Quantitative Analysis Software for X-Ray Fluorescence

Use with the MCA8000A Multichannel Analyzer

Figure 1: MCA8000A shown clockwise with Disk containing XRF-FP Quantitative Analysis Software and free MCA Software, Amptek XR-100CR X-Ray Detector, Amptek LASER-X X-Ray Generator, LASER-X Voltage and Current Controller and PX2CR Power Supply & Amplifier for the XR-100CR.

The XRF-FP software consists of a main program that provides the user interface and various machine input/output functions, together with two libraries (DLL's) that provide the spectrum processing and Fundamental Parameter (FP) calculations. The software runs on standard PCs and operating systems (Windows 95, 98, ME, etc.), with at least 32MB RAM of memory. Complete XRF analysis is possible, with or without standards, using an internal database of fundamental parameters such as absorption coefficients, fluorescence yields, transition probabilities, etc.
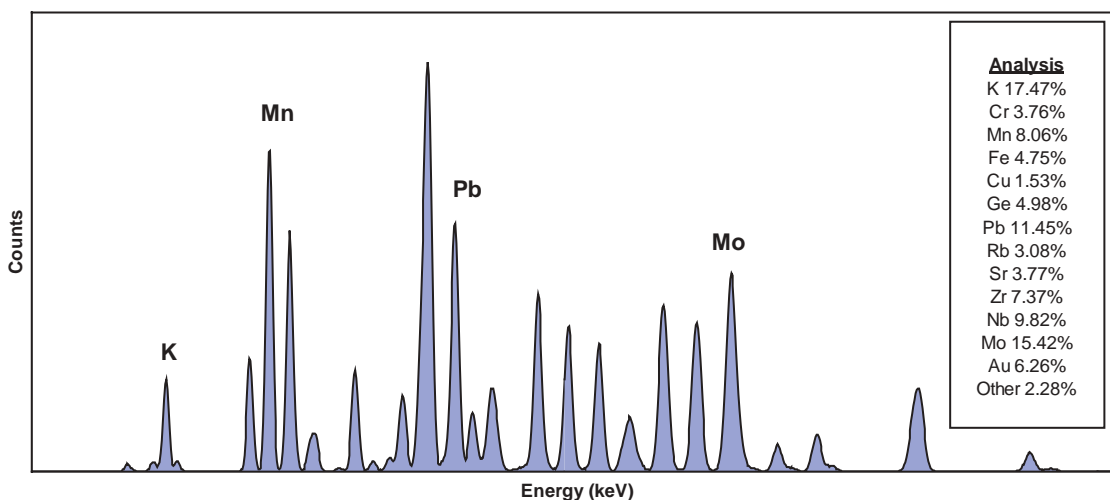

Analysis	
K	17.47%
Cr	3.76%
Mn	8.06%
Fe	4.75%
Cu	1.53%
Ge	4.98%
Pb	11.45%
Rb	3.08%
Sr	3.77%
Zr	7.37%
Nb	9.82%
Mo	15.42%
Au	6.26%
Other	2.28%

Figure 2: Spectrum using the MCA8000A, XRF-FP Quantitative Analysis Software and XR-100CR system imported into an Excel worksheet.

XRF-FP SPECIFICATIONS

FUNDAMENTAL PARAMETERS ANALYSIS

Elements/Components

The XRF-FP software analyzes up to 40 elements (can be configured for up to 100 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.
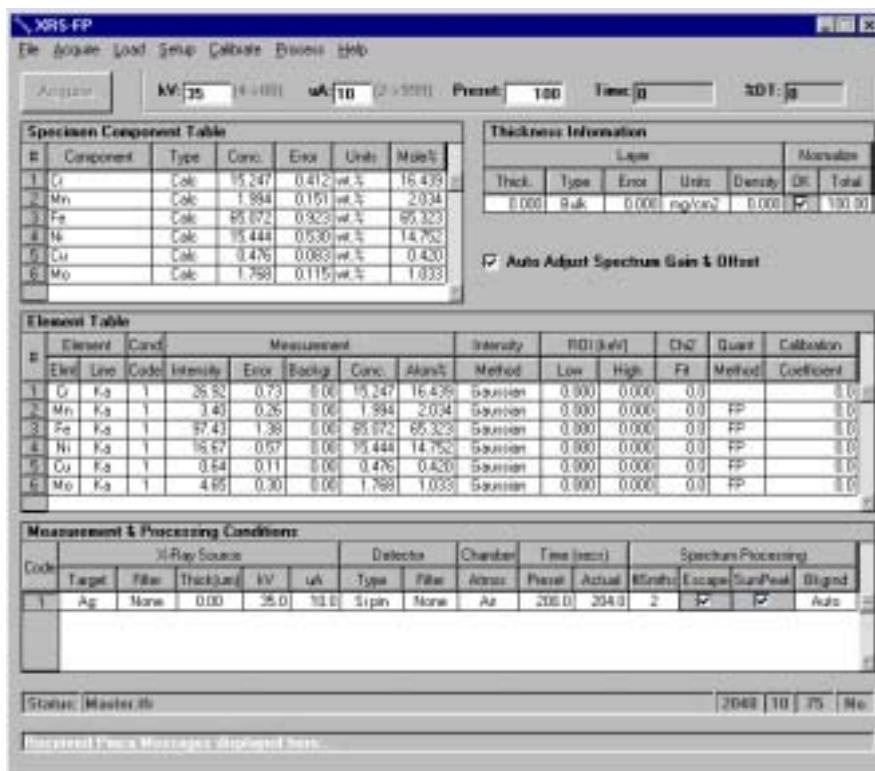

Figure 3: XRF-FP Main display window. The element table with the various parameters and chemical concentrations is shown.

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).

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.

XRF-FP SPECIFICATIONS (con't)

Excitation Sources

The XRF-FP software 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. 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., contact material and thickness, dead layer, etc.) associated with these detectors (and their windows).
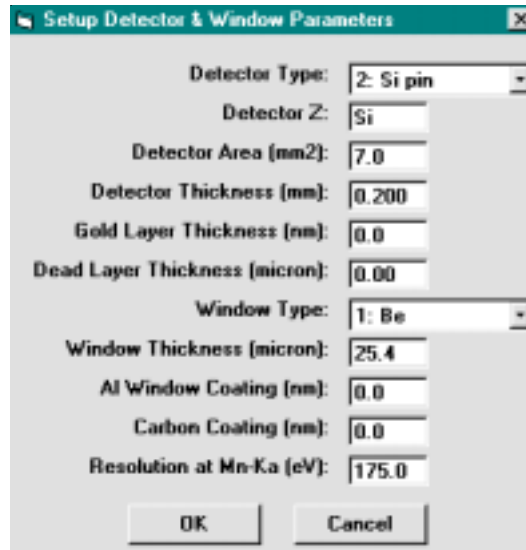

Figure 4: The Detector Setup dialog includes all the necessary parameters to model the detector.

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.
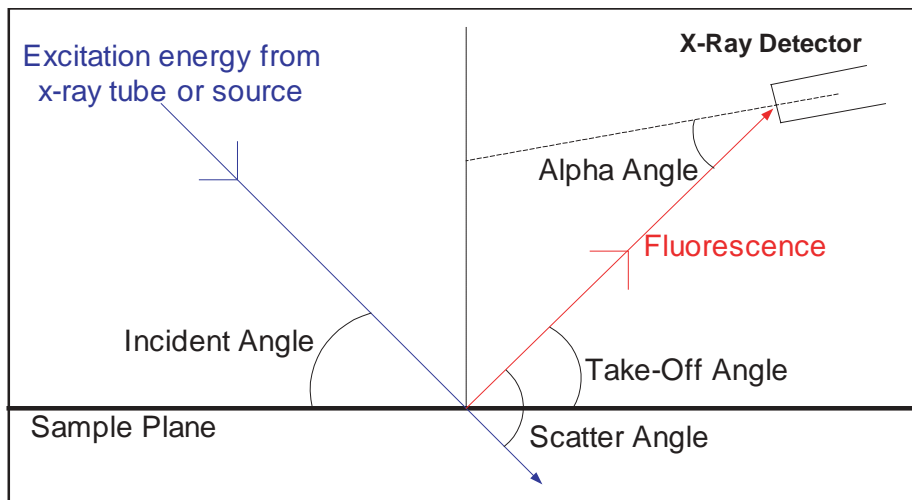

Figure 5: XRF-FP geometry parameters. A well defined geometry is critical for accurate results.

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.

XRF-FP SPECIFICATIONS (con't)

SPECTRUM PROCESSING SPECIFICATIONS

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 routinely 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.

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.
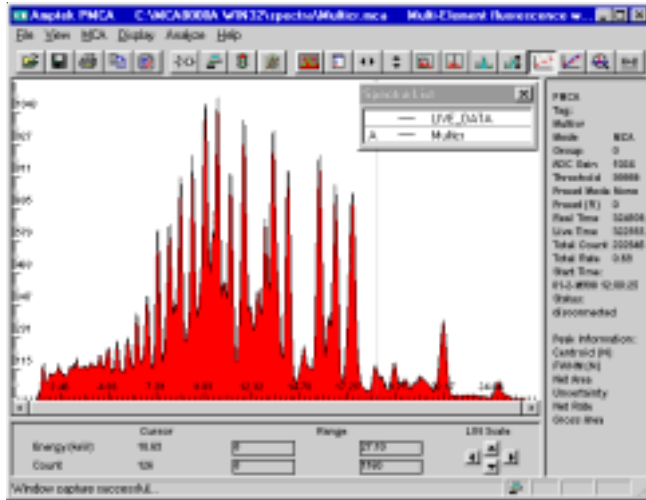

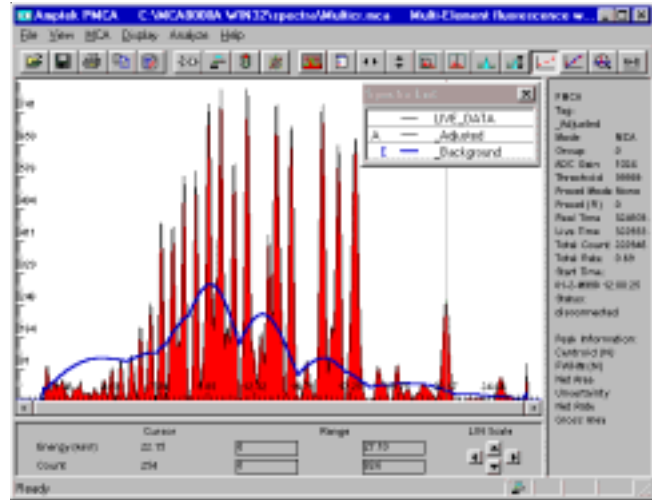*Figure 6: Original spectrum taken with the MCA8000A.*

Figure 7: Processed spectrum with the XRF-FP. The blue curve represents the removed background.

Escape Peak and Sum Peak Removal

Routines are available to optionally remove both detector escape and sum (pile-up) peaks.

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.
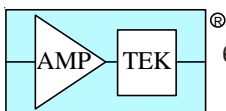
SOFTWARE OPTIONS

Spectrum Acquisition

There are two methods of spectrum acquisition. The first is to acquire with the Amptek Pmca application, which controls the MCA8000A. Once acquired, the XRF-FP can import the file and use the Pmca display for spectrum processing. The second uses the MCA8000A control DLL to acquire a spectrum directly into the XRF-FP software.

Automatic Peak/Element ID

Using the Amptek MCA8000A Pmca application, peaks can be identified and element tables imported into XRF-FP.