

CASE STUDY



SMALL SCALE REACTION CHEMISTRY AT LOW TEMPERATURES WITHOUT DRY ICE/ACETONE

During synthetic chemistry, when low temperatures are required for either a very slow or a stereospecific reaction step, it is extremely common for scientists to use a dry ice/acetone dewar to achieve temperatures of down to -78°C .

Although inexpensive and powerful, this solution presents a number of distinct disadvantages including: no control of reaction temperature, inability to run experiments overnight, necessity of planning to ensure you have dry ice to hand and the possibility that you either run out of, or do not have any dry ice available. Julabo came together with Asynt to design a solution that would address these issues in a single contained product, whilst also offering the ability to use a range of different vessels and vessel sizes for increased flexibility.

THE CHALLENGE

At the start of the project we canvassed a range of different synthetic chemists to see if we could define a product that could address the issues, and at the same time deliver a product with enhanced functionality. The key characteristics were defined as:

- Lowest temperature possible, at least -60 °C.
- Unattended operation, with the ability to run overnight.
- Dry, using no solvent or silicon oil.
- Flexibility to use different vessels up to 100 ml, including round bottom flasks and vials.
- From system activation to set temperature in short time and under 1 hour if possible.

To develop a solution, it was decided to see if we could combine two different existing technological solutions from the two companies, Julabo and Asynt. Julabo provide and supply temperature control solutions and one of their products – the FT900 range is a compressor based refrigeration system that is used to cool different probe designs for different applications. One of those products features a flexible hose that has been and can be used in a dewar of solvent to create -78 °C in solvent baths. Although a partial solution to the problem, the FT900 range did not address some of the agreed challenges. However, this product was used as the basis for the refrigeration source.

Asynt is a supplier of laboratory equipment who pride themselves on their flexible and reactive product development program to deliver new, sustainable and novel products. One of the key, constantly expanding Asynt product lines is the DrySyn range: a clean and safe alternative to oil and ice baths, and heating mantles. The comprehensive expertise in designing and machining metals and plastics allows the company to offer different custom solutions to user problems.



FT902

THE SOLUTION

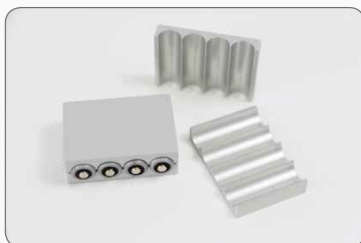
The development team trialled a number of different prototypes, arriving at a combined system that utilised a modified Julabo FT902 and an entirely new DrySyn block design to form the new FroSyn.

The FT902 features a Pt100 probe allowing direct temperature control of the machined aluminium plate, or in fact within the reaction vessel, and a unique cooling probe that can be fitted directly into the FroSyn block within a matter of seconds. Furthermore, the FT902 is on wheels to allow it to be moved rapidly and to be placed on the floor near the experiment, therefore not utilizing valuable lab and/or fume hood space.

The FroSyn plate is machined from the thermally efficient material of aluminium and is seated within a thermoplastic housing. The use of a specialist insulation around the cooling probe and plate on the FroSyn improves performance and keeps the apparatus ice-free whilst eliminating the possibility of operator cold burns during operation. The small footprint of the FroSyn allows for it to be positioned on top of a conventional magnetic stirrer plate for efficient sample mixing.

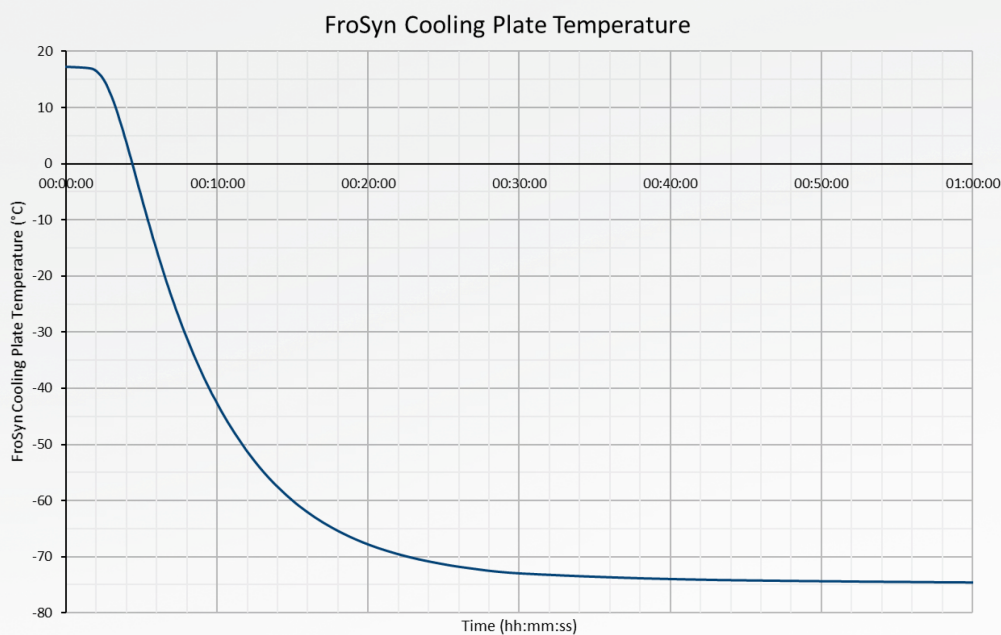


In addition a number of inserts have been specially designed for all of the standard laboratory reaction vessels. These include a set of standard inserts for round bottom flasks from 50 ml to 500 ml, as well as positioning plates for tubes and vials. The use of either weights and seals for the flask inserts, or an inerting dome with the vial plates, prevents the formation of ice between the glassware and the FroSyn cooling block. Other custom cooling accessories are also available to complement different applications, for example a 4-position holding module for battery research and development.



PERFORMANCE

The final product was evaluated over a number of weeks to ensure that it met the demands originally described.



A number of units have already been delivered to chemists, both in industry and academia, to give feedback on performance. Dr. Karim Bahou of the John Bower Group at Liverpool University stated:

"We are very pleased to have recently purchased a FroSyn for our lab. We have been using the FroSyn to conduct enantioselective catalytic reactions at low temperature for extended reaction times. By using the vial insert, we are able to set up several parallel reactions in order to test different reaction conditions at low temperature, at the same time. When we want to scale up, we are able to do this by simply switching the insert to allow larger flasks to be used. It is a neat and tidy solution, and occupies only a small part of the fumehood – no more cumbersome cooling baths!"

In conclusion the FroSyn system offers a self-contained, standalone solution to low temperature synthetic chemistry without the draw backs of dry ice/acetone baths. Whether you are using small scale screening, or low volume batch synthesis the system gives reproducible, unattended operation without the need for silicon oil baths, or large quantities of dry ice.

SHORT INFO ABOUT JULABO

JULABO GmbH, founded in 1967 in Germany, develops sophisticated temperature control technology and stands for innovation and competence in this area. Our units equipped with state-of-the-art control technology are used when highest temperature accuracy or fastest response to temperature changes is important. More than 600,000 JULABO units installed worldwide demonstrate the high acceptance among users in research and industry. With proven quality "Made in Germany" and fast and competent service by local contact partners, JULABO has developed to a market leading company for temperature control solutions.



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