

Analysis of Iron and Steel by Optical Emission

ARL 4460 Metals Analyzer

Key Words

- ARL 4460
- Optical Emission
- Metals Analyzer
- Iron & Steel

Introduction

Significant progress has been made in the analysis of iron and steel. Sample analysis time, sensitivity, precision and accuracy have all been substantially improved with the Thermo Scientific ARL 4460 Metals Analyzer.

Our long experience in metals analysis comes from an installed base of over 10'000 spectrometers worldwide. The ARL 4460 is the answer to your metallurgical analysis needs; whether they be incoming material control or metal QC and production analysis. Working 24 hours a day and 7 days a week, the ARL 4460 delivers dependable performance year after year.



The ARL 4460 Metals Analyzer can determine up to 60 elements simultaneously in about 50 seconds. Specific performance is detailed in this application summary.

Cast irons

From a metallurgical point of view, a distinction can be made between white cast iron and grey cast iron which contains free graphite. In alloyed cast irons, alloying elements such as Ni, Cr, Mn, Cu, etc. are added to improve hardness, corrosion resistance or engineering properties (Ni-hard, Ni-resist, etc.).

Steels

Typically steels are divided in two main qualities:

1. Low alloy steels (≥ 94 % iron)
2. High alloy steels (with notable quantities of Cr, Ni, Mn, Mo, Co, W, etc.)

1. Low alloy steels

The total content of the alloying elements is generally less than 5-7%. The main elements are C (≤ 1.5 %), Si (≤ 3 %), Mn (≤ 2 %), Cr (≤ 3 %), Mo (≤ 1.5 %), Cu (≤ 1 %), Ni (≤ 5 %), and V (≤ 1 %). The so-called "free-cutting steels" with a concentration of 0.2 - 0.3 % S or Pb, need a special calibration and a specific source condition.

2. High alloy steels

High alloy steels contain, in addition to iron and carbon, notable quantities of one or more of the following elements: Ni, Cr, Mn, Si, Co, W, Mo and V.

This category covers steels which are intended for a wide variety of uses such as:

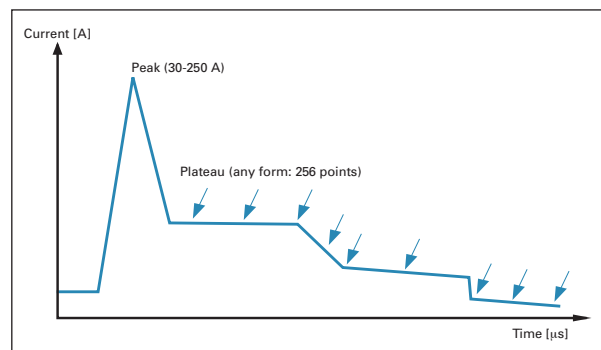
- Stainless steels
- Tool steels
- High manganese steels
- High speed steels
- Special high alloys steels, such as Ni-hard, Ni-resist, etc.

Current Controlled Source (CCS)

The Thermo Scientific Current Controlled Source presents significant advantages in comparison to any other spark generator currently used for OES. It is the only servo-controlled "digital source" on the market.

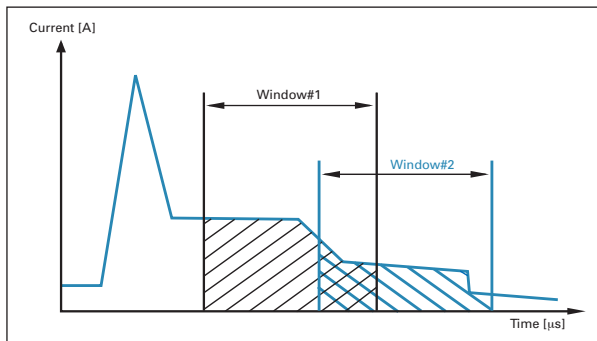
The current waveform is computer controlled and can be selected for each type of metal. The high degree of flexibility in selection of peak current (250 A max.), frequency (1000 Hz max.) and current waveforms enables the optimization of all analytical figures.

The very compact design close to the spark stand, in a Faraday cage, suppresses RF leakage and improves general stability (less current lost in connecting cables).



Time Resolved Spectroscopy (TRS)

In TRS mode, the acquisition takes place during a well-defined time window (“TRS window”) of each individual spark. The optimization of the start and the duration of the window make it possible to maximize the signal to noise ratio (e.g. in avoiding acquisition during the initial peak current causing high background), which results in lower detection limits and better precision. They also make it possible in certain cases to reduce interferences between atomic/low potential lines and ionic/high potential lines, improving thus accuracy.



Sample preparation

The sample is generally prepared by using a grinding machine. Milling machines are also recommended.

Sample analysis time

The analysis time is taken between the start of the analysis and the display of its result.

MATERIAL	CONVENTIONAL EXCITATION	ARL 4460 EXCITATION
Steels (except free cutting steels)	27 s	22 s
High- and low-alloyed cast irons (except grey cast irons)	31 s	22 s
Free cutting steels	68 s	57 s
Grey cast irons	50-120 s	50-95 s

5 to 11 seconds can be saved in an environment where the analysis time is critical.

Calibration summary

Numerous qualities are available for specific types of alloys, including:

- Low Alloy Steel
- Free Cutting Steel (with S and Pb \leq 0.3 %)
- Chrome Steel (ferritic stainless steel)
- Chrome-Nickel Steel (austenitic stainless steel)
- Manganese Steel (Mn \leq 20 %)
- High Speed Steel (Co \leq 10 %)
- Cast Iron (including nodular iron and Ni-hard - Ni \leq 7 %)
- High Alloy Cast Iron (Cr \leq 32 % and Ni \leq 16 %)
- Nickel Resist (Ni \leq 35 %)
- Global Iron (including all qualities except for free cutting steel)

- Global Iron "Small Samples" (including all qualities except for free cutting steel)
- Low Alloy Steel and Free Cutting Steel Small Samples

The global calibration offers more than a sorting program in terms of accuracy and number of analyzed elements. It can be considered as the cheapest solution to analyze all types of samples.

The use of a global calibration is recommended when the program choice function is used.

The global calibration for small samples can also be used for massive samples with some performance limitations.

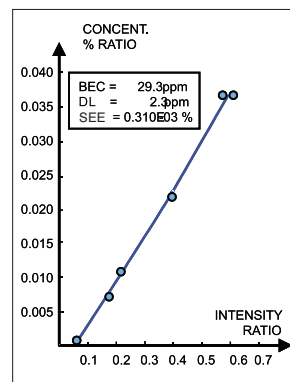
Factory calibration (CARL)

The Thermo Scientific optical emission spectrometers can be factory calibrated for iron and steel utilizing CARL, a very sophisticated multi-variable regression tool that corrects for matrix effects as well as spectral interferences. CARL can provide an immediate “turnkey” system which gives the user the highest accuracy possible. The calibrations are available for the different qualities given above. For each quality Thermo Fisher uses certified reference material (CRM) as standard samples and setting-up samples (SUS) are delivered with the instrument to maintain the accuracy of the calibration.

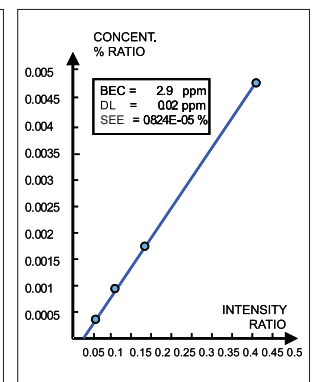
Accuracy

Precision is only a small part of providing accurate analyses. The most important factor is the accuracy and quality of the calibration standards. Next is the development of the calibration curve relative to a specific analytical task. Matrix matching and high energy pre-burns reduce or eliminate matrix effects and spectral interferences are significantly reduced by applying appropriate corrections.

The example shown below illustrates the significant improvement in the calibration curve thanks to the TRS feature.



Element: Cu without TRS



Element: Cu with TRS

ARL 4460 - Typical detection limits (3 sigma) and precision values (1 sigma) for IRON base

ELEMENT	Al	As	B	Bi	C	Ca	Ce	Co	Cr	Cu	La	Mg	Mn	Mo	N	Nb*
TYPICAL DL [ppm]	0.4	0.7	0.2	1	2	0.2	2	0.6	1.3	0.2	0.1	0.3	0.7	1.4	2.7	0.7
GUARANTEED DL [ppm]	<1	<1.5	<0.4	<2	<5	<0.3	<4	<1	<2	<0.4	<0.3	<0.5	<1.2	<2	<4.5	<1.5
Level [%]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.001	0.00003	0.00005	0.00001	0.00005	0.00007	0.00004	0.00015	0.00003	0.00005	0.00002	0.00002	0.00002	0.00005	0.00006	0.00008	0.00002
0.002	0.00004	0.00006	0.00002	0.0001	0.00008	0.00007	0.00015	0.00003	0.00005	0.00002	0.0001	0.00005	0.00007	0.00007	0.00009	0.00002
0.005	0.00006	0.00008	0.00003	0.0001	0.00015	0.00017	0.00015	0.00004	0.00008	0.00004	0.0003	0.00015	0.0001	0.0001	0.00016	0.00004
0.01	0.0001	0.0001	0.00005	0.00015	0.0002	0.0003	0.0003	0.00005	0.0001	0.00006	0.0006	0.00025	0.00012	0.00015	0.00025	0.0001
0.02	0.0002	0.00016	0.0001	0.0003	0.0003	0.0005	0.0005	0.0001	0.00015	0.0001	0.0012	0.0004	0.00015	0.0002	0.0003	0.0002
0.05	0.00045	0.0003			0.00055		0.0012	0.0002	0.00025	0.00025		0.0011	0.0003	0.0004	0.00055	0.0003
0.1	0.0009	0.00045			0.00075			0.00035	0.0004	0.0005		0.002	0.0005	0.0006	0.001	0.0005
0.2	0.002		0.003		0.001			0.0007	0.0007	0.001		0.004	0.0008	0.001	0.002	0.001
0.3	0.0025		0.007		0.0015			0.001	0.0009	0.0015			0.001	0.0015	0.003	0.0015
0.5	0.004		0.013		0.002			0.0016	0.0015	0.0025			0.002	0.002	0.006	0.0025
1	0.008				0.004			0.0035	0.0025	0.005			0.003	0.004		0.005
2					0.008			0.0065	0.004	0.01			0.006	0.006		0.01
3					0.013			0.01	0.005	0.015			0.008	0.008		0.012
4					0.016			0.013	0.0065	0.02			0.01	0.01		
5								0.016	0.008	0.025			0.013	0.012		
10								0.03	0.015				0.025	0.02		
20									0.02							
30									0.03							
40									0.04							

ELEMENT	Ni	O	P	Pb	S	Sb	Se	Si	Sn	Ta*	Te	Ti	V	W	Zn	Zr
TYPICAL DL [ppm]	1	20	0.3	0.5	0.7	2	1.5	3	1.5	5	1.5	0.3	0.7	5	0.6	0.6
GUARANTEED DL [ppm]	<1.7	<30	<0.8	<1	<1	<3.5	<3	<5	<3	<10	<3.5	<0.6	<1.5	<10	<1.2	<1
Level [%]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.001	0.00008		0.00003	0.00003	0.00003	0.00008	0.00007	0.00006	0.00005	0.0002	0.00006	0.00003	0.00004	0.0003	0.00003	0.0001
0.002	0.00008		0.00003	0.00004	0.00006	0.0001	0.0001	0.00006	0.00006	0.0002	0.00008	0.00003	0.00004	0.0003	0.00003	0.0001
0.005	0.00008	0.0005	0.00006	0.00008	0.00014	0.00012	0.0002	0.00008	0.00008	0.00025	0.00015	0.00005	0.00006	0.0003	0.00004	0.00015
0.01	0.0001	0.0006	0.0001	0.0002	0.00025	0.00015	0.00025	0.0001	0.0001	0.0003	0.00025	0.0001	0.0001	0.0003	0.00007	0.0003
0.02	0.0001	0.0001	0.0002	0.0005	0.0005	0.0002	0.0003	0.00015	0.00015	0.0004	0.0004	0.0002	0.00015	0.0003	0.00012	0.0006
0.05	0.0002	0.002	0.0005	0.0015	0.001	0.0004	0.0005	0.0003	0.0003	0.0008	0.0008	0.0005	0.0004	0.0004	0.00022	0.0015
0.1	0.0003		0.0009	0.003	0.0025	0.0007	0.0007	0.0005	0.0005	0.0022		0.001	0.0007	0.0006	0.0004	0.0025
0.2	0.0005		0.0015	0.008	0.006			0.001	0.001			0.002	0.0014	0.001		0.004
0.3	0.0007		0.002	0.012	0.01			0.0014				0.003	0.002	0.0015		0.006
0.5	0.0015		0.0035					0.0022				0.005	0.003	0.0025		0.009
1	0.0025		0.006					0.004				0.01	0.005	0.005		
2	0.005							0.008				0.02	0.009	0.01		
3	0.008							0.012				0.012	0.014	0.014		
4	0.01							0.015				0.015	0.017	0.017		
5	0.015											0.02	0.02	0.02		
10	0.025											0.1	0.035	0.035		
20	0.045													0.08		
30	0.075													0.15		
40	0.1															

Remarks: This data applies when homogeneous samples are prepared by recommended sample preparation methods.

The precision values given are typical. The guaranteed precision values are 1.5 times higher.

For multibase instruments, some analytical performance may vary based on the analytical line and grating selected.

Guaranteed DLs are calculated at 95 % confidence limit.

Performance for C, N and O can be improved with the so-called CNO option (see dedicated application note).

*DL of Nb if Ti > 0.2 % : 2.4 ppm typical and 3.7 ppm guaranteed.

*DL of Ta if not for low alloy steel analysis: 12 ppm typical and 18 ppm guaranteed.

Performance guarantee

Our company guarantees the precision shown in the above tables using homogeneous samples and recommended sample preparation. The list will be updated as improvements are announced. Please contact your nearest Thermo Fisher Scientific office for the most recent values.

The precision is calculated from the formula:

where:

$SD(1\sigma) = \pm \sqrt{\frac{\sum_{i=1}^{n-1} (X_i - \bar{X})^2}{n-1}}$

X_i the individual readings
 \bar{X} the arithmetic mean of the individual readings
 n the number of determinations

The DL (Detection Limit) is defined as three times the standard deviation of the background expressed in concentration units. The lower limits of quantification (LLQ) are alloy dependent and are defined in the calibration menus.

Options

1. PIM 2

The PIM 2 method permits the determination of soluble/insoluble aluminum and boron in low alloy steel. It does not need special skills from the operator and replaces a costly wet chemistry procedure.

2. Small samples analysis

This option permits the quantitative determination of wires and small samples with diameters down to 3 mm., thanks to a special excitation condition developed for this purpose. Small samples are normally analyzed with more labor intensive and costly methods (ICP, wet chemistry...).

3. CNO option

The CNO option permits the determination of C, N and O at ultra-low levels, as well as improves the analysis of these elements at low levels, so that no alternative analytical method like combustion analysis is required anymore. This option is required for the analysis of the quality micro-alloyed steel.

4. Spark-DAT on-line

The purpose of Spark-DAT (Spark Data Acquisition and Treatment) is to acquire the intensity of each individual spark instead of the integrated value and treat the signals via fast on-line algorithms. Spark-DAT offers improved performance for the determination of soluble/insoluble Al and B in low alloy steel. In addition, Spark-DAT is the fastest method to obtain information related to the amount, the size and the composition of inclusions. It can be used in production to control inclusions, even simultaneously with the steel composition. Spark-DAT can replace long or costly methods that measure steel cleanness indices, or other characteristics that depend on the presence of inclusions in the metal (e.g. fatigue resistance or total oxygen at low concentration).

Separate application notes are available on request for most options.

Stability

Stability of the instrument is of the utmost importance when performing routine analysis.

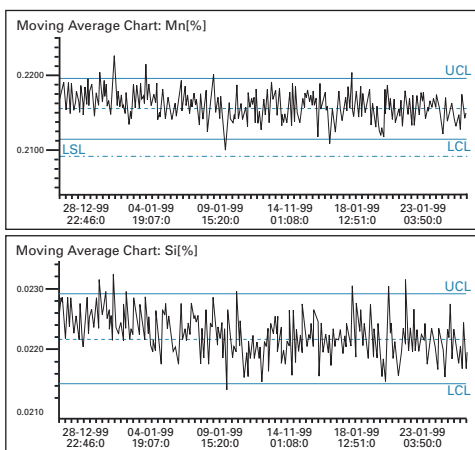
Mid-term stability measured over 24 hours shows that typical standard deviation achieved is below two times the precision value, which is excellent.

The example below shows the long term stability of two elements recorded over a period of 15 days without any intermediate drift correction. The values never went outside the control limits and no standardization was required.

To see our complete OES product portfolio, visit www.thermoscientific.com/oes.

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Conclusion

The ARL 4460 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
- Adaptable to the automatic Sample Manipulation System: ARL SMS-2000 and ARL SMS-3000
- 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 thanks to the exceptional and widely recognized instrument lifetime and to the continuous upgrade possibilities (software and hardware)
- Your investment costs are reduced with the capability of the instrument to cover your future needs
- Your production costs are reduced by the fact that more accurate and reproducible analyses are available faster
- Your production costs are reduced by the increased instrument availability thanks to its high stability and drift corrections being less frequently required
- Your operating and maintenance costs are reduced through low consumption of drift correction samples, and through simple maintenance
- Your overall cost management is reduced by optimum utilization of materials and extremely low running costs compared to other methods

With its over 70 years of experience and history of innovative technology, our company has become the world leader in OE metals analysis. We work with our customers to improve the efficiency of their analytical tasks and thereby increase productivity.

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

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