Application Note

A Tale of Two Refineries

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Fire assay is an established and proven method for testing the purity of gold jewelry and other objects. Nevertheless, it is a destructive and time-consuming process requiring the use of hazardous chemicals. X-ray fluorescence has emerged as a non-destructive alternative to the fire assay method, with comparable accuracy in just minutes. XRF instrumentation is now available in portable units ideally suited for countertop use in retail environments for accurate testing of any precious metal. In a test against two different refineries using fire assay to evaluate two gold ingots from the same melt, an XRF analyzer provided results just as accurate as one refinery, and even more accurate than the second refinery. The test results provided in this article were performed using the Thermo Scientific™ Niton™ DXL Precious Metal Analyzer.

What is Fire Assay?

The livelihood of businesses that buy and recycle gold, such as jewelry stores and pawn brokers, depends on being able to determine the accurate karat (K) weight of gold jewelry. Failing to detect just a small variation in composition can be an expensive mistake. Many of these businesses use fire assay testing to evaluate their gold, but this method, while an industry standard analytical process, requires many steps. Fire assay is a method aimed at separating the gold from all the other metals in a given piece of jewelry. Although the gold is retained at the end of the process, fire assay is a destructive method that requires the removal of a small sample from the jewelry item.

The sample is weighed and mixed with a flux (such as soda ash or borax) and lead (or silver) in a crucible. The flux reduces the melting temperature while the lead acts as a collector material. The sample is then heated and melted at a temperature range of 1000°C-1200°C, at which point the precious metals are collected by the molten lead and settle at the bottom of the crucible as a "lead button," leaving behind the base metals and other impurities in the slag.

Gold content analysis using the Niton DXL XRF analyzer (above) and the traditional fire assay method (below).

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Upon cooling, the lead button is separated from the slag and is placed into a refractory-made porous cup ("cupel") and heated in a cupellation furnace. During this step the lead is absorbed into the cupel's pores, leaving behind a precious metal bead ("prill"). To determine the gold content, the bead is dissolved in nitric acid, separating the gold from silver, or as an alternative, the bead can be dissolved in a mixture of hydrochloric and nitric acids ("aqua regia"). In the latter case the gold content can be determined by using one of several analytical methods (AAS, ICPMS, ICPAES).

While fire assay is widely used and considered to be one of the most accurate methods to determine the gold content in jewelry, it requires multiple preparation and processing steps. The method's reliability and accuracy can only then be guaranteed if these steps are carried out meticulously by skilled operators. Any slight procedural deviations in the course of the fire assay can accumulate to a significant error, as the following example demonstrates.

Refineries Provide Conflicting Results

The fire assay process is typically performed in large refineries equipped for this type of analysis. However, not all refineries are created equal as one pawn broker found out when two refineries came up with two significantly different results regarding the content of his precious metal. The broker decided to test out a new refinery by sending his current refinery and the new company ingots of gold scrap from the same melt. The new refiner was sent 850g, while the current refiner was sent 600g.The bar sent to the current refiner was double-fire assayed with a return of 52.7% gold. The new refiner, however, returned 51.2% gold. For this particular transaction, this represents approximately a $150 difference for the metals.

XRF to the Rescue

So how does portable XRF compare to fire assay? Prior to being sent to the refineries, both gold ingots were tested on the broker's Niton DXL machine. The DXL analyzer determined the samples had a gold content of 52.5%, nearly the same as the current refinery's results.

The accuracy of the Niton DXL and XL2 Precious Metal Analyzers is typically within 1/8 of a karat of the fire assay results. In fact, a recent correlation study comparing a Niton instrument to fire assay showed a correlation factor of 0.999. In addition to being a non-destructive method, portable XRF is a much more comprehensive analysis, yet simple, fast, and does not require any hazardous chemicals.

When a sample is measured using XRF, each element present in the sample emits its own unique fluorescent x-ray energy spectrum. By simultaneously measuring the fluorescent x-rays emitted by the different elements in the sample, XRF analyzers can rapidly determine those elements present in the sample and their relative concentrations — in other words, the elemental chemistry of the sample, typically in seconds.

Conclusion

XRF analyzers provide a fast, accurate, and nondestructive method to test the purity and composition of gold and other precious metals, with results comparable to the much more complicated, lengthy, and as we see in this case, possibly faulty fire assay method. XRF quickly provides the exact karat weight and percentages of all elements within an item — easily identifying non-standard, under-karated, and even advanced counterfeit material. What's more, some XRF instruments feature technology that can identify gold-plated items.

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