

Analysis of Small Samples of Steel and Iron Alloys by Optical Emission

ARL 4460 Metals Analyzer

Key Words

- ARL 4460
- Optical Emission
- Small samples
- Steel and Iron

Introduction

Significant progress has been made in the quantitative analysis of small samples of steel and iron. Sensitivity, precision and accuracy have been substantially improved with the Thermo Scientific ARL 4460 Metals Analyzer and the performance difference compared to “normal” samples is small. The preparation of small samples is simplified, and a faster analysis than with other techniques (ICP, chemical...) becomes possible, allowing for drastic reductions in analysis cost.

Small samples made of steel and iron

Several kinds of small samples made from steel and iron exist, distinguished by their shape, composition and use:

Shape:

- Wires with diameters down to less than 1 mm
- Sheets of various thickness
- Small samples of various shape

Composition:

Low alloy steel, Cr and Cr-Ni steel, tool steel, free cutting steel, special carbon steel, Ni-hard, Ni-resist...

Use:

Screws, nuts, axles, springs, cogwheels, rivets, cams, spade tag, rods, pistons... for many application fields.

ARL 4460 Metals Analyzer

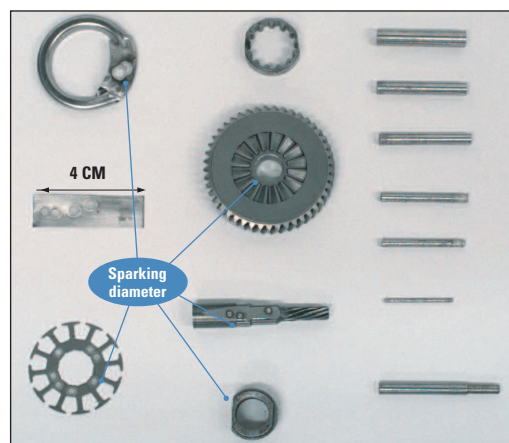
Using a dedicated tool and a specific sparking condition, the ARL 4460 Metals Analyzer can quantitatively determine up to 28 elements in small samples in less than two minutes. Analytical performance is detailed in this application note.

The Thermo Scientific Current Control Source (CCS) presents significant advantages in comparison to any other spark generator currently used for OES. The current waveform is computer controlled and the high degree of flexibility in selection of parameters enables the optimization of sensitivity, precision, and of the sparking diameter, which is important for the analysis of small samples. More details can be found in the application summary AN41220 “Analysis of Iron and Steel by Optical Emission - ARL 4460 Metals Analyzer”.

Small sample analysis

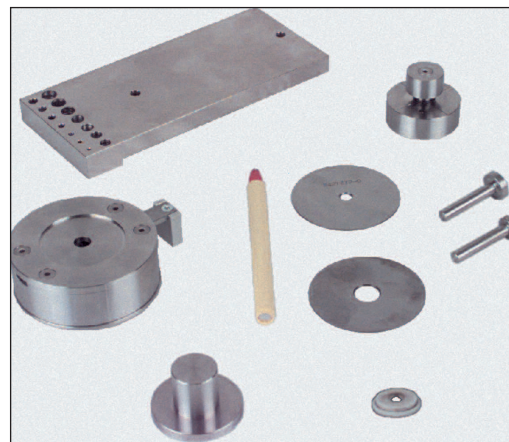
The ARL 4460 analyzes small samples of very different sizes and shapes as shown in the following picture:

- Wires: quantitative analysis down to 3 mm and semi-quantitative for smaller diameters.
- Sheets: quantitative analysis down to 50 μm thickness.



Kit for small sample analysis

A small samples analysis kit is supplied as shown below:



Sample preparation

The preparation depends on the sample shape:

- For wires: using a grinding machine and a special tool (included in the kit)
- For sheets: manual polishing with paper (grain 800)
- For samples of various shape: using a grinding machine
- In some cases, the sample can be pressed beforehand to obtain a larger surface area

Sample analysis time

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

MATERIAL	"NORMAL" SAMPLE EXCITATION	SMALL SAMPLE CCS EXCITATION
Steels with Nitrogen	19 s	44 s
Free Cuttings Steels	51 s	44 s

Factory calibration (CARL)

ARL 4460 spectrometers can be factory calibrated for iron and steel small sample analysis.

Our company proposes a specific global calibration for this OES application field, that covers all standard qualities available, and a specific quality covering low alloy steel and free cutting steel.

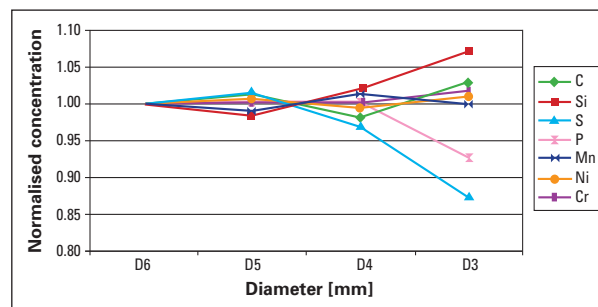
EI	GLOBAL SMALL SAMPLE CALIBRATION	LOW ALLOY STEEL + FREE-CUTTING FREE-CUTTING
	CAL-44-Fe-13	CAL-44-Fe-15
Fe	99.8 % - 42 %	99.9 % - 80 %
Al	0.003 - 1.5	0.001-0.3
As	0.003 - 0.1	0.001-0.1
B		0.0003-0.01
Bi	0.003 - 0.01	
C	0.01 - 4.2	0.001-1.3
Ca	0.0005 - 0.015	0.0005-0.01
Ce	0.002 - 0.015	
Co	0.002 - 12	0.001-0.15
Cr	0.005 - 32	0.001 - 5.0
Cu	0.005 - 6.5	0.005 - 0.7
Mg	0.001 - 0.15	
Mn	0.005 - 20	0.001 - 2.0
Mo	0.005 - 9	0.001 - 1.5
Nb	0.002 - 2.7	0.001 - 0.6
Ni	0.005 - 35	0.001 - 5.0
P	0.005 - 1.4	0.0005 - 0.1
Pb	0.002 - 0.19	0.001 - 0.19
S	0.002 - 0.31	0.001 - 0.3
Sb	0.005 - 0.15	0.002 - 0.07
Si	0.005 - 5.5	0.001 - 3.0
Sn	0.002 - 0.3	0.001 - 0.1
Ta		0.01 - 0.15
Te	0.002 - 0.03	
Ti	0.002 - 2	0.001 - 0.4
V	0.002 - 10	0.001 - 1.0
W	0.015 - 22	0.005 - 0.6
Zn	0.002 - 0.025	0.001 - 0.02
Zr	0.002 - 0.1	0.001 - 0.1

Accuracy

The most important analytical figure of merit is the accuracy. Typically, it is improved by a factor 2.5 in the analysis of 3 mm low alloy steel wires in comparison with classical sources. The next example illustrates the accuracy in the determination of two wire reference materials:

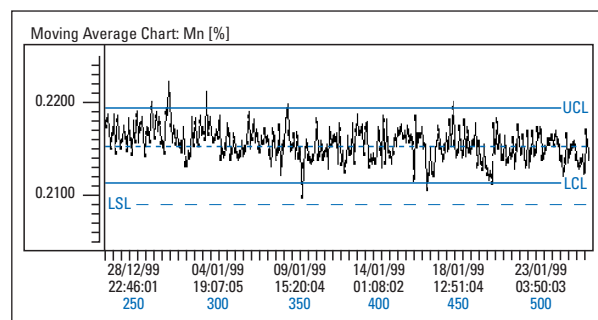
QUALITY	LALY		Cr-Ni	
Sample	NBS 662		NBS 447	
Diameter [mm]	3 mm		5.5 mm	
ELEMENT	CERTIFIED	ESTIMATED	CERTIFIED	ESTIMATED
C	0.16	0.16	-	-
Si	0.39	0.37	0.37	0.34
S	0.04	0.02	-	-
P	0.04	0.03	-	-
Mn	1.04	1.00	0.23	0.21
Ni	0.59	0.60	13.3	13.6
Cr	0.30	0.29	23.7	23.9

The accuracy depends on the wire diameter as shown in the next picture:



Stability

Instrument stability is of the utmost importance when doing routine analysis. The following example shows the long term stability of one element recorded over a period of 15 days without intermediate drift correction.



Sensitivity and precision

The fundamental figures of merit are given in the following tables. The values are valid for a wire with a diameter of 6 mm. At worst they can degrade up to a factor of 3 with a wire with a diameter of 3 mm.

ARL 4460 - Typical detection limits (3 sigma) and precision values (1 sigma) for small samples in iron base

ELEMENT	Al	As	B	Bi	C	Ca	Ce	Co	Cr	Cu	Mg	Mn	Mo	N
TYPICAL DL(PPM)	4.0	2.8	1.3	4.2	10 (4)	1.2	3.4	1.1	2.2	9 (4)	2.5	2.8	4.2	6.5
GUARANTEED DL (PPM)	≤ 8	≤ 5	≤ 3	≤ 8	≤ 20	≤ 3	≤ 9	≤ 3	≤ 5	≤ 15	≤ 5	≤ 5	≤ 8	≤ 15
Level %	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.001	-	-	0.00005	-	-	0.00007	-	0.00005	-	-	-	-	-	-
0.002	0.00012	0.00015	0.00005	0.0002	-	0.0001	0.00015	0.00005	0.00015	-	0.0001	0.0001	0.00015	0.0003
0.005	0.0002	0.00015	0.00005	0.00025	0.0003	0.0002	0.0002	0.00007	0.00015	0.0002	0.0002	0.0001	0.00015	0.0003
0.01	0.0004	0.0002	0.00005	0.0003	0.0004	0.00035	0.0004	0.0001	0.0002	0.0002	0.0004	0.00015	0.00017	0.0004
0.02	0.0006	0.00025	0.0001	0.00035	0.0007	0.0007	0.0007	0.00015	0.0003	0.0003	0.0008	0.0003	0.0002	0.0006
0.05	0.0009	0.00035			0.0012			0.0004	0.0005	0.0004	0.002	0.0005	0.0004	0.0009
0.1	0.0015	0.0005			0.0018			0.0007	0.0008	0.0006	0.0035	0.0008	0.0007	0.0015
0.2	0.0025				0.0028			0.0015	0.0012	0.0012		0.0012	0.0012	0.002
0.3	0.0035				0.004			0.002	0.0015	0.002		0.0016	0.0015	0.003
0.5	0.005				0.005			0.003	0.002	0.003		0.0025	0.0025	
1	0.008				0.008			0.006	0.0035	0.006		0.004	0.004	
2					0.016			0.01	0.005	0.01		0.008	0.008	
3					0.025			0.015	0.007	0.015		0.01	0.012	
4					0.035			0.02	0.008	0.02		0.012	0.015	
5								0.025	0.01	0.025		0.015	0.02	
10								0.05	0.015			0.03	0.035	
20									0.025			0.06		
30									0.035					
40									0.045					

ELEMENT	Nb	Ni	P	Pb	S	Sb	Si	Sn	Te	Ti	V	W	Zn	Zr
TYPICAL DL(PPM)	4.0	1.8	3.8	6.5	1.3	11	6.6	4.5	2.5	0.4	1.2	30	2.5	1.0
GUARANTEED DL (PPM)	≤ 8	≤ 4	≤ 7	≤ 15	≤ 3	≤ 25	≤ 12	≤ 9	≤ 4	≤ 1	≤ 3	≤ 80	≤ 6	≤ 3
Level %	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
0.001	-	0.00015	-	-	0.00005	-	-	-	-	0.00003	0.00004	-	-	0.0001
0.002	-	0.00015	0.00012	-	0.0001	-	-	0.00015	0.00025	0.00004	0.00004	-	0.0001	0.0001
0.005	0.00025	0.00015	0.00015	0.0007	0.0002	0.00045	0.00025	0.0002	0.0004	0.0001	0.00006	-	0.00025	0.00015
0.01	0.0004	0.00015	0.00025	0.0013	0.0004	0.00055	0.00025	0.00025	0.0007	0.0002	0.00008	0.001	0.0004	0.0005
0.02	0.0006	0.00015	0.0003	0.0025	0.0008	0.0007	0.0003	0.0003		0.0003	0.0001	0.001		0.001
0.05	0.0011	0.0002	0.0006	0.0065	0.0020	0.001	0.0006	0.0005		0.0008	0.00025	0.0013		0.0025
0.1	0.0017	0.0004	0.0009	0.013	0.0035	0.0012	0.0010	0.0008		0.0016	0.00045	0.0017		0.0055
0.2	0.0027	0.0008	0.0015	0.025	0.007		0.0015			0.003	0.001	0.0025		
0.3	0.0035	0.0012	0.002	0.04	0.01		0.0025			0.0045	0.0015	0.0032		
0.5	0.005	0.002	0.0035				0.005			0.0075	0.003	0.0045		
1	0.008	0.004	0.006				0.009			0.015	0.0065	0.0075		
2	0.015	0.007					0.015			0.03	0.016	0.013		
3	0.025	0.01					0.02				0.025	0.02		
4		0.012									0.035	0.03		
5		0.015									0.045	0.05		
10		0.035										0.10		
20		0.06										0.15		
30		0.09												
40		0.12												

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

The precision given is typical performance. Guaranteed values will be 2 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 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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