Application note | 41257

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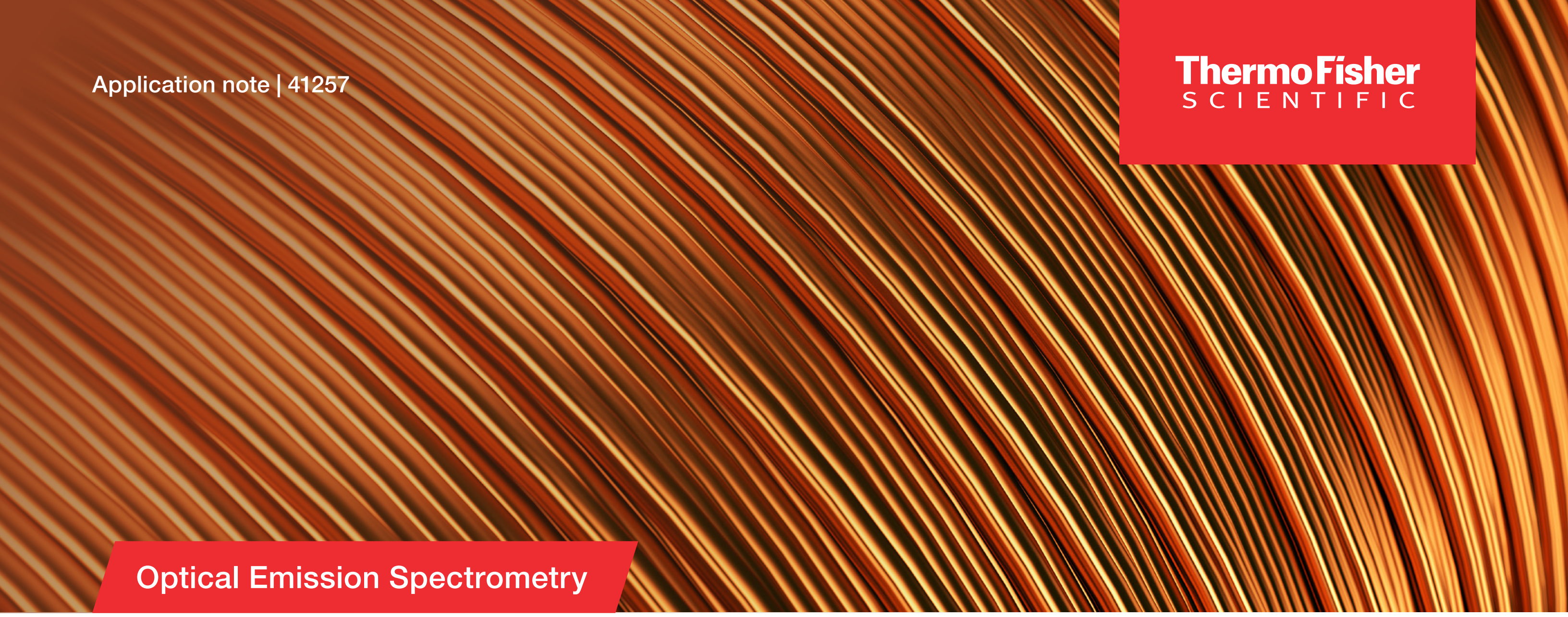

Optical Emission Spectrometry

Analysis of copper 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 all possible copper grades. 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
- 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

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.

Sample preparation

The sample surface is generally prepared by using a lathe or a milling machine. Grinding is not recommended because of risk of contamination.

Analysis time

The analysis time taken between the start of an analysis run and the display of its result is in average 25 s, with or without determination of the oxygen content.

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 certificate, 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.

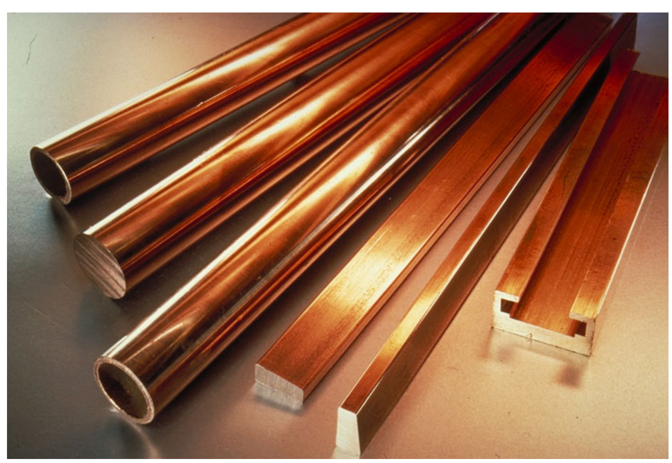

Table 1. ARL iSpark 8860 - Detection limits and precision values for copper

	Ag	Al	As	Au	B	Be	Bi	Cd	Co	Cr	Fe	Mg	Mn	Ni	O
Typical DL Pure Cu [ppm]	0.15	0.2	0.3	0.3	0.1	0.05	0.3	0.15	0.6	0.4	0.4	0.05	0.05	0.2	8
Guaranteed DL Pure Cu [ppm]	0.3	0.4	0.5	0.5	0.2	0.1	0.6	0.3	1	0.6	0.6	0.1	0.1	0.4	15
Typical DL ultra-pure Cu [ppm]	0.1	0.1	0.25	0.1	0.03	0.005	0.2	0.04	0.1	0.08	0.15	0.01	0.02	0.1	4.5
Guaranteed DL ultra-pure Cu [ppm]	0.15	0.2	0.35	0.15	0.06	0.01	0.5	0.1	0.25	0.15	0.2	0.03	0.03	0.2	8
Level [ppm]	**Precision (same unit as the concentration level)**														
0.5								0.03		0.02		0.03	0.04		
1	0.06	0.07	0.09	0.06	0.06	0.02	0.06	0.04	0.035	0.03		0.04	0.06	0.04	
2	0.09	0.1	0.12	0.08	0.11	0.035	0.1	0.06	0.06	0.06	0.1	0.075	0.09	0.06	
5	0.15	0.15	0.17	0.11	0.22	0.075	0.18	0.11	0.11	0.12	0.15	0.17	0.15	0.09	
10	0.25	0.2	0.22	0.17	0.37	0.13	0.3	0.17	0.18	0.2	0.2	0.3	0.25	0.12	
20	0.35	0.4	0.3	0.35	0.65	0.25	0.45	0.25	0.3	0.35	0.3	0.6	0.4	0.17	3.2
50	0.6	0.9	0.5	0.7	1.3	0.5	1.2	0.8	0.55	0.8	0.5	1.3	0.7	0.25	5.3
100	1.4	1.5	1.5	1.2	2.2	0.9	2.9	1.9	1.2	1.4	1	2.9	1	0.35	8
200	2.5		2.8				5.2	3.5	2.6		2.3	7.6	2	0.7	11
500	5.3		6.9					7.5	5.4		5.4	20	4	1.7	20
1000	9.4		13						9.5		8.5			2.8	30
2000															40
3000															50
5000															70

	P	Pb	Pd	Pt	Rh	S	Sb	Se	Si	Sn	Te	Ti	Zn	Zr
Typical DL Pure Cu [ppm]	0.2	0.5	0.05	0.8	0.3	0.2	1	0.2	0.2	0.3	1	0.15	1	0.15
Guaranteed DL Pure Cu [ppm]	0.4	1	0.1	1.5	0.6	0.4	2	0.4	0.5	0.6	2	0.3	1.5	0.3
Typical DL ultra-pure Cu [ppm]	0.1	0.2	0.03	0.6	0.1	0.1	0.4	0.1	0.2	0.2	1	0.1	0.2	0.1
Guaranteed DL ultra-pure Cu [ppm]	0.2	0.3	0.05	1	0.3	0.2	0.6	0.2	0.5	0.4	1.5	0.15	0.35	0.15
Level [ppm]	**Precision (same unit as the concentration level)**													
0.5	0.04													
1	0.06	0.15				0.04		0.06		0.06		0.03	0.055	0.11
2	0.09	0.2				0.07	0.25	0.09	0.15	0.09		0.045	0.08	0.17
5	0.15	0.3	0.83	0.3	0.1	0.15	0.35	0.15	0.27	0.15	0.38	0.09	0.12	0.3
10	0.25	0.4	0.25	0.5	0.18	0.25	0.45	0.23	0.4	0.22	0.53	0.15	0.17	0.45
20	0.4	0.5	0.4	0.7	0.33	0.45	0.55	0.45	0.65	0.32	0.8	0.25	0.25	0.7
50	1	1.2	0.9	1.3	0.73	1	0.75	1.1	1	0.55	1.8	0.5	0.7	1.1
100	3	2.2	1.4	2	1.3	1.7	1.5	2.1	1.5	1	3	0.83	1.3	1.7
200	5	4.1	2.4	3.1	2.4	3	3.9	4	2.2	2.8	5.1		2.4	2.6
500	8.5	9.4	4.8	5.6	5.2		11	9.5	5	6.6	10		5.5	
1000	12	24	8.1	8.7	9.3					18			10	
2000													19	

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 with the recommended method 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 are obtained with Ar 48 (99.998%) or higher purity.

Calibrations for copper

The following calibrations are available:

- Ultra-pure copper
- Pure copper

The two calibrations cover the same elements and have the same upper calibration range values. The ultra-pure copper offers lower calibration range values and is suitable for the purest copper grades, including BS EN 1978:1988 Copper Grade A and ASTM B115 – 10 Grade 1.

Please note that the following calibrations are also available for the analysis of copper and its alloys: low alloy copper, brass, bronze, Cu-Al, Cu-Ni, Cu-Zn-Ni, Gunmetal, Cu-Co-Be-Ag, general sorting (including all copper alloy qualities). A dedicated application note (AN41251) is available.

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.

Ultra-fast inclusion analysis

The Standard Inclusion Analysis option is available for the evaluation of non-metallic micro-inclusions in copper and its alloys. The inclusion data is obtained by processing the signal spark signals with Spark-DAT (Spark Data Acquisition and Treatment) algorithms. Inclusion analysis can be performed as stand-alone method or is preferably combined with the elemental analysis.

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 concentration, two to three conditioning runs may be necessary to eliminate contamination.

When the instrument is also used for the analysis of copper alloys, the memory effect may be more pronounced. We recommend using different sets of analytical table, electrode, and insulator for (pure) copper and for copper.

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**

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