

Analysis of Zinc and its Alloys by Optical Emission

ARL 4460 Metals Analyzer

Key Words

- ARL 4460
- Metals Analyzer
- Optical Emission
- Zinc

Introduction

Significant progress has been made in the analysis of zinc and its alloys. Sample analysis time, sensitivity, precision, accuracy and maintenance operations have all been substantially improved with the Thermo Scientific ARL 4460 Metals Analyzer.

Our long experience in metals analysis comes from an installed base of over 10,000 spectrometers worldwide. The ARL 4460 is the answer to your metallurgical analysis needs, whether they be incoming material control or metal QC and production analysis. Working 24 hours a day and 7 days a week, the ARL 4460 delivers dependable performance year after year.



The ARL 4460 Metals Analyzer can determine up to 60 elements simultaneously in less than 60 seconds. Specific performance is detailed in this application summary.

Zinc and its alloys

Zinc and zinc alloys are used in four main applications:

- Corrosion protection of steel (galvanization)
- In diecasting as injection of small and complicated mechanical parts with very high precision and high throughput
- Among zinc containing alloys, copper base alloys such as brasses are large zinc consumers
- Chemical applications present as zinc oxide such as rubber, paints, ceramics and as a plant nutrient in agriculture

Almost half of the zinc consumed in the world is used for corrosion-protection coatings on iron and low-alloy steels. Zinc anodes are also used to provide galvanic sacrificial protection in water.

Zinc foundry alloys are more and more used in numerous applications. A majority of casting alloys are Zn-Al alloys (Zamak). They can go up to 27 % Al for higher-strength applications.

Titanium (0.1-0.15 %) with a small amount of Copper and Manganese improves the quality of the zinc material. These alloys are used for forged or extruded zinc.

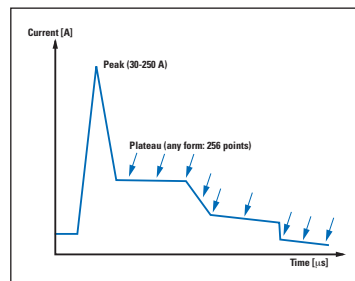
Alloys containing zinc, like brass, nickel silver, aluminum brass are manufactured using pure zinc.

Current Controlled Source (CCS)

The Thermo Scientific Current Controlled Source presents significant advantages in comparison to any other spark generator currently used for OES. It is the only servo-controlled “digital source” on the market.

The current waveform is computer controlled and can be selected for each type of metal. The high degree of flexibility in selection of peak current (250 A max.), frequency (1000 Hz max.) and current waveforms enables the optimization of all analytical figures.

The very compact design close to the spark stand, in a Faraday cage, suppresses RF leakage and improves general stability (less current lost in connecting cables).

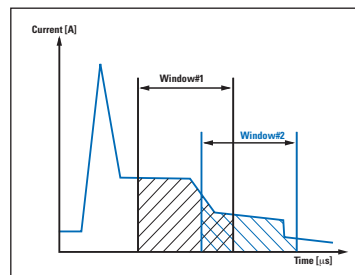


Time Resolved Spectroscopy (TRS)

During the time of an individual spark, the line-to-background ratio varies significantly. The selection of the appropriate period of the spark when data acquisition takes place improves sensitivity and precision for trace analysis.

TRS removes the first peak of current (high background), and selects a “window” (the best section of the “plateau” current) for each element.

In case of spectral line interferences, when there is a significant difference in the excitation potential of the analyte and of interfering lines, the accuracy of calibration can be substantially improved using TRS.



Small electrode

A new electrode has been introduced on the ARL 4460 which allows routine analysis without the need to brush after each run. This new small electrode achieves the same performance as the previous electrodes, drastically reduces the level of contamination in routine analysis and needs to be changed only every 2'000 runs.

A substantial gain of time is realized by not brushing the electrode and the spark chamber is less perturbed by the stirring of air.

Sample preparation

A lathe or a milling machine is used to prepare the samples. 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	CONVENTIONAL LVS EXCITATION	ARL 4460 CCS
Pure Zn, Galfan, Zamak	35 s	21 s
Spelter, Zintane	40 s	25 s

About 30 % of the sample analysis time has been saved in comparison to conventional excitations.

Factory calibration (CARL)

Thermo optical emission spectrometers can be factory calibrated for zinc 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 "turnkey" system which gives the user 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.

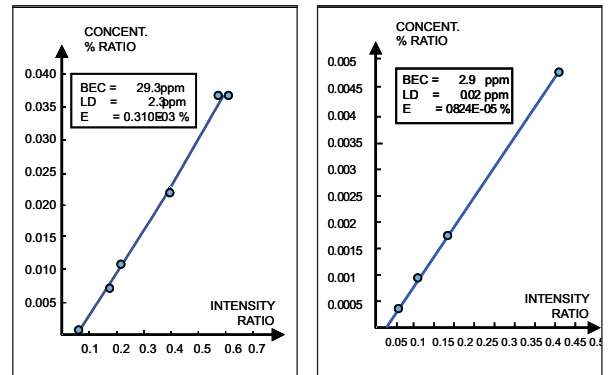
The calibrations available are for:

- Pure zinc
- Zn-Al (Al <4.7 %)
- Zn-Al-Cu (Al <5.1 %, Cu <1.5 %)
- ZA8 - ZA12 (Al <13 %, Cu <3.9 %)
- ZA27 (Al <29 %, Cu <3 %)
- Galfan
- Zamak 2
- Galva 5
- Zintane
- Spelter

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 in the calibration curve because of the TRS feature.



Element: Cu7 324.75x2 without TRS Element: Cu7 325x2 with TRS

The table below illustrates the accuracy of the Thermo Scientific factory calibration for a Zamak5 alloy:

ELEMENT	CALIBRATION CURVE RANGE [%]	NR. OF STANDARDS USED	STANDARD ERROR OF ESTIMATE (SEE)*[%]
Al	3.4 - 5.1	10	0.025
Cd	0.0001 - 0.03	9	0.000014
Cr	0.00015 - 0.01	8	0.000012
Cu	0.2 - 1.45	9	0.0067
Fe	0.0003 - 0.13	10	0.0019
In	0.0003 - 0.003	4	0.000014
Mg	0.0001 - 0.15	10	0.0011
Mn	0.0005 - 0.01	12	0.00017
Ni	0.0002 - 0.025	10	0.00017
Pb	0.0002 - 0.025	11	0.00023
Si	0.0001 - 0.08	13	0.00046
Sn	0.0003 - 0.03	9	0.00020
Tl	0.0002 - 0.004	6	0.00001

Table 1: ARL 4460 - Typical detection limits (3 sigma) and precision values (1 sigma) for zinc base

ELEMENT	Ag	Al	As	Be	Bi	Ca	Cd	Ce	Cr	Cu	Fe	Ge
Typical DL [ppm]	0.5	0.1	5	0.1	0.2	0.2	0.03	0.6	0.6	0.08	0.5	2
Guaranteed DL [ppm]	≤1	≤0.3	≤15	≤0.5	≤0.5	≤0.6	≤0.1	≤1	≤1	≤0.2	≤1	≤4
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
1		0.1					≤0.1			≤0.1		
2		0.2		<0.1	0.3	0.5	≤0.1			0.1		
5	0.3	0.5		0.1	0.5	1.5	≤0.1	0.4	0.2	0.15	0.6	
10	0.35	0.7		0.3	0.8	3.5	0.15	0.7	0.4	0.25	0.7	2
20	0.5	1.3		0.7	1		0.3	1	1	0.4	0.8	3
50	0.8	2.5	4	2.5	2		0.6	2.5	2.5	0.8	2	5
100	1.5	4	6	6.5	2.5		1	4	5	1	3.5	8
200	2.5	6	8	15	3.5		2	7	10	2	6.5	
500		12	12	60	6		5	15	30	5	15	
1000		18					10			10	30	

Level %												
0.2			0.003				0.002			0.0015	0.006	
0.5			0.005				0.004			0.0035	0.015	
1			0.01							0.006	0.025	
2			0.015							0.01	0.05	
5			0.025							0.015	0.15	
10			0.04							0.05		
20			0.065							0.08		
30			0.10									
40			0.15									
50			0.20									

ELEMENT	In	La	Mg	Mn	Ni	Pb	Sb	Si	Sn	Ti	Tl	V
Typical DL [ppm]	1.5	0.2	0.03	0.2	0.4	0.5	6	0.2	1.5	1.0	0.4	0.3
≤	≤2.5	≤0.4	≤0.1	≤0.5	≤1	≤1	≤10	≤0.5	≤3	≤1.5	≤0.6	≤1
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
1			≤0.1							0.7		
2			≤0.1	0.2				0.15		0.7	0.3	0.2
5			0.1	0.4	0.5	0.15		0.2		0.8	0.4	0.25
10	0.8		0.2	0.6	0.8	0.3		0.25	0.8	1	0.6	0.3
20	1.5		0.4	1.0	1.3	0.5		0.3	1	1.5	0.8	0.5
50	3		0.8	1.5	2.5	1	5	0.6	2	3	1.2	1
100	6		1.5	2.5	4	2	8	1.5	3	5	1.5	2.5
200	9		2	4	7	3	10	8	5	8	2.5	
500	20		5		15	7	20		9	15		
1000	35		8		25	15	25		15	25		

Level %												
0.2		0.006		0.0015		0.0040	0.0025	0.004		0.003	0.004	
0.5		0.015		0.003		0.0075	0.005	0.006		0.007		
1					0.01	0.01	0.01		0.015			
2					0.2	0.015	0.015		0.03			
5						0.025						
10												
20												
30												
40												
50												

Remarks : This data applies when homogeneous samples are prepared by recommended sample preparation methods.
 The precision given is typical performance. Guaranteed values will be 1.5 times higher.
 The precision is based upon 10 successive measurements.
 For multibase instruments, some analytical performance may vary based on the analytical line selected.
 Guaranteed DLs are calculated at 95% confidence limit.

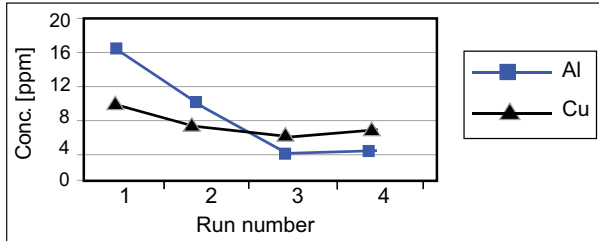
Option

Spark-DAT on-line

The purpose of Spark-DAT (Spark Data Acquisition and Treatment) is to acquire and treat the voltages of each spark instead of the global and integrated value. Spark-DAT allows mainly the determination of the composition and granulometry of inclusions. A separate product specifications sheet is available on request.

Memory effects

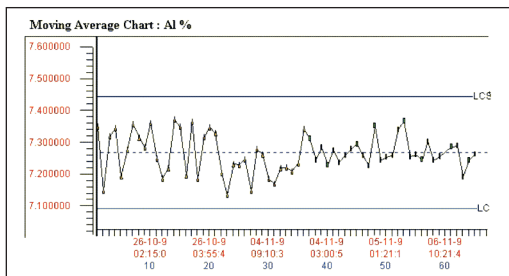
There is almost no memory effect. For testing purposes, alloyed samples (one with 4 % Al, then another with 1 % Cu) were analyzed before a pure Zn sample. The levels of the pure Zn samples are obtained after 3 runs.



Stability

Stability of the instrument is of the utmost importance when doing routine analysis. 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 one element recorded over a period of 15 days without any intermediate drift on sample SUS RA 18/1. The values never went outside the control limits and no standardization was required.



Performance guarantee

Our company guarantees the precision shown in Table 1 using homogeneous samples and recommended sample preparation. The list will be updated as improvements are announced. Please contact your nearest Thermo Fisher Scientific office for the most recent values.

To see our complete OES product portfolio, visit www.thermoscientific.com/oes.

www.thermoscientific.com

©2010 Thermo Fisher Scientific Inc. All rights reserved. All trademarks are the property of Thermo Fisher Scientific Inc. and its subsidiaries. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details.

The precision is calculated from the formula:

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

where:

- $\frac{X_i}{\bar{X}}$ the individual readings
- \bar{X} the arithmetic mean of the individual values
- n the number of determinations

The DL (Detection Limit) is defined as three times the standard deviation of the background expressed in concentration units, on a pure zinc sample.

Conclusion

The ARL 4460 provides not only state-of-the-art technology, but also has all the total system features which meet the critical needs of the metals analysis markets.

- Unmatched hardware for stability and reliability
- Exceptional performance in detection limits, precision, accuracy, stability and memory effects, all this in minimum analysis time
- Most advanced software technology with HTML/Internet simple to use tools
- Potential to cover your future analytical needs
- 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 continuous upgrade possibilities (software and hardware)
- Your investment costs are reduced with the capability of the instrument to cover your future needs
- 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 over 70 years of experience and history of innovative technology makes our company has become the world leader in OE 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.

Africa-Other
+27 11 570 1840

Australia
+61 3 9757 4300

Austria
+43 1 333 50 34 0

Belgium
+32 53 73 42 41

Canada
+1 800 530 8447

China
+86 10 8419 3588

Denmark
+45 70 23 62 60

Europe-Other
+43 1 333 50 34 0

Finland/Norway/Sweden
+46 8 556 468 00

France
+33 1 60 92 48 00

Germany
+49 6103 408 1014

India
+91 22 6742 9434

Italy
+39 02 950 591

Japan
+81 45 453 9100

Latin America
+1 561 688 8700

Middle East
+43 1 333 50 34 0

Netherlands
+31 76 579 55 55

New Zealand
+64 9 980 6700

Russia/CIS
+43 1 333 50 34 0

South Africa
+27 11 570 1840

Spain
+34 914 845 965

Switzerland
+41 21 694 71 11

UK
+44 1442 233555

USA
+1 800 532 4752



Thermo Fisher Scientific (Ecuublens) SARL, Switzerland is ISO certified.

AN41230_E 10/10C