

# Long-Term Synthesis in Medium of Organic Solvents using Xelsius+ Reflux Condenser

Relevant for:  
Academic Research, Pharmaceutical  
Chemistry, Industrial R&D



## Summary

This application note deals with the use of XELSIUS system with Reflux Condenser accessory for long term reactions using medium with volatile organic solvents.

Volatile organic solvents are largely used as a medium for organic synthesis in different application and temperatures. Keeping constant the characteristics and volume of medium is very important and the organic solvent cannot flow out of the vessel through evaporation due to the temperature of reaction.

However, there are so many reactions that need temperatures above boiling point of organic solvent, therefore in these cases we suggest to use the Reflux Condenser accessory. The Reflux Condenser creates a cold zone on top of the vessel. By using the same water chiller of Xelsius System, it keeps the top of the vessel below the solvent boiling point temperature and recondense the solvent to the vessel.

In this way the evaporated solvent is recondensed to medium and the volume does not decrease for long-term synthesis.

With a constant volume in a long-term synthesis, it is possible to develop new compounds or in some cases, to increase the reaction yield.

## Equipment

Item #	Description
8053 000 100	XELSIUS Basic Unit, Software Version: 2.44
8053 000 201	Reflux Condenser Module
8053 000 202	HV Vials Starter Kit, 1 - 30 ml
Chiller	LW 1000, Labtech Srl,
Balance	Analytical Balance, 4 digit (g)

## Chemicals

	Description
20 ml	Deionised Water
20 ml	Acetonitrile p.a
20 ml	Acetone p.a.
20 ml	Ethyl-Acetate p.a.
20 ml	Methanol p.a.

## Method

Four common solvents were used for an organic synthesis: Methanol, Acetonitrile, Ethylacetate, Acetone. Water was used as a reference too. 10 ml of the solvent was placed into a 30 mL vial with cap and septum, with a needle to release internal pressure and avoid overpressure during the tests.



The temperature set for each solvent was 10°C above the boiling point, to be sure of solvent evaporation.

The test was done with and without the usage of a reflux condenser at 10°C cooling liquid.

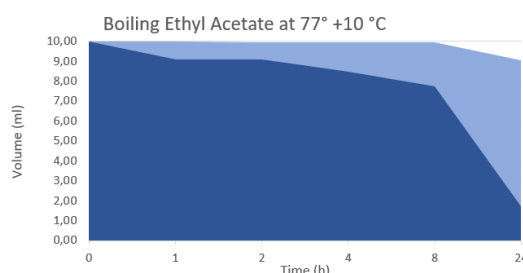
Loss of solvent will be measured after 1, 2, 4, 6 and 24 hours.

Temperature (°C)	Boiling Point	Reactor
Acetone	56	66
Methanol	65	75
Ethylacetate	77	87
Acetonitrile	82	92
Water	100	110

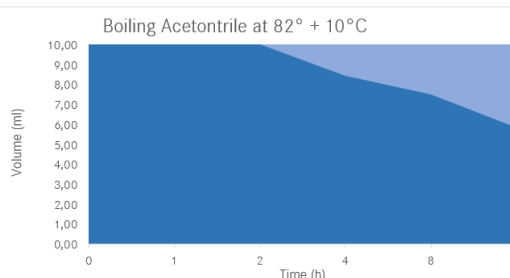
Table: 1 Solvent Temperatures

The results from experiments are shown in the graphics below show the volume of solvent with Reflux Condenser compared to the analyses without Reflux Condenser.

The recovery of solvent was very high and perfectly able to keep constant the volume inside the vial during longterm synthesis.



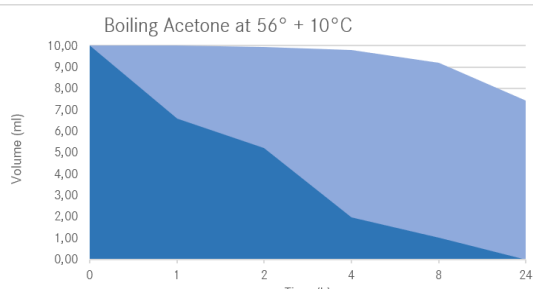
Graph 3: Ethylacetate results



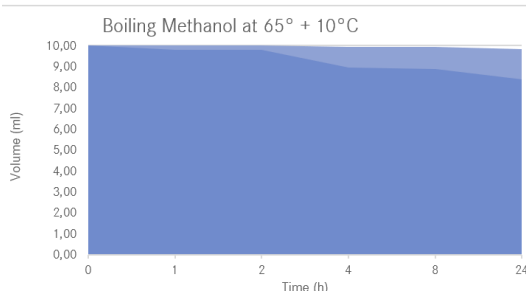
Graph 4: Acetonitrile results

### Conclusion:

It has been demonstrated that the use of Xelsius system combined with the Reflux Condenser enables to keep a constant volume (up to 95%) in long-term synthesis, at different temperatures, using volatile organic solvents.



Graph 1: Acetone results



Graph 2: Methanol results

### References:

(1) LABTECH End User Manual "Xelsius System"

(2) ORGANIC CHEMISTRY, Nick Greeves, Stuart Warren, Peter Wothers, Jonathan Clayden

Data provided by: nevoLAB GmbH in cooperation with LabTech Srl, IT [www.labtechsrl.com](http://www.labtechsrl.com)



nevolab

nevoLAB GmbH  
Am Gehrenbach 8  
D 88167 Maierhöfen  
[info@nevolab.de](mailto:info@nevolab.de)  
+49 8383 929 566 0