

Melting Point Apparatus SMP40

Instructions for use





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1. Introduction

Thank you for purchasing this piece of Stuart equipment. To get the best performance from the SMP40 please read these instructions carefully before use. Before discarding the packaging check that all parts are present and correct.

For your own safety and that of others please read and understand the safety advice given below before using the equipment.

2. Safety Advice Before Use

If the equipment is not used in the manner described in this manual and with accessories other than those recommended by Stuart, the protection provided might be impaired.

This equipment is designed to operate under the following conditions:

- For indoor use only
- Use in a well ventilated area
- ♦ Ambient temperature range +5°C to +40°C
- Altitude to 2000m
- Relative humidity not exceeding 80%
- Mains supply fluctuation not exceeding 10%
- Over-voltage category II IEC 60364-4-443
- Pollution degree 2

Never move or carry the unit when in use or connected to the mains electricity supply.

The unit should be carried using both hands with fingers under each side of the frame.

The ventilation slots at the back or side of the device should not be sealed or obstructed.

3. General Description

The device is designed to automatically measure and record the melting temperatures of crystalline samples held within capillary tubes. Up to three tubes can be accommodated in an illuminated chamber within an aluminium block. The tubes are monitored by a digital camera; the image of which can be viewed on the instruments display during the melting process. The device is designed to be independently operated using the colour touch screen allowing all operational and data management functions to be performed without the need to attach an external computer or keyboard. The unit can be split into two modules to allow the heating module to be located in an environment suitable for the types of samples being tested, while the control module can be kept in a more convenient location.

The temperature range of the device is ambient to 400°C and the heating rate is variable between 0.1°C and 20°C/minute. Temperature data and information on the instruments status is displayed on the screen at all times. All of the instruments functions and menus are accessed using the colour touch screen interface which allows fast and intuitive navigation throughout a substance's melting point determination (melt determination) The instrument has sufficient internal storage capacity for an almost unlimited number of preset method programs and typically between 100 and 200 result files* When required, result files can be transferred from the instrument to a PC or USB flash drive for external storage and archiving.

When a melt cycle is started the SMP40 heats up to the pre-programmed plateau temperature at the maximum rate before stabilising at the plateau temperature for 60 seconds. The SMP40 will then start to heat at the pre-programmed rate until the melt is complete or until the device reaches a pre-determined end temperature. Once a melt determination has been completed, cooling is automatically engaged until the instrument reaches 50°C.

* The number of result files that can be stored on the SMP40 is largely dependent upon the runtime and size of the video file for each sample.

The user is able to review the result file and video, reprocessing the automatically determined results if required.

3.1 Module Separation





Figure 1: SMP40 module separation

The SMP40 can be split into two separate modules using the split module release mechanism located at the rear of the "Control" module. Using the supplied accessory cables the modules can be placed in separate locations up to 1m apart.

Please ensure that the instruments electrical power supply is switched off or disconnected before commencing the following procedure.

1. Rotate the split module release mechanism clockwise until it cannot be turned any further.

2. Gently pull the "Control" and "Melt" modules apart taking care not to strain the connecting USB cable.

3. Disconnect the USB cable on the control module that connects the two modules.

4. Using the accessory cables supplied with your SMP40 reconnect the power and USB connections between the modules.

5. Once the devices power supply is re-established the unit can then be used as described in this manual.

6. Reconnecting the modules is the reverse of the above.

Please ensure that the extension cables are not removed from their connections when the unit is connected to the electrical power supply and switched on. The SMP40s touchscreen display is fitted with an accelerometer device.



Upright

2

3.2 Melting Point Determination

The capillary technique is a standard method for determining the melting point of a substance. In this method, thin glass capillary tubes containing prepared samples of a substance are introduced into the heating block of a suitable device. The block is then heated at a fixed rate over a temperature range that brackets the expected melting temperature of the substance, until the substance is completely melted (i.e. clear point).

During the heating cycle the substance under test will often undergo a number of observable physical changes:

- **1.** Fine droplets adhere uniformly to the inside of the capillary tube.
- **2.** A clearance between the sample and the capillary wall becomes visible.
- 3. The substance collapses and begins to liquefy.
- A meniscus is visible at the samples surface but solid particles remains visible. (Meniscus Point)
- **5.** The sample is a clear liquid and no solid particles remain visible. (Clear Point)

When determining the melting point of a substance it is necessary to select one stage in the melting process which will be used to define the melting point. The meniscus point or clear point is commonly used to define the melting point of a substance.

The SMP40 automatically reports the Clear Point (5); the point at which the sample is a clear liquid and no solid particles remain, as the melting point of a substance.

Other changes in the physical appearance of the substance are sometimes observed, such as sublimation (transition from the solid to gas phase with no intermediate liquid stage) or decomposition (the separation of a substance into its elements or smaller compounds). These changes (if observed) should be recorded manually.

Where a single melting point is difficult to define for a substance it is often useful to report a substances melting range. The melting range of a substance is defined as the interval between the start of a melt and the clear point.

3.3 Temperature Settings

The temperature settings used to determine a substances melting point are critical to the accuracy of the determination.

Start or Plateau Temperature:	The start temperature is usually set 5 or 10°C below the expected melting point of the substance.
End Temperature:	The end temperature is set at a temperature where all physical changes in the substance are expected to be complete.
Ramp Rate:	The ramp rate is the rate at which the sample block is heated during the melt determination. For accurate determinations a rate of between 0.5°C/min and 2.0°C/ min is recommended.

Ramp rate is the most important instrument parameter with regards to the accuracy of a substances melt determination. The use of fast ramp rates is the most common cause of inaccurate melt determinations.

3.4 Sample Preparation

For accurate melting point determinations the substance being tested should be in a dry and uniformly powdered form. Coarse crystalline and non-homogeneous samples should be crushed into a fine powder using a pestle and mortar before the substance is introduced into a capillary tube.

Each capillary tube in a substances melt determination should be prepared in the same way. The open end of a capillary tube is pressed gently into a small amount of powdered substance until the capillary contains sufficient material to form a compact layer of 2.5-4.5mm in height. The powder is then forced to the bottom of the tube by repeatedly tapping the bottom of the capillary against a hard surface.

Excessive amounts of sample in the capillary tubes will result in inaccurate melt determinations.

The SMP40 can accommodate up to three sample capillaries. For accurate melting point determinations it is recommended that three samples of the same material are prepared in the same way and analysed at the same time. The average of the three capillaries results can then be reported as the substances melting point.

4. Preparation for Use

4.1 Electrical Installation

) THIS EQUIPMENT MUST BE EARTHED

Before connecting the instrument, please read and understand these instructions and ensure that the electrical supply corresponds to that shown on the rating plate. This unit is designed for use with a supply rated at 115/230V, 50-60Hz. The power consumption of the unit is 60W. The unit is fitted with an IEC socket at the side of the instrument for connection of the mains lead.

Caution: Fuses are fitted in both live and neutral lines.

The unit is supplied with two mains leads fitted with IEC plugs. One lead has a UK 3 pin plug and the other has a 2 pin "Shuko" plug for connection to the mains supply. Choose the lead appropriate for your electrical installation and discard the other. Should neither lead be suitable take the lead with the UK plug and replace the plug with a suitable alternative. This involves cutting off the moulded plug, preparing the cable and connecting to the rewireable plug in accordance with its instructions.

IT IS IMPORTANT THAT THIS OPERATION SHOULD ONLY BE UNDERTAKEN BY A QUALIFIED ELECTRICIAN.

Refer to the equipment's rating plate to ensure that the plug and fusing are suitable for the voltage and wattage stated. The wires in the mains cable are coloured as follows:

LIVE	BROWN
NEUTRAL	BLUE
EARTH	GREEN/YELLOW

The appropriate mains lead should be connected to the instrument BEFORE connection to the mains supply. Should the mains lead require replacement, a cable of 1mm² of harmonised code H05VV-F connected to an IEC320 plug should be used. The UK mains lead is protected by a 10A fuse mounted in the plug top.

4.2 Installation

Connect the unit to the electricity supply but DO NOT SWITCH ON. Place the unit on a firm, level, non-slip surface ensuring that there is sufficient free space on all sides without coming into contact with anything else during use. Switch the unit ON at the mains On/Off switch on the side of the instrument. When switched ON, the touch screen display will illuminate and the interface software will load within 30 seconds.

IF IN DOUBT CONSULT A QUALIFIED ELECTRICIAN



The sample block will become hot during operation.

5. Instrument and Software Controls



5.2 Main Menu Screen

Figure 3:



When the instrument is first switched on the display will show "LOADING PLEASE WAIT" message. After 30 seconds the display will show the main menu screen and the four main menu options:

Measurement: This menu gives access to options that allow a new melt determination to be performed using new, previously stored or preset method parameters.

Results: This menu gives access to the result files stored on the device. The result files report and video file can be accessed from this menu.

File Management: This menu allows result files on the device to be copied or moved to an inserted USB flash drive. The devices storage information is also displayed in this menu.

Settings and Calibration: This menu gives access to the devices user adjustable settings and also allows the device to be recalibrated if required.

The Stuart SMP40 is controlled solely through the touch screen interface of the instrument. The header bar is located at the top of the screen in most menus within the software interface.



Back Button: Used to return to the previous screen at anytime

Block Temperature: Displays the actual temperature reading from the sample block Time and Date: Displays the current time and date in the format specified in the user settings Menu Title/Name: Displays the current menus title or name Instrument Status: Displays the current instrument status Home Button: Used to return to the main menu at anytime

The command toolbar is located at the bottom of the screen in most menus within the software interface. The command toolbar will give the user either one or two options based upon the menu shown.

Start the melt Cancel and go back

Figure 5: The Command Toolbar

If the number of options available in a menu exceeds the number that can be displayed on the screen, a scrollbar will be displayed on the right hand side. Pressing the arrows at the top and bottom of the scrollbar enables the list of options available in the menu to be seen.

The following controls are used to input data and adjust settings on the instrument:





Toggle Selection Field: Option is not selected or selected respectively.

Other keys used:

BCK Removes the character directly behind the cursor or the highlighted characters.

DEL Removes the character directly in front of the cursor or the highlighted characters.

SHIFT Toggles the keypad between higher and lower case letters for one character entry.

CAPS Toggles the keypad between higher and lower case letters for all characters entered.

CLR Clears the characters in the current entry.

To change the data in each of these fields the screen should be pressed within the border of the control. The interface used to add or change the setting depends on the type of control.





- 1 Selecting an Alphanumeric Entry Field opens the text keypad interface.
- 2 The alphanumeric entry field can be populated with letters (Abc), numbers (123) or symbols (SYM). Text, numbers and symbols are accessed using the Abc, 123 and SYM keys on each of the respective keypads.
- 3 Entries are confirmed by pressing the "Accept this text" button on the command toolbar.



5.2.2 Numerical Entry Field



- 1 Selecting a Numerical Entry Field opens the numeric keypad interface.
- 2 The numeric entry field can only be populated with numbers.
- 3 Entries are confirmed by pressing the "Accept this number" button on the command toolbar.

5.2.3 Date Entry Field



- 1 Selecting a Date Entry Field opens the date keypad interface.
- 2 The date entry field is displayed in the format specified in the User Settings menu.
- 3 Press the arrows above and below each number to increase or decrease the value displayed.
- 4 Entries are confirmed by pressing the "Accept this date" button on the command toolbar.



5.2.4 Time Entry Field

- 1 Selecting a Time Entry Field opens the time keypad interface.
- 2 The Time Entry Field is displayed in the 24 hour format.
- 3 Press the arrows above and below each number to increase or decrease the value displayed.
- 4 Entries are confirmed by pressing the "Accept this time" button on the command toolbar.



5.2.5 Listed Options Field

- 1 Selecting a Listed Options Field opens the listed options keypad interface.
- 2 The options available in the Listed Options entry field is specific to each individual setting. e.g. The Temperature Units setting gives the options Celsius (°C), Fahrenheit (°F) and Kelvin (K).
- 3 Use the scrollbar to view the full list of available options for each setting.
- 4 The option that is required is selected by pressing on the required option button.

5.2.6 Drop Down List Field

Search by	Completed date		-	
0	Comp	Completed date		
Criteria	Samp	Sample name		
Date	Batch	Batch/reference		
	Filename			
	Start			
	End t			
Apply this search Cancel and go back				

5.2.7 Toggle Selection Field



- 1 Selecting a Drop Down List Field displays the options available for that field.
- 2 Arrows keys may be displayed to indicate that further options are available. Use the arrow buttons to view the additional options.
- 3 Press the required option from the list to select it.

- 1 Pressing a Toggle Selection Field will add or remove the check mark in that field.
- 2 A check mark indicates that the option is active.

6. Setting up New Methods

Touching the measurement section on the main menu screen will open the measurement menu where three options are available:



Create a new measurement: This option allows a new melt determination to be started. Measurement parameters such as the Sample Name, Start Temperature and Ramp Rate need to be entered when using this option.

Use a preset from the library: This option allows a new melt determination to be started using measurement parameters stored previously on the device.

Carry out a rapid-melt: The device will roughly determine the melting point of the sample using preset measurement parameters.

6.1 Creating a New Method

In the Measurement menu screen touch the "Create a new measurement" section of the screen.

Block Temp. 45.9°C Create New Met	14:42 Oct 20 2008 thod Standby				
Sample Name	Sample Name Abc				
Start Temperature	88 Abc				
Ramp Rate	1 123				
Touch here to enter additional information					
Continue	Cancel and go back				



Save as a preset method?	Yes
A preset method can be quickly selected, removing	
the need to enter method details each time.	No

- 1 Enter a sample name for the samples by selecting the Sample Name field
- 2 Enter an appropriate start or plateau temperature for the sample by selecting the Start Temperature field.
- 3 Enter the required temperature ramp rate for the samples by selecting the Ramp Rate field.
- 4 Optional sample and method information is accessed by pressing the "Touch here to enter additional information" button. If this is not required press "Continue" and go to step 8.
- 5 The SMP40 will automatically detect the end of the melt when the toggle selection field has an X in the Automatically detect the melt end field. Removing this check mark allows a value to be entered into the End Temperature field.
- 6 Enter any batch or reference details in the Batch/Reference alphanumeric entry field.
- 7 To proceed to the melt determination select "Go back to main information" and then select "Continue".
- 8 The option of saving this new method as a preset method will be given. Select "Yes" to save the method for future use or select "No" to proceed without saving the method.

6.2 Preset Methods

In the Measurement menu screen touch the "Use a preset from the library" section of the screen.

Block Temp. 23.1° Preset Method	21:29 24 Oct 2008 Standby					
Sort/Search Open	Сору	Rename	Delete			
Acetanilide	Start tempera Ramp rate Date created	ture 1 1° 22/10	10.0°C 'C/min /2008			
CAFFEINE		ture 2 19 22/10	27.0°C 'C/min /2008			
Sample Name	Start tempera Ramp rate Date created	ture 1º 1º 24/10	88.0°C °C/min /2008			
Chemical name		ture 10 19 24/10	05.0°C rC/min /2008			
Select preset and continue Cancel and go back						

Block Temp. 45.9°C 14:42 Oct 20 200 Create New Method Standb					
Sample Name	Sample Name Abc				
Start Temperature	88 Abc				
Ramp Rate	1 123				
Touch here to enter additional information					
Continue	Cancel and go back				



- 1 In the preset methods screen select the method to be used for the melt determination by pressing on the method details.
- 2 Press "Select preset and continue".

- 3 Method parameters such as the Sample Name, the Start Temperature and the Ramp Rate can then be adjusted if required by selecting the relevant field.
- 4 Optional sample and method information is accessed by pressing the "Touch here to enter additional information" button.

- 5 Method parameters can be adjusted if required. The SMP40 will automatically detect the end of the melt when the toggle selection field has an X in the "Automatically detect the melt end" field. Removing this check mark allows a value to be entered into the End Temperature field.
- 6 Enter any batch or reference details in the Batch/Reference alphanumeric entry field.
- 7 To proceed to the melt determination, select "Go back to main information" and then select "Continue".

Yes
No
Yes
No

- 8 The option of saving this new method as a new preset method will be given if the sample name has been changed. Select "Yes" to save the method for future use or select "No" to proceed without saving the method.
- 9 If changes are made to the temperature settings the user will be asked whether they wish to overwrite the existing preset method of that name. Select "Yes" to save the new method settings for future use or select "No" to proceed without saving the new settings.

6.3 Carry out a Rapid-Melt

If the user is unsure of the start temperature that should be specified in a melt determination method they can use the rapid melt function to quickly determine an approximate melting temperature.

In the Measurement menu screen touch the "Carry out a rapid-melt" section of the screen.



Block Temp. 59.0°C

Report

Cooling

Temperature range analysed
50.0°C to 114.8°C

Ramp rate used
20°C per minute
Created time
14:55
Created time
14:55
Created date
Oct 21 2008
Calibration factor
0.963

Intensity Graph of Tube 0

View the video

Close and go to results

1 The rapid melt function will heat the sample from 50.0°C at 20.0°C/min until the sample has melted.

- 2 The results will be reported on the screen as shown in the example opposite.
- 3 The results from the rapid melt analysis can then be used to facilitate the setting of the start temperature on a more accurate melt determination.

7. Performing Melt Determinations

Once the analysis method and sample information have been entered into the SMP40 software the instrument will display the New Measurement screen.



Figure 6: New Measurement Screen

The Capillary tubes melt status is for <u>Information Only</u>. An accurate analysis of the data is performed after the melt determination has been completed.



1 Place 1-3 prepared melting point capillaries into the SMP40 sample compartments sample block.

The external surface of the sample capillaries must be wiped clean before being placed into the sample block as chemical residues may build up and affect the performance of the device.

2 Press "Start the melt" to initiate the melting protocol.







- 3 The instrument status will change to "Heating" and the command toolbar options will change.
- 4 The SMP40 will quickly heat the sample block to the set start temperature.

5 When the sample block reaches the start temperature the SMP40 status will change to "Stabilising" and the temperature of the sample block will remain constant for 60 seconds.

- 6 After the instrument has stabilised at the start temperature the status will change automatically to "Analysing" and the instrument will begin to heat the sample capillaries at the specified ramp rate.
- 7 The Capillary tubes melt status is shown to indicate the melt determinations progress.

The Capillary tubes melt status is shown for Information Only.







8 As the melt determination progresses the capillary tube melt status changes through:

Solid: No changes have been detected in the samples physical form.

Melting: The sample is showing the first signs of a change in its physical form.

Liquid: The change in the samples physical form is complete.

Finished: Once a capillary tube displays "finished" the melt determination is complete.

Once all of the sample capillaries are deemed to have finished, or the instrument reaches the previously specified end temperature, the instrument will complete the measurement and display the samples analysis report.

At any time in the melt determination pressing the "Stop and go back" button will end the melt determination and save any data collected up until that point.

Once the melt determination is complete the analysis report will be displayed. The analysis report displays the following information:

Statistical Analysis: The mean and standard deviation are calculated for the clear point values in the melt determination.

Method Information: Sample name, Filename, Batch reference, Start and end temperatures, Ramp rate, Time created, Date created, and Calibration factor.

Individual Capillaries Results: Intensity graph, melting range and clear point.

8. Results

Touching the results section on the main menu screen will open the results menu. Two options are now available:



View previous results: Displays the result files of melt determinations previously carried out on the device which are currently located on internal storage.

Re-use result parameters: A result can be selected and a repeat melt determination carried out using the method settings from that result.

8.1 View Previous Results

In the Results menu screen touch the "View previous results" section of the screen. The View Results screen allows the user to perform a number of actions with the result files stored on the instrument. The actions available are displayed in the Results Toolbar.

		np. 27.8°C Results		13:33 Oct 21 Sta	2008 andby	
Individual sample results	Sort/Search Rapid Meas	Open urement	View Video	Rename	Delete –	Results toolbar
	Completed 10/16, Sample Nan Batth/Reference: None Completed 10/21,	/2008 //2008			calculated (°C): 91.5 Melting point calculated (°C):	
Figure 7: View Results Screen	Select result	and continu	ie Ci	ancel and g	o back	

The View Results screen allows the user to perform a number of actions with the result files stored on the instrument. The actions available are displayed in the Results Toolbar.

Sort/Search: The Sort/Search function is used to display the stored result files in accordance with the rules specified in this option.

Open: The Open action is used to display the Report screen for the selected result.

View Video: The View Video action is used to display the Video Replay screen for the selected result.

Rename: The Rename action is used to change the Sample Name for the selected result.

Delete: The Delete action is used to delete the selected result from the instruments internal storage.

8.1.1 Sort/Search Function

Sort/Search



	Block Temp. 26.9°C View Results				14:15 Oct 21 2008			
					Standby			
	Sort By	Date	Ba Refe	tch/ rence	Sample	Other		
New Sample Name					Filename			
Batch/Reference: None Completed 10/21/2008					Start Temperature			
Rapi	d Meası	irement						
				End Temperature				
				Ramp Rate				
					Hide this menu			
Select result and continue Ca					ancel and g	o back		



In the menus that display the stored methods and results the user has the option to organise the displayed data using either a sort by or search for function.

Sort:

- 1 Pressing the "Sort/Search" button will change the tool bar to allow the user to specify which function is required.
- 2 Pressing the "Sort By" button will display the sort toolbar options.
- 3 The sort toolbar allows a method or parameter to be selected to organise the displayed data.
- 4 Select the parameter to be used to organise the displayed data by pressing the appropriate button.
- 5 Pressing the button twice will reverse the order or the displayed data.
- 6 Highlight the required result file and press the "Select result (or preset) and continue" button.
- 7 The "Other" button on the toolbar can be used to access additional parameters such as Filename, Start Temperature, End Temperature and Ramp Rate with which to sort the displayed files.

Search

- 1 Pressing the "Sort/Search" button will change the tool bar to allow the user to specify which function is required.
- 2 Pressing the "Search By" button will display the search options screen.
- 3 Pressing on a data entry field allows the user to define the search criteria for that field.







- 4 The required option should be selected from the displayed list or alternatively enter the search criteria using the keypad that is displayed.
- 5 When the search criteria have been finalised, press "Apply this search".

6 Only the results that match the search criteria specified will now be displayed.

- 7 Pressing the "Sort/Search" and "Search" buttons again will display the search results summary and allow a new search to be started.
- 8 Press the "Search again" button to apply new search criteria.
- 9 Press the "Clear search" button to remove the specified search criteria.

8.1.2 Opening Results Files



When a melt determination is complete or a result file is opened, the screen will display the Report Screen.

The report displays the following information:

The mean clear point: The average of the sample capillaries clear point values is automatically calculated.

The standard deviation of the clear points: The standard deviation of the sample capillaries clear point values is automatically calculated.

Method information: The Sample name, Filename, Batch reference (if entered), Analysed temperature range, Ramp rate, Time created, Date created, and Calibration factor are all displayed in the analysis report.

Intensity graphs for each capillary tube: The device continuously measures each capillary tubes image intensity value during the melt determination. These graphs show how the intensity value changes throughout the melt determination. Coloured lines indicate the start (green) and end (red) of the melting range.

Recorded events for each capillary tube:

Automatic and any manually recorded events are displayed for each capillary tube.

Use the scrollbar to review the data contained in the report: Use the arrows to scroll up and down the report.

If a video record of the melt determination has been stored it can be reviewed by pressing the "View the video" button on the command toolbar.



8.1.3 Viewing Videos

When a video record is accessed the Video Replay screen is displayed.



Figure 8: Video Replay Screen

The Video Replay screen is designed to allow the user to review the recorded video of a melt determination and reprocess the results if required.

Block temperature at specific time: The temperature of the sample block at the specific timeframe displayed in the video replay window.

Report information: Pressing the "Touch for automatically calculated values" button displays the screen below. Each capillary tube has its start temperature, end temperature and clear point displayed. In addition, the mean value of the start and end temperatures is calculated and displayed.

Press the "Hide" button to return to the Video Replay screen.

Tagged event selection: During the melt determination additional events may occur that are required to be recorded in the report. Pressing this button accesses the list of available events that can be added to the analysis report.

Capillary selection: In order to record a manual event you must first select the number of the capillary tube it applies to by using these buttons.

Video replay window: The Video replay window displays the images that were recorded during the substances melt determination. To view the image in full screen mode press the video replay window. Pressing the screen again will return the device to the normal Video Replay screen.

Video controls: The Video controls are used to control the playback of the substances melt determination video.





- To start the video replay press the play button
 (►) in the video replay controls. Once the play button has been pressed it will change to a pause symbol (■).
- 2 The video replay scrollbar will move and display the elapsed time in the format mins:seconds.
- 3 The scrollbar can be used to quickly move to any timeframe within the video file. Touch the scrollbar and whilst still touching the screen, drag the scrollbar to the required timeframe.
- 4 Pressing the **X2** button will double the speed at which the video is replayed.
- 5 The restart button (**I**◀) can be pressed to return the beginning of the video file.

Manual Events

Each melt determination may contain events of interest that require highlighting in the report. The SMP40 allows the user to review the melt video and add defined event information to specific timeframes for each individual capillary tube. Both the automatically determined events and any manually added events are shown in the analysis report.

The Tagged Events field can be used to add the following events:

Clear point: The sample is a clear liquid. Solid particles are no longer visible.

Collapse point: The substance collapses and begins to liquefy.

Decomposition point: The separation of a substance into its elements or smaller compounds. Often seen as discolouration or charring of the sample.

End of melt: The point at which no further changes in the physical form of the substance are being seen.

Start of melt: Droplets of liquid are clearly visible for the first time.

Meniscus point: A meniscus becomes clearly visible at the surface of the sample but solid particles remain visible in the liquid.

Sublimation point: The substance undergoes a transition from the solid to gas phase with no intermediate liquid stage.

Other: Use this tag if an event occurs that is not accurately described by the events listed previously.







1 Select the capillary tube to which the manual event is to be added by pressing the relevant "Capillary selection" button.

- 2 Select the timeframe in the video file, where the manual event is to be added, using the video replay scrollbar and the video controls.
- 3 A manually determined event is added to the timeframe by pressing the "Tagged Events" field and selecting the required event from the drop down list.

Manual events can only be used once in each capillaries report. e.g. A capillary cannot have more than one Meniscus Point.

- 4 Additional manual events can be added by repeating the process described in steps 2 and 3.
- 5 If a manual event needs to be deleted, pressing the X next to the manual event will delete that event.
- 6 After the manual events have been added for each capillary tube select "Save manual overridden tags". The screen will then go back to the previously displayed screen.



8.1.4 Renaming Result Files





The melt determination report is updated with any manual events that are added and saved. An asterisk next to the event name is used to denote that it has been manually added by the user.

CAUTION: IF A MANUAL CLEAR POINT EVENT IS ADDED BY THE USER, THE TEMPERATURE VALUE IN THE MANUAL EVENT WILL BE USED TO CALCULATE THE SAMPLES REPORTED MELTING POINT.

- 1 When the "Rename" button is pressed for a result a rename dialogue box is opened.
- 2 Press the alphanumeric entry field (Abc) in the dialogue box to enter a new Sample Name for the selected result.

3 The new Sample Name is then displayed in the alphanumeric entry field. Select "OK" to confirm the change to the results Sample Name. Select "Cancel" to retain the current Sample Name.

8.1.5 Deleting Result Files



- 1 To delete individual results from the instruments memory highlight the result to be deleted and press the "Delete" button on the Result Toolbar.
- 2 A confirm delete dialogue box will appear asking the user to confirm the action. Select "Yes" to confirm or "No" to cancel.

8.2 Re-use Result Parameters





In the Results menu screen touch the "Re-use result parameters" section of the screen.

- 1 In the Repeat Measurement screen highlight the result file to be repeated.
- 2 Press "Select result and continue".
- 3 The method parameters used previously will be loaded into the Create New Method screen.
- 4 Method parameters and information can be 4 adjusted and a new measurement initiated as described on page 14.

9 File Management

Touching the File Management section on the main menu screen will open the File Management menu. Two options are now available:



Manage files: Result files can be copied, moved or deleted from the devices internal storage.

View storage information: Displays status information on the internal storage or an inserted USB flash drive.

9.1 Manage Files

In the File Management menu screen touch the Manage files section of the screen.

	Block Temp. 28.3°C Manage Files	15:54 Oc	Standby	
	Sort/Search Select All	Copy Move	Delete	—— Manage files toolbar
Individual ——— sample results	Rapid Measurement () 10/16/2008 Melted at 223 <i>Rapid Measurement (1)</i> Rapid Measurement () 10/21/2008 Melted at 93	7.4°C	ame () Melted at 91.5°C	
Figure 9: Manage Files Screen	Cancel	and go back		

The View Results screen allows the user to perform a number of actions with the result files stored on the instrument. The actions available are displayed in the Result Toolbar:

Sort/Search: See page 18

Select All/Clear Selection: The Select All/Clear Selection action is used to select all of the result files on the instrument or it can be used to deselect the result files currently highlighted.

Copy: The Copy action is used to copy the selected result files to the inserted USB flash drive.

Move: The Move action is used to move the selected result files to the inserted USB flash drive.

Delete: The Delete action is used to delete the selected result files from the instruments memory.

9.1.1 Select All/Clear Selection



9.1.2 Copying Files



9.1.3 Moving Files



- 1 Pressing the "Select All" button in the Manage Files toolbar will highlight all of the result files.
- 2 If any of the result files in the Manage Files screen are selected, the "Select All" button will change to a "Clear Selection" button. Pressing the "Clear Selection" button will deselect all the highlighted result files.

- 1 Result files can be copied to an inserted USB flash drive by highlighting the files to be copied and pressing the "Copy" button in the Manage Files toolbar.
- 2 A window is displayed to show the progress of the copy action.

Copying a typical 5MB result and video file will take approximately 10 seconds.

- 1 Result files can be moved to an inserted USB flash drive by highlighting the files to be moved and pressing the "Move" button in the Manage Files toolbar.
- 2 A window is displayed to show the progress of the move action.

Moving a typical 5MB result and video file will take approximately 10 seconds.

Block Temp. 28.0°C 15:58 Oct 21 2008 Manage Files Standby				
Sort/Search	Select All	Сору	Move	Delete
Cancel and go back				

9.1.4 Deleting Files



3 Once the move action has been completed the files will no longer be available on the instrument so ensure that no further reprocessing of the results is required before using the move action as it is not possible to move the file back, from the USB flash drive to the instrument, once the action is complete.

- 1 To delete results from the instruments memory highlight the results to be deleted and press the "Delete" button on the Manage Files Toolbar.
- 2 A confirm delete dialogue box will appear asking the user to confirm the action. Select "Yes" to confirm or "No" to cancel.

9.2 View Storage Information

In the File Management menu screen touch the View storage information section of the screen.



1 The Storage Details screen displays information on the status of the internal memory.

Information displayed includes:

Total storage capacity.

Available storage capacity.

Details on the types of files stored on the instrument.

2 If a USB flash drive is inserted, a summary of the flash drives storage information can be displayed by pressing the Change to inserted USB flash drive section of the screen.

10 Settings and Calibration

Touching the Settings and Calibration section on the main menu screen will open the Settings and Calibration menu. There are three options available:



Change user settings: Settings that relate to how the device operates and displays information can be viewed and adjusted in this menu.

Change maintenance settings: The devices software can be upgraded and the touch screen can be recalibrated in this menu.

Manage device calibration: This menu can be used to recalibrate the device and view stored information on the devices calibration history.

10.1 User Settings

The following user settings can be accessed and adjusted by pressing the Change user settings section of the Settings and Calibration screen.

Date format: The devices displayed date can be set to UK or US format.

Time format: The devices displayed time can be set to 12 or 24 hour format.

Temperature unit: The devices displayed temperature unit can be set to centigrade (°C), fahrenheit (°F) or Kelvin (K).

Camera gain: The devices camera gain can be adjusted to make the image of the sample capillaries darker or brighter. Three settings of Low, Normal and High are available. The "Low" settings will reduce the brightness of the image compared to the "Normal" setting and the "High" setting will increase the brightness of the image compared to the "Normal" setting. The "Normal" setting should be suitable for most melt determinations.

Calibration frequency: The frequency at which the device is recommended to be recalibrated can be specified using this option. The available options are Never, Weekly, Monthly, Bi-annually and Yearly.

When a melt determination is initiated, the dialogue box below will be displayed if the time period specified in this setting has elapsed since the previous calibration. Pressing "OK" will allow the user to continue with the melt determination.

The dialogue box will be displayed each time a melt determination is initiated, until the device is recalibrated or the calibration frequency setting is changed.



Set time: The devices displayed time can be set using the time keypad interface.

Set date: The devices displayed date can be set using the date keypad interface.

Save melt video: If the video files of the devices melt determinations do not need to be stored, remove the check mark in this Toggle Selection field.

Use sounds for events: If sounds are not required for events that occur on the device, remove the check mark in this Toggle Selection field.

Use sounds for screen touches: If a sound is not required to confirm each time the touch screen is pressed, remove the check mark in this Toggle Selection field.

Block Temp. 21.9°C Change User Set	09:02 0 ttings	ct 22 2008 Standby	A
Date format	US format	E	1
Time format	24-hour	:=	
Temperature unit	Celsius (°C)	:=	
Camera gain	Low	:=	
Calibration frequency	Never	E	• ack
Set time	09:02:01	\odot	
Set date	Oct 22 2008	1 23 8 9 10 15 16 17 1	
Save melt video			
Use sounds for events			
Use sound for screen to	uches		

In the Settings and Calibration menu screen touch the Change user settings section of the screen.

- 1 The Change User Settings screen displays the current status of all the user adjustable instrument settings.
- 2 To adjust any of the displayed settings, touch the individual settings control field and a new setting can be entered by following the control instructions on pages 11 and 12.
- 3 When the required adjustments have been made press "Save these settings" on the command toolbar to apply them.

Block Temp. 21.9°C Change User Set	09:02 Oc ttings	t 22 2008 Standby	fi
Date format	US format	:=	
Time format	24-hour	:=	
Temperature unit	Celsius (°C)	E	
Camera gain	Low	:=	
Calibration frequency	Never	:=	✦
Save these setting	s Cancel and	d go ba	ack

10.2 Maintenance Settings

In the Settings and Calibration menu screen touch the Change maintenance settings section of the screen. The Maintenance screen allows the user to upgrade the version of software on the instrument and to recalibrate the touch screen.



1 To upgrade the instruments software, firstly download the new software version onto a USB flash drive. Insert the USB flash drive into one of the SMP40's USB ports and touch the "Press to upgrade" button. The new software version will be automatically installed onto the instrument.

Do not switch off the device or attempt to remove the USB flash drive during this process.

2 To recalibrate the touch screen touch the "Press to enter screen calibration" button and follow the on-screen instructions.

10.3 Instrument Calibration

To ensure that the instrument is operating correctly, periodic checks on the accuracy of the instruments temperature readings are commonly performed. If the accuracy of the instrument is found to deviate from the specification or if it is a requirement of your local quality policy, the following procedure can be used to recalibrate the displayed temperature on the instrument.

It is recommended that the recalibration procedure is performed using suitable certified reference materials (CRM's), as this will ensure the highest levels of confidence is maintained in the results generated with the instrument. The World Health Organisation (WHO) gives a list of suitable CRM's which includes the following materials:

Material Nominal Melting Point

Azobenzene	69 °C
Vanillin	83 °C
Benzil	96 °C
Acetanilide	116 °C
Phenacetin	136 °C
Benzanilide	165 °C
Sulfanilamide	166 °C
Sulfapyridine	193 °C
Dicyanodiamide	210 °C
Saccharin	229 °C
Caffeine	237 °C
Phenolphthalein	263 °C

For information on where to purchase suitable certified reference materials please contact us:

stuarthelp@bibby-scientific.com

Tel: +44 (0)1785 810433

In the Settings and Calibration menu screen touch the Manage device calibration section of the screen.



Figure 10: Calibration Screen

The Calibration screen shows the following information:

Current calibration factor: The current calibration factor of the device

Calibration type of the current calibration: Three types of calibration are possible: Single, Dual and Factory.

Time and date of current calibration: Displays the time and date of the current calibration.

Date when next calibration is due: Displays the date when the next calibration is due to be performed as specified in the user settings.

Previous calibration history: Displays a summary of the devices historical calibrations including the time, date, type and calibration factor.

10.3.1 Current Calibration Factor

The current calibration factor shows the factor which is used to calculate the displayed block temperature on the device.

Displayed Block Temperature = Instruments Sensor Temperature x Calibration Factor

To calculate a new calibration factor the instrument uses the following equation.

New Calibration Factor Calculation:

Calibribration = Expected Melting Point Factor $\left(\frac{\text{Actual Melting Point}}{\text{Results Calibration Factor}}\right)$

Please see the following calibration procedure for more details on calibrating the instrument.

10.3.2 Calibration Procedure

The SMP40 allows the user to perform either a single or dual point calibration by using the following procedure.

- 1 Using a suitable certified reference material, perform a melt determination as described in section 7.
- 2 If a dual point calibration is required, perform a second melt determination on a certified reference material with a different melting point to that used in step 1.
- 3 If manual events need to be added to a result file, they should be added using the steps described on page 22 before starting the recalibration procedure below.
- 4 In the Settings and Calibration screen select the Manage device calibration section of the screen.
- 5 In the Calibration screen press the "Calibrate this device" button on the Command toolbar.
- 6 The Set Calibrated Values screen is used to select the result files that will be used to calibrate the instrument.
- 7 Select the first result to be used by pressing the "Choose Result" field next to Value 1.

Block Temp. 106.7°C Set Calibrated V	2 10:3 alues	88AM Oct 22 2008 Cooling	f
Calculated Calibration Factor	Value 1	Choose Re	sult
	Expected	0	123
Previous Calibration Factor 1.0000			
Choose one or two previously completed melt measurements	Value 2	Choose Re	sult
and enter the expected values to have the device calculate a calibration factor.	Expected	0	123
Apply this calibration	n Cano	el and go ba	ick

Block Te	mp. 82.3° (C 1	.0:39AM Oct 22	2008
Selec	t Calibrat	ion	C	ooling 🎞
Sort/Search	Open	View Video	Rename	Delete
VANILLIN				79 7
Batch/Reference: S139 Completed 10/22	996-033 2/2008			Melting poir calculated (°C)
BENZANILI	DE			155.7
				Melting poir calculated (°C)
CAFFEINE				775 1
Batch/Reference: 134 Completed 10/22				Melting poir calculated (°C)
Select resul	t and contin	ue C	ancel and g	o back





- 8 In the Select Calibration screen highlight the result file to be used for the first calibration point.
- 9 Press the "Select result and continue" button on the Command toolbar.

- 10 The results melting point (mean clear point) is now displayed in the field. If the selected result needs to be changed press this field again and go to step 8.
- 11 If a dual point calibration is required press the "Choose Result" field next to Value 2 and repeat the selection process described in steps 8 to 10.

- 12 Enter the certified melting point (clear point) of the reference material/s in the respective "Expected" numerical entry field/s.
- 13 The new calibration factor is calculated and displayed on the screen.
- 14 To accept the new calibration factor press the "Apply this calibration" button on the Command toolbar.





15 The calibration screen is now updated to show the new calibration details. The Calibration history window will now update to display details of the previous calibration.

16 If a calibration record needs to be deleted, pressing the X next to the calibration record will display a confirm deletion dialogue box. Press "YES" to confirm or "NO" to cancel.

11. Maintenance & Servicing

WARNING: Ensure the unit is disconnected from the mains electricity supply before attempting maintenance or servicing.

This equipment does not require routine servicing. The only maintenance required is to clean internal and external surfaces using a damp cloth and mild detergent solution. Do not use harsh or abrasive cleaning agents.

11.1 Repairs and Support

Any repairs or replacement of parts MUST be undertaken by suitably qualified personnel. Only spare parts supplied or specified by Stuart or its agents should be used. Fitting of non-approved parts may affect the performance and safety features designed into the instrument. For a comprehensive list of parts required by service engineers conducting internal repairs please contact the service department quoting the model and serial number:

Email: <u>service@bibby-scientific.com</u> Tel. +44 (0)1785 810475 Fax: +44 (0)1785 810471

For any other technical enquiries please contact the Technical Support Department at;

Email: <u>stuarthelp@bibby-scientific.com</u> Tel: +44 (0)1785 810433

11.2 Cleaning the Sample Block

Occasionally it may be necessary to gain access to the sample block to allow broken capillary tubes or other debris to be removed.

Please ensure that the sample block is cooled to room temperature and the instruments electrical power supply is switched off or disconnected before commencing the following procedure.

1 Lift the cover of the capillary storage compartment and remove the two screws on either side of the recess.

- 2 Remove the plastic cover from the instruments melt module.
- 3 Remove the ceramic sample block cover and the metal plate covering the sample block chamber by lifting them clear of the instrument.
- 4 Unscrew the two thumbscrews on either side of the sample block and lift it out of the instrument. Take care not to damage the wiring that is connected to the sample block during its removal.
- 5 Using a pair of long nosed pliers or small tweezers remove the circlip from the sample blocks glass window.
- 6 The glass window is removed by gently agitating the sample block until the window is dislodged form the sample block.
- 7 Debris can now be removed from the sample block. Please ensure the window is clean before reassembly as any grease or dirt on the glass could be detrimental to the instruments performance.
- 8 Reassembly is the reverse of the above.

11.3 Accessories

The following accessories are available from your local distributor.

Description	Catalogue No.	
Capillaries, open at both ends (pack of 100)	SMP1/4	
Capillaries, open at one end (pack of 100)	SMP10/1	
Capillaries, closed at both ends (pack of 100)	SMP2/1	

12. Warranty

Stuart warrants this instrument (**excluding the VGA touch screen**) to be free from defects in material and workmanship when used under normal laboratory conditions, for a period of 3 years (**the VGA touch screen warranty period is 12 months**). In the event of a justified claim Bibby Scientific Ltd will replace any defective component or replace the unit free of charge. This warranty does NOT apply if damage is caused by fire, accident, misuse, neglect, incorrect adjustment or repair, damage caused by incorrect installation, adaptation, modification, fitting of non-approved parts or repair by unauthorised personnel.

Bibby Scientific Ltd. Stone Staffordshire, ST15 0SA United Kingdom Tel: +44 (0) 1785 812121

Fax: +44 (0) 1785 813748 E-mail: equipment@bibby-scientific.com Website: www.bibby-scientific.com

13. Specification

Overall Dimensions:

Height: 180mm Depth: 210mm Width: 340mm Weight: 3.9kg

Technical Specifications:

Temperature range	Ambient to 400°C
Temperature resolution	0.1°C
Ramp rate	0.1°C to 10°C/minute
Cooling time	350°C to 50°C in 10 minutes
Number of samples	Up to three
Display	5.7" Colour VGA touch screen
Sensor	PT1000
Memory storage	1GB
Electrical supply	115V / 230V, 50-60Hz

Notes

Notes

This product meets the applicable EC harmonized standards for radio frequency interference and may be expected not to interfere with, or be affected by, other equipment with similar qualifications. We cannot be sure that other equipment used in its vicinity will meet these standards

and so we cannot guarantee that interference will not occur in practice. Where there is a possibility that injury, damage or loss might occur if equipment malfunctions due to radio frequency interference, or for general advice before use, contact the Technical Department of Bibby Scientific Ltd.

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Declaration of Conformity			
Automatic Melting Point Apparatus, Model SMP40			
These products comply with the requirements of the EU Directives listed below:			
2004/108/EC EMC Directive.			
2006/95/EC Low voltage Directive (LVD)			
Compliance with the requirements of these Directives is claimed by meeting the following standards:			
EN 61326: 1997 + Amendments (Electrical Equipment for Measurement, Control and Laboratory use) EN 61010-1: 2001 (Safety Requirements for Electrical Equipment for Measurement, Control and Laboratory use) EN 61010-2-010: 2003 (Particular Requirements for Laboratory Equipment for heating of materials) CE mark affixed '08. Signed: (Mr D. E. Hicks CEng MIEE)			
Date: <u>10th Decess to 2008</u> Authority: Technical/Development Manager			
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