Chemical Analysis of metal samples using Optical Emission Spectroscopy (OES)

Glen Thiele
Spectrosourse Pty Ltd
What is Optical emission spectroscopy (OES) also known as Atomic emission spectroscopy (AES)?
Aggregate state of matter

Temperature (°C)

1. Solid (strong bond)
2. Liquid (particles close together)
3. Gas (total disorder)
4. Plasma (free electrons and ions)

Boiling
Melting
Phase 1
Phase 2

Adding energy
Removing energy

Energy
Atomic physics – How photons are emitted

Figure 1: Bohr model (1913) Lithium --> excitation --> relaxation --> photon emission

Figure 2: Lithium emission spectrum
Optical emission Spectrometers

ICP OES

LIBS OES

Arc/Spark OES
Why Use Spark OES Analysis?

• Most widely used, reliable and reproducible metal analysis technique.
• Wide choice of instruments.
• Excellent LOD
• CCD based spectrometers allow a high number of elements and bases.
• Detection of N is possible with high end portable and high end laboratory spectrometers.
• Detection of O is possible with high end laboratory spectrometers.
PMT based Spark OES architecture
CCD based Spark OES architecture
Spark Stand architecture
Typical spark sequence

<table>
<thead>
<tr>
<th>Argon-Preflush</th>
<th>High energy Prespark</th>
<th>Integration</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 Sec. No spark</td>
<td>9 Sec. 30μF, 1 Ohm</td>
<td>5 Sec. 10μF, 15 Ohm</td>
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**Sparkling**

**Shutter**
Grating properties

Prism:
- non-linear refraction
- affected by temperature due to influence of medium density

Grating:
- linear diffraction
- different orders
Reciprocal dispersion as criteria of line separation

Reciprocal dispersion [nm/mm] | focal length [mm] | lines/mm
---|---|---
0.51 | 1000 | 500
0.52 | 750 | 2400
0.51 | 500 | 3600

3600 grooves/mm grating
Spark OES Spectrum 130nm – 800nm
Calculation of results from Calibration Curve

Calibration curve for Carbon in low alloy steel

result comparison to previously measured set of calibration samples

Emission spectrometry is a relative measurement method

Light intensity ratio

Element concentration ratio

results of calibration samples

interpolation

calibration curve extrapolation
Recalibration, Standardization or Drift correction
Illustration of exaggerated concentration error $\Delta$ in a calibration curve where Type calibration correction can be used to improve accuracy.
Measurement accuracy and reproducibility
The analytical Process

Sample taking
Sample preparation
Chemical analysis
Type Calibration and measure program
Material Data Base / Grade Table

- Software compares analysis results with element concentration ranges and reports grade matches
- Virtually unlimited material data entries
- International material standards as well as customised data base can be loaded
- Specific material data base can be linked to a measuring program
Thank you.
For more information or feedback contact Glen Thiele
mobile: 0420323577
email: info@spectrosouce.com.au

• References


• (3) Various definitions and images: Wikipedia.

• (4) Melting Spectro Analysis Version 6: G Henderieckx – Gietech BV