



SPEX/Katanax

Automatic Fusion Machine

User Group

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Non-Wetting Agents in the Preparation of XRF Fusion Beads

In the preparation of a fusion bead for XRF analysis, numerous factors can affect the viscosity of the melt and how much it adheres to the platinumware. If factors such as flux type, fusion temperature, pouring motion and sample concentration have been optimised, a non-wetting agent (NWA) may need to be investigated if your beads are still cracking or not releasing from the mould.



What is a Non-Wetting Agent?

The role of an NWA is to promote cohesion of the melt by influencing their surface tension. By increasing the cohesiveness/surface tension of the molten glob of glass, it tends to release from the crucible better and leaves behind less residue. NWAs are halogenated compounds and are added in small amounts, typically $<0.5w/v$.

In practice, Iodine and Bromine are the only useful halogens for fusion beads. Fluorine and Chlorine are impractical as their smaller atomic radius results in them migrating towards the bulk, rather than remaining at the surface where they are needed. As a result, the most common NWAs are LiBr and LiI, while other compounds that can be used include KBr, KI, NaBr, NaI, CsBr, CsI etc.

Post Pouring Effects of NWAs



Since adding NWA increases surface tension, the addition of too much NWA will result in incomplete filling of the mould, highlighting the need for optimal addition levels whereby a balance must be met between mould filling and mould release.

In addition, optimising the amount of NWA extends the life of platinumware by not sticking to, warping or tearing platinum from the mould surface. This in turn also reduces maintenance for the technician and protects expensive platinumware.

NWAs and Analyses

The addition of an NWA will have 2 effects on your analyses.

1. As Br and I atoms are large they are primarily found on the surface of your bead. This will result in a small amount of XRF signal attenuation
2. The presence of additional elements such as Br and I may result in some line interference e.g. Br over Al, Mn over Rb, I over Ti and Ba over Sr.

Thus, the quantity of NWA needs to be optimised to minimise these effects. Furthermore, knowledge of the quantity of NWA added, can allow for it to be corrected for via instrument software.

Fluxes and NWAs

Many flux manufacturers have now introduced Lithium Borate blends that incorporate NWAs.

Before the NWA can take effect, the flux needs to be molten to release it. However, the incorporation of an NWA into the flux significantly reduces the evaporation speed of the flux, in particular for Iodine.

Some other factors to take into account are:

- NWA content may not be as accurate as the manufacturer specifies and may also vary from batch to batch.
- The amount of NWA in a blend may be too high for your specific application, so it may need to be mixed with plain flux

Some other things to be aware of:

1. NWAs may react with your sample creating compounds that evaporate at faster rates
2. Samples containing Fe, Cr and Cu can be quite sticky, requiring higher than recommended additions of NWAs

Br vs. I for NWAs

On an atom by atom basis Iodine has a greater non-wetting effect than Bromine. That said, Iodine compounds added separately (not incorporated into the flux blend) tend to evaporate faster and at lower temperatures, making brominated compounds a better choice for this scenario, although interference issues need to be taken into account when choosing your NWA.



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