

Analysis of Ultra-Pure Aluminum by Optical Emission

ARL 4460 Metals Analyzer

Key Words

- ARL 4460
- Metals Analyzer
- Optical Emission
- Ultra-Pure Aluminum

Introduction

There is a requirement in certain areas of the aluminum industry for the analysis of ultra-pure aluminum from 99.99 up to 99.999 %.

Thanks to the progresses made in optical emission spectrometry, in particular with the availability of the Current Controlled Source (CCS) and the Time Resolved Spectrometry (TRS), the Thermo Scientific ARL 4460 makes analysis at these levels possible.

The preparation of ultra-pure samples is simplified and a faster analysis than with other techniques (mass spectrometry, neutron activation, graphite furnace...) becomes possible, allowing for drastic reductions of analysis costs.

ARL 4460 Metals Analyzer



Using a specific sparking condition, the ARL 4460 Metals Analyzer can determine simultaneously up to 40 elements in ultra-pure aluminum in less than fifty seconds. Analytical performance is detailed in this application note.

The Thermo 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.

The Thermo Scientific Time Resolved Spectrometry (TRS) also presents significant advantages in comparison to other acquisition systems. The selection of the appropriate period of the spark allows acquiring data at best signal-to-noise ratio and improves the sensitivity for trace analysis.

More details can be found in the application summary “Analysis of Aluminum and its Alloys by Optical Emission – ARL 4460 Metals Analyzer”.

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, taken between the start of the analysis and the display of its result, is around 23 seconds.

Factory calibration (CARL)

Thermo Scientific optical emission spectrometers can be factory calibrated for ultra-pure aluminum.

The calibration covers all elements described in Table 1 on next page and covers ranges from the detection limit up to 100 ppm.

Elements such as C, Ce, Cl, La, Nd, O, U, W... can be added as long as the customer is able to provide reference samples.

Accuracy

The most important analytical figure of merit is the accuracy. The next example illustrates the accuracy achieved on the determination of some reference samples:

SAMPLES	EL.	REF. VALUE [PPM]	PRECISION REF. VALUE [PPM]	MEASURED VALUE [PPM]	DIFF. [PPM]
Pechiney 566	Si	4	0.5	3.6	0.4
VAW 1000	Si	15	1.8	13.2	1.8
VAW 1001	Si	48	2.4	46.5	1.5
Pechiney 566	Fe	7.5	0.6	7.7	-0.2
VAW 1000	Fe	3	0.3	2.2	0.8
VAW 1001	Fe	8.6	0.6	8.4	0.2
Pech 566	Cu	2.2	0.15	2.3	-0.1
VAW 1000	Cu	1.2	0.12	1.3	-0.1
VAW 1001	Cu	1.3	0.15	1.4	-0.1
Pechiney 566	Mg	3.5	0.3	3.6	-0.1
VAW 1000	Mg	2.3	0.3	2.6	-0.3
VAW 1001	Mg	14	1.4	14.4	-0.4
Pechiney 566	Zn	3.5	0.3	3.6	-0.1
VAW 1000	Zn	3.5	0.4	4.2	-0.7
VAW 1001	Zn	9	1	9.6	-0.6

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.

Table 1: ARL 4460 - Typical detection limits (3 sigma) and precision values (1 sigma) for Ultra-Pure Aluminum

ELEMENT	Ag	As	B	Ba	Be	Bi	Ca	Cd	Co	Cr	Cu	Fe	Ga	Hg	In	Li
TYPICAL DL [PPM]	0.007	1.5	0.08	0.02	0.001	0.2	0.03	0.09	0.05	0.05	0.02	0.1	0.06	0.25	0.1	0.02
GUARANTEED DL [PPM]	≤ 0.015	≤ 3	≤ 0.15	≤ 0.03	≤ 0.002	≤ 0.4	≤ 0.06	≤ 0.25	≤ 0.15	≤ 0.1	≤ 0.07	≤ 0.25	≤ 0.12	≤ 0.5	≤ 0.25	≤ 0.05
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.2	0.002		0.025	0.007	0.0006	0.06	0.009	0.03	0.018	0.015	0.006	0.04	0.02	0.08	0.03	0.006
0.5	0.002		0.03	0.008	0.009	0.067	0.015	0.03	0.02	0.015	0.006	0.04	0.02	0.08	0.03	0.008
1	0.003		0.044	0.01	0.0013	0.07	0.025	0.035	0.022	0.016	0.007	0.05	0.025	0.085	0.035	0.018
2	0.006	0.5	0.08	0.012	0.0025	0.075	0.045	0.04	0.025	0.02	0.01	0.055	0.027	0.09	0.037	0.037
5	0.013	0.65	0.19	0.02	0.006	0.09	0.1	0.055	0.04	0.03	0.02	0.07	0.04	0.12	0.05	0.1
10	0.003	0.7	0.4	0.04	0.02	0.1	0.2	0.07	0.06	0.05	0.04	0.1	0.06	0.15	0.07	
20	0.06	0.8	0.8	0.06	0.045	0.15	0.4	0.15	0.1	0.1	0.08	0.15	0.1	0.25	0.12	
50	0.2	1.3		0.15		0.3	0.9	0.3	0.25	0.2	0.2	0.3	0.25	0.45		
100	0.5	2				0.6		0.6	0.5	0.45	0.35	0.6	0.5			

ELEMENT	Mg	Mn	Mo	Na	Ni	P	Pb	Sb	Si	Sn	Sr	Ti	Tl	V	Zn	Zr
TYPICAL DL [PPM]	0.006	0.1	0.02	0.03	0.06	1.1	0.1	0.6	0.1	0.2	0.02	0.06	0.07	0.08	0.2	0.06
GUARANTEED DL [PPM]	≤ 0.015	≤ 0.2	≤ 0.05	≤ 0.06	≤ 0.2	≤ 2	≤ 0.25	≤ 1.2	≤ 0.2	≤ 0.4	≤ 0.04	≤ 0.12	≤ 0.2	≤ 0.15	≤ 0.4	≤ 0.2
Level [ppm]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.2	0.002	0.03	0.007	0.01	0.02		0.04		0.03	0.06	0.007	0.02	0.025	0.025	0.06	0.02
0.5	0.004	0.03	0.007	0.01	0.02		0.04		0.03	0.06	0.011	0.02	0.03	0.03	0.06	0.025
1	0.006	0.035	0.009	0.02	0.03	0.3	0.04	0.2	0.032	0.062	0.02	0.025	0.03	0.03	0.065	0.03
2	0.012	0.04	0.015	0.04	0.035	0.3	0.045	0.22	0.035	0.065	0.04	0.03	0.035	0.045	0.07	0.04
5	0.035	0.07	0.03	0.1	0.05	0.35	0.08	0.3	0.045	0.08	0.08	0.07	0.08	0.08	0.1	0.07
10	0.065	0.1	0.05	0.2	0.07	0.4	0.12	0.35	0.055	0.11	0.15	0.11	0.15	0.15	0.12	0.1
20	0.1	0.15	0.1	0.4	0.1	0.5	0.2	0.4	0.08	0.15	0.25	0.2		0.3	0.15	0.15
50	0.25	0.3			0.2		0.5	0.7	0.2	0.25		0.6		0.7	0.25	0.3
100	0.35	0.4					0.6	1.2	0.35	0.4		1.2			0.35	0.5

Remarks: This data applies when homogenous 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. Guaranteed DLs are calculated at 95% confidence limit.

Performance guarantee

The fundamental figures of merit are given in Table 1. Our company guarantees the precision shown 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. The precision is calculated from the formula:

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

where:
 X_i the individual readings
 \bar{X}_i the arithmetic mean of the individual readings
 n the number of determinations

The DL (Detection Limit) is defined as three times the standard deviation of the background expressed in concentration units.

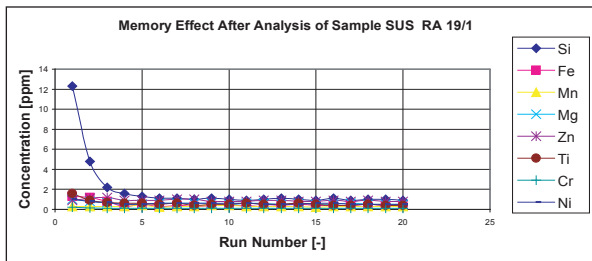
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Memory effects

Memory effects are determined by running an alloyed sample (Si 1.4 %, Zn 6.9 %, Mg 8.2%...) and then following the signal decrease on a pure sample. The figure below shows the evolution of the memory effect with time:



Silicon is the most difficult element to remove as it has the highest fusion temperature. On this element, there is a remaining effect of 18 ppm after 4 runs and 10 runs are required to be at the level of the pure sample. The other elements need no more than 4 runs to be at the desired level. Therefore when the instrument has to also analyze other aluminum qualities, it is recommended to have specific sparking chamber pieces for the ultra-pure analysis and to exchange them when needed.

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