NPL

NPL REPORT ENG 38

User's manual for NPL designed and supplied self-validating thermocouples, and the associated breadboard

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AUGUST 2012

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NOTES

This user's manual is adapted from the original protect-commercial report: TN5a: User's Manual, of the project with ESA "High temperature sensors with self-validating capabilities" with reference numbers:

NPL reference:	NPL Report EM (RES) 052/TN5
ESA contract number:	4000101740/10/NL/CP

ISSN: 1754-2987

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Approved on behalf of NPLML by Leon Rogers, Group Leader, Temperature & Humidity.

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ESA STUDY CONTRACT REPORT

ESA Contract No:	SUBJECT:	CONTRACTOR:
4000101740/10/NL/CP	**High temperature sensors with self-calibrating capabilities: User's manual (public)**	National Physical Laboratory, UK
* ESA CR( ) No: N/A	No. of Volumes: N/A This is volume No: N/A	CONTRACTOR'S REFERENCE: N/A

ABSTRACT:

User's manual for NPL designed and supplied self-validating thermocouples, and the associated breadboard

The work described in this report was done under ESA Contract. Responsibility for the contents resides in the author or organisation that prepared it.

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** NAME OF ESA STUDY MANAGER:	** ESA BUDGET HEADING:
DIV: DIRECTORATE:	

Signed on behalf of:	ESA	National Physical Laboratory, UK
Signature		
Name Position Date		Graham Machin Head of Temperature Standards 16 Oct '12

1 INTRODUCTION

This document is the user's manual for the self-validating thermocouples designed and supplied by NPL to ESA-estec (during the development project, contract number 4000101740/10/NL/CP). These sensors and associated equipment for measuring and analysing the thermocouple output form the supplied breadboard.

Implementation of self-validating thermocouples *in-situ* has been shown to be essential for improved user confidence when using Type C thermocouples above 1300 °C, through reliable measurement validation.

The self-validating thermocouple consists of two main parts: a W-Re thermocouple and a high-temperature fixed point (HTFP) cell. It is the reliable observation of the melting transition of this HTFP which enables validation of the temperature measurement *in-situ*. For each of the sensors, the calibrated melting temperature of the HTFP cell was traceably determined by radiation thermometry.

The user's manual is made up of the following sections:

- Description of breadboard
- Breadboard assembly and use instructions
    - Self-validating thermocouple assembly
    - Measuring equipment assembly
    - PC and software use instructions
    - Thermocouple measuring software: "TC Scanner"
    - Melting temperature determination software: "Best Fit"

It is recommended that the user read the associated final project report (public document) before implementing these sensors for the first time. Also note that in the original project this sensor was referred to as a "self-calibrating" thermocouple.

2 DESCRIPTION OF THE BREADBOARD

Five self-validating thermocouples have been provided:

- 5 W-Re thermocouples
- 5 Miniature crucibles containing high-temperature fixed point ingot(s) ("*the HTFP cells*")

The first four HTFP cells contain a single ingot of a eutectic metal-carbon alloy. One HTFP cell of each of the following is supplied:

Co-C (1323.73 °C ± 0.33 °C) **Pt-C** (1737.53 °C ± 0.86 °C)
Ru-C (1952.80 °C ± 1.03 °C) **Ir-C** (2289.76 °C ± 1.60 °C)

The fifth HTFP cell supplied contains two ingots:

Pt-C & Ru-C (1739.36 °C ± 1.19 °C and 1955.58 °C ± 1.88 °C respectively)

Calibration was determined by radiation thermometry at NPL. The procedure was completed according to ISO 17025 accredited procedures and results in a melting temperature and uncertainty traceable to NPL's realisation of the International Temperature Scale of 1990 (ITS-90). Uncertainties are expressed in terms of a 95 % confidence level, coverage factor $k = 2$.

The following items of equipment comprise the associated breadboard:

- 1 W-Re extension cable
- 1 PC with USB ports (Dell Latitude E5420)
- 1 Keithley 2182A digital nanovoltmeter

- 1 National Instruments IEEE 488.1 GPIB interface model NI GPIB-USB-HS
- 1 Hart Scientific 9101 zero point block calibrator (automatic reference temperature unit)
- 1 Foam-walled plastic box for connection of thermoelements to copper wires
- 6 Swagelok SS-2-UT-1-2 Ultra Torr Connector 1/8” OD – 1/8” NPT Male fitting to provide the sealed interface between thermocouple and furnace chamber
- 1 Custom data logging software “TC Scanner”
- 1 Custom software for determination of the point of inflection of the melting curve “Best Fit”
- 1 Carrying case to protect the self-validating thermocouples
- 3 Mains plug adaptors UK to EU

The following items are required to be in house, for implementation of the breadboard (note, these were **not** a deliverable part of the breadboard and must be provided locally):

- 1 High temperature furnace
- 1 High purity argon gas supply

A Webb “Red Devil” RD-G (www.rdwebb.com) high temperature graphite furnace was employed at NPL to realise the temperatures required to complete the breadboard test. The furnace contains a sealed argon gas atmosphere at a pressure of 1 atmosphere to prevent oxidation of the graphite and metal components inside. A schematic diagram showing the furnace and thermocouple arrangement is shown in Figure 1.

The furnace is controlled by a pyrometer which sights the crucible arrangement, either the blackbody or a graphite target. The thermocouple reference junction is maintained at 0 °C with a Peltier-cooled automatic ice point. The emf generated by the thermocouple is measured with a digital voltmeter, and recorded periodically (approx. every 10 s) by a PC. The connection regime is shown in Figure 2.

The fully operational breadboard, as arranged at NPL, is shown in Figure 3.

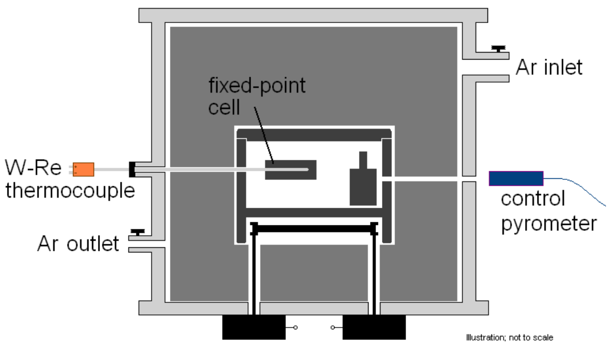

Figure 1: Schematic diagram of thermocouple and fixed-point cell location in furnace for measurements at NPL.

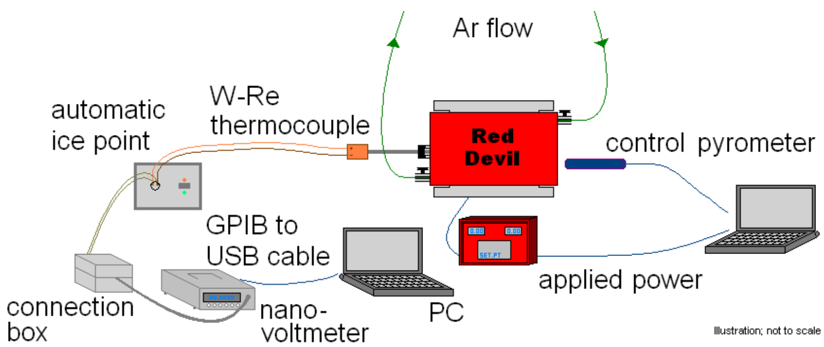

Figure 2: Schematic diagram of connection regime. This represents the ‘breadboard’ for testing the self-validating thermocouples.

Figure 3: Photograph of the 'breadboard' for testing the self-validating thermocouples. Left to right: PC, digital nanovoltmeter (with connection box on top), automatic ice point, high temperature furnace.

3 BREADBOARD ASSEMBLY AND USE INSTRUCTIONS

The breadboard consists of the items described in Section 2.

The Swagelok connectors provide a gas/vacuum tight seal between the furnace and thermocouple. These are provided as part of the breadboard and are suitable for use with a 1/8" OD – 1/8" NPT Female fitting on the furnace – if no such fitting is available, an alternative connector will be required.

It is assumed that all mains electrical equipment will be plugged into the local electrical supply. Mains adapters from UK to EU are supplied for this purpose. Assembly instructions now follow.

3.1 SELF-VALIDATING THERMOCOUPLE ASSEMBLY

The self-validating thermocouples are supplied in a presentation case. Each consists of a Type C thermocouple and a corresponding HTFP cell. <u>It is important to maintain the allocation of one thermocouple to one HTFP cell, to avoid cross-contamination. The thermocouples and cells **must** only be used in an inert atmosphere or under vacuum.</u> Additional Type C thermocouples may be ordered from Omega Engineering, www.omega.co.uk, with the product code: XTA-W5R26-U-125-30-H-Q-17.

The self-validating thermocouple is constructed by first placing the Swagelok Connector onto the thermocouple. This connector is supplied to provide a method of forming a vacuum tight seal into the user's furnace. If an appropriate connection on the user's furnace is not available, a different arrangement will be required.

After placing the thermocouple into the working position in the user's furnace, the HTFP cell should be positioned onto the tip of the thermocouple – by fully inserting the thermocouple tip into the well in the HTFP cell (the *thermowell*). This procedure should be performed with care to avoid breakage of the thermowell. The HTFP cell should also be positioned carefully in the user's furnace to subject it to the most uniform thermal environment possible along its length. As the HTFP cell is constructed from graphite (pure carbon); it requires an inert atmosphere in which to operate. The user must ensure that this atmosphere is maintained. High purity argon gas is recommended, at room pressure.

The self-validating thermocouple arrangement is shown in Figure 4.

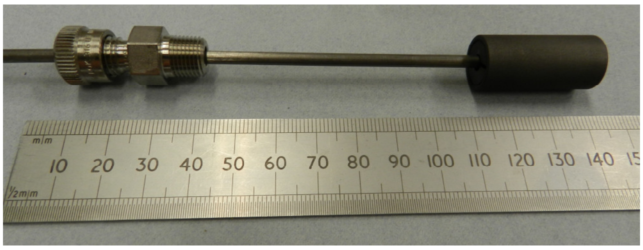

Figure 4: The self-validating thermocouple arrangement, showing the Swagelok adapter fitted to the Type C thermocouple, and fixed-point cell positioned on the thermocouple tip.

The thermocouple extension cable is supplied prepared for use. This unit consists of a red plug-cap connected to the thermocouple extension wires and a cold junction between the thermocouple extension wires and copper wires (positioned in a glass tube). The extension wire unit arrangement is shown in Figure 5 (and is as supplied).

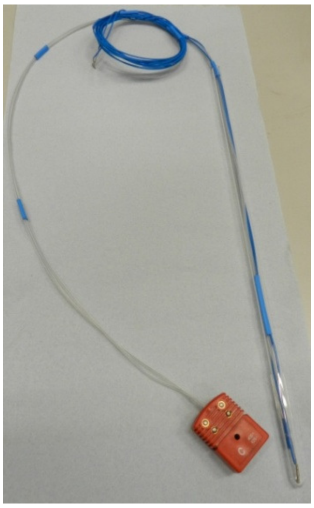

Figure 5: The extension wire unit arrangement (as supplied), showing the red cap and cold junction (in glass tube).

The red cap of the extension cable should be plugged onto the red cap of the self-validating thermocouple. Care should be taken to ensure the plugs are not subject to severe temperature gradients. It is essential that the cold junction remains fully inserted in the glass tube at all times – to maintain the proper temperature at the junction. The glass tube should be placed into one of the wells of the Hart Scientific 9101 zero point block calibrator.

The Hart 9101 device should be switched on, and will take approximately five minutes to reach its working temperature of 0 °C ± 0.02 °C. When ready to use, the "ready" light will illuminate.

3.2 MEASURING EQUIPMENT ASSEMBLY

The Keithley 2182A digital nanovoltmeter should be connected to the PC using the National Instruments IEEE 488.1 GPIB interface cable. Note that the GBIP address of this Keithley 2182A is already set to be "**7**".

The Keithley 2182A should now be switched on. This device should be switched on for a minimum of 8 hours before use to allow the readings to stabilise.

The Keithley 2182A is supplied with an input cable to connect to the thermocouple wires. The plug of the input cable should be inserted into the appropriate socket on the front of the Keithley 2182A. At the other end of this cable, the wires are labelled by colour and are terminated with crescent-shaped connectors. The green and white wires (channel 2) are not used, but should be kept connected (e.g. clipped together). The red and black wires (channel 1) are used for connecting to and measuring the thermocouple output.

The foam-walled plastic box is supplied to environmentally isolate the connection of the thermocouple extension wires to the red and black wires as circulating air can generate spurious measurement contributions. The user should connect the ends of the extension cable to the red and black wires, taking care to achieve the correct polarity – the supplied extension cable wires are labelled and should be connected black to black, and red to red. Once these steps have been completed, the self-validating thermocouples are ready for use.

3.3 PC AND SOFTWARE USE INSTRUCTIONS

The PC has been set up with Windows 7 Professional and an account named "estec-npl". To log in to this account the user password will be required (at time of delivery this password is "its90"). Two items of software have been installed for use:

- Custom data logging software: "TC Scanner"
- Custom software for determination of the point of inflection of the melting curve: "Best Fit"

These have been validated via the NPL QA process (May 2012) and are respectively located at:

estec-npl \ Documents \ estec \ builds \ TC Log \ TC Scanner
estec-npl \ Documents \ estec \ builds \ Best fit \ Best Fit

Instructions for use now follow.

3.4 THERMCOUPLE MEASURING SOFTWARE: "TC SCANNER"

This software is presented as an executable file (.exe) – a direct shortcut is provided on the pc desktop. The file is located at: estec-npl \ Documents \ estec \ builds \ TC Log \ TC Scanner

This software reads the thermocouple output via the Keithley 2182A, and therefore this must be connected properly to both the PC and the self-validating thermocouple (see Breadboard Assembly Instructions above).

Open the "TC Scanner" software. The user will be immediately prompted to enter a filename to store the results of the test. Upon pressing OK, logging of the thermocouple output will automatically start.

To pause the program, the user should press the "pause" button in the bottom left-hand side of the screen. To stop the program, the user should press the "stop" button in the lower centre of the screen. A screenshot of the "TC Scanner" software in use is shown in Figure 6. The software interface consists of three graphs; several display items and three user-controllable options. The sections of the interface are labelled.

Graphs 1 and 2 (as labelled) display the same data (thermocouple output versus time) but the scales can be adjusted independently. It is recommended that these are used to show real time information (default for Graph 1) and for viewing portions of the data collected (Graph 2). To adjust the scales, two options are available:

- Automatic, real time display – right click on the graph and select "AutoScale X" and "AutoScale Y"
- Manual scale setting – left click on the max/min values of the axes and set to the user's choice of value

Graph 3 displays the 100 most recent measurements recorded versus measurement number.

The display items show the progress of the measurements to the user. These include the Keithley 2182A output from the last measurement, the time elapsed since the program was begun, the current measurement number and the blue bar indicates progress towards taking the next measurement (where the user has defined a number of points to average).

The user-controllable options include the time interval between measurement points (in milliseconds) and the number of points to average before recording a measurement. This facility can be useful in electrically noisy environments. These values can be adjusted while the program is running. The user may also change the GPIB address in use; but this should not be necessary.

3.5 MELTING TEMPERATURE DETERMINATION SOFTWARE: "BEST FIT"

This software is presented as an executable file (.exe) – a direct shortcut is provided on the pc desktop. The file is located at: estec-npl \ Documents \ estec \ builds \ Best fit \ Best Fit

This software compares the data collected by the "TC Scanner" software against a best fit, third order polynomial expression to evaluate the point of inflection. For metal-carbon eutectic fixed points (i.e. the HTFP cells provided) the melting occurs over a range of fractions of a degree, so the point of inflection of the melting transition measurements is defined to be the melting temperature. This parameter is found to be extremely repeatable and is currently the *de-facto* standard method of determining the fixed-point value.

Upon opening the "Best Fit" software, the user will be immediately prompted to choose an existing file from which the program will source the measured data. A test file is included on the PC. This can be found at:

estec-npl \ Documents \ estec \ Best Fit \ Co-C_test_data

Upon pressing OK, the data will be displayed in the graph. This graph displays both the data set and a best-fit curve to the displayed data. The software fits a polynomial function to the data, with the general form described by the following equation:

$$f_i = \frac{1}{n}\sum_{j=0}^{m} a_j x_i^j$$

where *f* represents the output sequence **Best Polynomial Fit**, *x* represents the input sequence **X**, *a* represents the **Polynomial Coefficients**, and *m* is the **polynomial order**.

A screenshot of the "Best Fit" software in use is shown in Figure 7.
To obtain the 'best fit' curve, the user should adjust the scales ("min x" and "max x" or, alternatively "min y" and "max y"), to home-in on the melting curve plateau. These will be immediately changed within the graph, and the best-fit curve will be automatically adjusted. Figure 7 shows a good curve fit (white line) to the measured data (red points).

The 'mean squared error' (mse) panel gives an indication of the degree of fit: the software calculates the mse using the following equation:

$$mse = \frac{1}{n}\sum_{i=0}^{n-1} (f_i - y_i)^2$$

where *y* represents the input sequence **Y**, and *n* is the number of data points.

Once the user is confident that the best-fit curve is appropriate (that the point of inflection of the data and the curve match), the user can record the thermovoltage (emf) value at the inflection point – as shown in the "Best fit emf" panel. The thermocouple emf value for the inflection point can then be compared to the reference function value at the fixed-point cell melting temperature and a normalisation of the thermocouple emf can be performed to complete the process of self-calibration.

To save the best-fit curve, the user can click the "STOP and write to best fit file" button. This will prompt the user to choose a new filename to save the curve.

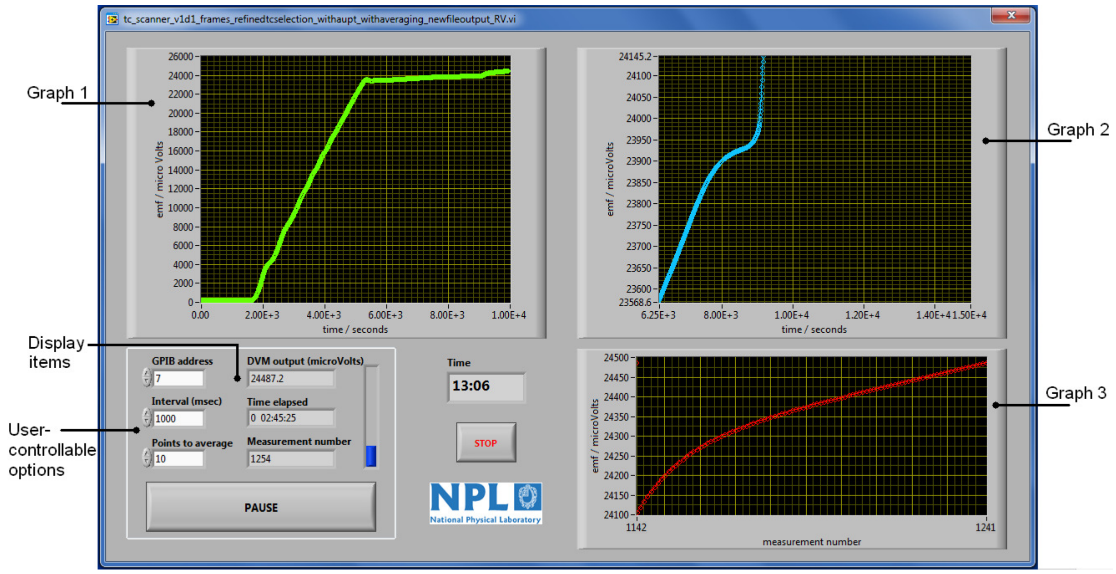

Figure 6: Screenshot of the "TC Scanner" software.

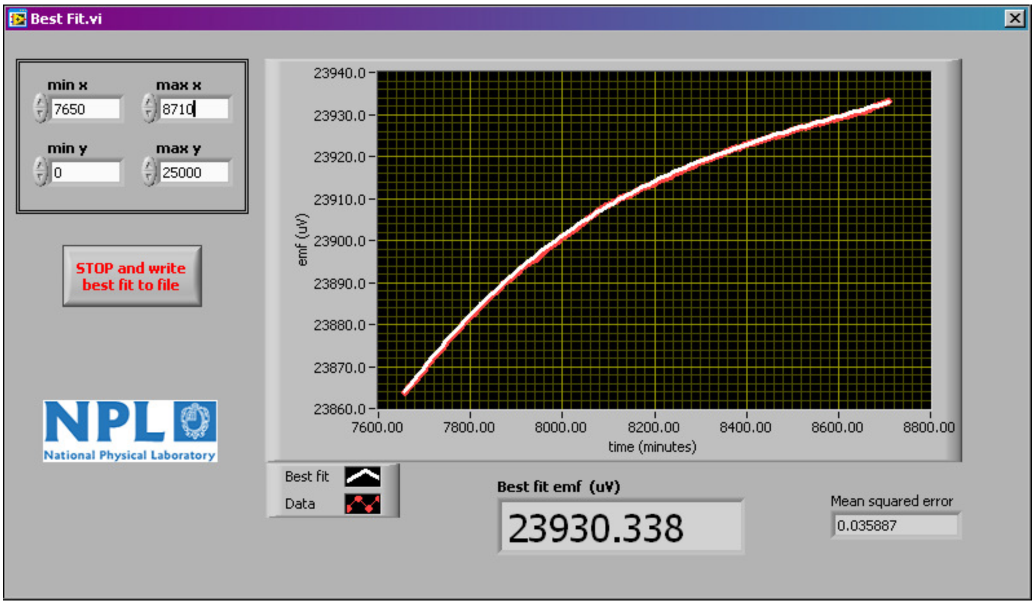

Figure 7: Screenshot of the "Best Fit" software.