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Evaluation of DrySyn MULTI and MULTI-M Reaction Blocks

Introduction

Asynt recently introduced the DrySyn Classic Reaction Blocks. These enable a standard stirring hotplate to be adapted to heat and stir a range of flask sizes from 1L down to 50mL. The DrySyn Classic comprises a machined aluminium base unit that accepts a standard 1L round bottomed flask and a set of inserts which accept 500mL, 250mL, 100mL and 50mL flasks respectively. This was followed by the DrySyn MAXI that uses the same idea, but the base unit accepts a 3L flask and additional inserts are provided to accept 2L and 1L flasks.

The DrySyn range has now been further expanded by the introduction of the DrySyn MULTI and DrySyn MULTI-M. These are multiple place units that allow the chemist to run 1,2 or 3 reactions simultaneously in round bottom flasks on scales up to 100ml (MULTI) or 250ml (MULTI-M). In addition other inserts are available for small vials etc and Asynt are able to supply custom inserts for special applications.

Equipment tested

Asynt DrySyn MULTI Reaction Block System comprising:
Base unit
Inserts for 100ml round bottom flasks

Asynt DrySyn MULTI-M Reaction blocks comprising:
Base unit
Inserts for 100ml and 250ml round bottom flasks

Note: All inserts (except the 250ml inserts) fit both bases. Additional inserts are available for round bottom flasks (50ml, 25ml and 10ml) and also for a wide range of vials and tubes.

Standard Schott and Quickfit round bottomed flasks

Heidolph MR 3001 K Stirring Hotplate fitted with EKT 3001 fuzzy logic temperature controller

Various stirring bars from Cowie Technology were also evaluated. These were:
Oval 001.635.13, 001.630.10
Pivot ring 001.330
Elliptical rare earth 001.2625RE
Cross shape 001.2402

Description

Like the standard DrySyn and DrySyn MAXI reaction blocks the DrySyn MULTI uses a standard stirring hotplate to stir the flask contents. Unlike the former the MULTI is (as the name implies) a multi position block so the stirring bars are not centrally placed over the hotplate (Unless performing one experiment at a time in the central position) nor are they around the periphery. The base fits over the top surface of the stirring hotplate with the 3 flask positions spaced at 120^o and is located securely with three pegs. Various inserts drop into cut-outs in the base to accept a wide range of round bottom flasks, vials and tubes. Currently inserts are available for 250ml r.b flasks (MULTI-M only), 100ml, 50ml, 25ml, 10ml and 5ml r.b flasks as well as several standard vials and tubes. Set up is very quick and a new 3-way clamp firmly and neatly secures all 3 flasks.

The base and inserts are all machined out of solid aluminium with a satin anodised finish that should be very durable under normal laboratory conditions. The components are machined to very close tolerances and the inserts are an excellent fit into the base, which is essential for good heat transfer. The base is designed to fit on stirring hotplates with a maximum hotplate diameter of 145mm (as is the Heidolph). It can therefore also be used with hotplates having a smaller diameter such as IKA who use a 135mm diameter hotplate.

The inserts are fitted with holes to accept a thermocouple probe or fuzzy logic temperature controller.

All the inserts are clearly marked with their appropriate sizes and there are warnings engraved on the base warning that it may be hot!

Experimental procedure

Initial evaluation of stirring bars

The stirring bars listed under '**Equipment tested'** were evaluated for stirring performance. The stirring bar was added to a single 250ml round bottomed flask containing water (125ml) and placed in position in one of the outer inserts in the DrySyn MULTI-M. The stirring speed was slowly increased and the stirring bar was evaluated for a) stirring performance and b) stability. The results are summarised in **Table 1**:

Table 1 – Stirring bars tested individually

Entry	Stirring Bar (all Cowie Technology)	Comments
1	001.635.13, 35mm x 13mm oval	Stayed centralised, little oscillation, excellent vortex. Stable to ~500rpm, lost coupling >500rpm
2	001.630.10, 30mm x 10mm oval	Stayed centralised, little oscillation, good vortex. Stable to ~700rpm, lost coupling >700rpm
3	001.330, 30mm x 6mm pivot ring	Failed to centre, tends to climb walls of flask and rotate into vertical plane. Lost coupling >500rpm
4	001.2625RE, 25mm x 14mm elliptical rare earth	Tumbles, failed to centre, poor vortex, unstable. Coupling lost >900rpm
5	001.2402, 20mm cross shape	Filed to centre, unstable, tumbles, no vortex.

The best stirring bar tested was entry 2, the Cowie oval bar. This centralised well, gave an excellent vortex at 500rpm and above and was very stable up to ~700rpm. Above 700rpm coupling was lost.

After testing the stirring bars individually, the three best performing bars (entries 1, 2 & 3) were tested together to look at how they interacted with each other. Only one sample of each bar was available so it was not possible to test three stirring bars of the same type together.

Table 2 – Stirring bars tested together (entries 6-8)

Entry	Stirring Bar (all Cowie Technology)	Comments
6	001.635.13, 35mm x 13mm oval	Stable, centred well, excellent full depth vortex at 400rpm. Lost coupling at >600rpm
7	001.630.10, 30mm x 10mm oval	Stable, centred well, excellent full depth vortex at 600rpm. Lost coupling at >700rpm
8	001.330, 30mm x 6mm pivot ring	Unstable, tends to stir in vertical plane!

In the presence of the other stirring bars the small oval stirring bar (entry 7) still performed best and was the most stable. No bars were tested in 100ml flasks as smaller sizes were not available. These stirring bars were used for all other tests in the 100ml flasks but were a little large and smaller versions should be tested.

Evaluation of heating performance

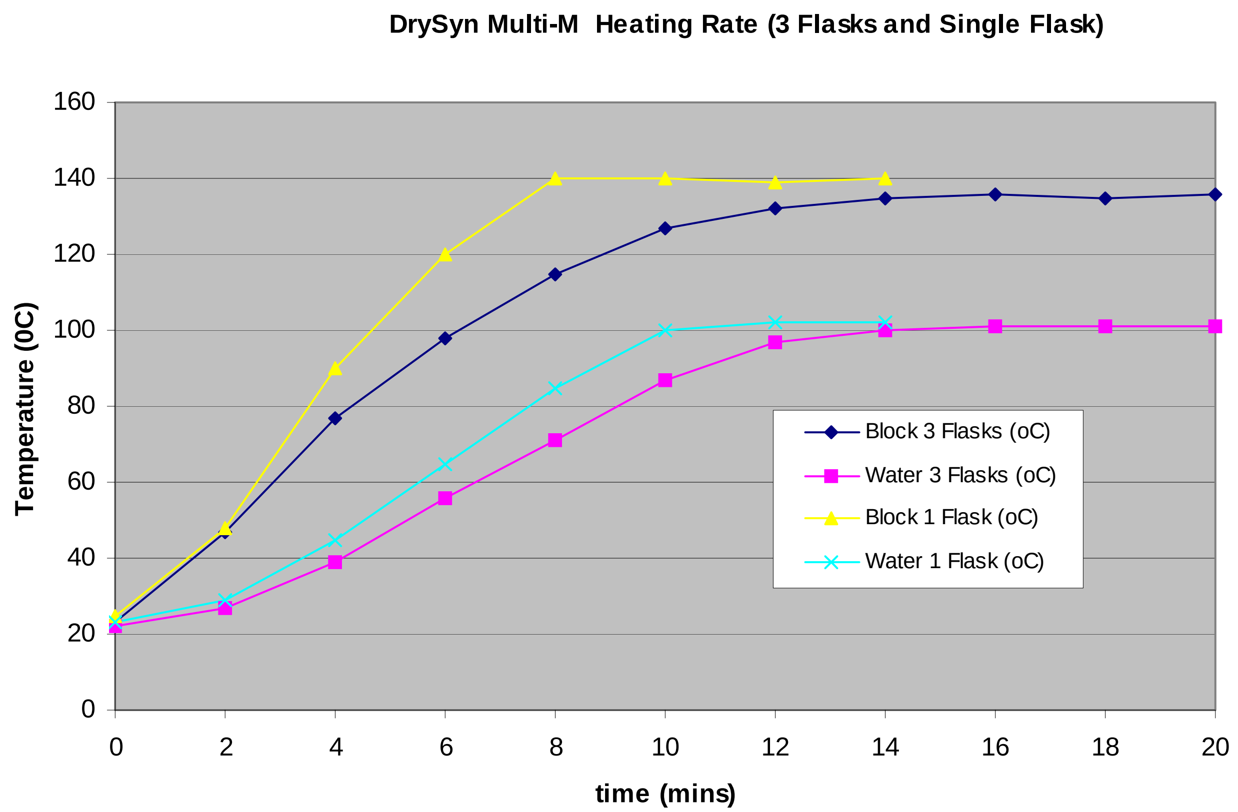
The heating performance of the DrySyn MULTI and DrySyn MULTI-M was evaluated using the fuzzy logic temperature controller for temperature control of the reaction block. The fuzzy logic temperature controller was set at 140^{o}C unless stated otherwise. The flasks contained water (half the flask's nominal volume unless stated otherwise) as a worst case. Organic solvents having appreciably lower specific heats will heat up significantly faster. The water was stirred using the stirring bars above (entries 1-3) Temperatures were measured via the fuzzy logic probe using the hole in the insert and a separate thermometer as appropriate immersed in the water.

Results

The results are shown in **Charts 1** to **4**.

Initially the heating performance was evaluated with 3 x 250ml flasks and then with a single 250ml flask placed centrally in the block of the DrySyn MULTI-M. **(Chart 1)** Each flask contained water (125ml) and the EKT 3001 fuzzy logic temperature controller was set at 140^{o}C. With 3 flasks all the flasks were boiling after 13mins 20 secs from ambient temperature. A single flask was slightly faster as would be expected and reached boiling after 10mins 15 secs.

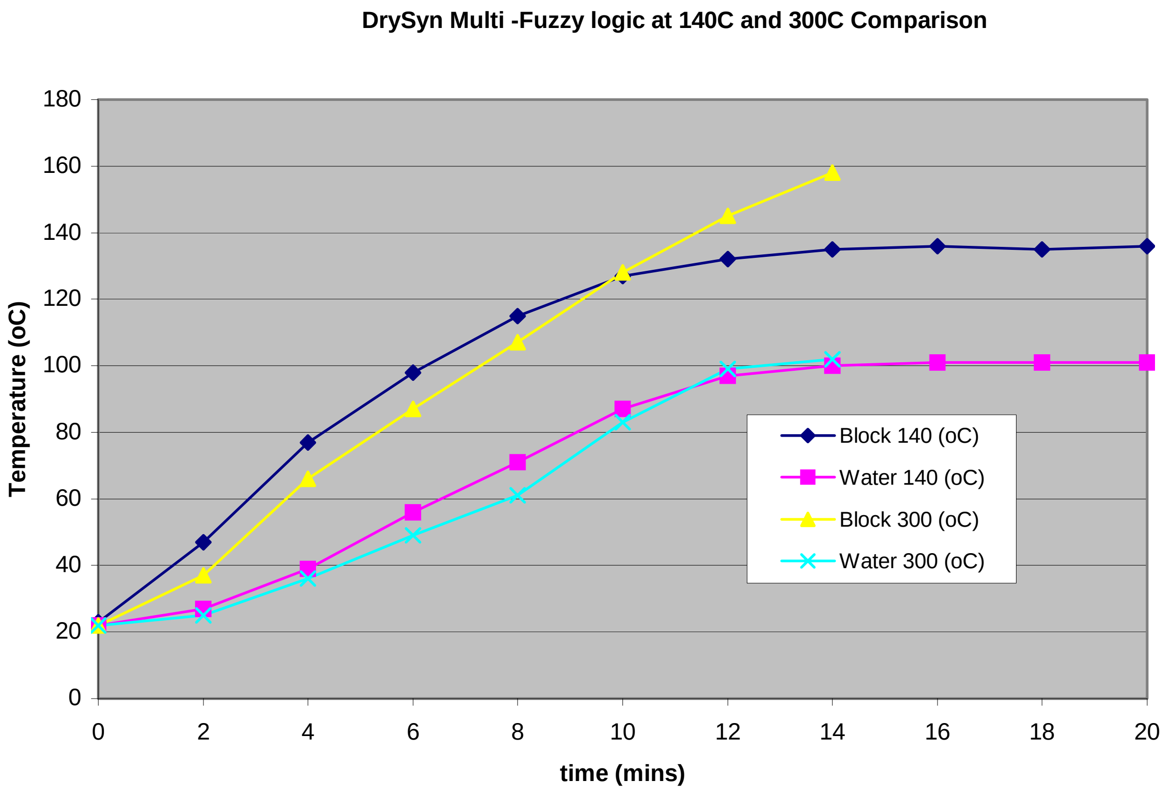
Chart 1



A heating test was also conducted with the MULTI-M under 'crash' conditions with the fuzzy logic control set at 300^{o}C and the heating control at maximum. Conditions were otherwise exactly as previously. The results are shown in **Chart 2.**

Interestingly with the fuzzy logic control set at 300^{o}C rather than 140^{o}C, the rate of temperature rise initially decreased (see **Chart 2**). The temperature of course kept rising beyond 140^{o}C so all the flasks were boiling after 12mins 30secs compared to 13mins 20secs with the fuzzy logic set at 140^{o}C. Running under these 'crash' conditions with a solvent can be hazardous with a great chance of a boilover and should be avoided. Normal practice should be to set the block temperature at between 10°C and 40°C above the b.pt of the solvent (higher range for high boiling solvents where heat losses are greater) for safe and efficient refluxing.

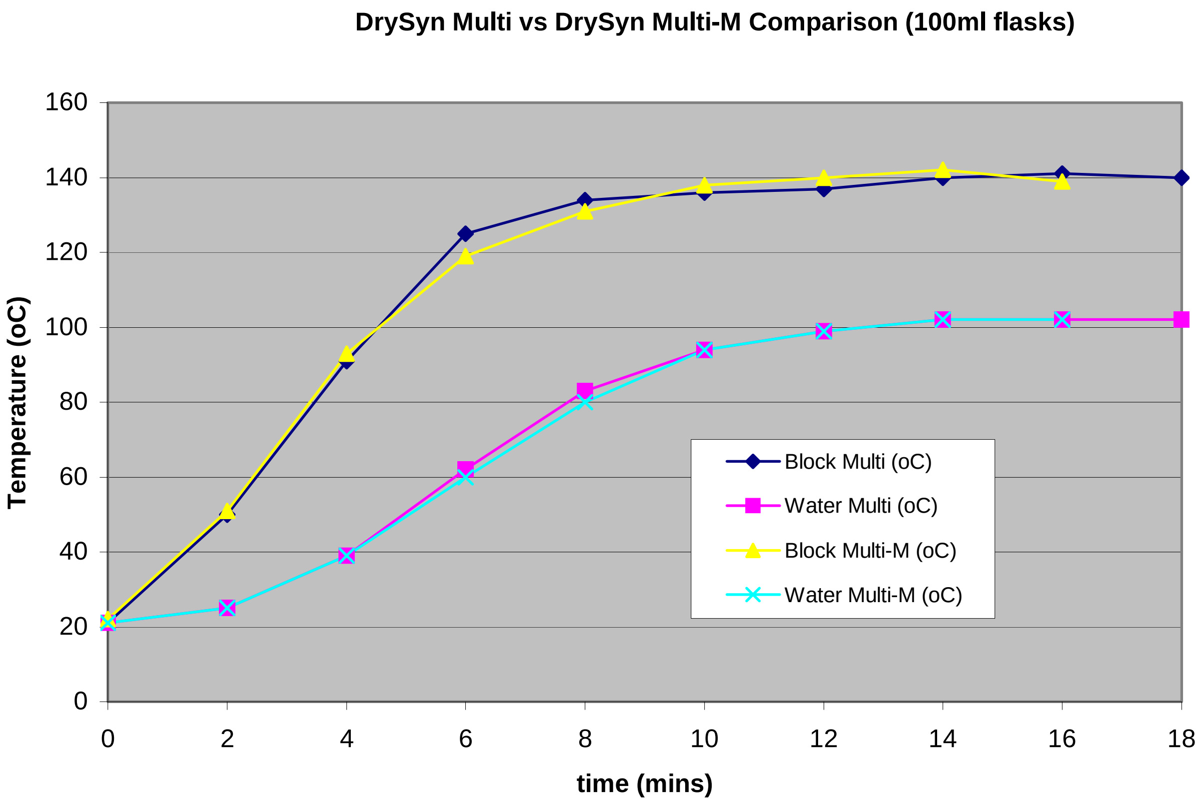
Chart 2



The DrySyn MULTI-M can accommodate round bottomed flasks up to 250ml and also accept inserts for smaller flasks down to 10ml as well as a wide range of tubes and vials with the appropriate inserts. The DrySyn MULTI will take round bottom flasks up to 100ml as well as smaller flasks and vials etc. The inserts fit both the MULTI and the MULTI-M but the larger diameter of the MULTI-M allows the use of 250ml flasks for chemists working on a larger scale.

The 100ml inserts were evaluated in both the MULTI and the MULTI-M with 50ml of water in each flask and the results are shown in **Chart 3.** As can be seen from **Chart 3** there was no significant difference in heating performance. The MULTI brought all the flasks to boiling after 13mins 15secs whereas the MULTI-M took 13 minutes exactly.

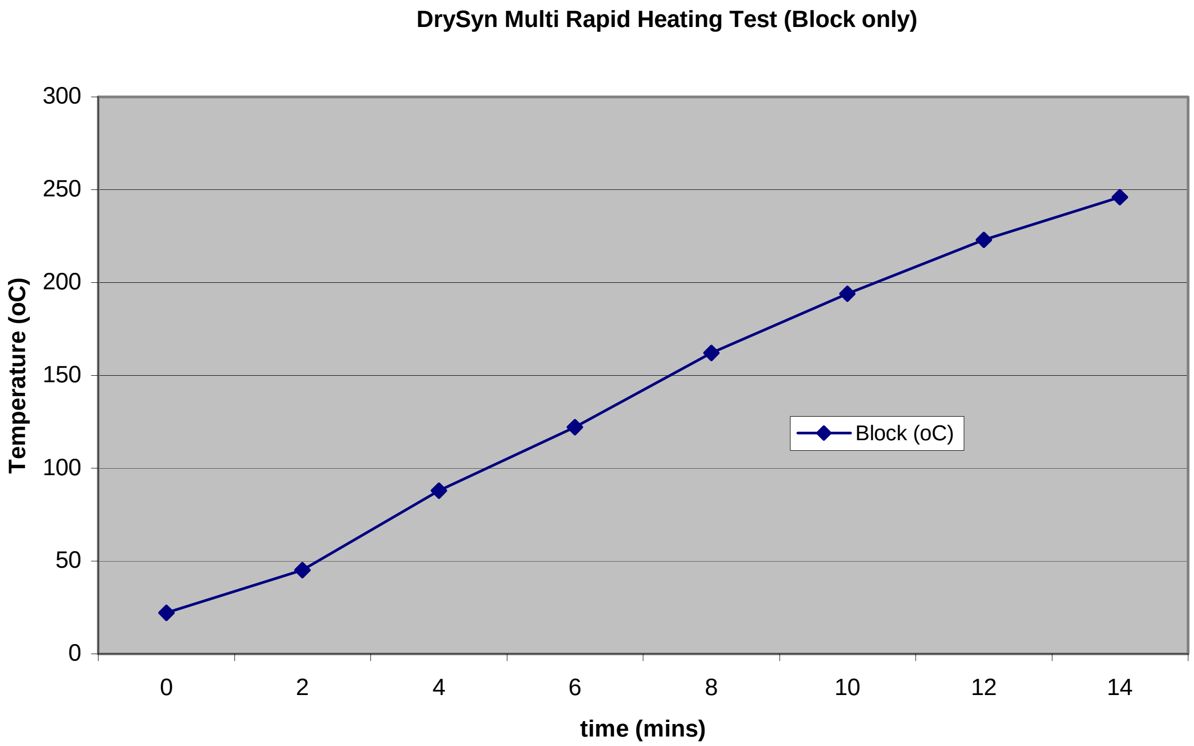
Chart 3



Finally a test was done to look at the maximum heating rate of the block alone without any flasks. The DrySyn MULTI-M base with 3 x 100ml inserts was set on the stirring hotplate and the fuzzy logic set at 300°C. The block temperature was measured using the fuzzy logic probe in the insert.

The rate of temperature increase was very rapid with a temperature of 246°C being reached after only 14 minutes (see **Chart 4**). This demonstrates the very efficient heat transfer between the stirring hotplate, base and inserts. However users should be aware of any limitations there are on using stirring hotplates at high temperatures for prolonged periods. If in any doubt consult the manufacturers!

Chart 4



Conclusions

The Asynt DrySyn MULTI and MULTI-M are two further very useful additions to the Asynt DrySyn family. They provide a very efficient means of performing parallel synthesis in a very cost effective and space saving manner. They neatly avoid the mess and safety concerns of oil baths and the cost and bulk of multiple mantles.