Application Note: 41202

Analysis of Copper and its Alloys by Optical Emission

ARL 3460 Metals Analyzer

Key Words

- ARL 3460
- Copper
- Metals Analyzer
- Optical Emission

Introduction

The Thermo Scientific ARL 3460 Metals Analyzer is custom designed to meet your specific requirements whether you are a small copper foundry or a large copper plant. Our long experience in metals analysis comes from an installed base of over 10,000 spectrometers worldwide. The ARL 3460 is the answer to your metallurgical analysis needs, whether they are incoming material control, or metal QC and production analysis. Working 24 hours a day and 7 days a week, the ARL 3460 delivers dependable performance year after year.

Pure Copper

Two main refining processes are used to produce pure and ultra pure copper:

- Pyrometallurgy
- Wetmetallurgy enrichment

Depending on the purity requested, a second pyrometallurgical procedure must also be applied.

Different levels of purity are required for various applications:

- The very high purity copper (electrolytic cathode copper) for electrical power transmission or electronic applications
- The fire-refined copper, mostly used to produce alloys like brass or bronze or Cupro-Nickel

It is worth to mention that it is difficult to produce a good representative sample of cathode copper. Different norms give sampling method procedures (i.e ASTM B115).

The ARL 3460 has the capacity to analyze pure copper. For ultra-pure copper some elements are below the limit of detection achievable with this instrument. It is recommended to consider the ARL 4460 if this level of analysis is requested.

Copper Alloys

Pure copper is mixed with other elements to produce a wide range of alloys:

- **Brasses** the most important non-ferrous alloy used for engineering
- **Bronzes** used for bearing and gears
- **Gun metal** used for casting due to its excellent fluidity
- **Aluminum bronzes** with good corrosion resistance but difficult to cast
- **Cupro-nickel** used for coins, tubes and wires
- **Nickel silver** used for marine applications, radiator, linen and fittings

Brass

Copper is capable of holding about 39 % of zinc in solid solution. Alloys containing less than 39 % Zn are known as α-brasses. This brass (70/30) is very widely employed for cartridges, cases, condenser tubes, etc.

From 39 to 46 % of Zn, a new β-solid solution gives αβ-brass. This alloy (60/40) is found in extensive engineering applications with enhanced corrosion resistance used in marine applications. Alloys containing more than 49 % Zn (γ-brass) are very hard and only used for brazing brasses.

Bronzes

There are a wide variety of bronzes containing various elements such as phosphorus, beryllium, silicon, etc:

- **Leaded bronzes** mainly used for bearings
- **Silicon bronzes** having high electrical conductivity and used for wires
- **Manganese bronzes** used for propellers and ship fittings
- **Phosphor bronzes** with up to 1 % P used for valves, bearings and gears

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ARL 3460 Metals Analyzer

The ARL 3460 optical emission spectrometer can determine simultaneously up to 60 elements. The HiRep source has the flexibility to customize the excitation parameters providing the best analytical conditions for a wide variety of sample types.

The source uses the HEPS (High Energy-Spark) technique to minimize the metallurgical effects and produce consistently accurate results. Printout of average concentration for the required elements from a repeat analysis is obtained in typically less than one minute.

Sample preparation

A lathe or milling machine is used to surface the sample. Grinding is not possible because of risk of contamination.

Sample analysis time

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

MATERIAL	ANALYSIS TIME [S]
Pure copper with oxygen	33
Alloyed copper	23

Factory calibration (CARL)

Thermo Scientific optical emission spectrometers can be factory calibrated for copper and its alloys utilizing CARL, a very sophisticated multi-variable regression tool that corrects for matrix effects as well as spectral interferences. CARL provides an immediate “turn-key” system with the highest accuracy possible. The calibrations are available for the different qualities given below. For each quality we use certified material as standard samples and setting-up samples are delivered with the instrument to maintain the accuracy of the calibration.

Ten qualities for specific alloy types and a general sorting programme are available:

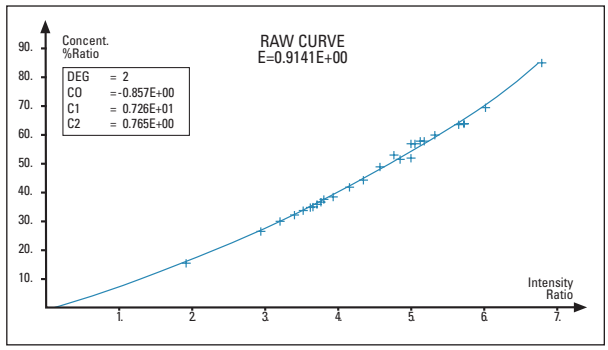
- Pure copper
- Brass
- Bronze
- Cu-Al
- Cupro-nickel
- Cu-Zn-Ni
- Gunmetal
- Cu-Co-Be-Ag
- Low alloy copper

The general sorting calibration offers more than a sorting program in terms of accuracy and number of analyzed elements. It can also be considered as the basic low cost calibration of the instrument, allowing the analysis of unknown samples.

Accuracy

Precision is only a small part of providing accurate analyses. The most important factor is the accuracy and quality of the calibration standards. Next is the development of the calibration curve relative to a specific analytical task. Matrix matching and high energy pre-burn reduce or eliminate matrix effects, and spectral interferences are significantly reduced by applying appropriate corrections.

The example shown below illustrates the significant improvement with the CARL calibration.



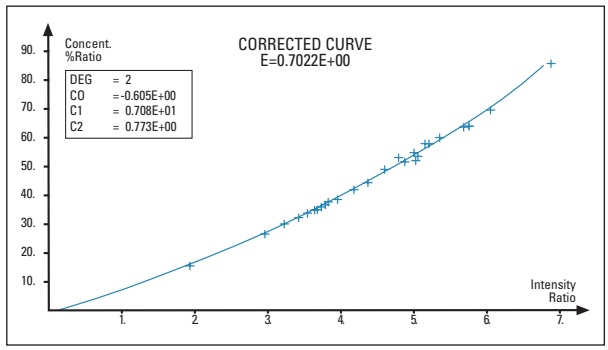


Table 2 below shows the analysis of the copper BAS 42.22-2 sample (International Certified Reference Material) and the comparison to the certified values:

ELEMENT	CONC.CERTIFIED %	SD CERTIFIED %	ARL 3460 MEASURED	DIFFERENCE %
Zn	26.32	0.04	26.28	-0.2
Sn	1.1	0.02	1.1	0.0
Pb	1.1	0.02	1.11	0.9
Fe	0.23	0.01	0.218	-5.2
Ni	0.061	0.002	0.061	0.0
P	0.177	0.003	0.178	0.6
Al	0.042	0.044	0.0364	-13.3
Si	0.042	0.034	0.039	-7.1
Mn	0.122	0.177	0.119	-2.5
Cu	70.37	70.408	70.6	0.3

Table 2

Table 1: ARL 3460 - TYPICAL detection limits (3 sigma) and precision values (1 sigma) for copper base

ELEMENT	Ag	Al	As	Be	Bi	Cd	Co	Cr	Fe	Mg	Mn	Ni	O	P
TYPICAL DL [ppm]	**0.2**	**0.8**	**0.4**	**< 0.1**	**0.8**	**0.2**	**0.8**	**0.7**	**0.7**	**0.4**	**< 0.1**	**0.5**	**8**	**0.3**
GUARANTEED DL [ppm]	**< 0.5**	**< 1.5**	**< 1.0**	**< 0.1**	**< 2.5**	**< 0.4**	**1.6**	**< 2**	**< 1.4**	**< 0.6**	**< 0.1**	**< 1.0**	**< 15**	**< 0.5**
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
10	1	1	1	1	1	1	1	0.4	1	1	1	1		0.5
20	1	1	1	1	1	1	1	0.5	1	1	1	2		0.5
50	2	2	2	1.5	1.5	1.5	1.5	0.7	2	2	2	3		0.7
100	2	2	4	2	2	2	3	1.2	3	2	3	4.5		1
200	4	4	8	4	4	4	5	2	8	4	5	6		2
500	8	10	20	10	10		8	5	11	8	8	12		5
1000	10	20	30	20	20		11		16	10	11	20		15
Level (%)														
0.2	0.002	0.003	0.005	0.004	0.004		0.003		0.0025	0.002	0.0015	0.003		0.005
0.5	0.005	0.007	0.008	0.01			0.006		0.0055		0.003	0.005		0.012
1	0.01	0.01					0.01		0.01		0.006	0.007		0.025
2		0.02							0.02		0.013	0.013		
4		0.04							0.04		0.025	0.016		
5		0.05							0.05		0.03	0.018		
10		0.1										0.02		
20												0.03		
30														
40														

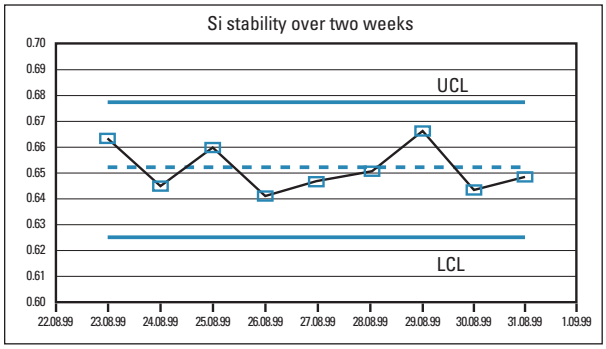
ELEMENT	Pb	S	Sb	Se	Si	Sn	Te	Zn	Zr
TYPICAL DL [PPM]	**4**	**0.2**	**1.3**	**0.3**	**0.7**	**0.4**	**1.5**	**2.5**	**0.15**
GUARANTEED DL [PPM]	**< 10**	**< 0.6**	**< 2**	**< 0.5**	**< 1.7**	**< 1**	**< 2.3**	**< 7**	**< 0.3**
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD
10	1	1	1	1	1	1	1	1	1
20	2	1	1	1.2	1	1	2	2	1
50	3	2	2	1.5	1.5	1.5	3	3	1.5
100	5	3	3	2	2	2	5	5	2
200	10	9	5		4	4	10	10	
500	20	9	10		8	8		20	
1000	50	18	19		20	20		30	
Level (%)									
0.2	0.01	0.0035	0.0025		0.004	0.004		0.004	
0.5	0.015		0.008		0.008	0.007		0.005	
1	0.02		0.017		0.015	0.014		0.006	
2	0.03					0.025		0.008	
4	0.05					0.05		0.012	
5	0.1					0.06		0.015	
10	0.2					0.1		0.03	
20						0.2		0.06	
30								0.10	
40								0.15	

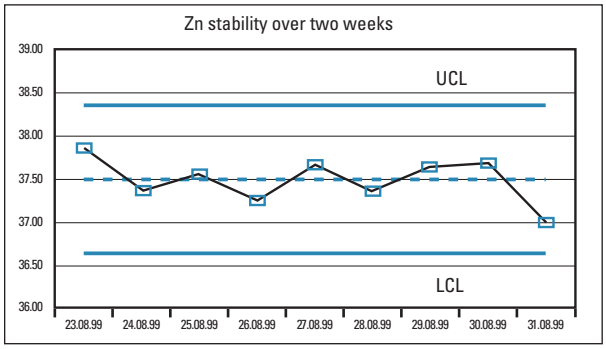
Remarks: This data applies when homogeneous samples are prepared by recommended sample preparation methods.
The detection limits were obtained with pure copper samples using the pure copper analytical conditions and the most sensitives lines.
For multibase instruments, some analytical performance may vary based on the analytical line selected.
Guaranteed DLs are calculated at 95 % confidence limit.

Stability

Stability of the instrument is of the utmost importance when doing 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 silicon and zinc channels recorded over a period of 15 days without any intermediate drift correction. The values never went outside the control limits.





Performance guarantee

Our company guarantees the values shown in Table 1 using homogeneous samples and recommended sample preparation methods. The precision is calculated from the formula:

$$SD\ (1s) = \pm \sqrt{\frac{\sum_{i=1}^{i=n} (X_i - \overline{X}_i)^2}{n - 1}}$$

where:

X_i the individual readings

$\overline{X}_i$ the arithmetic means of the individual readings

n the number of determinations

The precision given is typical performance, guaranteed values will be 1.5 times higher. The precision is based upon 10 successive measurements. The DL (Detection Limit) is defined as three times the standard deviation in intensities at the lowest point multiplied by the slope of the curve at zero concentration measured on a pure copper sample. The lower limits of quantification (LLQ) are alloy dependent and are defined in the calibration menus.

The performance list will be updated as improvements are announced. Please contact your nearest Thermo Fisher Scientific representative or consult our web site at **www.thermo.com/oes** for the most recent values.

Conclusion

The ARL 3460 has all the total system features which meet the critical needs of the metals analysis markets:

- Unmatched hardware for stability and reliability
- Excellent performance in detection limits, precision, accuracy, stability and memory effects, all this in minimum analysis time
- Most advanced software technology
- Easy operation by unskilled worker or research chemist
- Widest range of metals analysis
- Adaptable to the automatic Sample Manipulation System: ARL SMS-2000
- Advanced technical/service support
- Laboratory accreditation guidance
- Immediate access to parts inventory

All these features allow you to optimize your productivity and to achieve the shortest payback times:

- Your investment costs are reduced thanks to the exceptional and widely recognized instrument lifetime and to the continous upgrade possibilities (software and hardware)
- Your production costs are reduced by the fact that more accurate and reproducible analyses are available faster
- Your production costs are reduced by the increased instrument availability thanks to its high stability and drift corrections being less frequently required
- Your operating and maintenance costs are reduced through low consumption of drift correction samples, and through simple maintenance
- Your overall cost management is reduced by optimum utilization of materials and extremely low running costs compared to other methods

With its over 70 years of experience and history of innovative technology, our company has become the world leader in OES metals analysis. We work with our customers to improve the efficiency of their analytical tasks and thereby increase productivity.

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

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