A WHITE PAPER FROM
SPECTRO ANALYTICAL INSTRUMENTS



Analyzing Precious Metals

Introduction

Precious metals require — and reward — careful analysis. Their high monetary value means that purity is a prime consideration when trading in these metals or products made from them. Different alloys must be identified and their composition verified. Adulteration, while not always easy to detect, can dramatically affect value.

Analysts face various difficulties. The scope of precious metals analysis extends from trace levels to 100%. Most of these metals are resistant to dissolution by all but the strongest acids. Some traditional analytical methods like fire assay are time-consuming and demand a high level of skill.

Three modern techniques offer widely used solutions. Energy-dispersive X-ray fluorescence (ED-XRF) and optical emission spectrometry (OES) can be used without specialist analytical training to rapidly and accurately analyze bullion, jewelry, and alloys. A variation of OES — inductively coupled plasma optical emission spectrometry (ICP-OES) — is an ideal tool for the analysis of bulk materials such as ores, for the determination of trace impurities, and for the high-precision determination of major components in precious metal alloys and recycling products.

Several instruments available from SPECTRO Analytical Instruments represent the state of the art in these techniques. This paper describes their application to precious metals analysis.



CURRENCY TO CHEMISTRY: THE VALUE OF PRECIOUS METALS

The following are traditionally classified as precious metals: gold, platinum, iridium, palladium, osmium, silver, rhodium, ruthenium, and rhenium. All are metallic elements that have achieved high monetary value due to their rarity and special properties.

While the latest-known, rhenium, was discovered in 1925, gold has been known since antiquity, largely because it is found in nature as a free metal. Gold is the exemplar of an important characteristic of this class: resistance to corrosion and oxidation. This provides permanence, luster, and suitability for jewelry and coinage. Other precious metals like platinum and silver also occur naturally in metallic form, often alloyed with other metals.

Extraction can be very difficult, with only tiny concentrations present in most deposits. Precious metals are extremely rare — typically only a few parts per billion (ppb) in the earth's crust. Silver is the most abundant, at about 75 ppb, or 0.0000075%. This compares with aluminum at around 8%, and iron at 5%. Even "rich" deposits of precious metals have concentrations measured in parts per million (ppm). However, their value is not necessarily proportional to scarcity. Some are traded as commodities, and acquire inflated values via speculation in times of financial uncertainty. Indeed, four precious metals are regarded as convertible currencies and possess ISO 4217 currency codes: gold, silver, platinum, and palladium.

Other terms are sometimes used to describe these elements. Noble metals are characterized by their high resistance to corrosion. These can be precious metals, but may also include nonprecious metals such as mercury. Platinum group metals or elements are also known as PGMs or PGEs. These six transition elements — platinum, iridium, palladium, osmium, rhodium, and ruthenium — are clustered together in the periodic table, have broadly similar properties, and tend to occur in the same mineral deposits.

Whole industries exist to recover valuable metals from secondary sources such as scrap jewelry, electronic wastes, and junked vehicle catalysts. At the other end of the production chain, it's economically viable to exploit ores with quite low precious metal content. Improved extraction technology and higher prices make it practical to rework old mine dumps, extracting metals left behind by older, less efficient extraction methods. Evaluating these sources requires analysis to the sub parts-per-million level.



Mineral deposits worthy of extraction exist in few locations. Russia and South Africa are the world's major sources of gold and platinum. Where they occur, precious metals are often found alloyed with each other. They are also found in deposits of other base metals such as copper and nickel that are mined in huge quantities, which can justify the extraction of the precious metals as by-products. Although methods have improved, extraction and purification are still complex, capital-intensive processes, further increasing prices.

Estimates suggest that up to 30% of today's gold supply is recycled metal. Besides its financial merits, recycling is driven by legislation: in the EU, the Waste Electronic and Electrical Equipment (WEEE) Directive requires that suppliers and users of electrical and electronic equipment ensure that disposal at the end of the equipment's useful life is environmentally sound. Recovery of precious metals is often part of that disposal process. Similarly, the EU's End-of-Life Vehicles (ELV) Directive requires that potentially polluting components (including catalytic converters, which contain platinum and often palladium and rhodium) be removed and properly disposed of by an authorized processor.

Karats and Conventions

Precious metals in bulk that are traded by weight are known as bullion. This term also applies to gold coinage when the price depends on the purity and mass of the coin, not its face value. In jewelry, and other consumer goods such as silver tableware, a number of conventions have developed to describe the purity of the metal. These are more familiar within the industry than the concentration units used by analysts.

The essential measurement of gold is usually expressed in *karats* — a unit of *purity* — where pure gold (or fine gold) is defined as 24 kt. (Note that a *carat* — a unit of mass, where 1 carat equals 0.2 gram — instead expresses the *weight* of a precious stone, particularly a diamond.)

Pure 24 kt. gold is too soft to be practical for jewelry, being liable to scratching or deformation. So gold is often mixed with other metals; silver and copper are its most common alloying elements. Carefully controlling concentrations can produce alloys that have the appearance of gold, but quite low actual content. Metals such as zinc are also used in low concentrations, to increase hardness. These alloys may still be sold as "gold,"

% w/w	fineness	karat
99.999	999.99	24
91.67	916.7	22
75.0	750	18
58.5	585	14
33.3	333	8

although most countries set a limit for gold content below which the word cannot be applied. Another popular jewelry alloy is white gold, in which gold is alloyed with metals such as palladium or nickel. When used in jewelry, white gold is sometimes rhodium-plated; rhodium is also "white," and if overlooked could cause errors with some analytical techniques.

Platinum is also widely used in jewelry, alloyed with other PGMs such as palladium and iridium.

Most platinum jewelry is designated with a purity code of 950, or 95% pure; common platinum alloys are Pt950/Ru, Pt950/Ir, Pt900/Ir, and Pt950/Co. A common silver alloy, known as sterling silver, is 925, or 92.5% silver; coin silver is 900 or 90%. Whatever convention is used, in most countries precious metal artifacts are stamped with a hallmark to indicate their purity.

Non-Jewelry Applications

The chemical and physical properties of precious metals are useful in many other applications. For example, despite their apparent inertness, PGMs (platinum and palladium in particular) are excellent catalysts. Automobile catalysts (used in emission control systems) account for around half the platinum mined; PGM catalysts are also used in chemical synthesis and petroleum refining. The electronics industry is another major consumer: gold contacts are a familiar feature of printed circuit boards. Gold, platinum, and palladium are utilized in dental alloys for their insolubility and permanence. These and a host of other applications of precious metals not only require raw materials with analyzed com-position, but also produce waste that can be treated to recover these valuable materials.





THE ROLE OF ANALYSIS

Elemental analysis plays a central role in the precious metals industries. Typical tasks include:

- Verification of purity and composition, including hallmarking, for trading purposes
- Identification of alloys
- Measurement of impurities and adulteration
- Analysis of scrap and processed materials during recycling
- Process control
- Prospecting

These analyses involve a huge range of concentrations, from pure metal to sub-ppm. They also encounter a wide range of sample types, from bullion to jewelry to "sweeps" from working areas to scrap items to bulk geological samples.

A common requirement: quick assessment of the composition, and hence the value, of bullion, coins, and jewelry. In trading, an answer should ideally be available on the spot. Gold, for example, can be traded via a number of channels, including jewelers, dealers,



pawn shops, and so on. For small quantities, payment may be on the basis of hallmarks, or on the simple tests described below. Eventually, the scrap is melted, cast into bars, and sold to a refiner, often in the form of lots: bars of impure metal that might contain 40%–60% gold mixed with silver and other metals.

Two major sources of recycled precious metals are auto catalysts and electronic waste (e-waste). Components containing precious metals (e.g., catalytic converters or printed circuit boards) are removed and sent to specialist treatment companies. In the case of catalytic converters, the PGM catalyst is usually distributed on ceramic granules at up to 0.2% metal content. This is removed from its steel canister and milled to a fine powder containing the PGMs, for further processing. Printed circuit boards and similar electronic waste products are usually shredded, then subjected to smelting and/or leaching processes to extract precious metals. Prices are based on analyses of this powdered or shredded material.

Ultimately, payment must be based on analysis. Results that are of sufficient accuracy, and that can be obtained immediately, in-house, by personnel without special analytical skills, would be ideal. Clearly, it's a distinct disadvantage for any party in a transaction to rely on time-consuming and expensive external analysis, or on cheaper but perhaps less accurate internal testing. Unfortunately, traditional testing methods are either too inaccurate or time-consuming, or require a suitably equipped laboratory.

Traditional Analysis Methods

By nature, precious metals are difficult to analyze. The metals and jewelry trades have long relied on some simple tests, but these can give only approximate results for elemental composition:

- **The acid test.** With some variation, the basic test involves rubbing the sample on an abrasive stone to remove a minute trace of metal, which is left as a mark on the stone. This trace is then tested for solubility (indicated by a color change) in acids of different strengths, each matched to a different karatage of gold. This test can also distinguish some silver grades, and provide a yes/no test for platinum. It's cheap but very crude, limited to differentiating between commonly used jewelry grades.

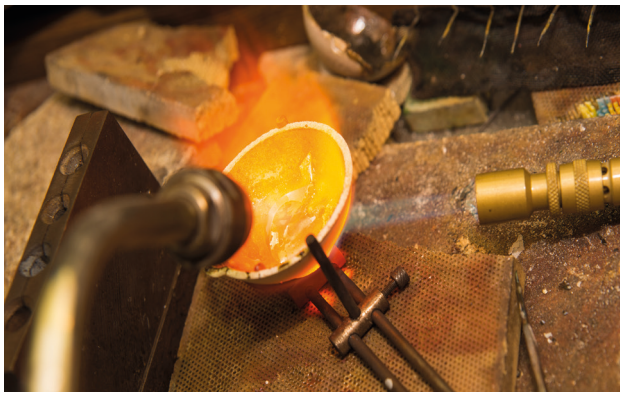
- **Conductivity**. This test meters the electrical conductivity of the sample. A conductive solution or paste is applied to the sample to establish a good electrical contact.

Neither of the above tests provides more than an indication of elemental composition. Note that both are also unsuitable for anything other than metallic samples.

- **Fire assay**. This is the traditional method for the accurate determination of the purity of gold. The weighed sample is heated with lead to around 1200° C in a porous crucible, made of compressed bone ash or magnesium oxide. This process is known as cupellation. Lead and other base metals are oxidized, and the oxides absorbed into the crucible, while the precious metal remains behind. Any silver in the solidified metal is dissolved in acid, and the remainder — gold — is weighed.

A skilled fire assay operator observing good laboratory practice can achieve accuracies of better than ±0.05%. However, the process takes several hours, so it's unsuitable when spot decisions must be made about an article's value. Although it's very accurate for simple gold/silver/copper jewelry alloys, more complex alloys containing other precious metals require further analysis. Fire assay is also usually regarded as a bulk analysis method, and may not be economical when trying to evaluate an individual jewelry item.

An extension of the fire assay technique, used to evaluate ores and other bulk materials containing low concentrations of precious metals, involves pre-concentrating precious metals by fusing the sample with a flux and a collector, such as lead for gold or nickel for PGMs. Precious metals are scavenged from the melt by the collector metal; other materials are fused into a slag. On cooling, the metals solidify as a button. This is easily separated from the glassy slag, and can then be subjected to fire assay or chemical analysis. Using this method, precious metals can be measured down to ppb levels. However, sampling must be done with great care to avoid errors.



Fortunately, modern instrumental methods have revolutionized the analysis of precious metals. They can achieve much more accurate results than acid or conductivity test methods, and in a fraction of the time needed for fire assay.

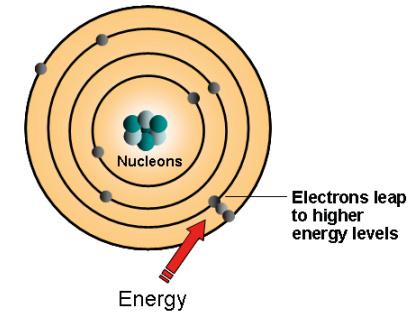
MODERN ELEMENTAL ANALYSIS TECHNOLOGIES

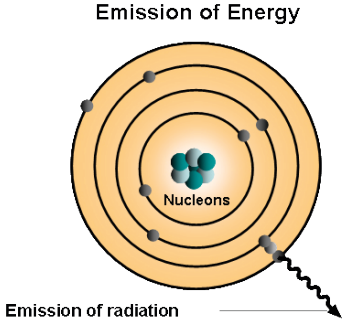
Energy-dispersive X-ray fluorescence (ED-XRF) and optical emission spectrometry (OES) can be used by operators without analytical knowledge to deliver accurate analyses of even complex precious metal alloys — in seconds rather than hours. **Inductively coupled plasma optical emission spectrometry (ICP-OES)** is an excellent tool for the analysis of bulk materials like ores, and for the determination of trace impurities.

All three methods work on the spectroscopic principle, which relies on the internal structure of the atoms of the material being analyzed. In emission spectroscopy, atoms of the sample are excited by an external source of energy, which is absorbed by and raises the energy level of the electrons in the sample atoms. This excited state is unstable, so the electrons rapidly return to their normal state, re-emitting energy as they do so. The energy emitted, or emission spectrum, is characteristic of the elements contained in the sample; its intensity is proportional to their concentration. In X-ray fluorescence, the excitation involves interaction with the inner shell electrons of the atom, which are ejected from the atom. The open position is filled by an electron from a more outer shell, and a characteristic fluorescence radiation is emitted.

The techniques differ in the type of energy used (which also governs the type of samples that can be handled), and in the technology used to analyze and detect the emitted radiation. The following table summarizes the main features of the techniques and their uses in precious metals analysis:

Technique	Excitation	Spectrum Analysis	Detection	Typical Analyses
ED-XRF	X-rays from low power (40 W or less) X-ray tube	Solid state Silicon Drift Detector (SDD) or Si PIN Detector, capable of discriminating between emissions from different elements.		Purity of solid metals eg bullion, pin samples, coins, jewelry, hallmarking. PM's in bulk recycled materials catalysts, electronic waste
OES	Electric arc or spark discharge	Optical poly-chromator using diffraction grating	CCD or CMOS detectors	Impurities in metals, eg bullion, pin samples
ICP-OES	Inductively coupled plasma	Optical poly-chromator using diffraction grating	CCD or CMOS detectors	Traces of PM's in fire assay "buttons". Impurities and major components in PM's and alloys





The spectrometric principle of optical emission: electrons become excited and subsequently emit characteristic energy as they return to normal.

ED-XRF Analysis of Precious Metals

ED-XRF is the most widely used analytical technique in the precious metals industry. Unlike many spectroscopic techniques, it doesn't require the sample to be atomized to enable excitation. So it can analyze solid samples directly. Unlike methods requiring fusion or dissolution, it's nondestructive. This is critical to its usefulness in analyzing jewelry and other valuable items without damage or removal of precious metal. ED-XRF is also capable of quantifying all the sample's elements of interest in a single measurement. Finally, it's ideal for the detection of counterfeiting and for other forensic work.



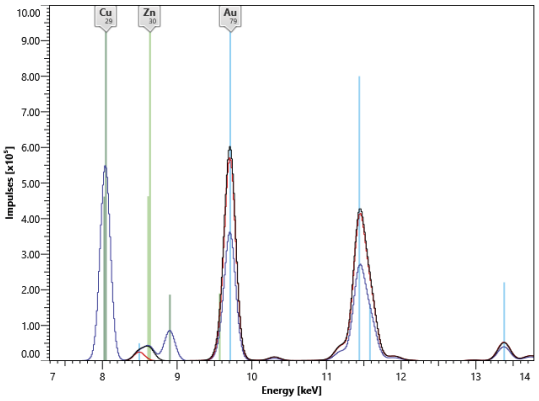
SPECTRO MIDEX and SPECTROCUBE

Users of the SPECTRO MIDEX and SPECTROCUBE small-spot ED-XRF spectrometers from SPECTRO Analytical Instruments report that these products approach fire assay in terms of precise results, while maintaining all XRF advantages. These midrange analyzers have recently been improved or newly launched with a number of technical innovations and user-friendly features. They are standard tools for laboratories where better precision or faster analysis is needed — in testing offices, assay offices, hallmarking centers, precious metal refineries, and more.

Components of both instruments include a 40 W molybdenum X-ray tube generating a standard measurement spot size of 1.2 mm. The analyzers also feature a large-area high-resolution silicon drift detector (SDD). In addition, a high-speed readout system provides an ultra-high count rate combined with excellent resolution. This also contributes to the systems' outstanding sensitivity, which is the basis for good precision and also good accuracy.



The fluorescence spectra of precious metals can be extremely complex; some XRF instruments have difficulty separating the spectral lines of individual elements in an alloy. By contrast, the high resolution achieved by SPECTRO MIDEX or SPECTROCUBE shows the clear separation of respective lines.

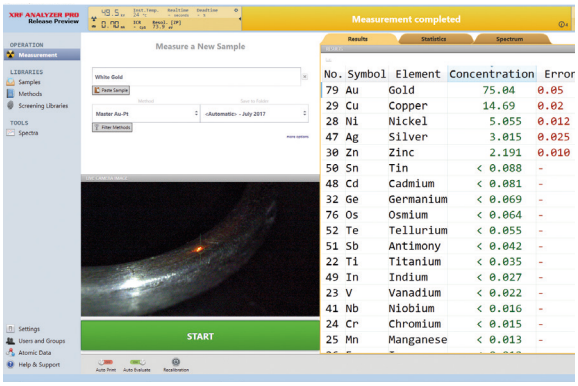


Typical part of spectra from a yellow gold, a white gold and a rose gold sample

In hallmarking centers, the SPECTRO MIDEX can deliver scanning results in as little as 15 seconds — still with good precision and accuracy. On the other hand, refiners may choose to take advantage of the unit's improved sensitivity to allow for lower detection limits for some trace concentrations — thus enabling refiners to avoid overpayment due to overmeasuring gold content. This is achieved within the short measurement times reached by previous models.

SPECTRO MIDEX was specifically designed to accurately analyze small objects such as jewelry. An optional software-controlled collimator changer goes beyond the instrument's standard 1.2 mm spot to allow measuring point sizes from 0.5 mm to 4 mm. (Larger spots can

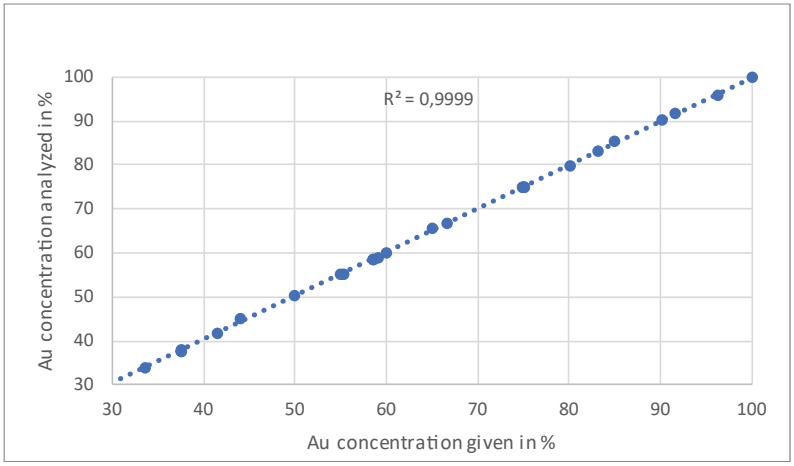
prove advantageous for silver, which, unlike gold, may not be uniformly homogenous throughout a sample's mass.) For irregularly shaped samples (such as slanted bars, high-relief jewelry, or ring inner surfaces), the 5 mm working distance permits focusing on sample points at varying heights. The integrated color video system allows clear imaging and positioning of the sample, plus documentation of the testing spot.



No.	Symbol	Element	Concentration	Error
79	Au	Gold	75.04	0.05
29	Cu	Copper	14.69	0.02
28	Ni	Nickel	5.055	0.012
47	Ag	Silver	3.015	0.025
30	Zn	Zinc	2.191	0.010
50	Sn	Tin	< 0.088	-
48	Cd	Cadmium	< 0.081	-
32	Ge	Germanium	< 0.069	-
76	Os	Osmium	< 0.064	-
52	Te	Tellurium	< 0.055	-
51	Sb	Antimony	< 0.042	-
22	Ti	Titanium	< 0.035	-
49	In	Indium	< 0.027	-
23	V	Vanadium	< 0.022	-
41	Nb	Niobium	< 0.016	-
24	Cr	Chromium	< 0.015	-
25	Mn	Manganese	< 0.013	-

The SPECTROCUBE was specifically designed for high-throughput applications such as hallmarking centers. With its bottom-up-geometry, positioning of jewelry samples is fast and simple. High throughput is also supported by measurement times as low as 15 seconds — twice as fast as other instruments in its class. So the analyzer easily allows testing of several hundred samples per day. A software-controlled collimator changer allows the user to choose from a range of different collimator dimensions (down to a spot size of 0.2 mm). SPECTROCUBE options and analytical performance are the same as those of the SPECTRO MIDEX spectrometer. These instruments are factory-calibrated and validated for an exceptionally wide range of precious metals samples. Their SPECTRO XRF Analyzer Pro operating software was recently optimized via third-party testing and user input to increase

Correlation for the Analysis of Au in Au Alloys (SPECTROCUBE)



Repeatability for Gold in gold alloys (SPECTRO MIDEX)

	Au	Ag	Cu	Zn	Ni
Replicate 1	75.03 ± 0.05	3.029 ± 0.025	14.67 ± 0.02	2.197 ± 0.010	5.072 ± 0.012
Replicate 2	75.05 ± 0.05	3.028 ± 0.024	14.67 ± 0.02	2.179 ± 0.010	5.057 ± 0.012
Replicate 3	75.08 ± 0.05	2.981 ± 0.024	14.67 ± 0.02	2.199 ± 0.010	5.071 ± 0.012
Replicate 4	75.04 ± 0.05	3.016 ± 0.025	14.68 ± 0.02	2.181 ± 0.010	5.083 ± 0.012
Replicate 5	75.08 ± 0.05	2.990 ± 0.024	14.67 ± 0.02	2.195 ± 0.010	5.066 ± 0.012
Replicate 6	75.04 ± 0.05	3.008 ± 0.025	14.66 ± 0.02	2.215 ± 0.010	5.074 ± 0.012
Replicate 7	75.06 ± 0.05	2.991 ± 0.025	14.67 ± 0.02	2.221 ± 0.010	5.049 ± 0.012
Replicate 8	75.09 ± 0.05	2.964 ± 0.024	14.67 ± 0.02	2.212 ± 0.010	5.056 ± 0.012
Replicate 9	75.03 ± 0.05	3.009 ± 0.025	14.67 ± 0.02	2.215 ± 0.010	5.072 ± 0.012
Replicate 10	75.04 ± 0.05	3.015 ± 0.025	14.69 ± 0.02	2.191 ± 0.010	5.055 ± 0.012
Average	75.05	3.003	14.67	2.200	5.066
Std dev	0.02	0.021	0.01	0.015	0.011

Gold Alloys (SPECTRO MIDEX)

Sample		BAM EB 506	BAM EB 507	BAM EB 508
Au	given in %	58.56 ± 0.06	75.10 ± 0.11	75.12 ± 0.11
	analyzed in %	58.71 ± 0.03	75.05 ± 0.03	75.16 ± 0.03
Ag	given in %	3.90 ± 0.05	3.02 ± 0.05	24.90 ± 0.05
	analyzed in %	3.86 ± 0.01	3.00 ± 0.01	24.80 ± 0.03
Cu	given in %	35.65 ± 0.06	14.69 ± 0.05	
	analyzed in %	35.50 ± 0.01	14.67 ± 0.01	
Ni	given in %		4.99 ± 0.04	
	analyzed in %		5.06 ± 0.01	
Zn	given in %	1.891 ± 0.018	2.107 ± 0.016	
	analyzed in %	1.921 ± 0.005	2.160 ± 0.005	

ease and effectiveness. The instruments' SPECTRO FP+ fundamental parameters calibration package not only provides excellent accuracy up to 100% concentration levels, but can analyze completely unknown precious metal alloys. To demonstrate its accuracy, SPECTRO MIDEX was used to analyze a variety of precious metal samples with certified compositions. The measurement time for these analyses was 60 seconds per sample.

SPECTRO xSORT

The SPECTRO xSORT is a portable, handheld XRF instrument designed for rapid identification and analysis of precious metals. The device is offered with two detector options: the Si-PIN detector provides an economical solution for high-throughput testing of precious metal alloys, while the optional Silicon Drift Detector (SDD) allows for shorter measurement times or enhanced precision during analysis due to its higher count rate capability. Additionally, the superior spectral resolution of the SDD enables lower detection limits, which is advantageous when analyzing possible contamination within alloys.



Both configurations feature methods employing either a large spot (5 mm) or a small spot (3 mm), allowing users to select the most suitable option based on specific analytical requirements. For instance, when testing larger silver samples with potential inhomogeneities, the larger spot is recommended. Conversely, for the assessment of finished jewelry pieces, the small spot is typically preferred. All relevant metals in the samples are analyzed at the same time. Results can be viewed either on the instrument's touchscreen or through the XRF Analyzer Pro software on an external computer, which is commonly used when the device is docked. The first set of results can be obtained within just a few seconds.

The table below presents analysis data for three international reference samples, measured using the SPECTRO xSORT equipped with an SDD and a 5 mm spot size, with each sample tested for 30 seconds.

	ERM-EB 506 Rose gold		ERM-EB 507 White gold		ERM-EB 508 Yellow gold	
Sample	Certified [%]	Analysis [%]	Certified [%]	Analysis [%]	Certified [%]	Analysis [%]
Au	58.56 ± 0.06	58.52 ± 0.06	75.10 ± 0.11	74.97 ± 0.07	75.12 ± 0.11	75.22 ± 0.08
Ag	3.90 ± 0.05	3.87 ± 0.02	3.02 ± 0.05	3.08 ± 0.02	24.90 ± 0.05	24.78 ± 0.04
Cu	35.65 ± 0.06	35.71 ± 0.04	14.69 ± 0.05	14.57 ± 0.01		
Ni			4.99 ± 0.04	5.25 ± 0.01		
Zn	1.891 ± 0.018	1.91 ± 0.01	2.107 ± 0.016	2.14 ± 0.01		

Analysis results for ERM-EB 506, 507, and 508 using the SPECTRO xSORT



SPECTROSCOUT

The SPECTROSCOUT is a portable XRF instrument designed for rapid identification and analysis of precious metals.

With technology based on the proven, popular SPECTRO xSORT handheld, the latest SPECTROSCOUT analyzer provides much of that instrument's ease of use and onsite transportability. But its robust construction and silicon drift detector (SDD) X-ray technology — with performance superior to that of the Si-PIN detectors used in many competitive instruments — also packs some of the analytical power of advanced benchtop laboratory analyzers like the SPECTRO MIDEX and SPECTROCUBE D. Thus SPECTROSCOUT can deliver fast, dependable, nondestructive analyses, truly lab-quality results on the spot — all at surprisingly low cost.

SPECTROSCOUT provides precision and accuracy over the wide range of concentrations typically found in precious metals alloys. Critically, it also offers rapid readout — only 30-90 seconds per measurement! So high performance and maximized throughput make SPECTROSCOUT a superb solution for small shops or portable use, or an ideal backup system for larger operations. The table below presents analysis data for two international reference samples, measured using the SPECTROSCOUT equipped with a 1 mm spot size, with each sample tested for 30 seconds.

Sample	kt	Certified Au in %	Analyzed Au in %
NCS HC 54918	22	91.69 ± 0.09	91.69 ± 0.13
NCS HC 54911	18	74.99 ± 0.04	75.19 ± 0.11

Example analysis results for the analysis of Au-based alloys using the SPECTROSCOUT



SPECTRO XEPOS

Even after preliminary processing, concentrations of precious metals in bulk samples such as catalysts and electronic wastes are much lower than in jewelry scrap, and require more sensitive techniques for analysis. While the SPECTRO MIDEX and SPECTROCUBE analyzers have limits of detection in the ppm range, sampling errors could still arise due to nonhomogeneous samples and relatively small measurement windows. Users tasked with these applications can turn to the high-end SPECTRO XEPOS HE XRF spectrometer—SPECTRO Analytical's most powerful XRF analyzer.

Its innovative components include a 50 W / 60 kV X-ray tube, an ultra-high-count SDD, and unique adaptive excitation technology. As a result, it can furnish previously impossible sensitivity boosts of up to 10x compared with previous models — optimized to target precious metals elemental groups. It can also achieve notably low limits of detection (LODs). This is a key advantage when analyzing for the relatively low concentrations found in automotive catalysts.

Besides high resolution and sensitivity plus low LODs, the SPECTRO XEPOS can provide reduced measurement times, low consumables use, and excellent long-term stability. Compared to small-spot excitation, the analysis area of the sample is larger using the SPECTRO XEPOS, especially when utilizing optional sample spinning during measurement. This helps to reduce the effects of inhomogeneities.

Example: To analyze recycled automotive catalyst, the sample is ground to approximately 100 μm or smaller, and the resulting powder is poured into a sample cup or pressed into a pellet after mixing with a binder. The SPECTRO XEPOS results below were obtained with NIST reference materials — and show excellent agreement with the certified values.

Element	Certified Values [mg/kg]	Analyzed Values [mg/kg]
Rh	135.1 ± 1.9	137.1 ± 1.0
Pd	233.2 ± 1.9	235.4 ± 0.6
Pt	1131 ± 11	1124 ± 2

Analysis results for validation sample NIST 2557 with SPECTRO XEPOS

Element	Certified Values [mg/kg]	Analyzed Values [mg/kg]
Rh	51.2 ± 0.5	47.2 ± 0.4
Pd	326 ± 1.6	312.8 ± 0.6
Pt	697.4 ± 6.3	676 ± 1.7

Analysis results for validation sample NIST 2556 with SPECTRO XEPOS

OES Analysis of Precious Metals



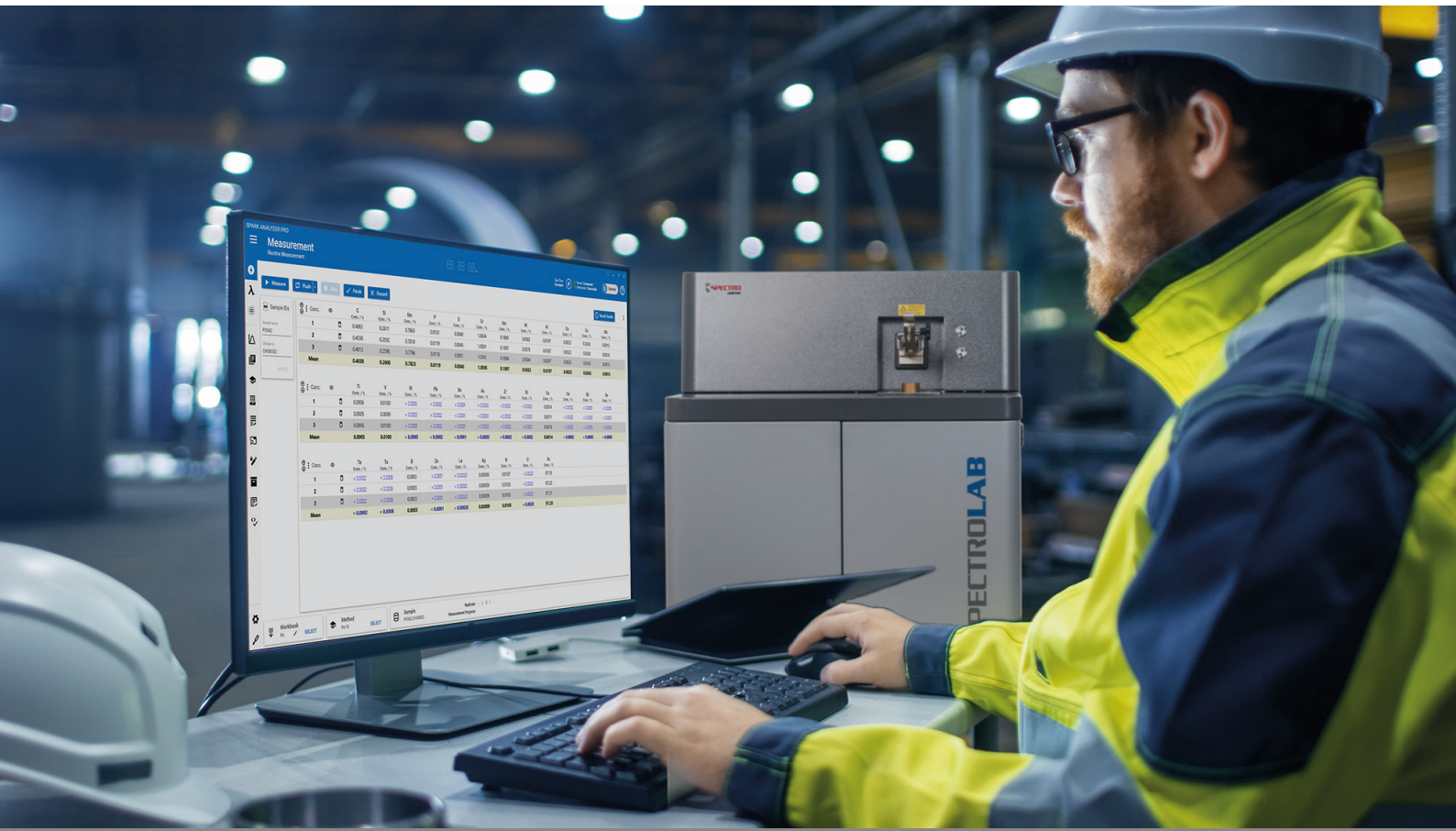
Refiners and alloy producers have particular requirements in determining precious metals impurities. Optical emission spectrometry (OES) is an ideal technique for their needs. Like ED-XRF, it can be used directly on metallic samples such as pins. It is, however, not completely nondestructive; a tiny amount of material is atomized by the spark used to excite the sample.

SPECTROLAB

The SPECTROLAB S high-performance arc/ spark OES analyzer offers a number of advantages for this work, even when compared to other OES instruments. The SPECTROLAB S perfected the use of solid-state detectors with the proprietary CMOS+T technology to revolutionize high-end arc/spark OES analysis. It's designed to supply the fastest possible measurements; lowest limits of detection; longest uptime; and exceptionally accurate, simultaneous analysis of precious metals.

The unit provides ultra-high speed of measurement, ultra-low LODs, ultimate elemental flexibility, outstanding stability, and affordable cost of ownership. Refineries must produce precious metals of very high purity as a starting point for new alloys.

The improved background correction and low detection limits achievable with SPECTROLAB S — combined with its speed of analysis, simple sample preparation, and ease of use — make



it suitable for refining quality control and similar tasks. SPECTROLAB S can be optimized for precious metal matrices of interest: gold, silver, platinum, palladium, rhodium, iridium and ruthenium. Typical LODs (in ppm) of a range of elements in different precious metal bases are shown in the accompanying table.

SPECTROLAB S: Limits of detection (LOD) for selected lines
Note: This data are from the SPECTROLAB Application Notes Arc/Spark No. 84, 85, 105 and 106.

Element	Gold Base [mg/kg]	Silver Base [mg/kg]	Platinum Base [mg/kg]	Palladium Base [mg/kg]
Ag	0.1		0.2	0.2
Al	0.06	0.03	0.1	0.5
As	0.5	0.3	1	2
Au		0.1	0.5	0.5
B			0.1	0.3
Ba			0.2	0.01
Bi	0.5	0.2	0.5	0.5
Ca	0.1		0.1	0.1
Cd	0.03	0.05	0.1	0.1
Co	0.05		0.4	0.5
Cr	0.07	0.2	0.3	0.2
Cu	0.1	0.1	0.1	0.2
Fe	0.2	0.2	0.4	0.5
Ir			2	1
Mg	0.05	0.1	0.1	0.1
Mn	0.05	0.1	0.2	0.2
Mo			0.2	
Ni	0.1	0.2	0.4	0.5
Os			1	1
Pb	0.3	0.5	0.5	1
Pd	0.1	0.2	0.3	
Pt	0.3	0.3		0.5
Rh	0.2		0.3	0.4
Ru			0.4	1
Sb	0.3	0.4	0.5	0.5
Se	0.3	0.2		0.2
Si	0.3		0.2	0.3
Sn	0.2	0.2	0.2	1
Te	0.4	0.5	1	0.6
Ti	0.05		0.5	0.2
Zn	0.05	0.05	0.1	0.5
Zr			0.3	
V			0.2	

ICP-OES Analysis of Precious Metals

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) is widely recognized for its high sensitivity and broad dynamic range, making it an essential technique for analyzing exploration samples such as ores, stream sediments, and impurities in precious metals. Because the method relies on dissolving comparatively large sample amounts, it effectively reduces the impact of sample inhomogeneities—an advantage over techniques such as ED-XRF and OES, provided that sampling protocols are properly designed. Advanced ICP-OES systems achieve detection limits (LODs) in the parts-per-billion (ppb, µg/kg) range while delivering accurate and precise results even for major components.



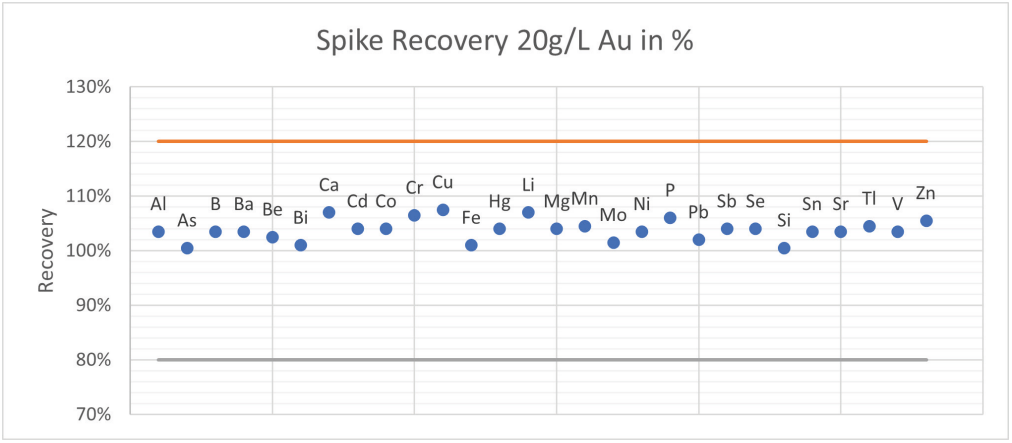
SPECTRO GENESIS

The SPECTRO GENESIS is a compact, fully simultaneous ICP-OES spectrometer featuring Dual Side-On Plasma (DSOI) technology. This design enhances sensitivity by up to a factor of two compared to conventional radial viewing systems. The instrument provides straightforward calibration, an exceptionally wide dynamic range, and precise, rapid analyses due to its simultaneous measurement capabilities. Although the GENESIS does not match the sensitivity of the highest-end instruments, the tables below demonstrate detection limits and spike-recovery performance, confirming its

Element	Wavelength [nm]	LOD (3σ) in solution [mg/L]	LOD (3σ) in the solid material [mg/kg]
Ag	328.068	0.04	2.1
Al	396.152	0.01	0.50
As	193.759	0.05	2.5
B	182.641	0.003	0.15
Ba	455.404	0.0009	0.05
Be	313.042	0.0001	0.005
Bi	223.061	0.02	1.0
Ca	396.847	0.003	0.15
Cd	228.802	0.001	0.06
Co	230.786	0.003	0.17
Cr	283.563	0.002	0.10
Cu	324.754	0.004	0.21
Fe	239.562	0.004	0.18
Ge	265.118	0.02	0.90
Hg	194.227	0.007	0.33
Ir	183.247	0.018	0.91
Li	670.780	0.01	0.60
Mg	280.270	0.001	0.05
Mn	259.373	0.0004	0.02
Mo	281.615	0.005	0.25

Element	Wavelength [nm]	LOD (3σ) in solution [mg/L]	LOD (3σ) in the solid material [mg/kg]
Ni	221.648	0.003	0.14
P	177.495	0.007	0.34
Pb	220.353	0.02	0.84
Pd	324.270	0.02	1.05
Pt	177.708	0.01	0.07
Rh	343.489	0.10	5.0
Ru	240.272	0.01	0.53
S	182.034	0.02	1.1
Sb	206.833	0.02	0.92
Se	196.090	0.03	1.5
Si	251.612	0.006	0.30
Sn	189.991	0.008	0.40
Sr	407.771	0.0007	0.03
Ti	336.121	0.001	0.05
Tl	190.864	0.02	0.80
V	311.071	0.002	0.10
W	207.911	0.09	4.5
Zn	213.856	0.002	0.095
Zr	343.823	0.01	0.50

Typical limits of detection in the original gold material prepared as a 1:50 dilution

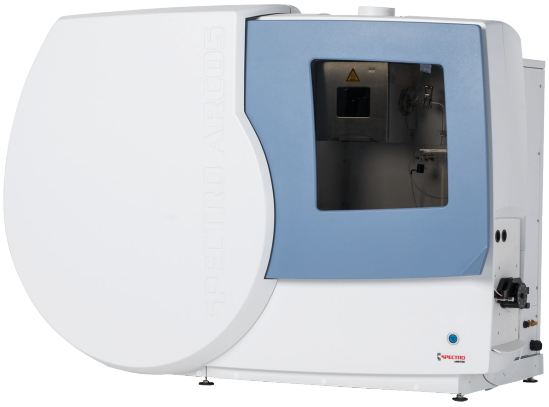


Recovery test of a gold sample spiked with 10 mg/kg (P 50 mg/kg) of the respective elements

suitability for monitoring gold with fineness levels up to 999.97 ‰.

SPECTRO ARCOS

SPECTRO ARCOS represents the high-end class of simultaneous ICP-OES analyzers and incorporates state-of-the-art technology for the most demanding applications in precious-metal analysis. A key advantage of the ARCOS is its MultiView capability, enabling true axial and radial plasma observation within a single instrument. This unique configuration combines the enhanced sensitivity necessary for trace-element determinations with the stability required for accurate major-component analysis. The addition of the DSOI technique further boosts sensitivity by up to a factor of two compared with standard radial observation, significantly extending the instrument's versatility.



The SPECTRO ARCOS achieves exceptionally low detection limits for gold, platinum, and other precious metals. The detection limit data below, obtained using axial viewing, demonstrate its ability to quantify trace impurities at extremely low levels.

Application reports (ICP176 for Ag, ICP177 for Au, ICP178 for Pd, ICP179 for Pt, ICP204 for Ir, ICP205 for Rh, and ICP206 for Ru) highlight the successful use of DSOI across a range of precious metal matrices.

Element	λ [nm]	LOD [mg/kg]
Ag	328.068	0.10
Al	167.078	0.07
As	197.262	1.1
Au	267.595	0.28
Ba	455.404	0.04
Be	313.042	0.004
Cd	226.502	0.02
Co	228.616	0.04
Cr	283.563	0.07
Cu	324.754	0.07
Fe	259.941	0.18
Ir	212.681	0.12
Li	670.780	0.02
Mg	280.270	0.04

Element	λ [nm]	LOD [mg/kg]
Mn	257.611	0.008
Mo	281.615	0.16
Ni	231.604	0.50
P	213.618	1.5
Pb	220.353	0.62
Pd	324.270	0.66
Rh	233.477	0.42
Ru	245.553	0.58
Sb	217.581	0.77
Sn	147.516	0.37
Sr	407.771	0.01
Tl	190.864	0.31
V	292.402	0.07
Zn	213.856	0.02

Typical limits of detection in the original platinum material prepared as a 1:100 dilution.

Element	λ [nm]	LOD [mg/kg]
Ag	328.068	*
Al	167.078	0.01
As	189.042	0.11
Ba	455.404	0.002
Be	313.042	0.001
Bi	223.061	0.11
Cd	214.438	0.006
Co	238.892	0.01
Cr	205.618	0.01
Cu	327.396	0.03
Fe	259.941	0.05
Ga	141.444	0.05
Hg	194.227	0.05
In	230.606	0.02
Ir	183.250	1.3
Mg	280.270	0.008

Element	λ [nm]	LOD [mg/kg]
Mn	259.373	0.006
Ni	221.648	0.04
Pb	220.353	0.10
Pd	340.458	--*
Pt	177.708	0.04
Rh	343.489	0.05
Ru	240.272	0.03
Sb	206.833	0.06
Se	204.050	0.52
Si	251.612	0.06
Sn	147.516	0.05
Sr	421.552	0.001
Te	214.281	0.27
Tl	190.864	0.08
Zn	202.613	0.004
Zr	339.198	0.02

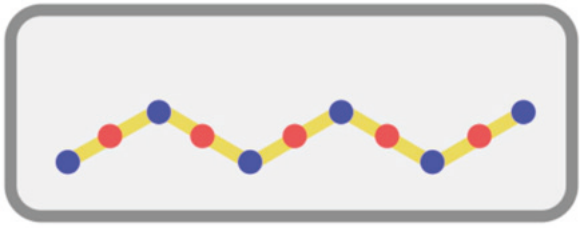
Typical limits of detection in the original gold material prepared as a 1:20 dilution

**Contaminated blank standard*

Bracketing Technique for High-Precision Analysis

To achieve the high precision demanded in precious metal industries, standardized analytical procedures such as the bracketing method have been established. ISO 11494 (platinum) and ISO 11495 (palladium) describe the use of bracketing in combination with yttrium as an internal standard. Historically, bracketing was used for single-element determinations; however, modern instrument software and advanced data-processing capabilities now support multicomponent bracketing in a single analytical run.

The bracketing approach alternates measurements between a low standard, the sample, and a high standard. One complete cycle consists of low standard → sample → high standard. A second cycle repeats the sequence, beginning with a sample measurement, followed by the low standard. This cycle-based approach compensates for instrumental drift and improves overall measurement accuracy. Typically, five cycles are performed. The ICP Analyzer Pro software automates all operational parameters, calculations, and preparation steps required for reliable bracketing.



Schematic drawing of a bracketing sequence. Blue dots represent calibration standards, red dots represent the unknown sample

Results and Practical Application

The excellent performance of multicomponent bracketing in both scrap-metal analysis and the characterization of certified reference materials such as the NIST catalyst standard is demonstrated by the results shown in the tables below. For the NIST standard, analyses of equally prepared samples show remarkably low RSD values and consistent accuracy.

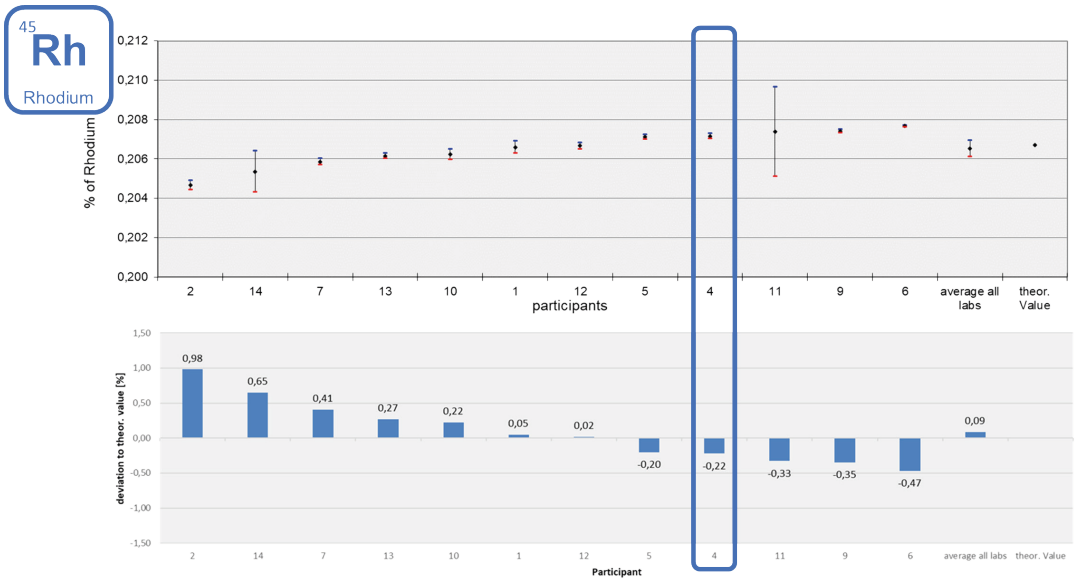
	Pt	Ag	Au	Pd	Ir	Rh	Ru
g/kg	207.94	32.83	81.85	73.87	47.92	11.82	18.66
mg/l	80.90	12.77	31.85	28.74	18.64	4.60	7.26
RSD in %	0.07	0.12	0.13	0.10	0.23	0.26	0.46

Multi-component bracketing from metal scrap

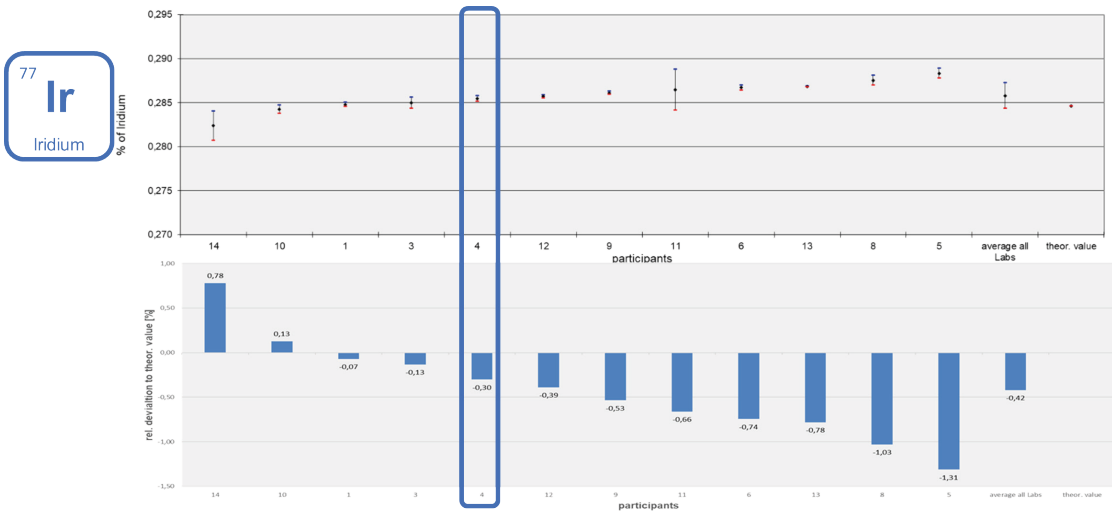
Element	Sample	Measured [mg/kg]	Certified [mg/kg]	Recovery [%]
Pt	1	1104	1131 ± 11	97.6
	2	1102		97.4
	3	1100		97.3
	4	1093		96.6
Pd	1	232.0	233.2 ± 1.9	99.5
	2	232.9		99.9
	3	230.0		98.6
	4	231.1		99.1
Rh	1	127.5	135.1 ± 1.9	94.4
	2	128.1		94.8
	3	127.8		94.6
	4	127.1		94.1

NIST 2557 certified reference material

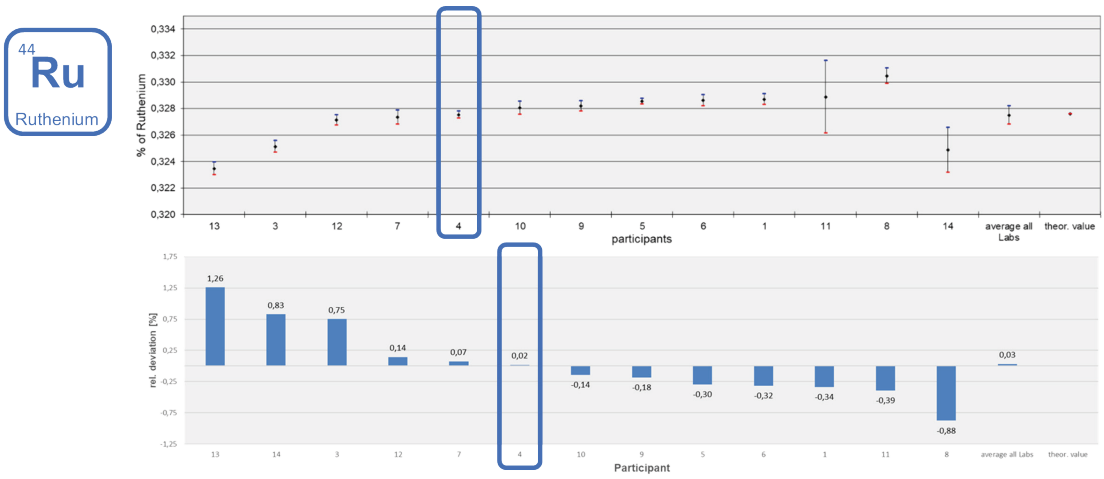
For precious metal alloys—where composition is usually well defined—the method can be optimized and adapted for specific alloy types. Interlaboratory comparison studies involving Rh, Ir, and Ru in alloys such as GDMB-AA-EM 2022-5 illustrate this capability. Measurements obtained with SPECTRO ARCOS using multi-component bracketing after aqua-regia digestion show excellent agreement with theoretical values and with results from other laboratories, confirming the robustness and reliability of the technique.



Rhodium concentration results in an alloy (GDMB AA-EM 2022-5) from a round robin test



Iridium concentration results in an alloy (GDMB AA-EM 2022-5) from a round robin test



Ruthenium concentration results in an alloy (GDMB AA-EM 2022-5) from a round robin test

CONCLUSION

From the rapid assessment of an item of jewelry to the determination of minute traces in ore, detecting and measuring precious metals presents substantial challenges. Traditional methods of analysis are either inaccurate or extremely time-consuming and skill-dependent.

XRF, OES, and ICP-OES analyzers from SPECTRO Analytical Instruments can meet all these challenges. Their performance can serve widely differing needs from the gold dealer to the refinery. These instruments represent the latest developments in analytical technology in their respective techniques. Often, where traditional methods may take several hours, operators without specialized analytical knowledge can complete detailed, accurate precious metals analyses in seconds.

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SPECTRO AMETEK

GERMANY
SPECTRO Analytical Instruments GmbH
Boschstrasse 10
D-47533 Kleve
Tel. +49.2821.892.0
spectro.sales@ametek.com

U.S.A.
SPECTRO Analytical Instruments Inc.
50 Fordham Rd
Wilmington 01887, MA
Tel. +1 800 548 5809
+1 201 642 3000
spectro-usa.sales@ametek.com

CHINA
AMETEK Commercial
Enterprise (Shanghai) CO., LTD.
Part A1, A4 2nd Floor Building No. 1 No. 526 Fute
3rd Road East; Pilot Free Trade Zone
200131 Shanghai
Tel. +86.400.022.7699
spectro-china.sales@ametek.com

Subsidiaries:
▶**FRANCE:** Tel. +33.1.3068.8970, spectro-france.sales@ametek.com ▶**GREAT BRITAIN:** Tel. +44.1162.462.950, spectro-uk.sales@ametek.com
▶**INDIA:** Tel. +91.22.6196.8200, sales.spectroindia@ametek.com ▶**ITALY:** Tel. +39.02.94693.1, spectro-italy.sales@ametek.com
▶**JAPAN:** Tel. +81.3.6809.2405, spectro-japan.info@ametek.co.jp ▶**SOUTH AFRICA:** Tel. +27.11.979.4241, spectro-za.sales@ametek.com

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