thermo scientific

XRF technology in the field

XRF technology for non-scientists

Thermo Fisher
SCIENTIFIC

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1

What is XRF?

X-ray fluorescence (XRF): a non-destructive analytical technique used to determine the chemical composition of materials

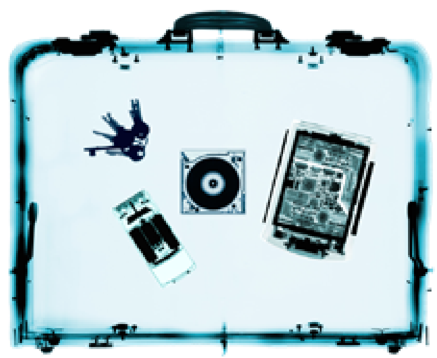
Overview

X-ray Fluorescence (XRF)

XRF occurs when a fluorescent (or secondary) X-ray is emitted from a sample that is being excited by a primary X-ray source. Because this fluorescence is unique to the elemental composition of the sample, XRF is an excellent technology for qualitative and quantitative analysis of the material composition. XRF spectrometry has a broad range of applications in industry, which we will discuss later in this ebook.

X-rays

X-rays are simply light waves that we can't see. Other light waves that we can't see include ultraviolet (UV) light (which gives you a sun tan), infrared light (which warms you up), and radio waves. X-rays have a very short wavelength, which corresponds to a very high energy.



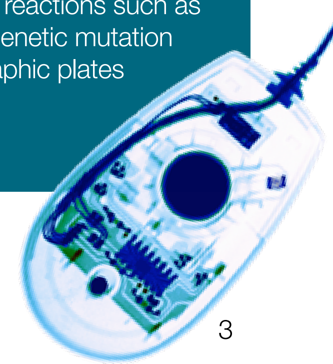
Properties of X-rays

They are:

- Propagated in straight lines at the speed of light
- Absorbed while passing through matter, depending on composition and density of the substance
- Emitted with energies characteristic of the elements present

They:

- Cause biological reactions such as cell damage or genetic mutation
- Darken photographic plates
- Ionize gases



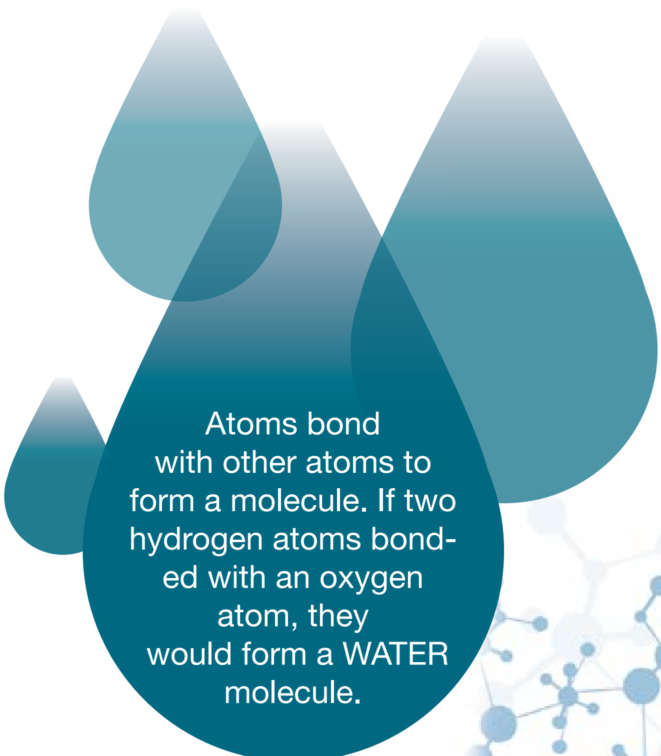
How XRF works

Fingerprints

Each of the elements present in a sample produces a unique set of characteristic X-rays that is a “fingerprint” for that specific element.

It all starts with the atom

Atoms are the extremely small particles of which we, and everything around us, are made. There are 92 naturally occurring elements and scientists have made more, bringing the total to 114 confirmed and at least 4 more claimed. Atoms are the smallest unit of an element that chemically behaves the same way the element does.



How XRF works

Anatomy of the atom*

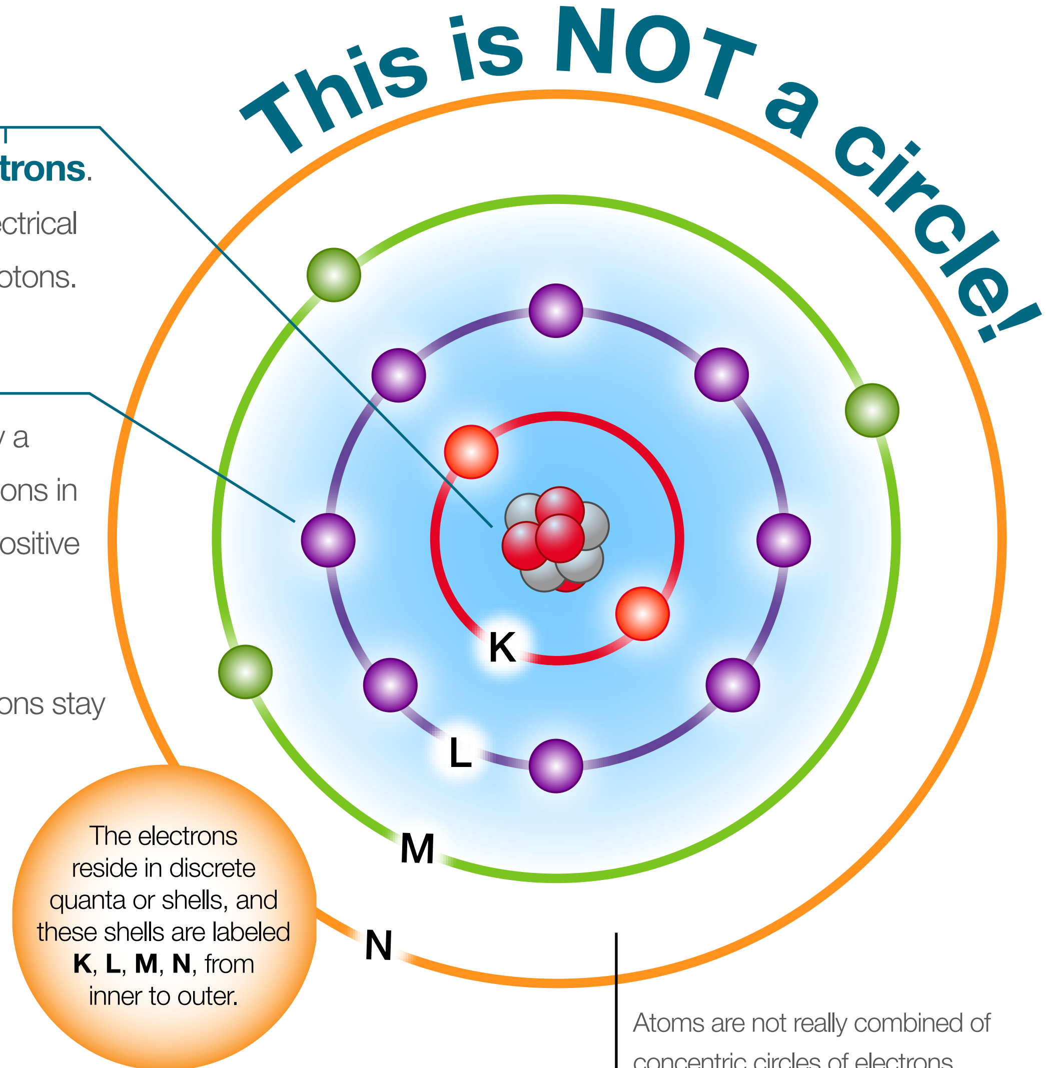
In the center of the atom is the nucleus, made up of **protons** and **neutrons**. Each proton carries a positive electrical charge, but neutrons carry no electrical charge, so the nucleus of an atom is positively charged because of its protons.

Electrons are particles that orbit the nucleus at a high speed and carry a negative charge, which balances the positive electrical charge of the protons in the nucleus. Since the total negative charge of electrons is equal to the positive charge of the nucleus, an atom is neutral.

The negative electrons are attracted to the positive protons, so the electrons stay around the nucleus in discrete shells.

When two chemicals react with each other, the reaction takes place between individual atoms at the atomic level. The outermost or covalent electrons are involved in this bonding.

The processes that cause materials to be radioactive occur at the atomic level, generally within the nucleus.



Atoms are not really combined of concentric circles of electrons… we just draw them that way to understand how the electrons orbit around the nucleus.

* Web site reference: http://www.epa.gov/radiation/understand/atom.html

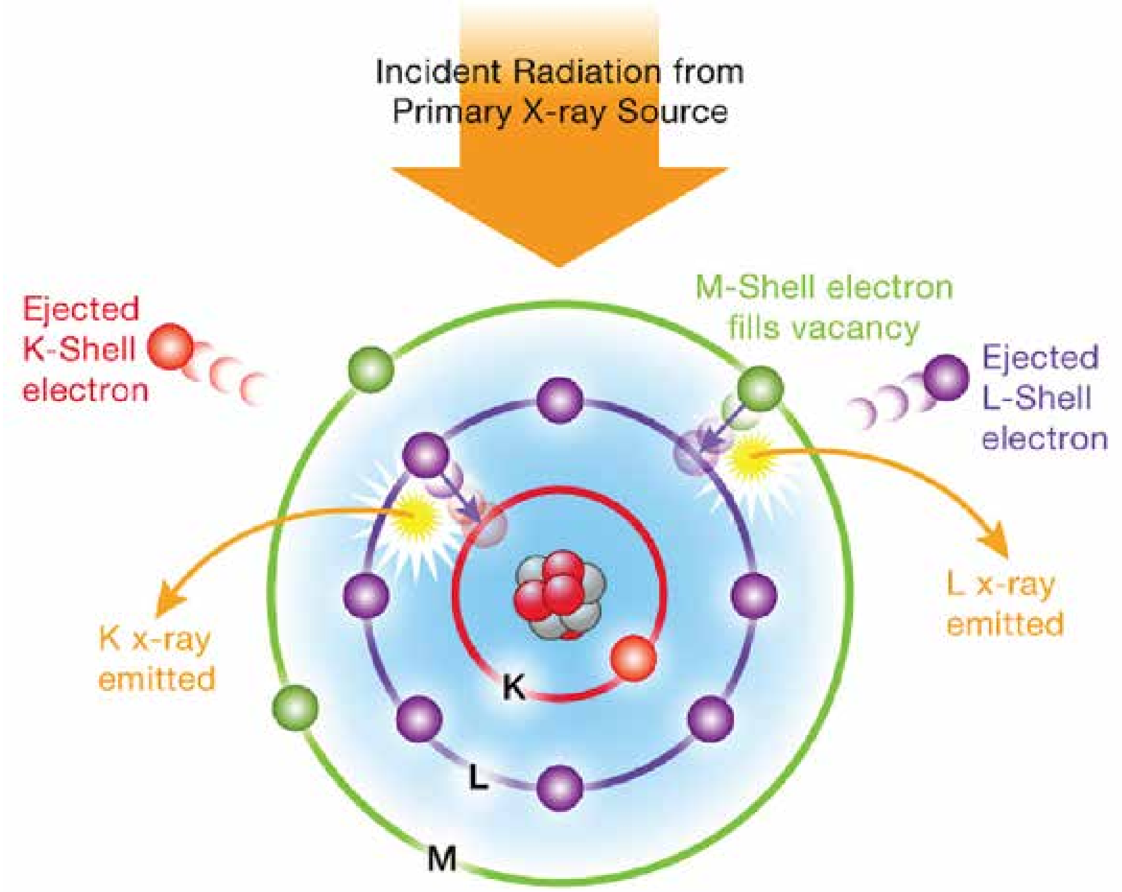
The X-ray fluorescence process

1 A solid or a liquid sample is irradiated with high energy X-rays from a controlled X-ray tube.

2 When an atom in the sample is struck with an X-ray of sufficient energy (greater than the atom's K or L shell binding energy), an electron from one of the atom's inner orbital shells is dislodged.

3 The atom regains stability, filling the vacancy left in the inner orbital shell with an electron from one of the atom's higher energy orbital shells.

4 The electron drops to the lower energy state by releasing a fluorescent X-ray. The energy of this X-ray is equal to the specific difference in energy between two quantum states of the electron. The measurement of this energy is the basis of XRF analysis.



The periodic table

Number of protons = atomic number (different for each element)

Number of electrons typically = number of protons (so the atom is neutral)

Number of neutrons is variable and is what allows some atoms to have isotopes

Electrons in shells closest to the nucleus are most strongly bound to the atom. Binding energy increases with the atomic number. The higher the number, the higher the weight.

An isotope of an element has the same number of protons but a different number of neutrons.

What is an element?

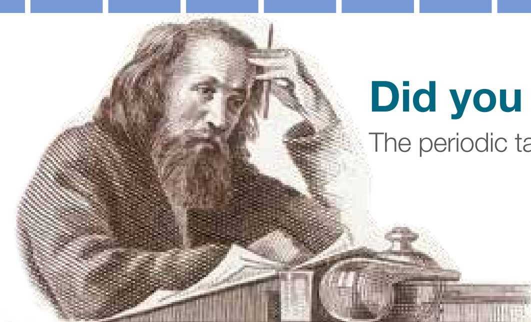
An element is a chemically pure substance composed of atoms.

Elements are the fundamental materials of which all matter is composed.

The elements are arranged in increasing order of their atomic weight (the number of protons in the nucleus of an atom).

1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba	57 *La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 +Ac	104 Rf	105 Ha	106 Sg	107 Ns	108 Hs	109 Mt	110 Ds	111 Rg	112 Cn	113 113	114 Fl	115 115	116 Lv		

58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr



Did you know?

The periodic table was created in 1869 by Dmitry I. Mendeleev.

List of periodic table elements

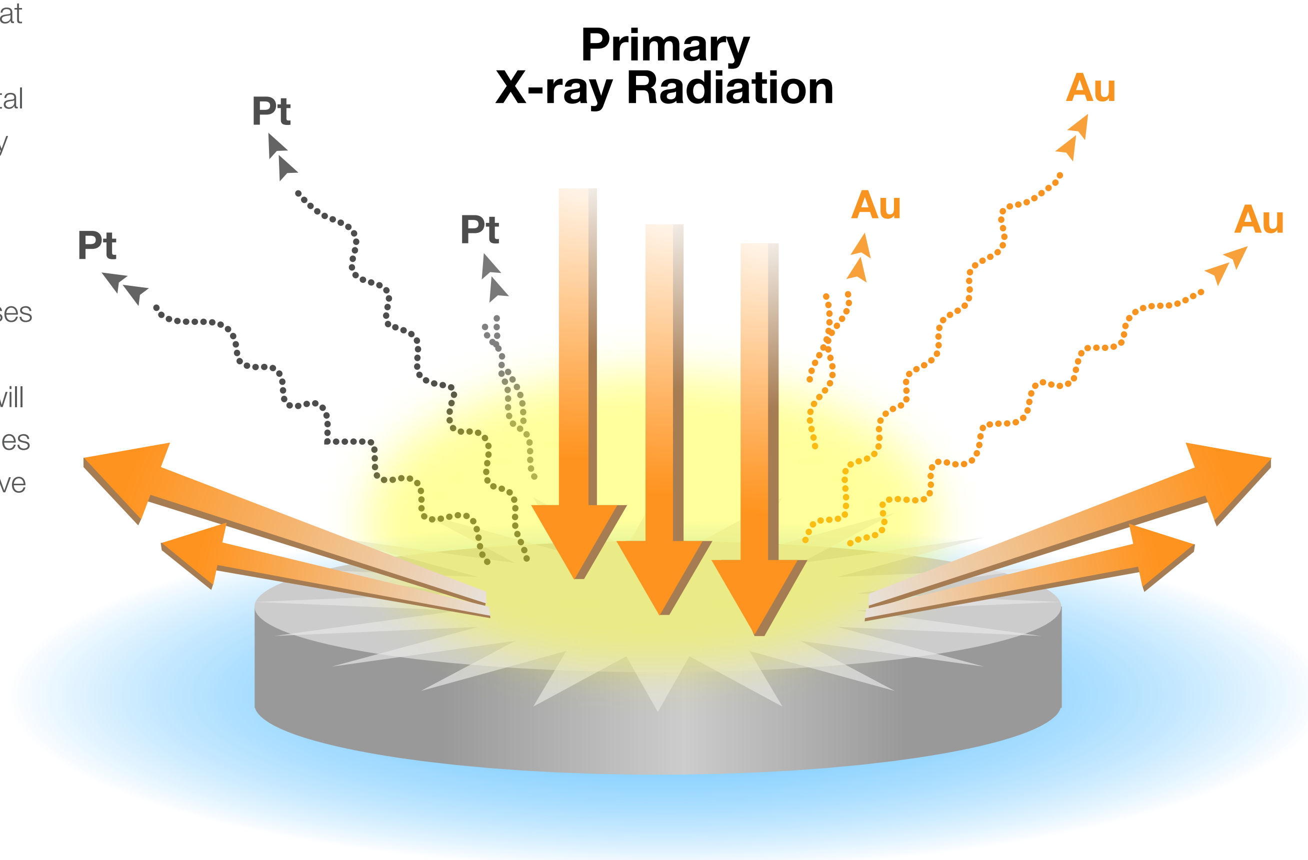
1 Hydrogen	**H**	21 Scandium	**Sc**	41 Niobium	**Nb**	61 Promethium	**Pm**	81 Thallium	**Tl**	101 Mendelevium	**Md**
2 Helium	**He**	22 Titanium	**Ti**	42 Molybdenum	**Mo**	62 Samarium	**Sm**	82 Lead	**Pb**	102 Nobelium	**No**
3 Lithium	**Li**	23 Vanadium	**V**	43 Technetium	**Tc**	63 Europium	**Eu**	83 Bismuth	**Bi**	103 Lawrencium	**Lr**
4 Beryllium	**Be**	24 Chromium	**Cr**	44 Ruthenium	**Ru**	64 Gadolinium	**Gd**	84 Polonium	**Po**	104 Rutherfordium	**Rf**
5 Boron	**B**	25 Manganese	**Mn**	45 Rhodium	**Rh**	65 Terbium	**Tb**	85 Astatine	**At**	105 Dubnium	**Db**
6 Carbon	**C**	26 Iron	**Fe**	46 Palladium	**Pd**	66 Dysprosium	**Dy**	86 Radon	**Rn**	106 Seaborgium	**Sg**
7 Nitrogen	**N**	27 Cobalt	**Co**	47 Silver	**Ag**	67 Holmium	**Ho**	87 Francium	**Fr**	107 Bohrium	**Bh**
8 Oxygen	**O**	28 Nickel	**Ni**	48 Cadmium	**Cd**	68 Erbium	**Er**	88 Radium	**Ra**	108 Hassium	**Hs**
9 Fluorine	**F**	29 Copper	**Cu**	49 Indium	**In**	69 Thulium	**Tm**	89 Actinium	**Ac**	109 Meitnerium	**Mt**
10 Neon	**Ne**	30 Zinc	**Zn**	50 Tin	**Sn**	70 Ytterbium	**Yb**	90 Thorium	**Th**	110 Darmstadtium	**Ds**
11 Sodium	**Na**	31 Gallium	**Ga**	51 Antimony	**Sb**	71 Lutetium	**Lu**	91 Protactinium	**Pa**	111 Roentgenium	**Rg**
12 Magnesium	**Mg**	32 Germanium	**Ge**	52 Tellurium	**Te**	72 Hafnium	**Hf**	92 Uranium	**U**	112 Copernicium	**Cn**
13 Aluminum	**Al**	33 Arsenic	**As**	53 Iodine	**I**	73 Tantalum	**Ta**	93 Neptunium	**Np**	113 Ununtrium	**113**
14 Silicon	**Si**	34 Selenium	**Se**	54 Xenon	**Xe**	74 Tungsten	**W**	94 Plutonium	**Pu**	114 Flerovium	**Fl**
15 Phosphorus	**P**	35 Bromine	**Br**	55 Cesium	**Cs**	75 Rhenium	**Re**	95 Americium	**Am**	115 Ununpentium	**115**
16 Sulfur	**S**	36 Krypton	**Kr**	56 Barium	**Ba**	76 Osmium	**Os**	96 Curium	**Cm**	116 Livermorium	**Lv**
17 Chlorine	**Cl**	37 Rubidium	**Rb**	57 Lanthanum	**La**	77 Iridium	**Ir**	97 Berkelium	**Bk**		
18 Argon	**Ar**	38 Strontium	**Sr**	58 Cerium	**Ce**	78 Platinum	**Pt**	98 Californium	**Cf**		
19 Potassium	**K**	39 Yttrium	**Y**	59 Praseodymium	**Pr**	79 Gold	**Au**	99 Einsteinium	**Es**		
20 Calcium	**Ca**	40 Zirconium	**Zr**	60 Neodymium	**Nd**	80 Mercury	**Hg**	100 Fermium	**Fm**		

Interpretation of XRF spectra

Spectral peaks

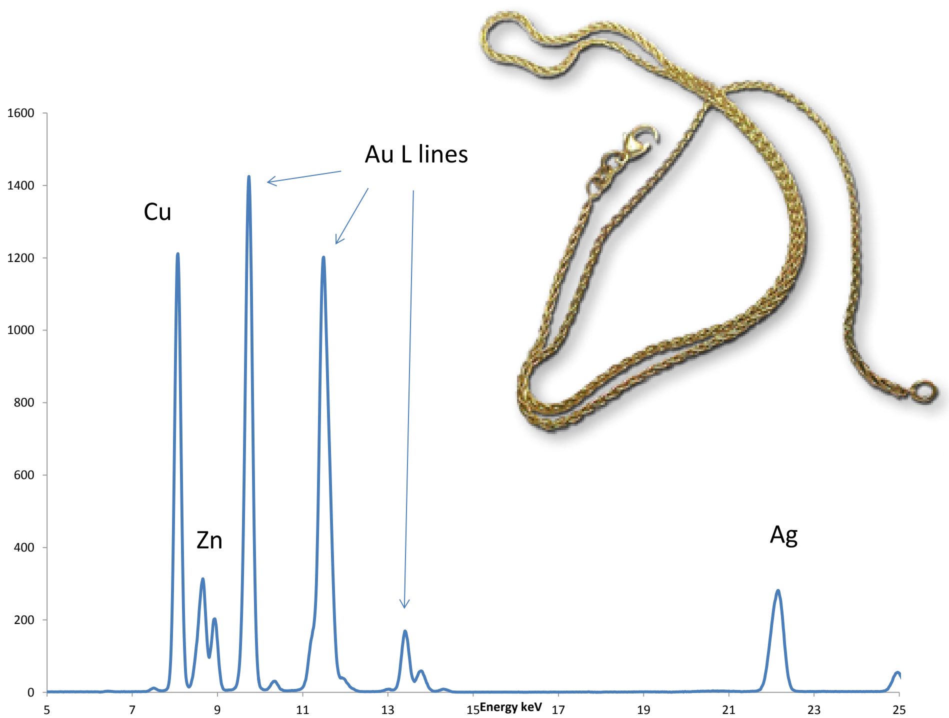
As we learned on the previous pages, each of the elements present in a sample produces a set of characteristic fluorescent X-rays that is unique for that specific element, which is why XRF spectroscopy is especially useful for elemental analysis. This elemental “fingerprint” is best illustrated by examining the X-ray energy spectrum and its “scattering peaks.”

Most atoms have several electron orbitals (K shell, L shell, M shell, for example). When X-ray energy causes electrons to transfer in and out of these shell levels, XRF peaks with varying intensities are created and will be present in the spectrum. The peak energy identifies the element, and the peak height/intensity is indicative of its concentration.

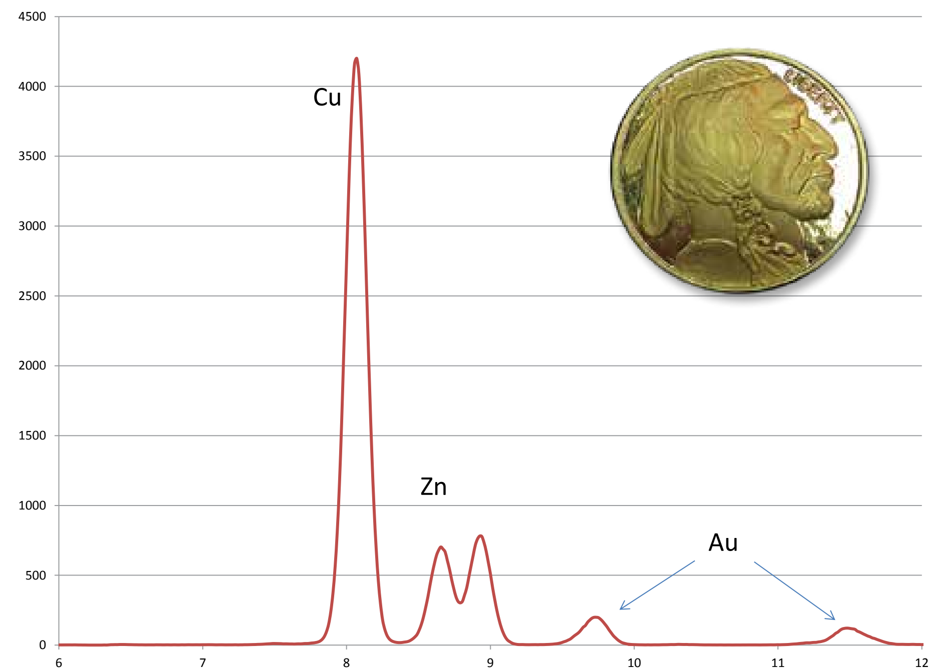


Examples of an XRF spectra

14k Gold (Au) chain



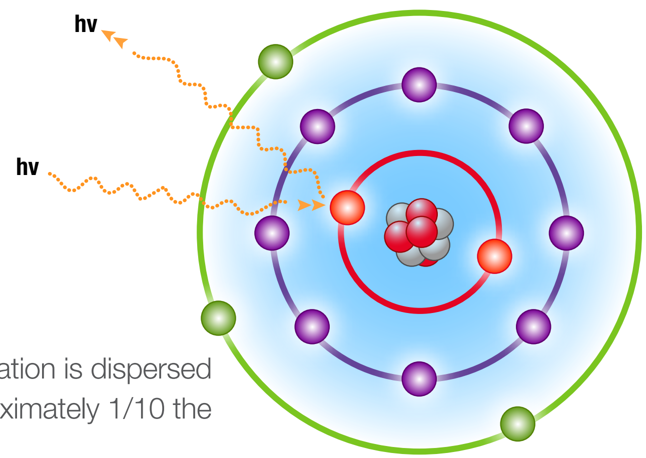
Gold (Au) plated replica 2011 American Buffalo coin



Rayleigh/Compton scatter peaks

Overview

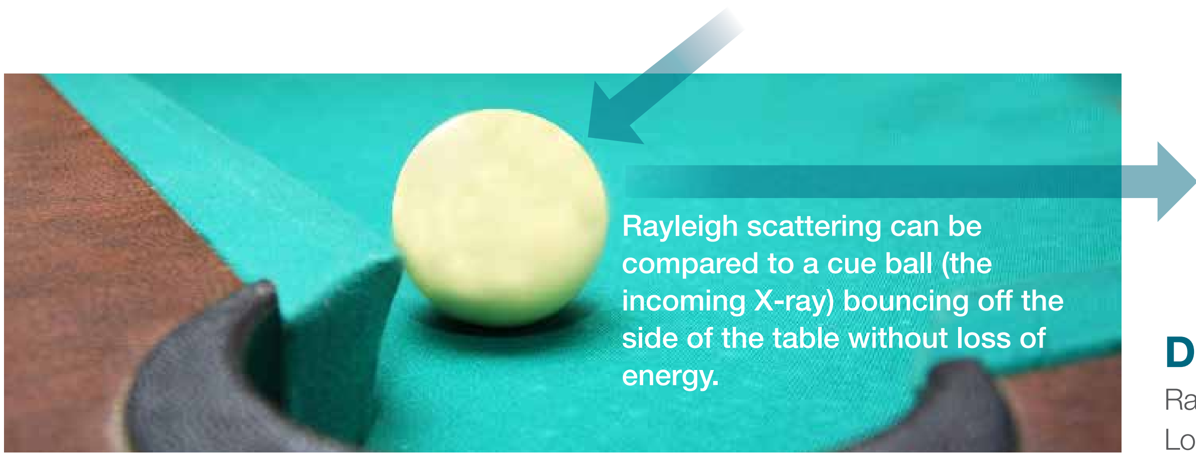
Scattering occurs when incoming X-rays do not produce fluorescence, but rather "collide" with the atoms of the sample which results in a change in the direction of motion of a particle.



Rayleigh scattering

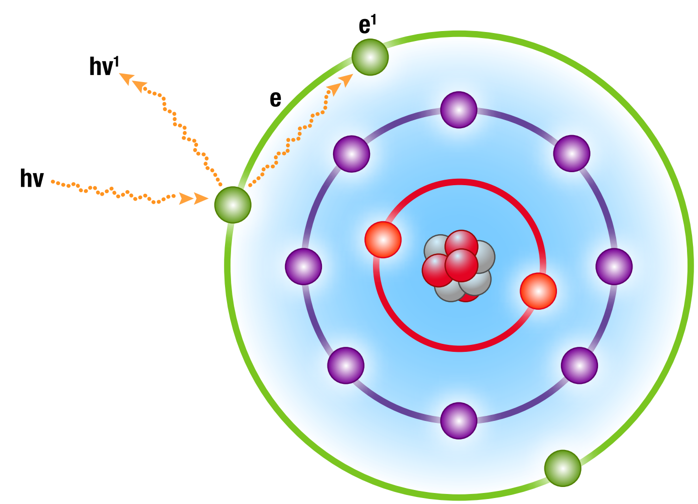
In Rayleigh scattering, electromagnetic radiation is dispersed by particles having a radius less than approximately 1/10 the wavelength of the radiation.

During the Rayleigh scattering process, photons are scattered by tightly bound electrons in which the atom is neither ionized nor excited. The incident photons are scattered with (essentially) an unchanged energy. Rayleigh scattering occurs mostly at low energies and for high atomic weight.



Compton scattering

In Compton scattering, the X-ray strikes an electron of the sample. Since some energy is transferred to the electron in the collision, the X-ray leaves the collision with less energy. That's why we see the Compton peak at an energy lower than the source excitation energy.



Did you know?

Rayleigh scattering is named after the British physicist Lord Rayleigh who discovered the process.

Limitations

Overview

Light elements analysis with handheld XRF can be challenging because the fluorescent X-rays from lighter elements (Z<18) are less energetic and are greatly attenuated as the X-rays pass through air. Also, sample preparation is highly recommended.

Spectral effects

Some elements have lines that overlap other elements. Fortunately the analyzer software will strip out and correct most of these overlaps (as long as the interfering element is in the mode being used), but limits of detection (LOD) may be worse when 2 overlapping elements are present.

Matrix effects

The matrix is any other element present in or on the sample other than the one element being considered. Enhancement and absorption effects are typically taken care of in the software if you're using a fundamental parameters based calibration with all the necessary elements present.

Enhancement effects
Some fluorescent X-rays have more energy than the binding energy of other elements present in the sample, and so their energy will excite those other elements. These elements will give a greater signal return to the detector, i.e. "enhancing" the reading.

Absorption effects
The fluorescent X-ray does not reach the detector as it is scattered or absorbed by other elements present in the sample, so the signal is weaker.

Sample effects
The surface of the material being analyzed is not representative of the entire sample (particle size, inhomogeneity, surface contamination, etc.) XRF is a surface analysis technique, so inhomogeneity or contamination will skew the results.

Calibration

Overview

Most handheld analyzers are pre-calibrated for immediate out-of-the-box analysis for a wide range of uses. Common calibration routines include the following:

Fundamental parameters

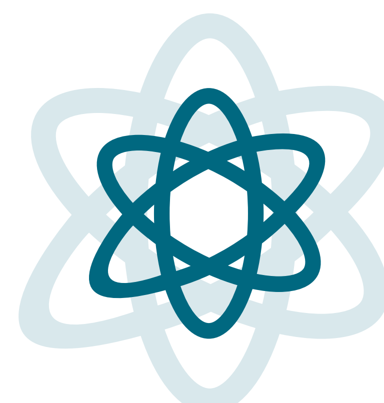
For measuring samples of unknown chemical composition in which concentrations of light and heavy elements may vary from parts per million (ppm) to high percent levels, fundamental parameters (FP) analysis is used to simultaneously compensate for a wide variety of geometric effects (including small and odd-shaped samples), plus X-ray absorption and enhancement effects as well as spectral overlaps. FP is the preferred analysis tool for mining and exploration, plastics analysis, precious metals analysis and all metal alloy testing applications.

Compton normalization

Compton normalization (CN) is a calibration technique that works well for a narrow range of sample types, basically soil samples that contain less than 5% of all elements $Z > 23$ (summing them together). It relies on using the ratio of the element peak to the Compton scatter peak, which gives a measure of density of the sample, and is ideal for the analysis of low concentrations of heavy metals such as lead (Pb) and copper (Cu) (monitored by the US Resource Recovery and Conservation Act) at contamination sites.

Empirical calibration

In empirical calibration, the user must first analyze known samples to obtain the count intensity, which is then plotted using off-line software to generate the calibration curve. This curve data is then put back into the analyzer which can then be run to give immediate results. Empirical testing modes are only suited for measuring samples for which chemical compositions will fall within the narrow calibration range, and interferences (spectral and matrix) must be taken into consideration within the calibration.



2

XRF analyzers in the field: Technology

Overview

Portable XRF analyzers

Handheld and portable XRF analyzers have become the standard for non-destructive elemental analysis in a wide range of applications. These systems are routinely used for rapid quality control inspection and analysis to ensure product chemistry specifications are met. Lightweight and easy to use, these instruments provide instant analysis in any field environment.

Since the late 1960s, portable XRF technology has evolved through seven generations of increasingly sophisticated analyzers. Each succeeding generation has added new capabilities, such as smaller size, increased speed, better performance, and greater ease of use. Today's portable XRF analyzers are miniaturized and designed for ultra high speed with lab-quality performance.

Niton XL5 Plus

Niton XL3t GOLDD+

Niton XL2 Plus

Niton XL2 100G

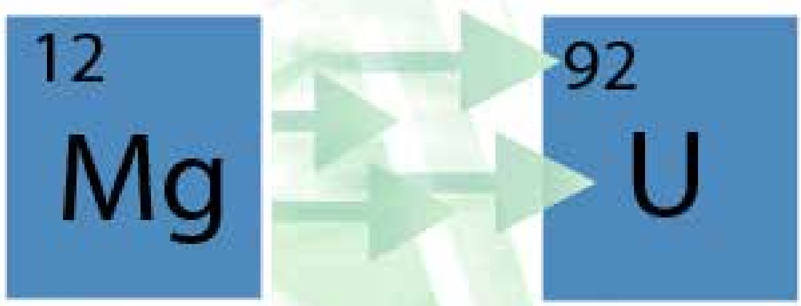
Niton DXL

Energy dispersive X-ray fluorescence

EDXRF

EDXRF is the technology commonly used in portable analyzers. EDXRF instrumentation separates the characteristic X-rays of different elements into a complete fluorescence energy spectrum which is then processed for qualitative or quantitative analysis.

EDXRF is a convenient way to screen all kinds of materials for quick identification and quantification of elements from magnesium (Mg) to uranium (U). EDXRF instruments may be either handheld or portable depending on user preference, making them the perfect tool for in-field analysis, and providing instant feedback to the user without the long trip to the laboratory. Low cost of ownership and rapid elemental analysis of any sample type make EDXRF an attractive front-end analysis tool.



Handheld XRF instrument

Working principle

1

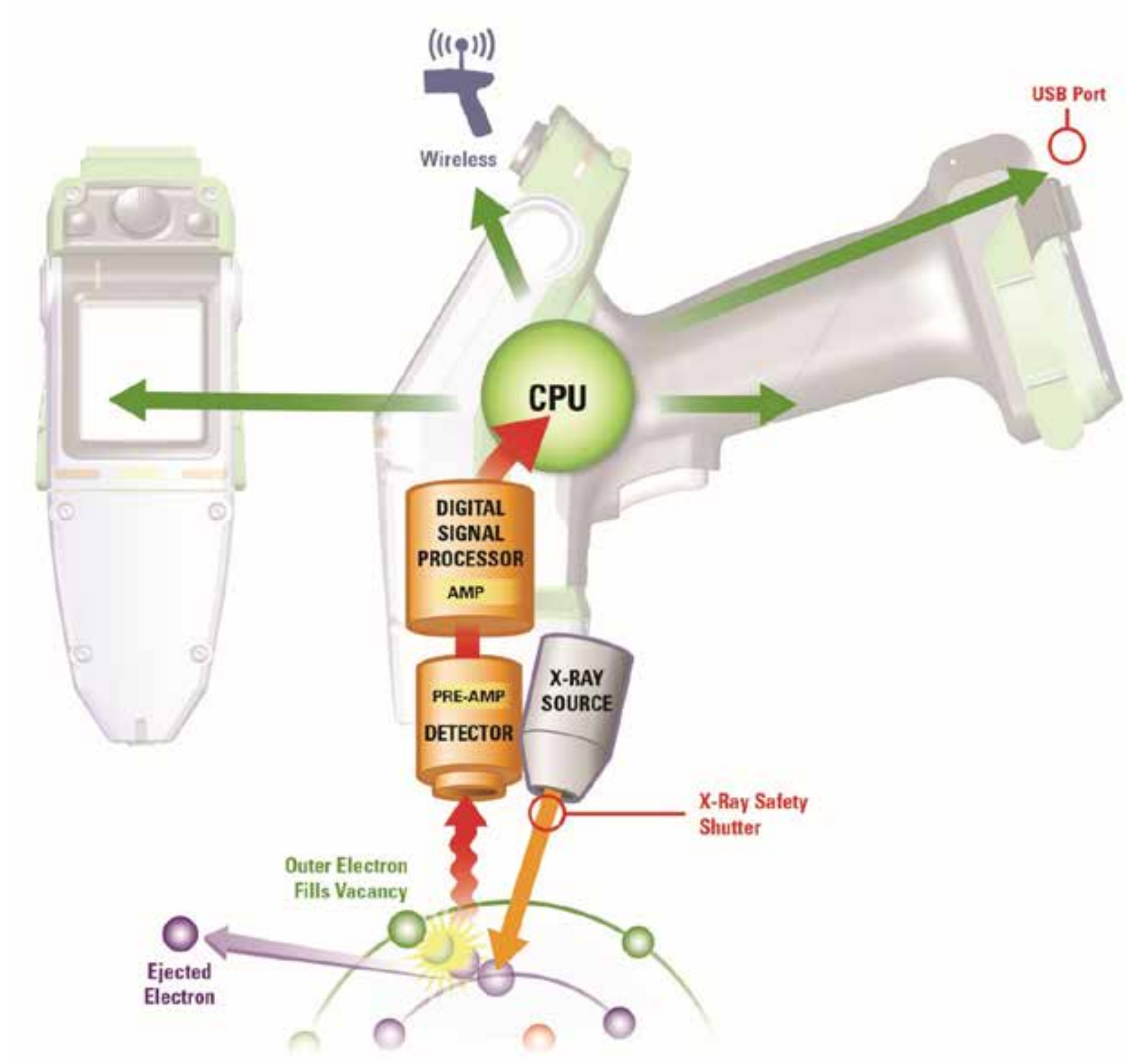
A sample is irradiated with high energy X-rays from a controlled X-ray tube. The energy causes inner-shell electrons to be ejected. Outer-shell electrons fill the vacancies and fluorescent x-rays are emitted.

2

The fluorescent x-rays enter the detector and send electronic pulses to the preamp. The preamp amplifies the signals and sends them to the Digital Signal Processor (DSP) to collect and digitize the x-ray events into channels of energy. Next, the “counts” for each channel (spectral data) is then sent to the main CPU for processing.

3

Using algorithms, the central processing unit (CPU) analyzes the spectral data and determines the concentration of each element present. Composition data and identified alloy grade are displayed in real time and stored via memory for later recall or download to an external PC.



Detector technologies

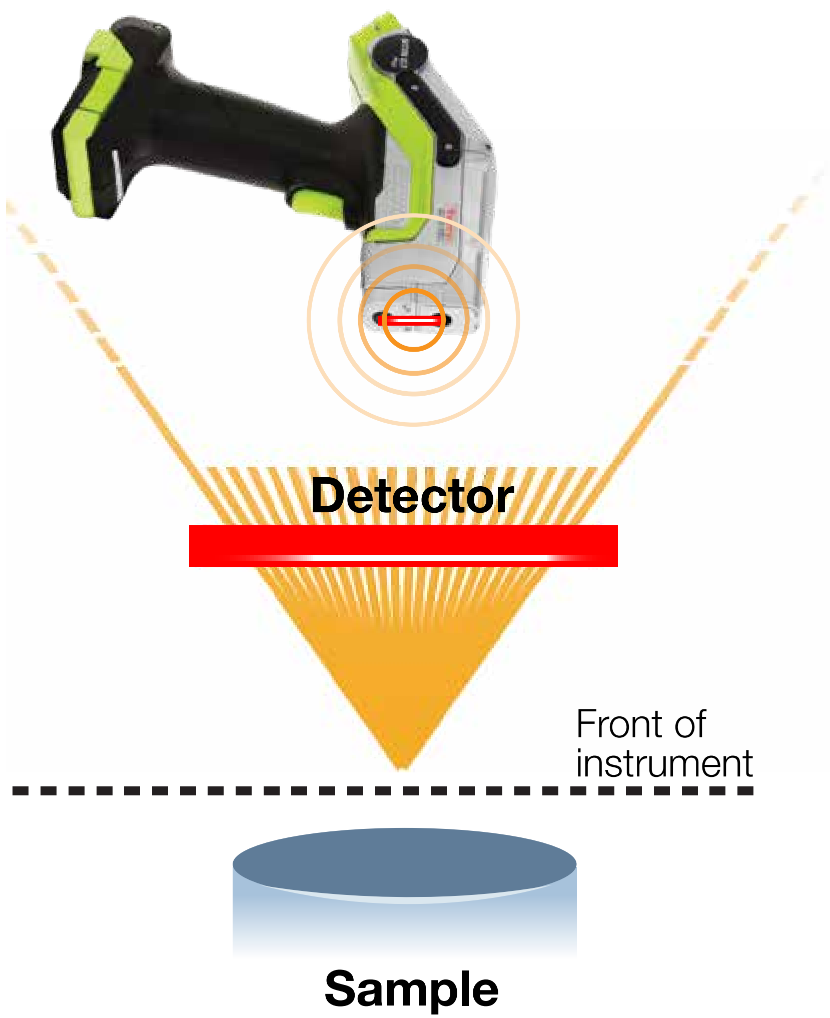
Both silicon drift detectors (SDDs) and PIN detectors employ a high-performance, high-resolution technology conventionally used in handheld and portable XRF instrumentation; they are appropriate for numerous industry applications.

Silicon Drift Detectors

The introduction of SDDs into handheld XRF instruments has produced significant performance improvements over traditional XRF capabilities. SDDs are high-resolution detectors that can be used in high count-rate applications. The larger the active area of the detector, the more efficiently it can gather and process X-ray counts.

XRF instrumentation employing SDDs can be used in applications that require extreme sensitivity, such as the detection of tramp elements in alloys that can degrade performance. Residual elements can be measured with a confidence once only possible in the lab.

SDDs are also required to analyze light elements such as magnesium (Mg), aluminium (Al), silicon (Si), phosphorus (P) and sulfur (S).



PIN detectors

PIN detectors measure the fluorescence radiation emitted from the sample after it has been irradiated, usually by an X-ray tube (occasionally by a radioactive source). Instruments with silicon PIN detectors are sensitive to X-rays that are higher on the periodic table than sulfur (S) and tend to be less expensive than instruments with SDDs.

Strengths

Overview

Portable handheld XRF analyzers are lightweight, easy to handle and can be operated with minimal training. They provide elemental analysis anytime, anywhere, in seconds rather than the hours or days it can take for a traditional testing laboratory.

Easy to use

Lightweight

Cost-effective

Nondestructive

Accurate results

Instant results

Portable

Used on-site

General use guidelines

Radiation

The analyzer emits a directed radiation beam when the tube is energized (tube based instrument) or when the shutter is open (isotope based instrument). Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure:

Time

Distance

Shielding

Did you know?

While the radiation emitted from a portable XRF analyzer is similar to the exposure received in a normal medical or dental X-ray, care must be taken to always point a handheld XRF analyzer directly at the sample and never at a person or a body part.

3

XRF analyzers in the field: Applications

Handheld and portable XRF analyzers have many applications for elemental analysis. Here are a few industries putting XRF technology to work in daily operations.

Metal alloy analysis, identification and testing

Mining/geology

Toys/consumer goods

Environmental analysis/ remediation

Art and archaeometry

Metal alloy analysis, identification and testing

Scrap metal recycling

Scrap metal recycling has become big business, but globalized trade in scrap metal, alloy stock and finished products has increased the costs of alloy mix-ups for suppliers, distributors and industrial consumers.

The exact chemical composition of scrap, including the existence of contaminants or hazardous elements, must be determined for quality, safety and regulatory compliance.

Scrap metal recyclers use handheld XRF to positively identify numerous alloys, rapidly analyze their chemical composition at material transfer points and guarantee the quality of the product for their customers.

Metal alloy analysis, identification and testing

Positive material identification

Wrong or out-of-specification metal alloys can lead to premature and potentially catastrophic part failures. Accidents within the refining and aerospace industries, for example, can happen when critical parts are made from the wrong metal alloy, or from a material that does not meet specifications.

The process of inspecting and analyzing individual component materials is called positive material identification (PMI). Portable XRF analyzers are indispensible tools for performing PMI of incoming raw materials, work in progress, and final quality assurance of finished parts.

Metal alloy analysis, identification and testing

Precious metals and jewelry analysis

Portable XRF analyzers are ideal for the retail environment. Many jewelers and pawn shops are using these instruments to test the purity and composition of precious metals. XRF quickly provides the exact percentages of all elements within an item – easily identifying non-standard, under-karated and even sophisticated counterfeit precious metals that acid testing is incapable of differentiating.

Did you know?

A karat is a unit of measure that describes the purity of gold alloys.

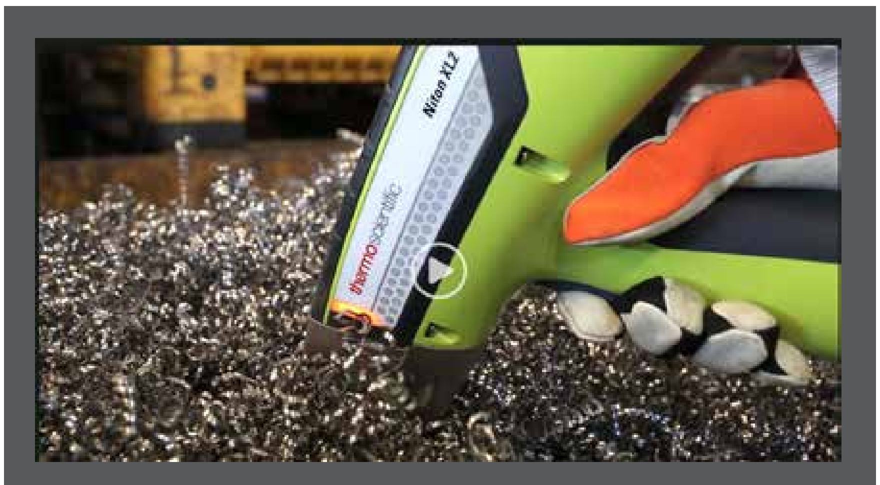
Infographic:
8 Reasons Jewelers Should Not Use Acid to Test Jewelry

Click to read or download pdf.

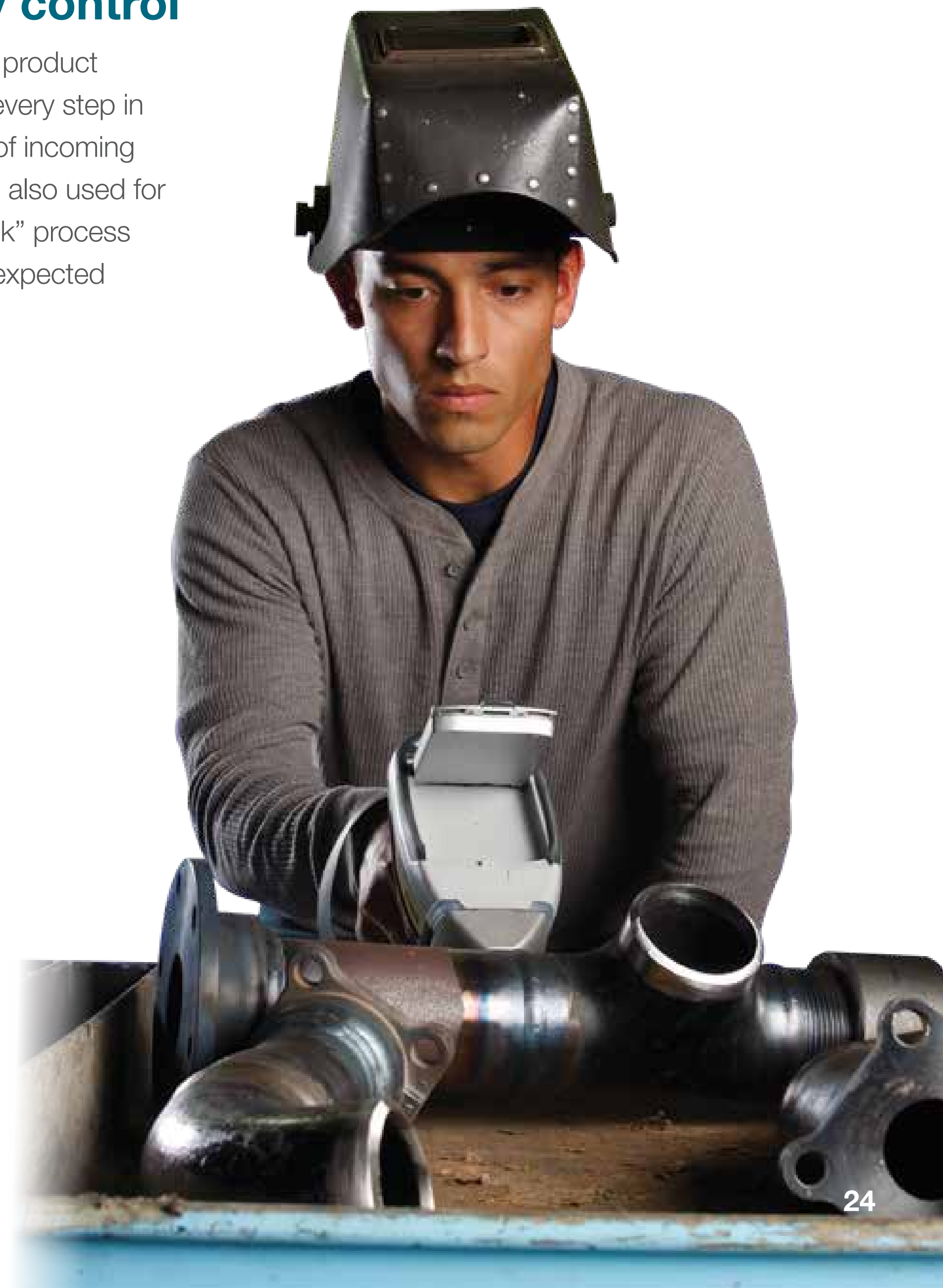
Metal alloy analysis, identification and testing

Metal fabrication: quality assurance and quality control

Material verification for alloy quality assurance and quality control (QA/QC) are critical to product safety. The potential for material mix-ups and the need for traceability are a concern at every step in the metal fabrication and manufacturing process. Handheld XRF is used for inspection of incoming raw material to verify the alloy grade and composition prior to product manufacture. It is also used for final quality inspection before finished parts are sent to the customer. This “double-check” process helps ensure that the incoming raw materials and the outgoing finished parts meet the expected engineering requirements.



Watch this video for evidence of the fast and accurate XRF analysis with the Thermo Scientific™ Niton™ XL2 Plus handheld XRF analyzer.



Mining/geology

Overview

Sample analysis with handheld XRF offers a substantial advantage in mining operations by providing immediate feedback for quick decision making on-site:

- Whether to stop or continue drilling
- When to make equipment relocation decisions
- Where to focus on the grid
- When to select a sample for laboratory analysis

Real-time analysis with handheld XRF analyzers is also a good way to prequalify samples for off-site lab analysis to ensure only the best samples are evaluated.

Portable handheld analyzers can be operated virtually anywhere on-site and easily accommodate a wide variation of samples, with little or no sample preparation.

Exploration

XRF analyzers quickly deliver exploration data for quantitative geochemical analysis of metal concentrations for mine mapping.

Mining/geology

Production and mineral processing

Fast, laboratory-grade sample analysis data for process control, quality assurance and other operational decisions.

Mine site analysis and extraction

Send data to quarry laboratory and operations management personnel for easy collaboration and informed decisions.

Industrial minerals evaluation

XRF analyzers can be used for in-quarry exploration and evaluating the composition of raw materials such as phosphate, potash, gypsum and limestone for industrial use.

Oil & gas exploration

XRF analyzers are valuable for upstream exploration and production, offering rapid, on-site chemical analysis of rocks, cuttings, and cores that can be used for identifying formations and determining mineral composition of the rock. Users can infer mineralogical properties favorable to oil and gas production from data collected in real time.

Toys/consumer goods

Overview

The US Consumer Product Safety Improvement Act (CPSIA) of 2008 was signed into law to combat the alarming amounts of lead found in children's toys. Now consumer goods such as toys, apparel, jewelry, cosmetics and furniture are routinely screened using XRF analyzers.

Worldwide Restriction of Hazardous Substances (RoHS) regulations continue to impact the manufacturers of electrical and electronic goods and their supply chains...as do the halogen-free initiatives. Handheld XRF analyzers help enforcement agencies screen goods for mercury, lead and other harmful materials.

Did you know?

The US Consumer Product Safety Commission (CPSC) and Europe's PROSAFE (Product Safety Forum) use XRF analyzers for screening toys and consumer goods.

Environmental analysis/remediation

Soil

Industrial and agricultural sites can become contaminated with lead, arsenic, cadmium, chromium and other toxic metals. The first remedial step in treating these hazardous areas is accurately assessing the scope and extent of the pollutants in the soil. XRF analyzers provide lab-grade performance in the field, permitting surgical delineation of contamination boundaries while in full compliance with US EPA Method 6200.

Did you know?

Regulatory agencies such as the US EPA use XRF analyzers.

Art and archaeometry

Overview

XRF analyzers can collect quantitative elemental data from archaeological samples. This data can be used to match pigments and other materials for restoration, help identify how objects have been preserved in the past, how to better conserve them for the future, glean important clues to the age of petroglyphs, identify alloys and other materials and help authenticate a variety of art and artifacts.

Did you know?

The US Native American Graves Protection and Repatriation Act (NAGPRA) requires that Native American cultural artifacts be returned to lineal descendants or affiliated tribes. XRF technology can be used to evaluate these objects for the presence of arsenic or other harmful preservatives before they are returned.

Related analyzer technology

Laser induced breakdown spectroscopy (LIBS)

LIBS complements handheld XRF as another analytical chemistry technique used for quantitative elemental analysis. It can help identify low alloy/ carbon steels and L- and H-grade steels.

When carbon detection and mobility are top of mind, industrial businesses rely on the Thermo Scientific™ Niton™ Apollo™ handheld LIBS analyzer for superior performance and enhanced productivity. The Niton Apollo analyzer transforms a traditional cart-mounted optical emission spectroscopy (OES) system into a highly portable, easy-to-use handheld analyzer.

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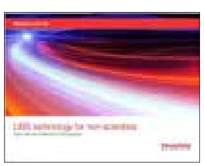
How LIBS technology works

Niton Apollo analyzer FAQs



Download the LIBS ebook

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