# Quantification of Thermo Scientific K-Alpha Data

The last ten years have seen the XPS technique develop from a technique used predominantly by experts of XPS to a tool used by application oriented scientists. The emergence of instruments such as the Thermo Scientific K-Alpha shows that there is a place for an instrument with a high performance to cost ratio and lower cost of ownership. Instruments with these characteristics offer a route for the XPS technique into application areas previous limited by the constraints of expensive high performance technology. There will always be a place for high performance systems run by experts, but these new low cost XPS systems can only be good for surface science.

The data from the Thermo Scientific K-Alpha is supported in version 2.3.15 of CasaXPS, where a number of enhancements are included to streamline the conversion and processing of the new ASCII files. The ASCII files exported from the K-Alpha include a new flag to indicate whether or not the intensities are corrected for transmission. Version 2.3.15 of CasaXPS detects the new flag and converts the ASCII file to the ISO 14796 format using the counts per bin computed from the transmission corrected data. The necessity of returning the data to counts per bin lies in the assumption that the noise in the data is governed by Poisson statistics, which is required when measuring the goodness-of-fit for and when performing Monte Carlo error analysis on peak models. The ISO file written by CasaXPS includes both the counts-per-bin and also the transmission functions from the ASCII file, therefore quantification of the spectra in CasaXPS proceeds exactly the same way as for other data acquired using the Thermo Avantage data system.

An attractive feature of the K-Alpha is the possibility of combining a new resource with existing high performance instruments, either in-house or available from external laboratories. To accommodate the use of more than one instrument, data converted from a K-Alpha have a unique source label assigned to each VAMAS block and hence entries specific to the K-Alpha can be added to the CasaXPS element library. This feature permits the default CasaXPS library to offer the appropriate RSFs for data from different instruments.
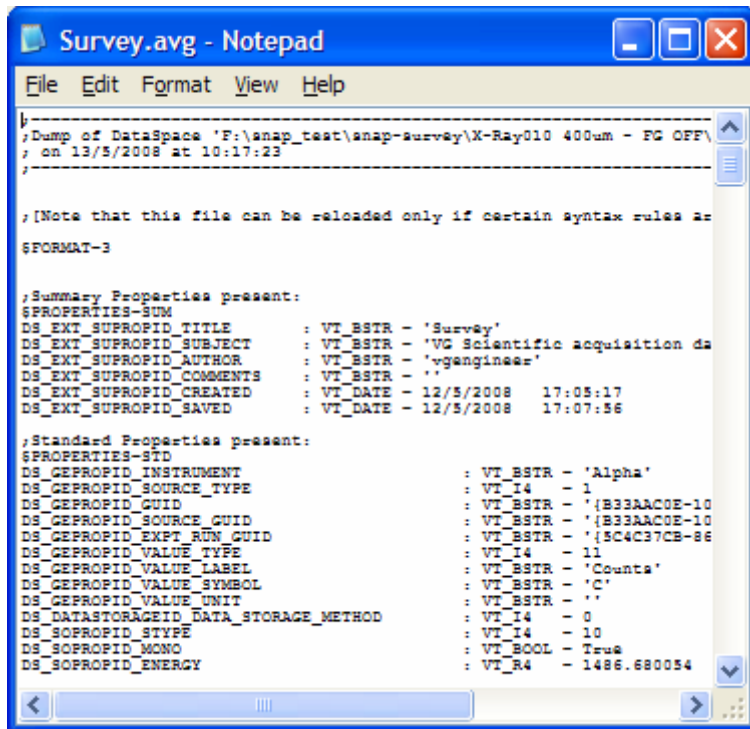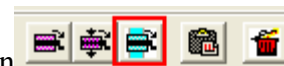
A new toolbar button provides a further means of creating quantification regions for a set of VAMAS blocks based on the element/transition fields in the VAMAS blocks. When converted from the Avantage ASCII files, the spectra are assigned the element/transition fields based on the name assigned to the data. For example, a carbon 1s transition might appear in the Avantage data file with the name "C 1s Scan" or "C 1s Snap", depending on the acquisition mode. On conversion to ISO format through CasaXPS, these strings are used to extract the element "C" and transition "1s". Armed with these strings, the new toolbar button identifies the entry corresponding to the photoelectric line in the element library and uses the element library information to create regions for each selected VAMAS block correctly assigned in terms of element/transition.

Thermo Scientific K-Alpha XPS Instrument

Converting K-Alpha Data

Data from the K-Alpha are exported from the Avantage system as ASCII file. The exported data files appear as shown in Figure 1. These ASCII files can be converted as follows.


Figure 1: Data file exported from Avantage.

Thermo Scientific Avantage Files

The Avantage binary files must be converted via an Avantage utility program (DataSpace_BatchDump.exe) to ASCII equivalent files. These ASCII files are created with the file extension .avg and can be converted to ISO 14976 (VAMAS) format using the Convert to VAMAS dialog window available from the File menu or the top toolbar of CasaXPS:

1. Using the Avantage utility program DataSpace_BatchDump.exe, convert all the .vgd binary files for an experiment to .avg ASCII files.
2. Select the Convert to VAMAS dialog window in CasaXPS either using the toolbar button or via the Convert menu item on the File menu.
3. Move to the directory containing the .avg ASCII files; the DataSpace_BatchDump.exe program offers the user the choice of where the ASCII files should be created.
4. Enter a name for the new VAMAS file using the file extension .avg and press the Open button. For example, if you wish to call your new file sample001.vms, then **you should enter sample001.avg** into the file name field on the Convert to VAMAS file dialog window. **Each .avg file found in the current directory will be read and entered into the new VAMAS file** named sample001.vms.

An example showing how to convert a directory of ASCII files can be seen in Figure 2, where a new ISO file named

steel_highres.vms will be created from the set of avg files in the current directory Scan_0. Note how the base-name for the ISO file is used together with the .avg conversion switch to instruct the set of .avg files in the directory to be read and then converted to the ISO format.
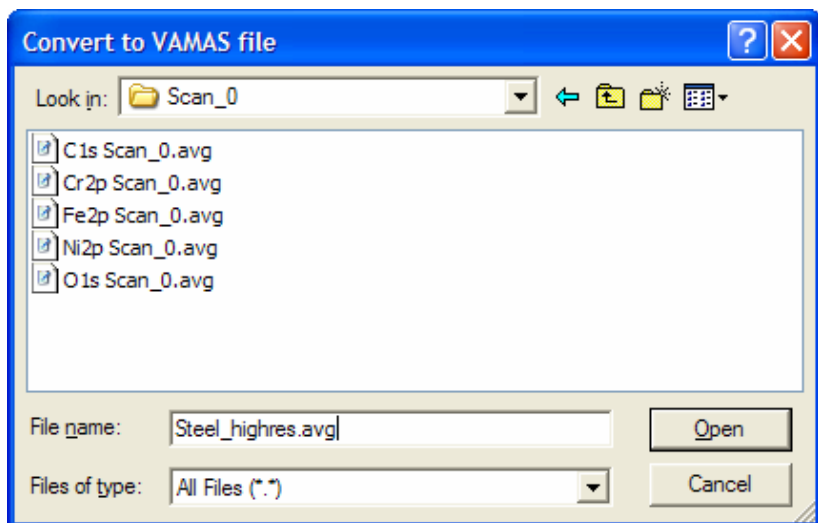
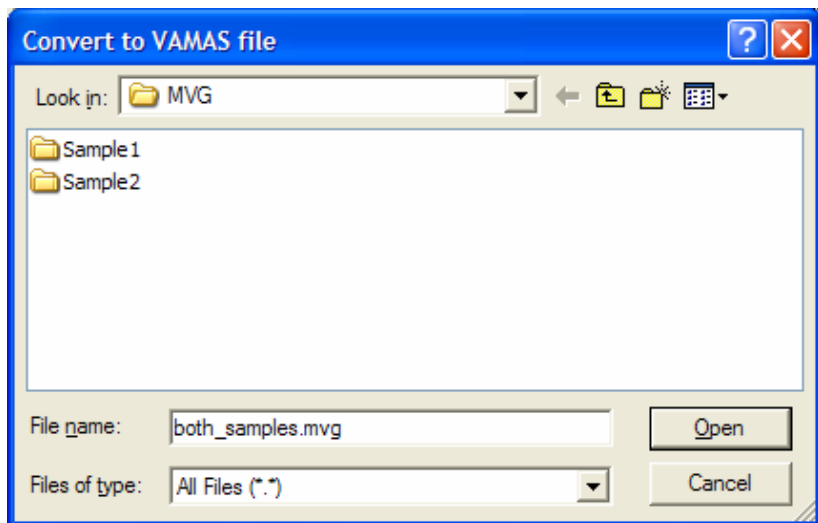**Figure 2: Convert to VAMAS Dialog illustrating a directory of ASCII avg files.**

The ISO file steel_highres.vms converted from the data files in Figure 2 is shown in Figure 3. Each avg file in Figure 2 contained two spectra per file, before and after etching the sample surface, hence the two rows of VAMAS blocks in the file in Figure 3.
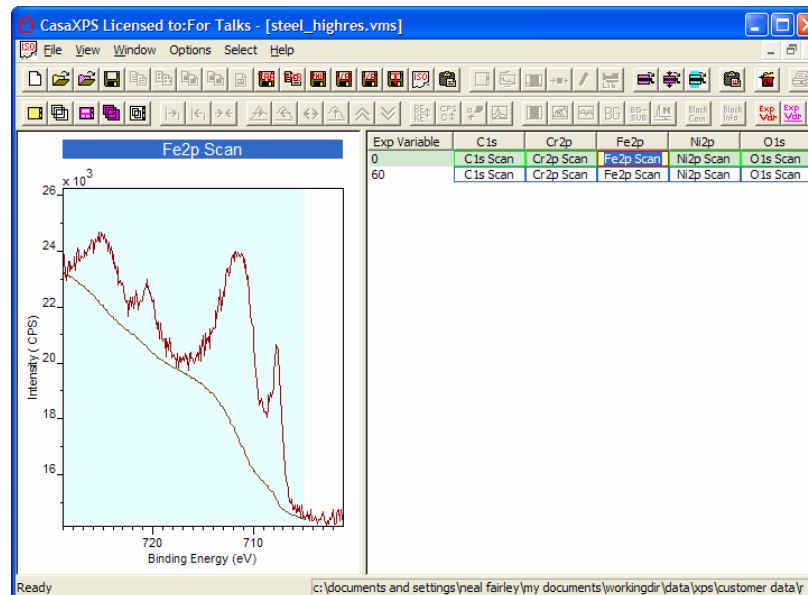
Figure 3: The ISO file converted from the data files in Figure 2.

Merging Sets of Experiments

The acquisition regions for each experiment are exported into separate ASCII files with the .avg extension, thus an experiment is typically recorded, once exported, as a set of ASCII files in a directory. If an experiment is one in a sequence of experiments, all of which might reasonably be processed as a unit, then a further import option allows a set of such data directories to be converted to a single ISO 14976 VAMAS file.

Figure 4: Merging data from two directories using the .mvg conversion switch.

assigned labels based on the names given to the sub-directories, which can be view using the Edit Mode toolbar button.
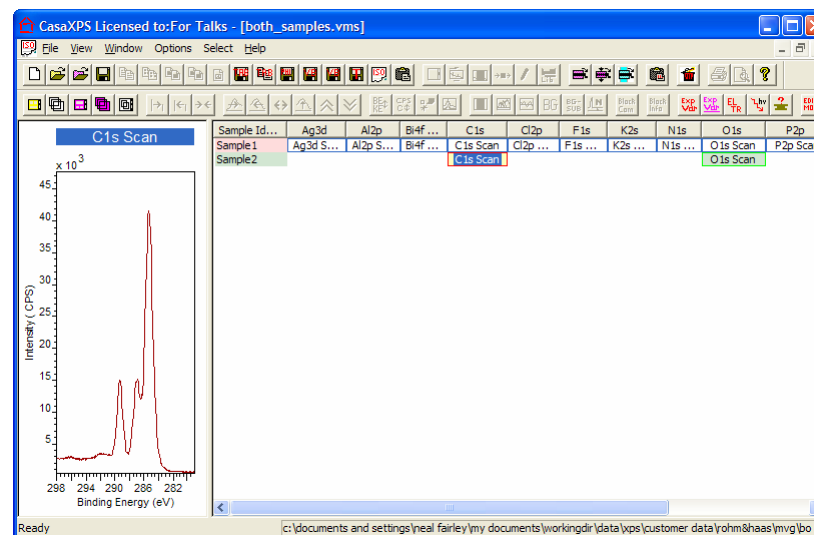
Figure 5: Single ISO file created using the .mvg conversion switch acting on the two directories in Figure 4.

Given a set of directories each containing exported Avantage data, a new VAMAS file is created by moving to the directory (using the Convert to VAMAS File Dialog, Figure 4) containing these data sub-directories and entering the new filename with an extension of .mvg. A VAMAS file with the extension .vms, shown in Figure 5, is created at the level of the directory containing the data sub-directories. The .avg files found in the sub-directories are converted and entered into the new VAMAS file with the experimental variable set to a Data Set index number; the index assigned to the experimental variable is dependent on the order of the sub-directories when read. In addition to the Data Set index, the rows are also

Quantification

The principal features used to quantify data from the K-Alpha can be discussed using survey spectra.
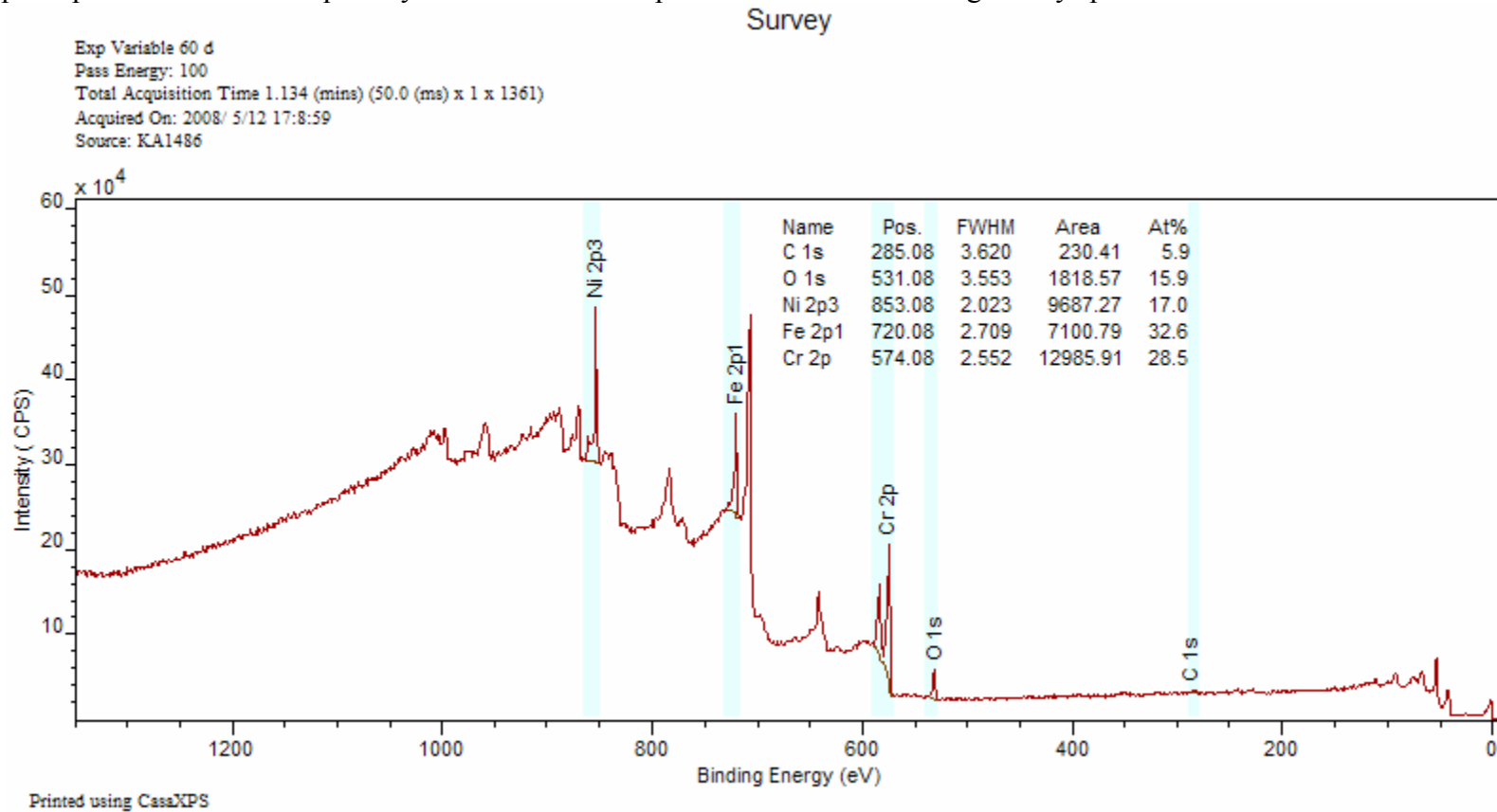

Figure 6: Survey spectrum from a steel standard.

Element Library Excitation Source String

Figure 6 is a survey spectrum measured using a K-Alpha instrument, exported to ASCII format and converted to ISO format using CasaXPS. The header above the spectrum in Figure 6 includes the source string assigned by CasaXPS to the aluminium anode, namely, *KA1486*. The significance of the unique source string assignment for the K-Alpha data is that the *KA1486* string allows a set of element library entries to be prepared for use with the K-Alpha data. When a spectrum is displayed in the active tile in CasaXPS, the element library entries visible on the Element Table property page shown in Figure 7 are selected based on a match between the source string stored in the VAMAS block for the spectral data and the Excitation Source string located in the element library file. The element library file may contain entries for use with different instruments, and provided the excitation source strings are different for the different instruments, only those entries appropriate for a given instrument will be displayed in the element table in Figure 7. Thus, if a laboratory owned a PHI Quantum 2000, say, and also a Thermo Scientific K-Alpha, the element library entries for the PHI instrument could be populated with the excitation source strings *Al* while the K-Alpha will be assigned *KA1486*. On loading data from the PHI Quantum, CasaXPS will use and display only the entries with excitation source *Al*, while similarly K-Alpha data in CasaXPS cause the display of the K-Alpha sensitivity factors on the Element Table property page.
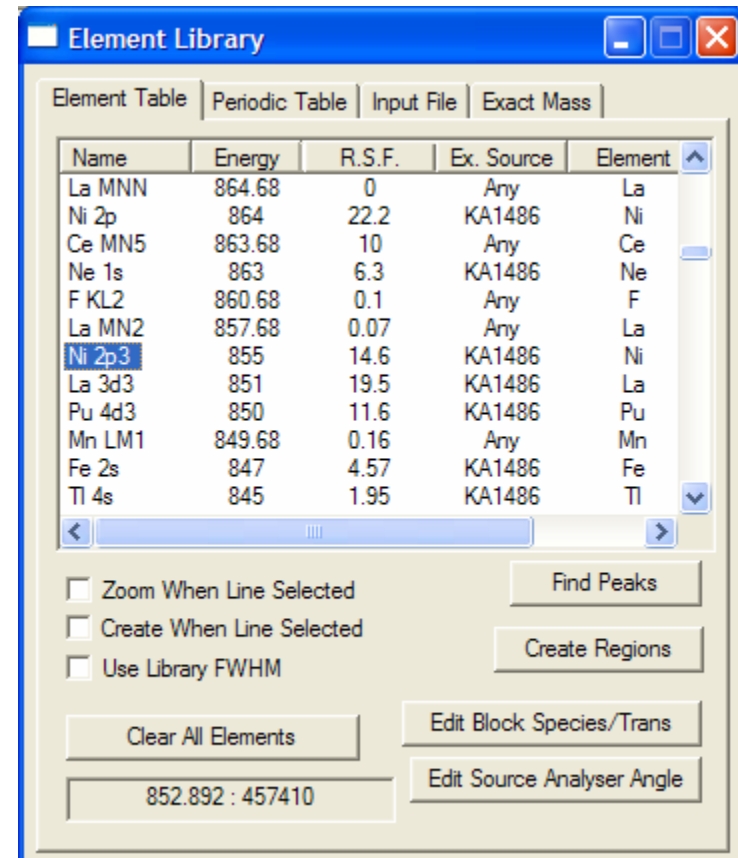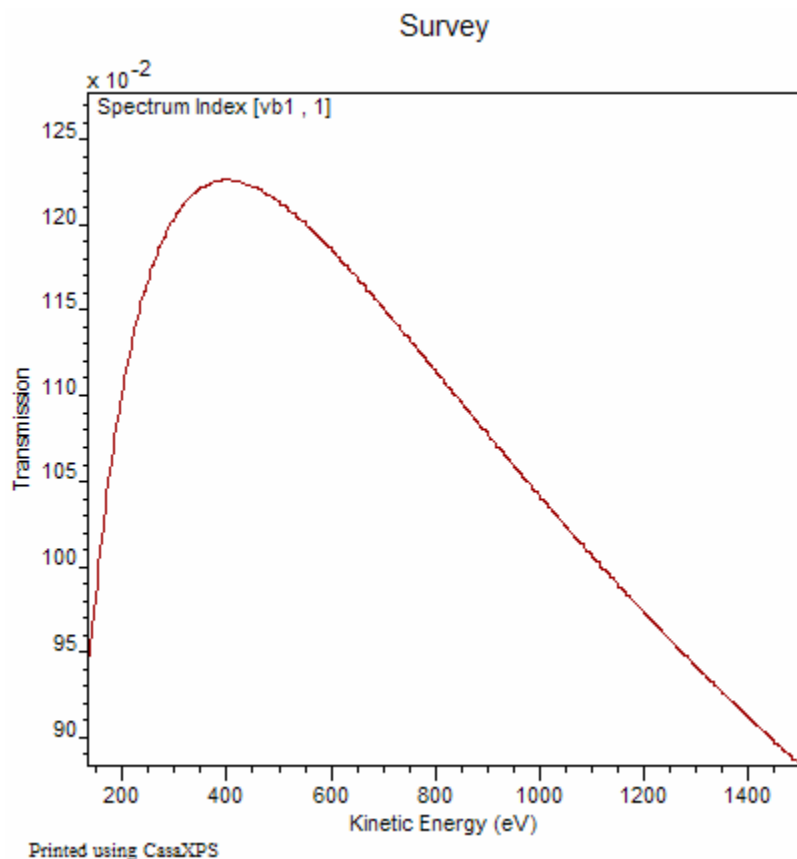

Figure 7: Element Table property page showing the Excitation Source string KA1486.

Transmission Function

Figure 8: Transmission curve for the data in Figure 6.

The transmission function for the K-Alpha is included in the ISO file created by CasaXPS. To view the transmission function in CasaXPS, hold down the Control key on the keyboard and press the PageUp or PageDown keys to switch between a display of the transmission curve or return to the spectral data. Figure 8 is an example of the transmission correction curve for the data in Figure 6.
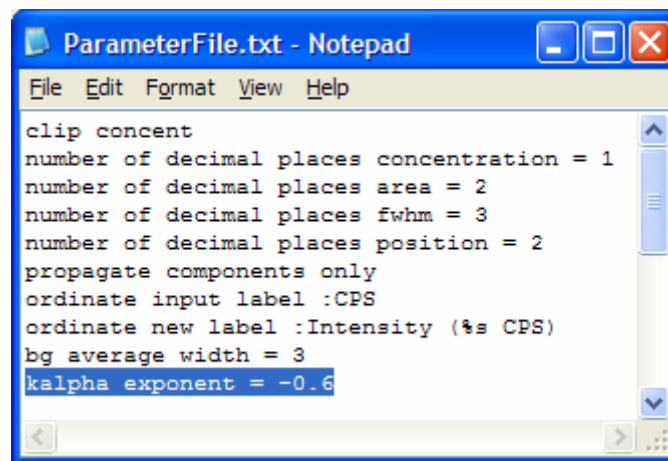
Figure 9: CasaXPS.DEF/ParameterFile.txt

The quantification table annotating the spectrum in Figure 6 computes the atomic concentrations using the raw area for each peak assigned a quantification region. CasaXPS divides the raw background subtracted area by the transmission function and the relative sensitivity factor, then applies the energy dependent correction calculated from the energy exponent (entered on the Regions property page) to the adjusted peak intensity. The default energy dependent exponent for the K-Alpha used with the Scofield cross-sections is -0.6, however the value used for the K-Alpha data is configured in the

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CasaXPS.DEF/ParameterFile.txt file, so other values for the exponent are possible. Figure 9 illustrates an example ParameterFile.txt file where the highlighted line is used to specify the energy exponent added to the data when converted from the Avantage ASCII files.
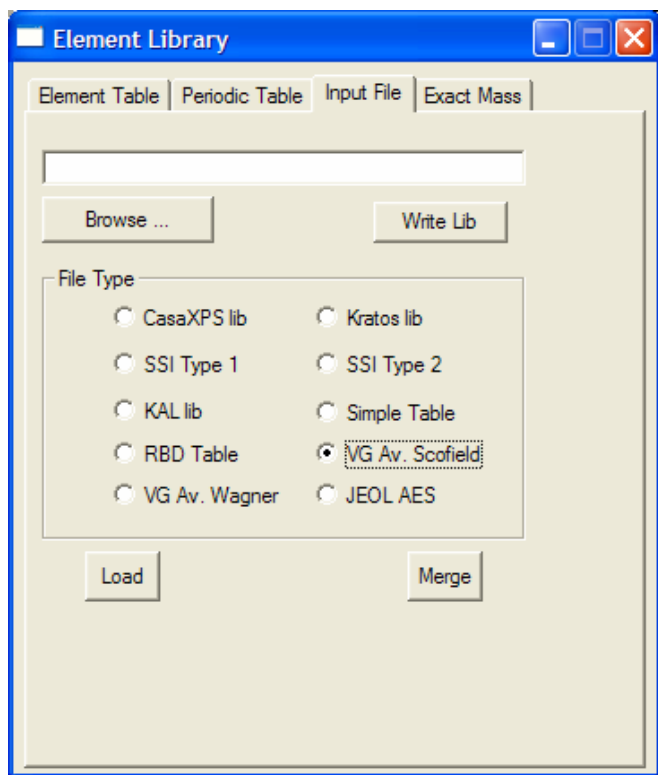

Figure 10: Input File property page on the Element Library dialog window.

Preparing an Element Library for use with the K-Alpha

The Avantage element library information is stored in a Microsoft Access database file. **The libraries supplied with Avantage are covered by the Avantage software license agreement and the original libraries or any converted formats must not be distributed. However, Thermo have made the library format available to enable individual users to obtain consistent results between Avantage and CasaXPS.**

Using Microsoft Access to open the .mdb file, select the Peaks table and copy the table into Excel. The Peaks table, viewed in Excel, can then be written to a Tab Delimited ASCII file for use in CasaXPS. Once the Tab Delimited ASCII file is available, the sensitivity factors from the Avantage system are loaded into CasaXPS using the Input File property page on the Element Library dialog window:

1. Use the Browse … button on the Input File property page (Figure 10) to identify the Peaks table file written from Excel.
2. Select the VG Av. Wagner or VG Av Scofield radio buttons on the same property page, depending of the sensitivity factors desired.
3. Press the Load button to replace the current CasaXPS library with the values from the Avantage Peak table file.
4. Exit CasaXPS, at which point CasaXPS will prompt for the filename used to save the modified element library.

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Save the element library to a new filename, e.g. CasaXPS_AvScofield.lib.

5. Open the newly created library file in Excel and globally change the source label strings as follows. The newly created file will contain rows for each transition in the original Peaks table file which will include entries for both Al and Mg anodes. Globally change the Al anode string to KA1486 and the Mg anode string to KA1253.

6. If another instrument using different RSFs is also in use, in Excel, open the current CasaXPS.lib file located in the CasaXPS directory containing the CasaXPS.exe executable file. Copy the entries from the newly created and source label adjusted CasaXPS formatted file corresponding to the K-Alpha library into the Excel file containing the current CasaXPS.lib file. The resulting spreadsheet should contain all the existing enters followed by all the entries from the Avantage file in CasaXPS format. After saving the resulting table of CasaXPS formatted entries to file, the combined file will need to replace the default CasaXPS library file called CasaXPS.lib in the directory containing the CasaXPS.exe executable file.

Once the new element library is prepared and in the correct location, CasaXPS will need to be restarted and data from each type of instrument examined. Load a spectrum form each of the instruments involved, open the Element Table property page and click on the tile displaying the spectra. Ensure the Ex. Source column displays the correct anode label for the data being viewed and also the RSF column is appropriate for the spectrum in the active tile.

Given an appropriate element library prepared as described above, data from either instrument will be quantified in exactly the same way. Each time a region or component is created using the element library, the appropriate RSF for the data will be retrieved from the combined element library.