

CAU Kiel - EPMA Lab
Technical Note No. 5
Calculation of Monazite Age Maps

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Abstract

This document deals with the problem of calculating and displaying age maps from count maps with the help of standard Jeol Epma software and in-house-programmed utilities. This task first is separated in two parts which are described to some detail: (1) generating concentration maps and (2) generating age maps from the concentration maps of U, Th and Pb. All information in this document is specific for Jeol EPMA software, version 3.03 which runs on a HP B180L workstation with system HP/UX 10.20.

1 The AgeMap Tools

Three utilities have been written which shall help you to perform the desired tasks. These are the following programs: “x2c”, “xlcor” and “x2am”. These tools can be downloaded as a tarball archive (“am-tools”) via anonymous ftp from <ftp://jxa2.min.uni-kiel.de/ftp/dist/sw/>

2 Generating Concentration Maps

From basic epma theory, concentrations are calculated from x-ray intensities using Castaing’s relation:

$$C_{i,unk} = C_{i,std} \frac{I_{i,unk}}{I_{i,std}} Z A F \quad (1)$$

where $C_{i,unk}$ and $C_{i,std}$ are the x-ray intensities per unit of probe current after interference and background correction.

Note, however, that in this equation, the ZAF correction factor is the ratio of the contributions from the standard and the unknown sample, according to:

$$Z = \frac{f_{Z,std}}{f_{Z,unk}}$$

(and equivalent expressions for A and F)

2.1 Applying a background correction

Because the underlying data of the MapDisplay software are raw counts rather than background corrected intensities, the latter first have to be calculated before applying further corrections. This can be performed by using the Calibration Line method (see Jeol-Manual section Element Map Software for a definition of the Calibration Line). In Calibration line method the concentration is related to the countrate of the standard by the simple relation

$$I = a C + b \quad (2)$$

thus

$$C = \frac{I}{a} - b \quad (3)$$

It is evident that in this model b (at $C=0$) yields the background intensity and a the slope of the calibration line. These two coefficients have to be calculated manually by the user from data of a pre-performed standardisation.

For the calculation of a it follows:

$$a = \frac{I_{net}}{C pc} \quad (4)$$

$$b = \frac{I_{bg}}{pc} \quad (5)$$

The unit of intensity is counts per milli seconds (ms) and the unit for the probe current (pc) is μA . Thus the unit of b is: counts (ms μA)⁻¹ and the unit of a : counts (ms μA %)⁻¹. Be careful to avoid mistakes with the units as this will result in meaningless data.

2.1.1 Calibration Line Method using internal standardisation

With this method, two quantitative analyses on the monazites are used as points on the calibration line. From the differences of composition and intensities of these two analyses the slope a and the b -value are calculated in the same way as described above. This method is equivalent to a linear behaviour of the ZAF coefficient between the two points (Fig. 1). Because the unknown has almost the same matrix composition as the standard no further ZAF correction has to be applied to the data. This method is integrated in the new version of the Jeol Epma software, running on SUN workstations and its use is recommended here.

2.1.2 Alternative approaches: Background mapping

It also could be considered to make a full mapping of the background calculate a net intensity map from this and the peak mapping by using the Mac Calculation software tool from Jeol Epma software. This, however is very time consuming but may be of advantage of polyphase maps with strongly varying matrix composition.

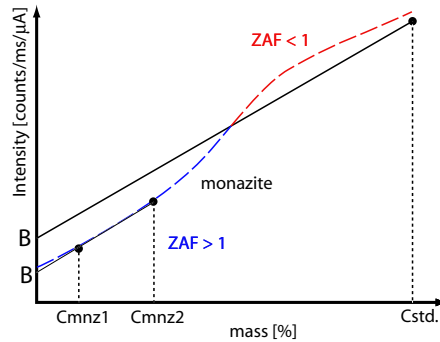


Figure 1: Derivation of the calibration line method. The figure shows schematically the relation between intensity and concentration and the influence of variation in matrix composition on the x-ray intensity.

2.1.3 Simple approach

Check a point on the map area where you assume that it does not contain the element of interest. Display the counts on this point and use this as the background. Make statistics: use an average of the counts over a line or area. This is easily performed with Map Analysis. This method has the disadvantage that the matrix is different, and the background intensity may also be different.

2.2 Applying a line interference correction

A possible line interference has to be eliminated, before the count rates can be used for further calculations according to eq. (1). The program “xlcor” performs a simple line interference correction on element 1 by the interfering element 2. As an example assume the interference of Th $M\gamma$ on U $M\beta$, which can be corrected with the equation

$$U = U - 0.012 Th_{net} \quad (6)$$

Th_{net} is the net intensity of Th $M\alpha$. The coefficient of 0.012 was determined for our probe measuring Th $M\alpha$ on spectrometer 3 and U $M\beta$ on spectrometer 2, both with a PET diffraction crystal. The program has to read in the two files with the counts of U and Th. Note that the program has to use the net intensity of Th $M\alpha$ for the correction. You therefore also have to enter the b-value for Th to enable the program performing the background correction. The count-data, corrected for line interference will be written to a file with the extension “.xlc”.

2.3 Applying a ZAF correction

In case that the standard and the unknown sample differ significantly in matrix composition, a ZAF correction has to be applied to account

for these effects. Use the ZAF coefficient obtained from a quantitative analyses of the monazite. Be aware that this coefficient is calculated according to equation (3) and it thus follows, that if you switch to a different standard, you also have to use a different ZAF coefficient. If you use a monazite as standard and calculate a monazite mapping, than you can assume that $ZAF=1$.

2.4 The program “x2c”

This program performs the background correction on the raw data and generates a concentration map. Enter the requested data after launching the program. The generated output file will get the extension “.con” to protect the original map files from being overwritten by accident. The program also reports a max. value, which can be used to set an upper limit of the level scale in the map display. Use this value in the 0.cnd file. Due to internal program design reasons all generated concentration data are in the unit ppm/10.

3 Generating the Age-Map File

Use the program “c2am” to perform this task. This program calculates a map which shows the distribution of apparent ages in a mapped monazite. After launching the program you will be asked for the names of the relevant concentration map-files for Th, U and Pb. After this the programm calculates for each pixel the apparent age by solving the decay equation iteratively. Depending on the size of the map and the speed of your machine this may take some time. The age map-file gets the file extension “.age”. The program also reports the max. value. You can use this number as the upper limit for the level scale by entering it in the 0.cnd file. The unit of the data which are calculated by c2am is Ma. To open and display the calculated map file with Jeol Map Display software you just have to rename the file so that it has the extension “.map”.

4 Howto

First calculate manually the calibration line coefficients a and b. For this you have to use two quantitative analyses on monazites which in an ideal case span up the range of concentrations in your unknown sample for the particular element. If you want to use the one-point calibration line method with a traditional standard you have to use the data as reported by Jeol epma software during the standard measurement. You will also find these data in */home/USER/.std/STDNAME/1.cor*. If you choose this method you have to correct for differences in the matrix between standard and unknown monazite by applying a ZAF coefficient. You can obtain this ZAF coefficient from a quantitative analyses using the same standard and accelerating voltage as during standardisation and during map analyses.

To my experience best results are received when using monazite as standard, because of the strongly matrix dependend background intensity.

/home/USER/GROUP/SAMPLE/.map/MAPNUMBER/0.cnd this is the condition file which has to be edited manually. Open this file with an editor. In the first line of this file increase the number of measured elementes by one, because internally the concentration map will be handeled just as an ordinary element map. Then, add a further mapping to the end of the file. Do this by copying one of the upper mapping blocks to the end. You can edit the specific fields. Jeol Map Display software needs this to recognise an additional map file and display it in the file selection dialog. At Max Value, enter the value as reported by x2c.

The map-files are compressed by default. You can easily expand them within your shell by using *uncompress {filename}*. *uncompress* restores the compressed files to their original form, renaming the files by deleting the “.Z” extension. For more information on this matter see the manpages of *uncompress*, *compress*, *zcat* and related UNIX tools.

Rename the concentration file with the extension “.map”. Jeol Map Display software recognises only files with this extension or their compressed form with the extension “.map.Z”.