

Provided as a resource for laboratories performing thermometry calibrations on 23 July 2021 by the North Carolina Metrology Laboratory. Changes made to this document after this date were not authorized nor approved by North Carolina metrology staff.

1 Introduction

1.1 Previous versions of this procedure were known as SOP 25a, Calibration of Platinum Resistance Thermometers (PRTs) and Thermistors.

This procedure details the method to test and calculate coefficients for PRTs and thermistors using Fluke 9938 “MET/TEMP II” software. Test results for the unknown are compared to those of a traceable laboratory standard when both are immersed in a comparison bath at temperatures ranging from -30 °C to 230 °C (Mercury Triple Point to Tin Freezing Point).

1.2 Prerequisites

- 1.2.1 Valid calibration certificates with appropriate values and uncertainties must be available for all the standards used in the calibration. All standards must have demonstrated metrological traceability to the international system of units (SI), which may be to the SI, through a National Metrology Institute such as NIST.
- 1.2.2 Standards must be evaluated to ensure that standard uncertainties for the intended level of calibration are sufficiently small. Reference standards should not be used to routinely calibrate customer standards using this procedure and should be reserved for the use of calibrating working standards only.
- 1.2.3 Verify that all equipment that is used is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart.
- 1.2.4 Verify that the operator is experienced in temperature measurement techniques, with a special emphasis on using the Met/Temp II software and developing ITS-90 compliant calibration data.
- 1.2.5 Laboratory facilities must comply with the following minimum conditions to meet the expected uncertainty possible with this procedure and to comply with the manufacturer’s operating conditions.

Table 1. Environmental conditions.

Temperature Requirements During a Calibration	Relative Humidity (%)
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Temperature Requirements During a Calibration	Relative Humidity (%)
Lower and upper limits: 18 °C to 23 °C Maximum changes: ± 5 °C / 12 h and ± 3 °C / h	40 to 60 ± 10 / 4 h

1.3 Safety

Review all applicable JSAs prior to performing this procedure. The danger of severe burns and freezing should be minimized to the fullest extent possible.

2 Methodology

2.1 Scope, Precision, Accuracy

This procedure is based upon the procedures demonstrated and described in the "Precision Thermometry Workshop" conducted by the NIST Temperature Group (March 2001). Multiple thermometers may be calibrated at the same time using this procedure. The procedure consists of an examination of the test probe(s), preparation of comparison baths, configuration of both hardware and software, calculation of coefficients, and measurement assurance.

This method is applicable to all resistance-based thermometry devices. The achievable precision using this procedure is appropriate. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the precision of the intercomparison.

2.2 Summary

The test probe(s), a standard, and a check standard are immersed in a temperature comparison bath and allowed to come to thermal equilibrium. A Fluke Superthermometer is used to read input of all probes. Fluke "MET/TEMP II" software is used to gather the test probes' resistances at various temperatures controlled by a working standard Platinum Resistance Thermometer (PRT). The "MET/TEMP II" software also calculates coefficients for the probes. Coefficients for the unknown standard derived from the results of the procedure are entered into the test device display and the indication is compared to that of the standard in the temperature bath for verification purposes.

The check standard monitors the precision and accuracy of the process. If the check standard detects an out of control situation, the metrologist attempts to

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discover the source, corrects it if possible, and repeats the calibration if necessary.

2.3 Apparatus/Equipment

- 2.3.1 Two standard temperature indicating devices, one used as the standard, the other used as the check standard, both with calibration certificates traceable to the SI and having an expanded uncertainty appropriate for the test thermometer's temperature range. Appropriate standards typically include a platinum resistance thermometer.
- 2.3.2 A comparison bath(s) with a standard deviation suitable to perform this procedure.
- 2.3.3 Sufficient clamping fixtures to hold the thermometers;
- 2.3.4 Calibrated environmental equipment to monitor temperature and relative humidity laboratory conditions;
- 2.3.5 A Superthermometer, Multiplexer, Met/Temp II software and laptop, and a Smart Switch for RS-232 communications during the calibration process.

2.4 Procedure – Configuration Steps

- 2.4.1 Check the probe carefully for foreign material and wear, i.e., nicks, dirt, frayed wires prior to beginning the Procedure process.
- 2.4.2 To prepare a test, configure the communications port, reference probe and module, scanner, heat sources and equipment as described in the configuration menu section of the Fluke 9938 "MET/TEMP II" User's Guide. Step-by-step instructions follow below.

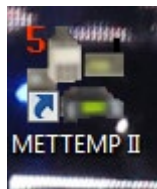


Fig. 1

Open a new test by clicking on the icon shown in Figure 1.

- 2.4.3 The dropdown menu will appear at the top of the screen as shown in Figure 2 and will provide the following menu options.

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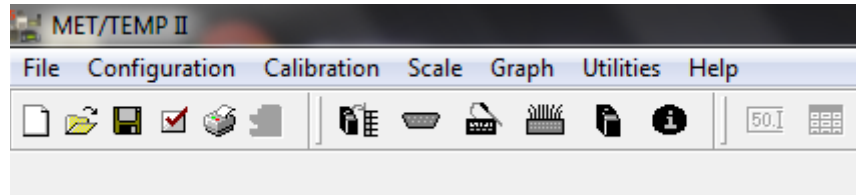


Fig. 2

- 2.4.4 Select the File dropdown menu and click on the Open option. The Configuration screen will be similar to the one shown in Figure 3, but since new configurations are always able to be added or removed, these exact configurations may not be present.

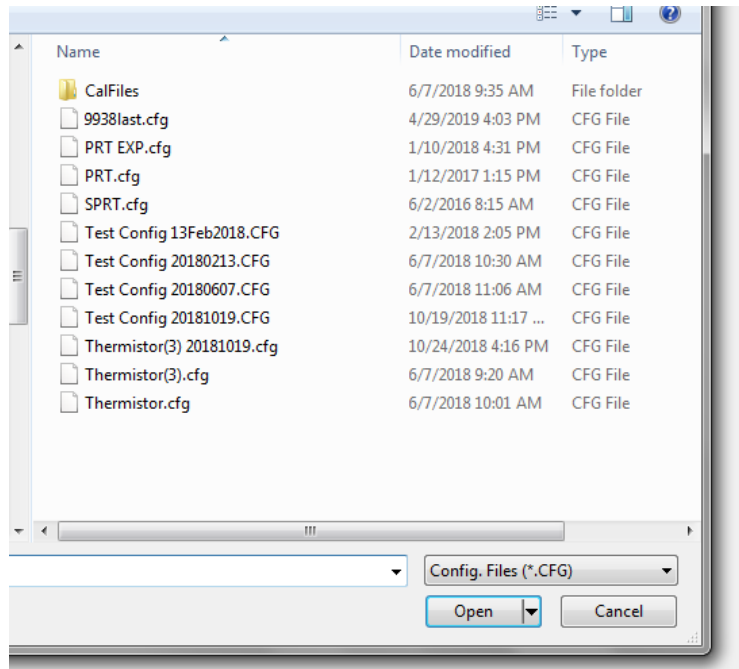


Fig. 3

- 2.4.5 Once a configuration is selected, a screen titled Equipment Info will automatically open. A total of six fields (Steps) are required to be filled in with appropriate information for a calibration configuration to be valid. Any steps that are in blue indicate that the step is fulfilled and does not require any further data input; likewise, any step that is red indicates an absence of required information for the calibration. Steps five and six will always appear red since the software intentionally blocks the carrying over of data in these steps when a configuration is saved. See the Equipment Info screen as shown in Figure 4.

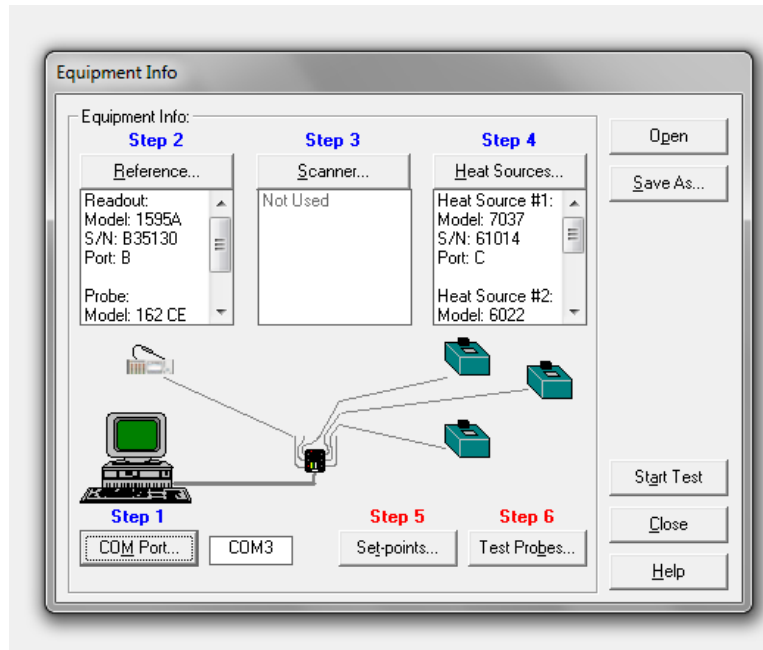


Fig. 4

2.4.6 Click on Step 5, Set-points. A Set-point Configuration will appear as shown in Figure 5. In order to meet ITS-90 calculation requirements, the following parameters must be followed:

- 1) A minimum of five set points is required for calculations
- 2) One of the set points must be 0 °C
- 3) Two to ten points must be selected between the Mercury Triple Point (-38.8344 °C) and the Gallium Melting Point (29.7646 °C)
- 4) Two to ten points must be selected between the Water Triple Point (0 °C) and the Tin Freezing Melting Point (231.928 °C)

For a reference that describes the parameters above, see the Fixed Points and Ranges of the ITS-90 graph.

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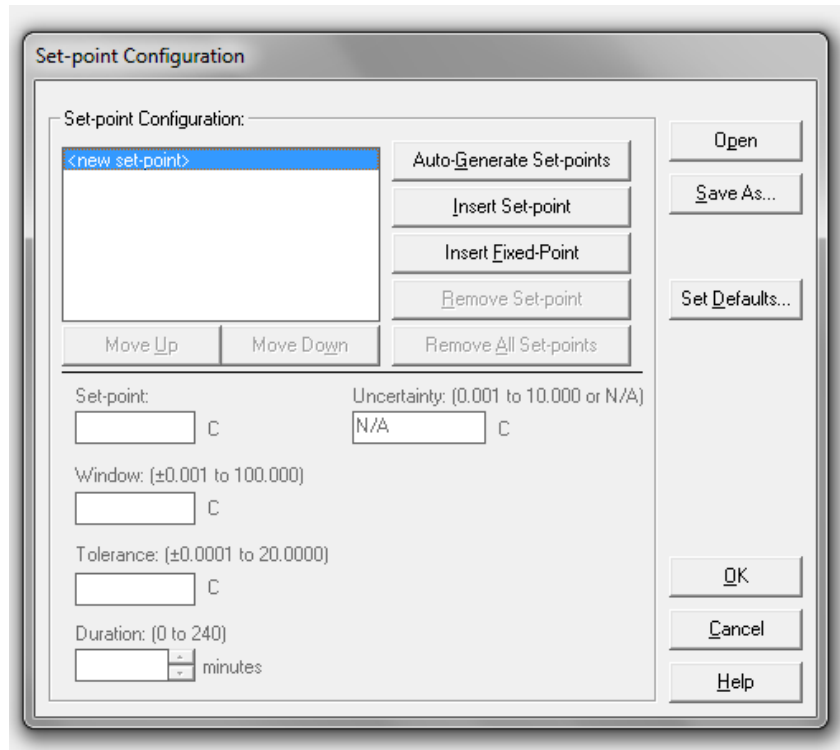


Fig. 5

- 2.4.7 Automatic generation of set-points is the simplest means to complete this portion of the configuration. If automatic generation is not used, then the “Insert Set-point” radio button can be selected to manually enter each set-point. In the example shown in Figure 6, six set-points have been selected with a range of 20 °C between each set-point and with a starting set-point of 0 °C. This example would be a typical setup for any thermistor. The “Prompt for heat source conflicts” option is useful but not required, as heat source “bath” selection can occur at later steps.

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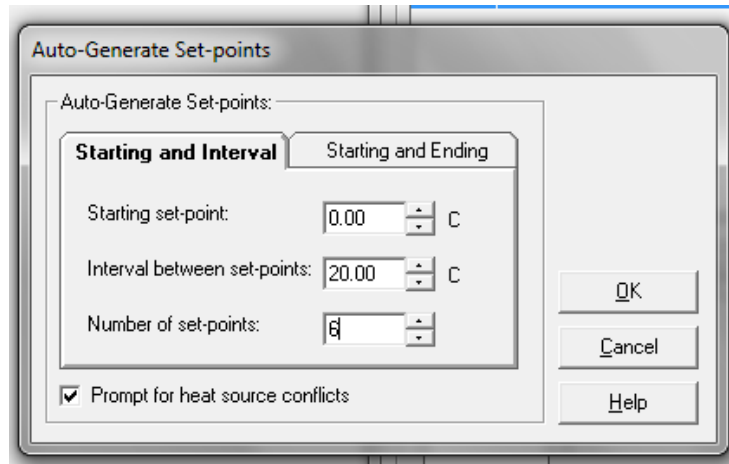


Fig. 6

2.4.8 After the previous step is complete and “OK” is selected, a new window will appear as shown in Figure 7. This window is similar to the one shown in section 2.4.6 except the fields are mostly complete. The Window, Tolerance, Duration, and Uncertainty should populate automatically based upon default settings. The Uncertainty value should be verified to be accurate based upon the latest published laboratory scope. The software has a lower limit of 10 mK for the uncertainty, so reporting anything lower than this is not possible. The “Heat Source to use” option will always default to the first available bath. This setting can be verified as correct or needing to be changed by clicking on “OK” and going back to the screen shown in step 2.4.5. Step 4, Heat Sources, will reveal the order of the baths established by the default settings. Each set-point must be individually clicked on to verify that the settings are correct.

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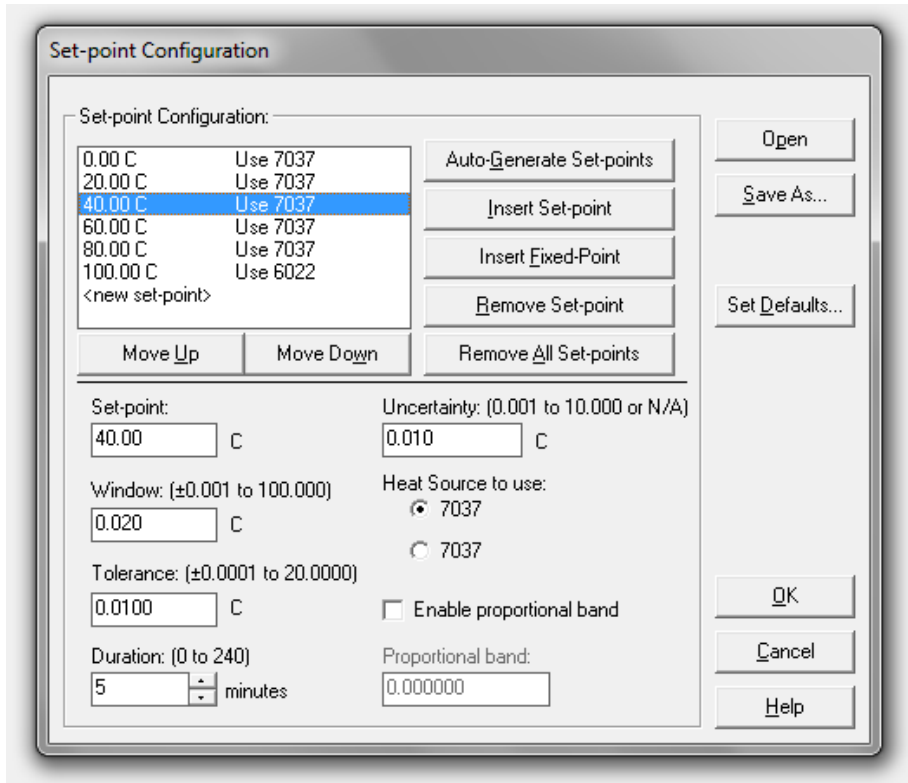


Fig. 7

- 2.4.9 As shown in section 2.4.8, selecting the 40.00 °C set-point will allow the insertion of the 25.00 °C set-point as required for a typical thermistor calibration. This is done by clicking on the “Insert Set-point” radio button and manually filling in the required information. In addition, the “Enable proportional band” should be enabled and the values entered according to the recommendations published in the bath user’s manual. This completed step is shown in Figure 8.

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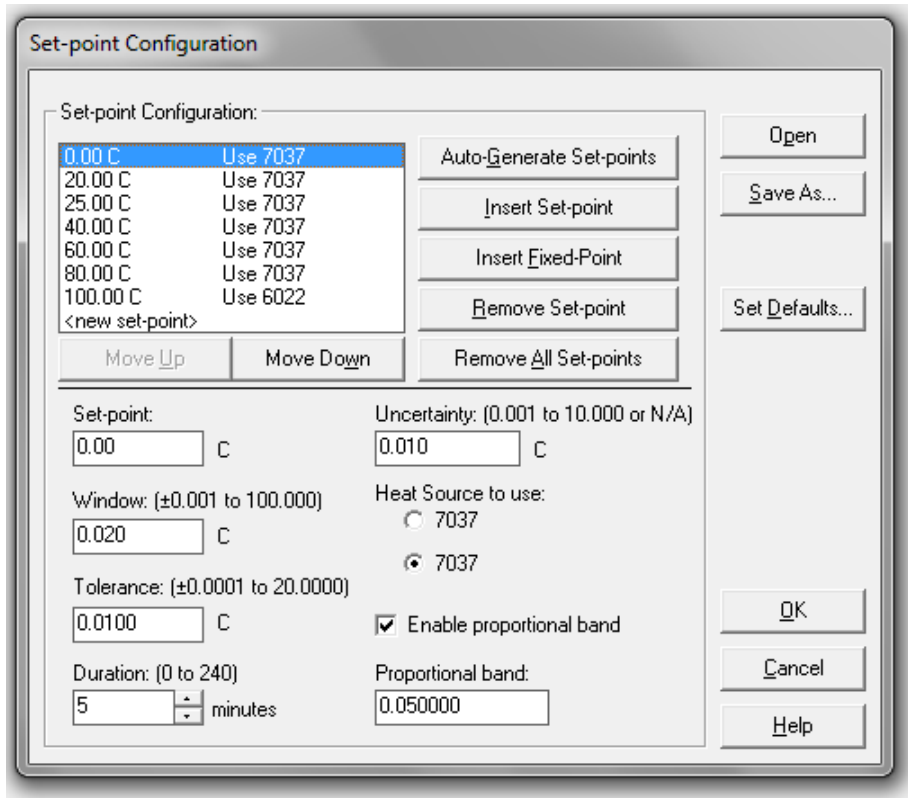


Fig. 8

- 2.4.10 Once the previous step is complete and “OK” has been selected, the final step of the configuration process is to complete step 6, Test Probes. As shown in Figure 9, select the appropriate channel based upon the desired channel placement of the unknown standard. Note that this selection must be on the Multiplexer (channels five through fourteen), as the first four channels are reserved for the standard, check standard, and connection from the Superthermometer to the Multiplexer. If the unknown standard has been previously calibrated, all of the remaining information fields will populate automatically. If the unknown standard is new, then all of the fields must be completed.

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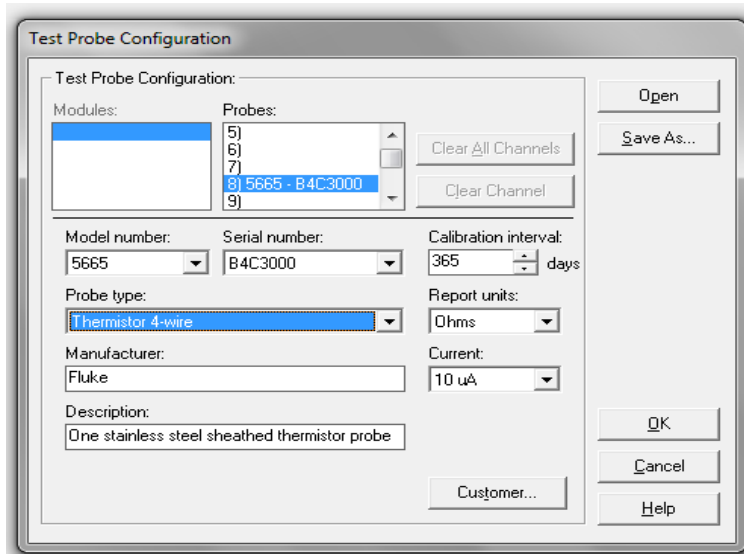


Fig. 9

2.4.11 Select the Customer radio button and a screen as shown in Figure 10 will appear. In most cases the dropdown option Recalibration will apply, unless this is a new unknown standard with no previous history available. The Customer order ID should represent the customer Purchase Order. The Customer name can be selected from the dropdown menu if they are a returning customer.

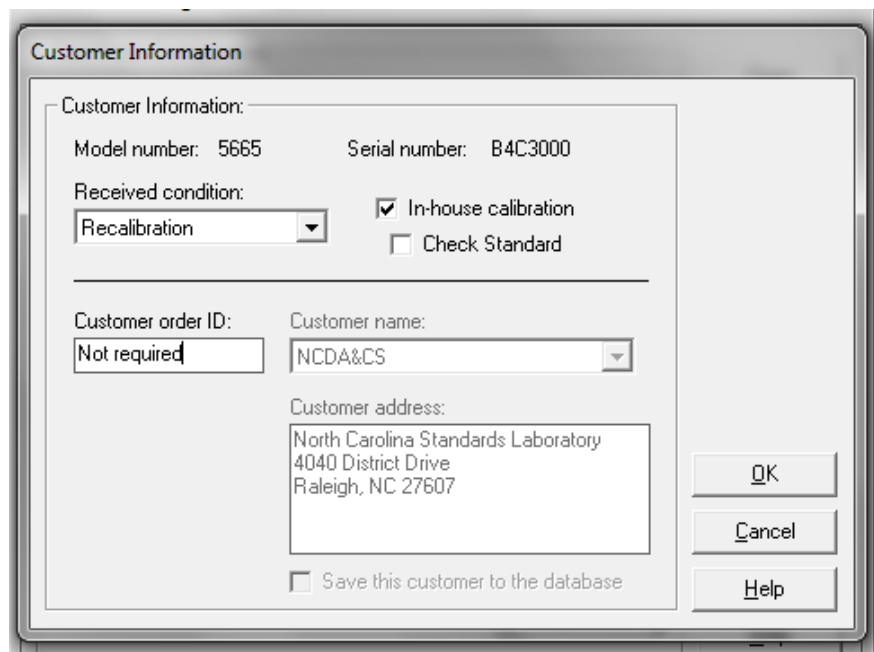


Fig. 10

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2.4.12 Select “OK” and then “OK” again on the screen as shown in Figure 9. This should take you to the screen as shown in Figure 4. Select the “Start Test” radio button to begin the test sequence.

2.5 Procedure – Calibration Sequence

2.5.1 The first confirmation screen will look like the example in Figure 11. The number of readings can be modified, if desired, in the programming configuration menu.

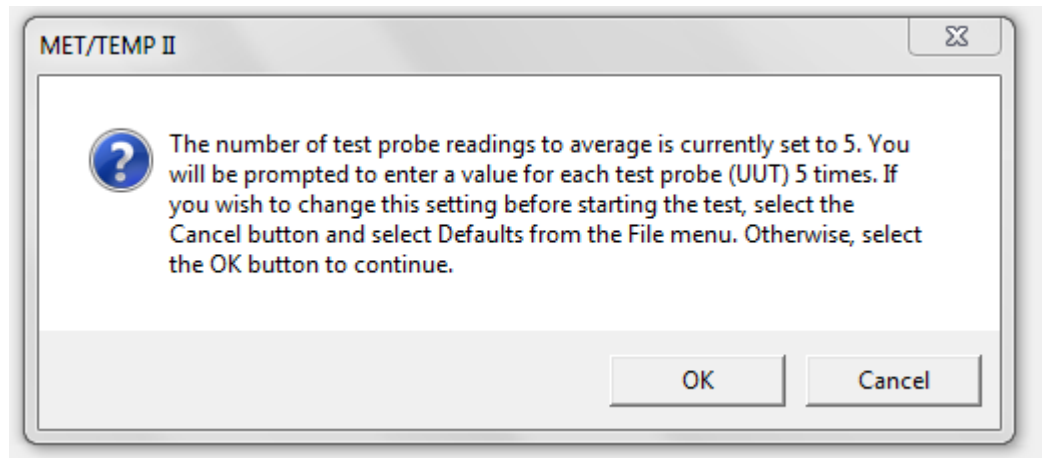


Fig. 11

2.5.2 The next confirmation screen is a verification of the data to be presented on the ITS-90 Report of Calibration. The name of the Metrologist, designation of a Full range or Partial range calibration, Test number, and ambient (environmental) conditions can be entered here.

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Test Information

Test Information:

Full range calibration Partial range calibration

Name of person performing test: Test date:

Test procedure: Preheat heat sources

Reference/UUT readings resolution: Example: -123.45679

Generate test number automatically

Test number:

Enter the ambient conditions:

Temperature: C F Humidity (%):

Fig. 12

- 2.5.3 Once “OK” has been selected on the previous screen, the calibration will begin. The first question in this sequence involves the unit readout of the standard and the unknown standard. The standard must be set to °C and the unknown standard must be set to ohms. Modify the unit readout if needed.

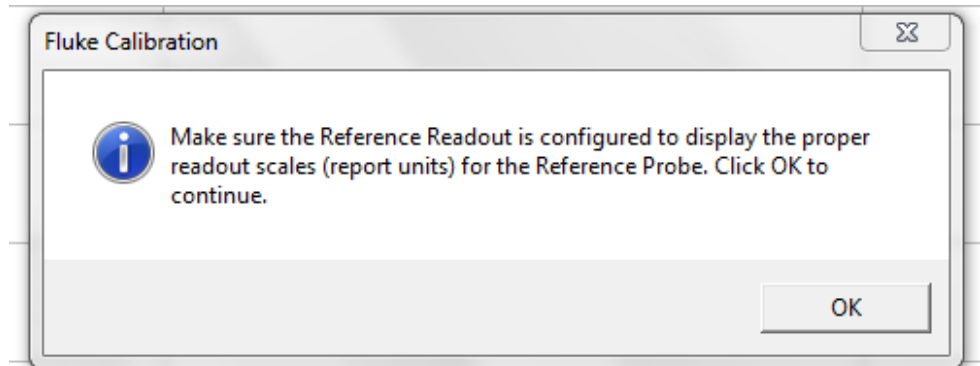


Fig. 13

- 2.5.4 Once “OK” has been selected on the previous screen, the second question in this sequence involves the confirmation that the probes are placed in the correct bath and ready to begin the calibration process.

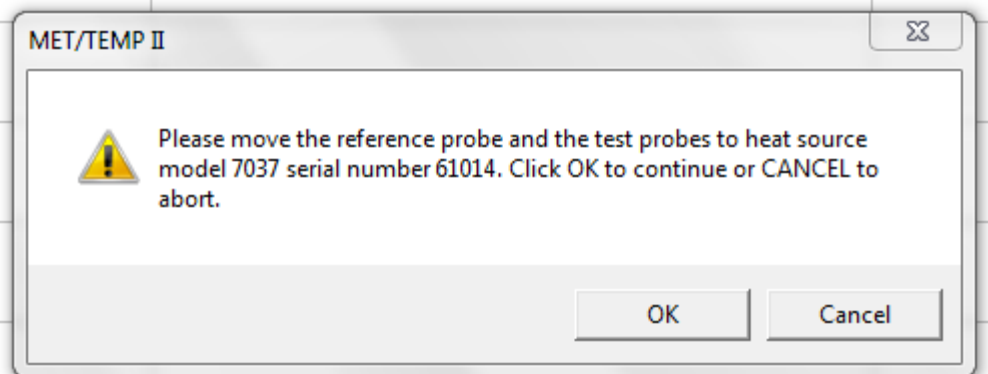


Fig. 14

- 2.5.5 Once the calibration begins, the process is automatic. When the program has determined stabilization has occurred (as defined in section 2.4.8) a prompt as shown in Figure 15 will appear. Data entries are permanent once the “OK” radio button is pressed.

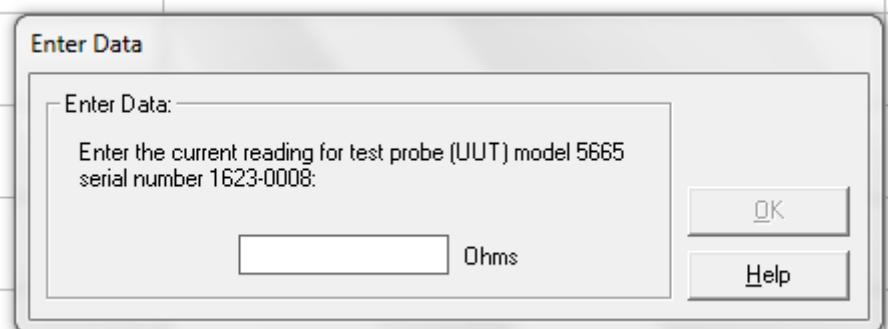


Fig. 15

- 2.5.6 Continue the calibration process and follow the prompts during the process. Once all of the data has been collected, a notification will appear stating “The test has been completed. Select Close Test from the File menu”.

2.6 Procedure – Calculating the Coefficients

- 2.6.1 Select the Utilities dropdown menu and scroll down to the Coefficients and Tables selection. Once this is clicked a new configuration screen will appear with new dropdown menu options.
- 2.6.2 Either select Coefficients from the dropdown menu or select the icon shortcuts. Follow these guidelines for determining coefficient calculation.

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- 1) Platinum Probe uses ITS-90 calculations and Subranges 5 and 9
- 2) Thermistors use Polynomial 3rd Order
- 3) The laboratory does not currently have the capabilities to produce coefficients for thermocouples

2.6.3 After clicking “OK” the next screen will direct the user to select a test number for calculation of coefficients as shown in Figure 16.

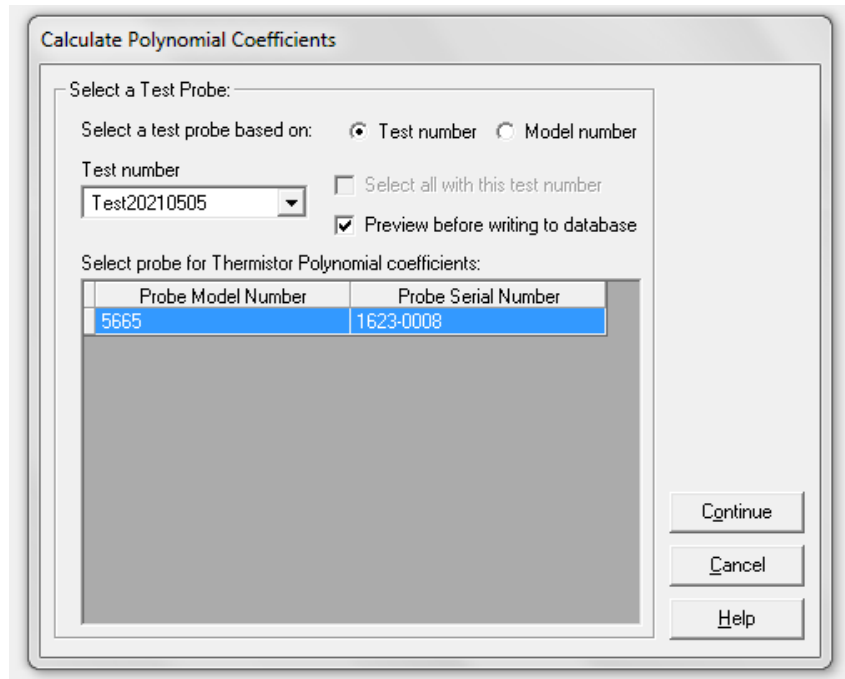


Fig. 16

2.6.4 Click continue. The next screen will appear as the one shown in Figure 17. Press the “Select All” radio button and then press the “Calculate” radio button. After reviewing the data, experienced users may elect to deselect one reading for the calibration curve to fit the model. This should only be done if one reading is out of line with the other readings and all of the other points fit the curve. Recalibration is a preferred option rather than this method in order to substantiate accurate readings.

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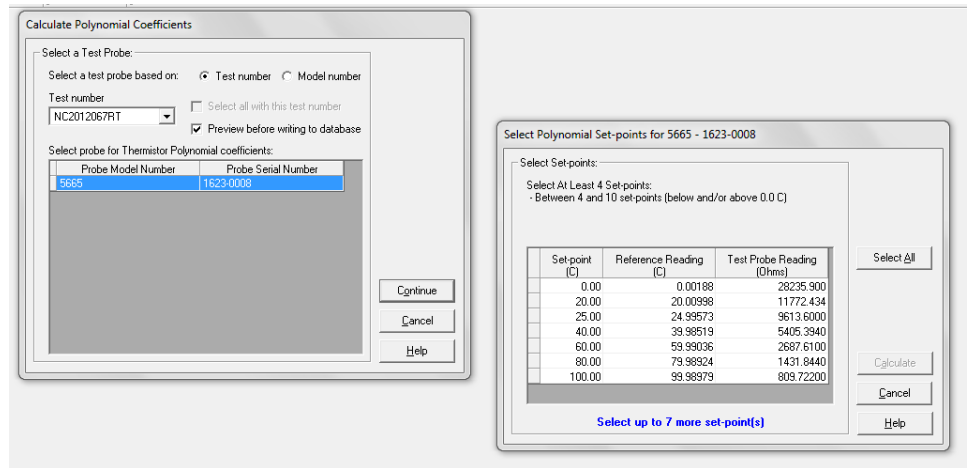


Fig. 17

2.6.5 The next screen will appear as the one shown in Figure 18. Click the “Save” radio button.

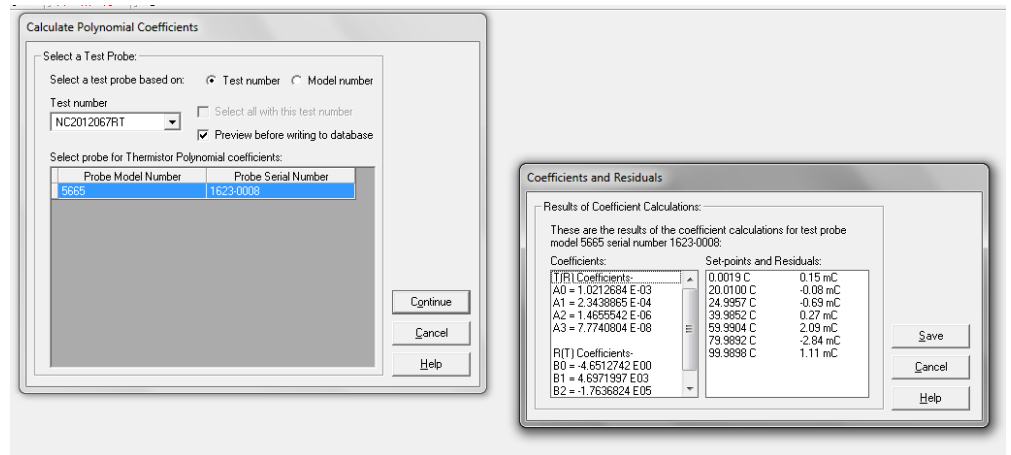


Fig. 18

2.6.6 The software will direct back to the main screen. From the File dropdown menu, select Export Coefficients. Select the correct test number, select the “Export” radio button, and then a new Save As screen will appear. Save the text file (EXPORT.TXT). Close this step by clicking on the “OK” radio button.

2.6.7 The software will then redirect back to the main screen. Follow a similar sequence as in section 2.6.6, except select Export Data. Highlight the test number and select the “Export” radio button. Save the file as in step 2.6.6. Note that even though the text files are named the same, the

data within these files is processed separately, and both text files are required for proper printing of the ITS-90 report.

- 2.6.8 The software will then redirect back to the main screen. Under the File dropdown menu, select Print Reports and Tables. Select the proper test number and then click on the “Print” radio button.
- 2.6.9 The first page of the Report of Calibration will display. Clicking on the printer icon at the top of the screen will print the first page. To print the subsequent pages, click on the Close icon at the top of the screen.
- 2.6.10 When all pages of the report have printed, click on the Close icon again and the program will return to the previous Print Reports and Tables screen. Select the “Cancel” radio button and then the Close option under the File dropdown menu. The Met/Temp II program can be closed by selecting the red X in the upper right corner.
- 2.6.11 In order to determine if the calibration was successful and accurate coefficients have been produced, all of the resistance values should be entered and plotted on that standard unknown’s history graph. Plotted points and uncertainties should overlap with previous values. If a particular data point does not overlap and it is outside the range of normal use of the customer, then the calibration can stand as is if it is limited to one such data point scenario. For more than one point as this, the unknown standard should be recalibrated.
- 2.6.12 If consecutive calibrations of the unknown standard reveal a shift in the data point values and these two calibrations agree with uncertainty overlap, then the new data point values will be considered the new resistance and T(R) values. If more than two calibrations are required to meet the history stability of the standard unknown, then the customer should consider replacing the artifact as long term stability is questionable.

2.7 Satisfaction Evaluation

An evaluation may be performed using the following definition: ITS-90 defines an instrument as satisfactory if it means the criteria that $W(302.914\text{ K}) \geq 1.11807$ or $W(234.3156\text{ K}) \leq 0.844235$. The value of W is calculated from the ITS-90 portion of the calibration certificate.

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3 Calculations

3.1 Since the Met/Temp II software is used for this procedure, no specific calculations or use of equations are performed outside the program, with the sole exception being the evaluation used for defining satisfactory instruments. Evaluation using this equation after the calibration is not a requirement but can be a useful tool.

3.2 W can be calculated using the following equation:

$$W = (((\Omega_{\text{fpc1}} - \Omega_{\text{fpc2}}) * \text{fixed point cell value from nominal}) + \Omega_{\text{fpc2}}) / \text{RTPW resistance}$$

Eqn. (1)

Where, Ω_{fpc1} = higher value of resistance at the fixed point cell
 Ω_{fpc2} = lower value of resistance at the fixed point cell
 Ω_{fpcnom} = resistance value of fixed point cell
RTPW = resistance value at the Triple Point of Water provided in ITS-90 section of calibration certificate

Example: $W(302.914 \text{ K}) = (((28.366277 \Omega - 28.356253 \Omega) * 0.7640 \text{ }^\circ\text{C}) + 28.356253 \Omega) / 25.366428 \Omega$

4 Measurement Assurance

4.1 Duplicate the process with suitable check standards for each nominal that is within the range of stem correction or unknown standard data gathering. See [NISTIR 6969 SOP 9, Control Charts for Calibration of Mass Standards](#) for general guidance on control charts; while these specifically reference mass control charts, many of the evaluations apply to the thermometry measurement assurance program.

4.2 Evaluate the value against the expected limits and plot the check standard value on the control chart to monitor changes over time.

All values must be entered in the control chart, even if failing this statistic, unless a mistake (e.g., typographical error) is identified and corrected, to ensure the variability obtained for the process is not unduly reduced over time. The observed value of the check standard is compared to the accepted mean value of the check standard and divided by the standard deviation for the check standard observations over time.

A t-test may be incorporated to check the observed value of the check standard against the accepted value using the following equation and a 95 % confidence

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level. The t-statistic is calculated for stability analysis. This equation monitors stability over time but should not be used to assess for bias. A calculated t-value less than two is within the warning limits of the process. A calculated t-value between two and three represents a value between the warning limits and control/action limits. A calculated t-value exceeding three represents a value outside of the control/action limits and suitable action must be taken. Calculated values of the t-statistic may also be monitored over time to determine the presence of drift.

$$t = \frac{(S_c - \overline{S_c})}{s_p} \quad \text{Eqn. (2)}$$

- 4.3 Check standard measurement results obtained over time are used to calculate the standard deviation of the measurement process, s_p .
- 4.4 The mean value of the check standard over time is also compared to an appropriate reference value of the check standard with respect to their applicable expanded uncertainties to evaluate bias and drift over time. Excessive drift or bias must be investigated and followed with suitable corrective action. See [SOP 9](#), Section 3.5.3 and Section 8.16 for assessment methodology.
- 4.5 If check standards were already checked on a given day and found to be in control, additional evaluations may be conducted, but are not required.

5 Assignment of Uncertainty

The limits of expanded uncertainty, U , include estimates of the standard uncertainty of the thermometry standards used, u_s , estimates of the standard deviation of the measurement process, s_p , and estimates of the effect of other components associated with this procedure, u_o . These estimates should be combined using the root-sum-squared method (RSS), and the expanded uncertainty, U , reported with a coverage factor of two ($k = 2$), to give an approximate 95 % level of confidence. See [SOP 29](#) for the complete standard operating procedure for calculating the uncertainty.

- 5.1 The expanded uncertainty for the standard, U , is obtained from the calibration certificate. The combined standard uncertainty, u_c , is used and not the expanded uncertainty, U , therefore the reported uncertainty for the standard will usually be divided by the coverage factor k . Where the coverage factor or confidence interval is not given, the laboratory should either contact the calibration provider to obtain the correct divisor or use a value of $k = 2$,

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assuming that the expanded uncertainty was reported with an approximate 95 % confidence interval (95.45 %).

- 5.2 Standard deviation of the measurement process from control chart performance (See [SOP No. 9.](#)) The value for s_p is obtained from the control chart data for check standards using direct comparison measurements. If the standard deviation of the measurement process from the control chart is less than the resolution of the Superthermometer (less than 0.00001 °C), the laboratory may round up to the value of the Superthermometer division or use the larger of the standard deviation of the process or the following estimate for repeatability is used to represent the standard deviation of the process:

$$s_p = \frac{d}{\sqrt{3}} \approx 0.6d \quad \text{Eqn. (3)}$$

- 5.3 Uncertainty associated with propagation of Triple Point of Water (TPW) cell. This is a fixed value that is established during the manufacturing and calibration of the cell.
- 5.4 Uncertainty associated with stability of the 1595A reference resistor (Superthermometer) based upon bath being used at the time of calibration of unknown standards. This is a fixed value provided by the manufacturer.
- 5.5 Uncertainty associated with the bias of the measurement process from the TPW cell. This uncertainty component is a comparison of the thermometry standard against an intrinsic standard.
- 5.6 Uncertainty associated with bath stability. This component is a fixed value obtained from the manufacturer.
- 5.7 Uncertainty associated with non-uniformity temperature gradients. This component is either the value obtained from the manufacturer or the laboratory observed value derived from internal studies, whichever is higher. Actual laboratory data may reveal a degraded stability less than claimed by the manufacturer, depending on bath.
- 5.8 Uncertainty associated with bias that is observed in the check standards as determined through analysis of the control charts. When bias is observed in the control charts, it must be assessed according to the equations provided in SOP 29 and may be incorporated as an uncorrected systematic error using the equations in SOP 29.

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5.9 Example components to be considered for an uncertainty budget table are shown in the following table.

Table 2. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standards (5.1)	u_s	Calibration certificate	Normal divided by coverage factor
Accepted standard deviation of the process (5.2)	s_p	Control chart, standard deviation chart	Normal or estimated rectangular if actual s_p is less than Superthermometer display resolution
Uncertainty of the propagation of the TPW Cell (5.3)	u_o	Calibration values from ITS-90 report	Normal divided by coverage factor
Uncertainty of the stability of the 1595A reference resistor (Superthermometer) (5.4)	u_o	Manufacturer supplied value in manual	Rectangular
Uncertainty associated with bias of the measurement process from the TPW cell (5.5)	u_o	Control chart	Rectangular (also see SOP 29)
Uncertainty associated with bath stability (5.6)	u_o	Manufacturer	Rectangular
Uncertainty associated with non-uniformity temperature gradients (5.7)	u_o	Manufacturer or from experimental data	Rectangular
Uncertainty associated with bias (5.8)	u_d	Control chart, proficiency tests	See SOP 29

5.10 Draft a suitable uncertainty statement for the certificate. For example:

The uncertainty reported is the root sum square of the standard uncertainty of the standard, the standard deviation of the process, and the uncertainty associated with other components corrections, multiplied by a coverage factor of 2 ($k = 2$) for an approximate 95 percent confidence interval.

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NOTE: Where inadequate degrees of freedom are available, k , is determined using the appropriate degrees of freedom and the 95.45 % column in the table from [Appendix A of NISTIR 6969, SOP 29](#).

6 Certificate

6.1 Report results as described in [SOP No. 1, Preparation of Calibration Certificates](#). Report the T(R) coefficient values, environmental conditions during the calibrations, and calculated expanded uncertainties with coverage factor(s).

6.2 Conformity assessment.

Compliance to applicable tolerances is not considered in this type of thermometry calibration as the measurements are not performed to a tolerance. Acceptability for use is defined in section 3.2.

6.3 In cases where a system is presented for calibration, a statement must be included on the certificate noting if the calibration was performed on the individual components of the system or if the system was calibrated as one whole entity.

6.4 A completed calibration packet includes the calibration certificate, the ITS-90 Report, history graphs of the points of calibration, and an uncertainty budget.

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