

# METAL PURITY TESTING

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**Abstract:** There is an increasing demand in precious metal like gold, diamond since they are expensive and cannot be created. Precious metal like gold are tested using acids for their purity which may lead to dissolving of gold in the acid. Hence to avoid corrosion of metal X-ray fluorescence analysis is proposed. The objective of the proposed method is to find the percentage of all the elements present in the any metal. Here X-rays are directed to precious metals(gold) without harming the metal in any way, the x-rays cause them to glow(fluoresce),like in a fluorescent light bulb. With advanced computer detection technology, the machine can separate the colors of the fluorescence into an analysis of the metal. Here the technique used is XRF (X-ray Fluorescence) where x-rays from a sealed tube are used to produce x-rays by secondary fluorescence in samples of interest. The Secondary target fluoresces

and excites the sample. The detector detects x-rays from the sample. Graph is plotted using processing techniques and the elements are detected.

## INTRODUCTION

It is important that NDT personnel have some background in material science for a couple of reasons. First, nondestructive testing almost always involves the interaction of energy of some type (mechanics, sound, electricity, magnetism or radiation) with a material. To understand how energy interacts with a material, it is necessary to know a little about the material. Secondly, NDT often involves detecting manufacturing defects and to find the percentage of elements in the metal and, therefore, it is necessary to understand how defects and damage occur.

Precious metals just as with most precious stones occur as mineral deposits in the ground and require extensive mining and

refining techniques to obtain a 'fine' finished product. Very often though precious metals are found in small pebble or grain deposits, popularized by gold panning in rivers.

A fine finished product would be a precious metal that has had all other materials / ores / impurities removed and is in a highest possible **Carat** and is in its most pure form. Although it is almost certainly impossible to remove every single impurity, fine metal is 99.99% pure.

Carat is the unit of measuring a precious metals compound mixture. (Not to be confused with carat weight of precious stones). Carat age of a metal is calculated in percentage. Although it is displayed and referred to in it's decimal value and / or out of a thousand parts. An example in silver most commonly used is known as 925 and is actually 92.5% fine silver and 7.5% alloy metals. I have described more details about each metals different carat ages and alloys in separate dedicated chapters for each metal. The cartage of gold is a little different..... [Read more about gold](#)

Precious Stones are mostly mounted in precious metals. Depending on the stone different styles of setting and metals are used to obtain a perfect and attractive finish. Most often harder metals are used to set precious stones if the color compliments the stone. Platinum and white-gold are the most popular settings for diamonds, rubies and sapphires not only for the color

and durability but also because a white metal setting can draw a little more light and white into a stone or create the illusion of a larger stone (illusion setting) or help create an *encrusted* effect.

In the past stones used to be set on a slice of foil, usually silver in color, this foil helped to draw light into a stone. This technique is not necessary today as the modern brilliant cut is far superior to the old cuts at drawing in light to the stone.

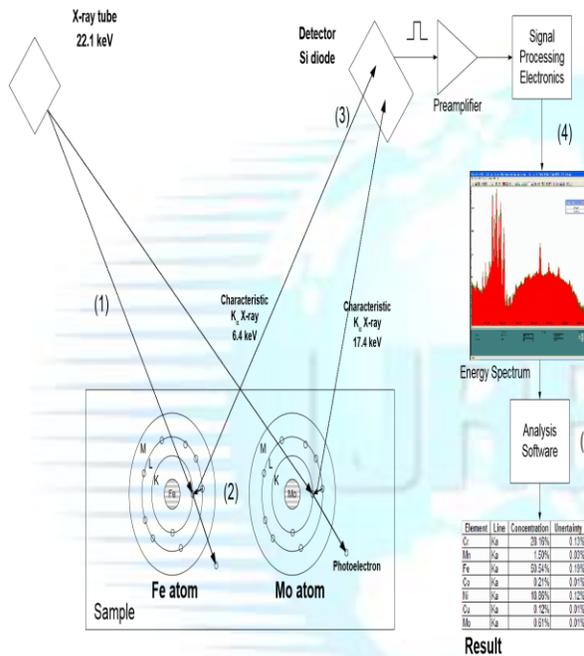
## XRF Spectra and Spectra Analysis Software

X-Ray Fluorescence (XRF) is a very simple analytical technique: X-rays excite atoms in a sample, which emit X-rays at energies characteristic of each element. A detector measures the energies and intensities of the emitted X-rays. From this, one deduces which elements are present and their concentrations.

Setting up a system for first order results is straightforward. One can easily identify the major constituents in simple samples and obtain approximate concentrations. This is sufficient for some applications. However, second order effects are quite important for many applications. To obtain high accuracy, identify constituents with low concentrations, or operate on a wide variety of samples, second order effects must be addressed. To remove these second order effects and obtain high accuracy, properly configuring both the hardware and software is critical.

Understanding the physical mechanisms responsible for these effects is helpful when one is configuring a system to obtain accurate results

HOW DOES AN XRF ANALYZER WORK?



1. Photons from the source irradiate the sample. Shown here is an X-ray tube with Ag anode.
2. Atoms in the sample are excited, then de-excite by emission of X-rays at energies characteristic of the elements in the sample. The intensities are related to the concentration of each element in the sample and to the excitation probabilities.
3. The characteristic X-rays interact in the detector. Each X-ray releases a pulse of charge, proportional to the energy deposited. The signal

processing electronics measures the total charge (i.e. total energy) of each pulse that is of each individual X-ray.

4. The signal processing electronics sum the results of many interactions into a histogram, showing the number of events in each of many energy bins. This is the energy spectrum.
5. The peaks in the spectrum correspond to the elements in the sample. The number of X-rays in each peak is proportional to the number of atoms. Analysis software detects which peaks are present, finds the intensity of each peak, and computes the concentration of each element in the sample.

HOW DOES THE ANALYSIS SOFTWARE WORK?

The matlab software uses the measured spectrum, such as that shown in Figure 13, to deduce the concentration of elements in the spectrum. It essentially inverts the effects described in the previous section. There are three major steps to the analysis:

1. Unravel the detector response to recover the incident photo peaks. The output of this step is a processed spectrum, ideally showing only the incident photo peaks. Figure 14 shows this.
2. Deconvolve the photo peaks to determine the intensity of the X-rays interacting in the detector. The output of this step is a table of the intensities in each photo peak to be analyzed.

Element	Intensity	Known Concentration	Measured Concentration		Chi <sup>2</sup>
Cr	316.6	25.3%	28.20%	+0.13%	32.8
Mn	19.6	17.2%	1.51%	+0.03%	9.9
Fe	444.4	53.2%	50.49%	+0.20%	87.0
Co	2.5	0.0%	0.21%	+0.01%	6.7
Ni	154.2	19.3%	18.85%	+0.12%	24.3
Cu	1.4	0.1%	0.12%	+0.01%	0.5
Mo	17.7	0.4%	0.61%	+0.01%	0.2

- Account for attenuation and matrix effects to determine the concentrations of the elements in the sample. The output of this step is a table of concentrations, as in Table 1.

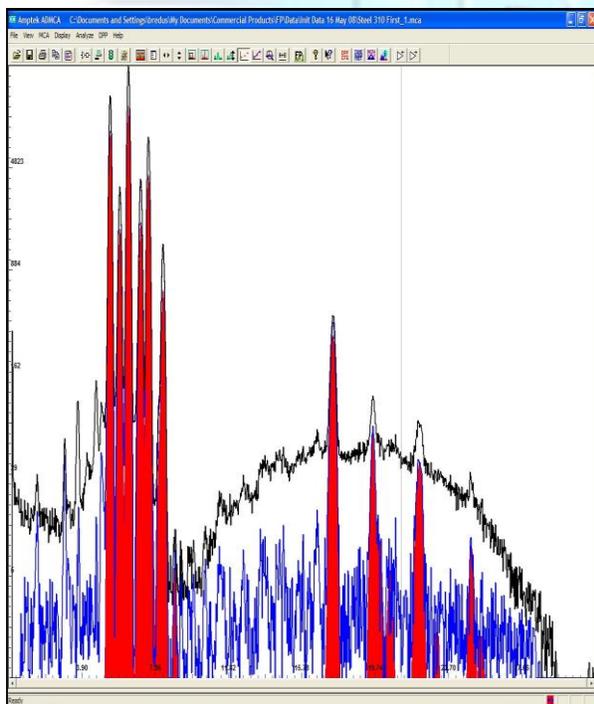


Figure 1: Plot showing the processing of the raw spectrum. The black trace is the original raw spectrum. The blue trace shows the processed spectrum, after correcting for escape peaks, sum peaks, background removal, etc. The red peaks are the result of

the deconvolution, showing the fitted Gaussian photo peaks. The intensities of the characteristic lines are the net area of the peaks.

Table: 1 showing concentration of every metals

## MATLAB STEP BY STEP

We will now examine in more detail the steps by which matlab goes from the raw spectrum to the final result. Before one can analyze a sample, there are several key actions required. They include (1) setting up the hardware, (2) launching and configuring the software, (3) performing an energy calibration, (4) calibrating the FP software using reference materials, and (5) acquiring the spectrum to be analyzed. This example assumes all these have been carried out, the system is configured and calibrated, and a spectrum has been acquired from the sample.

### Unraveling detector response

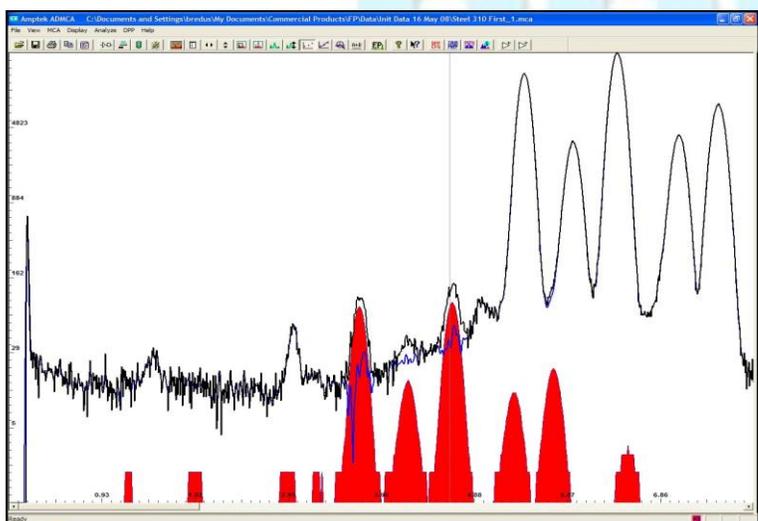
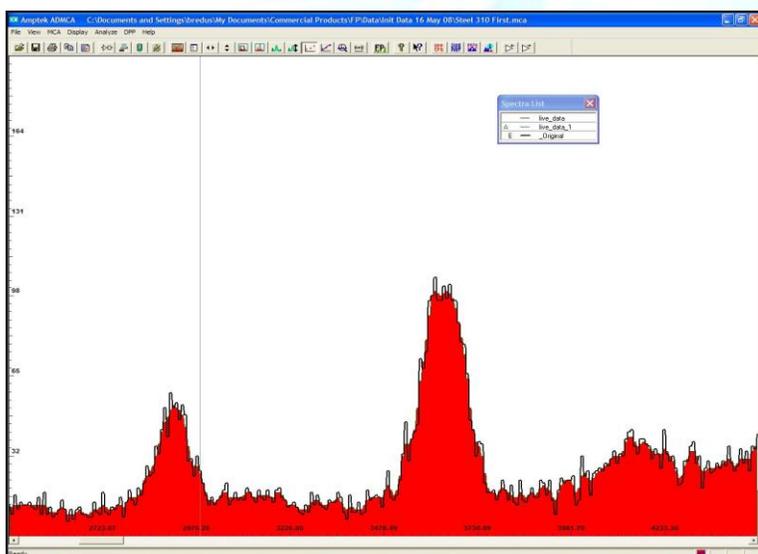
#### Smooth fluctuations

The first step in processing is to apply a filter which smoothes the spectrum. This reduces the effects of counting statistics and will improve the ability of the software to recognize the background and to remove the true peaks from background.

The plot on the right illustrates (in red) the smoothed spectrum derived from the raw data (black trace).

*Correct for escape peaks:* The software applies known corrections to remove the escape peaks and put the counts into the photo peaks. This removes possible interferences and corrects the photo peak net area for escapes.

The plot on the right illustrates (in red) the escape peaks. The black trace depicts the original spectrum, while the blue trace shows the result of the escape peak removal. These false peaks have been removed and their counts added back into the photo peaks.



### *Remove sum peak*

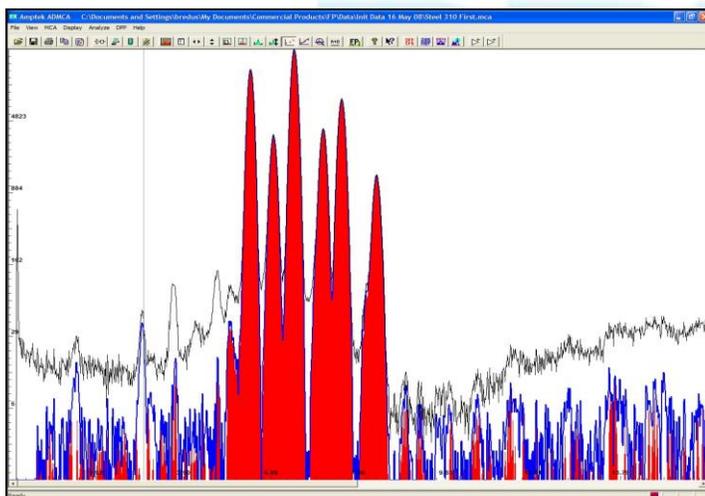
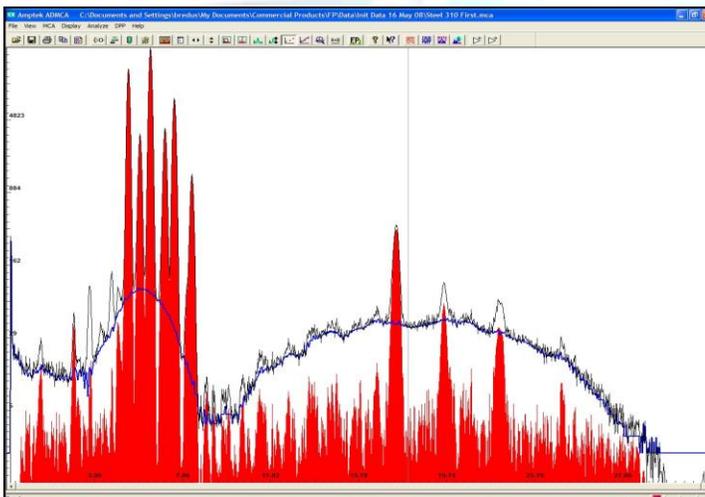
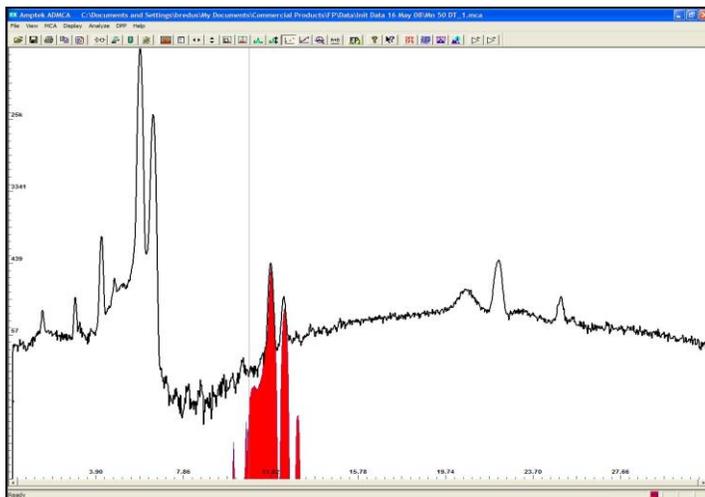
The software applies known corrections to remove the sum peaks and put the counts into the photo peaks. This removes possible interferences and corrects the photo peak net area. The plot on the right illustrates this, using data from a different spectrum, a pure Mn target measured at a 50% dead time. The black trace shows the raw data. The red peaks are the computed sum peaks, which are removed by using software.

### *Remove continuum*

#### *Background removal*

The first background removal step applies what is essentially a low pass filter to estimate the background, using a proprietary function to model the slowly changing background. Without attempting to separate the distinct physical mechanisms, the background is removed. The plot on the right shows the raw spectrum as the black trace, the modeled background as the thick blue trace, and the result in red. The smoothly varying continuum has been removed.

#### *Blank subtraction*



Blank subtraction removes artifacts

which do not vary significantly from one spectrum to another and cannot be handled with other processing. For example, it can remove Ar peaks from air, tube scatter peaks, or some contaminant peaks from the XRF system itself. In the plot on the right, the blue trace shows the result of blank subtraction. The Al and Ar peaks at 1.5 and 3.0 keV have been removed, along with some of the residual features above 10 keV.

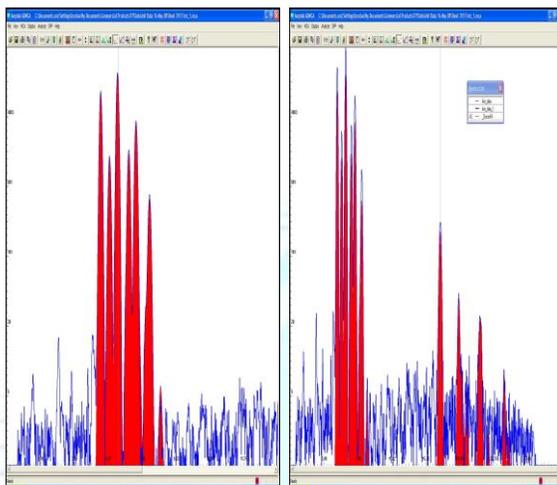
#### 4.1 Compton peak removal

Compton peak processing estimates the intensity of the Compton and Rayleigh scatter peaks. This is used during some of the deconvolution and quantitative analysis methods, depending on the options chosen. The spectrum is not shown here since this does not appear as a change in the spectrum. This processing step produces certain parameters used in deconvolution.

### Deconvolution

Deconvolution is the process of assigning the areas to the peaks of interest. The output of this step is a list of “elemental intensities”, shown in Table 1 as “intensity”. This is depicted in the spectrum display as the Gaussian peaks, with the appropriate areas and centroids. The results of the deconvolution for stainless steel 310 are shown in Figure 15. On the left, one can see the Gaussian fits to the major constituent elements: Fe, Cr, Ni, etc. The various other peaks and bumps are ignored.

On the right, one can see the fit to Mo and also to the scatter peaks arising from the Ag anode.



Plots showing nonlinear Gaussian

There are several options in the software which have a major impact on this deconvolution.

First, note that matlab does not automatically find the peaks in the spectrum. The user must supply a list of peaks to be analyzed (in the Specimen Component Table). The user also selects which peak is the basis for the deconvolution, i.e.  $K_{\alpha}$  line. The other lines will be shown and used in analysis, but the selected line is at the core of the intensity calculation. Matlab can deconvolve as many as 30 peaks. Limiting the peaks to those anticipated in the sample will generally provide the best results.

Second, matlab has three models for the photo peak shape: Gaussian, reference, and integrate. “Gaussian” is best under most circumstance. It does a Gaussian fit to the

photo peak. This is depicted in Figure. Even if the peak is somewhat non-Gaussian, the high quality of the fit to the Gaussian portion can provide excellent results. “Reference” is helpful when a peak is significantly non-Gaussian, such as higher energy peaks measured with CdTe detectors. “Integrate” sums the total counts within an ROI. For simple spectra, this approach is faster and can be quite precise. The user can select which of the three photo peak models will be used for any particular peak.

Third, matlab permits the user to select linear vs. nonlinear deconvolution. In a “linear deconvolution”, the energies, peak widths, and ratios of the intensities are all fixed. The software then does a linear regression to fit the net area (intensity). In the “nonlinear deconvolution”, the software is permitted to adjust the energies, peak widths, and line ratios (within limits) to improve the match.

Fourth, If Compton peak removal is used, and C/R ratio is checked, then matlab will distinguish (and separate deconvolve) peaks due to source backscatter versus the elemental peaks. In Figure these options were checked and so the scatter peaks are included in the deconvolution.

### Quantitative Analysis

This is the step which relates the net intensity of each peak to the concentration in the sample. This processing includes attenuation (including the detector’s sensitivity and losses in the Be window and in air) along with matrix effects, absorption and enhancement in the sample. The result

of the quantitative analysis step is not shown in the spectrum, but in the output table, such as in Table 1. In matlab there are three distinct correction methods that can be used and three different calibration options.

#### *Correction methods*

- **FP (Fundamental Parameters):** This method uses a set of parameters for each element and a matrix for inter-elemental effects. These parameters are based on the “physics” of the materials and geometry, though they may be tuned by calibration.
- **SIR-FP (Scattered Intensity Ratio):** This method uses information from the Rayleigh and Compton scatter peaks to provide additional information about elements (typically with low atomic numbers) that cannot be analyzed, because their x-ray lines are not detectable. Knowledge of these low Z elements is important because it permits one to better treat for matrix effects, thereby improving the accuracy of the elements which are detectable.
- **SLQ (Simple Least Squares):** This method uses a calibrated set of parameters and does a simple least square fit. This method requires calibration of the quantitative analysis parameters.

#### *Calibration Options*

Each option uses certain parameters, which account for attenuation and enhancement. There are default parameters which can be adjusted for a

particular configuration. This adjustment is “calibration” of the quantitative analysis parameters.

- **Standard less:** Uses default values for thickness, scattering, attenuation, etc. These are derived from the physics of the materials. It is extremely critical that the set-up information be accurate!
- **Single Standard:** Uses measured results from a known sample to “tune” parameters for the actual setup. Actually, it will use those elements in that standard to tune parameters for only those elements; others get analyzed via standard less parameters. For example, if one is measuring steel, you can start with a known steel standard, calibrate with that, and then get pretty good results from other, similar steel alloys.
- **Multiple standards (using single element standards):** One starts with many samples, each made of a “pure” element. You read them in, producing an array of coefficients.
- **Multiple standards (multi-element standards):** In this option, you have several standards, each with several elements (and with the same elements in several standards). Each standard will give some nominal parameters; the calibration sequence combines the result for the best fit combination.

There are two distinct calibration steps needed in matlab. First, one must perform

an energy calibration (also called a spectrometer calibration). This assigns an energy scale to the histogram recorded by the spectrometer. To do an energy calibration, one needs a spectrum with peaks at known energies. Second, one can calibrate the parameters used in the quantitative analysis. This requires one or more samples of reference materials, with known composition.

Standard less analysis is fairly quick and easy to set up but only gives approximate results. Using standards to calibrate the analysis requires more time to get started but gives much better results, as long as the sample is similar to the standards. If one uses several steel samples to calibrate, then matlab will provide excellent results on most steel alloys. However, the results will be less accurate for a brass alloy.

#### CONCLUSION

Hence the technique XRF(X-Ray Fluorescence) is used to detect the purity of the actual elements present in the metal.

#### ACKNOWLEDGMENT

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#### REFERENCES

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