Optical Emission Spectrometry

Analysis of iron and steel with ARL iSpark 8860 Optical Emission Spectrometer

Since 1934, 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. Continuing this long tradition of excellence, the Thermo Scientific™ ARL iSpark™ 8860 Metal Analyzer is the trusted standard, which also integrates the latest innovations to provide our customers with the optical emission solution they need today.

The ARL iSpark 8860 Metal Analyzer will accurately and rapidly measure all the elements of interest to cover your current and future needs in the analysis of iron and steel: white or grey cast iron, alloyed cast iron, low alloy steel and high alloy steel. It is the answer to your analytical needs, whether for incoming materials control, process QC, final product QC, certification, or investigation. Working 24 hours a day and 7 days a week, the ARL iSpark 8860 Metal Analyzer delivers dependable performance year after year. Specific performance is detailed in this application note.

The trusted standard

The ARL iSpark 8860 is based on Thermo Scientific's most trusted one-meter focal length, vacuum purged, PMT spectrometer with Paschen-Runge mounting. The spectrometer offers optimal resolution and stability and ensures outstanding performance for all the elements.

Highly innovative features and technologies also characterize the instrument, including:

- Advanced signal acquisition and processing for optimal performance and accuracy
- Ultra-fast analysis of non-metallic micro-inclusions
- The Thermo Scientific™ intelliSource™, a digital spark source with increased flexibility and efficiency
- An analytical stand that reduces maintenance and minimizes argon consumption
- ECOmodes to save argon when the instrument is idle
- Maintenance management software tool for maximum instrument performance and reliability with minimum maintenance

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IntelliSource digital spark source

The intelliSource is a very innovative spark source for OES. More flexible than other digital sources, this double current controlled source (CCS) helps our application specialists design efficient spark current shapes for sample surface preparation, material ablation, and light emission in each metal matrix. Optimized pre-integration spark current shape minimizes the effects of both the matrix and metallurgical structure, by optimally re-melting and homogenizing the sample before integration of the signal, while perfectly adjusted integration spark current shapes deliver optimal performance on elements in trace amounts or at major alloying concentrations.

Single Spark Acquisition (SSA) and signal treatment

The analysis is performed by repeating very short “single sparks” at high frequency. The signals emitted during each of the single sparks, the “single spark signals”, are collected by PMTs and digitized individually. Special signal treatments may then be applied on the single spark signals to maximize the benefits of the ARL iSpark:

- DISIRE (DIffuse Spark Intensity REmoval) and FAST (Flexible Acquisition STart/Stop) to maximize performance and accuracy
- Spark-DAT algorithms to evaluate micro-inclusions and soluble and insoluble contents.

Time Gated Acquisition (TGA)

TGA is an improved version of TRS (Time Resolved Spectroscopy). Signal acquisition is performed during specific TGA windows, in other words during short time windows defined within single sparks. Start time and duration of the window are optimized for each analytical line to maximize the signal of interest and minimize the amount of noise and interferences collected. This results in better detection limits, precision values, and accuracy on every element.

Performance

Our company guarantees the precision values and the detection limits (DL) of the ARL iSpark 8860 presented in table 1.

The precision expresses the closeness of the concentration values of the individual runs of an analysis. The lower the precision value, the fewer analysis runs are needed for high confidence in the result.

The DL is the smallest concentration that can be distinguished from a blank value with a given probability. It is defined as three times the standard deviation of the background expressed in concentration units. For quantitative analysis however, it is the limit of quantification (LOQ ≈ 3·DL) that must be considered. The LOQ is the smallest concentration that can be measured quantitatively. When low concentration calibration standards are available, the LOQ sets the lowest value in our calibration menus.

Accuracy and factory calibration

Accuracy, the most important characteristic of an OES spectrometer, expresses the agreement between the analytical result and the reference value. It depends on the quality of the reference materials used for calibration and that of their certification, 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.

Each ARL iSpark 8860 Metal Analyzer is individually calibrated by hand in our factory. The calibrations are performed using CRM’s or thoroughly tested and well accepted reference materials. The calibration curves are established with a powerful multi-variable regression (MVR) software tool which corrects for matrix effects as well as spectral interferences and ensures the highest possible accuracy. The same MVR model is included in the Thermo Scientific™ OXSAS Analytical Software for on-site calibration.

The measurement uncertainty based on the calibration curve and the precision value can be displayed for each sample analyzed. A dedicated product specification (PS41282) is available.

Sample preparation

The sample is generally prepared by using a grinding machine (e.g. stone grinding for cast irons and paper with grit size 40 to 80 typically for steels). Milling machines are also recommended for some critical qualities or for advanced inclusion determinations, in order to avoid any residual contamination by the abrasive material of the grinding machine.

Analysis time

The analysis time taken between the start of an analysis run and the display of its result is in average the following:

Material analyzed	Time [s]
Steels with/without N, O, low C	21
Cast irons and global	23
Free-cutting steel	47

Note: the analysis time is not changed if soluble/insoluble or/and inclusion analysis is/are added to the elemental analysis method.

Table 1. ARL iSpark 8860 - Detection limits and precision values for iron and steel

ELEMENT	Al	As	B	Bi	C	*C	Ca	Ce	Co	Cr	**Cu	La
Typical DL [ppm]	1	0.95	0.25	1.4	3	0.5	0.2	4	1.1	1.5	7	0.2
Guaranteed DL [ppm]	1.3	1.3	0.4	1.8	5	1	0.4	5	1.3	2	10	1
Level	Precision (same unit as the concentration level)											
0.5 ppm												
1 ppm							0.3					
2 ppm			0.15			0.2	0.4					
5 ppm	0.6	0.3	0.2			0.3	0.5		0.2			0.7
10 ppm	0.6	0.5	0.3		0.6	0.4	0.6		0.3	0.5		0.7
20 ppm	1	0.6	0.4	1	0.8	0.6	1	1.5	0.4	0.8	0.4	1
50 ppm	1.8	0.8	0.6	1.5	1.5	1	2	2.5	0.6	1	0.7	2
100 ppm	2.8	1	0.8	2	2	1.6	3	4	1	1.5	1	3.5
200 ppm	4	1.6	1.2	3.5	4	3	5	5	1.3	2	2	5
500 ppm	7	3	2	6	7			10	2.2	4	4	
1000 ppm	10	4.5	2.6	9	10			70	3.5	5	5	
0.2%	0.002	0.0005			0.0014				0.0007	0.0008	0.001	
0.3%	0.0025				0.002				0.001	0.001	0.0015	
0.5%	0.004				0.003				0.0016	0.0015	0.0025	
1%	0.008				0.004				0.0035	0.0025	0.005	
2%	0.013				0.008				0.008	0.004	0.01	
3%					0.012				0.012	0.005	0.015	
4%					0.014				0.015	0.007	0.018	
5%					0.016				0.016	0.008	0.022	
10%									0.026	0.014	0.04	
20%									0.04	0.022		
30%										0.03		
40%										0.04		

ELEMENT	Mg	Mn	Mo	N	*N	**Nb	Ni	O	*O	P	Pb	S
Typical DL [ppm]	0.12	1.1	2	4.5	2.9	4.3	1.5	20	15	1	1.4	0.6
Guaranteed DL [ppm]	0.2	1.5	3	5	3.5	5	2	30	20	1.5	1.8	1
Level	Precision (same unit as the concentration level)											
0.5 ppm	0.06											
1 ppm	0.1											
2 ppm	0.2											
5 ppm	0.3										0.5	0.3
10 ppm	0.4	0.5	0.5		0.7	0.4	0.8			0.3	0.5	0.4
20 ppm	0.5	0.7	0.7	1.5	0.9	0.4	1			0.6	0.8	0.8
50 ppm	1.5	1	1.2	2	1.4	1.0	1.5		3	1	1.5	1.5
100 ppm	4	1.2	2	2.5	2	1.5	2	6	4	1.5	3	3
200 ppm	6	2	3	3.5	2.5	3	3	10	7	2	5	5
500 ppm	11	3	5	7	6	4	4	20	15	5	10	10
1000 ppm	20	5	8	12	10	8	5	35	25	9	20	20
0.2%	0.003	0.0008	0.0012	0.002	0.0016	0.0013	0.0007			0.0015	0.008	0.004
0.3%		0.001	0.0015	0.003	0.0022	0.0017	0.001			0.002	0.012	0.006
0.5%		0.002	0.002	0.004	0.0035	0.0025	0.0015			0.005	0.015	0.025
1%		0.003	0.004	0.006	0.005	0.004	0.0025			0.008		
2%		0.006	0.006			0.01	0.005			0.018		
3%		0.008	0.009			0.012	0.008					
4%		0.01	0.01			0.015	0.01					
5%		0.012	0.012				0.015					
10%		0.02	0.02				0.025					
20%		0.025					0.045					
30%		0.055					0.065					
40%							0.08					

ELEMENT	Sb	Se	Si	Sn	**Ta	Te	Ti	V	W	Zn	Zr
Typical DL [ppm]	3.8	4	1.9	1.4	15	1.9	0.3	1.5	5.8	0.2	0.8
Guaranteed DL [ppm]	5.5	4.5	3	2.5	18	2.5	0.6	2	9	0.8	1.5
Level	Precision (same unit as the concentration level)										
0.5 ppm											
1 ppm											
2 ppm											
5 ppm				0.4			0.2				
10 ppm	0.5		0.5	0.5		0.8	0.3			0.3	1
20 ppm	0.8	1.2	0.7	0.6		1	0.4	0.5		0.3	1
50 ppm	1.2	2	0.8	1	3	1.6	0.5	0.8	3	0.4	1.5
100 ppm	2	2.5	1	1.5	4	2.5	1.5	1.3	3	0.7	3
200 ppm	3	4	1.5	2.2	6	4	2.5	2	3	1.2	6
500 ppm	6	10	3	3.5	10	10	5	4	4	2	15
1000 ppm	8	20	8	5	20		10	7	6		25
0.2%	0.002		0.0015	0.001	0.0025		0.002	0.0014	0.001		0.004
0.3%	0.003		0.002	0.002	0.003		0.003	0.002	0.0015		0.006
0.5%			0.0025	0.0045	0.005		0.005	0.003	0.0025		
1%			0.005		0.01		0.01	0.005	0.005		
2%			0.008				0.02	0.009	0.01		
3%			0.012				0.03	0.012	0.015		
4%			0.015					0.015	0.02		
5%			0.035					0.017	0.023		
10%			**0.07**					**0.03**	**0.04**		
20%									**0.07**		
30%									**0.09**		

Remarks

- The DLs and the precision values are based on at least six repeated measurements.
- The guaranteed DLs are calculated at 95 % confidence limit.
- The precision values are typical. The guaranteed precision values are 1.5 times higher.
- The guaranteed precision values apply to the concentrations covered by our standard calibrations. Precision values for concentrations not covered by our standard calibrations are given for information only.
- The values are valid for an ARL iSpark configured as recommended. For multi-matrix instruments, the performance may vary based on analytical lines and grating.
- These values apply to samples prepared according to recommendation and when the distribution of the elements is homogeneous. Homogeneity depends on the metallurgical structure of the sample, influenced by its composition and the production process. Other factors also have influence, including quality of sampling in the liquid melt and mechanical deformation by rolling. A measured precision higher than the guaranteed precision indicates, with a probability higher than 95%, that the element is segregated or has an inhomogeneous distribution.
- The values given in table are for Ar 48 (99.998%) or higher purity.
- The columns *C, *N and *O give the improved performance obtained with the CNO option.
- **Cu: typical DL is 0.3ppm and guaranteed is 0.4ppm if the most sensitive line is present
- **Nb: DL is lower with an alternative line used only when analyzing steels with Ti ≤ 0.2%. With this line, the DL typical is 0.75ppm, and the DL guaranteed 1ppm.
- **Ta: DL is lower with an alternative line used only for low alloy steel. With this line, the DL typical is 8.5ppm and the DL guaranteed 10ppm.

Calibrations for iron and steel

The following calibrations are available:

- Low alloy steel
- Free cutting steel (with S and Pb up to 0.3%)
- Chrome steel (ferritic stainless steel)
- Chrome-nickel steel (austenitic stainless steel)
- Manganese steel (Mn up to 20%)
- High speed steel (Co up to 10% and W up to 24%)
- Cast iron – including nodular iron with or without Ni-hard (Ni <7%)
- High alloy cast iron (Cr up to 32% and Ni up to 16%)
- Nickel resist (cast iron with Ni up to 35%)
- Global iron (including all qualities except free cutting steel)

Low alloy, CrNi steel and Global calibrations are also available with small spot conditions for the analysis of small samples and wires.

Our calibrations are delivered as turnkey, fully parameterized applications. Setting-up samples (SUS) are delivered with the instrument to maintain the accuracy of the calibration. Please contact your nearest Thermo Fisher Scientific office for more specific information on our calibrations.

Soluble / insoluble contents, ultra-fast inclusion analysis and CNO option

Options are available for the evaluation of soluble and/or insoluble contents, and evaluation of non-metallic micro-inclusions in iron and steel samples with the ARL iSpark 8860. The data is obtained by processing the single spark signals with Spark-DAT (Spark Data Acquisition and Treatment) algorithms.

The following options are analytical methods performed in conjunction with the elemental analysis. Basic Inclusion Analysis and Standard Inclusion Analysis options can also be run as stand-alone methods.

Soluble and insoluble contents

This method is used to determine the concentration of the insoluble and/or soluble part/s of Al, B, Ti, Ca or any other element forming inclusions in low alloy steel.

Basic Inclusion Analysis

This method is the entry-level inclusion analysis method. It allows qualitative determination of number and size of the most common non-metallic inclusions. See the dedicated product specification (PS41356) for more details.

Standard Inclusion Analysis

The Standard Inclusion Analysis allows qualitative determination of number and size of most non-metallic inclusions. A dedicated application note (AN41243) is available.

Advanced Inclusion Analysis

The Advanced Inclusion Analysis offers additional features to those of the Standard Inclusion Analysis, e.g., quantitative inclusions size/size distribution in micrometer, inclusions volume fraction and concentration. Determination of oxygen down to a few ppm in killed steels is possible under some conditions. A dedicated application note (AN41244) is available.

CNO option

The CNO option allows the determination of C, N and O at ultra-low levels, so that utilization of alternative analytical method like combustion analysis can be eliminated or reduced to a minimum (see detection limits and precision table for specifications).

Stability

Stability of the instrument is of the utmost importance when performing routine analysis. High stability reduces the frequency of maintenance and drift correction operations. Standard deviation of mid-term stability is typically less than three times the short-term precision value at the measured concentration.

Memory effect

The memory effect is due to the contamination of the analytical stand after analysis of samples containing elements at relatively high concentration. Artificially high concentrations may be measured for some elements in the subsequent samples. Depending on the concentration of the alloying elements, up to six conditioning runs may be necessary to eliminate contamination.

When the instrument is used for the analysis of low and high alloy steels, the memory effect may be more pronounced. We recommend using different sets of analytical table, electrode, and insulator for low alloy steel and for high alloy steels.

Conclusion

The ARL iSpark 8860 Metal Analyzer provides not only state-of-the-art technology, but also has all the total system features to meet the critical needs of the metal markets:

- Unmatched hardware stability and reliability
- Exceptional performance in detection limits, precision and accuracy with minimal analysis time
- Individual true calibration
- Advanced software technology
- Easy operation
- Widest range of metals analysis
- Analysis of non-metallic micro-inclusions
- Automation solutions with ARL SMS products
- Advanced technical/service support

All these features allow you to optimize your productivity and 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 in the shortest possible time
  - Increased instrument availability due to high stability and low frequency of drift correction
- Your operating and maintenance costs are reduced by:
  - Low consumption of drift correction samples and simple maintenance
  - Significant argon savings during and between analyses
- Your overall cost management is reduced by:
  - Optimum utilization of materials
  - Extremely low running costs compared to other methods.

Learn more at **thermofisher.com/ispark**