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MELTING POINT APPARATUS

CAT NO. CH0801

Instruction Manual

MELTING POINT APPARATUS

INTRODUCTION

1. GENERAL DESCRIPTION

The instrument is designed for the determination of single or mixed melting points of solids up to a maximum temperature of 350°C.

In use the sample is contained in a capillary tube which is placed with a thermometer in a heating block within the apparatus. Internal illumination allows the melting process to be viewed through a magnifying lens on the front of the case and when this occurs, the melting point temperature is then read off from the thermometer.

Heating System

The aluminium heating block has a through-hole with a glass window at each end to prevent air convection. Heating elements are clamped to either side of the block and are controlled via a solid-state circuit by a variable-heat control knob on the front panel. A rapid-heat switch is fitted to give full power when heating up and the variable-heat control enables fine adjustment of the heating rate as the melting point temperature is approached.

Illumination and Viewing

A lamp at the rear of the heater block provides bright and shadow less illumination of the sample tubes. Viewing is through a full field magnifying lens. A light or dark background can be selected using the slide knob on the rear of the casting to obtain optimum contrast for the sample.

Thermometers

The thermometer selected should be calibrated for 30mm immersion and should be a reasonably close fit in the hole in the top of the heating block. Suitability and accuracy can best be established by carrying out trial melting point determinations on standard substances of known melting points. These substances can also be used for checking replacement thermometers and students technique. (Note: Thermometers are not supplied with the Melting Point Apparatus).

2. INSTALLATION

Thermometer Support

A thermometer support is provided, and this should be fitted on the back of the apparatus using the two crosshead screws provided.

3. CONTROLS

Mains Switch

Main Switch is provided at the rear of the unit for switch is the apparatus ON & OFF.

Variable heat Control

The unit has a large Knob in the front of the case for heat control. (See Figure 1.) This Knob is basically a potentiometer controlling the amount of energy delivered to the block and has a range suitable for operation front room temperature up to 350^0 C.

A guide to the setting required for a given Melting Point temperature can be obtained from figure 3.

Note : - Control point 'O' does not equate to zero input to the heater. Left at zero the block temperature may rise up to 50^0.

It is important to therefore switch off the apparatus when not in use.

1 amp. Rapid Heat Switch

This is located on the front panel next to the heat control knob. When the switch is set to the 'ON' position the lamp within its bezel will light. The variable control is overridden and maximum power is delivered to the heaters.

Important note

This power is greater than that obtainable from the variable control set to maximum. Thus it will give the fastest heating but, for the same reason it must not be left on for longer than required because the block or its heaters may be damaged by overheating.

Background Screen Control

The background color is controlled by the small button on the rear of the casting. It slides from side to side, giving a light or dark background as required to give optimum contrast for the sample.

NOTE: If working at elevated temperatures for long periods, care should be taken not to touch the area of the heating block.

4. DETERMINING MELTING POINTS

Melting Point determination is in principle straightforward but optimum results will only be obtained through the exercise of care and correct technique.

Basic Procedure

Load sample crystals into a capillary tube to the depth of about 2mm. and tap the bottom of the tube gently on the bench a fixed number of times; say five times. Sample depth and packing can affect the result and should be kept as consistent as possible.

Insert the sample tube (s) into the top of the heating block and insert empty sample tubes into any holes not being used.

If the approximate melting point of the sample is not known it will be quickest to find it by heating the block rapidly - turn the control to 6 or 7 initially - and noting where the sample melts.

Remember that at high heating rates a difference will exist between the thermometer reading and the sample temperature of up to as much as 15°C when rapid heat is in use. The apparent melting temperature is usually a little high.

When the approximate melting point is known, the graph of Figure 3 can be used as a guide to the variable heat control setting required to achieve a temperature rise of 1°C/ minute at the given temperature; a more precise setting must be found by experiment - see section “Heat rate and controls”.

Once the control is set, the block will begin to heat. If the block temperature is more than 15-20°C lower than the expected melting point, the rapid heat switch may be used to increase the heating rate. There is a short delay in response time and a difference between the sample temperature and the thermometer reading will develop. Thus it is easy to exceed the required temperature. With a little practice the right technique can soon be acquired.

NOTE: **The rapid heat switch must not be left on for prolonged periods.**

Once a suitable heating rate has been established, the melting event may be observed through the lens and the temperature recorded. See also section “Melting points of Solids”.

Cool the heating block appropriately for the next sample. If this is a repeat or similar sample it is only necessary to drop the temperature a few degrees. If the next sample has a considerably lower melting point a considerable saving in time may be achieved by inserting the water cooled plug available as an accessory.

Melting Points of Solids

It must be realised that even with very slow heating rates the melting event of a substance does not occur instantaneously but over a small range of temperatures. It is normally possible to distinguish two points in the process with a reasonable degree of consistency.

These are:

A) The meniscus point
Definable as the temperature at which partial liquefaction occurs and which is indicated by the formation of a define meniscus between the sample crystals and the capillary tube.

B) The liquefaction point
Indicated by the final disappearance of the solid crystals within the molten sample.

Sample Preparation

It has already been mentioned that the depth of sample and its packing density in the capillary tube can affect the results obtained and that care should be taken to achieve consistency. In addition, for optimum results the sample should be in the form of a finely divided power which should be dried in a suitable desiccator for 24 hours if it is at all hygroscopic.

Heat Rate and Controls

Generally speaking, the lower the rate of rise of temperature at the melting point, the more accurate will be the result. This is because of the different delay times involved in heat transfer to the sample and to the thermometer. In practice a compromise can be reached and a heating rate in the range 0.5°C/min to 2°C/min will normally be suitable, according to the accuracy required. Note, however, that the same rate must be used for comparative work on similar samples. The rate of 1°C/min suggested previously represents a rate which, for most samples, gives a satisfactory result in a reasonable time. The graph in Figure 3 gives the approximate dial settings required to achieve a 1° /min rate at given temperature.

Precise settings vary slightly for different apparatus, supply voltages and ambient temperature and must be determined for each installation. The rapid heat switch can be operated for a few seconds to raise the block temperature one or two degrees or it can be left on for longer if the temperature is to be raised through a larger Interval. Either way, there will be a small delay time, whose characteristics will quickly be learnt through experience. Also, as previously mentioned, the sample and thermometer temperatures will only be equal if the heating rate is vary low. The rapid heat switch will, therefore, need to be turned off up to 15 °C before the melting point, depending on how long it has been on for and what rise rate has been attained. The switch off point and setting of the variable control should be chosen so that the heating rate falls to the required value, (normally 1°C/min) as the melting temperature is approached.

Higher temperature considerations

At temperatures below about 150° C thermal equilibrium is readily established. Above this, however heat loss by radiation begins to become significant and as the temperature gradient between the block and its surroundings also builds up, small temperature gradients may occur inside the air bath.

For this reason, at higher temperatures small discrepancies in results may be noticed in the different tube positions. To obtain the highest accuracy it may be necessary to calibrate each position with a sample of known melting point, although even at 350°C, the discrepancy is unlikely to exceed 1°C.

Materials which sublime

Some materials sublime and so when testing these, the sample should not be put into the block until perhaps 10°C before its melting point, otherwise it may disappear before it melts. With some materials a better result can be obtained by the use of a smaller sample, e.g. 1mm deep, in the bottom of the tube.

Mixed melting points

The apparatus is eminently suitable for the identification of organic substances by the method of mixed melting points. Three capillary tubes can be observed at the same times enabling the whole determination to be carried out in one heating run. The procedure when using three tubes is similar in all other respects to that when a single tube is used.

5. SERVICING

BASIC EQUIPMENT MAINTENANCE MUST BE CARRIED OUT BY A QUALIFIED /COMPETENT ELECTRICIAN.

Viewing Lamp

This is located behind the heater block under the top casting. It is supplied at well below its nominal rated voltage and should have a good lifetime. Should it fail, replace as follows.

WARNING

Although the lamp is a low voltage type its terminal are at high potential when the mains supply is switched on.

Loosen the four crosshead screws located between the fins of the top casting (they have retaining washers and will not lift out). Lift the casting from the base.

Carefully unscrew the old lamp and fit a replacement, making sure it is screwed fully in. Replace the casting and tighten down the four screws.

Heating Elements

Two heating elements, connected in series for 240v (and parallel for 110v), are clamped one on either side of the heating block. Should a heating element fail, the heating rate at a given control setting will fall considerably and the maximum temperature attainable will also drop significantly. If this should occur, a replacement heater block assembly is available from your usual distributor and can be fitted by a qualified person.

To do this proceed as follows.

Remove the casing as stated previously, then remove the 4 corner screws which secure the heater block assembly and the 2 nylon heat breaks.

Unscrew the top nut from the EARTH SCREW and lift off the earth lead terminal.

Lift off the top plate and turn over to reveal the terminal connecting strip for the heaters and lamp.

Noting carefully the connections on the connector strip, disconnect the old heater block assembly and connect the new heater assembly leads.

Re-fit the earth lead and re-assemble the heater block assembly and the 2 nylon heat breaks to the top plate, noting that the three small holes in the top of the heater block assembly should be positioned towards the front of the unit.

Warning - insure that the earth lead is replaced correctly and is firmly fitted in position. Re-fit the top casing.

Power Control PCB Calibration

Should the performance of the apparatus be found to differ from that shown on the heating curves, it may be desirable to adjust the power controller. This requirement is most likely to result from high or low local mains voltage, or following the repair of a circuit board.

RECALIBRATION OF THE POWER CONTROLLER BOARD

Disconnect the apparatus from power supply, then remove the plastic capillary tube holder by squeezing the right hand corners together and easing forward when the retaining teeth have cleared the case. (A little difficulty may be experienced and it may be found easier to release first the top tooth, and then the bottom one).

On removing the plastic capillary tube holder the controller PCB will be visible. On the PCB tube BLUE coloured / YELLOW coloured multi turn Preset are visible. Looking from front side **RHS** Preset is Rv2 meant for setting the lower level voltage 20V ± 2V A.C. & the left hand side preset is Rv3 meant for setting the higher level voltage i.e. 200V ± 2V A.C. Remove the top black casting as described earlier then the connector strips will be visible. On this strip only two connectors are there, where heater wires are connected (one wire for screw) (2 wires per screw for 120V).

Connect these two terminals to an Avometer (Analog meter). set to 250V A.C. range., Using insulated probes, and taking great care to ensure that they do not touch each other or to any other metal part.

As described earlier make the position of apparatus such that RV2 & RV3 are easily visible and approachable.

* Make sure that everything is electrically safe, then switch on the apparatus & turn the dial setting to '0'. The meter should read 20 ± 2V A.C. (10V A.C. ±1 V A.C. for 120V version), if it does not, carefully adjust RV2 so that the meter read the required voltage.

* Turn the dial to setting to 10. The meter should read 200V ±2V A.C. (100V ±1V A.C. for 120VAC version), if it does not carefully adjust with RV3 so that the meter read the described voltage.

Please repeat the above steps* as described to recheck again.

Note: If the mains input supply is prone to variation these accurate settings may not be attained.

Switch off the apparatus and disconnect from the supply. Remove the probes and check all the wire are still firmly in place. Replace the top black casting and put the capillary tube holder back in position.

6. CLEANING

The outside of the Melting Point Apparatus needs to be cleaned occasionally. Proceed as follows:

1) Disconnect the apparatus from the electricity supply.
2) Wipe the surface with a mild detergent solution, such as washing up liquid.
3) Wipe dry and buff with a soft cloth.

Do not use solvents or abrasive cleaners on the apparatus !
Do not allow water to run in to the apparatus at the top of the unit !

7. CORRESPONDENCE

In the event of any correspondence concerning this apparatus, place quote the catalogue number and apparatus number given on the apparatus nameplate, also the voltage and frequency of the local mains power supply. This will help to avoid unnecessary delays.

8. SPECIFICATIONS

Overall dimensions	W x D x H: 120 x 155 x 210mm
	Height with thermometer tube
	guard attached: 390mm
Mass	Approximately: 1.9Kg
Power rating	50w max.
Recommended	Ambient to 350 °C
Operation	
Temperature range	
Humidity	MRH 80% up to 31°C decreasing
	linearly to 50% RH at 40°C
Altitude	Up to 2000M
Accuracy	±0.5°C at 0°C and ±2°C at 350°C
Fuse (5 dia x 20L)	1 amp

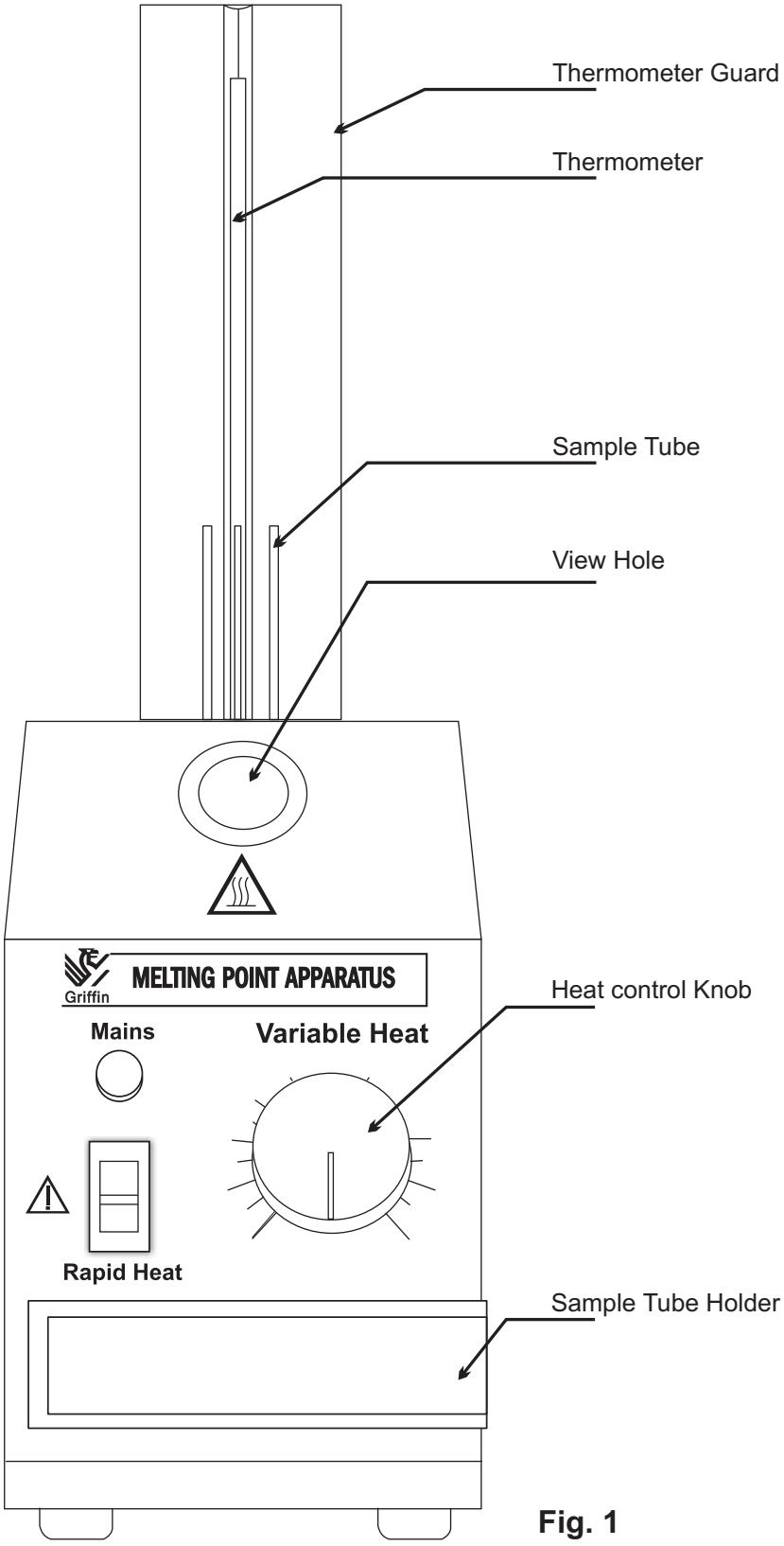

Fig. 1

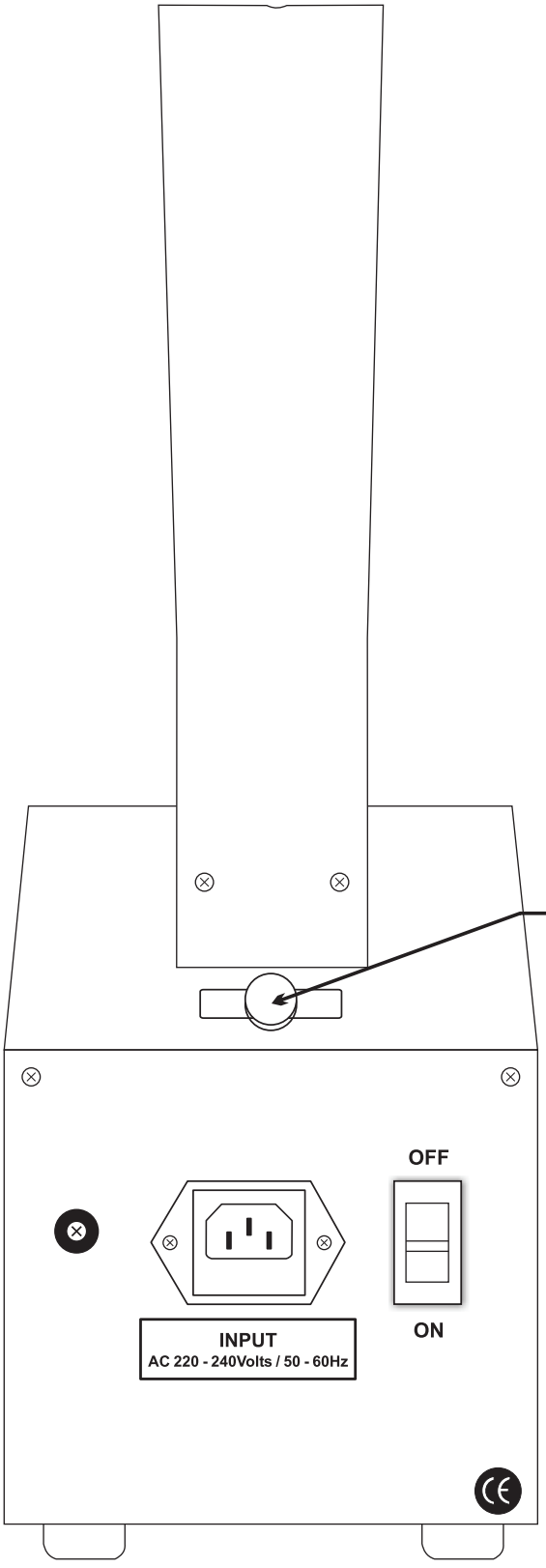

Fig. 2

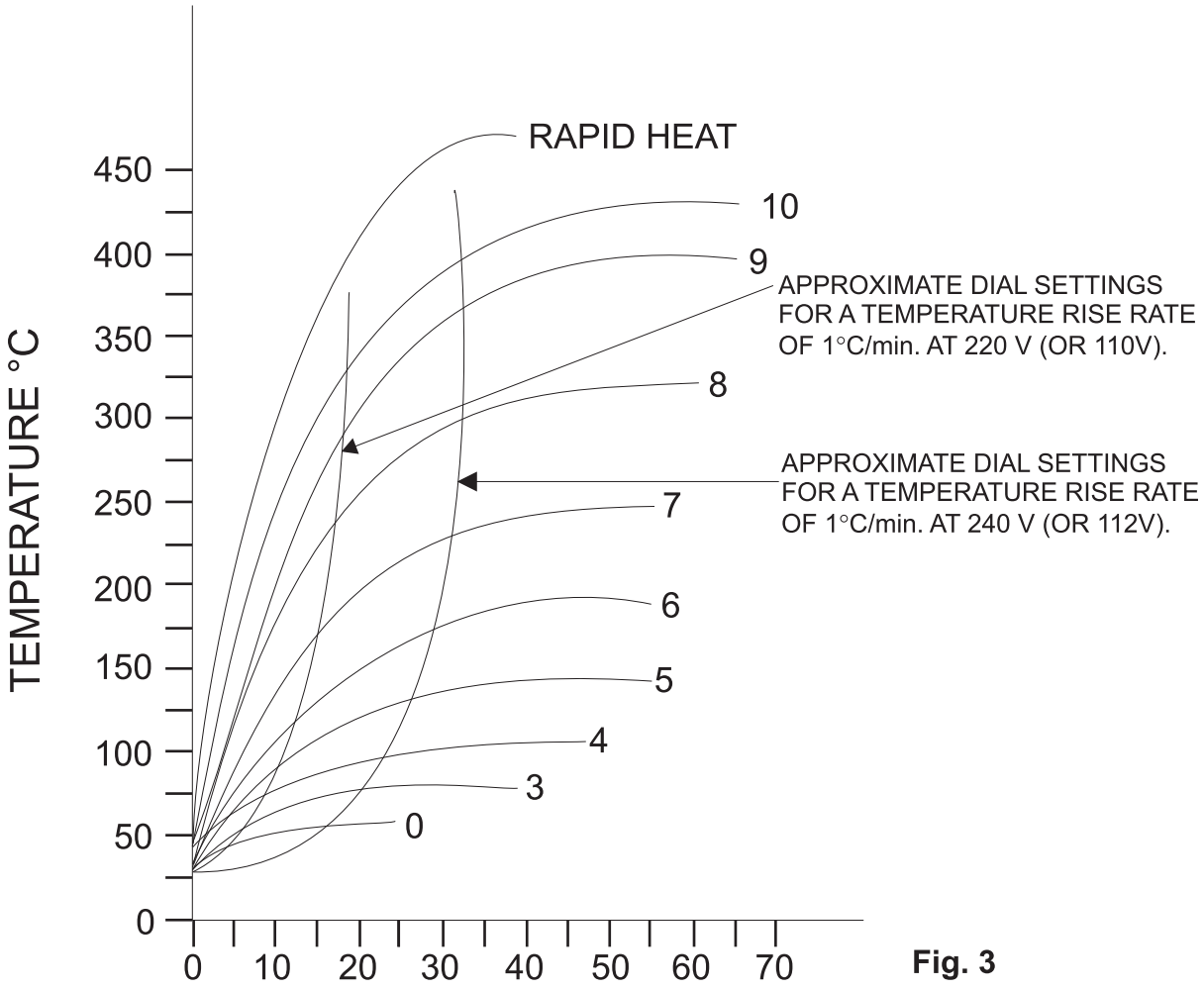

Fig. 3

Manufactured by :
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