

Optical Emission Spectrometry

Analysis of fine platinum and fine palladium 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 for fine platinum and fine palladium analysis. 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, and analysis on LBMA reference samples are shown.

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



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 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 24 s per run for fine platinum and fine palladium analysis.

Performance

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.

Precision is demonstrated with 4-run analyses of LPPM (London Platinum and Palladium Market) fine platinum and palladium certified reference materials (see tables 1, 3, 5 and 7).

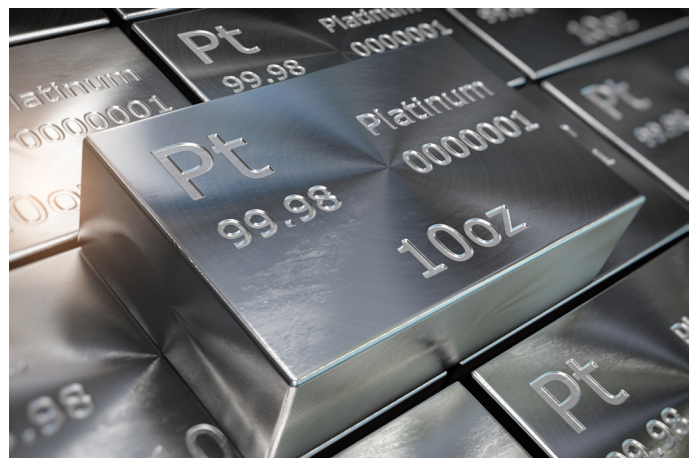
Accuracy and factory calibration

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.

Accuracy is demonstrated with 4-run analyses of LPPM (London Platinum and Palladium Market) fine platinum and palladium certified reference materials (see tables 2, 4, 6 and 8).

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.



Analysis of LPPM fine platinum certified reference materials

Table 1. Sample LPPM PtRM1 - Precision

El.	Average conc. [ppm]	SD [ppm]	RSD
Au	10.9	0.13	1.2
Ag	14.5	0.07	0.5
Pd	10.9	0.09	0.8
Rh	14.6	0.11	0.8
Ir	192.6	0.99	0.5
Ru	10.9	0.12	1.1
Al *	99.9	0.37	0.4
As	50.7	0.67	1.3
Cr	8.9	0.08	0.9
Cu	26.8	0.20	0.7
Fe	12.4	0.12	1.0
Ni	11.8	0.11	0.9
Pb	11.1	0.13	1.2
Sb	17.3	0.63	3.7
Si	47.4	0.56	1.2
Sn	39.5	0.38	1.0
Tl	5.5	0.11	1.9
Zn	46.6	0.26	0.6
Zr	1.0	0.04	4.1

Table 2. Sample LPPM PtRM1 - Accuracy

El.	Measured	Certified		Accuracy	
	Average conc. [ppm]	Conc. [ppm]	U [ppm]	Δ [ppm]	$ \Delta / U $
Au	10.9	11.1	2.4	-0.2	0.1
Ag	14.5	13.9	1.3	0.6	0.5
Pd	10.9	10.5	1.3	0.4	0.3
Rh	14.6	13.7	2.4	0.9	0.4
Ir	192.6	204	17	-11.4	0.7
Ru	10.9	11.4	1.4	-0.5	0.4
Al *	99.9	93.9	7.1	6.0	0.8
As	50.7	58.2	5.4	-7.5	1.4
Cr	8.9	9.3	0.5	-0.4	0.8
Cu	26.8	26.1	1.5	0.7	0.5
Fe	12.4	12.4	1.8	0.0	0.0
Ni	11.8	11.1	2.3	0.7	0.3
Pb	11.1	12.3	3	-1.2	0.4
Sb	17.3	19.5	2.3	-2.2	1.0
Si	47.4	47.5	4.9	-0.1	0.0
Sn	39.5	47.6	5	-8.1	1.6
Tl	5.5	6.2	0.8	-0.7	0.8
Zn	46.6	44.9	2.2	1.7	0.8
Zr	1.0	1.2	0.2	-0.2	1.0

Table 3. Sample LPPM PtRM2 - Precision

El.	Average conc. [ppm]	SD [ppm]	RSD
Au	49.8	0.11	0.2
Ag	46.3	0.28	0.6
Pd	95.6	0.55	0.6
Rh	102.8	0.61	0.6
Ir	58.3	0.54	0.9
Ru	50.6	0.41	0.8
Al *	36.0	0.09	0.3
As	25.6	0.41	1.6
Bi	16.7	0.41	2.4
Co	18.9	0.18	1.0
Cr	47.4	0.24	0.5
Cu	90.1	0.40	0.4
Fe	85.7	0.44	0.5
Ni	41.1	0.13	0.3
Pb	31.0	0.71	2.3
Sb	44.6	0.49	1.1
Si	13.7	0.17	1.2
Sn	6.6	0.42	6.3
Ti	12.4	0.02	0.2
Tl	36.9	0.67	1.8
W	23.7	0.32	1.3
Zn	9.7	0.07	0.8
Zr	36.4	0.12	0.3

Table 4. Sample LPPM PtRM2 - Accuracy

El.	Measured	Certified		Accuracy	
	Average conc. [ppm]	Conc. [ppm]	U [ppm]	Δ [ppm]	$ \Delta / U $
Au	49.8	51.9	6	-2.1	0.3
Ag	46.3	44.8	4.5	1.5	0.3
Pd	95.6	95.3	5.9	0.3	0.0
Rh	102.8	101.6	5.1	1.2	0.2
Ir	58.3	59.2	7	-0.9	0.1
Ru	50.6	51.5	3.9	-0.9	0.2
Al *	36.0	34.7	4.4	1.3	0.3
As	25.6	28.7	6	-3.1	0.5
Bi	16.7	18.6	2	-1.9	1.0
Co	18.9	19.5	1.4	-0.6	0.4
Cr	47.4	47.2	1.7	0.2	0.1
Cu	90.1	90.8	5.4	-0.7	0.1
Fe	85.7	85.4	7.9	0.3	0.0
Ni	41.1	41.4	3.6	-0.3	0.1
Pb	31.0	33.3	3.7	-2.3	0.6
Sb	44.6	49.9	6	-5.3	0.9
Si	13.7	13.1	3.7	0.6	0.2
Sn	6.6	7	3.1	-0.4	0.1
Ti	12.4	11.8	0.8	0.6	0.8
Tl	36.9	39.2	6.1	-2.3	0.4
W	23.7	20.2	1.5	3.5	2.4
Zn	9.7	9.5	0.5	0.2	0.5
Zr	36.4	36.5	3.8	-0.1	0.0

* with extended calibration range

Notes:

- U = Uncertainty; Δ = (measured concentration - certified concentration); Δ / U = accuracy ratio
- The accuracy is outstanding when parameter $|\Delta / U|$ is less than 1 and good when it is less than 3.

Analysis of LPPM fine palladium certified reference materials

Table 5. Sample LPPM PdRM1 - Precision

El.	Average conc. [ppm]	SD [ppm]	RSD
Rh	53.4	0.38	0.7
Pt	52.3	0.29	0.6
Ir	9.2	0.18	2.0
Ru	10.1	0.09	0.9
Au	99.3	0.55	0.6
Ag	11.2	0.07	0.6
Al	12.3	0.15	1.2
B	2.5	0.28	11.4
Co	8.9	0.14	1.5
Cr	47.2	0.17	0.4
Cu	11.1	0.07	0.6
Fe	10.1	0.43	4.3
Mg	24.1	0.24	1.0
Mn	23.3	0.12	0.5
Ni	53.0	0.36	0.7
Pb	12.6	0.13	1.1
Sb	8.5	0.23	2.8
Si	53.8	1.42	2.6
Sn	12.7	0.15	1.2
Zn	9.7	0.08	0.8

Table 6. Sample LPPM PdRM1 - Accuracy

El.	Measured	Certified		Accuracy	
	Average conc. [ppm]	Conc. [ppm]	U [ppm]	Δ [ppm]	$ \Delta / U $
Rh	53.4	56.3	3.4	-2.9	0.8
Pt	52.3	54.4	2.9	-2.1	0.7
Ir	9.2	9.7	2.5	-0.5	0.2
Ru	10.1	10.3	0.9	-0.2	0.3
Au	99.3	99.2	5.7	0.1	0.0
Ag	11.2	11.1	2.3	0.1	0.0
Al	12.3	12.4	1.9	-0.1	0.1
B	2.5	2.5	0.6	0.0	0.0
Co	8.9	8.8	0.8	0.1	0.1
Cr	47.2	45.5	1.9	1.7	0.9
Cu	11.1	11	2	0.1	0.0
Fe	10.1	11.1	1.9	-1.0	0.6
Mg	24.1	22.3	0.9	1.8	2.0
Mn	23.3	23.8	1.2	-0.5	0.4
Ni	53.0	54.1	2.9	-1.1	0.4
Pb	12.6	13.2	2.2	-0.6	0.3
Sb	8.5	9.2	1.1	-0.7	0.7
Si	53.8	50	11	3.8	0.3
Sn	12.7	12.3	1.9	0.4	0.2
Zn	9.7	9.5	1	0.2	0.2

Table 7. Sample LPPM PdRM2 - Precision

El.	Average conc. [ppm]	SD [ppm]	RSD
Rh	12.7	0.12	0.9
Pt	224.7	0.82	0.4
Ir	39.0	0.41	1.1
Ru	43.9	0.25	0.6
Au	16.0	0.08	0.5
Ag	98.3	0.26	0.3
Al	5.0	0.13	2.5
Ca *	7.7	0.24	3.1
Co	16.7	0.15	0.9
Cr	9.7	0.71	7.3
Cu	92.3	0.13	0.1
Fe	45.4	2.66	5.9
Ga *	18.4	0.09	0.5
Ni	10.4	0.45	4.3
P	27.3	0.51	1.9
Pb	62.8	0.08	0.1
Sb	42.3	0.42	1.0
Si	126.3	1.71	1.4
Sn	43.4	0.30	0.7
Zn	18.2	0.01	0.1

Table 8. Sample LPPM PdRM2 - Accuracy

El.	Measured	Certified		Accuracy	
	Average conc. [ppm]	Conc. [ppm]	U [ppm]	Δ [ppm]	$ \Delta / U $
Rh	12.7	13.3	1.2	-0.6	0.5
Pt	224.7	228	13	-3.3	0.3
Ir	39.0	43.4	5.7	-4.4	0.8
Ru	43.9	45.1	3.4	-1.2	0.4
Au	16.0	16	2.3	0.0	0.0
Ag	98.3	95.5	7.9	2.8	0.4
Al	5.0	5	1.3	0.0	0.0
Ca *	7.7	9.5	2	-1.8	0.9
Co	16.7	17.5	1.1	-0.8	0.8
Cr	9.7	9.3	1.5	0.4	0.3
Cu	92.3	92.1	4.4	0.2	0.1
Fe	45.4	45.1	2.8	0.3	0.1
Ga *	18.4	18.6	2.3	-0.2	0.1
Ni	10.4	10.1	0.9	0.3	0.4
P	27.3	20.1	4	7.2	1.8
Pb	62.8	66	5	-3.2	0.6
Sb	42.3	42.8	5	-0.5	0.1
Si	126.3	119	29	7.3	0.3
Sn	43.4	44.4	5.2	-1.0	0.2
Zn	18.2	17.4	1.4	0.8	0.5

* with extended calibration range

Notes:

- U = Uncertainty; Δ = (measured concentration - certified concentration); Δ / U = accuracy ratio
- The accuracy is outstanding when parameter $|\Delta / U|$ is less than 1 and good when it is less than 3.

Fine platinum and fine palladium calibrations

The standard calibrations that we propose for fine platinum and fine palladium include 30 and 29 elements, respectively. They comply with the chemical requirements of ASTM B5689-94 (2017) Standard Specification for Refined Platinum and ASTM B561-94 (2012) Standard Specification for Refined Palladium for the common elements and most of the others. More elements and extended calibration ranges can be offered on demand.

Our calibration is 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 fine platinum or palladium 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 Standard Inclusion Analysis is a method that allows qualitative determination of number and size of most non-metallic inclusions. The inclusion analysis is preferably performed in conjunction with the elemental analysis, but it can also be performed stand-alone.

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

Due to low energy of the analytical condition and pureness of material analyzed, the memory effect is negligible.

Superposition of runs

It is possible to measure up to four times without moving the sample, i.e., by superimposing burns, and without cleaning the electrode. This allows for quick quantitative analysis on precious samples that are often small, without introducing analysis bias.

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.

