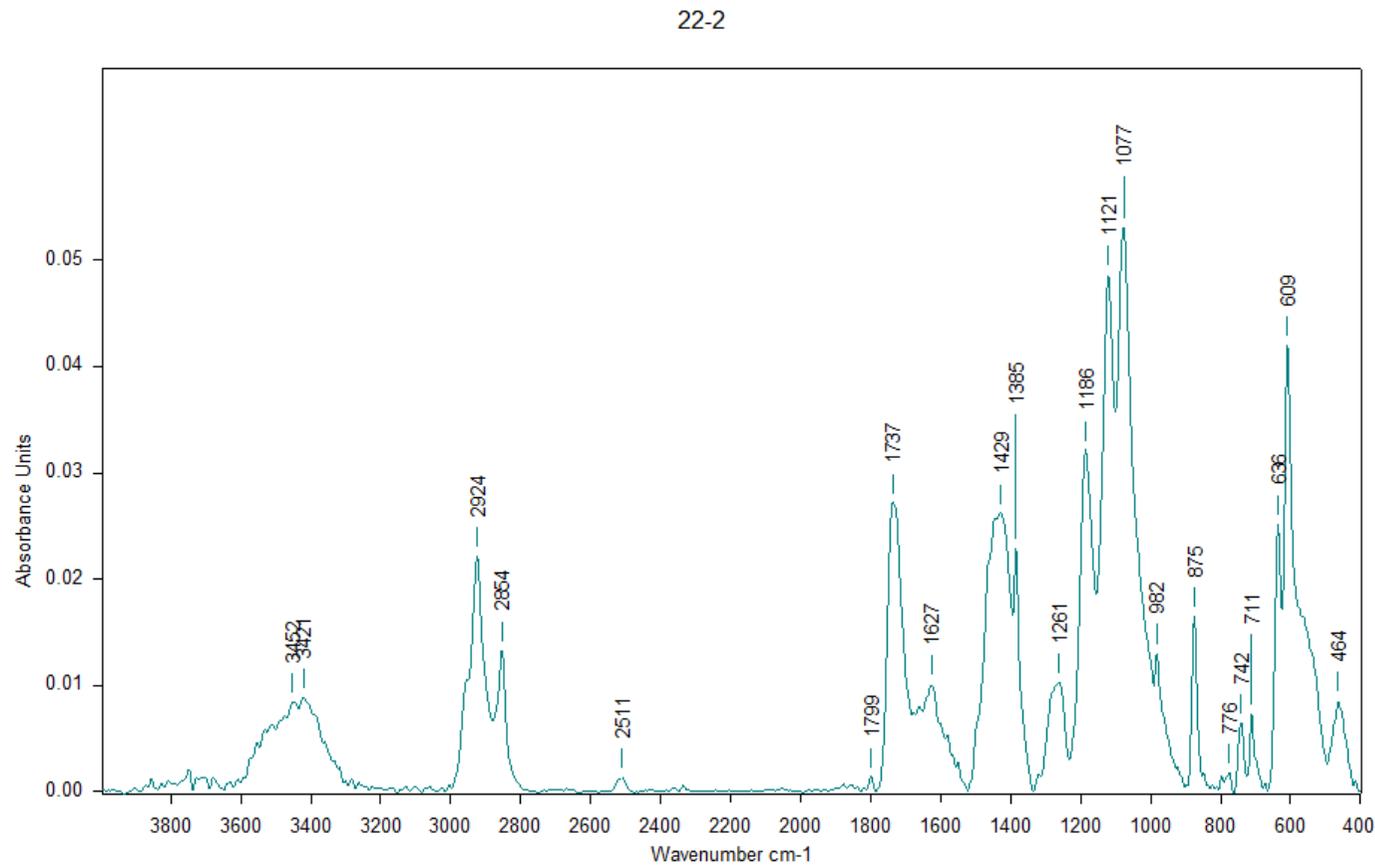
Sample 22/2 – optical microscopy (magnification 70X). Visually the same two layers of coatings (red base coat and black top coat) as in sample 22/1 were recognized.

The red base coat contains mostly chalk, red ochre and titanium oxide. Black top coat consists in barite, possibly zinc oxide and silicates. Both layers contain alkyds.

Sample 22/2 - SEM/EDS analysis

| Electron Image 52 | | | Spectrum 161 | Spectrum 162 | Spectrum 163 | Spectrum 164 | | | | | |
|---|--------|-----------|--------------|--------------|--------------|--------------|--------|------|-------|--------|------|
| | Wt% | Wt% Sigma | Wt% | Wt% | Wt% | Wt% | | | | | |
|  | | | | | | | | | | | |
| O | 44.45 | 0.86 | C | 45.60 | 0.64 | O | 32.89 | 0.89 | O | 36.82 | 1.06 |
| C | 29.32 | 0.80 | O | 28.00 | 0.57 | Ba | 26.90 | 0.73 | C | 28.90 | 0.96 |
| Ca | 16.40 | 0.35 | Ba | 12.90 | 0.30 | C | 17.12 | 1.23 | Ca | 21.68 | 0.51 |
| Fe | 9.44 | 0.35 | Fe | 3.93 | 0.16 | Fe | 10.98 | 0.44 | Fe | 10.85 | 0.43 |
| Ti | 0.40 | 0.11 | S | 3.54 | 0.10 | S | 6.01 | 0.23 | Ti | 1.02 | 0.15 |
| Total | 100.00 | | Cl | 2.45 | 0.08 | Cl | 2.21 | 0.16 | Cl | 0.40 | 0.09 |
| | | | Zn | 1.45 | 0.18 | Zn | 1.77 | 0.41 | S | 0.33 | 0.09 |
| | | | Si | 0.83 | 0.06 | Si | 1.18 | 0.14 | Total | 100.00 | |
| | | | Ca | 0.76 | 0.06 | Ca | 0.94 | 0.13 | | | |
| | | | Mg | 0.35 | 0.06 | Total | 100.00 | | | | |
| | | | Al | 0.20 | 0.05 | | | | | | |
| | | | Total | 100.00 | | | | | | | |

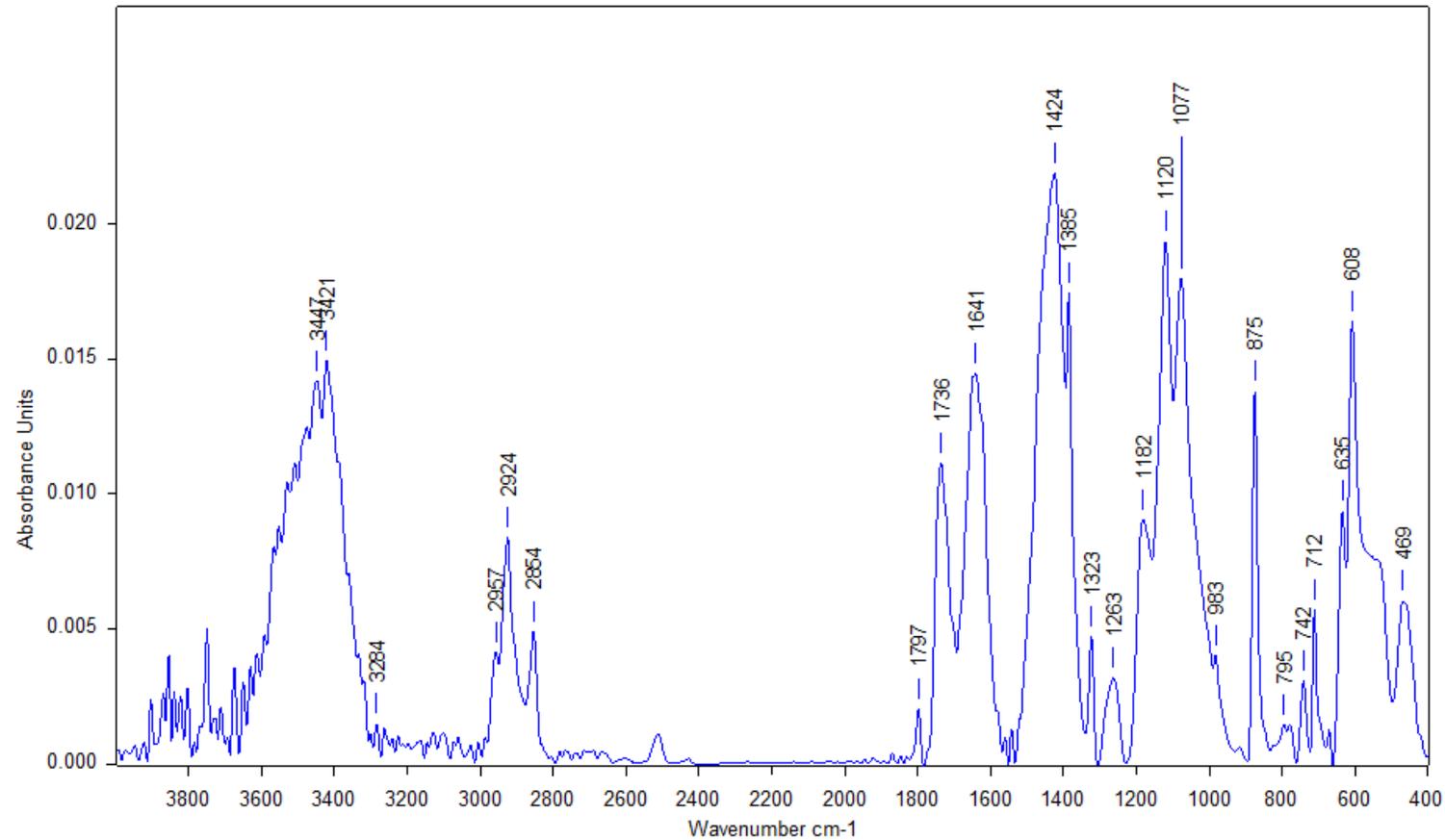
Sample 22/2 - FTIR analysis



In the obtained spectrum peaks assignable to alkyds, barite, chalk and nitrates are assignable. Weak peaks attributable to quartz are also present.

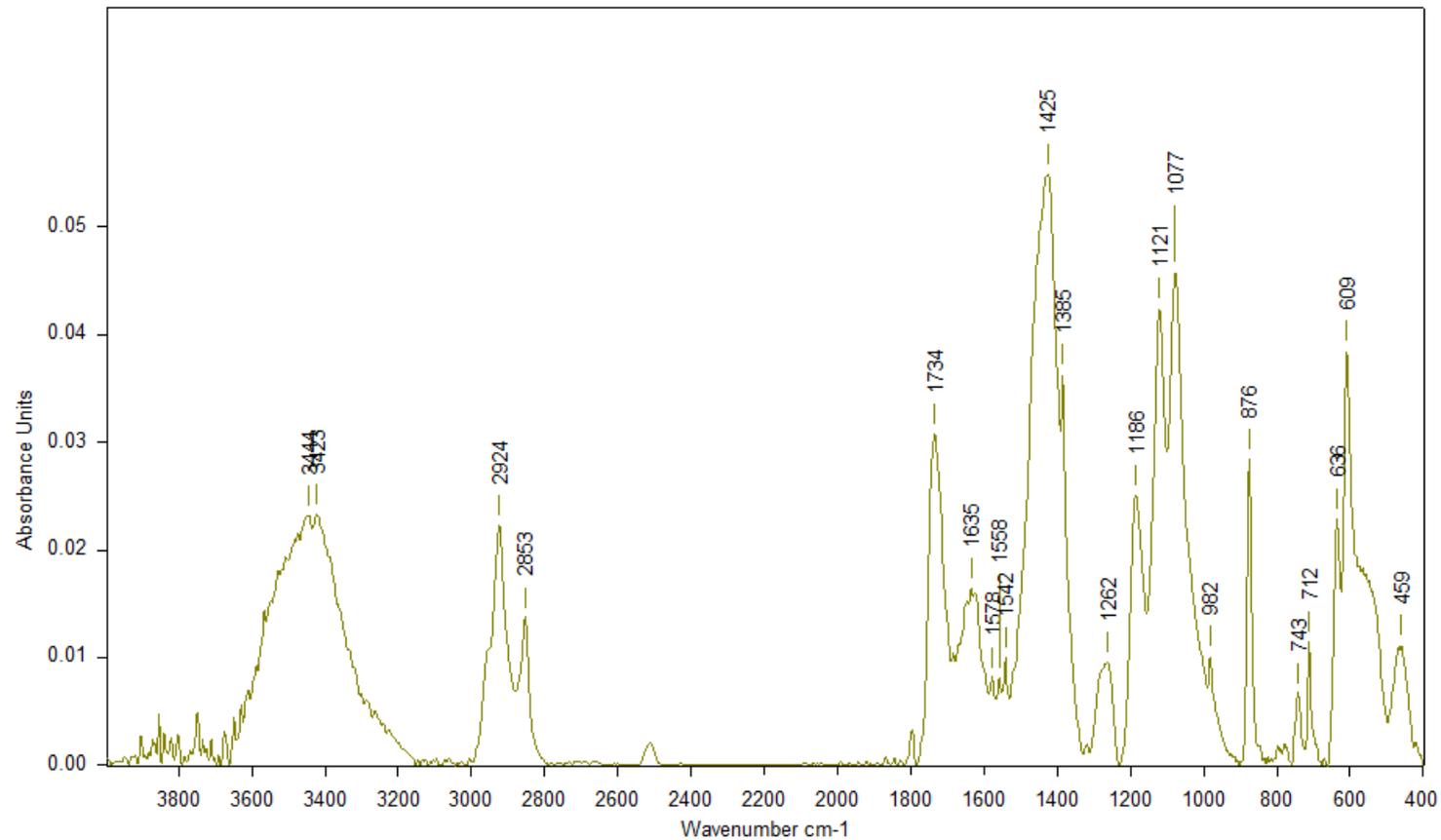
Sample 22/3 - FTIR analysis

22-3



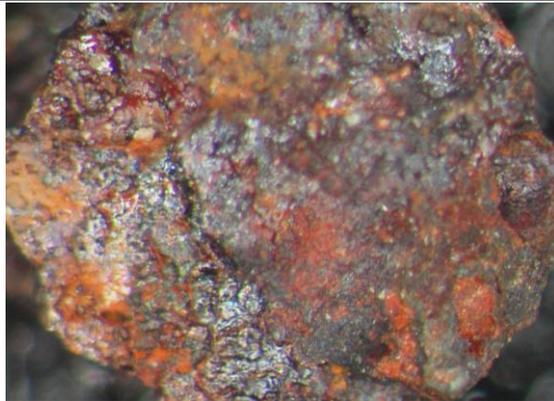
The sample was analysed as whole (all the layers included). In the obtained spectrum peaks assignable to alkyds, barite, chalk and nitrates are assignable. Weak peaks attributable to quartz are also present.

Sample 22/4 - FTIR analysis

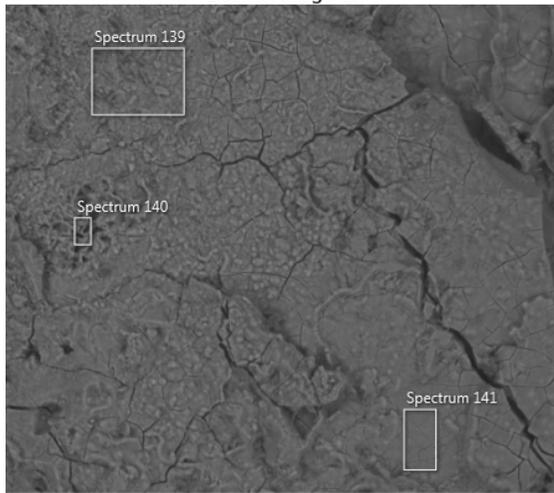


The sample was analysed as whole (all the layers included). In the obtained spectrum peaks assignable to alkyds, barite, chalk and nitrates are assignable. Weak peaks attributable to quartz are also present.

Sample 22/5 - SEM/EDS analysis



Electron Image 46



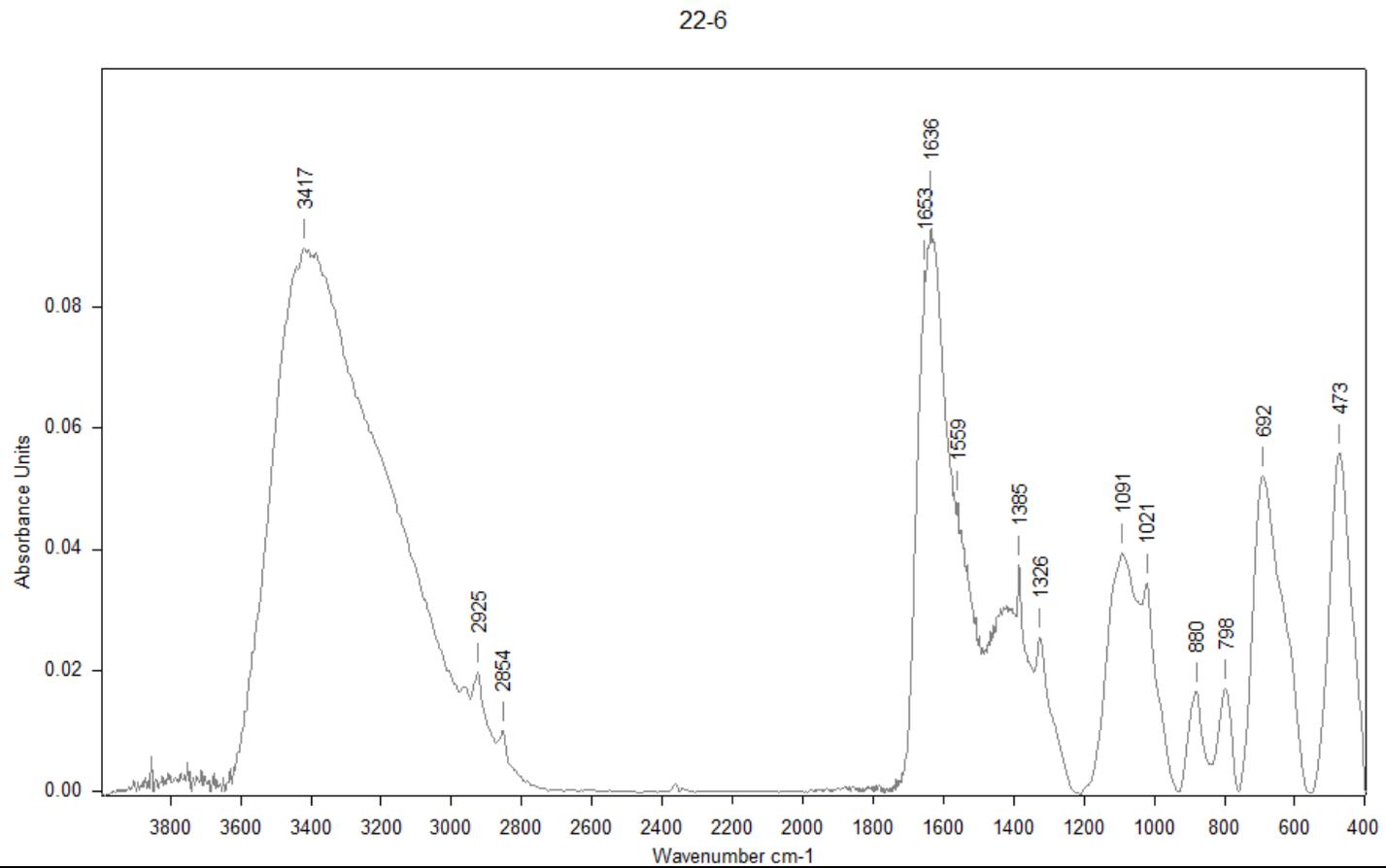
| Spectrum | Wt% | Wt% Sigma |
|----------|--------|-----------|
| 139 | | |
| O | 45.30 | 0.63 |
| Fe | 37.43 | 0.51 |
| C | 12.53 | 0.95 |
| Ca | 2.23 | 0.07 |
| P | 1.00 | 0.07 |
| Cl | 0.67 | 0.05 |
| K | 0.35 | 0.04 |
| Si | 0.32 | 0.06 |
| S | 0.18 | 0.04 |
| Total | 100.00 | |

| Spectrum | Wt% | Wt% Sigma |
|----------|--------|-----------|
| 140 | | |
| O | 43.82 | 0.93 |
| Fe | 38.81 | 0.80 |
| C | 11.23 | 1.49 |
| Ca | 1.62 | 0.09 |
| P | 1.26 | 0.11 |
| Na | 1.04 | 0.24 |
| Cl | 0.74 | 0.07 |
| K | 0.54 | 0.06 |
| Si | 0.37 | 0.08 |
| Al | 0.35 | 0.10 |
| S | 0.22 | 0.06 |
| Total | 100.00 | |

| Spectrum | Wt% | Wt% Sigma |
|----------|--------|-----------|
| 141 | | |
| Fe | 53.37 | 0.46 |
| O | 40.67 | 0.47 |
| P | 1.82 | 0.12 |
| Na | 1.63 | 0.26 |
| Ca | 1.16 | 0.08 |
| Cl | 0.69 | 0.08 |
| K | 0.65 | 0.07 |
| Total | 100.00 | |

The corrosion products are consisted mainly in iron oxides. The relatively high concentration of carbon and other elements in several points of the sample surface, suggests that coatings might be also represented in the taken sample.

Sample 22/6 - FTIR analysis



In the obtained spectrum peaks attributable to water are present. Peaks assignable to iron oxide, nitrates and silicates are also visible.

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