![Swedish National Heritage Board logo] SWEDISH NATIONAL HERITAGE BOARD
RIKSANTIKVARIEÄMBETET

Datum: 2022-09-12
Dnr: RAÄ-2021-1228
ÖLM 2021.34
Handläggare: Kaj Thuresson

Instrument Report: μ- XRF, Microscopy, SEM/EDS

Sample Identification Code
RAÄ Dnr RAÄ-2021-1228   **Object no. OLM-39358-771 and OLM-39358-772**
Sample
Description of sample: Small dark chips/pieces with a powdery white substance on surface.
Material: Leather

Point of analysis

Picture 1. The two samples with their own Eppendorf tube

Purpose

Investigation of the composition of the powdery substance on the leather.

Method

Sample preparation   The whole sample was at first analysed with μ-XRF. A smaller portion of each sample was then taken with a scalpel and transferred to carbon tape, mounted on a SEM-stub, for investigation with optical light microscopy and SEM/EDS.

Instrument Parameters μXRF

[X] μXRF Artax 800, Mo X-ray tube with polycapillary lens, Bruker; Berlin, Germany

    [X] single point analysis (spot size <100μm)

    [ ] line scan (lateral resolution <100μm)

    [ ] elemental 2D mapping

    [ ] quantification, MQuant Calib, Bruker; Berlin, Germany

    [ ] quantification with standards

Voltage 50 kV

Current 600 µA
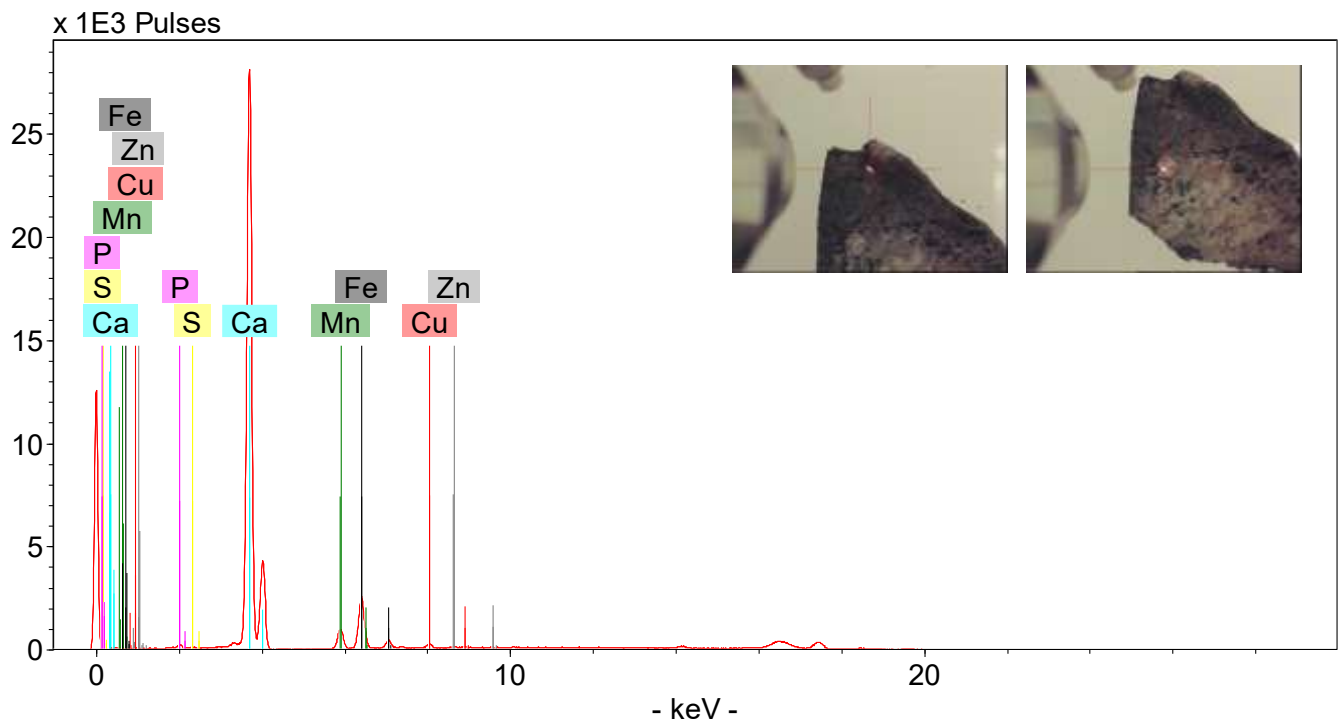
Scan time per point 10 s

Number of measurement points 8 + 8

Filter [X] no filter [ ] Al 315 µm [ ] Mo 12.50 µm [ ] other _____

Lens 0.060

Atmosphere [X] air [ ] He for light element detection

Qualitative Results µ-XRF

x 1E3 Pulses

Picture 2. Sample OLM-39358-771 was analysed at eight µ-points. All spectra were comparable (no difference between light or dark areas on sample). Mostly calcium (Ca), small amounts of iron (Fe) and manganese (Mn), and possibly phosphor (P), were detected in addition to trace amounts of copper (Cu), zinc (Zn) and sulfur (S).
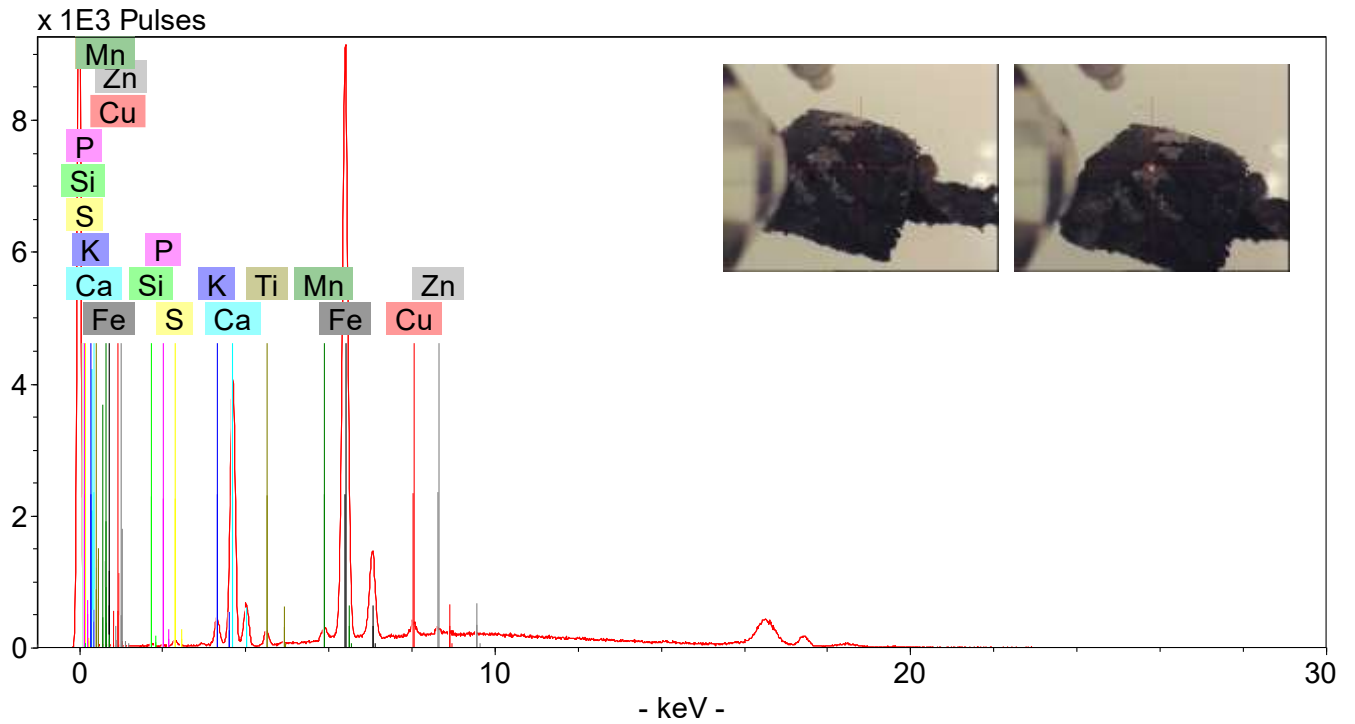
Picture 3. Sample OLM-39358-772 was analysed at eight µ-points. All spectra were comparable (no difference between light or dark areas on sample). Mostly iron (Fe) and calcium (Ca), small amounts of manganese (Mn), copper (Cu), potassium (K) and titanium (Ti) were detected, in addition to trace amounts of zinc (Zn), sulfur (S) and phosphor (P).

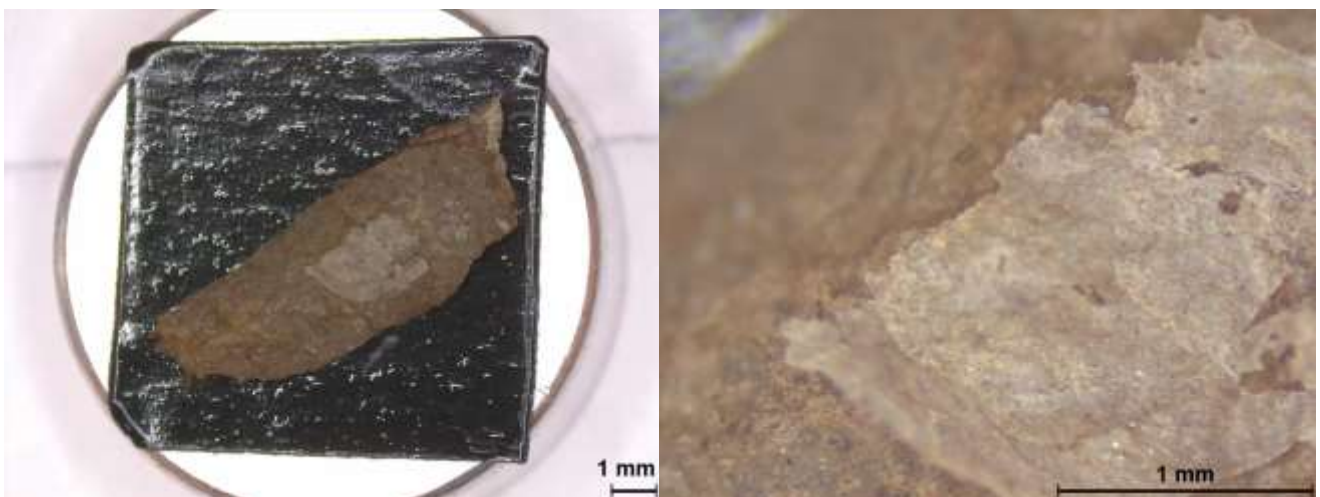![Swedish National Heritage Board - Riksantikvarieämbetet logo]

Microscopy results

Microscope used: LEICA S9i

External light source: VWR VisiLight™ CL-150
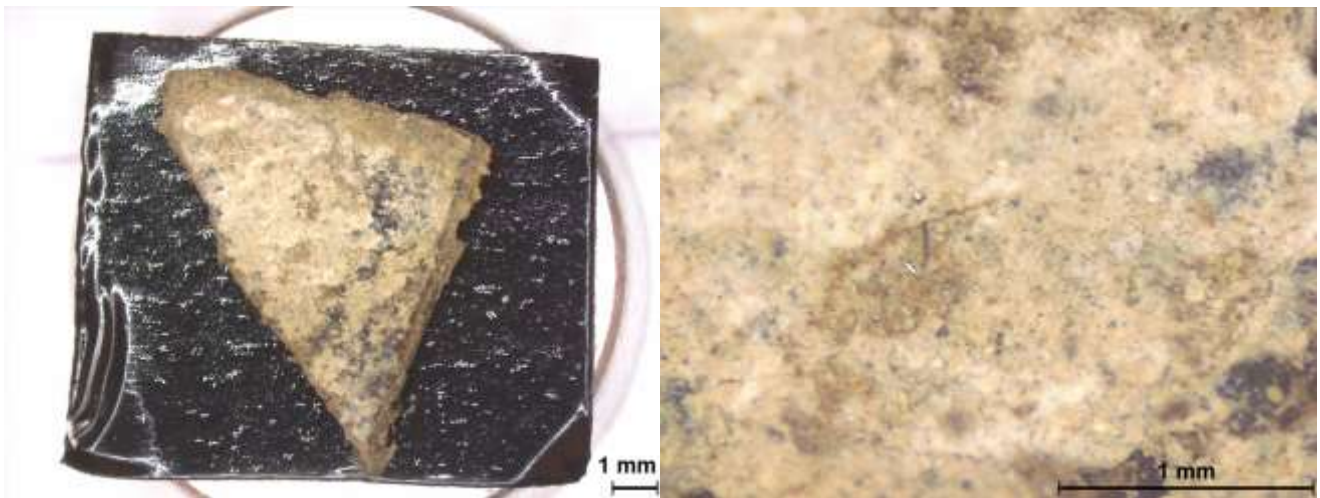
Picture 4. OLM-39358-771 sample on carbon tape in two different magnifications.
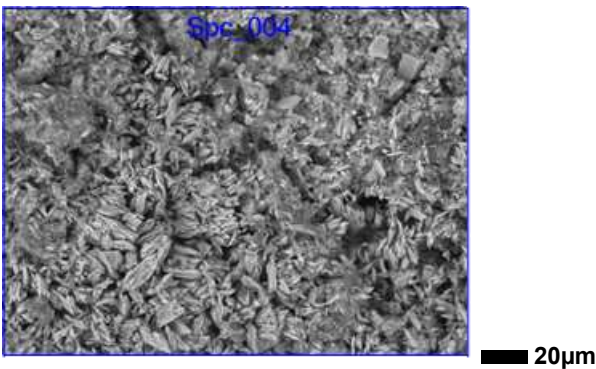

Picture 5. OLM-39358-772 sample on carbon tape in two different magnifications

SEM/EDS results

Instrument used: JEOL JSM-IT500

▬ **20mm**

Sample OLM-39358-771 and OLM-39358-772 in SEM chamber

▬ **20μm**

Signal BED-S

Landing Voltage 15.0 kV

WD 10.9 mm

Magnification x600

Vacuum Mode Low Vacuum

Items	Value
measurement conditions	
Acceleration voltage	15.00 kV
Probe current	-
Magnification	x 600
Process time	T2
Measurement detector	First
Live time	58.85 seconds
Real time	60.09 seconds
Dead time	2.00
Count rate	4713.00 CPS

Element	Line	Mass%	Atom%
O	K	50.67±0.26	70.98±0.36
Si	K	3.39±0.06	2.70±0.05
P	K	3.84±0.06	2.78±0.04
Ca	K	42.11±0.23	23.55±0.13
Total		100.00	100.00
Spc_004			Fitting ratio 0.6814
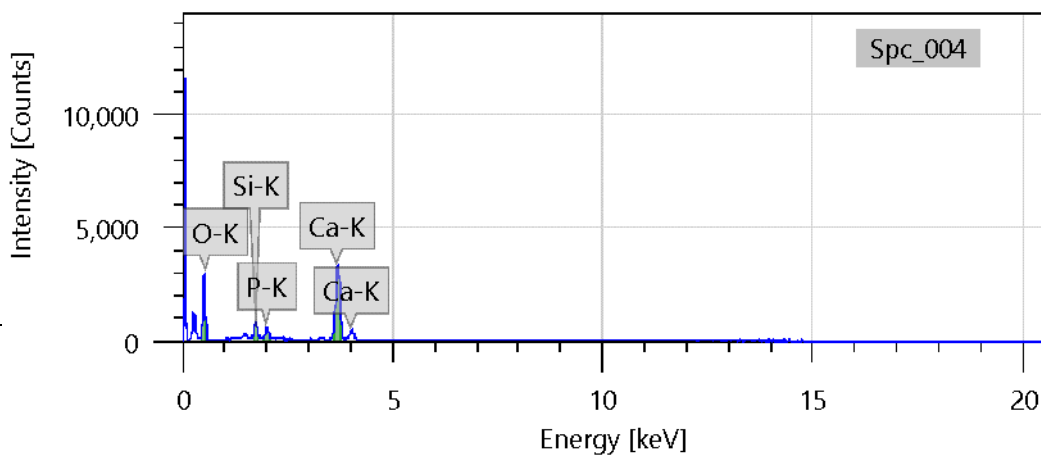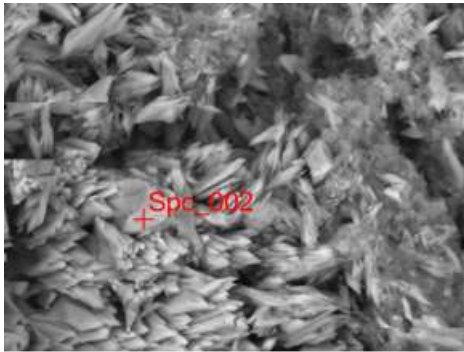
10 µm

Signal BED-S

Landing Voltage 15.0 kV

WD 10.9 mm

Magnification x600

Vacuum Mode Low Vacuum

Items	Value
measurement conditions	
Acceleration voltage	15.00 kV
Probe current	-
Magnification	x 2000
Process time	T2
Measurement detector	First
Live time	38.20 seconds
Real time	39.03 seconds
Dead time	2.00
Count rate	4846.00 CPS

Element	Line	Mass%	Atom%
O	K	47.87±0.29	69.54±0.42
P	K	1.40±0.05	1.05±0.03
Ca	K	50.73±0.27	29.41±0.15
Total		100.00	100.00
Spc_002			Fitting ratio 0.5913
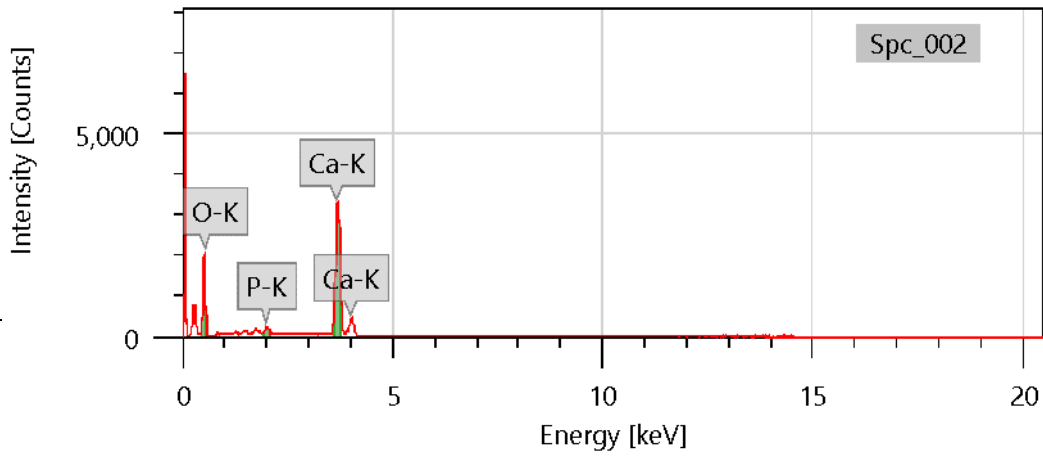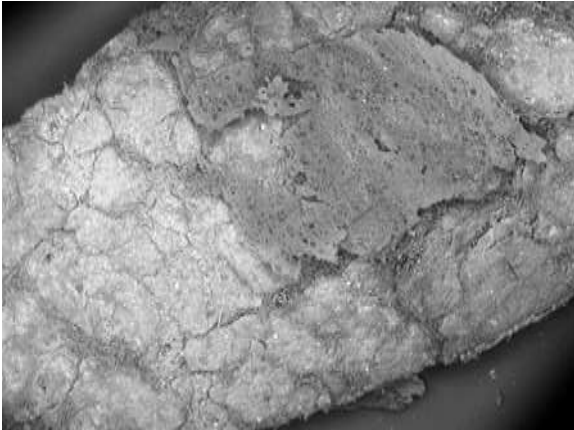
Signal BED-S

Landing Voltage 15.0 kV

WD 14.0 mm

Magnification x30

F.O.V. 4.267 x 3.200 mm

Probe Current Std. 65.0

Scan Rotation 191.4°

Vacuum Mode Low Vacuum

Pressure 36 Pa

◼━━ **500 µm**
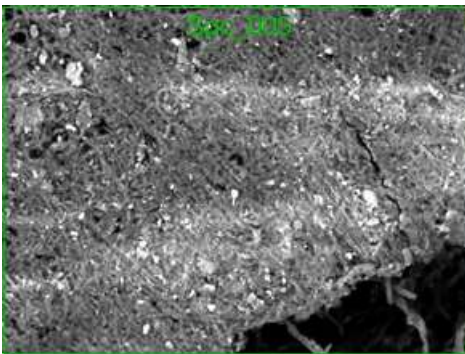
◼━ **20 µm**

Signal BED-S

Landing Voltage 15.0 kV

WD 13.8 mm

Magnification x600

Vacuum Mode Low Vacuum

Items	Value
measurement conditions	
Acceleration voltage	15.00 kV
Probe current	-
Magnification	x 600
Process time	T2
Measurement detector	First
Live time	43.92 seconds
Real time	44.78 seconds
Dead time	2.00
Count rate	4610.00 CPS

Element	Line	Mass%	Atom%
C	K	52.81±0.10	60.69±0.12
O	K	43.95±0.19	37.92±0.17
Si	K	1.89±0.04	0.93±0.02
Ca	K	1.36±0.04	0.47±0.02
Total		100.00	100.00
Spc_005			Fitting ratio 0.5666