

A BCA Industrial Group XRF Application.

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Uses and abuses of platinum ware in the XRF laboratory

The accuracy and precision of data reported by an XRF laboratory reflects care taken by the analyst to ensure that appropriate techniques are adopted to prepare the sample and calibrate the spectrometer. Many laboratories have systems in place to compensate for short and long term drift in the spectrometer with analysts following quality procedures for calibration maintenance. However, the maintenance of sample preparation equipment must not be overlooked and where fusion techniques are adopted, the care of platinum ware is vital. Good analytical practice should minimise cross contamination between samples, but in a busy laboratory fusing a wide range of powdered materials careful handling and cleaning regimes will reduce errors and extend the life-time of the platinum ware.

Platinum ware for XRF analysis

Platinum is used for laboratory ware because it is resistant to chemical attack, even at high temperatures and it can be readily fabricated. The addition of 5% gold gives an alloy with improved high temperature strength and non wetting properties. Crucibles and casting dishes vary in shape and size and are selected to satisfy the needs of the fusion process adopted. Techniques may be manual or automated with heat provided by gas burner or electric muffle furnace.

Samples in the form of oxides, carbonates and silicates may be readily fused with borate fluxes at temperatures up to 1250 °C with users reporting more than 1,000 beads cast from one dish. However, some samples such as ferro-alloys, carbides and spent catalysts from oil refineries represent a greater risk. Samples with incomplete history and “unknowns” presented for semi-quantitative analysis may also challenge the platinum ware and laboratory budget.

The advantages of presenting a sample for analysis in the form of a fused bead are well documented. However, it is perhaps important to highlight situations that are harmful to the platinum / 5% gold alloy.

- Inappropriate handling and accidental physical damage should be avoided.
- Platinum ware should only be handled with platinum tipped tongs
- Avoid prolonged unnecessary heating; this will limit crystal growth and cracking.
- Avoid reducing gas flames
- Fusion should be performed under oxidising conditions
- Crucibles must not come into contact with SiC heating elements
- Regularly remove any particles of SiC that have spalled from the heating elements on to the floor of the furnace.
- Ensure that metals such as Cr, Fe, Ni, Cu, Zn & Pb do not come into direct contact with the platinum ware.

- Fine metal powders should be pre-oxidised before fusion using an oxidant such as sodium nitrate, potassium nitrate or strontium nitrate.
- Use separate crucibles for samples requiring pre-oxidation
- Minimise cross contamination using a thorough cleaning regime for all platinum ware.

Cleaning platinum ware

Routine cleaning of the platinum ware will reduce cross contamination and extend working life. A crucible or casting dish should never be hit or tapped with any implement to remove residual fused sample, stuck to the surface. Any residual material should be removed by one of the following methods:

1. Soak in 10% solution of citric acid at 50°C in an ultrasonic bath. This method can be slow for some materials but it is thorough and gentle.
2. To shorten the cleaning cycle, use 20% w/v citric acid in a beaker at 80°C
3. For a more aggressive method use 10% HCl
4. If all else fails, boil in Cl free HNO_3
5. After acid cleaning, wash thoroughly with clean water and dry

WARNING: Never use Aqua Regia.

To check for possible cross contamination when developing sample preparation methods, fuse and analyse a blank (100% flux) bead. Crucibles used to prepare samples containing copper compounds are particularly difficult to clean. Fortunately, the presence of a trace of copper will be revealed by a blue discolouration in a blank bead.

Polishing platinum ware

Spectroscopists using semi-quantitative programmes for the analysis of fused beads will be familiar with the presence of trace levels of both Pt & Au in a reporting list. Gold lost from the surface will reduce the non wetting properties of the ware. The sheen from the surface of new platinum ware will have been replaced by a dull appearance; crucibles and fusion dishes may be deformed and the surface of the casting dish will no longer reflect the analysts face. Scratches and the “orange peel” effect showing the presence of grain boundaries become familiar sights. The crucibles will be more difficult to clean and deformations in the casting dish may cause difficulty in releasing the bead and will increase errors in the reported result.

Some degree of maintenance on the platinum ware can be carried out by the user. With a suitable die and hydraulic press, casting dishes can be re-pressed to keep them perfectly flat and formers may be available from the supplier for crucibles and dishes. The most effective form of maintenance however is polishing. A diamond paste of the correct grade can be used to polish the surface of the platinum alloy. This will remove a small amount of the surface to get back to the original alloy. It will also have the effect of smoothing out any minor scratches or imperfections on the surface. For more serious

scratches and surface damage, a coarse diamond paste can be used, prior to final polishing with a fine paste.

The life of platinum ware can be extended by careful use and polishing, but it should be noted that polishing will reduce the weight of the platinum, subsequently reducing the amount of credit on replacement. In time, the platinum will crystallise and crack to which the only solution is replacement. Polishing should primarily be considered as a means of maintaining the quality of results and not as a means of artificially extending the life of the alloy.

The polishing kit supplied by Analysco comprises one or three grades of polishing paste.

Grade A – for routine polishing to maintain surface and finish

Grades B and C – Two stage grinding and polishing for surface damaged casting dishes

Lubricant and cylindrical felt bobs for polishing casting dishes are also included.

A small amount of diamond paste should be placed onto the flat, end surface of the polishing bob. Taking care to keep the surface of bob flat on the casting dish, polish with a circular motion, adding small quantities of lubricant from the dispenser, as needed to keep the mixture fluid. After polishing, the platinum should be washed in warm water with detergent and dried. If stored carefully, the polishing bob can be used many times over. Make sure however if you are using different grades of polishing paste, to keep the bobs separate and appropriately labelled.

Polishing by hand can be quite labour intensive and therefore some users may consider using a power tool. The bobs are fitted with a shank for fitting in the chuck of an electric drill which could be mounted in a suitable stand. Appropriate safety rules should be observed in this case. Additionally, it must be stressed that a great deal of care needs to be taken when using a power tool. Platinum will be removed much more quickly and if the bob is not perfectly flat against the surface of the casting dish, gouges can be made in the surface. Further advice on this method is available if required.

The basic polishing kit comprises

1. 20g syringe of grade A polishing paste
2. 20g syringe each of grade B and C grinding pastes
3. Set of 10 felt polishing bobs, 32mm or 38mm diameter
4. 1 litre of oil based lubricant
5. 1 x 250ml pump up lubricant dispenser

Margaret West
West X-Ray Solutions Limited