

MARK WAINWRIGHT ANALYTICAL CENTRE UNSW

XRF FACILITY

METHODOLOGY:

PREPARATION of GLASS BEADS for XRF Major Element ANALYSIS:

40mm glass beads are prepared using the following weights*, weighed out onto wax paper and poured into a glass vial:

1. 1.0000 grams of powdered sample which has been pre-dried.
2. 10.0000 grams of either Type 1222 or 2212 flux, depending on sample matrix

*These are typical weights; the ratios are sample type and calibration program dependent and might need to be altered.

The three ingredients are mixed thoroughly in a glass vial and transferred to a Pt/Au crucible. The crucible is placed in a high temperature muffle furnace (either the Fusilux or Eagon) at a temperature of 1050 degrees Celsius and allowed to melt and mixed for 15 minutes. The molten flux and sample is poured into 40mm Pt/Au casting dishes and left to cool before labeling. L.O.I. (loss on ignition) is done on sub-samples at the same fusion temperature as the beads. This loss indicates the presence of organics, volatiles and adsorbed moisture.

Prepared glass beads are typically measured on either the PW2400 WDXRF Spectrometer or the Axios Advanced WDXRF Spectrometer. The appropriate analytical program is selected, which has been calibrated using certified reference materials and using optimal instrumental operating conditions (e.g. choice of crystal, counting times, line overlaps, background measurement and so on). An in-house secondary control is used to ascertain machine performance and drift monitors are run daily. All elements analysed are expressed as wt. % oxides.

Instrumentation:

PanAlytical PW2400 WDXRF, end window Rh tube, 3 kW

PanAlytical Axios Advanced WDXRF, end window Rh tube, 4 kW