

Platinum Group Metals

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The platinum group metals (PGMs) consist of six elements: platinum, palladium, rhodium, ruthenium, iridium, and osmium, which are used as jewelry, catalysts, and stores of wealth. These elements are all located in the same region of the periodic table. Consequently, they all exhibit similar chemistries, which explains why they are found together in the earth's crust, and why their production and refining pipeline is much longer than that of other metals. The uses, mining sources, market supply, primary extraction, and recycling of these metals are described in this chapter.

USES OF PLATINUM GROUP METALS

PGMs are used in jewelry, coinage, and investment bars, and as powerful catalysts for industrial reactions. Over the long term, the requirement for lower emissions from cars and trucks has driven an increase in demand for these metals. In the United States, legislation was first introduced in 1975 requiring light vehicles to be fitted with autocatalysts. Other countries followed this lead, and it is estimated that at least 95% of all new vehicles are fitted with autocatalysts. Other uses of the PGMs are given in Table 1.

SOURCES AND SUPPLY

Production

PGMs are produced from primary sources in only five countries of the world, as shown in Table 2. Most platinum is produced in South Africa, while most palladium is produced in Russia. The annual global production of platinum, palladium, and rhodium are shown in Figures 1 and 2. The global demand for iridium and ruthenium is shown in Figure 3.

Occurrence

The economically viable sources of platinum are concentrated in South Africa and Russia. The platinum-bearing ores of South Africa occur in a massive igneous intrusion referred to as the Bushveld Igneous Complex. The main mining region in Russia is the area around Norilsk and Talnakh, which produces large quantities of nickel and copper in addition to PGMs. Small amounts of PGMs are produced from alluvial deposits at Kondyor and Koryak in the Urals. PGMs are also mined at

Table 1 Demand by application for 2013*

Uses	Platinum (Pt)	Palladium (Pd)	Rhodium (Rh)	Iridium (Ir)	Ruthenium (Ru)
Autocatalyst	3,125	6,970	801	—†	—
Chemical	540	530	79	20	104
Dental	—	510	—	—	—
Electrical	205	1,055	7	36	531
Electrochemical	—	—	—	59	125
Glass	235	0	40	—	—
Investment	765	75	—	—	—
Jeweler	2,740	390	—	—	—
Medical and biomedical	235	—	—	—	—
Petroleum	155	—	—	—	—
Other	420	100	89	83	68
Total demand	8,420	9,630	1,016	198	828

Adapted from Johnson Matthey 2003–2015b

*Units per 1,000 troy ounces.

†A dash indicates minimal usage in that field or usage is unquantified.

Table 2 Supply of platinum group metals by country for 2013*

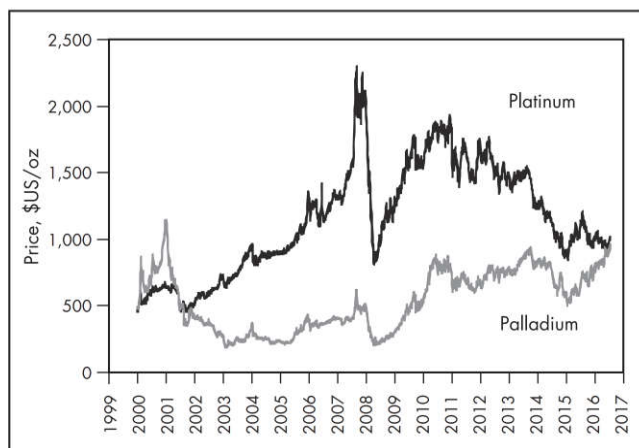
Country	Platinum (Pt)	Palladium (Pd)	Rhodium (Rh)
South Africa	4,120	2,350	574
Russia	780	2,700	85
Canada	209	541	24
United States	106	389	—
Zimbabwe	400	310	33
Others	125	140	5
Total supply	1,620	4,080	147

Adapted from Johnson Matthey 2003–2015b

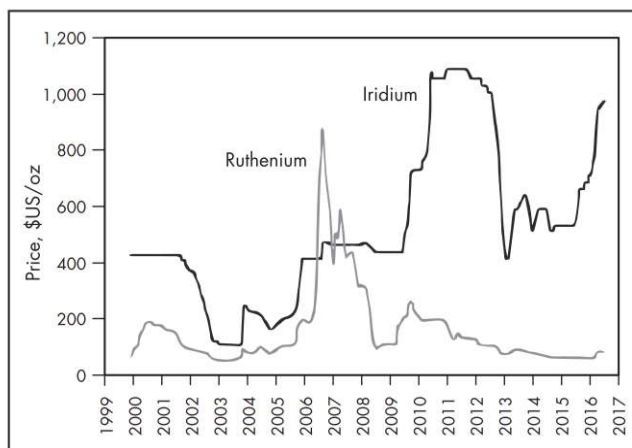
*Units per 1,000 troy ounces.

Stillwater and East Boulder, in Montana, United States, and from the Great Dyke area of Zimbabwe.

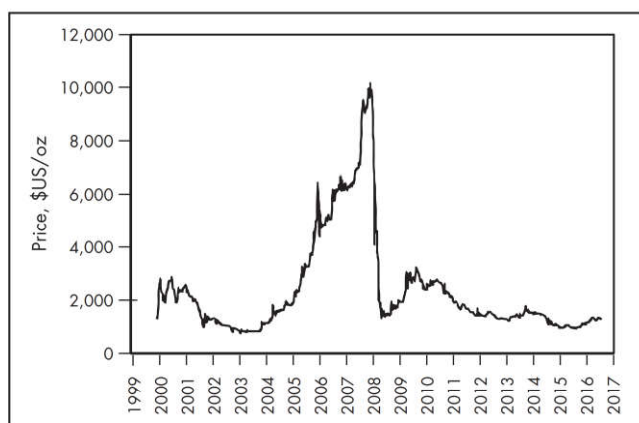
A significant amount of PGMs are produced as by-products of nickel production. All the Canadian production and the



Adapted from Johnson Matthey 2003–2015a

Figure 1 Global production of platinum and palladium

Adapted from Johnson Matthey 2003–2015a

Figure 3 Global demand for iridium and ruthenium

Adapted from Johnson Matthey 2003–2015a

Figure 2 Global production of rhodium

production from countries that toll refine nickel-bearing ores, such as Norway, falls into this category. The locations of primary mining, smelting, and refining facilities are given in Table 3 and Figure 4.

PRICES AND MARKETING

The prices of PGMs, from January 1, 2004, to August 20, 2015, are shown in Figures 5 to 7. Prices dropped dramatically during the global credit crisis of August 2008, recovered over the following two years, and have been in decline since 2011.

PRIMARY EXTRACTION

The primary extraction of PGMs from the Bushveld Igneous Complex in South Africa is the most complex with the longest processing pipeline of all metals. The mined ore is milled and the sulfide fraction is removed by flotation. This sulfide concentrate is smelted to remove most of the silicate material, and the resulting matte is converted to reduce the sulfur and iron content. The converter matte can be treated in a base metals refinery, or it can be treated by slow cooling and magnetic separation. After either the base metals refinery or the magnetic separation plant, the platinum-containing fraction is then transferred to the precious metals refinery, where each of the metals is separated into their pure forms.

Table 3 Location of mining, smelter, and refining facilities for the production of platinum group metals

	Location	Mine	Concentrator	Smelter	Base Metals Refinery	Precious Metals Refinery
1	Bushveld Igneous Complex, South Africa	~20	20	6	4	3
2	Sudbury Basin, Canada	~10	2	2	1	*
3	Norilsk/Talnakh, Russia	7	2	3	1	*
4	Stillwater, United States	2	1	1	1	*
5	Lac des Iles, Canada	1	1	*	*	*
6	Selous, Zimbabwe	1	1	1	*	*
7	Kola Peninsula, Russia	1	1	1	1	*
8	Kondyor, Russia	1	*	*	*	*
9	Koryak, Russia	1	*	*	*	*
10	Port Elizabeth, South Africa	*	*	*	*	1
11	Hanau, Germany	*	*	1	1	1
12	Hoboken, Belgium	*	*	1	1	1
13	Acton, England	*	*	*	*	1
14	Royston, England	*	*	*	*	1
15	Kristiansand, Norway	*	*	*	1	1
16	Prioksk, Russia	*	*	*	*	1
17	Yekaterinburg, Russia	*	*	*	*	1
18	Novosibirsk, Russia	*	*	*	*	1
19	Krasnoyarsk, Russia	*	*	*	*	1
20	Niihama, Japan	*	*	*	*	1

* Indicates that no production of that type is undertaken at that site.

PGMs are extracted from Southern African sources primarily for their precious metals values, even though nickel and copper are produced as by-products. However, PGMs are produced from Russian sources as a by-product of nickel

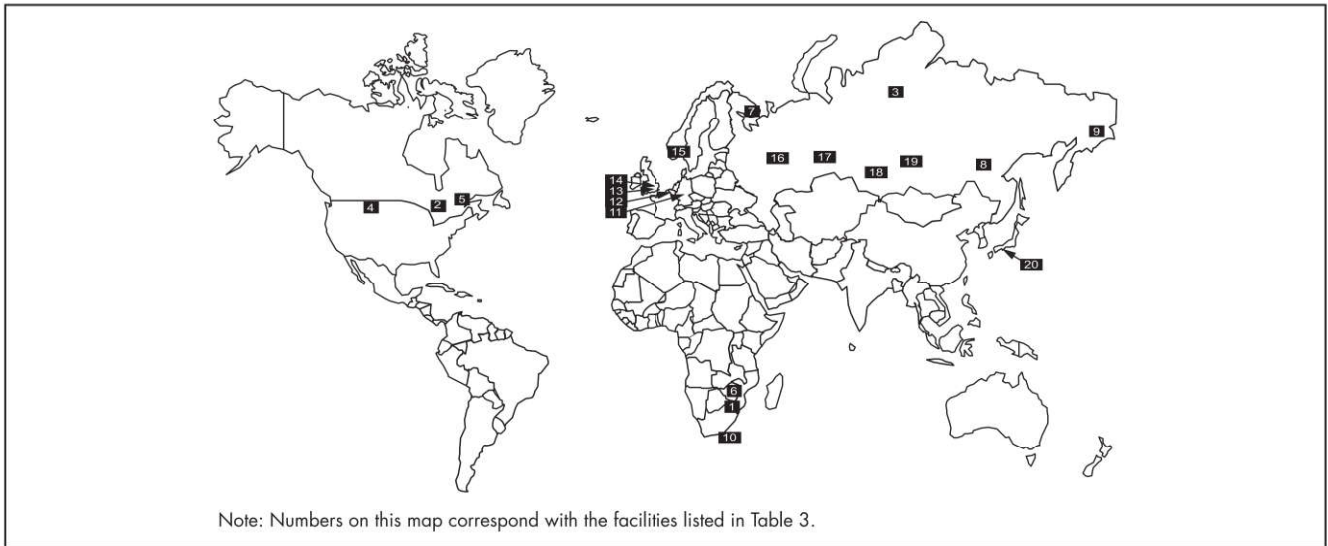
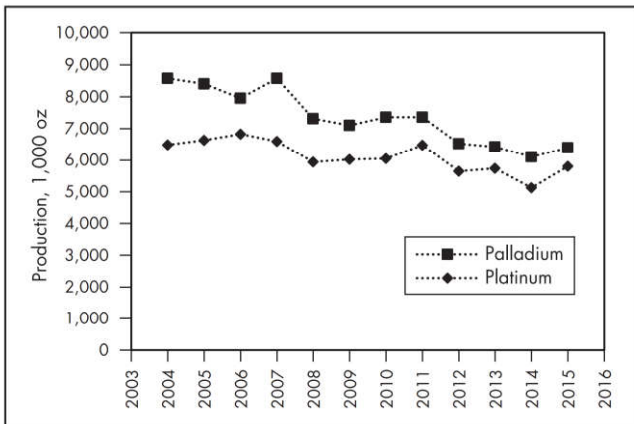
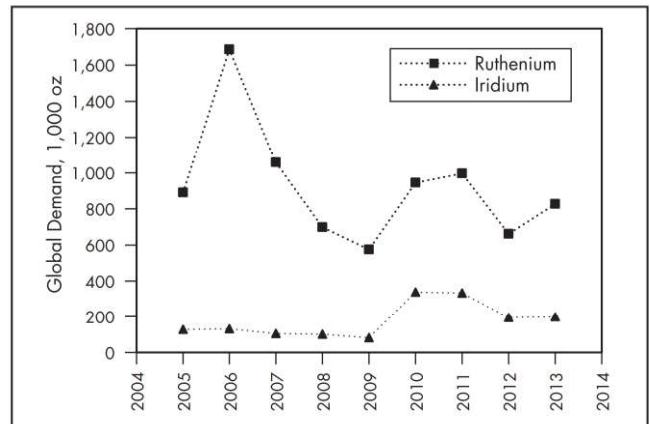


Figure 4 Locations of the major platinum group metals mines, smelters, concentrators, and refineries around the world



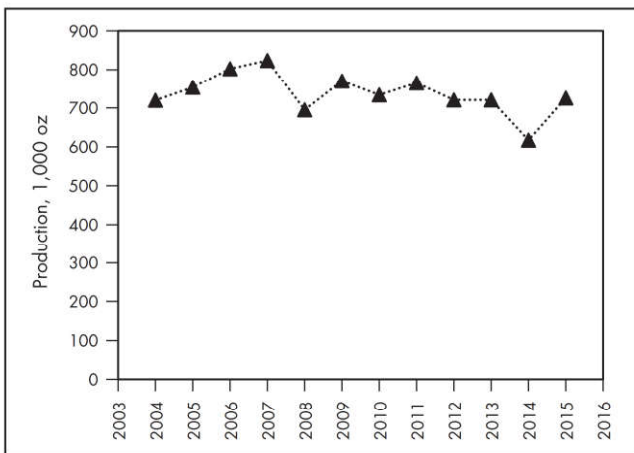
Adapted from Johnson Matthey 2003–2015a

Figure 5 Daily price for platinum and palladium from 2004 to 2015



Adapted from Johnson Matthey 2003–2015a

Figure 7 Prices of iridium and ruthenium for 2005 to 2013



Adapted from Johnson Matthey 2003–2015a

Figure 6 Price of rhodium from 2004 to 2015

production. Thus, a flotation concentrate is produced from the mined ore, which is smelted; the matte is cast into anodes used in electrowinning to produce nickel metal. The insoluble precious metals report to the anode slime, which is refined in the precious metals refinery.

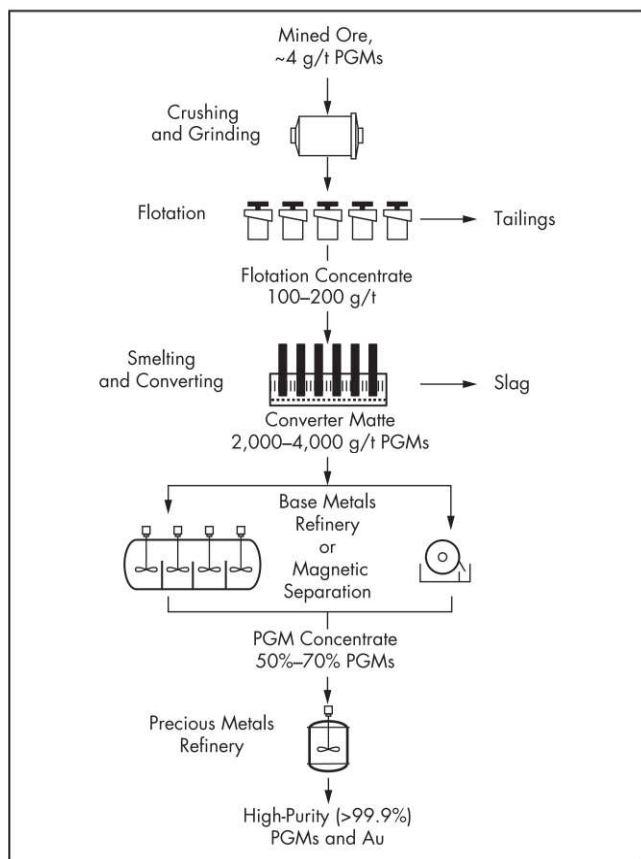
PGM extraction from both South African and Russian sources is discussed in further detail in the following sections.

Primary Extraction from South African Ores

The overall process for the extraction of PGMs from South African sources is shown in Figure 8. The mined ore typically contains between 1 and 10 g/t of PGMs in either metallic form or as sulfides or tellurides. These PGMs are associated with the sulfide of copper and nickel.

Flotation

The mined ore is ground and floated to produce a nickel-copper concentrate. The PGM concentration is between 100 and 200 g/t (Cramer 2008), which means that the concentration has been upgraded by a factor of approximately 20. However,



Source: Crundwell et al. 2011

Figure 8 Overview of the processing of PGMs from South African ores, showing the major steps of flotation, smelting, base metal refining, and precious metal refining

the recovery of PGMs is typically between 75% and 85%, which means that this step represents the biggest loss of value in the process.

The ores of the Merensky Reef have been typically treated by conventional sulfide flotation, in a rougher, cleaner, and re-cleaner circuit configuration. The rougher tailings are discarded, while the cleaner tailings are recycled to the rougher feed. The conventional flow sheet is given in Figure 9.

The ores of the Upper Group 2 Reef are generally more challenging, because these ores have a higher content of chromite (between 20% and 60% chromium oxide [Cr_2O_3]), which is deleterious in the downstream smelting process. Therefore, reducing the content of chromite in the flotation product to less than 3% is important. The main mechanism by which chromite reports to the flotation concentrate is by entrainment of fine particles of chromite between rising bubbles (rather than by true flotation) (Knights and Bryson 2009). The effective control of grinding is critical in reducing such entrainment. In addition, the circuit is configured in an MF2 (mill-float-mill-float) configuration, shown in Figure 10, in which the tailings from the rougher stages are reground and refloats. The objective is to achieve a relatively coarse grind (35% passing 75 μm) in the first milling stage, which reduces the chromite entrainment while managing to recover the fast-floating fraction. The rougher tailings are reground and refloats to recover the slow-floating fraction.

Because the density of the platinum minerals is higher than that of the host rock, separation techniques based on density can be effective. A gravity concentrate can be produced by cycloning the primary mill fines. This concentrate can be of a sufficiently high concentration to be sent directly to the precious metals refinery.

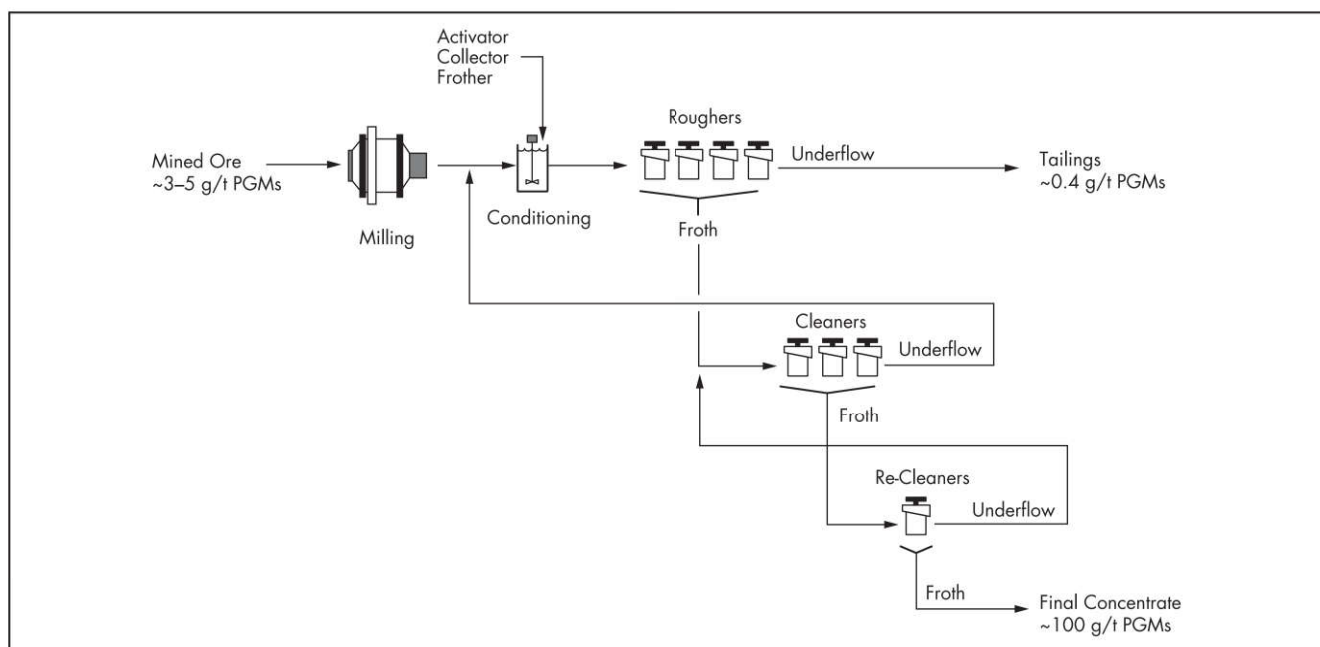
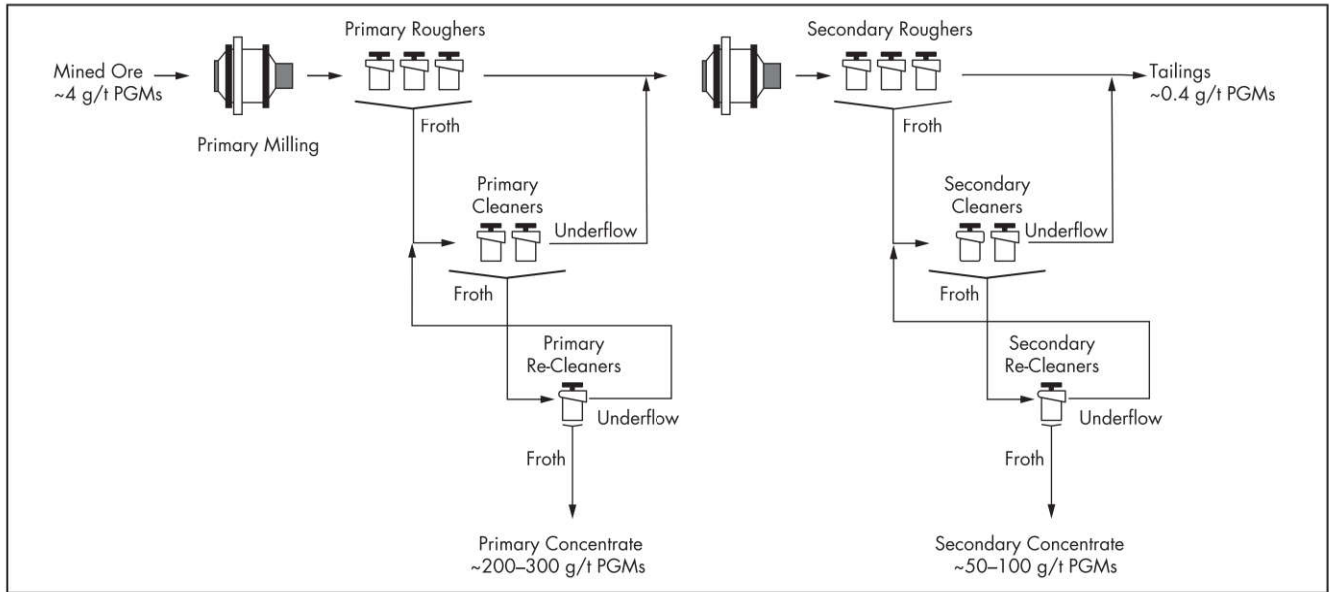
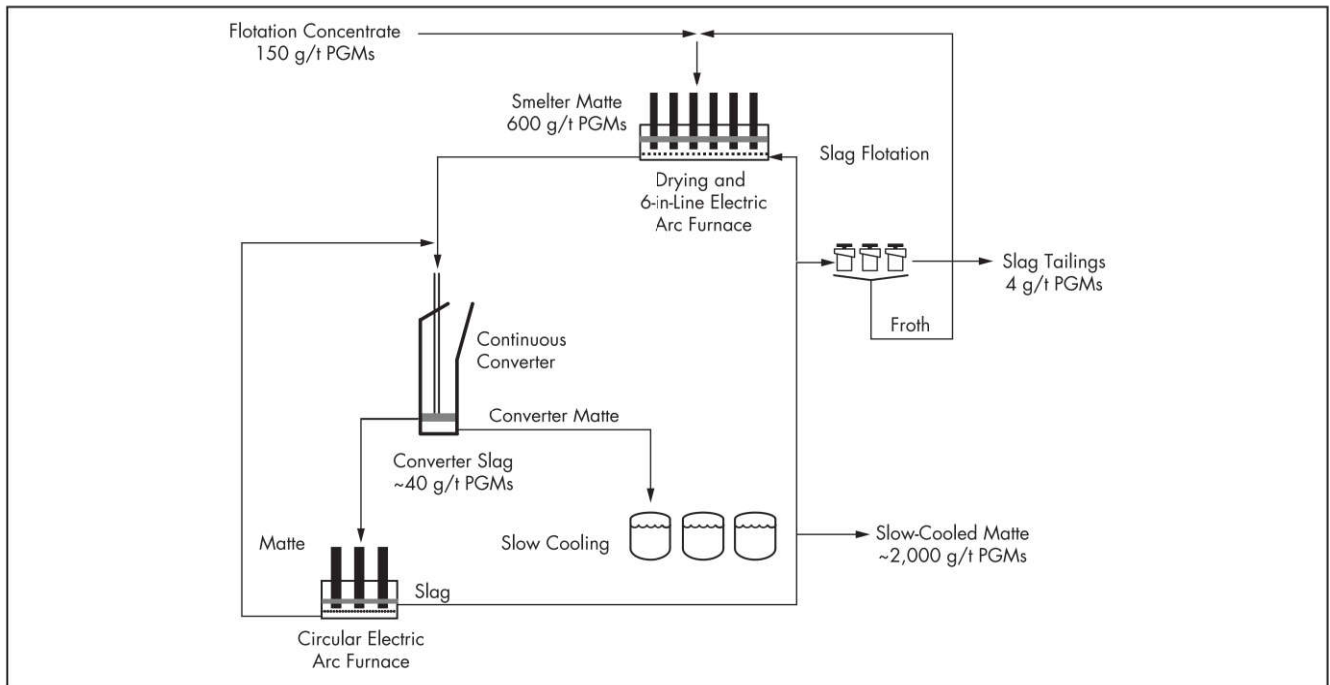


Figure 9 Process flow sheet used to produce flotation concentrates from the Merensky Reef



Adapted from Knights and Bryson 2009

Figure 10 Process flow sheet used to recover flotation concentrates from ores containing increased concentrations of chromite



Adapted from Jacobs 2006

Figure 11 Smelting of platinum-containing sulfide concentrates at Anglo American Platinum's Waterval smelter

Smelting

The flow sheet for the smelter circuit at Anglo American Platinum is shown in Figure 11. Moist or wet concentrates are first dried in flash dryers or spray dryers to a moisture content of less than the requirement of 0.5% (Jacobs 2006). The dried concentrate is fed to the smelter, where the objective is to separate the PGM matte phase from a slag phase containing the gangue minerals. The concentrate is heated to approximately 1,600°C in an electric arc furnace. This temperature is about 250°C hotter than that of typical nickel-copper smelters

because of the higher melting points of magnesium oxide (MgO) and Cr_2O_3 slags associated with these ores. The melt separates by gravity into two phases: namely, the denser matte phase at the bottom of the furnace and the less dense slag phase above it. The furnace is operated semicontinuously, with matte and slag phases being tapped off at regular intervals.

The minerals containing the PGMs are small, typically less than 10 μm in size. Because of their size, droplets of these minerals would take a long time to settle through the slag. It is thought that the larger droplets of sulfide collect these

platinum-containing minerals as they descend. In this manner, PGMs are collected by the “rain” of sulfide minerals falling through the slag (Nelson et al. 2005). This means that it can be more difficult to recover PGMs in ores in which the content of sulfides is particularly low.

The smelter matte is fed to the converter, which can be of the Peirce–Smith, top-blown horizontal, or vertical-blown lance type. The iron content of the feed to the converter is typically 40%, and this is reduced to between 0.6% and 3% in the converter. The removal of the majority of the iron in the converter greatly simplifies the downstream hydrometallurgical processing.

Slow-Cooled Matte and Magnetic Separation

Most producers prefer to send the converter matte directly to the base metals refinery. However, one of the producers (Anglo American Platinum) employs the slow-cooled process, in which molten matte is poured into large molds that are covered to reduce the rate of heat loss. The matte in these molds cools over several days, during which time, crystals of heazlewoodite (Ni_3S_2) and chalcocite (Cu_2S) grow to a size of approximately 100 μm . The converter matte contains less than the stoichiometric amount of sulfur for the formation of these two sulfide phases. As a result, an alloy phase also forms. It is this alloy phase that collects the PGMs.

The slow-cooled matte is crushed and milled, and passed through a series of magnetic separators. The magnetic

concentrate, which contains virtually all the PGMs, is transferred to the precious metals refinery, while the nonmagnetic fraction is sent to the base metals refinery.

Base Metals Refining

As mentioned, most producers send the converter matte to the base metals refinery, where it is milled and leached. In this case, the feed to the refinery is the converter matte, which contains typically 46% Ni, 33% Cu, and 19% S. The first two stages are leaching steps, and the leach residue from these steps (after some further batch dissolution steps to remove base metals and amphoteric elements) forms the feed to the precious metals refinery.

There are different processes for the refining of the base metals, and these processes might be classified on the basis of the form in which the nickel and sulfur are produced. Different options have been selected by different producers, as shown in Table 4.

Flow sheets for the three processes shown in Table 4 are given in Figures 12 to 14. The least complex of these flow sheets is the nickel sulfate process shown in Figure 12.

Nickel sulfate process. This process is implemented by Lonmin Platinum and Northam Platinum, both in South Africa, and by Stillwater in Montana, United States. The converter matte is contacted with copper spent in the first-stage leach at 85°C, which consists of five tanks in series. Oxygen is supplied to the first three tanks. The Ni_3S_2 phase in the converter matte reacts as follows:

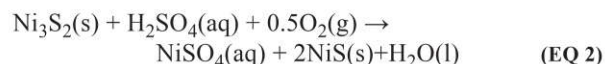
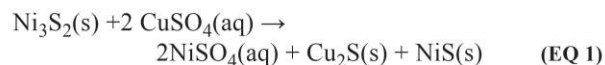


Table 4 Products from different base metal refineries

Process	Nickel Product	Sulfur Product	Producer
1	Nickel sulfate	Nickel sulfate	Lonmin Platinum, Northam Platinum, Stillwater
2	Nickel powder	Ammonium sulfate	Impala Platinum
3	Nickel cathode	Sodium sulfate	Anglo America Platinum

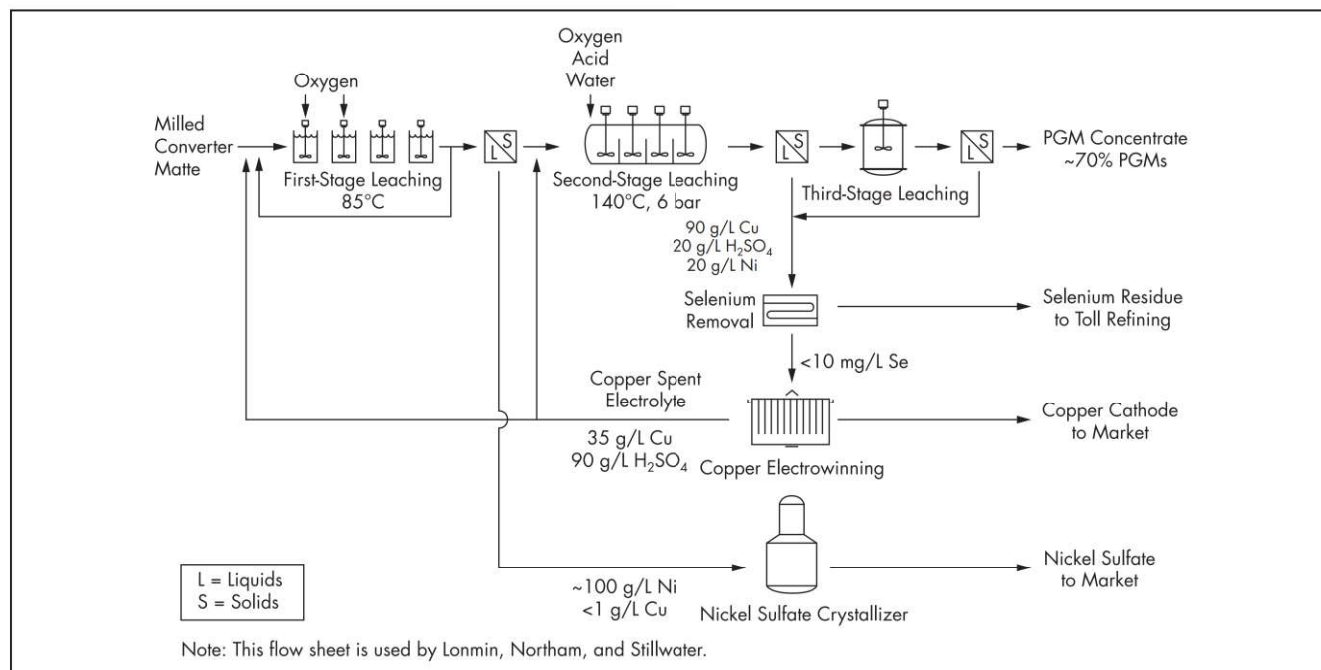


Figure 12 Process flow sheet for the base metal refinery where nickel is produced as nickel sulfate

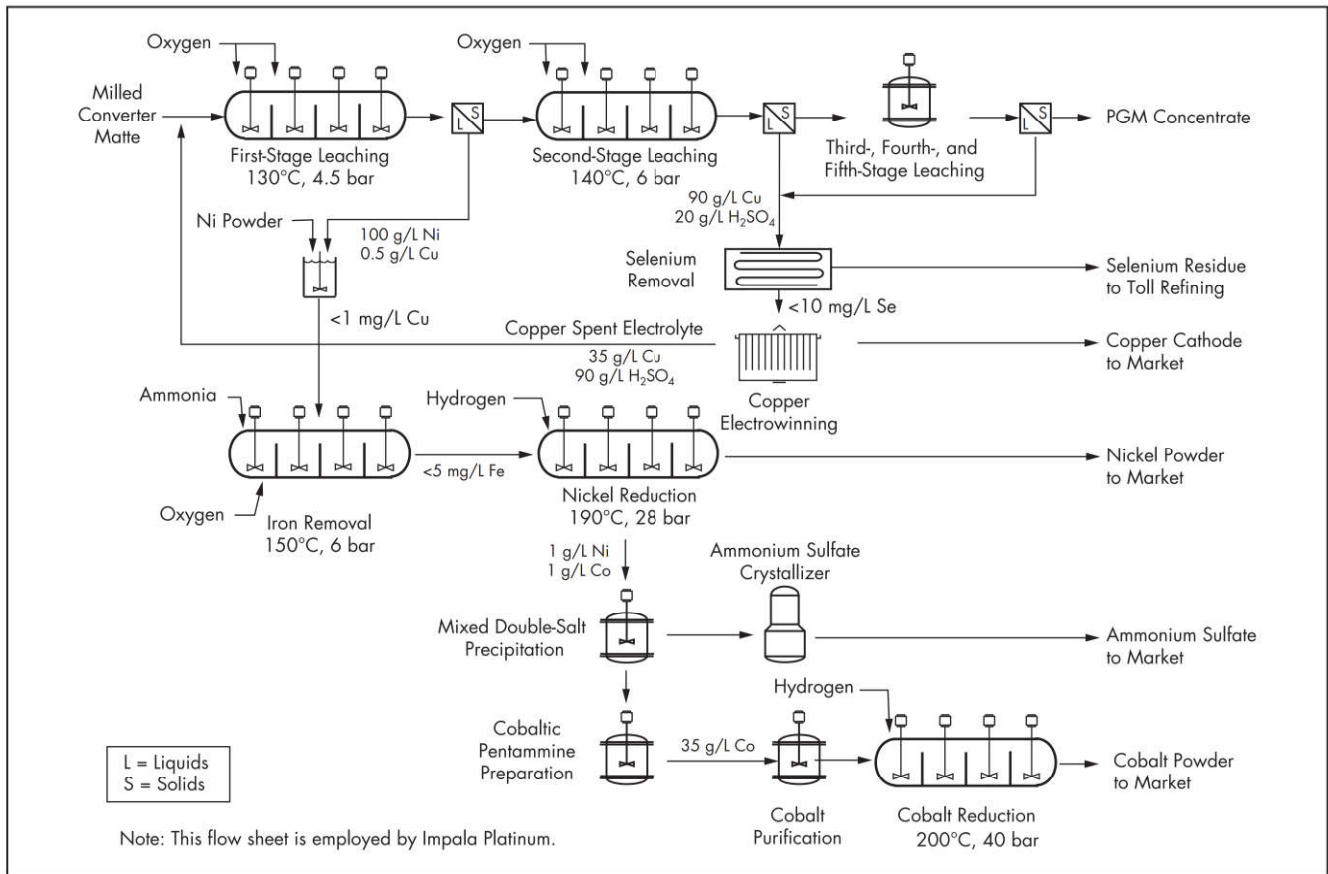


Figure 13 Process flow sheet for the base metals refinery where nickel is produced as nickel powder

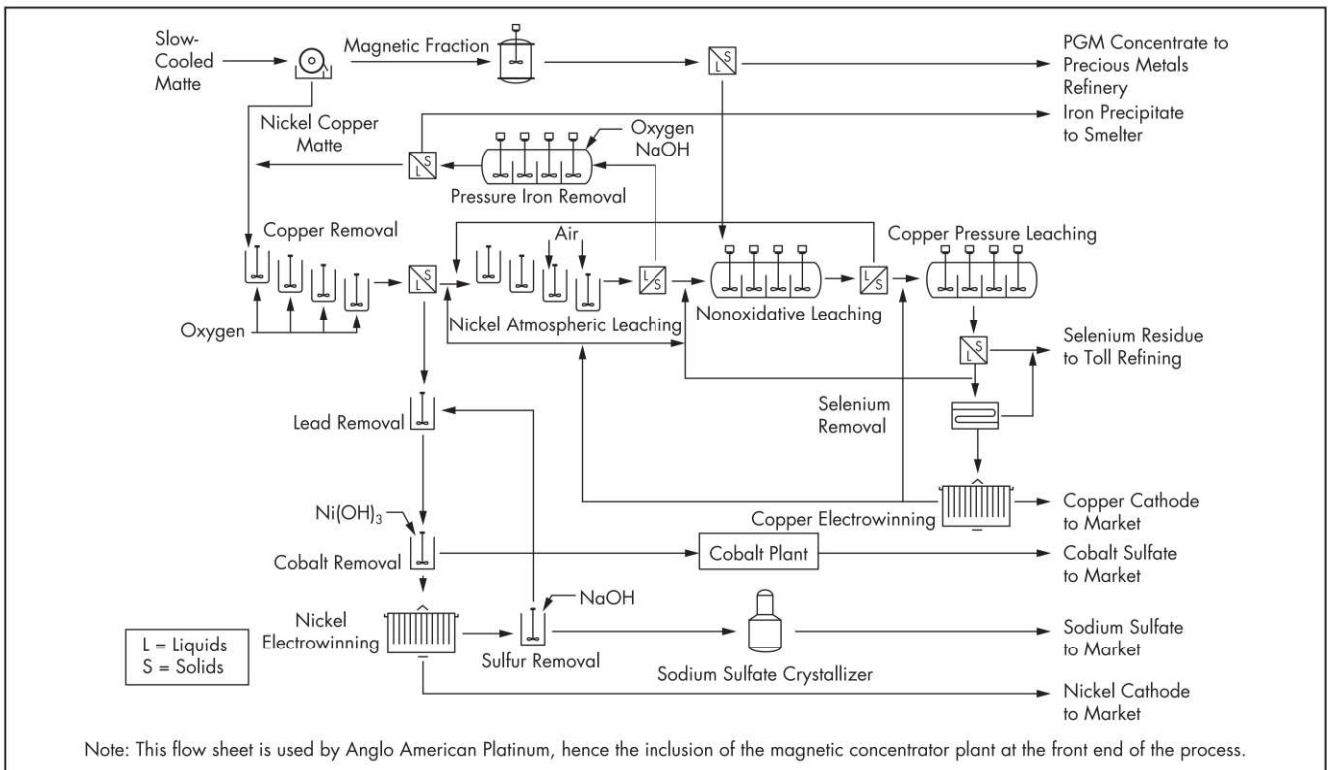
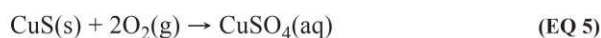
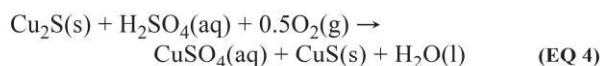
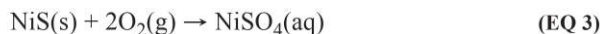


Figure 14 Process flow sheet for the base metals refinery in which nickel is produced as nickel cathode

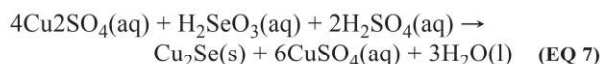
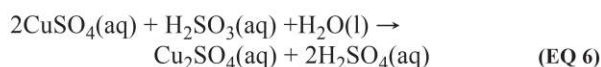
Reaction 1 is the primary method used for separating copper from nickel in these base metals refineries. The consequence of these two reactions is that approximately 75% of the nickel dissolves, reaching about 90 g/L. Copper precipitates to less than 0.5 g/L, and acid is consumed to the point where its concentration is less than 1 g/L.

The reaction conditions are intensified in the second-stage leach by increasing the temperature and the oxygen partial pressure. The reactions are carried out in a horizontal autoclave, which is separated into various compartments so that internally it forms a series of continuous stirred-tank reactors (CSTRs). The reactions that occur are the following:



As a result of these reactions, all the remaining nickel and copper dissolves, leaving a residue rich in PGMs. This residue is leached in formic acid to remove additional copper and nickel. In another step, it is leached in caustic soda with oxygen to remove sulfur, selenium, and tellurium. The remaining residue is transferred to the precious metals refinery, where the individual PGMs are separated.

The solution from second-stage leaching is purified by reducing the selenium and tellurium using sulfurous acid. The reactions are as follows:



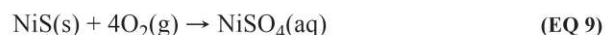
This reaction occurs at approximately 90°C. The initial contacting is in a pipe reactor, after which the reactions continue in two tanks with residence times of the order of 24 hours. The resulting slurry is filtered, and the filtrate, containing between 60 and 75 g/L Cu and 15 g/L H₂SO₄, is sent to copper electrowinning.

Copper is plated onto stainless-steel cathodes (or copper starter sheets), and oxygen evolves at the lead anode. The solution discharge from copper electrowinning, referred to as *copper spent*, contains 25–35 g/L Cu and 80–100 g/L sulfuric acid (H₂SO₄). This copper spent is recycled to all the leaching stages.

The solution from the first-stage leach, containing 100 g/L nickel sulfate along with impurities of copper, iron, and acid, is sent to the nickel crystallizer, where nickel is crystallized as nickel sulfate heptahydrate. Because the feed to the crystallizer is impure, the nickel sulfate product is not a pure product. If a purer nickel product is desired, significantly more processing steps are required, as is evident from a comparison of the nickel sulfate circuit with the nickel powder circuit shown in Figure 13, which is discussed next.

Nickel powder process. This process, shown in Figure 13, is implemented at Impala Platinum. The first-stage leach is performed in an autoclave at 130°C. Oxygen is added to the first two compartments of the autoclave, and none to the last two. As a result, the last two compartments are referred to as *nonoxidizing*. The final conditions are similar to those for

the nickel sulfate process, that is, approximately 0.5 g/L Cu and 15 g/L H₂SO₄. The reactions that occur in the first stage are similar to those of the first stage in the nickel sulfate process, given by Reactions 1 and 2, but because of the harsher conditions in the first-stage leach, two additional reactions occur:



As a result, the nickel extraction reaches typical values of 85%, which is higher than that achieved in the nickel sulfate process.

Slurry from the thickener underflow from first-stage leach is diluted with process water and fed to the second-stage leach. The reactions occurring in this section are similar to those in the nickel sulfate process, that is, Reactions 3 to 5. The autoclave is operated at a temperature between 120° and 140°C and at a pressure of 9.5 bar gauge. Over the last five years, the operating temperature has been lowered to reduce the dissolution of PGMs in this stage.

The leach residue from the second stage is leached in a series of batch stages to remove base metals and amphoteric elements. The amphoteric elements of interest are selenium, tellurium, sulfur, and arsenic. The residue from these leaching steps is the feed to the precious metals refinery.

The solution from the second-stage leach contains approximately 90 g/L Cu and 20 g/L H₂SO₄. This solution is purified in the selenium-removal section in a manner similar to that described previously, and sent to copper electrowinning. The spent solution from copper electrowinning goes to the first-stage leaching section.

The solution from the first-stage leach, containing 100 g/L Ni, 0.5 g/L Cu, and 1.5 g/L H₂SO₄, is purified by removing copper and iron in separate steps. Copper is removed by cementation with nickel powder. The resulting solution contains less than 1 mg/L Cu. Iron is removed from this solution in an autoclave operated at a temperature of 150°C and a pressure of 7.5 bar gauge using oxygen. Ammonia is added to precipitate the iron as ammonium jarosite, NH₄Fe₃(SO₄)₂(OH)₆. Lead and arsenic are also removed in this autoclave. Lead precipitates in the form of plumbojarosite, Pb_{0.5}Fe₃(SO₄)₂(OH)₆, and arsenic in the form of scorodite, FeAsO₄·2H₂O. The solution, after filtering, is pumped to nickel reduction.

The solution is diluted to 60 g/L Ni using a solution containing ammonium sulfate. Ammonia is added so the molar ratio of nickel to free ammonia is approximately 1.9. This solution is fed to a batch autoclave that operates at a temperature of 190°C and a pressure of 28 bar gauge. Nickel sulfate is reduced to nickel metal using hydrogen gas. The operation is as follows: Solution is pumped into the reactor with seed crystals, heated and pressurized until the concentration of nickel is reduced to between 2 and 3 g/L. The reactor is depressurized to about 2 bar gauge, and the solution is drained while leaving the nickel powder in the reactor. Fresh solution is then pumped in. This cycle is repeated about 60 times, referred to as *densification*. After approximately 20–50 t (metric tons) of nickel powder is produced, the reactor is depressurized, flushed with nitrogen, and the slurry of nickel powder is drained from the reactor. The powder is filtered, washed, and dried. This dried powder can be mixed with a binding agent and briquetted, which is the final product.

The solution from nickel reduction contains 2–3 g/L nickel and about 0.5 g/L cobalt. The nickel and cobalt is precipitated from solution as a mixed double salt of nickel diammine disulfate hexahydrate, $\text{Ni}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, and cobalt diammine disulfate hexahydrate, $\text{Co}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$. The mixed double salt is filtered and dissolved in water so that the concentration of nickel is 85 g/L and that of cobalt is 20 g/L. In the next step, ammonia is added to the solution. This ammonia forms stable cobaltic pentammine complexes with the cobalt. The solution is cooled and acidified so that nickel precipitates as $\text{Ni}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$. This leaves a solution containing about 35 g/L of cobalt.

The reduction of cobalt is achieved using hydrogen reduction in a similar manner to that of nickel, except at higher temperature and pressure. The temperature is 200°C and the pressure 40 bar gauge. After washing and drying, the cobalt powder is briquetted.

The solution from the reduction of cobalt and from mixed double-salt precipitation contains about 450 g/L ammonium sulfate. This ammonium sulfate is crystallized in a crystallizer, and the crystals are sold into the fertilizer market.

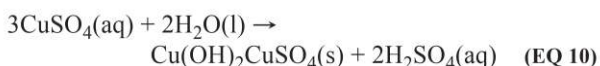
Nickel cathode process. This process, shown in Figure 14, is implemented at Anglo American Platinum. It is significantly more complex than the other two processes because of the five leaching stages arranged in a countercurrent configuration, although the main separation techniques are similar. The implementation is also different from that of the other producers in that the slow-cooled matte and magnetic separation process lies between that of the converter matte and the base metals refinery.

The slow-cooled converter matte is crushed, milled, and separated by magnetic concentration in three stages. The mass pull of the magnetic concentrate is between 13% and 17% of the mass of the slow-cooled matte. The nonmagnetic fraction, referred as the *nickel-copper matte*, forms the feed to the base metals refinery.

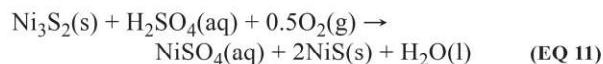
The magnetic concentrate is leached in three different solutions to remove nickel, copper, and iron. The first of these three leaching steps is performed at 160°C and 9 bar gauge using acid and oxygen to remove copper. The second leaching step is performed at atmospheric pressure using formic acid to remove iron from spinel-type minerals such as trevorite (NiFe_2O_4). The objective of the third leaching step is to dissolve any remaining copper, nickel, or iron. This leaching stage operates under conditions similar to those of the first-leach.

The residue from these leaching steps contains about 50% PGMs and forms the primary feed to the precious metals refinery. The filtrate, containing approximately 100 g/L Ni and 30 g/L Cu, is pumped to the base metals refinery.

The first step in the base metals refinery is referred as *copper removal*. In this step, the nickel-copper matte from the magnetic separation plant is contacted with solution from iron removal in a series of four CSTRs. The solution contains about 70 g/L Ni^{2+} and 12 g/L Cu^{2+} at a pH value of 2. Copper precipitates as antlerite, $\text{Cu}(\text{OH})_2 \cdot \text{CuSO}_4$, so that the concentration of copper solution is less than 0.5 g/L by the following reaction:

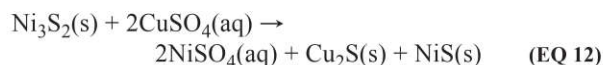


At the same time, acid is consumed by the partial leaching of Ni_3S_2 :

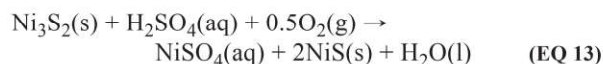


The precipitation reaction is at equilibrium, which means that the concentration of copper is dependent only on the temperature and the pH. However, if the pH rises above a value of 6, nickel precipitates from solution as nickel basic sulfate, $\text{Ni}(\text{OH})_2 \cdot \text{NiSO}_4$, so careful control of pH is needed. The slurry is thickened, and the overflow solution is pumped to lead removal, while the underflow solids are pumped to nickel atmospheric leaching.

In the nickel atmospheric leaching step, the slurry from copper removal is mixed with copper spent, which contains about 30 g/L Cu and 90 g/L H_2SO_4 , and solution from the non-oxidizing leaching, which contains about 20 g/L Cu and 35 g/L H_2SO_4 . The equipment consists of four CSTRs in series, and air is injected to some of the tanks. The concentration of copper in solution is reduced by about half in this step, while all the remaining Ni_3S_2 dissolves. The reactions that occur are the metathesis leaching of Ni_3S_2 :

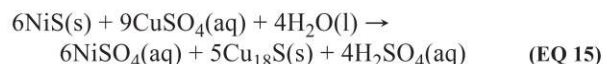


and the dissolution of Ni_3S_2 by oxygen:



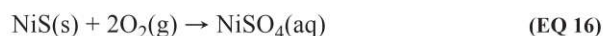
In addition, all of the antlerite that is precipitated in copper removal redissolves in nickel atmospheric leaching. The slurry is thickened, the overflow is pumped to iron removal, and the underflow solids are pumped to the nonoxidative leaching stage.

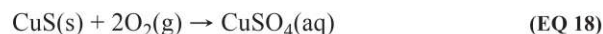
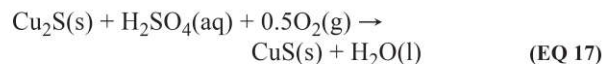
The next step is the nonoxidative leaching stage, in which the slurry from nickel atmospheric leaching is combined with solution from the magnetic concentrator plant and with solution obtained from the feed to copper electrowinning, referred to as *copper advance*. A four-compartment autoclave operated at 150°C and 6 bar gauge is used to dissolve as much of the nickel as possible. The reactions that occur in this reactor are the metathesis of millerite (NiS) in the following two reactions:



Because this last reaction produces acid, the concentration of acid rises across nonoxidative leaching. The solution leaving nonoxidative leaching contains about 20 g/L Cu^{2+} and 30 g/L H_2SO_4 . The leach slurry is thickened, the overflow is pumped to nickel atmospheric leaching, and the underflow slurry is pumped to copper pressure leaching.

The next step is copper pressure leaching, in which the slurry from the nonoxidizing leach step is contacted with copper spent from electrowinning in an autoclave operating at 140°C and 6 bar gauge. Oxygen is sparged into each of the four compartments of the autoclave. The following reactions occur under these conditions:





The solution leaving the copper pressure leaching autoclave contains about 90 g/L Cu, 40 g/L Ni, and 15 g/L H_2SO_4 . The slurry is filtered, the filter cake is collected for toll refining to recover the value of residual PGMs, and the filtrate is pumped to selenium and tellurium removal.

Selenium and tellurium removal and copper electrowinning are similar in nature to these same steps for the nickel sulfate and nickel powder processes discussed earlier.

The solution from copper removal is purified further by the removal of lead and cobalt from solution. Lead is removed by precipitation with barium hydroxide. The cobalt in this product stream is oxidized to the cobaltic state using nickelic hydroxide. This causes cobaltic hydroxide to precipitate. The precipitate is dissolved, and a pure cobalt stream is obtained by solvent extraction using D2EPHA as the extractant. The cobalt-lean solution from cobaltic precipitation is fed to the nickel electrowinning tank house.

The feed solution to nickel electrowinning contains about 90 g/L Ni and 140 g/L sodium sulfate (Na_2SO_4) at a pH of 3.5. The solution is fed individually to the cathode compartment of a cell, which is divided into compartments using a cathode bag. Solution flows from the cathode compartment into the anode compartment, from which it exits the cell. The flow through the cathode bag opposes the diffusion of H^+ ions from the anode compartment to the cathode compartment. The nickel plates onto titanium cathodes, and once the plate is sufficiently thick, the cathodes are harvested and stripped. The nickel cathode is washed, strapped into bales, and shipped to market. The solution from nickel electrowinning goes to sulfur removal.

In sulfur removal, the flow from nickel electrowinning is split into two streams. In one stream, all the contained nickel is precipitated as nickel hydroxide and filtered. The filtrate goes to the sodium sulfate crystallizer. The cake is dissolved in the fraction of the stream from nickel electrowinning that was not subject to precipitation.

The nickel-free solution from sulfur removal is transferred to the sodium sulfate crystallizer. The product crystals of sodium sulfate are sold as filler in detergents.

Comparison of different operations. A comparison of the different processes is presented in Table 5.

Precious Metals Refinery

The typical feed composition to a precious metals refinery is shown in Table 6. From this feed, PGMs must be produced at a purity of equal to or greater than 99.9% with as high a recovery as possible. The flow sheet of platinum metals refineries generally follow a pattern in which there is a sequence of primary separations, in which each of the PGMs is removed sequentially from the feed, secondary purification of each the products is made from the primary separations, and finally reduction of the salt to metallic form suitable for sale to the market is performed. The *sharpness* of the separation processes used affects the *first-pass yield*, which is a measure of the fraction of PGMs in the feed that emerges from the refinery without having to be reprocessed.

There are three main types of modern refineries, classified by the methods used in the primary separations, particularly the separation of platinum and palladium: (1) those based on precipitation, (2) those based on solvent extraction, and (3) those based on ion exchange. Each of these will be discussed in turn.

Precipitation-based refining. An overview of the precipitation process as implemented at Lonmin is shown in Figure 15. The platinum-metal concentrate as received from the base metals refinery contains 68%–70% PGMs. This material is digested in a solution of 6 M HCl at 65°C for about 6 hours. Chlorine is continuously sparged through the glass-lined reactor while the slurry is agitated using a pitched blade turbine.

Once the digestion is complete, air is blown through the solution to remove the dissolved chlorine. The slurry is filtered, and the remaining solids are redissolved using similar conditions. The dissolution efficiency of the platinum and palladium is in excess of 99%. The dissolution efficiency of the other metals is in excess of 95%, except that occasionally the extraction of iridium may drop to 85%.

Gold, which is in solution following the digestion step, is precipitated using hydrazine. Following this, the pH of the filtrate is adjusted to a value of 1 by the addition of sodium hydroxide. This results in the precipitation of approximately 40% of the base metals in this solution.

In the next step, ruthenium is removed from solution by distillation. Sodium chlorate and sodium bromate are added to the solution to oxidize the ruthenium and osmium to RuO_4 and OsO_4 . In this form, ruthenium and osmium are volatile, and are easily stripped from solution into the gas phase. This stripping is achieved by passing a stream of air through the vessel. This gaseous stream, now laden with RuO_4 and OsO_4 , flows through a packed column where it is contacted with a solution containing hydrochloric acid. The solution causes the reduction of the gaseous ruthenium, which then reports to the aqueous phase. The gaseous phase passes through a second column where it is contacted with a solution containing caustic soda, which scrubs chlorine and osmium from the gas phase.

The pH of the solution remaining after ruthenium distillation is increased to about a value of 5, resulting in the precipitation of rhodium and iridium hydroxide. These salts are filtered from the solution and transferred to this sidestream.

The solution remaining after pH 5 contains mainly platinum and palladium. These two elements are separated from one another by the addition of hydrochloric acid and hydrogen peroxide, which causes the precipitation of ammonium hexachloroplatinate, $(\text{NH}_4)_2\text{PtCl}_6$. This salt is filtered from solution and ignited to produce an impure platinum sponge of between 92% and 97% platinum. The main impurity in the platinum sponge is palladium. The sponge is purified in the platinum sidestream.

The pH of the solution remaining after platinum precipitation is increased to 0.9 by adding sodium hydroxide. The solution is boiled, during which time the pH increases to about a value of 3. Ammonium acetate is added and the solution boiled until the pH reaches a value of 4.2. When this solution is cooled, palladium precipitates as diammino-palladous dichloride, $(\text{NH}_3)_2\text{PdCl}_2$. This precipitate is filtered off and transferred to the palladium purification sidestream.

Table 5 Comparison of the different base metals refineries

	Nickel Sulfate Process	Nickel Powder Process	Nickel Cathode Process
Operation	Lonmin Platinum	Impala Platinum	Anglo American Platinum
Year of first production	1971	1969	1954
Converter matte capacity, t/a (metric tons per annum)	10,000	30,000	45,000
Nickel Leaching			
Temperature	85°C	150°C	NAL*: 85–90; NOX†: 150°C
Pressure	Atmospheric	4.5 bar	NAL: ambient; NOX: 6 bar
Nickel extraction	75%	85%	80%–85%
Copper Leaching Stage			
Temperature,	140°C	130°–140°C	140°C
Pressure	6 bar	6 bar	6 bar
Copper extraction	90%	>95%	>95%
Iron Removal Step			
Product	Ferric hydroxide	Ammonium jarosite	Sodium jarosite
Temperature	85°C	140°C	140°C
Cobalt Product			
Purification method	None	Cobaltic pentammine	Cobaltic hydroxide
Product	None	Cobalt powder or briquette	CoSO ₄ ·6H ₂ O
Nickel Product			
Production capacity, t/a	3,500	16,000	33,000
Process route	Nickel crystallizer	Hydrogen reduction	Electrowinning
Product type	NiSO ₄ ·6H ₂ O	Nickel powder or briquette	Nickel cathode
Copper Product			
Electrowinning capacity, t/a	2,500	9,000	18,000
Platinum Group Metal Product			
Composition of PGMs	68%–70%	50%–60%	50%

Source: Crundwell et al. 2011

*NAL = nickel atmospheric leaching

†NOX = nonoxidative leaching

This completes the description of the primary separation step. The most important sidestreams are those of platinum and palladium, which are discussed next.

The feed to the platinum sidestream is the impure platinum sponge. This sponge is digested in hydrochloric acid using chlorine gas. The solution is heated, the pH is increased to a value of 3, and sodium chlorate and sodium bromate are added. The pH is increased to a value of 5, which results in the precipitation of iridium and rhodium hydroxides. The pH of the solution remaining after this treatment is increased to 8, and any precipitated base metals are filtered off. After this step, dimethylglyoxime is added, which results in the precipitation of palladium from solution. Following these steps, the solution should contain only platinum. This solution is conditioned by adding hydrochloric acid and hydrogen peroxide, and then ammonium chloride is added, which causes (NH₄)₂PtCl₆ to precipitate. This salt is filtered off, redissolved in water, and the dissolved platinum reduced to metal using hydrazine. The metal is calcined to produce a platinum sponge with a purity of 99.99% platinum.

The feed to the palladium sidestream is the impure palladium salt. The main impurity in this salt is platinum. The salt is dissolved in a solution of pH 9, and the redox potential is adjusted using hydrogen peroxide. Under these conditions, the

Table 6 Composition of the feed to a South African PGM refinery

Element	%
Pt	24.6
Pd	13.3
Au	1.12
Rh	4.12
Ru	5.03
Ir	1.49
Ag	2.20
Ni	1.20
Cu	3.00
Fe	2.60
Pb	1.90
SiO ₂	12.9
As	0.43
Se	1.20
Te	2.40
Ba	0.20
Na	1.70
O, H	~20.00

Source: Asamoah-Bekoe 1998

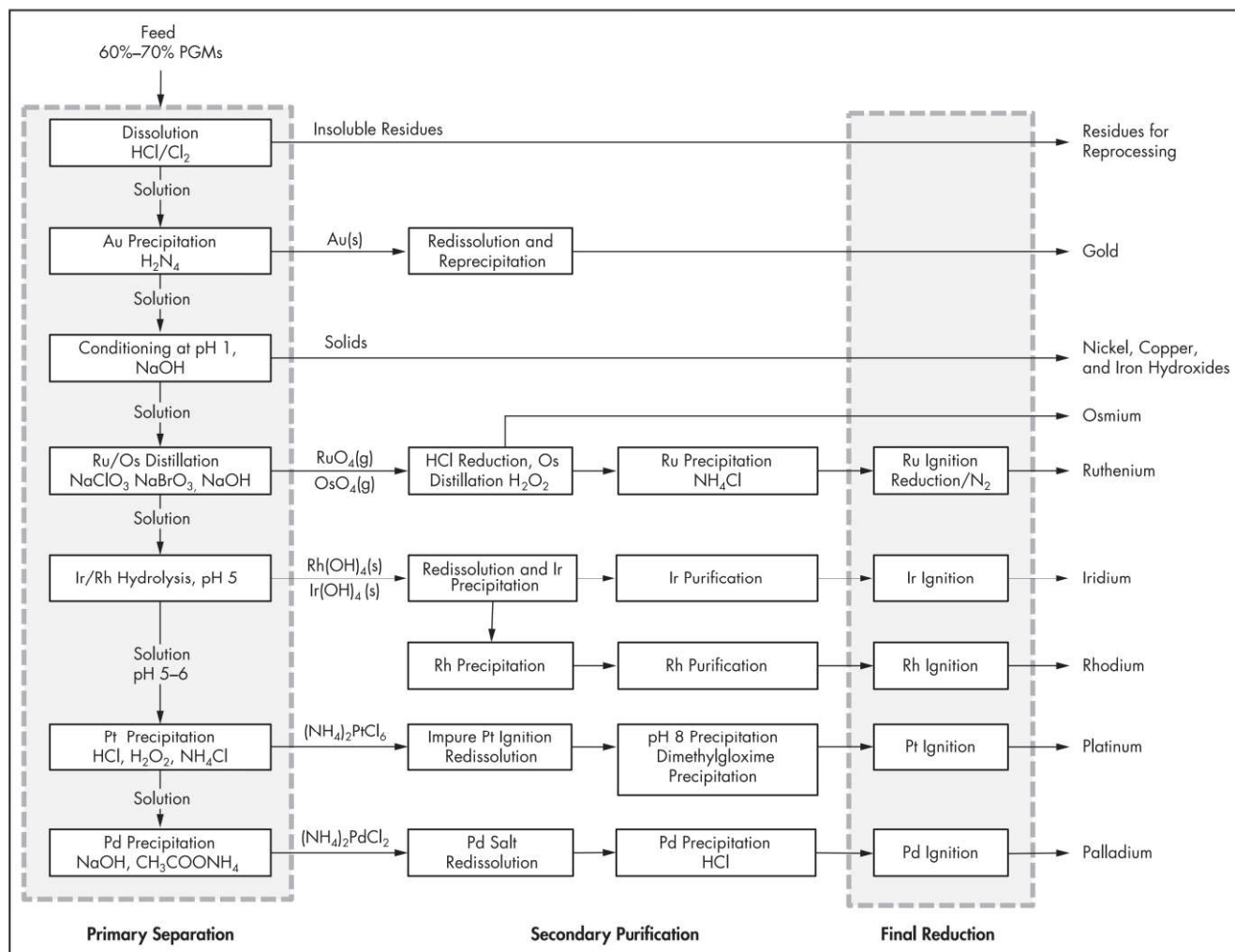


Figure 15 Process flow sheet used for refining PGMs at Lonmin's Western Platinum Refinery. As is typical in refineries, the flow sheet consists of primary separation steps, secondary purification steps (also called *sidestreams*), and final metal production or reduction steps.

palladium salt dissolves, but the platinum in the salt does not. After filtering, the solution contains primarily palladium. The solution is acidified to a pH of 0.6, which causes $(\text{NH}_3)_2\text{PdCl}_2$ to precipitate. This precipitate, ignited in air, produces palladium metal with a purity of between 99.98% and 99.99% palladium.

Solvent-extraction-based refining. An overview of the solvent-extraction-based process implemented at Vale's refinery at Acton in England is shown in Figure 16. The plant treats anode slimes and secondary materials in addition to primary concentrates (Barnes and Edwards 1982). The first step in the process at Acton is the smelting of the feed with iron, which collects PGMs. This iron is dissolved from the smelted solids, which is the feed to the rest of the process.

The residue from iron dissolution is dissolved in hydrochloric acid using chlorine gas at 90°C and at ambient pressure. The residues from this digestion step contain silver and lead that are treated with nitric acid. After this nitric acid treatment, the residues are fused with sodium hydroxide at temperatures between 500° and 600°C, and then subject to a second dissolution step with hydrochloric acid and chlorine gas. The resulting solution is neutralized using sodium hydroxide.

In the next step, ruthenium is removed by distillation. Sodium bromate is added to oxidize ruthenium in solution to ruthenium tetroxide at a temperature of about 90°C. The ruthenium tetroxide is stripped from solution by passing a stream of air through the solution. The ruthenium tetroxide in the gas phase is reduced by contacting the gas with a solution containing hydrochloric acid. Ammonium chloride is added to this solution, resulting in the precipitation of ammonium ruthenium chloride, $(\text{NH}_4)_3\text{RuCl}_6$. This salt is heated and reduced using hydrogen gas with a purity of 99.9% ruthenium.

The pH of the solution remaining after ruthenium distillation is increased using sodium hydroxide, resulting in the precipitation of base metals, such as copper and nickel.

Hydrochloric acid is added to the solution remaining after base metal precipitation so that the concentration is about 3 M HCl. This solution is fed to the gold-solvent-extraction circuit, where it is extracted by dibutyl carbitol in two stages: The loaded organic is washed with a solution of hydrochloric acid and stripped from the loaded organic using oxalic acid. The oxalic acid reduces the gold to the metallic state. This gold powder is melted into grains with a purity of 99.99% gold.

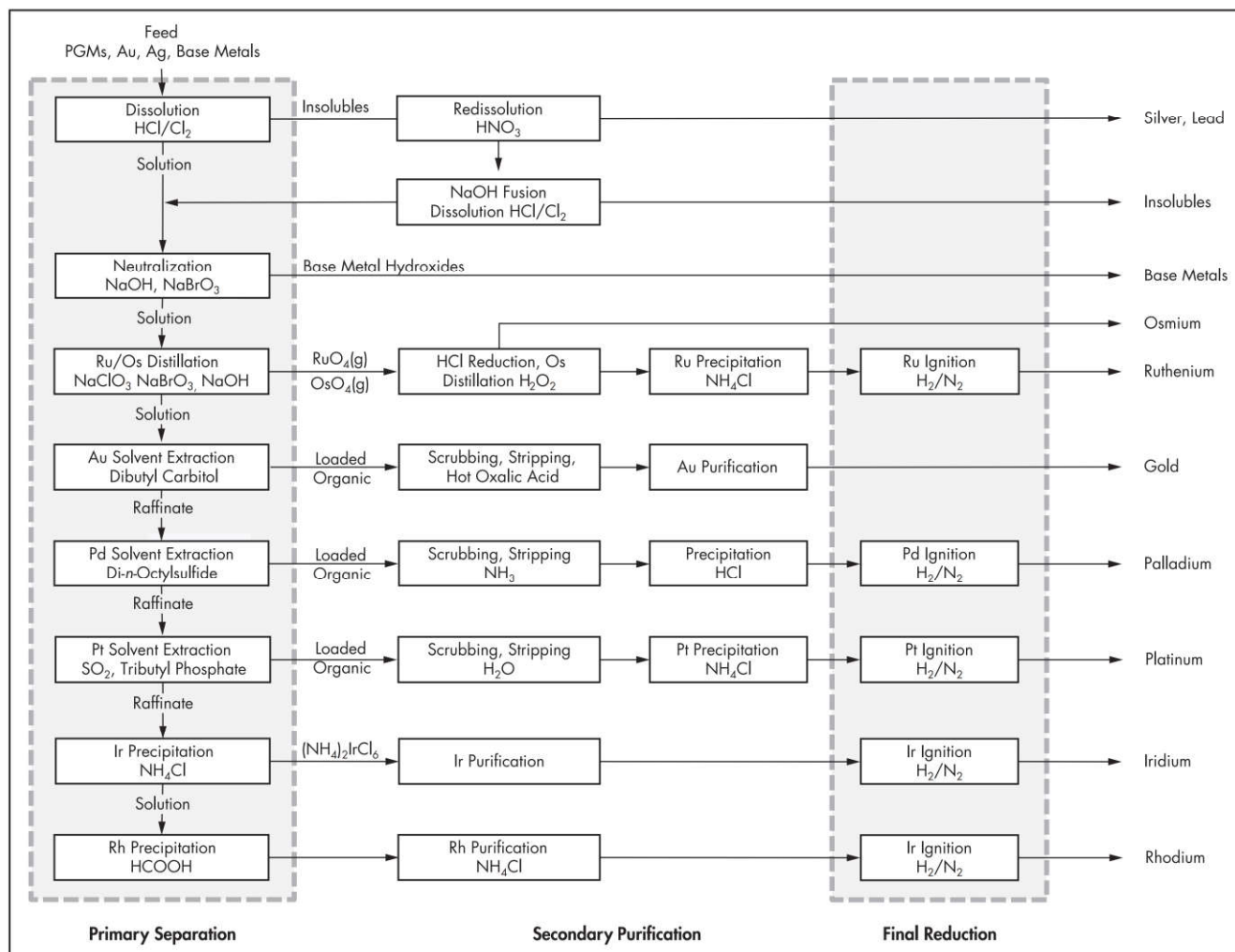


Figure 16 Process flow sheet used at Vale's refinery at Acton, London, England. This flow sheet is typical of solvent-extraction-based processes.

The raffinate from gold extraction is fed to palladium solvent extraction, where the palladium is extracted from the solution using di-*n*-octyl sulfide. The raffinate contains less than 1 mg/L. The loaded organic is scrubbed with hydrochloric acid and stripped from solution, using ammonium hydroxide. Palladium is precipitated as $(\text{NH}_3)_2\text{PdCl}_2$ from the strip solution using hydrochloric acid. This salt is reduced at 1,000°C by hydrogen. The final metal sponge has a purity of greater than 99.95% palladium.

The raffinate from palladium extraction forms the feed to platinum solvent extraction. The solution is conditioned using SO_2 to reduce iridium from the tetravalent state to the trivalent state. The platinum in this solution is extracted using tri-*n*-butyl phosphate in four stages. The loaded organic is scrubbed with a solution of 5 M hydrochloric acid and stripped with water. Platinum is recovered from the strip solution by adding ammonium chloride, which causes $(\text{NH}_4)_2\text{PtCl}_6$. This salt is ignited in air to produce a sponge with a purity of greater than 99.95% platinum.

The raffinate from platinum solvent extraction is conditioned so that the iridium is oxidized from the trivalent state to the tetravalent state. Ammonium chloride is added to the solution, causing ammonium hexachloroiridate, $(\text{NH}_4)_2\text{IrCl}_6$, to precipitate. This salt is heated in the presence of hydrogen

gas, which results in iridium metal with a purity of greater than 99.9% iridium.

Rhodium is precipitated from the solution remaining after iridium precipitation by formic acid. This results in *rhodium black*, which is heated to form a powder with a purity of greater than 99.9% rhodium.

Ion-exchange-based refining. An overview of an ion-exchange-based process implemented at the Impala Platinum refinery at Springs in South Africa is shown in Figure 17. The refinery treats primary material from the base metals refinery. Impala Platinum reprocesses a significant amount of materials that contain PGMs, but these materials are added to the smelter rather than as direct feed to the precious metals refinery. The feed to the refinery contains about 65% PGMs.

The initial step is the digestion of the feed in hydrochloric acid using chlorine gas at 70°C. Digestion is carried out in two steps to ensure complete conversion. Gold is extracted from this solution using an ion-exchange resin. The columns are arranged in a lead-middle-trail configuration. The gold is stripped from the resin and precipitated as gold metal using a reductant. The final gold metal has a purity of 99.95% gold.

The raffinate from gold ion exchange is fed to the palladium ion-exchange step. The media that is used, SuperLig 2,

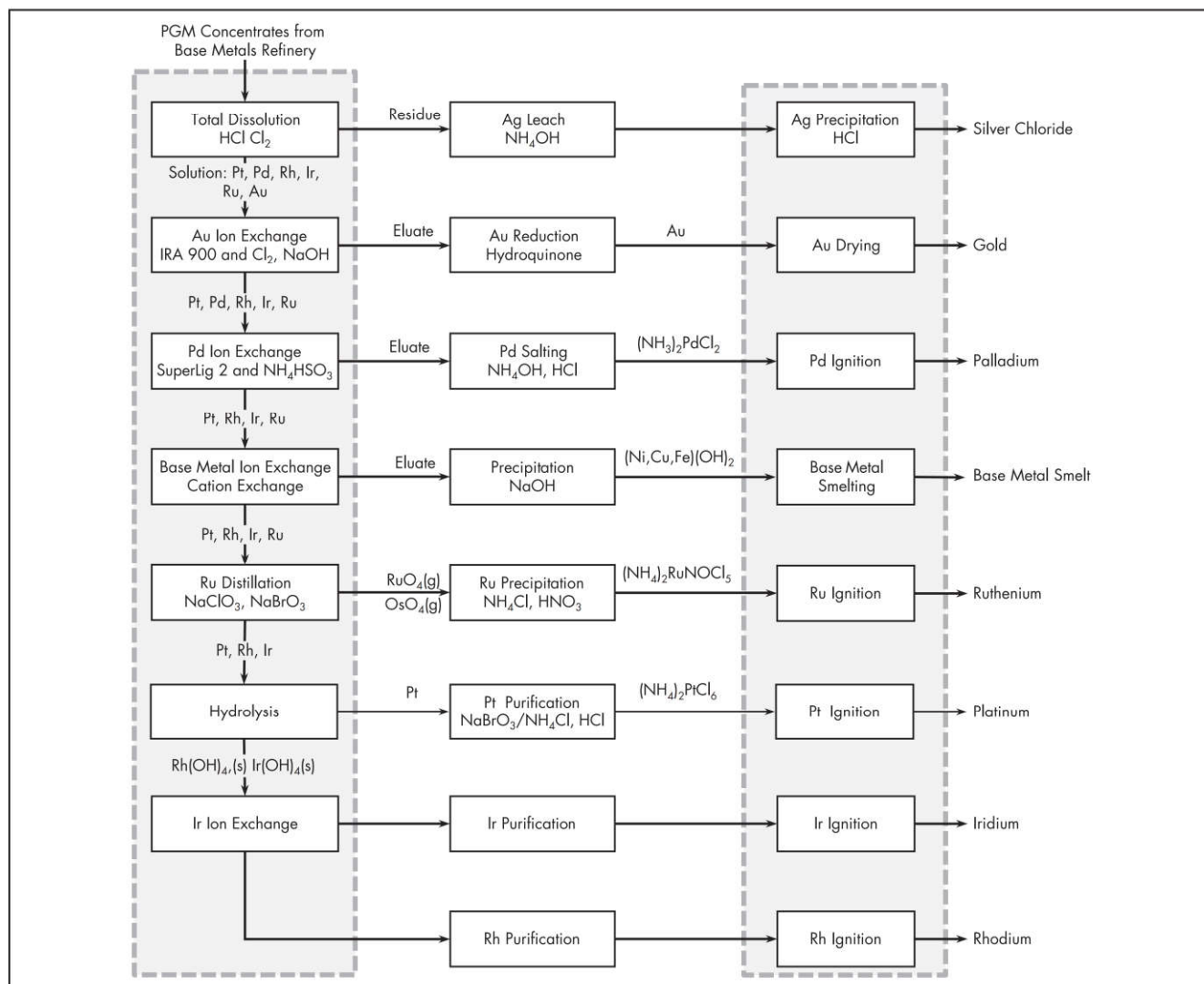


Figure 17 Process flow sheet used at Impala Platinum's refinery in Springs, South Africa. This process is based on ion-exchange-type separations.

is a molecular-recognition technology compound bound to a silica-based substrate. This step is particularly effective: Less than 1% of the platinum in the feed is co-extracted with the palladium, and less than 1 mg/L palladium remains in the solution. Loaded solution is eluted using ammonium bisulfite. Palladium is precipitated as $(\text{NH}_3)_2\text{PdCl}_2$ from the eluate by the addition of ammonium hydroxide and hydrochloric acid. The palladium salt is ignited to produce palladium sponge.

The raffinate from palladium ion exchange is boiled down to increase the concentrations of the metals in solution. Following boil-down, base metals are removed from the solution using ion exchange.

This solution is fed to the ruthenium distillation section, where ruthenium is oxidized to ruthenium tetroxide by the addition of sodium chlorate and sodium bromate. The volatile RuO_4 is stripped from the solution into the gas phase by passing a stream of air through the solution. The ruthenium is removed from the gas phase by contacting it with a solution of hydrochloric acid. The ruthenium in the resulting solution is precipitated as a ruthenium nitrosyl salt, $(\text{NH}_4)_2\text{RuNOCl}_5$, by

the addition of nitric acid and ammonium chloride. The ruthenium nitrosyl salt is ignited to produce pure ruthenium sponge.

The pH of the solution left behind after ruthenium distillation is increased to a value greater than 5 by the addition of sodium bicarbonate to precipitate rhodium and iridium as hydroxides. These hydroxides are filtered from solution and treated in the rhodium-iridium circuit. The filtrate is transferred to the platinum recovery section.

The pH 5 hydrolysis is repeated to further remove rhodium and iridium from the solution. Finally, the solution is boiled to dryness in a glass-lined vessel, the dry contents resolubilized with demineralized water, and the pH adjusted to a value of about 8. After filtration, ammonium chloride is added to the solution so that $(\text{NH}_4)_2\text{PtCl}_6$ precipitates. The salt is ignited to produce platinum sponge with a purity of greater than 99.95% platinum.

Primary Extraction from Russian Ores

The source of PGMs of the Russian deposits is mainly from ores mined primarily for their nickel content. These ores

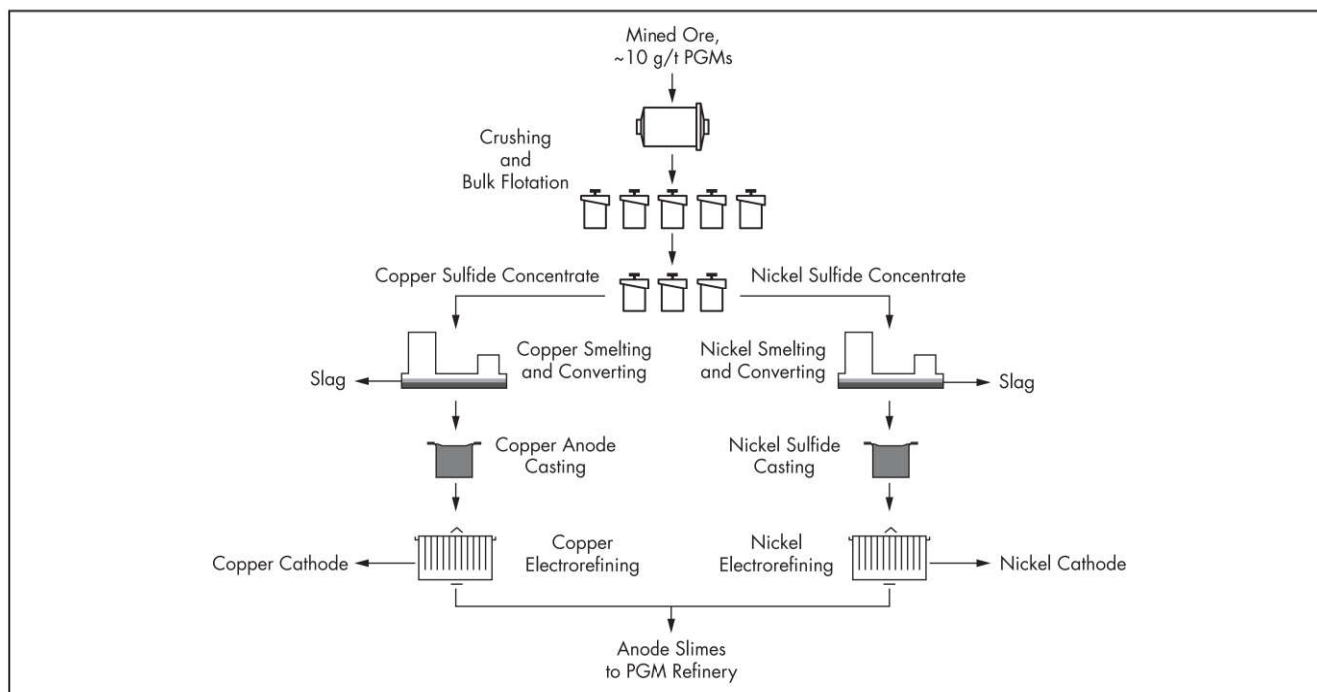


Figure 18 Process overview for the production of PGMs from Russian ores

contain perhaps 50 times more nickel, copper, and cobalt than ores mined in South Africa for their PGMs. Another advantage is that they are more concentrated in PGMs than the South African ores. However, the South African ores have a higher ratio of platinum to palladium, which is advantageous because platinum usually yields a higher price than palladium.

An overview of the extractive metallurgy of these ores is shown in Figure 18. The ore is mined, and after a bulk concentration step, a copper-rich concentrate and nickel-rich concentrate are prepared. Each of these concentrates is smelted separately and cast into anodes. The anodes are purified in an electrorefining step. Precious metals report to the anode slimes, which are then the feed to the platinum refinery.

An overview of the Krastsvetmet refinery at Krasnoyarsk is given in Figure 19. The feed material is sampled and separately routed. Feed rich in platinum and palladium is routed to the platinum–palladium circuit, while that rich in the other PGMs or of low grade is routed to the smelting circuit.

The first step in the platinum–palladium circuit is the digestion in a solution of hydrochloric acid using chlorine gas. From the resulting solution, gold is precipitated using a small quantity of fresh feed. The precipitated gold is redissolved in aqua regia and precipitated by reduction using sucrose, which results in gold with a purity of 99.99% gold.

Ammonium chloride is added to the solution remaining after gold precipitation. This causes platinum to precipitate as $(\text{NH}_4)_2\text{PtCl}_6$. An impure sponge is formed by igniting this salt, which forms the feed to the platinum purification section.

Ammonium hydroxide and hydrochloric acid are added to the solution remaining after platinum precipitation. This results in the precipitation of $(\text{NH}_3)_2\text{PdCl}_2$. This salt is transferred to the palladium purification section.

Feeds that are low in platinum and palladium and all internal recycle streams are smelted. The alloy from the smelting step is dissolved in a solution of hydrochloric acid and

chlorine gas. Ammonium nitrite is added to the solution to precipitate rhodium in the form of ammonium rhodium nitrite, $(\text{NH}_4)_3\text{Rh}(\text{NO}_2)_6$. This salt is redissolved and the solution is fed to a solvent extraction step in which tri-*n*-butyl phosphate is used to remove impurities. Rhodium metal is produced by electrowinning from the raffinate.

Solution remaining after the precipitation of rhodium nitrite is fed to the ruthenium circuit. Platinum and palladium are extracted from this solution using tri-*n*-butyl phosphate, and the strip solution is transferred to the platinum and palladium circuit. Ruthenium is precipitated from the raffinate by the addition of sodium chlorate.

The residue from the alloy dissolution step contains iridium. This residue is alloyed with barium peroxide and then dissolved in a solution of hydrochloric acid using chlorine gas. Barium is removed by precipitation as the sulfate. Impure ammonium chloroiridate is precipitated by the addition of ammonium chloride. After several redissolution and precipitation steps, this salt is ignited to form iridium with a purity greater than 99.9% iridium.

Comparison of Refineries

A comparison of the major features of some precious metals refineries is given in Table 7.

SECONDARY PRODUCTION

It is estimated that approximately 25% of the consumption of PGMs is supplied from recycled sources (Jollie 2010). The supply of secondary production of PGMs into the market from different sources is given in Table 8. The largest source of the recycling in the PGM market is that of autocatalysts (or catalytic converters) used in trucks and cars to catalyze the conversion of hydrocarbons, carbon monoxide, nitrous oxide, carbon dioxide, water, and nitrogen.

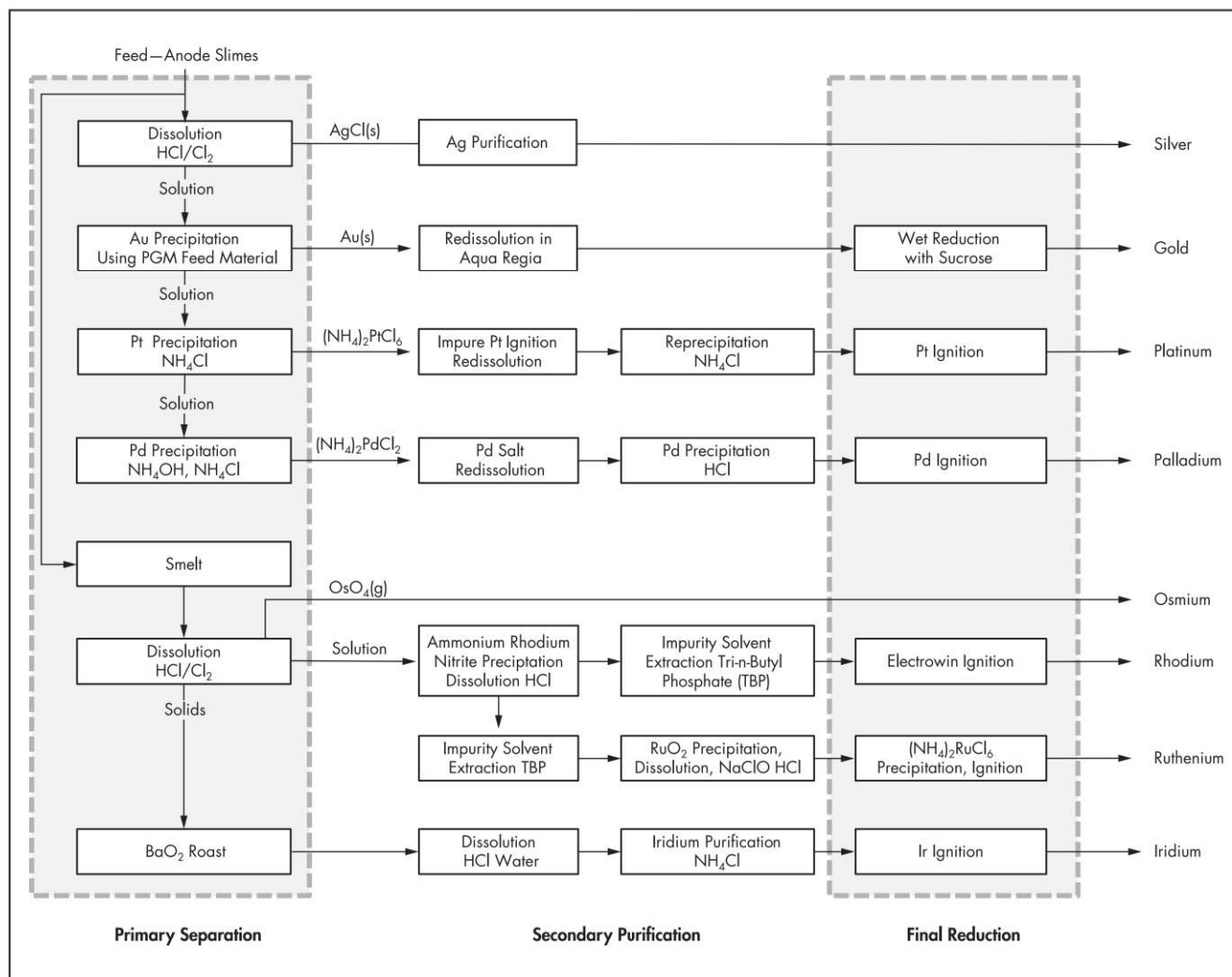


Figure 19 Overview of the Krastsvetmet's precious metals refinery at Krasnoyarsk, Russia

Table 7 Comparison of the major features of several refineries

Feature	Lonmin	Krastsvetmet	Vale (Acton)	Anglo American Platinum	Impala Platinum
Capacity	32,000 kg 1 million oz Pt		32,000 kg 1 million oz Pt	112,000 kg 3.5 million oz Pt	71,000 kg 2.2 million oz Pt
Year started	1975/1987	1943	1975	1989	1998
Production	Primary	Primary	Mainly toll refining	Primary	Primary with large-scale toll refining
Technology	Precipitation	Precipitation	Solvent extraction	Solvent extraction	Ion exchange
Feed quality	65%–75% platinum group metals (PGMs)		<15% PGMs	30%–50% PGMs	60%–65% PGMs
Dissolution	HCl/Cl_2 at 65°C and atm pressure	HCl/Cl_2 at 70°C, 3 bar pressure	HCl/Cl_2 at 65°C and atm pressure	HCl/Cl_2	HCl/Cl_2 at 85°C, 1 bar pressure
Order of extraction	Au, Ru, Pt, Pd, Rh, and Ir separated after Ru distillation (pH 5 cake)	Ag, Au, Pt, Pd, Rh, and Ir separated after smelting, dissolution	Ru, Au, Pd, Pt, Ir, Rh	Au, Pd, Pt, Ru, Rh, and Ir separated after Ru distillation (pH 5 cake)	Au, Pd, Ru, Pt, Rh, and Ir separated after Ru distillation (pH 5 cake)

Source: Crundwell et al. 2011

Table 8 Secondary production of platinum group metals for 2013*

Source	Platinum	Palladium	Rhodium
Autocatalyst	1,275	1,860	281
Electrical	25	420	0
Jewelry	775	180	0
Total recycling	2,075	2,460	281

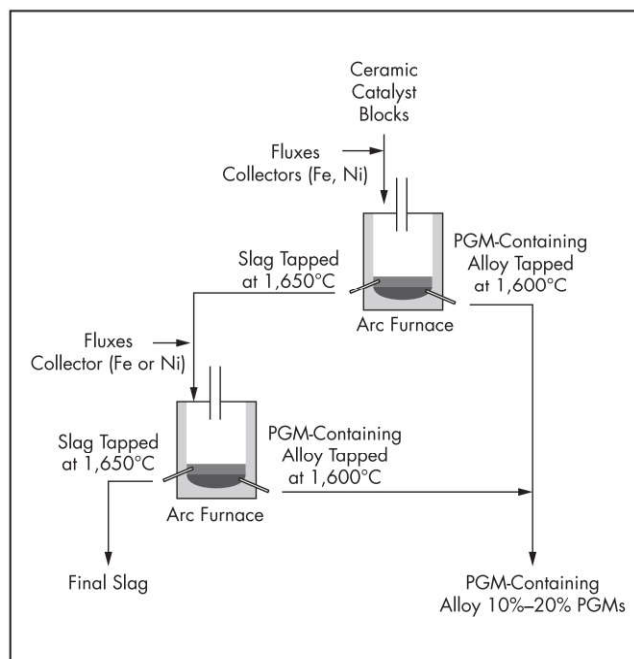
Adapted from Johnson Matthey 2003–2015b

*Units per 1,000 troy ounces.

Table 9 Recycle smelters and recycle refineries around the world

Smelters	Description
Anniston, United States (Multimetco)	Catalyst recycle smelter
Hanau, Germany (Heraeus)	Precious metals smelter
Hoboken, Belgium (Umicore)	Base and precious metals smelter
Kosaka, Japan (Dowa Mining)	Catalyst recycle smelter
Niihama, Japan (Sumitomo)	Copper smelter and refinery, nickel-cobalt refinery, precious metals refinery
Rustenburg, South Africa (Impala Platinum)	Primary platinum group metal (PGM) smelter with catalyst recycle
Stillwater, United States (Norilsk)	Primary PGM smelter with catalyst recycle
Williston, United States (Sabin)	Catalyst recycle smelter
Refineries That Produce PGMs and/or Chemicals	
Acton, England (Vale Limited)	Primary and recycle refinery
Badia Al Pino, Italy (Chimet)	Recycle refinery
Balerna, Switzerland (Valcambi)	Recycle refinery
Castel San Pietro, Switzerland (PAMP)	Recycle refinery
Chikusei, Ibaraki, Japan (Furuya)	Recycle refinery
Hanau, Germany (Heraeus)	Feed from Hanau smelter
Hoboken, Belgium (Umicore)	Feed from Hoboken smelter
Hong Kong (Heraeus)	Recycle refinery
Los Angeles, United States (Heraeus)	Recycle refinery
Mendrisio, Switzerland (Argor-Heraeus)	Recycle refinery
Neuchâtel, Switzerland (Metalor)	Recycle refinery
Newark, United States (Heraeus)	Recycle refinery
Niihama, Japan (Sumitomo)	Feed from Niihama base metals plants
Rochester, United States (Sabin)	Feed from Williston smelter
Rome, Italy (BASF)	Recycle refinery
Royston, England (Johnson Matthey)	Primary and recycle refinery
Sao Paulo, Brazil (Umicore)	Recycle refinery
Seneca, United States (BASF)	Recycle refinery
Sodegaura, Chiba, Japan (Tanaka K.K.)	Recycle refinery
Springs, South Africa (Impala Platinum)	Feed is from Impala Rustenburg smelter
Tainan, Taiwan (Solar Applied Technology)	Recycle refinery
Wayne, United States (Johnson Matthey)	Recycle refinery

Source: Crundwell et al. 2011

**Figure 20 Process overview employed at many recycling smelters for the collection of PGMs as an alloy of either iron or copper**

PGMs are deposited on the internal surfaces of a porous ceramic block, which contains between 0.1% and 0.3% PGMs, within the autocatalyst. This ceramic block is encased in a steel container. Recycling of the autocatalyst requires the steel container to be opened and the ceramic block to be extracted. The crushed ceramic blocks can either be fed to the smelter of a primary PGM producer, such as Impala Platinum, or they can be processed in a recycle smelter. A list of some recycle smelters and refineries is given in Table 9.

The typical process followed in recycle smelting is shown in Figure 20. Crushed ceramic blocks, together with a collector and fluxes, are fed to an arc furnace. Iron is usually used as a collector, but copper and nickel might also be used. The furnace is operated at temperatures as high as 1,700°C. The alloy phase and the slag phase are tapped intermittently. The slag is crushed and resmelted in a slag-cleaning furnace. Dusts from the furnaces are also collected. The alloy phase, containing 10%–20% PGMs, and the dusts are sent to the precious metals refinery.

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