Overview of the 4200 MP-AES

Shane Elliott
Marketing Manager : AA, MP-AES and ICP-OES
Agilent Technologies
Agenda

• What is MP-AES, and how does it work?
• Key benefits of MP-AES compared to flame AA
• Ease of Use benefits over flame AA
Agenda

• What is MP-AES, and how does it work?

• Key benefits of MP-AES compared to flame AA

• Ease of Use benefits over flame AA
Microwave Plasma Emission Overview

EXCITATION

Ground State → Excited State → Relaxation

Emission

Atom Formation

Sample

Microwave excited Plasma

Monochromator & Detector

Quantitation
How does the MP-AES work?

- 4200 MP-AES runs on nitrogen extracted from air using the Agilent 4107 nitrogen generator

- Axial magnetic and radial electrical fields sustain the nitrogen plasma

- Sample aerosol is introduced into nitrogen plasma.
How does the MP-AES work?

• Axial emission from the vertical oriented nitrogen plasma is directed into the fast scanning monochromator optics.

• Wavelength specific emissions are detected using a high efficiency CCD.
Agenda

• What is MP-AES, and how does it work?

• Key benefits of MP-AES compared to flame AA

• Ease of Use benefits over flame AA
Key benefits of MP-AES

**Lowest cost of ownership**
- Runs on air!
- Eliminates on-going gas supply costs

**Safer Laboratory Operation**
- No flammable gases
- Unattended multi-element operation

**Higher Performance**
- Ideal flame AA alternative
- Handles major, minor & most trace levels
- Wider linear dynamic range
- Lower detection limits

**Easy Operation**
- New generation software
- Simultaneous auto background correction
- Plug & Play torch

Agilent Technologies
Introducing the next generation Agilent 4200 MP-AES

Optimized, second generation plasma system and new torch design: Superior analytical performance and torch robustness suiting a broader range of complex matrix samples.

MP Expert v1.2: Intuitive software interface, now with advanced features in the ‘PRO’ pack.

Mass flow control of nebulizer gas and robust sample introduction: Enhanced accuracy and long term stability in tough samples.
Low cost of ownership

• Supply of gases is the major on going cost for any routine elemental spectrometer.

• 4200 MP-AES uses nitrogen extracted from air using Agilent 4107 nitrogen generator.

• Example, big USA fruit juice manufacturer doing 100 FAAS samples per day, 3 elements per sample.

Online cost estimator available at: www.agilent.com/chem/runsonair
Safety

- Using inert nitrogen gas to sustain the atomization/ionization source eliminates the need to use combustible gases like acetylene.

- Not having to ever replace a gas cylinder reduces manual handling issues.
### 4200 MP-AES Performance – Detection Limits

*DL’s in ppb, clean water samples*

<table>
<thead>
<tr>
<th>Element</th>
<th>Flame AA</th>
<th>4200MP-AES</th>
<th>Element</th>
<th>Flame AA</th>
<th>4200 MP-AES</th>
</tr>
</thead>
<tbody>
<tr>
<td>K</td>
<td>0.8</td>
<td>0.65</td>
<td>As*</td>
<td>60</td>
<td>57</td>
</tr>
<tr>
<td>Ca</td>
<td>0.4</td>
<td>0.04</td>
<td>Cd</td>
<td>1.5</td>
<td>1.4</td>
</tr>
<tr>
<td>Mg</td>
<td>0.3</td>
<td>0.09</td>
<td>Cr</td>
<td>5</td>
<td>0.3</td>
</tr>
<tr>
<td>Na</td>
<td>0.3</td>
<td>0.12</td>
<td>Mn</td>
<td>1.0</td>
<td>1.05</td>
</tr>
<tr>
<td>Au</td>
<td>5</td>
<td>2.1</td>
<td>Pb</td>
<td>14</td>
<td>2.5</td>
</tr>
<tr>
<td>Pt</td>
<td>76</td>
<td>6.1</td>
<td>Sb</td>
<td>37</td>
<td>12</td>
</tr>
<tr>
<td>Pd</td>
<td>15</td>
<td>1.6</td>
<td>Se*</td>
<td>500</td>
<td>77</td>
</tr>
<tr>
<td>Ag</td>
<td>1.7</td>
<td>1.2</td>
<td>Zn</td>
<td>1.6</td>
<td>3.1</td>
</tr>
<tr>
<td>Rh</td>
<td>4</td>
<td>0.5</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* 30 second integration time used for these elements

3 sigma DLs using a 10 second integration time with dewar nitrogen
Agenda

• What is MP-AES, and how does it work?
• Key benefits of MP-AES compared to flame AA
• Ease of Use benefits over flame AA
Ease of Use

Simple hardware

- Torch installation
- Sample Introduction System
Ease of Use

Simple software

• Applet quick start methods
• Auto optimization tools
• FLIC and IEC tools
Ease of use—reduced sample preparation

Optimum working range comparison between FAAS and MP-AES:

<table>
<thead>
<tr>
<th>Element</th>
<th>4200 MP-AES, mg/L</th>
<th>Linear correlation coef. MP-AES</th>
<th>FAAS, mg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Au 242.8</td>
<td>0.05 – 50</td>
<td>0.9999</td>
<td>0.1 – 30</td>
</tr>
<tr>
<td>Ca 422.6</td>
<td>0.01 – 20</td>
<td>0.9999</td>
<td>0.01-10</td>
</tr>
<tr>
<td>Mg 518.3</td>
<td>0.05 – 100</td>
<td>0.99988</td>
<td>0.15-20 (202.6nm)</td>
</tr>
<tr>
<td>Na 589.5</td>
<td>0.01 – 20</td>
<td>0.999996</td>
<td>0.01-2</td>
</tr>
<tr>
<td>K 767.8</td>
<td>0.05 – 100</td>
<td>0.99968</td>
<td>1-6</td>
</tr>
</tbody>
</table>

Example: Au 242.8nm calibration for 4200 MP-AES
Ease of Use - MP-AES verses FAAS

- Simple sample preparation
  - Reduced chemical and ionization interferences minimizes need for addition of extra reagents
  - Lower costs, higher sample throughput
  - Reduces introduction of possible contaminants

<table>
<thead>
<tr>
<th>Element</th>
<th>Possible chemical interferences</th>
<th>FAAS specific sample preparation</th>
<th>MP-AES specific sample preparation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca</td>
<td>Refractory compounds</td>
<td>Lanthanum releasing agent</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>Ionization effects</td>
<td>Cesium ionization buffer</td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>Refractory compounds</td>
<td>Lanthanum releasing agent</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>Ionization effects</td>
<td>Cesium ionization buffer</td>
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</tr>
<tr>
<td>Na</td>
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<td>Cesium ionization buffer</td>
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<td>K</td>
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<td>Cesium ionization buffer</td>
<td>None</td>
</tr>
</tbody>
</table>
4200 MP-AES - Summary

• Agilent’s 4200 MP-AES is the second generation of proven MP-AES technology.

• The 4200 offers safe, cost effective elemental analysis, with an application range that makes it an ideal flame AA alternative (upgrade!).

• 4200 MP-AES is very easy to use and has simple sample preparation.
CHANGE IS IN THE AIR
LEAD THE WAY WITH SAFE, COST EFFECTIVE ELEMENTAL ANALYSIS.

Introducing the next generation of **MP-AES** technology – it’s changing the way you work. While still offering safer, more cost effective elemental analysis, the new 4200 **MP-AES** is now suited to an even wider range of sample types and applications. The 4200 **MP-AES** will put you on the path to success.

The Agilent 4200 MP-AES for Geochemistry, Minerals, Mining and Metals Analysis.

Shane Elliott
Marketing Manager
AA, MP-AES, ICP-OES
Agenda

• The Agilent 4200 MP-AES

• Major elements in copper alloys

• Determination of major and minor elements in geological samples
Key benefits of MP-AES

- **Lowest cost of ownership**
  - Runs on air!
  - Eliminates on-going gas supply costs

- **Safer Laboratory Operation**
  - No flammable gases
  - Unattended multi-element operation

- **Higher Performance**
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Determination of major elements in copper alloys using the Agilent 4200 MP-AES
Major elements in copper alloys by 4200 MP-AES

Copper alloy sample preparation:

1 g SRM drillings (3 different alloys) was digested in open test tube (pyrex) digestion / 1.5 ml HNO3 and 4.5 ml HCl made to 50ml (2% TDS).

For determination of Cu and Zn, the samples were diluted further 1:50 (0.04% TDS, 2500 x dilution)

Calibration solutions were prepared from commercial bulk standards.
## Analysis of brass and bronze – major elements Cu and Zn

These analysis results had Neb flow optimized on the diluted bronze sample

<table>
<thead>
<tr>
<th></th>
<th>Element</th>
<th>Certified value (%)</th>
<th>4200 MP-AES result (Measured value, % recovery)</th>
<th>Wavelengths</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Aluminium Brass</strong></td>
<td>Cu</td>
<td>75.1</td>
<td>75.2 (100.1%)</td>
<td>219 nm</td>
</tr>
<tr>
<td><strong>SRM 1118</strong></td>
<td>Zn</td>
<td>21.9</td>
<td>22.3 (101.8%)</td>
<td>481 nm</td>
</tr>
<tr>
<td><strong>Bronze Standard</strong></td>
<td>Cu</td>
<td>93.0</td>
<td>92.4 (99.4%)</td>
<td>219 nm</td>
</tr>
<tr>
<td><strong>NBS C1117</strong></td>
<td>Zn</td>
<td>6.8</td>
<td>6.8 (100%)</td>
<td>481 nm</td>
</tr>
<tr>
<td><strong>Naval Brass</strong></td>
<td>Cu</td>
<td>65.0</td>
<td>65.9 (101.4%)</td>
<td>219 nm</td>
</tr>
<tr>
<td><strong>NIST C1108</strong></td>
<td>Zn</td>
<td>34.4</td>
<td>34.3 (99.7%)</td>
<td>481 nm</td>
</tr>
</tbody>
</table>
Discussion

• Major element determination of the copper alloy CRMs showed high degree of accuracy using the 4200 MP-AES.

• The copper alloy analysis employed a simple sample preparation and a simple analysis regimen.

• Simplification of the analysis reduces labor costs and enhances the sample throughput.
Conclusion
4200 MP-AES is the ideal FAAS alternative

- **Improved Performance**
  - Increased working range
  - Lower detection limits

- **Reduced Running Costs**
  - Runs on Air!
  - Simple sample prep

- **Increased safety**
  - No acetylene

- **Ease of Use**
  - MP Expert
  - Simple sample prep

19 March 2014
Determination of major and minor elements in geological samples using the 4200 Microwave Plasma-Atomic Emission Spectrometer (MP-AES)
Analytical challenges of analyzing geochemical samples

• Wide working range to cover major, minor and trace concentrations
• High levels of total dissolved solids, complex acid sample prep
• Spectral interferences due to emission line overlaps
• Varying sample composition
• Non-spectral interferences due to EIE
Common challenges facing atomic spectroscopy users

1. Increased need for multi-element determination over a wide dynamic range
2. Desire to reduce the overall cost of analysis due to rising costs (instrument supplies and consumables, power, labor etc.)
3. Difficulty in sourcing gases — especially in remote areas and emerging geographies
4. Availability of suitably trained personnel to develop methods, perform sample measurement and interpret results
5. Laboratories under pressure to improve safety by removing flammable gases
Sample preparation

Two CRMs prepared
• GBM398-4 Low grade Cu/Pb/Zn with Laterite
• GBM908-14 Cu-Zn-Pb Sulphide Ore

Acid digestion
• HNO$_3$-HCl-HClO$_4$-HF four-acid ‘total’ digestion

Sample dilution
• 0.4 g nominal sample weight brought to 100 mL final volume with 30% HCl

Calibration standards
• All calibration standards in 6% HNO$_3$ and 19% HCl
  - No modifiers or ionization buffers required
  - No further sample dilutions required
Analyte line selection, and background and interference correction methods

Table 2. The emission line selection, background and interference correction methods

<table>
<thead>
<tr>
<th>Element</th>
<th>Wavelength (nm)</th>
<th>Type</th>
<th>Background Correction</th>
<th>Interference Correction</th>
<th>Possible Interferences</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>305.082</td>
<td>Analyte</td>
<td></td>
<td>FLIC</td>
<td>La</td>
</tr>
<tr>
<td>Ag</td>
<td>328.068</td>
<td>Analyte</td>
<td>Auto</td>
<td>IEC</td>
<td>Cu, Ti</td>
</tr>
<tr>
<td>Ti</td>
<td>334.940</td>
<td>IEC element</td>
<td>Auto</td>
<td></td>
<td>x</td>
</tr>
<tr>
<td>Pb</td>
<td>405.781</td>
<td>Analyte</td>
<td></td>
<td>FLIC</td>
<td>La and Ti</td>
</tr>
<tr>
<td>Zn</td>
<td>481.053</td>
<td>Analyte</td>
<td></td>
<td>FLIC</td>
<td>La, Sr, and Ti</td>
</tr>
<tr>
<td>Cu</td>
<td>510.554</td>
<td>Analyte</td>
<td></td>
<td>FLIC</td>
<td>Al₂O₃, and La</td>
</tr>
<tr>
<td>Lu</td>
<td>547.669</td>
<td>Internal standard</td>
<td></td>
<td>FLIC</td>
<td>Ni, Ti</td>
</tr>
</tbody>
</table>

- Wavelengths were selected to minimize spectral interferences and have widest dynamic range
- All calibration curves are linear with correlation coefficient >0.999 and <10% calibration fit error
- Lu internal standard was 10 ppm and delivered via Y-connector to the sample tubing
Spectral interference correction – Fast Linear Interference Correction (FLIC)

Nickel and Ti interference on Lu 547.669nm line in FLIC

(background: blue, Lu; red, Ni: green, and Ti: dark blue)

Table 3. FLIC sequence

<table>
<thead>
<tr>
<th>Anayte (nm)</th>
<th>Blank</th>
<th>Analyte mg/L</th>
<th>Interferent-1 Al mg/L</th>
<th>Interferent-2 Ti mg/L</th>
<th>Interferent-3 La mg/L</th>
<th>Interferent-4 Sr mg/L</th>
<th>Interferent-5 Ni mg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni (305.083)</td>
<td>Cal Blank</td>
<td>10</td>
<td>x</td>
<td>x</td>
<td>100</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>Zn (481.053)</td>
<td>Cal Blank</td>
<td>10</td>
<td>x</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>x</td>
</tr>
<tr>
<td>Cu (510.554)</td>
<td>Cal Blank</td>
<td>10</td>
<td>1000</td>
<td>x</td>
<td>100</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>Lu (547.669)</td>
<td>Cal Blank</td>
<td>10</td>
<td>x</td>
<td>100</td>
<td>x</td>
<td>x</td>
<td>100</td>
</tr>
</tbody>
</table>

Extra interference models can be added, even after results have been collected.
Spectral interference correction – Conventional IEC

IEC factors are developed using intuitive MP Expert software.

IEC solutions used:

- 1 ppm Ag (analyte)
- 100 ppm Ti (interferent)
- 1000 ppm Cu (interferent)

These concentrations represent the expected concentrations in the sample.

MP Expert IEC factors and Ag calibration
Results and discussion

Table 4. MP-AES and certified CRM results

<table>
<thead>
<tr>
<th>Reference</th>
<th>MDL</th>
<th>GBM398-4</th>
<th>GBM908-14</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>MP-AES</td>
<td>Certified</td>
</tr>
<tr>
<td>Ag (mg/kg)</td>
<td>1</td>
<td>45.8</td>
<td>48.7</td>
</tr>
<tr>
<td>Cu (wt %)</td>
<td>0.002</td>
<td>0.37</td>
<td>0.39</td>
</tr>
<tr>
<td>Ni (wt %)</td>
<td>0.002</td>
<td>0.39</td>
<td>0.41</td>
</tr>
<tr>
<td>Pb (wt %)</td>
<td>0.002</td>
<td>1.08</td>
<td>1.17</td>
</tr>
<tr>
<td>Zn (wt %)</td>
<td>0.002</td>
<td>0.50</td>
<td>0.51</td>
</tr>
</tbody>
</table>

All results are shown as results in the original sample.

Results demonstrate the ability of the 4200 MP-AES to:

- Measure Ag at low concentrations, with interferences corrected by IECs
- Achieve excellent recoveries across a wide concentration range (0.39% to 2.37% for Cu, and 0.51% to 4.27% for Zn)
- Determine all elements in a single sample preparation
Potential cost savings with the 4200 MP-AES

Total savings of more than US$150k over an 7 year evaluation period.

- FAAS with air compressor and 1 year consumables
- MP-AES with air compressor, SPS 3 and 1 year consumables
- 5 elements measured under method conditions
- 350 samples per week

This example is intended to help you compare the running costs & savings of the MP-AES vs. flame AA. The applied formulas and parameters are correct to the best of our knowledge, but we cannot guarantee the results. Savings may vary depending on factors such as local gas and electricity costs, operator costs, number and types of elements. For this calculation operator labor costs were set to US$25/hour and electricity costs were set to US$0.18 per kW.
Conclusion
The Agilent 4200 MP-AES is the ideal FAAS replacement.

Improved performance
• Increased linear working range
• Lower detection limits

Reduced running costs
• Runs on air

Increased safety
• No acetylene
• No nitrous oxide

Ease of use
• MP Expert
• IEC and FLIC
• Simple sample prep
4200 MP-AES - Summary

- Agilent’s 4200 MP-AES is the second generation of proven MP-AES technology.

- The 4200 offers safe, cost effective elemental analysis, with an application range that makes it an ideal flame AA alternative (upgrade!).

- 4200 MP-AES is very easy to use and has simple sample preparation.