CIP Project

Report of the CIP Forensic Review Board

23 April 2008
The Hague
The Netherlands
The CIP project report can also be accessed at http://www.enfsi.eu/page.php?uid=96

Contents

1. Summary

Precious metal producing companies world-wide are under continuous threat of theft of their products. When alleged stolen material containing platinum group elements (PGE) is recovered, the source of the material has to be determined and scrupulous and effective scientific analysis needs to be carried out to provide reliable information that will enable a court of law to arrive at a fair judgment.

A procedure (Complex Identification Procedure – CIP) has been developed in Russia that incorporates a variety of chemical and mineralogical techniques for such source determination. In order for the CIP results and the expert opinions based on them to be accepted in future court proceedings, it was deemed necessary to have the procedure analytically and forensically validated by the European Network of Forensic Science Institutes (ENFSI). An independent Forensic Review Board comprised of experienced forensic scientists, academics and legal experts was assembled to review the CIP.

This comprehensive report covers the finding of the Forensic Review Board and discusses:

- the composition and the activities of the Forensic Review Board
- background information on PGE production
- a description of the CIP
- the verification process
- the testing of the CIP
- forensic and legal aspects of the use of the method
- recommendations for further improvement.

The board concluded that the CIP is based upon accepted, reliable analytical methods. The methodology is regarded as fit for purpose and well-suited for the identification and source attribution of Russian PGE-bearing materials.

It was agreed, however, that effective interpretation of CIP results within the context of legal casework requires specialist knowledge and an understanding of the mining and beneficiation of PGE-bearing materials. This is crucial for the identification of components in a mixture and in situations where the materials do not fully match those in the database because of undocumented variation. The continued development of analytical protocols for the discrimination of PGE-bearing materials is encouraged, and the CIP could be regarded as a starting point for development of methods by which consistent, comparable data can be obtained across the various producers.

2. Introduction

Partially processed platinum group element (PGE)-bearing material from Russian and South African producers is being stolen on a large scale and illegally supplied to specialised platinum group metal (PGM) refineries worldwide for further processing[1]. Together, these two countries produce over 80% of the world's PGE-bearing intermediate materials. The estimated annual value of such stolen material worldwide is 150 million Euros. A number of international organisations in Europe and North America have noted that the proceeds from the sale of these materials are being used to finance illegal arms sales, drug trafficking and terrorist activities.

A number of leading Russian scientific research institutes have developed a procedure to characterise intermediate beneficiation process PGE materials. This incorporates the use of a variety of techniques, including microscopy, inductively-coupled plasma optical emission spectroscopy (ICP-OES), inductively-coupled plasma mass spectrometry (ICP-MS), X-ray diffraction (XRD) and scanning electron microscopy with energy-dispersive X-ray analysis (SEM).

The full title of this procedure is the "Complex Analytical Procedure for Identification of the Nature and the Source of Origin of Precious Metals Containing Products of Mining and Metallurgical Operations". For the purpose of this project, the title is shortened to Complex Identification Procedure (CIP)[2].

Russian law enforcement agencies have initiated legal actions in Western Europe against companies suspected of dealing in stolen PGE-bearing materials. In order for the CIP results and expert opinions derived from them to be accepted in future court proceedings, it was deemed necessary to have the procedure analytically and forensically validated by an independent international body. A project was initiated to this end under the auspices of European Network of Forensic Science Institutes (ENFSI)[3]. It is supported by the Ministry of Justice of the Russian Federation and the International Platinum Association[4] (IPA).

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[1] See Appendix 1 for acronyms, terms and definitions.

[2] In earlier documentation describing this procedure, it was called the "Complex Methods of Identification" or CIM.

[3] See Appendix 2 for the ENFSI letter of invitation

[4] Now called the International Platinum Group Metals Association.

3. Formation of the Forensic Review Board

An independent international Forensic Review Board (from here on referred to as the ‘Board’) comprised of experienced forensic scientists, academics and legal experts was formed to review the CIP. Most members of the Board had no previous experience with PGE-bearing materials, so six specialists were selected to advise the Board. The board members and the advisers were invited by ENFSI to participate in the project, with the Russian Federal Centre of Forensic Science (RFCFS) as the facilitator of the project.

The CIP validation project as envisaged by ENFSI consisted of two parts:

1. The validation of the analytical methods of the CIP by an independent international organisation.

2. The forensic validation of the CIP by a Forensic Review Board.

The Netherlands Organisation for Applied Scientific Research (TNO) was commissioned by RFCFS, on the advice of the Board, to perform an independent analytical verification and testing of the CIP.

The Board was comprised of nine members from the United Kingdom, Germany[5], Sweden, The Netherlands, The United States, South Africa and The Russian Federation. The chairman of the Board was Prof. Ingvar Kopp, former Director of the Swedish National Laboratory of Forensic Science (SKL). A four person Executive Board was created to facilitate the activities of the Board.

The members of the Board and the Executive Board are listed in Appendix 3 and the advisers in Appendix 4.

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[5] The German member of the Board participated only in the first part of the project.

4. Overview of Board Activities

Meetings of the Board were held on:

11-12 July 2006 (The Hague, The Netherlands),
20-21 September 2006 (Moscow, Russia),
23-24 October 2006 (The Hague, The Netherlands),
20-21 November 2006 (Munich, Germany),
21-22 March 2007 (Bakubung, South Africa),
14-15 June 2007 (Brussels, Belgium),
17-18 October 2007 (Paris, France),
13-14 February 2008 (The Hague, The Netherlands),

Advisers were invited to Board meetings when required; representatives from TNO attended all but the last two meetings. Time was spent during the first few meetings familiarising the Board with the occurrence and processing of PGE ores. Reports presented by the advisers are attached as Appendices. In addition to the Board meetings, visits were made to laboratories and production facilities.

Some Board members visited the Institute of Criminalistics of the Federal Security Service (ICFSS) of the Russian Federation in Moscow on 18-19 September 2006 to carry out a study on the practical application of the CIP. A report on this visit is provided as Appendix 5.

Preceding the meeting in South Africa, the Board visited the Anglo Research laboratory in Germiston, the Anglo Platinum Waterval Smelter and the Precious Metal Refinery. The purpose of the visits was to obtain an understanding of the Anglo Platinum operations and their methods used for characterising their PGE-bearing materials.

Background information is important for evidence evaluation, whatever profiling methodology is used. To explore the extent to which useful information could be obtained from geological data, the Board commissioned a study (see Appendix 6). Based in part on the results of this study, the Board concluded that, because of the mixing of various ore types during processing, the variation in the composition of partially processed PGE-bearing materials is less than that in geological samples. Converter matte material appeared to be a good material for characterisation of producers, so a secondary project to acquire data on converter mattes from various producers was initiated (see Section 5.3). The detailed characterisation of the converter mattes obtained was undertaken by Anglo Research, funded by Anglo Platinum (Appendix 8).

In order to get a better understanding of the practical implications of the procedure, TNO and the forensic laboratory of the Bundeskriminalamt tested the CIP on a simple 50/50 mixture of Norilsk materials (Appendices 10 and 11). Their results are discussed in Chapters 7 and 8.

In order to explore the application of the CIP to mixed samples, the Board decided to conduct a collaborative exercise. Three mixed samples were distributed to laboratories in Russia and South Africa. The information provided to participating laboratories was that the mixed samples contained PGE-bearing materials from Norilsk Nickel (NN) and other producers. The participants were asked to use the CIP to identify the individual components of the mixtures. Results of this collaborative exercise are discussed in Chapter 8.

5. Geological, mining and beneficiation background

5.1 Introduction

At present, most of the worlds supply of platinum group elements (platinum, palladium, rhodium, ruthenium, iridium and osmium) is obtained from mines within four major layered igneous intrusions, namely, the Bushveld Complex in South Africa, the Stillwater Complex in the USA, the Great Dyke in Zimbabwe, and the Norilsk Complex in Russia (Figures 1 and 2).

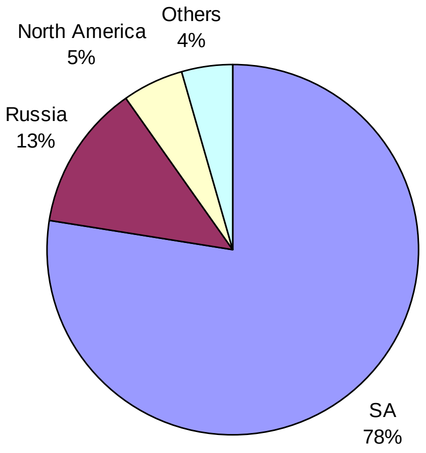

Figure 1: 2006 Platinum supply (7 million oz).

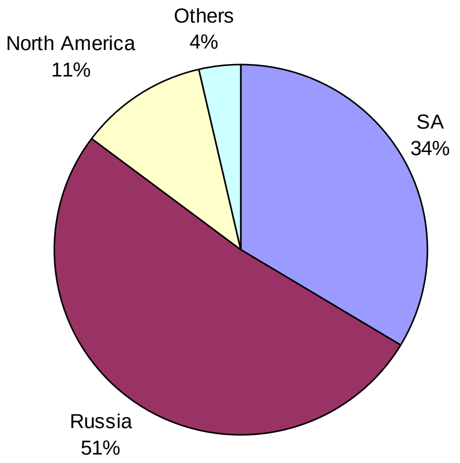

Figure 2: 2006 Palladium supply (8.48 millior oz).

It is generally believed that the magmatic Ni-Cu sulphide deposits within these layered igneous intrusions form as the result of segregation and concentration of droplets of liquid sulphide from mafic or ultramafic magma, and the partitioning of chalcophile elements into these from the silicate melt. Sulphide saturation of a magma is not enough in itself to produce an ore deposit. The appropriate physical environment is required so that the sulphide liquid mixes with enough magma to become adequately enriched in chalcophile metals, and then is concentrated in a restricted locality so that the resulting concentration is of ore grade.

Magmatic sulphide deposits fall into two broad groupings, those valued primarily for their Ni and Cu and those for their PGE. The principal magmatic sulphide deposits of the world are shown in Figure 3 (see Appendix 6), distinguished according to grouping. Placer deposits, resulting from weathering processes concentrating PGE from igneous rocks or sulphide deposits, are also shown. The most significant PGE producers are ringed.

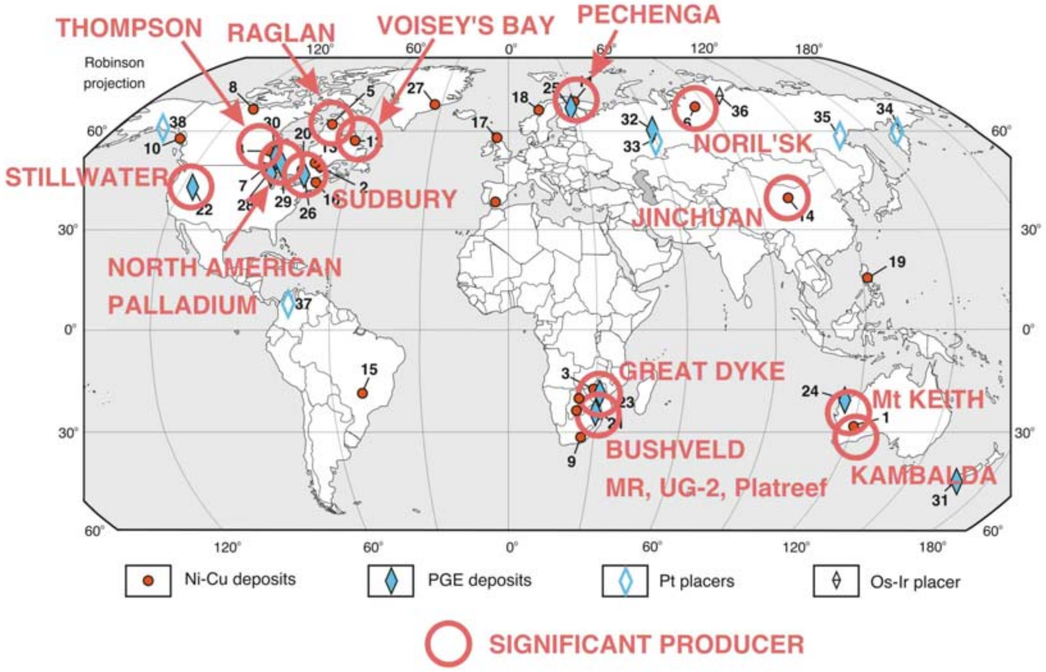

Figure 3: Location of magmatic sulphide deposits, along with other types of PGE deposits.

The proportions of the different elements in each deposit vary from deposit to deposit, with more variation discernible with differences in geographic location and genetic type (see Appendix 6).

5.2 Russian and South African producers

The major sources of at-risk materials are producers from the Bushveld Complex in South Africa and from Norilsk and Pechenga in Russia.

5.2.1 Russia - Pechenga and Noril’sk

There are two major production complexes united under the MMC Norilsk Nickel name – the Polar Branch in the Taimyr Peninsula and the Kola MMC located in the Kola Peninsula. Both production complexes use massive Cu-Ni sulphide ores as primary raw material. Mineral resources of the Polar Branch include Norilsk-1, Talnakh and Oktyabrsky. The Talnakh and Oktyabrsky mineral deposits form the so-called Talnakh ore cluster. The Kola MMC resource base includes a number of Cu-Ni deposits located in the Pechenga District of the Kola Peninsula. The most significant among them are Zhdanovskoye – Pilgujarvi, Zhdanovskoye – Severny, and Zhdanovskoye – Central.

Ore types in a large number of mineral deposits vary dramatically in their composition and processability. For instance, in the deposits of Norilsk Industrial Area there are three principal ore types categorized according to their sulphide content and metal grades:
Rich ores – sulphides over 70% with Ni – 2-5%; Cu – 2-25%; PGE – 5-100 g/t.
Disseminated ores – sulphides from 5 to 30% with Ni – 0.2-1.5%; Cu – 0.3-2%; PGE – 2-10 g/t.
Cupreous ores – sulphides from 5 to 30% with Ni – 0.2-2,%; Cu – 1-15%; PGE – 5-50 g/t. Up to 30% of all cupreous ores are refractory (difficult to concentrate).

Processing

After mining, the ore is crushed, milled and upgraded (Figure 4). Although some of the ores being mined are amenable to direct smelting, most are beneficiated by normal froth flotation geared to the recovery of the sulphide minerals. The proportion of the Polar Branch to the total metal output of the Company is close to 75%. The Polar Branch operates two ore concentrators: the Talnakh concentrator and the Norilsk concentrator. Both produce several types of final products:

> Copper concentrate (Cu 23÷27.5%, Ni 1.1÷1.7%): feed for the Copper Plant;
>
> Nickel concentrate (Cu 0.6÷4.5%, Ni 4.7÷9.2%): feed for the Nickel Plant and the Nadezhda Metallurgical Plant;
>
> Pyrrhotite concentrate (Cu 1.5%, Ni 2.2%): feed for the Nadezhda Metallurgical Plant;
>
> Gravity concentrate (Pt 500 g/t): feed for the Metallurgical Shop (Copper Plant).

Three metal-producing plants (smelters and refineries) further process the various concentrates.

Nickel Plant

The lower grade concentrates are smelted and converted, after which the converter matte is slow-cooled in large ingots. The converter matte, containing base metals and PGE, is crushed and milled followed by magnetic separation and flotation to form a copper and nickel concentrate. The copper concentrate after the matte separation stage is sent to the Nadezhda Metallurgical plant. The nickel concentrate is roasted to remove sulphur, followed by anode smelting and electrowinning. The final products from the Nickel Plant are nickel cathodes and residues (sludge accumulating at the bottom of electrowinning tanks). The residues are transported to the Metallurgical Shop of the Copper Plant for further recovery of precious metals.

Nadezhda Metallurgical Plant

The copper concentrate received from the nickel plant is converted into blister copper. The blister copper is cast into anodes which are transported to the Copper Plant’s tank house for copper electrolysis.

Copper Plant

Copper-rich concentrates from the Talnakh and the Norilsk concentrators are smelted and converted into blister copper in the copper plant. The copper anodes are further processed using an electrowinning process. Final products of the Copper Plant are copper cathodes and residue that accumulates at the bottom of the electrowinning tanks. The residue is transported to the Metallurgical Shop of the Copper Plant for further recovery of precious metals.

Electrolytic residues (sludge) from the Nickel Plant and Copper Plant tank houses, as well as the gravity concentrate from the Norilsk concentrator, are processed in the Metallurgical Shop (Figure 5). After roasting of the various feeds, two types of calcines are produced. These are further leached with sulphuric acid resulting in an insoluble cake that contains the precious metals; this is sent for anode smelting to produce secondary anodes which are electrolytically refined. The final product of the Metallurgical Shop has a Pt and Pd content in excess of 59 %.

Metals such as copper, silver, rhodium, ruthenium (together with some platinum and palladium) precipitate at the surface of the cathodes during electrolysis. After acid leaching, washing, drying and grinding this product contains 18 to 38 % Pt and Pd Platinum concentrates and it is shipped for further refining to the Krasnoyarsk Precious Metals Plant. According to Norilsk specialists the typical Pt to Pd ratios of Norilsk and Kola ores are retained during processing up to PGE separation and refining stages (Krasnoyarsk Precious Metals Plant).

The general process flow from mining to the production of high-grade concentrates of precious metals is very similar in both the Polar Branch and Kola MMC, and is graphically summarised in Figures 4 and 5.

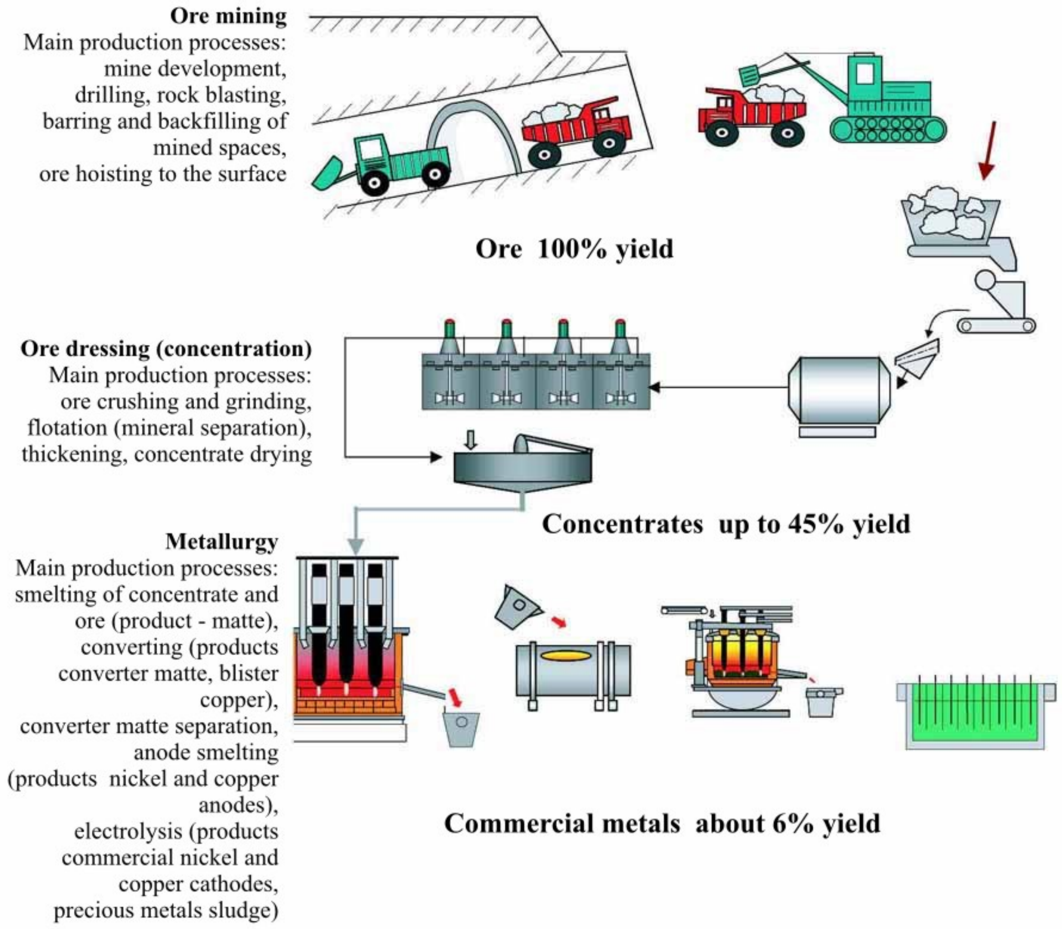

Figure 4: Metal production at MMC Norilsk Nickel.

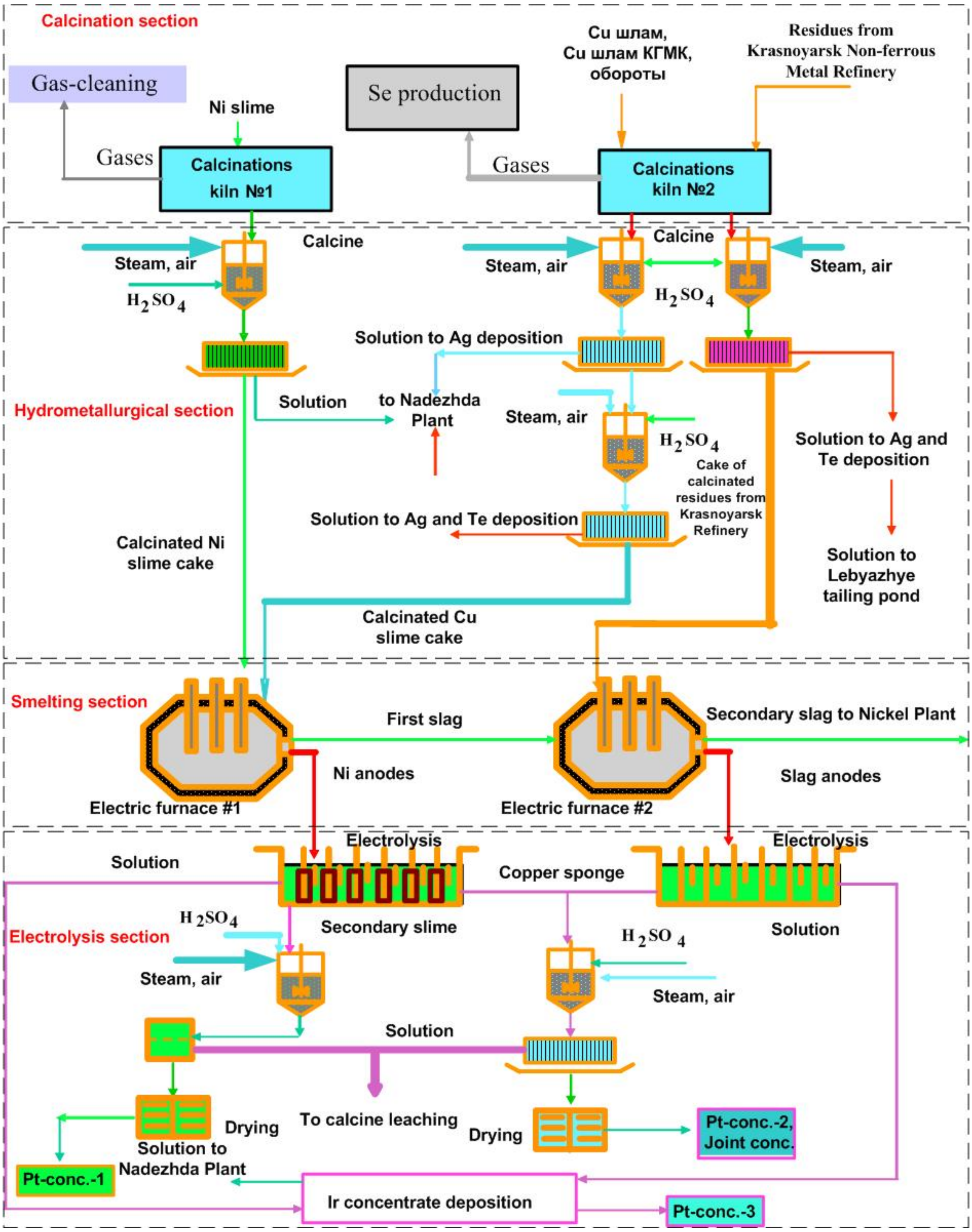

Figure 5: Metallurgical Shop process flow sheet.

5.2.2 South Africa - Bushveld Complex

The Bushveld Complex is the world's largest layered intrusion, and hosts the largest concentration of PGE in the world. Three different reefs are currently mined for their PGE content — the Upper Group Chromitite No. 2 (UG-2), the Merensky Reef and the Platreef. The abundances of the PGE for all the mines that exploit the Merensky Reef on both western and eastern limbs of the Bushveld Complex are relatively uniform over 100 km of strike length, at 5 to 8 g/t. The relative proportions of the various PGE vary little over this same distance. In contrast, the platinum-group minerals (PGM) vary enormously. On a regional scale around the western limb, the mineralogy ranges from alloy-dominated in the northwest to sulphide- or telluride-arsenide dominant in the southeast. Local features that are important in terms of mining are potholes and discordant ultramafic bodies. This variation in the mineralogy around the complex gives rise to differences in processing which are important in the distinction between the different smelter products from the different producers.

Most of the production is currently sourced from the western limb of the Bushveld complex (*cf.* Figure 6). The Rustenburg area hosts the Lonmin, Impala and Anglo Platinum smelter complexes (smelter and converter operations). Anglo Platinum operates additional smelters at Mortimer (northwestern limb) and Polokwane (eastern limb). The Northam smelter is also in the northwestern limb (Northam mine).

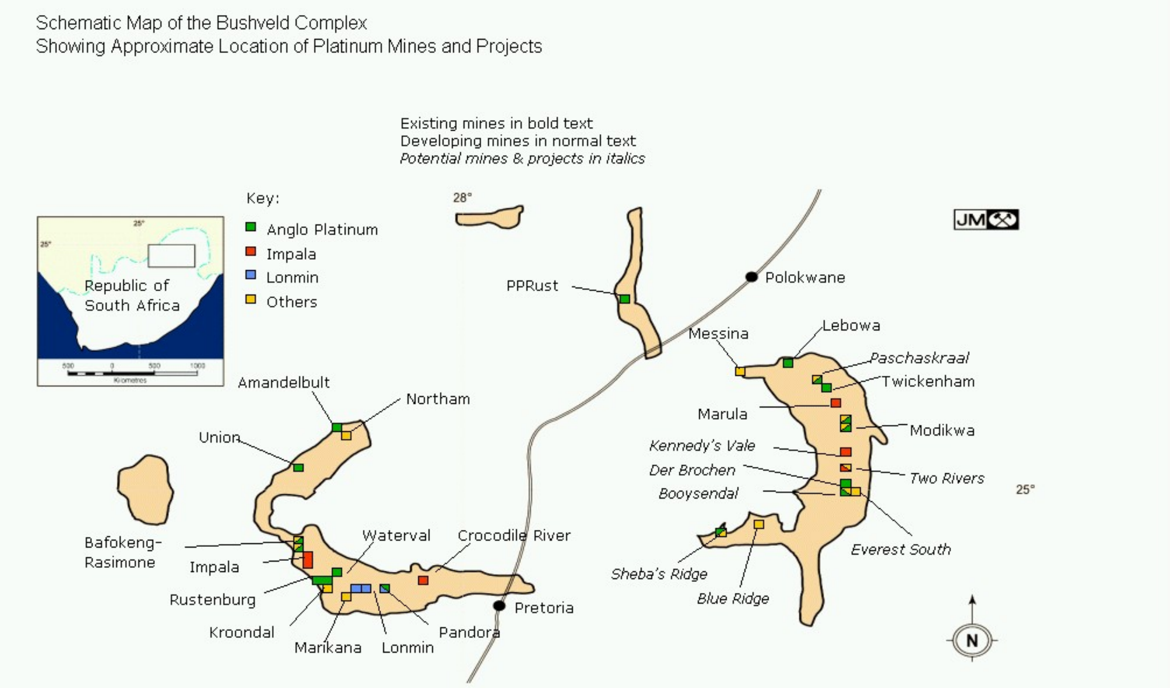

Figure 6: Map of the Bushveld Complex showing the location of the producing mines and projects.

There are two base metal refineries around the Rustenburg area (Lonmin and Anglo Platinum). The Northam base metal refinery is located at the Northam smelter, whereas the Impala base metal refinery is situated on the East Rand. The Anglo Platinum Precious Metal Refinery is also located in Rustenburg, whereas both the Lonmin and Impala Precious Metal Refineries are located east of Johannesburg. The Impala precious metal refineries also treat Zimplats matte and recycle catalysts. The Northam PGE are further refined by Heraeus (based in Germany, but has recently opened a new refinery in Port Elizabeth – Eastern Cape).

Processing

The extraction of the platinum group metals from the various ores generally follows a comminution process, followed by froth flotation. Although some losses of PGE do occur during the flotation process, the ratios of the various elements in the ores are maintained. At Anglo Platinum's Rustenburg and Amandelbult Merensky Reef operations, the coarse and dense PGE alloys (about 30% of the total platinum in the Merensky) are gravity concentrated into a very high-grade stream, which bypasses the smelter stages and is an early feed to the refinery. Apart from these exceptions, the successive concentration processes do not differ radically from one mine to another. Figure 7 is a generalised graphic depicting the usual grades for the various process steps.

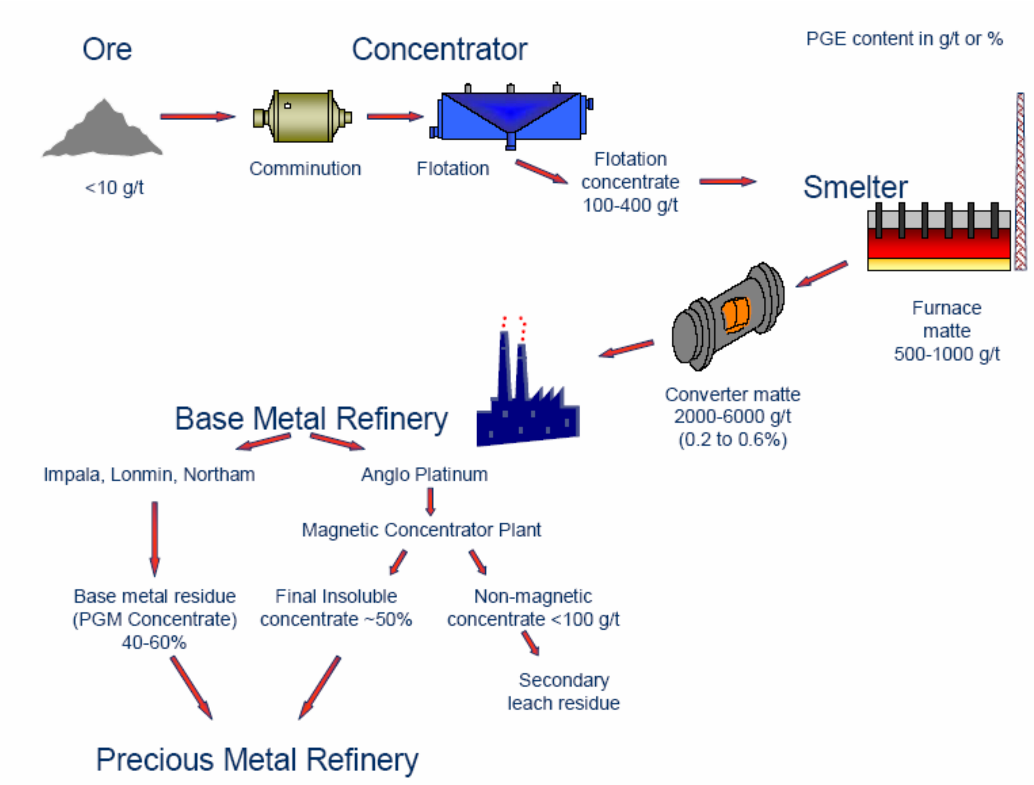

Figure 7: Generalised flowsheet for PGE production in the Bushveld and Great Dyke deposits.

The grades of flotation concentrates from producers in the Bushveld Complex vary, with UG-2 concentrates being generally higher than Merensky and Platreef concentrates. The upgrade during this process is around 20 to 80 times, with concentrates varying between 100 to 400 g/t. Base metal contents of these concentrates are low, varying between 1 to 4% Cu and 1.5 to 7% Ni for the Merensky reef and the Platreef, and between 0.3 to 1.5% Cu and 0.6 to 2.0% Ni for the UG-2.

The flotation concentrate forms the feed to the smelting process. During the smelting step the PGE-rich matte is separated further from the non-valuable gangue minerals. As for flotation, the smelting process is common to all major platinum producers and the PGE-rich product contains high levels of nickel, copper, iron and sulphur. For Bushveld and Great Dyke producers, the PGE content will vary between 500 to 1000 g/t. Losses in the smelter are low: recoveries vary between 95 and 98% and the characteristic PGE ratios are maintained. The molten furnace matte is transported in ladles to the converter (Impala, Lonmin, Northam), or granulated (Anglo Platinum Waterval Smelter) or cast into ingots (Anglo Platinum Polokwane and Mortimer Smelters) and crushed before being sent to converters for further upgrading. The physical product of the smelting process (feed to the converter) will thus vary between different producers but the chemical signature will be similar.

In the converting process, the PGE are further upgraded by blowing down the iron and sulphur contents. This also upgrades the nickel and copper levels. However, as for the smelting process, the base metal ratios (Ni to Cu) as well as the PGE ratios are quite consistent. Anglo Platinum slow-cool their converter matte which requires a specific iron and sulphur content to produce a quality product. The other Bushveld producers granulate their converter matte. At Anglo Platinum the slow-cooled matte is crushed for subsequent processing and so the converter product exhibits different physical characteristics from other Bushveld producers.

At Anglo Platinum the crushed converter matte is further upgraded by a comminution and magnetic concentration process. The PGE-rich magnetic concentrate is leached at a separate section at the Base Metal Refinery. This process further removes the base metals, producing a concentrate containing in excess of 50% PGE still having a typical Bushveld PGE ratio. The other producers leach the granulated converter matte at their respective base metal refineries to electrowin the base metals, also ending up with a PGE-rich concentrate. The PGE-rich concentrates are further treated at the various Precious Metal Refineries. Here various chemical processes remove the different metals, forming PGE-rich streams. Products from these processes have lost their typical Bushveld signature.

5.3 Provenance of beneficiation products (converter matte study)

Utilising a variety of elemental and isotopic ratios, it is possible to discriminate the majority of ore deposits from each other (see Appendices 6 and 7). It is, however, difficult to use ore deposit characteristics to prove the provenance of recovered material, because of the variability in chemical element distribution within a mining area and between mining areas. This requires a comprehensive database of all ores from all deposits, which is not practical, and published information is of a variable quality and thus usefulness. This makes it very difficult to identify a recovered process product with a specific producer based on ore type characteristics with any certainty. Although it might also be possible to trace back from a specific point in the beneficiation process, this would require a database of the characteristics (chemical and mineralogical) of all products originating from the various operations.

A material which retains the distinctive chemical characteristics of the ore deposit (e.g. Pt to Pd ratios, Cu and Ni contents), but is a representative sample of a particular deposit, is therefore the material of choice for database purposes. Converter mattes have been identified as the ideal material, as they are common to all producers and retain the essential chemical signature of the deposits from which they are derived.

The reason for choosing converter matte is the value of this material. In South Africa, smelter products are most at risk of being stolen, because the feed to the smelter is of too low a grade to warrant further processing. This also has the advantage that the product might acquire additional differentiating characteristics imposed by the specific process route. The majority of producers follow a pyrometallurgical route with converter matte forming the feed to the subsequent hydrometallurgical process. Converter mattes are a common denominator and an averaging factor in the process because the feed to the smelter is generally a blend of ore types. Converter mattes from different PGM producers were examined (Appendix 8) to determine whether they are sufficiently dissimilar to enable discrimination using a variety of chemical and mineralogical techniques. Some of these characteristic signatures would be maintained throughout the hydrometallurgical process to the point of further refining and separation of the platinum group metals. It should be borne in mind, however, that these findings are based on a limited number of samples which are not representative of all producers, and can be expected to vary with time.

Superficially, the granulated converter mattes look similar, as do the slow-cooled converter mattes; however it is possible to differentiate between pure converter matte from the various producers using a combination of techniques. The following are the most characteristic features:

1. Converter mattes from primary PGE-producers are characterised by much higher PGE concentrations.
2. Converter mattes from primary southern African PGE-producers exhibit Pt to Pd ratios in excess of 1.0.
3. In pure form, some macro-physical characteristics (granulated vs. crushed and particle size distributions) can be used to differentiate further between mattes from some of the southern Africa primary PGE-producers.
4. Major and trace element data might aid in identification and differentiation, but apart from varying Ni to Cu ratios for Norilsk and Bindura, and an anomalous high Pb level in Stillwater, chemistry will not be conclusive.
5. Apart from the Bindura mattes, which are characterised by a very high alloy content combined with a shift in the alloy peak position (due to the different composition of the alloy phase), the remainder of the converter mattes studied have similar mineral abundances, resulting in similar XRD patterns. As a result one cannot differentiate between all producers using X-ray diffraction.
6. Similarly, bulk modal compositions will not be conclusive in differentiating different converter mattes from one another.
7. Different mattes, although having similar compositions and therefore containing similar phases, exhibit different textural characteristics. These differences are mainly manifested by the distribution, grain sizes and shape of the alloy phases.
8. Specific phases present in converter mattes, such as alloy and spinels, have different compositions, and can be used to differentiate further between mattes from various producers.

Further beneficiation of the converter mattes can lead to a situation where differences between producers could become more pronounced, as differences become accentuated, or conversely that the differences become lost, and products from different producers become indistinguishable. It is thus necessary that, in order that a distinct characterisation of a producer’s material be made, unique characteristics which are diagnostic to that specific material from the specific producer be identified. It might also be found that specific characteristics which can identify most materials from a specific producer exist. For both situations, studies are necessary in order to identify these unique markers and to determine how many samples are needed to be representative, for database and identification purposes.

6. Description of the Complex Identification Procedure (CIP)

The intended use of the CIP is the characterisation in a consistent manner of materials of questioned origin in order to compare them with materials of Russian origin in the Reference Data Base (RDB). The data can also be compared with that for materials from other regions of the world, where available.

The CIP consists of six analytical protocols (CIP 1 to CIP 6), referring to individual preparation and analytical techniques (Appendix 9). Various analytical techniques are used to determine the following:

Bulk elemental compositions
Phase compositions of materials
Elemental compositions and morphology of individual particles to define groups of particle types.

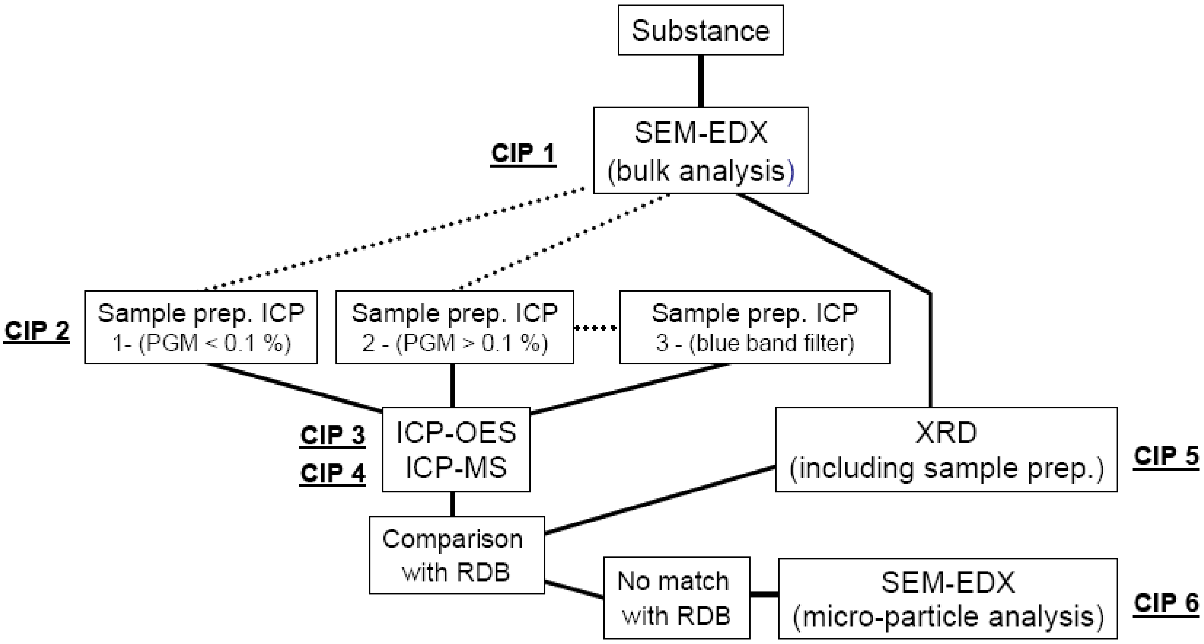

Figure 8. Analytical scheme as described in the CIP, showing the workflow of sample analyses utilising the six analytical protocols.

The stepwise approach of the CIP is as follows:

1. Bulk elemental composition of the material is determined by SEM (**CIP 1)**.
2. The results are used for the preliminary identification of the material and determination of the sample preparation method (**CIP 2**) for ICP-MS and ICP-OES analysis.
3. Bulk elemental composition is determined by ICP-OES (**CIP 3**) and ICP-MS (**CIP 4**).
4. Phase composition is determined by XRD (**CIP 5**).
5. The results are compared against the RDB.
6. In the case of a match with one of the RDB materials (i.e. when all diagnostic features coincide), the conclusion is made that the analysed sample came from the same source as the corresponding material in the database.
7. If the analysed sample does not match any of the materials in the RDB, then the hypothesis that the sample is a mixture containing an RDB material is tested by determining the elemental composition and morphology of individual particles of the sample using SEM (**CIP 6**).
8. If some particles in the sample correspond to particles of one or more materials in the RDB, these materials are presumed to be present in the sample. This is confirmed by comparing the particle suite and other measured characteristics of the sample with those of the corresponding materials in the RDB.

7. Verification of the analytical methods

This chapter summarises two activities utilised to verify the analytical methods: the verification study that was carried out by TNO and the BKA report on their investigation of mixtures.

The relevant reports are contained in Appendices 10 (TNO Report) and 11 (BKA Report).

7.1 TNO verification study

The objectives of the TNO study were to:

- Confirm the CIP’s fitness for purpose;
- Test the suitability of the RDB for determining the origin of a PGE-bearing material (the plant, the shop, the technological section).

TNO performed a verification of the CIP (Appendix 10) by auditing the documentation submitted by Norilsk Nickel, testing and revising the CIP protocols, statistically evaluating the RDB and observing the application of the CIP at the Norilsk St Petersburg Laboratory.

TNO evaluated the CIP by using it to analyse three samples (N1 - copper sludge from Kola MMC, N2 - nickel sludge from Kola MMC, and N3 - a 1:1 (w/w) mixture of N1 and N2). The identity of the samples was unknown to TNO during testing, although they were told that N3 was a mixture of the other two samples. The results are discussed in Section 8.3.

The CIP consists of a detailed analytical procedure for PGE-bearing materials. Results of these analyses are compared with those in the RDB. The RDB, as evaluated by TNO, contained analytical results obtained using the CIP for about 70 PGE-bearing materials. The RDB was statistically evaluated using fuzzy c-means clustering (FCM) to determine the extent to which the materials within the RDB could be differentiated.

TNO reached the following conclusions and recommendations (note that these do not necessarily reflect the views of the Board which are discussed in Chapter 10):

Conclusions and recommendations from the desk audit and analyses

1. On the basis of the desk audit and the analytical testing and verification it was concluded that the CIP was fit for purpose.
2. The individual protocols were revised by a team consisting of TNO, specialists from the involved Russian institutes and the Board. The current versions are attached as appendices.
3. It is recommended that the recorded XRD diffractograms should be compared with the diffractograms in the RDB instead of those in the ICDD PDF-2 database.
4. For identification of unknown materials it is necessary to interrogate the RDB in which all the Norilsk information is stored, e.g. chemical analyses, particle composition, and XRD diffractograms. To date this RDB is not available in an English version.
5. Results of certified reference materials should be made available and included in the digestion, ICP-OES and ICP-MS protocols.
6. The explanation of the qualitative and quantitative analyses given in CIP 6 should be illustrated with practical examples.

Conclusions and recommendations from the statistical evaluation of the RDB

1. The first three stages of the analytical procedure are of sufficient accuracy and precision to narrow down the provenance of the test-specimen to groups of a few materials, which can be further assessed in analytical stages 4 and 5.
2. Based on the bulk elemental data, not all materials in the RDB can be uniquely separated from each other. Fifteen clusters, each containing up to 10 materials, but generally about 4 materials, can be discerned.
3. Using the full RDB differentiation into more than 15 clusters is likely.
4. By adding additional analyses of all materials to the RDB, separation should be improved, although overlap in compositional signature is still to be expected for some materials.
5. With additional analyses available for all materials, each material should ideally be able to be assigned to its own cluster. In that case, discriminant function analysis (DFA) can be used to test the statistical significance of the separation.
6. One advantage of the FCM approach is that the focus is on the relevance of the compositional differences (cluster centres), rather than just on statistical significance of threshold boundaries that may change with advances in analytical techniques.
7. A second major advantage of FCM is in the allocation of test specimens to clusters. This allocation is allowed even for partial similarity to a cluster or placement between two or more clusters.

7.2 BKA verification study

The BKA evaluated the CIP by using it to analyse the same set of samples that were studied by TNO. The results are discussed in Section 8.3. The BKA analyses were performed following the 2005 version of the CIP, with slight modifications to accommodate the instrumentation available in the BKA laboratory. Analytical techniques used were XRF, XRD, ICP-MS and SEM.

It was known by the analysts that sample N3 was a mixture of N1 and N2. Considering only this hypothesis, XRF results were sufficient to indicate that sample N3 was a mixture of a 1:1 ratio by mass of N1:N2. SEM was also used for screening of the samples, although the BKA considers XRF a better alternative in this case. The elemental compositions determined in this way are, within the limitations of this screening method, similar to TNO and BKA results by other bulk methods.

The phases identified in the samples by TNO and BKA differ, even though the XRD diffractograms for each sample are very similar in peak positions and relative intensities. The differences in phase identification may reflect the very complex character of the samples that contain many similar phases.

ICP-MS results for the three samples were found to be very similar to those obtained by TNO: sample N3 measurements were very close to the average of N1 and N2, as would be expected for an equal mass mixture.

SEM was used to determine the compositions of particles in samples N1, N2 and N3 according to CIP 6, except that only approximately 100 particles were analysed for each material.

7.3 Conclusions from the verification studies

The CIP is suitable for discrimination of NN PGE-bearing materials and, within an appropriate closed universe, the identification of NN PGE-bearing materials by characterising questioned materials and comparing the results of this characterisation with data in the RDB.

This conclusion is based on the results obtained by TNO and BKA in a limited testing of the CIP, both for single-source materials as well as for a simple 1:1 mixture of these materials. Such a test appears to be suitable to test the methods and competence of laboratories that are new in the field of analysis of PGE-bearing materials. For the general interpretation of results, however, much more experience and intimate knowledge of the production processes and materials are required.

8. Testing of the CIP for mixtures

Several tests of the CIP to identify materials in the RDB in mixtures have been performed and are summarised below.

8.1 SAR test

In 2003, 24 samples of “at-risk” materials from four South African producers (Anglo Platinum, Impala Platinum, Northam Platinum and Lonmin Platinum) were supplied to the ICFSS, together with three mixtures containing some of the supplied 24 samples in order to test the method. Samples were rendered in the form of finely dispersed powders.

The analysis of the mixtures was conducted on the basis of quantitative elemental analysis, XRD and SEM. After identification of the different components present in the mixtures, elemental analysis data of the mixed samples and comparison samples was processed using linear regression. According to the report, the mixtures were successfully identified and quantified (Appendix 12).

8.2 JM test

In a pilot study in 2003, Johnson Matthey prepared three samples for testing which consisted of at-risk South African materials as supplied by NN which were “masked” by a mixture of process intermediates from Johnson Matthey’s own refining process. The CIP was used to investigate the samples and correctly identified the materials supplied, as well as providing an approximate quantification (Appendix 13).

In this case the analysts were looking for material which corresponded with the material that they had supplied. No information has been supplied as to the nature of the masking materials used by Johnson Matthey, so the ease or difficulty of the test cannot be assessed, especially when considering the variations and similarities in materials from different producers, such as converter mattes as discussed in Chapter 5. After receiving the results of the analysis of their prepared mixtures, Johnson Matthey stated (see Appendix 14) as follows:

“The results obtained from this preliminary test of the “tracking procedure”, demonstrated that it showed some promise. Since then JM have been broadly supportive of the method and have recommended:

1. further development of the techniques within the procedure take place to reflect “best available technology”;
2. that consensus for implementation of the procedure should be sought from experts within the member companies of the International Platinum Association; and
3. that a more thorough validation of the “tracking procedure” be designed where a much wider range and variability of the target materials be employed.”

8.3 Initial testing of the CIP in this project

In the initial stages of this project it was decided to test whether the analytical protocols (**CIP 1- 6**) were suitable for the purpose for which they had been designed. TNO and the BKA received three samples, two of RDB samples and the third a 50/50 mixture of the two. Details of these tests are given in Chapter 7 and Appendices 10 and 11. Both laboratories were able to identify the two pure samples and to determine the composition of the mixture. However, all that this testing showed was that the analytical methods were fit for purpose. The applicability of the CIP to real world situations, where the material was unknown, and could be represented in the RDB or not (as in the JM test), was not tested here.

It was therefore decided to perform a test of the CIP methodology under a more real-world environment using Norilsk materials as well as materials from other producers and from other deposits, in order to test the RDB and the forensic application of the method.

8.4 “Blind” Test of the CIP

The purpose of this test was:

1. Experimental confirmation of the CIP's suitability for characterisation of NN at-risk materials in mixtures with each other and with North American and South African PGE-containing materials.
2. The confirmation that the information contained in the NN RDB of at-risk PGE-containing materials was sufficient to enable the identification of materials that have been analysed using the CIP.
3. Each participating laboratory was to use the RDB to identify the at-risk materials within the test mixtures. Each participant was also asked to provide an estimate of the amount of each material in the mixture.

The composition of the test samples was agreed to as follows:

1. The test samples would consist of three mixtures containing NN materials and/or materials from other producers.
2. The content of each material would be greater than 10%. The reason for this was that in most real life situations the suspect material usually consists of only a few components.

The samples used to prepare the 3 test mixtures were selected from the following:

- N35 Leached secondary sludge from NN Polar Branch
- N25 Leached copper sponge from NN Polar Branch
- N37 Secondary sludge before leaching and washing from NN Polar Branch
- N38 Copper sponge before leaching and washing from NN Polar Branch
- N22 Calcined copper sludge cake from NN Polar Branch
- N23 Calcined nickel sludge cake from NN Polar Branch
- N24 Secondary anodes from NN Polar Branch
- N27 Slag anodes from NN Polar Branch
- N18 Nickel sludge from NN Polar Branch
- N19 Copper sludge from NN Polar Branch
- SMC1 Converter matte from Stillwater Mining Company
- Converter matte from Lonmin
- Converter matte from Impala Platinum
- Converter matte from Anglo Platinum (Waterval Smelter)

The sample preparation, using the supplied samples of 100 g each, did not involve any further processing of the sample – they were all used as received. The mixes were prepared by weighing the individual components of the mixtures and then homogenising in a mixer. 30 g of each prepared mixture was given to each participating laboratory.

Originally, the testing procedure was to involve four different laboratories. However, because of unforeseen circumstances, only two laboratories were able to participate: the ICFSS (Russia) and Anglo Research (AR) (South Africa).

The composition of the mixed samples as well as the result of the participating laboratories is given in Table 1. The AR methodology for determining particle type differs from that of the CIP. Moreover AR uses automation that enables them to measure more particles as well as specific phases within a particle and so achieve better counting statistics.

The reasons behind the choice of the materials in the three test samples were:

Mix 1: Three converter mattes, as supplied were all received in a milled form, and not as they would have been stolen, so some of the physical properties of the particles had been changed. Here the aim was to discriminate between similar materials from different producers. It should have been fairly easy to recognise the different ore types (Stillwater vs. Bushveld) on the basis of chemistry. The more difficult test here was to be able to discriminate between two producers processing similar ore from the same ore body.

Mix 2: In this mixture, two sets of two similar materials from NN were put together to see if the method could discriminate between materials that were not easily distinguishable on the basis of their chemistry. The purpose here was to see if the database entries were representative of the materials, and thus the reliability of the identification could be ascertained.

Mix 3: This was a mixture of material from three different ore deposits and refineries. The two converter mattes are very distinct from each other as the one is granulated and the other slow-cooled. The anode materials are similar but are clearly discriminated in the RDB. The aim was to determine whether the particle size variation together with the chemical and phase characteristics would allow a clear discrimination between the different components of the mix.

Commentary to the results

This test was based on a limited number of materials, the origins of which were known to the investigators, and so the test was in fact not entirely “blind”, but rather a “closed universe” (see Chapter 9).

Mix 1	**Make-up**	**ICFSS**	**AR**
Stillwater Converter Matte	32	50	30
Lonmin Converter Matte	44	50(in total)	30
Impala Converter Matte	24		40
Mix 2			
N35 Leached secondary sludge	34	50	20
N37 Secondary sludge before leaching and washing	35	*	40
N23 Calcined nickel sludge cake	12	25	10
N19 Copper sludge	20	25	30
Mix 3			
N24 Secondary anodes	38	60	70
N27 Slag anodes	29	*	10
Stillwater Converter Matte	14	20	10
Anglo Platinum Converter Matte	20	20	10

*Table 1. Results of the analysis of the three test mixtures by ICFSS and AR. * signifies that the material was identified as being a potential component but was not confirmed and/or quantified in the findings*.

ICFSS:

The ICFSS focused on the identification of the NN materials (including Stillwater). Identification of other materials was for exclusion purposes, and was successful given the limited nature of the available material. They were unable to discriminate between similar materials from NN (Appendix 15).

The CIP allows for identification of the NN materials. However, some pairs of materials close to each other in the refining process are poorly discriminated when they are found in mixtures. This impeded reliable quantification of the mix components. This result is in agreement with the statistical analysis of the RDB by TNO as discussed in Sections 7.1 and 9.3.

AR:

The AR focused on the identification and discrimination of all the materials in the three mixes. This was successful given the limited nature of the available material. They were able to discriminate between similar materials from NN (Appendix 16).

The information contained in the NN RDB of at-risk PGE-containing materials was not suitable for the approach employed by AR for the identification and discrimination. Materials obtained from NN and other producers were used as a reference database because the NN RDB did not contain sufficient unique criteria that could be used for differentiation and confirmation. AR investigators also did not have the list of possible components, but were only aware that NN materials could be present.

It was found that bulk chemical composition was not very useful when trying to identify components in a mixture. Physical characteristics were found to be far more important, and here the use of automated particle classification enabled accurate identification of all phases within particles, even when only one reference sample was available.

9. Forensic inferences

9.1 Introduction

The purpose of the CIP protocol is to identify PGE-bearing materials that are represented in the RDB. During the course of discussions among members of the Board and the originators of the CIP protocol, it was apparent that the use of English terminology had different meanings to different people. The differences in the meaning of terms such as “identification” arise from linguistic, cultural, and legal differences. Therefore, to avoid misunderstandings we will define a few terms that will be used in this report. We use the term “identification” in the sense of assigning a questioned sample to a particular origin. The term “origin” can refer to any of a hierarchy of sources. For example, the data derived from use of the CIP could be used to identify a material of unknown origin as being an intermediate product in PGE beneficiation, or identify the material as being a PGE-bearing material of Russian origin, or in the most explicit application, to identify the material as originating from a specific point in the processing of PGE intermediate products in a particular manufacturing facility in Russia. Each of these identifications is an inference which is made with some degree of uncertainty; and the degree of uncertainty increases with increasing specificity of the identified source. The Board recognises that this definition of identification is not universally accepted. In fact, not all Board members would necessarily use the term identification when uncertainty is associated with it. However, this usage is adopted here because the term “identification” is inherent in the CIP protocol as indicated in its title and its previous usage in Russia and it is most consistent with accepted western European legal practice.

Another term that is commonly used in forensic science that bears an intimate relationship with the concept of identity is that of “indistinguishability”. When two items are compared with each other, such as a recovered material and a potential source material present in a reference data base, they are declared analytically indistinguishable when the values of all measured parameters in both samples are in one-to-one agreement with each other according to a set of documented match criteria. The requirement that a specimen is indistinguishable from a potential source is a prerequisite for identification of that as the true source. However, indistinguishability is not in itself enough for identification, in that it does not provide information about the number of other potential sources that may exist. The Board believes that the CIP produces information that provides excellent discrimination among many of the world’s sources of PGE. However, the ability to identify uniquely a single source is limited by the lack of completeness of reference databases, particularly for materials of non-Russian origin.

The Board believes that the CIP, when properly applied, provides data that can be interpreted by an expert to provide reliable inferences concerning source identification, provided that the expert has good knowledge of the PGE processing procedures and has access to appropriate reference data. In many forensic cases, these inferences will provide valuable investigative leads and may also serve as expert opinions at trial. The degree of uncertainty associated with inferences drawn from CIP data depends upon the makeup of the questioned sample and the specific details of the case at hand, particularly the number and identity of possible sources. In the following, several hypothetical scenarios are discussed to illustrate how inferences based on the CIP results can be formed. The examples are not meant to be all-inclusive. The formulation and wording of inferences in a particular case will depend heavily upon the nature of the evidence, the questions being asked of the expert, and the knowledge of the expert.

9.2 Single product samples

Initially, we will consider a questioned sample that consists of a single PGE-bearing material and no other components. The analysis of such a questioned sample proceeds according to the protocols described in the CIP and the analytical results are compared with data contained in the RDB by means of the documented criteria. If two samples are found to be not different according to the criteria, then they are said to be indistinguishable, or that they "match". We consider the case where the questioned sample is found to be indistinguishable from one, and only one, of the materials in the RDB. First, we note that the Board does not believe that all 69 materials or even that all 33 at-risk materials identified in the RDB can be unambiguously identified. Various statistical studies conducted by Board members as well as those of TNO and BKA presented in Chapter 7 and Appendices 10 and 11, indicate that several of the listed materials are so similar that they should be grouped into a single category from the standpoint of source identification by the CIP. Depending upon the statistical test used, the 33 at-risk materials logically group into approximately 25 distinguishable groups. Therefore, one can at best place a questioned material into a material group that may contain materials from several closely related stages in a single manufacturing process.

The inference to be drawn from a match result depends on the circumstances of the case. If these are such that the questioned sample must have come from one of the sources represented in the RDB – a so-called "closed universe" - then there is very strong support for the conclusion that the questioned sample must necessarily have come from the same source as the material that it matches. An example of this type of case follows:

Case Example 1: Materials were confiscated during customs inspection of passengers on a domestic flight to one of Moscow's airports. Among the questions posed to the forensic experts of the Institute of Criminalistics Special Equipment Centre of the Russian Federal Security Service were, "What is the substance, what is its origin, and how was it produced?" The seized material was analysed following the protocols of the CIP. The circumstances of the case (the fact that the given material had not crossed the Russian Federation's border) allowed the use of the available database on Russian materials to identify the source of the material. The forensic experts determined that the material presented for examination was a product obtained by processing copper-nickel sulphide ores, specifically the material designated as secondary anodes produced in a metallurgical workshop of the Norilsk Nickel company's Polar Branch. In response to the third question, the forensic experts stated that the description of the technology was not within their field of expertise (although presumably it would be within the expertise of engineers in the manufacturing plant).

This example is representative of several cases in which the CIP was effectively utilised within the Russian legal system. In fact, prior to the actions of this Board, it appears that the CIP had already received widespread acceptance in Russian courts. The task of this Board was to evaluate the acceptability of the CIP and inferences drawn from it in a Western European legal system. An instance of a closed universe for a Western European case could be where the two parties in a legal action each claim an alternative origin for the questioned sample. If reference samples are available for each of the proposed origins, a comparison with the questioned sample can be made. In such a case, when the questioned sample is found to be indistinguishable from one of the proposed origins, but distinguishable from the other, then the inference is straightforward.

When the circumstances of the case do not present a closed universe of possible sources, then the inference is not so straightforward. In such a case, it is necessary to refer to whatever information is available relating to PGE mining and production worldwide (see Chapter 5), and to RDBs from other regions of the world, such as South Africa, or by analysis of exemplar samples representing potential sources. Any inferences drawn in such a case must take into account the uncertainties arising from the incomplete compilation of source information.

Case Example 2: A material containing precious metals was delivered to a European refinery by a Western European firm. Terms of delivery and a number of other factors led to a suspicion of a criminal nature of the consignment and a possible Russian origin. The firm that supplied the substance referred to it as a secondary raw material for precious metal production representing spent catalysts and copper-nickel sludge originating from an African country, for which no reference samples were available. A portion of the material was provided to the ICFSS for evaluation. The questions posed to the forensic experts included the following: “What is the material that is presented for analysis?”; “What is its chemical composition?”; “Does the material contain precious metals and, if so, in what amounts?”; “What is the country of origin of the material?”; “Is the material a product of one of the Russian manufacturing enterprises and, if so, what is the name of the product and the location at which it was manufactured?”; and “What is the value of the precious metals in the samples presented for analysis?” In other words, the Russian experts were asked to identify the material to the highest level possible.

Using the results of the CIP and the information in the RDB, the forensic experts at ICFSS concluded that the substance presented for examination was a material obtained by processing copper-nickel sulphide ores containing PGE. As evidence for this conclusion, the experts cited the material’s basic elements (copper, nickel, sulphur and oxygen), the phase composition of the material (largely made up of copper and nickel oxides and sulphides), and the presence of a number of specific elements (selenium, tellurium and arsenic) typical of materials obtained from processing copper-nickel sulphide ores. NN has compiled a database of industrial wastes that contain precious metals. The levels of selenium, tellurium and, to a lesser degree, arsenic determined in the questioned material using the CIP are significantly greater than in any of the industrial wastes contained in this specific database. Most industrial waste samples, which typically contain aluminium, silicon and iron oxides, carbon and aluminosilicates, are also quite different from the questioned samples. Therefore, they concluded that the examined material was obtained by processing ores and was not a previously observed industrial waste. Moreover, the relative concentrations of platinum and palladium in the examined material were atypical of materials made from South African ores, but were typical of Russian-made intermediate products of the PGM beneficiation process. Finally, using the measured precious metal concentrations and current world market prices, the experts were able to place a value on the recovered materials.

Case example 3: A joint investigation by Russian and foreign law-enforcement agencies led to the detection of a suspicious material on offer on the Western European market. A preliminary examination revealed that the chemical composition of the material closely resembled one of the samples of previously stolen materials, which were delivered to a major European refining company for processing. An investigation conducted by the ICFSS in which the CIP was utilised led to the conclusion that the material was not of Russian origin.

9.3 Multiple product samples

Evaluation of the results for cases in which the questioned sample is a mixture of materials from two or more origins is more problematic. Although the CIP protocols indicate that they can be used to identify the presence of Russian materials in mixtures, specifically the use of the SEM protocol

CIP 6, they do not include exact criteria for apportioning measured data among components of a mixture. For example, the protocols for bulk elemental analysis only give match criteria for comparison of element concentrations for pure material. Additional information presented to the Board by Russian scientists suggests that various forms of linear combinations are used to assess the composition of mixtures. These protocols were not evaluated by the Board because they are not formal parts of the CIP as it currently exists.

The use of the CIP for mixtures is discussed in the testing described in Chapter 8. As shown in the blind tests, the success rate depends upon how much is known about the pure materials that make up a mixture. In all tests discussed in Chapter 8, the study was not completely blind, that is, the analyst had some knowledge of a group of possible end-member components. The initial TNO and BKA verification studies using the two component mixture demonstrate, as expected, that physical mixtures result in a composition that is a linear combination of the measured values for the end members. In simple cases the CIP can be used to quantify accurately the components of a mixture. Therefore, it could produce very strong evidentiary support in simple mixtures of two PGE-bearing materials, or a mixture of a PGE-bearing material with a masking or diluting agent provided the end-members are available for testing or their compositions are otherwise known accurately.

In the case of mixtures of several components of unknown origin, the success of the CIP in identifying components depends heavily upon the availability of accurate RDB data. When a mixture consists only of materials present in the Russian RDB of PGE-containing materials, one should be able to determine the approximate amounts of each component provided the pure materials are fairly represented in the database. In order to assess the success rate for these mixtures, we must first look at some limitations of the current RDB data and the uncertainties that they place upon expert opinions. The version of the RDB provided to the Board comprised 68 materials, 33 of which were characterised as “at-risk”. The Board and several technical advisors attempted various methods of statistical classification of the data in the database and were unable to identify confidently each material in the RDB to the exclusion of all others. As discussed in Chapter 7, using the data alone, we could only subdivide the 33 at-risk samples into roughly 25 distinguishable groups. Further subdivision among material groups would require data that is not quantitatively represented in the RDB and so is not suitable for statistical analysis. Additionally, the RDB does not contain enough data to provide a good overview of the range of compositions that occur for each material category. The database contains only a few replicate measurements acquired over a short time period for many of the samples. The match criterion cited in the CIP protocols is range overlap. Ranges can only increase as new measurements are added to a given material. The Board expects that with an increasing amount of data, several of the now distinct materials will merge into a single group. We do not see this as a problem in utilising the CIP to form expert opinions, merely a question of linguistics. Rather than say that the CIP is capable of identification of each individual point in the manufacturing process as defined by the sample identifiers in the RDB, we believe that it is proper to classify each material as unique within the RDB or a member of a small group of indistinguishable materials. The indistinguishable materials within each grouping consist of intermediate products collected in close proximity in the production process. For example, N35 and N37 are secondary sludges from the NN Polar Branch process that have very similar compositional, mineralogical, and physical characteristics. It is not surprising that these two materials are similar since the difference between them is that N35 was collected before leaching and washing and N37 was collected after. These two materials are two that were not differentiated by the ICFSS in Mix 2 of the blind test discussed in Section 8.4.

The Board was not able to obtain a good feel for the degree of variability which would be expected within each material group when samples are collected across production runs over a long period of time. It is possible that with continued data collection, trends will be spotted over time reflecting changes in ore sourcing or production processes. No opinions can be offered for the variation to be expected in materials from other regions of the world besides Russia. For these reasons, it is important that addition of samples, both from Russia and other locations, into a broad data base be continued. It is our expectation that as more knowledge of world-wide production sources is obtained, including their variability, opinions formed in the evaluation of evidentiary samples will become more refined.

Most pairs of materials in the RDB are readily distinguishable and we expect that they will always remain so regardless of the number of reference measurements that are made. In general, samples that are widely different arise from different stages of beneficiation, so they have readily distinguishable levels of PGEs or base metals.

Forensic use of CIP for mixtures relies primarily on particle analysis (CIP 6). Other methods such as bulk analysis are less important because it is often difficult to deconvolute bulk data into individual component proportions. Again, the CIP protocol does not address this directly, nor does it appear to be designed to provide quantitative determination of mixture components. Rather, it is designed to provide identification of and the approximate proportioning of component materials. When a suite of particles is found which is indistinguishable from a material within the RDB, a statement can be made that there is support for the presence of that material within the mixture. As the percentage of a component material decreases within a mixture, the ability to detect its presence decreases and the uncertainty associated with a positive opinion increases. The Board cannot provide specific guidance as to how the expert should express an opinion in such cases, because it depends very much on the experience of the expert and the complexity of the questioned sample. Expert knowledge which exists within the PGM producers would be required to assess CIP results for more complicated cases.

It may be necessary to utilise methods outside of those described in the CIP protocols to increase the level of confidence in attribution of mixture components, *e.g.* the use of gravimetric procedures or isotope ratios. Although the Board has not evaluated other protocols, we recommend their use to provide supporting or additional information to that provided by the CIP.

10. Recommendations for improvements

The instrumental methods used in the CIP are modern and well-suited for the purpose of comparison and/or identification of PGE-bearing materials. Methods and procedures can, of course, be modified and developed. We feel that development of the analytical methods should be focused mainly on:

1. an automated determination of particle types by SEM;
2. the development of the RDB;
3. statistical evaluation of matching criteria for the bulk analysis methods – ICP-MS, ICP-OES and XRD;
4. other methods for characterisation of PGE-bearing materials e.g. isotope measurements.

An in-depth publication in English on the analytical methods of the CIP and its forensic application would be most welcome.

There is need for enhanced cooperation between producing companies in order to get a good understanding of the background data on which the forensic use of the CIP is based. This cooperation needs to be extended to the refining companies in order to introduce procedures that will make it possible for them to identify stolen materials. The Board also supports the initiative taken by NN to initiate a public-private partnership to counter illegal precious metal trafficking used to fund organised crime.

The adoption of the following specific suggestions and recommendations should encourage and enhance the adoption of the CIP internationally.

10.1 The protocols

We support the following recommendations made by TNO (Appendix 10):

1. Results obtained from the analysis of process-specific certified reference materials should be made available and included in the digestion, ICP-OES and ICP-MS protocols.
2. The explanation of the qualitative and quantitative analyses in CIP 6 should be illustrated with some practical examples.

The Board recommends that CIP 1 be modified to include X-ray fluorescence (XRF) with other sources besides SEM for the determination of bulk elemental composition.

From the testing of the CIP in mixtures it was observed that additional methods were used in some tests to identify and quantify components. From the test described in Section 8.5 it was found that, because of the complex nature of these materials, quantification may be inaccurate. The Board recommends that a 7th protocol should be developed and validated to describe how analytical results may be used to identify and quantify the components of a mixture.

10.2 Introduction of automation into particle type determination

The analysis of large quantities of particles is time-consuming and the method as presently applied in the CIP can only be carried out by highly skilled and experienced scientists. An automated SEM method for the detection and classification of particles should be utilised. This should ensure that:

1. the results are less expert dependent;

2. sufficient particles are analysed to characterise the material fully;
3. matching criteria for particle type determination are based on statistical evaluation of the data obtained; and
4. the consistency of results among laboratories increases.

10.3 Improvements to the RDB

10.3.1 More replicates

At present the database includes data for about 70 materials, with the number of analysed samples for each varying considerably. The Board is of the opinion that, ideally for each material, data for at least ten samples acquired over time should be included in the database. In this way the variation within each material may be better described. Moreover, the changes in the composition of the materials should be monitored and the RDB updated when necessary.

10.3.2 More parameters

Additional data that should be collected for each sample in the RDB should include physical characteristics such as colour, shape of particles, particle size analysis, magnetic properties and phase composition of particles. The RDB should be improved with the addition of distinguishing characteristics that will uniquely identify each material. This will most probably be a combination of characteristics that could be arrived at by a step-wise process.

10.3.3 More materials

The forensic inferences that may be drawn from the results of a CIP analysis depend, as we have seen, on the data that are available. The present RDB consists only of NN materials. For effective source attribution, data on materials from other producers and PGE-bearing scrap materials would be required. Creation of a worldwide database containing information on the composition of PGE-bearing materials, produced by all or most of the producers of these materials is desirable.

10.3.4 Statistical evaluation

When the database has reached sufficient size, it should be re-evaluated statistically to compare the variation within each material (product type) with the variation between materials (see the conclusions and recommendations given in the TNO report in Appendix 10). The aim of statistical evaluation would be to assess the extent to which materials in the RDB can be clearly distinguished from each other. It should include all quantitative data sets: data measured by ICP-OES and ICP-MS, data measured by SEM, XRD information, definition and distribution of all the particle types defined by SEM analysis.

10.3.5 Digital format

The present RDB does not contain X-ray diffractograms in a digital format. The creation of a digital database would allow other laboratories to carry out analyses according to the CIP and compare these results with the RDB more readily. Software packages are available that incorporate various algorithms, such as fuzzy clustering described in Section 7.1, for matching the observed composition with the diffractograms in the database.

10.3.6 English version

For identification of unknown materials it is necessary to interrogate the RDB in which all the NN information is stored, *e.g.,* chemical analyses, particle composition and description and XRD diffractograms. For international acceptance and usage an English version of the RDB is required.

10.4 Publication of the CIP methodology in English

At present there are the following publications in English: Perelygin *et al.* (2008), Karpov *et al.* (2007) and Karpov *et al.* (2006). The Board recommends that a comprehensive paper be prepared for publication in a reputable analytical chemistry journal, *e.g.* Journal of Analytical Atomic Spectrometry.

10.5 Isotope measurements

In addition to the analytical procedures included in the CIP, some other methods for characterisation of the PGE-bearing materials may be considered. From geological investigations of ores originating from different mines in the world, it is known that they show differing isotopic compositions for elements including sulphur, lead and strontium (see Appendix 6). For pure materials the isotopic composition might further strengthen the evidence that the examined material originates from a specific source. The beneficiation process, however, may alter the isotopic composition of the material. Studies should be undertaken of possible changes in isotopic compositions of materials through various beneficiation steps, particularly converter mattes.

10.6 Converter matte study

As we stated in Section 5.3, a project has been started to characterise converter mattes from various producers. It is important that this work be continued. The success of this project is heavily dependent on the continuing participation of the production companies worldwide.

10.7 Co-operation between Norilsk Nickel, other production companies and refining companies

In March 2006 representatives from NN, AR and the Forensic Science Laboratory of the South African Police Service participated in a workshop on the identification and databasing of “at-risk materials”. At the workshop it was seen that NN and AR used similar methodologies to build databases of PGE-bearing materials from their metallurgical operations and that a combined approach could be successfully applied to the identification of PGE-bearing materials. It was also agreed that the NN and AR methodologies should serve as a basis for further bilateral cooperation in the field of the identification of illegal trade in precious metals, including those materials supplied to large refining companies.

The parties expressed their intention to undertake the following steps:

1. Further development and continuous updating of a representative database of PGE-bearing materials of metallurgical operations, especially “at-risk” materials.
2. Promotion of information transfer through the exchange of standards and summary documents characterising individual “at-risk” materials (material data sheets or “passports”)
3. Continuous improvement of the developed methodology and the application thereof, supported by the exchange of information between the various parties.
4. Assistance to each other in attaining recognition (validation) for the methodology developed by NN and AR.

The Board considers that this type of cooperation is very important for the development of methods to counteract theft of PGE-bearing materials. It would therefore be of great value if this cooperation could be extended to include other producers as well.

10.8 Public- Private partnership

At the 1st Moscow G8 Global Forum on Public-Private Partnership to Counter Terrorism in 2007 NN initiated an international project to enhance public-private partnership to counter trafficking in stolen gold and PGE-bearing materials. The expected result of this is the development of internationally accepted approaches for the establishment of an efficient mechanism to facilitate cooperation between industry, governmental institutions and law-enforcement bodies to counter illegal worldwide trafficking in precious metals. The Board supports this action.

10.9 Quality assurance

In the Boards’ opinion any laboratory that uses the CIP should be accredited in accordance with ISO 17025 (or equivalent level) to perform such analyses. Such a laboratory should also take part in regular proficiency testing similar to that organised by the Board (see Section 8.4) *i.e.* that unknown samples are prepared containing mixtures of PGE-bearing materials from different sources.

11. Conclusions

1. We find that the CIP is a comprehensive system of procedures that is well-suited for the identification and source attribution of Russian PGE-bearing materials.
2. The CIP is based upon accepted, reliable analytical methods.
3. The CIP protocols are well-documented and formatted.
4. The CIP has been verified by an independent laboratory.
5. We encourage the continued development of analytical protocols for the discrimination of PGE-bearing materials by implementation of our recommendations made in Chapter 10. The CIP is a potential starting point for development of methods by which consistent, comparable data can be obtained across the various producers.
6. Each producer, or perhaps PGM production region, should maintain a database of their PGE-bearing materials. Sharing of this information, for example through a body such as the IPA, would help considerably in forming expert opinions for forensic investigations.
7. Effective interpretation of CIP results within the context of legal casework requires specialist knowledge and an understanding of the mining and beneficiation of PGE-bearing materials. This is crucial in the identification of components in a mixture and in situations where the materials do not fully match the RDB because of undocumented variation.
8. Further research, in particular to characterise within-source variation, will enable formal methods for assigning probabilistic statements of uncertainty to identification and quantification.

12. Literature

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13. Appendices

Appendix 1. Terms and definitions

Appendix 2. The ENFSI letter of invitation

Appendix 3. Composition of the Board

Appendix 4. Industrial and scientific advisers to the Board

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

Appendix 6. On the use of geochemistry to characterise mineralisation of magmatic sulphide deposits from around the world

Appendix 7. Paper by Nikolay Kharkov entitled: “Technological processes’ peculiarities of mineral raw materials treatment used to substantiate the unique character of “Norilsk Nickel” products”

Appendix 8. Report by R.P. Schouwstra (2008) entitled Converter matte characterisation

Appendix 9. Protocols (CIP 0 - CIP 6)

Appendix 10. TNO report

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

Appendix 12. Report by Nikolay Kharkov on the 2003 SAR test entitled: “Report about analysis results of the production samples of the SAR mining metallurgical complex and decoding results of the check samples formed on the basis of production samples.”

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

Appendix 14. Letter from Geoffrey Otterman of Johnson Matthey to Nikolay Kharkov about the results of the 2003 JM test.

Appendix 15. Report by ICFSS on the “Blind” test of the CIP

Appendix 16. Report by AR on the “Blind” test of the CIP