Application Note: 41200

Analysis of Iron and Steel by Optical Emission

ARL 3460 Metals Analyzer

Key Words

- ARL 3460
- Iron & Steel
- Metals Analyzer
- Optical Emission

Introduction

The Thermo Scientific ARL 3460 Metals Analyzer is designed to meet the specific requirements of a wide range of customers from the smallest cast iron foundries to the largest steel plants. Our long experience in metals analysis comes from an installed base of over 10,000 spectrometers worldwide. The ARL 3460 satisfies all your metallurgical analysis needs; whether they be incoming material control or metal QC and production analysis. Working 24 hours a day, 7 days a week, the ARL 3460 delivers dependable performance year after year.

Iron

Several kinds of iron exist, distinguished by their composition and use. They belong to two main categories:

- Pig irons (hot metal); basic product for the manufacture of steel.
- Cast irons; used for the production of semi-manufactured products.

From a metallographic point of view, a distinction can be made between white cast iron with a cementite structure, and grey cast iron which contains free graphite, either in the form of laminae or nodules. Alloy cast irons also exist where alloying elements such as nickel, chromium, manganese, copper, etc., are added to improve hardness, corrosion resistance or engineering properties (Ni-hard, Ni-resist, etc.).

Steel

Typically steels are divided in two main qualities:

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

1. Low alloy steels

The total element alloying is generally less than 5-7 %. The main elements are: Mn (≤ 2 %), Cr (≤ 3 %), Mo (≤ 1.5 %), Cu (≤ 2 %), Ni (≤ 5 %), and V (≤ 1 %). The so called “free-cutting steels” with a concentration of 0.2 - 0.3 % sulfur or lead, need a special calibration.

2. High alloy steels

High alloy steels contain, in addition to iron and carbon, notable quantities of one or more of the following elements: nickel, chromium, manganese, silicon, cobalt, tungsten, molybdenum and vanadium. This category covers steels which are destined for a wide variety of uses such as :

- Stainless steels (ferritic or austenitic 18/8, maraging and all types of special stainless steels)
- Tool steels
- High manganese steels
- High speed steels
- Special high alloys steels, such as Ni-hard, Ni-resist, etc.

ARL 3460 Metals Analyzer

The ARL 3460 Metals Analyzer can determine up to 60 elements simultaneously. The HiRep source has the flexibility to customize the excitation parameters providing the best analytical conditions for a wide variety of sample types.

The source uses the HEPS (High Energy Pre-Spark) technique to minimize the metallurgical effects and produce consistently accurate results. The printout of average concentration for the required elements from a repeat analysis is obtained in typically less than one minute.

Sample preparation

The sample is generally prepared by using a grinding machine. Milling machines are recommended for some critical qualities.

Sample analysis time

LOW ALLOY STEELS WITH NITROGEN & SOL./INSOL. Al	LOW ALLOY STEELS WITH NITROGEN	CAST IRON	FREE CUTTING STEELS
27s	25s	31s	68s

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Calibration summary

Numerous “qualities” are available for specific alloy types:

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

The global calibration offers more than a sorting program in terms of accuracy and number of analyzed elements. The global calibration can be considered as the basic low cost calibration of the instrument, allowing the analysis of unknown samples.

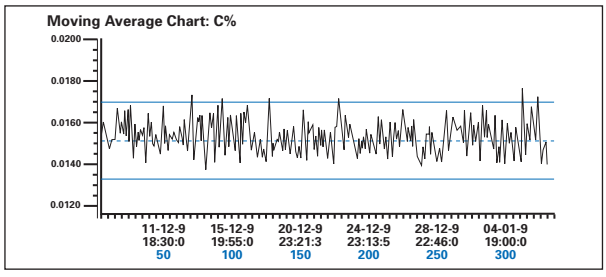
Factory calibration (CARL)

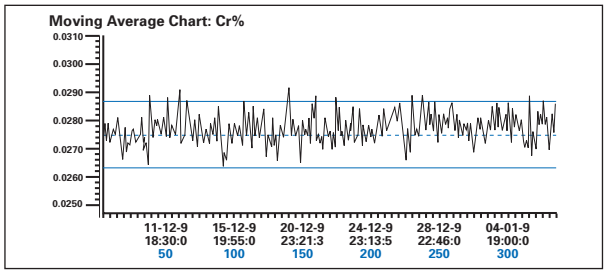
Thermo Scientific optical emission spectrometers can be factory calibrated for iron and steel utilizing CARL, a very sophisticated multi-variable regression tool that corrects for matrix effects as well as spectral interferences. CARL can provide an immediate "turnkey" system which gives the user the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

Stability

Stability of the instrument is of the utmost importance when doing routine analysis. Typical mid-term stability measured over 24 hours shows that the 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 more than 15 days without any intermediate drift correction. The values almost never went outside the control limits.

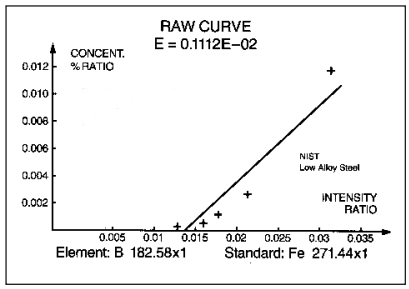




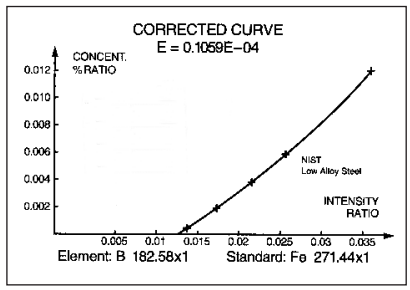
Accuracy

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-burn reduce or eliminate matrix effects, and spectral interferences are significantly reduced by applying appropriate corrections.

The example shown below illustrates the significant improvement with the CARL calibration.



Before CARL



After CARL

Performance guarantee

Our company guarantees the precision shown in the attached 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 or consult our web site at **www.thermo.com/oes** for the most recent values.

The precision is calculated from the formula:

$$SD(1\sigma) = \pm\sqrt{\frac{\sum_{i=0}^{i=n}(X_i - \overline{X})^2}{n-1}}$$

where:

X_i the individual readings

$\overline{X}$ 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.

Option

PIM 2

This option permits the determination of soluble/insoluble aluminum and boron in low alloy steel during the normal OES analysis.

ARL 3460 - Typical detection limits (3 sigma) and precision values (1 sigma) for iron base

ELEMENT	Al	As	B	Bi	C	Ca	Ce	Co	Cr	Cu	Mg	Mn	Mo	N	Nb
TYPICAL DL [ppm]	**1**	**1.6**	**0.3**	**4**	**4.5**	**0.2**	**4.5**	**0.8**	**1.5**	**0.6**	**3**	**0.8**	**1.6**	**2.8**	**2.7**
GUARANTEED DL [ppm]	**< 1.8**	**< 2.7**	**< 0.6**	**< 6.5**	**< 7.5**	**< 0.5**	**< 7**	**< 1.3**	**< 3**	**< 1.3**	**< 5**	**< 1.3**	**< 2.5**	**< 4.6**	**< 4.5**
Level [%]	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.001	0.00006	0.00015	0.00002			0.00007		0.00005	0.00015	0.00006		0.00005	0.0002	0.0002	
0.002	0.00009	0.00015	0.00004	0.00026	0.00023	0.00014	0.00025	0.00005	0.00015	0.00007	0.0001	0.00005	0.0002	0.0003	0.00016
0.005	0.00016	0.00015	0.00012	0.00042	0.00023	0.00033	0.00025	0.00005	0.00015	0.00009	0.00025	0.00005	0.0002	0.00045	0.00022
0.01	0.00025	0.0002	0.00038	0.00062	0.00023	0.00065	0.0003	0.0001	0.0002	0.00012	0.0005	0.00008	0.0003	0.0006	0.00035
0.02	0.00038	0.00025	0.001	0.00085	0.0003	0.0013	0.0005	0.00015	0.0003	0.00018	0.0009	0.00015	0.0006	0.0008	0.0005
0.05	0.0007	0.00035			0.0006		0.0013	0.0004	0.0005	0.0003	0.0022	0.00032	0.001	0.0013	0.001
0.1	0.0012	0.0005			0.001			0.0007	0.0008	0.0006	0.0043	0.0006	0.0015	0.0018	0.0015
0.2	0.002				0.002			0.0015	0.0012	0.0012	0.008	0.0011	0.002	0.0025	0.0025
0.3	0.003				0.0025			0.002	0.0015	0.002		0.0025	0.003	0.003	0.005
0.5	0.005				0.0037			0.003	0.002	0.0035		0.005	0.005		0.009
1.	0.008				0.0062			0.006	0.0035	0.0065		0.009	0.008		0.017
2.					0.01			0.01	0.005	0.01		0.013	0.01		0.03
3.					0.015			0.015	0.007	0.015		0.017	0.012		
4.					0.02			0.02	0.008	0.02		0.02	0.015		
5.								0.025	0.01	0.025		0.03	0.025		
10.								0.05	0.015			0.04	0.04		
20.									0.03						
30.									0.04						
40.									0.05						

ELEMENT	Ni	O	P	Pb	S	Sb	Si	Sn	Ta	Te	Ti	V	W	Zn	Zr
TYPICAL DL [ppm]	**1.4**	**30**	**1.8**	**2.8**	**1.4**	**5**	**3**	**1.5**	**15**	**2.6**	**0.4**	**1**	**9**	**2.4**	**1.8**
GUARANTEED DL [ppm]	**< 2.4**	**–**	**< 3**	**< 4.7**	**< 2.3**	**< 10**	**< 4.5**	**< 3**	**< 22**	**< 4.5**	**< 0.6**	**< 1.6**	**< 14**	**< 3.3**	**< 3**
Level [%]	SD		SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.001	0.00015		0.00008		0.00009			0.00008			0.00003	0.00007		0.0002	0.00012
0.002	0.00015		0.00009	0.0002	0.00014		0.00015	0.0001		0.00025	0.00005	0.00007		0.00025	0.00012
0.005	0.00015		0.0001	0.00035	0.00028	0.0003	0.00015	0.0001	0.0005	0.0004	0.00012	0.00007	0.0005	0.0003	0.00013
0.01	0.00015		0.00013	0.0007	0.0005	0.00033	0.0002	0.00015	0.001	0.0007	0.00022	0.0001	0.0005	0.00035	0.0003
0.02	0.00016		0.00025	0.0014	0.0009	0.00045	0.00025	0.00025	0.0015		0.0005	0.0002	0.00055	0.00038	0.0006
0.05	0.0003		0.0006	0.0035	0.002	0.001	0.0003	0.0004	0.0025		0.001	0.0005	0.00065	0.00042	0.0015
0.1	0.0005		0.0016	0.01	0.004	0.0022	0.0007	0.00065	0.0035		0.002	0.0008	0.00085		0.0052
0.2	0.0009		0.0033	0.02	0.0075		0.0012				0.003	0.0015	0.0015		0.021
0.3	0.0013		0.005	0.025	0.012		0.002				0.005	0.002	0.002		0.45
0.5.	0.002		0.009				0.003				0.01	0.003	0.003		0.01
1.	0.004		0.015				0.005				0.02	0.006	0.005		
2.	0.007						0.01					0.011	0.01		
3.	0.011						0.015					0.015	0.015		
4.	0.013						0.02					0.02	0.02		
5.	0.016											0.025	0.025		
10.	0.035												0.045		
20.	0.065												0.09		
30.	0.1												0.15		
40.	0.15														

Remarks: This data applies when homogeneous samples are prepared by recommended sample preparation methods.
The precision given is typical performance. Guaranteed values will be 1.5 times higher.
For multibase instruments, some analytical performance may vary based on the analytical line selected.
Guaranteed DLs are calculated at 95 % confidence limit.

Conclusion

The ARL 3460 has all the total system features which meet the critical needs of the metals analysis markets:

- Unmatched hardware for stability and reliability
- Excellent performance in detection limits, precision, accuracy, stability and analysis time
- Most advanced software technology with HTML/Internet simple to use tools
- Easy operation by unskilled worker or research chemist
- Widest range of metals analysis
- Adaptable to the automatic OES Sample Manipulation System (ARL SMS-2000)
- Advanced technical/service support
- Laboratory accreditation guidance
- Immediate access to parts inventory

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 continous upgrade possibilities (software and hardware)
- 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 OES 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.

Africa
+43 1 333 5034 127
Australia
+61 2 8844 9500
Austria
+43 1 333 50340
Belgium
+32 2 482 30 30
Canada
+1 800 530 8447
China
+86 10 5850 3588
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Sweden/Norway/ Finland
+46 8 556 468 00
Switzerland
+41 21 694 71 11
UK
+44 1442 233555
USA
+1 800 532 4752

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