Guide to DeltaT-iQ

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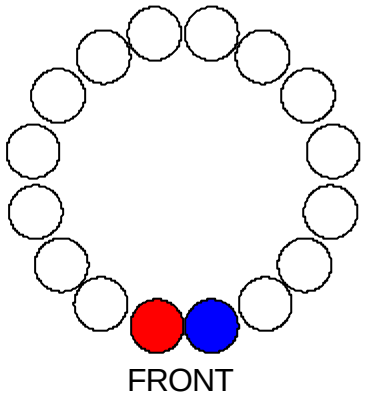
Contents

About this Guide

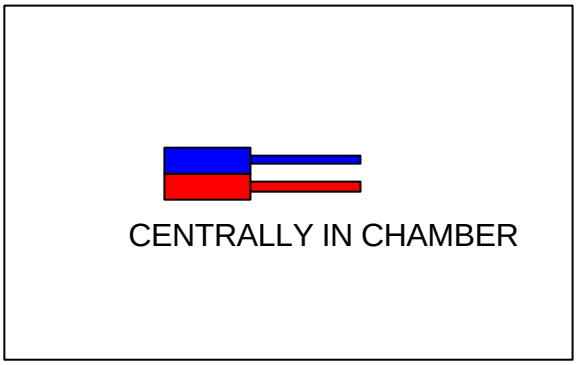
DeltaT-iQ provides the user with two additional features to help users with their GMP requirements. These features are the Calibration and Qualification tools and are explained in more detail below. It also provides details on customising the system so that it is appropriate to the users' own profiles. Familiarity with the basic operation of DeltaT is assumed.

Calibration Report

There are two stages to the calibration of a freezer; an adjustment followed by a calibration. In the adjustment phase, the purpose is to determine the difference between a reading given by the freezer and a reference standard and then to adjust the freezer to minimize this error. In the calibration phase, the purpose is to record the remaining errors and determine that the error is within acceptable limits. In order to calibrate a freezer, the adjustment run is performed using a traceable reference probe placed in the chamber. The position of the probe depends upon the freezer in question but standard positions are shown below. Note the reference probe and measurement system should have an accuracy better than ±0.05 °C



1.7 AND 3.3 LITRE CHAMBERS

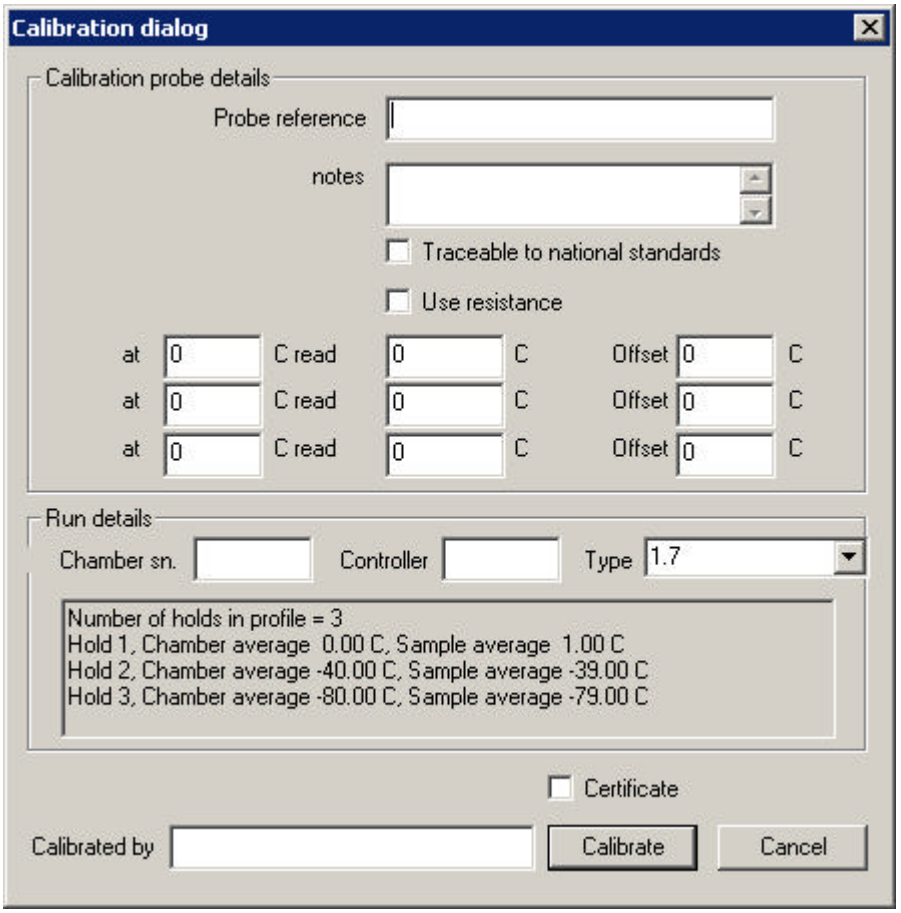


16 LITRE CHAMBERS AND ABOVE

SAMPLE PROBE

REFERENCE PROBE

The adjustment run normally comprises just one hold at the adjustment temperature with a duration of at least 10 minutes; for Planer freezers this is normally 0 °C. Using the resulting KryoFile, a **Calibration report** can be produced which will also provide the appropriate adjustment values for the freezer. To produce the report, follow the instructions below:
While the KryoFile is displayed, select **Calibration** from the **Tools** menu or click on the **Calibration** button to open the **Calibration Dialog** window.



When opened, the window automatically analyses the first 3 holds in the KryoFile to allow a calibration report to be generated. In order to produce a calibration report you will need the temperature data from the reference probe that was placed in the chamber during the run and from which the temperatures were recorded during the holds

The window allows the reference measurements to be entered in either degrees C or, if a PT100 probe was used, ohms.

In the Calibration probe details section enter the following

Probe reference Serial number or other code identifying the reference probe used to perform the calibration,

Notes Any notes you wish to add to the report

Traceable to national standards Select this check box if the reference is traceable.

Use resistance Select this if the reference probe is a PT100 probe and you want to enter the temperatures as measured resistances.

The program allows entry of measurements for up to 3 holds depending on the number of holds in the profile. For each of the holds enter the following:

at Nominal temperature at which the calibration measurement was taken. This should be the same as the nominal temperature of one of the holds.

read Temperature (or resistance) as measured by the reference probe at this temperature.

Offset Offset that should be applied to the measured temperature. This figure is added to the temperature entered in the previous field (or calculated from the entered resistance)

For example, if the nominal hold was 0 C and the probe read 0.12 C and it is known form the probe's own calibration that at 0 C it actually reads 0.21 C, you would enter:

at 0 C read 0.12 C Offset -0.21 C

The offset is entered as minus 0.21 as this is what needs to be added to the probe's reading to obtain the correct temperature. (*At 0C the probe reads +0.21 C*)

In the Run details section enter the following:

Chamber sn. Serial number of the controller.

Controller sn. Serial number of the chamber.

Type From the drop down list select the parameters appropriate to the system to be calibrated.

Certificate Select this check box if you want to produce a certificate.

Calibrated by The name of the person who performed the calibration.

When complete click the **Calibrate** button to generate a calibration report. You will be prompted to enter a filename under which to save the report. Note that reports are created as HMTL files and once created will be opened in your default browser. The report will also include adjustment values for each of the holds. These will be provided in units appropriate to the freezer type. These values can be entered into the freezer's controller to reduce the error at the hold temperatures. Note that although adjustment values are provided for up to three holds, the Planer freezers only allow

adjustment figures to be entered for one temperature. As such the adjustment figures for the temperature of main concern should be used; this is typically 0 °C. These values should be entered into the freezer (refer to the freezer's documentation for details on how to enter the values.)

Once the freezer has been adjusted, the routine can be repeated but this time with a calibration profile. The calibration profile can contain up to 3 holds and is normally designed to reflect the use of the freezer and the temperatures at which calibration data are available for the reference probe. Each hold should have a duration of at least 10 minutes and the rate between each hold should not exceed 5 °C/minute. Note that in the first pass, a set of adjustment figures for a single point were calculated to allow the freezer's readings to be adjusted. In the second run, the data from the three holds are being collected to allow the difference between the reference probe and the freezer to be documented and checked.

It is important to note that DeltaT uses the profile to analyse the run. The run must not include seeding of any type as this will result in deviations from the profile.

Qualification Report

The Qualification report allows statistical data to be collected from a KryoFile. While a KryoFile is displayed, select **Qualification** from the **Tools** menu or click on the **Qualification** button to open the **Qualification Dialog** window.
When the **Qualification dialog** appears, enter the following:

Chamber sn.	Serial number of the chamber
Controller sn.	Serial number of the chamber.
Qualification by	The name of the person performing the qualification.
Type	From the drop down list select the parameters appropriate to the system to be calibrated.

These details need to be re-entered for each analysis.

When complete, click the **Start** button to generate a qualification report. You will be prompted to enter a filename under which to save the report. Note that reports are created as HMTL files and once created will be opened in your default browser.

The report analyses the KryoFile to generate the following data.

Note that the definition of chamber error differs depending on the controller type.

- For MR3, Compact and Kryosave controllers the chamber error is the Chamber temperature minus the Profile temperature.
- For MRV and PID Engine controlled units such as the Kryo250 and Kryo750Plus, the chamber error is the Profile temperature minus the chamber temperature.

The sample error is the Sample temperature minus the Chamber temperature for all controllers.

N Readings	Number of data points in the trace
Minimum C	Minimum error
Maximum C	Maximum error
Mean	Mean error
Variance	Sample variance
Max. 5 min mean	The maximum magnitude of the mean calculated over any 5 minute period.
Max. 5 min variance	The maximum variance calculated over any 5 minute period.

Using this raw data the program calculates the following parameters for quantifying the performance of the freezer.

95% limit	mean + 1.96 * sqrt(variance)
Rolling 95% limit	Maximum 95% limit calculated over any 5 minute period.
Stable 95% limit	Same as the 95% limit but ignoring any points less than 2 minutes after a rate change of more than 1 C/minute.
Min error.	Minimum recorded error.
Max error.	Maximum recorded error.
Min stable error.	Minimum error but ignoring any points less than 2 minutes after a rate change of more than 1 C/minute.
Max stable error.	Maximum error but ignoring any points less than 2 minutes after a rate change of more than 1 C/minute.
Max. first sig. error	Maximum error that occurs within two minutes of the first rate change of more than 1 C / minute.
Chamber error C	Mean chamber error calculated over a 2 minute period from the centre of the hold.
Sample error C	Mean sample error calculated over a 2 minute period from the centre of the hold.

The program will also grade the system. There are 10 possible grades.

1 to 3	The system meets the performance expected for a new unit under controlled factory conditions.
4 to 6	The system meets the performance expected from a new unit when installed.
7 to 9	The unit meets the requirements for normal operation under the less controlled environment that might be expected in the field.
10	The system does not meet the expected performance criteria

Note that the criteria depend upon the profile to be analysed and may not be applicable to all profiles. It is also important to note that the program uses the profile to analyse the run. The run must not include seeding of any type as these will result in deviations from the profile.
More details on setting the parameters are given in the next section.

Customising the Qualification Parameters

The parameters used for grading the KryoFile are contained in the **DeltaTQA.ini** file that must be installed in the same folder as DeltaTv6.exe. This is a simple text file that you can modify using a simple text editor such as Notepad to allow you to grade your own runs. The format of the file contains a qualification section and up to 99 freezer type sections.

The qualification section begins with the line

```
[Qualifications]
```

This contains a `Count` entry defining how many freezer types are contained within the file and then a keyword entry for each freezer type to identify its own corresponding section. Each type is defined with an entry `Typexx=keyword` where x is the number of the entry; and keyword is a unique name to identify the freezer type; the number must have a leading 0 if less than 10 and the keyword can be any unique name. For example if the file contained two freezer types the qualifications section might look like:

```
[Qualifications]
Count=2
Type01=MRV
Type02=750
```

For each freezer type defined in the qualifications section there must exist an associated freezer type section. The freezer type sections begin with the keyword defined in the qualifications section, enclosed in square brackets. So for the example above the ini file would look like the following (note the contents of the freezer type sections are not shown).

```
[Qualifications]
Count=2
Type01=MRV
Type02=750

[MRV]
.
.
[750]
.
.
```

The first entry in each section is the description. This is the text that appears in the dropdown list when selecting the parameters in the **Qualification dialog** in DeltaT.

For example:
`Description=MRV Freezer System`

The next entries in the Freezer type section are the parameters themselves. For each parameter there are two possible probes (chamber and sample) and three possible values, factory, installation and operation. The three values are used to determine the grade of the unit.

These entry names are all entered in the following format:

`Name&$` where & is the probe (0 = chamber, 1 = sample) and $ is the setting, 1 = factory, 2 = installation and 3 = operation.

So for an entry for the **95 limit** which has an entry name of Conf95, there would be six entries:

```
Conf9501
Conf9502
Conf9503
Conf9511
Conf9512
Conf9513
```

All of the entries which must be defined in each freezer section are shown in the table below. NB. all entries are in 1/100ths of a degree Centigrade so a value of 200 is equivalent to 2.0 C.

Entry name	Parameter	Description
Conf95&$	95% limit	mean + 1.96 * sqrt(variance)
RollConf95&$	Rolling 95% limit	Maximum 95% limit calculated over any 5 minute period.
StableConf95&$	Stable 95% limit	Same as the 95% limit but ignoring any points less than 2 minutes after a rate change of more than 1 C/minute.
MinError&$	Min error.	Minimum recorded error.
MaxError&$	Max error.	Maximum recorded error.
MinStableError&$	Min stable error.	Minimum error but ignoring any points less than 2 minutes after a rate change of more than 1 C/minute.
MaxStableError&$	Max stable error.	Maximum error but ignoring any points less than 2 minutes after a rate change of more than 1 C/minute.
MaxFirstError&$	Max. first sig. error	Maximum error that occurs within two minutes of the first rate change of more than 1 C / minute.
MaxAbsOffsetHold&$ MaxAbsPercentHold&$	Hold error C	These two parameters are combined to calculate the limits for the chamber and sample errors on a hold . The hold error is calculated as the mean over a 2 minute period from the centre of the hold. The maximum permissible error is calculated as `MaxAbsOffsetHold&$ + |T| * MaxAbsPercentHold&$ / 10000` where |T| is the magnitude of the current temperature in degrees C. This allows a wider error band to be defined when the hold temperature is further away from 0 C.
MaxCalError	Calibration error.	This parameter is not used in the Qualification mode but is used in the Calibration. It should always be set to 200 for MRV and PID Engine system. There is only one entry per section. i.e no &$ parameters

Note that the sample trace can show very large errors on fast ramps due to the time lag for the sample as it follows the chamber. In many situations it is not appropriate to use the sample trace when analysing the KryoFile. In these situations the sample parameter should be set to 99.99 to identify that it is not in use and to prevent it skewing the results.

When calculating the freezer grade, the program uses linear interpolation between the three parameters, factory, installation and operation . An example is shown below for the `Conf95&$` parameters