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 much 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 and therefore care needs be taken not to touch the units, or inserts, at any time unless you are certain that they are cool. The nature of these heating blocks is such that the equipment will retain the heat for some time and 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. 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** for the safe use of heating block systems.

All DrySyn® bases are supplied with adjustable feet to offer compatibility with any hotplate stirrer.

A: Setting Up Your Heating Block Apparatus

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

With the DrySyn® heating blocks, the handles simply screw into the base.

2. Locate the heating block, or base unit, on a hotplate stirrer, as per the manufacturer's instructions.

   All DrySyn® bases are supplied with adjustable feet to offer compatibility with any hotplate stirrer.

3. If the design allows interchangeable inserts, select the appropriate size for the flask being used and carefully place this 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 cracks and etching, which occur when the flasks are carelessly placed into glass washers or otherwise mishandled. 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 should also not be used.

2. Ensure that your flask fits properly.

   Use a gauge to check that your flask does not exceed the maximum size as specified by DIN ISO 4797.

   When selecting a flask, ensure that it fits correctly by rolling it around inside the heating block. If the flask feels tight, scratches the side of the block, or feels like it is sitting on an edge, it may be too large. In this case, choose another flask and check it accordingly.

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

Flasks should be manufactured to ensure that the diameter of the body does not exceed the stated DIN ISO 4797 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. In general, there will be 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 DIN ISO 4797 standardisations.

Round-bottomed flasks with a volume of less than 100 mL do not abide to DIN ISO 4797 standards, however ALL glassware must be checked, for proper fit in the block, prior to heating.

Heating Block Size	Max. Flask Diameter	DIN ISO Specified Diameter	Covered by DIN ISO 4797
50 mL	51.4 mm	N/A	No
100 mL	65.2 mm	64 mm	Yes
250 mL	85.4 mm	85 mm	Yes
500 mL	105.5 mm	105 mm	Yes
1000 mL	131.3 mm	131 mm	Yes

Round-bottomed flasks with a volume of less than 100 mL do not abide to DIN 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 advisable to also clamp the neck to stop the flask from tilting. If using a reflux 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 user to lower the heating block away from the flask when cooling, as well as ensuring that in an emergency the heat source can easily be separated from the reaction flask. Lowering the heating block away from the flask by just 2-3 mm during cooling will ensure that, in the unlikely event that an oversized flask has been used (see Point 2, overleaf), it will not jam and potentially crack in the heating 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 heating insert, as 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 in the heating block.

7. Add a condenser or other glassware as required. 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 setup. 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 panel.

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. If using a fluid-cooled reflux condenser, ensure that there is adequate coolant supply to minimise the loss of solvent.

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

Insulating the flask reduces the thermal gradient across the glass thereby reducing stress on the flask at high temperatures.

D: Post Reaction Cool Down

1. If you are leaving it unattended, please make your colleagues aware that the heating block could still be hot for some time after the hotplate has been switched off.
2. If available, it is recommended that you lower the hotplate stirrer and heating block away 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, Head of the School of Chemistry Teaching Laboratories at the University of St Andrews (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.DrySyn.com

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