Application Note: 41203

Analysis of Lead and its Alloys by Optical Emission

ARL 3460 Metals Analyzer

Key Words
- ARL 3460
- Lead
- 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 foundry or a large lead 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 be 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.

Lead

With particular properties, lead is the unique material for solving a variety of problems. The most interesting are:

- High density (113 g/cm^3)
- High malleability
- Corrosion resistant
- Low melting point (328° C)
- Unusual electrical properties

Currently the major applications are:

• Car batteries	45 %
• Industry	18 %
• Building	15 %
• Energy	14 %
• Miscellaneous	8 %

Batteries remain the primary use of lead. The recycling market from battery scrap represents a large volume of the world lead production. Lead oxide is used for crystal manufacturing as well for battery and for chemical process.

Lead is used in the following main applications :

- Pure lead for cable shielding, tubes, primary material (DIN 719)
- Lead-calcium for new batteries (ASTM B29)
- Lead antimony for common batteries and storage batteries (DIN 17641)
- Lead-Sn with 7-15 % Sn for coating alloys (ASTM B32)
- Lead-Sn with 5-63 % Sn for soldering (with Ag for electronic applications)
- Lead-Sb-Cu-As for bearings (DIN 1703)
- Lead 99.9 % for radio-protection (or sound attenuation)

ARL 3460 Metals Analyzer

The ARL 3460 optical emission spectrometer can determine 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. The printout of average concentration for the required elements from a repeat analysis is obtained in typically one minute.

Sample preparation

A lathe or a milling machine is used to prepare the samples. Grinding is not possible due to the risk of contamination.

Sample analysis time

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

ALUMINUM MATERIAL	ANALYSIS TIME
Pure lead and lead alloys	19 s

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Factory calibration (CARL)

The Thermo Scientific optical emission spectrometers can be factory calibrated for lead and its alloys utilizing CARL, a very sophisticated multi-variable regression tool that corrects for matrix effects as well as spectral interferences. CARL can provide an immediate “turn-key” system which gives the user the highest accuracy possible. For each quality we use uses certified reference material as primary calibration samples and setting-up samples are delivered with the instrument to maintain the accuracy of the calibration.

We provide calibrations for several qualities of lead and lead alloys:

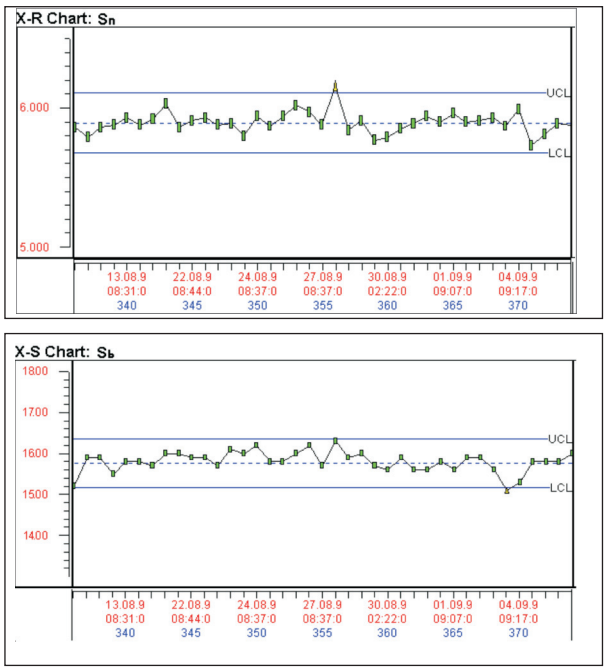
- Pure lead (Pb <99.5 %)
- Low alloy lead
- Pb-Sn (Sn 9-65 %)
- Pb-Sn-Sb (Sn <50 %; Sb <15 %)
- Pb-Sb battery lead (Sb <12.5 %)
- Pb-Ca battery lead (Ca <0.14 %)
- Global calibration

Please note that global calibration offers more than a sorting program in terms of accuracy and number of analyzed elements. It can be considered as the basic low cost calibration, allowing the analysis of unknown samples. The use of a global calibration is recommended when the program choice function is used.

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 examples below show the long term stability of the antimony and tin channels recorded over a period of 20 days with intermediate drift corrections. The values almost never went outside the control limits.



Accuracy

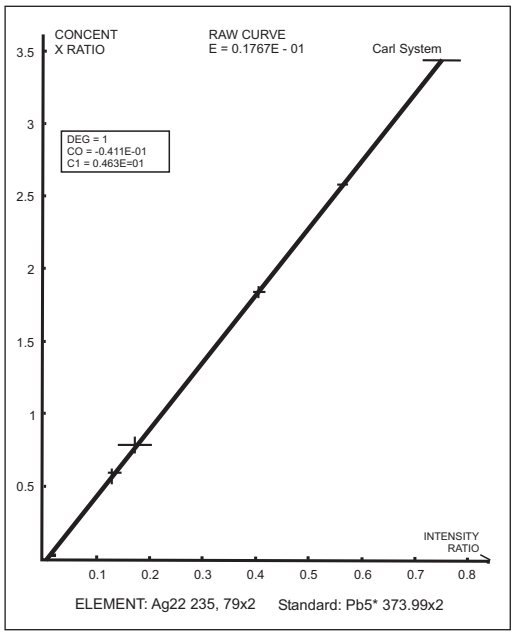
Precision is only a small part of providing accurate analyses. The most important factor is the accuracy and quality of the calibration standards. Next is the development of the calibration curve relative to a specific analytical task. Matrix matching and high energy pre-burn reduce or eliminate matrix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

With the ARL 3460 the calibration curves are linear along a large range of concentrations.

For example :

- Ag in the Pb-Sn alloys is linear up to 3%
- Sb in Pb-Sb is nearly linear up to 15%

Both curves show an excellent fit.



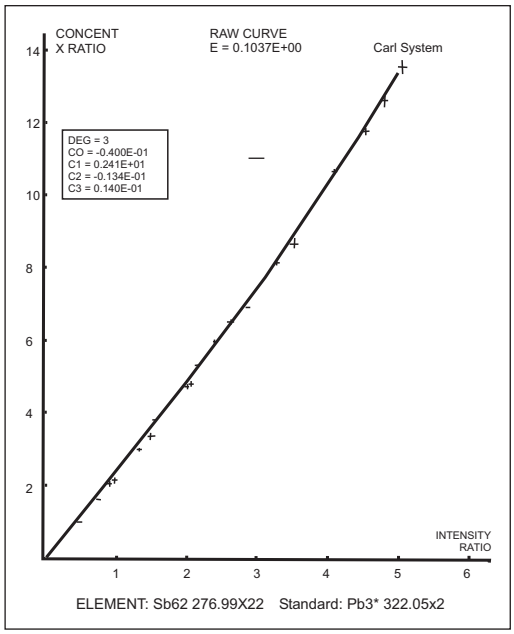


Table 1: Arl 3460 - Typical detection limits (3 sigma) and precision values (1 sigma) for lead base

ELEMENT	Ag	Al	As	Bi	Ca	Cd	Cu	Fe	Ni	S	Sb	Se	Sn	Te	Zn
Typical DL [ppm]	**0.01**	**0.2**	**0.5**	**0.05**	**0.05**	**0.02**	**0.01**	**0.2**	**0.01**	**1**	**0.5**	**0.5**	**0.5**	**0.5**	**0.2**
Guaranteed DL [ppm]	**≤0.05**	**≤1**	**≤1**	**≤0.2**	**≤0.2**	**≤0.05**	**≤0.1**	**≤0.6**	**≤0.1**	**≤2**	**≤1**	**≤2**	**≤1**	**≤1**	**≤1**
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
2	0.1	0.2	0.3	0.04	0.1	0.1	0.1	0.5	0.1		0.5		0.4	1	0.5
5	0.15	0.3	0.5	0.06	0.2	0.2	0.2	1	0.2	1	0.5	1	0.6	1	1
10	0.2	0.4	1	0.1	0.3	0.3	0.3	2	0.3	2	1	1	0.8	1	1
20	0.3	0.6	1.5	0.2	0.5	0.5	0.7	4	0.4	4	1.5	2	1	1.2	1.2
50	1	1	2.5	0.5	1	0.8	1.5	8	0.6	8	2	3	2	1.5	2
100	2	2	4	1	1.5	1.2	3	15	1.5	15	2.5	4	3.5	2	4
200	3	3	6	2	2.5	2.0	4	25	3	25	3	6	5	3	10
500	8	6	10	5	5	5	10	60	10		4	8	10		15
1000	15		15	10	10	10	20		20		5		20		20
Level %															
0.2	0.003		0.002	0.0015	0.0015		0.004		0.004		0.001		0.0025		
0.5	0.006		0.004	0.0035			0.01				0.002		0.007		
1	0.01		0.005								0.004		0.01		
2	0.03										0.007		0.02		
5	0.06										0.02		0.05		
10											0.05		0.06		
20											0.2		0.07		
30											0.3		0.08		
40													0.09		
50													0.1		

Remarks: This data applies when homogeneous samples are prepared by recommended sample preparation methods.
For multibase instruments, some analytical performance may vary based on the analytical line selected.
Guaranteed DLs are calculated at 95% confidence limit.

Performance guarantee

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

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

$$RSD=\frac{SD}{X_i}$$

where:

X_i the individual readings

$\overline{X}_i$ the arithmetic mean 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 lead 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/elemental 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 analysis time
- Most advanced software technology with HTML/Internet simple to use tools
- 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 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.

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