

Thin Film Analysis

XRF is well suited to the analysis of coatings and thin films, particularly if the coating is predominantly one element or one is only interested in the total quantity (mg/cm^2) of one element. Amptek's XRF systems (XR100 and PX4, Mini-X, and the XRS-FP software) can easily be used for such analyses, but there are a couple of subtleties in the setup. Most of the XRS-FP documentation is focused on measuring composition, i.e. the percent of each element, so this note discusses the differences when measuring areal density.

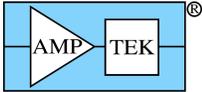
A typical application is measurement of lead in paint. There are usually multiple layers of paint on a substrate. One typically does not know how many layers of paint or the thickness of each layer, but this does not matter. The standards for lead paint state that remedial action must be taken whenever the total amount of lead on a painted surface exceeds some threshold, typically $1.0 \text{ mg}/\text{cm}^2$. An XRF measurement yields the total intensity in the lead peaks, which is proportional to the total amount of lead in the region being analyzed, i.e. the mg/cm^2 . So XRF is ideally suited to this measurement, but the FP software must be calibrated in these units and setup to yield results in mg/cm^2 rather than percent. There are many other applications which are very similar. For example, users often measure the total lead or cadmium in the coatings on toys. Users often want to verify coatings on metals, for example zinc chromate and strontium chromate are primers in the marine and aerospace industry. XRF can be used to verify that an adequate coating thickness has been applied.

There are two major differences when using XRS-FP to analyze a film in units of mg/cm^2 , versus bulk and film analyses yielding percent composition. First, one cannot use a standardless analysis. The relative intensities of the X-ray lines can be estimated from first principles, but the actual intensities of the lines, the cts/sec, depend strongly on the details of the set-up. One must calibrate the parameters. Second, the relative intensities are only weakly dependent on many instrument details. For example, if the flux from the X-ray tube varies by 5%, then the intensity of each line varies by 5%, so the relative intensities are unchanged. The percent composition is unchanged. However, the actual intensities vary by 5%, so the mg/cm^2 results scale directly.

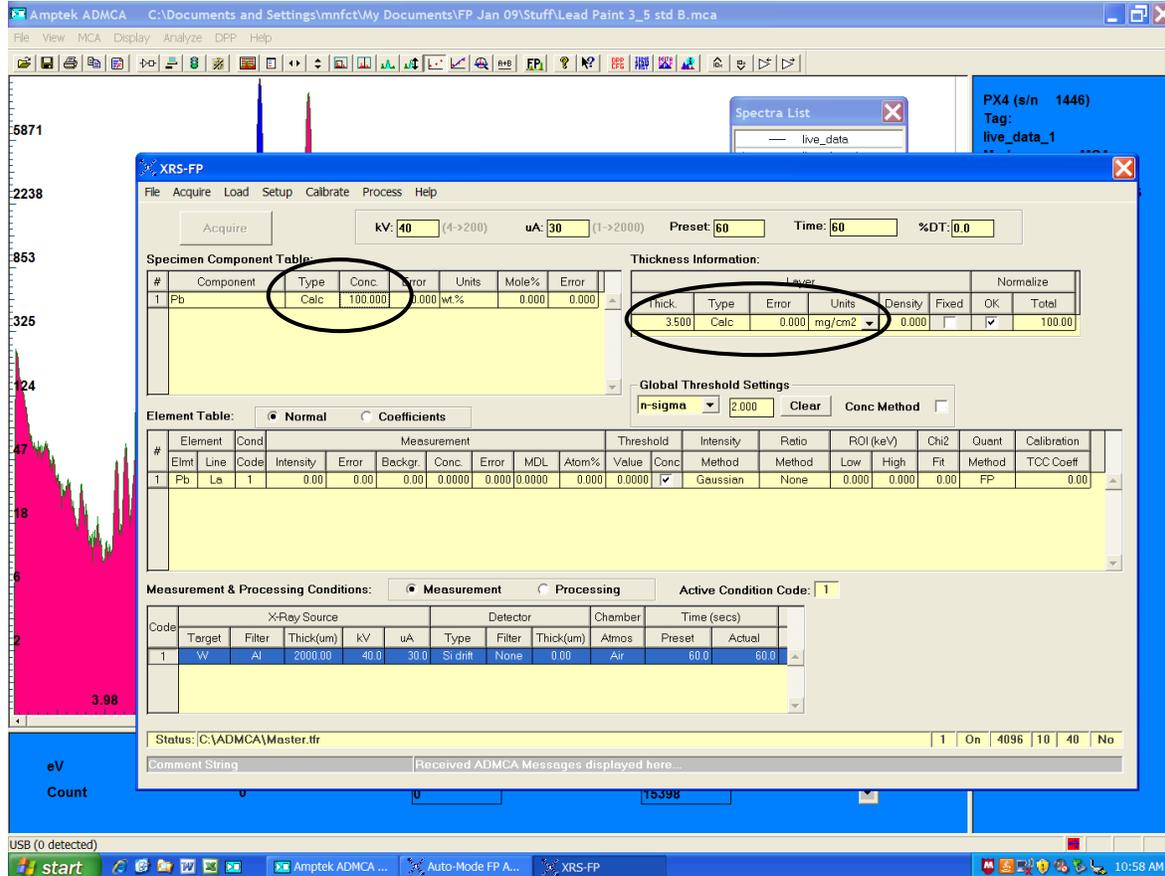
Lead Paint Example

As a specific example, we will show how to set up a system to measure lead in paint. This system used a silicon drift diode (SDD) in an XR100 with a PX4, and Amptek Mini-X tube with a tungsten anode set to 30 kVp, and Amptek's standard base plate to obtain a backscatter geometry. Calibrated lead paint standards were obtained with 1.0, 1.6, and $3.5 \text{ mg}/\text{cm}^2$ lead and were placed on a piece of $\frac{3}{4}$ pine board. Set-up and energy calibration are described in the "FP Quick Start Guide".

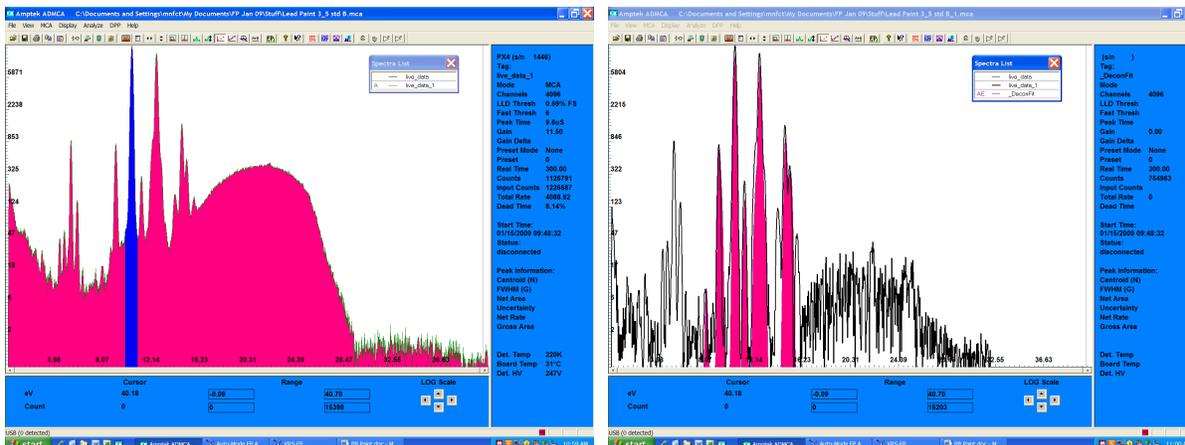
- 1) Obtain a calibration spectrum (we used the $3.5 \text{ mg}/\text{cm}^2$ sample). Open FP and set up parameters for detector, geometry, etc.
- 2) In "Setup", "Quant", set the analysis to "Fundamental Parameters", "One Standard".
- 3) In the "Component Table", enter the element (Pb) set "Type" to "Calc", and set "Conc" to "100%". In the "Layer Table", set the units to " mg/cm^2 ", "Thickness" to the known value (3.5 in this case), "Type" to "Calc", and check the "OK" for "Normalize". These are the key steps to calibration, and are illustrated below.



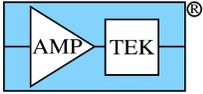
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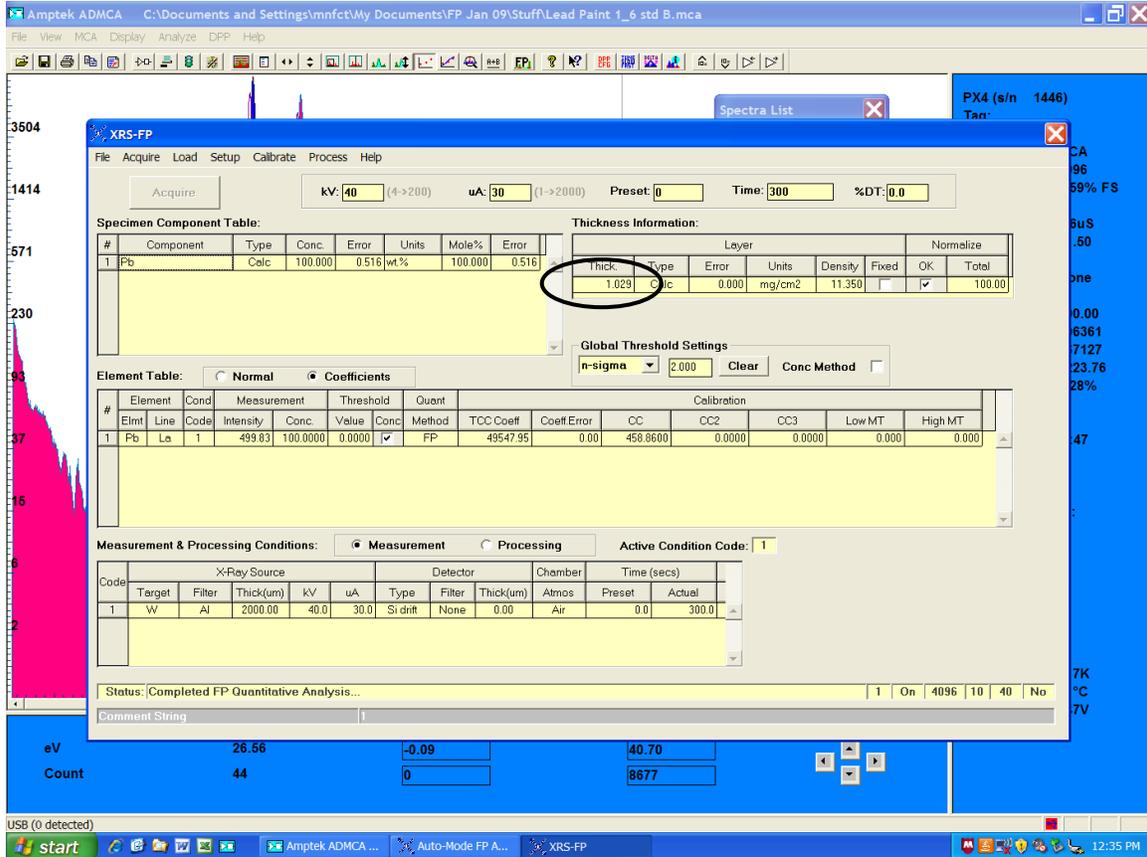
- 4) Load the calibration spectrum into FP, and process as usual. The plots below show the raw calibration spectrum (on the left) and the processed spectrum (on the right). The deconvolved peaks, in red on the right, are lead. There are other peaks visible, corresponding to Ti, Cr, and Sr, which are presumably other components of the paint. It is the net area of these deconvolved Pb peaks which is proportional to the total lead on the surface.



- 5) After the spectrum is processed, select "Calibrate", "FP". Verify that the calibration coefficients are changed (see in the coefficients portion of the element table).
- 6) Insert another sample and take a spectrum. In this case, a 1.0 mg/cm2 standard was used. Load the spectrum, process it, and then click "Analyze". The result, the thickness in units of mg/cm2, will be displayed in the "Thickness Information Table", in the "Thickness" cell. This is shown below.



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The plot below shows the result of measurements taken using several different standards. The dashed line represents the ideal measurement, the circles with error bars the results of the XRF analysis.

