APPLICATION NOTE

Analysis of Lead and its Alloys with Thermo Scientific ARL iSpark 8860 Optical Emission Spectrometer

For over 80 years, our company has set the standard of quality for spectrochemical analysis of metals. Throughout these years, accuracy, performance, stability, reliability and longevity have been the key attributes of our optical emission spectrometers. The Thermo Scientific™ ARL iSpark™ 8860 metals analyzer combines these guiding principles with our experience and technical innovation to bring our customers, the complete value based solution they have come to expect from our company.

The ARL iSpark 8860 will determine the elements necessary in your current and future applications, in all possible lead and lead alloy grades. It is the answer to your analytical needs, whether for incoming goods control, metal sorting, process QC, final product QC, certification or investigation. Working 24 hours a day and 7 days a week, the ARL iSpark 8860 Metals Analyzer delivers dependable performance year after year. Specific performance is detailed in this application summary.

ARL iSpark 8860 – experience and performance

The ARL iSpark 8860 is based on Thermo Scientific famous one-meter PMT spectrometer in Paschen-Runge mounting operated under vacuum. The spectrometer offers optimal resolution and stability and ensures outstanding performance for all the elements. Highly innovative features and technologies characterize further the instrument, among which:

- Advanced signal acquisition and processing improving performance and accuracy
- IntelliSource, a digital spark source with increased flexibility and efficiency
- Spark stand with improved design to reduce maintenance and minimize argon consumption during the analysis
- ECOmode and super ECOmode allowing significant argon savings when the instrument is idle
- Optimal instrument design increasing its functionality.

ThermoFisher SCIENTIFIC

intelliSource digital spark source

The Thermo Scientific™ intelliSource is a double current controlled source (CCS), the most innovative excitation source for Spark-OES. More flexible than the other digital sources, it allows discharge shapes to be tailored for most efficient control of the sample surface preparation, material ablation and light emission in various metal matrices.

Matrix-optimized pre-integration sparks significantly reduce the effects of matrix and metallurgical structure (in re-melting the sample surface before the integration spark), which improves the accuracy of the analysis. Other innovative features contribute to the performance of the intelliSource, e.g. DISC (Discharge Interrupt by Short-Circuit) that improves the repeatability in trace analysis.

Time Gated Acquisition (TGA)

TGA is a high precision version of the TRS (Time Resolved Spectroscopy). The acquisition is performed during TGA windows, i.e. very short time windows synchronized with the single sparks. Optimized start and duration of the window allow collecting the signal of interest when unwanted signals (e.g. noise, high background emission or spectral interference) have minimal deleterious effect. This results in lower detection limits. In some cases, TGA also reduces the need for interference correction, therefore improving accuracy.

Signal processing

The signals integrated during the TGA windows are digitized for each individual spark. Several signal processing methods contribute to the performance with the PMTs:

- The algorithm FAST (Flexible Acquisition STart/stop) allows acquiring an optimal subset of single spark intensities - the most steady part of the signal - for each channel
- The algorithm DISIRE (DIffuse Spark Intensities REmoval) improves the precision by discarding abnormally low intensity signals

Sample preparation

The sample surface is generally prepared by using a lathe or a milling machine.

Sample analysis time

The analysis time taken between the start of the analysis and the display of its result is in average 13.5s per run.

Performance guarantee

Our company guarantees the precision and the detection limit (DL). The precision expresses the closeness of the concentration values of the individual runs of an analysis. The lowest the precision value, the smallest the number of runs needed for high confidence in the average result. The DL is the smallest concentration that can be distinguished from a blank value with a given probability.

For quantitative analysis, however the lower limit of quantification (LLQ ≈ 3·DL) must be considered. The LLQ is also the lowest possible value in our calibration menu, when calibration standards are available for calibration down to this limit.

Accuracy and factory calibration

The accuracy is the most important figure of merit of a spectrometer. It expresses the agreement between the result and the reference value. It depends on the quality of the reference materials used for calibration, on some instrumental attributes and parameters (e.g. the optical resolution, the spark source condition or the TGA window), and on the mathematical model used to calculate the calibration curves.

The ARL iSpark 8860 is fully calibrated in our factory by using thoroughly tested and well accepted certified reference materials (CRM's) and reference materials (RM's). The calibration curves are established utilizing CARL (Calibration ARL), a very sophisticated multi-variable regression (MVR) software tool that corrects for matrix effects as well as spectral interferences and ensure the highest possible accuracy. The same MVR is included in OXSAS analytical software for on-site calibration.

Calibration summary

Seven calibrations are available for analysis of lead and lead alloy samples:

- Pure lead
- Low alloy lead
- Pb/Sn
- Pb/Sn/Sb
- Pb/Sb
- Pb/Ca
- Global lead

The pure lead and low alloy lead calibrations typically cover lead grades with Pb ≥ 99.5% and 95%, respectively. The alloy specific calibrations offer optimal accuracy for each class of alloys. The global lead calibration is an excellent solution to analyze most of the lead and lead alloy grades with a single calibration and is perfect for sorting.

Our calibrations are delivered as turn-key, fully parameterized applications. Setting-up samples (SUS) are delivered with the instrument to maintain the accuracy of the calibration. Special calibrations are also available upon request. Please contact your nearest Thermo Fisher Scientific office for more specific information of our calibrations. Based on the calibration curves and the measurements repeatability, the measurement uncertainty can be displayed for each sample. A dedicated product specification is available.

ARL iSpark 8860 - Detection limits and precision values for lead matrix

	Ag	Al	As	Au	Bi	Ca	Cd	Co	Cr	Cu	Fe	Ge	In	Mg	Ni	P	Pd	Pt	S	Sb	Se	Sn	Te	Tl	Zn
Typical DL [ppm]	0.004	0.01	0.3	0.2	0.01	0.02	0.006	0.05	0.08	0.006	0.2	0.025	0.03	0.002	0.01	2	0.01	0.2	0.5	0.2	0.7	0.2	0.35	0.02	0.1
Guaranteed DL [ppm]	0.008	0.025	0.5	0.3	0.03	0.05	0.01	0.1	0.2	0.015	0.4	0.05	0.05	0.005	0.02	3		0.3	1	0.5	1	0.4	0.5	0.03	0.2
Level [ppm]	**Precision [ppm]**																								
0.5	0.035														0.03									0.02	
1	0.055			0.1	0.015		0.045	0.15	0.2	0.073	0.35		0.04	0.06	0.05		0.08	0.13						0.03	
2	0.095	0.23	0.25	0.15	0.03		0.075	0.3	0.3	0.13	0.63	0.15	0.065	0.1	0.075		0.11	0.23	0.4	0.15		0.28	0.2	0.045	0.4
5	0.2	0.33	0.5	0.23	0.07	0.6	0.15	0.7	0.55	0.28	1.4	0.35	0.13	0.15	0.15	0.68	0.18	0.5	0.68	0.25	0.7	0.5	0.3	0.075	0.7
10	0.3	0.45	0.8	0.33	0.13	1	0.23	1.3	0.9	0.5	2.4	0.7	0.23	0.25	0.25	0.95	0.23	0.9	1	0.43	1.1	0.8	0.5	0.11	1.1
20	0.5	0.65	1.3	0.45	0.25	1.6	0.38	2.3		0.95	4.5	1.3	0.38	0.38	0.4	1.3	0.3	1.6	1.5	0.7	1.7	1.3	0.75	0.18	1.6
50	1	0.95	2.5	0.75	0.6	1.5	1.4	5.3		2	9.5	3	0.75	0.65	0.75	2.1	0.5	3.5	3.8	1.4	3	2.3	1.3	0.28	2.9
100	1.7	1.3	4	1.3	1	2	2.5	10		3.7	17	5.5	1.3		1.4	3	0.7	6.3	10	2.3	8	3.8	2.2	0.43	4.4
200	3.5	1.8	6.3	3.5	2	3.3	4.2	18		6.7	30	10	2.3		2.6	4	1	11	30	3.8	20	6	3.2		6.8
500	9	2.8	12	9	5	6.4	9			15	68	24	4.4		5.8		1.4	24		7.5	57	11	5.8		12
1000	16		19	18	9.5	10	16			27			7.5							13		18			18
Level [%]	**Precision [%]**																								
0.2	0.003		0.003	0.0037	0.002	0.0018	0.003			0.005			0.0013							0.0023		0.0028			0.003
0.3	0.0043		0.004	0.0055	0.0025		0.004			0.007			0.0018							0.0033		0.0038			
0.5	0.0068		0.006	0.0095	0.004		0.006			0.01										0.005		0.0053			
1	0.05		0.0095		0.008					0.02										0.009		0.0083			
2	0.25																			0.015		0.015			
3	0.35																			0.02		0.02			
4	0.5																			0.028		0.028			
5																				0.033		0.033			
10																				0.058		0.058			
20																				0.2		0.11			
30																				0.4		0.15			
40																						0.2			
50																						0.23			
60																						0.28			
70																						0.3			

Remarks:

- The precision values given in the table are typical instrumental repeatability. The guaranteed precision values are 1.5 times higher.
- The detection limits and the precision values are based on ten repeated measurements, typically.
- Guaranteed DLs are calculated at 95% confidence limit.
- The values are valid for all single matrix ARL iSpark instruments configured as recommended. For multi-matrix instruments, the performance may vary based on analytical line and grating.
- Homogeneity of the elements depends on the metallurgical structure obtained through the sampling procedure and on the metallurgical history including mechanical deformation by rolling. These values apply when homogeneously distributed elements are present in samples which are prepared by recommended sample preparation methods.
- A measured precision higher than the guaranteed precision indicates that the element is segregated or has an inhomogeneous distribution over the sample volume.

thermo scientific

Stability

Stability of the instrument is of the utmost importance when performing routine analysis. High stability reduces the frequency for maintenance and drift correction operations.

Mid-term stability tests over 2-5 days show typical standard deviation below two times the guaranteed precision value, which is excellent.

Memory effect

The memory effect is due to the contamination of the analytical stand after analysis of samples containing elements at relatively high concentration. Too high concentrations may be measured in the next sample if it has substantially lower concentration on these elements.

This memory effect is not significant when measuring a lead sample after other (unalloyed) lead sample(s) with higher element concentrations. For the sake of analytical reliability, 2-3 decontamination runs with a pure sample may however be performed before analyzing the purest samples.

When the instrument is also used for the analysis of lead alloys, the memory effect may be more pronounced. We recommend using a different set of analytical tables, electrodes and insulators for (pure) lead and for lead alloys.

Conclusion

The ARL iSpark 8860 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
- Potential to cover your future analytical needs
- Easy operation by unskilled worker or research chemist
- Widest range of metals analysis
- Advanced technical/service support.

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

- Your investment costs are reduced by:
  - Exceptional instrument lifetime and continuous upgrade possibilities (software and hardware)
  - Instrument capability to cover your future needs
- Your production costs are reduced by:
  - More accurate and reproducible analyses made available faster
  - Increased instrument availability thanks to its high stability and less frequently required drift corrections
- Your operating and maintenance costs are reduced by:
  - Low consumption of drift correction samples and simple maintenance
  - Significant argon savings during analysis and in stand-by
- Your overall cost management is reduced by:
  - Optimum utilization of materials
  - Extremely low running costs compared to other methods.

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
Korea +82 2 3420 8600
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

Find out more at **thermofisher.com/ispark**

ThermoFisher SCIENTIFIC

AN41258 0717