A WHITE PAPER FROM
SPECTRO ANALYTICAL INSTRUMENTS

SPECTRO

When results matter

Improved Spectrometric Analysis for Positive Material Identification (PMI)

Mobile and Portable Spectrometers for Infrastructure Integrity Testing in the Process Industries

Introduction

Explosions, fires, and other incidents at oil refineries, petrochemical complexes, power plants, and similar installations receive wide and often sensational publicity, particularly when there is loss of life or personal injury. Apart from the human cost, financial losses to operators and insurers can run to many millions of dollars. Often described as "accidents," these incidents are not infrequently traced to the use of piping, valves, and similar components made of inappropriate materials.

In many cases, the presence or absence of a particular alloying element in a steel component can be critical to its performance, but impossible to detect by a physical inspection of the item. In recent decades, positive material identification (PMI) has become accepted practice in the process and equipment supply industries.

Modern spectrometric analyzers are vital components for the performance of PMI in the demanding process plant environment.

AMETEK®
MATERIALS ANALYSIS DIVISION

The Role of PMI

A few examples of situations that can arise from the use of inappropriate materials, sometimes with disastrous results, will illustrate the need for PMI.

Hydrogen gas is frequently encountered in petroleum and chemical processes. However, at high temperatures and pressures, it can cause high temperature hydrogen attack (HTHA) in steel piping and other components. If undetected, this can lead over time to component failure — with fire and explosion the likely result. At elevated temperatures (above about 400 °F (204 °C)), hydrogen atoms can readily diffuse into carbon steels. The diffused hydrogen reacts with the carbon in the steel to form methane gas. This can accumulate at grain boundaries, weakening the steel and leading to cracks, fissures, and ultimately component failure. There are well-documented cases where HTHA has led to catastrophic component failure due to the incorrect installation of a low-alloy steel component. The use of steel alloys containing elements such as chromium, which has a stabilizing effect on the iron carbides, can effectively prevent HTHA.

Another example: ultrapure water would not normally be thought of as particularly corrosive. But under certain conditions, it can be responsible for a mechanism known as flow-accelerated corrosion, or FAC. This has been experienced particularly in power generation, where ultrapure water and wet steam run through condensate lines in cooling systems. The factors giving rise to FAC are complex, but essentially, when hot water or steam with a low oxygen content flows along a carbon steel pipe, a protective oxide film (Fe_3O_4) normally found on the steel surface can be dissolved. (In a chemical reaction, the metal actually creates corrosion to regain this oxide.) Over time, metal is gradually eroded, leading to thinning and weakening of the pipe — and ultimately to failure. FAC has been responsible for several high-profile accidents in the nuclear power industry. Like HTHA, it can be prevented by the use of steel alloyed with chromium.

These two examples might suggest that chrome steels are always the answer to chemical plant corrosion. The situation is more complex. Type 316 stainless steels, widely used in the construction of chemical plants, all contain chromium. However, their mechanical strength and durability depend also on their carbon content. Type 316 stainless steel contains up to 0.07% carbon, whereas 316L contains a maximum of only 0.03%. This small absolute difference is enough to give the alloys clearly different intergranular corrosion behavior. Welded seams formed with low-carbon 316L are more durable than welds with the higher-carbon alloy.

Other elements besides carbon can also play a critical role. In a third example,

General corrosion (flow-influenced) (Photo courtesy of the Energy Institute, London)

sulfidation corrosion can occur when some grades of steel piping are exposed to process fluids containing hydrogen sulfide (H_2S), or to hydrogen-free (H_2-free) fluids containing certain hydrocarbons with sulfur compounds. Chromium is certainly one answer: generally, the lower the chromium content, the faster the corrosion. However with carbon steels, the silicon content is also important. In the presence of those H2-free hydrocarbons with sulfur compounds, carbon steels with silicon content below 0.1% experience a high rate of corrosion. Steel grades with higher silicon levels experience much lower corrosion rates.

To prevent corrosion or other adverse developments, PMI plays a vital role not only during the construction, operation, and maintenance of process plants, but at all stages of the plant equipment supply chain. Incorrect identification of raw materials, poor record keeping, or missing documentation could all result in the use of inappropriate components. Even when the correct materials are used, traceability and validation of the materials used in formulation and synthesis processes are required parts of good manufacturing practices (GMP) in tightly regulated industries such as pharmaceuticals and foods.

The Challenge

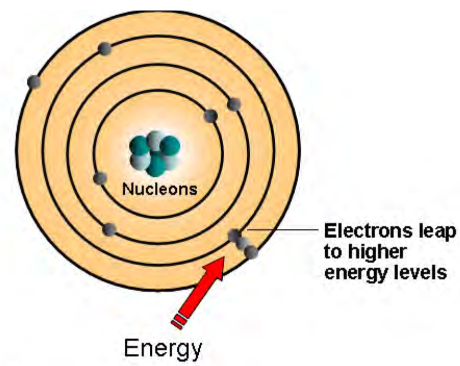
The industry-standard approach for achieving efficient PMI is via elemental analysis of materials. Traditional analytical techniques involve taking a process sample and subjecting it to analysis in a laboratory. This always involves delays and expense, and may well be difficult or impossible while a process plant is in operation. Shutting down an oil refinery to take a metal sample is hardly a realistic proposition.

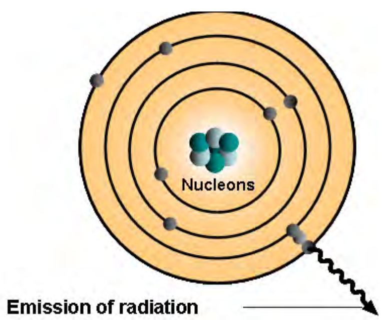
The ideal analytical technique for PMI should be fast, simple to use on-site, and preferably nondestructive, so as not to compromise the integrity of the component tested. The ability to test components while the plant is operational, which may involve coming into contact with piping surface temperatures of several hundred degrees, would be an added bonus. Minimal sample preparation should be required.

Instrumental Solutions

The right handheld X-ray fluorescence (XRF) spectrometer can satisfy most PMI analysis requirements. Example: the SPECTRO xSORT handheld analyzer employs the latest XRF technology to provide a comprehensive, easy-to-use solution for numerous process industry PMI applications.

For even more challenging requirements — such as the measurement of nitrogen or boron, or of carbon as in the stainless





The spectrometric principle in action: X-rays excite the inner electrons, which emit characteristic energy as they return to normal.

steel type differentiation above — the answer may be larger, more powerful, but still field-deployable models such as the SPECTROTEST mobile analyzer or the SPECTROPORT portable analyzer. Both are based on optical emission spectrometry (OES), and both are fully at home in process industry PMI working conditions.

All three analyzers are manufactured by SPECTRO Analytical Instruments, and employ the latest technologies in their respective techniques. Together, they provide a comprehensive solution for PMI.

XRF and OES instruments can be used by operators without analytical expertise to return accurate analyses of even complex alloys in seconds rather than minutes or hours, and to deliver reliable alloy identification.

Both techniques work on the spectroscopic principle, which relies on the internal atomic structure of the material being analyzed. The atoms of the sample are excited by an external source of energy, which is absorbed by and raises the energy level of the electrons in the sample atoms. This excited state is unstable. So the electrons rapidly return to their normal state, emitting energy as they do so.

This emitted energy, or emission spectrum, is characteristic of the elements contained in the sample. Its intensity is proportional to their concentration. The two techniques, XRF and OES, differ in the type of energy used to excite the sample atoms: in the former, it's a beam of X-rays; in the latter, an electric arc or spark. The relevant aspects of the instruments are covered below.

The SPECTRO xSORT Alloy and AlloyPlus

The design, performance, and simple operation of the SPECTRO xSORT handheld X-ray fluorescence spectrometer make it ideal for PMI in the petrochemical and other process sectors. Its XRF spectrometry is a well-proven technique for metals analysis, popular since its introduction in the 1950s.

The technique works by irradiating the surface of the sample with a beam of X-rays. This induces fluorescence of the atoms in the sample, which is then reemitted as X-rays of a lower energy. Each element emits X-rays of a different and unique energy or wavelength, whose intensity is proportional to the concentration of that element in the sample. The analyzer's detection technology can discriminate between the energies emitted, and measure each one's intensity — hence determining the concentration of the different elements in the sample.

SPECTRO Analytical Instruments has supplied X-ray spectrometers to the metallurgical industries for many years. That experience is built into SPECTRO xSORT. So it can discriminate quickly and easily between many alloy types, and also identify specific alloys within those groups.

Convenience and portability
The latest SPECTRO xSORT models have been optimized for fatigue-free, on-site analysis.

Complete with battery pack, the SPECTRO xSORT weighs less than 4 lb (1.64 kg) and features an ergonomically designed "pistol-grip" handle. To operate, the user grips the handle, presses the flexible X-ray safety gasket onto the surface of the test sample, and pulls the trigger. Usually little or no sample preparation is required, but where conditions might affect results (because X-ray analysis detects both surface and underlying materials), surfaces of uneven or corroded samples may be improved by grinding with a suitable abrasive. The user interface and results are displayed on an optimally positioned touchscreen. The instrument is robust, with a housing of shock-resistant ABS plastic. When not in use, it can be carried in a convenient holster.

Analytical performance
SPECTRO xSORT can provide metal grade analysis for most metals and alloys in only 2 seconds.

Of course, light elements such as silicon, phosphourus, and sulfur are traditionally more difficult to measure via XRF than heavy alloying elements like chromium and copper.

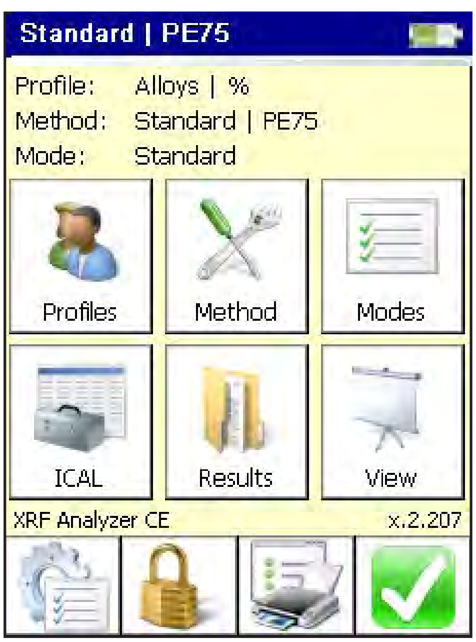
However, the latest SPECTRO xSORT models utilize a miniaturized, low-power X-ray tube rated at 50 kilovolts (kV), with a rhodium anode, providing exactly defined excitation for excellent stability and precision. This is complemented by a new high-count readout system and a high-resolution, high-sensitivity silicon drift detector (SDD). (These technologies are versions of those used in high-performance laboratory instruments like the top-of-the-line SPECTRO XEPOS analyzer.) Combined, they enable the handheld SPECTRO xSORT to furnish accurate, high-productivity spectrochemical analysis and screening of numerous elements in PMI work — including difficult-to-analyze light metals such as silicon.

For example, SPECTRO xSORT can thus differentiate between various steel alloys on the basis of their silicon content in 7 seconds.

This speed of analysis is conducive to high productivity, and to operator comfort in keeping a handheld instrument stationary on a sample, particularly in challenging plant conditions. For instance, it minimizes the time the unit (and operator) needs to spend near any high-temperature surface. So reliable measurements can be obtained from surfaces as hot as 930°F (500 °C).

Note that some XRF handhelds had difficulty in measuring light elements. Readings are at or below their limits of detection (LODs). Such instruments required a substantially longer time — perhaps 60 seconds — for reliable measurements in these cases.

Also, other instruments may require the path of the X-ray beam to be flushed with helium gas, or to be maintained in a vacuum, during the measurement. SPECTRO xSORT measures every sample — even those containing light elements — in air, with no method switching, helium purge, or vacuum.



Easy to Use

It doesn't take a skilled analyst to perform successful PMI with SPECTRO xSORT. The easy-to-understand graphical interface allows operations to be selected directly on the touchscreen using a finger or stylus.

Users may choose a complete analysis, or have the instrument compare the analysis against its extensive internal alloy library and display only the material's grade specification on the screen. Even more conveniently, in some situations SPECTRO xSORT can show a simple pass/fail message, using the first sample measured as a reference and comparing all subsequent test pieces to it.

The predefined grade library delivered with the instrument can be easily extended by adding data from the optional SPECTRO Metals Database. Customized new grade libraries may also be created. All results can be stored in the instrument, or sent to a USB drive, network, or printer as XML or PDF files, via wireless Bluetooth interface. An optional integrated camera can even add images of the sample measured.

SPECTRO Metals Database

The instrument uses stored calibrations combined with SPECTRO's unique iCAL (intelligent calibration logic) software. With any XRF instrument, these calibrations must be checked periodically against a known standard to standardize the system. SPECTRO xSORT is fitted with an automatic shutter that uniquely fulfills two purposes: to protect internal components, and to serve as sample material for the iCAL standardization. Any calibration adjustments are made automatically. No separate sample or tedious operator standardization routine is required.

AISI 304			
Element	Measurement time: 2 Sec		
	Certified Value [%]	Average Value [%]	2 * SD [%]
Mn	1.48	1.46	0.048
Cr	18.37	18.33	0.10
Mo	0.20	0.21	0.014
Ni	8.09	8.06	0.13
Cu	0.40	0.43	0.027

AISI 316			
Element	Measurement time: 2 Sec		
	Certified Value (%)	Average Value [%]	2 * SD [%]
Mn	1.78	1.89	0.051
Cr	16.56	16.60	0.075
Mo	2.11	2.18	0.025
Ni	10.38	10.41	0.123
Cu	0.17	0.19	0.011

Differences in the molybdenum content clearly distinguish the two alloys.

Corrosion by an acidic process chemical (scale inhibitor) (Photo courtesy of the Energy Institute, London)

Results of PMI With SPECTRO xSORT

The previous results illustrate the ability of SPECTRO xSORT to distinguish between different alloys. In each case, the average values quoted are based on three measurements, but a single measurement would suffice to identify the alloy. SPECTRO xSORT calculates and displays the result's calculated error automatically, if required. The unit doesn't display all analytical results unless instructed, given its ability to identify the alloy automatically.

As mentioned, minor differences in composition can significantly affect the performance of different alloys. One of the most popular steels for chemical plant construction is Type 304 stainless, the standard austenitic chrome-nickel steel alloy. Considered practically indestructible and characterized by high corrosion resistance, it's especially suitable for vessels and piping that contain aggressive liquids at high temperature and pressure. However, it's not a universal solution: hot acetic acid causes accelerated corrosion of Type 304 steel. Type 316 stainless steel, with a higher molybdenum content, is more resistant to this corrosion. Fortunately, SPECTRO xSORT can differentiate between these alloys easily.

[Note: Concentration values in the tables below are expressed as percentages; the precision of the measurement is expressed by SD or 2*SD, also in percent concentration. SD is the standard deviation of a number of measurements, and indicates the spread of the data about the average (or mean) result. The smaller the SD, the better the precision of the measurement. 2*SD, also known as "two sigma," is twice the standard deviation; it indicates that 95% of readings will fall within this range.

In the examples given, these values are calculated on the basis of three separate measurements, but SPECTRO xSORT can also use the data collected during a single measurement to calculate an SD. Clearly any concentration reported that is of the same magnitude as the measurement error will be unreliable — which leads to the concept of limit of detection (LOD): the lowest concentration that can be reliably measured. This is conventionally three times the SD. SPECTRO xSORT can be set up to either display the < symbol, or not to report a result, if it detects a concentration lower than the LOD.]

Survival in most chlorine environments requires alloys with a higher content of chrome and molybdenum. In addition, a high level of tungsten, as in alloys C-22 and C-276, results in good resistance to pitting and crevice corrosion.

Alloy C-22			
Element	Measurement time: 2 Sec		
	Certified Value [%]	Average Value [%]	2 * SD [%]
Cr	21.32	21.48	0.090
Mo	13.02	12.76	0.075
W	2.87	2.93	0.035
Fe	3.63	3.54	0.055
Co	0.59	0.57	0.033
Ni	57.30	57.96	0.084

Alloy C-276			
Element	Measurement time: 2 Sec		
	Certified Value [%]	Average Value [%]	2 * SD [%]
Cr	15.70	15.74	0.052
Mo	15.70	15.44	0.109
W	3.25	3.27	0.019
Fe	5.25	5.09	0.052
Co	0.23	0.23	0.046
Ni	59.00	59.13	0.53

AISI 321			
Element	Measurement time: 2 Sec		
	Certified Value [%]	Average Value [%]	2 * SD [%]
Mn	1.52	1.52	0.061
Cr	17.45	17.46	0.11
Mo	0.36	0.36	0.018
Ni	9.42	9.39	0.064
Ti	0.63	0.62	0.036
V	0.13	0.13	0.020
Cu	0.30	0.32	0.019

The significant elements in these alloys are heavy elements, easily determined by many XRF instruments. Lighter elements may be more difficult to measure, and it is here that the superior performance of SPECTRO xSORT becomes apparent. AISI 321 is a general-purpose austenitic stainless steel, with added titanium to reduce intergranular corrosion. With its high signal throughput, SPECTRO xSORT can reliably determine titanium and differentiate between 321 and 304 in only 2 seconds.

AISI 303			
Element	Measurement time: 2 Sec/additional 5 Sec for Si and S		
	Certified Value [%]	Average Value [%]	2 * SD [%]
Si	0.63	0.74	0.10
Mn	1.87	1.84	0.031
S	0.38	0.32	0.024
Cr	17.35	17.43	0.07
Mo	0.58	0.58	0.015
Ni	8.64	8.62	0.085
V	0.11	0.11	0.022
Cu	0.51	0.53	0.029

Type 304 austenitic chrome-nickel steel, while offering good corrosion resistance, is difficult to machine. Type 303 austenitic chrome-nickel steel is excellent for machining, and has a lower corrosion resistance. Cause: different sulfur contents. Type 304 contains a maximum of 0.03% sulfur, whereas 303 contains a minimum of 0.15%. The measurement of sulfur is even more of a challenge for XRF technology than the measurement of silicon, but with a slightly longer measuring time of only 7 seconds, SPECTRO xSORT AlloyPlus can identify the higher sulfur content and separate the 303 and 304 grades.

1117						
Measurement time: 2 Sec						
	Mn	Cr	Ni	Mo	Cu	Fe
1	1.03	0.064	0.051	0.016	0.063	98.4
2	1.04	0.071	0.054	0.017	0.066	98.6
3	1.04	0.066	0.037	0.017	0.044	98.6
4	1.01	0.077	0.056	0.013	0.052	98.6
5	1.06	0.076	0.051	0.017	0.064	98.6
6	1.03	0.073	0.051	0.01	0.051	98.6
7	1.08	0.066	0.049	0.017	0.059	98.6
8	1.04	0.077	0.042	0.017	0.041	98.6
9	1.06	0.073	0.056	0.011	0.055	98.6
10	1.02	0.073	0.054	0.02	0.066	98
Average Value [%]	1.04	0.072	0.05	0.015	0.056	98.5
2 * SD [%]	0.02	0.005	0.007	0.003	0.009	
Given	1.07	0.076	0.042	0.016	0.085	

Analysis of Cr, Cu, Mo in a carbon steel for FAC applications

1117				
Element	Measurement time	Analysis [%]	2 * SD [%]	Given [%]
Si	5s	<0.08		0.05
	10s	<0.056		
	20s	0.066	0.012	
	30s	0.076	0.01	
Mn	2s	1.04	0.02	1.07
Cr	2s	0.072	0.005	0.076
Ni	2s	0.05	0.007	0.042
Mo	2s	0.015	0.003	0.016
Cu	2s	0.056	0.009	0.085
Fe	2s	98.5		

Quick grade ID and analysis of low Si in a carbon steel (analysis is done out of 5 repeats)

For cases where such light-element analysis requires utmost precision, the instrument now delivers dependable results in only 22 seconds. But for most users, its most significant improvement comes in the routine grade identification and sorting necessary for the great majority of analyses. Here, the new model can determine low silicon levels with twice (2X) the precision of previous models — and in half the time. Thus sorting and identification of light-element alloys can be accomplished in only 7 seconds.

In addition, precision in determining other alloying elements — including corrosion-inhibiting chromium — improved 3X to 5X over previous models. As is the case with most alloys, lower chromium content levels in steel were accurately measured in only 2 seconds.

SPECTROTEST and SPECTROPORT

As the above results show, SPECTRO xSORT can handle most process industry PMI tasks. However, some cases may require more precise analysis, or measurements of elements outside the scope of XRF technology (such as carbon, boron, or nitrogen), for comprehensive PMI.

These circumstances may call instead for use of the SPECTROTEST mobile arc/spark optical emission spectrometry (OES) analyzer, or its smaller, lighter relative: the SPECTROPORT portable arc/spark OES metals analyzer. Compared to SPECTRO xSORT, both offer larger but still field-deployable form factors, combined with superior analytical performance.

Both SPECTROTEST and SPECTROPORT analyzers utilize the OES principle. In this technique, the atoms in the sample are excited not by X-rays but by an electric arc or spark, so that each element emits light of characteristic wavelengths in ultraviolet and visible regions of the spectrum. The arc or spark is generated at a sample probe on a flexible umbilical cord up to 8 meters (m) long. As with SPECTRO xSORT, the operator simply places the probe in contact with the sample to take a measurement. (Because metal atoms are expelled during an ark spark measurement, a small burn mark occurs on the sample surface.) Light emitted by the sample is transferred via fiber optic to the optical system, where it is separated into its different wavelengths using a diffraction grating. Individual intensities are then measured with a suitable detector.

SPECTROTEST and SPECTROPORT both employ a state-of-the-art detector: highly sensitive, fast, multiple CCDs deliver high-speed analysis and generate high-quality data. This enables the same sophisticated approach to data handling as in SPECTRO xSORT. So all three instruments use unique iCAL procedures, and can identify and verify´alloys automatically in seconds.

AISI 316			
Element	Measurement time: 10 Sec		
	Certified Value [%]	Average Value [%]	2 * SD [%]
C	0.042	0.045	0.002
Si	0.26	0.29	0.013
Mn	1.25	1.17	0.024
P	0.022	0.019	0.004
S	0.020	0.021	0.003
Cr	16.64	16.16	0.174
Mo	2.11	2.05	0.067
Ni	10.66	10.80	0.119
V	0.13	0.13	0.007
Cu	0.09	0.11	0.004

AISI 316L			
Element	Measurement time: 10 Sec		
	Certified Value [%]	Average Value [%]	2 * SD [%]
C	0.019	0.022	0.001
Si	0.44	0.46	0.004
Mn	1.21	1.18	0.012
P	0.026	0.023	0.003
S	0.020	0.023	0.003
Cr	17.36	17.05	0.047
Mo	2.11	2.04	0.001
Ni	11.86	12.09	0.082
V	0.05	0.06	0.001
Cu	0.09	0.11	0.004

Duplex Alloy 1.4462 (X2CrNiMoN 22-5-3)			
Element	Measurement time: 10 Sec		
	Certified Value [%]	Average Value [%]	2 * SD [%]
C	0.013	0.019	0.001
Si	0.47	0.55	0.011
Mn	1.74	1.62	0.016
P	0.023	0.014	0.004
S	0.002	<0.002	
Cr	22.41	21.64	0.046
Mo	2.89	2.64	0.020
Ni	6.01	5.88	0.076
V	0.15	0.12	0.001
N	0.103	0.139	0.019

Results of PMI with SPECTROTEST and SPECTROPORT

As mentioned, the only difference between Type 316 and Type 316L stainless steel is carbon content: the 316 alloy contains up to 0.07% carbon; the 316L, a maximum of 0.03%. Both SPECTROTEST and SPECTROPORT can easily differentiate between them. (See SPECTROTEST results at left; SPECTROPORT results would be similar.)

Nitrogen is yet more difficult to analyze — beyond the capabilities of xSORT or even SPECTROPORT. However, SPECTROTEST meets the challenge. It can conduct on-site identification of austenitic (duplex) steels alloyed with nitrogen. Nitrogen stabilizes the austenitic structure without decreasing durability. In high-alloy, chemically resistant steels, it also somewhat increases resistance to corrosion, especially pitting corrosion. Among duplex steels, the X2CrNiMoN 22-5-3 (1.4462) grade has found wide acceptance. The nitrogen content in duplex steels usually ranges from 0.10% to 0.22%.

Conclusion

PMI has become an essential part of good practice and risk reduction in the process industries. Elemental analysis of alloy samples — including previously difficult-to-measure light elements — can improve efficiency and add value to PMI efforts. The SPECTRO xSORT handheld XRF analyzer, SPECTROPORT portable OES analyzer, and SPECTROTEST mobile OES analyzer provide a complete solution. They offer accurate, rapid, and reliable alloy identification on site for PMI in this demanding sector.

Choosing a handheld XRF analyzer

Handheld XRF spectrometers are not created equal. Make sure the instruments you consider can meet the needs of your specific PMI tasks with the right mix of proven performance, innovative features, and tested convenience. Look for the following benefits:

Field-proven performance and speed. Consider models that have proved they can perform well in challenging plant or field locations. One key for highly reliable yet high-volume PMI: the ability to deliver dependable results in seconds.

Operating flexibility. Some older models require time-consuming procedures such as switching analytical methods between samples, or demand helium purges or vacuum for accurate operation. Find an instrument that lets you analyze the alloys you need: simply, easily, and quickly.

Documentation/connection flexibility. Why get stuck with limited choice of results formats to document compliance? Flexible SPECTRO xSORT lets you save results in different formats at different destinations simultaneously. Save to USB drive, network, or printer as XML or PDF, and (via an integrated camera) combine with images of the sample measured.

Easy standardization and built-in protection. Try to find instruments that avoid tedious multiple-sample standardization. Example: SPECTRO xSORT provides unique one-sample, one-time standardization. The shutter even functions as the system's standardization sample, while also offering built-in protection of detector and tube, even when analyzing light elements.

Large metals database. Choose devices that can easily accommodate new alloys (e.g., with light elements) or materials. For instance, SPECTRO xSORT lets you extend prepackaged libraries and/or create new customized grade libraries.

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