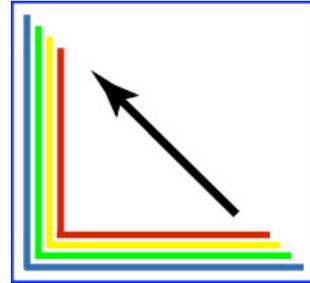


MigrationCorrection[©]

A program to correct for element migration.



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1 - Introduction

Some elements, such as Na and K, will migrate away from the heat of the electron beam during an electron microprobe analyses. This is particularly a problem when the material being analyzed is non-crystalline, like a glass. A crystal structure will greatly reduce the ability of an element to migrate away from the beam, but in a glass the elements are not constrained to particular sites. Therefore on heating, the more mobile elements will migrate away. This is particularly a problem for sodium, though other elements like potassium will also exhibit this phenomenon. During the time it takes to make an analysis, the measured counts for Na will drop (Fig. 1).

This obviously will cause a serious problem when trying to quantify the amount of Na in a glass. There are a number of strategies that have been used in the past to overcome this problem. These include:

- 1) Broaden the beam. If the beam diameter is wider, the electron density will be less and therefore less heating will take place.
- 2) Lower the beam current. This will also reduce the amount of heating in the sample. However it also will reduce the number of X-rays produced. Therefore longer count times will be required.
- 3) Use a cold stage to reduce heating in the sample (Nielsen and Sigurdsson, 1981).
- 4) Measure the count rate over time and back calculate the count rate at time zero. Then use this new calculated count rate to calculate the abundance of Na in the sample.

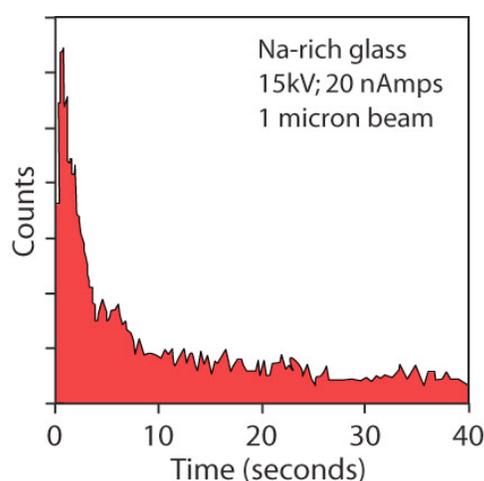


Figure 1. Na counts over time in a Na-rich glass.

In many cases, the fourth choice is the analyst's only option, particularly when the material being analyzed are small volcanic glass shards. In these cases, the beam cannot be broadened because the shards are too small; cold stages are not typically available, and even lowering the beam current won't stop the loss in counts. This program, MigrationCorrection, allows a user to collect a series of element measurements and then back-calculates the count rate at time zero. The user then runs the JEOL Off-Line Correction program to convert the new calculated count rate to weight percents.

2 – Analysis Setup

In order to collect a series of count rates for an element over time, the JEOL Quantitative Analysis program must be setup properly. Each element that is expected to exhibit this problem of element migration has to be analyzed multiple times. In the example shown in Figure 2, Na and K were selected five times each in the JEOL WDS Element Condition window. All other elements of interest were selected in a normal manner. How many times that Na and K are selected, and therefore analyzed, is up to the user. In most circumstances, analyzing each migrating element five times should be adequate. However the MigrationCorrection software will allow each element to be analyzed up to 50 times.

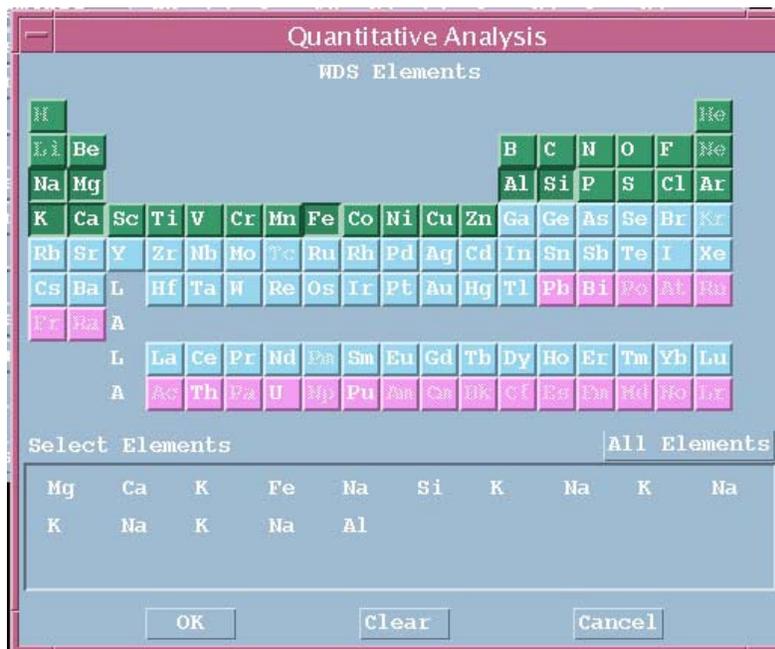


Figure 2. Selecting elements in the JEOL Element Condition window.

Next open the JEOL Measurement Order window, and arrange the elements in a manner similar to that show in Figure 3. All of the K analyses are listed on one spectrometer and all of the Na analyses are listed on a second spectrometer. The non-migrating elements were collected onto the remaining spectrometers. If the user cannot avoid putting non-migrating elements onto separate spectrometers, and have to put them on the same spectrometers as the migrating elements, then they must be run after the migrating elements.

The user has to now set the element conditions for each element (Fig. 4). For this example, the Na and K should be setup as shown below. The first 4 times that Na and K are analyzed, the backgrounds positions must be set to "0". The fifth time these elements are analyzed, the background positions must be set to their normal position. This will cause the background measurements to be skipped the first

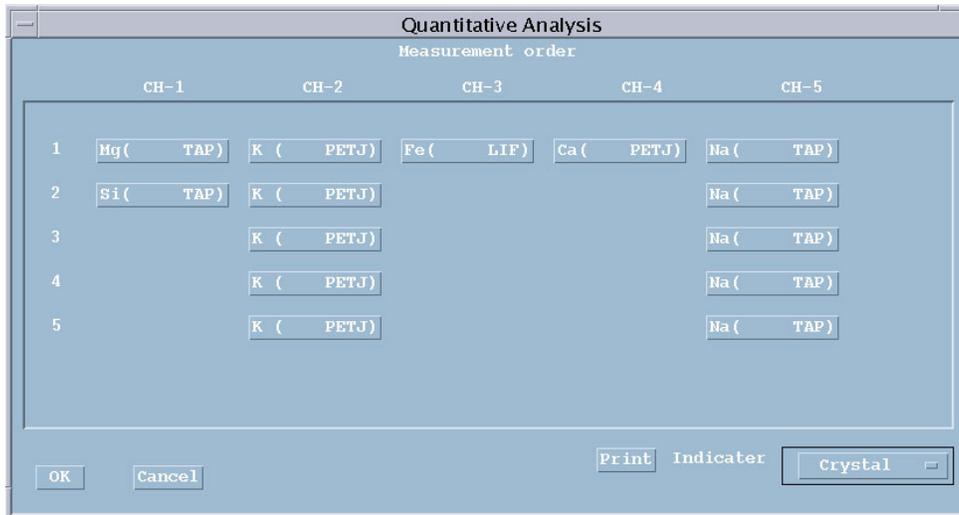


Figure 3. JEOL Element Order window.

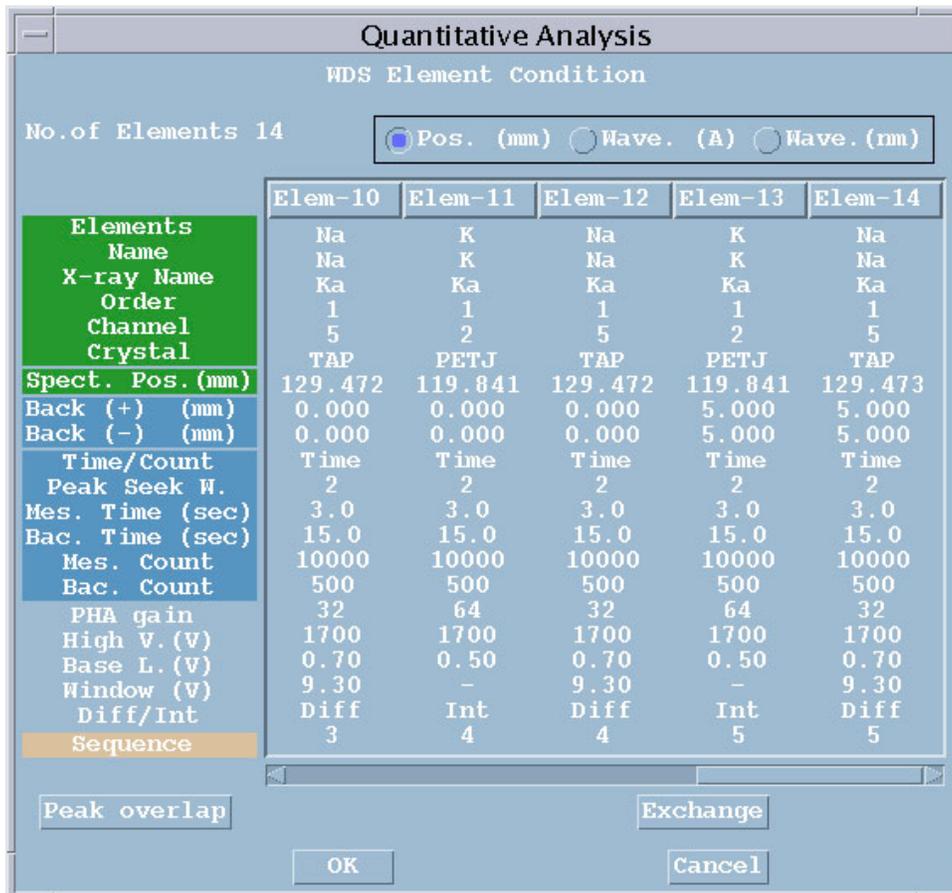


Figure 4. JEOL Element Condition window.

four times Na and K are measured. The background will only be analyzed after the last time each element is run. The length of the counting time for each element is up to the user. Counting times of 2-5 seconds have been found to work well. However it is critical that the same counting times are used for each time the migrating element is measured.

The quantitative analyses can now be run as normal. The JEOL software will average the counts from the elements that have been measured multiple times. Therefore the results will not reflect the true concentrations of the migrating elements. However the data has been stored in files for each measurement, allowing us to extract this data and to interpolate what the count rate must have been at time zero.

3 – Interpolating Data

The program MigrationCorrection will correct for the lost counts due to the migration of certain elements away from the electron beam during analysis. When the program is first run, it will display the window shown in Figure 5.

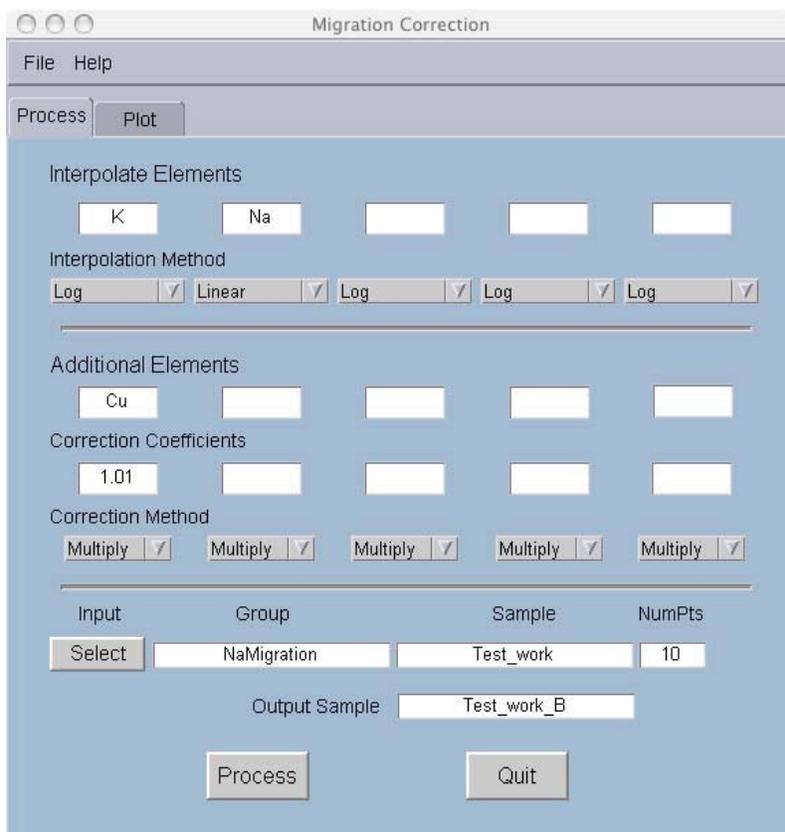


Figure 5. The main window of the program MigrationCorrection .

At the top of the window, the elements to be interpolated are entered, and then for each element, the interpolation method is selected. The user can choose to use a linear, a polynomial or a logarithmic function to interpolate the data.

If the user has some other corrections that they would like to perform on any of the elements in analyses, these can be done in the “Additional Elements” section. The user simply enters the element to be corrected, correction coefficient and selects whether the coefficient is to be added or multiplied to the raw X-ray counts for that element. This allows a user to make small corrections to the data that cannot be made in any other manor.

Finally the Group, Sample and Stage Numbers to be corrected have to be selected. To do this the user clicks on the Select button. The window shown in Figure 6 will open. The Group and Sample of interest can be selected.

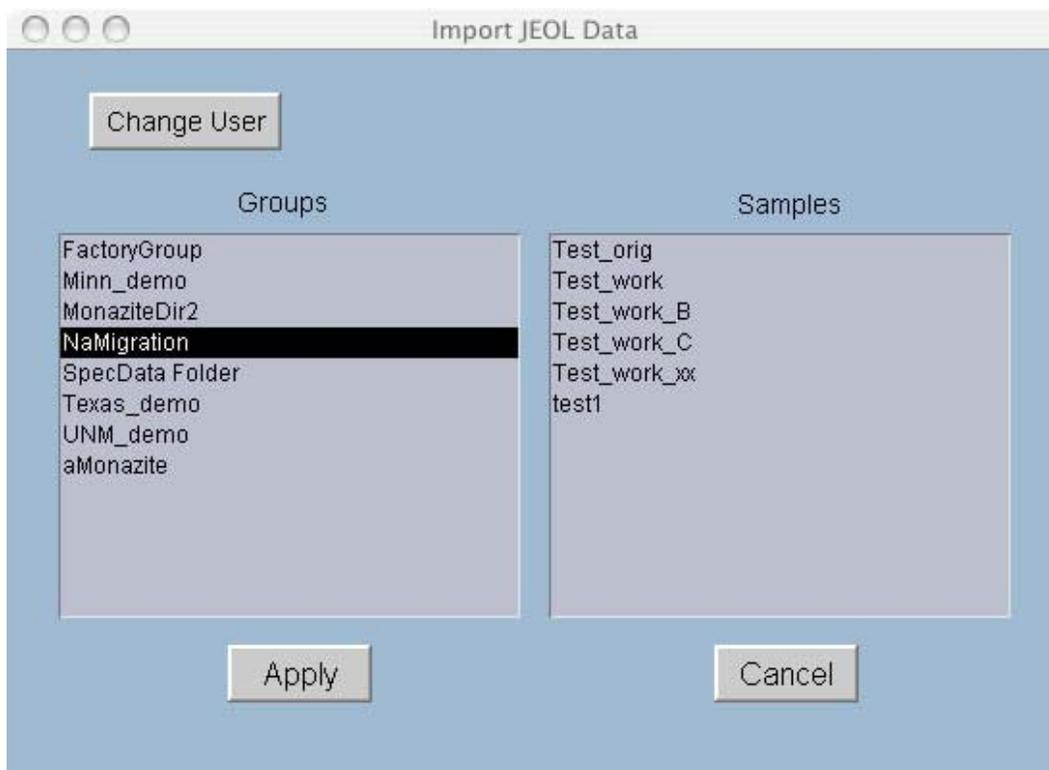


Figure 6. Import JEOL Data window allows the user to select the JEOL Group and Sample containing the quantitative data of interest.

The Apply button will open the Select Stage Position window (Fig. 7) allowing the user to select the stage positions of interest. Individual stage positions can be selected or unselected using mouse clicks. Multiple stage positions can be selected or unselected using <control> <shift>, or by using the Select All, Select, Clear All and Unselect buttons.

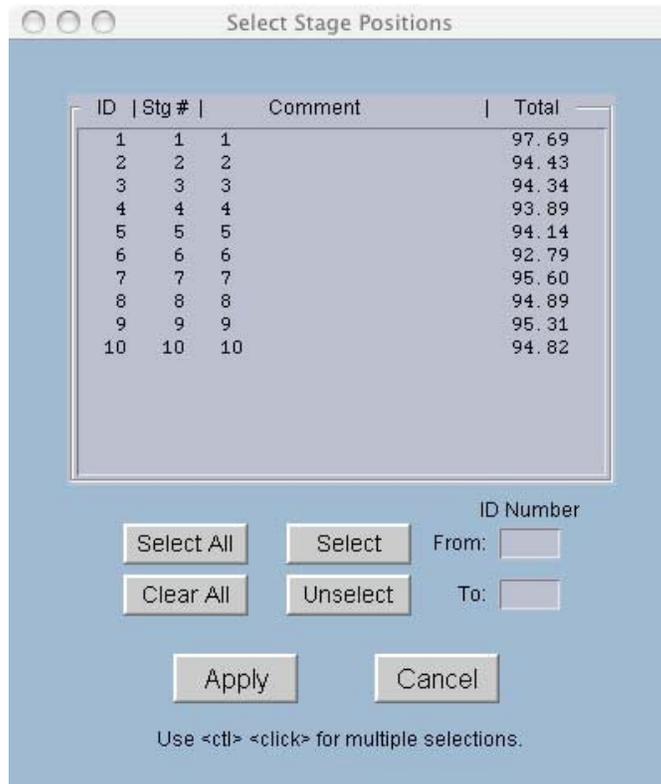


Figure 7. Select Stage Positions window allows the user to choose which analyses are to be processed.

The user directory can also be changed from the window shown in Figure 6 (Change User button). The resulting Select New User Directory window (Fig. 8) allows the user to navigate through the file system and switch into a different user directory. The user can move up through the file structure using the up arrow button or by using the pull down menu. Moving down deeper into the file structure can be achieved by selecting a directory and then by using the down arrow or the Open button. Once the new user directory has been located, it needs to be selected using the Select button.

The Process button will now read the original data and will interpolate the data to determine the counts rate at time zero. The old data will then be replaced by the new data in the files.

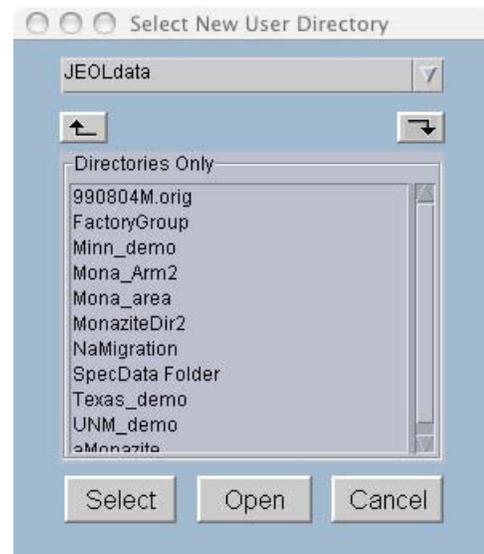


Figure 8. Select New User Directory window

4 – Checking Results

The quality of the interpolations can be checked by clicking on the Plot tab in the main window. This will change the main window to that shown in Figure 9. By clicking on one of the element buttons, the raw data and the interpolation curve will be displayed for the first stage position. To step through the remaining stage positions, either the Up or Down Arrow buttons can be successively clicked on.

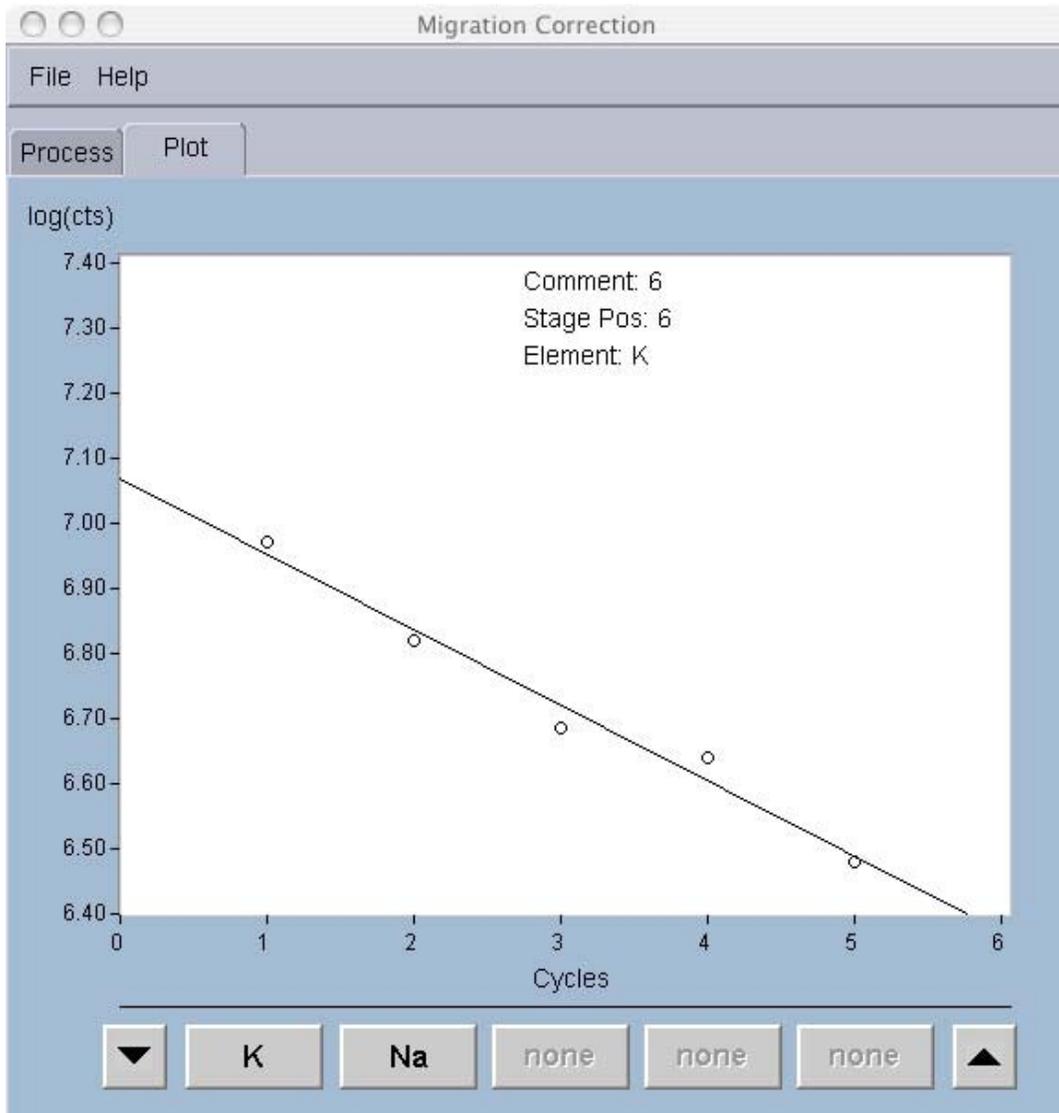


Figure 9. MigrationCorrection Plot window.

5 – Final Processing

The program MigrationCorrection only writes out the new interpolated raw counts into the original quantitative analysis files. It does not calculate new weight percents for the interpolated elements. This must be done by running the JEOL Off-line Correction program.

References:

Nielsen, C.H., and H. Sigurdsson, 1981, Quantitative methods for electron microprobe analysis of sodium in natural and synthetic glasses. *American Mineralogist*, vol. 66, p. 547-552.

Notes:

1) The MigrationCorrection program only works with WDS analyses, not with EDS analyses.

Installation Procedures

Installation instructions:

- 1) If it doesn't already exist create the directory:
 /opt/McSAssoc
- 2) Use the File Manager to copy the folder *MigrationCorrection* from this CD into the above directory.
- 3) Add the program to the EPMA menu:
 - a) Go to the main EPMA menu and select:
 > Initialize > Soft Config > Util Menu
 A file will open up.
 - b) Add the following line to the bottom of the file.

```
T :MigrationCorrection : /opt/McSAssoc/MigrationCorrection/runMigrationCorr.sh: 3:
```

- c) Now go to File > Save(needed) and save the file.
 - d) Close the window.
 - e) Go to main EPMA menu > Configure > Update Menu ...
- 4) The program is now ready to run. Go to the Utility menu and at the bottom of the list should be the new item "MigrationCorrection". Select it and the program should run.