RSB
PGM Plant Cleaning

Maximizing the value from Nitric Acid plant cleaning.

Recover more metal

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Introduction

Cleaning of Nitric acid plant can deliver huge benefits for owners and operators, firstly by enabling significant value recovery from the return of precious metals but also from increased plant efficiency associated with the removal of scale and deposits from the tube surfaces. Efficiency increases of up to 10% can be expected from successful cleaning operations.

This report outlines positive ways in which the plant operator can have an impact on the amount of metal recoverable and recovered from their plant; either during a clean down, while the plant is in operation and at end of life, when a plant is decommissioned and scrapped.

The report is presented in 5 sections as follows:

1 / Management of clean down projects and how to ensure that your contractor is working in your best interest.

2 / Simple yet often overlooked factors that have a significant influence on the amount of metal recovered.

3 / The hidden costs of Nitric Acid Plant Cleaning. Considerations for calculating and understanding the total costs of a plant clean down.

4 / Recovery Methodology – Pros and Cons and reasons for selection. Subtle differences that can have a significant bearing on the amount of metal recovered or how long the plant is offline for clean down.

5 / End of Life Destructive Cleaning – Recover 100% of the precious metal from your plant once it reaches the end of its useful operational life.

The author: R S Bruce Metals and Machinery Ltd developed the first commercial processes for precious metal recovery from Nitric Acid plants back in the 1980s.

The report will discuss and offer best practice opinion based on 33 years' experience covering 700 successful plant / equipment clean down projects and over 40,000 kg of precious metal recovered.

"This guide is based on over 33 years' experience from the world's leading nitric acid plant metal recovery specialist. The purpose of this guide is to give you practical and usable advice, to increase the amount of metal recovered and returned to you from your plant."

Management of Clean Down Projects:

In a critical path programme to repair, maintain or replace plant components the cleaning contractor should be consulted early on in the process to give help and advice.

Consideration must be given to the following points:

- Are PGM recovery and plant efficiency improvements considered within the aims and objectives of the project?
- What equipment can be cleaned in the allocated shutdown window?
- What is the best available technology for cleaning? Acid liquor - HPJ - Water?
- How can the contractor's time be best scheduled to work in sync with the shutdown programme?
- Where and how should clean down plant and equipment be placed?
- How will the communication between contractor and the plant engineering staff be managed during the works?
- How will you as the plant operator ensure that you have a permanent presence and working relationship with the contracting team?
- How will the acceptance of the contractors RAMS be managed?

Treat the clean down as a major aspect of the project; delays and problems in the clean down could impact the entire shutdown and restart of your plant. Agree a project plan with your contractor and discuss the project's critical path. What does the contractor need you to do to ensure that they are not delayed?

Communicate regularly: at the start and end of each shift make sure the contractor is on plan and ensure that they can get in contact with your personnel if there are urgent issues. We find that an ideal time for this is during the morning safety briefings.

During the clean down.

The precious metal oxides coating the surfaces of your plant and equipment will be transferred during the clean down process into a large volume of wash liquid from where they will be extracted by filtration, creating a residue / filter cake. This filter cake now contains all the recovered precious metal.

Remember – Filter cake can contain up to 200 grams / kg of precious metal. At a conservative $30,000 / kg for the precious metal, that's potentially $6,000 per kg. (In the filter press the moisture content can be between 40% and 50%.)

As the exact amount of precious metals that will be recovered is unknown and can only be estimated, it is down to you as the plant operator to ensure transparency with your contractor from the outset.

A few simple rules to follow can make this easier:

- Agree early on with your contractor that you want to be present at key steps of the clean down and when the residues are being handled.
- All filter presses should be locked and tagged, and the tags only broken in the presence of the client.
- All equipment brought on to site by the contractor should be cleaned and inspected prior to leaving site; pumps and hoses checked, filter presses emptied etc. Any material collected here must be added to the residues.
- Operators' representatives should not bring bags, containers etc. onto your site. If they do, they should be inspected before leaving. Your contractors must agree to a search policy.
- All precious metal residues leaving site should be weighed, sealed with tags in drums or tubs and listed on a site protocol document, only to be opened again in the presence of the client or the client's representative at the next stage of processing.
- Under current legislation, due to the nickel oxide content (threshold value 0.1%), cleaning residues should be transported to the refiner as a Hazardous Waste Material; failure to transport the residues correctly, especially across country borders, can lead to delays and substantial fines. Pre-planning and applying for the correct transport documentation in good time is the key to smooth completion and the fastest time from clean down to metal value delivery. For transport of residues across country borders it is recommended to start the permission process a minimum of 3 – 6 months before the cleaning. Your cleaning contractor should be able to help you with this process.

Simple yet often overlooked factors that have a significant impact on the amount of metal recovered.

1 - Research potential contractors. Unfortunately, as in any industry a significant gap exists between the best and worst service providers in terms of capabilities. Do your research, check references and speak with other operators to get recommendations. Plants have been damaged by companies attempting chemical cleaning without fully understanding the chemistry and metallurgy involved.

In the field of metal recovery, especially where there is material containing significant and unknown value, the opportunity exists for theft and dishonesty. The internet can be a useful resource checking for lawsuits and prosecutions relating to metal fraud.

2 - Share plant information with your contractor. Ensure that contractors have correct and up-to-date drawings and usage data of the plant. Doing this ensures that they know the materials of construction and catalysts used and allows an appropriate cleaning method.

Your contractor will also be able to prepare an estimation for you of the amount of metal that they would anticipate from the clean down process. It is not possible to be totally accurate here as some metal may have passed further downstream and also be in the product: however, this is usually a good benchmark figure for post-cleaning evaluation.

3 - Good coordination between facilities-management companies, plant operatives and your clean down contractor to ensure that requirements of the project's critical path are understood. The cleaning process is often not seen as critical in the grand scheme of the shutdown. It is important however, to ensure that your contractor has made it clear to you what they need access to, and when, during the shutdown, so that all the work can be completed, and you are not left waiting to get back online. They also need to understand when they must be able to hand back each item of plant they are working on. If your contractor has everything they need, then there is no reason why they cannot perform their job to the best of their ability. If you delay them, it is then difficult to hold them to the deadline or work schedule and have an effect on the metal recovery.

4 - Understand the metal recovery process and how many parties are involved and keep the number of parties to a minimum. The plant management should ensure that they are aware of the EXACT process to recover the elemental metals at the end of all the clean down work. It is a risk if the residues are being treated and conveyed through numerous contractors, refiners etc. Plant management should be aware of the diminishing value of their metals as they pass through 2nd and 3rd party contractors. There is an obvious risk here.

The hidden costs of Nitric Acid Plant Cleaning:

When compared on a direct basis, water cleaning is cheaper than chemical cleaning. This price difference is due to the costs of the chemicals and subsequent effluent disposal.

However, the comparison is not straight forward as, when carried out correctly, chemical cleaning will remove more coating from the plant surfaces, which in turn means significantly improved heat transfer being achieved after cleaning. This has a direct cost benefit to the operation of the plant.

Chemical cleaning methods remove more of the oxide coatings, leading to increased time between the need for cleaning, meaning the plant can run for longer uninterrupted.

Through our own testing and experience, gathered over more than 33 years, we know that chemical cleaning recovers more precious metal than any other method, and when done correctly causes no damage to the plant.

Recovery Methodology:

Not all clean down processes recover the same amount of metal from a plant. We have performed in house research to determine the levels of metal recovery from a variety of techniques:

Chemical Clean followed by High Pressure Jetting	>95% of Metal
High Pressure Jetting	90-95 % of Metal
Chemical Fill Cleaning with Agitation / Micro Bubbles	60-65 % of Metal
Recirculation Chemical Flood Spraying	50 % of Metal
Mechanical Cleaning (HTX - Tube Side Only)	45-50 % of Metal
Water Fill Cleaning with Agitation / Micro Bubbles	15-25% of Metal

Furthermore, not all methods are suitable for all circumstances. High pressure jetting is excellent for shell and tube heat exchangers and open surfaces but is not effective on obstructed surfaces, e.g. Lamont coils, where the surfaces of the lower coils cannot be accessed.

Cleaning involving chemicals must only be carried out by experts, using chemicals that are non-corrosive to the base metals of the plant, and that will not react with the residues in the plant. Where the design weight of the plant will not allow filling, Flood Spraying, which introduces significantly less weight of cleaning liquid, may be more appropriate.

Addressing plant operators' concerns

The common concerns about plant clean down are:

1 – Will this damage my plant?
2 – How long will it take?
3 – How much of the metal in the plant will be removed / recovered?

To answer these questions….

When carried out by a competent contractor, the cleaning methods listed above will NOT damage the plant, however in the hands of an inexperienced contractor they all have potential to cause damage, water washing without an inhibitor will leave oxidation on the mild steel; use of micro bubbles and agitation can cause deposits to be left behind baffle plates where they can seed corrosive damage; cleaning with a chemical that is too aggressive or incompatible with the metallurgy of the plant and catalyst deposits could cause significant damage: mechanical cleaning can cause surface wear and cause precious metal to fuse to the steel of the plant rather than removing it. It is therefore essential to ensure the contractor is experienced and has a good track record with the techniques they are suggesting.

The length of time required will depend on factors such as the contractor's experience, access to your plant and the method used. As a rule of thumb Chemical Treatments should be expected to be about three times faster than Water Washing. So, if a boiler clean takes one day using the

Chemical Cleaning method, then factor in three days for Water Washing. For single pass High-Pressure Jetting (after the equipment is set up) you should expect 100 – 120 tubes per hour to be completed.

The amount of metal removed from the surfaces of the plant will vary depending on the cleaning method, but the numbers listed previously are a good guide on which to base expectations. These are based on 33 years of experience and over 700 successful plant / equipment clean down projects and over 40,000 kg of precious metal recovered.

End of Life Destructive Cleaning - The only way to recover all of your metal

Once your plant has reached the end of its working life it can be cleaned destructively to recover up to 100% of the precious metal oxides. At this stage damage to the plant is of no consequence.

Our top tips for ensuring you get the best return here are:

- As with any clean down choose your contractor wisely.
- Invite the contractor to site well in advance of the removal so that they can discuss the recovery method with you. At this stage you will be discussing at least the disconnection and extraction of the unit, potentially the demolition of the whole plant and even the building. Here you are making sure that there are plans in place to safeguard the precious metal in the plant.
- Protect the redundant plant items from the environment once they have been removed, allowing rain into the plant can wash away any loose metal oxides just like water washing. Shrink-wrapping is an excellent way to protect from wind and rain and help protect the value.
- Safeguard metal during transportation, as a minimum any unsealed openings need to be wrapped in protective coverings, i.e. shrink wrap.
- Consider how you are going to manage the representation and inspections at the contractor's site whilst they are recovering the precious metal from your plant.
- Ensure that the contractor has a recovery plan that that will outline how the maximum amount of precious metal will be recovered from your plant.
- Confirm the contractor's sampling and analysis plan. Do they have on site laboratory facilities or do they use a third party?
- Understand whether your contractor has their own site with dedicated and clean, covered spaces where your equipment can be processed. Is this site secure 24/7?
- Check how many parties are involved. Does your contractor work directly with a refinery? Every set of hands that your material moves through potentially loses metal and costs you money. The risk in the care, custody and control of the PGM increases.

- If the plant / equipment is to be re-sized for shipping, be sure that during this process all of the cutting dust is collected and shipped with the plant. This dust contains precious metal.

An analogy once used during a presentation at an AN-NA conference: -

"When giving a contractor a nitric acid plant for PGM recovery, it is like handing over suitcases full of wet $100 bills to be dried, pressed and bundled. And then asking the contactor to tell you the total value once they have counted them, together with their costs"

We hope that you have found our guide interesting and thought provoking and that you feel it has been a worthwhile read. On Page 10 & 11 of this guide you will find a "Plant Questionnaire" detailing the information required to enable us to estimate the potential PGM recovery from your plant. This service is provided free and without any obligation by sending the completed form to: enquiries@rsbruce.com. Please contact us if you prefer to sign a confidentiality agreement before providing the plant details.

To bring home some of the points detailed in this guide, we have attached an Annex with some stories and anecdotes that we have heard over the years that may help to re-enforce some of what we are saying.

With warmest regards,

Richard Bruce

Richard Bruce.
Chairman and Founder R S Bruce (Metals and Machinery) Limited

ANNEX:

Over the years we have heard many stories and anecdotes that highlight the importance of vigilance by the client. Here are a few examples….

- A contractor **used the wrong acid on a plant that had been operated with a copper-based catalyst**. Significant damage was caused to the plant when an exotherm occurred delaying the re-start and incurring costs for repairs. These costs were in excess of $5M.

- Contractor used **dilute sulphuric acid when cleaning a mild steel boiler** with Lamont coils. Significant corrosion damage was caused which also delayed the entire shutdown and re-start of the plant.

- **Filter presses used on plant clean down removed from a customer's site full of residue**. Only by chance did the customer ask to have the filter press checked as it was leaving site on a lorry after completion of the job. The contractor had "*forgotten"* to empty it. We also heard that a contractor left **residue within the lining of the portable collapsible Tank** used on the job. They had "*forgotten*" and packed it away with their equipment.

- Due to **incorrect selection of chemicals and insufficient ventilation** one operator was surprised by a loud bang and blue flames as the Hydrogen generated ignited in the plant. Luckily no one was hurt. The plant was damaged as a result of poor knowledge of basic chemistry.

- A clean down contractor **pricing work at an initial loss**, so cheap that they were sure to win the work. The customer thought they were getting a great deal however; the contractor unlawfully took a portion of the customer's residues, without the customer's knowledge or permission once they had completed the job.

- Operator and client had **not fully discussed the program of works and agreed on an appropriate plan**. As a result, when the time came to clean the Nitric Acid storage, the operator had not emptied the tanks and they could not be cleaned out during the shutdown as planned. There were additional costs incurred by the client for the contractor to return and complete the clean down at a later date.

- **Fraudulent analysis of residues**. Reporting the incorrect metal content so that the full value of metal was not known to the client. This could have been avoided if the client had taken more care on the chain of custody of their material. The issue became apparent when mistakes were made, and contradictory analysis was discovered.

- **Oversampling**. One contractor was understood to be taking excessive samples of the dry precious metal bearing residue. During a customer visit to the contractor's site, the question was raised as to why so many samples were taken and why they were not returned to the batch after testing. There was significant metal in the samples. This highlights the value of representation at the contractor's site to ensure that correct procedures are followed.

R. S. Bruce Plant Clean Down Questionnaire - Page 1

To receive a free estimation of the potential PGM recovery from your Plant, please complete

Site Address	Contact Name	Contact E-mail	Contact Telephone

Plant Details

Boiler Manufacturer	Date Plant Commissioned	Plant Pressure	Operational Yes / No	Number of Burners

Design Output per Burner of Acid in Tonnes Per Day (TPD)	Production Time Days Per Year (DPY)	Primary Metal Loss (Mg/ton 100%) Acid (PML)	Gauze supplier

Actual Output per Burner of Acid Per Day	Basic Nett Loss Calculation (Years x TPD x DPY x PML)

Tonnage Produced Since Commissioning "Or" Since Last Clean Down

Catchment Installed	Date Catchment Installed If Applicable	Catchment Efficiency (%)

Previous Clean Downs (If Applicable)

Date of Clean Down (Month and Year)	List of Components Cleaned (Boiler, tail gas heater etc...)	Cleaning Method (Chemical Treatment, High Pressure Jetting etc.)

PGM Recovered in Last Clean Down (kg)	
Platinum	
Palladium	
Rhodium	

Any Noticeable Efficiency Improvements

this form and send it to: enquiries@rsbruce.com

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Storage Tanks

Number and capacity of storage tanks

Tank 1	Capacity	M^3	Diameter	Height
Tank 2	Capacity	M^3	Diameter	Height
Tank 3	Capacity	M^3	Diameter	Height
Tank 4	Capacity	M^3	Diameter	Height

Date of any previous cleaning of the storage tanks

Other Equipment

Any Redundant Components on site? Including service history and removal date

Any further discussion or relevant information

Campaign Lengths, Next Planned Shutdown etc.

Drawings

P&I Drawing for the plant. (Or basic Flow Sheet)
Detailed Drawing of Boiler, Tail Gas Heaters, Economisers, Cooler Condensers and Storage Tanks showing surface areas, tube dimensions, tube diameters, metals composition and inlet/outlet temperatures.

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To request your own copy of this guide visit:

https://www.rsbruce.com/nitric-acid-plant-cleaning-best-pra

Maximising the Value From Nitric Acid Cleaning Version 1 - November 2019