

**Appendix 11. Report by Becker et al on the BKA preliminary testing of the CIP entitled: “analysis round robin.”**



# **Report on the analysis of PGM-bearing samples N1, N2, and N3**

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## 1.) Introduction and comments

Three samples were forwarded by the NFI and were received on August 10<sup>th</sup> 2006. The samples were packed in transparent Whril-Pak bags. They consisted of black powder that was finely grained.

The weights of the samples as received were the following:

Sample name	Weight [g] ± 0,1
N 1	12,6
N 2	12,8
N 3	12,7

It was stated by Dr. Alexander Usov in an email dated August 2<sup>nd</sup> 2006 that:  
Packet # 1 is copper slime; Packet # 2 is nickel slime;  
Packet # 3 is a mix of copper slime and nickel slime.

Concerning sample # 3 it was requested to accomplish the following identification task:  
*Which concrete substances (#1 or #2 or (#1 & #2) are part of the mix #3?*

**Aim of this exercise was to gain first experiences with the analysis of PGM-bearing materials in order to foster the discussion on the analytical issues of the validation process.**

This report should be regarded as a documentation of the first attempts of analysing PGM materials. Therefore, it has draftlike character and should not be regarded as a final statement.

The following analytical methods were applied to the samples:

- 1.) X-ray analysis (XRD & XRF)**  
Carried out by KT 11 Central Laboratory I
- 2.) Inductively coupled plasma – mass spectrometry (ICP-MS)**  
Carried out by KT 13 Inorganic Material Analysis
- 3.) Scanning electron microscopy (SEM)**  
Carried out by KT 11 Central Laboratory I

## 2.) X-ray analysis

For characterisation of the above mentioned sample materials the following methods were applied:

Energy dispersive X-ray fluorescence /EDXRF (Spectro XEPOS)

Powder X-ray diffraction/XRD (Bruker AXS D8-Advance)

### 2.1 EDXRF analysis

#### Sample preparation for EDXRF analysis

The sample material was placed in a XEPOS sample carrier, which was sealed with two layers of Ultralen<sup>®</sup> foil.

#### EDXRF (XEPOS) measurement parameters

EDXRF (XEPOS)		
Radiation	Pd	
Atmosphere	vacuum	
Targets	HOPG	Mo
Voltage	20 kV	45.1 kV
Current	0.5 mA	0.5 mA
Measurement time	300 s	300 s

#### Results of XRF analysis

The results of the XRF analysis of N1, N2, and N3 are listed below:

Sample	Identified elements Main elements printed in <b>bold</b> letters, trace elements printed in <i>italic</i> letters and listed in brackets
N1	<b>S, Cl, Ni, Cu, Se, Ag</b> , Si, Pd, As, Pb, ( <i>Ca, Ti, Fe, Co, Pt, Au, Nb, In, Sn, Sb, Te, Cs</i> )
N2	<b>S, Cl, Ni, Cu, As</b> , Si, Se, Pb, Pd, Ag, ( <i>P, Ca, Ti, Cr, Mn, Fe, Co, Pt, Au, Nb,, Sn, Sb, Te, Cs</i> )
N3	<b>S, Cl, Ni, Cu, As, Se, Ag</b> , Si, Se, Pb, Pd, Fe, ( <i>P, Ca, Ti, Cr, Mn, Co, Pt, Au, Nb, Sn, Sb, Te, Cs</i> )

## 2.2 XRD analysis

### Sample preparation for powder XRD analysis

After grinding of the sample material, approximately 20 mg were placed between two layers of Kapton® foils and fixed in a brass XRD-sample carrier.

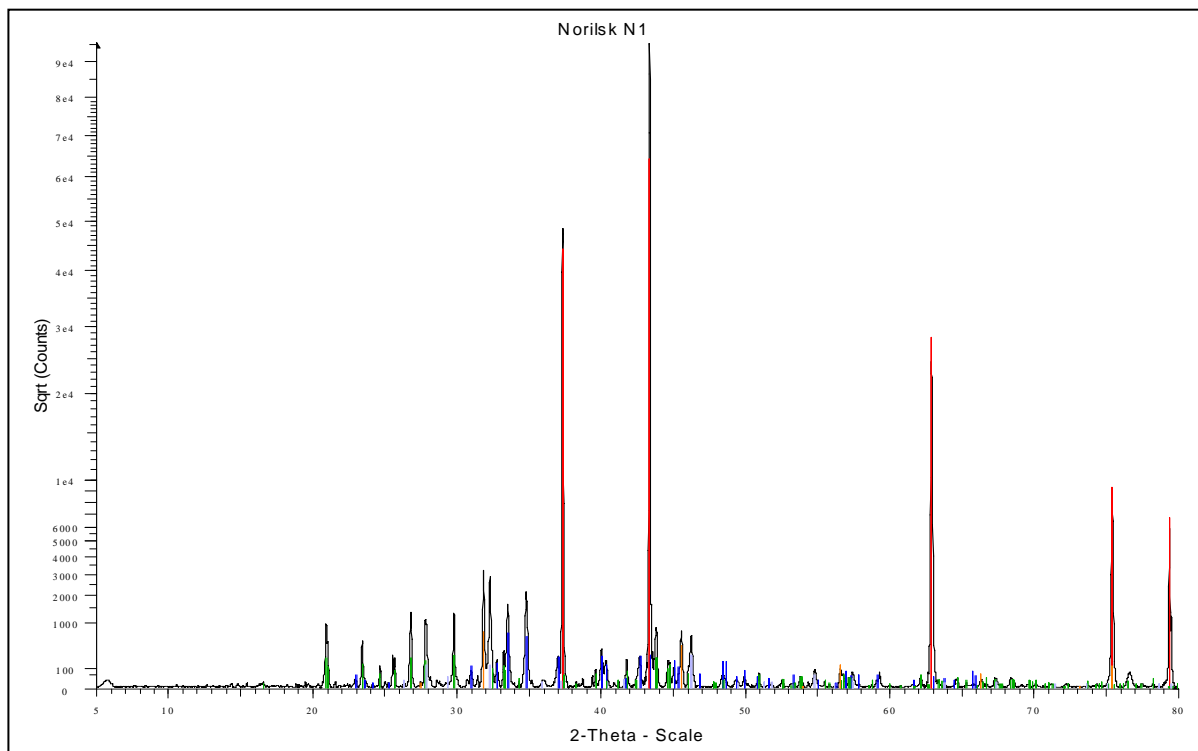
### Instrumental parameters for XRD-measurement

Radiation	Cu K $\alpha$ 1
Voltage	40 kV
Current	40 mA
2 $\theta$ -range	5-80 °
Collecting time:	15 min.

A second set of measurements was carried out with a collecting time of 4 h.

### Results of XRD analysis

Diffract grams of samples N1, N2 and N3:



**Figure 1: Diffractogram of sample N1**

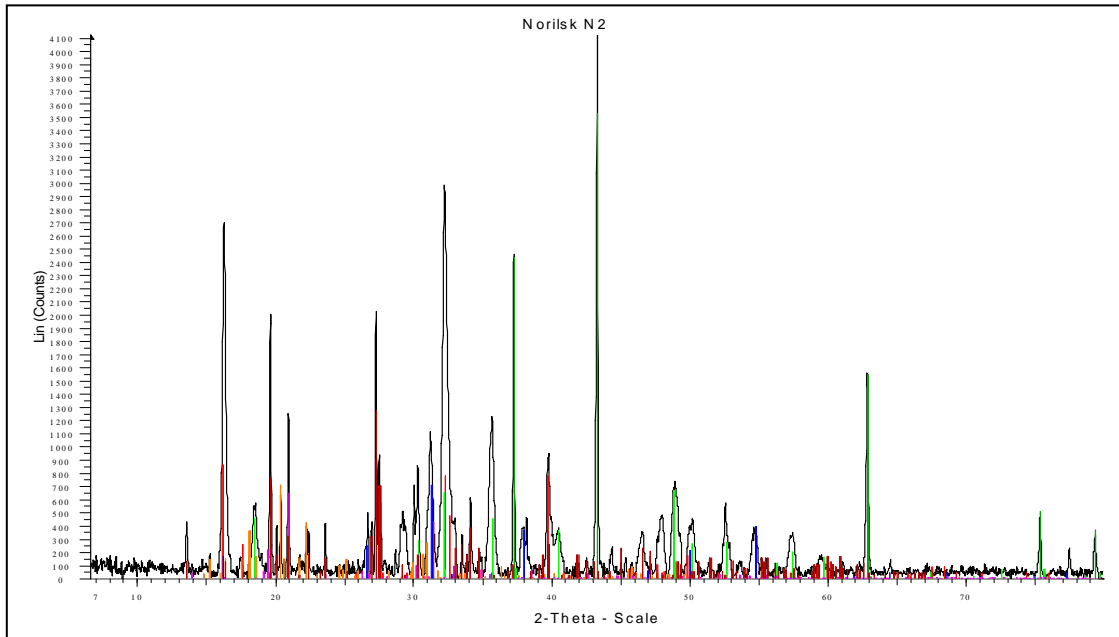


Figure 2: Diffractogram of sample N2

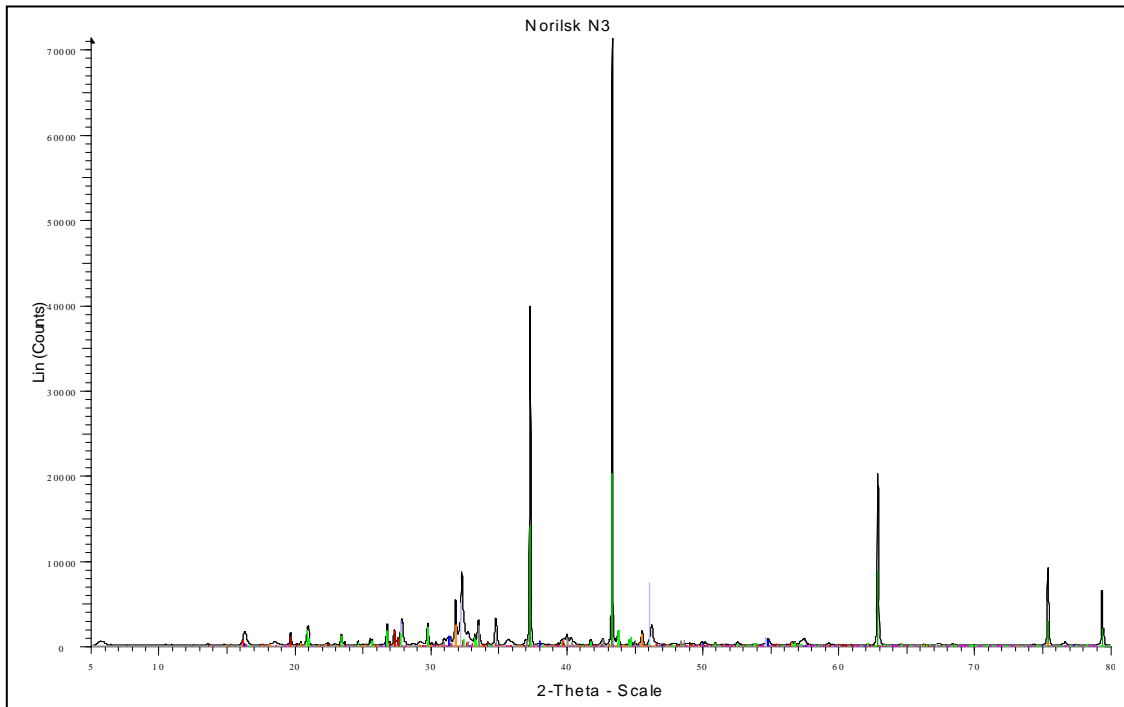


Figure 3: Diffractogram of sample N3





For the interpretation of the diffraction data, results from the XRF analysis were taken into account in order to limit the selection of possible mineral phases.

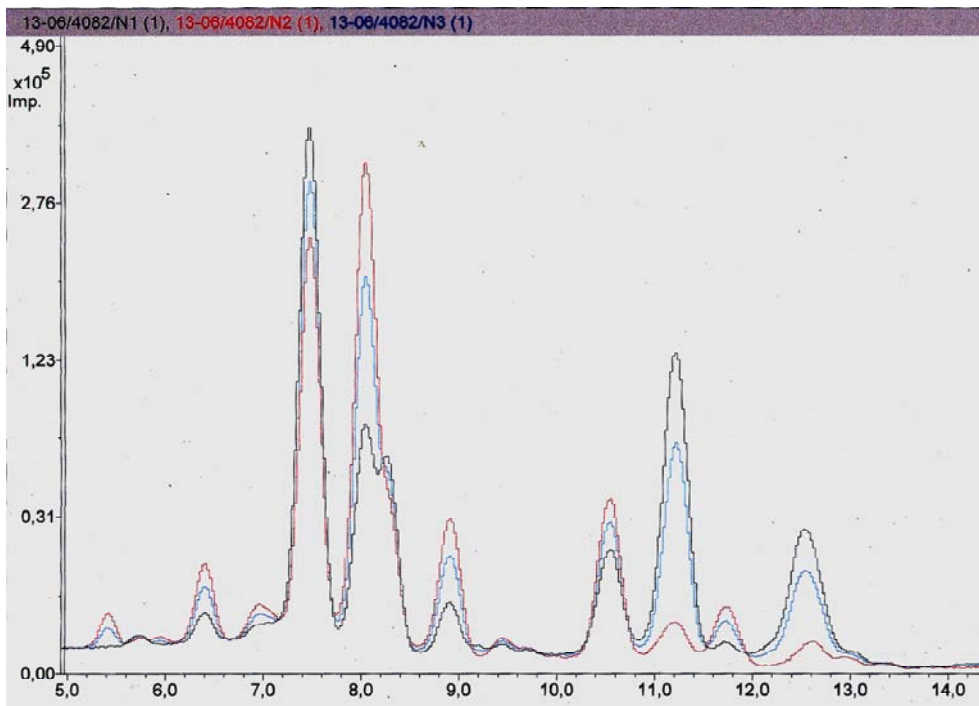
The XRD analysis identified the following mineral phases to be present as main components in the samples (descending by level of concentration):

<b>Sample</b>	<b>Mineral phases</b>
Norilsk N1	$Ni_{1-x}Cu_xO$ – nickel copper oxide –major component $PbSO_4$ – lead sulphate (Anglesit) $Ag_2Se$ – silver selenide (Naumannit)  <u>Additional phases identified by 4 h collecting time:</u> none
Norilsk N2	$Ni_{1-x}Cu_xO$ – nickel copper oxide –major component $(CuCl_2)_2 \cdot (Cu(OH)_2)_5 \cdot H_2O$ – (Atacamite) $NiS$ – nickel sulfide (Digenit)  <u>Additional phases identified by 4 h collecting time:</u> $NiSO_4 \cdot 6 H_2O$ – Nickelsulphate-hexahydrate $Na_2Ni(SO_4)_2 \cdot 4 H_2O$ – Nickelblodite
Norilsk N3	$Ni_{1-x}Cu_xO$ – nickel copper oxide–major component $PbSO_4$ – lead sulphate (Anglesit) $Ag_2Se$ – silver selenide (Naumannit)  <u>Additional phases identified by 4 h collecting time:</u> $NiSO_4 \cdot 6 H_2O$ – Nickelsulphate-hexahydrate $Na_2Ni(SO_4)_2 \cdot 4 H_2O$ – Nickelblodite $Na_{1-x}Ag_xCl$ – silver-doped sodiumchloride

Discussion of XRD and XRF results

Given the fact that sample N3 contains components identified in sample N1 as well as in sample N2, sample N3 seems to be a mixture of N1 and N2.

This finding is supported by EDXRF elemental analysis, indicating a ratio of N1:N2 = 1:1 (approximation). An example of an overlay of part of the spectra of all three materials is given below:



**Figure 4: Part of the EDXRF spectra of measurements of N1 (black line), N2 (red line), and N3 (blue line)**

### 3.) ICP-MS

#### 3.1 Sample preparation / digestion

Three samples of each material were prepared for liquid digestion based on the method described in *Complex Analytical Procedure, Moscow 2003*, page 5-12.

Due to the lack of larger Teflon beakers only 0,1 g ( $\pm 0,02$  g) - instead of the stated 1 g for each sample as described - were used for the liquid digestion procedure. Therefore, all sample steps were adapted / corrected by the factor of ten. An open digestion using 5 mL Teflon cups was employed. Heating was carried out by using an aluminium block.

Elemental concentrations were determined using an Agilent 7500cs ICP-MS applying the following instrumental parameters. Due to the fact that no mass-to-charge ratios for the ICP-MS analysis are stated in *Complex Analytical Procedure, Moscow 2003* they were selected according to our experience.

ICP-MS parameter (Agilent 7500cs)	
RF power	1390 W at 27,12 MHz
Cool, aux. and nebuliser gas flow	14 / 0,9 / 1 L argon per minute
Torch position, ion optics	Optimised for sensitivity and stability
Data acquisition	peak jump, integrated
channels per mass, measurement time per mass	3 channels, 100 ms dwell time
Measured isotopes/masses	<sup>49</sup> Ti, <sup>60</sup> Ni, <sup>63</sup> Cu, <sup>82</sup> Se, <sup>105</sup> Pd, <sup>107</sup> Ag, <sup>118</sup> Sn, <sup>121</sup> Sb, <sup>125</sup> Te, <sup>137</sup> Ba, <sup>182</sup> W, <sup>195</sup> Pt, <sup>197</sup> Au, <sup>208</sup> Pb
Analysis per sample (parallel measurements)	3
Measurement time per sample	24 s

The measurement time per sample differs from the stated protocol as they are instrumental / vendor specific. Due to a lack of standard solutions the following elemental concentrations couldn't be determined: **Rh, Ru, and Ir.**

In a first set of analysis the following observations were made:

- 1.) In many cases (for many elements) the concentrations of the digestion solutions were way above the concentration range of the calibration solution, in some cases up to 3 dimensions.
- 2.) This resulted in several cases in A/D conversion problems (concentration range of the calibration curve measured in pulse count/PC, whereas the digestion solution measured in analogue mode).
- 3.) This leads to incorrect quantitative results.

Therefore, a second measurement with an additional dilution step of a factor of 20 was introduced. The above mentioned problems (1. to 3.) were then not observed for most elements.

**3.2 Results of ICP-MS measurements (all concentrations in µg/g)**

Sample N1	N1 1	N1 2	N1 3	Mean	SD	RSD%
Titanium / 49	464	638	596	<b>566</b>	91	16
Nickel / 60	344200	342600	360700	<b>349167</b>	10020	3
Copper / 63	59950	59840	60630	<b>60140</b>	428	0,7
Selenium / 82	84590	83260	86460	<b>84770</b>	1608	2
Molybdenum* / 95	27	26	28	<b>27</b>	1	3
Palladium / 105	7567	7461	7507	<b>7512</b>	53	0,7
Silver / 107	68190	72130	74960	<b>71760</b>	3400	5
Tin / 118	3257	3295	3156	<b>3236</b>	72	2
Antimony / 121	886	879	894	<b>886</b>	8	0,9
Tellurium / 125	6018	5912	6049	<b>5993</b>	72	1,2
Barium* / 137	29	33	29	<b>30</b>	2	7
Tungsten / 182	70,73	63,14	60,15	<b>64,67</b>	5,45	8
Platinum / 195	1111	1114	1132	<b>1119</b>	11	1,0
Gold / 197	647	640	628	<b>638</b>	10	1,5
Lead / 208	23410	23000	22910	<b>23107</b>	267	1,2

Sample N2	N2 1	N2 2	N2 3	Mean	SD	RSD%
Titanium / 49	1216	1237	1297	<b>1250</b>	42	3
Nickel / 60	188800	192800	194700	<b>192100</b>	3012	2
Copper / 63	212100	220600	223300	<b>218667</b>	5845	3
Selenium / 82	1852	1508	1370	<b>1577</b>	248	16
Molybdenum / 95	1205	1256	1261	<b>1241</b>	31	2
Palladium / 105	14940	15420	15590	<b>15317</b>	337	2
Silver / 107	2752	2782	2880	<b>2805</b>	67	2
Tin / 118	7007	7278	7240	<b>7175</b>	147	2
Antimony / 121	1168	1215	1208	<b>1197</b>	25	2
Tellurium / 125	1676	1698	1707	<b>1694</b>	16	0,9
Barium* / 137	20	22	23	<b>22</b>	2	8
Tungsten / 182	1288	1312	1338	<b>1313</b>	25	2
Platinum / 195	2450	2482	2450	<b>2461</b>	18	0,8
Gold / 197	627	594	567	<b>596</b>	30	5
Lead / 208	1552	1552	1548	<b>1551</b>	2	0,1

Sample N3	N3 3	N3 4	N3 5	Mean	SD	RSD%
Titanium / 49	939	1027	1046	<b>1004</b>	57	6
Nickel / 60	284100	281900	279000	<b>281667</b>	2558	0,9
Copper / 63	131700	137000	134200	<b>134300</b>	2651	2
Selenium / 82	43410	44830	43300	<b>43847</b>	853	2
Molybdenum / 95	571	595	581	<b>582</b>	12	2
Palladium / 105	10780	11140	10910	<b>10943</b>	182	2
Silver / 107	42910	46380	47450	<b>45580</b>	2373	5
Tin / 118	5091	5275	5068	<b>5145</b>	113	2
Antimony / 121	978	1023	995	<b>999</b>	23	2
Tellurium / 125	3658	3831	3701	<b>3730</b>	90	2
Barium* / 137	30	29	28	<b>29</b>	1	4
Tungsten / 182	666	680	663	<b>669</b>	9	1,4
Platinum / 195	1645	1761	1710	<b>1705</b>	58	3
Gold / 197	460	539	552	<b>517</b>	50	10
Lead / 208	11770	12310	12260	<b>12113</b>	298	2

Concentrations of elements that are indicated by an asterisk (\*) such as Barium for N1, N2, and N3 are reported from the first set of measurements. Due to the additional dilution step concentrations of these samples were reported below detection limit in the second set of measurements.

Deviation to reference values may be explained by the following facts:

- Homogeneity issues due to the smaller sample amount (factor 10) cannot be ruled out.
- In a few cases the concentrations of the digestion solutions were way above the concentration range of the calibration solution, in some cases up to two dimensions.
- In a few cases in A/D conversion problems occurred (concentration range of the calibration curve measured in pulse count /PC, whereas the digestion solution measured in analogue mode).
- Sample preparation issues (open digestion vs. closed high pressure digestion).

Based on the above stated reasons an error of 10 % should be assigned to all results of ICP-MS analysis (except the RSD stated in the table exceeds this value).

Based on the elemental concentrations of N1, N2, and N3 an equal mix of N1 and N2 can be assumed for sample N3 ( $\pm 10\%$ ).

## 4.) Scanning electron microscopy

### 4.1 Bulk analysis by SEM

#### Sample preparation

One sample of **N1, N2, and N3** was prepared and measured based on the method described in *Complex Analytical Procedure, Moscow 2003*, page 14-17. Two deviations from the protocol were carried out: Due to technical reasons a different magnification was used for the integral / bulk measurements (50x instead of 20x magnification).

Also the measurement time was different from the stated 50 sec in the above mentioned procedure. In order to evaluate the impact on the variation of the measurement time, analyses were carried out with a measurement time of 200 sec and 500 sec.

#### Instrumental parameters for SEM measurements

SEM: Nova NanoLab with field emission gun (FEG);

Energy dispersive X-Ray micro analysis (EDX) Oxford INCA

As stated in the given procedure five areas have been measured for bulk elemental composition.

#### Results (all concentrations stated in wt %)

For sample N1 the following bulk concentrations were achieved by SEM:

Measurement time 200 sec											
	O	Na	Si	S	Cl	Ca	Ni	Cu	Se	Ag	Te
mean	17,22	3,17	1,35	5,91	4,86	0,41	15,44	14,34	18,22	17,90	1,17
SD	0,69	0,37	0,18	0,19	0,24	0,06	1,12	0,33	0,37	0,42	0,16
Min	16,26	2,71	1,09	5,78	4,54	0,33	13,98	13,83	17,89	17,28	0,95
Max	18,14	3,63	1,57	6,20	5,13	0,46	16,91	14,64	18,77	18,31	1,39

**Se<sub>18-19</sub>-Ag<sub>17-18</sub>-O<sub>16-18</sub>-Ni<sub>14-17</sub>-Cu<sub>14-15</sub>-S<sub>6-6</sub>-Cl<sub>4.5-5.1</sub>-Na<sub>2.7-3.6</sub>-Si<sub>1.1-1.6</sub>-Te<sub>1.0-1.4</sub>-Ca<sub>0.3-0.5</sub>**

Measurement time 500 sec													
	O	Na	Si	S	Cl	Ca	Ni	Cu	Se	Ag	Te	As	Pt
mean	17,86	3,48	1,46	5,58	4,98	0,41	15,21	12,79	18,30	17,32	1,13	1,09	0,41
SD	0,41	0,33	0,02	0,09	0,07	0,06	1,70	0,06	0,20	1,00	0,03	0,05	0,58
Min	17,57	3,24	1,44	5,51	4,94	0,37	14,01	12,75	18,16	16,61	1,11	1,06	0,00
Max	18,15	3,71	1,47	5,64	5,03	0,45	16,41	12,83	18,44	18,03	1,15	1,13	0,82

**Se<sub>18-19</sub>-Ag<sub>17-18</sub>-O<sub>16-18</sub>-Ni<sub>14-17</sub>-Cu<sub>14-15</sub>-S<sub>6-6</sub>-Cl<sub>4.5-5.1</sub>-Na<sub>2.7-3.6</sub>-Si<sub>1.1-1.6</sub>-Te<sub>1.0-1.4</sub>-As<sub>1.1-1.1</sub>-Pt<sub>0.0-0.8</sub>-Ca<sub>0.3-0.5</sub>**



For sample N2 the following bulk concentrations (wt %) were measured by SEM:

Measurement time 200 sec														
	O	Na	Si	P	S	Cl	Cr	Fe	Ni	Cu	As	Pd	Sn	Pt
mean	26,39	1,72	0,91	0,52	13,34	5,24	0,73	1,91	14,56	26,82	3,36	2,33	0,92	1,26
SD	0,71	0,21	0,16	0,19	0,25	0,16	0,14	0,18	0,41	0,63	0,25	0,48	0,22	0,30
Min	25,26	1,45	0,72	0,36	13,01	5,08	0,55	1,65	14,13	25,81	2,98	1,63	0,69	1,02
Max	27,12	1,94	1,08	0,77	13,67	5,49	0,87	2,16	15,24	27,37	3,59	2,70	1,14	1,76

**Cu<sub>26-27</sub>-O<sub>25-27</sub>-Ni<sub>14-15</sub>-S<sub>13-14</sub>-Cl<sub>5-5</sub>-As<sub>3.0-3.6</sub>-Pd<sub>1.6-2.7</sub>-Fe<sub>1.6-2.2</sub>-Na<sub>1.4-1.9</sub>-Pt<sub>1.0-1.8</sub>-Sn<sub>0.7-1.1</sub>-Si<sub>0.7-1.1</sub>-Cr<sub>0.6-0.9</sub>-P<sub>0.4-0.8</sub>**

Measurement time 500 sec														
	O	Na	Si	P	S	Cl	Cr	Fe	Ni	Cu	As	Pd	Sn	Pt
mean	25,79	1,77	0,97	0,41	13,27	5,24	0,86	1,98	15,07	26,75	3,22	2,41	1,04	1,23
SD	0,53	0,18	0,03	0,05	0,11	0,13	0,01	0,02	0,07	0,28	0,19	0,04	0,01	0,16
Min	25,41	1,65	0,95	0,38	13,20	5,15	0,85	1,96	15,02	26,56	3,08	2,38	1,03	1,11
Max	26,16	1,90	0,99	0,44	13,34	5,33	0,87	2,00	15,12	26,95	3,35	2,44	1,05	1,35

**Cu<sub>27-27</sub>-O<sub>25-26</sub>-Ni<sub>15-15</sub>-S<sub>13-13</sub>-Cl<sub>5-5</sub>-As<sub>3.1-3.3</sub>-Pd<sub>2.4-2.4</sub>-Fe<sub>2.0-2.0</sub>-Na<sub>1.6-1.9</sub>-Pt<sub>1.1-1.3</sub>-Sn<sub>1.0-1.0</sub>-Si<sub>0.9-1.0</sub>-Cr<sub>0.8-0.9</sub>-P<sub>0.4-0.4</sub>**

For sample N3 the following bulk concentrations (wt %) were measured by SEM:

Measurement time 200 sec													
	O	Na	Si	S	Cl	Cr	Fe	Ni	Cu	As	Se	Pd	Ag
mean	21,86	2,34	0,83	9,89	4,23	0,51	1,07	22,11	19,48	2,34	7,22	1,66	6,46
SD	0,53	0,33	0,07	0,50	0,16	0,10	0,13	0,57	0,65	0,28	0,45	0,16	0,37
Min	21,23	1,93	0,77	9,27	4,07	0,39	0,96	21,22	18,75	1,84	6,66	1,46	6,10
Max	22,34	2,71	0,93	10,53	4,44	0,60	1,26	22,70	20,32	2,52	7,71	1,88	7,01

**Ni<sub>21-23</sub>-O<sub>21-22</sub>-Cu<sub>19-20</sub>-S<sub>9-11</sub>-Se<sub>7-8</sub>-Ag<sub>6-7</sub>-Cl<sub>4.1-4.4</sub>-Na<sub>1.9-2.7</sub>-As<sub>1.8-2.5</sub>-Pd<sub>1.5-1.9</sub>-Fe<sub>1.0-1.3</sub>-Si<sub>0.8-0.9</sub>-Cr<sub>0.4-0.6</sub>**

Measurement time 500 sec															
	O	Na	Si	S	Cl	Cr	Fe	Ni	Cu	As	Se	Pd	Ag	Pt	P
mean	22,61	2,24	0,85	9,77	4,13	0,44	0,95	20,91	18,99	2,24	7,09	2,06	6,59	0,94	0,21
SD	0,38	0,29	0,02	0,03	0,16	0,03	0,11	0,74	0,16	0,08	0,27	0,19	0,33	0,12	0,00
Min	22,34	2,03	0,83	9,75	4,02	0,41	0,87	20,38	18,87	2,18	6,89	1,92	6,35	0,85	0,21
Max	22,88	2,44	0,86	9,79	4,24	0,46	1,03	21,43	19,10	2,30	7,28	2,20	6,82	1,02	0,21

**O<sub>22-23</sub>-Ni<sub>20-21</sub>-Cu<sub>19-19</sub>-S<sub>10-10</sub>-Se<sub>7-7</sub>-Ag<sub>6-7</sub>-Cl<sub>4.0-4.2</sub>-As<sub>2.2-2.3</sub>-Na<sub>2.0-2.4</sub>-Pd<sub>1.5-1.9</sub>-Fe<sub>0.9-1.0</sub>-Pt<sub>0.9-1.0</sub>-Si<sub>0.8-0.9</sub>-Cr<sub>0.4-0.5</sub>-P<sub>0.2-0.2</sub>**

## Comments

It should be stated that these concentrations differ from the content (as determined by ICP-MS) due to:

- 1.) the lack of proper quantification using matrix matched external standards
- 2.) no representative results for the bulk material, due to the fact that SEM/EDX only measures surface near areas; as stated on page 3 of the material data sheet H19 large particles are often layered with fine particles, which then leads to wrong results.

As stated in *Complex Analytical Procedure, Moscow 2003*, page 4 the results of this measurement are: “used in the preliminary identification of a substance, and in determination the methods of sample preparation for further tests”.

Even though XRF can be considered as a better alternative in this case, the use of SEM fits the purpose of fast screening.



## 4.2 Particle analysis

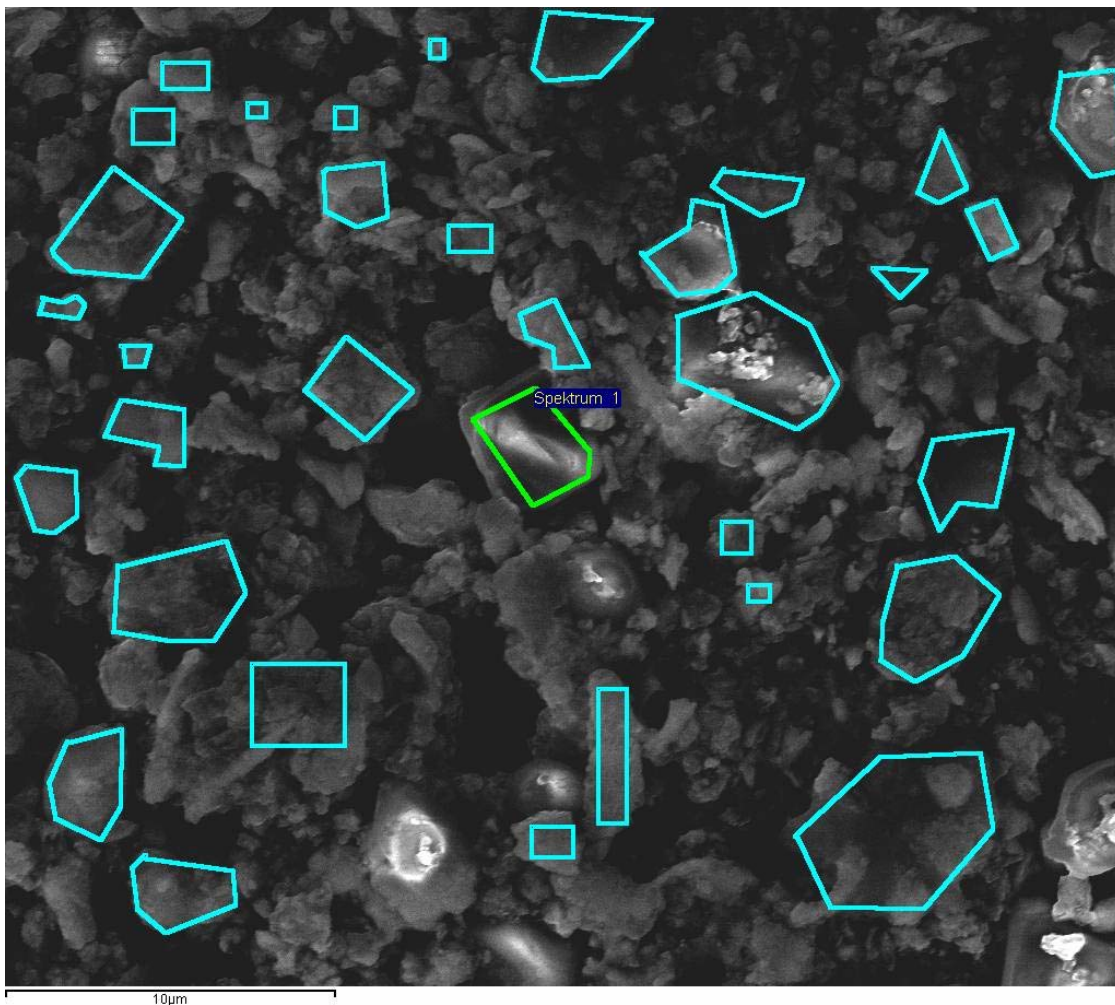
### Sample preparation

One sample of **N1**, **N2**, and **N3** were prepared and measured based on the method described in *Complex Analytical Procedure, Moscow 2003*, page 14-17. In order to achieve a monolayer it was necessary to carry out a third dilution step.

### Instrumental parameters for SEM measurements

SEM: Nova NanoLab with field emission gun (FEG);

Energy dispersive X-Ray micro analysis (EDX) Oxford INCA



**Figure 5: Example of the selection of areas for particles analysis by SEM of sample N1**

Approximately 100 particles were analysed for each material.

## Results

The following type classes were found for N1:

<b>N1</b>	<b>findings</b>	
<b>Type class</b>	<b>n =</b>	<b>%</b>
Se-Ag	85	64,4
Cu-Ni-Se-Ag	7	5,3
Ni-O	19	14,4
Pb	11	8,3
Sn	2	1,5
Ca	2	1,5
unclassified	6	4,5
<b>total</b>	<b>132</b>	<b>100</b>
<b>N2</b>	<b>findings</b>	
<b>Type class</b>	<b>n =</b>	<b>%</b>
Se-Ag	1	1,0
Cu-Ni	41	41,0
Cu-Ni-As	22	22,0
Cu-Ni-As-Pd	18	18,0
Cu-Ni-Pd	1	1,0
Cu	10	10,0
Ni-O	4	4,0
Ni-S	2	2,0
unclassified	1	1,0
<b>total</b>	<b>100</b>	<b>100</b>
<b>N3</b>	<b>findings</b>	
<b>Type class</b>	<b>n =</b>	<b>%</b>
Se-Ag	29	30,2
Cu-Ni	22	22,9
Cu-Ni-As-Pd	12	12,50
Ni-O	8	8,3
Cu-Ni-As	6	6,3
Cu-XX	5	5,2
Cu-Se	3	3,1
Ni-S	2	2,1
Pb	1	1,0
unclassified	8	8,3
<b>total</b>	<b>96</b>	<b>100</b>

### Comments

In the course of particle analysis by SEM a better insights were gained on the principle and philosophy of procedure.

However, it remained unclear whether the reproducibility of the results can be achieved by operators with different levels of experience regarding the database.

## **5.) Concluding remarks**

This report is meant to foster the discussion on the analytical validation of the CIM/CIP method. Certainly it will be very helpful to compare the data included in this report with the data supplied by TNO and the Russian data contained in the Reference database (RDB).