Appendix 13. Report by Nikolay Kharkov on the 2003 JM test entitled: "Testing of Johnson Matthey's control samples."

FSS Criminalistics Institute of Russia

Testing of Johnson Matthey's control samples

Moscow July, 2003

In July, 2003 the Institute of Criminalistics of the Russian Federal Security Service in collaboration with Johnson Matthey plc (UK) conducted experimental testing of the Complex Analytical Procedure for Identification of the Nature and the Source of Origin of Precious Metals Containing Products of Mining and Metallurgical Operations developed by several Russian institutions in cooperation with MMC Norilsk Nickel. In the framework of this collaborative exercise three control samples from JM were tested, composed of secondary raw material that the company received for processing. Samples were marked A, B and C accordingly. It was necessary to determine if their compositions included any products of South-African mining/metal companies. Also provided was a list of 8 previously tested S.A. samples, potentially used in the preparation of control samples (see Table 1). Sample numeration in the Table corresponds to that in the S.A. production databank.

TESTING

The samples were tested using our Complex analytical procedure for determining the nature and the source of origin of precious metal-bearing products of mining and metallurgical operations, and initial database of S.A. production.

In the phase composition testing it was established that all three samples had the same basic structure (difractogram on Fig. 1), formed of copper oxides and a phase based on copper and/or copper-based intermetallides. Natrium chloride was also present in all three compositions at the level of 3-5 mass. %.

In the analysis of impurity phase composition the “fingerprints method” was used. Each control sample spectrum was compared with spectra of individual S.A. products from the database. One line, common for all three control samples, was taken as a basic line of difractograms.

It was established that the A-sample composition included A11 (Anglo Platinum concentrate) in the amount exceeding 3 mass. % (detection limit for this method) (see part of difractogram on Fig. 2).

In the B-sample spectrum, the lines (diffraction maximums) of A1, A5 and A19 were detected (Fig. 3). The difractograms of these S.A. products are practically the same. Thus, based on the phase analysis of B-sample it was established that its composition could include over 3 mass. % of substance of one of the samples (A1, A5 or A19), or their mixture.

In the phase analysis of C-sample, S.A. products in the amount over 3 mass. % were not detected.

Results of the gross element composition determination, as well as previous data on S.A. samples are shown in Table 2.

The data on the gross element composition of the control samples was analyzed by the way of iterative composition modelling.

It was established that the closest to the experimental data was a model mixture, containing:

- for control sample A: about 5 mass. % of A11 sample (Anglo Platinum Concentrate);
- for control sample B: about 5 mass. % of A1 sample (Impala Platinum Converter Matte);
- for control sample C: A11 sample (Anglo Platinum Concentrate) and A1 sample (Impala Platinum Converter Matte) with total content of about 5 mass. %.

To verify the modeling results, the microparticles in the control mixture samples were examined, in the amount not less than 1 000 microparticles from each control sample (A, B and C).

The examination results show that the material of all samples is basically formed of particles and their aggregates which include copper oxide, copper, and copper oxide with precious metals (Pd, Ag, Pt, Ru, Rh) and other specific elements (Te, As, Se, etc.) Typical view and element composition of such particles are

shown on Fig. 4 and Fig. 5. Their proportion in the base material of control samples is not less than 95%.

Along with the base microparticles some other specific microparticles were detected.

Sample A

In the material of the sample A over 50 microparticles of the following types were found:

Si–Mg–O–Fe–Ca;

Cr–Fe–Al–Mg–Si–O–Ti;

Fe–Cu–S–Si–Mg–Al–O.

Such particles are typical for Anglo Platinum concentrate (sample A11). Their total share in the material of the concentrate amounts to 95%. No other microparticles (typical for S.A. products examined in the past) were found in this control sample.

Based on the test results it was concluded that the material of A-control sample contained Anglo Platinum concentrate (sample A11).

Sample B

In the material of this sample over 50 microparticles of nickel and copper sulphides were found (in 60:40 ratio), referred to the following types:

Ni–Cu–Fe–Co**–S–**(O);

Cu–Ni–Fe–Co**–S–**O

These particles represent over 95% of particles in other S.A. products, examined earlier: Impala Platinum converter matte (A1), Anglo Platinum's WCM (A5), Lonmin Platinum's matte after granulation (A19). No other typical S.A. microparticles were not found in this control sample.

Particles of the type **Cu-Ni**-Fe-Co-**S**-O are common for all three S.A. samples, so in order to differentiate materials from A1, A5 and A19 we took particles **Ni-Cu**-Fe-Co-**S**-(O).

Comparing the element composition of these microparticles in the B-sample with compositions of similar microparticles in the products listed above, the following was established:

In most microparticles of the **Ni-Cu**-Fe-Co-**S**-(O) – type found in the B-sample, the presence of cobalt was continuously registered. In the Lonmin Platinum's matte after granulation (A19) there was no cobalt (or its content was below the detection limit). This allows to exclude this product from those that might be present in the B-sample.

Copper and cobalt contents in these microparticles (in the B-sample) were very close to those contained in the Impala Platinum converter matte (A1). In the Anglo Platinum's WCM (A5), the copper content is much higher and that of cobalt is lower than in the particles of this control sample.

Based on the test results it was concluded that the material of B-control sample contained Impala Platinum converter matte (sample A1).

Sample C

In the composition of this sample over 50 microparticles of the following types were found:

Si–Mg–O–Fe–Ca ;

Cr–Fe–Al–Mg–Si–O–Ti;

Fe–Cu–S–Si–Mg–Al–O;

Ni–Cu–Fe–Co**–S–**(O);

Cu–Ni–Fe–Co**–S–**O.

These particles were identical to those present in A and B control samples but their quantities in C-sample were much lower.

Based on the test results it was concluded that the material of C-control sample contained microparticles of Anglo Platinum concentrate (A11) and Impala Platinum converter matte (sample A1).

CONCLUSIONS

The results of complex testing carried out with A, B and C control samples submitted by Johnson Matthey, and their matching with the initial databank of S.A. products have led to the following conclusions:

1. The A-sample composition includes about 5 mass. % of the Anglo Platinum concentrate.
2. The B-sample composition includes about 5 mass. % of the Impala Platinum converter matte .
3. The C-sample composition includes Anglo Platinum concentrate and Impala Platinum converter matte in the total amount of approx. 5 mass. %.

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Table 1.

ANGLO PLATINUM SAMPLES			
SAMPLE NUMBER	**DESCRIPTION OF SAMPLE**	**WEIGHT**	**DESCRIPTION OF PROCESS**
Sample 1	Granulated Matte	50.0 g	Furnace Process
Sample 2	WCM	50.0 g	Furnace Process
Sample 3	Concentrate	50,0 g	Mill plus Flotation Process

NORTHAM PLATINUM SAMPLES			
SAMPLE NUMBER	**DESCRIPTION OF SAMPLE**	**WEIGHT**	**DESCRIPTION OF PROCESS**
Sample 1	Final Concentrate	50,0 g	Electroletic Copper / Nickel Refining Process

LONMIN PLATINUM SAMPLES			
SAMPLE NUMBER	**DESCRIPTION OF SAMPLE**	**WEIGHT**	**DESCRIPTION OF PROCESS**
Sample 1	Pt Sponge WPR 2	50,0 g	Primary Separation Platinum Process after Ignition
Sample 2	Matte after Granulation	50,0 g	Oxidising to extract Sulphur (not metal) - Water granulation
Sample 3	Pressure Leach	50,0 g	Oxidising plus Sulpheric Acid Process

IMPALA PLATINUM SAMPLES			
SAMPLE NUMBER	**DESCRIPTION OF SAMPLE**	**WEIGHT**	**DESCRIPTION OF PROCESS**
Sample 1	Converter Matte	50,0 g	Mill Float and Smelt Convertor Process - Mixed Merensky plus UG2 Ore - 60/40 split

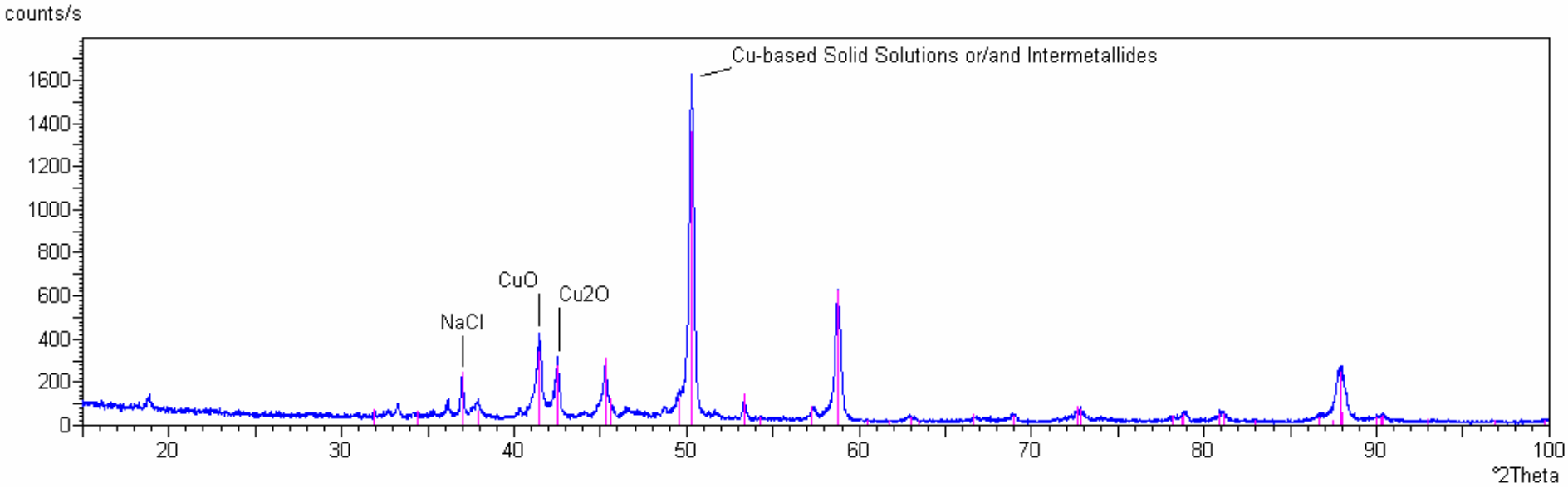


Fig. 1 Diffractogram of the base material of control samples

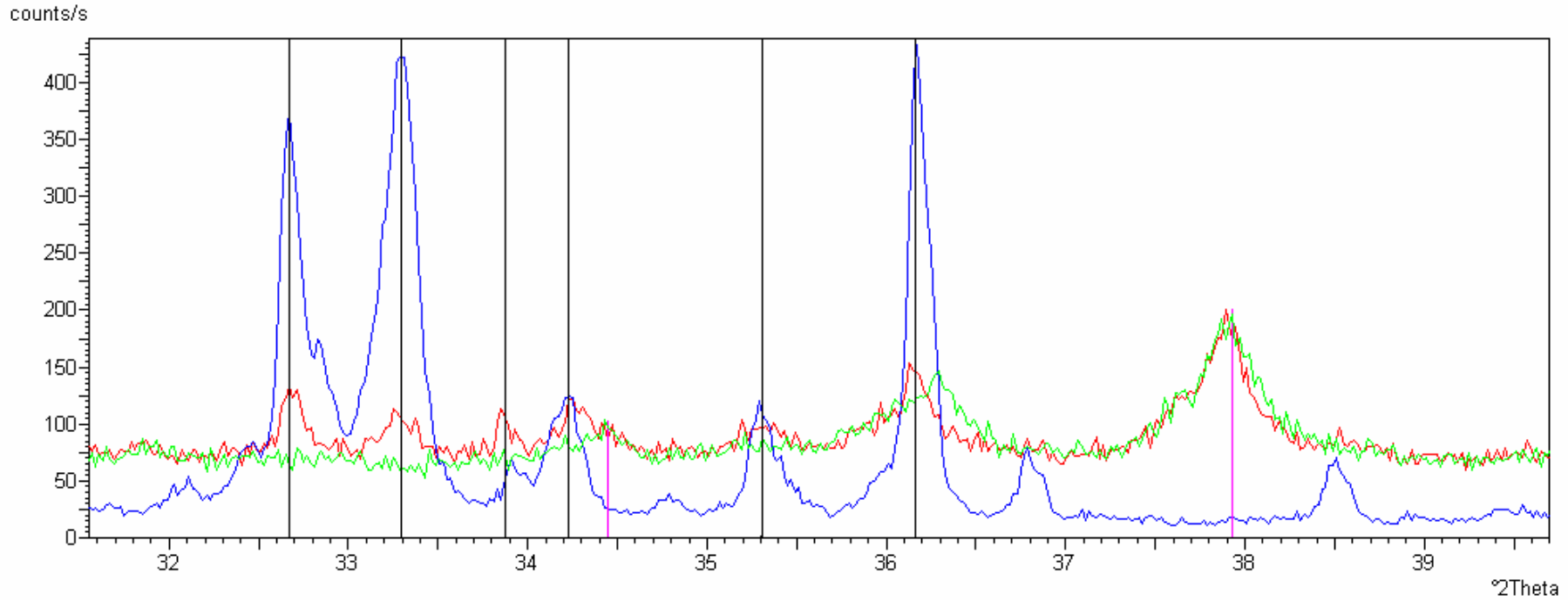


Fig. 2. Superposition of spectral fragments (A-sample – red, A11 – blue, base material – green). Black lines show the major peaks for A11 and rose lines show the peaks for base components.

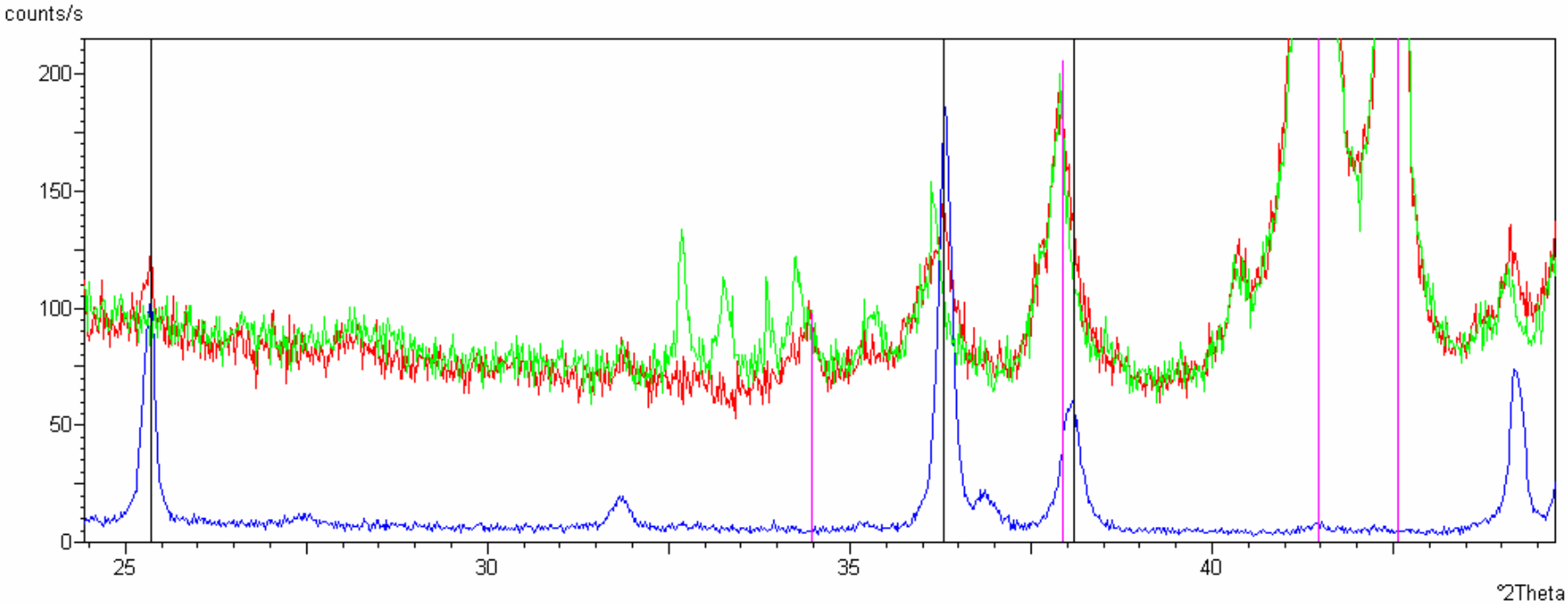


Fig. 3. Superposition of spectral fragments (B-sample – red, A1 – blue, base material – green). Black lines show the major peaks for A1 and rose lines show the peaks for base components.

Table 2

Composition of S.A. products and control samples

sample #	Product	Ni	Ni	Cu	Cu	Au	Au	Ag	Ag	Pt	Pt	Pd	Pd	Rh	Rh	Ru	Ru
A1	converter matte	44.1	%	31	%	56	ppm	106	ppm	1900	ppm	1010	ppm	195	ppm	244	ppm
A3	granulated matte	15.9	%	9.66	%	12	ppm	34	ppm	646	ppm	387	ppm	89	ppm	52	ppm
A5	WCM	53.5	%	19.2	%	113	ppm	113	ppm	986	ppm	1548	ppm	286	ppm	422	ppm
A11	concentrate	6875	ppm	3894	ppm	1.3	ppm	6	ppm	13	ppm	13	ppm	1	ppm	<10	ppm
A13	final concentrate	3.93	%	9.68	%	8900	ppm	7500	ppm	21.3	%	8.96	%	1.01	%	6254	ppm
A15	Pt sponge WPR 2	5	ppm	74	ppm	95	ppm	82	ppm	98.11	%	8790	ppm	345	ppm	<10	ppm
A19	matte after granulation	45.6	%	32.2	%	64	ppm	100	ppm	4035	ppm	2024	ppm	535	ppm	913	ppm
A21	(Pressure leach)	5.76	%	6.4	%	2543	ppm	4624	ppm	13.3	%	6.34	%	2.53	%	2720	ppm
A		3500	ppm	55.4	%	934	ppm	5950	ppm	1.98	%	5.48	%	4090	ppm	323	ppm
B		2.22	%	57.1	%	1040	ppm	6450	ppm	2	%	5.48	%	4450	ppm	607	ppm
C		1.29	%	55.6	%	1070	ppm	6230	ppm	1.99	%	5.29	%	4620	ppm	476	ppm

table 2 cont.

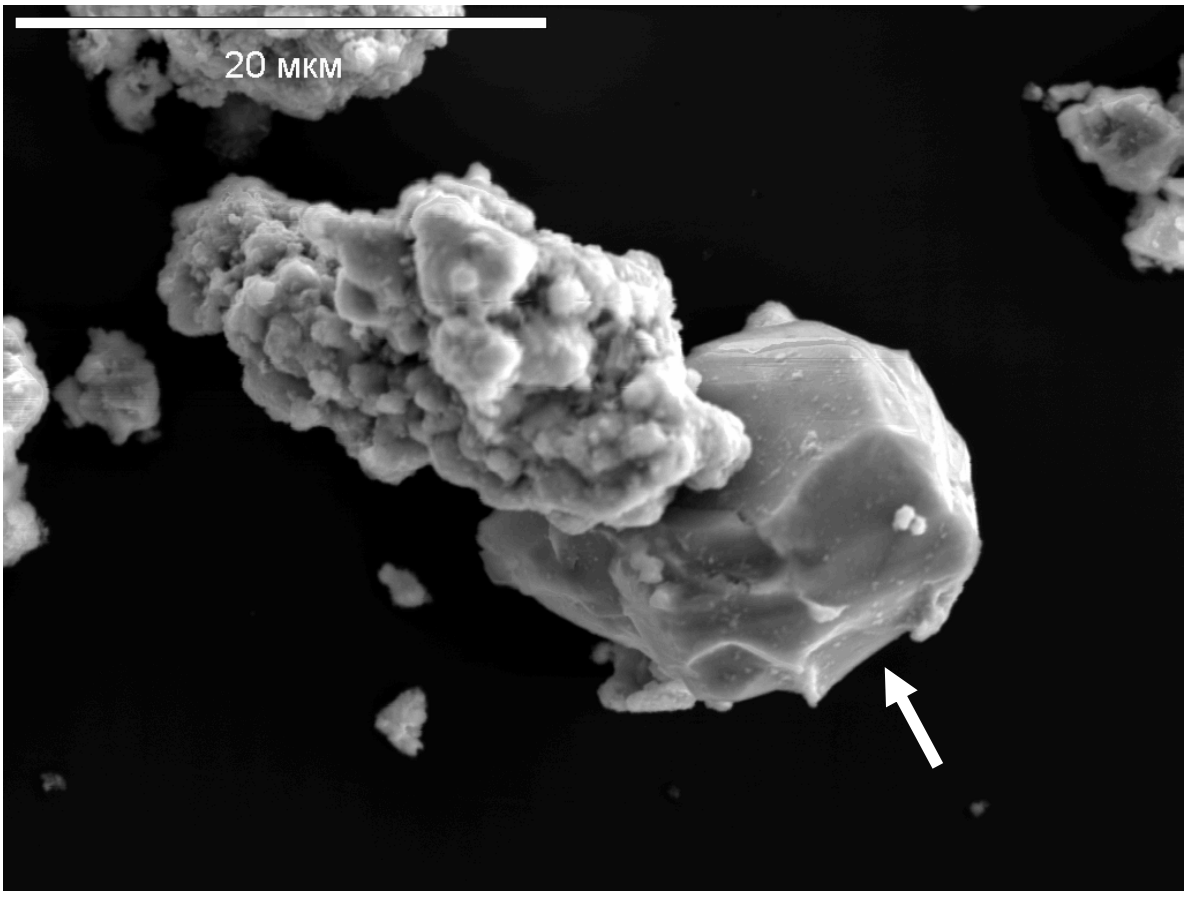
№ образца	Наименование продукта	Co	Co	Se	Se	Te	Te	Fe	Fe	S	S	Pb	Pb	Sn	Sn	As	As
A1	converter matte	3380	ppm	551	ppm	141	ppm	7500	ppm	14.1	%	882	ppm	1.81	ppm	227	ppm
A3	granulated matte	3660	ppm	70	ppm	<10	ppm	41.3	%	21	%	578	ppm	25	ppm	62	ppm
A5	WCM	4865	ppm	377	ppm	134	ppm	2.37	%	10.8	%	700	ppm	4	ppm	142	ppm
A11	concentrate	210	ppm	<1	ppm	<10	ppm	7.63	%	7084	ppm	75	ppm	4.2	ppm	4.5	ppm
A13	final concentrate	891	ppm	3.88	%	1.63	%	3.71	%	5.46	%	3656	ppm	293	ppm	6776	ppm
A15	Pt sponge WPR 2	1.2	ppm	<1	ppm	<10	ppm	90	ppm	<10	ppm	13	ppm	8	ppm	<1	ppm
A19	matte after granulation	1634	ppm	517	ppm	110	ppm	3052	ppm	11.28	%	827	ppm	4.3	ppm	302	ppm
A21	(Pressure leach)	736	ppm	1.31	%	4960	ppm	1.4	%	11.6	%	1562	ppm	191	ppm	1.05	%
A		16	ppm	3930	ppm	1.88	%	2.13	%	2020	ppm	2540	ppm	487	ppm	6810	ppm
B		151	ppm	3840	ppm	1.75	%	1.85	%	8370	ppm	2620	ppm	642	ppm	6720	ppm
C		87	ppm	3910	ppm	1.72	%	1.98	%	5490	ppm	2610	ppm	688	ppm	6920	ppm

table 2 cont.

sample #	Product	Ba	Ba	Cr	Cr	Mn	Mn	Ca	Ca	Ti	Ti	W	W	Mo	Mo	Re	Re
A1	converter matte	<1	ppm	1.6	ppm	1.34	ppm	15.3	ppm	1.5	ppm	<1	ppm	<1	ppm	<10	ppm
A3	granulated matte	8	ppm	1350	ppm	738	ppm	432	ppm	9.6	ppm	<1	ppm	7.12	ppm	85	ppm
A5	WCM	<1	ppm	23	ppm	3.7	ppm	28	ppm	7.2	ppm	<1	ppm	<1	ppm	<10	ppm
A11	concentrate	10	ppm	2528	ppm	940	ppm	1.37	%	843	ppm	<1	ppm	9	ppm	<10	ppm
A13	final concentrate	15.5	ppm	2413	ppm	63	ppm	204	ppm	277	ppm	79	ppm	2043	ppm	5	ppm
A15	Pt sponge WPR 2	<1	ppm	1.7	ppm	<1	ppm	<10	ppm	<1	ppm	<1	ppm	<1	ppm	<10	ppm
A19	matte after granulation	<1	ppm	2.4	ppm	4.72	ppm	52	ppm	2.3	ppm	<1	ppm	<1	ppm	<10	ppm
A21	(Pressure leach)	14.3	ppm	2085	ppm	90	ppm	713	ppm	309	ppm	4.9	ppm	130	ppm	2.2	ppm
A		140	ppm	605	ppm	134	ppm	800	ppm	274	ppm	29	ppm	156	ppm	34	ppm
B		133	ppm	476	ppm	82	ppm	96	ppm	249	ppm	21	ppm	161	ppm	37	ppm
C		137	ppm	542	ppm	110	ppm	467	ppm	274	ppm	22	ppm	149	ppm	38	ppm

Table 2 cont.

sample #	Product	Na	Na	K	K	Mg	Mg	Sc	Sc	Y	Y	Sr	Sr	Zr	Zr	Zn	Zn
A1	converter matte	9	ppm	<10	ppm	4	ppm	<1	ppm	<1	ppm	<1	ppm	<1	ppm	<1	ppm
A3	granulated matte	170	ppm	93	ppm	<1	ppm	<1	ppm	<1	ppm	<1	ppm	3	ppm	276	ppm
A5	WCM	14.4	ppm	3	ppm	14	ppm	4.3	ppm	1.14	ppm	<1	ppm	<1	ppm	1	ppm
A11	concentrate	1140	ppm	687	ppm	7.83	%	13.6	ppm	2.6	ppm	20	ppm	8	ppm	72	ppm
A13	final concentrate	144	ppm	424	ppm	344	ppm	<1	ppm	<1	ppm	2	ppm	10	ppm	<1	ppm
A15	Pt sponge WPR 2	1070	ppm	620	ppm	10	ppm	<1	ppm	<1	ppm	<1	ppm	<1	ppm	<1	ppm
A19	matte after granulation	9	ppm	<10	ppm	41	ppm	4.6	ppm	<1	ppm	<1	ppm	<1	ppm	<1	ppm
A21	(Pressure leach)	378	ppm	107	ppm	3053	ppm	1.6	ppm	1	ppm	4.1	ppm	8.4	ppm	<1	ppm
A		1.59	%	44	ppm	6160	ppm	13	ppm	<1	ppm	18	ppm	17	ppm	25	ppm
B		1.6	%	11	ppm	46	ppm	12	ppm	<1	ppm	19	ppm	19	ppm	14	ppm
C		1.6	%	26	ppm	3130	ppm	11	ppm	<1	ppm	19	ppm	18	ppm	18	ppm



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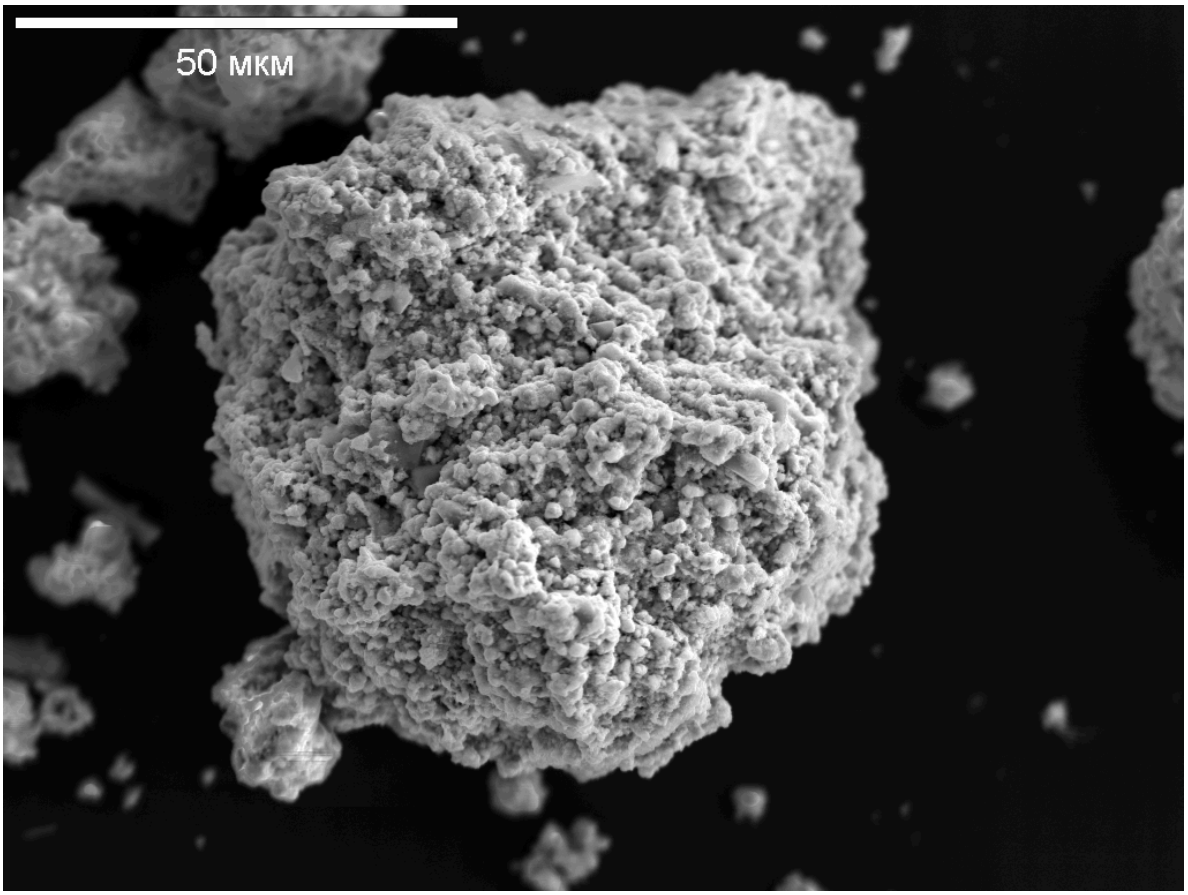

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Fig. 4. Typical shapes of microparticles in the base material of control samples:
a – particles of Cu-O type (arrow) – composition see on Fig. 5a;
b – particles of Cu-O type (precious metals) – composition see on Fig. 5

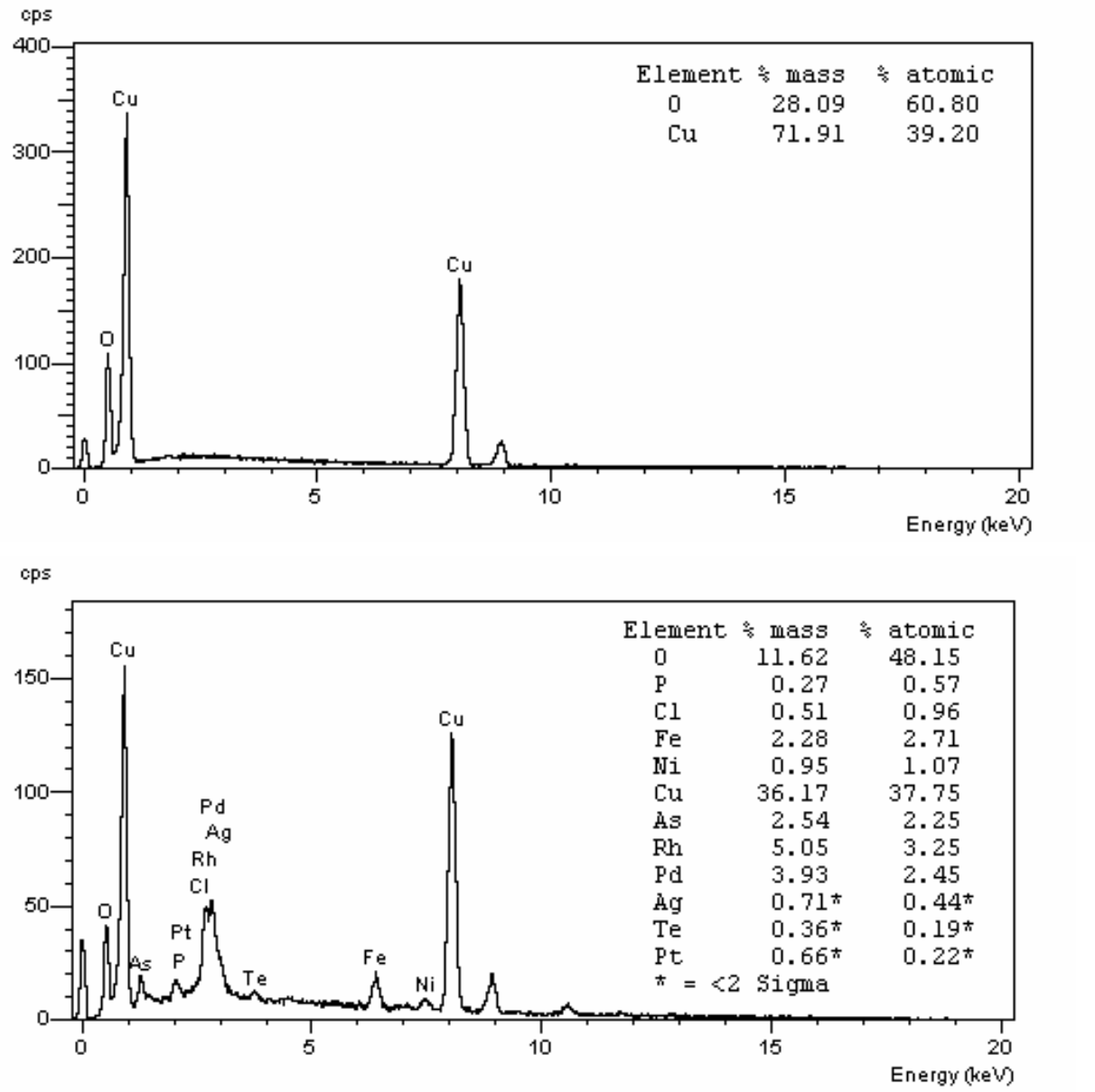

Fig. 5. Typical composition of microparticles in the base material of control samples.