A Safety Guide for using Laboratory Heating Blocks

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Heating blocks provide a safe, convenient and productive alternative to heating mantles and oil baths for heating round bottomed flasks, tubes and vials. Used in combination with a standard hotplate stirrer, heating blocks such as the Asynt DrySyn® Range have proved themselves with their ability to outperform the heat-conducting properties of oil baths. They pose a far lower fire risk and their use makes the clean-up of glassware far easier as there is no residual oil contamination on the outside of the flasks. In addition to accelerating your chemical reactions, heating blocks ensure a safer, cleaner, healthier working environment.

However solid aluminium heating blocks can reach temperatures of over 250°C. Therefore care needs be taken not to touch the units, or inserts, at any time unless you are positive that they are cool. The nature of these heating blocks is such that the equipment will remain hot and hold the heat for some time. Larger heating blocks that have been used at high temperatures could stay hot enough to cause a burn even after over half an hour has elapsed. Ideally users should use a thermocouple probe in the heating block to monitor temperature during the cooling phase. However in the absence of a thermocouple probe it is recommended that any block that has been heated should be left for a minimum of one hour. In the case of Asynt DrySyn® Heating Blocks you could also use an additional DrySyn® Safety Heat Sticker which shows when the heating block has cooled sufficiently to touch without causing a hazard. This document has been written to provide laboratory staff with an **IN BEST PRACTICE GUIDE** to safe use of heating block systems.

Designed to fit all standard hotplates, in multiple configurations

A: SETTING UP YOUR HEATING BLOCK APPARATUS

1. Assemble and set up the heating block including the attachment of any safety handles to the block as per the manufacturer's instructions.

With the DrySyn® Heating Blocks, the handles simply screw into either side of the base

2. Locate the heating block, or base unit, on a hotplate stirrer to manufacturer's instructions. (Please note that some manufactures heating blocks have an adjustable location system to allow secure locking to various hotplate manufacturers.)

3. If the design allows interchangeable inserts, then select the appropriate size for the flask being used and carefully place into the base.

4. If available, place the hotplate stirrer's temperature probe into the heating pocket of the block.

B: SETTING UP YOUR REACTION

1. Inspect your glassware

Please ensure glassware is free of star cracks and visible scratches. Flasks can become significantly weakened by etching which occurs with repeated use to all glassware. Please be advised that etched glassware should not be used in solid heating blocks. Flasks that have had star cracks repaired in the lower body also should not be used.

2. Ensure your flask fits properly

Ideally use a gauge such as Asynt DrySyn® Flask Size Gauge to check your flask conforms to the required ISO standard size

When selecting a flask, ensure that the flask fits correctly by rolling it around in the insert. If the flask feels tight, scratches the side of the block, or feels like it is sitting on an edge, the flask may be too large. Choose another flask.

As of 2005, all round bottom flasks manufactured in Europe should conform to ISO 4797. However this ISO standard may not be conformed to by manufacturers outside of Europe, the USA and Canada, especially in locations such as India and China. Japan also has their own method of standardisation called JIS.

Flasks should be manufactured to ensure that the outside diameter of the body does not exceed the stated ISO outside diameter. You will find that some heating block manufacturers produce their units with a tolerance to allow some oversize from standard. This prevents the cracking of any glassware within the block. There is generally no marking on the individual flasks so you should check with your supplier about the flasks you are purchasing, or check your flask against a gauge to be sure. Some manufacturers will provide you with size guides for your round bottomed flasks. This will enable you to see if they abide to ISO standardisations

Heater Block adapter size	Max flask outside diameter	ISO Specified Outside Diameter	Covered by ISO 4797
50ml	51.4 mm	None	No
100ml	65.2 mm	64 mm	Yes
250ml	85.4 mm	85 mm	Yes
500ml	105.5 mm	105 mm	Yes
1000ml	131.3 mm	131 mm	Yes

Round bottomed flasks under 100mls don't abide to ISO standards, however ALL glassware must be checked, for proper fit in the block, prior to heating.

3. Carefully place the correct sized stirring bar into the round bottomed flask.
(Do not drop stirring bars into the flask as this can cause breakage!)

4. Clamp your flask securely at the neck

Although the flask is supported from beneath, it is also advised to clamp the neck to stop the flask from tilting. If using a condenser, it is essential to support the flask with a clamp to hold the weight of the condenser and reduce the pressure on the flask.

5. Use a lab jack if available

We strongly advise that you support the hotplate stirrer and heating block on a lab jack. This allows the ability to lower the heating block away from the flask when cooling post synthesis, as well as ensuring in an emergency the heat source can be easily separated from the reaction flask. Lowering the heating block away from the flask by just 2-3mm during cooling will ensure that in the unlikely event that an oversize flask (See point 2) has been used it will not jam in the block. If an oversized flask has been used it may jam in the heating block if cooled in situ.

6. Ensure that the flask is vertically positioned in the insert

   Flasks may not be spherical if inserted at an angle. You could increase stress on the flask if it is not vertically positioned as expansion at different temperatures will not be even within the heating block. This could then cause the flask to jam or crack.

7. Add a condenser or other glassware as desired. Solvents and reagents can be added more easily using a 2-neck flask.

8. Adjust the stirring speed to a suitable level for good mixing.

9. Perform a final visual and mechanical check to verify the integrity of your system set-up. Make certain that all clamps are tight, all supports are solidly placed, and that the equipment is positioned to allow the manipulations needed to run your chemistry.

C: START YOUR REACTION HEATING

1. Set the appropriate temperature on the temperature controller (if fitted) or via the hotplate control.

2. Do not set the temperature too high

   For low boiling solvents 5-10°C above the boiling point is sufficient for reflux. For higher boiling solvents 10-20°C above the boiling point will give good refluxing. Some hotplate stirrers offer the ability to control solution temperature by putting a probe directly in to the reaction medium.

3. Ensure that there is adequate coolant supply to the condenser to minimise loss of solvent. (See Julabo FL series of chiller circulators for further information)

4. If your operating temperature is above 150°C we recommend that you insulate the flask

   This reduces the thermal gradient across the glass and reduces stress on the flask at high temperatures.

Important note!

The maximum recommended temperature of some hotplate stirrers for prolonged operation is 250°C. Exceeding this temperature may reduce the life of the stirring hotplate, please check with the manufacturer

D: POST REACTION COOL DOWN

1. Please make your colleagues aware that the heating block could still be hot for some time after the hotplate has been switched off if you are leaving it unattended.

2. If available, it is recommended that you lower the hotplate stirrer and heating block from the reaction flask on a lab jack during cooling.

3. You may transport the heating block using the handles, however this is only recommended if the temperature of the block is less than 65°C. Otherwise, please use insulated gloves to avoid burns!

E: CONCLUSION AND ACKNOWLEDGEMENTS

This in best practice guide has been written in order to offer guidance for the safest way of using laboratory heating blocks. The guide includes contributions from acknowledged expert - Dr I Smellie, Senior Teaching Fellow, University of St Andrews Chemistry Teaching Laboratories (St Andrews, UK) and from Dr N Langerman of Advanced Chemical Safety, Inc, (California, USA) a leading consultant well-versed in the practical application of regulations and industry standards to achieve compliance and safety in a cost-effective manner.

You can find further information on the design, and range of heating blocks available from Asynt at http://www.asynt.com/product-category/chemistry/drysyn-range/

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