APPLICATION NOTE No. 41242

Analysis of iron and steel with Thermo Scientific ARL iSpark 8860 optical emission spectrometer

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Introduction

For over 85 years, our company has set the standard of quality for spectrochemical analysis of metals. Throughout these years, 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 Metals Analyzer will determine all the elements necessary in your current and future applications, in all possible qualities 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 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 note.

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 even for all the critical trace 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.

ARL iSpark 8860 – Detection limits and precision values for iron matrix

Table 1.

Element	Al	As	B	Bi	C	C*	Ca	Ce	Co	Cr	Cu	La
Typical DL	1	0.9	0.2	1.4	3	0.6	0.2	2	1.1	1.6	0.25	0.6
Guaranteed DL	1.3	1.3	0.4	1.8	5	1	0.3	3	1.3	2	0.4	1
Level %	Precision											
0.001	0.00006	0.00005	0.00003	0.00006	0.00006	0.00004	0.00006	0.00012	0.00003	0.00005	0.00003	0.00007
0.002	0.0001	0.00006	0.00004	0.0001	0.00008	0.00006	0.0001	0.00015	0.00004	0.00008	0.00004	0.0001
0.005	0.00018	0.00008	0.00006	0.00015	0.00015	0.0001	0.0002	0.00025	0.00006	0.0001	0.00007	0.0002
0.01	0.00028	0.0001	0.00008	0.0002	0.0002	0.00016	0.0003	0.0004	0.0001	0.00015	0.0001	0.00035
0.02	0.0004	0.00016	0.00012	0.00035	0.0004		0.0005	0.0005	0.00013	0.0002	0.0002	0.0005
0.05	0.0007	0.0003	0.0002	0.0006	0.0007			0.001	0.00022	0.0004	0.0004	0.001
0.1	0.001	0.00045	0.00026	0.0009	0.001			0.007	0.00035	0.0005	0.0005	
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		
20									0.04	0.022		
30										0.03		
40										0.04		

Table 2.

Element	Mg	Mn	Mo	N	N*	Nb*	Ni	O	O*	P	Pb	S
Typical DL	0.1	0.5	1.5	3	3	3.5	1.5	20	15	0.6	1.4	0.5
Guaranteed DL	0.2	1	2	4.5	3.5	5	2	30	20	1	1.8	1
Level %	**Precision**											
0.001	0.00004	0.00005	0.00005	0.00012	0.00007	0.00004	0.00008			0.00003	0.00005	0.00004
0.002	0.00005	0.00007	0.00007	0.00015	0.00009	0.00004	0.0001			0.00006	0.00008	0.00008
0.005	0.00015	0.0001	0.00012	0.0002	0.00014	0.0001	0.00015		0.0003	0.0001	0.00015	0.00015
0.01	0.0004	0.00012	0.0002	0.00025	0.0002	0.00015	0.0002	0.0006	0.0004	0.00015	0.0003	0.0003
0.02	0.0006	0.0002	0.0003	0.00035	0.00025	0.0003	0.0003	0.001	0.0007	0.0002	0.0005	0.0005
0.05	0.0011	0.0003	0.0005	0.0007	0.0006	0.0004	0.0004	0.002	0.0015	0.0005	0.001	0.001
0.1	0.002	0.0005	0.0008	0.0012	0.001	0.0008	0.0005			0.0009	0.002	0.002
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		
1		0.003	0.004	0.006	0.005	0.004	0.0025			0.008		
2		0.006	0.006			0.01	0.005					
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							0.065					
40							0.08					

Table 3.

Element	Sb	Se	Si	Sn	Ta*	Te	Ti	V	W	Zn	Zr
Typical DL	2	2.3	2	1.1	14	1.3	0.4	1.5	5	0.2	1
Guaranteed DL	3.5	3	3	1.5	18	2	0.6	2	8	0.8	1.5
Level %	**Precision**										
0.001	0.00005	0.00009	0.00005	0.00005		0.00008	0.00003	0.00004		0.00003	0.0001
0.002	0.00008	0.00012	0.00007	0.00006	0.0002	0.0001	0.00004	0.00005	0.0003	0.00003	0.0001
0.005	0.00012	0.0002	0.00008	0.0001	0.0003	0.00016	0.00005	0.00008	0.0003	0.00004	0.00015
0.01	0.0002	0.00025	0.0001	0.00015	0.0004	0.00025	0.00015	0.00013	0.0003	0.00007	0.0003
0.02	0.0003	0.0004	0.00015	0.00022	0.0006	0.0004	0.00025	0.0002	0.0003	0.00012	0.0006
0.05	0.0006	0.001	0.0003	0.00035	0.001	0.001	0.0005	0.0004	0.0004	0.0002	0.0015
0.1	0.0008	0.002	0.0008	0.0005	0.002		0.001	0.0007	0.0006	0.0003	0.0025
0.2	0.002	0.0035	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				0.005	0.003	0.0025		0.009
1			0.005				0.01	0.005	0.005		
2			0.008				0.02	0.009	0.01		
3			0.012					0.012	0.015		
4			0.015					0.015	0.02		
5								0.017	0.023		
10								0.03	0.04		
20									0.07		
30									0.09		
40											

- The precision values (given in percent) are typical instrumental repeatability. The guaranteed precision values are 1.5 times higher
- The detection limits (given in ppm) and the precision values are based on ten repeated measurements typically
- Guaranteed DLs are calculated at 95% confidence limit
- The columns C*, N* and O* give the improved performance obtained with the CNO option
- Nb*: DL for Nb can be lower with a line to be used only for alloyed steels with Ti max 0.2%. DL typical 0.75ppm, DL guaranteed 1.5ppm
- Ta*: DL of Ta can be lower with a line that can be used only for low alloy steel. DL typical 0.75ppm, DL guaranteed 1.5ppm
- The values are valid for all single-matrix ARL iSpark instruments configured as recommended. For multibase instruments, the performance may vary based on analytical line and grating used
- Homogeneity of the elements depends on the metallurgical structure obtained through the sampling procedure (cast, forged or rolled) and on the metallurgical history including mechanical deformation by forging or rolling and heat treatments. 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, with a probability higher than 95%, that the element is segregated or has an inhomogeneous distribution over the sample's surface

IntelliSource digital spark source

The Thermo Scientific™ intelliSource is the most innovative excitation current controlled source (CCS) 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, like 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 and better precision values. 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 precision by discarding abnormally low intensity signals

Spark-DAT methods

SSA (Single Spark Acquisition) allows acquiring individually the single spark signals processed by Spark-DAT (Spark Data Acquisition and Treatment) algorithms in order to determine the soluble/insoluble fraction of an element or characterize non-metallic inclusions.

The Spark-DAT methods are extremely fast – they run in parallel with the elemental analysis – and can therefore be used to control inclusions in production.

Sample preparation

The sample is generally prepared by using a grinding machine (e.g. stone grinding for cast irons and paper 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.

Sample analysis time

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

Table 4.

Material analyzed / type of analysis	Time [s]
Steels (without N, O)	17
Steels (with N, O)	21
Cast iron and high alloy cast iron	24
Free-cutting steel	53

Notes: Insoluble and inclusion analysis methods do not change the time.

Performance guarantee

Our company guarantees the precision and the detection limit (DL) as shown on page 2.

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. It is defined as three times the standard deviation of the background expressed in concentration units. 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 availability allows calibrating down to this point.

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 individually calibrated in our factory. The calibrations are performed 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 ensures the highest possible accuracy. The same MVR model is included in OXSAS analytical software for on-site calibration.

Calibration summary

The following calibrations are available for cast irons and steels:

- 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 available with small analysis spot. Our calibrations are true calibrations, delivered as turn-key, 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.

Based on the calibration curves and the repeatability of the measurements, the measurement uncertainty can be displayed for each sample. A specific Product Specification is available.

Analytical options

The following options are analytical methods performed during – and without additional time – the standard analysis in concentration. The Basic Inclusion Analysis and Standard Inclusion Analysis options can also be run as stand-alone methods.

Insoluble

The Insoluble Spark-DAT method is used to determine the concentration of the insoluble or soluble fraction of Al, B, Ti, Ca or any other element forming inclusions in low alloy steel.

Basic inclusion analysis

This Spark-DAT method is the entry-level inclusion analysis method, allowing qualitative determinations of the most common non-metallic inclusions in terms of number and size. See PS41356 for more details.

Standard inclusion analysis

This Spark-DAT method (application note AN41243 available) allows qualitative determinations of most non-metallic inclusions in terms of number and size.

Advanced inclusion analysis

The Advanced Inclusion Analysis Spark-DAT method (application note AN41244 available) offers additional features compared to the Standard Inclusion Analysis method, e.g. quantitative inclusion size determination, and determination of oxygen down to a few ppm in killed steels.

CNO

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 for maintenance and drift correction operations.

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

Memory effects

The memory effect is defined as the number of runs necessary to reduce the apparent concentrations of elements after measuring high alloyed samples, such as stainless steels. For the ARL iSpark 8860 the electrode is completely cleaned and the instrument will measure very low concentration of the alloying elements after 5 runs.

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 scientist
- Widest range of metals analysis
- Ultra-fast analysis of micro-inclusions
- Adaptable to the automatic sample manipulation systems ARL SMS-2500 and ARL SMS-3500
- 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

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

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