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A WHITE PAPER BY
SPECTRO ANALYTICAL INSTRUMENTS

Surprising New Capabilities of ED-XRF Technology

Abstract

In this paper, industry experts will outline ED-XRF spectrometers' technological evolution; their comparison to more costly WD-XRF devices (as well as to lower-end ED-XRF models); and the current advantages users worldwide find in the best ED-XRF analyzers. These include major benefits in sensitivity and precision, cycle time and throughput, ease of use, compactness, and cost of ownership.

The paper will also discuss how a high-performance ED-XRF spectrometer (example: SPECTRO XEPOS) provides high-quality measurement of most elements in the periodic table, from uranium (U) to fluorine (F). So it can offer effective solutions for a wide array of both routine and challenging real-world analytical applications, including petrochemicals, chemicals, environmental and geological samples, clinker/cement/slag, cosmetics, food, pharmaceuticals, and many more.



Dr. Joachim Heckel

Introduction

ED-XRF analyzers were once known as “poor man’s X-ray fluorescence spectrometers.” Their relatively low price was matched by underdeveloped technology and comparatively low performance. They were considered suitable mostly for qualitative analysis or analyzing a few simple sample matrices. Some standard models still operate at this level.

So if you could shop for X-ray spectrometers in the supermarket, for example, some more demanding users would automatically head for the WD-XRF aisle.

But today, that would be a mistake. In recent years, a truly surprising amount of improvements have been achieved in high-end ED-XRF technologies and applications — so that, for the majority of people in the market, the most advanced ED-XRF spectrometers now represent the best buys in the store.

(This report relies extensively on information graciously supplied by Dr. Joachim Heckel, now senior consultant and formerly director of research and development for SPECTRO in Kleve, Germany; Alexander Seyfarth, Global XRF Technology Manager for SGS North America Natural Resources Geochemistry Group in Lakefield; and Mario Van Driessche, owner of X-Ray Services BV and consultant on spectroscopic analysis for the petrochemical and automotive industries as well as for analytical instrument makers, in Ertvelde, Belgium. Naturally, they hold no responsibility for any errors.)



Alexander Seyfarth



Mario Van Driessche

Background

In *X-ray spectrometry*, the instrument begins by sending radiation from its X-ray tube into the sample. When the X-rays hit an atom in the sample, they ionize it. This causes the atom to eject an electron from its inner shells.

An outer-shell electron drops down to fill the gap. This movement emits fluorescence radiation. The energy of that radiation equals the energy difference between the two electron shells. And that difference is unique for every element. Thus you can identify each element by its characteristic energy.

Wavelength-dispersive X-ray fluorescence (WD-XRF) spectrometers, the earliest type of X-ray measurement analyzers, were developed in the 1950s. A modern WD-XRF operates in much the same manner as its predecessors. Portions of the signal emitted when the sample is excited by X-rays are directed toward a set of analyzer crystals using collimators. These crystals diffract the radiation depending on its wavelength, like light passing through a prism. Two or more detectors are used to sequentially detect each analyte's characteristic signal. Most modern WD-XRF instruments utilize this sequential technology. A small number of high-end models instead possess a tuned crystal/detector set for each element, and therefore can analyze multiple elements at the same time.

However, these instruments still can analyze only the preselected elements for which they have channels. Sequential units can analyze the content of elements in the range from beryllium to uranium — but, by definition, only one at a time.

WD-XRF spectrometers are expensive, complicated devices. Due to signal losses from diffraction and a long beam path, a higher-power (3-4 kW) excitation tube is needed. This requires extensive cooling. A supply of compressed gas is also necessary for one of the detectors; sometimes compressed air for the sample movement must be provided as well. Finally, the hardware as a whole is complex, with many moving parts.

Energy-dispersive X-ray fluorescence (ED-XRF) spectrometers were developed in the 1980s, and have undergone continuous development. In today's ED-XRF, the X-ray energy emissions from the sample are collected by a semiconductor detector. The different energies are sorted, and then processed at high speed in a multichannel analyzer. The resulting spectrum shows the intensity of each element's peaks in counts per second on the y-axis, and the emission energies on the x-axis.

Today, the best ED-XRF analyzers deliver multi-elemental analysis of major, minor, and trace element concentrations with very good resolution, even in samples of unknown composition — liquid, powder, or solid. They require no external chilling equipment. Low-volume helium purging is used only when required for light elements in liquids and powders, with a vacuum system for solid samples. The most advanced ED-XRF spectrometer typically requires half the purchase price of a top-flight WD-XRF, and is significantly less expensive in terms of ongoing operation and maintenance.

At a basic level, any spectrometer is designed to provide qualitative data — determining what elements are present in the sample. In addition, ED-XRF technology

using traditional direct excitation has long been applied to deliver quantitative data — measuring how much of each element is present — for single-element applications such as analyzing sulfur (S) content in mineral oil or at most for analyzing a handful of elements. Many standard ED-XRF units are still designed for these levels of functionality.

But now, the best modern ED-XRF spectrometers can also determine the concentrations of multiple elements present in the sample, delivering quantitative data on each. Employing newer quasi-monochromatic excitation for different energy ranges, an advanced ED-XRF spectrometer such as SPECTRO XEPOS can handle complex interferences and deliver concentration values starting from sub-ppm levels.

ED-XRF Versus WD-XRF: a Shifting Balance

Factors like their high cost and greater complexity aside, WD-XRF analyzers once enjoyed superiority over ED-XRF units in a number of performance areas. Years ago, a top-of-the-line WD model possessed lower limits of detection (LODs), greater resolution, and better precision, as well as higher sample throughput rates and shorter measurement times than its ED-XRF counterpart.

That balance has decisively shifted (as the rest of this report makes evident) when comparing today's leading ED-XRF models with sequential WD-XRF instruments, which make up the major portion of the WD-XRF market.

Note that WD-XRF, especially simultaneous WD-XRF, remains the recommended choice for a few analytical tasks.

“Cement and steel, that’s still typically a WD application,” says Mario Van Driessche. “They use simultaneous WD, so you have a very fast analysis of major elements.”

“Nickel laterites has been another exception,” adds Alexander Seyfarth. Reason: the composition of such samples can be complex. They may contain high percentages of iron (Fe) with a percentage of nickel (Ni), but also low amounts of cobalt (Co) — perhaps 100 to 400 ppm. In an ordinary spectrometer analyzing these ores, the primary peak for cobalt is overlapped by the neighboring peak for iron. And the secondary peak for cobalt is overlapped by the neighboring peak for nickel. It’s easier for a WD-XRF analyzer to distinguish these particular closely neighboring peaks.

However, the best modern ED-XRF instruments can now address these challenges. Their detectors supply high count rates, which are induced in the sample: an important factor. But that’s not the whole story. In an application such as laterites, even models with high count rates may still deliver spectra with unsatisfactory differentiation.

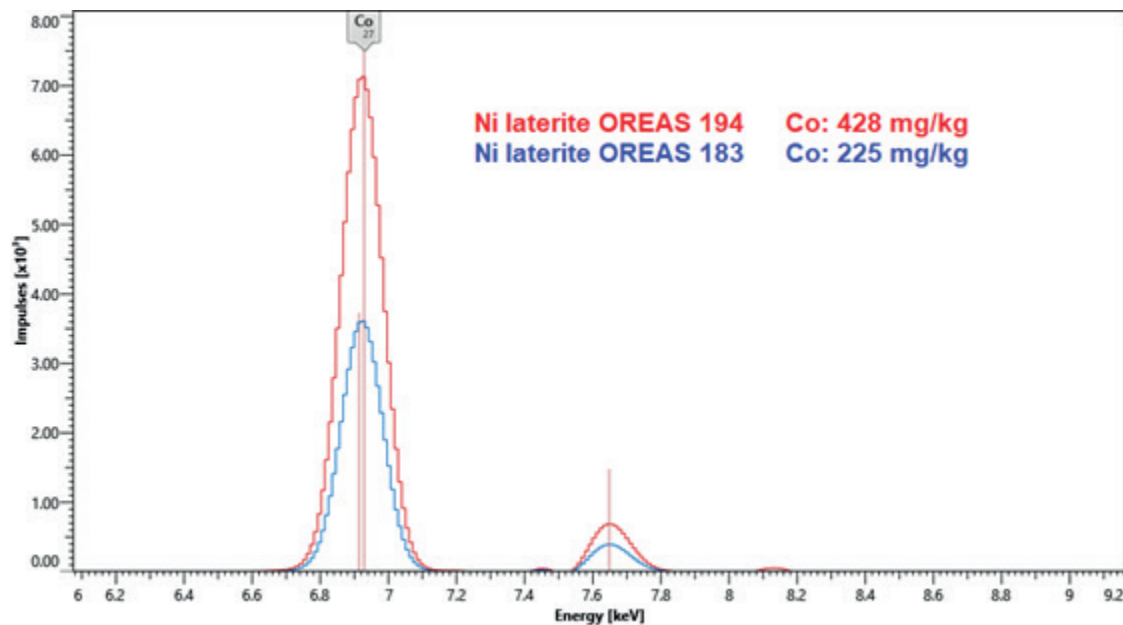
The key is the addition of sophisticated software optimized to resolve these problems. Recently, using technologies known from photographic image processing, at least one model has been able to provide extremely stable peak positions and resolution in these demanding circumstances.

Examining a recent test run spectra, Alexander Seyfarth had one word for the clarity of its cobalt peaks:

“Wow!”

His later, somewhat lengthier verdict: “When looking at cobalt, the SPECTRO XEPOS processing software demonstrates how an excellent signal can be read out of the spectrum when measuring a laterite sample.”

“And older decision-makers sometimes claim that energy-dispersive is not as quantitative as wavelength-dispersive, which once was and now is absolutely not true,” says Mario Van Driessche. “Some users still think there’s a resolution issue, or a limits of detection issue with energy-dispersive. Not everybody is aware about high-end ED-XRF’s recent detector improvements, and the improvements of the instrument in general. But the new generation of lab managers do their



Challenging applications aside, a choice for WD-XRF analysis also may be mandated by legacy laws, regulations, or standards. (See “Approved standards” below.) This is changing as standards committees and regulatory bodies catch up with realities on the analytical benchtop.

Another possible factor: inertia. Not in physics, but in human nature. “People often think that bigger and more expensive is better,” says Alexander Seyfarth. “Not so true in this case anymore.”

homework, and they see the improvements that energy-dispersive models have made.

“I have a petrochemical facility client that used a WD for most analyses — with extremely high running costs. They had a backup instrument: an older energy-dispersive instrument. They replaced their old ED system with a recent ED instrument from SPECTRO, and now, the funny thing is — they use their WD as a backup instrument.



“For most applications,” Mario Van Driessche states, “top-of-the-line ED-XRF technology has absolutely reached parity with WD-XRF. The limits of detection are better and the repeatability is at least the same.”

“Actually, I think only 20% or 25% of my customers really compare WD and energy-dispersive when purchasing,” he continues. “That’s often budget-wise: because WD is a lot more expensive. The competition between ED and WD is rather between ED and low- or medium-power WD, cheaper WD instruments.”

“And I have to say for the petrochemical industry these days, there is no difference in performance,” Mario Van Driessche concludes. “SPECTRO offers a calibration, which I normally do at the factory, of 27 elements that covers nearly all our applications. And ED-XRF is a factor of two faster. You do a complete analysis in 8 minutes; high-end WD-XRF takes about 15 minutes and low-power WD-XRF about 25 minutes. Also, the levels of detection nowadays with the best ED-XRF instruments are a factor of two better than WD instruments.”

Additionally, the simple fact is that WD-XRF technology has reached a dead end.

“For example, a WD-XRF maker cannot increase the power of their tube and reduce the optical path,” says Dr. Heckel, “because they already produce a lot of heat at the sample. Any more and they burn or cook their sample. And there’s not much improvement to be made in their detectors or electronics.”

The greatest change to WD-XRF technology in recent years has involved reconfiguring the standard WD analyzer’s feature set — and capabilities — to create a low-power, midrange benchtop version. This class of device meets a lower price point and eliminates the need for a chiller, though it is still typically priced somewhat above a top-line ED-XRF model. But slashing the traditional WD spectrometer set, number of crystals, and level of tube power also compromises the new unit’s capabilities. Result: for most points of comparison, a midrange WD-XRF performs below (sometimes well below) the level achieved by an advanced ED-XRF.

“Actually, the direction forward for WD-XRF manufacturers is probably to combine with other technologies,” Dr. Joachim Heckel says. “Some manufacturers have already combined WD with ED in one instrument, although with some issues. But in the future, in those minority cases where you need it, you might have an analyzer with two or three WD-XRF channels for your main or difficult elements, and then the rest are covered by an ED-XRF assembly.” Future possibilities aside, currently the direction for the large majority of XRF users is clear: the best ED-XRF instruments offer convincing benefits in cost and performance.

ED-XRF Advantages

Today’s most advanced ED-XRF models furnish features and benefits that far surpass the performance available at the technology’s beginnings — or indeed, possible only a few years ago.

The latest SPECTRO XEPOS provides recent technological improvements that include a silicon drift detector (SDD) combined with a new high-speed readout system that furnishes an ultra-high count rate — up to 1.5 million counts per second (cps). Traditional ED-XRF units are limited by designs that, for example, can offer only direct excitation of a sample’s atoms. But here, an innovative air-cooled X-ray tube design and unique new adaptive excitation technology plus beam conditioning optics furnish the highest possible sensitivity to target elements of choice. An optimized combination of a thick binary palladium/cobalt alloy anode X-ray tube with filtered direct excitation, excitation via a BRAGG bandpass filter, and polarized monochromatic excitation helps provide optimized performance for different elements. (The effect is similar

to having two tubes in one instrument.) Fundamentally, this new class of ED-XRF instrument delivers the fast, accurate, affordable results that many users need. (See “ED-XRF Applications” section below for partial list of relevant uses.)

Sensitivity / precision / LODs

Compared to previous models, the latest SPECTRO XEPOS ED-XRF analyzer, for example, furnishes dramatically improved sensitivity, often by 10x or more — and up to 3x better precision. So it can achieve exceptionally low detection limits (often less than 1 ppm) for critical trace amounts — including analysis of heavy elements such as cadmium (Cd). Additionally, the analyzer’s long-term stability now matches that of WD-XRF models. These qualities make it what SPECTRO evaluates as the best available ED-XRF spectrometer for applications requiring multi-element analyses of major, minor, and trace element concentrations.

“I can give you precise examples of LODs,” Mario Van Driessche reports, “because every year I calibrate both WD and ED instruments for my clients. For real-world lube oil blending applications, the SPECTRO XEPOS instrument’s latest lower limits of detection are a factor of two better than WD. For silver and cadmium — if you want the LOD figures, WD has an LOD of around 4 or 5 ppm. With the ED system, you have about 1.5 ppm — that’s better than twice as good. For low sulfur analysis, its detection limit is about a factor of three better than a high-end WD system. And for titanium in polymers, you have a factor of six better than WD!”

“Really, the most important issue has been the detector: how fast it is,” says Dr. Joachim Heckel. “The development of these SDD detectors over time now allows us to have really absolutely stable conditions. If you measure a standard today, you will get the same spectrum 1 week later, or 1 month later, or 1 year later. The peaks are at the same position at the same resolution as before, due to the absolute stability of the new electronics.”

“Based on that,” he explains, “our sensitivities are improved together with our excitation modes, using polarized radiation or monochromatic excitation to achieve low or no background signals. That low background and high count capability give you extremely good sensitivities and detection limits.”

“Even a newer ED model that just uses direct excitation is like a sports car on a racetrack,” says Alexander Seyfarth. “But with an instrument like the SPECTRO XEPOS, these new excitation modes get you four or five times better results. It’s like a Formula One racer.”

Sample throughput / measurement time / readiness

The speed of ED-XRF analyzers has seen immense improvement since the early days of the technology.

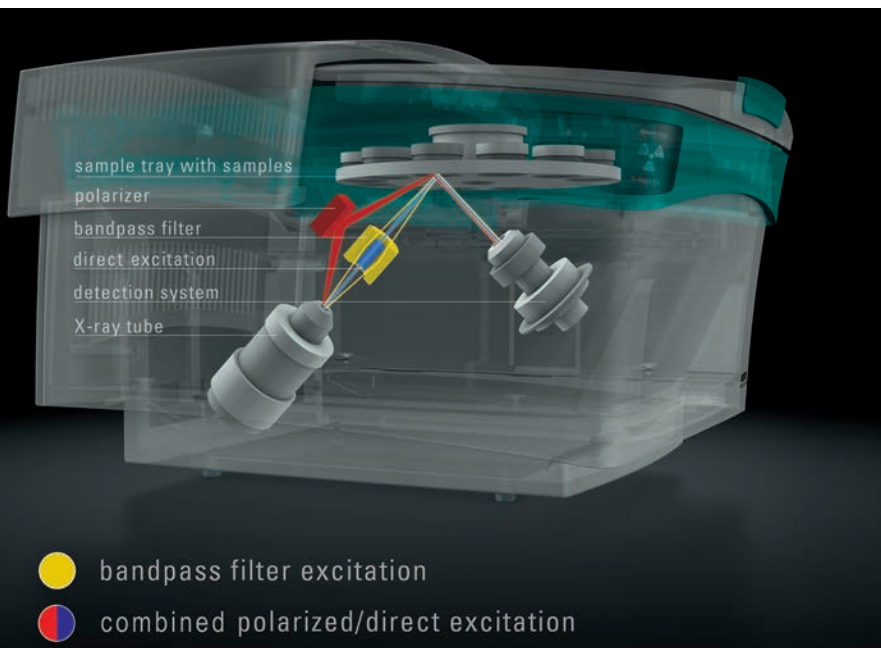
“In the beginning,” says Dr. Heckel, “we might require 1 hour measurement time per sample. For many applications, we are now at round about 3 minutes.”


“Runtime becomes fairly secondary in most of our work,” says Alexander Seyfarth. “WD-XRF is faster for a single element. But the moment I have a complex multielement sample and many elements to report to the client — the more elements I run, the better it’s going to be for a good ED-XRF in comparison. It’s throughput and cost of ownership that count.”

Ease of use / software

An advanced ED-XRF model is also easy to use. Its workflow provides proven, effective procedures for achieving accurate results in minimum time, allowing high productivity. For a new method, users first calibrate the instrument, test the precision of the analysis and the unit’s limits of detection, and validate its measurement against known reference samples.

“SPECTRO XEPOS makes things easy in ways that are particularly critical for an organization like ours, which is operating multiple instruments in a number of sites worldwide,” says Alexander Seyfarth. “Methods can be deployed remotely from a ‘master’ setup to the sites — which ensures all setups are the same. The software functionality that allows this import/export





is part of their standard delivery. Since these advanced ED systems are much closer in terms of production variation to WD units, a complete calibration can be transferred just by using stable glass samples — maybe not for pellets or powders, but certainly for metals, fusions, and liquids. This facilitates the validation, and reduces our startup/deployment time and effort.

“Factory-made packages — such as for lube oils or sulfur in oil — are also available to facilitate the startup for users beginning with XRF as a technique. Even without adding your own samples, you can get started. Though a local QC sample of the material should always be used.

“The user interface in the software can be customized with access rights to different roles,” Seyfarth continues. “This allows us to train people quickly to use the unit. Within less than 10 or 15 minutes I can get an operator to run samples, check the results, and transfer them onto our LIMS system. Having multiple languages available, and being able to easily switch between them like on a smart phone, makes support in places such as Kazakhstan easy.”

“In most cases — for a quick screening analysis, for example — sample pulverization isn’t required,” Seyfarth adds. “TurboQuant II is SPECTRO’s universal screening tool. It’s more along the lines of ‘press button get results’ than the traditional standardless programs used on WD instruments, which dominated the ‘standardless universal screening approach’ in the past. But with TurboQuant, users just place the sample into the measurement compartment and run the screening test and get meaningful results.”

SPECTRO XEPOS operating software is optimized for multi-element precision and specific application packages — while its TurboQuant II software screens unknown samples without extensive setup or degradation by matrix effects (even at low concentration levels).

“Unlike WD-based software packages, most of which struggle at low concentration ranges due to their scan approach, TurboQuant-based evaluation is very good even at those low ranges,” Alexander Seyfarth concludes.

“Broadly speaking, compared to other types of spectrometers, both WD and ED analyzers are simple to use,” says Mario Van Driessche. “Any plant person can learn it with 30 minutes’ training. I will sometimes joke to lab managers, ‘It’s so easy that even you can do it.’”

Compactness / simplicity

Laboratory space is often at a premium. So many ED-XRFs now exhibit a small-as-possible benchtop form factor.

“A small footprint is important,” says Mario Van Driessche. “Most old labs are really too small to fit all their new instruments. An ED-XRF like SPECTRO XEPOS is relatively compact. And it has related advantages. You need no extra water-cooling unit and only if your instrument is equipped with vacuum will you have to make space under the bench for the pump. And you have a limited gas consumption. Plus you need only one gas — with WD-XRF, you need two gases.”



SPECTRO XEPOS

Alexander Seyfarth agrees that lack of simplicity comes with consequences: “In a service lab in a populated area, with well-trained technicians and service people not too far away, you may be able to handle the complexity of having to deal with compressed gases, and with water chillers. But the moment you have to work somewhere else, such as a remote mine site in Africa or Australia — then life becomes difficult.

“Whereas a good ED doesn’t require the water chiller, doesn’t need compressed gas, is small enough to fit in most places — and is much more affordable.”

Lower price / cost of ownership

Even top-line ED-XRF models deliver a significantly lower cost of investment and ownership than WD-XRF spectrometers — often by a factor of two.

For instance, the advanced yet affordable SPECTO XEPOS analyzer offers cost savings at a number of points:

1. Lower analyzer purchase price, with fewer moving parts
2. No chiller needed — which contributes to reduced purchase price, as well as cutting ownership costs otherwise due to continual algae buildups and breakdown-prone pumps
3. No compressed gasses needed (except helium (He) for some liquid and loose powder analyses) — which generates substantial savings and eliminates the maintenance costs of a flow detector and compressor
4. Lower cost for associated uninterruptible power supply (UPS)
5. Lower installation costs / less installation time onsite / remote setup capability
6. Air-cooled, low-power X-ray tube (only 200 watts, versus up to 9,000 watts for high-end WDs including their required water chillers), plus small vacuum pump

The instrument's long component life cycle often extends to 5-7 years. And when components finally must be replaced, they're often less expensive: a new SPECTRO XEPOS tube is around one-third the cost of a high-power WD-XRF tube.

Bottom line: performance better than or similar to a WD-XRF in many applications, but at half the price.

This affordability is accomplished via numerous strategies. For example, a low-volume helium purging can handle light elements in liquids and powders, while a vacuum system can measure solid samples.

"An ED unit like that is good for us from a price perspective alone," says Alexander Seyfarth. "It allows us to easily double up the systems, run two machines instead of one, which we often do in many areas of the world. Because we can't afford to just stop analyzing if there's a problem. The client expects — and contracts for — high availability.

"To achieve a high level of redundancy," he says, "we often have two wavelength-dispersive units in a location, and sometimes three if we already need two for the throughput. But if you compare the pricing, that is much easier done — much less expensive — on the ED side."

ED-XRF Applications

Advanced ED-XRF technology now allows fast, accurate, efficient measurement of most elements in the periodic table, from uranium to fluorine. That makes it the ideal method for multi-element analysis of a really wide array of materials.

Success stories

Current successful ED-XRF applications

- Petrochemicals
- Chemicals
- Environmental
- Geology/minerals
- Soils
- Ceramics/refractories
- Additives in oil / lubricants
- Used oils
- Filter materials
- Wear metals / cooling liquids
- Low-sulfur fuels
- Food
- Biofuels/biomass
- Wastes
- RoHS / WEEE / ELV compliance screening
- Polymers
- Cements
- Metal alloys
- Metal alloy powders
- Steel / aluminum coatings
- Precious metals
- Ores/concentrates/tailings
- Clinker/cement/slugs
- Ferroalloys
- Pharmaceuticals
- Foods
- Cosmetics

One of the first practical ED-XRF applications, in the 1980s: incineration waste plant analysis. "The analytical challenge was to measure a lot of elements in an unknown matrix," recalls Dr. Heckel. "Previously, ED-XRF wouldn't work for this. The matrix could be inorganic material, organic material, or a mixture of both. But we were able to develop software based on a new algorithm that could do it.

And that was the ancestor of the SPECTRO TurboQuant software — which has been

much further developed, and which we're still improving to this day."

(The current version, TurboQuant II, handles a much greater range of samples — now including any type of liquids, plus solids from tree leave to plastics, granite to glass — with a single calibration.)

The petrochemical industry also used the technology for measurement of sulfur at high concentrations. An early ED-XRF success that has prevailed to the present day is analysis of oil additives in fresh lubricants. "That's a case where the highest resolution is not necessary, because you don't get overlaps in the signals for those elements," says Dr. Heckel.

"For many users, wavelength-dispersive was and is simply too expensive," says Mario Van Driessche. "Over €200,000 for an instrument in the early 1990s; way too high for smaller blending plants. But with ED-XRF we have an alternative that was less than 50% of that price — and it does a good job."

Additionally, metals industries also began using ED-XRF technology to analyze steel and slags — a use that continues strongly today.

Other current applications include a number that use pelletized samples. Crumbling of pelletized materials during analysis is unfortunately not an uncommon occurrence. In the great majority of XRF analyzers (which measure "bottom-up," as opposed to only a few "top-down" models), the resulting powder will fall into critical components below. In most WD-XRF models, that means instant contamination

of the main chamber, secondary optics, pump, and more. This necessitates disassembly and a major cleanup effort.

"But with an ED-XRF — or at least, with a SPECTRO XEPOS — you basically take a vacuum cleaner, clean the powder off the plate, and get right back to analyzing," says Alexander Seyfarth.

Additionally, detection limits are no longer an ED-XRF weakness in the great majority of applications such as mining. "Uranium and thorium was one of the earliest quantitative applications we ran exclusively on the SPECTRO XEPOS," he recalls. "Because it had hit the 10 ppm detection limit we needed.

"Now, years later, we just did an application where it could actually quantify fluorine! Not in some marketing literature, but fluorine in a fusion bead, diluted 1 to 10. If I look at that, and I look at the performance for rubidium, strontium, yttrium, zirconium, niobium, molybdenum," Seyfarth says, "basically the energy-dispersive technology leaves the wavelength models in the dust. That is, any energy-dispersive unit will leave a wavelength unit in the dust. The SPECTRO XEPOS will leave the other energy-dispersive units in the dust!"

Approved standards

Today, ED-XRF results meet a growing number of standards and regulations worldwide.

Examples of standards / test methods / technical reports currently citing ED-XRF

Test Method	Description/Title	Test Method	Description/Title
DIN 51418-1:2008-08	X-ray spectrometry - X-ray emission- and X-ray fluorescence analysis (XRF) - Part 1: Definitions and basic principles	IP 532	Determination of the sulfur content of automotive fuels - Polarized X-ray fluorescence spectrometry method
DIN 51418-2:2015-03	X-ray spectrometry - X-ray emission and X-ray fluorescence analysis (XRF) - Part 2: Definitions and basic principles for measurements, calibration and evaluation of results	GB/T17040	Determination of Sulfur in petroleum and petroleum products- Energy dispersive X-ray Fluorescence Spectrometry
GB/T 31364	Test methods for main performance of Energy Dispersive X-ray Fluorescence Spectrometer	JIS K 2541-4	"Crude oil and petroleum products—Determination of sulfur content Part 4: Energy-dispersive X-ray fluorescence method "
JIS K 0119	General rules for X-ray fluorescence analysis	ASTM F2617 - 15	Standard Test Method for Identification and Quantification of Chromium, Bromine, Cadmium, Mercury, and Lead in Polymeric Material Using Energy Dispersive X-ray Spectrometry
ASTM D4294 - 16e1	Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry	ASTM C114 - 18	Standard Test Methods for Chemical Analysis of Hydraulic Cement
ASTM D7220 - 12(2017)	Standard Test Method for Sulfur in Automotive, Heating, and Jet Fuels by Monochromatic Energy Dispersive X-ray Fluorescence Spectrometry	DIN EN 196-2:2013-10	Method of testing cement - Part 2: Chemical analysis of cement
ASTM D8252 - 19e1	Standard Test Method for Vanadium and Nickel in Crude and Residual Oil by X-ray Spectrometry	DIN EN ISO 12677:2013-02	Chemical analysis of refractory products by X-ray fluorescence (XRF) - Fused cast-bead method
ASTM D5059 - 20	Standard Test Methods for Lead and Manganese in Gasoline by X-Ray Spectroscopy	DIN 51001:2003-08	Testing of oxidic raw materials and basic materials - General bases of work for X-ray fluorescence method (XRF)
ASTM D6481 - 14(2019)	Standard Test Method for Determination of Phosphorus, Sulfur, Calcium, and Zinc in Lubrication Oils by Energy Dispersive X-ray Fluorescence Spectroscopy	ASTM D5839 - 15	Standard Test Method for Trace Element Analysis of Hazardous Waste Fuel by Energy-Dispersive X-Ray Fluorescence Spectrometry
SH/T 0631	Standard Test Method for Determination of Phosphorus, Sulfur, Calcium, and Zinc in Lubrication Oils by Energy Dispersive X-ray Fluorescence Spectroscopy	ASTM D6052 - 97(2016)	Standard Test Method for Preparation and Elemental Analysis of Liquid Hazardous Waste by Energy-Dispersive X-Ray Fluorescence
ASTM D7751 - 16	Standard Test Method for Determination of Additive Elements in Lubricating Oils by EDXRF Analysis	EPA 6200:2007-02	Field Portable X-RAY Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment
DIN EN ISO 8754:2003-12	Petroleum products - Determination of sulfur content - Energy-dispersive X-ray fluorescence spectrometry	ISO 18227:2014-03	Soil quality - Determination of elemental composition by X-ray fluorescence
DIN EN ISO 20847:2004-07	Petroleum products - Determination of sulfur content of automotive fuels - Energy-dispersive X-ray fluorescence spectrometry	ISO TS 16996:2015	Solid biofuels — Determination of elemental composition by X-ray fluorescence
DIN EN ISO 13032:2012-06	Petroleum products - Determination of low concentration of sulfur in automotive fuels - Energy-dispersive X-ray fluorescence spectrometric method	JIS K 0470	Determination of arsenic and lead in clay and sand using energy-dispersive X-ray fluorescence spectrometry
DIN 51577-4:1994-02	Testing of mineral oil hydrocarbons and similar products; determination of chlorine and bromine content; analysis by energy dispersive X-ray spectrometry with low cost instruments	DIN EN 15309:2007-08	Characterization of waste and soil - Determination of elemental composition by X-ray fluorescence
IP 336	Petroleum products - Determination of sulfur content - Energy-dispersive-X-ray fluorescence method	IEC 62321-3-1:2013	Determination of certain substances in electrotechnical products - Part 3-1: Screening - Lead, mercury, cadmium, total chromium and total bromine by X-ray fluorescence spectrometry
ASTM D4326 - 13	Standard Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence	DIN EN 62321-3-1:2014-10	Determination of certain substances in electrotechnical products - Part 3-1: Screening - Lead, mercury, cadmium, total chromium and total bromine by X-ray fluorescence spectrometry

Test Method	Description/Title	Test Method	Description/Title
VDE 0042-1-3-1:2014-10	Determination of certain substances in electrotechnical products - Part 3-1: Screening - Lead, mercury, cadmium, total chromium and total bromine by X-ray fluorescence spectrometry	ASTM C1254 - 18	Standard Test Method for Determination of Uranium in Mineral Acids by X-Ray Fluorescence
GB/T 28020	Adornment- determination of Baneful elements-X-ray fluorescence spectrometric method	ASTM C1255 - 18	Standard Test Method for Analysis of Uranium and Thorium in Soils by Energy Dispersive X-Ray Fluorescence Spectroscopy
GB/T 33352 - 2016	General rules of screening application of restricted substances in electrical and electronic products -- X-Ray fluorescence spectrometry	ASTM C1343 - 16	Standard Test Method for Determination of Low Concentrations of Uranium in Oils and Organic Liquids by X-ray Fluorescence
DIN/TS 51012:2020-04	Screening of substances of very high concern (SVHC) - General principles	VDI 3405 Blatt 2.3:2018-07	Additive manufacturing processes, rapid manufacturing - Beam melting of metallic parts - Characterisation of powder feedstock
EPA IO-3.3 - 1999	Determination of Metals in Ambient Particulate Matter using X-RAY Fluorescence (XRF) Spectroscopy	DIN EN ISO/ASTM 52907: 2020-05	Additive manufacturing - Feedstock materials - Methods to characterize metal powders
HJ829-2017	Ambient air -Determination of inorganic elements in ambient particle matter- Energy dispersive X-ray fluorescence spectroscopy (ED-XRF) method	ASTM F2980 - 13(2017)	Standard Test Method for Analysis of Heavy Metals in Glass by Field Portable X-Ray Fluorescence (XRF)
ISO/DIS 23345 - 2020	Jewellery and precious metals — Non destructive precious metal fineness confirmation by ED-XRF	ISO/TR 17276:2014-05	Cosmetics — Analytical approach for screening and quantification methods for heavy metals in cosmetics
ASTM B568 - 98(2014)	Standard Test Method for Measurement of Coating Thickness by X-Ray Spectrometry	CAIA/SH001-2015	"Rice - Determination of cadmium - X ray fluorescence spectrometry method"
DIN EN ISO 3497:2001-12	Metallic coatings - Measurement of coating thickness - X-ray spectrometric methods	GB/T 36226-2018	Stainless steel-Determination of Mn, Ni, Cr, Mo, Cu andTi- Handheld Energy Dispersive X-ray Fluorescence Spectrometric method (Semiquantitative method)
GB/T 16921	Metallic coatings - Measurement of coating thickness - X-ray spectrometric methods	GB/T 36168-2018	Turquoise-testing
JIS H 8501	"Methods of thickness test for metallic coatings Appendix 3"		

Of course, not every organization or locality has caught up, and adjusted its standards and regulations to the new reality.

“There are still applications in some areas which will require WD-XRF because of things like ISO standards,” explains Alexander Seyfarth. “Iron ore, for example. There’s no point putting ED-XRF on a mine site if the client needs ‘trade’ grade analysis approved per ISO. Or at the harbor — often the government needs to run all the assays very quickly to assess customs and tax, and their regulations specify WD. However, for applications where that standard is not

required, we’re putting energy-dispersive units on iron ore mines sites around the world today.

“But when we’re supplying commercial laboratories,” he says, “those are the last holdouts where we can switch over, because they have to deal with all sorts of samples. So you have to have a system or systems that can analyze virtually anything.

“For the last 2 or 3 years, though, in total, 70% of our new projects for geochemical analysis are being specified for ED-XRF.”

Seyfarth reports that his organization uses advanced ED-XRF analyzers wherever possible for applications such as mine tailings and for process control, using pelletized samples and detecting concentrations down to 10 ppm or lower of tungsten, tin, copper, uranium, thorium.

The ED-XRF Future

ED-XRF technology has room to grow. Not only in its number of users, but in the nature and performance of its technology.

For instance, light elements remain a challenge. “Both WD-XRF and ED-XRF have this same limitation,” says Dr. Joachim Heckel. “At the moment, they are still the domain of the scanning electron microscope. To overcome this would require developing an X-ray tube that delivered better excitation of these elements, as well as using more rigorous sample preparation methods. These are possible. You would then be able to analyze light elements such as lithium, boron, oxygen, nitrogen, and so on. Certain specific applications also could be targeted. We might seek improvements in the measurement of sodium with ED-XRF technology. That would be a new frontier of interest to, for instance, cement plants.”

“Earlier I talked about the brand-new application we’ve been running — analyzing fused beads of fluorine with SPECTRO XEPOS,” says Alexander Seyfarth. “We can actually do that! And it should be possible also for fused beads with low levels of elements like sodium, phosphorus, and sulfur. Also the rare earth elements, and low levels of uranium next to high levels of molybdenum. Was that on anybody’s radar for the future of XRF? You’d think it could only be done with WD-XRF at that

degree of dilution. Now it starts changing the perception. Approaching how that kind of high-level ED-XRF analyzer might accommodate different ways of doing sample prep could be one path forward.”

Generally, the experts expect further developments in ED-XRF throughput and in speed of measurement. These offer promising avenues for improvement before the technology reaches certain limits in the physics of silicon.

Seyfarth is looking forward to what he sees coming. “Unlike wavelength dispersive, where we are basically at a very mature technology and not much is happening — on the energy-dispersive side, there is lots of stuff happening,” he says.

“Right now I’m very excited about the prospects for a graphene window between the sample and the high-vacuum detection system, because that gets me more sensitivity. And I’m excited about new post-processing capabilities like the ones I see in SPECTRO XEPOS because that gets me more throughput — and gets me to applications which, before that, from a statistical point of view I had a hard time running. If the vendors continuously invest in the update of the technology, then for me, the reach of ED-XRF can only grow.”

Dr. Heckel sums up the prospects: “You could improve the element range if you look to the tubes. You could expand the complete element list if you look to the electronics — redesigning them to handle even higher count rates. You could improve the instrument’s mechanics, and thus help improve precision. And perhaps, even further in the future, you might be able to take advantage of superconductivity to create new detector technologies.”

CONCLUSION

From relatively modest beginnings, ED-XRF has emerged as a leading solution to a wide array of analysis requirements. No longer in the shadow of WD-XRF technology, advantages such as sensitivity and precision, throughput and readiness, ease of use, compactness, and affordability make an advanced ED-XRF instrument the leading choice for most users' X-ray spectrometry needs.

Literature

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