



Electrothermal



OMNISTATION

Application note: A01-001A

Preparation & performance of novel catalyst complexes using a 16 reactor well Omnistation from Electrothermal for fast reaction screening.

■ Introduction

Electrothermal's Omni-Reacto Stations can accommodate a wide range of sample volumes when using the different combinations of interchangeable heating blocks, glassware and reflux heads that are available. The instrument's compact footprint ensures that it can be placed into any fume hood or workspace and the modular design and the extensive range of glassware and accessories ensures that it can be used in a wide range of research and development applications.



A research group headed by Prof. Jonathan Williams, of the University of Bath, recently purchased a custom built Omnistation unit from Electrothermal.

■ Equipment

The central control unit of the Omnistation supplied to Prof. Williams was a standard specification unit which, by the addition of removable aluminium heating & cooling blocks, allows screening of reactions in vessel volumes ranging from 1 to 1000ml. However, in this case, the removable aluminium heating and cooling blocks were specifically designed to accommodate a range of Young's tap ampoules (made at the customer's site by the university glassblower Mr Phil Jones). The aluminium units each had 16 holes with a diameter of 13mm.

■ Application

The reactions carried out by Prof. Williams's research group with the Electrothermal unit, usually comprise a total volume of less than 2 ml, a volume which fits nicely in the 4 cm deep bore holes of the heating unit.

The group's reactions are normally carried out under inert conditions, which can be achieved directly on the Omnistation by connection of the gas inlet to an inert gas supply. It is also possible to place tubes under vacuum, which is useful when handling air sensitive reaction components.

Most of the reactions carried out require heating at a specific temperature and for a defined time, both parameters being controlled directly on the Omnistation and maintained accurately by the Omnistation central control or base control. Stirring is also a feature built into the Omnistation.

Prof. Williams's group uses the Omnistation to screen various catalyst precursors and ligand combinations under identical conditions for a chosen reaction.

info@electrothermal.com

www.electrothermal.com

Tel: +44 (0) 1702 303350



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"We are able to test various reaction parameters for a catalyst or test a range of substrates for a particular catalyst system. The use of the Omnistation saves us time and the use of a massive amount of glassware. It also affords us consistent results, which can sometimes be hard to obtain. Consistency is the key parameter in catalyst, substrate and reaction condition screenings."

The William's group specialise in a research strategy referred to as "Borrowing Hydrogen". We take a simple substrate, like an alcohol, and temporarily convert it to an aldehyde then an ester as shown in Figure 1.

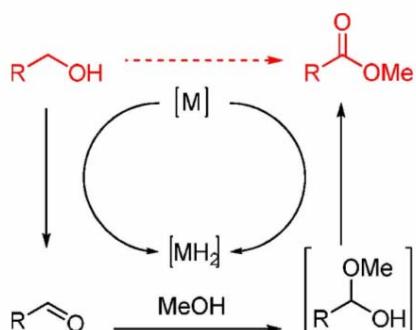


Figure 1. Conversion of an alcohol to an ester.

Method

An example of a general 16 experiment parallel reaction run on the Omnistation is:

To all of the ampoules add a magnetic stirrer bar. The catalyst precursor and a ligand are also added and placed under inert conditions (the Omnistation is connected to a Schlenk line, allowing the tube to be placed under vacuum then filled with inert gas). The catalyst and ligand are dissolved in 0.5 ml of dry toluene and heated at reflux, with stirring, for 1 hour to activate the catalyst complex.

Although the Omnistation can cool down to -30°C, the ampoules were only cooled to room temperature. The substrate, an alcohol (0.5 mmol), the hydrogen acceptor (3 eq.), 0.5 ml methanol and water (2 eq.) are then added to each ampoule. The ampoule's are then sealed and heated at reflux for 24 hours with continuous stirring.

A number of these reactions (maximum of 16) are run at the same time with one component changed to compare the reactivity.

Conclusion

Electrothermal's Omni-Reacto Station is a versatile, modular instrument that can be utilised in a wide variety of industrial and development applications. The instrument enables research and development professionals to investigate and optimise the reaction chemistry of both classical and novel organic chemistry applications.