

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

Anglo Research
Mineralogical Research Department

Converter matte characterisation
(A project for the CIP Forensic Review Board)

Report No: M/07/238

Reported by: R.P. Schouwstra

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Converter matte characterisation

IMPACT STATEMENT

Using a combination of techniques it is possible to differentiate between pure samples of normal converter mattes as supplied by the different producers participating in this study.

Ladle skulls, spillages, and reverts will have different characteristics, and will not have the characteristics features of the normal product.

Mixing mattes from different producers will complicate the identification process. It will not be possible to identify and quantify the different components in a mix using bulk chemical and bulk mineral contents. Textural and compositional studies using automated techniques on sectioned particle mounts will be essential for identification.

Although some of the distinguishing characteristics identified will persist in subsequent processing, one will need to use a combination of chemical and mineralogical techniques, as well as have a thorough understanding of the various processes, to try and identify the product as well as the producer.

Converter matte characterisation

Synopsis

Partially processed PGE-rich materials from Russian producers are stolen on a large scale and illegally supplied to specialized PGE refineries worldwide for further processing. Similar problems occur in South Africa.

Russian law enforcement agencies have initiated legal actions in Western Europe against companies suspected of dealing in stolen PGE materials from Norilsk. As part of the legal proceedings it is important to identify and prove that the recovered PGE-bearing materials originated from Norilsk. For this purpose Norilsk and the Russian Institute of Criminalistics (Federal Security Service) developed a procedure for the characterization of PGE materials (Complex Identification Method or CIP).

In order for the CIP results and expert opinions derived there from to be accepted in future court proceedings, it was thought necessary to have the method validated analytically and forensically by an independent international body. An international forensic project was initiated to this end under the auspices of ENFSI (European Network of Forensic Science Institutes). This CIP project is supported by the Ministry of Justice of the Russian Federation and the IPA (International Platinum Association).

R. Schouwstra was invited to serve as a scientific adviser to assist and advise the Forensic Review Board by providing comments on the Russian methodology, prepared documents and (interim) reports and by answering technical questions posed by the board members.

One of the early hypothesis that was investigated as part of the CIP validation project was the use of ore deposit characteristics to prove the provenance of recovered material. However, it was recognized that the wide spread in chemistry and mineralogy on some of the ore types, as well as the lack of information on others would make this an impossible task. It was for this reason that Anglo Platinum sponsored a supplementary project on the characterization of converter mattes.

The primary objective of this supplementary project was to determine whether converter mattes from different producers of PGE are sufficiently dissimilar to enable discrimination using a variety of chemical and mineralogical techniques.

It was also postulated that 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, possibly further aiding in determining the provenance of recovered material other than converter mattes.

Thirdly, in South Africa, smelter products are most at risk of being stolen, and additional information on converter mattes could assist South African producers to identify recovered material.

This report summarises the results of this study, and was submitted to the Forensic Review Board for incorporation in their final report.

Key words: Converter matte, PSD, RD, chemistry, mineralogy.

Converter matte characterisation

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Converter matte characterisation

1 INTRODUCTION

One of the early hypothesis that was investigated as part of the CIP validation project was the use of ore deposit characteristics to prove the provenance of recovered material. However, it was soon recognized that the variability in chemical element distribution within the same mining area is too wide, resulting in overlaps between different mining areas. To complicate matters further, different mining companies mine the same reef types. For example, different producers operating on the Bushveld complex in South Africa mine both the Merensky and UG-2 reefs. This makes it impossible to link a recovered process product to a specific producer based on ore type characteristics.

Although it might also be possible to trace back the origin of recovered material 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 third option is to characterise a product within the beneficiation process that still retains most of the chemical characteristics of the ore types treated (e.g. Pt to Pd ratios, Cu and Ni contents). This also has the advantage that the product might acquire additional differentiating characteristics imposed by the specific process route used by individual producers. The majority of producers follow a pyrometallurgical route with converter matte forming the feed to the subsequent hydrometallurgical process. With converter matte thus as a common denominator and an averaging factor in the process (generally the feed to the smelter is a blend of ore types) a project was initiated to determine whether converter mattes from different producers of platinum group metals are sufficiently dissimilar to enable discrimination using a variety of chemical and mineralogical techniques. If this is indeed possible, 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.

Another reason for choosing converter matte is the value of this material. In South Africa, smelter products are most at risk of being stolen, with the feed to the smelter of too low a grade to warrant further processing.

Converter matte characterisation

To test the above hypothesis the following producers were approached to participate in the project:

Bushveld Complex (South Africa)	<i>Anglo Platinum</i>	AP
	<i>Impala Platinum</i>	IP
	<i>Lonmin Platinum</i>	LP
	<i>Northam Platinum</i>	NP
Great Dyke (Zimbabwe)	<i>Zimplats (Impala Platinum)</i>	ZP
Sudbury Complex (Canada)	<i>Vale Inco</i>	
	<i>Xstrata Canada Corp. (Falconbridge)</i>	FB
Stillwater Complex (USA)	<i>Stillwater (Norilsk Nickel)</i>	SW
Norilsk (Russia)	<i>Norilsk Nickel</i>	NN
Pechenga (Russia)	<i>Norilsk Nickel</i>	P
Jin Chuan (China)	<i>Jin Chuan</i>	
Bindura (Zimbabwe)	<i>Bindura</i>	B
Selibi Pikwe (Botswana)	<i>Selibi Pikwe</i>	

Northam and Bindura both submitted two converter mattes. The one Northam matte (NP a) represents normal production, whereas the second sample (NP b) was taken during a period when a significant amount of another ore type was treated through the Northam converter. Bindura submitted a nickel-rich desulphurised matte (BSR – normal product) and a copper-nickel desulphurised matte (BSL – containing reverts). Vale Inco and Selibi Pikwe declined participation, whereas requests to Jin Chuan went unanswered.

Permission has been obtained from the participating producers to incorporate this information in the final report of the CIP Forensic Review Board.

2 METHODOLOGY

Participating companies were asked to submit 0.5 kg of unpulverised converter matte for chemical and mineralogical characterisation. Since the majority of producers granulate their matte for subsequent processing, most of the material received was in granulated form. Anglo Platinum, Norilsk and Pechenga slow-cool their converter matte to form ingots and these producers submitted material in crushed form, with lumps approximately 3 to 5 cm in size.

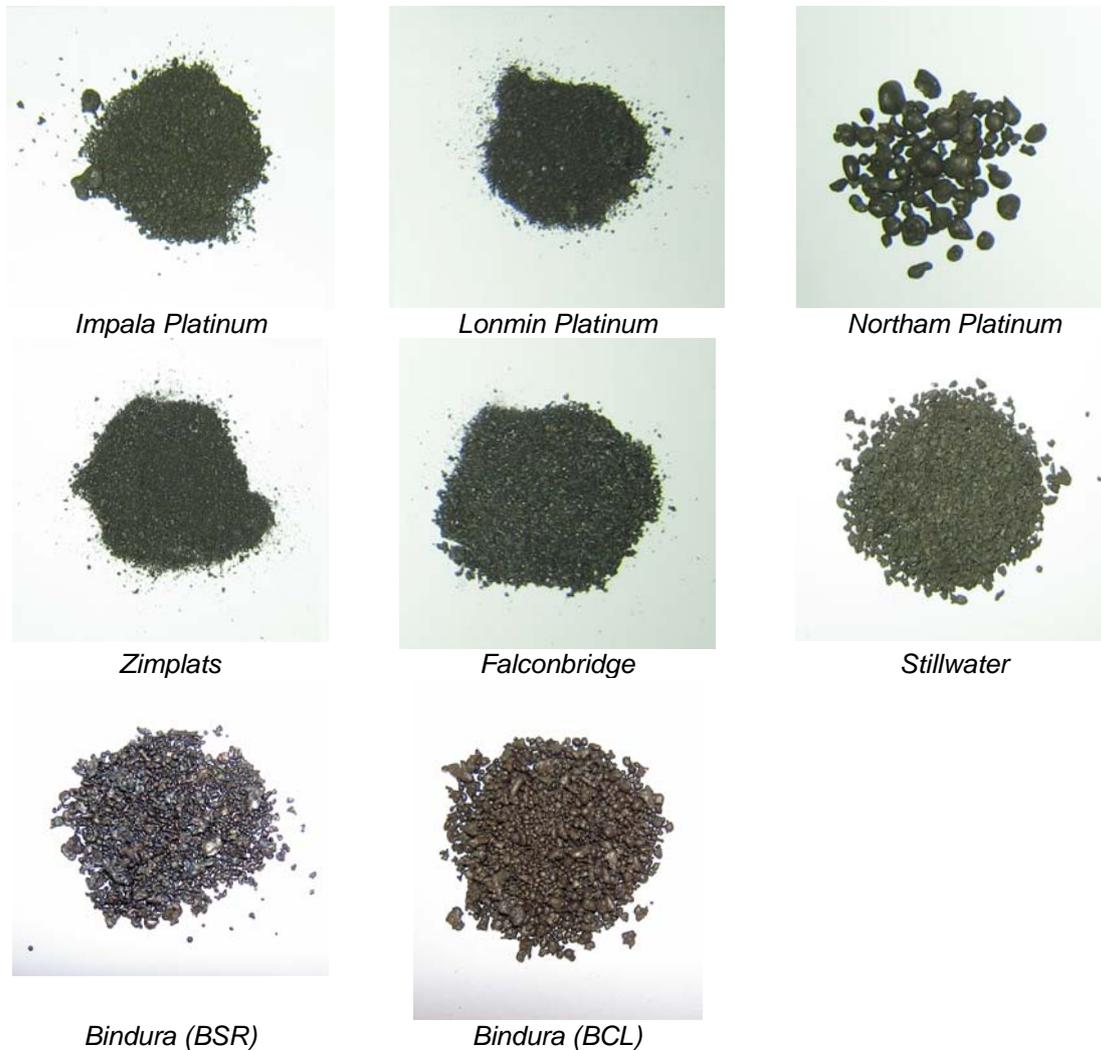
The particle size distribution of the granulated converter matte was determined by screening. The lumps of slow-cooled converter matte were crushed to -3 mm. Representative aliquots of the granulated converter matte and the -3 mm slow-cooled converter matte were pulverised and analysed for various major, minor and trace elements. Similarly, representative portions were used to determine the relative density of the various mattes. X-ray diffractometry on pulverised material was used to identify the phases present in the various samples.

Representative aliquots of granulated converter matte as well as the -3 mm slow-cooled converter matte were mounted in resin, and ground and polished to section the matte particles for detailed characterisation using optical microscopy, bulk modal analysis by QEMSCAN and phase specific analysis by SEM-EDX.

3 RESULTS

3.1 Macro-physical characteristics

Figure 1 highlights the difference in appearance between granulated and crushed slow-cooled matte, whereas Figure 2 exhibits the particle size distribution of the various mattes studied. Note that the Northam Platinum matte is much coarser than the other granulated mattes, which have a much narrower size distribution.



Converter matte characterisation



Figure 1: Photographs showing the appearance of the granulated and crushed converter mattes received. Note that the granulated converter mattes look quite similar, apart from the Northam converter matte which is much coarser (both the Northam a and b samples are very similar). The slow-cooled converter mattes also have a similar appearance. All photographs approximately at the same scale.

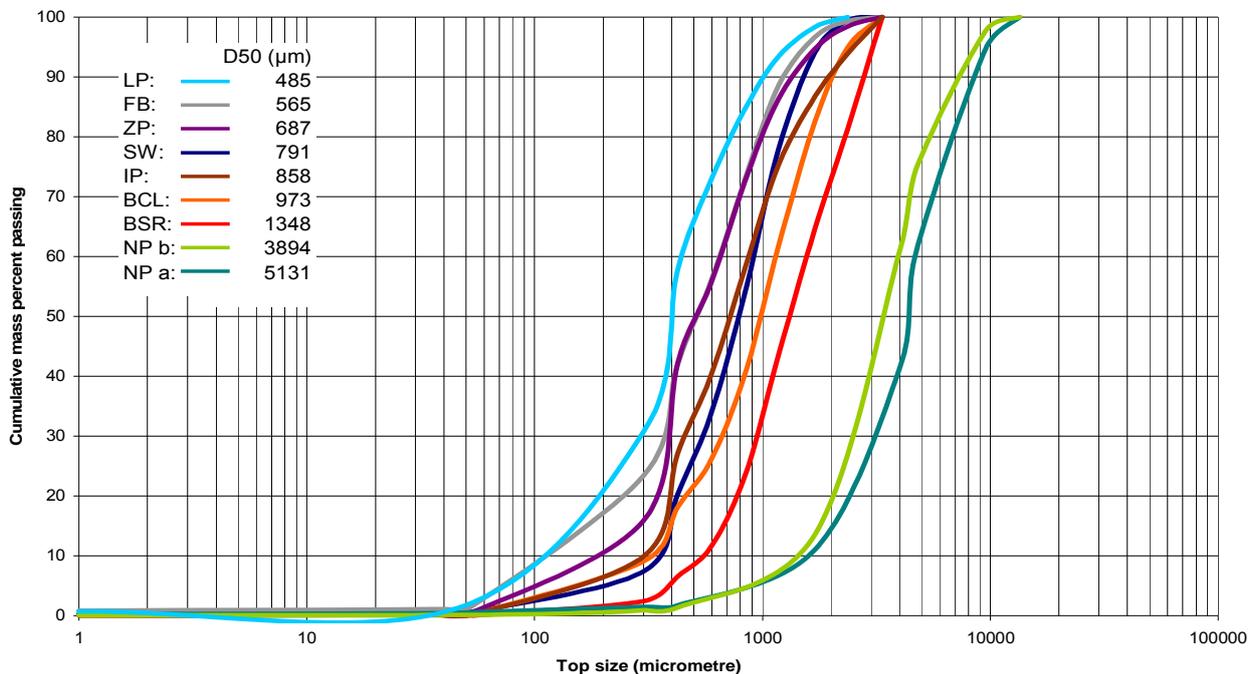


Figure 2: Particle size distribution of the granulated converter mattes (note the much coarser size distribution of the two Northam granulated converter mattes).

Table 1 presents the relative densities of the converter mattes studied. Note that the Bindura mattes exhibit the highest relative densities. However, most of the mattes exhibit relative density values between 6.1 and 6.6.

Table 1: Relative densities of the various converter mattes received – as determined by Grabner Minidens instrument.

AP	IP	LP	NP a	NP b	ZP	SW	FB	NN	P	BSR	BCL
6.2	6.6	6.3	6.1	6.1	6.4	6.6	6.6	6.3	6.1	8.4	8.6

3.2 Chemical characteristics

Table 2 summarises the chemical data for the various converter mattes. Although overall more than 30 elements were analysed, only the most prominent ones are presented in this table. The most characteristic features are the high PGE contents of the primary PGE-producers (Anglo Platinum to Stillwater) compared to those operations that produce PGE as a by-product, and the difference in platinum to palladium ratios of the southern African primary PGE-producers (Anglo Platinum to Zimplats) vs. the others (Stillwater to Bindura).

Compared to the South African PGE-producers, the following characteristics are evident:

A high PGE, Se, Te and Pb concentration for Stillwater.

A high Ni to Cu ratio, and high Co, Sn and Bi values and a low Te concentration for Falconbridge.

A low Ni to Cu ratio and a low Te concentration for Pechenga.

A low Te concentration for Norilsk.

The Bindura converter mattes are characterised by different but very distinctive Ni to Cu ratios, low Te contents and, for the BSR product, an abnormally high As content.

Zimplats is characterised by increased Bi levels.

Table 2: Chemical composition (selected elements only) of the various converter matte samples investigated.

	AP	IP	LP	NP a	NP b	ZP	SW	FB	NN	P	BSR	BCL
Cu	27.8	29.4	31.9	25.5	26.6	30.8	29.6	20.8	26.8	35.3	17.3	41.9
Fe	2.7	0.7	0.5	2.2	1.7	1.0	2.5	1.7	2.4	3.0	0.5	0.9
Ni	45.6	46.8	49.1	49.5	49.2	44.3	43.9	53.0	46.2	35.3	69.8	48.2
Co	0.3	0.3	0.2	0.7	0.6	0.6	0.5	1.7	1.0	0.7	0.7	0.1
S	22.1	20.1	17.7	20.5	20.0	21.5	20.0	20.6	21.7	22.2	8.6	6.4
Pt	1931	2280	2740	2040	2013	734	6930	20	25	3	9	14
Pd	1164	1268	1207	1007	946	593	12000	96	153	11	30	55
Rh	301	325	384	265	238	69	563	2	15	2	3	4
Ru	501	451	628	394	358	58	130	2	4	2	6	2
Ir	95	106	154	88	80	28	13	1	1	1	1	1
Os	44	53	77	43	40	4	5	2	5	5	1	1
Au	80	77	78	64	83	82	171	9	4	2	2	4
Ag	226	149	107	73	84	171	125	44	128	135	54	129
Se	647	613	612	508	507	563	820	335	175	399	545	481
Te	262	178	171	142	137	300	578	69	23	28	78	76
Sn	9	3	3	2	2	2	24	204	9	4	4	22
Bi	64	42	22	12	16	142	47	129	12	3	12	16
As	348	248	411	81	102	105	447	341	39	325	5630	159
Sb	34	39	68	12	13	4	34	34	5	7	86	6
Pb	503	332	179	509	556	121	2300	467	135	60	29	32

PGE and trace elements by ICP-MS, base metal analysis by BMfuse ICP, S by Leco.

3.3 Mineralogical characteristics

3.3.1 X-ray diffractometry (XRD)

Figures 3 to 5 compare the XRD patterns of the granulated mattes, slow-cooled and granulated Bindura mattes. All converter mattes studied comprise a matrix of nickel sulphide (heazlewoodite – Ni_3S_2), copper sulphide (djurleite or chalcocite – $\text{Cu}_{1.96}\text{S}$ or Cu_2S) and, if the matte contains significant amounts of iron, also copper-iron sulphide (bornite – Cu_5FeS_4). The base metal alloy phase (Ni,Cu-Fe-alloy) is similar in structure to awaruite. The following differences were noticed in the diffraction patterns of the mattes studied:

Amongst the granulated converter mattes the Stillwater (SW) matte shows a shift in the alloy peak position.

Amongst the primary PGE-producers Lonmin (LP) has the highest and Zimplats (ZP) the lowest alloy content.

Compared to the granulated converter matte from primary PGE-producers, the Bindura mattes (BSR and BCL) can be distinguished by their very high nickel-alloy content. The alloy peak positions also do not coincide with those of the primary PGE-producers.

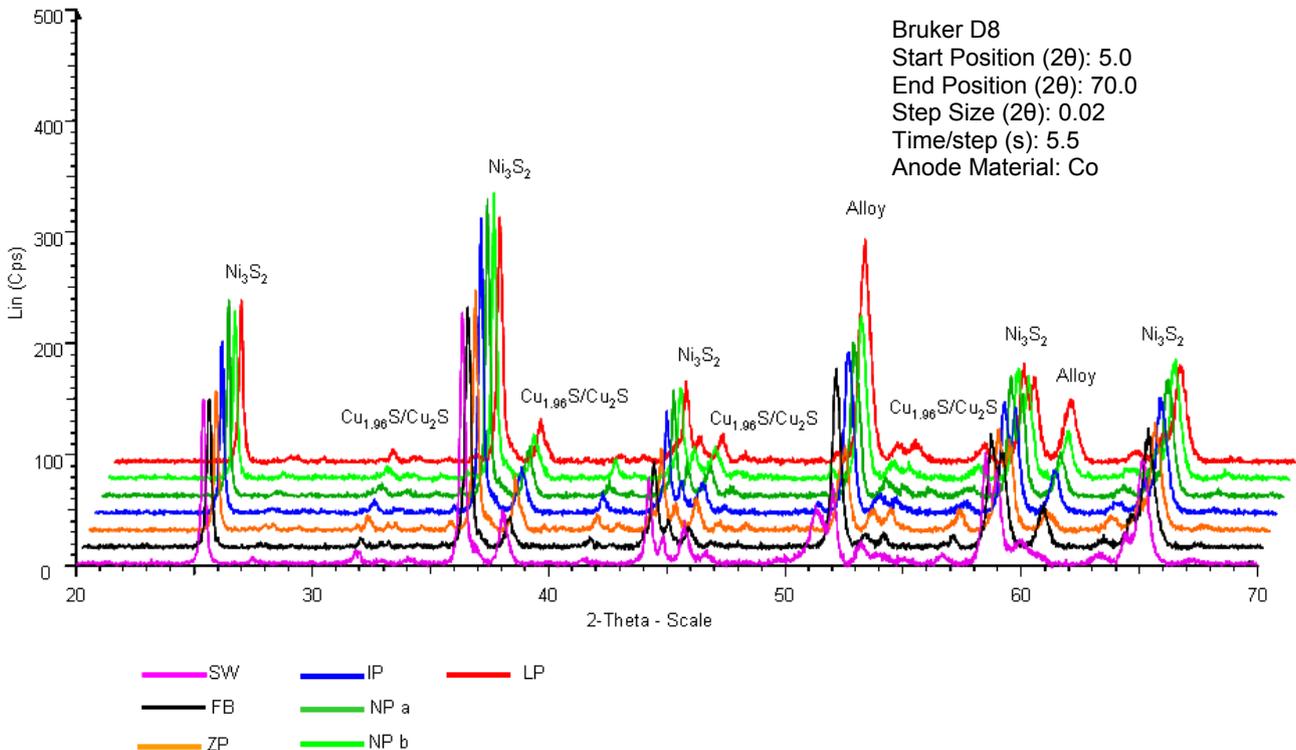


Figure 3: XRD patterns - Granulated converter mattes (annotation highlights the most important peak positions only).

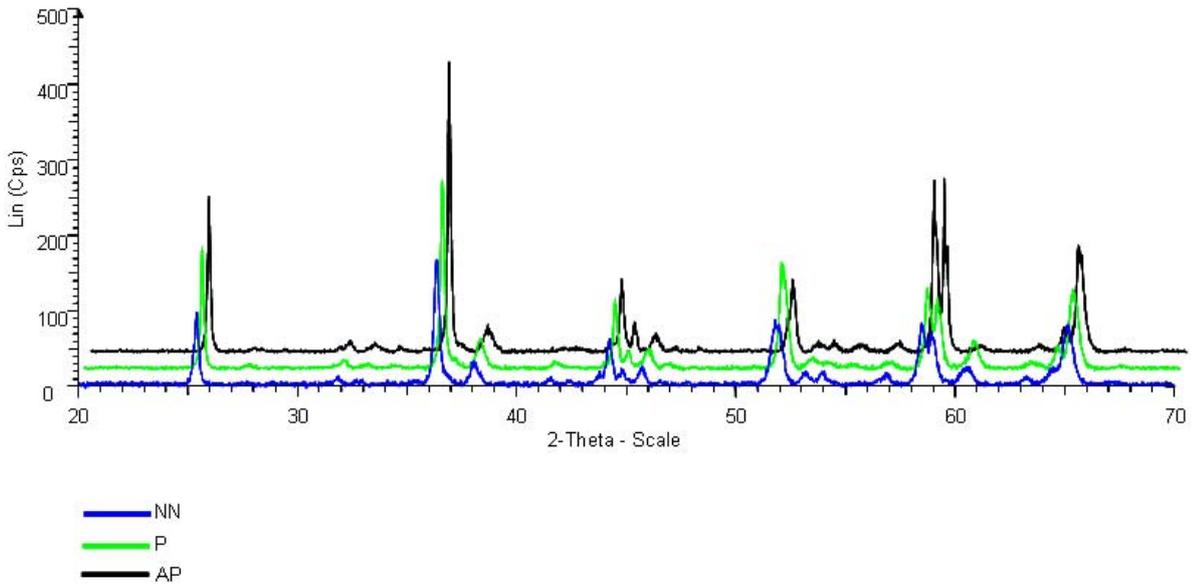


Figure 4: XRD patterns – Slow-cooled converter mattes (annotations and conditions as for Figure 3).

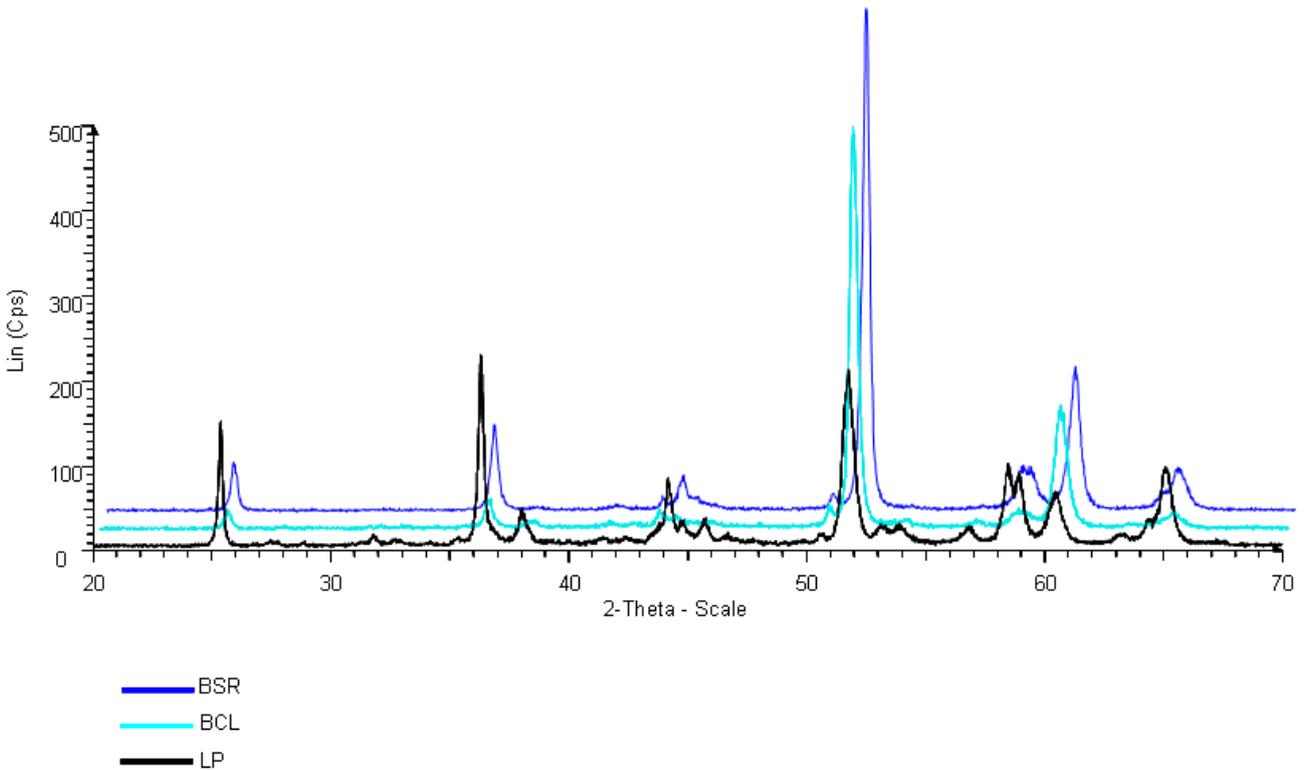


Figure 5: XRD patterns – high alloy Bindura converter mattes compared to a typical granulated converter matte from a primary PGE-producer (annotations and conditions as for Figure 3).

3.3.2 Bulk modal composition

The detailed bulk and phase compositions of the various converter mattes were determined using a combination of QEMSCAN and SEM-EDX techniques (Table 3). As already highlighted in the XRD study, the converter mattes contain similar phases, but at different abundances. The most characteristic features are the differences in alloy

content, with the Lonmin (LP) converter matte having the highest alloy content of the primary PGE-producers from southern Africa, and Bindura (BSR and BCL) exhibiting the highest overall alloy contents.

Table 3: Bulk modal composition of the various converter mattes – determined using QEMSCAN (accelerating voltage 25 kV, specimen current 5 nA, running at 200000 counts per second on the Au standard).

	AP	IP	LP	NP a	NP b	ZP	SW	FB	NN	P	BSR	BCL
Nickel sulphide	56.3	49.4	45.9	50.8	49.9	55.4	47.9	53.0	56.7	45.3	22.0	15.2
Cu(Fe)-sulphides	32.9	29.0	27.0	30.0	29.3	32.6	28.2	31.1	31.7	46.6	13.0	9.0
Alloy	8.9	20.9	25.9	18.5	19.1	10.6	22.6	15.4	9.1	6.1	64.2	75.0
Other	1.9	0.7	1.2	0.7	1.7	1.4	1.3	0.5	2.5	2.0	0.8	0.8

Figure 6 highlights the textural differences between the various converter mattes as photographed in section.

3.3.3 Phase composition

Semi-quantitative SEM-EDX analysis of the various phases comprising the converter mattes indicates that the copper sulphides, copper-iron sulphides (where present) and the nickel sulphides are similar in composition.

However, the alloy phases collect most of the PGE and are characterised by varying PGE contents and different copper to nickel ratios, which is likely to be a function of the overall matte composition. For primary PGE-producers Pt-alloys occur associated with base metal alloy (frequently forming the core to base metal alloy phases). The Stillwater alloy is characterised by a high Pd concentration.

Table 4 summarises the composition of the various alloy phases. In the case of the slow-cooled converter mattes, the alloy composition refers to the base metal alloy rim and not the cores of the base metal phase which are generally higher in PGE. Table 4 reports the base metals only. The small grain size of the base metal alloy in granulated mattes impacted on the quality of the analyses, and significant amounts of background sulphur and possibly nickel were detected.

Converter matte characterisation

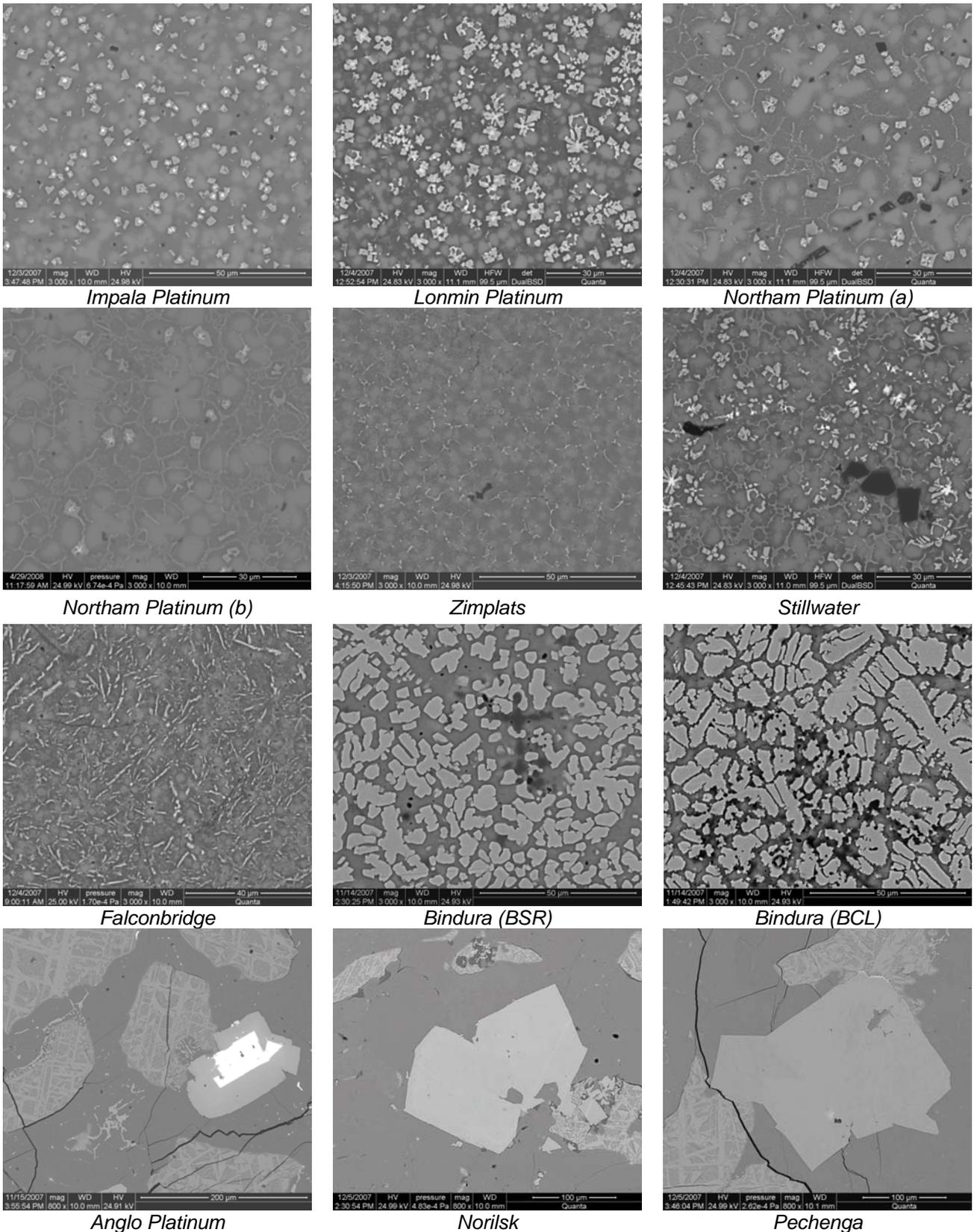


Figure 6: Backscattered electron images highlighting the differences in alloy shapes and alloy size distribution for the various converter mattes. The brighter phases are alloy set in a darker sulphide matrix. Note that the alloy from different producers exhibit different shapes (square, star-shaped or worm-like). White specks in the granulated converter mattes are PGE-rich alloys. The images of the Bindura matte highlight the abundance of alloy. The alloy in the slow-cooled converter mattes occur as larger plates. The white core to the base metal alloy phase in the slow-cooled Anglo Platinum converter matte is Pt-rich.

Converter matte characterisation

Table 4: Composition of the major alloy phase in the various converter mattes determined using SEM-EDX (a minimum of 30 points for each sample investigated, accelerating voltage 20kV, 70 000 counts per analysis).

	AP	IP	LP	NP a	NP b	ZP	SW	FB	NN	P	BSR	BCL
Ni	78.7	66.9	66.2	71.4	72.8	65.4	53.0	69.4	78.7	79.1	83.1	53.7
Fe	12.4	0.4	0.4	5.9	3.7	1.4	0.1	1.4	12.4	10.0	0.1	0.3
Cu	8.0	28.2	31.5	13.5	18.9	19.4	35.6	13.7	8.0	10.0	15.9	45.6
Co	0.9	0.5	0.5	1.5	1.4	1.1	0.4	2.1	0.9	0.9	0.9	0.4

Comparing the alloy compositions the following distinguishing features are evident:

The base metal alloy of the slow-cooled converter mattes (AP, NN and P) is similar in composition.

The Impala (IP) and Lonmin Platinum (LP) base metal alloys are similar in composition, but both are distinctly different from the other primary PGE-producers.

The Northam (NP a and b) base metal alloy exhibits higher iron contents than the other granulated converter mattes.

The Stillwater (SW) alloy phase is characterised by high copper and lower nickel contents compared to that of the other primary PGE-producers. It is also characterised by a higher Pd content.

Both Bindura mattes (BSR and BCL) have alloys with distinctive (although very different) nickel to copper ratios.

One of the minor phases occurring in converter matte is spinel (mainly Fe,Ni-oxide), which forms during the converting process and reports in small amounts to the converter matte. These spinels highlight the blowing conditions during converting and if present can possibly be used as a distinguishing characteristic. In the samples investigated, the Lonmin spinel exhibited the highest Ni-content. Although the Stillwater spinel also has high nickel contents, the Stillwater mattes contained four different types of spinel, including a CaFe-oxide. This wide variation in spinel composition has not been observed in any of the other converter mattes.

Table 5: Composition of the major spinel phase present in the various converter mattes determined using SEM-EDX (a minimum of 30 points for each sample investigated, accelerating voltage 20kV, 70 000 counts per analysis).

	AP	IP	LP	NP a	NP b	ZP	SW	FB	NN	P	BSR	BCL
NiO	9.6	18.5	78.0	7.3	10.4	28.0	71.1	6.7	4.0	7.1	76.8	75.8
CuO	0.9	0.8	0.8	1.3	3.1	4.1	4.6	0.9	0.9	2.3	1.8	3.5
FeO	87.6	78.3	16.3	85.7	83.1	59.9	16.5	89.1	90.2	87.0	13.5	18.2
CoO	1.1	0.7	3.7	1.7	1.6	2.8	5.3	2.4	1.6	1.2	6.9	1.5

4 SUMMARY

Although superficially the granulated converter mattes look similar (as do the slow-cooled converter mattes), it is possible to differentiate between pure converter matte from the various producers using a combination of techniques.

The following are the most characteristics 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 further differentiate 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 different Ni to Cu ratios for Pechenga 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 spinel, have different compositions, and can be used to further differentiate between mattes from various producers.

5 CONCLUSIONS

Using a combination of techniques it is possible to differentiate between pure samples of normal converter mattes as supplied by the different producers participating in this study (Figure 7).

The following aspects need to be taken into account:

1. The study is based on 1 or 2 samples from each producer. Variation and changes in the ore type treated, as well as the process used, will impact on the characteristics of the converter mattes. It is recommended that producer data is acquired on a regular basis.
2. This study was performed on final converter matte products. Ladle skulls, spillages, and reverts will have different characteristics, and will not have the characteristics features of the normal product.
3. Mixing mattes from different producers will complicate the identification process. It will not be possible to identify and quantify the different components in a mix using bulk chemical and bulk mineral contents. Textural and compositional studies using automated techniques on sectioned particle mounts will be essential for identification.
4. Although some of the distinguishing characteristics identified will persist in subsequent processing, one will need to use a combination of chemical and mineralogical techniques, as well have a thorough understanding of the various processes to try and identify the product as well as the producer.

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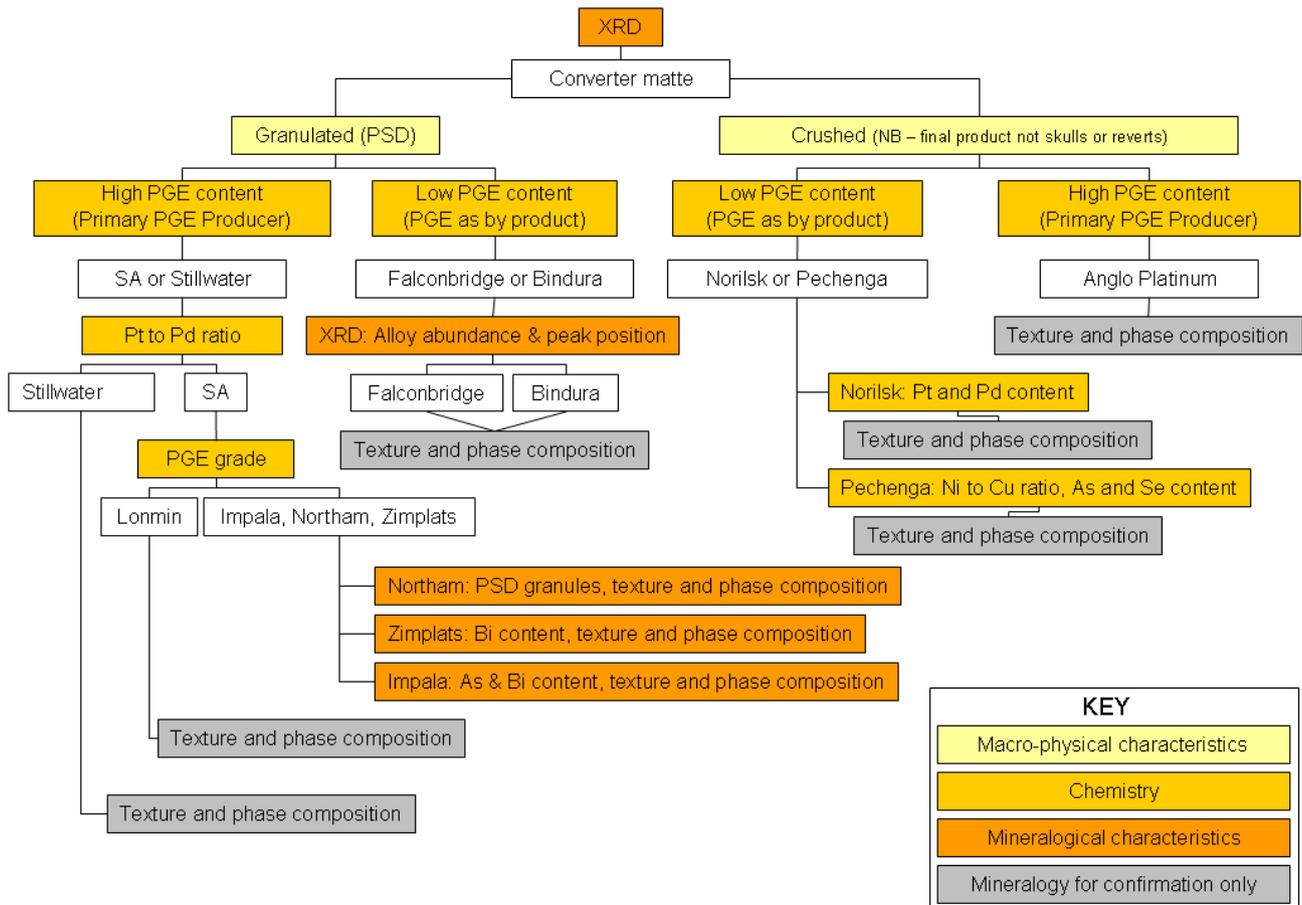


Figure 7: Diagram highlighting the information needed to identify converter mattes from various producers.