

Report

“XEPMA analysis of the samples received from Proton21 laboratory”

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Summary

Sample description. The samples (accumulating screens numbered 7550 and 7203) represent copper disks each approximately 1.2 cm in diameter with a hole in the middle. As a result of the target explosion, the target material is deposited at the surface of the screen in the general form of radial rays pointing away from the target position. The microphotographs presented with the samples allow identifying the spots for analysis.

Aim of the analysis. To confirm the results by Proton21 regarding the presence of elements different from the principal components of target material and accumulating screen material in the provided samples.

Device description. The scanning electron microscopy was performed on a Philips ESEM XL 30, equipped with energy dispersive X-ray spectroscopy (EDS), EDAX DX-4. The microphotographs were taken with a backscattering detector. The EDS x-ray spectra were used for element identifications and element normalized quantifications.

Measurement parameters. The microscope was used in its high-vacuum mode. The acceleration voltage of 30 kV was used for all spots. The measurement parameters of interest are presented at the bottom of each microphotograph.

General remarks. The aim of this analysis was to verify the results obtained by Proton21 regarding the elemental composition of foreign inclusions deposited on the surface of the accumulating screen after the explosion of the target. As the surfaces of the samples presented for the analysis is highly non-uniform, with the elemental composition of the inclusions drastically varying from spot to spot, the verification procedure was chosen as follows.

1. Proton21 presents the microphotographs of the samples with the selected spots clearly marked for the analysis, alongside with the spectra obtained at these spots
2. We find the marked spots and take spectra. To ensure consistency we present the microphotographs of the samples with the spots we took for analysis (also clearly marked at the photographs), alongside with our spectra taken at these spots.

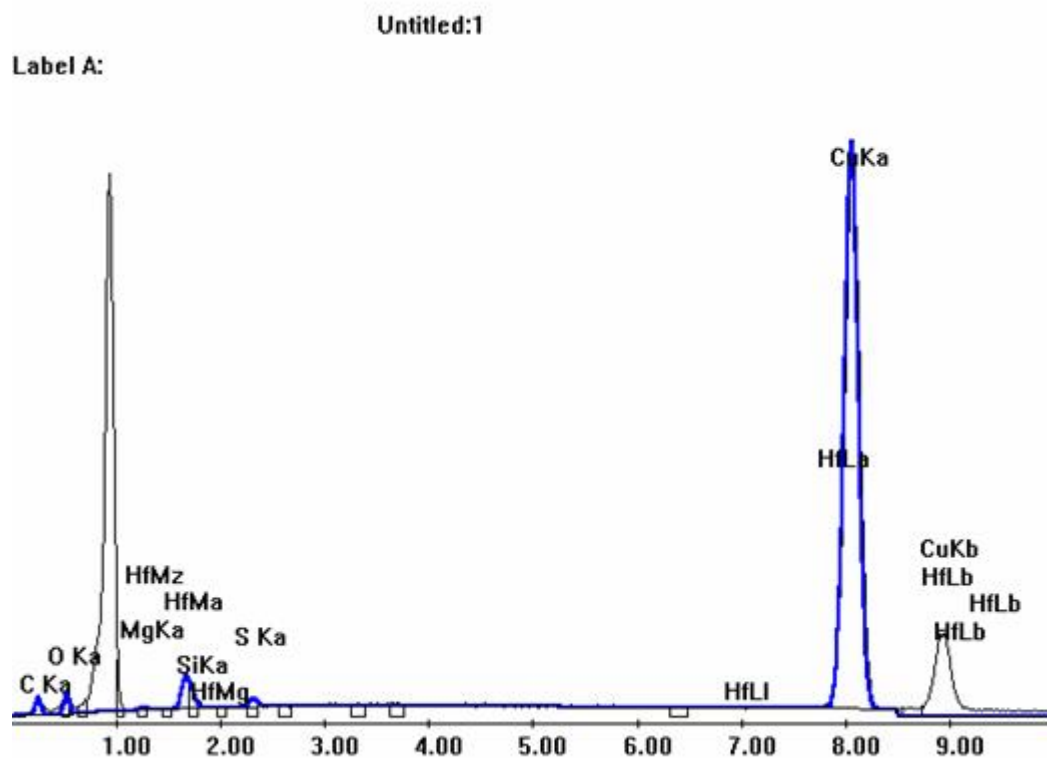
The spectra and microphotographs by Proton21 allowed clear identification of the analysis points. The “spot analysis” mode (not “area analysis” mode) was used to ensure stronger signal from non-uniform inclusions.

Overall conclusion. We were able to identify most of the elements that were registered by Proton21, therefore **confirming** the presence of foreign inclusions at the surface of the accumulating screen that differ from the declared composition of the screen and the target materials. The discrepancies between some of the spectra supplied by Proton 21 and the corresponding spectra from this analysis may be explained by slight shifting the observation point. However we were able to show the presence of foreign inclusions roughly of the same elemental composition as in the original spectra. Also, because of the above error in locus determination the concentrations of elements found in this analysis may differ from the ones presented in the Proton21 spectra.

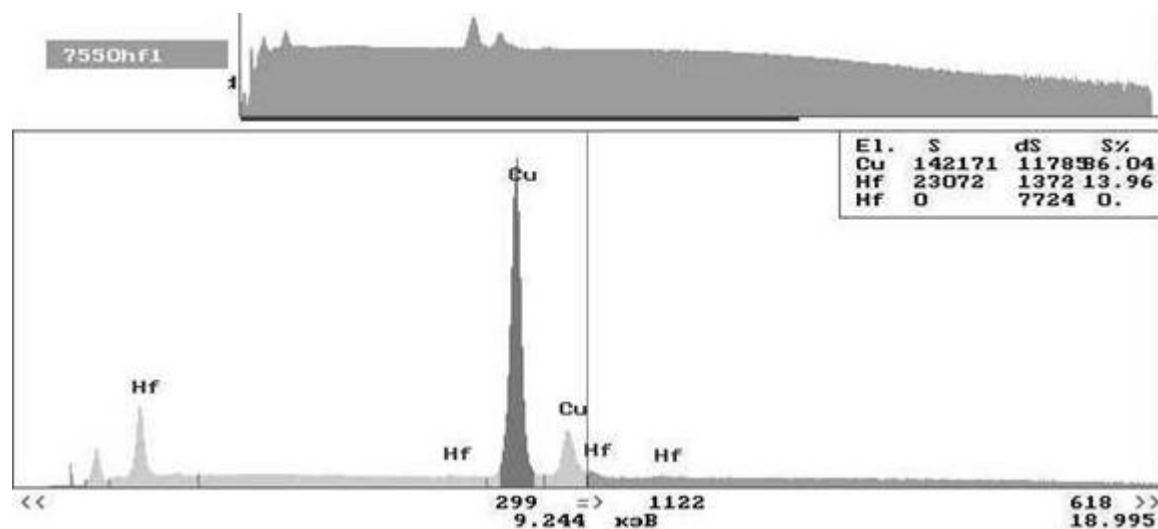
Results of the analysis: Sample 7550

Point 7550hf1 (t4).

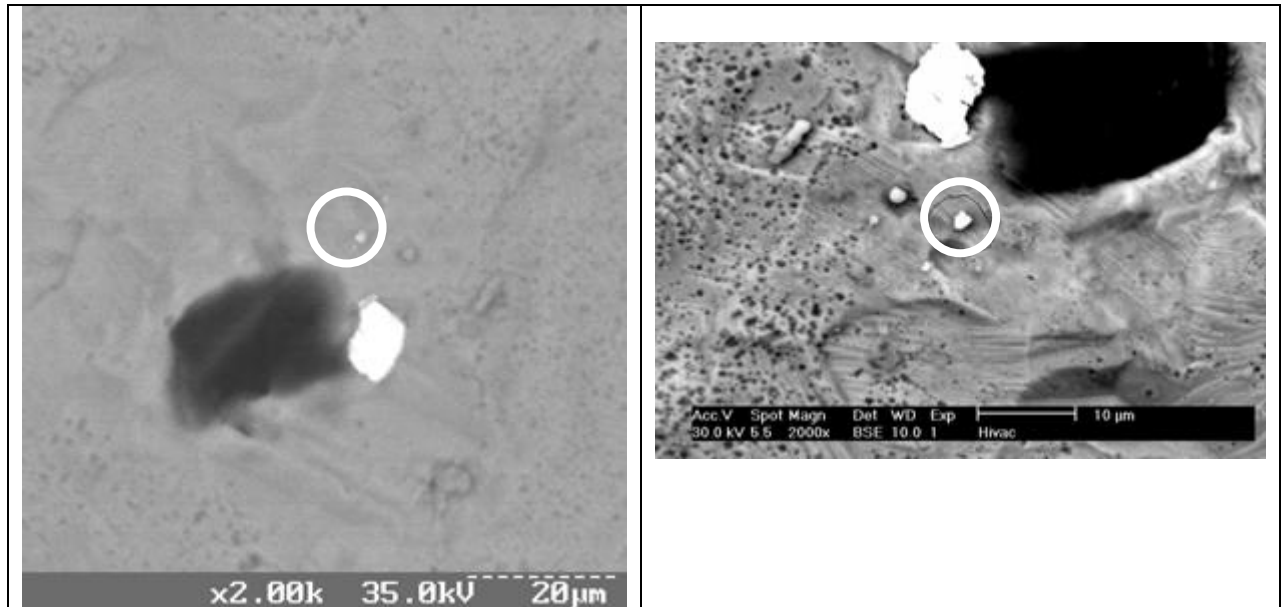
Spectrum from this analysis:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



Quantitative analysis:

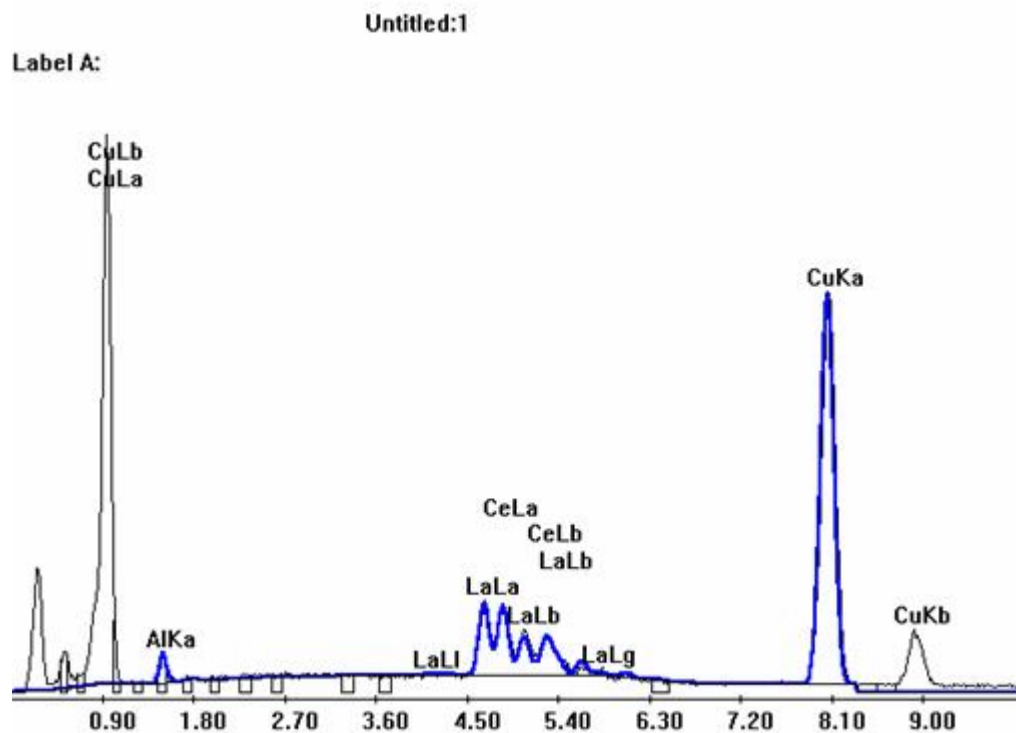
**EDAX ZAF Quantification (Standardless)
Element Normalized**

Element	Wt %	At %	K-Ratio	Z	A	F
C K	6.63	24.91	0.0102	1.1624	0.1317	1.0004
O K	3.37	9.52	0.0092	1.1457	0.2360	1.0033
MgK	0.60	1.11	0.0008	1.1036	0.1158	1.0001
SiK	0.69	1.11	0.0017	1.1041	0.2213	1.0001
S K	0.54	0.75	0.0023	1.0966	0.3967	1.0000
HfL	0.00	0.00	0.0000	0.8373	1.1045	1.0000
CuK	88.17	62.60	0.8630	0.9748	1.0041	1.0000
Total	100.00	100.00				

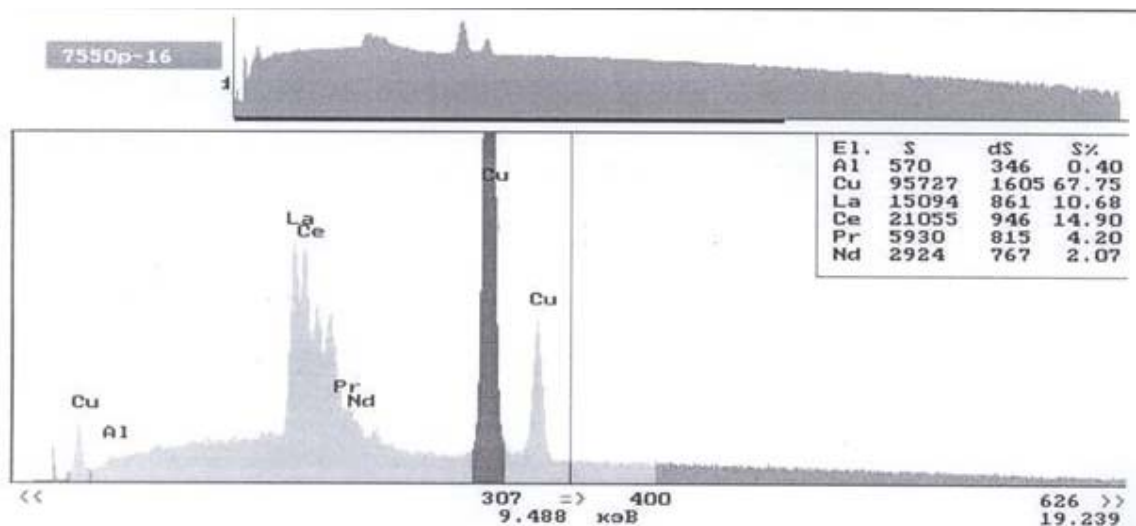
Comment. We were able to identify trace amounts of **Hf**, although much less than in the original spectrum supplied by Proton21.

Point 7550p16

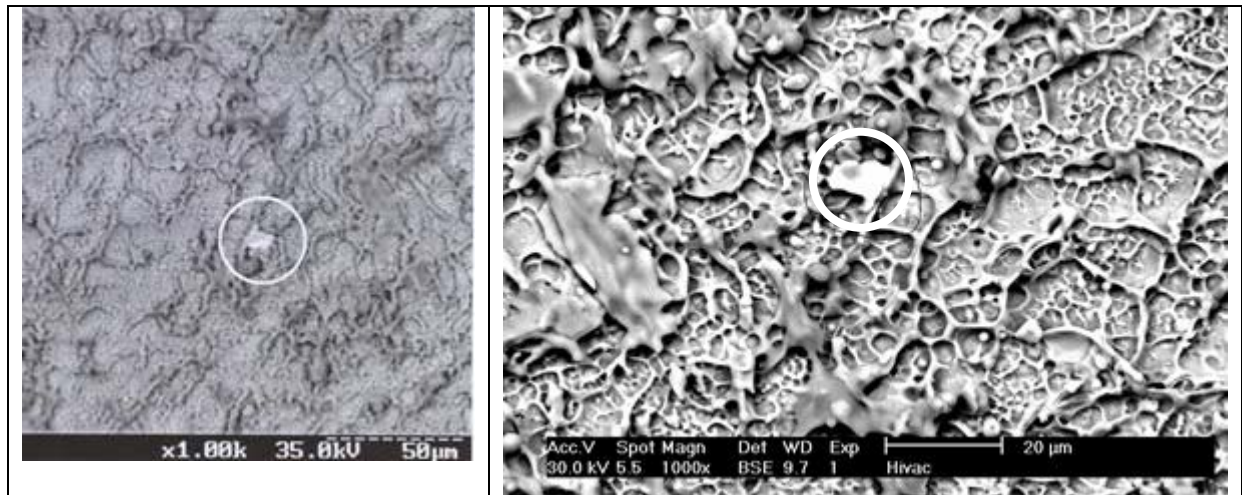
Spectrum from this analysis:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis

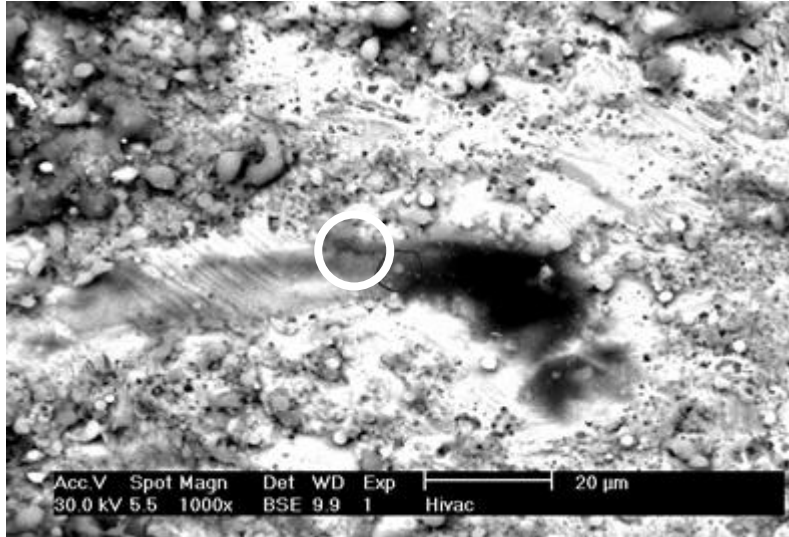


Quantitative analysis:

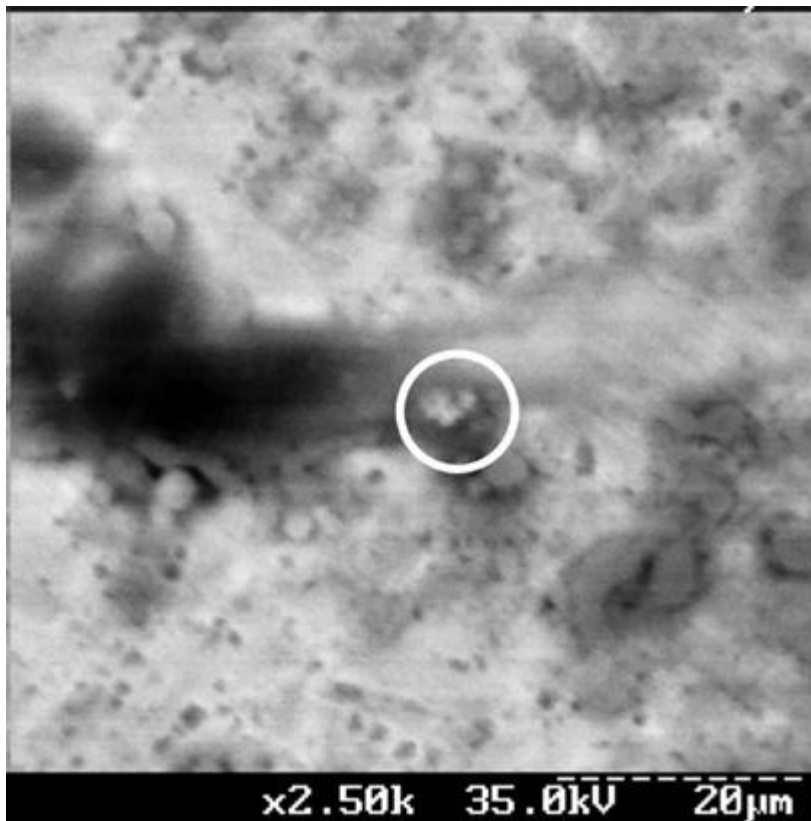
Comment. We may confidently register **Al**, **La** and **Ce**. We could not with the same level of confidence register **Nd** and **Pr**.

Point 7550t1

Microphotograph from this analysis



At this point we found out that the particle marked for analysis was not present at the sample surface, as it has possibly fallen off during shipment (compare to the original picture below). Therefore, no spectra were taken.

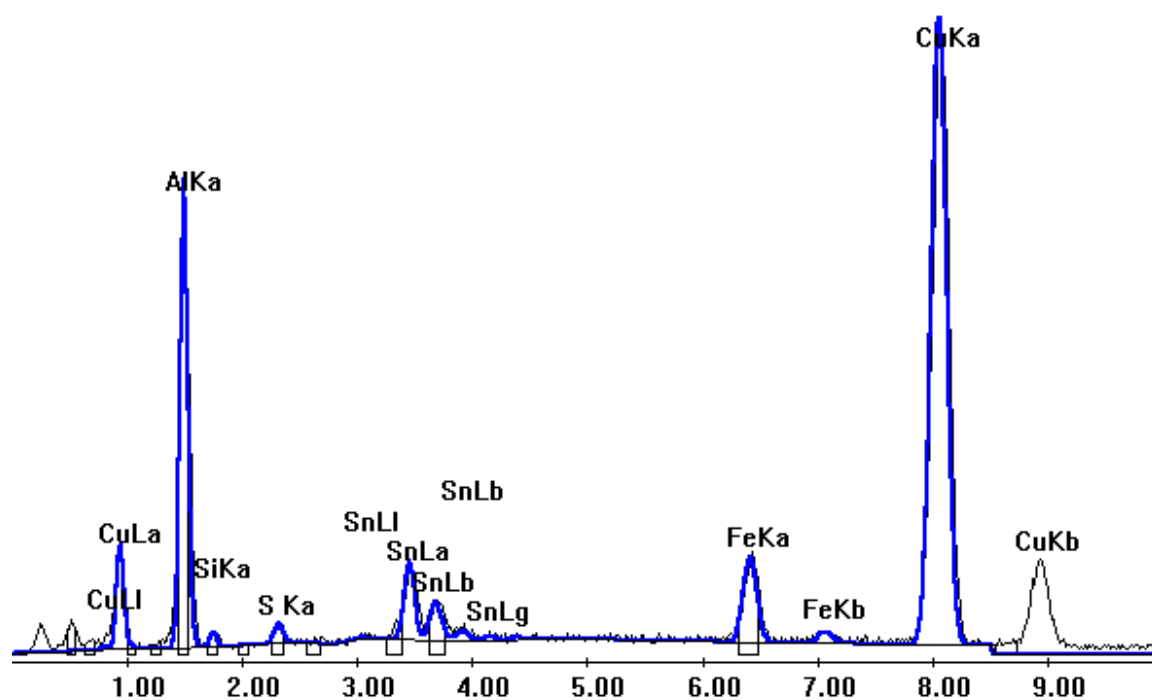


Point 7550t2.

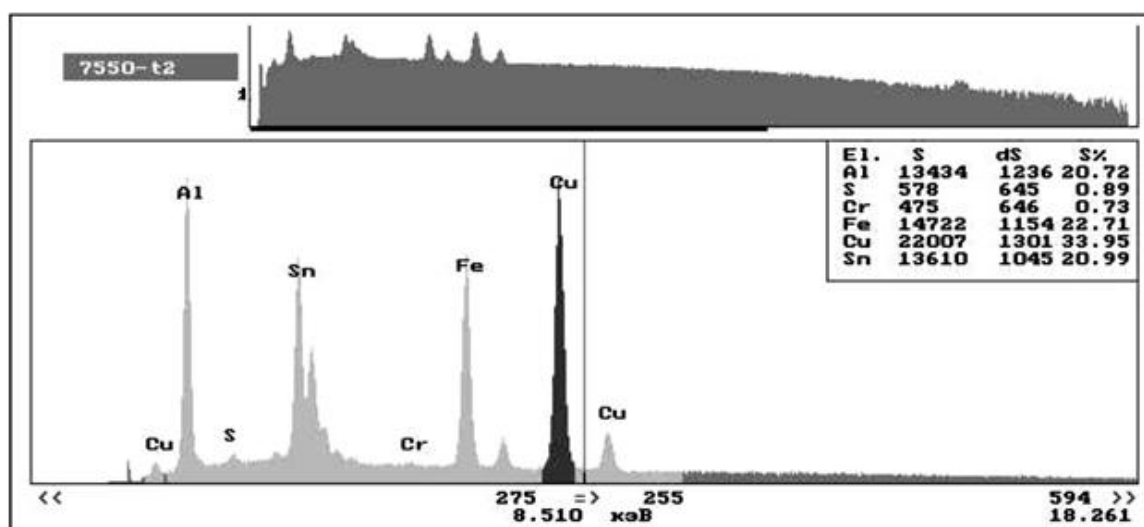
Spectrum from this analysis:

Untitled:1

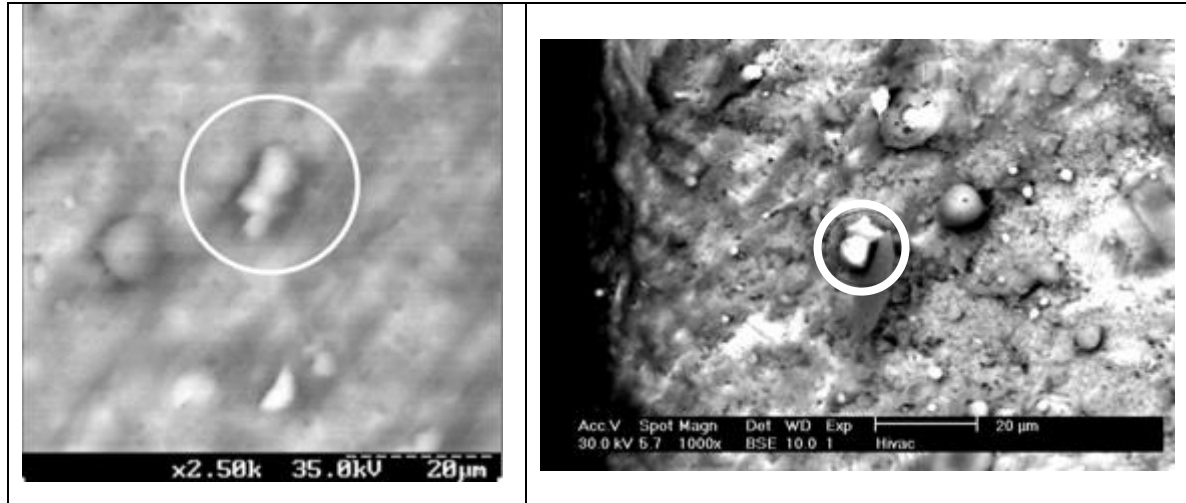
Label A:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



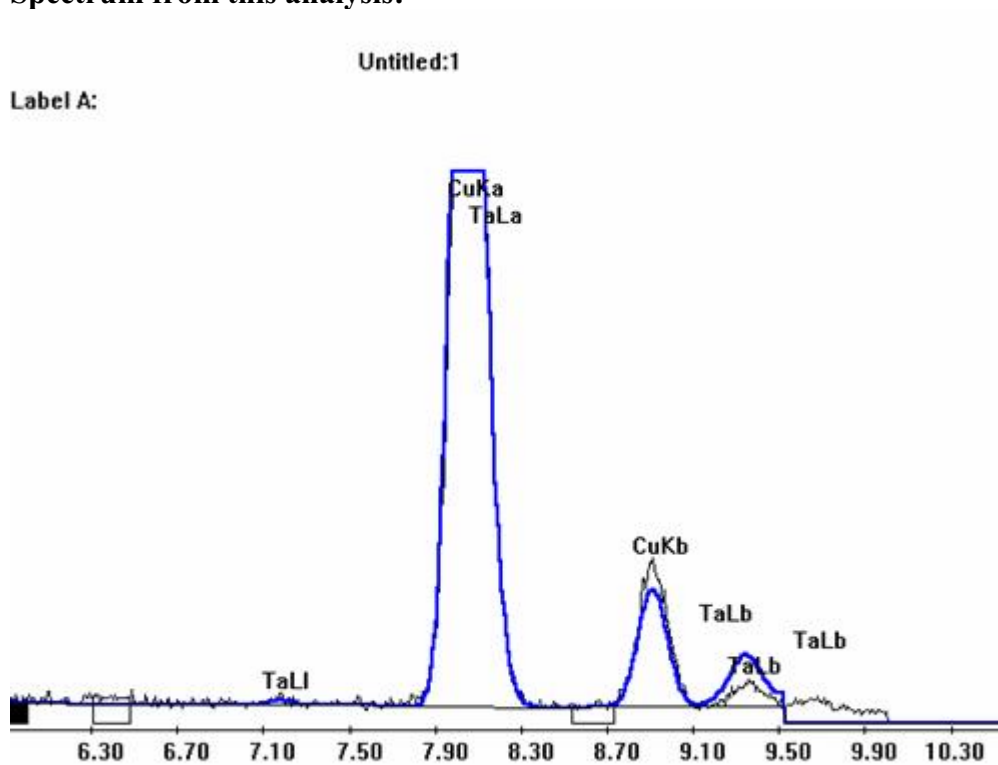
Quantitative analysis:

EDAX ZAF Quantification (Standardless)						
Element Normalized						
Element	Wt %	At %	K-Ratio	Z	A	F
AlK	28.60	48.77	0.0631	1.0665	0.2066	1.0007
SiK	0.94	1.54	0.0021	1.0984	0.2062	1.0009
S K	0.83	1.19	0.0034	1.0910	0.3699	1.0022
SnL	6.99	2.71	0.0538	0.8653	0.8879	1.0017
FeK	4.30	3.54	0.0466	0.9943	0.9501	1.1474
CuK	58.34	42.24	0.5519	0.9687	0.9766	1.0000
Total	100.00	100.00				

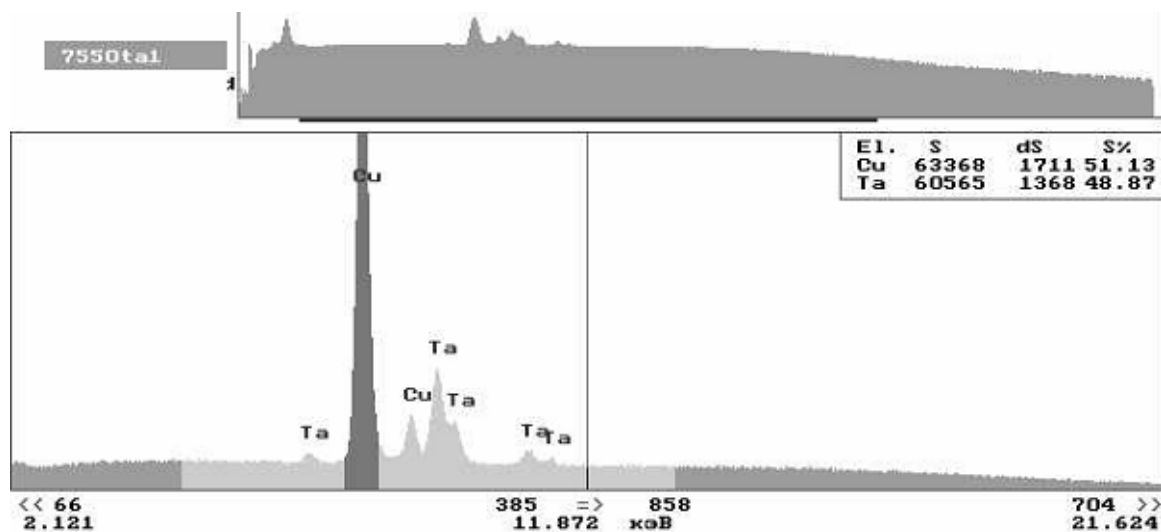
Comment: We confidently register Al, Sn, Fe, S, Si. We cannot confidently identify Cr.

7550t3 (ta1).

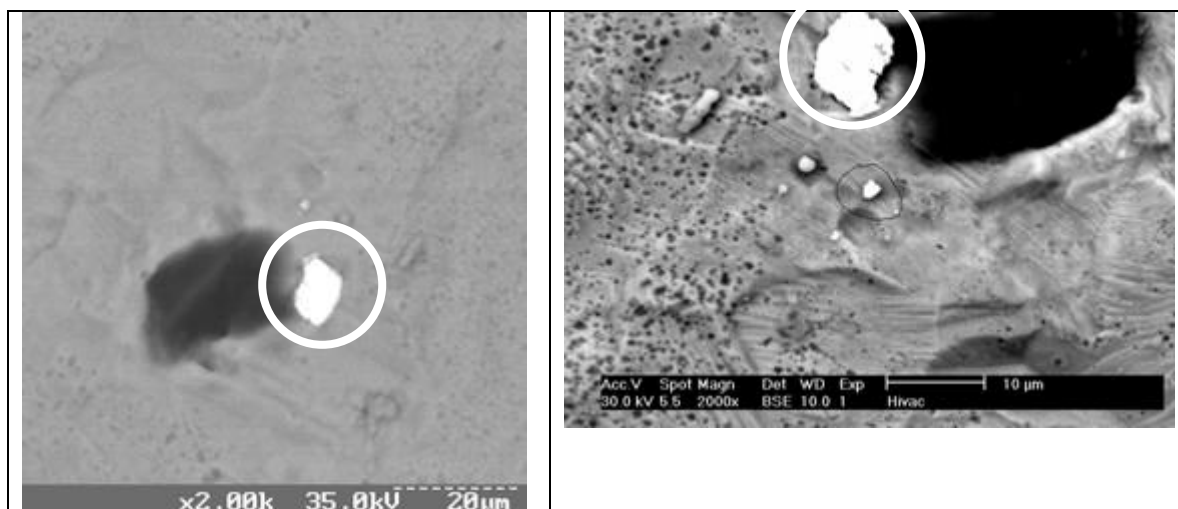
Spectrum from this analysis:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



Quantitative analysis:

EDAX ZAF Quantification (Standardless) Element Normalized

Element	Wt %	At %	K-Ratio	Z	A	F
SiK	37.09	61.31	0.1406	1.0859	0.3492	1.0000
CuK	47.57	34.76	0.4480	0.9601	0.9808	1.0000
TaL	15.34	3.94	0.1361	0.8231	1.0778	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
CuL	146.12	3.44	0.76	42.44
SiK	266.23	5.26	0.56	50.62
CuK	264.10	4.87	0.56	54.20
TaL	33.48	4.82	1.68	6.95

Comment. Ta is confidently registered

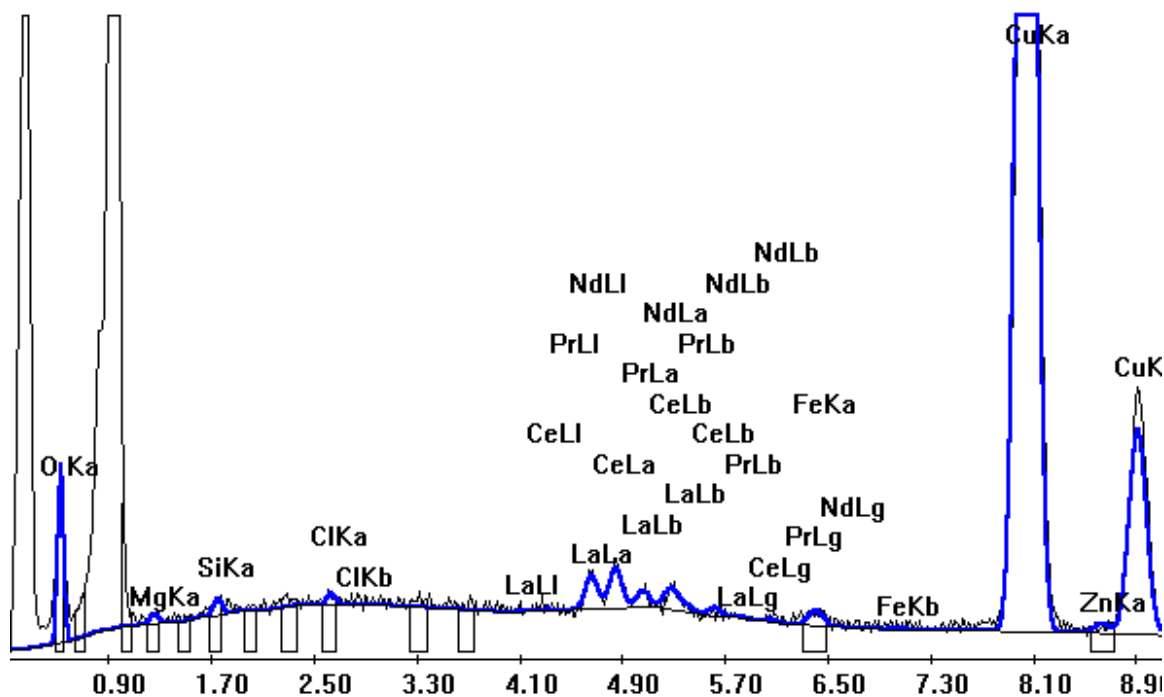
Results of the analysis: Sample 7203

Point 7203P10

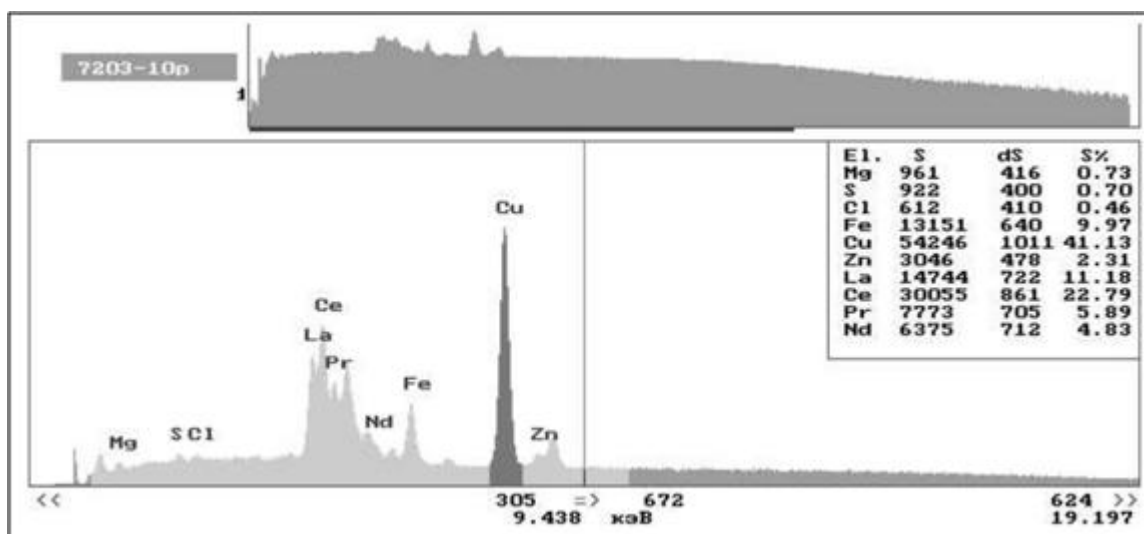
Spectrum from this analysis:

Untitled:1

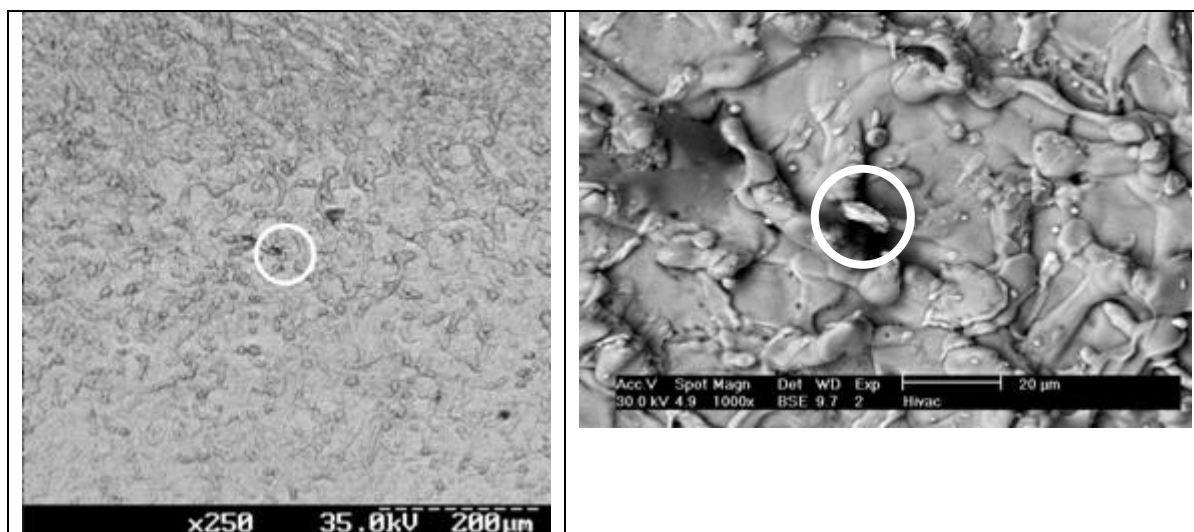
Label A:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, Right – this analysis



Quantitative analysis:

**EDAX ZAF Quantification (Standardless)
Element Normalized**

Element	Wt %	At %	K-Ratio	Z	A	F
O K	6.60	21.95	0.0202	1.1591	0.2631	1.0029
MgK	0.61	1.33	0.0008	1.1164	0.1120	1.0001
SiK	0.57	1.09	0.0014	1.1168	0.2119	1.0002
ClK	0.22	0.34	0.0012	1.0621	0.4855	1.0009
LaL	1.75	0.67	0.0175	0.8745	1.0685	1.0706
CeL	2.19	0.83	0.0226	0.8835	1.0798	1.0853
PrL	0.00	0.00	0.0000	0.8947	1.0889	1.1026
NdL	0.00	0.00	0.0000	0.8895	1.0962	1.1233
FeK	0.37	0.35	0.0045	1.0129	0.9605	1.2501
CuK	87.11	72.97	0.8518	0.9876	0.9902	1.0000
ZnK	0.58	0.47	0.0057	0.9911	0.9960	1.0000
Total	100.00	100.00				

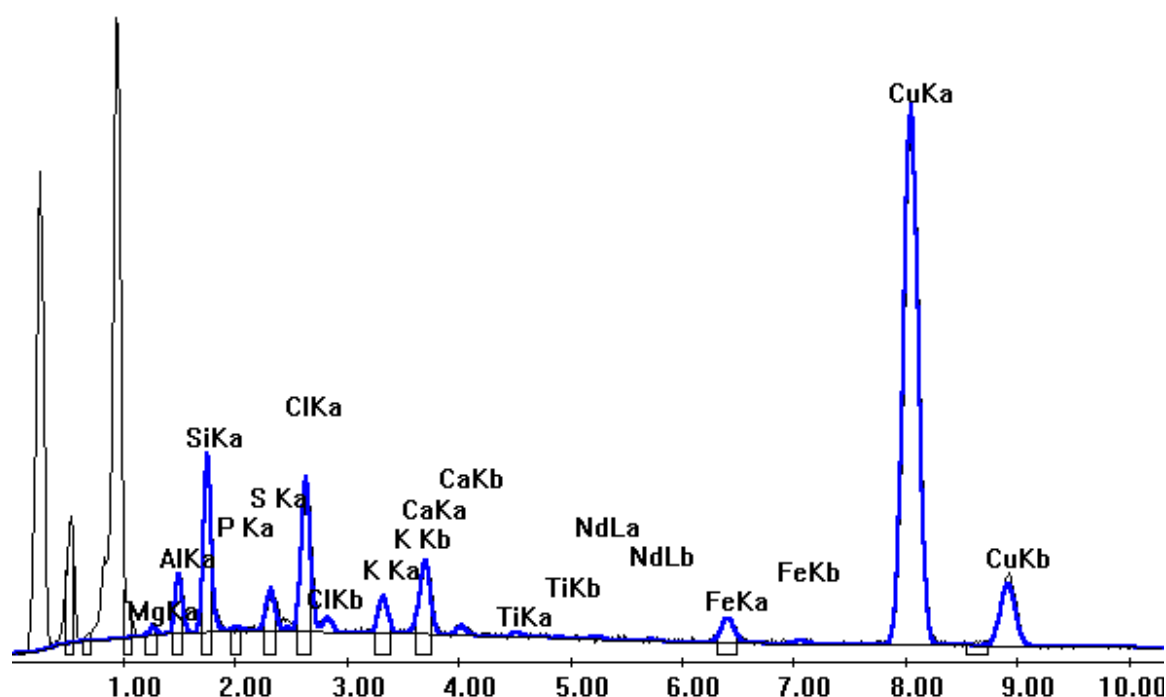
Comments: We confidently identify **Mg, Si, La, Ce, Cl, Zn, Fe, O**. We could not identify **Nd** and **Pr**.

Point 7203p14

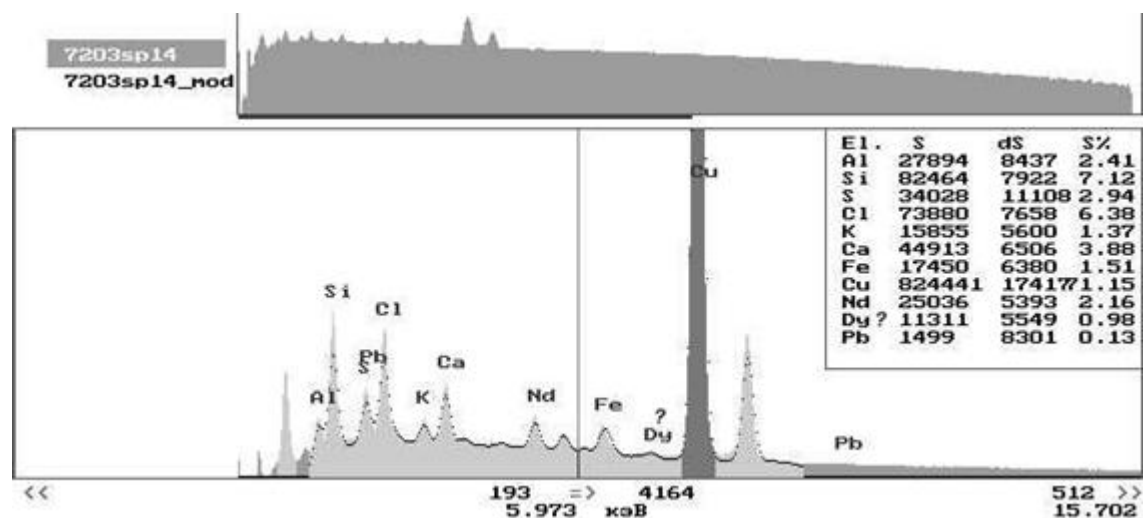
Spectrum from this analysis:

Untitled:1

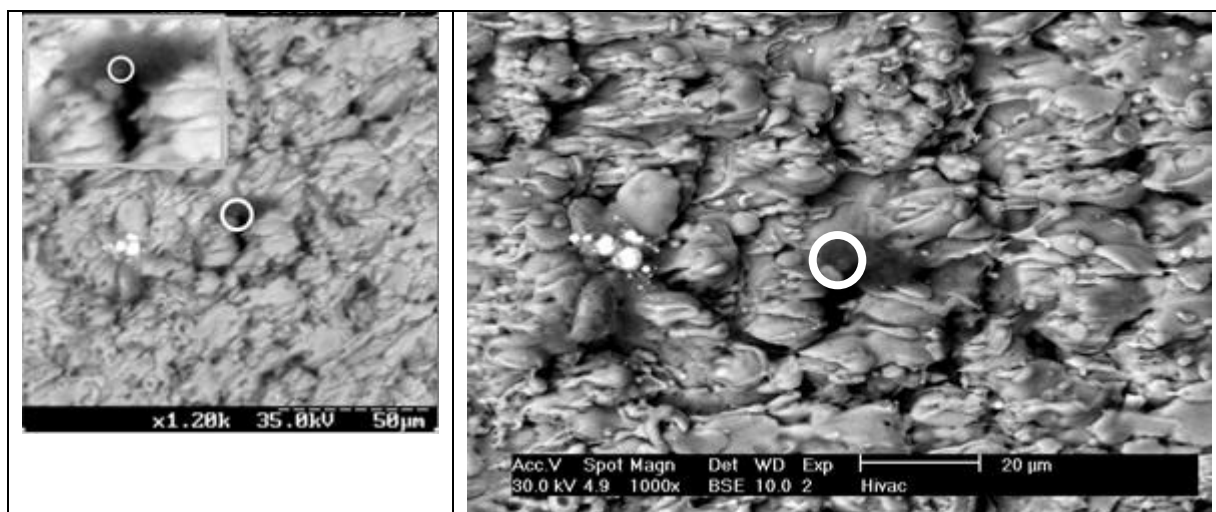
Label A:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



Quantitative analysis:

**EDAX ZAF Quantification (Standardless)
Element Normalized**

Element	Wt %	At %	K-Ratio	Z	A	F
MgK	1.15	2.23	0.0018	1.0883	0.1430	1.0026
AlK	4.91	8.56	0.0102	1.0572	0.1964	1.0037
SiK	12.14	20.34	0.0338	1.0888	0.2554	1.0024
PK	0.19	0.29	0.0006	1.0535	0.2968	1.0041
SK	2.22	3.26	0.0095	1.0815	0.3919	1.0054
ClK	7.32	9.72	0.0368	1.0357	0.4835	1.0027
KK	1.57	1.89	0.0107	1.0516	0.6372	1.0130
CaK	3.18	3.74	0.0247	1.0754	0.7108	1.0150
TiK	0.23	0.23	0.0019	0.9816	0.8208	1.0361
NdL	0.25	0.08	0.0025	0.8655	1.0483	1.0831
FeK	1.55	1.31	0.0171	0.9854	0.9519	1.1747
CuK	65.28	48.35	0.6167	0.9597	0.9843	1.0000
Total	100.00	100.00				

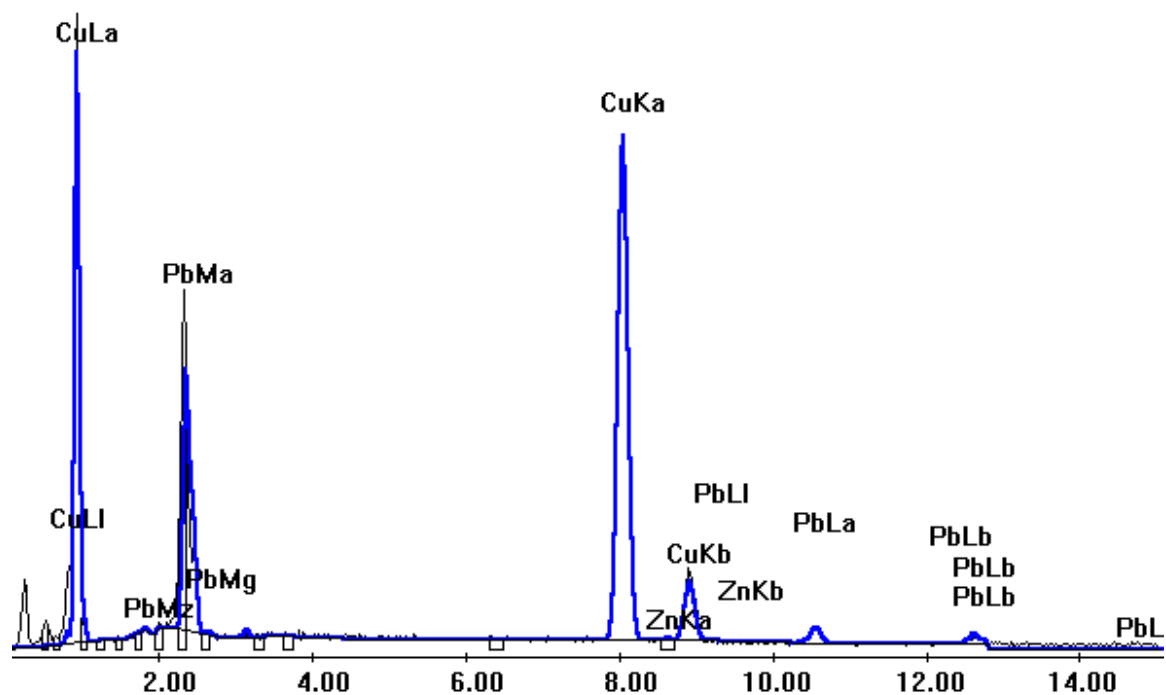
Comment. We may confidently identify all the listed elements except **Nd**, which is registered with little certainty.

Point 7203x1

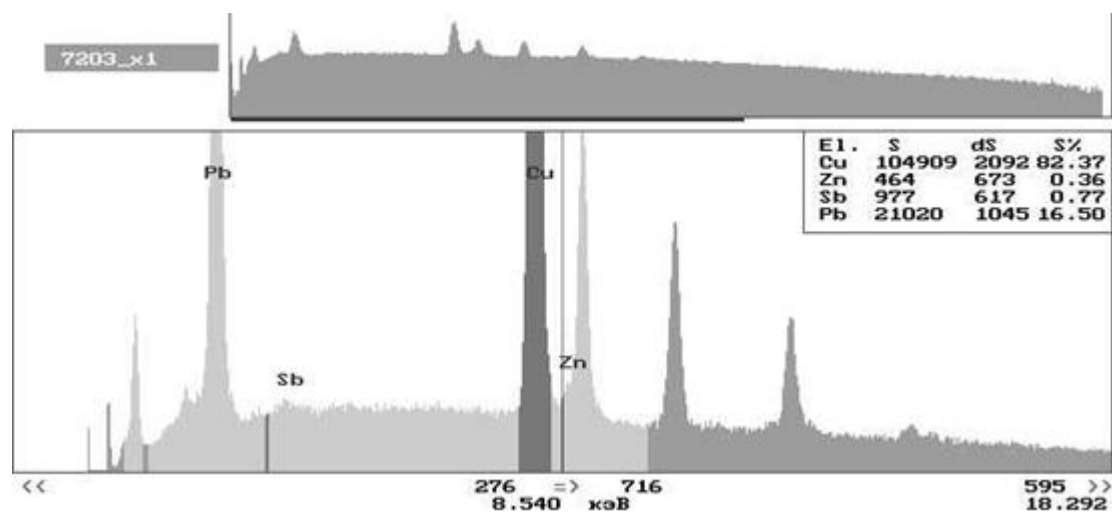
Spectrum from this analysis:

Untitled:1

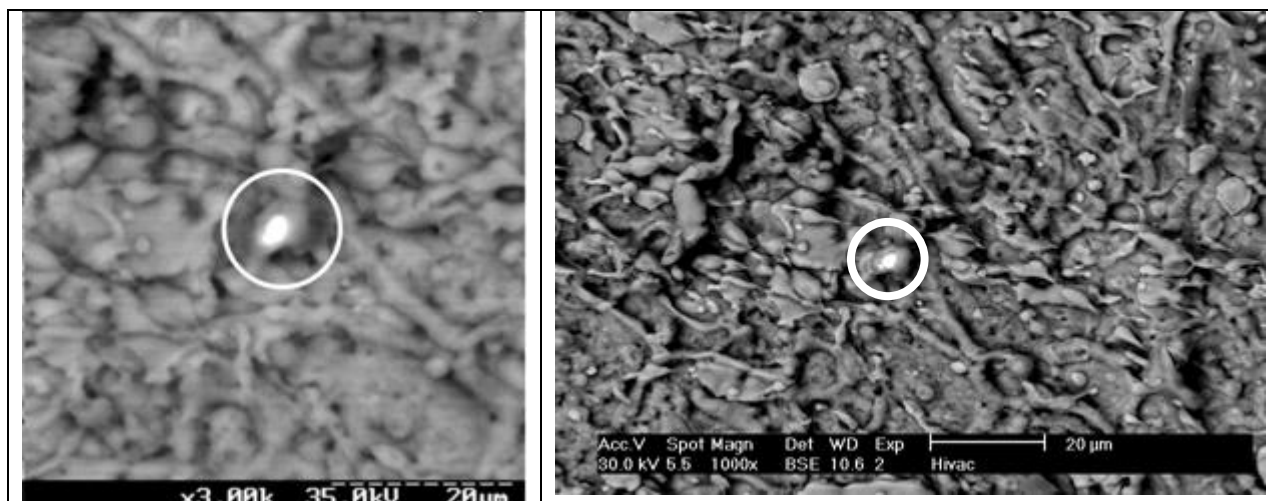
Label A:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



Quantitative analysis:

**EDAX ZAF Quantification (Standardless)
Element Normalized**

Element	Wt %	At %	K-Ratio	Z	A	F
CuK	85.16	94.57	0.8574	1.0176	0.9775	1.0122
ZnK	0.51	0.55	0.0052	1.0221	0.9855	1.0167
PbL	14.33	4.88	0.1133	0.8340	0.9485	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
CuL	290.92	3.08	0.73	94.59
PbM	168.44	13.00	0.98	12.96
CuK	523.92	10.48	0.54	49.97
ZnK	2.77	10.61	16.24	0.26
PbL	18.26	6.74	3.37	2.71

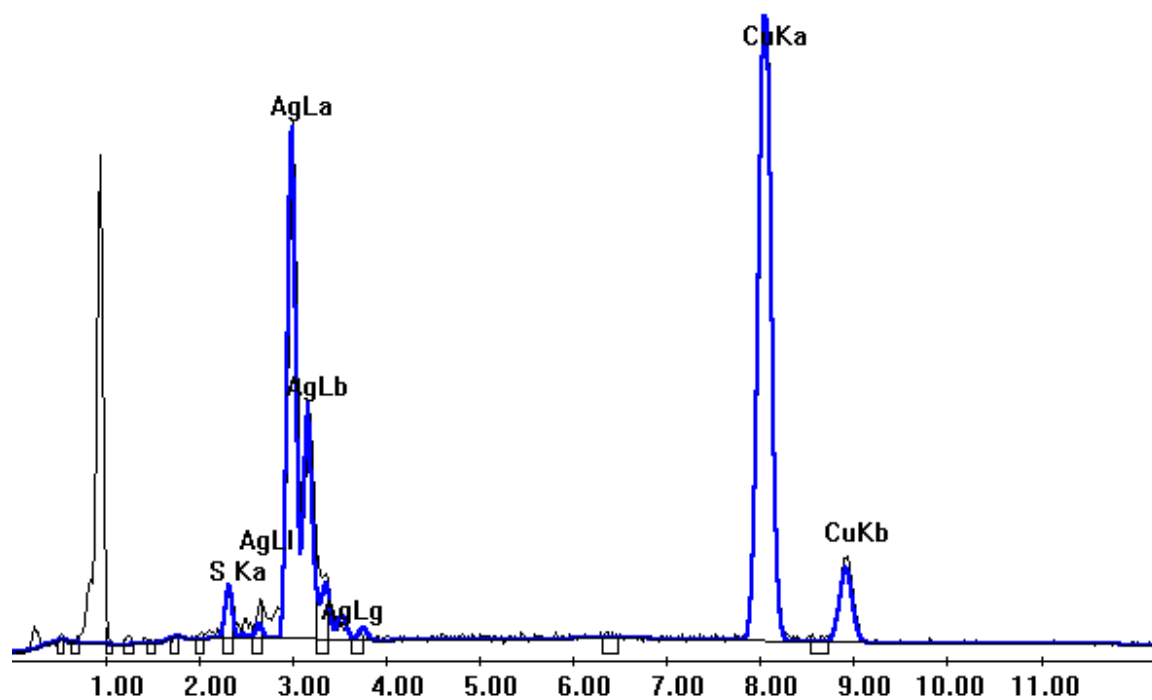
Comment. We may confidently register **Zn** and **Pb**. We could not with the same level of confidence register **Sb**.

Point 7203x2

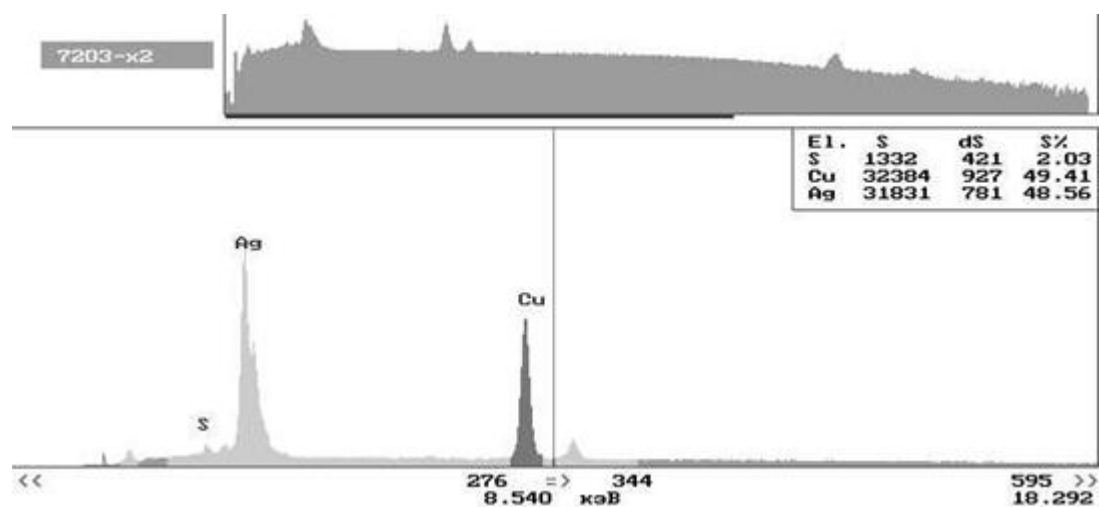
Spectrum from this analysis:

Untitled:1

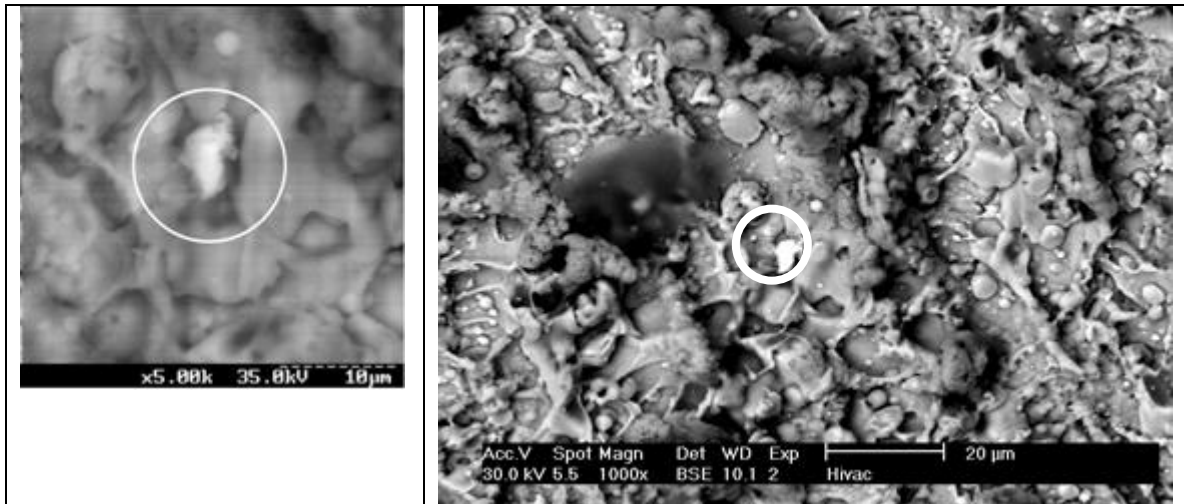
Label A:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



Quantitative analysis:

EDAX ZAF Quantification (Standardless) Element Normalized

Element	Wt %	At %	K-Ratio	Z	A	F
S K	1.88	4.33	0.0095	1.1396	0.4385	1.0153
AgL	38.78	26.59	0.3110	0.9512	0.8432	1.0000
CuK	59.34	69.08	0.5723	1.0209	0.9446	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
S K	18.25	5.92	2.77	3.08
AgL	192.93	6.51	0.75	29.63
CuK	362.15	8.18	0.55	44.26

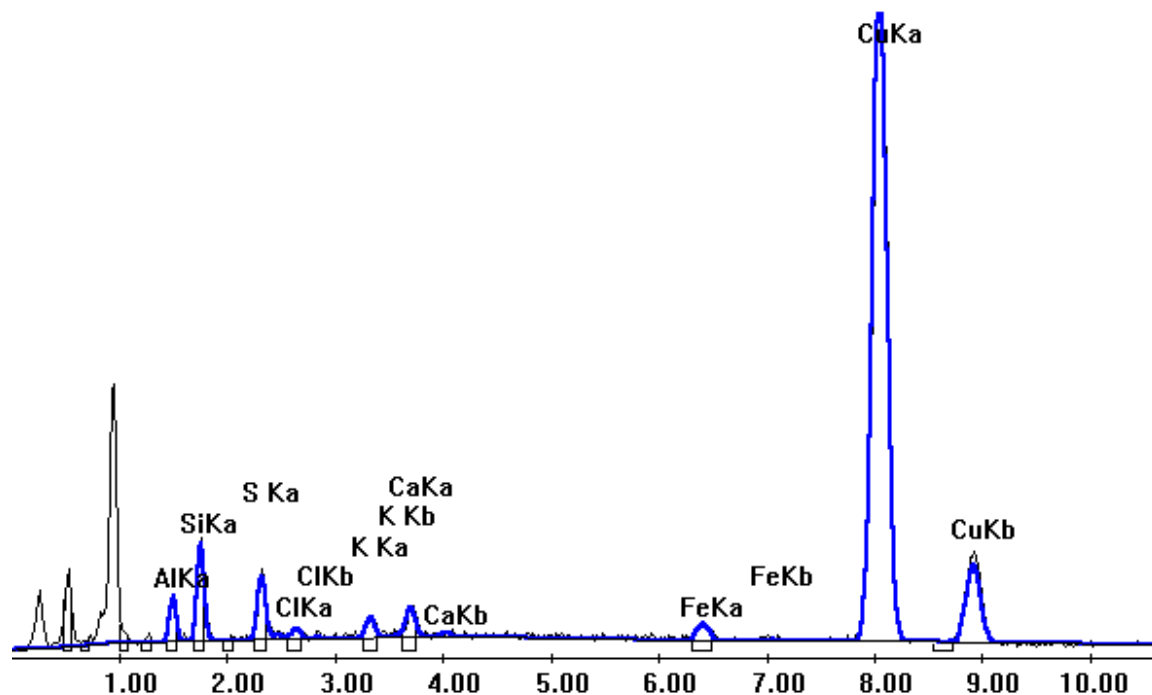
Comment. We confidently register Ag and S.

Point 7203x3

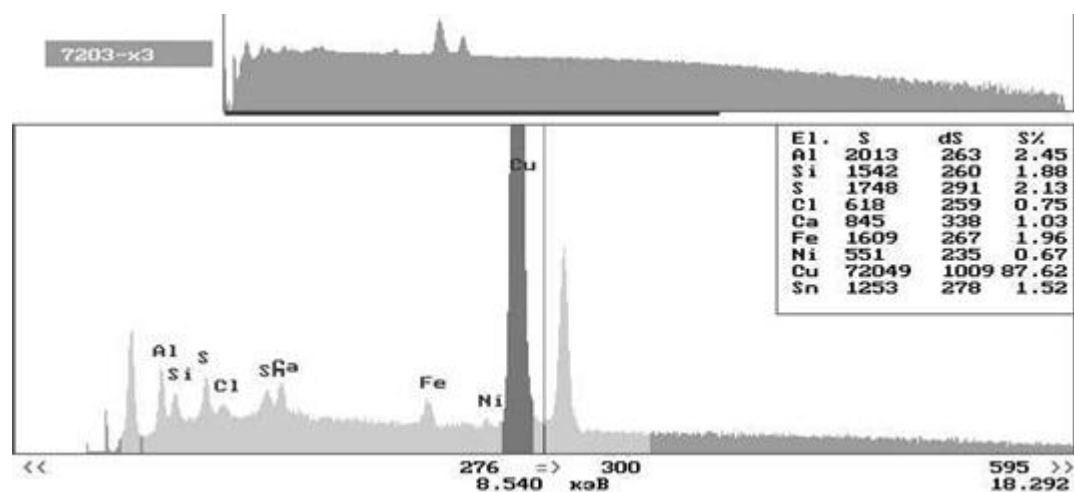
Spectrum from this analysis:

Untitled:1

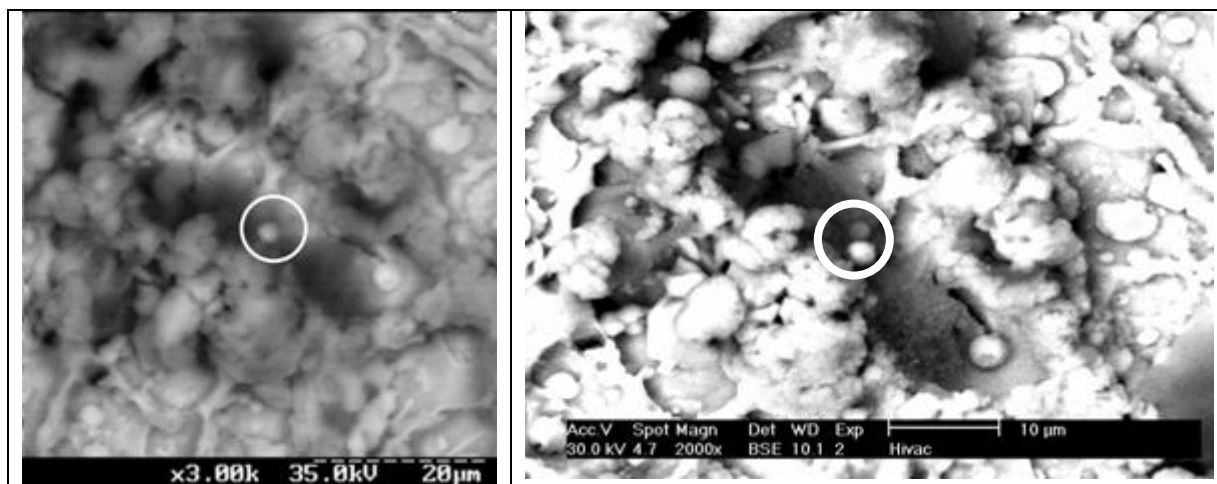
Label A:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



Quantitative analysis:

**EDAX ZAF Quantification (Standardless)
Element Normalized**

Element	Wt %	At %	K-Ratio	Z	A	F
AlK	4.39	8.57	0.0081	1.0727	0.1719	1.0019
SiK	7.54	14.13	0.0189	1.1047	0.2266	1.0011
S K	3.59	5.88	0.0148	1.0972	0.3756	1.0010
ClK	0.53	0.79	0.0026	1.0507	0.4591	1.0012
K K	0.81	1.10	0.0058	1.0699	0.6545	1.0131
CaK	1.20	1.57	0.0098	1.0939	0.7316	1.0190
FeK	1.01	0.95	0.0121	1.0010	0.9654	1.2422
CuK	80.92	67.01	0.7839	0.9754	0.9931	1.0000
Total	100.00	100.00				

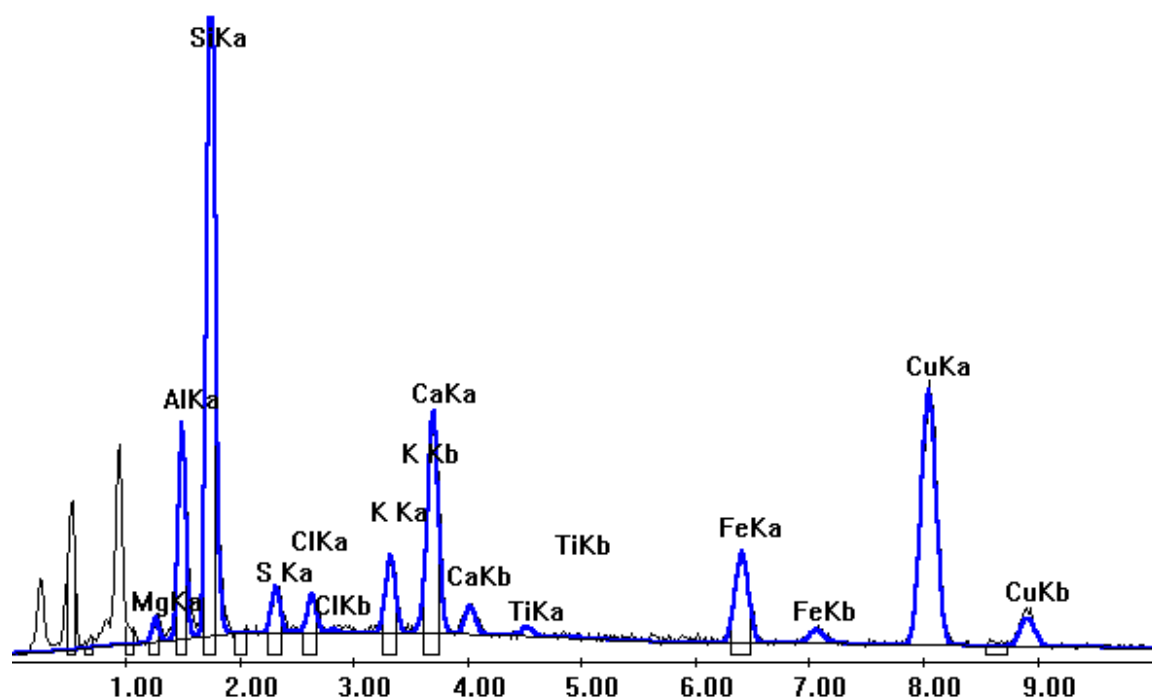
Comment. We may confidently register **Al, Si, S, Cl, K, Ca, Fe**. We could not with the same level of confidence register **Ni** and **Sn**.

Point 7203x4

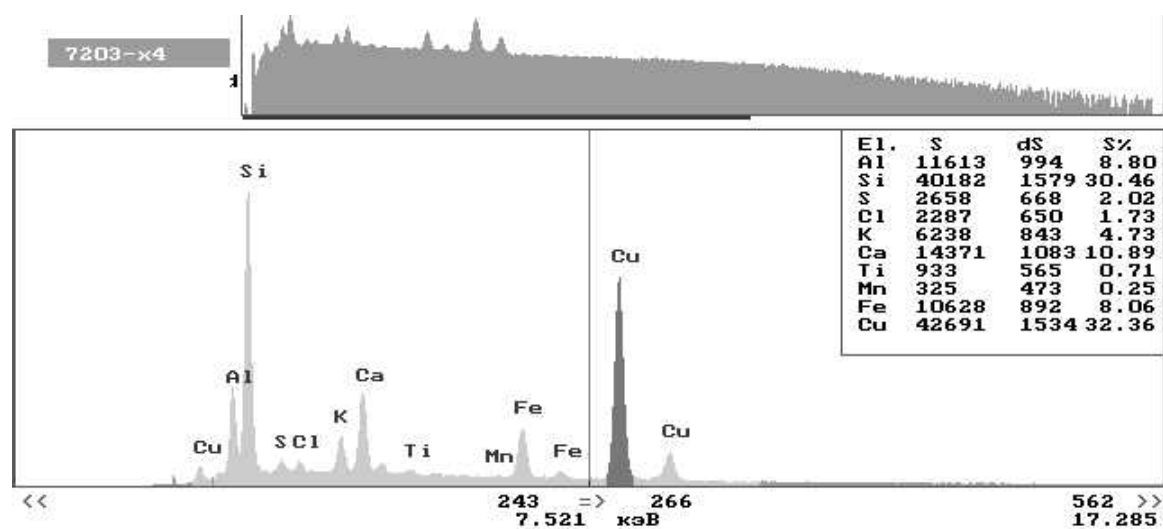
Spectrum from this analysis:

Untitled:1

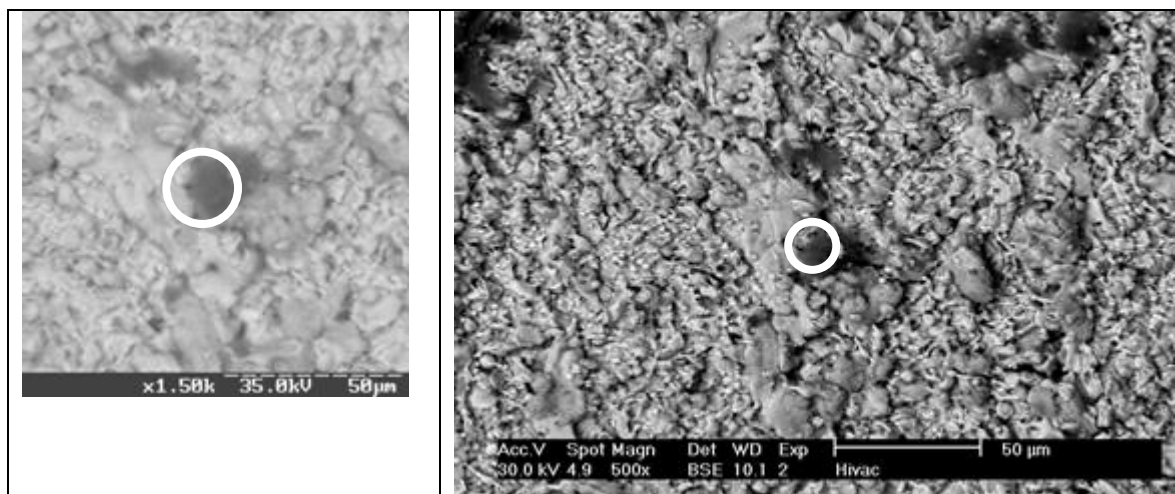
Label A:



Spectrum presented by Proton21:



Microphotographs: Left – original from Proton21, right – this analysis



Quantitative analysis:

EDAX ZAF Quantification (Standardless) Element Normalized

Element	Wt %	At %	K-Ratio	Z	A	F
MgK	1.57	2.35	0.0038	1.0450	0.2311	1.0082
AlK	10.24	13.77	0.0330	1.0152	0.3138	1.0116
SiK	37.12	47.97	0.1388	1.0456	0.3567	1.0024
S K	2.39	2.71	0.0093	1.0388	0.3705	1.0049
ClK	1.75	1.79	0.0080	0.9949	0.4591	1.0066
K K	3.01	2.79	0.0198	1.0030	0.6464	1.0160
CaK	8.90	8.06	0.0654	1.0260	0.7098	1.0087
TiK	0.49	0.37	0.0037	0.9388	0.7897	1.0191
FeK	5.80	3.77	0.0547	0.9435	0.9379	1.0662
CuK	28.74	16.42	0.2551	0.9177	0.9673	1.0000
Total	100.00	100.00				

Comment. We may confidently register **Mg, Al, Si, S, Cl, K, Ca, Ti** and **Fe**. We could not with the same level of confidence register **Mn**.