NFU 498 Digital Melting Point Apparatus

Purpose:

Determining the melting of small quantities of substances, in the range 25° to 380°C, to aid the identification and assessment of purity of the substance.
Melting point test for up to a maximum of two samples can be conducted simultaneously. The heating rates can be varied from 0°C/min to a maximum of 20°C/min using keypad to heat the sample to its melt temperature. The maximum temperature range is 380°C. The magnifying lens in the eyepiece aided by the illumination provided by a bright white LED gives clear view of the sample during its melting phase. The melting point temperature is displayed on LCD. Other features include an indication buzzer in case of temperature sensor disconnection and in case of crossing the maximum temperature of 400°C.

Theory:

The criterion of the purity of a solid organic substance is its melting point.
This may be defined as the temperature at which the compound changes from solid to the liquid stage.

If an organic compound is pure, it will usually melt within a range of 1° at the most. If it is impure, the melting will occur at a lower temperature or over a wider range. If a pure compound is mixed with a small amount of another pure compound, the melting point is generally lowered. This fact is useful in establishing the identity or non-identity of any two solids.

Operating Procedure

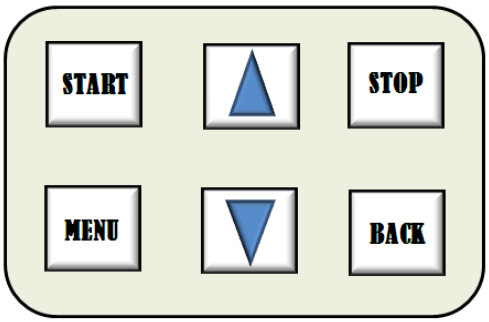
Mains On/Off: A rocker switch to control main power supply to the unit.

Heater Block: This aluminium block can hold two capillary sample tubes.

Viewer / Eye Piece: Comprises a magnifying lens encased in a plastic eye viewer. A bright white LED illuminates the slot where sample capillaries are inserted.

LCD Screen: This display shows programming information / menu options and stored melting points.

Keypad: This membrane keypad controls the equipment via the following buttons:-



1. START: This is for starting the heater initially from ambient to plateau & then from plateau to melt temperature. Also it is used to record up to two melting point values when in ramp state by pressing this key.

2. STOP: This is for stopping the heater once the melt temperature is reached.

3. UP ARROW: This is for browsing through the menu options and setting the plateau & ramp rates.

4. DOWN ARROW: This is for browsing through the menu options and setting the plateau & ram rates.

5. MENU: This is for entering into the menu options screen and for selecting or conforming current menu/option. Also it is used to record up to 2 melt temperatures.

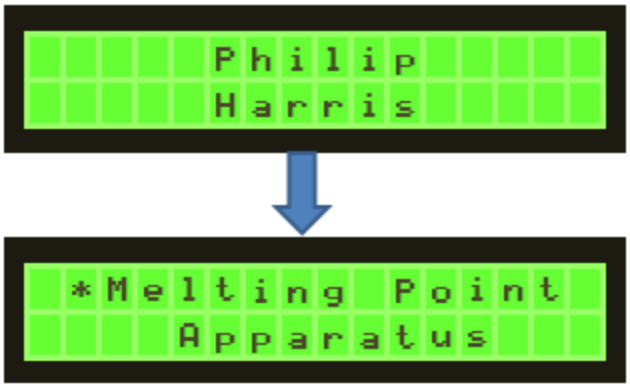
6. BACK: This is for returning the control back to the previous options menu.

Initial set-up

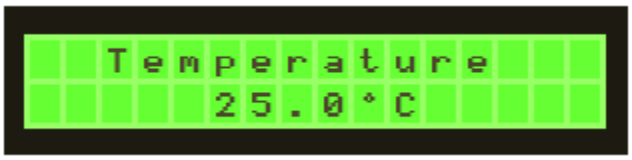
1) Place the unit on a firm, level, non-slip surface. The unit is to be used indoors in a well ventilated area and at a minimum distance of 20cm from walls or any other substances.

2) Connect the mains electricity supply (230V, 50Hz) & switch the unit ON at the mains On/Off switch. When switched ON,the LCD display will show power up messages followed by the current temperature.

3) The initial power up screen is followed by a buzzer beep that indicates readiness of system for experiment start.



Current temperature of the heating block/sample will be displayed when the equipment is left in idle condition after power up.



Menu Screen

The menu screen can be selected by pressing the 'MENU Key'. Before starting the experiment, all the parameters on the menu options are to be set. Otherwise default parameters will be considered for the heating process.

Default Parameters:

a) Plateau – 100°C
b) Ramp Rate – 2°C/min
c) Unit Scale – Celsius
d) Last Records – None
e) Audio Feedback Enabled
f) LCD Backlight Enabled
g) Calibration Enabled
h) System Info – Model and firmware versions

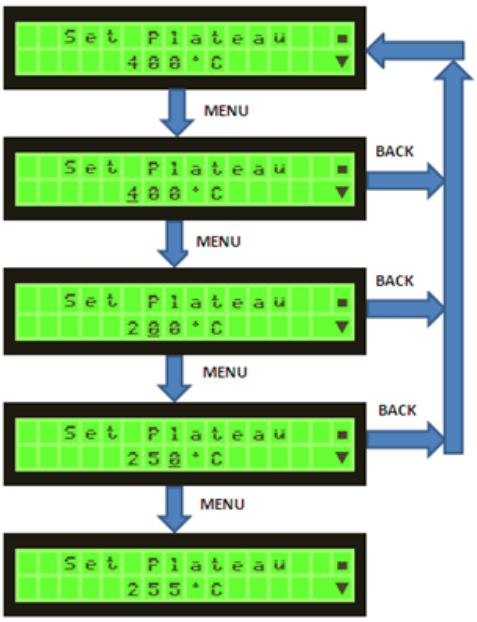
Pressing the 'MENU Key' will take you to the First Option in MAIN MENU as shown below.

Set Plateau

a) Press 'MENU Key' again will take you inside the 'SET PLATEAU' option. The cursor will be on the first digit and the digit will be blinking.

b) Using UP / DOWN key, the desired value for the digit is selected.

c) Pressing the MENU key again will take the cursor to the second digit. Follow the steps as mentioned above for setting the first digit and then press MENU key to take the cursor to the third digit.



d) Follow the same steps as for setting the digit one and two followed by MENU key to set the plateau value. The control will go back to the MAIN MENU.

e) Pressing the BACK key anywhere inside the SET PLATEAU menu will take the control to the MAIN MENU and the plateau value will be same as previous.

f) User can set only values that are within the maximum and minimum temperature range ie; 0°C to 400°C or the corresponding values in Fahrenheit scale or Kelvin scale.

Pressing the DOWN key will take you the second option in the MAIN MENU.

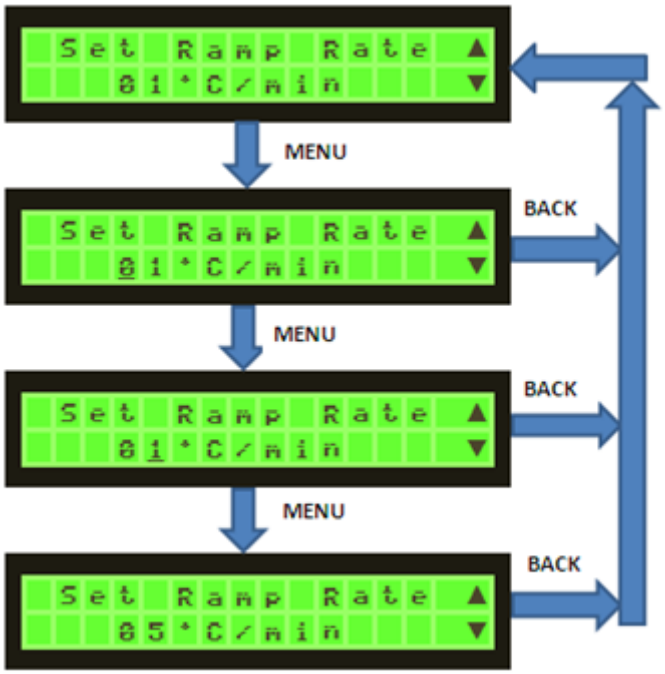
Set Ramp Rate

a) Press ‘MENU Key’ again will take you inside the ‘SET RAMP RATE’ option. The cursor will be on the first digit and the digit will be blinking.

b) Using UP / DOWN key, the desired value for the digit is selected.

c) Pressing the MENU key again will take the cursor to the second digit. Follow the steps as mentioned above for setting the first digit and then press MENU key to set the new ramp rate and the control will be transferred to the MAIN MENU.

d) Pressing the BACK key anywhere inside the SET RAMP RATE menu will take the control back to the MAIN MENU and the ramp rate value will be same as previous.



e) The ramp rate can be set to any value between 1°C/min to 10°C/min or 1°F/min to 10°F/min or 1K/min to 10K/min (including both the end values).

Pressing the DOWN key will take you the third option in the MAIN MENU.

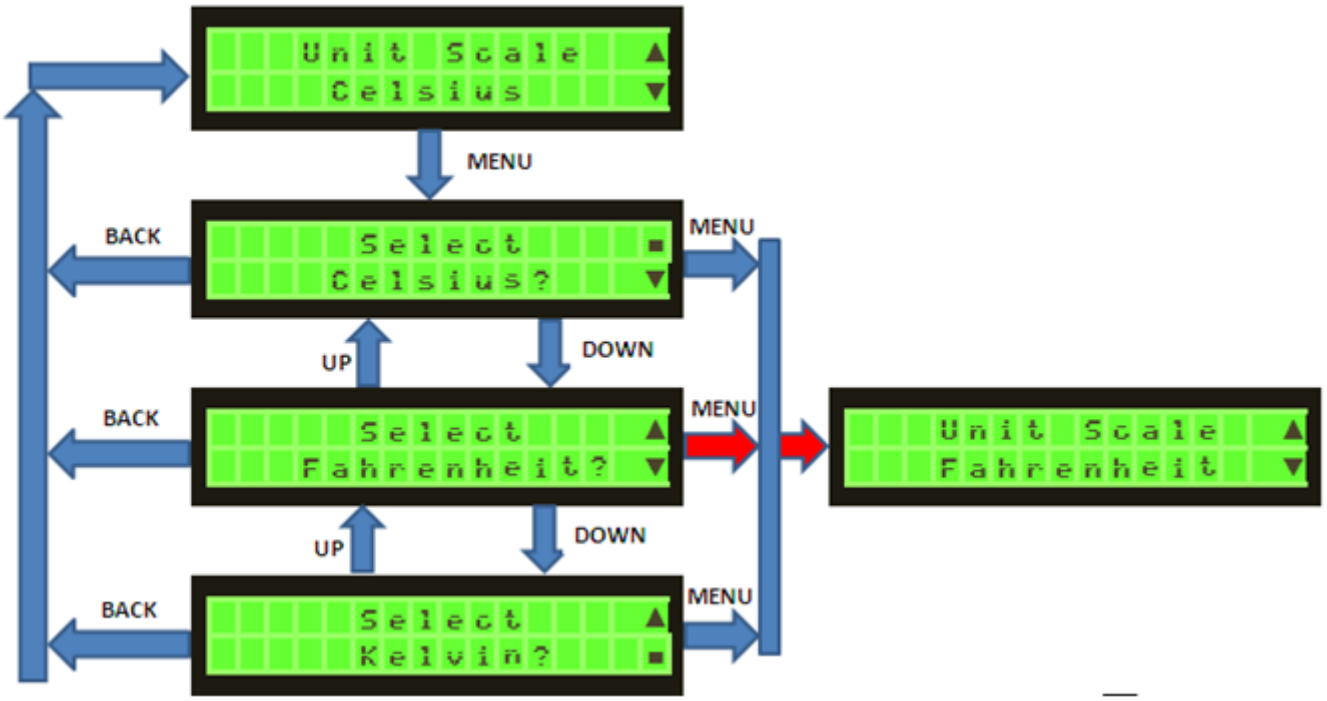
Unit Scale Selection

a) Press ‘MENU Key’ again will take you inside the ‘UNIT SCALE’ selection option.

b) Using UP / DOWN key scroll through the different options of temperature scales ie; degree Celsius or Fahrenheit or Kelvin.

c) Pressing the MENU key when a particular scale name is displayed on the LCD will set that particular scale and the control will be transferred to the MAIN MENU.

d) Pressing the BACK key anywhere inside the UNIT SCALE menu will take the control back to the MAIN MENU and temperature scale will be same as previous.



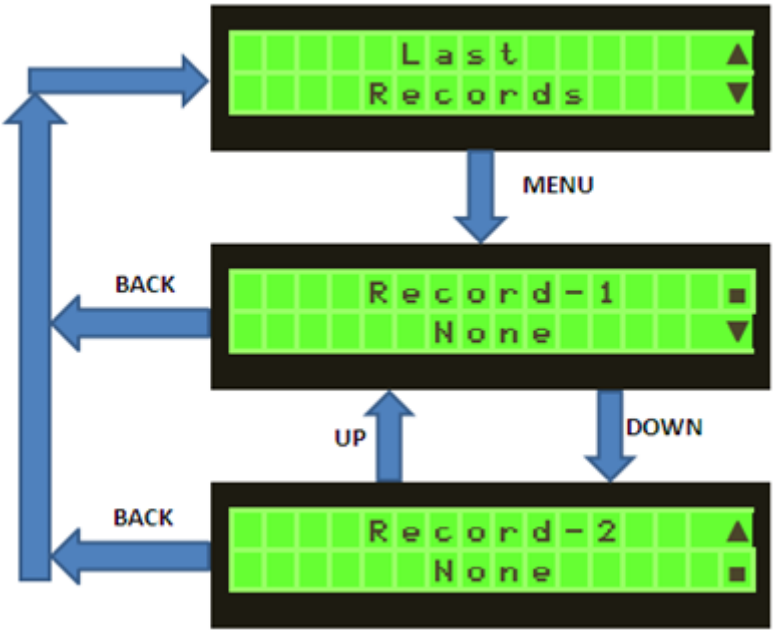
Pressing the DOWN key will take you the fourth option in the MAIN MENU.

Last Records

a) Press 'MENU Key' again will take you inside the 'LAST RECORDS' option.

b) Using UP / DOWN key scroll through the SUB MENU showing Record -1 and Record – 2 values ie; the previously stored melting point temperature values. If no previous values are stored, 'None' will be displayed on the screen.

c) Press back key to exit from SUB MENU to MAIN MENU.



Pressing the DOWN key will take you the fifth option in the MAIN MENU.

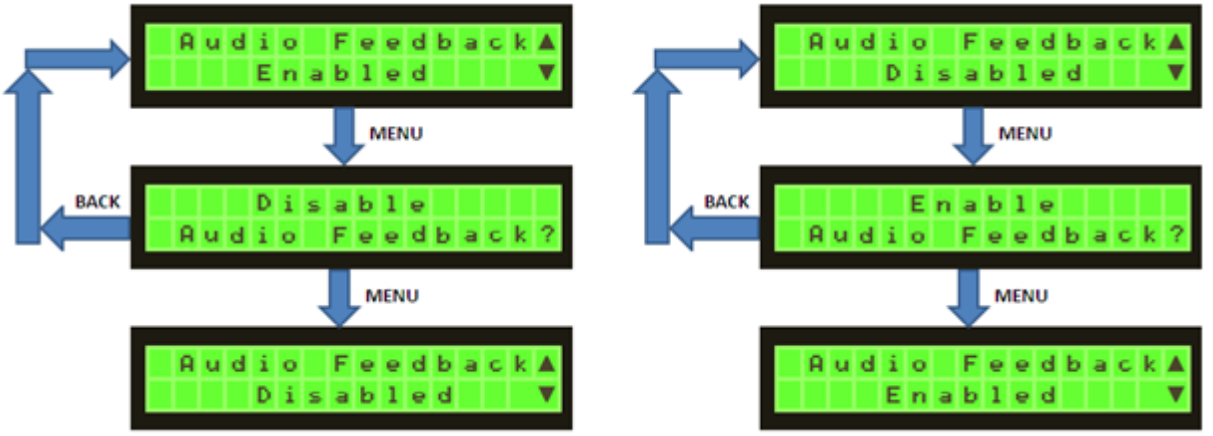
Audio Feedback Control

a) Press 'MENU Key' again will take you inside the 'AUDIO FEEDBACK ENABLED / DISABLED' menu.

b) If the audio feedback was enabled before, a message will be displayed on LCD asking for confirmation to disable the audio feedback. If the audio feedback was disabled before, a message will be displayed on LCD asking for confirmation to enable the audio feedback.

c) Pressing the MENU key will enable / disable the audio feedback and the control will be transferred to the MAIN MENU.

d) Pressing the BACK key while inside the SUB MENU will take the Control back to the MAIN MENU and the Audio feedback will retain its previously set condition.

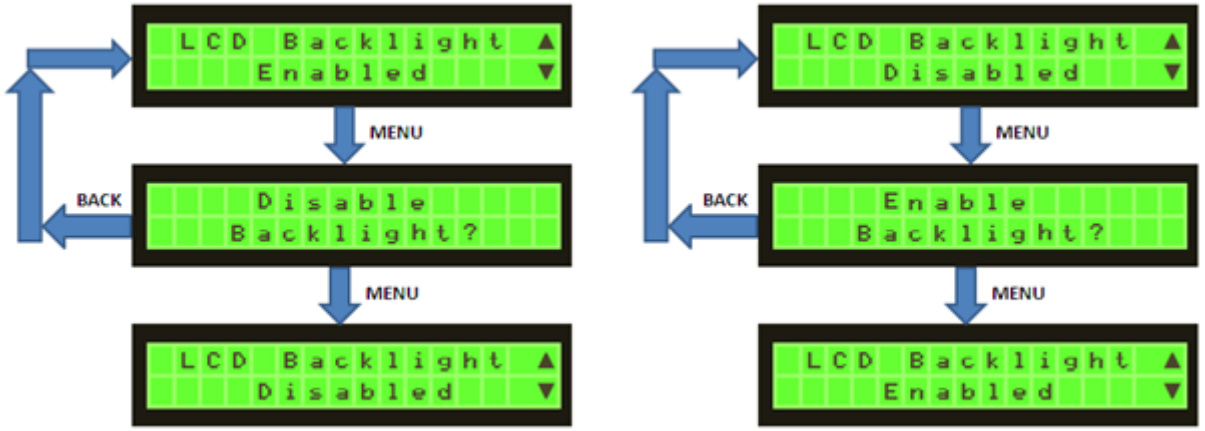


Pressing the DOWN key will take you the sixth option in the MAIN MENU.

LCD Backlight Control

a) Press 'MENU Key' again will take you inside the 'LCD BACKLIGHT ENABLED / DISABLED' menu.

b) If the LCD backlight was enabled before, a message will be displayed on LCD asking for confirmation to disable the LCD backlight. If the LCD backlight was disabled before, a message will be displayed on LCD asking for confirmation to enable the LCD backlight.



c) Pressing the MENU key will enable / disable the LCD backlight and the control will be transferred to the MAIN MENU.

d) Pressing the BACK key while inside the SUB MENU will take the Control back to the MAIN MENU and the LCD backlight will retain its previously set condition.

Pressing the DOWN key will take you the seventh option in the MAIN MENU.

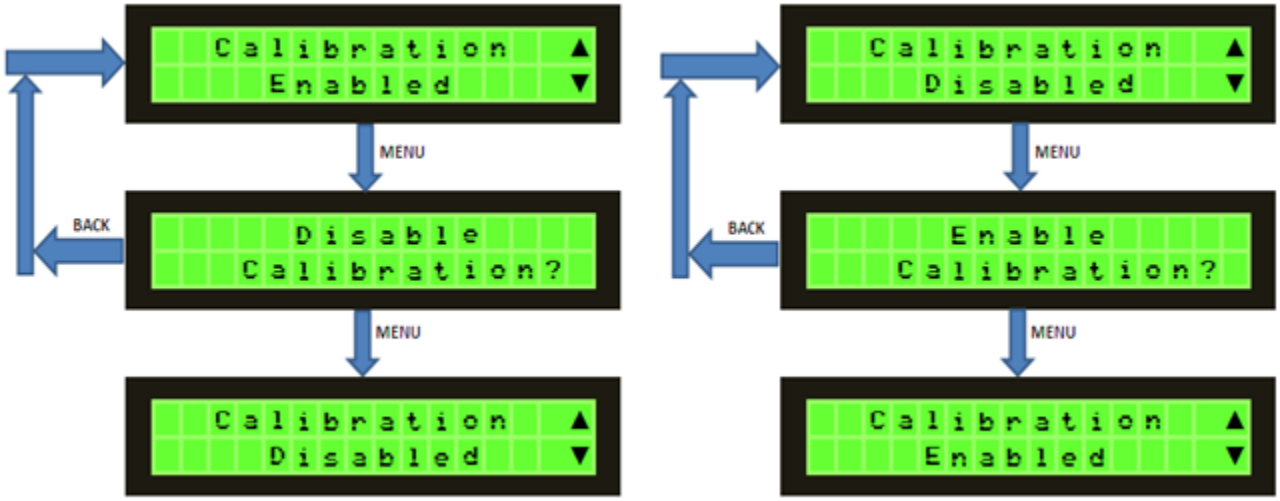
Calibration Control

a) Press ‘MENU Key’ again will take you inside the ‘CALIBRATION ENABLED / DISABLED’ menu.

b) If the Calibration was enabled before, a message will be displayed on LCD asking for confirmation to disable the Calibration. If the Calibration was disabled before, a message will be displayed on LCD asking for confirmation to enable the Calibration.

c) Pressing the MENU key will enable / disable the Calibration and the control will be transferred to the MAIN MENU.

d) Pressing the BACK key while inside the SUB MENU will take the Control back to the MAIN MENU and the Calibration status will hold its previously set condition.



Note:

Calibration is normally enabled by default. User should disable calibration only in case he/she is using the device to find the melting points of Certified Reference Standards (Substances which are very pure and has accurate melting point values) which are to be used for calibrating the device. Pressing the DOWN key will take you the eighth option in the MAIN MENU.

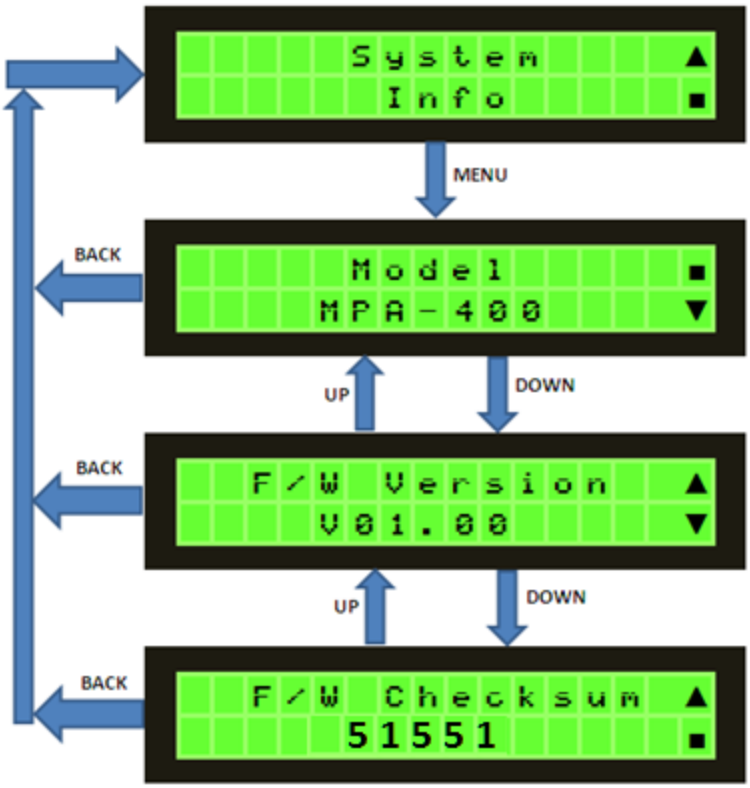
System Info

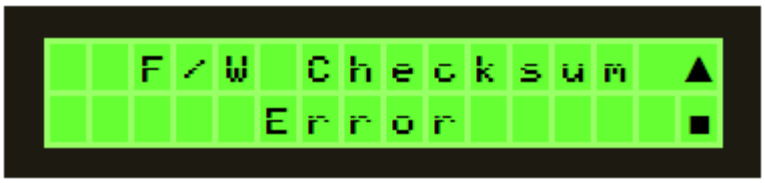
a) Press ‘MENU Key’ again will take you inside the ‘SYSTEM INFO’ option.

b) Using UP / DOWN key scroll through the SUB MENU showing Model, Firmware Version and Firmware Checksum.

c) Press back key to exit from SUB MENU to MAIN MENU.

d) In case any checksum error was found, instead of checksum value 'Error' will be displayed





Pressing the UP key while in MAIN MENU will take you to the previous option in the MAIN MENU list. Pressing the BACK key will take you to the idle screen displaying the current temperature.

Measuring a Melt Temperature

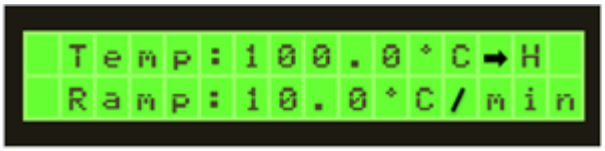
1) Switch on the power button on the side of the instrument. The instrument turns on with a beep sound followed by Power Up Messages. Then the bright white light LED turns ON and the LCD displays current temperature.

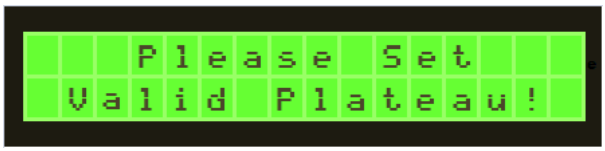
2) Insert the sample filled capillary into the slot on the side of the heater. A maximum of 2 capillary tubes can be inserted at one time.

3) Position the capillary tube, looking down the lens, so that the sample is observed clearly.

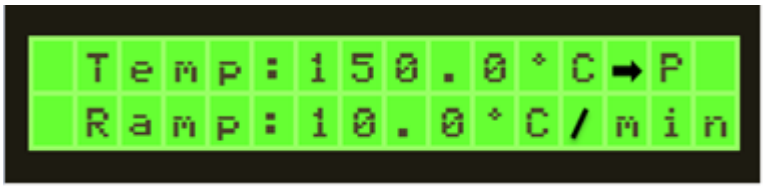
4) Set the Plateau and Ramp rate. The plateau to be set about 20°C below the expected melt temperature and the ramp rate preferably 1 to 2 °C/min.

5) Start heating by pressing the START key. The heater start heating at the maximum rate till the plateau is reached. The LCD will display the current temperature of the sample and the rate at which the sample is being heated. The letter H keeps blinking indicating that the PLATEAU process is going on. If the set plateau is less than current temperature, an error message will be displayed and the heating process will not start.

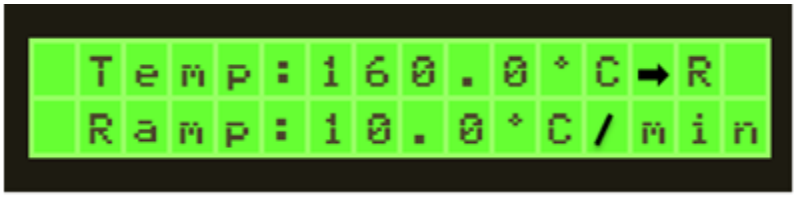




6) On reaching the set plateau temperature, the buzzer beeps and the device maintains the sample at that temperature. The letter P keeps blinking indicating that the PLATEAU process is going on.



7) Press the START key again will start the RAMP phase where in the sample is heated at a temperature set by the user. The letter R keeps blinking indicating that the PLATEAU process is going on.



8) On observing the sample melt press the START button to store the melting point value. Two such records are stored by the device.

9) Press the STOP key to stop the heater and bring the control back to idle process displaying the current temperature of the sample/heater block.

10) Pressing the STOP key any time during the heating process will stop the heater and bring the control back to idle process displaying the current temperature of the sample/heater block.

General Safety Instructions & Usage Tips

The equipment is for indoor use only with area well ventilated.

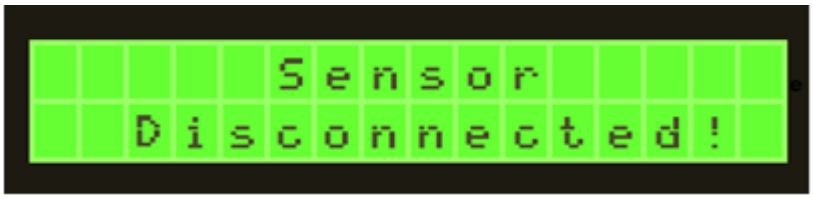
1) Ambient temperature 5°C to 40°C,altitude 2000m, relative humidity not exceeding 80%.

2) Mains supply fluctuation not >10%.

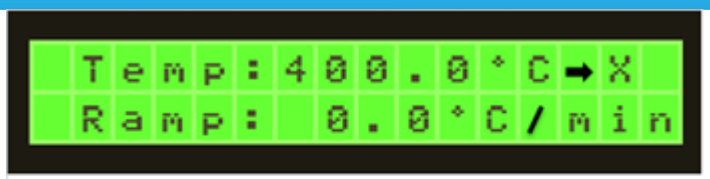
3) Use within a minimum distance of 200mm from walls and other items.

4) Before connection to mains supply ensure that lines supply corresponds to that stated on the rating label. The equipment must be properly earthed.

5) In case the buzzer beeps followed by 'Sensor Disconnected!' message on LCD screen, the reason is that the pt100 sensor is disconnected. If the pt100 connection is restored, then the device restarts. Switch off the mains supply & restore the connection in case there is a broken connection.



6) The equipment is programmed such that, the heater temperature will not go above 400°C. However the user is supposed to be aware of the maximum heating temperature limit. On reaching the maximum temperature, a message will be popped up in the screen every 5seconds for 1 second followed by a buzzer beep. Otherwise the screen will display the maximum temperature value and ramp rate as 0°C/min. The Letter X keeps blinking while at the max temperature.



7) The sample used for testing should be dry & finely powdered.

8) Ensure there is no sample on the outside of the tube before inserting it into the melting point apparatus.

9) A sample should be used only once for melting point determination as some substances chemically decompose on heating.

10) Do not heat the sample too fast; generally 1-2ºC/minute is recommended. If the melting temperature is unknown, first perform a rapid melt with a ramp of up to 20ºC/minute. Once the approximate melting point is known, follow the instructions described in "Operating Procedure" section.

11) If the unit has been used recently the block may be too hot for your sample. If this is the case, turn the apparatus off and allow the unit to cool before proceeding.

12) Ensure the unit is disconnected from the mains electricity supply and allowed to cool before attempting any cleaning or servicing.

13) Periodically clean the instrument using a damp cloth and mild detergent solution. Do not use harsh or abrasive cleaning agents.

Calibration Procedure

Though the melting point apparatus has been calibrated and checked during production phase, to make sure that the apparatus is measuring the melting point temperature accurately, calibration of the apparatus can be done by the user. The procedure involves the use of Certified Reference Standards(CRS) which are chemicals of high purity that have been tested and certified by standard laboratories. In case the accuracy of the instrument is found to deviate from the expected, the following procedure can be performed to recalibrate the instrument to display the accurate temperature.

1) Disable Calibration by following the procedure as mentioned in section 3.7.

2) Find out the melting point values of certified reference samples by following the steps as mentioned in Section.4. Select the reference standards so that the whole melting point range is covered. A few of the CRSs with their melting points are as below. Also ensure that the 'DEGREE CELSIUS' option is selected.

Please note that the melting point of a minimum of 3 CRSs have to be found out to be able to perform the calibration procedure. The more the number of sample the better the accuracy of calibration will be. Also, please ensure that the CRSs are selected to cover the whole melting point range of the instrument.

3) Enable Calibration by following the procedure as mentioned in section 3.7.

4) Switch OFF the device and turn it ON again keeping the 'MENU' button pressed.

5) Once the 'Calibration Mode' message pops up on the screen, you are good to take your fingers off the 'MENU' button that you were pressing.

6) The calibration MAIN MENU is displayed which has the following options:

a) View Calibration Coefficients
b) Set Calibration Coefficients
c) Calibration Info

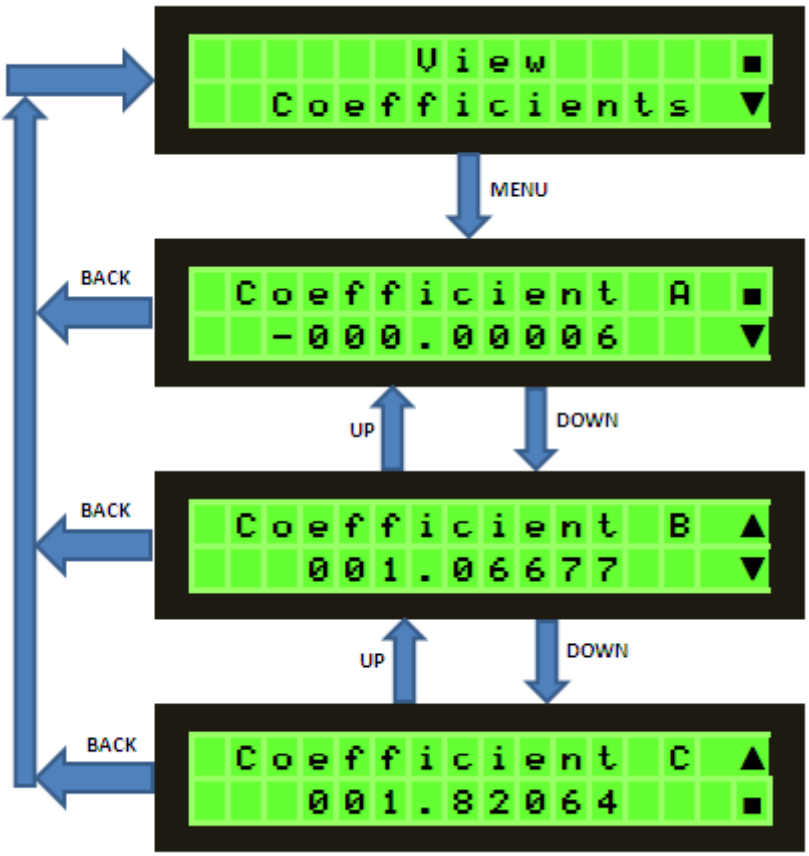
View Calibration Coefficients

This menu displays the calibration coefficient values ie; A, B and C coefficients' values. The menu can be accessed by pressing the 'MENU Key'.

a) Press 'MENU Key' again will take you inside the 'VIEW CALIBRATION' option.

b) Using UP / DOWN key scroll through the SUB MENU showing Coefficients A, B and C respectively.

c) Press back key to exit from SUB CALIBRATION MENU to MAIN CALIBRATION MENU.



Pressing the DOWN key will take you the second option in the MAIN MENU

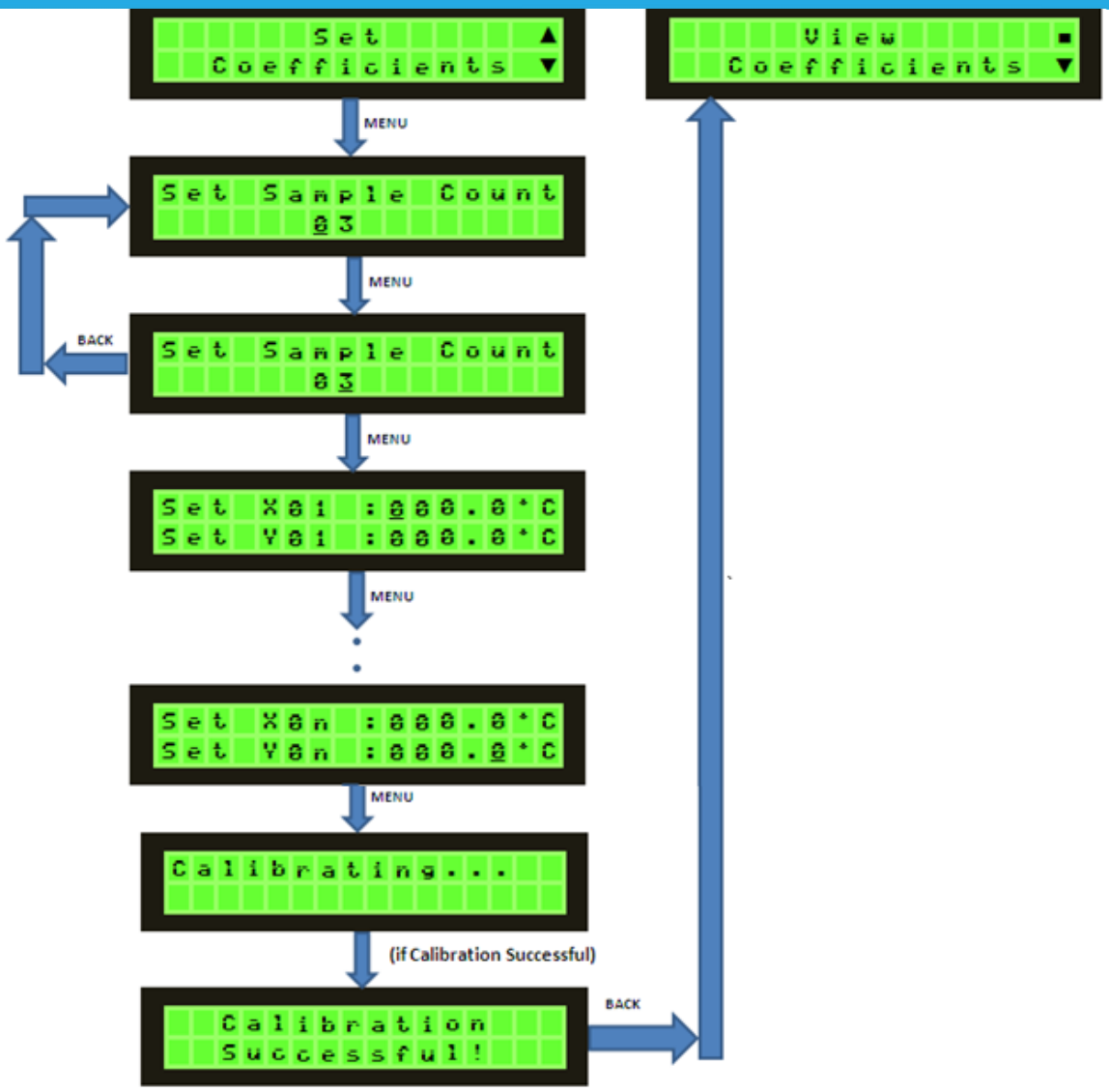
Set Calibration Coefficients

a) Press 'MENU Key' again will take you inside the 'SET COEFFICIENT' option. The cursor will be at the first digit and the digit will be blinking.

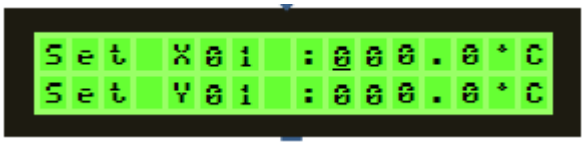
b) Using UP / DOWN key, the desired value for the digit is selected. The minimum set value is 03 and maximum value is 12.

c) Pressing the MENU key again will take the cursor to the second digit. Follow the steps as mentioned above for setting the first digit and then press MENU key to set the new sample count and the control will be transferred to the 'Set X & Y value MENU'. Please ensure that the number of sample count same as the number of CRSs for which the melting point experiment has been conducted in step 2 above.

   Pressing the BACK key when the cursor is at the second digit, will move the cursor back to the first digit and enables the user to change the first digit. Pressing the BACK key when the cursor is at the first digit has no effect.

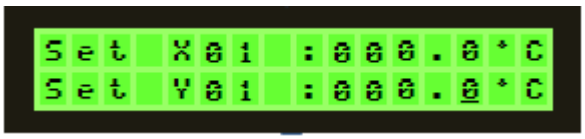


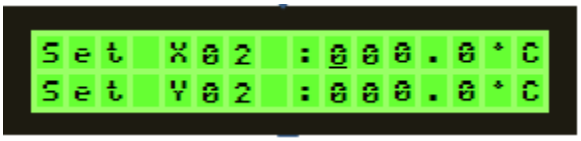
d) Now the X01 and Y01 values can be set. The cursor will be at the first digit of X01 and the digit will be blinking. Using UP / DOWN key, the desired value for the digit is selected. X and Y can have values between 0 t0 400°C (including both the values**). X corresponds to the observed melting point value of the CRS and Y corresponds to the actual melting point value of the same CRS.**



e) Pressing the 'MENU Key' will move the cursor forward through the digits of X and Y. First the 4 digits in X and then the 4 digits in Y.

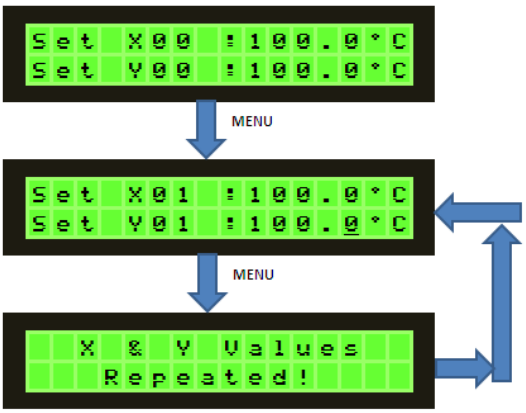
f) Pressing the 'Menu Key' after the 4th digit of Y(Y's decimal place digit), that particular value of X and Y are stored and the screen displays next set of X and Y values to be set. The user has to set 'n' number of X and Y values where n = sample count.





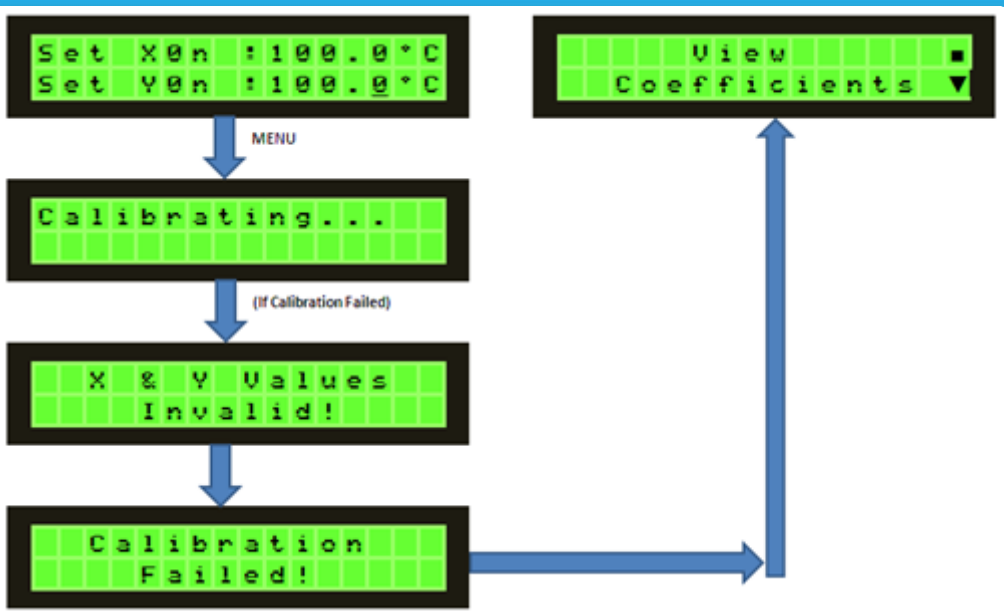
g) No two values of X ie; any X value and any previously set X value cannot be the same. The same is mandatory with Y values also. If any X value equals any previously set X value, screen displays 'X VALUE REPEATED' and the user has to re-enter the X and Y values. The screen 'Y VALUE REPEATED' is displayed if Y value is repeated and the screen 'X & Y VALUES ARE REPEATED' is displayed if both X & Y values matches to any previously set X and Y value respectively.

h) No two values of X ie; any X value and any previously set X value cannot be the same. The same is mandatory with Y values also. If any X value equals any previously set X value, screen displays 'X VALUE REPEATED' and the user has to re-enter the X and Y values. The screen 'Y VALUE REPEATED' is displayed if Y value is repeated and the screen 'X & Y VALUES ARE REPEATED' is displayed if both X & Y values matches to any previously set X and Y value respectively.



i) Pressing the 'BACK Key' will move the cursor back to the previous digit's position while setting X and Y values. An exception is that it has no effect when the cursor is at the first digit of X value, ie; pressing the same key has no effect.

j) Pressing the 'Menu Key' after entering the nth sample's Y value will display the screen 'CALIBRATING..' followed by 'CALIBRATION SUCCESSFUL' screen if the calibration is successful. If calibration is not successful due to invalid values of X and Y, the Screen displays 'X & Y VALUES INVALID' followed by 'CALIBRATION FAILED' message.



k) If calibration is successful, the coefficients are set and the main calibration menu is displayed on LCD screen. User can see the newly set coefficients from the 'View Coefficients' Menu.

l) If calibration fails, the coefficients will retain its previously set values and the user has to follow the same steps as above once again to recalibrate the apparatus.

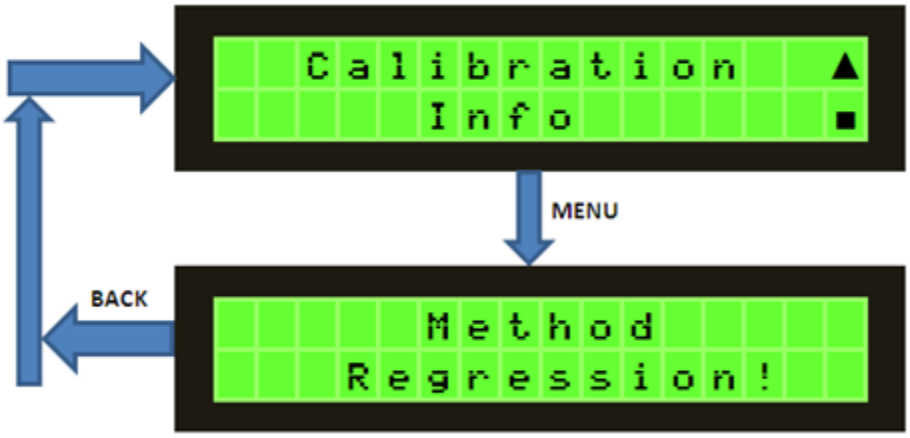
m) In case the user has set the sample count and pressed the 'MENU Key' the same cannot be reset, the only way doing the same is to restart the device. The same is true for any value of X and Y.

Calibration Info

This menu displays the calibration Information ie; the method used for calibrating the device. The menu can be accessed by pressing the 'MENU Key'.

a) Press 'MENU Key' again will take you inside the 'VIEW CALIBRATION' option.

b) Press back key to exit from SUB CALIBRATION MENU to MAIN CALIBRATION MENU.



CAUTION – the heating block will get hot when left on for long periods of time. Please be aware on this when handling the unit.

An alarm will sound when the temperature exceeds 400°C. To turn the alarm off, press STOP key and allow the heating block to cool down.

Warranty, repairs and spare parts:

The Digital Melting Point Apparatus is guaranteed for a period of one year from the date of delivery to the customer. This warranty does not apply to defects resulting from the action of a user such as misuse, improper wiring, any operations outside of its specification, improper maintenance or repair, or unauthorized modification.

Our liability is limited to repair or replacement of the product. Any failure during the warranty period should be referred to Customer Services.

In the event of a fault, apart from replacing the instrument fuse in the fuse socket, the unit should be referred to the Philip Harris recommended repair agent.

Please contact Customer Services or techsupport@philipharris.co.uk for advice

Disposal of Waste Electrical and Electronic Equipment (WEEE)

Do not dispose of this product with household waste

- For the proper treatment, recovery and recycling please take this product to an appropriate collection point.
- If you are unsure where this is, contact your Local Authority.
- By disposing of this product correctly you will be providing positive help to the environment.

Supplier details:
Philip Harris Education, 2 Gregory Street, Hyde, Cheshire SK14 4RH

Orders and Information	Tel: 0845 120 4521
	Fax: 0800 138 8881
Repairs	Tel: 0845 120 3211

E-mail: techsupport@philipharris.co.uk

Website: www.philipharris.co.uk