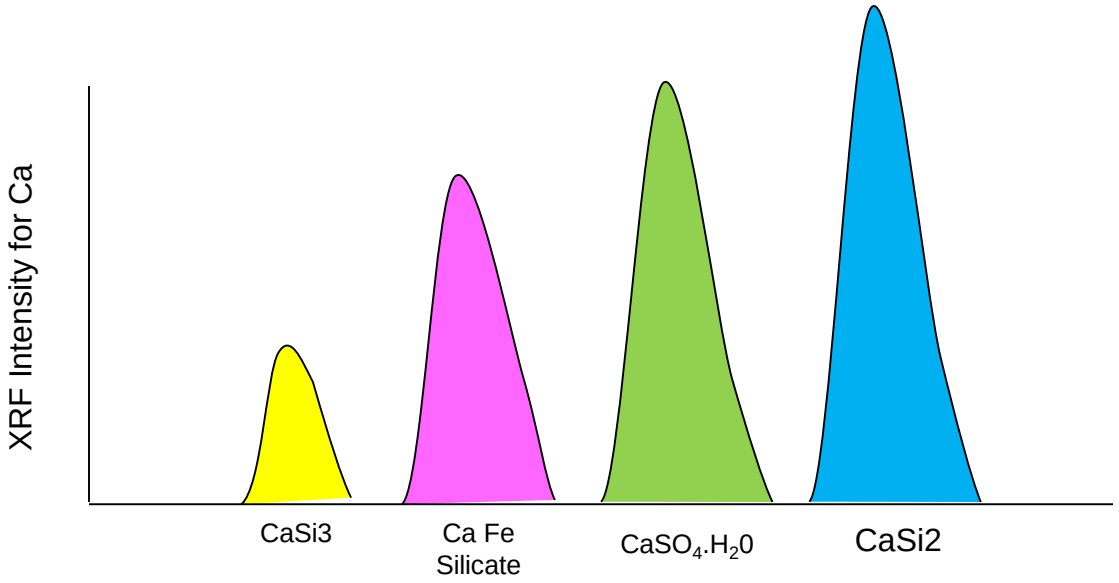
INCREASING THE EFFICIENCY OF FUSIONS FOR XRF

NAVAS INTRUMENTS – JOSE LAS NAVAS

USING FUSION FOR XRF ANALYSIS IN THE CEMENT INDUSTRY

Due to complex chemistry of Portland and Pozzelanic Cements dissolution of the clinker phases necessary to get meaningful XRF results

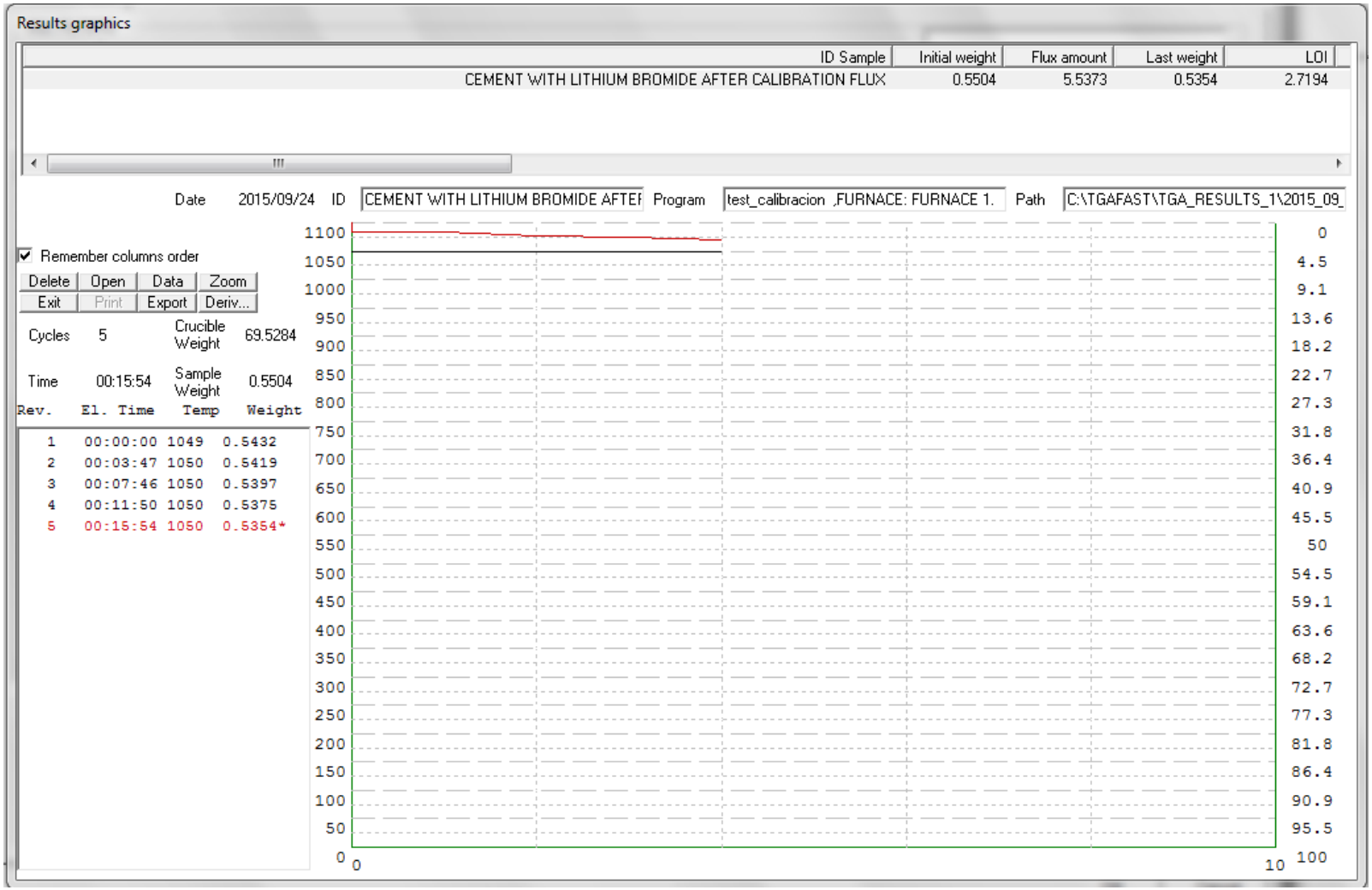
ONLY WAY TO ELIMINATE OR COMPENSATE FOR THIS EFFECT IS FUSION



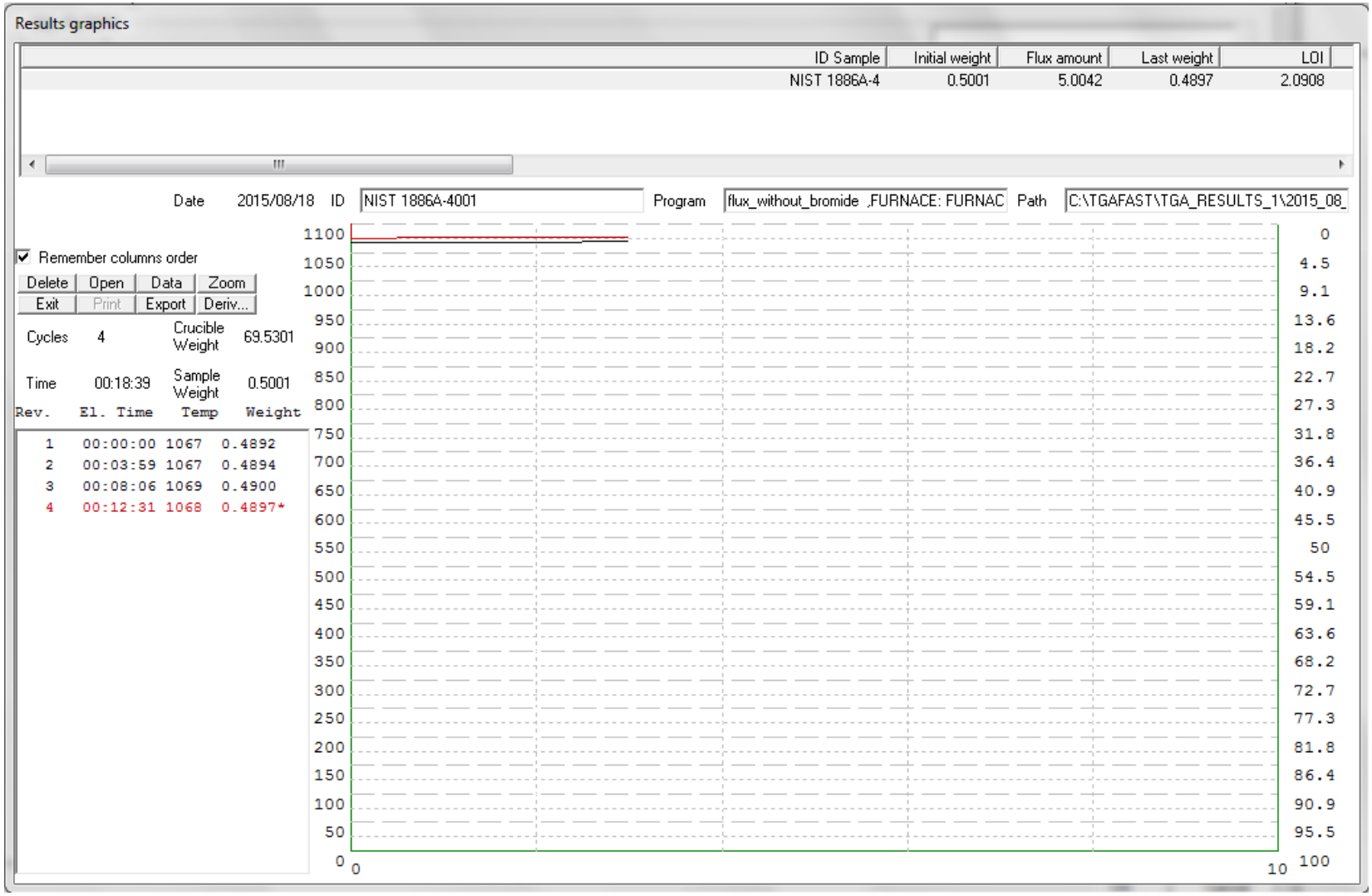
Different x-ray intensities for the same concentrations of Ca in different Ca bearing compounds in cement

NAVAS INSTRUMENTS

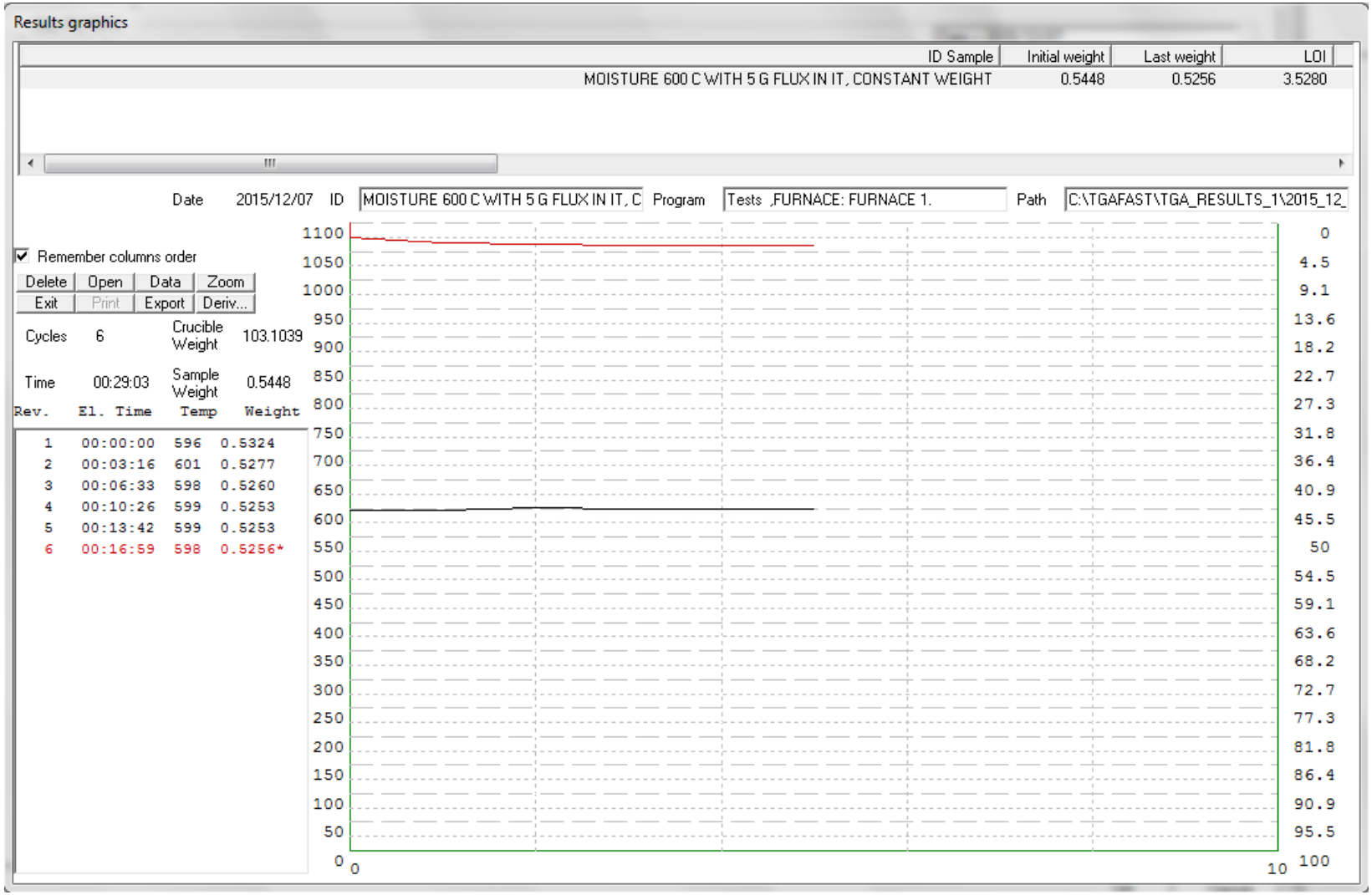
CEMENT WITH LITHIUM BROMIDE



NIST SAMPLE


Results graphics
ID Sample | Initial weight | Flux amount | Last weight | LOI
NIST 1886A-4 | 0.5001 | 5.0042 | 0.4897 | 2.0908
Date 2015/08/18 ID NIST 1886A-4001 Program flux_without_bromide ,FURNACE: FURNAC Path C:\TGAFAST\TGA_RESULTS_1\2015_08_
Remember columns order
Delete Open Data Zoom
Exit Print Export Deriv...
Cycles 4 Crucible Weight 69.5301
Time 00:18:39 Sample Weight 0.5001
Rev. El. Time Temp Weight
1 00:00:00 1067 0.4892
2 00:03:59 1067 0.4894
3 00:08:06 1069 0.4900
4 00:12:31 1068 0.4897*
1100 1050 1000 950 900 850 800 750 700 650 600 550 500 450 400 350 300 250 200 150 100 50 0
0 4.5 9.1 13.6 18.2 22.7 27.3 31.8 36.4 40.9 45.5 50 54.5 59.1 63.6 68.2 72.7 77.3 81.8 86.4 90.9 95.5 100
0
10

MOISTURE AT 600 °C

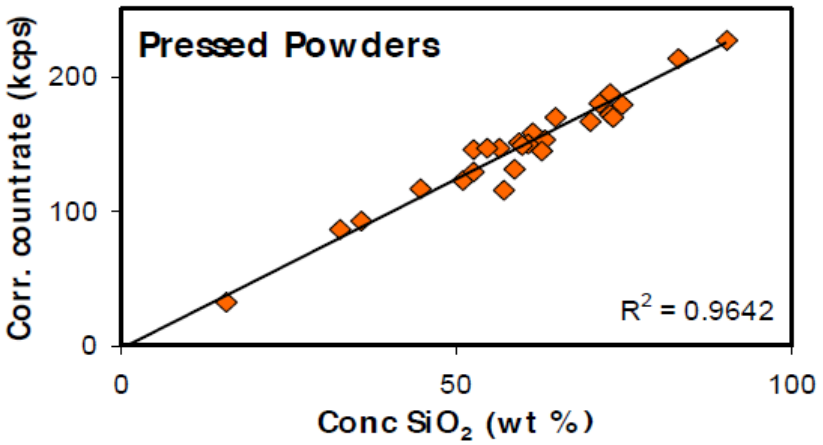


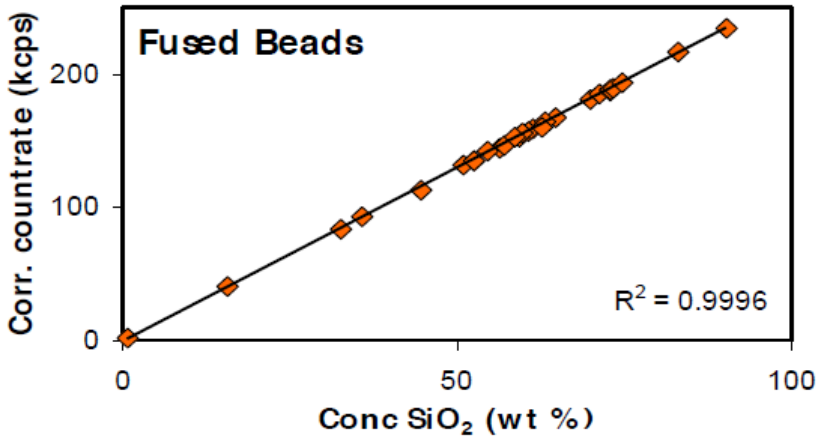
THE NEED FOR FUSION

Advantages:

- Very homogeneous sample
- No mineral phases or particles
- Inter-element matrix effects reduced by dilution
- Any remaining inter- element matrix effects easily corrected
- Easy to handle and store

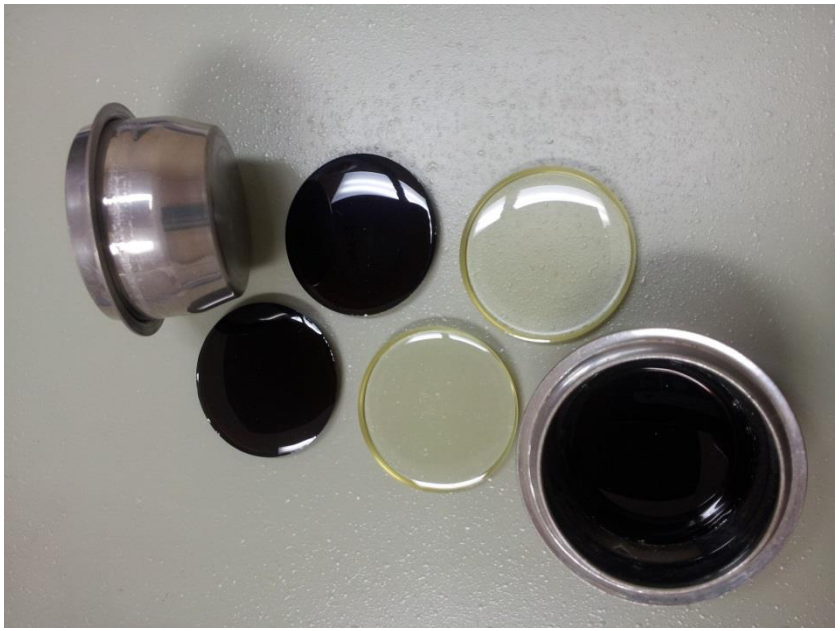
Essentially the ideal XRF sample





CHARACTERISTICS OF A GOOD FUSED BEAD

- All solid material completely dissolved into glass
- No loss of sample, flux or additives during fusion – **EXACT** ratio is **KNOWN**
- No residue left behind in crucible
- Comes out of the casting mold or moldable crucible cleanly



FUSION MAKERS-DESIGN CONSIDERATION

Precise Temperature Control

Advantages of circular VERSUS linear designs for multiple sample prep stations in how you measure the temperature

Sufficient agitation for good mixing

Rotation as well as horizontal and vertical movements – ideal figure of eight movement

Atmosphere control – sufficient air for complete oxidation

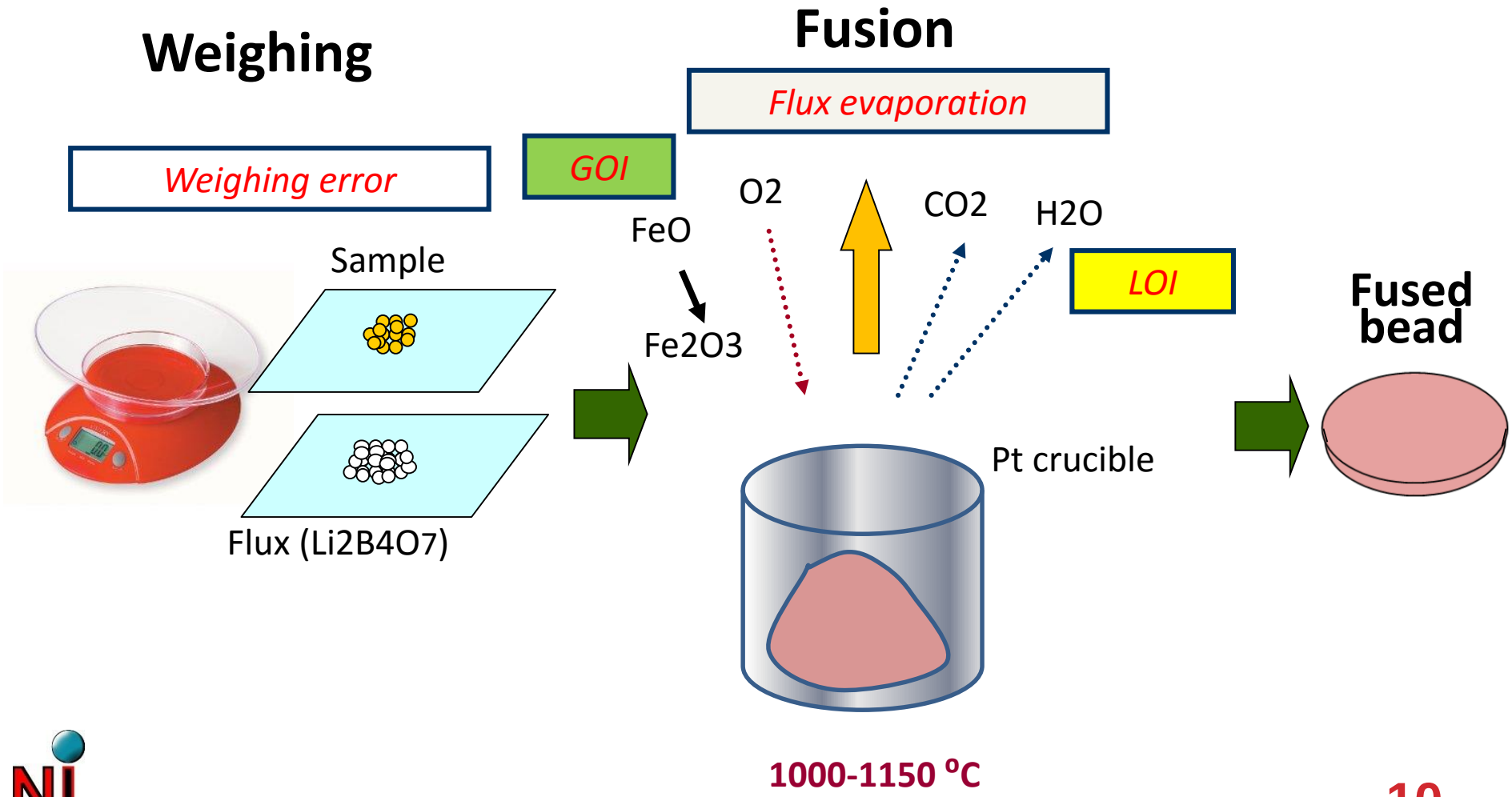
Inject air to keep uniform temperature and air circulation via convection

UNIQUE ASPECTS OF THE NAVAS FUSION SYSTEM

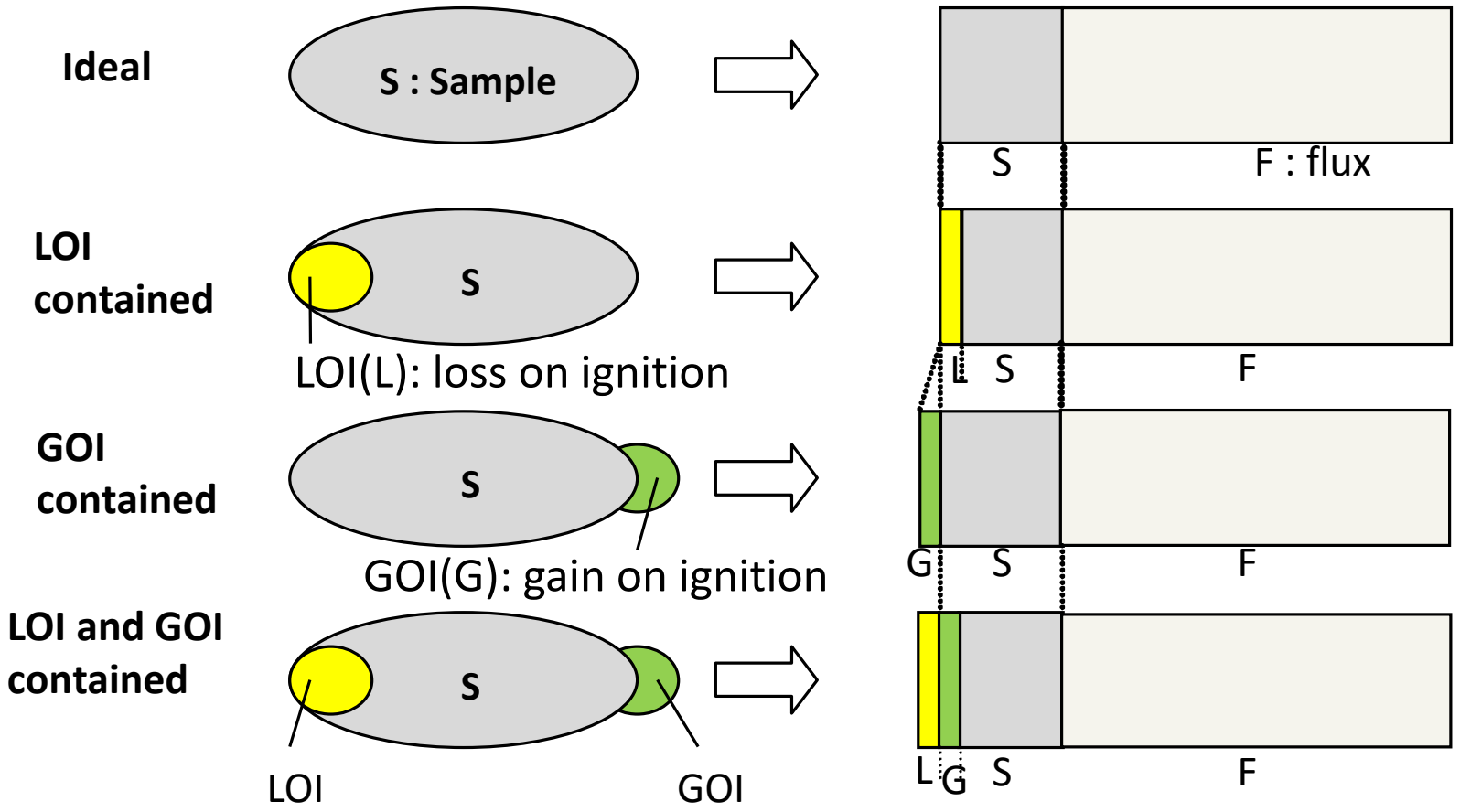
- Electric furnace, NO danger of explosion, No gas
- Analyzes loss on ignition and provides beads in the same instrument, simultaneously
- Simple, Reliable, MODULAR, Robust construction
- Bench type instrument with high throughput at a reasonable price
- No pouring, only moldables used
- Automation, requires little operator involvement
- Minimal moldable cleaning needed, only after 20 - 30 uses, not required after each use
- Easy to use software in Windows
- Up to 8 samples simultaneously

Navas Instruments AFS-5000-6, Automatic Fusion & LOI System For XRF Spectrometry

FACTORS IN FUSION

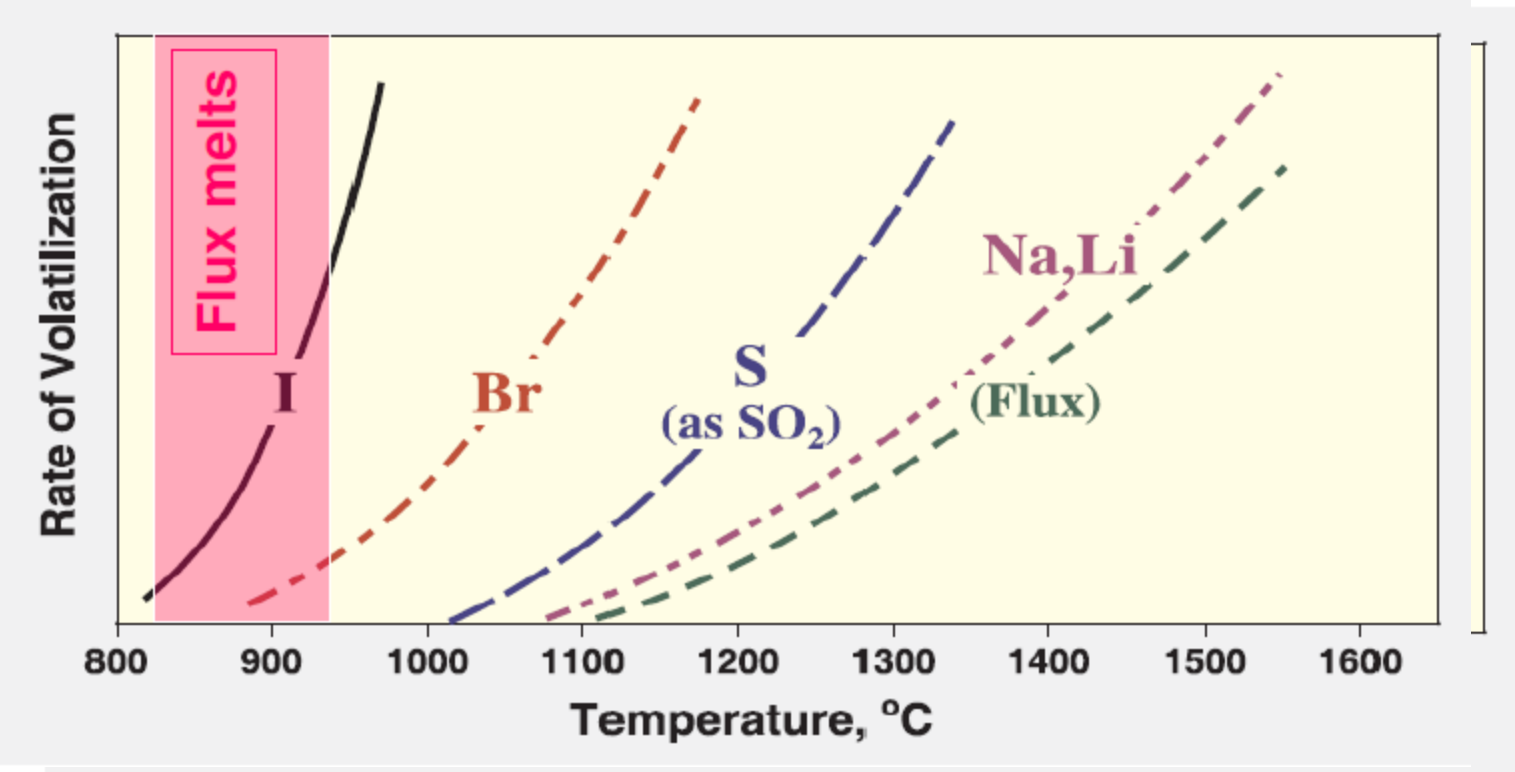


CONTROLLING FLUX, SAMPLE, OXIDANT AND NON-WETTING AGENT RATIOS



Dilution ratio: weight ratio of flux to sample

TEMPERATURE VS LOSS OF FLUX, ADDITIVES AND COMPONENTS



HOW TO CONTROL LOSSES

- **Use Pre-fused fluxes**
- **Monitor weight in-situ in furnace**
- **Add releasing agent just before fusion is completed**

  Reduces loss of material associated with LiBr evaporation

- **Measure Fused Bead Weight**
- **Use Fused Bead Weight to Sample Weight to account for LOI, GOI and LOF(Loss of Fusion)**

DILUTION RATIO CORRECTION

Calibration equation with dilution ratio correction

$$W_i = (b I_i + c)\left(1 + \sum_{j \neq L} \alpha_j W_j + \alpha_F R_F + K_F\right)$$

$$K_F = -\alpha_F \overline{R}_F$$

General calibration equation

$$W_i = (b I_i + c)\left(1 + \sum \alpha_j W_j + \alpha_F \Delta R_F\right)$$

$$\Delta R_F = R_F - \overline{R}_F$$

ΔR_F : Difference between the actual and standard ratio

R_F : Actual dilution ratio

$\alpha_F R_F + K_F$ is the correction term for the difference between the actual and standard dilution ratio.

HOW USING THIS APPROACH IMPROVES YOUR ACCURACY

10 replicate beads read on XRF

	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	K2O	CaO	TiO2	MnO	Fe2O3
	mass%	mass%	mass%	mass%	mass%	mass%	mass%	mass%	mass%	mass%	mass%
Using actual sample weight and bead weight											
Std dev.	0.024	0.023	0.016	0.051	0.009	0.008	0.007	0.086	0.007	0.002	0.012
RSD(%)	**13.9**	**1.48**	**0.28**	**0.25**	**4.44**	**0.25**	**1.25**	**0.13**	**2.41**	**2.63**	**0.28**
Using sample weight and flux weight											
Std,Dev	0.036	0.023	0.018	0.079	0.010	0.011	0.008	0.086	0.008	0.004	0.013
RSD(%)	**15.92**	**1.58**	**0.33**	**0.42**	**5.23**	**0.37**	**1.46**	**0.15**	**2.75**	**5.01**	**0.31**

SUMMARY

The Navas Automated Fusion System has distinct advantages over current automated fusion machines available in the market place

- The ability to record and update weights of the sample, flux and fused bead in situ allows for accurate tracking of the fusion process. Not available in other instruments

- Possible to do accurate LOI measurements at the same time as fusing the sample into a bead

- The use of fused bead weights in the final XRF calculation greatly improves the accuracy and precision of data used to control critical processes in cement manufacture

CONCLUSIONS

This novel approach that allows LOI and Fusion in the same process

- Saves time and thereby improves productivity which translates into financial savings
- The ability to improve both the accuracy and long term precision of the measurements, improves the product reliability which also translates into cost savings and intangible assets with respect to product reliability

THANK YOU FOR YOUR ATTENTION