Appendix 5. Final report of the expert group visit to the ICFSS

During the period from September 18th to 19th a group consisting of Dr. Gerard van der Peijl (NFI), Dr. Jan Andrasko (SKL), Dr. Ludwig Niewoehner and Dr. Stefan Becker (both BKA) visited the Institute of Criminalistics of the Federal Security Service (IC FSS) of the Russian Federation.
Aim of the visit was a preliminary study on the application of the CIM methods, which were developed and applied within the institute. This visit was aimed at a more profound understanding of the structure and use, rather than an audit type visit concerning quality assurance issues.

The group would like to thank the ICFSS for the time and effort, which all participating members put into our visit. We encountered a high degree of openness and competence. Also the flexibility of the Russian colleagues reacting to our various requests enabled an effective evaluation. Certainly, we have all learned a lot and gained from this visit.

During our two-day visit, the issues of sample preparation, instrumental analysis, the so called related reference database (RDB), and interpretation were covered. Also an expert witness report dated from March 10th 2005 dealing with the analysis of PGM bearing material was supplied in a Russian and English version.

The reviewed methods were:
- Scanning Electron Microscope (SEM-EDS) for bulk material analysis including sample preparation
- Inductively coupled Plasma Optical Emission Spectroscopy (ICP-OES) and Inductively coupled Plasma Mass Spectrometry (ICP-MS) including sample preparation
- X-ray Diffractometry (XRD)
- Microparticle analysis using SEM-EDS
- Concept, structure and application of the related reference database

We have the impression that the equipment for sample preparation and instrumental analysis is fully adequate. In our opinion based on the two day visit, the staff is highly competent.
The concept and stepwise approach of the analysis scheme seems appropriate.
We used a fair amount of time to understand the principle and outline of the database.
The methodology of the classification of a sample based on the semi-quantitative results of large amounts of microparticles measured by SEM-EDS seems efficient.

Based on our investigation and provided that the assumptions are correct
a) that 70 product types can be distinguished and
b) that these materials are different in composition/character from all other global sources,
we are convinced that pure materials derived from the Norilsk and Kola sites can be identified. Also, by the application of the combined analytical methods it may be possible to identify two component mixtures of these materials.

Further steps necessary to validate the CIM

Considering the information gained during this visit and given the fact that TNO will carry out an analytical evaluation of the CIM methods, we believe the following steps are necessary:

Comparison of the variation within a product type (i.e. up to ten repeat measurements from the same locations at different times) with the variation between the 70 existing product types.

This needs to be done for all 70 product types. For this statistical evaluation several data sets have to be examined: quantitative data measured by ICP-OES and ICP-MS, quantitative data measured by SEM, diffractometry information, definition and distribution of all types defined by SEM microparticle analysis.

The statistical evaluation may cover descriptive statistic to provide an improved visualisation of range and means (such as box whisker plot) and should certainly contain chemometric modules such as ANOVA.

Aim of the statistical evaluation is to prove that all 70 product types can be distinguished from each other and that the within product type variation does not interfere with that result.

Suggestion

Even though the focus of this visit was the evaluation of the existing methods, we would still like to take the opportunity to state the following:
We suggest that further development may be focussed on a classification based on the semi-quantitative results of a large amount of microparticles measured by SEM EDS.
The analysis of 100 (1000) particles is very time consuming and the applied method can only be carried out by a highly skilled and experienced operator/scientist.
We suggest the development of an automated detection and classification of the microparticles measured by SEM EDS (with software such as INCA or similar products).
This would save time and make the method more operator/expert independent.
It would also enable other laboratories to repeat this analysis, and in this way enhance the possibility of the international application of this method.

Wiesbaden, September 28th 2006

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