

# **“Platinum & palladium recovery from Catalytic converters”**

**A practical manual for first  
time refiners**

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## Preface

### Introduction :

Welcome to the world of extractive metallurgy! In many ways the modern day equivalent of alchemy. In this manual are disclosed the methods used to recover platinum group metals (PGM's) from scrap automotive catalytic converters (C/C's).

Most people know that C/C's contain platinum; it's fairly common knowledge that in some mysterious way, the metal reacts with exhaust emissions, and purifies them, and so the planet will be saved.

That's the theory, anyway, and to this end, it was decreed, in 1992, that all vehicles manufactured from that date should be fitted with catalytic converters. This has resulted in a dramatic rise in the cost of PGM's; in 1998, the world market price of platinum stood at \$372.50 per troy ounce. At the time of writing, (early 2007), the price has soared to a staggering \$1186 per oz., an increase of over \$800.

Every platinum mine in the world is at full blast, recycling has come into its own, and still the price rises inexorably. Filter into the equation the mushrooming economies of China, and India, both heavily committed to producing their own vehicles for world consumption, and the future looks bright for those who can help supply the burgeoning demand.

Designed specifically to apply to a `small footprint` type of operation, this guide can be your introduction to the lucrative world of precious metal recovery.

Unfortunately, things have changed somewhat in recent years; by which I mean the threat of terrorism. Obtaining the necessary materials, the essential acids, required to enable recovery has become more difficult.

Nowadays, chemical suppliers can only supply acids, and other commodities vital to metal recovery operations, to bona-fide firms. Bona-fide means registered, in this context. Don't reel back in horror; registering a company as a concern primarily stated as a recycling/metal recovery business is simplicity itself. Check the ads in a publication such as `Exchange & Mart`; you'll see how simple it is. Many will still balk at this; of forming a registered company, and all the perceived hassle involved. That is their choice, but for those of you who grasp the nettle, the opportunity is there. The end result will be a reduction in the numbers competing for the platinum source; the catalytic converters. This is simple business economics; the decision is yours.

Although this manual is primarily aimed at the individual scrapper, who may simply want to know what is involved in getting at the PGM's contained in the converters, I have also mentioned the mechanical aids available to the larger, more organised recyclers. These can take your operation to

a new level; it all depends on the amount of C/C's that come your way.

If you take the plunge, and register your business, my last piece of advice is to remember the golden rule: the investment of any profits in expansion.

From here, the choice is yours, as to your level of involvement; there are still good profits to be harvested in the field, without having to register a company in order to do so. I refer to the practice of simply decanning your cats, and selling the PGM bearing innards on to a refinery concern, in the form of concentrates (cons). It has to be said, however, that the real money lies in extracting the precious metals on your own behalf. Good luck to you, whichever way you choose.

## Chapter I

### Regarding Catalytic Converter



In compiling this manual, I have ensured that every step can be performed by a person working alone, and without access to the mechanized aids available to the professionals. For those who wish to set up a bona-fide secondary refinery, I have also described the equipment required to do so. Little acorns grow into big oak trees, as the old saw has it.

The most commonly asked question has to be “How many cats do I need to produce an ounce of platinum?”, or variations on that theme. The answer is that it is impossible to quantify, for a variety of reasons; different manufacturers using different PGM loadings, the actual grade of C/C, diesel or petrol, pellet or honeycomb construction, fresh or spent, and the methods of extraction used, will all play their part.

Generally speaking, there are three grades of C/C's, in terms of PGM content; this is shown below, in which the various grades are expressed in ppm (parts per million).

Grade 1: 1200 ppm platinum.  
          200 ppm palladium.  
          300 ppm rhodium.  
Grade 2: 1000 ppm platinum.  
          200 ppm palladium.  
          100 ppm rhodium.  
Grade 3: 875 ppm platinum.  
          250 ppm palladium.  
          30 ppm rhodium.

The lowest yields of all are found in the type of C/C's known as `aftermarkets`, which are often re-used cores; cheap, spot-welded construction usually betrays them. Learn to identify them, and if they're more than a fiver, avoid them.

To further confuse the issue of what can be expected in the way of returns, the fact is that PGM composition of C/C's varies from one manufacturer to the other.

There are also basic differences in manufacture; some converters are two-way, and just oxidise carbon monoxide and hydrocarbons. More expensive vehicles tend to have three-way converters, which also remove nitrous oxide. Mercedes/BMW's, and the like fall into this category. (That's right, they're the ones you want, for stripping; so does everyone else, though.)

The PGM bearing materials usually take two forms; either a monolith of the honeycomb type made from a ceramic material, and carrying a mix of platinum, palladium and rhodium; or pellets formed from a substrate of gamma alumina, on the surface of which has been deposited the metals required. Combs are better, in terms of yields.

It is very difficult to judge possible returns when faced with a pile of ten or a dozen cats; take an overall view, based on exterior condition, which is as good a way as any of estimating whether the things are spent or fresh (fresh, or newer, is vastly superior, both in yields, and ease of extraction), whether they are pellets or H/C, (picking them up and shaking them will determine this; you'll soon learn to distinguish the difference). Any `aftermarkets` among them can usually be identified by the cheap, spot-welded construction.

Having transported them back to your work area, the next step is to open them up; easier said than done, in many cases. This process is known as `decanning`, in which the PGM bearing innards are retrieved, and preparations for recovery can begin.

## Chapter II

### Decanning your cats.

There are various methods employed in the opening, or decanning of C/C's. Most common are hand-held circular (chop) saws, used with a fibre blade; flame cutters (propane, or plasma, with the latter giving a finer cut), all used in conjunction with various hammers, bolt cutters, grips, and so on. In all cases, the correct protective equipment is an absolute must; goggles, heavy gloves, and steel toe-capped boots, if using a chop saw.

Decanning boils down to personal choice, coupled with expediency, in the end. Professional concerns often have the luxury of heavy industrial guillotines, of necessity; because they are dealing with much larger volumes than the small secondary refiner will be faced with. Even so, they bring as many problems as they solve, from what I've seen; I would still choose to have them decanned singly, and by hand.

C/C's containing pellets mostly have fill-holes, sealed with a metal plug; the simplest way to deal with these is just to drive them through with a metal punch, or a cold chisel.

After extracting the pellets (do it over a container of some sort, they go everywhere), spread them out, and go over them with a powerful magnet, in order to remove any ferrous contamination caused during the decanning process. (Metal shards, filings, etc.).

Having extracted the PGM bearing innards from your cats, you have reached the stage where many secondary refiners call it a day, and simply grade, pack into drums, and ship to a refinery, or sell to a scrap yard. This arrangement is pretty well-known; many small concerns are happy with it, and it has been going on for some time. It is even being sold as a `business plan` on the web. You simply have to obtain a

Mere twenty or so cats per day, decan them, and grade them. How hard can that be? Especially if, as the promoter of the plan advocates, you pay no more than a pittance, or nothing for them, as he apparently does. Try that approach, and see how you get on.

However, even back in the real world, if you can obtain enough cats, at the right price, merely decanning and grading them (simply keeping separate drums for pellets, or honeycomb matrix), should still show you a healthy profit. How much depends on the deal you strike with your chosen refinery concern; you can either sell direct, or take a price based on the PGM returns. This last requires a certain amount of trust between you and the refiner; and is why many will choose to strip the metal values out for themselves.

Deciding, at this point, will probably depend on the amount of C/C's you have to deal with. Obviously, cost will prove decisive; if you are fortunate enough to be at the head of the pecking

order, you will have the best chance. By that, I mean those who can come by their cats at the lowest possible price; end-of-life vehicle tow-away guys; scrap yards, exhaust centers, and so on. These people can decide their best course fairly simply. For others, cost acquisition is absolutely vital; pay too much, and it's all uphill from there.

The variables are; cost of the chemicals and reagents used, plus the time spent in refining your PGM's to the point of sale, as against the relatively simple operation of decanning.

A word of warning; scrap yards are notoriously optimistic about the amount of platinum group metals obtainable from C/C's; trust your own judgment. Not to digress; now that you have your PGM innards to hand, what's the next step?

## **Chapter III**

### **Grinding and Incineration.**

Whenever you are engaged in extracting precious metals from a bulk of material, you have to concentrate the values. In the case of, for instance, a mass of electronic scrap, you would be confronted with a lot of unwanted dross. To minimize this, incineration is used to rid the mass of everything that will burn, in order to reduce the material to workable proportions, whilst retaining your core values.

The same applies to C/C refining. Although you can only reduce by a negligible amount the actual physical mass, incineration is necessary to get rid of the unburnt hydrocarbons, and sulphur that they contain, which would interfere with the leaching process that follows the grinding and roasting of your PGM bearing materials.

There really isn't any need to grind pellets; the PGM's sit on the surface of them, and it's just unnecessary wear and tear on whatever grinding tools you use.

The ideal method is to employ a commercial ball-grinder; this will ensure a complete breakdown of material, but I appreciate that no-one taking their first steps into secondary refining will have access to one. Since you don't need one for pellets, that only leaves the honeycomb matrix to deal with. That's enough, since you have to reduce the ceramic material to a size that will pass through a fine-mesh sieve.

The minimum recommended size for ground H/C matrix is minus 60 (minus, in trade terms, means `no larger than`) i.e., no larger than sixty holes to the inch. It seems to be generally accepted that the finer the grind, the better the returns. Don't concern yourself overmuch with that; as long as your grind passes through a 60 mesh sieve, you'll be ok. Without the aid of mechanization, it can be hard work, though.

Ideally, your pre-roast, ground honeycomb matrix would resemble instant coffee, of the powdered variety, not the granulated stuff. Easier said than done, given that you aren't likely to have access to a ball-grinder straight off. The alternative is to fragment the matrix with a hammer, and then to reduce the fragments to as fine a constituency as you can, using a cast-iron mortar and pestle. Goggles, or similar eye protection, are essential if this is the way you choose.

A ball-grinder, or ball-mill, is a fairly simple piece of machinery; consisting of a drum which rotates horizontally, and into which your material is placed, along with a number of steel balls, or circular steel bars. Commercially available, but pricey for what they are; I've seen home-made ones that perform their function perfectly well, but the noise factor can be considerable. After every grind, use the magnet; ferrous contamination will adulterate the purity of your end-product.

Having reached the point where you're satisfied with your grinding results, it's time to proceed to the next stage; roasting, or incineration, to remove all unburnt hydrocarbons and sulphur from the mix.

Prior to that, there has to be a few words on your workshop arrangements. You will be working with acids (so-called wet chemistry), so care must be taken at all stages of your operation. Get into safe working mode from the word go; that is to say, use all the protection available; eye-shields (goggles, or full-face), rubber apron, and gloves.

Regulations have altered, along with accessibilty to supplies; fume-hoods are now required equipment, but they can be fabricated fairly simply. Sooner or later, you will need to visit a chemical supply company; pick one with a showroom, and take a look at what is on offer.

## The Work Area.

First and foremost, you must provide for the removal of fumes; in the initial stages, your work can be done outside, taking care to stay upwind of any fumes. Never work with any chemicals in your house, or anywhere children or pets may find access to.

A separated, outside structure makes an ideal workshop, or a garage will do, provided it can be well ventilated. You must also have a washbasin, access to running water, and a means of getting rid of fumes.

This means a fume hood, which is essential for working indoors. Here again, first-timers are

unlikely to have the odd fume-hood laying about, but a temporary arrangement can be cobbled up, by the simple use of a three-sided box, equipped with an exhaust fan that expels to the outside. Temporary is the operative word here, because the fumes expelled will have to go through the fan motor, which will be digested, in short order.

Past the initial stages, however, there is no substitute for a bona-fide enclosed fume hood, equipped with glass doors, and an efficient extraction system. If you scale up your operation in time to come, fumes become a problem, and that will be the time to start looking at a workable fume-scrubber. For now, I'll assume that you are simply at the testing-out stage, so I'll keep it fairly straightforward.

Far superior to the simple extraction fan, is a blower type exhaust system. This arrangement is as simple to construct as the ordinary fan suction type, and is a lot cheaper in the long run. It utilizes a blower to provide a suction draught, and has the advantage of avoiding corrosive fumes passing through the fan motor. The blower tube enters the hood on one side, and is set to intrude into the exhaust tube opposite, some six or eight inches. This method is known as Venturi tube extraction.

Apart from anything else, a fume hood is necessary to avoid exposing your other tools and machinery to corrosive vapours from your chemical operations. These can be destructive in the extreme; much more than you would imagine. Once you have progressed past the initial, or beginner's stage, your chemical equipment dealer is the best bet to advise on hoods, lab furniture, fume scrubbers, etc., to suit whatever size operation you intend to run.

Acids are expensive, when purchased in small quantities, but considerably cheaper in bulk, although correspondingly more difficult to handle. In the operation in which you are engaged, the requirement is for two main types: hydrochloric acid (HCl), and Nitric acid (HNO<sub>3</sub>).

Together, these form Aqua Regia (Royal Water), which is the leaching solution for the concentrates we wish to strip. Correct proportions are given later, but for the moment, I wish to emphasize the safety side, which is crucial.

Acids are highly corrosive; if mishandled, they can be dangerous. If you ever get any on your skin, or near your eyes, flush immediately with lots of water (a basin should always be kept to hand, filled, in readiness) and then neutralise with a solution base, such as sodium bicarbonate in water, which should always be kept handy, and well-labelled.

Safety arrangements in your working area cannot be stressed too strongly; both in protective clothing, and in your layout. Common sense and an awareness of the pitfalls make for a safe, secure working environment. Employ both.

## **Supplies and Equipment.**



To begin with, I've given a short list of what you will need, as an absolute minimum. There are other supplies you will find it necessary to obtain, as time goes on, but as a starter kit, the following should see you operational.

#### **Workshop Equipment.**

2 packs 25-cm filter papers (medium porousness).  
1 set pH papers: range 4/14 pH.  
1 large glass funnel (at least 5 inch) plus stand.  
Assorted Pyrex beakers and flasks.  
1 large crucible (refractory type).  
1 pair tongs.  
2/3 glass stirring rods (avoids contamination).  
Rubber apron/gloves/eye protection.

#### **Dry reagents.**

1 pound sodium chlorate ( $\text{Na}_2\text{CO}_3$ ) (sodium bicarbonate, or common baking soda).  
1 pound ammonium chloride ( $\text{NH}_4\text{Cl}$ ) (sal ammonia).  
1 pound sodium chlorate ( $\text{NaClO}_3$ ).

#### **Liquid Reagents.**

Distilled water ( $\text{H}_2\text{O}$ ).  
Hydrochloric acid ( $\text{HCl}$ ) (CP or reagent grade).  
Nitric acid ( $\text{HNO}_3$ ) (aqua fortis).

### **WARNING:**

**Never attempt to add water to an acid: when combining the two, always add the acid to the water, slowly. Water added to concentrated acid will cause the acid to react violently, spitting. Always wear eye protection when mixing the two. Allow the mix to cool down between additions of acid, in order to prevent an**

**excessive buildup of heat.**

**Never store acids in the same area as any product that may contain cyanide. (e.g., pest-control products, such as rat poison; some may contain cyanide). The addition of acids such as hydrochloric to cyanide produces cyanide gas (hydrocyanic gas), which is lethal. Take every precaution in storing your acids.**

### **The “Wet Chemical” method: an overview.**

The so-called wet chemical method used to retrieve the PGM's is known as solvent extraction, or SX. What happens is that the platinum and palladium are dissolved into the solution; the metal ions are held in suspension. Liquid ionised solution is the proper term for it, but SX will serve.

The chemical mix used is Aqua Regia, as previously stated, or Nitrohydrochloric acid. This is composed of three parts hydrochloric acid, and one part nitric acid. To this is added (by volume) four parts distilled water. Mix your acids separately; first one, then the other, with the distilled water required by volume, and then combine the two, whilst wearing eye protection.

This mixture will gasify continuously; never cap the solution, or attempt to store for long periods. Just formulate the amount needed for the job at hand. Aqua regia is a non-volatile mixture, but the gasifying can cause pressure reaction, if tightly contained. A glass-stoppered bottle is ok, but ensure that pressure can escape, by simply lifting the stopper, if not used at once. Never use a screw-capped container. Keep under the fume-hood till required, in any case, or outside.

Having organized your working area to suit, and finished grinding the combs to your satisfaction, you are ready to proceed with the incineration, or roasting. There is one final step, however; to know when the roasting is done; you must test-roast a small sample. In the case of pellets, simply place 100 gms (I use a level tablespoon), into the dent in a fire-brick, and subject to a fierce flame. (One of those small propane torches is ideal). Do this outside, or under the hood, as the stench of unburnt hydrocarbons and sulphur coming off is highly obnoxious; do not breathe the fumes; if outside, make sure you're upwind.

As you burn the pellets, note the color change; they will turn beige, or pinky-brown, or similar. It doesn't really matter; the reason you're doing a test-roast is to enable you to know when you're finished, when roasting larger amounts. Wispy, white fumes will rise as you heat your cons; stop when these cease, and note the color change. This will be the color you want for this particular batch.

Don't use a torch on ground honeycombs. For these, place the sample into a heated crucible, and stir as the fumes rise. Again, note the colour change; this is the colour you want to see when you roast the remainder of the batch.

Don't worry about losing values when roasting; platinum melts at 1773C.

Incineration is probably the stickiest part of your operation: it's far easier to accomplish a satisfactory roast with an electric oven (temperature control is better), but I appreciate that they are an expensive piece of equipment, and unlikely to be available to the fledgling refiner. Assuming that you're on a budget, however, the simplest way to roast your cons is to fabricate a 'bucket' roaster. This will serve to roast the average batch (one C/C), and is the cheapest method available.

The main thing is to stir the pellets, a lot, while roasting, making sure that a good, even roast is obtained. Continue until all the pellets are pretty much uniform in the colour you're looking for, and that the wispy fumes have ceased. As a rule, this happy state takes at least an hour, at between 800F/1000F in an electric oven (which I appreciate doesn't help if you don't have one), but judicious and careful roasting, and a lot of stirring, will give you the desired result.

For combs, you must use a crucible to achieve the same result; the finer grind means that the wire basket won't contain them. You may also find it necessary to fabricate a larger roaster for some combs; a large three-way comb can be quite bulky, and if you want to achieve the roast as a complete batch, simply replicate the bucket roaster on a larger scale. More later, about this.

Simple to construct, and effective enough for our purpose, the bucket roaster is really the best alternative available to the first-time individual scrapper. The principle is the same as a blacksmith's bellows; a forced draught to produce the intense heat needed. Until such time as you can lay your hands on an electric oven, the bucket roaster will serve to get the job done, burning off all the unburnt hydrocarbons and the sulphur, prior to leaching.

### **Fabricating a Bucket Roaster.**

In the simplest terms, a bucket roaster is just a forced-draught smelter. Easy to make, and portable, for the secondary refiner on a budget, it is an essential piece of kit.

### **Parts List.**

1 metal bucket.  
1 10 or 12 inch clay pot (a flowerpot).  
1 length of iron pipe (threaded at one end).  
90 degree elbow fitting/ a bush reducer/ threaded nipple.  
Small bag (or 2) concrete. Ready-mixed is ok.  
Wire-mesh: size 20 mesh. (20 x 20 holes to the inch).  
4/6 firebricks. (heat-reflecting type).

### **Assembly.**

Ensure that the clay pot fits inside the bucket with at least one and a half inches to spare all round, in order to leave space for the firebrick/cement insulating layer. Screw the pipe into the 90 degree elbow fitting, and then screw the threaded nipple into that. (See photo). Now punch a hole in the bucket, ensuring that the pipe has a level entry. Fragment the brick, and arrange a layer in the bottom, around the pipe. Cover the nipple with something to ensure that no dust or rubble can enter while you're placing fragments in the bottom, around the pipe. Test-assemble pot so that it sits squarely over the flange, with the nipple protruding into the hole. When satisfied that all is flat and level, remove the pot, and add concrete up to flange level; nipple should show

No more than half an inch above concrete level. (see photo). Don't make your concrete mix too stiff for this stage; after pouring, tap lightly to remove any air bubbles. Too stiff a mix makes this difficult. Leave to set, and remove protective nipple cover.

Ideally, when assembly is complete, the pot rim should be about half an inch or so above that of the bucket. This should be ascertained during the dry test assembly, in order to know where to punch the hole. This will assure a level entry of the pipe, and ensure an even air flow.

When the concrete has set, re-insert the pot, with the nipple protruding into the drainhole. Pack more crumble all around pot, up to just below the bucket rim, and pour more mix up to the desired level, again tapping lightly to remove any air bubbles. Leave to set completely before attaching the blower.

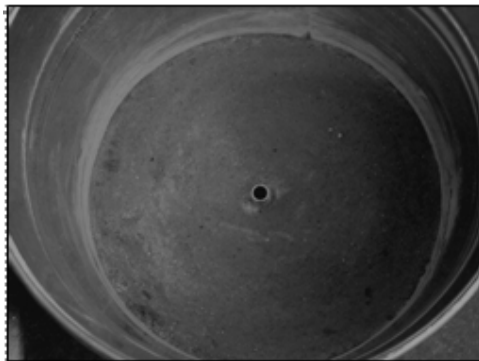
The ideal source for the air is a small leaf blower, but a vacuum cleaner with variable speeds, used on exhaust will do. If the blower pipe doesn't fit tightly over the iron inlet pipe (Sod's Law dictates that it rarely will), fabricate an adapter from sheet tin. This is simply a tube, rolled to accommodate the size you require at each end. Longer is simpler, and secure with jubilee clips. Close any gaps along the length with filler, to prevent air-loss. More jubilee clips where necessary along the length also make life easier.

All that remains to complete the bucket roaster is to make a wire-mesh container, to fit snugly inside the pot. I find the easiest way is to use a smaller clay pot (one that fits comfortably inside the main one), as a former. Aluminium mesh is simpler to form, but will need replacing sooner.

Steel mesh is the ideal: 20 x 20 is the maximum mesh size; any bigger, and the pellets will fall through. Combs must be roasted in a crucible; the mesh will not contain the finer grind.



View inside bucket, showing pipe in position

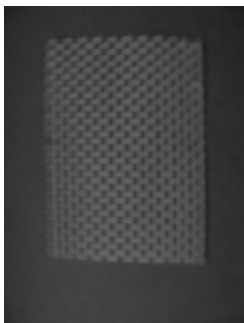


Fire-brick crumble and cement added.

Note: nipple protrudes half an inch above cement level.



Finished Pellet roaster, minus wire-mesh basket.



Left: sample of 20 x 20 wire-mesh required for basket container.

Steel mesh is ideal, but aluminium is easier to form, although it will need replacing sooner.

Roll mesh top over to form makeshift handles, or bind top with wire, and form loops on either side to serve.

For roasting ground honeycomb (i.e., the larger three-way type), then the same arrangement will apply, except on a larger scale. Sometimes it's hard to find a big enough container to replace the bucket, but a paint drum will do; you will also need a much bigger clay pot. Construct in the same way, pipe, nipple, brick-crumble, cement, etc., but the roasting container needs to be at least a 12 inch crucible.

Obviously, roasting in this way has to be done outside; and a firebrick hearth needs to be put together for holding the charcoal, or smokeless solid-fuel used for the heat-source (charcoal is best, and cheapest).

Experience is the best teacher; persevere, even if at first you run up against snags. I realize that working space is often a problem; that is, having somewhere that supports what amounts to a small draught-smelter, likely to emit foul smelling fumes on a regular basis, is often a difficult call. (At least the bucket roaster is portable, offering the choice of doing the necessary elsewhere).

I should also point out that utilising home appliances such as hair-dryers for a blower source, or using food-processors/blenders for grinding, is also hazardous, and may result in divorce.

While devices such as a bucket roaster will suffice during testing periods, should you decide to give secondary refining your full attention, there is really no alternative to an electric oven. Ease of operation, and better results are a given; the same benefits will come from mechanisation of other aspects of the procedures involved: a ball-mill for grinding, added to your electric oven, will result in a more professional operation. Fume-hoods, and fume-scrubbers are also a necessity, if you progress past the testing stage.

#### **Solvent Extraction. (SX).**

Also known as the `wet chemical` method, solvent extraction is just one of several ways employed to recover PGM`s from scrap C/C`s. Larger refinery concerns use autoclaves, (in which a cyanide solution is heated under pressure, to get at the metals), although this is becoming less common these days, because of by-product difficulties. Others have turned to gas-flow extraction, in which the material is subjected to heat, and then a non-volatile gas (there are several mixes of these), is used to transport the metal ions to a collection point. This is a much-favoured method because of the low costs (relatively speaking) involved. Apart from the initial investment in plant and equipment, that is.

Since this manual is primarily concerned with the lowest cost/ simplest method available to a small-footprint operation, I won`t cloud the issue with details of how industrial refinery concerns get their results; all that you may want to know is readily available on the internet.

For someone who wants to process smaller amounts, the SX method is ideal. You can scale up easily, if required, simply by duplicating the equipment.

To understand what takes place requires some knowledge of chemical process; put as simply as possible, it is just a leaching operation, in which the platinum and palladium are formed into an ionised solution. This is simply a liquid in which the PGM`s are held in suspension, in the form of ions. The gangue (be it in the form of a slurry, or pellets), remains at the bottom of your charge receptacle, (whatever vessel you use to perform the operation), and the resultant liquid is your ionised solution. All that remains is to separate the ions from this liquid, and re-process back to PGM`s.

### **The Leaching Process.**

A good habit to get into is to number each batch separately. I keep a small notebook for the purpose, in which I record the make and model (if known) of each cat: plus the weight of decanned pellets, or combs, before I start. Say, for example, you have six C/C`s to strip; simply label them A, B, C, D, E, and F. Use your notebook to record weights/colour changes following test burns/final weights of PGM metals obtained from each. Over a period, this small book will prove invaluable in many ways; not least in improving your judgment in assessing the value of any cats you may come across.

First thing is to mix the leach solution; the proportions are given on page thirteen, below the warning panel. Read both before you go any further. Also remember that the mixture will gasify continuously once mixed: never cap the solution, or keep it tightly contained. (See page fourteen).

When mixed, and slightly warmed, aqua regia will turn a reddish brown colour. Now add your PGM bearing materials to the solution, in a ratio of 18% solids to liquid. Do not exceed 25% solids by volume. This mixture is now heated up to boiling point, and kept there, at a steady simmer just below boiling, for at least two and a half hours.

This may seem like a long time, but remember that platinum has some odd qualities in its makeup.

It has to be heated, for chemical reaction to take place. Platinum is only minimally soluble in cold aqua regia, whereas gold will dissolve instantly, for instance.

During this extended boiling process, it is necessary to keep the solution agitated, in order to ensure complete solubisation of all susceptible metal values.

In this case, `complete solubisation` means platinum, and palladium. Rhodium is not soluble in aqua regia; I'll come to that presently.

Once again, mechanisation makes life simpler for the more seasoned refiner. They will employ a magnetic stirrer, which is simply a ceramic-topped hot plate, equipped with a motor beneath. This turns at pre-set revolutions, and the thing comes with a metal spin bar, which is placed into the charged solution above. The bar turns in accordance with the corresponding bar in the motor, via magnetism, providing steady agitation. For the first-time trier, this may not be an option, so stir very often, with a glass stirring rod.

At the end of this procedure, you should have an ionised solution, in which both the platinum and palladium ions are held in suspension. When cooler, siphon or decant the aqua regia from the gangue, and set aside. Do this carefully, avoiding picking up any sediment. The slurry, or pellets, must then be rinsed in clean water through the filtered funnel; do this in small doses, say, about thirty grams at a time, so that the funnel is no more than half full when you run the water through. When completely rinsed to your satisfaction, spread this out to dry. Best way to achieve this is to make a draining board; simply a flat panel, covered in a strong sheet of polythene, and set at a drainage angle, to allow the water to run off. When completely dry, put this aside, stored in a drum. This material contains the rhodium, for which extraction is both difficult and expensive. This should definitely be sent to a professional refinery concern for treatment. The alternative is boiling it in sulphuric acid, and undertaking a completely different method of filtering. Filter papers are no good; the acid is too strong, and will simply destroy the paper. Plus, the amount you are likely to see returned is practically infinitesimal, because rhodium is only used as a hardener for the other two. Disposal of the spent acid is also a problem you don't need.

Having said that, don't despair; the current spot bullion price of rhodium is about \$6,200 per troy ounce. Even small amounts are worth pursuing, at that price. Over a period, allow the dried gangue to accumulate and then ship to a refiner. Half a loaf is better than none, and half of any rhodium extracted from your gangue can be regarded as a bonus.

Now you have to separate the PGM's held in solution. This process is called precipitation. The chemical used to achieve this, in the case of platinum, is ammonium chloride ( $\text{NH}_4\text{Cl}$ ), more commonly known as sal ammoniac. It is essential that you purchase a good grade for this purpose; a cheap grade will produce a muddy solution, which will have to be filtered before use.

You will need a concentrated solution; try to dissolve as much ammonium chloride as possible into a small quantity of hot distilled water (a cupful, say). Bring your aqua regia back to almost boiling point, and pour the ammonium chloride slowly into it, stirring as you go. When you see a



colour change taking place, stir well, and leave to settle. The platinum will precipitate as a bright orange powder, which is ammonium chloroplatinate. Wash this powder onto a filtered funnel, and allow it to dry completely.

To recover the palladium from the solution, repeat the same procedure, using the precipitate for palladium, which is sodium chlorate ( $\text{NaClO}_3$ ). The palladium will settle as a bright red powder, which is palladium ammonium chlorate.

Now for the final stage; when completely dry, place the platinum into a glazed refractory evaporating dish, and heat strongly. Take care that the heat does not become too fierce; if the fumes coming off are white, then your heat is correct. If they are black or yellow, your heat is too strong, and you are losing platinum. (This is called 'loss to fuming', and is a refiners nightmare). All you want to achieve is to drive off the ammonium chloride, leaving the residue as a grey metallic powder, which is called platinum sponge.

The bright red powder (palladium ammonium chlorate) is then treated in exactly the same way as the platinum residue; heated carefully in a glazed evaporating dish, until the white chlorine fumes are produced. Dark fumes mean that your heat is too high, and that you are losing values.

A simple method of ensuring that the heat isn't evaporating your metal is to hold a cold stainless steel spoon over the dish. A white deposit is fine, but if a grey, or dark surface forms, then the heat is too high. The resulting residue is palladium sponge.

This is the point at which first-time refiners should call it a day. The conversion of platinum and palladium sponge into pure metal is achieved simply by the application of heat. Except that it isn't simple at all; the melting point of platinum is  $1773^\circ\text{C}$ , and that of palladium is  $1554^\circ\text{C}$ .

Never attempt to heat the metal with a torch of any kind, in particular an oxy-acetylene torch. Acetylene gas is almost pure carbon, which will cause metallic carbides to form, making the metal hard, and brittle. Also, heating the metal with a torch produces a brilliant white light, which is dangerous to your eyes: no-one should ever attempt that without using cobalt goggles for protection.

Following extraction of the PGM's, the spent acid must be disposed of according to regulations. The tables overleaf are for the disposal of aqua regia; for any other acid type disposal, you must refer to a laboratory safety manual.

### **Neutralisation & Disposal procedure for Aqua Regia.**

Equipment needed for neutralizing acids and bases.

1. Sodium carbonate (Soda ash), baking soda, or diluted inorganic base (hydroxide) for

neutralisation of an acid, or a diluted inorganic acid for neutralisation of a base.

2. Polyethylene bucket- 1 or 2 gallon size (personal choice).

(1 gallon weighs approx 8 lbs, or more).

3. Protective equipment: (goggles, apron, gloves).

4. 500 ml beakers.

5. pH indicator strips, or other pH test method.

6. Work in a fume hood.

**Warning:** Remember that extreme heat can be produced by this procedure unless it is done very slowly. Closely monitor the amount of heat produced by touching the outside of the neutralisation container.

#### **Neutralisation procedure for Acid.**

1. Make a saturated solution of sodium carbonate (soda ash) in a beaker or use an inorganic base diluted in water (1:10 ratio).

2. Put tap water into a 1 or 2 gallon polyethylene bucket.

3. Dilute acid at least 1:10 (1 part acid to 9 parts of water) by slowly pouring and stirring the acid into the water.

4. Slowly add soda ash or other basic solution into diluted acid with stirring, or save diluted acid to neutralise bases, as described below.

5. Monitor pH with pH meter, pH indicator strips, or other pH test method.

The recommended time for the neutralisation/disposal procedure is when the wastewater flow is at a peak: e.g., 9:00a.m

#### **Summation.**

By now, you should have a fair idea of what is involved in the business of extracting PGM's from scrap converters. This manual, which is aimed at the small, individual scrapper, may also serve as a sound business plan/model for larger, established businesses, already involved in some form of recycling.

Don't forget, it wasn't so very long ago that the recovery of PGM's was regarded as something of a hit or miss affair, because of the high cost of chemicals and reagents. The soaring price of platinum, in particular, has altered this perception somewhat.

Other considerations include the fact that many of the first vehicles which were liable for compulsory fitting of C/C's, (in 1992), are now at the 'end of life' stage, which translates into a lot more converters reaching the recycling market.

Even if you balk at the chemical process, and decide simply to decan, grade, and ship to a refiner, remember that they will assess your material as to worth. You will be docked for dirt and grease, as a matter of course. You can circumvent this by supplying top-grade cons: material already ground and roasted, in other words. Costs saved by the refinery will be reflected in the price of

your concentrate assessment.

Yields of PGM's are notoriously difficult to quantify, as previously discussed. Taken on a large scale, I believe a reliable estimate to be somewhere in the region of 80/100 troy ounces per ton recoverable from C/C innards. I'll leave you to do the math, but that's a lot of money, isn't it?

For those seriously contemplating a move into C/C recycling, there are other matters to consider. Where to sell, is one, and how much can you expect to receive, is another. For anyone involved in the recovery of noble metals, the bible is the 'Precious Metals Data Book', which is published by Metal Bulletin Books Ltd., Surrey, U.K. This has it all; it is a mine of information, detailing smelters, refineries, and buyers telephone numbers, along with what they wish to buy.

Another tome which is worth its weight in gold is the 'Chemical Technicians Ready Reference Hand Book', published by McGraw-Hill Book Co.. This is an invaluable, virtually indispensable aid for anyone lacking chemical bench experience. All types of lab equipment, and their use and operation are covered.

There you have it; hopefully, enough information to enable you to form a decision. I wish you luck, whichever way you decide, whether it be full-on recovery, or the simpler, but still lucrative, supply of refinery concentrates.

## Glossary.

**Ammonium Chloride (NH<sub>4</sub>Cl):** Sal Ammoniac, a water-soluble, white, crystalline powder used as a reagent.

**Aqua Fortis (HNO<sub>3</sub>):** "Strong water"- nitric acid.

**Aqua Regia:** "Royal water" Can vary, but usual composition is one part nitric acid, and three parts hydrochloric acid.

**Base:** A substance that reacts with an acid to form a salt and water only. A base is the opposite of an acid; it turns litmus blue.

**Crucible:** A refractory vessel for melting, fusing, or containing high-temperature reactions.

**Cyanide:** A salt of hydrocyanic acid (HCN); extremely poisonous.

**Decantation:** The separation of a liquid from a solid by letting the solid settle, and then carefully pouring off the liquid.

**Dilute:** Of concentration of a solute- a solution that contains a large amount of solvent.

**Dilution:** The act of adding additional solvent to a solution.

**Distilled Water:** Water that has been purified by distillation.

**Electrometallurgy:** The field of study dealing with plating, refining, extracting, and forming metals by electrical means.

**Ferric:** Containing iron.

**Ferro-:** A prefix denoting "iron".

**Filter:** A semi-permeable device for separating solids from liquids; usually paper, but not always.

**Filtrate:** The solution left after filtration.

**Gangue:** The bulk material remaining after extraction.

**Hydrochloric Acid:** A solution of hydrogen chloride gas (HCl) in water; also known as muriatic acid.

**Insoluble:** Not able to dissolve, or go into solution in a particular solvent.

**Ion:** An electrically charged atom, or group of atoms.

**Kilo:** A prefix denoting 1000. Kilogram, for example, means 1000 grams.

**Leaching:** Washing out (dissolving) a soluble constituent from an insoluble matrix.

**Litmus:** Turned red by acids, and blue by bases; used as a pH indicator. Normally used in paper strip form.

**Metallurgy:** The science and study of metals.

**Neutral:** Neither acidic nor alkaline.

**Nitric acid (HNO<sub>3</sub>):** A colourless, corrosive, water-soluble acid.

**Ounce (avoirdupois):** A weight equivalent to 28.3 grams.

**Ounce (troy):** A weight equivalent to 31.1 grams.

**pH:** A measure based on hydrogen ion concentration in a solution; a key to the relative acidity, or alkalinity, of the solution.

**Precipitate:** An insoluble substance formed in a solution as the result of a chemical reaction.

**Pyrex:** The trade name for a heat-resistant glass widely used in chemical ware.

**Reagent:** A chemical substance used to bring about a reaction.

**Refine:** To purify.

**Refractory:** Able to withstand extreme heating without damage or physical alteration.

**Saline:** Substance or solution containing salt.

**Silicate:** A salt of silicic acid (H<sub>2</sub>SiO<sub>3</sub>). Silicates are usually considered to be composed of silica and the oxide of whatever metal is present.

**Sodium Chlorate (NaClO<sub>3</sub>):** A water-soluble, colourless solid.

**Solute:** A substance that dissolves in a solvent.

**Solvent:** A fluid having the power to dissolve solutes.

**Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>):** Vitriol, or oil of Vitriol.

**Toxic:** Poisonous.

**Valence:** The chemical combining power of an atom.

The above glossary is limited to terms that you may encounter in this manual, or that you may come across in general research; it is by no means complete. Fuller knowledge can be accessed by the acquisition of a chemical glossary, or a technical handbook.