Recommended Workflow for Quantitative Analysis

Introduction

Within AZtec "standardless" method is the default method for quantitative analysis, and if used in conjunction with the Oxford Instruments Recommended Workflow, it will give fast, accurate results for all but the most challenging and complex samples.

This recommended workflow describes several considerations that are important for ensuring that quantitative analysis gives the correct results.

Contact us:

1. Sample preparation and stage set up

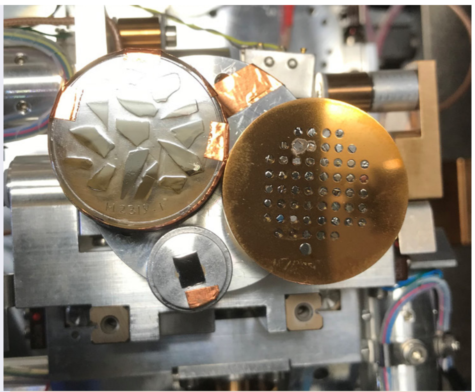
i) Samples should be:

- **Flat**
- **Polished**
- **Conductive** (e.g. C coating)

ii) Prepare and load a pure metal
(e.g. Cu, Co, Mn, Si) for **un-normalised** quantitative EDS analysis

- **Copper tape on a sample stub is a low cost and readily available pure metal standard**

iii) Insert samples and pure metal sample/standard into the SEM



Tip: Try to align any samples/standards so that they are the same height on the stage

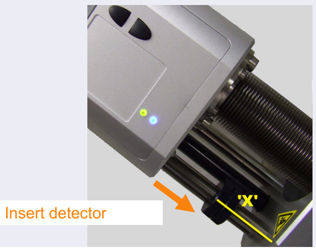
2. SEM and EDS detector setup

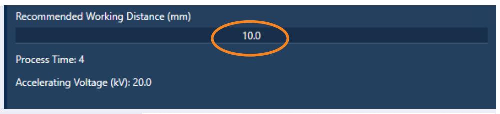
i) Set the sample at the **recommended working distance**

- Set the accelerating voltage (20 kV recommended for standardless microanalysis)
- Remove lens hysteresis
- Check and set the **recommended WD** for your system
- Use the stage Z position to bring the sample into focus

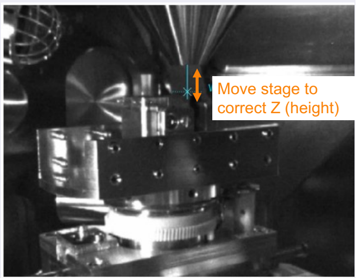
Sample height is important for determining/maintaining the correct **take off angle**

ii) EDS detector should be fully inserted





Recommended Working Distance (mm)
10.0
Process Time: 4
Accelerating Voltage (kV): 20.0

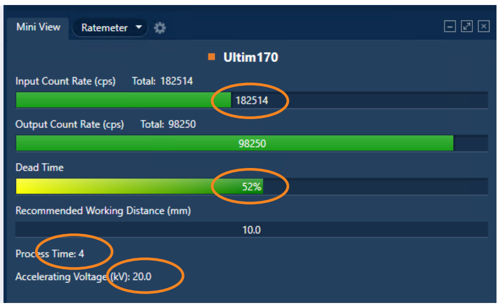


Tip: Each point should be carefully brought into focus at the recommended WD by adjusting the stage Z position

3. SEM analytical conditions

Beam current

- Adjust to give input count rate of between 50 and 200 kcps
- Set process time (PT) to between 3-6 and ensure dead time is ~50% or below



Ultim170
Input Count Rate (cps) Total: 182514
182514
Output Count Rate (cps) Total: 98250
98250
Dead Time
52%
Recommended Working Distance (mm)
10.0
Process Time: 4
Accelerating Voltage (kV): 20.0

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OXFORD INSTRUMENTS

4. Beam measurement - for un-normalised quant (EDS)

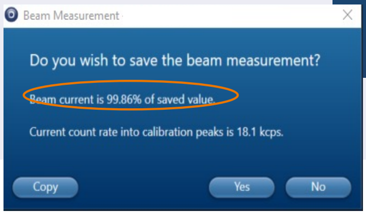
i) Perform a beam measurement on the pure metal

- Check focus (adjust stage Z)
- Set magnification to >10,000x

Accelerating voltage and beam current should not be changed between the beam measurement and the unknown sample measurement

ii) Beam currently stability

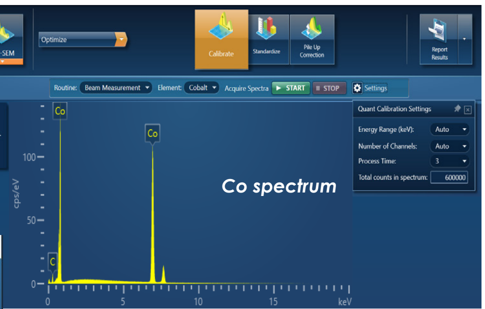
- Repeat beam current measurement to check beam is stable


Beam Measurement

Do you wish to save the beam measurement?

Beam current is 99.86% of saved value.

Current count rate into calibration peaks is 18.1 kcps.


Routine: Beam Measurement
Element: Cobalt

Quant Calibration Settings
Energy Range (keV): Auto
Number of Channels: Auto
Process Time: 3
Total counts in spectrum: 600000

Tip: It is **good practice** to perform beam measurements throughout the analytical session to correct for any change in the beam current over time

5. Measuring the sample

i) For coated samples, enter the appropriate **coating information**

ii) The electron beam should be either in:

- Spot mode
- Scanning over a very small region
- High magnification (e.g. >10,000x)

iii) Check EDS Acquire Spectrum Settings:

- Set count limit to 1,000,000 counts
- Ensure Pulse Pile Up Correction is on


EDS Acquire Spectrum Settings
Energy Range (keV): Auto 20
Number of Channels: Auto 2048
Process Time: 4
Acquisition Mode: Counts
Count Limit: 1000000
☑ Pulse Pile Up Correction

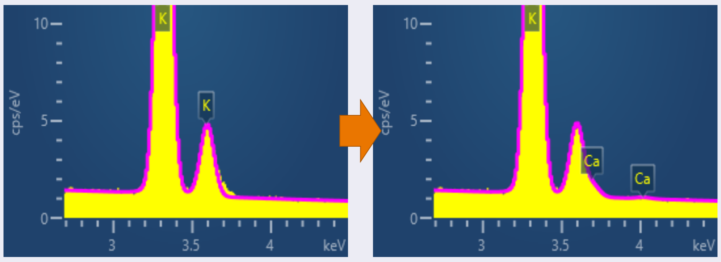
Specimen Coating Information:
☑ Specimen is coated
☑ Beam calibration element is coated
Coating element: Carbon
Thickness (nm): 10.00
Density (g/cm³): 2.25

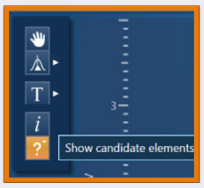
6. During/post acquisition

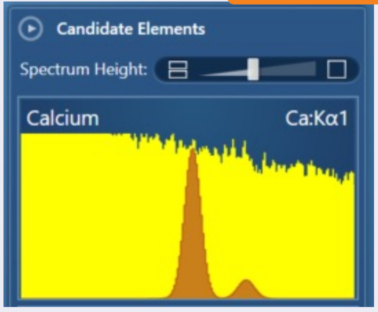
Verify the elements that have been identified correctly by AutoID. This can be done using:

- Fitted spectrum (pink),
- Analytical total, normally 100% ± 2%

EDS will never yield accurate results unless all the major and minor elements are correctly identified and in the element list



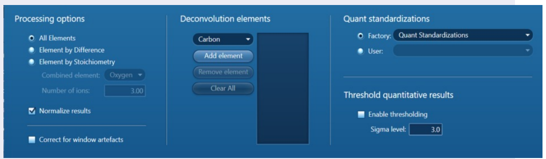




7. Use ‘Calculate Composition’ to view and interrogate the result

Check the quant settings:

- Processing options
  - Normalised vs. Un-normalised
  - Select the appropriate quant standardisations database – Factory or User


Processing options
○ All Elements
● Element by Difference
● Element by Stoichiometry
Combined element: Oxygen
Number of ions: 3.00
☑ Normalize results
☐ Correct for window artefacts

Deconvolution elements
Carbon

Quant standardizations
○ Factory: Quant Standardizations
● User:

Threshold quantitative results
☐ Enable thresholding
Sigma level: 3.0

Tip: ‘Quant standardisations’ for 10-30 kV
‘Quant standardisations (5 kV set)’ for 5-10 kV