EDX vs WDX: Head to Head

Welcome

Today’s Topics:

• How energy-dispersive (EDX) and wavelength-dispersive (WDX) X-ray spectrometry work
• Capabilities and advantages of EDX
• Capabilities and advantages of WDX
• Audience poll – which technique wins for your applications?
• New S8 DRAGON
• Q & A
X-ray Fluorescence Analysis
X-ray Spectrometry

...is the method to do qualitative and quantitative analysis of elemental composition by excitation of atoms and detection of their characteristic X-rays
X-ray Fluorescence Analysis
X-ray Spectrometry

...is the method to do qualitative and quantitative analysis of elemental composition

by excitation of atoms and detection of their characteristic X-rays

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X-ray Fluorescence Analysis
X-ray Spectrometry

...is the method to do

qualitative

and

quantitative

analysis

of elemental composition
by excitation of atoms and
detection of their
characteristic X-rays

• Qualitative analysis
  • identification of elements
  • "What's there?"

• Quantitative analysis
  • determination of concentrations
  • “How much?”

• Semi-quantitative analysis
  • estimation of concentrations
  • “About how much?”
Sample Excitation by X-Ray Tube

Each element present in the sample emits secondary fluorescence of a unique energy.

For WDX
- Soller slit
- Analyzer crystal
- Detector

For EDX
- Detector only
Wavelength-Dispersive X-ray (WDX) Spectrometry

- An analyzer crystal separates the various wavelengths, $\lambda$ (energies, $E$)
- The detector records only the number ($N$) of X-ray photons at a given wavelength (energy)
Wavelength-Dispersive X-ray
A Closer Look
Wavelength-Dispersive XRF (WDX) Analyzer Crystal and Bragg´s Equation

\[ n\lambda = 2d \sin \theta \]
Wavelength-Dispersive XRF (WDX)
Sequential Spectrometers
Collimators

- Sample emits X-rays in all directions
- Sequential spectrometers require (nearly) parallel beams
- Collimator suppresses X-rays which are not close to parallel
Benchtop Energy-Dispersive XRF (EDX)

- The detector is used to record both:
  - Energy of X-ray photons
  - Number of X-ray photons at a given energy
Benchtop Energy-Dispersive XRF (EDX) A Closer Look
What elements can be measured?
What elements can be measured? WDX

WDX: Boron – Uranium

Periodic Table

- H
- Li
- Be
- Na
- Mg
- K
- Ca
- Sc
- Ti
- V
- Cr
- Mn
- Fe
- Co
- Ni
- Cu
- Zn
- Ga
- Ge
- As
- Se
- Br
- Rb
- Sr
- Y
- Zr
- Nb
- Mo
- Tc
- Ru
- Rh
- Pd
- Ag
- Cd
- In
- Sn
- Sb
- Te
- I
- Xe
- Cs
- Ba
- La
- Hf
- Ta
- W
- Re
- Os
- Ir
- Pt
- Au
- Hg
- Tl
- Pb
- Bi
- Po
- At
- Rn

- Ce
- Pr
- Nd
- Pm
- Sm
- Eu
- Gd
- Tb
- Dy
- Ho
- Er
- Tm
- Yb
- Lu
- Th
- Pa
- U
- Np
- Pu
- Am
- Cm
- Bk
- Cf
- Es
- Fm
- Md
- No
What elements can be measured?
EDX

EDX: Sodium – Uranium
with X-Flash LE: Fluorine – Uranium

<table>
<thead>
<tr>
<th>H</th>
<th>Li</th>
<th>Be</th>
<th>B</th>
<th>C</th>
<th>N</th>
<th>O</th>
<th>F</th>
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<td>Yb</td>
<td>Lu</td>
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<td>U</td>
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<td>Cf</td>
<td>Es</td>
<td>Fm</td>
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</table>
What are the requirements for sample size?
Sample Size

WDX

- Sample must fit into the sample cup

- Standard Template
  - 60 available positions
  - Mask sizes: 5, 8, 23, 28, and 34 mm
  - Sample cannot be larger than 51.5 mm in diameter

- Easy Loader Template
  - 75 available positions
  - Mask sizes: 5, 8, 23, 28, and 34 mm
  - Sample cannot be larger than 40 mm in diameter

- Bare Sample Template
  - 108 available positions
  - 40 mm samples
Sample Size
EDX

- S2 RANGER manual unit: Single position
- S2 RANGER automation unit: 28 available positions
- Ring Size
  - Aperture: 30 mm
  - Sample cannot be larger than 40 mm in diameter
- Can run larger, irregular-shape samples
  - Handheld XRF
  - ARTAX µXRF
What is the resolution of the technique?
EDX resolution depends on detector capabilities

- Typical EDX resolution: <150 eV for Si(Li) detector
- X-Flash® Silicon Drift Detector (SDD)
  - Resolution of <129 eV FWHD for Mn Kα @ 100,000 cps
  - Peltier-cooled (no need for liquid nitrogen)
  - Resolution not affected by increases in count rate
Resolution

WDX

- WDX resolution is:
  - Dependent on the collimator and crystal selected
  - Not dependent on the detector like EDX is

- Typical resolution is <15 eV FWHD at Mn Kα

- 8-10 times better resolution than EDX with SDD
  - Less spectral overlaps
  - Lower background intensity
  - Better detection limits
What options are available for dealing with overlapping elements?
Overlaps

WDX

- Better resolution means less spectral line overlap and more accurate calibration

- If a spectral line cannot be resolved from the spectral line of interest, then:
  - Try an alternate crystal
  - Use a finer collimator

- If the above does not resolve the overlapping lines, then:
  - Choose an alternative line (Kβ, Lβ, etc.)
  - Apply mathematical overlap correction; need sufficient number of calibration standards
Crystal Comparison
Si: PET vs. XSCEM vs. InSb
Overlaps - WDX
Na Kα1 and Zn Lα1

XS-55 Crystal with 0.23 Collimator

TIAP Crystal with 1.0 Collimator

TIAP Crystal with 0.46 Collimator

TIAP Crystal with 0.23 Collimator
Overlaps - WDX Collimators

- "Coarse" collimator: 0.46°
  - High intensity
  - Overlaps may not be separated

- "Fine" collimator: 0.23°
  - Low intensity
  - Good resolution
  - Good separation of lines

- "Very Fine" collimator: 0.12°
  - Lower intensity
  - Better resolution
  - Better separation of lines
Overlaps
EDX

- EDX will show all spectral lines of the elements from the measurement region defined.

- If a spectral line cannot be resolved from the spectral line of interest, then:
  - Choose an alternative line (Kβ, Lβ, etc.)
  - Apply mathematical overlap correction; need sufficient number of calibration standards.
Overlaps - EDX
Slag Interference Fe Kα & Mn Kβ
Overlaps - WDX
Slag Interference Fe Kα & Mn Kβ
What are the count rate limitations?

What is the maximum count rate?
Count Rate Limitations
EDX

- Total Counts:
  - 100,000 – 120,000 cps
  - For entire spectrum that is being excited from measurement conditions selected
Count Rate Limitations

WDX

- Can achieve a lot higher count rates compared to EDX
  - Flow Proportional Detector: 1.5 million cps per analytical line
  - Scintillation Detector: 2 million cps per analytical line
What is the precision?
Cement Precision Study
WDX

WDX is the most precise analytical technique available

<table>
<thead>
<tr>
<th>Time</th>
<th>Na₂O [%]</th>
<th>MgO [%]</th>
<th>Al₂O₃ [%]</th>
<th>SiO₂ [%]</th>
<th>P₂O₅ [%]</th>
<th>SO₃ [%]</th>
<th>K₂O [%]</th>
<th>CaO [%]</th>
<th>Mn₃O₄ [%]</th>
<th>Fe₂O₃ [%]</th>
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<td>18:35:36</td>
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<td>1.021</td>
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<tr>
<td>Std.Dev.</td>
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<td>0.006</td>
<td>0.016</td>
<td>0.02</td>
<td>0.001</td>
<td>0.006</td>
<td>0.002</td>
<td>0.05</td>
<td>0.001</td>
<td>0.004</td>
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</table>
| RSD.     | 1.03     | 0.27    | 0.25      | 0.10     | 0.62     | 0.14    | 0.22    | 0.08    | 0.74     | 0.19       

One sample run 50 times: Total time per sample was 113 seconds
Roughly 10 seconds per element
Cement Short Term Precision Study
EDX

<table>
<thead>
<tr>
<th>Sample</th>
<th>MgO [wt. %]</th>
<th>Al₂O₃ [wt. %]</th>
<th>SiO₂ [wt. %]</th>
<th>SO₃ [wt. %]</th>
<th>K₂O [wt. %]</th>
<th>CaO [wt. %]</th>
<th>TiO₂ [wt. %]</th>
<th>Mn₃O₄ [wt. %]</th>
<th>Fe₂O₃ [wt. %]</th>
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<tr>
<td>Rep-1</td>
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<td>21.0017</td>
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<td>Rep-8</td>
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<td>2.70</td>
<td>0.12</td>
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</table>

One sample run 12 times: Total time per sample was 200 seconds
What are the typical detection limits?
Lower Limit of Detection (LLD)

- LLD is dependent on sample matrix

- Oil matrix
  - EDX: 3 – 15 ppm
  - WDX: 0.2 – 3 ppm

- Metal sample
  - EDX: 10 - 25 ppm
  - WDX: 1 – 8 ppm

\[
\text{LLD} = 3 / S \times \sqrt{I(BKG) / T}
\]
## Trace Elements in Geological Sample
### Lower Limit of Detection

<table>
<thead>
<tr>
<th>Element Compound</th>
<th>LLD (10C, 3σ)</th>
<th>Upper Calibration Range</th>
<th>Analysis Time [s]</th>
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<tr>
<td>Sc</td>
<td>0.9 ppm</td>
<td>100 ppm</td>
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<tr>
<td>TiO₂</td>
<td>0.001 %</td>
<td>2.6 %</td>
<td>10</td>
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<td>V</td>
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<td>Cr</td>
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<tr>
<td>MnO</td>
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<td>1 %</td>
<td>5</td>
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<tr>
<td>Fe₂O₃</td>
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<td>As</td>
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<tr>
<td>Th</td>
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<td>Th</td>
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<tr>
<td>U</td>
<td>T</td>
<td>&lt;1</td>
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What is the capability for measuring light elements?
New detector technology has now made it possible to see even lighter elements with EDX

- **X-Flash LE** can measure down to the element Fluorine

**X-Flash LE** provides:
- More than 8 times the sensitivity for Na (compared to standard SDD)
- More than 4 times the sensitivity for Mg (compared to standard SDD)
Light Elements

WDX

- Better light element analysis with WDX (B) compared to EDX (F / Na)

- New crystal design for analysis of super light elements (B, C, N, O)
  - LLD for C in low alloy steel is now around 70 ppm
  - Assuming surface preparation is properly done

XS-B crystal: Boron in glass
How long does it take to run a sample?
Analysis Time

WDX

- Time to analyze a sample depends on the following:
  - Element(s) to be measured
  - Concentration of the element(s)
  - Required detection limit
  - Required precision

- Sequential WDX
  - Measure for each element
    - Time to measure peak
    - Additional time may be necessary to measure background

- Multichannel WDX
  - Fixed-element channels
  - Reduces measurement time drastically
  - But lose ability to measure background
Analysis Time
EDX

- Time to analyze a sample depends on:
  - Element(s) to be measured
  - Concentration of the element(s)
  - Required detection limit
  - Required precision

- Measure by regions
  - Typical to measure with one or two measurement regions depending on the elements of interest
    - < Ca: 20kV with no filter
    - > Ca: 40kV with Al filter
    - Heavy elements: 50kV with Cu filter
  - Typical measuring time for slag, cement, and steel samples is 5 - 10 min per sample, depending on precision requirement
What about protection of the instrument from sample spillage or breakage?
Protection - WDX
Sample Care and Easy Loader

- **Sample Care (4x Protection)**
  - Two contamination shields to protect tube window and goniometer (3)
  - Dust reservoir to collect sample particles and droplets (6)
  - DuraBeryllium shield for tube window protection (2)
  - Unique vacuum seal with high-transmission window for goniometer protection (5)

- **Easy Loader**
  - Prevents liquid samples from running under vacuum mode
Protection - EDX Dust Reservoir

- Dust reservoir to collect sample particles and droplets
- Easy access to sample chamber
Are there any ASTM, ISO, or DIN norms that suggest which technique to use in the analysis of cement and petrochemicals?
ASTM, ISO, and DIN Norms

WDX

- Cement
  - ASTM C114 – Chemical Analysis of Hydraulic Cement (does not specify either EDX or WDX, but typically it is done by WDX)

- Petrochemicals
  - ASTM D2622 – S in Petroleum Products by WDX
  - ASTM D4927 – Elemental Analysis of Lubricant and Additive Components (Ba, Ca, P, S, Zn) by WDX
  - ASTM D5059 – Lead in Gasoline
  - ASTM D6334 – S in Gasoline by WDX
  - ASTM D6376 – Determination of Trace Metal in Petroleum Coke by WDX
  - ASTM D6443 – Determination of Ca, Cl, Cu, Mg, P, S, & Zn in Unused Lubricating Oils and Additives by WDX
• Petrochemicals (continued)
  • ISO 14596 – Determination of S Content
  • ISO 14597 – Determination of V & Ni Content
  • ISO 15597 – Determination of Cl & Br Content
  • ISO 20884 – Determination of S Content of Automotive Fuels

• DIN 13723 – Lead in Automotive Fuels
• DIN 51363 – P in Lubricating Oils & Additives
• DIN 51391 – Zn & Ca in Lubricating Oils
• DIN 51431 – Mg in Lubricating Oils
ASTM, ISO, and DIN Norms
EDX

- Petrochemicals
  - ASTM D4294 – S in Petroleum and Petroleum Products by EDX
  - ASTM D5839 – Trace Element Analysis of Hazardous Waste Fuel by EDX
  - ASTM D6445 – S in Gasoline by EDX
  - ASTM D6481 – Determination of P, S, Ca, & Zn in Lubrication Oils by EDX

- ISO 8754 – Determination of Sulfur Content
- ISO 20847 – Determination of Sulfur Content of Automotive Fuels
What peripherals are required or optional for each technique?
Peripherals
WDX

- Location of instrument and peripherals
  - Computer with instrument software
  - P-10 for Flow Proportional Detector
  - Haskris for 3 or 4 kW instrument
  - Correct country-specific power
  - UPS/TVSS (optional)
  - Helium for liquid and loose powder samples
Peripherals
EDX

- Location of instrument and peripherals
  - All-in-one design
    - Can be used as standalone unit (computer not required to run samples)
    - Correct country-specific power
    - Vacuum pump
    - Printer
  - UPS/TVSS (optional)
- Helium for liquid and loose powder samples
What is the cost difference between a benchtop EDX and a sequential WDX?
Cost Difference

• Typically, WDX is 2 -3 times more expensive than EDX

• EDX is more attractive because of price, but always remember to

"Choose the Right Tool for the Job"
Audience Poll

Use your mouse to answer the question on your screen:

Which technique is the winner for your applications?
- EDX
- WDX

We will share the results with you before of the Q&A session.
New truly simultaneous XRF S8 DRAGON

- Truly simultaneous XRF spectrometer combining single element channels with the new Multielement Channel™
- Covers almost the whole periodic table from C upwards in one run in less than 40 seconds
- 4 kW high excitation power
- Unmatched precision for all relevant elements in metal production and foundries
- Footprint more than 25% smaller than traditional instrument
S8 DRAGON
Spectrometer Setup
Essential benefits of the unique Multielement Channel™

- Elemental fingerprinting
  - Identification and analysis of all elements from Na upwards
- Analytical flexibility
  - Contaminations can be traced
  - Analysis of non-routine samples
  - Upgrading of analytical methods with additional elements in minutes - no further installation of new hardware
- Dual-mode data acquisition
  - Internal backup for data safety with a second internal source
Parallel measurement of SEC (WDX) and Multielement Channel
Identification of trace elements, internal backup (second information source)
Precision Test for Steel
40 s measurement time - Dual Mode

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<th>RSD [%]</th>
<th>LLD [PPM]</th>
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<td>Zr KA1</td>
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Iron Concentration
200 measurements: Fe 47.065 +/- 0.013
Iron Concentration
200 measurements: P 0.031 +/- 0.001

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<td>101</td>
</tr>
<tr>
<td>0.031</td>
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P Repeatability

April 18, 2012
Thank you for attending

- Results of audience poll

Which technique is the winner for your applications?
- EDX
- WDX

- Q & A

Please type any questions you may have for Luke (EDX) or Dan (WDX) in the Q&A panel on your right and then click Send.