Quantitative Analysis Software
for X-Ray Fluorescence

XRF-FP

XRF-FP is a full-featured quantitative analysis package for XRF

APPLICATIONS

- X-Ray Fluorescence
- Thin-film Analysis
- RoHS/WEEE Analysis
- Teaching and Research
- Art and Archaeology
- Process Control
- X-ray Tube Characterization

XR-100CR and Mini-X performing XRF.

FEATURES

- Fundamental parameters (FP)
- Analysis with or without standards
- General bulk and thin-film analysis
- Analyze up to 40 elements
- Supports all Amptek detectors, X-ray tubes, and electronics
- Auto-mode for continuous or repeated analysis
- CdTe escape peak correction
- Non-destructive analysis

XR-123 and Mini-X performing XRF.

XRF analysis with Fundamental Parameters (FP) converts elemental peak intensities to elemental concentrations and/or film thicknesses. The analysis can be performed standardless, where all variables are based upon theoretical equations, the fundamental parameter database, and precise modeling of the detector, X-ray tube, and geometry. Standardless mode is possible for simple bulk or single-layer thin-film samples when the film thickness is known. The FP analysis can also be performed with standards. This is achieved through a calibration step where the XRF response function for each element is measured using a known standard of some kind. A with-standards analysis provides the most accurate results.

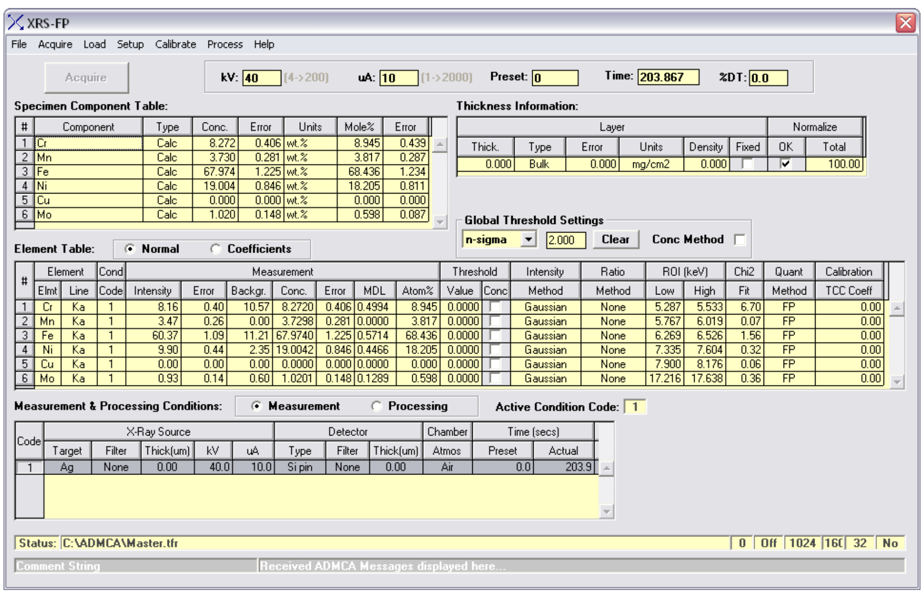

Figure 1: XRF-FP Main display window. Shows the element table with the various parameters and chemical concentrations.

AMPTEK INC. 14 DeAngelo Drive, Bedford, MA 01730-2204 U.S.A.
Tel: +1 (781) 275-2242 ***Fax:*** +1 (781) 275-3470 ***email:*** sales@amptek.com **www.amptek.com**

XRF-FP SPECIFICATIONS

Introduction

There are only two steps in XRF analysis whether or not the fundamental parameter (FP) method is used. The first step is to calibrate the response function for each element from one or many standards (called the "Calibration" step). The second step is to produce the sample analysis of a given material, using the previously stored calibration coefficients, and the FP algorithms given a definition of the sample (i.e., number of layers, and which elements are in which layers).

The software will support single layer or bulk composition and thickness analysis of up to 40 elements, calculated as either elements and/or compounds. Up to 4 or more excitation "conditions" are allowed per analysis. Each condition describes a separate analysis, and can be freely defined with any combination of experimental conditions, such as kV, tube anode, filter, detector filter, environment (air, vacuum, He) and acquisition time. This allows the analyst to measure some elements with one condition, and others completely differently, such that each analysis can be optimized for the specific element, or group of elements. Likewise, the spectrum processing steps can also be freely defined, and are all part of the condition code setup.

The FP analysis software will support a single or multiple standard calibration scheme, or completely standardless analysis if the tube, detector, environmental and geometry parameters are known. Calibration standards should be passed one at a time and the merging of the calibration standard information is handled internally. After each calibration step, a set of calibration coefficients and associated information for each of the defined elements is returned, which can immediately be used if only one standard is employed. When using multiple calibration standards, all the coefficients are merged into one set, and then this final set is available for subsequent quantitative analysis.

The layer thicknesses must be fixed for standardless analysis. Results can be normalized to any value, and MUST be normalized for standardless analysis or when the layer thicknesses are calculated. Elements (or compounds) can be calculated, fixed, or determined by difference. Elements can also be determined by stoichiometry from the compound formulae. Composition results can be calculated in units of Wt% or ppm, and for thin films, units such as $\mu g/cm^2$ and mg/cm^2 are used for mass thickness. The latter can be converted to thickness (microns, microinches, nm, etc.) if the density is known. The density may be input or optionally calculated theoretically.

All the appropriate FP calculations are made both during calibration and for Quantitation, using calculations based upon the Sherman equation. Tube spectra, required for the direct fluorescence calculations, can be supplied by the user or calculated from built-in models (Ebel, Pella et al.). These tube spectra can be convolved with experimental transfer functions to derive the expected tube spectrum passing through an optic such as a polycapillary bundle. The presence of air paths will also be calculated from the input geometry parameters for both the source and detector paths. Single-element filters can also be inserted between the tube and the sample or between the sample and the detector, and the software can accommodate both.

The detector parameters (window, thickness, area, etc.) will also be used to calculate the various absorption and efficiency effects when X-rays pass through the window and get deposited in the detector material. This is only strictly necessary when doing standardless analysis, but the calculations are always done this way for consistency, and to make it easier to compare calibration coefficients between elements. If the theory were perfect all the calibration coefficients would have the same value. In practice, differences should be relatively small, especially in comparison with coefficients that did not fully compensate for the detector efficiencies. Usually when calibrating elements that all use the same line series (e.g., K), the coefficient variation is small (<30%), but is often larger when calibrating from mixed lines (e.g., K and L) because it is difficult to make absolute calculations that include the line series information (e.g., fluorescence yields).

It is not necessary to collect pure-element spectra for FP analysis as no direct ratioing is necessary for the elemental intensities. The calculations are done this way to make it easier to do standardless analysis. Of course, it is possible to use pure-element standards if desired, and the complete FP calibration may be done this way without any "type" standards being used at all. This is useful if the analyst does not have type standards readily available.

Both direct and secondary fluorescence effects are considered in the FP calculations. Included in the FP database are all the required parameters to calculate or recall absorption coefficients, fluorescence yields, jump factors, Coster-Kronig transitions, line energies, line ratios, transition probabilities, etc.

The software consists of a main program window that provides the user interface. It runs on standard PCs (Windows XP and later) with at least 256MB RAM of memory. The XRF-FP software is completely compatible and integrated with the Amptek ADMCA display and acquisition software. It can also directly control all Amptek electronics to provide an auto/repeat/continuous mode of operation.

Fundamental Parameters Analysis

Elements/Components

Can analyze up to 40 elements as individual elements and/or compounds. Unanalyzed elements can be specified stoichiometrically bound with an analyzed element (e.g., oxides or carbonates). Elements can be analyzed in one or more compounds within the same analysis. One compound (or element) can be analyzed by difference. Any number of compounds (or elements) can be "fixed." For example, solutions, binders and/or hydrated crystals can be analyzed this way.

General Bulk and Thin-Film Analysis

Any bulk or single-layer (unsupported) thin-film sample can be analyzed by either standardless or a calibration-with-standards FP approach. Each analysis may use up to 6 excitation conditions per analysis. Each excitation condition can vary almost any analysis setup, including the kV, acquire time, tube (or secondary) target, detector type, detector or tube filter, source focusing optic, atmosphere (air, vacuum, helium), and spectrum processing (e.g., deconvolution type, background removal, sum & escape peak removal).

Optional software is available to handle multilayer samples up to 6 layers for simultaneous film thickness and composition analysis using FP with standards (please contact Amptek).

XRF-FP SPECIFICATIONS (con't)

Analysis With or Without Standards

Many detectors and windows can be fully modeled. This allows analysis without any standards with normalization to 100% (or any specified factor). This is only possible when a single excitation condition is used.

When more than one excitation is used, at least one of the elements for each condition must have been calibrated. Calibration factors may be generated using any type of standard (e.g., pure element or analytical "type" standard). A single "type" standard may be used, or the calibration may be done with a different standard for each element, or any combination of standards may be used. If some elements are calibrated and some are not, the latter can use calibration coefficients derived from the former group.

The mass thickness of the sample can either be specified or calculated. If the latter, then the analysis cannot be standardless. Several units are possible for thickness measurement, and the density can be calculated theoretically or specified in the case of linear thickness calculations. Composition units may be ppm or wt%, with the additional output of atomic and mole percent.

Excitation Sources

Can model different x-ray tube types ("reflection" or transmission) using two analytical models (Pella or Ebel), or by the use of a supplied source spectrum for complete polychromatic source modeling. Different tube windows and filters may also be included. The tube window can be of any composition (e.g., BeO or glass). Any tube anode element may be specified, as well as the tube (electron) incidence and take-off angles. The kV may range from 3 to 60 kV. Provision is provided for including a transmission efficiency file for use with, for example, polycapillary optics placed between the source and the sample.

Radioisotopes can be used using a source file describing the relative line ratios. For secondary target excitation, monochromatic excitation is assumed.

Detectors

Various detectors (Si-PIN, SDD, CdTe, Si(Li), and Ge) and windows can be fully modeled. The software has provision for the user to input all the required parameters (e.g., thickness, area, dead layer, etc.) associated with these detectors and their windows. For more information on CdTe detector analysis please see http://www.amptek.com/epeaks.html.

Geometry

The complete system geometry can be specified including the sample incidence and take-off angles, the source-to-optic and/or source-to-sample distances, the sample-to-detector distance, as well as the environmental factors

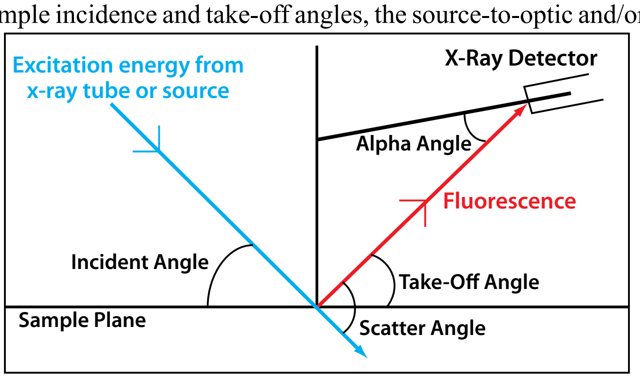

Figure 2: XRF-FP geometry parameters. A well defined geometry is critical for accurate results.

Elements, Lines, and Interelement Corrections

The software includes full corrections for absorption and both thick and thin-film secondary fluorescence. All possible lines are considered for both excitation and fluorescence. The analysis can be performed for all elements from H through Fm, using K, L or M lines in the energy range from 0.1 keV up to 60 keV.

Spectrum Processing

Spectrum Calibration

Using two known peaks in the spectrum, the software can calculate the effective gain (eV/channel) and offset (zero shift) for the spectrometer. These factors can then be applied to subsequent spectra prior to other spectrum processing. It is vital that peaks are located at their expected energies, otherwise the spectrum processing cannot function correctly. The calibration can be specified in the XRF-FP software or in the ADMCA software. XRF-FP can automatically import the calibration from ADMCA.

Background Removal

This module uses iterative filtering to remove all peaks, leaving behind the spectral background. The background spectrum may be displayed or removed from the original spectrum.

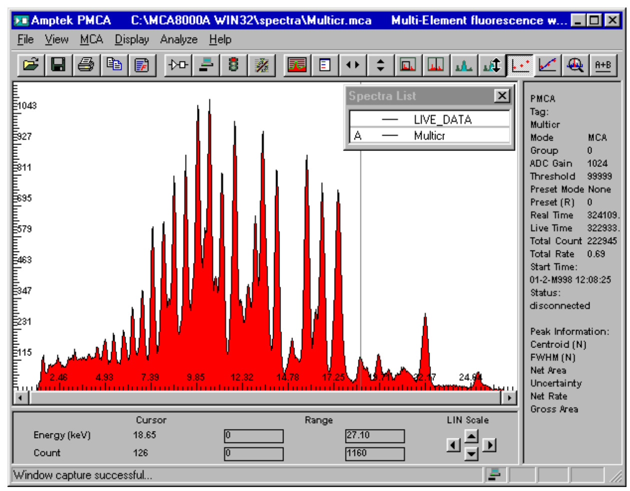

Figure 3: Original spectrum taken with the MCA8000A.

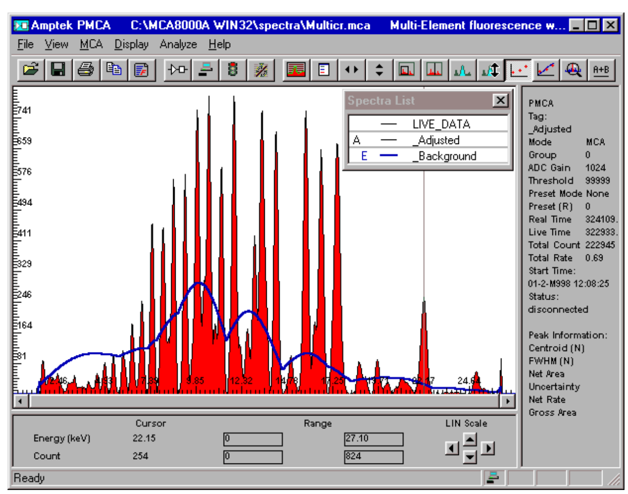

Figure 4: Processed spectrum with the XRF-FP. The blue curve represents the removed background.

XRF-FP SPECIFICATIONS (con't)

Escape Peak and Sum Peak Removal

Removes, at the user's to option, both detector escape and sum (pile-up) peaks.

For information on Escape Peak Corrections for CdTe detectors see http://www.amptek.com/epeaks.html. This is important when characterizing the output X-ray spectrum of an X-ray tube or performing XRF with a CdTe detector.

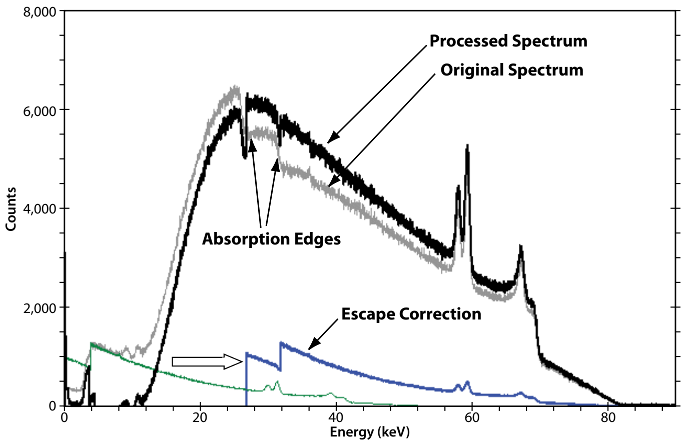

Figure 5. Plot showing a tungsten (W) X-ray tube output spectrum taken with a CdTe detector after processing to remove escape events. The gray trace shows the original spectrum. The green trace illustrates the escape events in the original spectrum. These are subtracted from this original spectrum, then the correct energies are computed (by adding in the energy which escaped). The blue trace shows the corrected escape events, which are then summed with the gray trace. The dark black trace shows the final result of the processing with the events in their correct channels.

Smoothing

A specified number of 1:2:1 Gaussian smooths can be applied to a spectrum.

Intensity Extraction

Specified element peaks may be integrated over a fixed Region Of Interest (ROI), or the complete spectrum can be fit using synthetic Gaussians for every possible line in the regions of interest. One of six major lines (Ka, Kb, La, Lb, Lg, Ma) is selectable as the main analysis peak for intensity extraction. All relevant lines required for deconvolution are then automatically included by the software for full overlap correction using a least-squares fitting procedure.

All required line energies and resolutions are calculated automatically from the specified analyte line. The Gaussian peak fitting can be done with a linear or non-linear least-squares approach. The latter allows constrained changes in the peak positions, intra-series line ratios, and peak widths, from their nominal starting points.

In addition to calculating elemental intensities, the software automatically calculates the estimated uncertainty and background values, which allows uncertainty and Minimum Detection Limit (MDL) calculations to be performed during the FP analysis.

Software Options

Spectrum Acquisition

There are two methods of spectrum acquisition. The first is to acquire with the Amptek ADMCA acquisition application, which controls the MCA8000A, DP4 Digital Pulse Processor, X-123, or PX4 Digital Pulse Processor and Power Supply. Once acquired, the XRF-FP can import the file and use the ADMCA display for spectrum processing. The second uses the MCA8000A control DLL or the DP4/X-123/PX4 control DLL to acquire a spectrum directly into the XRF-FP software, which can then be automatically processed. A repeat measurement capability is provided.

Automatic Peak/Element ID

Using the Amptek ADMCA application, the user can automatically mark peaks (ROIs) for analysis. If the appropriate element library is loaded into ADMCA the software will associate the marked peaks with elements. The corresponding elements can then be automatically imported into XRF-FP element table.

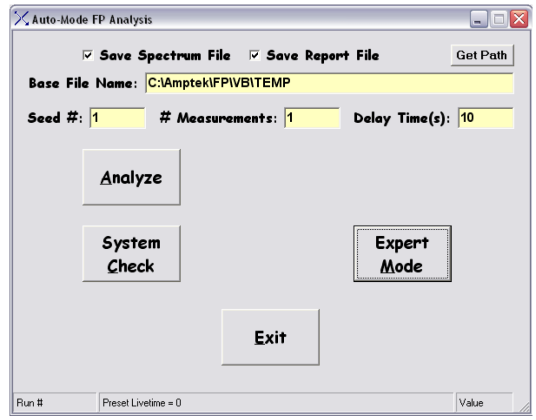

Figure 6. Auto and Repeat Mode.

Complete XRF Systems

Complete XRF System 1 Includes:
- **XR-100CR** X-Ray Detector
- **PX4** Digital Pulse Processor, MCA, & Power Supply
- **Mini-X** USB Controlled X-Ray Tube
- **XRF-FP** Quantitative Analysis Software

Complete XRF System 2 Includes:
- **X-123** Complete X-Ray Spectrometer
- **Mini-X** USB Controlled X-Ray Tube
- **XRF-FP** Quantitative Analysis Software