



XRF Technology in the Field





Table of Contents



What is XRF?

Overview	3
How XRF Works	4
The X-ray Fluorescence Process	6
The Periodic Table	7
List of Periodic Table Elements	8
Interpretation of XRF Spectra	9
Examples of an XRF Spectra	10
Rayleigh/Compton Scatter Peaks	11
Limitations	12
Calibration	13



XRF Analyzers in the Field: Technology

Overview	14
Energy Dispersive X-ray Fluorescence	15
Detection of X-Rays	16
Strengths	18
General Use Guidelines	19



XRF Analyzers in the Field: Applications

Metal Alloy Analysis, Identification and Testing	21
Mining/Geology	25
Toys/Consumer Goods	27
Environmental Analysis/Remediation	28
Art and Archaeometry	30

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 X-rays Are:

- Propagated in straight lines at 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:

- Affect the electrical properties of liquids and solids
- Cause biological reactions such as cell damage or genetic mutation
- Darken photographic plates
- lonize 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.

Atoms bond with other atoms to form a molecule. If two hydrogen atoms bonded with an oxygen atom, they would form a WATER molecule.

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.



The X-Ray Fluorescence Process



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

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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.



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.



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The Periodic Table



different number of neutrons.

What is an Element?

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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).

la	Mg											AI	51	Р	5	CI	Ar
K	²⁰ Ca	21 SC	²² Ti	23 V	²⁴ Cr	²⁵ Mn	Fe	²⁷ Co	²⁸ Ni	²⁹ Cu	³⁰ Zn	³¹ Ga	Ge	³³ As	³⁴ Se	³⁵ Br	³⁶ Kr
2b	³⁸ Sr	³⁹ Y	⁴⁰ Zr	⁴¹ Nb	42 Mo	43 Tc	⁴⁴ Ru	⁴⁵ Rh	Pd	A7	⁴⁸ Cd	49 In	⁵⁰ Sn	⁵¹ Sb	⁵² Te	53 	Xe
S	⁵⁶ Ba	⁵⁷ *La	⁷² Hf	⁷³ Ta	74 W	⁷⁵ Re	⁷⁶ Os	77 Ir	⁷⁸ Pt	⁷⁹ Au	⁸⁰ Hg	81 TI	Pb	⁸³ Bi	⁸⁴ Po	⁸⁵ At	⁸⁶ Rn
-r	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	Sm	Eu	Gd	⁶⁵ Tb	⁶⁶ Dу	67 Ho	68 Er	⁶⁹ Tm	⁷⁰ Yb	⁷¹ Lu
⁹⁰	⁹¹	92	⁹³	94	95	°°	97	98	99	¹⁰⁰	¹⁰¹	¹⁰²	103
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

Did you know?

The Periodic Table was created in 1869 by Dmitry I. Mendeleev.

He

Ne

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F

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List of Periodic Table Elements

1 Hydrogen	Н	21 Scandium	Sc	41 Niobium	Nb	61 Promethium	Pm	81 Thallium	TI	101 Mendelevium	Md
2 Helium	Не	22 Titanium	Ti	42 Molybdenum	Мо	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	Ро	104 Rutherfordium	Rf
5 Boron	В	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	Ν	27 Cobalt	Co	47 Silver	Ag	67 Holmium	Но	87 Francium	Fr	107 Bohrium	Bh
8 Oxygen	0	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	Те	72 Hafnium	Hf	92 Uranium	U	112 Copernicium	Cn
13 Aluminum	AI	33 Arsenic	As	53 lodine	1	73 Tantalum	Та	93 Neptunium	Np	113 Ununtrium	113
14 Silicon	Si	34 Selenium	Se	54 Xenon	Xe	74 Tungsten	W	94 Plutonium	Pu	114 Flerovium	FI
15 Phosphorus	Ρ	35 Bromine	Br	55 Cesium	Cs	75 Rhenium	Re	95 Americium	Am	115 Ununpentium	115
16 Sulfur	S	36 Krypton	Kr	56 Barium	Ва	76 Osmium	0 s	96 Curium	Cm	116 Livermorium	Lv
17 Chlorine	CI	37 Rubidium	Rb	57 Lanthanum	La	77 Iridium	lr	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	ו 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 in 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

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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 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 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 1 element being considered. Enhancement and absorption effects are typically taken care of in the software if you are 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) (and other RCRA elements) 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 onto 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.



Energy Dispersive X-ray Fluorescence

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 technology 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.





Detection of X-Rays

Detectors

In order to "read" fluorescent x-rays to accurately and nondestructively determine elements present in a given sample - they must be detected, thus the detector is an important part of any kind of XRF instrumentation. When the characteristic x-rays enter the detector, their electromagnetic energies are converted to electrical pulses. These pulses then are sorted into element channels in a Digital Signal Processor (DSP).

Next, the "counts" from each element are sent to the microprocessor, which contains the algorithms for calculating the concentration of each element from the count rate data.



Detection of X-Rays

There are 2 main types of detectors in handheld XRF technology:

Silicon Drift **Detectors (SDDs)**

The introduction of silicon drift detectors (SDD) 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 SDD can be used in applications that require extreme sensitivity, such as the detection of tramp elements in alloys that can degrade their performance. Residual elements can be measured with a confidence once only possible in the lab. SDD's are also required to analyze light elements such as Magnesium (Mg), Aluminium (Al), Silicon (Si), Phosphorus (P) and Sulfur (S).



PIN Detectors

A high-performance, high-resolution technology conventionally used in handheld and portable XRF instrumentation and appropriate for numerous industry applications. 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 Silicon Drift Detectors.

Nondestructive

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.



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:



Distance



Radioactive material is considered a hazardous material (HAZMAT) for the purposes of transport. This means that the transportation of a portable XRF device containing radioactive sources is regulated.

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.





Mining/Geology

Toys/Consumer Goods



Environmental Analysis/ Remediation



Art and Archaeometry

3 XRF Analyzers in the Field: Applications

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



Scrap Metal Recycling

Scrap metal recycling has become big business, but globalized trade in scrap metal, alloy stock and finished products have increased the costs of alloy mix-ups to 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, including light alloys, rapidly analyze their chemical composition at material transfer points and guarantee the quality of their product to their customers.

Positive Material Identification (PMI)

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.



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Precious Metals & 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.

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Metal Fabrication; Quality Assurance & Control

Material verification for alloy quality assurance and alloy 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 a video about Chip Ganassi Racing and how they use the Thermo Scientific[™] Niton[™] XL3t GOLDD+ 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 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 U.S. Consumer Product Safety Commission (CPSC) and Europe's PROSAFE (Product Safety) use XRF analyzers for screening toys and consumer goods.

Environmental Analysis/Remediation

Overview

From hazardous site modeling and risk assessment to on-site contaminant screening and lead paint abatement, handheld XRF analyzers provide on-site analysis of environmental contaminants.

Lead Paint Inspection

Government regulations, such as the U.S. EPA-issued Renovation, Repair and Painting (RRP) Rule, mandate lead-safe work practices for contractors performing renovations. XRF analyzers provide conclusive results for lead in samples associated with the abatement and control of lead-based paint for compliance with RRP and other state and federal requirements.



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.



Thermo Scientific[™] Niton[™] XL3t GOLDD+ Handheld XRF analyzer highlighted in *USA TODAY* "Ghost Factories" investigation of contaminated soils.



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.



The 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.



Debbie Schatzlein, MRSC, is a Senior Applications Chemist within the R&D department for Thermo Scientific Portable Analytical Instruments, based in Tewksbury, MA, USA. She was born and educated in England but has made Massachusetts her home for over 20 years.

Debbie has been practicing chemistry over 35 years in a variety of environments, including R&D laboratories, 24/7 foundry operations and a variety of instrument manufacturers. She has specialized in atomic spectroscopy, particularly ICP, arc-spark OES and XRF; with the last 12 years focused on improving handheld XRF instrumentation and making it easier to use in the field. She has traveled around the world to train users on how to get the best out of their instruments.

Debbie is the only woman to have served as President of the U.S. Section of the Royal Society of Chemistry, which serves several thousand members within the USA.



About the Author Debbie Schatzlein

Company Intro

About Thermo Fisher Scientific

Thermo Fisher Scientific is the world leader in serving science. Our mission is to enable our customers to make the world healthier, cleaner and safer. With revenues of \$17 billion, we have approximately 50,000 employees and serve customers within pharmaceutical and biotech companies, hospitals and clinical diagnostic labs, universities, research institutions and government agencies, as well as in environmental and process control industries. We create value for our key stakeholders through three premier brands, Thermo Scientific, Fisher Scientific and Unity Lab Services, which offer a unique combination of innovative technologies, convenient purchasing options and a single solution for laboratory operations management. Our products and services help our customers solve complex analytical challenges, improve patient diagnostics and increase laboratory productivity.

Contact Us

For additional information or to schedule a demo, please click below.

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