



IRP

INSTITUTO DE
RESTAURACIÓN DEL
PATRIMONIO
INSTITUTO UNIVERSITARIO DE INVESTIGACIÓN

FESEM-EDX study of the painted surface of the Mikado 141F engine

R+D Project AICO/2021/095

“Developing strategies against climate change for the preventive conservation of Valencian railway-tramway heritage”

Financial support by *Consellería de Innovación, Universidades, Ciencia y Sociedad Digital. Dirección General de Ciencia e Investigación. Subvenciones para la realización de proyectos I+D+i desarrollados por grupos de investigación consolidados 2021*

Authors:

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Miguel F. Silva

Date:

Valencia, 11th July 2022

Instrumentation:**Instrumentation:**

Field emission scanning electron microscope coupled to a focused ion beam Zeiss (Orsay Physics Kleindiek Oxford Instruments) model Auriga compact equipment with an Oxford-X Max system controlled by Aztec software.

Working conditions:

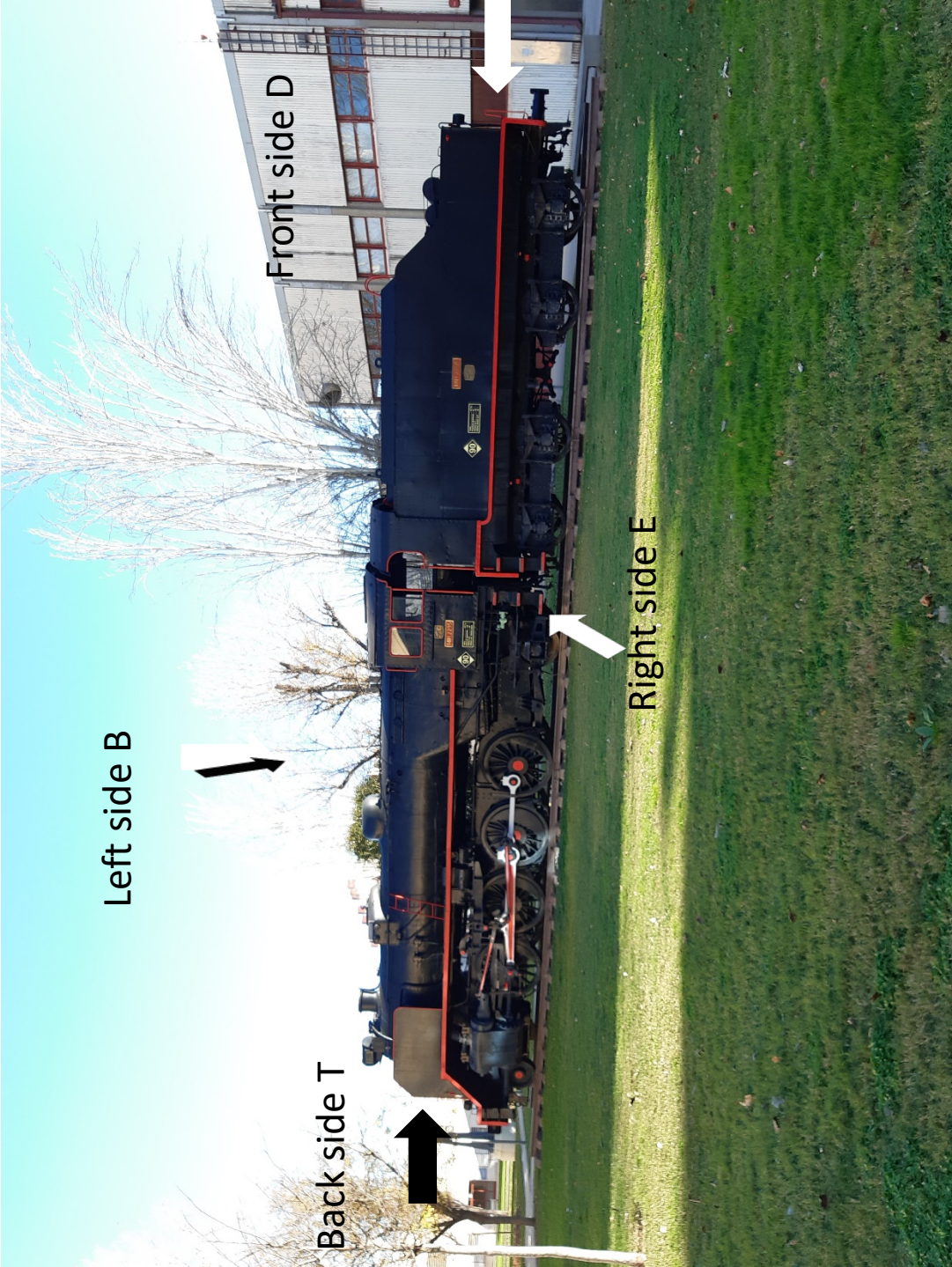
FESEM: voltage of 3 kV for acquiring secondary electron images.

FESEM-EDX: voltage of 20 kV, working distance of 6–7 A mm were applied.

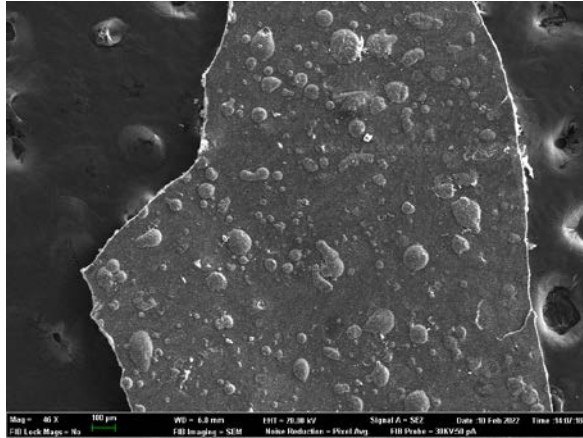
ZAF method to correct interelemental effects in semiquantitative microanalysis was carried out.

Counting time of 100 s.

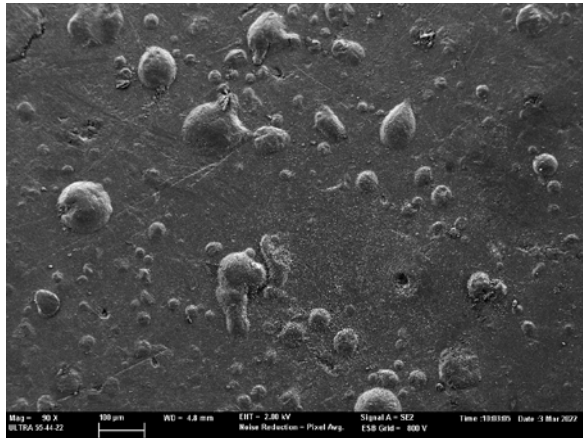
Map of the engine:



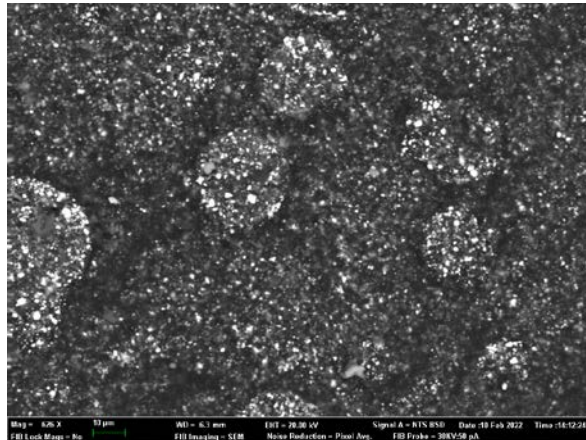
Sample: black paint
Ref: MIK-2
Sampling point: side E



Secondary electron image of the surface of the paint



Secondary electron image of the surface of the paint

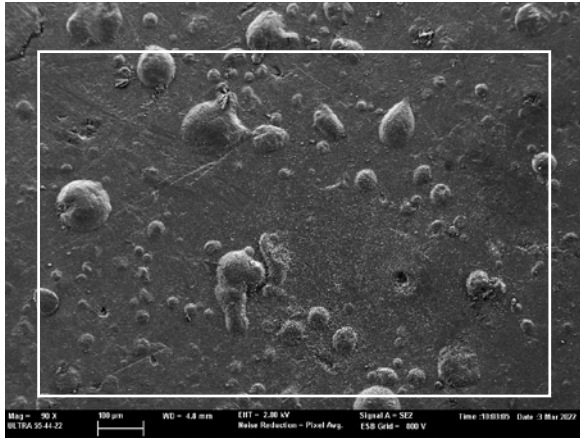


Backscattered electron image of the surface of the paint

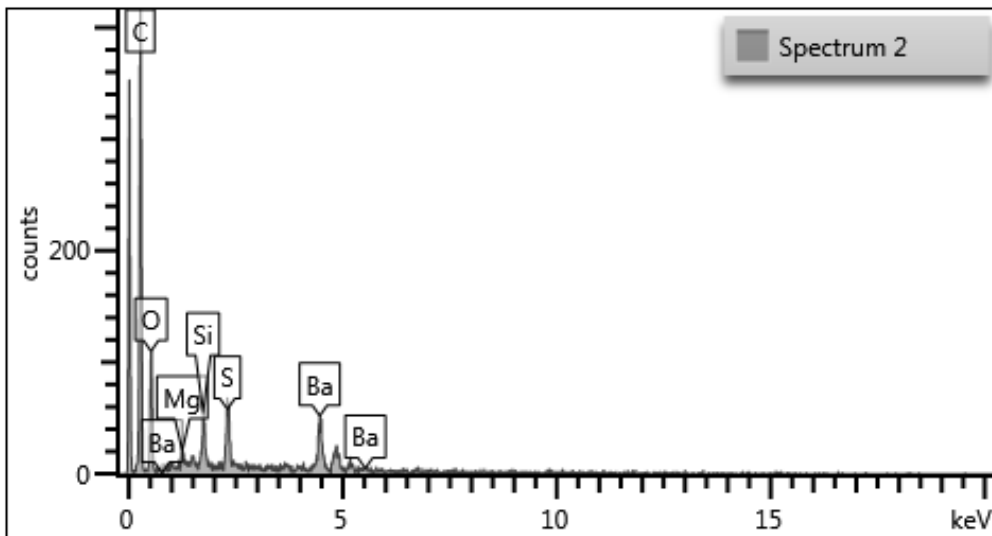
Sample: black paint
Ref: MIK-2
Sampling point: side E



Acquisition
 area



X-ray spectrum

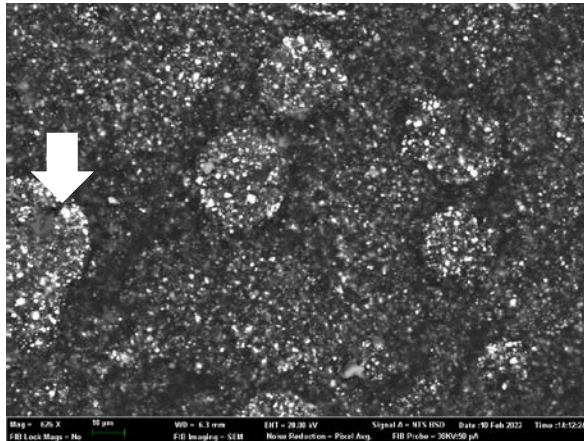


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O	33,6		65,77		
Mg	3,24	0,84	4,17	5,36	1,4
Si	7,77	0,9	8,66	16,61	1,92
S	11,71	0,96	11,44	29,25	2,4
Ba	43,69	2,02	9,96	48,77	2,26
Total:	100		100	100	

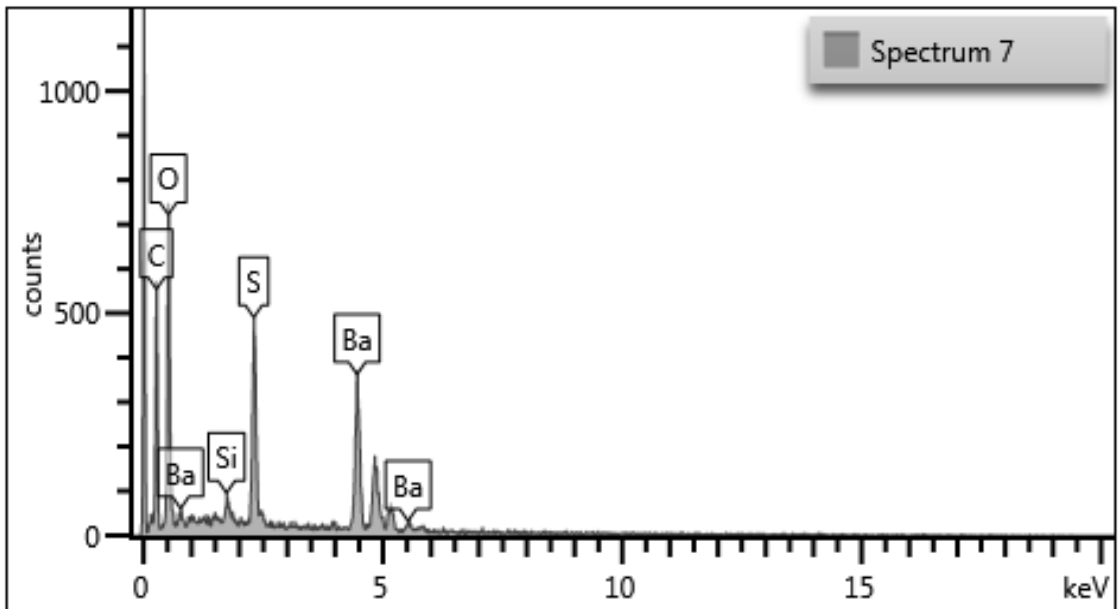
Sample: black paint
Ref: MIK-2
Sampling point: side E



Acquisition
point



X-ray spectrum

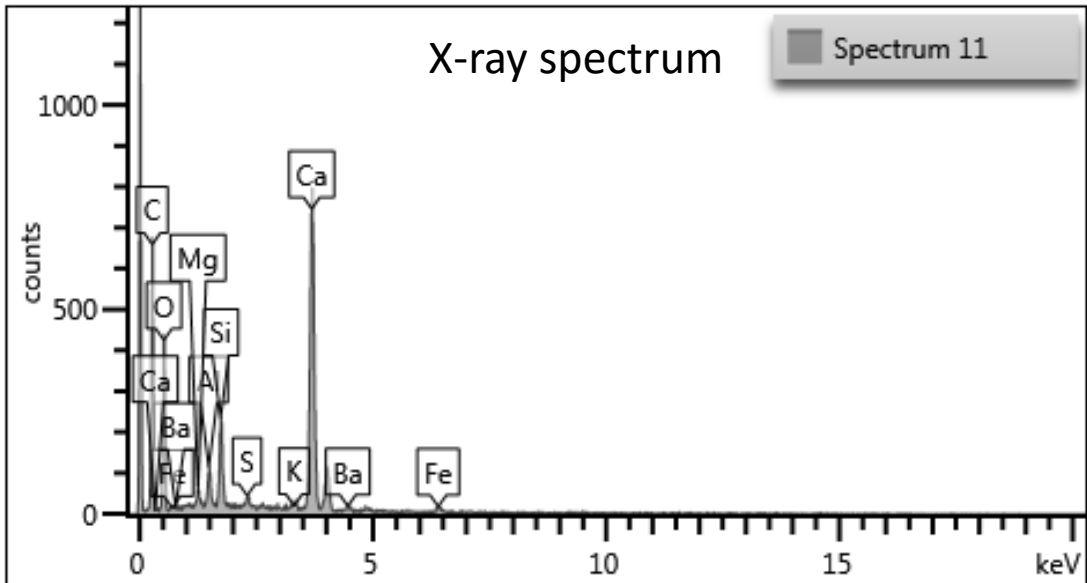
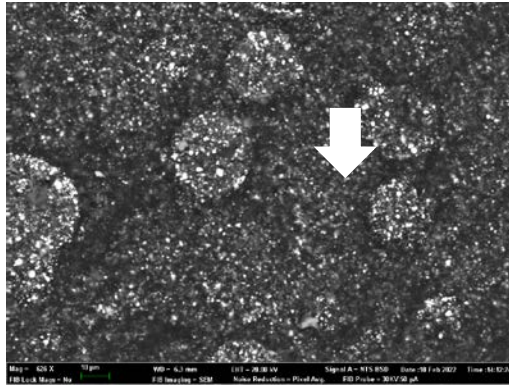


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O	29,47		67,24		
Si	1,48	0,24	1,92	3,17	0,51
S	14,3	0,4	16,28	35,71	1,01
Ba	54,75	0,72	14,55	61,13	0,81
Total:	100		100	100	S/Ba = 1,1189

Sample: black paint
Ref: MIK-2
Sampling point: side E

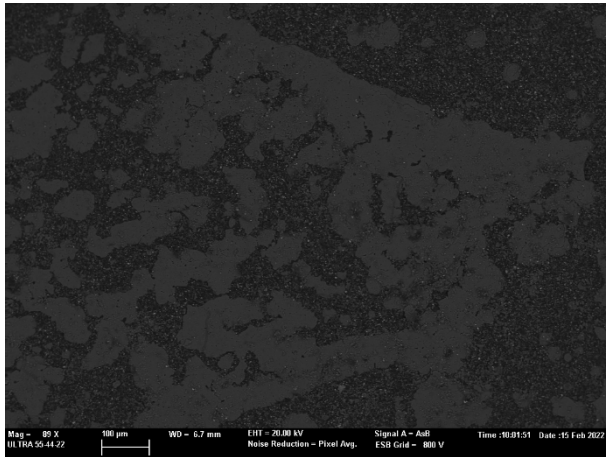


Acquisition
point

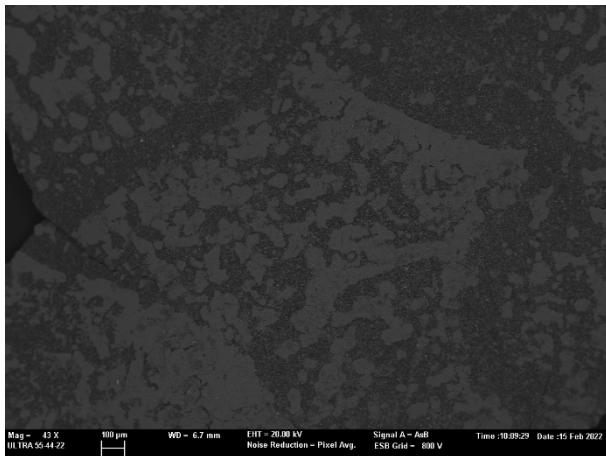


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O	34,13		55,04		
Mg	1,39	0,27	1,48	2,31	0,44
Al	3,57	0,29	3,42	6,75	0,54
Si	7,68	0,34	7,06	16,44	0,73
S	1,02	0,2	0,82	2,55	0,49
K	0,99	0,2	0,65	1,19	0,24
Ca	46,58	0,74	29,99	65,17	1,03
Fe	2,43	0,4	1,12	3,12	0,52
Ba	2,21	0,67	0,41	2,46	0,75
Total:	100		100	100	

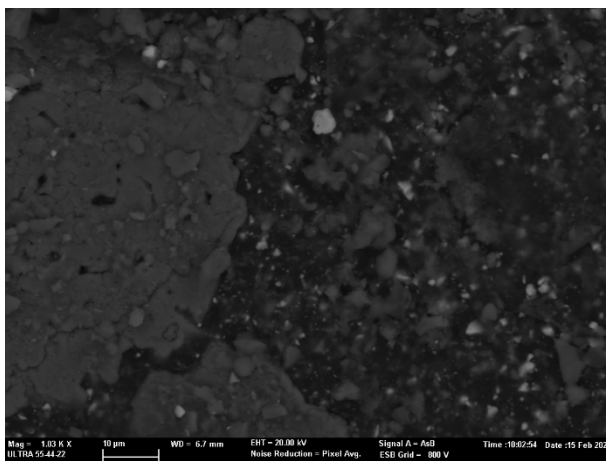
Sample: black paint
Ref: MIK-6
Sampling point: side E



Backscattered electron image of the surface of the paint



Backscattered electron image of the surface of the paint

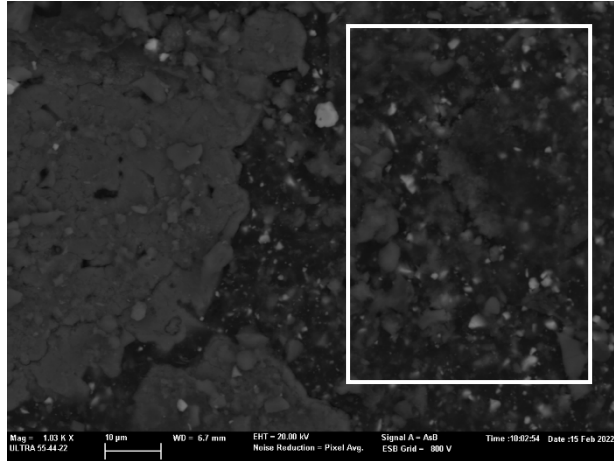


Backscattered electron image. Detail of the above image of the surface of the paint

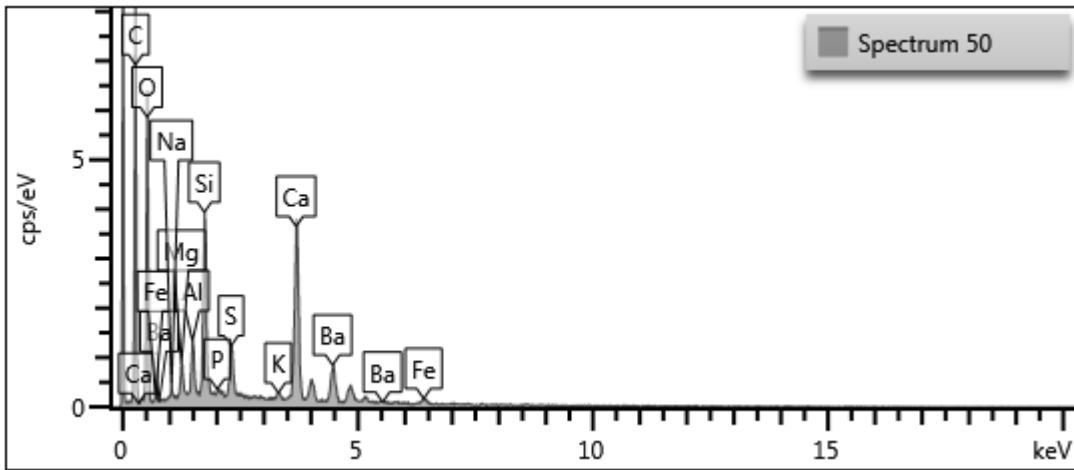
Sample: black paint
Ref: MIK-6
Sampling point: side E



Acquisition
 area



X-ray spectrum

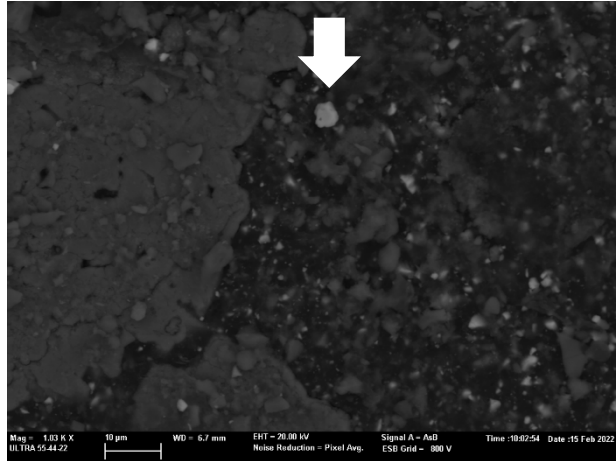


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		36,79		60,02		
Na	K series	0,64	0,19	0,73	0,86	0,25
Mg	K series	2,66	0,19	2,86	4,42	0,32
Al	K series	4,03	0,2	3,9	7,62	0,37
Si	K series	12,5	0,28	11,61	26,74	0,59
P	K series	0,26	0,14	0,22	0,6	0,33
S	K series	4,13	0,18	3,36	10,31	0,46
K	K series	0,66	0,12	0,44	0,8	0,15
Ca	K series	19,79	0,34	12,89	27,69	0,47
Fe	K series	1,61	0,25	0,75	2,08	0,32
Ba	L series	16,92	0,53	3,22	18,89	0,6
Total:		100		100	100	

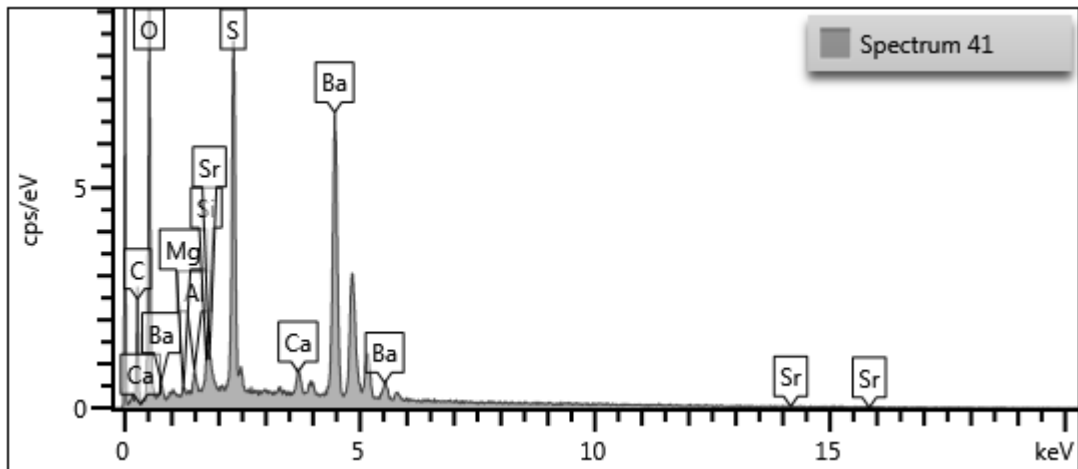
Sample: black paint
Ref: MIK-6
Sampling point: side E



Acquisition
point



X-ray spectrum

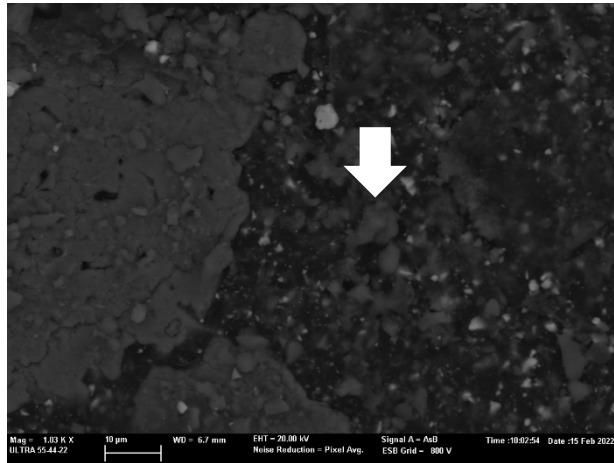


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		28,39		65,79		
Mg	K series	0,28	0,09	0,42	0,46	0,14
Al	K series	0,88	0,09	1,21	1,66	0,17
Si	K series	1,08	0,11	1,43	2,31	0,24
S	K series	12,78	0,18	14,77	31,91	0,45
Ca	K series	1,15	0,08	1,06	1,61	0,12
Sr	L series	2,27	0,3	0,96	2,69	0,35
Ba	L series	53,16	0,38	14,35	59,36	0,43
Total:		100		100	100	S/Ba = 1,03

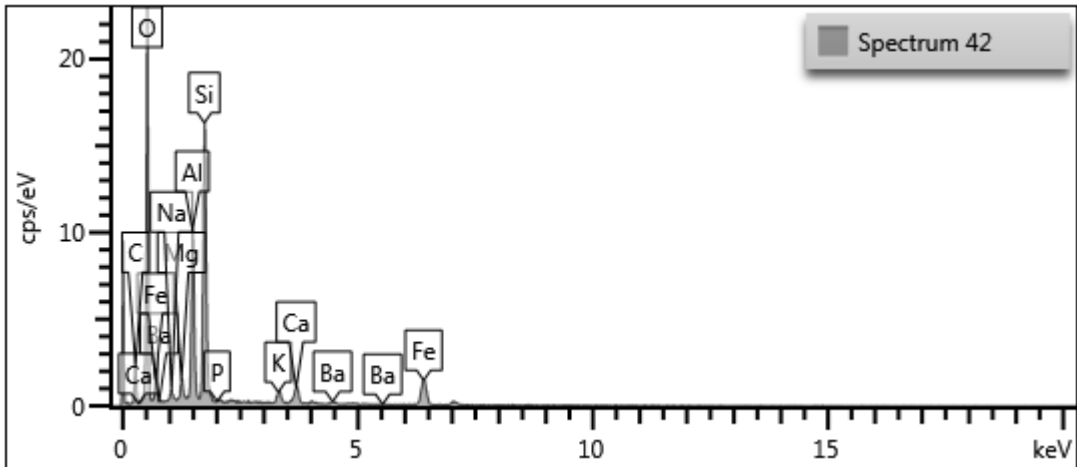
Sample: black paint
Ref: MIK-6
Sampling point: side E



Acquisition
point

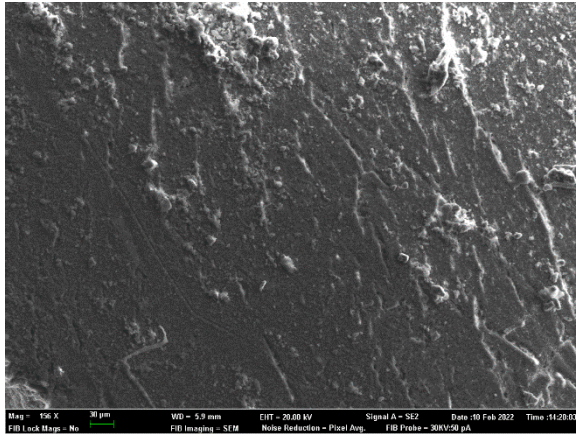


X-ray spectrum

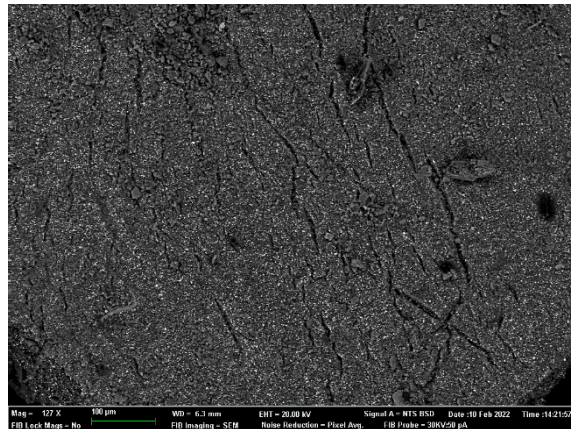


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		44,78		61,62		
Na	K series	0,83	0,15	0,79	1,12	0,2
Mg	K series	1,74	0,13	1,58	2,89	0,21
Al	K series	13,34	0,23	10,89	25,21	0,43
Si	K series	23,69	0,29	18,57	50,67	0,63
P	K series	0,08	0,09	0,05	0,17	0,22
K	K series	1,64	0,11	0,92	1,97	0,13
Ca	K series	2,3	0,13	1,27	3,22	0,18
Fe	K series	10,48	0,31	4,13	13,48	0,39
Ba	L series	1,13	0,24	0,18	1,26	0,27
Total:		100		100	100	

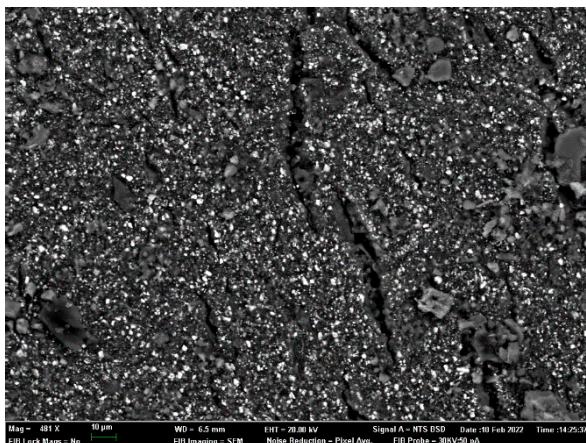
Sample: red paint
Ref: MIK-3
Sampling point: side E



Secondary electron image of the surface of the paint



Backscattered electron image of the surface of the paint

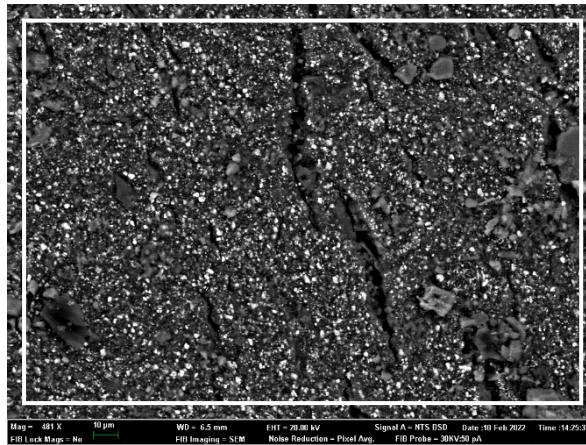


Backscattered electron image. Detail of the above image of the surface of the paint

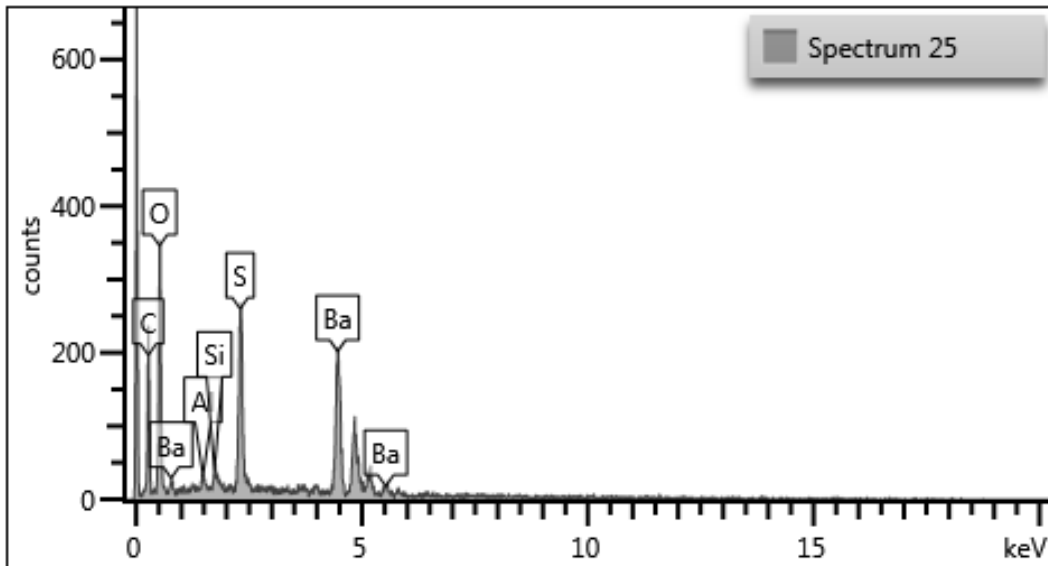
Sample: red paint
Ref: MIK-3
Sampling point: side E



Acquisition
 area



X-ray spectrum

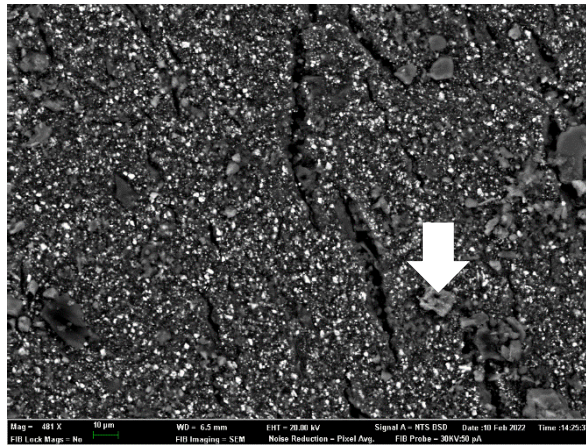


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
C	16,98	0,81	27,06	62,2	2,98
O	55,6		66,55		
Al	0,37	0,1	0,26	0,69	0,18
Si	0,34	0,09	0,23	0,73	0,19
S	4,74	0,19	2,83	11,84	0,49
Ba	21,97	0,63	3,06	24,53	0,71
Total:	100		100	100	

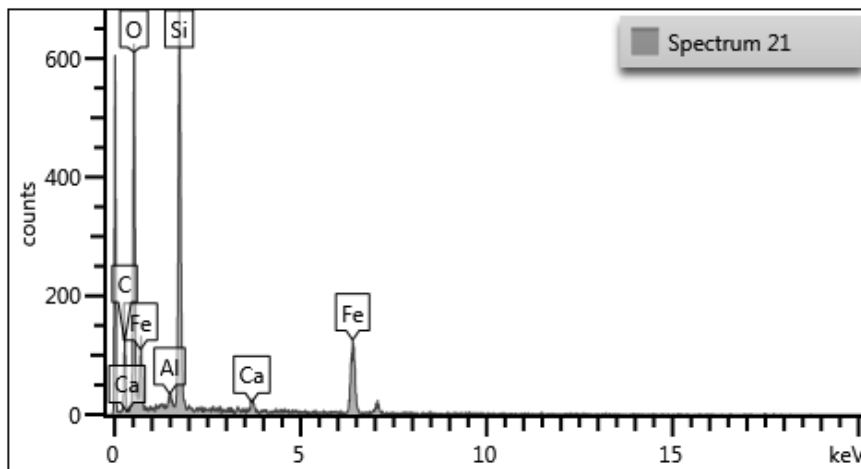
Sample: red paint
Ref: MIK-3
Sampling point: side E



Acquisition point



X-ray spectrum

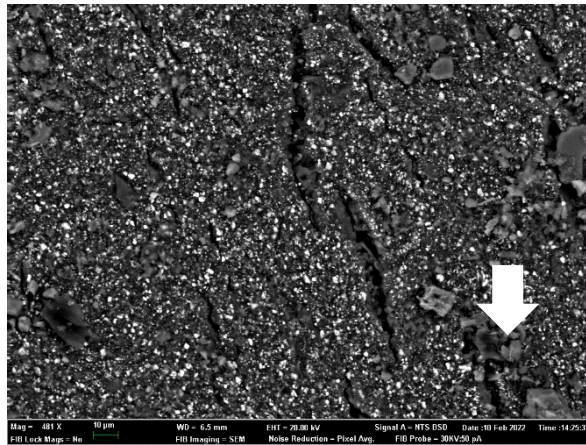


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
C	15,65	1,06	22,9	57,35	3,9
O	59,45		65,29		
Al	0,37	0,1	0,24	0,69	0,19
Si	12,1	0,38	7,57	25,89	0,81
Ca	0,69	0,1	0,3	0,97	0,14
Fe	11,73	0,48	3,69	15,1	0,61
Total:	100		100	100	

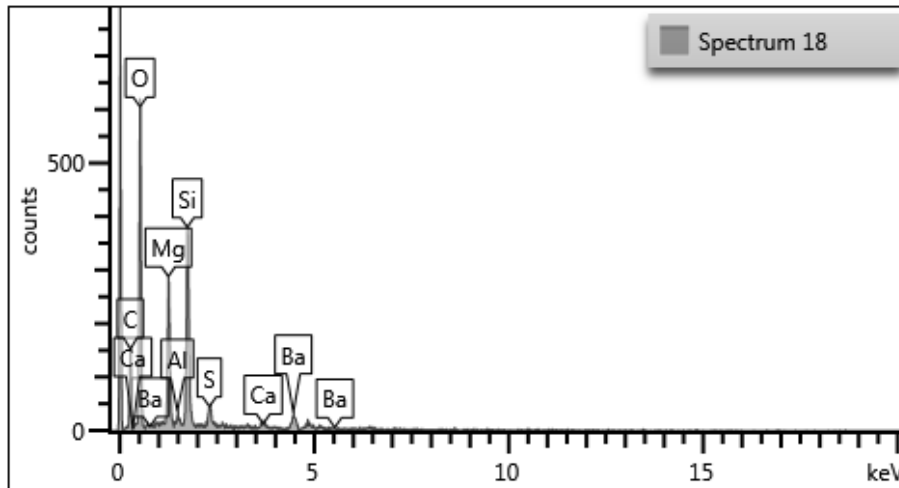
Sample: red paint
Ref: MIK-3
Sampling point: side E



Acquisition point



X-ray spectrum

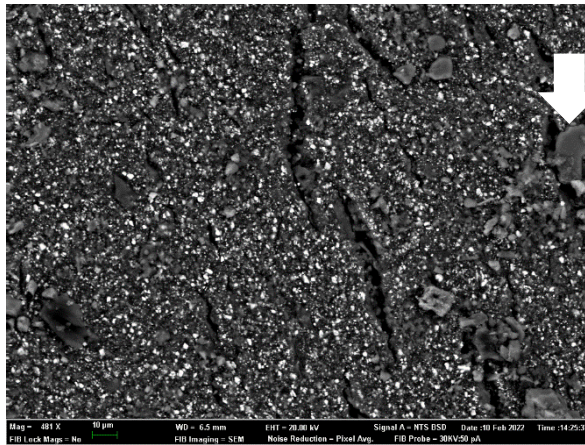


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
C	18,36	1,12	25,18	67,28	4,12
O	63,35		65,22		
Mg	6,13	0,28	4,16	10,17	0,46
Al	0,36	0,1	0,22	0,69	0,19
Si	7,21	0,28	4,23	15,42	0,6
S	0,89	0,11	0,46	2,23	0,27
Ca	0,34	0,09	0,14	0,47	0,13
Ba	3,36	0,34	0,4	3,75	0,38
Total:	100		100	100	

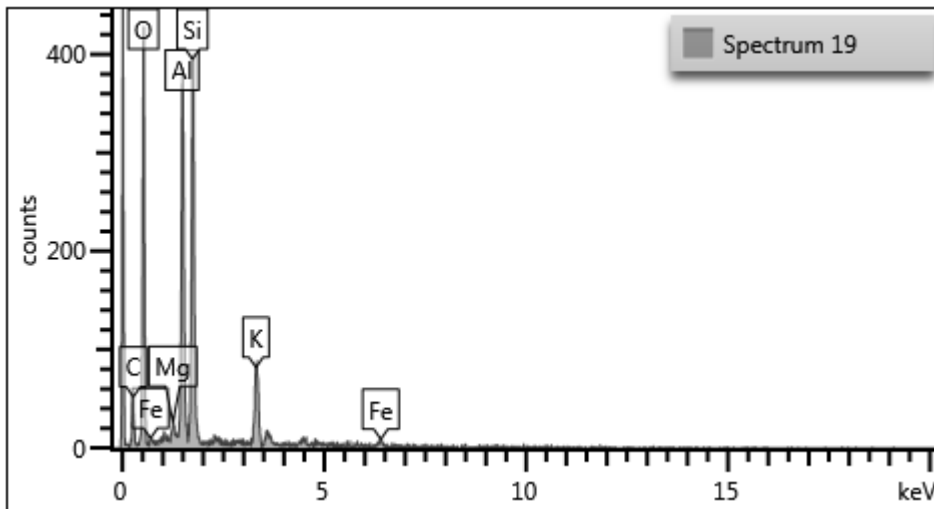
Sample: red paint
Ref: MIK-3
Sampling point: side E



Acquisition
 point



X-ray spectrum

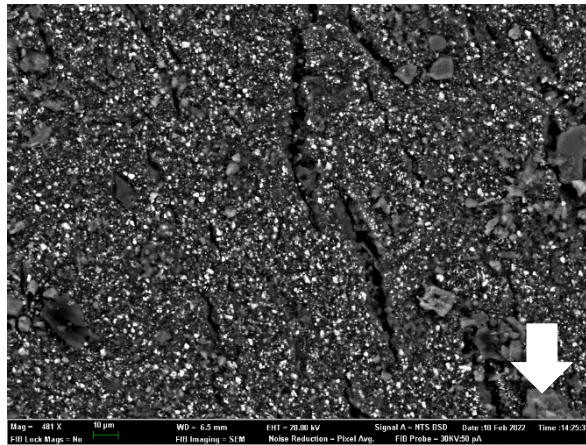


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
C	12,55	1,68	18,35	46	6,15
O	58,62		64,33		
Mg	0,59	0,14	0,42	0,97	0,23
Al	10,77	0,49	7,01	20,35	0,93
Si	12,37	0,55	7,73	26,46	1,18
K	4,13	0,27	1,86	4,98	0,32
Fe	0,96	0,22	0,3	1,24	0,28
Total:	100		100	100	

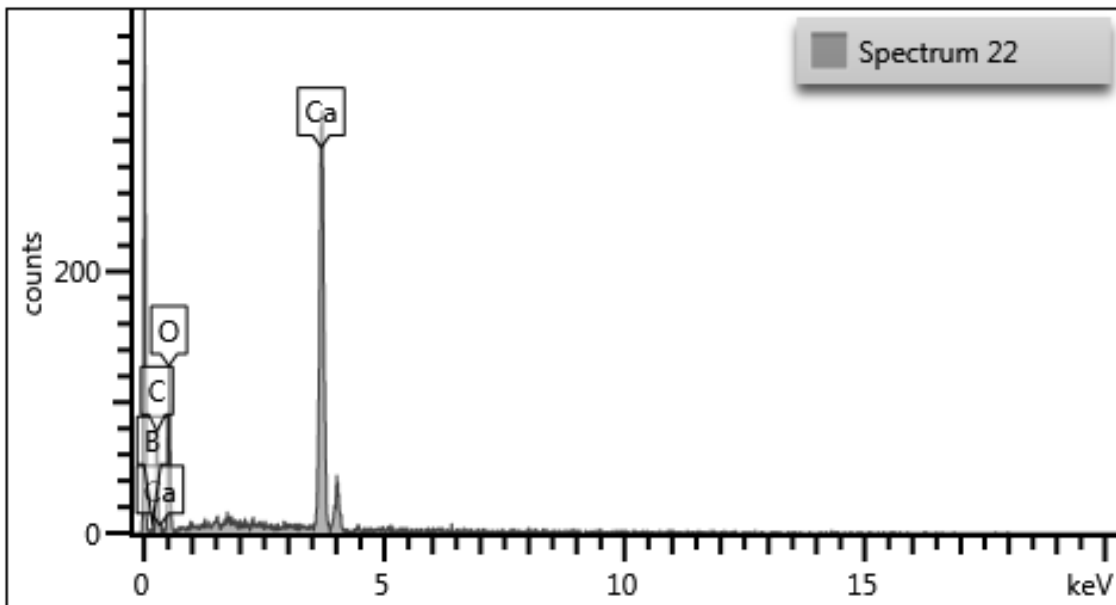
Sample: red paint
Ref: MIK-3
Sampling point: side E



Acquisition
 point

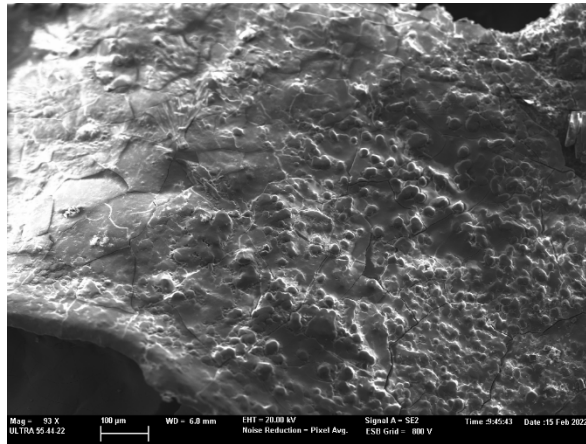


X-ray spectrum

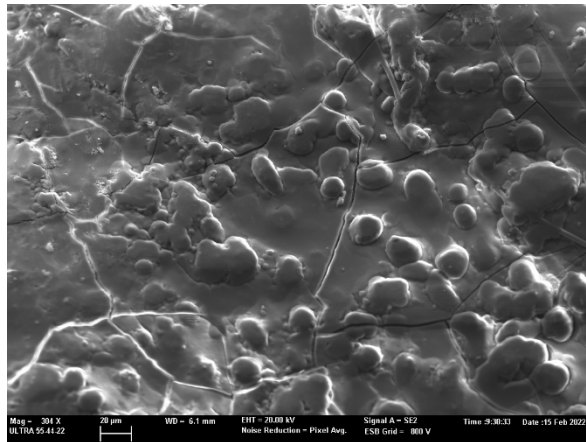


Element	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
B	7,33	1,94	11,19	23,6	6,25
C	13,77	1,31	18,92	50,46	4,79
O	60,36		62,26		
Ca	18,54	0,92	7,63	25,94	1,29
Total:	100		100	100	

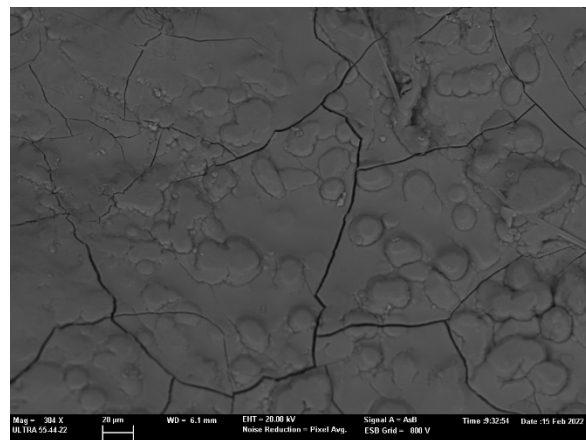
Sample: red paint
Ref: MIK-5
Sampling point: side E



Secondary electron image of the surface of the paint



Secondary electron image of the surface of the paint

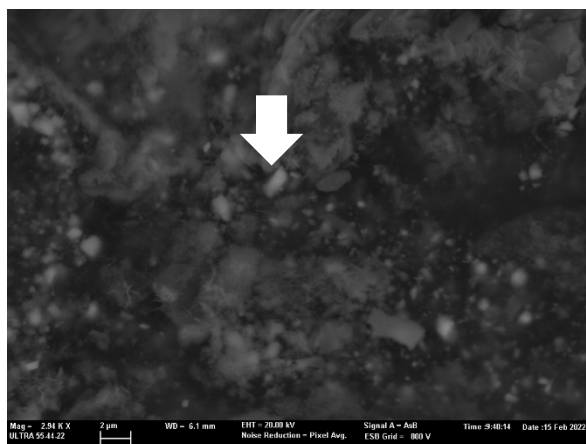


Backscattered electron image of the surface of the paint

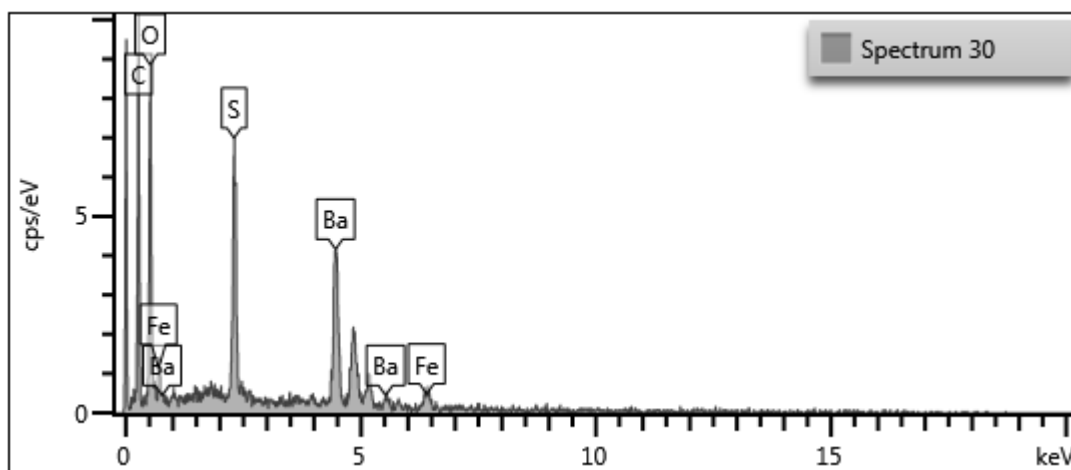
Sample: red paint
Ref: MIK-5
Sampling point: side E



Acquisition point



X-ray spectrum



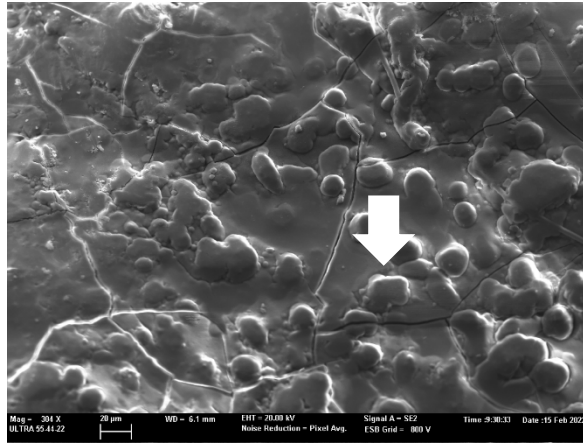
Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		29,66		66,92		
S	K series	15,03	0,73	16,92	37,53	1,83
Fe	K series	4,21	0,78	2,72	5,42	1,01
Ba	L series	51,11	1,36	13,43	57,06	1,52
Total:		100		100	100	S/Ba = 1,26

The higher value of the S/Ba molar ratio than that ideal of 1,00 corresponding to the barite is associated to rests of the reagent iron(II) sulfate heptahydrate used in the solid state reaction that takes place in the roasting process of synthesis of iron oxide red. The oxidative calcination of this reagent produces α -Fe₂O₃

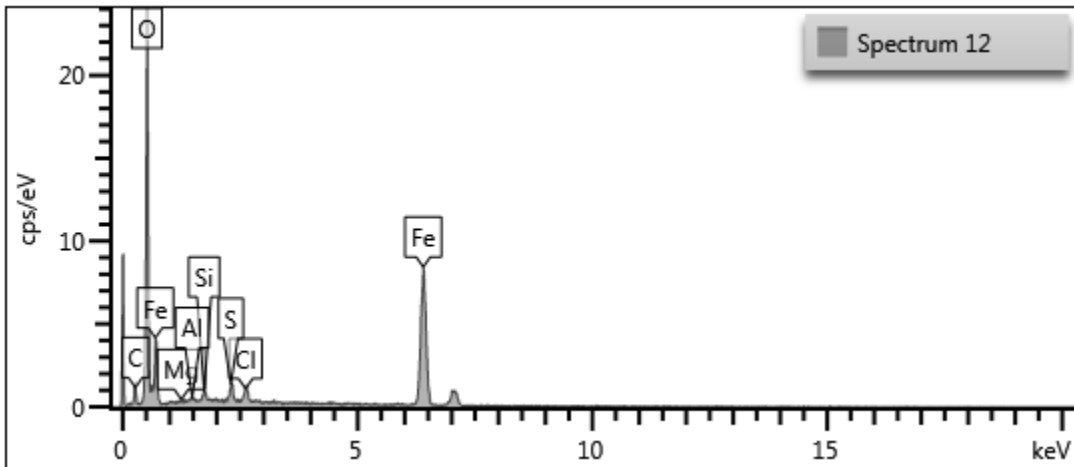
Sample: red paint
Ref: MIK-5
Sampling point: side E



Acquisition
point



X-ray spectrum

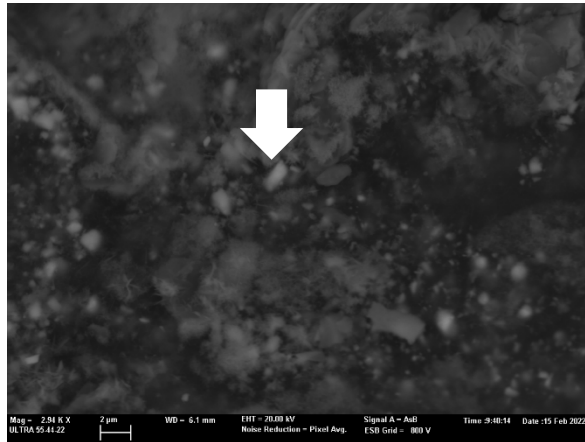


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		25,07		52,3		
Mg	K series	0,22	0,24	0,31	0,37	0,41
Al	K series	0,48	0,21	0,59	0,91	0,4
Si	K series	1,31	0,2	1,55	2,79	0,43
S	K series	2,23	0,2	2,33	5,58	0,5
Cl	K series	2,03	0,19	1,91	0	0,19
Fe	K series	68,65	0,7	41,02	88,32	0,9
Total:		100		100	97,97	

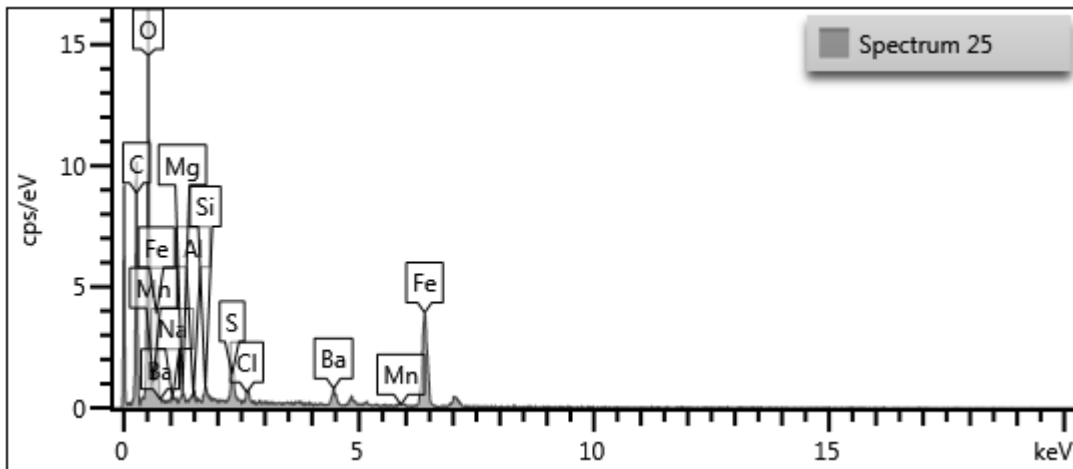
Sample: red paint
Ref: MIK-5
Sampling point: side E



Acquisition
point

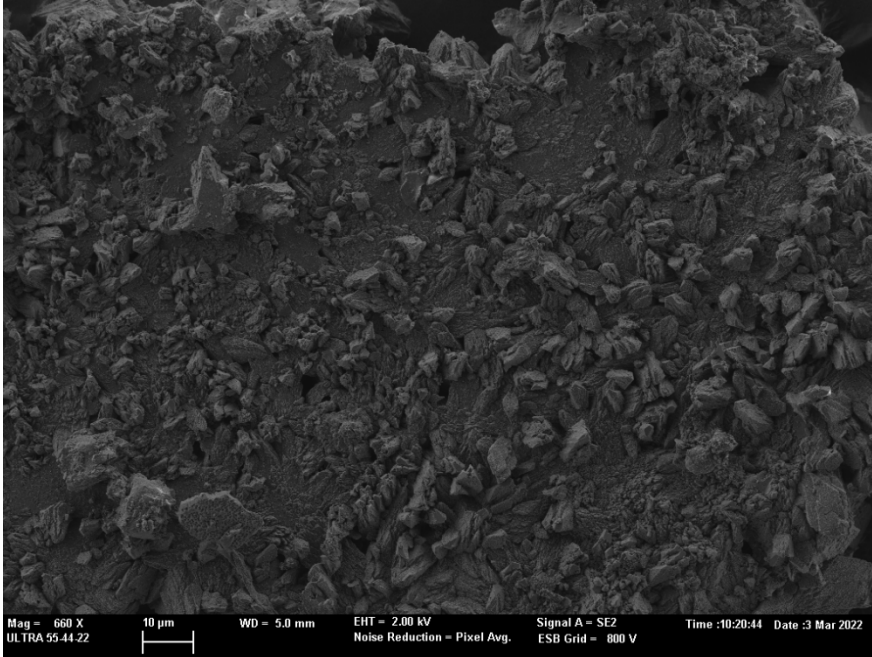


X-ray spectrum

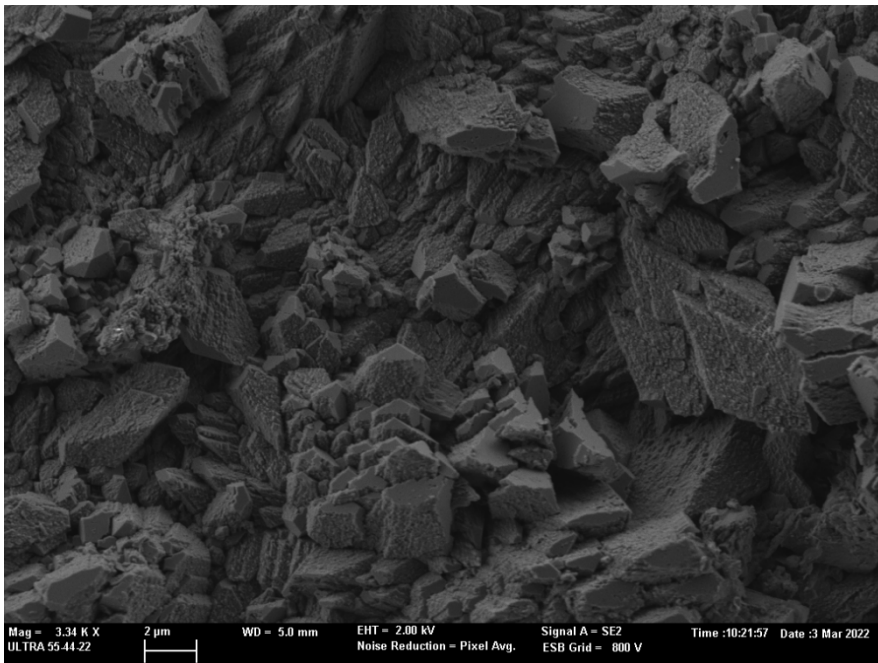


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		26,24		54,56		
Na	K series	0,32	0,62	0,47	0,44	0,83
Mg	K series	1,49	0,43	2,04	2,47	0,72
Al	K series	0,78	0,35	0,96	1,48	0,65
Si	K series	1,63	0,32	1,94	3,5	0,68
S	K series	4,17	0,35	4,32	10,4	0,87
Cl	K series	1,83	0,26	1,72	0	0,26
Mn	K series	1,35	0,39	0,82	1,74	0,5
Fe	K series	51,28	1,13	30,54	65,97	1,46
Ba	L series	10,9	0,81	2,64	12,17	0,91
Total:		100		100	98,17	

Sample: mineral crusts
Ref: MIK-8
Sampling point: side E

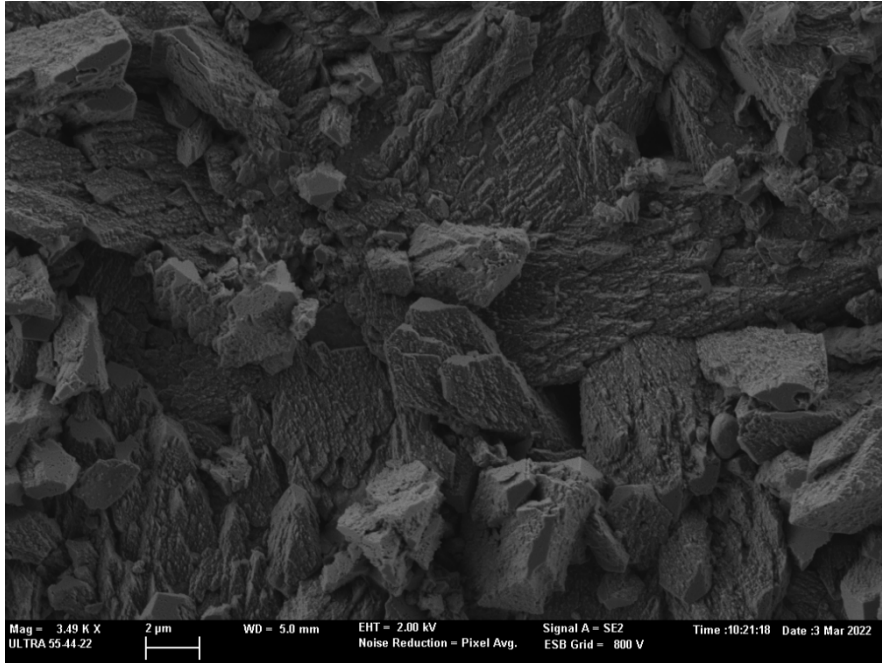


Secondary electron image of the crusts formed on surface of the paint

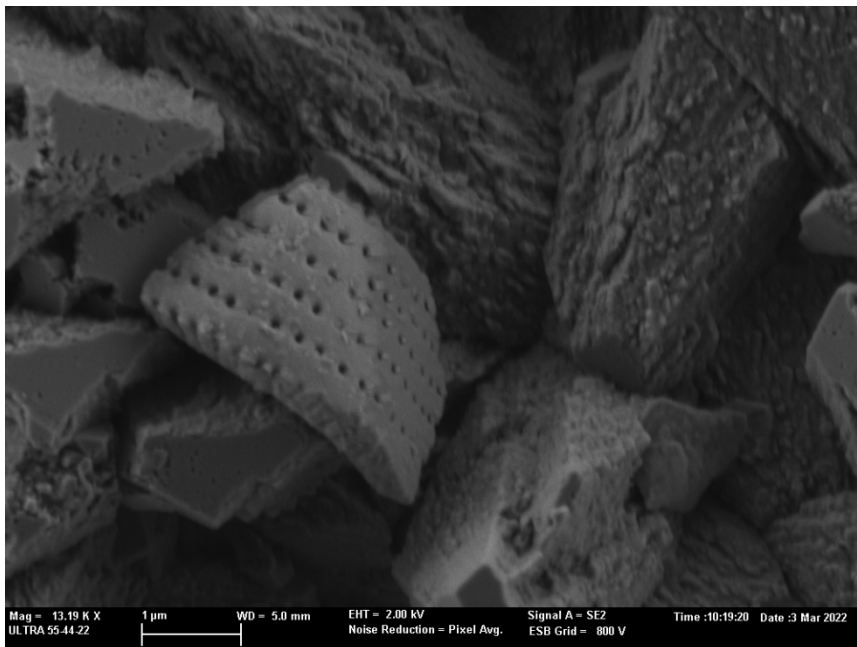


Secondary electron image of the crusts formed on surface of the Paint Detail.

Sample: mineral crusts
Ref: MIK-8
Sampling point: side E



Secondary electron image of the crusts formed on surface of the Paint. Detail of the surface of the cryptocrystalline grains

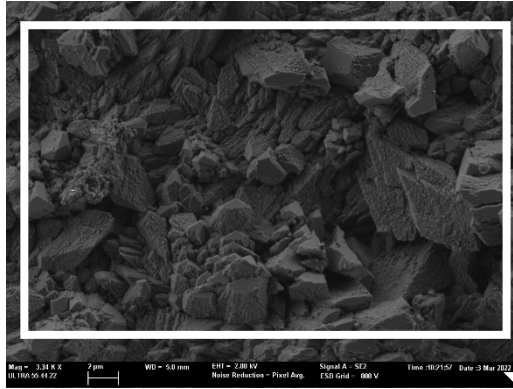


Secondary electron image of the crusts formed on surface of the Paint Detail of a fragment of diatomea.

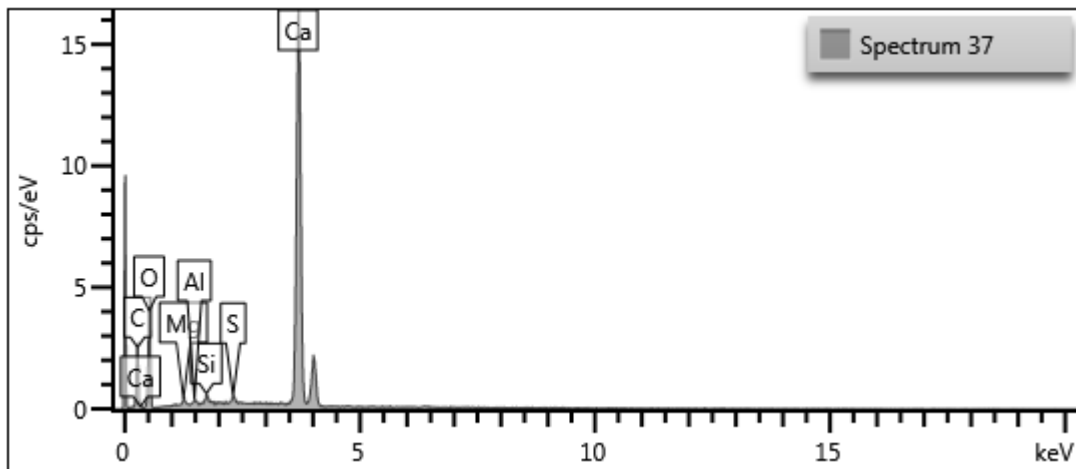
Sample: mineral crusts
Ref: MIK-8
Sampling point: side E



Acquisition
 area



X-ray spectrum

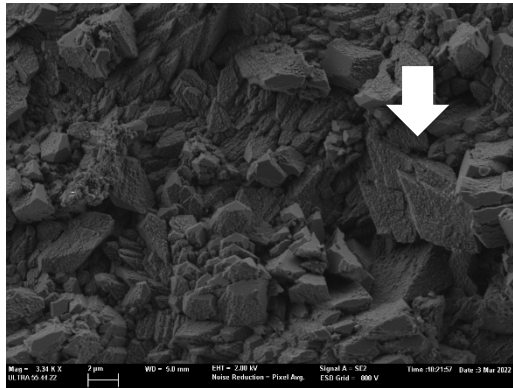


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		29,79		51,09		
Mg	K series	0,79	0,11	0,9	1,32	0,18
Al	K series	0,45	0,09	0,46	0,85	0,16
Si	K series	0,75	0,09	0,73	1,6	0,19
S	K series	0,71	0,09	0,61	1,78	0,22
Ca	K series	67,51	0,32	46,22	94,46	0,44
Total:		100		100	100	

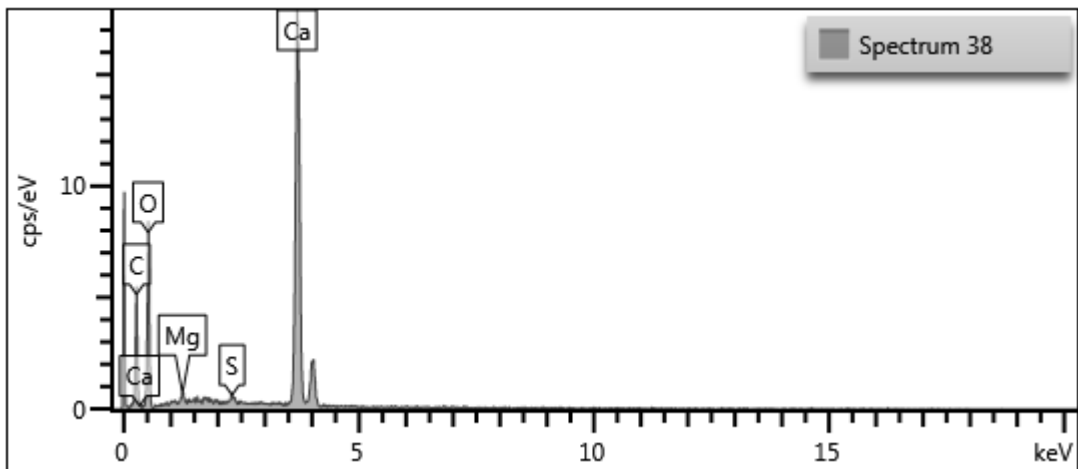
Sample: mineral crusts
Ref: MIK-8
Sampling point: side E



Acquisition
 point



X-ray spectrum



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide %	Oxide % Sigma
O		29,43		50,73		
Mg	K series	1,25	0,26	1,41	2,07	0,44
S	K series	0,85	0,18	0,73	2,13	0,46
Ca	K series	68,48	0,59	47,12	95,81	0,83
Total:		100		100	100	