Platinum Care

Extending the life of your Labware

Introduction

The harsh environments that platinum labware is exposed to can significantly reduce the lifetime of the product and the accuracy of the measurement processes associated with it. In this document a brief summary is given on how to look after your labware to ensure optimum lifetime.

Platinum is one of the least reactive metals due to its high chemical resistance even at elevated temperatures. It does not suffer attack by any single acid nor can it be oxidised in air at any temperature. These properties make this metal an excellent choice for sample preparation of fusion samples or wet chemical digestions. However there are many substances that can attack platinum and combine with it at relatively low temperatures.

Conditions to Avoid

On occasion, platinum crucibles can suffer cracking or holes during fusion processes or sample ignitions. Not only is this an annoying expense for the analyst, it can also cause damage to fusion furnaces due to leaking molten mixtures. The failure of the crucible is not necessarily due to manufacturing defects or poor metal quality. The cause of these detrimental effects is the chemical reaction and alloying of the platinum crucible with reduced metals due to incorrect procedure or a failure to maintain oxidising conditions during fusion processes.

Conditions to Avoid

The detrimental effects of different substances can vary greatly. In some cases, a platinum crucible can suffer considerable reaction and alloying with a certain metal over a long period of time without significantly limiting its usability. Apart from cross contamination of samples due to leaching, the crucible can still maintain its physical integrity. In other cases however, even a few hundredth of a milligram of a different type of metal can completely destroy a platinum crucible.

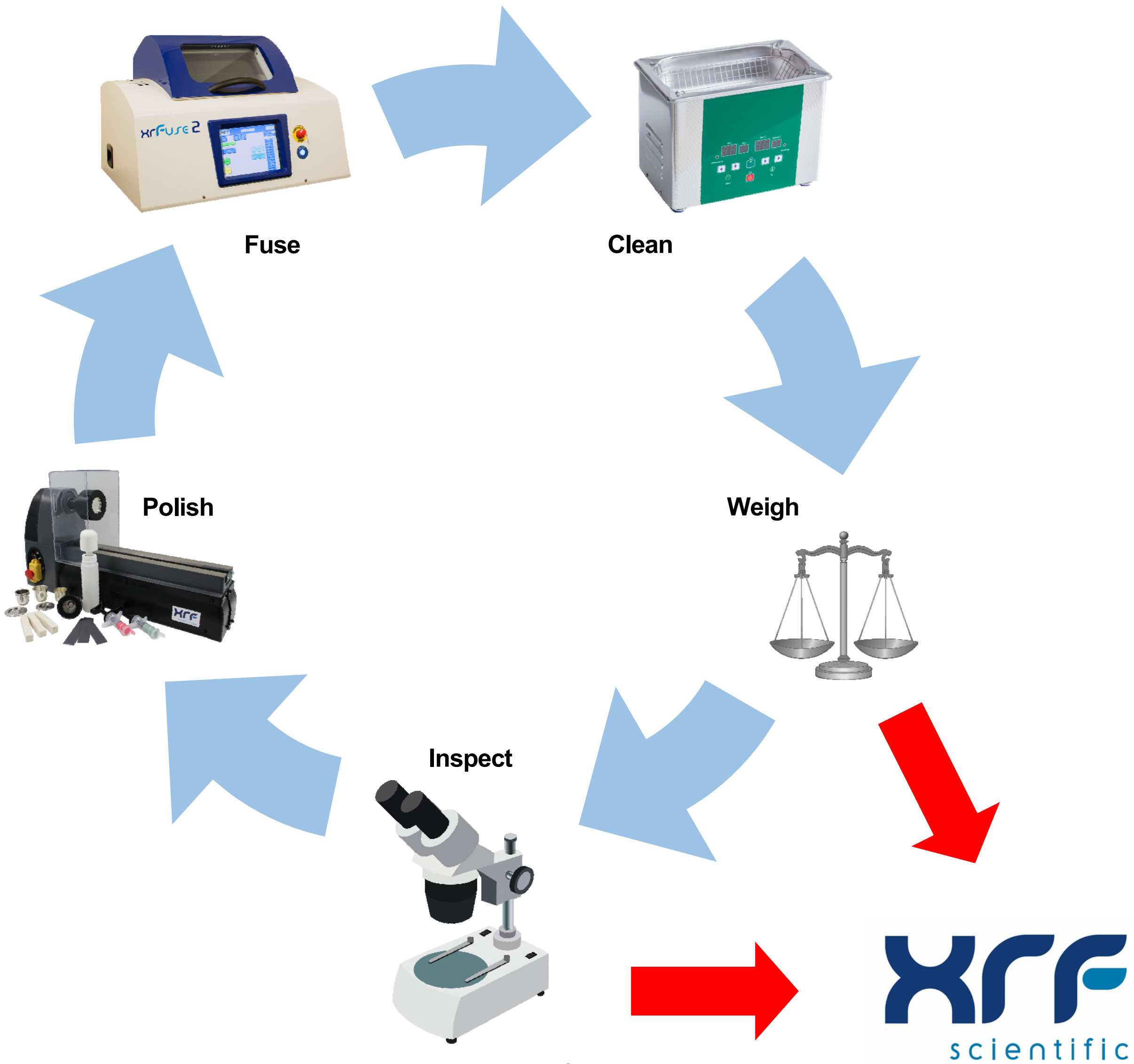
Cr, Mn, Fe, Co, Ni, Cu, Zn are metals that will easily react with platinum alloys at elevated temperatures however do not significantly cause damage to the crucible unless the contamination is very high or the crucible is subjected to temperatures above recommended operating conditions i.e. 1200°C. These metals tend to subsequently leach out of the platinum alloy and contaminate other samples. Evidence of high contamination will be small cracks in the base or lower walls of the crucible.

Ag, Sn, Sb, Pb, Bi are metals that will readily alloy with the platinum crucible even at quite low temperatures and reduce the melting point of the platinum causing holes. A common cause of failure is when these metals are present during low temperature ashing and then the crucible is heated to fusion temperature causing the platinum to melt. Even a minute amount of these metals is enough to cause severe ‘corrosion’ of the crucible inner walls and base resulting in destruction.

Si, P, S, As, SiC are non-metals but can still have devastating effects on platinum alloys. These elements also form low melting alloys with platinum but their effect is very destructive. In particular, silicon and phosphorus have a strong affinity for the platinum metal and forms alloys along the grain boundaries. This alloying effect occurs very fast and subsequently causes cracks. If the crucible crack is examined under a microscope, the metal will appear to have a crystalline structure where the metallic grain has ‘unzipped’. If the contamination is more severe, the crucible will simply fall apart. This type of contamination is the most common cause of platinum Labware failure despite the added caution on the part of the operator. For example, even small amounts of organic material or a momentary lack of oxidising conditions is enough to reduce silicon dioxide to the elemental form which quickly reacts with the platinum.

Process Summary

The following steps provide detailed instructions on how to follow the XRF Scientific Platinum Care process:



Handling

Tools:	Platinum tipped tongs
Consumables:	None
How often?	Every time

When picking up a hot crucible / mould, platinum tipped tongs must be used to prevent contamination. Platinum Labware should be placed on clean surface at all times to prevent base contamination and corrosion. A clean sheet of paper is a good substitute.

Fusion

Tools:	Fusion machine
Consumables:	Ammonium Iodide tablets or Flux containing oxidants and release agents
How often?	Every fusion run

During the fusion process, always maintain oxidising conditions to prevent metal formation particularly when fusing reducible matter. This can be achieved by either pre-oxidising the sample before fusion or adding an oxidant to the flux/sample mixture. If any metals are present in the sample e.g. Fe, Cr, Ni, Cu, Zn, Pb, Sb, Sn, As, P, S, Si, they will readily react with the platinum crucible often forming brittle alloys and “corrosion”. The crucible will then subsequently fail with repeated heating and cooling. The addition of ammonium iodide tablets to the fusion process ensures the best outcome when releasing a bead from the mould and reduces release/sticking related damage**.** This is particularly effective where the tablet is added after the process has started. Alternatively a release agent can be added to the flux/sample mixture prior to fusion. Do not carry out direct fusions with caustic alkalis, nitrates, cyanides or nitrides in platinum labware.

Cleaning

Tools:	Ultrasonic Bath, drying oven, platinum tipped tongs, Crucible cleaning holder, Mould cleaning holder
Consumables:	Citric acid
How often?	Every fusion run

Crucibles and Moulds should be placed in a custom holder to ensure no cross contamination and to maximize cleaning effectiveness. Using citric acid (20%) in an ultrasonic bath set to vibrate at 50C for 10 minutes will remove any adhering flux. The citric acid solution should be replaced regularly particularly if the solution is dark yellow or viscous. The labware should be dried by using a clean air stream or placed in a drying oven.

Weighing

Tools:	Weigh scale
Consumables:	none
How often?	Daily

The weight of the labware should be measured on a daily basis. This is the best indicator that the product has reached a point that it should be remade. If the mould has reduced in weight by more than 10% of specification due to polishing and/or usage, it should be returned to the manufacturer for remake.

Inspection

Tools:	Magnifying glass/ microscope
Consumables:	None
How often?	Daily

The moulds should be inspected for holes, scratches and blemishes and if required, should be polished to restore an unmarked mirror surface. Polishing will also renew the mould surface and reduce the probability of sticking beads. After each polishing session, the moulds should be tested for straight edge reflection to ensure that the surface is flat. Crucibles should be regularly checked for integrity of shape and/or alloy 'corrosion'. If crucibles are damaged or exhibiting cracks/corrosion, they should be returned to the manufacturer for remake.

Polishing

Tools:	Polishing jig
Consumables:	Polishing oil, 1000 grit paper, soft tissue, 14, 8, 3 micron diamond paste, felt pads.
How often?	Based on inspection

Polishing is designed to correct for minor distortions and blemishes to the surface. Major structural issues require remake. When polishing is required, the following process should be followed:

1. Place the mould in a mould polishing jig and screw on the retaining cap.
2. Place the mould polishing jig into position by screwing it onto the polishing lathe spindle.
3. Ensure the polishing jig is firm by holding the shaft steady with the push rod.
4. Remove the push rod from shaft.
5. Start the polishing lathe and adjust the speed to 60%.
6. Lightly spray some polishing oil onto the 1000 grit paper.
7. Use 1000 grit paper to lightly prepare the surface, and then clean the surface with a soft tissue
8. Squeeze about 3-4mm of the 8 micron diamond paste onto some clean felt pad and press onto the mould surface before starting the polishing machine. This will minimize the diamond paste from 'flicking' off the polishing surface. Now start the polishing machine

and introduce the felt pad to the mould surface. Do not use the polishing oil at this stage. Always use a circular motion with a backwards and forward motion across the surface of the mould and try to concentrate equal effort on the outside and centre of the mould while polishing machine is spinning.

9. Spray a small amount of oil onto the mould and reintroduce the felt pad. The oil will thin out the diamond past, giving a finer finish and will be easier to remove completely using a soft tissue while the polishing machine is turning.
10. Once the 8 micron diamond paste has been removed, repeat stage 8 and 9 using the 3 micron diamond paste and a new felt pad. Once again, finishing by cleaning with a soft tissue.

Note:
When "working" the surface of the mould with either grit paper or diamond paste ensure the centre of the mould receives equal attention due to speed differences as you move outwardly towards the edge. To maintain good release of glass disks, hold the felt pad perpendicular to the bevelled surface during the polishing process and clean up as required. The use of polishing oil should not be used at the start of the diamond paste stages as it reduces its cutting effectiveness and more effort and diamond paste will be required. Note excessive pressure is NOT required, let the grit paper and the felt pads do the task for you by applying light - firm pressure only. If a 'wobbling' effect occurs whilst applying the felt pad then you are applying too much pressure. Be very careful in between stages not to cross contaminate the abrasive compounds. Ensure the 1000 grit paper, the 8 micron and the 3 micron polishing media are kept separate. Cleaning the mould surface completely with a clean soft tissue should also help to minimize this. If a finer mould surface finish is required, the polishing process can include a three stage diamond paste procedure i.e. 14um – 8um – 3um.