FORUM BOOK #2 FINAL (updated links for phpBB3)

Gold Refining Community
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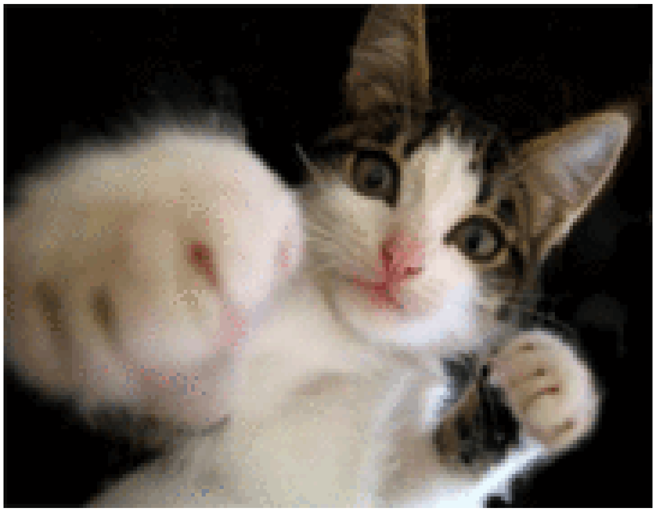

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REV: 6-24-09

LINKS

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Testing for silver

Making Sodium Nitrate ?

Crucible

RH Test

Gold Prospecting in Ireland CD

CHEMISTRY (GOLD REFINING) HANDBOOKS

GOLD REFINING BOOKS ALL KINDS INFO

Gold Refining Forum Chat Room
http://www.goldrefiningforum.c.....shchat.php

Where to sell your gold
http://goldrefiningforum.com/phpBB3/viewtopic.php?p=9998&highlight=#9998

Flatpacks
http://goldrefiningforum.com/p.....=flatpacks

http://goldrefiningforum.com/p.....mp;start=0

http://goldrefiningforum.com/p.....=flatpacks

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Burning mercury off in nitric Acid? Danger !!!!

Recovering precious metals from polishing sweeps

Making Nitric acid question?

Incineration

Checking black sand for Au

Separating gold from black sand/useless particles

Washing the gold powder after using SMB to drop

General Waste Disposal

Refining Waste Disposal

Stannus chloride formula and use

Gold Fingers

Perioxide & HCL color (AP)

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Where can I find smb(sodium metabisulfite) (not sulfate) ?
http://www.chemistrystore.com/sodium_metabisulfite.htm

SpreadSheet Calculator (For estimating gold content value$$)

486 Yields

Plated CPU tops and bottoms Yield

Multi Layout Periodic Table >>>> Download

Electromotive Series

What are Ishor's chemicals

How to estimate the amount of gold in electronics and jewelry

What to pay for gold plate and gold filled?

Chemical for circuit boards (to remove soilder mask)

Refine gold plated, filled or rolled products.
http://goldrefiningforum.com/phpBB3/viewtopic.php?p=20845#20845

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Stannous Chloride... POWDER (???) ***How to make***

AP using high percentage peroxide- BE CAREFUL! ***AP Process***

Testing with Stannous

Making Sodium Nitrate ? ***From Calcium Nitrate***

Concentrated Hydrogen Peroxide and Pins

Digesting Gold Plated Stainless Items in Nitric

HCL - clorox

Maximum metals solubility in Aqua Regia ?

SMB mixing

Dropping list???

Processing anode slime from parting cells?

Gold Recovery from PINS, How to do it?

Clorox Acid Experiment - Help

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Question concerning the History of the AP process

AP. What Did I do Wrong?

Ap Process Help

Preview of Electrolytic Process- Equipment List Shor

What chemical will precipitate gold from ammonium chloride Shor

Evaporating Auric Chloride

Removing stones from scrap jewelry?

Aqua Regia process with nitric acid

How to refine kt gold (for dummies)

Final ammonia water wash necessary ?

Iodine 7% Leach for Gold

Clarification on moles and acid Concentration

Silver cyanide reduction

Is gold bromide reduction feasible using SMB?

Making Nitric acid... what did I do wrong?

Removing lead from parts before processing

Percipitate questions

Stannous Chloride Testing Solution

Iodine gold leaching

Fume scrubbing

Rhodium Tests.

Testing for Rhodium #2

Refining 10kt and 14kt Gold

Concerning Nitric..and jewelry

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Iodine solution questions

Concentrate Nitric Acid ?

Which would be the best/most inexpensive way Stripping gold plate

Diluting Stripping cell acids

Refining electroplating solutions

Making Nitric acid questions

[Electrolisis] First cycle: disaster! Hundreds of questions shor experiment.

Should i drop the Palladium first before the Gold?

Reuse of (AP) HCL

Refining gold sulphide ore concentrate

Cell Acid Processing questions.

Silver Cell

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Help! New to forum! Have media from plating operation

Making Stannous chloride from 95/5 plumbing solder?

How to clean a crucible or melting dish.

AR pH value before dropping gold

unpopulated telecom boards

Is this Palladium?

What to refine in a computer ?

SMD Identification *(Monolithics)*

Solder Mask Removal

Can potassium metabisulfite be used as a precipitant?

Alchemy and The Big Con *Alchemistry vs. Chemistry*

Do you use Butyl Diglyme ?

Urea ➡ http://goldrefiningforum.com/p.....hp?id=2019

-

Palladium Precipitation Test: Comments and Questions

Steve,
How long do you think saturated ammonium chloride solution will last? Could you make a large bottle before hand to have ready?

Jim,

Absolutely, since ammonium chloride is a stable salt it will keep indefinitely. You will need to make sure the bottle is sealed air tight when not in use to keep the water from evaporating. If the water evaporates just add enough water so that the solid ammonum chloride just dissolves again.

Steve,
I dissolved a small amount of the palladium wire that I got from organs and got a blood red solution with nitric. My question is do I have to nuetralize the nitric before I use ammonium chloride and sodium chlorate? Or do I dilute it? I am probably going to add the 6 grams of tiny little wire from the contacts also so I have some more to work with. Right now there is probably a gram or less in solution. I just want to see what the results are from pure palladium before I try any scrap from electronics. I also would like to make sure the palladium wire is pure, if it is it will be better to just keep it as it is or melt it without refining it, do you agree?
Jim

Jim,

The precipitation reaction should work with your nitirc acid solution. Harold or Chris need to confirm this one for me. I have only worked with Palladium in AP solutions. The Palladium Ammonium Chloride drop as the proper levels of ammonium chloride and sodium chlorate are reached in the solution. Concentrating the solution is still a requirement.

As a side note, the Palldium could be cemented from the solution using copper or silver metal once all the free nitric acid is removed via evaporation. I don't think the Pd from this method won't be as pure as that attained from the above reaction.

Steve

I'm of the opinion that you should evaporate the nitric----but it's just that, an opinion. I am not basing it on any chemical knowledge, but I'd not be too pleased to work with the salt if it was contaminated with traces of nitric, which it will be. In spite of a fair amount of washing, it's pretty hard to totally eliminate almost anything that's combined with the salt, and palladium salts don't lend themselves to much washing without dissolving to some degree. If you were to ask my advice, I'd recommend you evaporate the solution. The choice is yours.

I question that it will precipitate with nitric present------so if you give it a go without eliminating the nitric, I'd appreciate hearing the results. Hoke may have commented in that regard, but it's been too long for me to remember, and my copy is not available to me at this time.

Do keep in mind that palladium won't precipitate from a dilute solution. This point can't be over stressed. The more concentrated the solution, the better the results. As I recall, Hoke calls for an ounce of ammonium chloride per ounce of palladium expected to be recovered, and an excess is advised.

Harold

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Harold,
I have been using the smallest amount of nitric possible to dissolve the palladium. I just added the 6 grams of contacts to that, and will see if it is dissolved in the morning. If not I will add a little more nitric, I am trying to keep it as full of palladium as I can.
By evaporating do you mean heat it untill it is like syrup? If so how do I get it back to liquid without making it a dilute solution? Steve mentioned that the salts are water soluble. Do I add ammonium chloride saturated water?, and then sodium chlorate? With heat.

Thanks. Jim

For starters, you'll have better luck using AR instead of straight nitric. Be careful if you started with just nitric----it's likely to come alive very quickly when you introduce the HCL, but that's what you'll have to do. You won't believe how quickly palladium dissolves that way.

While I don't know that I can say what constitutes a good level of concentration, it wouldn't be out of line to expect a liter of solution to contain two, maybe three ounces of palladium. Think accordingly. When I refined gold, I'd generally run my concentration level up around four ounces/liter (when re-refining).

You can have an abundance of HCL and not have problems. Keep that in mind. The worst problem it creates is that it can cause filter failure when too heavily concentrated.

Fact is, one of the purification processes for further refining the red salt is to dissolve it in ammonium hydroxide, then re-precipitate it with nothing more than HCL.

It's hard for me to know what you have at your disposal, so what I'll do is tell you how I did the job-----and you can pick and choose methods that will lead you down the same path.

Using an evaporating dish helps the process go faster, but I eventually moved away from them because I had limited burner space and could fit more beakers on the hotplate. Choose what works best for your case here.

Evaporate the solution at a low heat----do not boil it----and do not allow vapors to come off that have color. Hold a cold object (a porcelain spatula works well) in the "steam" coming off to insure that only clear fluid condenses. As the solution evaporates, it will get darker and darker, eventually turning a blackish brown color, and thickening. It often will start forming a hard edge, but it's best that you not let it get that far, but if you do, it's not fatal. If in doubt, and it's gone too far, drip in a drop of nitric and a few drops of HCL, which will re-dissolve anything that may have gone too far. Don't go crazy with the nitric-----remember----you're trying to get rid of it. As it thickens and darkens, drip a few drops of HCL in the solution. When it's that concentrated, if you have free nitric, you'll see those familiar brown fumes come from the beaker. If, when you add the HCL, you see no fumes, and it's hot and very dark, you can stop the evaporating process at any time-----for the material in question is very water soluble.

Dilute it enough that the acid is not heavily concentrated, and stop. You'll further dilute the solution when you wash down the filter. It helps to wet the filter well before you introduce any of the solution. Once fully filtered, wash the filter with tap water until you're happy with the amount remaining in the filter. If you try for a perfectly clean filter, you'll have added a lot of water, which can be troublesome when it comes to precipitation. The filter can be incinerated and values recovered later, so don't worry about leaving traces behind. You can also toss it in your stock pot and incinerate it with the recovered sludge. The values are not lost if you do things properly.

If you find that your solution is too dilute, there's nothing wrong with heating it to evaporate water---just give it time to do so, and keep it clean, so it doesn't require filtration again.

If you know your solution contains no platinum, if necessary, evaporate until it's quite dark again, then introduce the dissolved ammonium chloride (filter it if it's dirty). You can't recover the palladium when the solution is cold----it's done right under a boil. Heat the solution until it just starts boiling, then dribble grains of sodium chlorate in. Not too fast, they often boil up on you and can easily overflow the beaker. Scratch the side of the beaker with a glass rod if you don't see a change in color, and tiny specks of red salt starting to form. Sounds crazy, but they often seed where you scratch the beaker. Make sure the one you use is no more than half full, and work slowly with the sodium chlorate. Once you've seen it done, it will make sense and you'll have a general idea of what works, and what to avoid.

When you introduce the ammonium chloride solution, if you have platinum present and it's in the right concentration, you get an instant cloud of platinum salts. It's a real fun thing to watch happen. Should you find yourself in that situation. allow the salts to settle well, then decant the bulk of the solution to another beaker, and filter the last bit, combining the solution with the decanted solution for precipitation of the palladium.

From this you probably understand that when you have them both in solution, you recover platinum first, always, using this system.

A tip on dissolving ammonium chloride. The reaction is endothermic, so requires heat to work. Start with hot water (tap water is fine) and use less than you think you'll need. Allow it to dissolve everything it will, and stir regularly. As the solution gets concentrated, you'll notice it is now cold. Stir a little longer to insure that it won't dissolve any more of the ammonium chloride, then you can pour off the solution and introduce a little more hot water to the remaining material. Do this until all of it has dissolved. By following this advice, you'll create a heavily concentrated solution, keeping the overall volume small.

One more tip. An Erlenmeyer flask is cute as hell, but it will get you in trouble faster than any other container I can think of when you have a situation at hand that can boil over. There's no room for any expansion, so the slightest problem guarantees it will overflow. I'd suggest you not use them.

Hope this helps.

Harold

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It stands to reason that you'd avoid what I said about using HCL (AR) if you get involved with an alloy that contains silver. You'd find yourself in the same position as when trying to dissolve gold that has silver within, whereby the silver forms a hard crust of silver chloride and the action coming to a stop. I should have been a little more specific.

In such a case, you may find yourself having to deal with silver chloride. Otherwise, it's hard to separate the silver from the palladium. Once dissolved, you'd precipitate the silver as a chloride, then wash the palladium out with ample water. It's a lot of work, but I don't know of another way to separate the two. It's not even a good idea to do it in a silver cell because the ratio of palladium to silver is too high and that creates problems in the cell.

I've been through this exact thing when processing sludge from a silver cell, where you have a high percentage of silver mixed with other values, so if you'd like to talk about it, feel free to ask. No sense me going on about it if it serves no purpose.

Harold

Harold,
The wire from the organs is pure palladium. I added AR to that solution and it disolved the rest of the contacts. I used your calculations for the volume, and transfered it to a clear pyrex coffee pot, that should make heating easier. I will probably do it tomorrow, following your directions as the weather will be nicer tomorrow. Today I did a batch of processors in poormans AR, so today was a gold day, and I made up some saturated ammonium chloride and put it in a wash bottle to be ready to do the palladium precipitation.

The rings I know are a different story, I shouldn't confuse the topic by bringing them into it like I plan on doing the same thing with them.
I planned on using straight nitric for the precium ring as that is 75% silver, as I know the silver will cause problems, although isn't it inquarted by being 75% silver already? If I drop the silver out as silver chloride with salt, that should leave me with the pd in the nitric solution. Could I just save that as my standard palladium test solution? I still need to save some gold as a standard to test my stannous before using, I have been making it up when needed.

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The palladium/plus rings are unknown alloy, but I am thinking they are 90% or 95% palladium with ruthenium or maybe silver. These I plan on trying AR with. I will try one first before I do any others as a test.
Thanks. Jim

jimdoc wrote:

> although isn't it inquarted by being 75% silver already?

In a sense it is, but palladium requires no inquartation under normal conditions. If you had a batch of nothing but impure palladium that was contaminated with a trace of silver (solder, perhaps), you'd be able to dissolve it directly in AR, or even nitric if you don't mind it working slower.

Quote:

> If I drop the silver out as silver chloride with salt, that should leave
> me with the pd in the nitric solution. Could I just save that as my standard palladium test solution?

It would work, but you're working with an unknown solution. When you build standard solutions, they should be made to rigid guidelines, so the reaction you get is constant and predictable. They are often used to make comparison tests to help determine the level of concentration in a solution. That can be important when there are other metals, often not of value, that are effecting the color of a solution and you don't know what it may be. Palladium, far more so than either gold or platinum, reacts differently with different levels of concentration-----right down to different colors being displayed. I've had it confused with gold, but with brown and even green highlights in the test. It's really screwy, at least that was my experience over the years. With that in mind, I'd advise that you make a standard solution from pure palladium, to a formula that you repeat when you have to make new solution. If you know for sure the wire you've been processing is pure----what better place to find some? You don't need much-----can't recall what Hoke had to say about it, but I did everything as she directed. It sets a standard for you so your tests are meaningful.

Quote:

> I still need to save some gold as a standard to test my stannous before using, I have been making it up when needed.

I trust you do the same thing with your gold solution. If you know the precise reaction you should expect, it, too, can help you determine if a solution is concentrated, although not nearly as well as with palladium. For sure, it's a great way to know when your stannous chloride is dying. I used to mix mine @ 2 ounces, plus I'd add some pure tin as Hoke suggested. About the time it was hitting the bottom of the dropping bottle, it would fall off. Likely because it was oxidizing quite rapidly because of the low volume. When I'd test some gold, it wouldn't show much, so I'd know it was past its useful life.

Quote:

> The palladium/plus rings are unknown alloy, but I am thinking they are 90% or 95% palladium with ruthenium or maybe silver. These I plan on trying AR with. I will try one first before I do any others
> as a test.

That's exactly what I'd do, too. I remember processing one of the new alloy class rings, but I was on my way out of refining before they started getting available to any degree, so I have no experience with them aside from that one, and I don't remember much about it. I'd like to hear what comes from the first one if you have time to post a report.

Harold

IODINE LEACH - What Kind?

Hi,

what kind of iodine can be used in an iodine leach?

I know it should technically be:

Iodine crystals; iodine sublimed; iodine bisublimate CAS No.: 7553-56-2

but since this kind of iodine are also being used to make prohibited substances, they are hard to come by. Can we use other forms of iodine instead?

Thanks

Alberto

Sodium or potassium iodide will work.

AR Nightmare with Shore

In the last 2 weeks we've run 3 batches of gold bearing electronic scrap based on the Shor Muriatic/Subzero method. Nothing has worked as advertised. We are now having better luck with the acid/peroxide method and have our 1st finished gold in hand. The big question is , what do we do with the 5 gallon pail of Aqua regia loaded with dissolved metals sitting in the corner? There is quite a bit of gold in there, along with copper, silver, solder,etc. How do we get our values out...? We've learned our lesson and made lots of mistakes early. We feel the AP and AC methods on Lazersteve's website have been great...!

Welcome to the forum,

Here's a quick overview of a test process:

- You should test the AR to be sure you have gold.
- If you have gold then separate out a small volume of the solution to test with.
- Filter out any solids from the test solution.
- Add urea until the pH is 1, **or** evaporate the solution. If evaporating, add HCl and evaporate again. Repeat evaporations until no brown fumes are evolved.
- Add enough water to double the volume of the solution.
- Dissolve in water an amount of SMB equal to the expected amount of gold in the solution.
- Add the SMB solution to the test solution with stirring and observe if a brown powder drops after the SMB is added.

Let us know what happens from here.

Steve

Below are the steps I performed on the AR batch last evening...

1) Removed spent circuit board material from solution.
2) filtered solution through several coffee filters to remove solid debris.
3) added pinch of urea and observed no reaction.
4) filtered solution 2 more times to remove any potential contaminants.

5) estimated 4 ounces of dissolved metals in solution
6) mixed 6 tablespoons of SMB with hot water and poured into solution.
7) Waited 1 hour and poured entire bucket of solution through coffee filter.
8) There was no mud or solid debris in bucket or filter at all.
9) Waited until next morning , still no precipitate.

This has been our experience on all three batches of AR we've run. The Shor test liquid shows plenty of gold present in solution.

---

unexpected precipitates - help identify

So I completed my first batch of fingers all the way through, and now have a very small gold BB to show for my efforts! Not much, but not bad for a first attempt with a small amount of source material.

I have left over about 4 liters of spent acid peroxide solution, with is a dark green color.

Just to make sure no gold had dissolved into the acid, and since I do not have any tin to make stannous cloride, I went ahead and put SMB in the green liquid to see what might drop, and left it overnight.

This morning, we have a small amount of a silvery-gray precipitate, which I filtered out.

First question - what is it, and is it silver in some form?

Second, I figured I would go ahead and drop the copper from the solution, so I put into the solution two pieces of metal from a computer that I thought was a mild steel, but now think might have been aluminum.

Immediately upon adding this metal, the solution reacted violently with a strong fizzing and a very distinct sulfur smelling gas discharge.

After a few hours, I decided to remove the metal, which was covered in black residue, and filter out the black precitate. It is clearly NOT copper.

What is this black precipitate? Could it be gold, that was dropped by the addition to the aluminum to the acid peroxide/copper/SMB solution?

Any insight as to what this is would be helpful!

-David

Dave,

Congratulations on your first nugget!

Now on to your questions and a few comments.

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First, you can reuse AP if you just bubble air thru it for 24 hours or add enough peroxide to lighten the color back to transparent green. If gold is dissolved it will eventually be pushed out as the copper content increases in future batches.

You should buy some of the pure tin metal I sell on my website so you can test all types of solutions for gold, palladium, and platinum.

Never add more SMB than you expect gold to return from **copper chloride** soluitons. If you suspected a gram of gold in the solution, then add 1-2 grams of SMB to drop the gold. Excess SMB can react with the copper in solution to produce solid by products especially if water is used to rinse the precipitated solids, which can lead to copper contamination. To avoid this use minimal amounts of SMB and always rinse throughly with HCl before rinsing with water.

Gold will drop with SMB as a dark brown to almost black powder and it sinks to the bottom. Sometimes it may appear tinted purple if finely divided. Always test small batches before loading up your solution with more chemicals. You may have been better served by separating a small volume of the suspect solution and adding a small dash of SMB, if nothing showed up by morning, you should have moved on to regeneration or disposal of the main solution. The silvery gray percipitate could be contaminants from the fingers, especially if they came from slotted cpus. These fingers tend to get fouled with the heatsink compound used on the cpus which forms a silver gray sludge.

Silver will form silver chloride in the inital reaction and end up as a white powder that darkens to a purple gray when exposed to light, nothing except HCl and dissolved silver is required for this to occur. If you filter your solution BEFORE you add any SMB or scrap metal you can be sure no solids are present prior to adding the scrap metal. Typically fingers **do not** produce any silver chloride. Silver chloride is soluble in hot household ammonia if it ever ends up in your gold and you need to remove it. You can test your suspect powder by trying to dissolve a small amount in ammonium hydroxide (household ammonia). I doubt it's silver chloride, but who knows.

The second precipitate sounds to me like an aluminum compound which commonly forms in the presence of HCl. Copper (II) oxide is a black compound also. You should find some pc case part that you know is iron and put it in the solution, if a spongy dark red moss forms on the iron in 10-15 minutes that's definitely copper. This will happen without the violent reaction as you described. Some cases have a galvanized zinc layer over the steel which intially reacts quickly but tapers off very soon after adding the scrap. You described what appears more to be an aluminum and HCl reaction.

To sum everything up:

- Test small samples before commiting to a larger reaction.
- Identify your unknowns

- Think clean..
    - Fewer chemicals in the mix
    - **GIGO** (**G**arbage **I**n **G**arbage **O**ut)
    - Reuse when possible

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Let us know how this turns out.

Steve

____
The proof is in the pudding.
http://www.goldrecovery.us

Remember also that gold reacts with copperas, and platinum does not. Therefore, if your stannous chloride test is confused, it may be helpful to repeat the test using copperas instead.

GOLD AND PLATINUM HYDROXIDES

Hi,

I used sodium hydroxide to precipitate gold and platinum from a polycarbonate solution. I have the precipitates which look like dark colored powders.

Can I use nitric acid or aqua regia or muriatic acid / clorox or muriatic acid / hydrogen peroxide to dissolve these hydroxides and then start parting them into their individual metal forms?

Thanks

Alberto

Alberto,

The metal hydroxides are not purely the precious metal as an hydroxide, they can also be combined with the Sodium ion from the sodium hydroxide depending on the reaction conditions. For this reason you may have mixed results with the dissolving reagents you mentioned.

Heating is all that is required to reduce the hydroxide precious metal compounds to the native metal forms in some cases.

A second drawback to using lye as a precipitant is that it drops all the metals in the solution without discretion. This can lead to impurities if your source solution was contraminated with base metals initially. This is because sodium is near the top of the activity series. For this reason you should avoid using bases that are high in the activitiy series to drop PMs from solutions.

The pure metal hydroxides will most likely dissolve as you have asked, but will require more reaction acids to overcome the basic conditions again wasting your resources.

In short you are adding extra steps and chemicals to the process by dropping the PM's with lye. Try to eliminate steps that don't purify the target metals as these steps merely waste your resources and don't work to purify your PM's in the process.

Steve

Silver chloride and ammonia

How do you recover all the silver from an ammonia solution you have used to dissolve silver chloride (used in refining of karat jewelry)?

Thanks

Alberto

The silver ion formed in the reaction is Diamminesilver (I) otherwise known as Tollens' Reagent. Adding Karo syrup to the basic solution and gently heating will return the elemental silver. You can make a silver mirror using this very same reaction if done properly.
Steve

Thanks for the answer Steve, I appreciate it.

as a follow up,

I read that ammonia and silver chloride when mixed and dried can create something explosive. Is this true?

If so, then I guess I have to use Karo syrup to recover the silver right away before anything dries up. Please confirm.

I am always wondering if sugar syrup instead of corn syrup (in Karo syrup) can be used instead. Please confirm.

thanks for all the help

Alberto

Alberto,

Yes silver chloride and ammonia will form an explosive mixture if allowed to react for a long period of time.

You can use sodium hydroxide to convert the silver chloride as well. If ammonia is used be sure the solution is made basic before adding the Karo.

Sugar syrup will not promote the conversion.

Steve

Over the years, I have witnessed several explosions created from ammoniacal silver (azides can form) and gold solutions. Elsewhere on this forum, I suggested that, immediately after dissolving the silver chloride in ammonia, that one should acidify the solution with HCl. This re-precipitates the silver chloride, which can then be treated with Karo syrup and NaOH. **Never let the ammonical silver solutions sit around without acidifying first.**

Since witnessing these explosions, some 40 years ago, I have never used ammonia to dissolve silver chloride, except for using it as the final leach for pure gold powder.

Karo syrup (corn syrup) is glucose. I think that some other types of sugar can be used but, experimentation would be required. For 31.1 grams of silver chloride, it takes 10 mL of Karo syrup, 15 grams of sodium hydroxide, and about 100 mL of water. The sodium hydroxide converts the silver chloride to silver oxide and, then, the Karo syrup converts the silver oxide to silver metal. Chris

GSP,

On the subject of exploding silver compounds.. do you know of a way to render the ammonical silver to a safe state if you have formed any? Is acid addition a viable option after the explosive silve has formed or only prior to it forming?

Thanks,

Steve

I really don't know the answer to that, Steve. I think that once you've made it, you're stuck with it but, I may be wrong. I decided to never use ammonia, except for the final cleaning of gold powder. Silver azides form only under certain conditions but, I've never gotten a straight answer as to what those conditions are.

You can also use sodium thiosulfate (photo hypo) to dissolve silver chloride. Zinc dust or steel wool can then be used to drop out the silver. Separating the silver from the excess zinc or iron is another story. Chris

---

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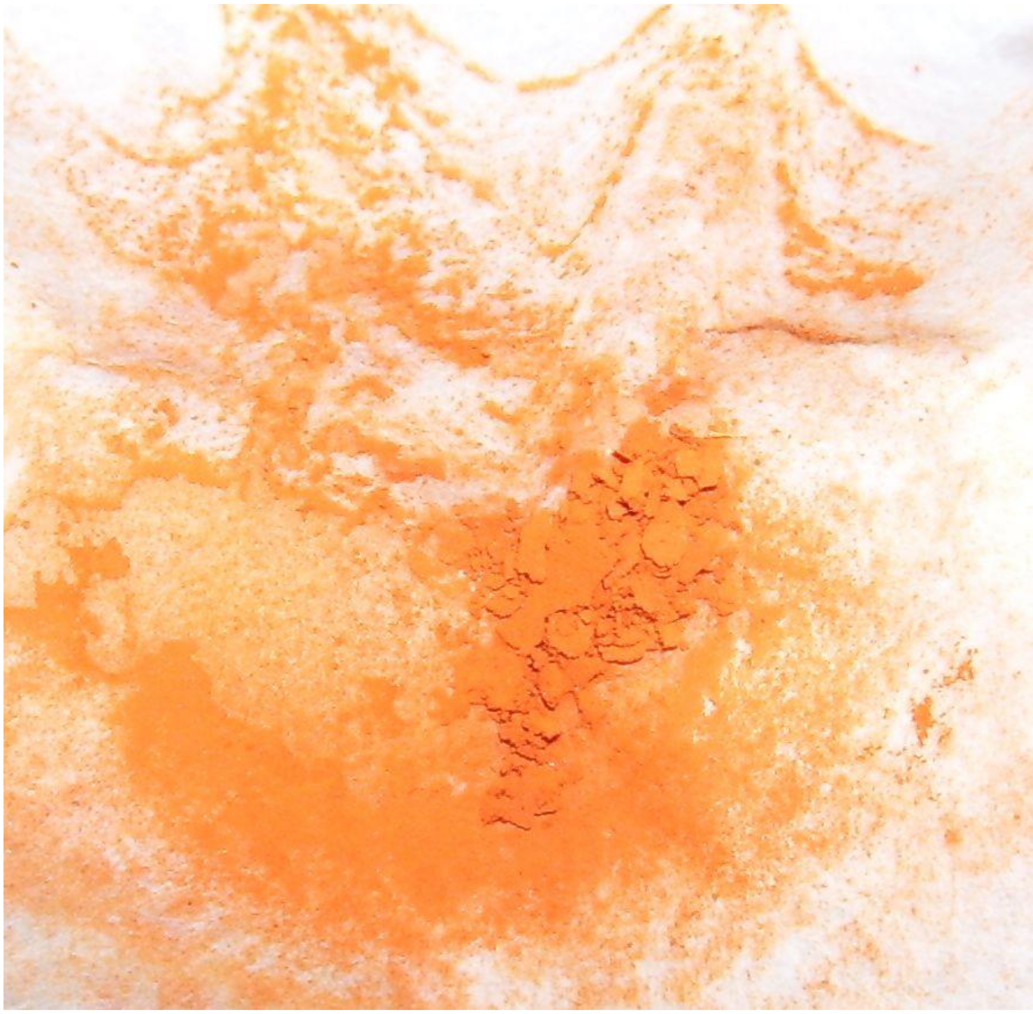
Identify Suspected Platinum Salt

Last night I tested a small sample of metal that I suspected to be Platinum and precipitated the AR solution with ammonium chloride. This is the salt that precipitated.:



Can anyone confirm the orange color of the salt as Platinum Ammonium Chloride ?

Are any of the Palladium Ammonium salts orange?

I hunted the internet for a good photo of the platinum salt and found nothing.

Thanks in advance,
Steve

Steve,
In the Precious Metals Science and Technology book from IPMI they show a color picture with crystals on the top and solutions on the bottom. The platinum chloride and platinum chloride solutions look very similar. With the crystals the palladium looks a little more red or brownish and the platinum an orange-red. It looks like it may be platinum in your picture to me, but palladium is also a possibility,and I think Harold or GSP will be the ones that will know. Jim

In picture left to right are; silver nitrate, gold chloride,platinum chloride,
palladium chloride,rhodium chloride, iridium chloride, ruthenium chloride,
and there crystals on top.

are brazed in vacuum at 950°C

act of gold-silver/silver-palladium/

Crystals (top) and solutions (bottom) of various precious metal compounds. From left to right: silver nitrate, gold chloride (chloroaurate), platinum chloride (chloroplatinic acid), palladium chloride, rhodium chloride

The only color variants with palladium are when you precipitate with DMG, or when you dissolve the characteristic red salt with ammonium hydroxide, and re-precipitate with HCL, at which time it comes out a yellow color, but much lighter than that from DMG. I posted a picture some time ago that shows each of them. You'll find it in the Pt group section of the board.

Regardless of the degree of purity of solution, when I precipitated palladium, it was always red, but varied in depth of color. It would often be a much darker red, which I attributed to the dirty solution. It would brighten with washing.

The color of your platinum salt can and will vary widely, which I think you can confirm in Hoke's book. I noticed a distinct relationship between the cleanliness and concentrations of solutions to color. When I re-refined platinum the few times I did so, the salt came down a nice light yellow color. It was often an orange color when precipitated from dirty solutions.

Take a small sample of your salt and test it with stannous chloride. I'm certain you'll see a platinum reaction (coffee color). Dissolve the tiny sample with a touch of water before testing.

Harold

Randy,

Here's how I made the orange powder:

1. I dissolved a very small amount of the suspected Platinum metal in a test tube of hot AR.
2. I filtered the orange colored solution and rinsed the filter throughly.
3. I added approximately a 1/4 teaspoon of solid ammonium chloride
4. I evaporated the solution with heating until the orange powder formed along with solid ammonium chloride crystals.
5. After cooling, Iadded, dropwise, just enough distilled water to dissolve the solid ammonium chloride.
6. I filtered the orange powder out of the solution.

Steve

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GENERAL

HI, Just joined this great site.
I have been running some small test batches of AR.
First I dissolved 50g of gold fingers (cut from pcb's) in a 10ml nitric/45ml HCl AR mix, worked great every time.
I then filtered solution and added urea till no fizz the quantities ranged from 15g urea to 31ml water up to 40g urea to 80ml hot water.
I then filtered solution again.
The amount of dissolved metals ranged from 6.16g/50g fingers to 7.2g/50g fingers.
Now i added smb. I started with 1g smb to 1g dissolved metals and went as low a 0.6g smb to dissolved metals.
Now onced dropped a white precipitate formed on top of mud and in water. I washed with HCl afew times then water many times, the white disappeared and left mud, now the colour of this mud ranges from golden/light brown to a dark brown/purple. I tested all solutions and they were negative for gold, but the last few batchs where i have used less smb, no white forms but a molten looking gold layer on top of solution forms (won't sink) and dark brown/purple mud at bottom. but every time i test the solution the top molten layer sticks to rod and thus test is postive for gold. Its hard to get to clean solution to test and the sides of my beakers are covered in this same golden/brown so i can't see clarity of solution. Is this what happens or have i done somthing wrong?

I cleaned these last batchs anyway thru repeated filtering and water washing, there seemed to be lots of it for a small amount of scrap, burt once i dried over low flame it shrank dramatically and left fine dark brown cracked mud like web, which peeled off or moved easily, once turned over it shined a gold like colour in the light, weighed in at 0.39g.
Now the other first batchs cleaned with HCl all left about 0.19g-0.23g of light to dark brown mud. Do these results seem like I'm on track and going in the right direction ?

I Plan on putting all these batchs thru a 2nd AR process.

And finally what is the best way to determine the amount of smb needed ?

Thanks Damon
(I come from a land down under)

Damon,
Welcome to the forum.

Your best bet with such small batches of fingers is a treatment with acid peroxide to remove the base metals first. I typically recommend processing a minimum of 1 pound of card fingers in a batch of AP. After AP the foils can be dissolved with a small amount of HCl-Cl or AR whichever you desire. With the HCl-Cl method there is no need to use UREA.

Your descriptions of the experiments sound very normal for AR reactions. The problems you had with the powder sticking could be contamination in your reaction vessel. When using AR I mix up a solution of SMB that has at least 1 gram of SMB per gram of gold expected in the batch.

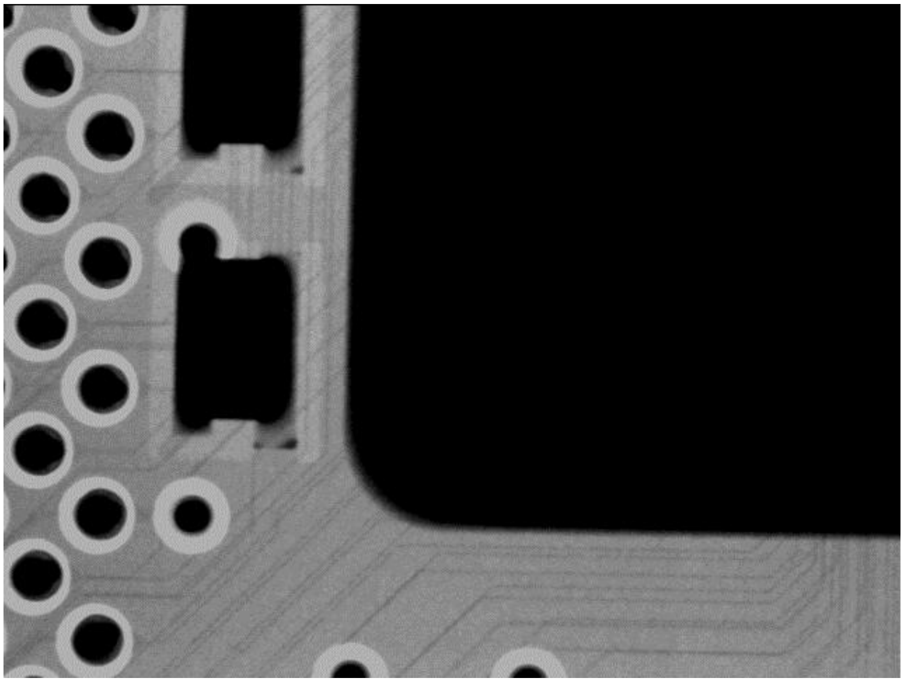
The white stuff could have been cuprous chloride which tends to drop from dirty solutions when excess SMB is used, cuprous chloride is soluble in HCl. Two things cause this, too much SMB combined with copper (II) chloride in the solution. If you strip your base metals off the foils first you won't have this problem.

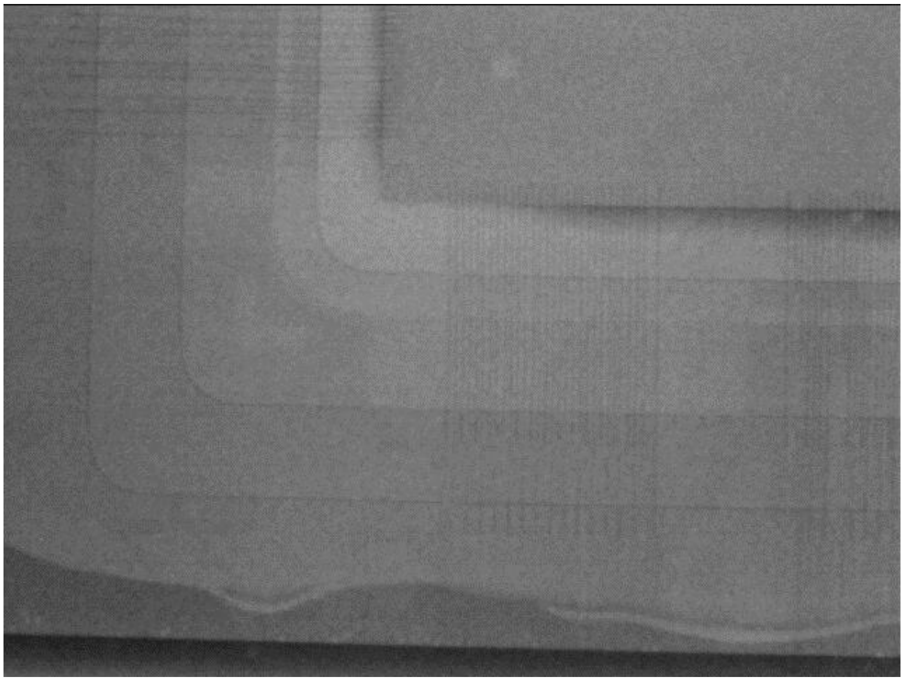
Your melting experiment sounds like you had contaminations as well. If you had a lot of white smoke that was the cuprous chloride. If so your gold is most definitely contaminated with copper and needs to be processed again.

Thanks for the post and welcome aboard.
Steve

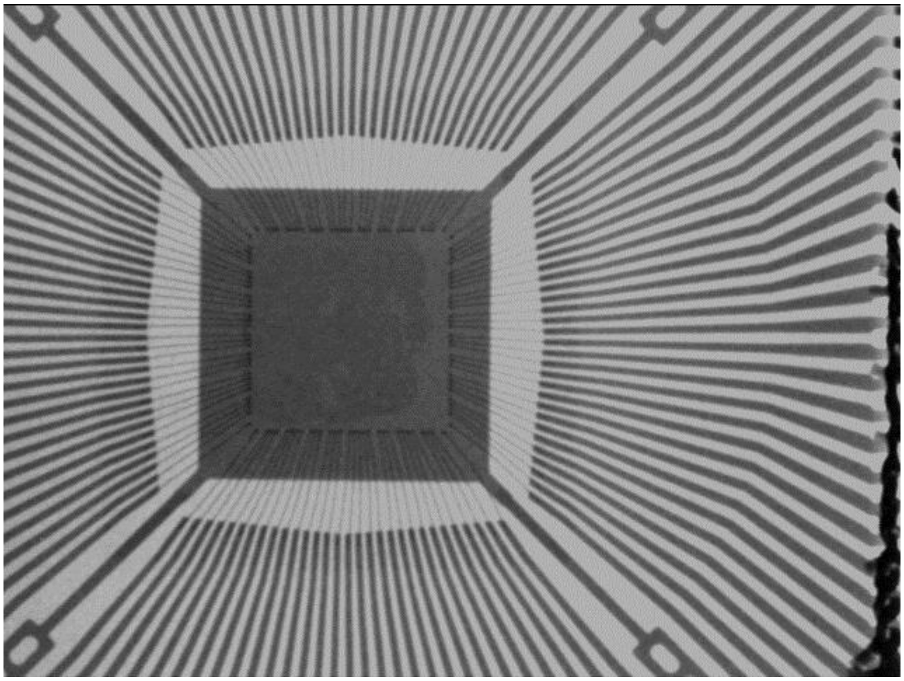
X-ray micrographs of various chips

Pentium MMX fiber chips





flat pack from creative



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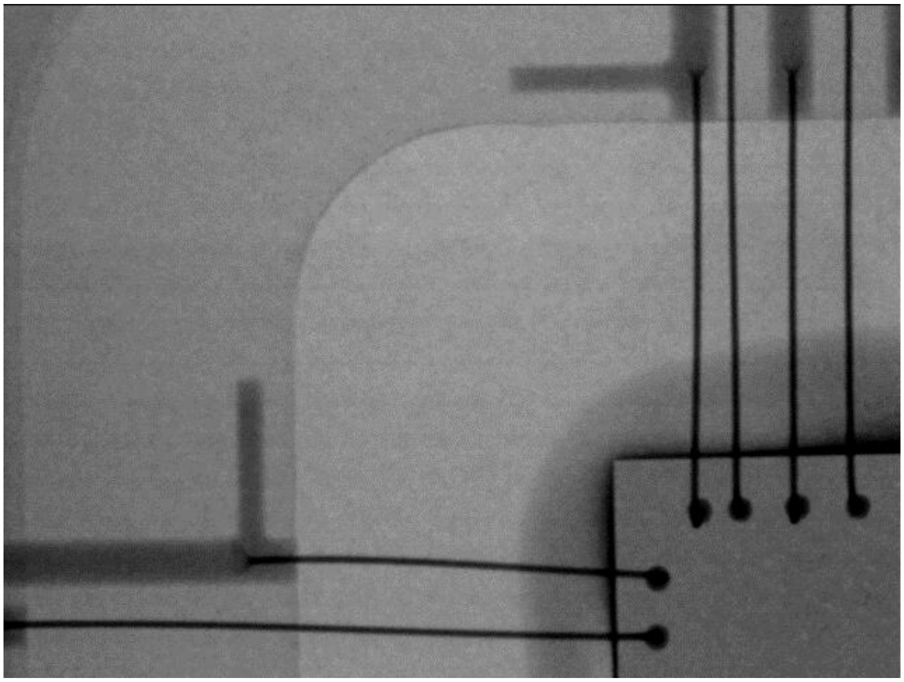

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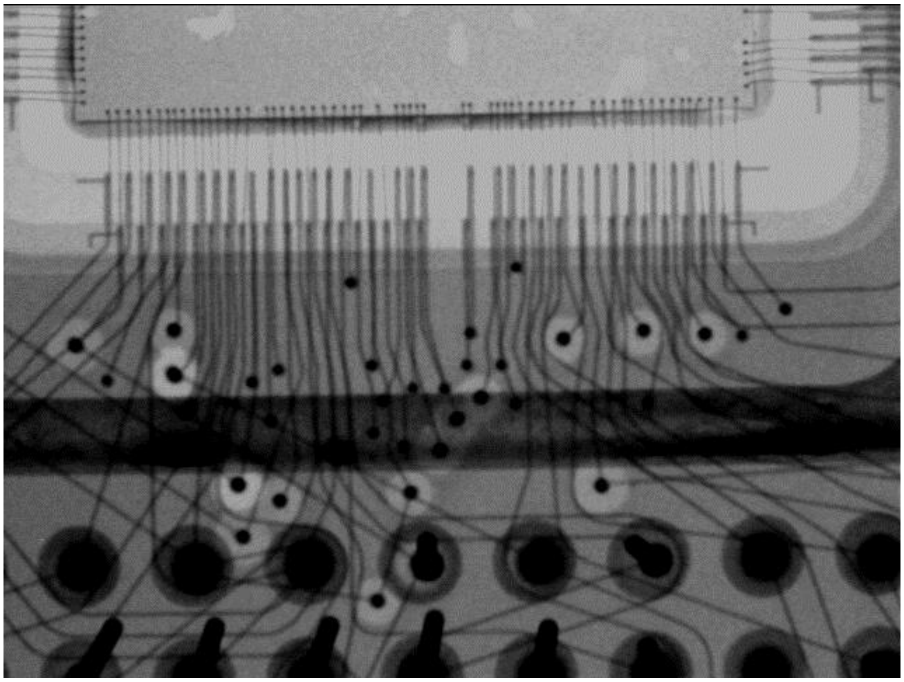
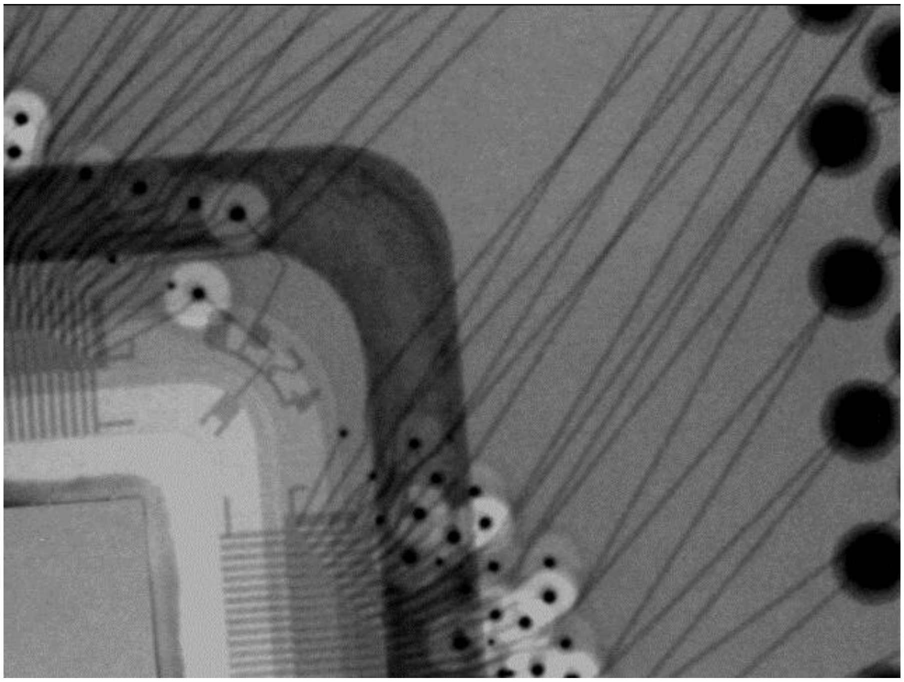


Pentium



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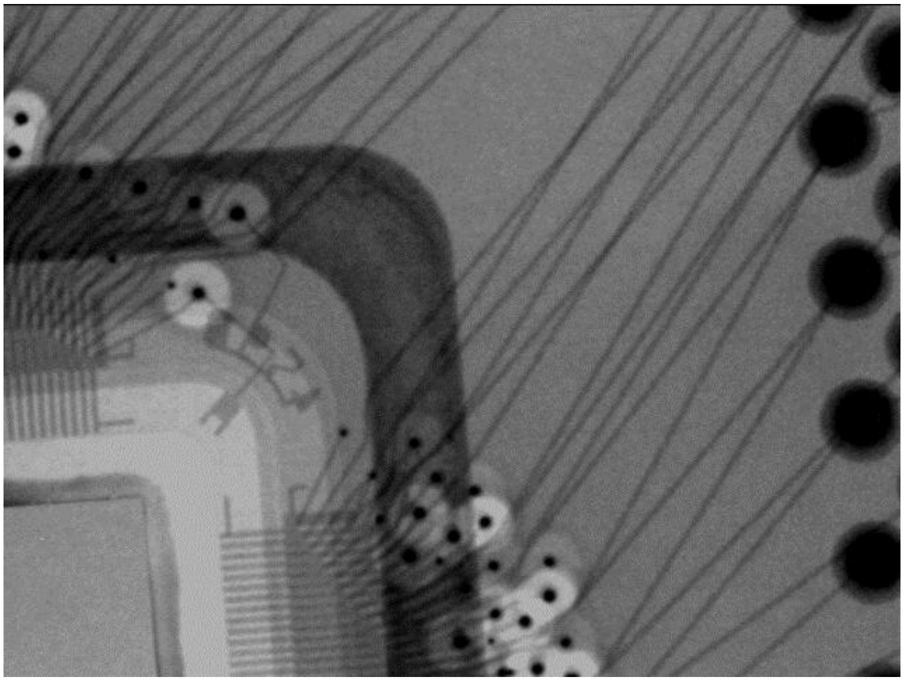
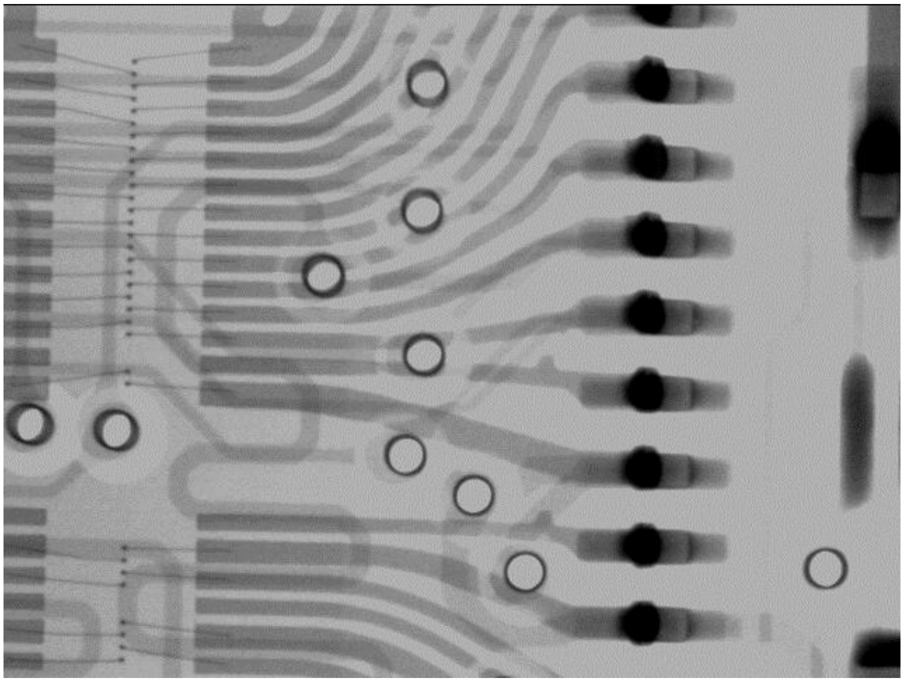


memory chip from new ram



Shor topic >>>> **http://tinyurl.com/2vkzgs**
http://tinyurl.com/yw6opr

http://goldrefiningforum.com/phpBB3/viewtopic.php?t=1143&start=0&postdays=0&postorder=asc&highlight=

He clearly stated that he dissolved his material in AR-----silver will have already been precipitated as silver chloride, so nothing would be recovered, assuming there was any present in the beginning. Having started with AR instead of nitric, virtually none of the silver would have gone in to solution.

The problem I see with this entire operation is the fact that AR was used prior to eliminating base metals. The fact that there was base metal remaining virtually assures that there will be little, if any, precious metal left in solution. The crud described on the one side of the copper is likely the values, having precipitated on base metals. Why would **anyone** process by that method?

I'm mystified by the lack of knowledge in folks that are attempting recovering precious metals. There's an abundance of good and proper information on this list, free for the reading, which makes mistakes like this not excusable.

There is no good reason to process base metals with AR, with a few exceptions. If your material copper, or a copper based alloy, if you don't strip the gold, or eliminate the base metal by dissolving it in nitric, or an equivalent substitute, you're spinning your wheels and have much to learn about refining.

Harold >

> **Noble Metals Recovery, LL wrote:**
>
> I did some testing to see if the bead is silver. I remelted it. No oxides formed. (I think the first time I melted it it did "spit" some). I then put the bead in boiling HCL. No reactions. If it were tin, zinc, or aluminum it would react strongly.

A test with Schwerter's solution will determine, instantly, if you have silver. Schwerter's is made of distilled water, reagent grade nitric and sodium or potassium dichromate. It reacts blood red when applied to silver.

Harold>

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Processing gold polishing sweeps >>>

http://goldrefiningforum.com/phpBB3/viewtopic.php?t=1253&postdays=0&postorder=asc&start=15

My approach was very simple.

1} Incinerate in a shallow pan, heating from below. Ignite the material to eliminate the majority of smoke and stink. It will readily burn once hot enough. Incinerate until all carbonaceous material has been eliminated. There should be no glowing embers. Allow them to burn completely. Insure the pan is hot enough by heating with a torch from the bottom. Don't burn through the pan. An old stainless fry pan works very well for this operation. Find them at Goodwill or other second hand stores. DO NOT USE ALUMINUM.

2} Screen after cooling, to remove any sizeable pieces that would be difficult to dissolve without inquartation.

3} Run a magnet through the material to remove free iron particles. They should be placed in the stock pot, not discarded. They generally will transfer miniscule traces of values.

4} Place screened wastes in a beaker, or a container that will tolerate being heated.

5} With an approximate mixture of 50/50 tap water and HCl, cover the contents with solution, then place on hot plate. Heat slowly until contents are brought to a boil. Stir regularly to prevent the container from overheating and breaking from thermal shock.

6} After a reasonable boil period, (contents will change color), remove from heat and fill container with tap water. Stir well.

7} After solids have settled well, decant solution. Test for values, then discard if barren. It is rare to find values in the solution unless you have allowed nitrates to be included.

8} Repeat the above operation until the rinse water is clear, or nearly so.

9} Apply AR @ 5 ounces per troy ounce of values expected to be recovered. Use only what is necessary, to minimize the amount of nitric that must be evaporated later. Heat to boiling, and stir regularly. Do not allow the material to dry on the bottom of your container, which will lead to thermal shock and cracking, as above. Make your AR @ 4 HCl/1 nitric, not less. If in doubt, move up to 5 HCl/1 nitric. Too much HCl does no harm, unlike too much nitric.

10} When values are in solution (note color change of solids, and the yellow color of the solution), and you see no fumes coming from the container, remove from heat and add a few ounces of water. Stir, then allow contents to settle.

11} Decant, then add more water to solids and stir well. Repeat this operation until you have removed the bulk of values. Note the color of the solution, which will slowly shift towards clear as it is washed.

After the final decantation, place the solids in a filter (Sharkskin works very well for this operation) and extract all solution possible.

Combine all solutions from the AR process and evaporate to eliminate water and nitric. Add a few drops of sulfuric to the evaporation process early on, which will precipitate any traces of lead that may be included in the process. Be very careful about adding sulfuric to hot solutions. It is best added while they are cold. Stir as it is added to prevent local generation of steam.

After evaporation, take up with water, filter, precipitate, and wash appropriately.

I suggest you do none of the extraction processes until you've read Hoke's book---so you have a general understanding of what you're doing, and why. None of this is difficult----it becomes routine after you've done it a few times. Do not allow the outline to discourage you.

Dry the waste solids by heating, then save them. Do not store them wet----they will give off fumes endlessly and destroy anything of value in the immediate vicinity by corrosion. Please take my word on this part (the solid waste material, after acid processing) ---it is a wonderful savings plan. The values remaining are not substantial, but they add up. Remember------you will have left silver behind in this operation, not just traces of gold.

Note that this process will not be suited to processing wastes high in silver. It will remain in the solids. If you process waste from the silver bench, it becomes somewhat more complicated. I can talk about that if and when it is an issue. It is a waste of time to pursue traces of silver with each batch.

Harold

AR Chemistry http://goldrefiningforum.com/phpBB3/viewtopic.php?p=6946#6946

Any solution containing chlorine ions is capable of dissolving gold.

Oxygen from the air can get absorbed into HCl and oxidize gold. The gold ions then form complexes with the chlorine ions to give auric chloride.

The truth is a very tiny amount of gold will dissolve in HCl until all available oxidizers are used up. This amount may not be detectable.

Acid peroxide is just a weak form of aqua regia. Aqua regia works by having the nitrate ion oxidize gold as ions and allowing chloride ions to form complexes. This is why you don't need nitric acid to make a gold dissolving solution: just use sodium nitrate (which is an oxidizer) for poor man's AR. Those brown fumes come from the nitrate ions being reduced by the gold as the nitrate ion gives up oxygen to become NO.

Acid peroxide is the same idea. Instead of nitrate as an oxidizer, you're using hydrogen peroxide, which is also a powerful oxidizer. The hydrogen peroxide oxidizes the gold and the chloride ions form soluble complexes. Also happening is the dissolution of oxygen into the acid, which helps dissolve gold.

Nitric acid will actually dissolve a tiny undetectable amount of gold into ions, but since gold will not form soluble compounds with the nitrate ion, the gold does not continue to dissolve.

The reason why nitric acid will dissolve base metals such as copper very fast while hydrochloric acid will not dissolve it fast is because HCl is a non-oxidizing acid.

HCl cannont oxidize copper metal into an ion so the copper stays intact. It will react with oxides of copper forming water with the oxygen and hydrogen ion leaving a copper ion to form a compound with the chloride ion. If there is oxygen in the air that can dissolve into the acid, then that dissolved oxygen can oxidize the copper which then forms compounds with the acid. In effect, the acid makes the copper corrode from the air faster because the chlorine is stripping away the protective oxide layer.

Nitric acid is different because it can oxidize copper. The nitrate ion plays a dual role. Free nitrate ions give up oxygen to oxidize copper. The oxidation of copper simultaneously reduces the nitrate ion into nitrogen monoxide gas. Free hydrogen ions combine with oxygen to form water and free nitrate ions combine with the copper ion to form copper nitrate.

The reason why AP is prefered over AR is because it doesn't give off horrible fumes. Gold is least likely to dissolve in both AR and AP, but each metal does not dissolve precisely sequentially. There is some overlap. Galvanic action can help make the process more sequential, but not everything is electrically connected so it doesn't happen effectively.

You can in theory dissolve base metals only with aqua regia by controling the amount of nitrate ions you put into the acid. The reason why AR dissolves everything is because we make it such that it will dissolve everything, but if we limit the amount of nitric acid or NaNO3, then we mimick the effect that acid peroxide does.

AP will dissolve everything like AR does if and only if you put in wayyyyyyyy too much peroxide without diluting it too far. It will take a long time but it will happen.

When an AP batch is done, the reason why gold flakes remain is because the oxidizer is all used up, and gold chloride displaces base metals and gets deposited as a black powdery film.

Recovering precious metals from polishing sweeps

> Ok Harold we did the final process, as we are letting it dry and before we melt which flux will suffice?

I trust you went through the proper wash procedure? While gold that comes from this process tends to be of quite high quality, I don't recommend you melt any gold that has been precipitated without first washing properly. I've discussed the washing process at length in previous posts. Be certain you follow that, or a comparable, procedure. The wash should include at least one application of ammonium hydroxide, even if you don't feel it is necessary. It performs a valuable operation in dissolving substances that may not, otherwise, get washed from the precipitated gold.

Regards the flux you use. Use nothing but borax for melting pure gold. The use of other chemicals has the potential to reduce oxides, recombining them with the pure gold, defeating your purpose in processing the gold. If your gold is clean, you need only a film of borax covering the dish, which will serve to "lubricate" the molten gold and allow it to flow well. If your flux discolors when you melt your gold, the gold is not clean. The flux should remain clear of color, shifting towards purple. If any other color develops, the gold is contaminated. Once melted, the surface of your gold should remain shiny, with on oxide skin forming, and will cool with a large crystalline pattern. It should also pull a very deep hole in the center, as it solidifies. If it frosts up and refuses to form the hole (pipe), the gold is not pure.

Quote:

> we tried melting whilst still wet using borax and it seemed like it did the job fine. I think we didnt do the last bit correctly thow as it still has a brown mud on the bottom along with the gold and other metals.

This is a little confusing. For starters, while melting the mud while it's wet will work, you risk breaking your melting dish, depending on how long the wet material is in intimate contact with the melting dish, and how well it is coated with flux.

There should be noting on the bottom of your container aside from gold. If other metals have precipitated with the gold, something has gone woefully wrong. Can you tell me what you think is there with the gold?

If you've read my washing procedure, you know that the gold that is ready for melting has been well washed and dried. It never leaves the beaker in which you precipitate the gold------ not until it has been through all of the process. By following this procedure, the gold gets washed and dried, and acts as its own collector for the very fine particles of gold that can be troublesome. The only time it doesn't collect all the dusty particles is when the gold is very dirty, from dirty solutions. In that case, it is usually accompanied by a dark color, but not always.

Quote:

> If you dont mind could you go into abit more detail from the AR process onwards? we are going to try some more tomorrow night.

I'm going to assume you have done a good hot wash with HCl and water, and have rinsed the mud enough times (tap water) that the rinse water is coming off the mud in an almost clear color. The mud at this point should be a maroon to purple color, depending on the volume of gold it contains, and what medium was used for polishing. If the benchman did a lot of platinum work, it's possible the mud could even be green in color. I've seen them vary widely, so color alone is not a good indicator of contents.

It's hard to guess the amount of value in the mud, for various reasons. The entire objective is to dissolve all the values, which can then be rinsed from the mud via a few tap water washes. Start with AR (mixed @ 4 parts minimum HCl and 1 part nitric). If you have a hunch on content, assume that amount to dissolve 1 troy ounce. Do not use too much AR----if you add unneeded nitric to the mix, it must be eliminated before recovering the gold. The negative side is that if you don't use enough, you won't get total extraction. The best policy is to estimate your recovery and use slightly less acid than is required. Let it work, heated, until it has done all the work it can, then add a little HCl to insure that you did not run out of it in the process, leaving some nitric unconsumed. If you get no further reaction (fumes) by adding the HCl, add a small amount (half ounce or so) of nitric and observe fuming, if any. Repeat this process until further additions of acid yield no reaction. At this point you're wise to allow the mud to stew for a while, to insure that you have dissolved larger bits of gold. Polishing waste usually contains bits of gold from the use of abrasives at the wheel, and they take a little longer to dissolve. The mud should have changed color completely, and reflect a cream color. Solution, if you've done your washing properly before dissolving the gold, will now be a nice deep yellow orange color, a sign of dissolved values and few, if any, base metals. If the solution is green, or darker, the preliminary wash was not carried out well, and an abundance of base metal was left in the mud.

I never used urea, so I can't discuss it's proper application. My method was to evaporate the solution, which eliminates unused nitric acid, plus was an indicator that I had used enough AR to dissolve all of the contents of the mud. Had I not, there would be no unused nitric present. I proved the unused nitric by adding a weighed button of gold to the solution, which gradually dissolved as the solution was evaporated. By weighing before and after, you can determine how much gold you added to the process, which should be subtracted from the gross yield to determine the net yield. By using the button, evaporation need not be carried to a totally thick solution, so it saves time in evaporation. Note that a few drops of sulfuric acid should be included in the evaporation process, which will serve to precipitate any traces of lead as lead sulfate. this is an important part of the process. Do not leave it out. Lead is death on gold's ductility. When the solution has been evaporated until it is well concentrated, and has shifted color towards a dark red, some HCl is added. If there is no reaction at the gold button (considerable bubbling and some brown fumes), you can consider that there is no more nitric present, and the solution can be diluted with tap water and filtered. Once filtered, it can be precipitated with the precipitate of your choosing. I favored SO2 from a bottle, but you can use any of a wide variety of precipitants. One that is easy to use and is readily available at a garden shop is ferrous sulfate. Hoke's book discusses the application in fine detail. The ferrous sulfate, if that be your choice, should be a nice green color. If the color has shifted to brown (exposure to excessive moisture), it will not precipitate gold.

Quote:

> Also for the filter we used the coffee filters which seemed like it did a very good job (have you ever used that before)??

No, I did not use coffee filters, although I did use something that is very similar (Shark Skin, made by S&S) for filtering the solution from the mud. Once my solutions were evaporated, I preferred a Whatman #2 paper, which I purchased in the 32 cm size if memory serves correctly. They fit, when properly folded, the filter funnels that are readily available from chemical supply houses. I found it to be the best filter on the market for filtering solutions prior to precipitation.

The exception to this is that when I filtered gold that was re-refined, I then switched to a Whatman #5, same size. That is a very tight and slow filter paper, and will remove the smallest of contaminants. Unless you're working with very clean solutions at the outset, they perform very poorly. They're quick to stop flowing, but very good at removing miniscule particles from otherwise clean solutions. That describes the gold that I re-refined perfectly, so it was a perfect match for the process.

There's nothing wrong with using coffee filters aside from the fact that they tend to allow very fine particles to pass. As long as your solution is crystal clear (of flocculence, not color), it doesn't matter if you use a coffee filter or don't filter at all, for that matter. The important thing is to separate all the particulate matter from the solution before gold is precipitated, otherwise you recombine unwanted materials with your processed gold. Gravity alone can be used to separate solutions from insoluble materials, it just takes more time, and it makes retrieving the last little bit more difficult. I was way too busy to take the time, so fast and good filtration was important to my operation. I learned to turn out solutions that would filter readily. How well a solution filters revolves around doing the proper washes before you ever dissolve the gold. By doing so, compounds that are formed by acids and difficult to filter are eliminated in the preliminary washes and rinses. These are things you'll learn as you progress.

Ageo308 wrote:

> Aha i feel like an idiot but we did miss a couple of things.
>
> 1: the ammonium hydroxide which we didnt add but will do next time.
>
> 2: the precipitation process, i feel like a dummy as i skipped this bit (i will go and buy some ferrous sulfate.
>
> If you dont mind could you go into some detail with both these things, i.e how much ammonium needs to be used and do i just add it to the rinse? (with tap water)?

Regards the ammonium hydroxide, be certain you understand when this process is used. It should **NEVER** be combined with **gold chloride solution**. The ammonium hydroxide operation, for me, came after the first HCl wash and rinse of the precipitated gold powder. Cover the gold powder with ammonium hydroxide and then some tap water, and heat it. You'll see a change in the color of the solution, indicating it is dissolving unwanted compounds. Usually a light blue color. If you get no color change, great! That's a pretty good indicator that the gold is quite clean. Boil for a few minutes, then decant and rinse with more tap water, bringing it to a boil once again. Decant, and add enough HCl to cover the gold. Add a little water and boil for a while. If the color of the solution doesn't change, the gold is as clean as you're going to get it by washing. That isn't necessarily an indicator that the gold is pure; it may not be. Only a second processing will put more of the contaminants in solution, where they can be left behind by a second precipitation and wash cycle. That improves gold tremendously and is worth the effort if you're striving for 4 9's quality. You should get close, if not there, by this method.

Quote:

> Also with the precipitation you mention take up water, filter, precipitate, and wash appropriately so once i filter it and have the brown mud sitting on the bottom is that when i add the sulfate? and if so how much? these 2 are the only areas i havent done and will do tonight.

I mentioned ferrous sulfate only as an example. As I may have mentioned, there are many things that will act as a precipitant. If you are precipitating by any other means, that's fine, so if you have brown mud sitting on the bottom of your beaker, you've already accomplished that operation.

Quote:

> as for the wash procedure, we basically filled the beaker with tap water, stirred it then let it settle, decanted it and made sure no value were in the liquid then emptied it and repeated the process until the rinse water was clear. Sound right? We did the same rinse process with the HCI/water and the AR mix.

I maybe should have been more specific. I have posted on this procedure and covered anything of importance in fine detail. Aflac has provided a link in the post, above. Please follow that link and read the various posts. There is a lot of information there that will benefit you far beyond the time it takes to read the comments.

Regards what you did with your wash operation, if leaves a great deal to be desired. It doesn't come close to washing the gold in the way it needs to be washed. You may not understand that until you've actually seen what comes from the gold, and how it is changed by the procedure I've mentioned. There's no doubt in my mind, you'll rarely, if ever, produce gold that rivals gold that is washed properly after precipitation. Only when it comes from a very clean solution do you have a remote chance, for drag-down (of contaminants) is the enemy. Prolonged boiling in acid and ammonium hydroxide insure that traces are removed as much as is possible. I advise that you do not short cut the process by avoiding the wash cycle.

Please read the provided link, and tell me what you feel you've learned. It might help in fine tuning what you're trying to learn.

For the record-----gold that comes from this process (polishing wastes that are well washed before the gold is dissolved) tends to be of very high quality because you start with material that lends itself to easy dissolution of the metals. Your quality may be quite good now----but washing is good insurance. You'll see that when you process dirty material, such as filings.

Harold

Getting pure gold (shining) *(Acid Wash)*

This process is used to do your final wash to remove any impurities (Trash) that you may have carried over from your precipitation. This is one of the most important things you will learn in gold refining.

Learn it and live it.

There are NO shortcuts. If you want pure gold, you MUST follow accepted practice. Remember, those that went before us have gone through this time and again----and have perfected a process that will yield good quality------so if you're interested in achieving that level, follow instructions that are at your disposal. Don't experiment-----leave that for others with time on their hands that are content to re-invent the wheel. This is particularly important for you, considering you're trying to establish a refining service.

Depending on what you're starting with, you may have a hard time achieving a good level of purity the first time through. That's to be expected. Part of the problem is mechanical drag-down, where you're working with very heavily contaminated solutions, part of which follow the gold. They're not always easy to wash out, even when boiling with HCL.

My advice to you is this:

Process your gold the first time, using conventional means. Allow as little in the way of contaminations (metals) as is humanely possible, to be included with the gold. Use only clean vessels. Wash them well between operations-----and use BonAmi cleanser if necessary to remove stubborn stains. Do not use other cleansers, for they scratch the glass. Filter before precipitating, and insure that the filter does not allow particulate matter to pass. If it does, re-filter, or go to a better filter grade. A Whatman #2 does an outstanding job for first refined metals, although they are not cheap. I used them exclusively for filtering first run gold chloride. I felt they were worth the extra money. At all times, keep your beaker covered with a watch glass of appropriate size, to prevent contamination from other sources.

Precipitate your gold using conventional methods, collect it and wash it well. It's best to use a precipitant that does not add contamination. I preferred SO2, but there are others that work equally as well, I'm sure.

First wash should be in boiling HCL and tap water. Boil for a prolonged period of time. Take up the solution with tap water, decant after the gold has settled, then rinse with tap water, which should again be brought to a boil. After it has boiled for a period of time, add more tap water to cool the lot, then decant as before. Rinse again, and do it until the wash water comes off clear. Next, wash the gold with ammonium hydroxide and tap water. Heat it until it boils. You'll notice that the solution gets discolored. How much is determined by how dirty your gold was when you started. The ammonia evaporates fairly quickly, so you can't boil very long. Add tap water to cool, decant, and follow up with a tap water rinse, again, boiling the water. Add tap water to cool, then decant. You now repeat the HCL and tap water wash. You'll be amazed that suddenly more contaminants will come off. Boil well, add water to cool, decant, repeat the water rinse, decant, then, and this is important-------start the refining process all over again by dissolving the well washed gold powder in aqua regia. I'll talk about that in a minute, but these are indicators you should have observed along the way, aside from the fact that you could see contaminants being washed from your gold powder.

The gold powder, which started out quite dark, is now much lighter in color.

It may have been rather loose and flocculent, but now is eager to agglomerate, so it forms lumps and settles quickly.

While these instructions sound like a lot of wasted time, when you process what you think is pure gold, you'll see evidence in the remaining solutions that it was not. The evidence of which I speak will be in your solution when you've extracted the gold for a second time. It's a lot of work which is why I re-refined my gold in large lots, often as much as 200 ounces at each refining, so the time spent yielded a large return.

When you precipitate for a second time, one of the things that you can do to help eliminate unwanted elements is to use a different precipitant than the first time. I never bothered with that, but it's good advice if you're fighting with a given contaminant and can't get rid of it by other means. That was never a problem for me.

Wash the gold powder the second refining by the same methods and sequences as the first refining. Make sure all your equipment is scrupulously clean. Force dry the gold when it's washed by heating the beaker at a very low heat for a long period of time. Avoid allowing the beaker to achieve boiling temperature---you get minor steam explosions that blow gold out of the container. As the gold dries, it's a good idea to swish it around the beaker occasionally, which prevents the gold from bonding to the beaker. The residual matter in water tends to form around the gold and bond it to the beaker. While it's a contaminant of sorts, it does NOT alloy with your gold, so it is not a source of lowering the fineness unless you have strange elements in your water. For me, it was not a problem.

Once dry, you can then melt your gold. A clean, never used for anything but pure gold, dish can be used. It should be covered with a coating of borax, but nothing else. DO NOT USE ANY SODA ASH. If there are any oxides present, they'll be reduced and absorbed by the molten gold instead of locked in the flux.

Your torch should be cleaned of loose crud before melting your gold. Run fine abrasive cloth over it until it's very clean, then wipe it down with a damp cloth to remove any traces of dust. Think clean every inch of the operation-----otherwise you undo all the hard work you've done getting your gold pure.

Your other option is to operate a small gold cell, but you must have several ounces of pure gold for the electrolyte.. That is likely not a viable choice for you at this point in time.

Harold

> **Ageo308 wrote:**
>
> Ok let me explain what we have done so far, We incinerated the sweeps, then sifted it (removed the larger pieces and stored them), then ran a magnet through it to remove an iron particles.
>
> * Once that was done we boiled it in 50/50 HCL/water until it changed colour (cream brown then green), basically from there we added some water to cool then decanted the solution, then added some more water but didnt bring it to boil

> (wasnt sure) after a quick stir and let it settle we rinsed it again (5 times roughly).

At this point you've done quite well. The whole idea is to dissolve anything that will dissolve, but not the precious metals. If your solution tested barren, it can be discarded. The five rinses would indicate that you had removed the vast majority of unwanted materials, so when you dissolved the gold and other values, the solution would then be quite clean. Well done to this point.

> **Quote:**
>
> * From there we put the mud into AR and did the same process as the HCL which is wrong i know but im just explaining what we did.

No, it's not wrong--it's exactly what you were supposed to do. What you did with the solution is what I think you did wrong. If you discarded it, you tossed your gold. AR dissolves gold, which is EXACTLY why you introduced it to the mud. It is intended to dissolve the gold and other values, so they can be separated from the dirt, which has no value. You get a much better extraction by dissolving than you do by melting, unless you use litharge in melting, but that involves cupellation, which is beyond the average guy's ability, and is also very hazardous because you're burning lead and producing large volumes of lead fumes.

> **Quote:**
>
> Tonight we basically put the dried dirt into a 50/50 HCL/water mix and boiled for about 20min then added water, decanted (the solution was yellow), and rinsed again with new tap water and again brought to boil. This time we repeated that process about 8 times and had to stop but the rinse water is becoming much clearer and the mud is now becoming a nice smooth fine dust (lightly brown) with no sludgyness. I can see gold particles in there so im guessing thats a good thing.

If you combined all these wash waters with the first (yellow) solution and have started evaporating them, you're on the right track. If you discarded the wash water, as I said before, you tossed the values. If you can still see gold in the mud, it's because you didn't use enough acid to dissolve all of it. Fact is, the mud is what you should have been trying to eliminate, not keep. By washing it until you had rinsed out all of the yellow color, you would have removed all of the gold, assuming you had dissolved all of it in the first place. For some reason you seem to think that the dirt is the target-----**it is not**.

> **Quote:**
>
> * Tomorrow night we are going to finish off the rinsing process then start with the ammonia rinse and do that until its clear and then finally back to HCL/water with a final rinse to make sure there is nothing left to clean.

Don't do that. You should have evaporated the golden solution to expel the unused nitric, diluted the evaporated solution with water, filtered, then precipitated the gold FROM the solution. You're spinning your wheels working on worthless dirt, assuming you dissolved all of the gold in the first place. Your only hope is if you had enough sense to keep all of those solutions aside from the very first one, where you used HCl and water the first time, plus the rinses. If you did not, you have lost your gold.

> **Quote:**
>
> So tell me where i went wrong and if we are going in completley the wrong direction. From what i can see thow the powder is looking much nice then it did before.

It may look "nicer", but what does that mean? You've possibly tossed the baby with the bath water. I hope you haven't.

Can you see why I suggested you not do anything until you understand the process? Do you see why I suggested you hold off until you had Hoke's book?

Had you kept the instructions near you while you were working, and checked each step to verify what you were doing, you wouldn't be where you are right now.

Let me know if you saved your gold colored solutions. If so, you must turn your attention to them.

If you think you're seeing gold in the mud, you may not have dissolved all of the values. Heating with a little more AR will disclose if you did, or did not. If there's values remaining, you'll see the solution turn yellow to orange, depending on the degree of concentration.

I haven't heard you mention stannous chloride much in your report. Did you ever bother to check solutions?

NEVER DISCARD A SOLUTION WITHOUT TESTING WITH STANNOUS CHLORIDE THAT IS KNOWN TO BE ACTIVE. That means you should have on hand a standard gold solution, which is used to test the stannous chloride. It's all in Hoke.

Harold

> **Ageo308 wrote:**
>
> Ok Harold, we havent discared any solution and all of them have been filtered.

I'm pleased to hear that. I feared you took a wrong turn and had discarded your gold, as you may have noticed.

> **Quote:**
>
> I just re-read hoke's book and revised some of the important areas. A quick question on what she says.
>
> Basically she mentions that you should Incinerate, sift, magnet then if there is shellac/grease etc.. to boil in caustic soda/lye (to remove it). Then she mentions to wash in nitric/water acid unlike your procedure which is HCL/water, it says in her book you can use HCL but it doesnt dissolve copper or nickel (which will be present in these sweeps), the only draw down with using nitric/water is that it will dissolve silver but you can store that and recover it at another time.
>
> * so im interested to know why you choose HCL over the nitric?

It was my reasoning that there is far more iron in the waste (from polishing compounds) than copper or nickel, each of which will be dissolved to some degree because they are oxidized by incineration. HCl is an excellent solvent for iron, unlike nitric, although it, too, will dissolve iron. The solutions are very different, however, with the nitric solution eager to precipitate the iron as rust, which would occur when you rinsed the material. That can complicate your processing, and leaves behind unwanted iron.

My experience in running my wastes by this manner tended to support that logic in that the solution, after precipitation of gold, was relatively clean. If you test your initial wash solution for copper and nickel, I'm quite sure you'll find that a good deal of it does, indeed, get dissolved with the HCl operation. A drop of ammonium hydroxide in a drop of the solution will turn dark blue if there is copper present. Likewise, a drop of the solution, along with a drop of ammonium hydroxed and a drop of DMG (see Hoke) will disclose nickel with a pink display. It left the silver behind either as a chloride, or in the elemental state, although I imagine it was converted to chloride by the gold chloride solution if that was the case. You would be unlikely to be able to recover any silver by this process, but it is not lost, it's still in the dirt. More on this, below.

Note that I did NOT process filings by this method. Only floor sweeps and polishing wastes. Filings are processed with nitric acid, then AR.

You see considerable evidence of my claim in the color of the original HCl wash. Beyond that, what you'll come to discover is that solutions that come from dirty substances and a nitric wash tend to be difficult to filter. Without the HCl wash, your gold chloride solution has a tendency to plug filters. I've had a liter of solution take as long as three days to filter. From that, I learned to incinerate suspect materials and then do an HCl wash, even if the material had already undergone a nitric wash. In that case, the material must be incinerated a second time, to kill any nitric that is present. I found nothing to equal HCl's ability to clean material of substances that were difficult to filter. That is particularly true of refining gold filled objects in volume. There is often a little lead and tin included in gold filled objects. They respond to the treatment I mention in a very positive way.

What you have been instructed by me is a process that evolved through trial and error and was used for many years. To be quite frank, I don't recall what Hoke recommended, so it's been interesting to hear that her version varies from mine. I assure you, if you follow my instructioins, you'll achieve a level of quality and extraction that is very good, although if you have doubts, don't hesitate to follow her instructions. I've commented time and again, I learned refining from her book, and know that aside from her recommendation to use gasoline freely when incinerating, her information is trustworthy.

> **Quote:**
>
> * And why not boil in caustic as she mentioned to remove the shellac/grease etc?

It's redundant. A boil in lye will remove a good deal of the unwanted material, but it won't eliminate everything that will be troublesome. In the end you still must incinerate, so I see no good reason to duplicate operations unless there is some gain. Incineration performs every possible useful function, and eliminates the hazard of using lye. Should you splash lye in your eye, blindness is guaranteed, yet a drop of nitric in the eye, while painful, is not serious. I've been through that and came out fine. Learned from that to wear eye protection.

> **Quote:**
>
> And i cant see anywhere that stannous chloride is mentioned in hoke's book, is this to determine what metals are present in the solutions???

That's correct. Read the chapter that involves testing of precious metals, and follow her instructions in buying and building the necessary solutions. In her book, I recall that she refers to stannous chloride as testing solution A, but I could be wrong. I haven't looked at a copy in well over 12 years.

If you do not have stannous chloride, immediately work in the direction of obtaining it. Do a search on the board if you must, for there's a lot of information available. Many of the guys here are making it with 95/5 solder. Personally, I preferred to use pure tin and stannous chloride crystals, which was my routine. Without it's use, you run blindly, not knowing the status of any of your solutions. Also, do yourself a favor and discard solutions by proper means the moment you conclude they have no value. It takes very little time to find yourself inundated with a huge variety of unknown and unwanted solutions, so you not only trip over them, but you spend a good amount of time screwing with them unnecessarily when you finally decide on disposal.

> **Quote:**
>
> P.S the sweeps we are using is my partners and he has plenty to spare and experiment with.
>
> P.SS i think im getting you now Harold

I hope so. I'd like to see you succeed. This method is quite good. I have serious doubts that you could come up with one any better.

> **Quote:**
>
> Correct me if im wrong but when you say combining the wash waters (yellow) solution is the colour yellow because its suppose to be gold?

That's correct. Gold chloride that isn't contaminated with copper or nickel is yellow, deepening to a dark red/orange, depending on concentration. The wash water is nothing more than dilute gold chloride, which is why you combine it with the darker yellow solution. If you had stannous chloride, you'd see by testing that the solution would react with a purple stain. That's what gold looks like when combined with stannous chloride. As the concentration deepens, so too does the color. Very slight presence of gold will yield a faint purple reaction.

> **Quote:**
>
> we havent discarded this solution and if im understanding right we are suppose to evaporate that yellow solution to basically have clean gold??? Is this correct???

No----evaporation does NOTHING to improve gold quality. That's determined by the washes you do prior to dissolving the gold, by the precipitant you choose to use, and by the washing you do after the gold has been precipitated. Evaporation serves two purposes. One is to concentrate voluminous solutions so you aren't working with gallons when a liter is adequate, but most importantly, it, when carried out properly, eliminates unused nitric acid. That is a requirement to recovering your gold. If you leave it behind, it will react with not only the precipitant, slowing or stopping the operation, but assuming you are successful in precipitating your gold, when you wash it, the residual nitric will re-dissolve some of your gold, until it has been consumed. That's not desirable, as you might understand. Please read Hoke on evaporation of solutions, and learn to do so. Also, if you have a button of gold at your disposal, it really helps to add one, as I've already mentioned. Weigh it before introducing to the solution you're evaporating, and weigh it afterwards if you want to know how much of the button was dissolved in the process of eliminating any free nitric. The use of a button will avoid evaporating the gold solution down to a heavy syrup, so it saves a lot of time. If you choose to use a button, you must still add some free HCl as the solution thickens. That way it will form AR and consume the free nitric by dissolving the gold button. Very slick way to process, if I must say so myself.

> **Quote:**
>
> If it is i will add more AR to the mud to dissolve any more undissolved gold then wash with the HCL.

I'm not convinced you understand the wash purpose in using HCl. It is NOT used to wash the material once you have introduced AR. In this case, if you subject the material to more AR, simply boil the material in the AR and water mix, then add a small amount of HCl to insure that you haven't depleted what you introduced with AR. If, when you add the HCl, you see brown fumes coming off the beaker, that's a sign that you did just that. If you see no reaction, the next thing to do is add a few drops of nitric. If there is any undissolved gold present, you should see brown fumes coming from the beaker. If you do not, you can safely assume that the dirt is now barren of undissolved gold, and needs no further processing. At this point, add a little water, stir well, and allow the solution to settle.. Decant the solution, then add a generous amount of water and stir, again allowing the contents to settle well. Your objective here is to rinse the mud clear of all dissolved gold-----which requires NO HCl. Just plain old tap water will suffice. Wash the mud until you're satisfied that you are not getting a return worth the trouble, then get the dirt in a coffee filter and extract the last of the liquid, which should be combined with the balance of the yellow solutions we've been dealing with.

The dirt, at this point, is relatively valueless, although it will contain traces of values that you haven't recovered. Included in that there can possibly be platinum, which dissolves very slowly, and silver in the way of silver chloride. I suggest you dry this material by heating, just as if you would to incinerate. You'll see nitric fumes coming off at the very end of drying. Heat until you no longer see the fumes, then cool the material and store in a covered drum. If you collect enough of the polishing waste in this fashion, it has market value, or, down the road, when you have enough, you can run it in cyanide and extract the silver, along with traces of gold, all of which are never fully recovered, try as you might.

DO NOT STORE WET WASTE MATERIALS. THEY WILL RUST EVERYTHING IN YOUR WORK AREA THAT WILL RUST.

Hope some of this helps.

Keep us all informed on your progress.

Harold

Gold Refining Community
Gold Refiners helping one another !

> **Ageo308 wrote:**
>
> Just re-read hoke's washing:
> Correct me if im wrong but when you say combining the wash waters (yellow) solution is the colour yellow because its suppose to be gold? we havent discarded this solution and if im understanding right we are suppose to evaporate that yellow solution to expel the excess nitric then once that is all done

When evaporation is concluded, you dilute the gold chloride solution <u>and filter the solution until it is clear of flocculance.</u> Do not precipitate any solution that is cloudy---all you do is recombine the gold with unwanted junk.

> **Quote:**
>
> make a seperate iron sulfate solution with water and add some HCL to make it a clear green (as the book says) and then slowly add it to the main gold contents to precipitate the gold. Once the dark cloud fumes stop then it should be done.

Do <u>NOT</u> assume it's done. Check with stannous chloride. This is important for a solution that has iron dissolved within will still be yellow in color. You'd have no way of knowing if the color was gold or iron without testing. You may also detect the presence of the platinum group when gold has been precipitated. If you get a yellow to brown display, that's a sign of platinum in solution.

> **Quote:**
>
> So i filter the gold then rinse it with water a few times and finally a few times with hcl/water mix until its the gold powder is clean. Then filter again and let it dry in the funnel. After this its ready to be melted.....
>
> This sound about right???

That is one place where I depart from anything you may read, and I encourage you to listen to me. Do NOT filter the gold powder. There is nothing to be gained by doing so, and you lose some in the process, although you would recover it in future processing.

What I would like to see you do is, after you have precipitated your gold, and tested the solution and know that it is barren, to allow the gold to settle by gravity alone. This may take a day, but you can see the progress by the stratification that comes from the finest of particles, which are slow to settle. When the solution has settle totally, decant with a hose, then place the beaker on a burner and add some HCl and water. You will do the three wash and rinse phases without ever removing the gold from the beaker. You will also dry the gold in the same beaker. If you do your work properly, when the gold is dry, it will be a light brownish gold color, and will cling in clumps, making handling very easy. Again, I strongly encourage you to follow my advice in this regard. It is the fastest and easiest way to deal with your gold.

Harold

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Silver >>

I never filtered my silver nitrate solutions when I was recovering silver, even if I had traces of silver chloride (which I routinely had). I never processed silver for the purpose of refining it-----it was used for inquartation. Only when it was used in that fashion and recovered with copper did it get a final refining. Totally irrelevant in the scheme of things, but there was method to my madness.

I found that even if I missed my inquartation and the gold broke up in fine powder, all it took was a few hours of settling, far less time than filtering. I also didn't filter when I washed the collected cement silver. Only when I was ready to dry it did it see a filter. I'd simply wash it with tap water, allow it to settle, then decant. I'd do this until the wash water was clear (usually only three times or so). It took almost none of my time aside from siphoning the solution.

When the wash was clear, I'd siphon off the last wash, then get the entire lot of silver in a large buchner. At this point, it filters very well, so it takes no time to pull the remaining water out. At that point it would have shrunk considerably, and pulled cracks in the silver. I'd compress the entire lot with the butt end of a pestle, then wash down the funnel well to insure all the silver was collected.

This process shrinks the volume of the silver tremendously, making it much easier to handle. When the water was extracted to my satisfaction, I'd them dump the silver in a large evaporating dish, where it was force dried on a hot plate. The silver was then ready to be melted with flux (borax only-----no soda ash). The silver, once molten, was poured into a large cone mold, so the flux could be separated.

When cooled, the buttons were chipped free of flux, then re-melted and cast as anodes, ready for the parting cell. The fluxing operation removed anything that was unwanted aside from traces of copper, so if you have a trace of silver chloride, it is removed in that phase of the operation. No big deal, really.

I reprocessed all my flux, including it with low grade wastes when processing residues in my large furnace. The flux reduced any chloride present, with the resulting silver acting as a collector for higher values. In a sense, the process I developed worked to my advantage, because I needed a collector anyway.

You might keep this in mind if you see more refining in your future. The benefit is you don't waste valuable time screwing around with a few cents worth of silver chloride, and it pays huge dividends when you recover it by collecting traces of values from low grade waste materials that might, otherwise, be lost.

That's my story, and I'm stickin' to it!

Harold

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Ammonium chloride

Pave as the name implies it's a simple mixture of ammonium hydroxide and hydrogen chloride in a 1 to 1 molar ratio:

*Making Ammonium Chloride with 32% Muratic Acid and Household Ammonia (3% Ammonia by weight = Ammonium Hydroxide)

Formula (ignoring water) : $HCl + NH_3 \longrightarrow NH_4Cl$

Mass:36.5 g/mol + 17 g/mol --> 53.5 g/mol

Percents: HCl = 32%;NH_3 = 3% ; NH_4Cl= 100%

Weights for 1 Mole:

HCl =~ 36.5/.32=114.1 gm;

NH_3 =~ 17/.03= 567 gm;

NH_4Cl=~ 53.5gm

Specific Gravities:

32% HCl=~ 1.18 g/mL ;

NH_4OH (3% NH_3)= ~ 1.02 g / mL ;

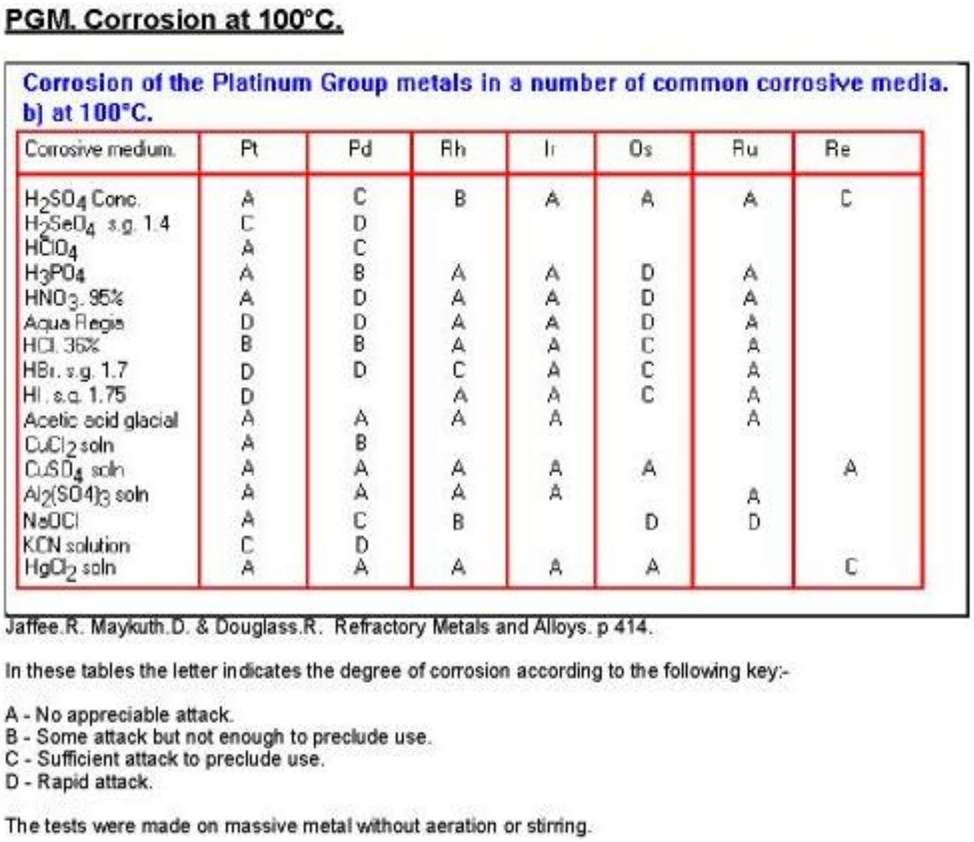
Calculate Volumes required per mole (alternately you can just weigh the liquids in the graduated cylinder with the scales tared for the empty cylinder and fill to the proper weights for 1 Mole):

32% HCl = 114.1 / 1.18 =~ 97 mL

3% NH4OH = 567 / 1.02 =~ 556 mL

Reaction:

1. Obtain a clean, dry, empty 1 Liter container
2. Use a graduated cylinder to measure 556 mL of 3% ammonia being careful not to inhale the fumes. Adjust this volume with the above equations if not using 3%.
3. Carefully decant the 3% ammonia into the container.
4. Throughly rinse the graduated cylinder with water, dry, and measure out 97 mL of 32% muratic acid.
5. Add a 10ml of the HCl to the NH3 and swirl
6. Swirl the vessel until the vessel cools and no longer heats up when swirled.
7. You must wait for the vessel to cool completely between additions or the vessel may break due to the heat build up!
8. Check the pH of the cooled mixture, it should read 7.0 +/- 0.1, if lower add a drop of 3% NH3, if high add a drop of 32% HCl and retest. Alternately, if the solution smells of ammonia add a drop of HCl, if it smells of HCl add a drop of ammonia. BE CAREFUL NOT TO INHALE THESE FUMES!!! Waft your hand over the vessel to get the scent.
9. Gently evaporate the solution to dryness in a well ventlated area to obtain pure 53 gms of NH4Cl or concetrate until crystals form and use hot for precipitation of Platinum and Palladium. If evaporating to dryness pour the liquid into a square casserole dish with low sides and gently heat unitl all moisture and fumes are gone. Scrape the powder from the sides of the dish with a knife. Steve



PGM. Corrosion at 100°C.

Corrosion of the Platinum Group metals in a number of common corrosive media.
b] at 100°C.

Corrosive medium.	Pt	Pd	Rh	Ir	Os	Ru	Re
H_2SO_4 Conc.	A	C	B	A	A	A	C
H_2SeO_4 s.g. 1.4	C	D					
$HClO_4$	A	C					
H_3PO_4	A	B	A	A	D	A	
HNO_3. 95%	A	D	A	A	D	A	
Aqua Regia	D	D	A	A	D	A	
HCl. 36%	B	B	A	A	C	A	
HBr. s.g. 1.7	D	D	C	A	C	A	
HI. s.g. 1.75	D		A	A	C	A	
Acetic acid glacial	A	A	A	A		A	
$CuCl_2$ soln	A	B					
$CuSO_4$ soln	A	A	A	A	A		A
$Al_2(SO_4)_3$ soln	A	A	A	A		A	
NaOCl	A	C	B		D	D	
KCN solution	C	D					
$HgCl_2$ soln	A	A	A	A	A		C

Jaffee.R. Maykuth.D. & Douglass.R. Refractory Metals and Alloys. p 414.

In these tables the letter indicates the degree of corrosion according to the following key:-

A - No appreciable attack.
B - Some attack but not enough to preclude use.
C - Sufficient attack to preclude use.
D - Rapid attack.

The tests were made on massive metal without aeration or stirring.

PALLADIUM AND AMMONIUM CHLORIDE (Hoke)

Place in a small beaker a half ounce or less of the palladium solution you prepared in Chapter IX. This was made by dissolving pure palladium in a little aqua regia, evaporating off the excess acid, and adding a little water; its chemical name is "palladium chloride." In appearance it resembles both gold chloride and platinum chloride. To this add a little of the ammonium chloride solution you prepared for the previous test a quarter ounce, more or less. Does anything happen ?

If your palladium is pure, and if your standard solution was prepared as directed, nothing will happen. This experiment shows that palladium differs from platinum in that it is not precipitated with ammonium chloride from an aqua regia solution from which excess nitric acid has been boiled off.

Warm the mixture gently, and add a single crystal of sodium chlorate. Or, dissolve a pennyweight of chlorate in a little warm water, and add a drop at a time. There will be considerable fizzing, and as it subsides, add another small crystal or another drop. The solution should be just short of boiling. After the addition of a little more of the chlorate, a change in color will be noted. Stir the mixture with a glass stirring rod; soon a dark red precipitate will be seen, floating on the surface and then sinking. Continue to add chlorate, a little at a time until the liquid is almost colorless, and then let cool. This red powder is palladium ammonium chloride chemically much like the orange powder above.

REMOVING GOLD WITH AQUA REGIA

All the gold is now to be dissolved. A little platinum will dissolve unavoidably at the same time. For every Troy ounce of gold, more or less, add

1 fluid ounce nitric acid,
1 fluid ounce water,
4 fluid ounces hydrochloric acid.

Do not add sulphuric acid yet. Use enough aqua regia to cover the filings well. Do not heat this (unless you are in a great hurry) because the hotter it is the more platinum it dissolves, and we do not want to dissolve any more platinum than necessary. If your material is mainly gold, add the aqua regia slowly, as it may bubble over.

Let this stand till there is no more bubbling when you stir. Usually 24 hours are enough, but the thicker the lumps of gold the longer it takes. In hot weather it works more rapidly. You can hasten action by stirring frequently, by heating, and by having all pieces rolled very thin. In some cases two hours are long enough to dissolve all the gold. If you have much gold, put on a little aqua regia at first, let it act all it will, then pour it off and add more. It is better to do this than to add all the aqua regia at once. To the liquid now add a little sulphuric acid, to throw down the lead, if any is present, as white lead sulphate. Use say an ounce or less of concentrated sulphuric acid to each quart of solution, add it

slowly, stirring all the while. The lead sulphate settles slowly.

The next step is to precipitate platinum by means of ammonium chloride. For each quart of solution you will need about half a pound of ammonium chloride, dissolved in hot water to make a concentrated solution. Good quality (C. P. or T. P.) chloride is best; cheaper stuff contains trash which must be filtered out. Add the strong ammonium chloride solution to your aqua regia solution, and stir well. Use plenty; for each pennyweight of platinum that is in solution, you will need a little over 1/2 a pennyweight of ammonium chloride, and a definite excess is advisable. Immediately the orange powder of platinum ammonium chloride will appear. About 95 per cent, more or less, of the platinum that was in solution will be precipitated in this form. The color will range from canary yellow through orange to a deep maroon. Do not be surprised if the shade is different every time; it depends upon the temperature at which it forms, the impurities present, and other factors as well.

In some cases this is a satisfactory method that is, some workers are willing to obtain all their platinum-group metals together in this way, and then repurify the powder as required. Other workers, including Whitely and Dietz, vary the procedure thus: just before adding ammonium chloride, they stir some alcohol into the hot solution from which the chlorine gas has just been expelled. This reduces the palladium and iridium compounds to the "ous" condi tion, in which they stay in solution while the platinum salt comes down. The palladium and iridium are recovered later from the Stock Pot, as usual.

Four parts HCl to 1 Part HNO3,; If I remember correctly, Hoke states 4 fluid ounces of HCl + 1 fluid ounce HNO3 to 1 troy ounce gold scrap.

I haven’t been able to find borax. Can someone point me to the right isle in the grocery store? Answer >>>> Walmart Laundry detergent isle ‘20 mule team borax ’, blue-green and yellow colored box.

For silver, one gallon of 70% nitric (2 gallons of 50/50 nitric) will dissolve about 7 pounds - 100 troy ounces.

8 oz of Nitrate Soda mixed with 480ml of very hot water and dissolve it. 960ml of HLC (32%) mixed with the nitrate soda and H_2O solution. Stir the solution and let it set for about 5 minutes. Salt will form on the bottom of the container. I just pour off the clear solution. This amount of (sic) Aqua Rega will dissolve 140 grams of metal (plated or filled Material) or 48 grams of solid karat gold.

Interesting facts about Activated Carbon and Adsorption.

Comparison of Particle Sizes
1-inch ball 25.4 mm (millimeters) = 25,400 um or µm or “microns” (micro-meters)
pollen 10 - 100 microns
smallest item visible to naked eye 40 microns
fog droplet 2 - 50 microns
“dirt” 40+ microns
silt and clay ½ - 20 microns
pathogenic protozoan cysts 3 - 20 microns
Cryptosporidium oocysts 3 - 7 microns
Cyclospora cysts 8 - 10 microns
Giardia cysts 8 - 12 microns
Entamoeba cysts 12 - 20 microns
red blood cells 7 ½ microns
most bacteria and algae ½ - 5 microns
“turbidity” 0.1 - 5 microns
colloids 0.1 - 5 microns
wavelengths of visible light 0.40 (blue) – 0.77 (red) microns = 400 (blue) – 770 (red) nm (nanometers)
cigarette smoke 10 – 1000 nm
viruses 10 – 250 nm
protein molecules 2 – 50 nm
individual atoms 0.05 – 0.25 nm

Adsorption: Not to be confused with absorption (which is what a sponge does), adsorption is the attraction of tiny particles or dissolved molecules to a solid surface and holding them there by weak intermolecular forces. It is similar in concept to magnetism and the attraction due to static electricity, but much weaker. In theory, every atom in the universe has some degree of affinity for every other atom in the universe, just like gravity. But, just as gravity requires enormous masses like planets and stars to show its effects, adsorption requires extremely tiny distances to show its effects. In adsorption, the particle in question is randomly bounced around the solution by collisions with water molecules and other molecules in the water. (This is called Brownian motion.

It is estimated that an atom or molecule in water is involved in a million-billiontrillion or 1027 collisions with other atoms or molecules every second. This is part of the definition of temperature.) Eventually, by chance, it will be bounced so close to the surface of a wall or another larger particle that there are very few water molecules separating it from the surface. When that happens, those few molecules produce only a few collisions from that side, and the particle is overwhelmed by collisions from the other sides and tends to become “plastered” to the surface by a continual barrage of collisions from the solution. This is the “physical” half of adsorption. The “chemical” half occurs if there is any chemical affinity between the particle and the material of the surface. If there is, the particle will become attached (adsorbed) and stay there; if not, it will bounce off right away or just diffuse away, later.

The adsorptive forces (called van der Waals or London forces) are so weak that adsorbed substances can become desorbed rather easily—by adding certain acids, by heating the system, or by merely removing the contaminant from the influent water. For example, activated carbon filters or ion exchange beds nearing exhaustion are subject to desorption if the water quality suddenly changes for the better. That shows that these treatment techniques are equilibrium (balance) phenomena in which sorption and desorption both occur and achieve an average condition, like a well-matched tug-of-war.

Since adsorption requires a surface, commercial adsorbent materials have very large surface areas and are exemplified by activated carbon, activated alumina, and fine powders such as baking soda. But many substances are so very insoluble or otherwise so readily adsorbable that even small surface areas can make a big difference. For example, most heavy metal ions (lead, mercury, copper, cadmium, silver, chromium) adsorb so strongly to the walls of both glass and plastic sample bottles that more than half of the total contamination can be missed in an analysis if the sample bottles are not treated with nitric acid first, to cause desorption. Similarly, many chlorinated hydrocarbons like the polychlorinated biphenyls (PCBs) adsorb so readily to both metal and plastic plumbing and filter materials that even coarse prefilters remove them very well.

The adsorption and reduction of disinfectant chlorine by activated carbon is a special case. Activated carbon is a mild reducing agent and chlorine is a strong oxidizing agent, so after chlorine becomes adsorbed, it then actually reacts with the carbon. The chlorine is reduced to chloride ion (as in table salt and sea water), one atom of carbon is oxidized to carbon dioxide, and both are released to the solution (desorbed). Meanwhile, most of the spots on the activated carbon where all this took place become “auto-regenerated” back to their original, like new condition, ready to adsorb again. For free available chlorine (FAC), this takes only about fifteen minutes, which means that a small amount of carbon can achieve an acceptable steady-state condition if the flow rate is slow or intermittent. For “combined chlorine” (monochloramine), the reaction is much slower, and more carbon or more contact time is needed to achieve equivalent reductions. The chemical reactions between activated carbon’s “active sites” (C*) and these forms of chlorine are shown below. Note that any surface oxides on the carbon are recycled when reacted with monochloramine, while they are oxidized to CO2 and lost when reacted with free chlorine.

Free Chlorine
Cl2 + H2O ⇔ HOCl + H+ + Cl − (forming “aqueous chlorine”)
C* + 2Cl2 + 2H2O ⇒ C*O2 + 4H+ + 4Cl− (the overall reaction)
C* + HOCl ⇒ C*O + H+ + Cl−
C*O + HOCl ⇒ C*O2 + H+ + Cl−

Combined Chlorine: Monochloramine

$C^* + NH_2Cl + H_2O \Rightarrow C^*O + NH_3 + H^+ + Cl^-$
$C^*O + 2NH_2Cl \Rightarrow C^* + N_2 + H_2O + 2H^+ + 2Cl^-$
Finally, most dissolved/suspended particles and molecules in drinking water that are highly adsorbable to something usually do become adsorbed to a larger particle before reaching the point of use. Thus, adsorbable contaminants can often be removed by mechanical fine-filtration because the contaminant in question is already adsorbed to a larger particle. If you remove the particle, you remove the adsorbed contaminants along with it. This commonly applies to heavy metal ions, many pesticides, other chlorinated hydrocarbons, viruses, and asbestos fibers. About Activated Carbon: Granular activated carbon (GAC) and powdered activated carbon (PAC) are the predominant adsorbents used in our industry.

They can be made from nearly anything organic: coal, petroleum, wood, coconut shells, peach pits, ion exchange resin beads, fabrics, even waste plastics. The starting material is first charred—heated without air or oxygen, so it doesn’t burn up. Everything that can be vaporized or melted bubbles out as tar or pitch, leaving many holes and channels. Then the charred material is heated further, to above 1000陣C (hot enough to melt aluminum and lead), with the introduction of live steam or other activating chemicals. The superheated water vapor is extremely corrosive, etching more holes and extending channels to an amazing degree. Metallic impurities are preferentially attacked and washed out, resulting in a significant purification of the original material.

However, the heat of activation does more than extend holes and channels and increase the surface area of carbon; it also changes the fundamental crystal form from amorphous “carbon black” to the perfect crystalline array of graphite plates. The carbon atoms in graphite are arranged in sheets or plates of interlocking six-atom rings that look like slices through a honeycomb. Such a perfect arrange-ment causes the London forces to focus and concentrate at the surface, making activated carbon the best (strongest and most general) adsorbent known.
After activation, the carbon may be treated further to produce specific chemical qualities on the surface. For example, an acidic environment produces carbon with maximum capacity for heavy metals but minimal capacity for chlorinated organics, while an alkaline environment does the opposite. Most grades used in our industry are made for organic adsorption. When activation is complete, the carbon is a delicate, airy material that is so full of holes, it can barely hold together. It is crushed to a powder, and then proprietary binders are added to form granules of the desired size. The final product has a total internal and external surface area of more than 1000 square meters per gram, or half a football field inside a piece the size of a pea.

Activated carbon adsorption is useful because the material has strong chemical affinities for several important classes of contaminants that are common in water. These are:

1. Disinfectant chlorine: “Free available chlorine” (FAC) is readily adsorbed, then chemically reduced, and finally desorbed as chloride ion along with one molecule of carbon dioxide, with auto-regeneration of most of the carbon’s active sites and nearly infinite capacity. “Combined chlorine” (monochloramine) is less easily adsorbed, requiring more carbon or reduced flow rate for equivalent performance.

2. Organic compounds containing chlorine and other halogens: Simple halogenated hydrocarbons are highly adsorbable to activated carbon. This includes a great many pesticides (DDT, Endrin, Lindane, Chlordane, etc.), industrial solvents (trichloroethylene, trichloroethane, tetrachloroethylene, carbon tetrachloride, etc.), and disinfection byproducts (THMs including chloroform, chloral hydrate, etc.).

3. Organic compounds containing benzene rings: These include some of the most toxic chemicals, such as benzene, toluene, dioxins, polychlorinated biphenyls (PCBs), and phthalate esters (plasticizers for vinyls).

4. Heavy metals: Lead, cadmium, and mercury adsorb readily, both as dissolved ions and colloidal oxide or carbonate particles, but the capacity is limited—similar to the capacity for THMs.

5. Taste and Odor (T&O) compounds: The substances produced by microbes that are responsible for the common musty-earthy-mildewy T&O are extremely well adsorbed and with very great capacity

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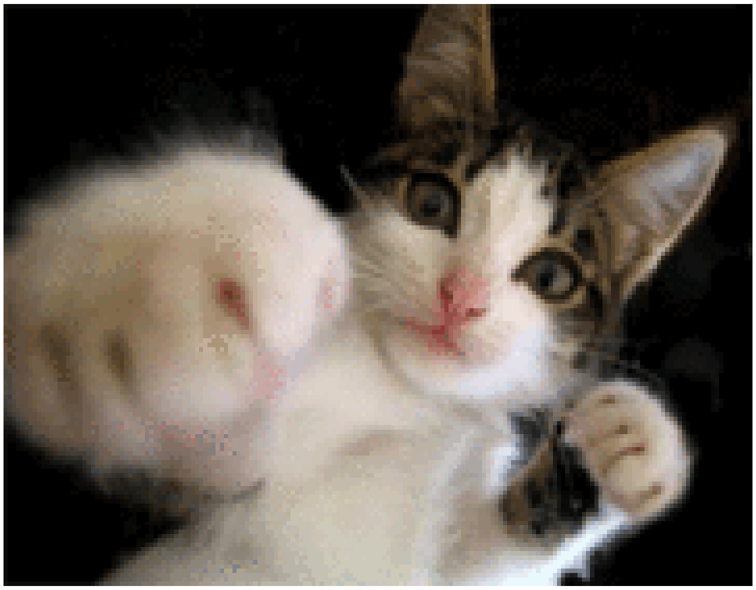

Urea Purification

Disclaimer: This tutorial comes from www.goldrefiningforum.com and can't be copied or reproduced without my authorization. This CAN'T be sold. By reading this, you agree to these terms. I'm not responsible of what you do with this stuff. This tutorial is only for informative purpose. Remember: Always wear good protection if you deal with chemicals. At the minimum, chemical resistant gloves and a respirator with acid gases cartridges.

Safety: **Urea is not considered as corrosive. Toxic ammonia gases can be released when heating Urea.**

Parts list:
-Your Urea
-One 4 liters bottle
-One measuring cup (1000ml or more)
-Coffee Filters
-Funnel
-Hotplate (optional but recommended)
-Mortar and Pestle

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1. First of all, put 500ml of Urea in your measuring cup and add hot water until you reach the 1 liter line. Swirl until all the urea dissolves.

2. When everything is dissolved, let sit 1 hour (you want the particles to settle). Your liquid will be very brown and will clear a little when it settles.

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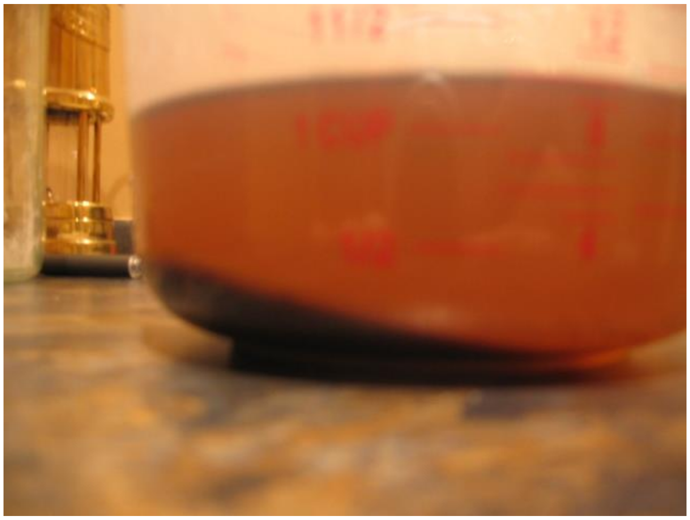

This is the sediments you don't want ;)

3. Now filter the liquid but try not to disturb the sediments. Don't expect getting a clear liquid with coffee filters. If you have lab grade filters, you may get better purity. You should get a brown liquid with little or no sediments.

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Interesting thing: I've let a coffee filter dry over night and look, Urea crystals have grown !

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4. You now have two choices. You can keep the liquid and use it like this to neutralize the acid, or get back the Urea. Personally, I do both. I think the best way to recover the Urea is to evaporate the liquid with a vacuum. But I do not own a vacuum bell so I had to heat the liquid. The thing is, Urea decomposes at 120°C so you must be careful. It releases CO2 and ammonia when it decomposes. So if it smells ammonia, you know you are heating too much. But even if you go higher than 120°C, you won't loose that much Urea...

Ok dried crystals should look like this (or better):

Now, put all that stuff in your mortar and crush it with your pestle. (Maybe one could use a coffee grinder but I did not tried)

It looks much better right ?

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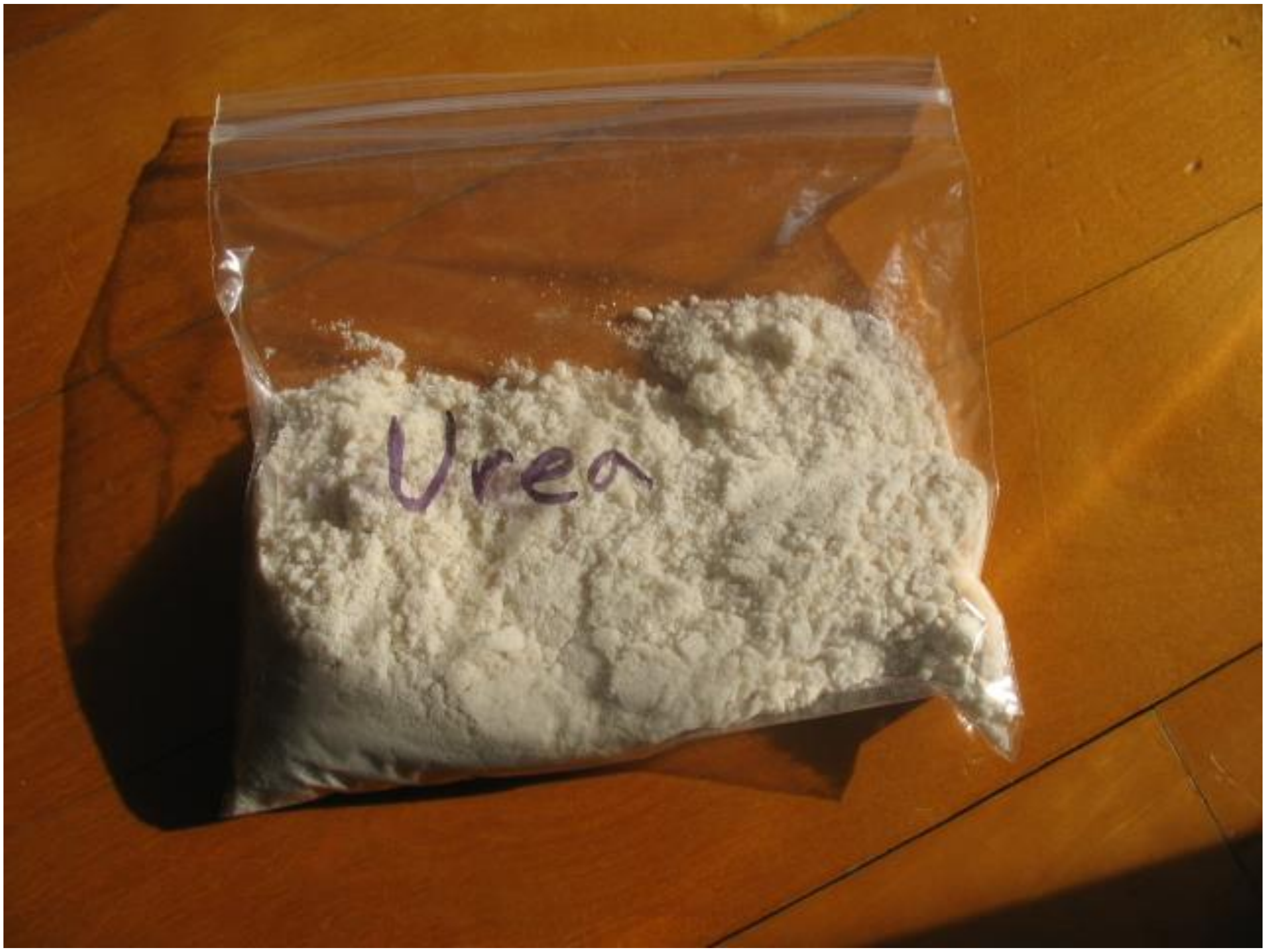
Final product !:



I hope you enjoyed this tutorial. More will come soon.

Feel free to Donate to help maintain this Forum. PM me (Noxx) for more info.

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NITRIC ACID BURN SAFETY

Your sample is to be dissolved in concentrated nitric acid while heating the solution on a hot plate as shown at the left. the student's hand really ought not to be exposed to the mouth of the other flask. The solutions of dissolved brass generally have a low volume and high acid and salt concentrations. "Bumping" or little explosions of steam in the superheated liquid can occur. You don't want your hand to be close to the mouth of the flask should the solution suddenly "bump" because drops of acid (not to mention part of your sample) will fly out of the flask and possibly onto your hand.

The image on the left shows the results of just such a spattering five days after it occurred. This student was holding a flask with the rubber Hot Hand and as the flask was removed, it "bumped", splattering her hand. The student washed her hand immediately with copious quantities of water and the instructor helped her to rub a slurry of sodium carbonate all over the spots. She then went to the student health center for treatment, but the hot concentrated acid had already done its work. The use of crucible tongs, as shown on the right, is a safer method for moving around flasks containing these solutions.

A similar spattering occurred recently in which five seconds passed between the time the droplets of hot nitric and sulfuric acid spattered onto the student's arm and hand and when she began rinsing off the acid in copious quantities of running water. The top pair of photos show where the droplets hit minutes after it happened. She thereafter visited the Student Health Center for medical treatment.

One week later.

Two weeks later

So as not to leave anything to chance, we can't emphasize too strongly that strong acids are to be treated with respect and care. The image on the right shows the result of a splash and extended contact of 3M sulfuric acid six months after it occurred. The student was working in a non-university laboratory. His supervisor spilled and splashed some 3M sulfuric acid but was not aware that any had gone beyond the immediate work bench area. The student was standing a few feet away. An hour after the splash occurred the student felt a burning sensation near his ankle. He was wearing elastic sweat pants with direct contact with his skin in that area. When he tried to pull up his pant leg to find out what was causing the burning sensation he noticed that the fabric had bonded (more or less) to his skin at the place producing the burning sensation. He realized what had happened and soon thereafter cut away the bottom of his pant leg and sought medical treatment. The remaining scar is a good indication of the long-lasting appearance of such an injury.

Any acid which contacts the skin must be washed off immediately. Acid which dribbles down the outside of laboratory stock bottles offers a familiar peril to students who use bottles of reagents in common.

One is not immediately conscious of such contact because even in the case of concentrated acids there is some delay before the reaction between acid and skin raises the temperature sufficiently for one to feel the effect. Students are advised to be conscious at all times of potential mishaps caused by their procedures and to be prepared to take immediate and appropriate action. If fingers or other areas having exposed skin feel wet when they ought not to be wet, the surface should be washed immediately in running water. The images at the left and right show the effect of either concentrated sulfuric or nitric acids which stayed on the skin for more than a few seconds (but less than a minute) before being washed off. Fortunately most discolorations caused by such contact disappear within a few days, but constant vigilance in the laboratory is the rule which should be followed at all times.

Continuing with the presentation of helpful hints, the presence of the concentrated sulfuric acid aids in the exclusion of the remaining nitrogen oxides from the hot solution. Those red oxides first boil off, then one sees fumes of sulfur trioxide beginning to escape, as at the left