

ARTIST:	Branko Ružić
TITLE OF THE WORK and YEAR OF EXECUTION:	Vrata / Door (1984)
MATERIALS:	Painted steel

	Name and description of the sample	Analytical methods	Notes					
1	24/1 – coatings (cross section)	Optical microscopy, SEM/EDS, micro FTIR	Samples 24/1 and 24/2 were prepared as cross					
2	24/2 – coatings (cross section)	Optical microscopy, SEM/EDS, micro FTIR	sections in order to get the insight of the stratigraphy					
3	24/5 – corrosion products	SEM/EDS	by means of optical microscopy, micro FTIR and					
4	24/6 – corrosion products	FTIR	SEM/EDS analyses.					

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.

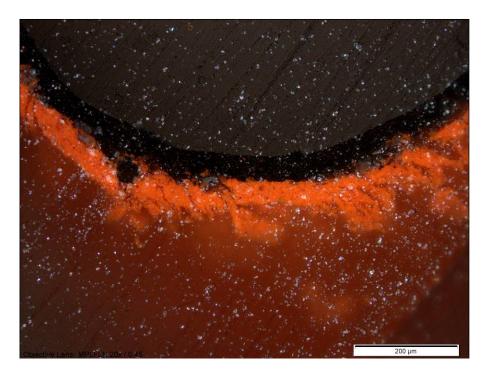
-Fourier Transform Infrared Spectroscopy (FTIR): analysis performed using KBr pellets preparation (2 mg sample + 120 mg Kbr). Each spectrum is a result of 64 scans taken at resolution of 4 cm⁻¹ in the range from 4000 to 400 cm⁻¹. Collected spectra were baseline corrected and when necessary smoothed according 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 distance10 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 24/1

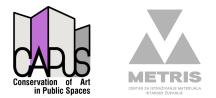


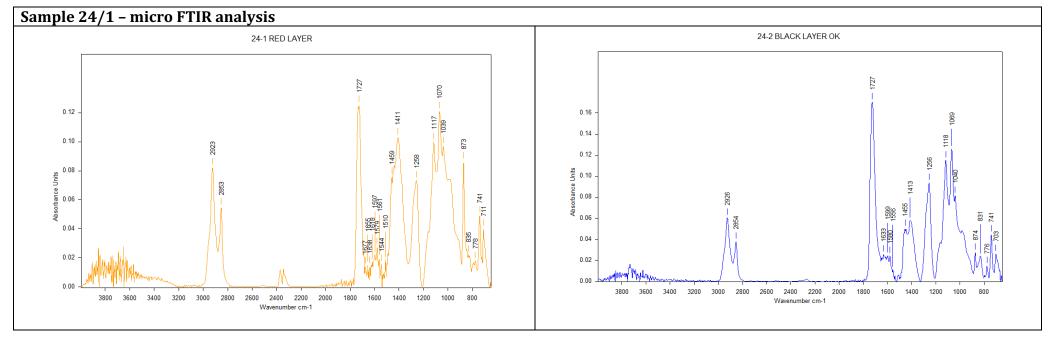
Sample 24/1 – optical microscopy (magnification 200X). The sample consists of two layers. The lower layer red and the upper one black. SEM/EDS and micro FTIR analysis has shown the following structure and composition

- 1- Red base coat, irregular thickness about 50 μm (by scratching off the sample the layer has been twisted), containing alkyds, chalk and red ochre most probably. Bottom-most layer of the base coat possibly missing since no metal support is visible in the taken sample. Particles consisting in zinc and oxygen are also present suggesting. Particles of titanium white have also been detected in this layer. The binder is alkyd resin.
- 2- Black top coat of regular thickness about 70 µm, consisting of an unidentified organic black pigment and alkyd resin.



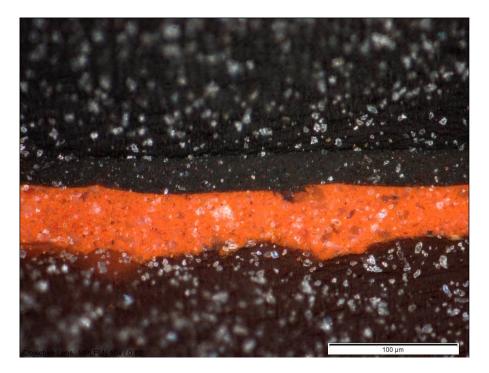
Sample 24/1 – SEM/EDS analysis												
Electron Image 56	Spectrum 177	Wt%	Wt% Sigma	Spectrum 178	Wt%	Wt% Sigma	Spectrum 179	Wt%	Wt% Sigma	Spectrum 180	¹ Wt%	Wt% Sigma
	0	45.78	0.90	Zn	37.73	1.52	Ti	67.27	1.18	С	97.89	0.26
A Contraction of the second	С	34.69	0.84	0	31.71	1.37	С	32.18	1.19	Ca	1.66	0.22
Spectrum 180	Ca	15.48	0.36	С	23.37	2.17	Ca	0.55	0.14	Si	0.46	0.14
	Fe	3.72	0.26	Na	5.18	1.32	Total	100.00)	Total	100.00)
Spectrum 179	Si	0.33	0.08	Κ	0.77	0.16						
spectrum 179	Total	100.00)	Ca	0.73	0.18						
				Cl	0.50	0.14						
Spect&practRom 177				Total	100.00)						







Sample 24/2



Sample 24/2 – optical microscopy (magnification 500X). The sample consists of two layers. The lower layer red of thickness about 80 μm, and the upper one black with thickness about 50 μm. SEM/EDS and micro FTIR analysis has shown the following structure and composition

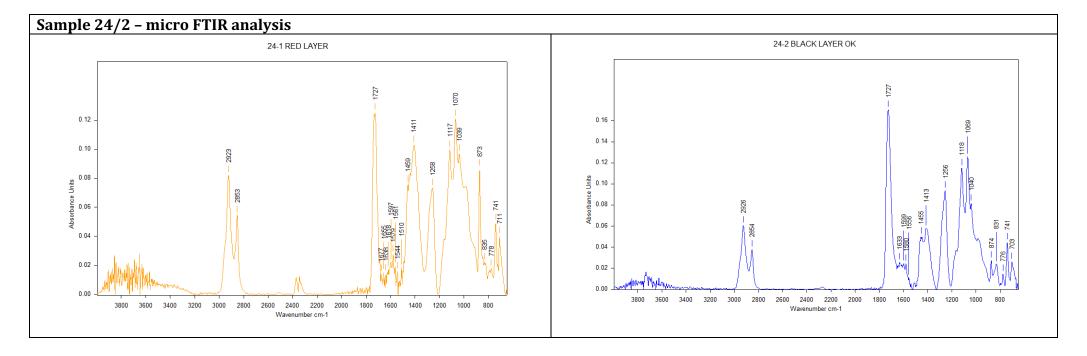
- 1- Red base coat, irregular thickness about 70 μm (by scratching off the sample the layer has been twisted), containing alkyds, chalk and red ochre most probably. Bottom-most layer of the base coat possibly missing since no metal support is visible in the taken sample.
- 2- Black top coat of regular thickness about 50 μm containing barite, chalk and alkyd binder. The black colour is most probably given by carbon black.

Obtained micro FTIR spectra of the layers are affected by signals of nearby areas.



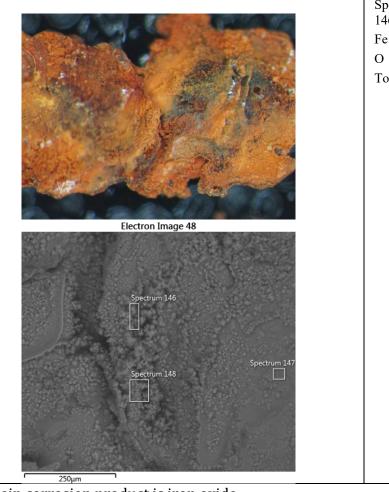
Sample 24/2 – SEM/EDS analysis											
Electron Image 57	Spectrum 181	Wt%	Wt% Sigma	Spectrum 182	Wt%	Wt% Sigma	Spectrum 183	Wt%	Wt% Sigma		
	0	41.30	1.00	0	55.10	1.11	С	69.18	0.98		
	С	37.21	0.94	Si	34.67	0.79	0	25.91	1.00		
	Ca	12.30	0.34	С	10.23	1.50	Ca	2.08	0.13		
the second s	Fe	8.19	0.37	Total	100.00)	Si	1.27	0.10		
Spectrum 181	Ti	0.58	0.12				Ba	1.05	0.24		
	Si	0.41	0.10				S	0.51	0.08		
Spectrum 182 Spectrum 183	Total	100.00)				Total	100.00)		







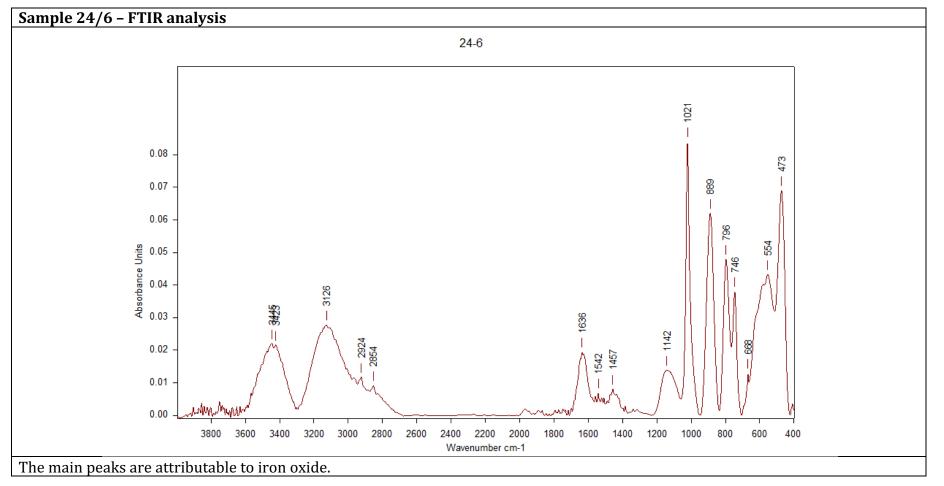
Sample 24/5 - SEM/EDS analysis



Spectrum 146	Wt%	Wt% Sigma	Spectrum 147	Wt%	Wt% Sigma	Spectrum 148	Wt%	Wt% Sigma
Fe	64.64	0.64	Fe	52.04	0.62	Fe	67.95	0.78
0	35.36	0.64	0	41.17	0.58	0	32.05	0.78
Total	100.00)	С	6.80	0.80	Total	100.00	
			Total	100.00				

The main corrosion product is iron oxide.









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