# XRF ANALYSIS: A QUICK GUIDE

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Atoms of different elements absorb any incident X-rays and then emit X-rays of characteristic frequencies dependent on their atomic number. The aim of this experiment is to use this property of elements to determine unknown concentrations of elements in samples. This property of elements can also be used in thin-film analysis and give us the thickness of thin-films on a substrate.

Apparatus setup:
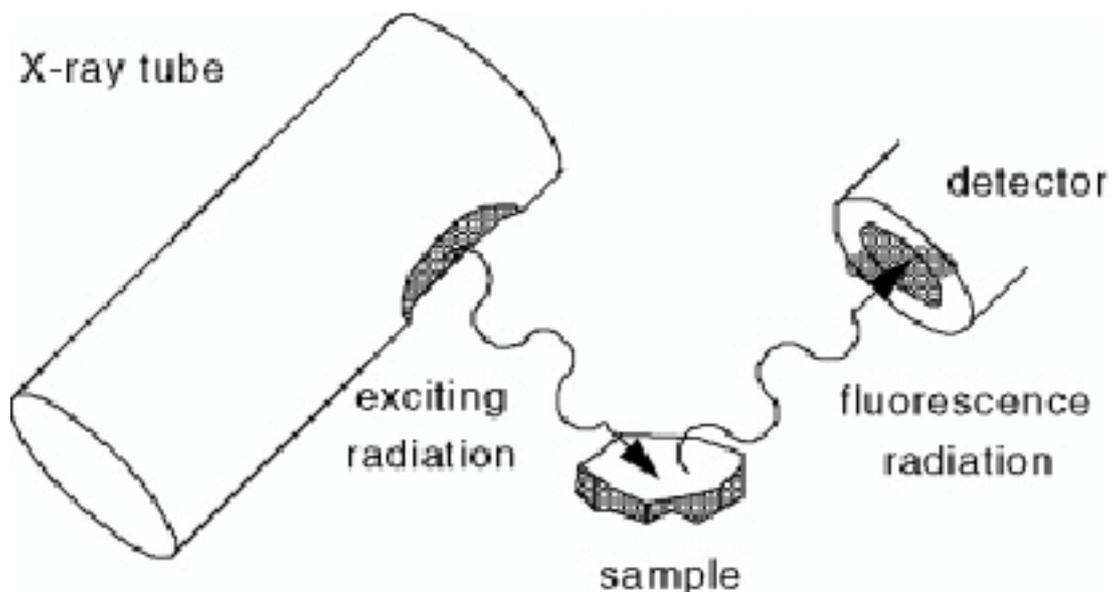
1. List of apparatus:
   a. PX4 Digital Analyzer
   b. MiniX X-ray tube

c. XR-100 X-ray detector
d. Radiation sealed box to place sample in with opening for detector/emitter

2. List of software:
a. ADMCA (Acquisition software)
b. XRS-FP (Quantitative analysis software)
c. MiniX tube controller

Procedure:
1. Setup and Installation:
a. Insert the Amptek 4.00 Winusb disk into your desktop PC running Windows XP.
b. Install the XRF Analysis software (XRS-FP) from the disk.
c. Copy the entire 'ADMCA 2.0' folder from the disk to the hard disk.
d. Connect MiniX and install the driver from the disk
e. Install the HASP key software from the disk and connect the HASP USB key
f. Connect the PX4 and allow it to install its driver
g. Open ADMCA and choose connect to PX4. You will see a green USB icon in the bottom right
h. Make sure you have complete administrator access on the computer you are using
i. The setup is now complete and ready for calibration

2. Calibration of acquisition software:
a. Open the MiniX software on your desktop
b. Open ADMCA
c. Load the SS-316 stainless steel sample into the sample holder
d. Make sure the sample is properly sealed in the box
e. Turn on the X-ray tube from MiniX controller
f. Press 'Start Acquisition' in the toolbar in ADMCA
g. Wait until the 'Total Count' goes above 50,000 and then stop acquisition
h. In the toolbar select 'Define ROI'

      i. Highlight the highest peak (Fe) and add it. Do the same with the rightmost peak (Mo)

     j. Choose 'calibrate' from the toolbar and assign the values 6.4 and 17.4 to Fe and Mo respectively

     k. Write keV in the units box and click ok

     l. You will see the horizontal scale change from channel to keV. Your ADMCA calibration is complete

    m. Save the spectrum as SS-316

3. Calibration of analysis software:
   a. Open ADMCA and open the SS-316 spectrum file
   b. Click on analyze in the menu bar and click load library. Navigate to ADMCA 2.0>library>Xray-Ka1 and open the file
   c. Use the define ROI feature to highlight all prominent peaks
   d. Click on Analyze>ROI detail to obtain names of all elements which have peaks present in the spectrum
   e. Open XRS-FP and click on expert mode
   f. Open a new file in XRS and enter the known concentrations of the elements (the names of which were obtained in step d.) in SS-316
   g. Import the spectrum from MCA into XRS
   h. Click on Process>All (the functions of this command will be described later)
   i. When the processing in complete, press calibrate>FP to obtain the calibration coefficients for the elements present
   j. Save this file as SS316calib.tfr
   k. Repeat for all samples, which contain elements you want to test for in the future. XRS will need all the calibration coefficients of elements A, B and C in one file to analyze a sample containing said elements

4. Functions of the Process>All command
   a. Smoothing-Each peak is fit to a gaussian. This fitting is done as many times as specified in XRS

b. Escape peak removal-The detector material contains Si. As a result, fluorescence also takes place inside the detector material. This results in an energy loss of 1.75 keV per escape photon. The peak corresponding to this is removed.

c. Sum peak removal-The peaks that result from two photons arriving together are filtered out

d. Background removal-This involved the removal of Bremsstrahlung radiation

e. Blank Subtraction-This involves the removal of peaks that are present in every reading e.g. argon

f. Attenuation correction-Lower energy-rays may interact with detector window or the intervening air. Higher energy x-rays may not interact at all. The software has to correct for these two cases

5. Analysis
   a. Load the sample into the tray and obtain its spectrum
   b. Identify the elements present using the ROI detail feature
   c. Open a calibration file containing calibration coefficients for those elements in XRS-FP
   d. Import the spectrum into XRS and click Process>All
   e. Click Process>Analyze to obtain the elemental concentrations

6. Thin-film calibration:
   a. Apply a uniform coating of an element (Pb/Au) on a uniform substrate (preferably Silicon)
   b. Use an ellipsometer to measure the thickness of the coating. The method for this is:
      i. Place the sample on the loading try on an ellipsometer
      ii. Open the CompleteEase software on your PC
      iii. Make sure the ellipsometer emitter and detector are bent at the same angle as specified in the 'measure' options on CompleteEase
      iv. Take a measurement. This will give you a Psi Delta curve

         v. Click on the analysis tab

       vi. Click on 'Open' in the model option and open the 'Si with absorbing film' as your model. This will give the details of the model in the model window

     vii. Click on 'Fit' in the analysis tab. This will vary the parameters of the model curve to fit the obtained psi delta curves

    viii. The model window will now show you the thickness of the layer

  c. Place it into the sample holder and obtain its spectrum

  d. Import the spectrum into XRS and process it

  e. Enter the known element and its concentration as 100% in the specimen component table.

  f. In the thickness table set the type to 'Calc'

  g. Select sensible units (mg/µg per cm^2) depending on the value of the thickness

  h. Calculate the thickness in terms of the above mentioned units and enter it into XRS

  i. Press Calibrate>FP

  j. Save the file as ElementNamecalib.tfr

7. Thin-film Analysis:

  a. Open the calibration file in XRS

  b. Obtain the spectrum of the sample, load into XRS, and process it

  c. Press Process>Analyze