SAMPLE PREPARATION WITH ULTRAWAVE IMPROVES STANDARD DEVIATION WHEN DETERMINING LOW CONCENTRATION OF CA IN STEEL WITH ICP-OES

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OVERVIEW

- Presentation of Analytical Chemistry Laboratory
  - Sandvik SMT in Sandviken
- Introduction
  - Demand, Objective and Challenge
- Experimental
- Results and Discussion
- Summary

SANDVIK MATERIAL TECHNOLOGY SANDVIKEN

Developing advanced stainless steels and special alloys which can make industrial processes more efficient, profitable and safer.
Application areas such as Oil and Gas, Aerospace and Nuclear power.

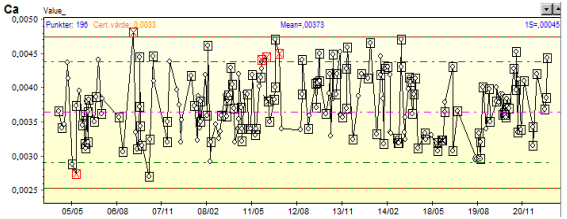
INTRODUCTION
DEMAND, OBJECTIVE AND CHALLENGE

OBJECTIVE

Determination of Ca in steel

- Sample Preparation
  Dissolved in a solution
- Measurement by ICP-OES
- Specification for the material
  Ca 0.0020-0.0060 %
  Srw 0.0002 %

Demand



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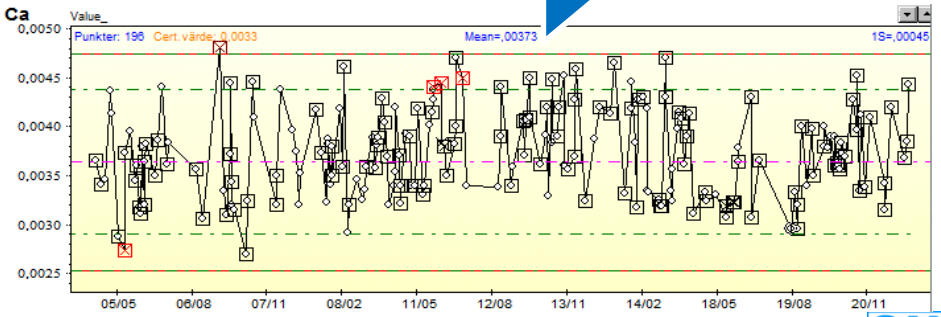
PRESENT ROUTINE METHOD

Sample preparation,

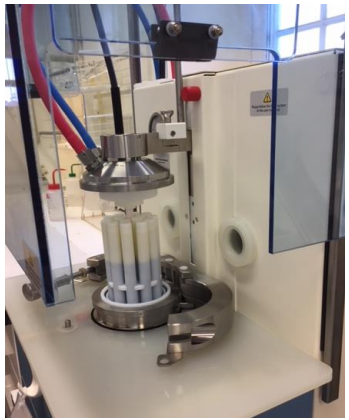
in acids on hot plate

Control Chart shows srw 0.00045 %



How to improve?

CHALLENGE



UltraWAVE

Sample preparation,

Microwave digestion technique.

Better precision for the control sample??

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EXPERIMENTAL

EXPERIMENTAL

Ref material
JK27A
SDN69

1. Routine, hot plate

0.5 g sample dissolved in 6 mL HCl 3 mL HNO_3. Diluted to 100 mL with UPW in a plastic flask.

15 mL tube for injection to instrument

2. Modified, hot plate

3. UltraWAVE

0.25 g sample dissolved in 6 mL HCl 3 mL HNO_3. Diluted to 50 mL with UPW in 50 mL PP-tube.

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PREPARATION TIME

1. Routine, hot plate

~ 30 min

2. Modified, hot plate

~ 20 min

3. UltraWAVE

~ 60 min

The UltraWAVE preparation takes longer time but can be unattended, that´s positive!

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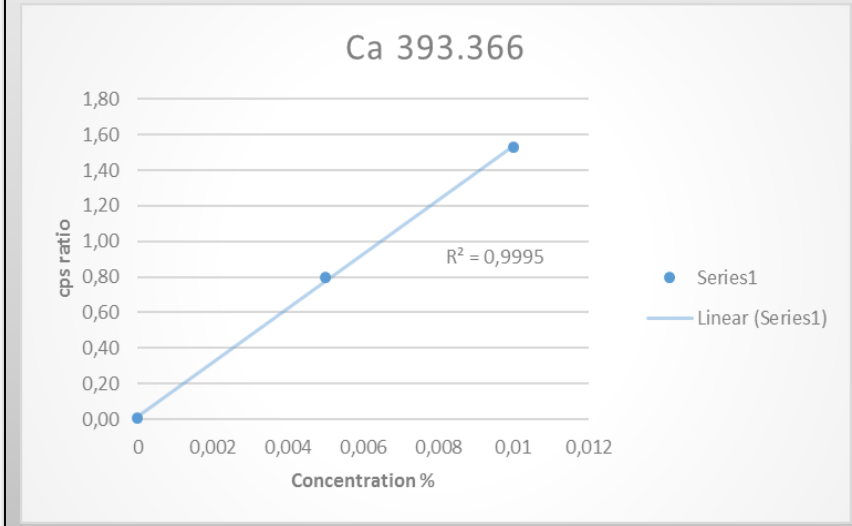
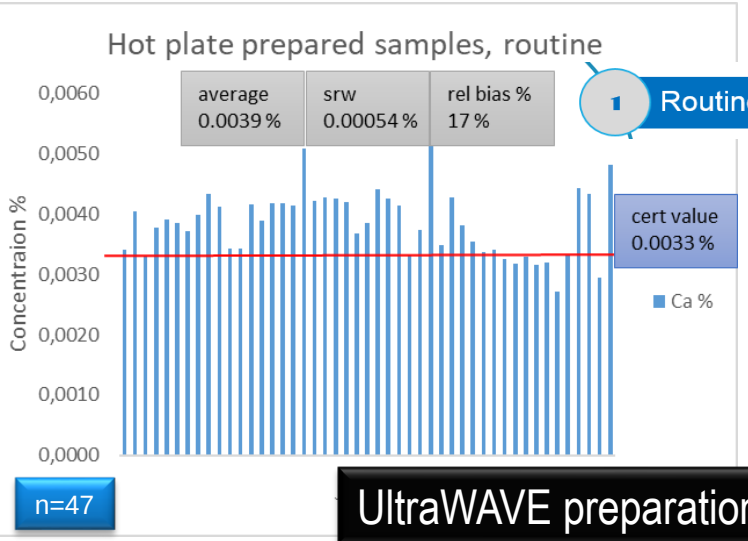
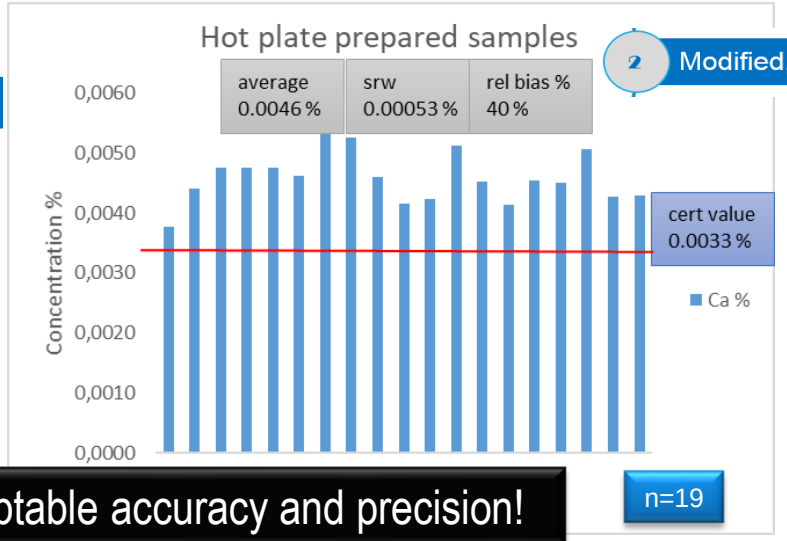
ICP-OES ANALYSIS

- Calibration
  - ➢ Fe matrix spiked with certified standard solution of Ca with high purity.
  - ➢ New calibration for every run.
  - ➢ 0-0.01 % Ca
- Spectral line
  - ➢ Ca 393.366
- Internal standard
  - ➢ Sc 361.384
- ICP-OES instrument
  - ➢ Spectro Arcos



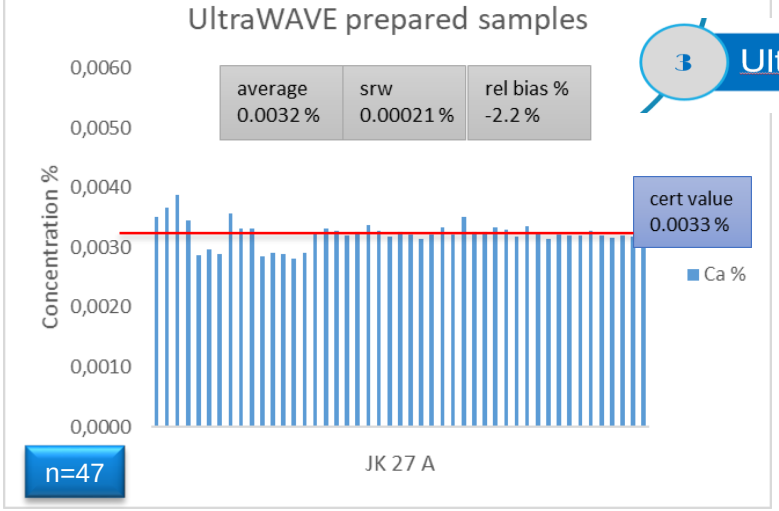
RESULTS AND DISCUSSION





UltraWAVE preparation – acceptable accuracy and precision!

RESULTS



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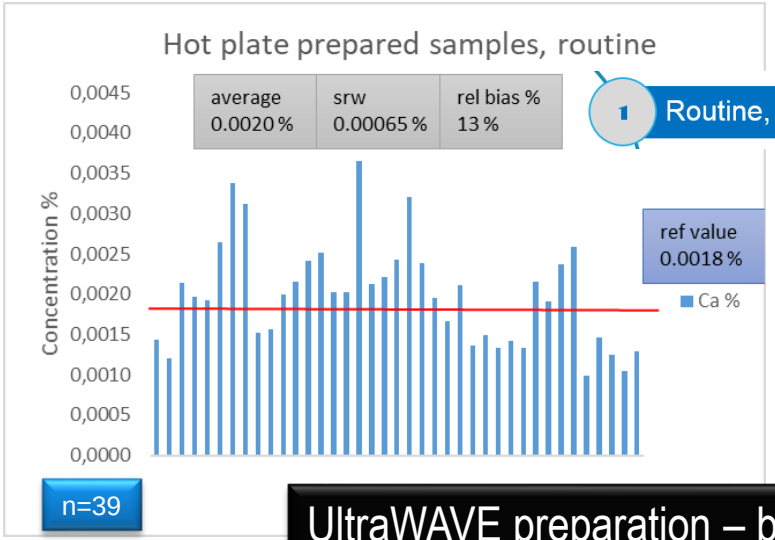
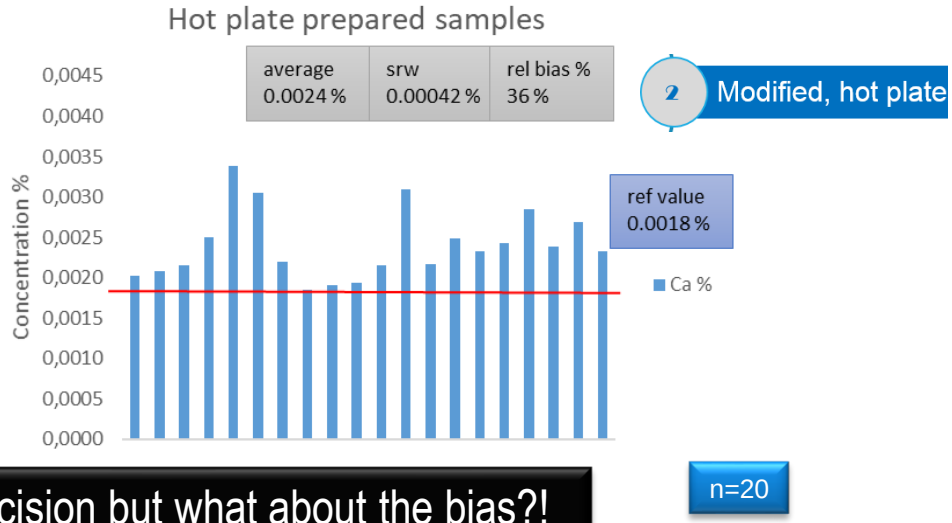
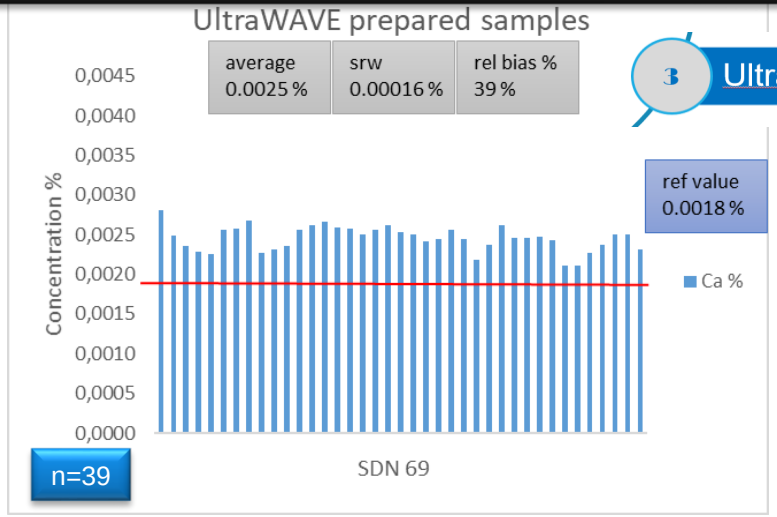




UltraWAVE preparation – better precision but what about the bias?!

RESULTS



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SUMMARY

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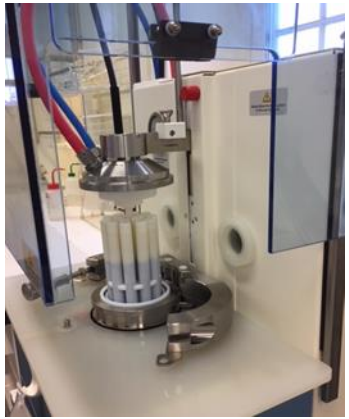
With UltraWAVE digestion;



- Significant improved precision for JK 27A and SDN69.
- Significant improved accuracy for JK 27A.
- Reduced risk of undissolved sample.
- Improved safety with less sample handling.
- Longer sample preparation time but can be left unattended.

Sample preparation with UltraWAVE improves standard deviation when determining low concentration of Ca in steel by ICP-OES

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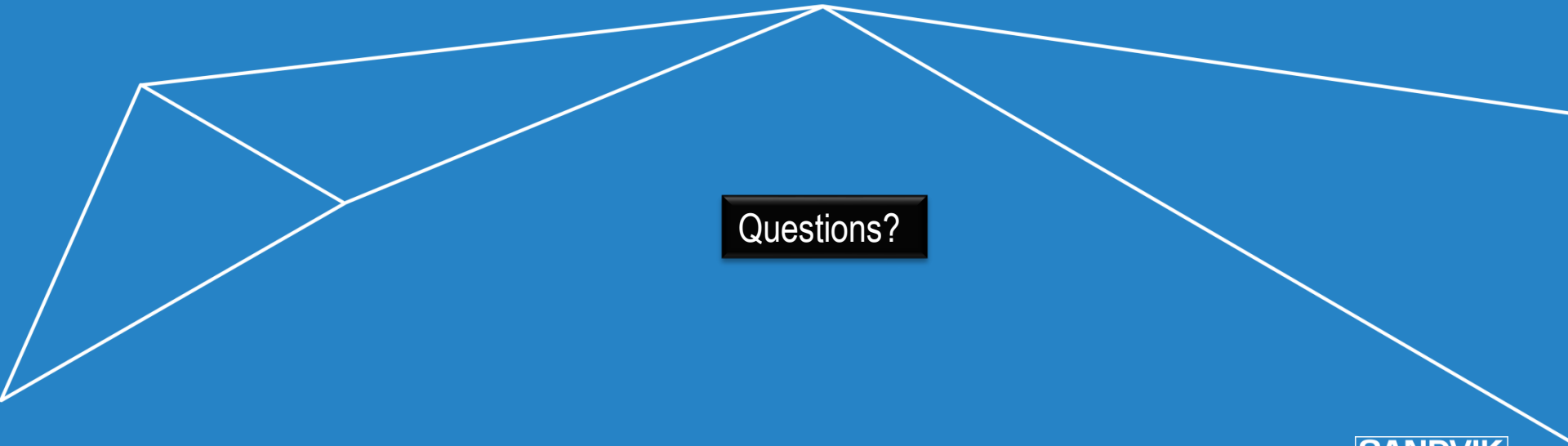
Next–Interesting… ”ColdBlock” IR-digestion?!

Anyone who has any experience?



Questions?

The End
Thanx for your attention ☺