Protocol on Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

Water Extraction Regale:

Weigh approximately 100 mg of sample in an Erlenmeyer flask,
Add 2.5ml H_2O milli-Q, 7.5ml HCl conc. 2.5ml HNO_3 conc.
Allowed to react overnight.
Reflux for 2 hours then filter with 0.45um.
Make up to 50ml.
Analyse by ICP_EOS.

Hydrofluoric Acid Extraction:

Weigh approximately 100 mg of sample in a Teflon container,
Add 10ml of HNO_3 conc. Wait 2h reaction
Place the tubes in the microwave oven
Add 5 ml of hydrofluoric acid
Place the tubes in the microwave oven and then check if all is well dissolved, otherwise add 5ml of hydrofluoric acid, and then return to the microwave oven.
Add 20ml of boric acid to neutralize the hydrofluoric acid
Place the tubes in the microwave oven
Threading 0.45um and made up to 50ml.
Analyse by ICP_EOS.

Contamination:

- Concerning the major components:
Some elements are different or not detected with HF, I suppose an evaporation reaction as with silica.
Like the reaction:

SiO_2 (s) + 4 HF (aq) = SiF_4 (g) + 2 H_2O (l)

- Concerning the trace elements:
For extractions ER no impurities detected in your sample in the composition of Pyrex, apart from the Si, it shows that the glass does not contaminate the samples.
For extractions with HF we use TEFLON which contains no element to be quantified for your analyses, so no possible contamination.

- For the case of Si:
L'ER does not dissolve the Si-O, so we underestimate the silica values.
HF dissolves the Si-O bonds, but there is a loss in gas form according to the reaction:

SiO2 (s) + 4 HF (aq) = SiF4 (g) + 2 H2O (l)

Over- or under-estimation:

The final results are given in mg/kg, this means that we report the results in mg / l on the mass of starting samples.
In the case of the ER results correspond to the portion of the sample dissolved by a strong acid.
In the case of HF results correspond to the total sample because everything is dissolved.

Improvements:

There are no other means in our laboratory to improve these results.