

Application of the WDXRF AR35 on-stream analyzer for determination of the content of Fe, Co, Ni, Cu and S in slurry products

INTRODUCTION

1.1 Determination of average elements in polymetallic ores

The analysis of the content of iron Fe, cobalt Co, nickel Ni and copper Cu during the technological process of polymetallic ore enrichment by X-ray fluorescence analysis

11	12																	13	14	15	16	17	18
Na	Mg																	Al	Si	P	S	Cl	Ar
22,98976928	24,305																	26,9815385	28,0855	30,9737619	32,06	35,453	39,948
19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36						
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr						
39,0983	40,08	44,9559	47,88	50,9415	51,996	54,938	55,847	58,9332	58,69	63,546	65,38	69,72	72,59	74,9216	78,96	79,904	83,8						
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54						
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe						
85,4678	87,62	88,9059	91,22	92,9064	95,94		101,07	102,905	106,42	107,868	112,41	114,81	118,71	121,75	127,6	126,9054	131,29						

(XRF) using the analyzer AR-35 is a typical task and does not present any difficulties.

1.2 Determination of light elements in polymetallic ores

11	12																	13	14	15	16	17	18
Na	Mg																	Al	Si	P	S	Cl	Ar
22,9897	24,305																	26,9815	28,0855	30,9737	32,06	35,45	39,948
19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36						
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr						
39,0983	40,08	44,9559	47,88	50,9415	51,9961	54,938	55,845	58,9332	58,69	63,546	65,36	69,72	72,56	74,9216	78,96	79,904	83,8						
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54						
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe						
85,4678	87,62	88,9059	91,22	92,9064	95,94		101,07	102,906	106,42	107,868	112,41	114,82	118,71	121,75	127,6	126,905	131,29						

The determination of light elements by the XRF method is complicated by the fact that the fluorescent lines of these elements are strongly absorbed by air, as well as by the materials of

the separation windows all the way from the sample to the detector.

Direct analysis of sulfur S without additional improvements that reduce the absorption of lines of light elements is impossible due to the almost complete absorption of lines $SK_{\alpha,\beta}$ by the volume of air between the sample and the working surface of the detector.

EQUIPMENT AND METHODS

To measure the pulp samples, the AR-35 analyzer configuration was used, including:

- X-ray tube with Pd anode;
- spectrometric channels on the line FeK_{α} , CoK_{α} , NiK_{α} , CuK_{α} , SK_{α} and scattered radiation Pd_{inc} ;
- pneumatic shutter with primary X-ray filter;
- measuring cuvette with PET working and protective film.

Two measurement modes were used, which differ in the use of primary X-ray filtration.

TESTING ON REAL SLURRY SAMPLES

A set of 16 samples presented by four technological products was used as test objects:

- Condensed nickel Concentrate;

- Nickel-pyrrhotite concentrate;
- Metal-containing product;
- Condensed nickel sulfide concentrate.

Parameters of the constructed calibration dependencies:

Element	Content range, %	Regression equation	Residual calibration error σ , % macc.	Linear correlation coefficient, R
Fe	from 26.6 to 42.5	$C_P = a_0 + a_1 I_{FeK\alpha} + a_2 I_{CuK\alpha} + a_3 I_{Pdinc}$	0.503	1
Co	from 0.034 to 0.371	$C_P = a_0 + a_1 I_{CoK\alpha} + a_2 I_{CuK\alpha} + a_3 I_{FeK\alpha}$	0.0055	1
Ni	from 0.87 to 8.51	$C_P = a_0 + a_1 I_{CoK\alpha} + a_2 I_{FeK\alpha} + a_3 I_{CuK\alpha}$	0.113	1
Cu	from 0.39 to 3.58	$C_P = a_0 + a_1 I_{CuK\alpha} + a_2 I_{FeK\alpha} + a_3 I_{Pdinc}$	0.058	1
S	from 17.1 to 30.1	$C_P = a_0 + a_1 I_{SK\alpha} \cdot I_{SK\alpha} + a_2 I_{SK\alpha} \cdot I_{CuK\alpha} + a_3 I_{SK\alpha} \cdot I_{FeK\alpha} + a_4 I_{Pdinc}$	0.393	1

The values of the detection limits calculated from measurements of real slurry samples:

Element	Fe	Co	Ni	Cu	S
Background intensity, imp/s	13.0	9.0	19.0	61.0	15.0
Cipher of the initial sample	11	22	33	44	55
Intensity of the line from the slurry sample, imp/s	8650	44.3	564	429	73
Concentration of the element in the sample, %	26.6	0.034	0.87	0.39	17.1
Exposition T, s	40	40	40	40	40
Definition limit, %	0.018	0.005	0.011	0.013	1.81

CONCLUSIONS

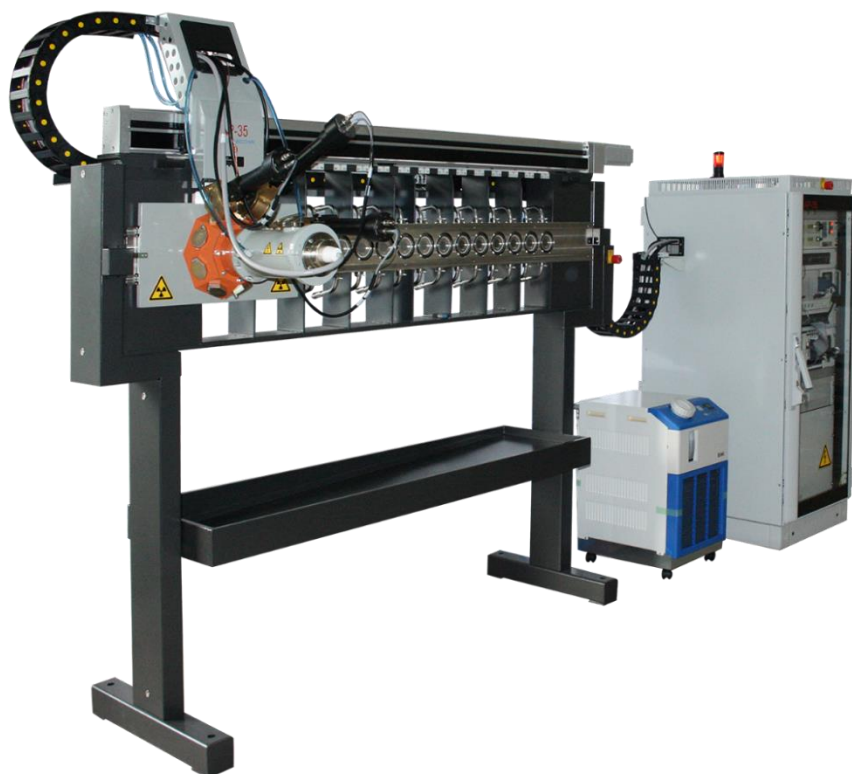
1. The on-stream WDXRF analyzer AR-35 makes it possible to determine the concentrations of iron Fe, cobalt Co, nickel Ni and copper Cu in the solid phase of liquid pulp samples with an intrinsic analysis error of up to 10% relative.

The determination of sulfur S can be carried out after the completion of the AR-35 analyzer and the installation of a spectrometric channel. The error of the sulfur S analysis will range from 5% to 20% relative, depending on the concentration range.

2. The values of the detection limits for Fe, Co, Ni and Cu are at the level of 0.01%, and for S the detection limit was 1.8%.

The value of the limits of determination and the final error of the analysis depends on the properties of the product: slurry density, fineness of grinding, mineralogical composition, etc. Methodological recommendations have been developed to improve the analysis indicators, which are selected individually for each enterprise.

3. The measurement time of one product starts from 40 seconds, the total cycle time for 15 lines is no more than 15 minutes.

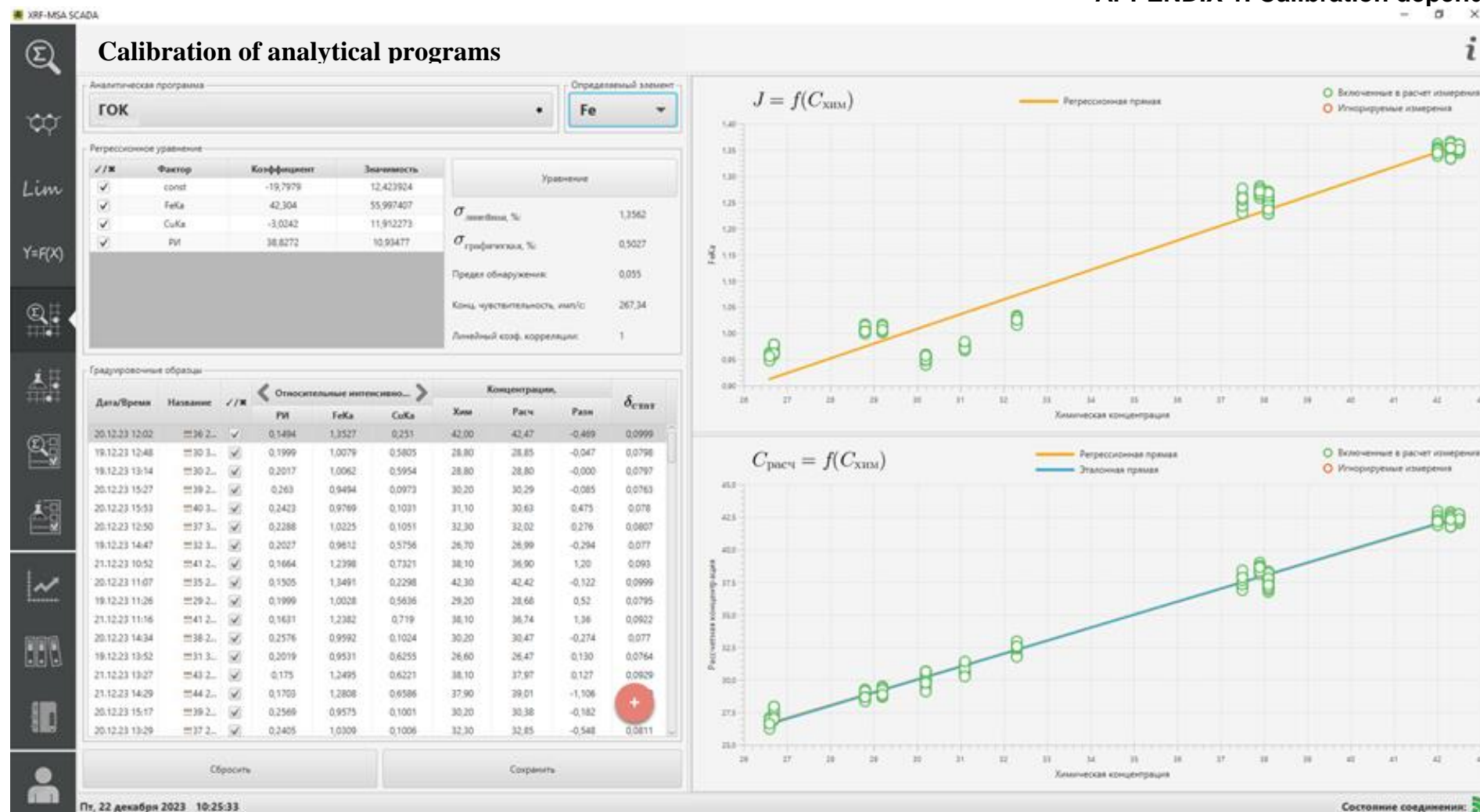


In the photo: AR-35 analyzer for 15 measuring cell, cooling system, automatic control stand

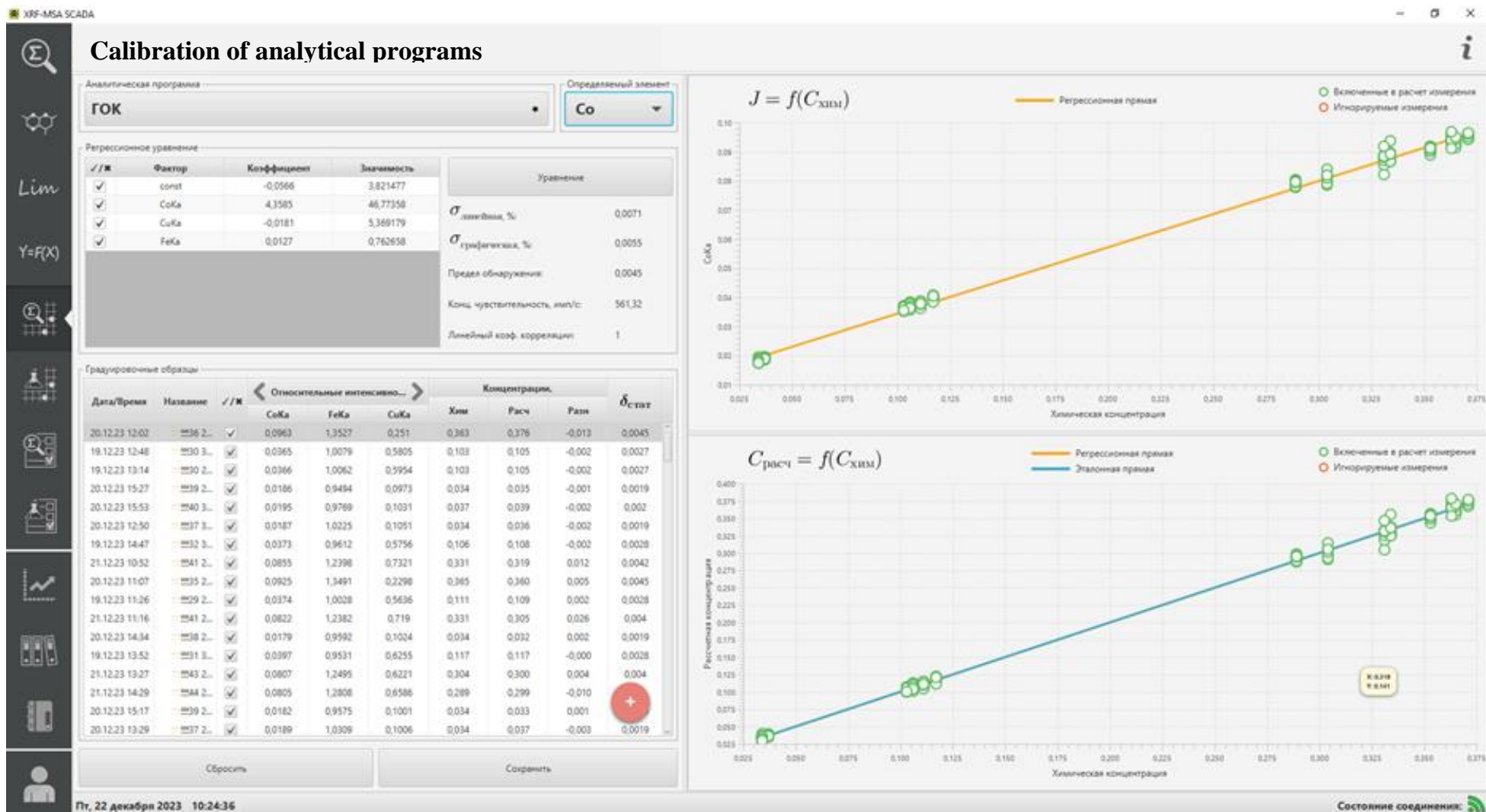
Applications:

Appendix 1. Visualization of calibration.

APPENDIX 1. Calibration dependencies



Picture 1 – Calibration dependence for Fe.



Picture 2 – Calibration dependence for Co.

XRF-MSA SCADA

Calibration of analytical programs

Аналитическая программа: **ГОК** Определяемый элемент: **Ni**

Регрессионное уравнение

✓/✗	Фактор	Коэффициент	Значимость	Уравнение
✓	const	-1,9386	6,896599	$\sigma_{\text{линейная, \%}}$ 0,3552 $\sigma_{\text{графиковая, \%}}$ 0,1129 Предел обнаружения: 0,0123 Конц. чувствительности, мкг/с: 567,03 Линейный коэф. корреляции: 1
✓	NiKa	14,4567	51,767863	
✓	FeKa	1,9961	6,660037	
✓	CuKa	-1,0009	9,641617	

Градуировочные образцы

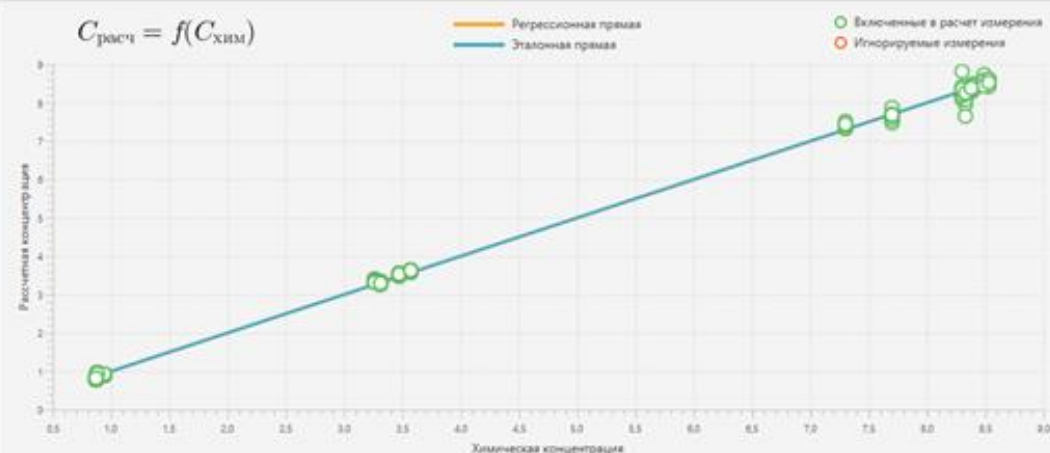
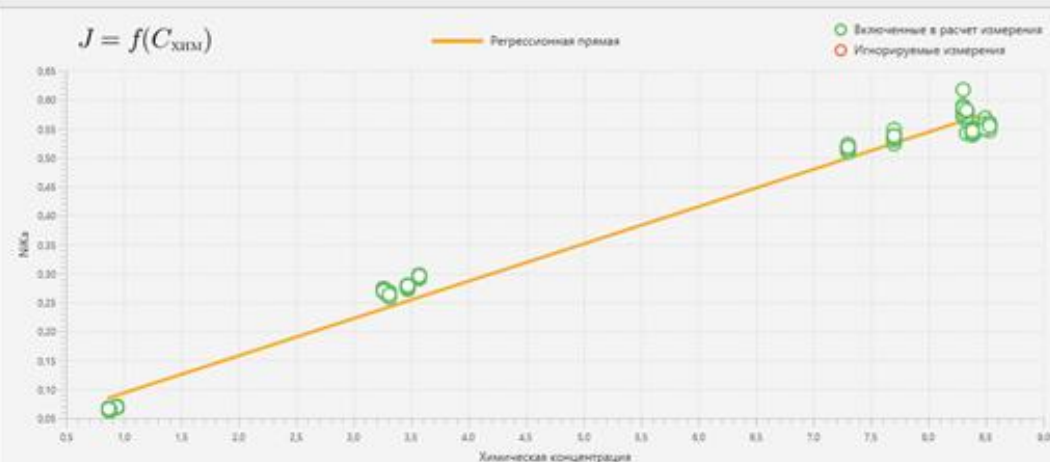
Дата/Время	Название	✓/✗	Относительные интенсивности...			Концентрации			$\delta_{\text{стат}}$
			NiKa	FeKa	CuKa	Хим	Расч	Разн	
20.12.23 12:02	№36 2...	✓	0,5576	1,3527	0,291	8,49	8,57	-0,081	0,0242
19.12.23 12:40	№30 3...	✓	0,2643	1,0079	0,5805	3,31	3,31	-0,004	0,0151
19.12.23 13:14	№30 2...	✓	0,2674	1,0062	0,5954	3,31	3,34	-0,029	0,0152
20.12.23 15:27	№39 2...	✓	0,0644	0,9494	0,0973	0,87	0,79	0,080	0,0068
20.12.23 15:53	№40 3...	✓	0,0689	0,9769	0,1031	0,94	0,90	0,035	0,0071
20.12.23 12:50	№37 3...	✓	0,0658	1,0225	0,1051	0,88	0,95	-0,069	0,0069
19.12.23 14:47	№32 3...	✓	0,2732	0,9612	0,5756	3,26	3,35	-0,094	0,0154
21.12.23 10:52	№41 2...	✓	0,564	1,2390	0,7321	8,33	7,96	0,373	0,0242
20.12.23 11:07	№35 2...	✓	0,5488	1,3491	0,2298	8,40	8,46	-0,057	0,024
19.12.23 11:26	№29 2...	✓	0,2795	1,0028	0,5636	3,47	3,54	-0,070	0,0156
21.12.23 11:16	№41 2...	✓	0,542	1,2382	0,719	8,33	7,65	0,68	0,0231
20.12.23 14:34	№38 2...	✓	0,0649	0,9592	0,1024	0,87	0,81	0,059	0,0069
19.12.23 13:52	№31 3...	✓	0,2949	0,9531	0,6255	3,57	3,60	-0,032	0,0161
21.12.23 13:27	№43 2...	✓	0,5322	1,2495	0,6221	7,70	7,63	0,074	0,0229
21.12.23 14:29	№44 2...	✓	0,5226	1,2808	0,6586	7,30	7,51	-0,214	
20.12.23 15:17	№39 2...	✓	0,0652	0,9575	0,1001	0,87	0,81	0,055	
20.12.23 13:29	SP-00137 2...	✓	0,0641	1,0309	0,1006	0,88	0,95	-0,066	0,0068

Сбросить

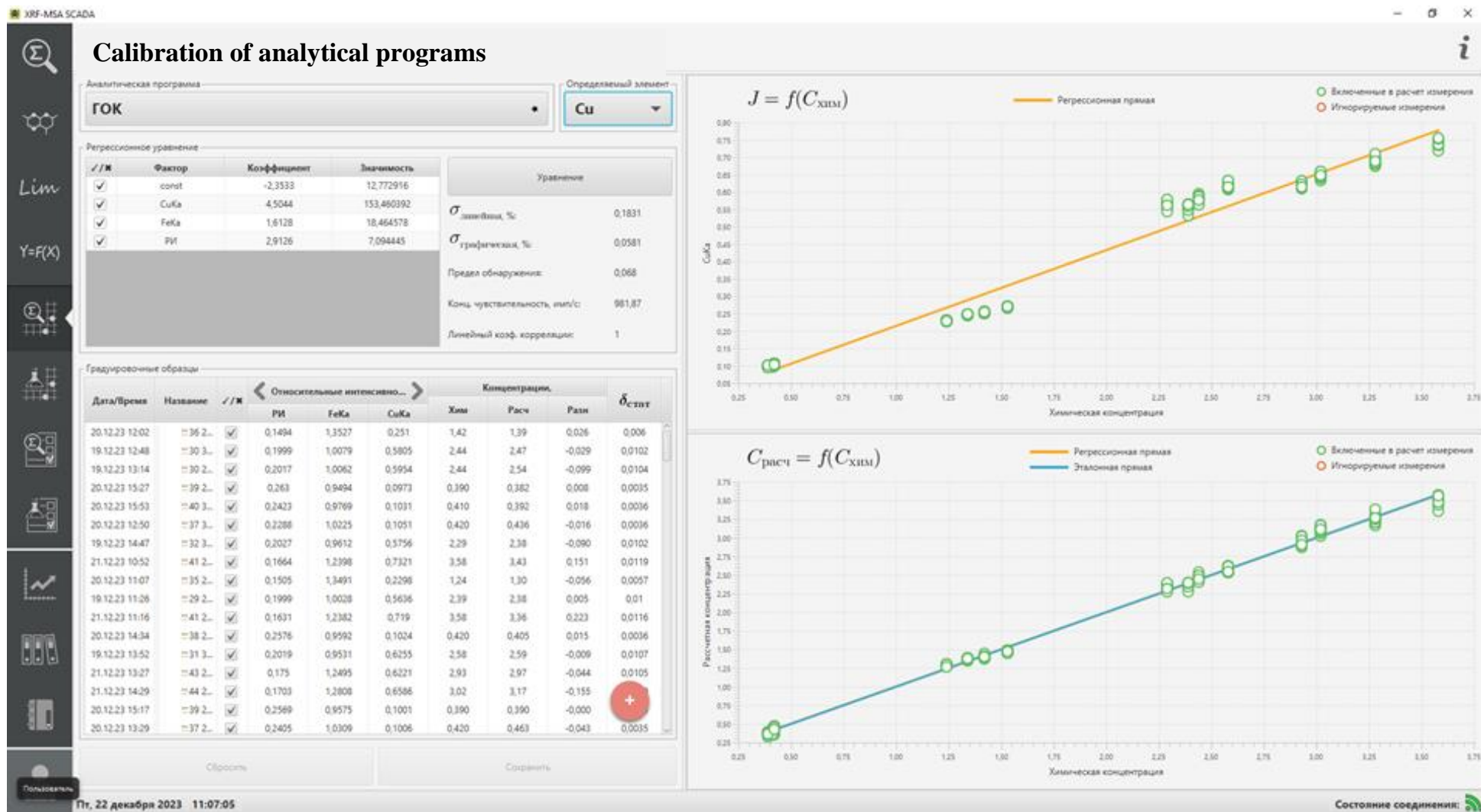
Сохранить

Пт, 22 декабря 2023 10:23:49

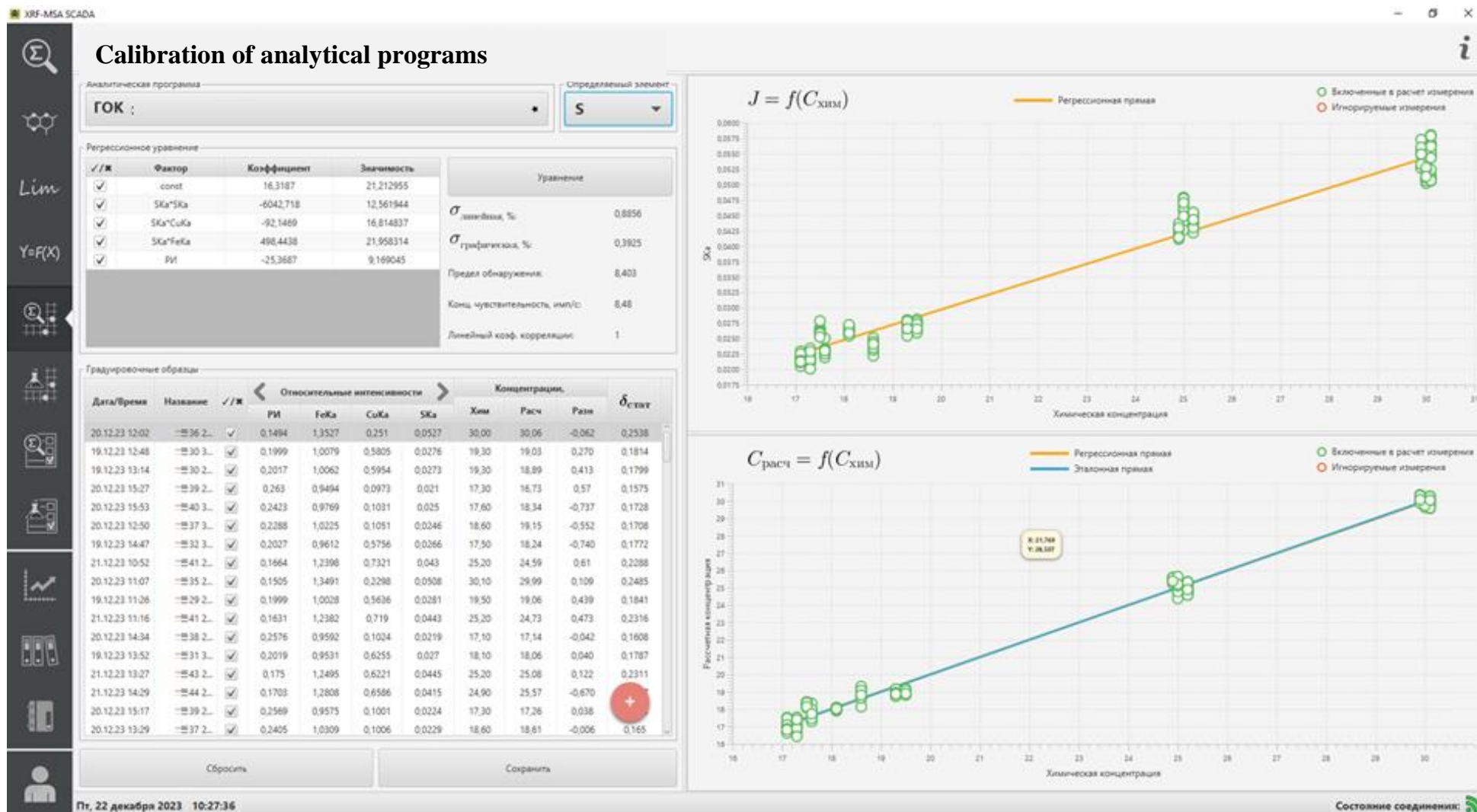
Состояние соединения:



Picture 3 – Calibration dependence for Ni.



Picture 4 – Calibration dependence for Cu.



Picture 5 – Calibration dependence for S.