10

DRIFT CORRECTION OF THE INSTRUMENT

10 Drift Correction of the Instrument

10.1 Introduction

The instrument is subject to long-term drift. The **Drift Correction** is an operation aimed at correcting for the drift and should be performed regularly.

- Drift Correction for **Quantitative analyses**

Remark:

Some images are taken form XRF instruments, but they are identical except sample position and names.

10.2 Quantitative Analyses

10.2.1 Explanation of the Drift Correction

Within the element description for each element of the Method the user specifies the samples to be used for this operation.

Drift correction uses the principle of recording a **nominal reference** or **DAY 0 intensity** in the Setting-up Samples file, and also an **actual (current) measured intensity,** which is updated each time the setting-up sample is run for all the channels using that sample.

The drift in the intensity of the setting-up sample for a particular channel is the difference between these two recorded values. There are four ways in which this can be used to drift correct intensities measured during an analysis:

10.2.1.1 No drift correction

If there are **no setting-up standards** defined in an element description of a Method, then no drift correction takes place for that element.

$$\boxed{I_{corrected} = I_{measured}}$$

10.2.1.2 Two point drift correction

If both a **Top and a Bottom Standards** are entered, then that element uses two points drift correction. Interpolating the intensity drift for both standards gives an estimate of the drift in an intensity value measured during an analysis, and the reading may then be corrected by removing the computed drift.

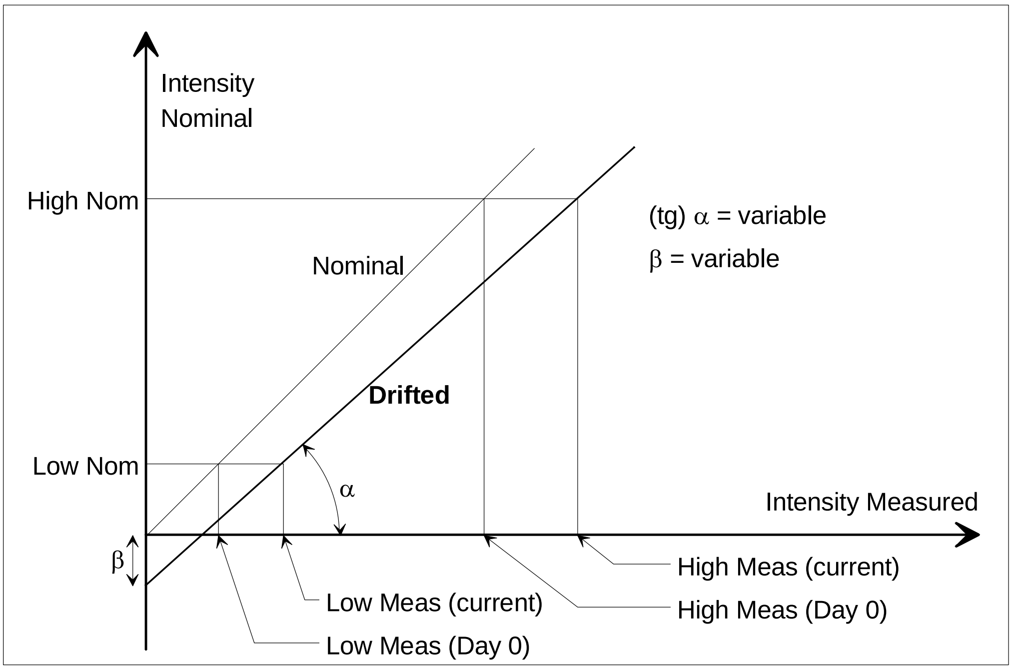

Figure 13.1

$$I_{corrected} = I_{nominal\ low} + (I_{measured} - I_{measured\ low}) \frac{(I_{nominal\ high} - I_{nominal\ low})}{(I_{measured\ high} - I_{measured\ low})}$$

$I_{corrected}$	Drift corrected intensity
$I_{measured}$	Measured intensity (eventually background corrected)
$I_{nominal\ high}$	Initial (day zero) intensity for the high (top) setting-up sample
$I_{nominal\ low}$	Initial (day zero) intensity for the low (bottom) setting-up sample
$I_{measured\ high}$	Current measured intensity for the high (top) setting-up sample
$I_{measured\ low}$	Current measured intensity for the low (bottom) setting-up sample
α	Slope of the curve
β	Displacement of the curve at the origin

Rotational drift correction

If only a **Top Standard** is entered and the bottom standard is left blank, the element is drift corrected by rotational correction. In this case, the intensity measured during an analysis is multiplied by the proportional drift in the top standard.

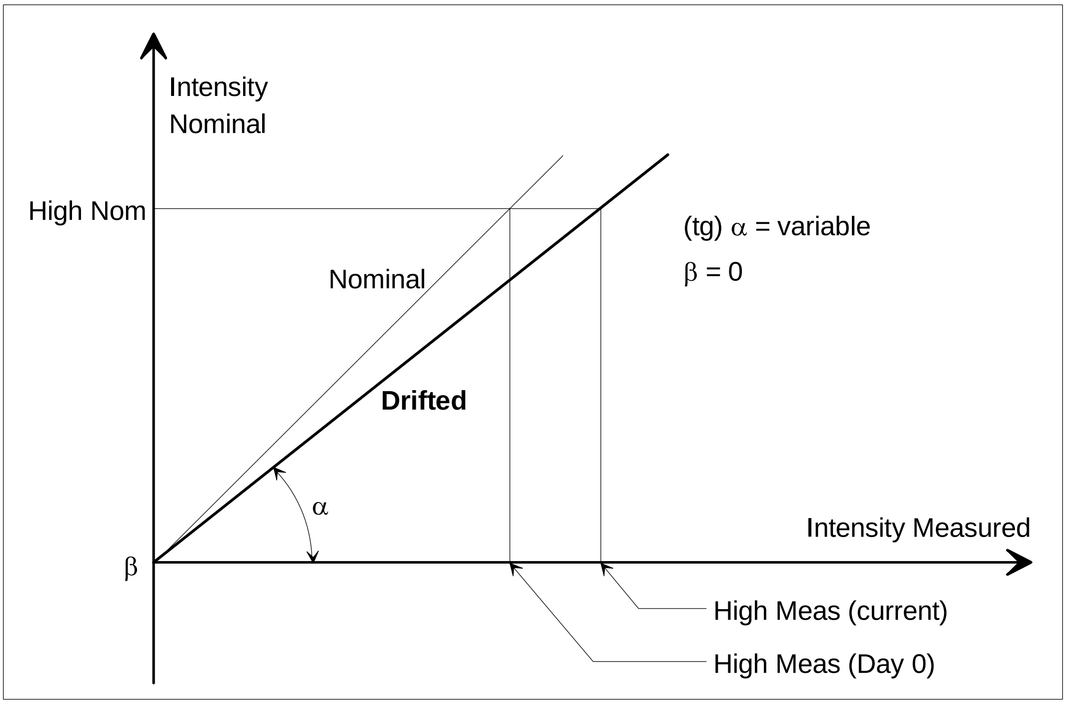

Figure 13.2

$$I_{corrected} = I_{measured} * (I_{nominal\ high} / I_{measured\ high})$$

Translational drift correction

If only the **Bottom Standard** is entered, translational parallel shift occurs. A constant value corresponding to the drift measured in the bottom standard is removed or added from all the intensity readings for that element in the program.

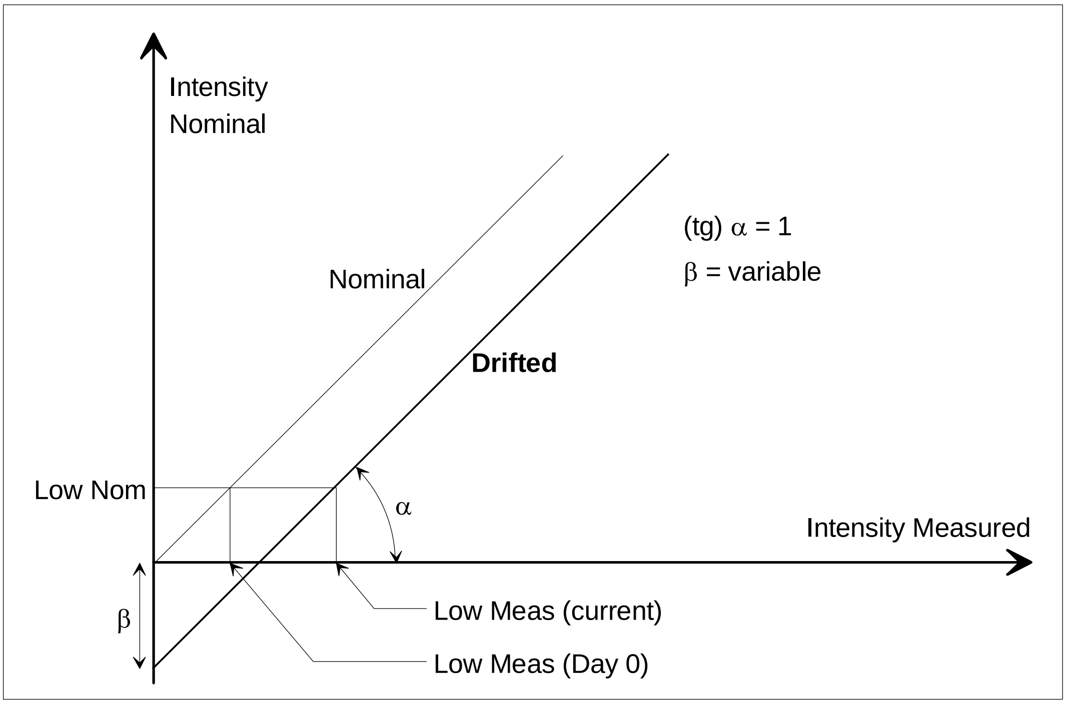

Figure 13.3

$$\boxed{I_{corrected} = I_{measured} + (I_{nominal\ low} - I_{measured\ low})}$$

Elements in the same program may use any of the above types of drift correction.

10.3 Drift Correction Update (Standardization)

At regular intervals, and at the discretion of the user, the Top and Bottom standards should be analyzed using the drift correction analysis mode. After performing a drift correction measurement each channel description within the **Setting-up Samples** file is scanned for the sample just analyzed. For each channel, OXSAS uses the stored **Absolute SIGMA** value to determine whether the measured intensity is to be unchanged, adjusted or replaced by the new measured intensity. This absolute SIGMA value is derived from the standard deviation obtained when the channel was measured for the first time (**Drift Correction Initialization**) and represents a drift tolerance.

10.3.1 Short Term Drift

During a **Drift Correction Update** the system first of all checks the difference between the actual (new) measured and the intensity that was measured the last time (old) for each channel specified in the Setting-up Sample.

This may be represented as:

$$\mathbf{DIFF = | I_{new} - I_{old} |}$$

where:

I_{new}	new (actual) measured intensity
I_{old}	old (last) measured intensity.

Note that the vertical lines mean that an absolute, unsigned value is taken for the value DIFF. For example:

$|\ 0.561\ | = 0.56, \qquad |\ -0.3451\ | = 0.345$

Adjustments are then made according to the following criteria:

Criteria	Value stored	Explanation
DIFF < SIGMA	I_{old}	last measured intensity remains unchanged
1 * SIGMA < DIFF < 2 * SIGMA	$(I_{new} + I_{old}) / 2$	last measured intensity is adjusted "halfway"
DIFF > 2 * SIGMA	I_{new}	last measured intensity is fully updated

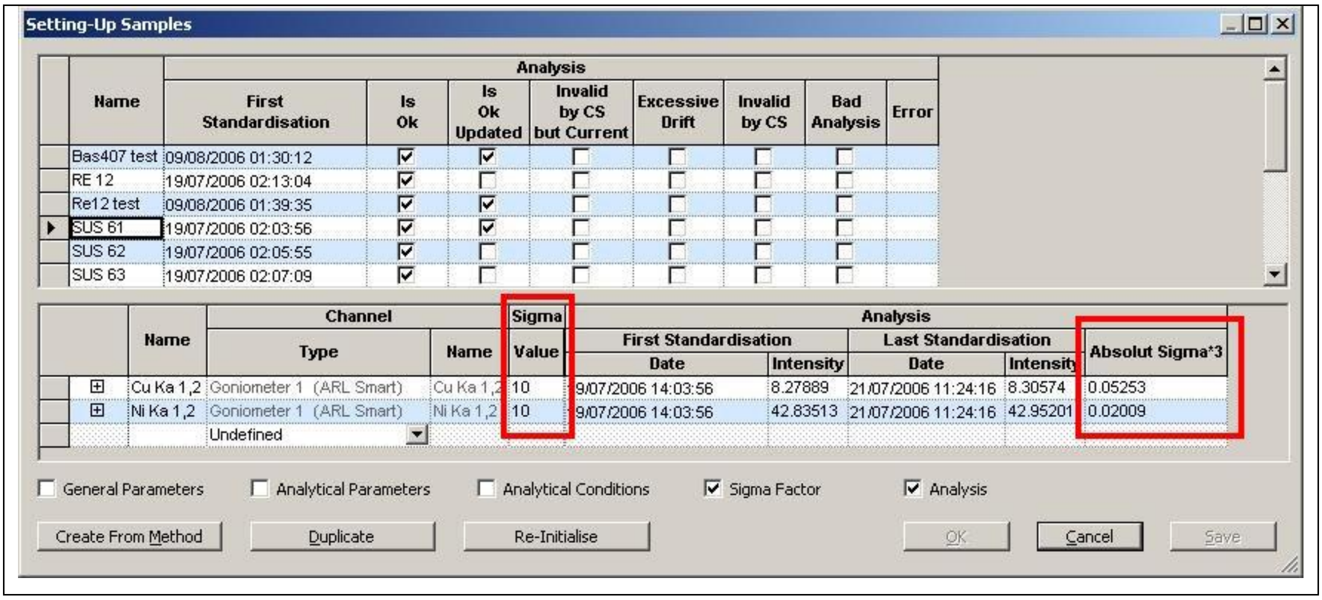

Figure 13.4

Note:	Here, the value called **SIGMA** represents 3 times the Absolute Sigma (calculated σ) for the channel, derived when the setting-up sample was measured the first time and recorded in the Setting-up Samples file.

This rule for adjustment is designed to take account of the precision of the instrument in performing the fine-tuning of the setting-up sample parameters.

If the difference between new and old measured intensity is greater than:

$$\textbf{DIFF} = \textbf{Sigma Value} * \textbf{SIGMA}$$

$$= \textbf{Sigma Value} * 3 * \textbf{Calculated } \sigma$$

then a warning is given to indicate an excessive drift.

A **SIGMA Factor** typically **10** or **20** is recommended to suppress the excessive drift warning, unless there is a reason for it (wrong sample).

10.3.2 Long Term Drift (Drift Tolerances)

The observation of the instrumental drift using the sigma factor and absolute sigma value only gives knowledge about the drift between the latest and the previous measurement of the setting-up samples. Normally, this short term drift is not big enough to trigger a drift warning and therefore, the operator may think that there is no or only a negligible drift. This might also be confirmed by the stability of the analyses. It is, nevertheless, important to know how the instrument drifts over a long period of time, due to ageing of different parts in the instrument, like the phototubes. This could be checked by comparing the nominal intensities (reference values) with the actual intensities, which is quite time consuming.

The **Alpha** and **Beta** values give a good indication of how much the instrument drifted since the initialization. If the instrument did not drift the alphas would be equal to1 and the betas to 0.

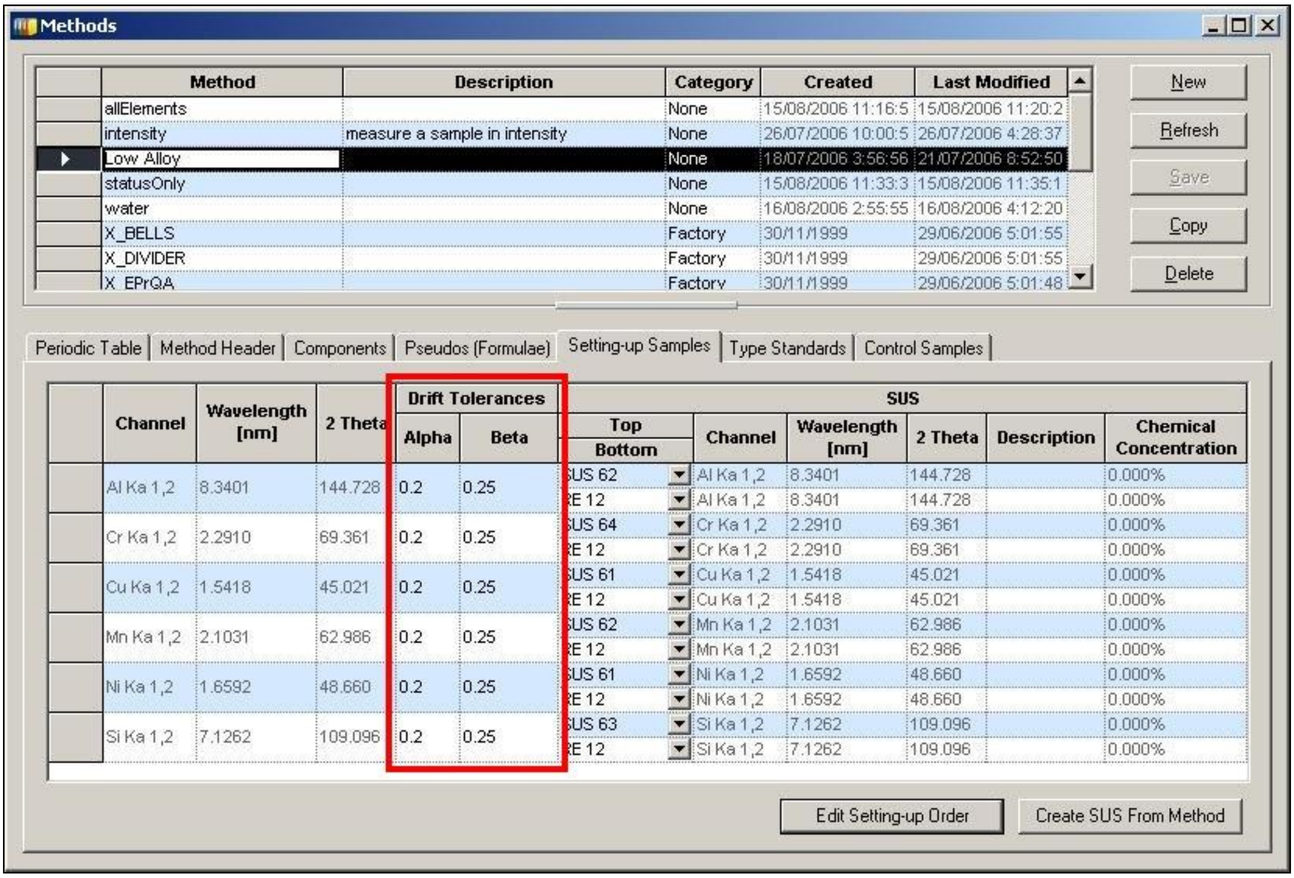

Figure 13.5

Their relationship with the general form of the drift correction equation is described hereafter.

$$\boxed{I_{corrected} = I_{nominal\ low} + (I_{measured} - I_{measured\ low})\,\frac{(I_{nominal\ high} - I_{nominal\ low})}{(I_{measured\ high} - I_{measured\ low})}}$$

Above equation can also be expressed in the following way:

$$\boxed{I_{corrected} = (\alpha * I_{measured}) + (\beta * I_{nominal\ low})}$$

with:

$$\alpha = (I_{nominal\ high} - I_{nominal\ low}) / (I_{measured\ high} - I_{measured\ low})$$

$$\beta = I_{nominal\ high} - (\alpha * I_{measured\ high})$$

where:

$I_{corrected}$	Drift corrected intensity
$I_{measured}$	Measured intensity (eventually background corrected)
$I_{nominal\ high}$	Initial (day zero) intensity for the high (top) setting-up sample
$I_{nominal\ low}$	Initial (day zero) intensity for the low (bottom) setting-up sample
$I_{measured\ high}$	Current measured intensity for the high (top) setting-up sample
$I_{measured\ low}$	Current measured intensity for the low (bottom) setting-up sample
α	Slope of the curve
β	Displacement of the curve at the origin

Alpha expresses the deviation from the slope when no drift existed and therefore, it indicates how the sensitivity of the instrument changes. For no sensitivity change Alpha is equal to 1, for a sensitivity loss Alpha is greater than 1 and for a sensitivity gain Alpha is smaller than 1.

Beta expresses the deviation from the origin when no drift existed and therefore, it indicates how the background of the instrument changes. For no background change Beta is equal to 0, for a background decrease Beta is positive and for a background increase Beta is negative.

The further Alpha moves away from 1 and Beta away from 0 the bigger the drift. There is, however, no absolute rule that indicates when it is appropriate to undertake actions to correct the situation. The ultimate requirement has after all to be judged by the quality of the analytical results.

10.3.3 Performing the Drift Correction Update (Standardization) in Manual Mode

Put the setting-up samples into the cassettes and place the cassettes onto the previously specified positions on the sample loader.

The analysis for the Drift Correction Update is invoked by selecting **Analysis and Data ¦ Standardize** as shown in the figure below.

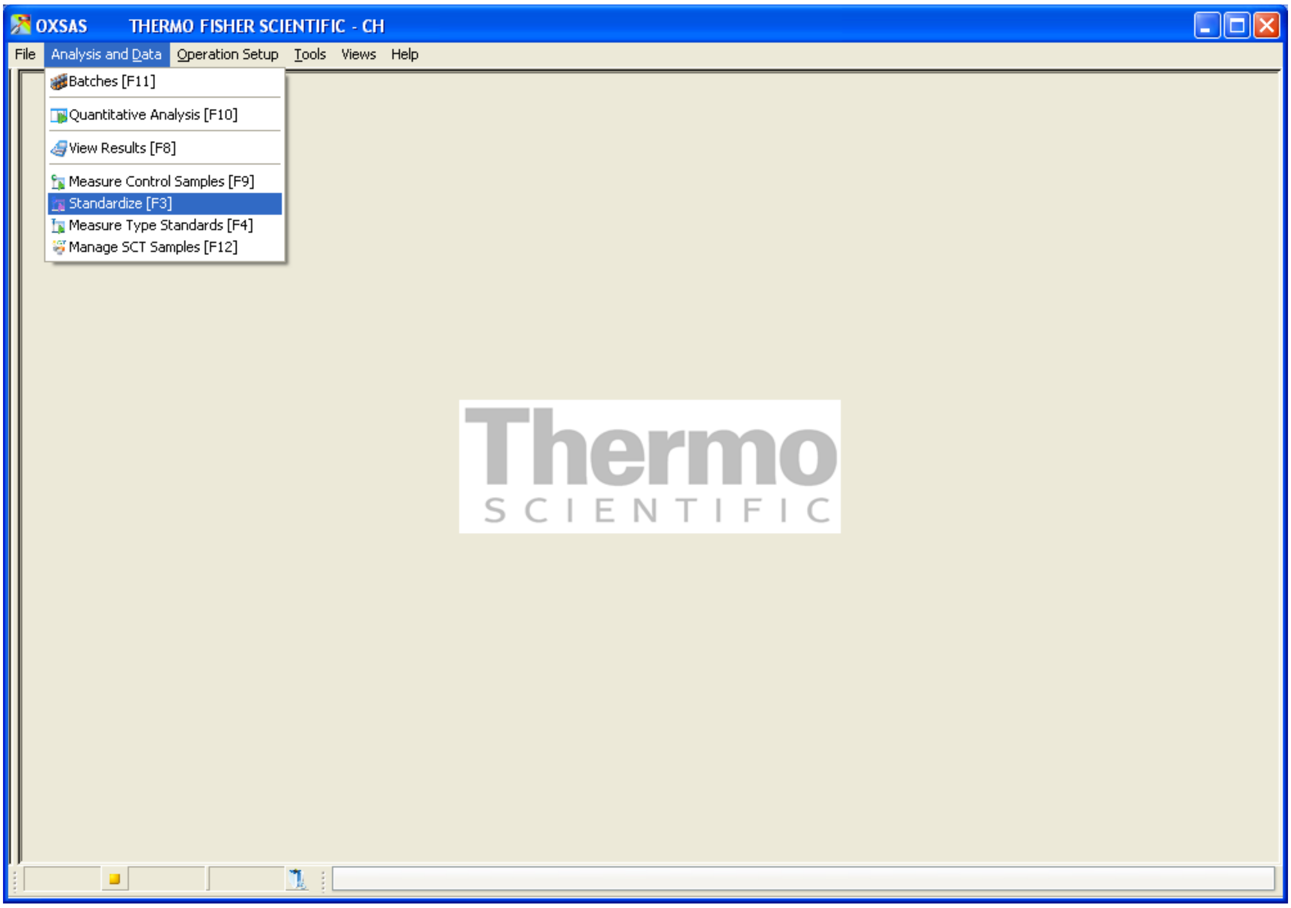

Figure 13.6

The dialog box shown in the next figure is then displayed.

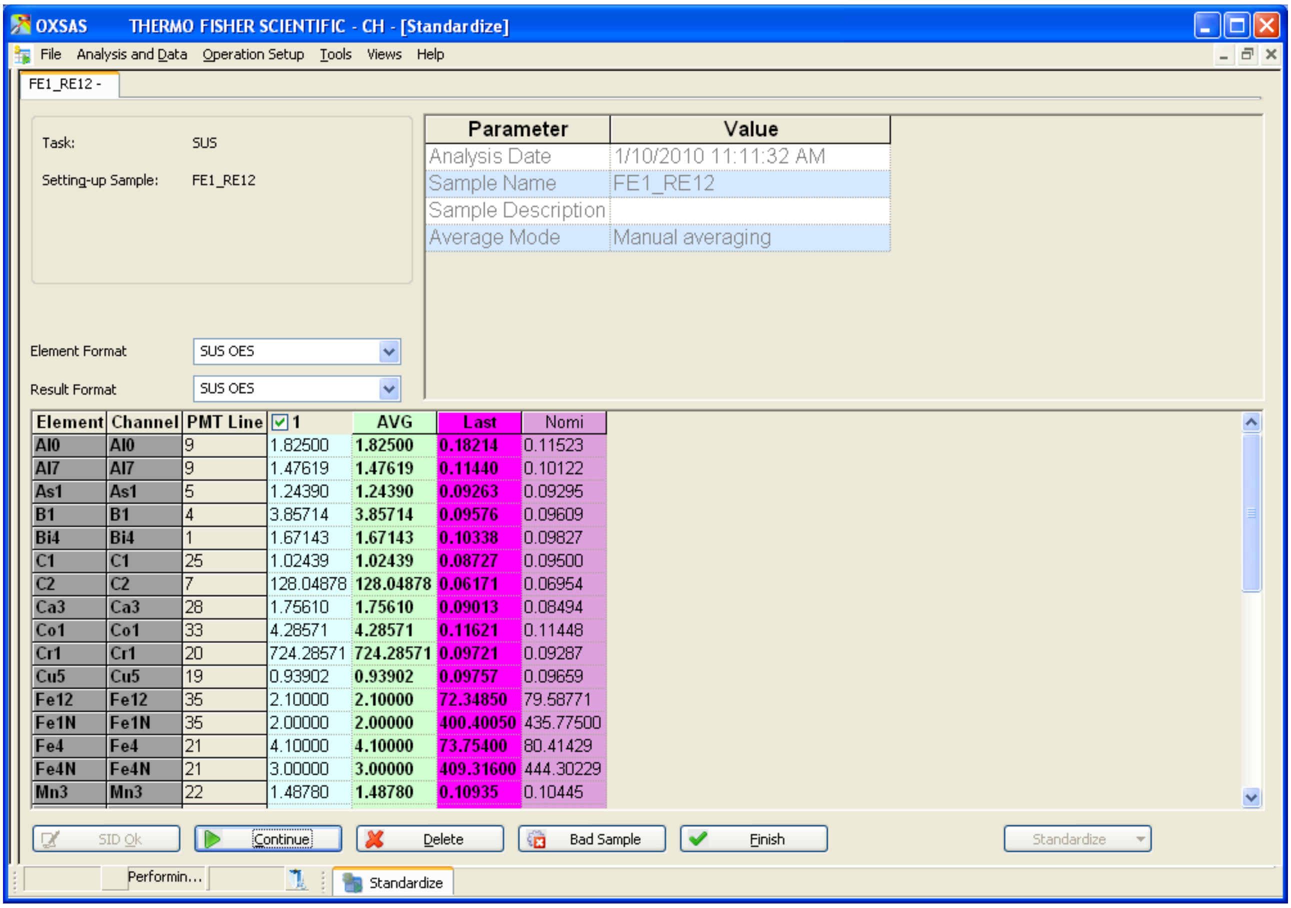

Figure 13.7

- From the drop down list **Task** select the task for the measurement of the setting-up samples. **SUS** is the default task made for the drift correction initialization and update. The parameters and values are displayed in the **Parameter / Value grid**.
- From the drop down list **Setting-up Sample** select the first sample to be measured.

To start the measurement of the first sample, press on the button ***SID OK***.

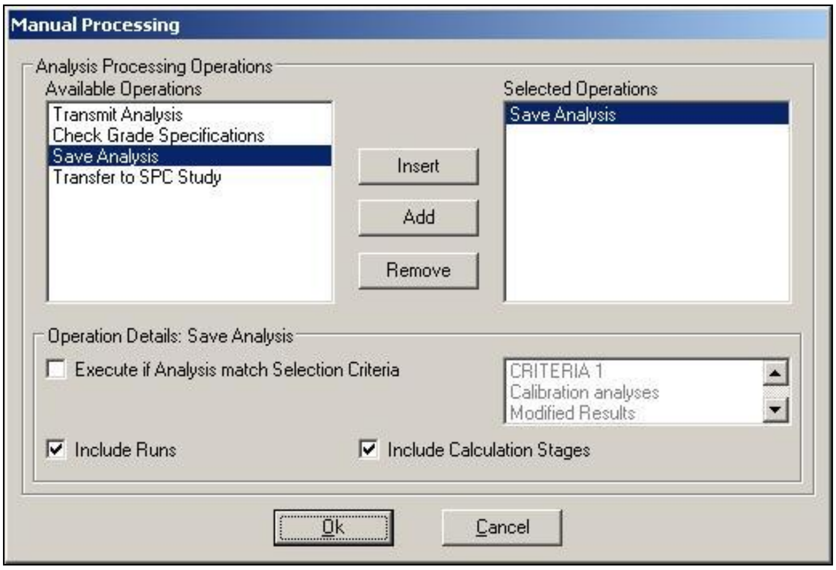

Figure 13.8

Depending on the Processing option (manual or automatic) the dialog box shown in the previous figure will be displayed or not. For manual processing you have the possibility to select whether you want to transmit the analysis (can also be used to print the analysis), save the analysis or transfer to SPC.

Figure 13.9

At this stage if an excessive drift is detected, a warning message is issued.

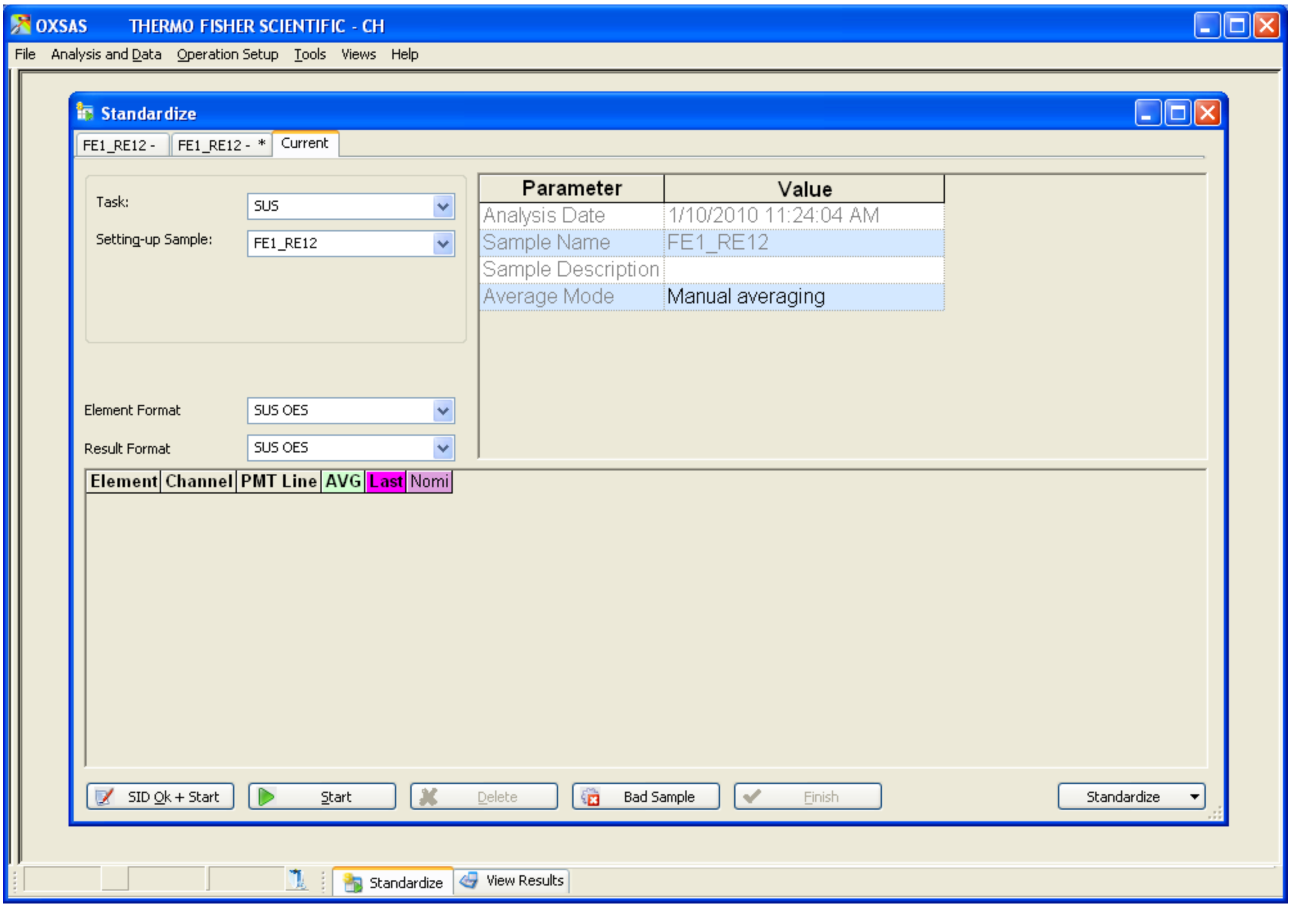

Figure 13.10

➽ The Standardize window is presented again. Click on **Standardize** and choose between the 2 options:

Finish Standardization: The system updates or not the intensities according to the rules explained under section *Short Term Drift* and stores the date and time of the new drift correction update. These intensities will be used to compute the alpha and beta for any subsequent analysis of unknown samples. The system will also warn if excessive drift has been detected and let the operator decide whether to update the setting-up sample or not.

➽

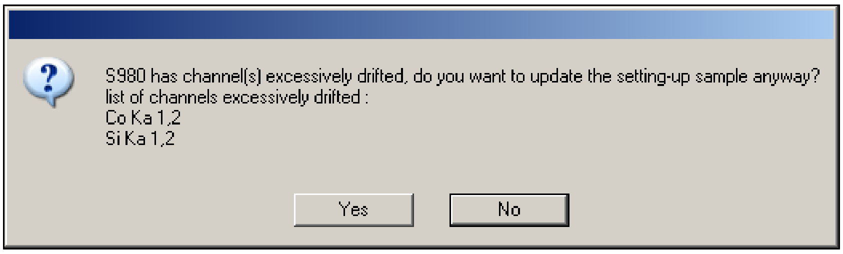

Figure 13.11

Flush Standardization: the newly acquired intensities are discarded, the setting-up sample intensities are not updated.

10.3.4 Performing the Drift Correction Update (Standardization) in Batch Mode

Put the setting-up samples into the cassettes and place the cassettes onto the previously specified positions on the sample loader.

If a Batch to measure the concerned setting-up samples already exists you can directly run the Batch to start with the measurements. If not, you first have to create it. This is done by selecting ***Analysis and Data*** ¦ ***Manage SCT Samples*** as shown in the figure below.

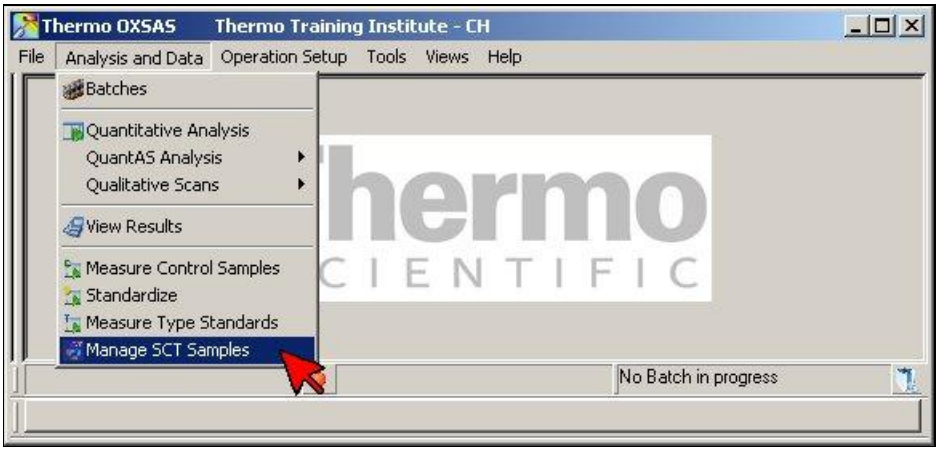

Figure 13.12

The dialog box shown in the next figure is then displayed.

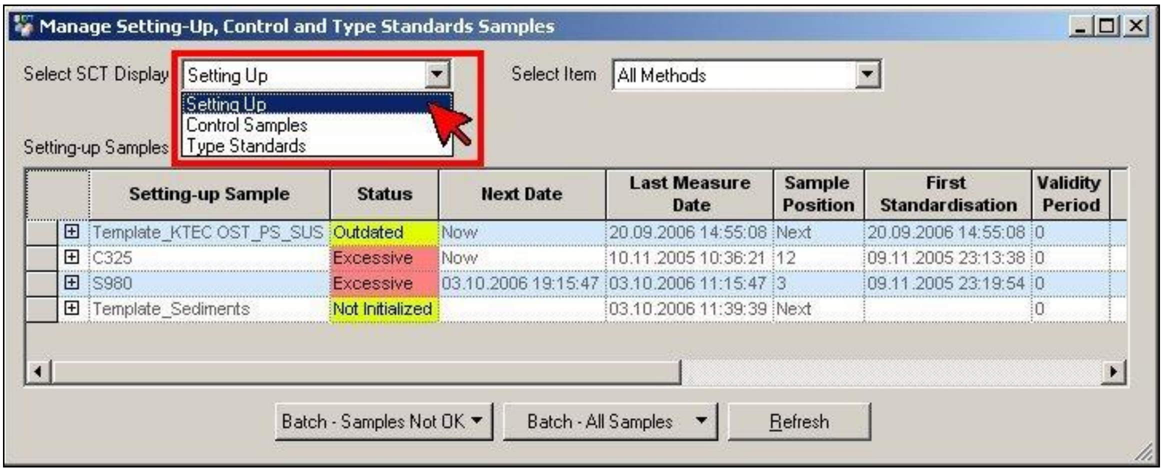

Figure 13.13

Here open the drop down list **Select SCT Display** and select ***Setting-Up***. The status for all setting-up samples defined in the system is shown. By selecting a particular Method you can see the setting-up samples status for that Method.

Possible status

Error — This indicates that a system error has occurred unrelated to analysis errors (shown in red).

Bad Analysis — This indicates that the last analysis of this sample was a bad analysis (shown in red).

Excessive Drift — This indicates that the latest analysis showed excessive drift. Excessive Drift is recorded if the drift exceeds the absolute sigma value multiplied by the sigma factor (shown in red).

Invalidated (by Control) — This indicates that the analysis of a Control Sample, associated to a Method used by this Setting-up sample, is out of control (shown in yellow).

Outdated — This indicates that the delta time between setting-up sample analyses has expired (shown in yellow).

Invalidated but current — **For Automation only**: This indicates that the analysis of a Control Sample, associated with a Method used by this Setting-up sample, is out of control. However the validity time defined for this Setting-up sample has not expired so its readings are still considered to be valid (shown in green).

OK Updated — This indicates that the last setting-up sample measurement was used to update the intensities for the setting-up sample (shown in green).

OK — This indicates that the last analysis of this sample did not show a significant drift and its latest intensities did not need updating (shown in green).

In the same dialog box you can also see under **Next Date** when the setting-up samples are due to be analyzed.

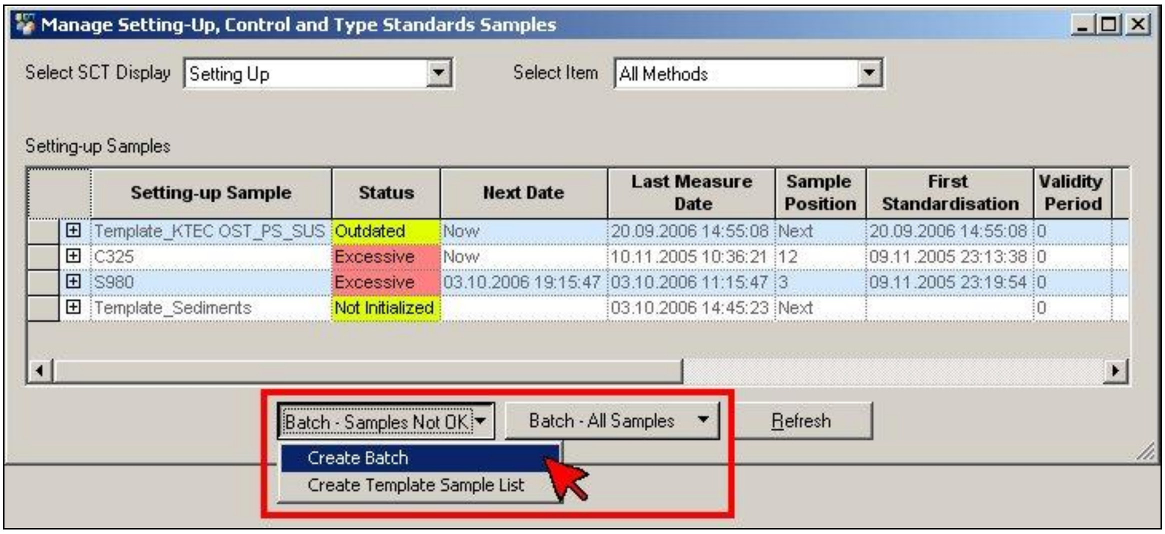

Figure 13.14

The next step consists in creating a Batch for the measurement of the desired setting-up samples.

Press on the button ***Batch – Samples Not OK*** and select ***Create Batch***. A new Batch called **$MethodName$SUS** is automatically created and it is containing all previously displayed setting-up samples with a status different than **OK** and **OK Updated**.

Note: The other button ***Batch – All Samples*** allows you to create a Batch to measure all previously displayed setting-up samples regardless to their status.

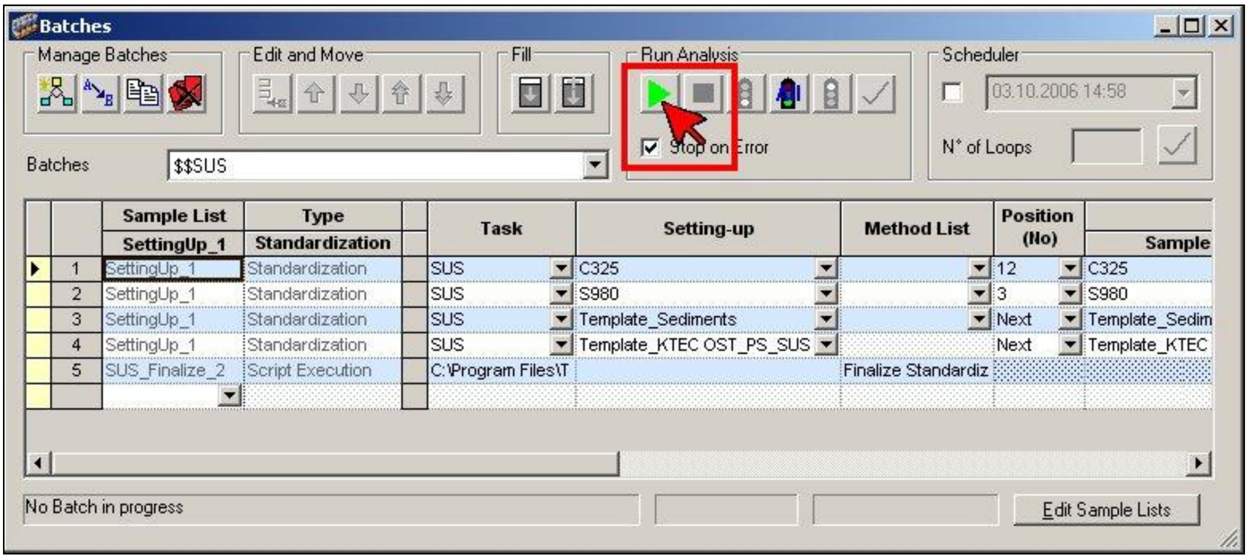

Figure 13.15

Press on the ***Green Arrow*** button to start the measurement of the first setting-up sample.

Note: You will see that the script **FinalizeStandardization** has been added to the Batch. It is executed after the last analysis and finalizes the standardization.

During the measurements the analysis screen is presented and therefore you can see the current and previous results if necessary.

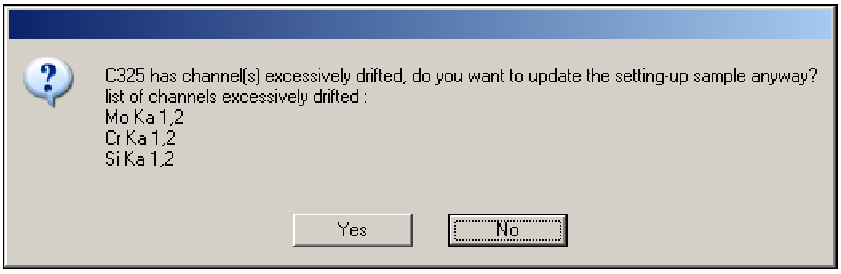

Figure 13.16

In case one or several setting-up samples show an excessive drift you have the choice to store or not the new intensities.

Note:	If later you want to view the measurement results, open the Standardize dialog (***Analysis and Data*** ¦ ***Standardize***), according to the type of samples that you have measured.