Application note | AN41423

Steel analysis

ARL X900 Simultaneous/Sequential X-Ray Fluorescence Spectrometer

Keywords
ARL X900, XRF, WDXRF, steel, ferrous base, X-ray fluorescence

ARL X900 Simultaneous/Sequential X-Ray Fluorescence Spectrometer.

Goal

Describe the analytical performance of Thermo Scientific™ ARL™ X900 Series for steel analysis.

Introduction

Ferrous base materials are very important products in our world because they are the foundation in many applications such as building, automotive and many manufacturing processes. It is important to accurately analyze these materials to confirm compliance with their chemical specifications and allow for high quality and efficient production.

Irons

There are several kinds of irons which are distinguished by their composition and use. They belong to two main categories:

- Pig irons, also called hot metal, form the basic production for the manufacture of steel
- Cast irons, are used to produce 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. These make grey cast iron inhomogeneous and therefore difficult to analyze. 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.

Low alloy steels

This category covers steels which are destined for a wide variety of uses, such as the production of:

- Steel castings, rails, axels, boiler and ship plates, automobile bodies
- Girders, all kinds of bridge and structural sections
- Wires, nuts, bolts, and forgings of any description
- Springs, cutting steels

From a compositional point of view, these steels can be distinguished by the fact that the alloying elements generally total less than 5 to 7 %. Typically, the main alloying elements are present at less than the following concentrations:

Mn 2 % ; Cr 3 % ; Ni 5 % ; Cu 1.5 % ; Mo 1.5 % ; V 1 %.

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. Included under this heading are: Stainless steel types such as 18/8, austenitic, maraging, martensitic and all types of special stainless steels, tool steels, high speed steels, and high manganese steels.

Instrument parameters and conditions

The ARL X900 XRF Spectrometer can accommodate up to 24 fixed monochromator channels alongside the goniometer or up to 32 fixed monochromator channels when no goniometer is fitted. Optional high counting fixed channel monochromators are available to further improve the precision of analysis, especially for elements like Ni, Co, and Mo.

Our unique proprietary Moiré fringe goniometer ensures speed, flexibility, and reliability of analysis thanks to the clever friction-free positioning system. Up to nine crystals and four collimators can be fitted. With the two detectors (flow proportional and scintillation counters), precise elemental analysis from boron to californium is possible.

The ARL X900 XRF Spectrometer can be calibrated using commercially available certified reference material (CRM) standards or well analyzed samples from the user. Calibrations can be delivered from the factory, hence reducing the commissioning time at the customer's site.

This application note only includes data obtained on fixed channels. Please refer to application note 41425 for steel data using the goniometer.

Typical performance in steel samples

Table 1 is a summary of limits of detection (LoD) determined by repeat analysis of a blank sample (pure iron RE12) using 20 seconds and 100 seconds counting time per element on fixed channel monochromators at high power (4200W). The limits of detection are calculated as 3 times the standard deviation of 21 repeatability runs.

Twenty seconds is the selected counting time for fixed channels used in the steel industry, allowing for a final result to be obtained in less than a minute. Hundred seconds counting time is often used to express limits of detection in X-ray fluorescence technique as a point of comparison.

Element	Line	Empirical LoDs	Empirical LoDs
		20s fixed channel	100s fixed channel
Al	Kα	11.3	5.1
As	Kβ	3.3	1.5
Ca	Kα	3.6	1.6
Co	Kα	8.9	4.0
Cr	Kα	5.9	2.7
Cu	Kα	4.1	1.9
Mn	Kα	8.2	3.7
Mo	Kα	1.7	0.8
Nb	Kα	1.8	0.8
Ni	Kα	6.5	2.9
P	Kα	2.7	1.2
S	Kα	2.1	0.9
Sb	Kα	7.6	3.4
Si	Kα	15.9	7.1
Sn	Kα	8.4	3.8
Ta	Lβ	20.7	9.3
Ti	Kα	4.0	1.8
V	Kα	3.3	1.5
W	Lα	11.6	5.2
Zr	Kα	2.8	1.3

Table 1: Typical limits of detection in ferrous matrix for 20 fixed monochromator channels at two different counting times.

Typical precision tests

The stability of an instrument reflects the precision that can be obtained. Calibration curves were obtained with a large set of international steel standards at X-ray tube settings of 50 kV and 70 mA. Overlap and matrix corrections were made according to our state-of-the-art recommendations. A short-term test consisting of eleven runs of 20 seconds was performed using a couple of steel samples. Fixed monochromators channels analyze simultaneously. Hence, up to 32 elements can be measured in less than one minute, providing they are all fitted in the spectrometer. This includes loading and pumping the sample into the vacuum of the spectrometer.

Table 2a and 2b provides summaries of analytical results from short-term repeatability tests. The ARL X900 Spectrometer is fitted with 21 fixed channels. Only the relevant data is shown.

The innovative, high counting fixed channels permit to measure high concentration levels of Ni, Cr and Mo without the need of an attenuation filter; hence the precision of analysis is dramatically improved.

A long-term repeatability test over 38 hours was performed consisting of one analysis of 20 seconds every second hour at X-ray tube settings of 50 kV and 70 mA (Table 3). All elements are measured with fixed channel monochromators. The high linearity channels for Ni, Co and Mo permit analysis without attenuation filter, which improves the precision of analysis.

Elem	Si	S	P	Mn	Ni	Cr	Mo	V	Cu	W	Ti	Sn	Co	Al
	%	%	%	%	%	%	%	%	%	%	%	%	%	%
1	0.3334	0.0037	0.0109	0.4365	43.021	23.424	2.713	0.0449	1.748	0.0030	0.713	0.0060	0.0813	0.1127
2	0.3330	0.0036	0.0110	0.4372	43.023	23.435	2.713	0.0448	1.751	0.0010	0.714	0.0061	0.0812	0.1115
3	0.3329	0.0039	0.0102	0.4352	43.023	23.430	2.714	0.0455	1.749	0.0026	0.714	0.0059	0.0815	0.1115
4	0.3336	0.0037	0.0111	0.4363	43.025	23.433	2.714	0.0456	1.750	0.0019	0.713	0.0060	0.0807	0.1122
5	0.3334	0.0040	0.0106	0.4362	43.033	23.427	2.714	0.0451	1.746	0.0031	0.715	0.0061	0.0811	0.1114
6	0.3348	0.0039	0.0106	0.4349	43.021	23.429	2.714	0.0456	1.749	0.0022	0.713	0.0061	0.0810	0.1123
7	0.3344	0.0039	0.0108	0.4353	43.017	23.431	2.713	0.0454	1.749	0.0020	0.713	0.0061	0.0819	0.1115
8	0.3348	0.0038	0.0110	0.4374	43.020	23.423	2.713	0.0452	1.748	0.0026	0.715	0.0060	0.0811	0.1119
9	0.3335	0.0037	0.0109	0.4368	43.024	23.420	2.713	0.0459	1.749	0.0023	0.714	0.0061	0.0823	0.1118
10	0.3329	0.0038	0.0109	0.4353	43.024	23.423	2.712	0.0453	1.749	0.0022	0.715	0.0060	0.0820	0.1132
11	0.3343	0.0040	0.0106	0.4348	43.021	23.440	2.713	0.0455	1.750	0.0020	0.715	0.0060	0.0808	0.1123
Avg %	0.3337	0.0038	0.0108	0.4360	43.023	23.429	2.713	0.0453	1.749	0.0023	0.714	0.0059	0.0813	0.1120
% std dev	0.00074	0.00014	0.00026	0.0009	0.0040	0.0059	0.0004	0.0003	0.0013	0.0006	0.0009	0.0001	0.0005	0.0006

Table 2a: Short term precision test on a NiFeCr high temperature alloy using 20 seconds counting time (3500W).

Elem	Si	S	P	Mn	Ni	Cr	Mo	V	Cu	W	Ti	As	Sn	Co	Al	Sb
	%	%	%	%	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
1	0.504	0.0182	0.0218	0.81	9.19	18.337	100	90	251	206	16	49	38	344	385	33
2	0.502	0.018	0.0218	0.811	9.189	18.365	99	93	249	210	19	50	38	345	388	35
3	0.504	0.0184	0.0222	0.811	9.189	18.341	100	97	251	216	17	51	37	357	378	36
4	0.503	0.0182	0.0221	0.811	9.187	18.354	100	92	250	209	17	48	37	347	388	30
5	0.503	0.0181	0.0221	0.811	9.179	18.352	100	94	251	207	18	49	37	344	386	33
6	0.504	0.0183	0.0222	0.811	9.189	18.348	99	95	252	206	16	46	36	349	383	31
7	0.504	0.0182	0.0221	0.811	9.186	18.346	101	95	255	214	20	46	37	346	380	37
8	0.504	0.0181	0.0222	0.812	9.18	18.353	100	98	252	205	21	49	39	348	374	34
9	0.503	0.018	0.0224	0.81	9.194	18.349	100	87	257	209	20	49	38	345	378	29
10	0.502	0.0183	0.0222	0.811	9.19	18.345	99	97	251	208	19	49	36	348	384	38
11	0.502	0.0181	0.0223	0.811	9.189	18.343	101	92	253	210	14	48	36	344	377	40
Avg %	0.503	0.0182	0.0221	0.811	9.188	18.349	100	94	252	209	18	49	37	347	382	34
% std dev	0.0008	0.00012	0.00018	0.0007	0.0035	0.0075	6	3	24	4	2	1	1	4	5	3

Table 2b. Short term precision test on a stainless steel using 20 seconds counting time (3500W).

	Ni	Co	Mo	Ti	Cu	Al	Cr	Si	Mn	V	Ta	Zr	Ca
	%	%	%	%	%	%	%	%	%	%	%	%	%
Aug 9	18.343	9.198	4.789	0.658	0.216	0.1286	0.1077	0.0719	0.0366	0.0300	0.0161	0.0143	0.0039
Aug 9	18.345	9.203	4.793	0.657	0.217	0.1280	0.1076	0.0718	0.0366	0.0302	0.0149	0.0143	0.0037
Aug 9	18.340	9.203	4.792	0.656	0.217	0.1283	0.1085	0.0730	0.0367	0.0302	0.0160	0.0144	0.0035
Aug 9	18.345	9.199	4.787	0.655	0.216	0.1276	0.1082	0.0741	0.0361	0.0307	0.0156	0.0143	0.0038
Aug 9	18.346	9.200	4.790	0.655	0.216	0.1289	0.1078	0.0752	0.0362	0.0306	0.0142	0.0142	0.0037
Aug 10	18.340	9.198	4.791	0.656	0.216	0.1276	0.1083	0.0759	0.0361	0.0307	0.0149	0.0142	0.0033
Aug 10	18.340	9.194	4.789	0.655	0.216	0.1300	0.1082	0.0772	0.0365	0.0303	0.0151	0.0144	0.0034
Aug 10	18.336	9.193	4.788	0.655	0.216	0.1293	0.1076	0.0787	0.0360	0.0302	0.0142	0.0142	0.0037
Aug 10	18.330	9.200	4.790	0.655	0.216	0.1286	0.1083	0.0799	0.0364	0.0306	0.0148	0.0143	0.0035
Aug 10	18.343	9.200	4.790	0.655	0.216	0.1285	0.1081	0.0805	0.0365	0.0306	0.0140	0.0143	0.0037
Aug 10	18.341	9.201	4.792	0.657	0.216	0.1282	0.1089	0.0814	0.0368	0.0306	0.0149	0.0144	0.0036
Aug 10	18.336	9.192	4.790	0.655	0.216	0.1290	0.1078	0.0827	0.0356	0.0298	0.0157	0.0144	0.0038
Aug 10	18.338	9.194	4.792	0.652	0.217	0.1267	0.1078	0.0833	0.0364	0.0304	0.0150	0.0143	0.0035
Aug 10	18.333	9.202	4.795	0.655	0.216	0.1283	0.1083	0.0847	0.0368	0.0303	0.0155	0.0142	0.0037
Aug 11	18.337	9.191	4.791	0.656	0.216	0.1280	0.1088	0.0849	0.0364	0.0302	0.0167	0.0142	0.0034
Aug 11	18.337	9.197	4.793	0.658	0.216	0.1289	0.1088	0.0864	0.0359	0.0296	0.0149	0.0141	0.0033
Aug 11	18.339	9.200	4.793	0.657	0.215	0.1295	0.1082	0.0877	0.0365	0.0299	0.0144	0.0142	0.0036
Aug 11	18.343	9.196	4.793	0.655	0.216	0.1290	0.1081	0.0888	0.0365	0.0301	0.0155	0.0142	0.0040
Average %	18.339	9.198	4.791	0.656	0.216	0.1285	0.1082	0.0799	0.0364	0.0303	0.0151	0.0143	0.0036
% std deviation	0.0042	0.0036	0.002	0.0013	0.0004	0.0008	0.0004	0.0055	0.0003	0.0003	0.0007	0.0001	0.0002

Table 3: Precision test over 38 hours using fixed channels on a high alloy steel sample.

Accuracy of analysis

The accuracy of analysis can be assessed by measuring steel certified reference materials (CRM) and comparing the found results with the recommended values of the certificate. Table 4 shows such a comparison for five different steel alloys.

It should be stressed that an XRF spectrometer is a very accurate comparator, but the accuracy of the final analysis is entirely dependent on the quality of the standards used for calibration and on the care and reproducibility of sample preparation which must be identical for CRMs and for routine samples as well.

	Low alloy steel NIST 1763b			Manganese steel BAS 493/3			Nimonic 901 BAS 387/1			Maraging steel BS 161A			Tool steel BS32c	
	%	*certif %*		%	*certif %*		%	*certif %*		%	*certif %*		%	*certif %*
Mn	1.63	***1.61***	Mn	11.12	***11.15***	Ni	41.2	***41.2***	Ni	18.35	***18.4***	W	6.24	***6.3***
Si	0.627	***0.628***	Ni	3.25	***3.24***	Cr	11.20	***11.35***	Co	9.21	***9.22***	Mo	4.83	***4.85***
Ni	0.505	***0.508***	Mo	0.99	***1.04***	Mo	5.85	***5.83***	Mo	4.79	***4.82***	Cr	3.86	***3.98***
Cr	0.500	***0.504***	Si	0.868	***0.861***	Ti	3.04	***3.00***	Ti	0.66	***0.65***	V	1.98	***2.03***
Mo	0.495	***0.491***	Cr	0.284	***0.259***	Al	0.22	***0.24***	Cu	0.217	***0.22***	Ni	0.34	***0.35***
Ti	0.298	***0.313***	P	0.132	***0.120***	Si	0.056	***0.06***	Al	0.13	***0.14***	Co	0.32	***0.31***
V	0.309	***0.308***	Al	0.046	***0.035***	Mn	0.016	***0.025***	Cr	0.11	***0.12***	Si	0.33	***0.29***
Nb	0.098	***0.100***	V	0.022	***0.025***	Co	0.024	***0.02***	Si	0.033	***0.032***	Mn	0.28	***0.29***
As	0.053	***0.054***							Mn	0.030	***0.031***	Cu	0.13	***0.13***
Zr	0.041	***0.045***												

Table 4: A comparison for five different steel alloys.

Conclusion

Analysis of irons and steels can be performed with ease using the ARL X900 Simultaneous-Sequential XRF Spectrometer. Appropriate calibrations for steel alloys can be delivered turnkey from the Thermo Fisher Scientific factory. In this case the commissioning time of the spectrometer is reduced to a minimum. The precision and accuracy are excellent in these matrix types for routine or R&D analysis, especially when the new high counting fixed channel monochromators are used for elements like Ni, Co and Mo.

The famous Moiré fringe goniometer can also be fitted alongside the series of fixed channels. The performance of this goniometer is such that it can be used for analysis of any elements that are not fitted as fixed channels. Analysis on the goniometer and the fixed channels are started at the same time. Hence they occur simultaneously. Obviously, the goniometer can be used as a backup in case of failure of any of the fixed channels.

Furthermore, operation is made easy through the advanced Thermo Scientific™ OXSAS™ Software which operates with the latest Microsoft Windows® package.

Learn more at **thermofisher.com/X900**