

# Analysis of Small Samples of Copper and its Alloys by Optical Emission

## ARL 4460 Metals Analyzer

### Key Words

- ARL 4460
- Copper
- Optical Emission
- Small samples

### Introduction

Significant progress has been made in the quantitative analysis of small samples of copper and its alloys. Sensitivity, precision and accuracy have been substantially improved with the Thermo Scientific ARL 4460 Metals Analyzer and the performance difference compared to “normal” samples is small. The preparation of small samples is simplified, and a faster analysis than with other techniques (ICP, chemical...) becomes possible, allowing for drastic reductions in analysis cost.

### Small samples made of copper

Several kinds of small samples made from copper and its alloys exist, distinguished by their shape, composition and use:

#### Shape:

- Wires with diameters down to less than 1 mm
- Sheets of various thickness
- Small samples of various shape

#### Composition:

Low alloy copper, brass, bronze, Al-bronze, new silver, gun metal, copper-nickel...

#### Use:

Wire cables, tubes, screws, nuts, axles, cogwheels, rivets, cams, rods,... for many application fields.

### ARL 4460 Metals Analyzer

Using a dedicated tool and a specific sparking condition, the ARL 4460 Metals Analyzer can quantitatively determine up to 24 elements in small samples in less than two minutes. Analytical performance is detailed in this application note.



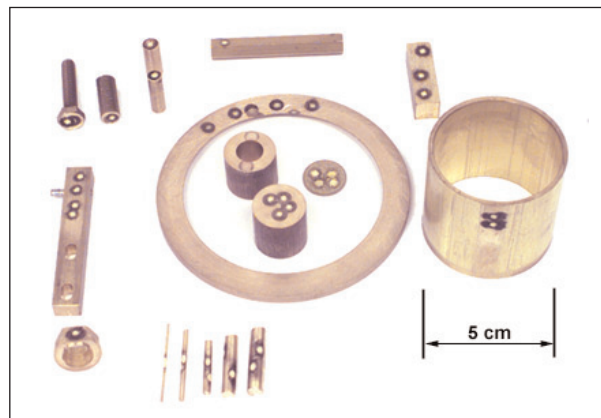
The Thermo Scientific Current Control Source (CCS) presents significant advantages in comparison to any other spark generator currently used for OES. The current waveform is computer controlled and the high degree of flexibility in selection of parameters enables the optimization of sensitivity, precision, and of the sparking diameter, which is important for the analysis of small samples.

More details can be found in the application note AN 41224 "Analysis of Copper Alloys by Optical emission - ARL 4460 Metals Analyzer".

### Small sample analysis

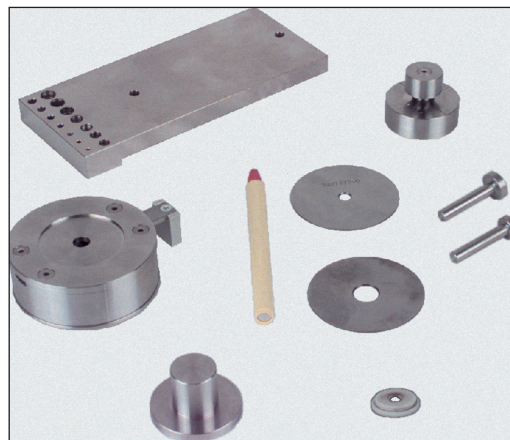
The ARL 4460 analyzes small samples of very different sizes and shapes as shown in the following picture:

- Wires: quantitative analysis down to 4 mm and semi-quantitative for smaller diameters
- Sheets: quantitative analysis down to 50 µm thickness



### Kit for small sample analysis

A small samples analysis kit is supplied as shown below:



## Sample preparation

The preparation depends on the sample shape:

- For wires: using a grinding machine and a special tool (included in the kit)
- For sheets: manual polishing with paper (grain 800)
- For samples of various shape: using a grinding machine
- In some cases, the sample can be pressed beforehand to obtain a larger surface area

## Sample analysis time

The analysis time is taken between the start of the analysis and the display of the result:

| MATERIAL     | "NORMAL" SAMPLE EXCITATION | SMALL SAMPLE CCS EXCITATION |
|--------------|----------------------------|-----------------------------|
| Copper alloy | 23 s                       | 37 s                        |

## Factory calibration (CARL)

ARL 4460 spectrometers can be factory calibrated for copper small sample analysis.

Our company proposes a specific global calibration for this OES application field, that covers all standard qualities available, with the exception of the ultra pure and pure copper qualities.

Calibration extensions with B and Be can be done at special request.

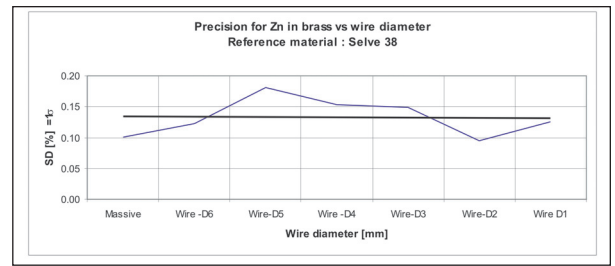
The standard pure copper wires with a diameter of 8 mm can be analyzed without any problem using a chuck and the analysis conditions for pure massive samples.

## Accuracy

In order to provide an idea of the accuracy, a massive reference material (Selve 38) was analyzed with the normal conditions and then machined to wires from 6 mm down to 1 mm and analyzed with the small sample conditions. The accuracy remains good as shown in the table below:

| EL. | CERTIF% | MASSIVE | 6 MM  | 5MM   | 4 MM  | 3 MM  | 2 MM  | 1 MM  |
|-----|---------|---------|-------|-------|-------|-------|-------|-------|
| Sn  | 0.15    | 0.151   | 0.152 | 0.152 | 0.146 | 0.145 | 0.146 | 0.122 |
| Zn  | 35.96   | 36.04   | 36.06 | 35.72 | 35.71 | 35.36 | 35.05 | 33.88 |
| Pb  | 0.065   | 0.064   | 0.069 | 0.067 | 0.070 | 0.071 | 0.073 | 0.085 |
| Fe  | 0.082   | 0.081   | 0.083 | 0.086 | 0.093 | 0.096 | 0.105 | 0.114 |
| Ni  | 2.26    | 2.25    | 2.24  | 2.26  | 2.23  | 2.25  | 2.28  | 2.29  |
| Al  | 3.25    | 3.25    | 3.27  | 3.43  | 3.50  | 3.68  | 3.75  | 3.81  |
| Si  | 0.090   | 0.089   | 0.091 | 0.092 | 0.090 | 0.090 | 0.088 | 0.085 |
| Mn  | 0.170   | 0.170   | 0.169 | 0.169 | 0.164 | 0.162 | 0.161 | 0.142 |
| Cu  | 57.97   | 57.90   | 57.86 | 58.01 | 57.98 | 58.13 | 58.34 | 59.47 |

The next figure shows the evolution of the precision for Zn in brass. The precision is almost stable down to 2 mm.

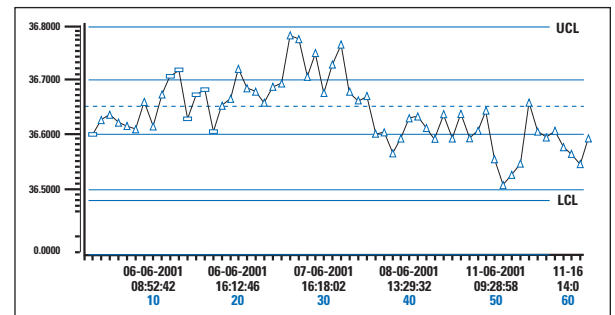


It is also worth mentioning that the accuracy is improved by a factor of at least three in the analysis of 3 mm wire in comparison with classical sources.

## Stability

Stability of the instrument is of the utmost importance when performing routine analysis. Typical mid-term stability measured over 24 hours shows that the standard deviation achieved is below two times the precision value, which is excellent.

The example below shows the long term stability of the zinc channel recorded over a period of 5 days without any intermediate drift correction. The values never went outside the control limits at 2 times the precision.



## Sensitivity and precision

The fundamental figures of merit are given in Table 1, on next page. The values are valid for a wire with a diameter of 6 mm. At worst they can degrade up to a factor of 3 with a wire with a diameter of 3 mm.

**Table 1: ARL 4460 - Typical detection limits (3 sigma) and precision values (1 sigma) for small samples in copper base**

| Element                    | Ag           | Al            | As           | Au           | B              | Be           | Bi           | Cd           | Co           | Cr           | Fe           | Mg             | Mn             | Ni           |
|----------------------------|--------------|---------------|--------------|--------------|----------------|--------------|--------------|--------------|--------------|--------------|--------------|----------------|----------------|--------------|
| <b>Typical DL [ppm]</b>    | <b>2</b>     | <b>6</b>      | <b>2</b>     | <b>0.7</b>   | <b>0.6</b>     | <b>0.6</b>   | <b>4</b>     | <b>1</b>     | <b>3</b>     | <b>1</b>     | <b>3.5</b>   | <b>0.2</b>     | <b>0.5</b>     | <b>1.7</b>   |
| <b>Guaranteed DL [ppm]</b> | <b>&lt;5</b> | <b>&lt;10</b> | <b>&lt;5</b> | <b>&lt;2</b> | <b>&lt;1.5</b> | <b>&lt;1</b> | <b>&lt;5</b> | <b>&lt;2</b> | <b>&lt;5</b> | <b>&lt;3</b> | <b>&lt;8</b> | <b>&lt;0.6</b> | <b>&lt;1.5</b> | <b>&lt;4</b> |
| Level [ppm]                | SD           | SD            | SD           | SD           | SD             | SD           | SD           | SD           | SD           | SD           | SD           | SD             | SD             | SD           |
| 10                         | 0.6          | 2             | 0.6          |              | 0.3            | 0.2          |              | 0.3          | 1.0          | 0.3          | 1.2          | 0.5            | 0.4            | 0.5          |
| 20                         | 0.7          | 2.5           | 0.8          |              | 0.5            | 0.25         | 1.3          | 0.7          | 1.0          | 1            | 1.2          | 1              | 0.6            | 0.5          |
| 50                         | 0.8          | 3             | 1.0          |              | 0.8            | 0.3          | 1.8          | 1.0          | 1.5          | 2            | 1.3          | 2              | 1.0            | 0.6          |
| 100                        | 1.5          | 4             | 2.0          |              | 1.5            | 0.4          | 2.5          | 2            | 1.7          | 4            | 1.5          | 3              | 2              | 0.7          |
| 200                        | 3            | 5             | 3.5          |              |                | 0.6          | 5            | 4            | 2.0          | 10           | 3            | 5              | 3              | 0.8          |
| 500                        | 6            | 7             | 7            |              |                | 1.2          | 12           |              | 5            | 20           | 5            | 9              | 5              | 2            |
| 1000                       | 11           | 12            | 15           |              |                | 2.3          | 25           |              | 8            |              | 10           | 12             | 10             | 4            |
| Level %                    |              |               |              |              |                |              |              |              |              |              |              |                |                |              |
| 0.2                        | 0.002        | 0.0020        |              |              |                | 0.0005       | 0.005        |              | 0.0025       |              | 0.002        | 0.0020         | 0.0015         | 0.0010       |
| 0.5                        | 0.005        | 0.0045        |              |              |                | 0.001        |              |              | 0.0035       |              | 0.004        | 0.0035         | 0.003          | 0.0025       |
| 1                          | 0.01         | 0.008         |              |              |                | 0.002        |              |              | 0.007        |              | 0.008        | 0.005          | 0.01           | 0.006        |
| 2                          |              | 0.015         |              |              |                | 0.004        |              |              | 0.011        |              | 0.015        |                | 0.02           | 0.013        |
| 5                          |              | 0.03          |              |              |                |              |              |              |              |              | 0.04         |                |                | 0.04         |
| 10                         |              | 0.05          |              |              |                |              |              |              |              |              |              |                |                | 0.09         |
| 20                         |              |               |              |              |                |              |              |              |              |              |              |                |                | 0.18         |
| 30                         |              |               |              |              |                |              |              |              |              |              |              |                |                | 0.25         |
| 40                         |              |               |              |              |                |              |              |              |              |              |              |                |                |              |

| Element                    | P            | Pb            | S              | Sb            | Se           | Si           | Sn           | Te            | Zn            | Zr           |
|----------------------------|--------------|---------------|----------------|---------------|--------------|--------------|--------------|---------------|---------------|--------------|
| <b>Typical DL [ppm]</b>    | <b>2</b>     | <b>15</b>     | <b>0.4</b>     | <b>8</b>      | <b>1.5</b>   | <b>2</b>     | <b>2</b>     | <b>10</b>     | <b>12</b>     | <b>0.5</b>   |
| <b>Guaranteed DL [ppm]</b> | <b>&lt;4</b> | <b>&lt;40</b> | <b>&lt;0.6</b> | <b>&lt;20</b> | <b>&lt;5</b> | <b>&lt;5</b> | <b>&lt;5</b> | <b>&lt;50</b> | <b>&lt;25</b> | <b>&lt;2</b> |
| Level [ppm]                | SD           | SD            | SD             | SD            | SD           | SD           | SD           | SD            | SD            | SD           |
| 10                         | 0.6          |               | 0.5            |               |              | 0.7          | 0.6          |               |               | 0.2          |
| 20                         | 0.8          |               | 1.0            | 2.5           |              | 0.8          | 1.0          | 3             | 4             | 0.3          |
| 50                         | 0.9          | 5             | 1.7            | 2.8           |              | 1            | 1.2          | 4             | 5             | 0.5          |
| 100                        | 1.6          | 6             | 2.7            | 3.2           |              | 1.5          | 2            | 5             | 6             | 1            |
| 200                        | 3            | 7             | 4.5            | 5             |              | 3            | 3            | 6             | 7             | 2            |
| 500                        | 7            | 8             | 8              | 8             |              | 6.5          | 5            | 10            | 8             | 6            |
| 1000                       | 15           | 15            | 13             | 13            |              | 12           | 10           |               | 12            | 11           |
| Level %                    |              |               |                |               |              |              |              |               |               |              |
| 0.2                        | 0.003        | 0.003         | 0.002          | 0.002         |              | 0.0022       | 0.002        |               | 0.002         |              |
| 0.5                        | 0.01         | 0.006         | 0.004          | 0.004         |              | 0.005        | 0.004        |               | 0.004         |              |
| 1                          | 0.025        | 0.012         |                | 0.1           |              | 0.01         | 0.0075       |               | 0.007         |              |
| 2                          |              | 0.025         |                |               |              | 0.018        | 0.013        |               | 0.012         |              |
| 5                          |              | 0.05          |                |               |              | 0.03         | 0.03         |               | 0.03          |              |
| 10                         |              | 0.1           |                |               |              |              | 0.05         |               | 0.06          |              |
| 20                         |              |               |                |               |              |              |              |               | 0.1           |              |
| 30                         |              |               |                |               |              |              |              |               | 0.15          |              |
| 40                         |              |               |                |               |              |              |              |               | 0.2           |              |

Remarks : This data applies when homogeneous samples are prepared by recommended sample preparation methods.  
 The precision given is typical performance. Guaranteed values will be 2 times higher.  
 For multibase instruments, some analytical performance may vary based on the analytical line selected.  
 Guaranteed DLs are calculated at 95 % confidence limit.

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