

# FDVS - Freeze Drying Vial System\*

A Lyophilisation System Designed to Optimise Your Freeze Drying Protocols



\*Prototype

## Benchtop System with One to Seven Vials

Optimise your freeze drying protocols

## Industry Standard Lyophilisation Vials

2R, 4R, 8R, 10R, 15R and 20R

## Lyophilisation Imaging

Track the front throughout the freeze drying process

# Introducing the FDVS

Optimising freeze drying protocols is a time consuming and expensive process which can be wasteful. The Freeze Drying Vial System (FDVS) has been optimised for simulating the industrial freeze drying process in a compact and efficient form. By incorporating the vials, the FDVS works with a small sample volume and uses enough to simulate large scale industrial processes while still minimising sample wastage. It saves time, money and the unit has also been designed to save space.

The system was designed with world leading lyophilisation experts and as such comes with several specialised components and features which are essential to the freeze drying process. The optical system can be controlled in the X, Y, Z axis with a 10x darkfield lens mounted in line with the freeze drying chamber. With this specially designed optical set-up, monitoring and tracking the position of the freeze-drying front can be done simply and efficiently.

A cold trap has been built into the system to condense and remove any excess moisture. The system is fitted with two vacuum sensors, a capacitive and a pirani-type, which allows accurate sensing throughout the drying process and detection of the end point of primary drying. The top of the stage also features a vial stopper which allows the user to close the open vials once the freeze drying process is complete. Up to three thermocouples can be fitted in the chamber to measure the temperature of three vials or to observe the temperature gradient in one vial.

## Features

### **SIMULATE INDUSTRIAL PROCESSES**

Along with vacuum, temperature (-100°C to 125°C) can be accurately controlled and programmed to simulate industrial procedures and determine ideal drying parameters. The process is quick and utilises small volumes of sample, ultimately reducing sample wastage and saving on costs.

### **ACCURATE TEMPERATURE AND VACUUM SENSING**

The system is fitted with two vacuum sensor types ensuring accurate detection of the transition between primary and secondary drying. The system is also fitted with three dipping thermocouples which accurately measure the temperature within the lyophilisation vials and a platinum sensor in the shelf which provides accurate stage temperature monitoring.

### **REAL TIME IMAGING**

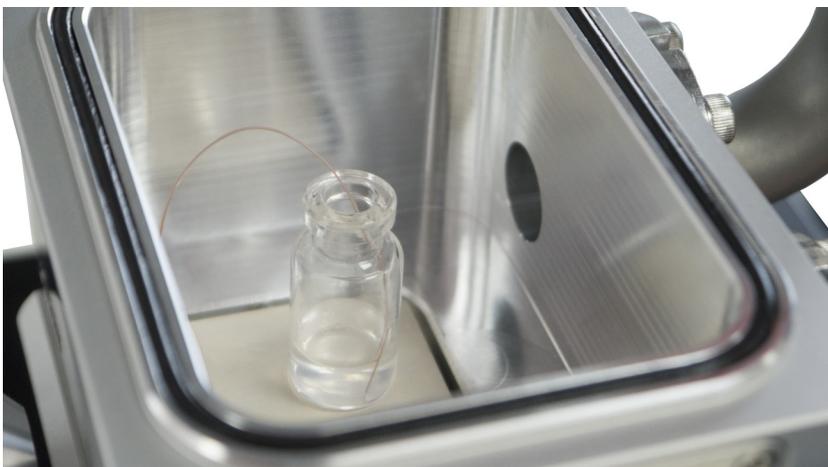
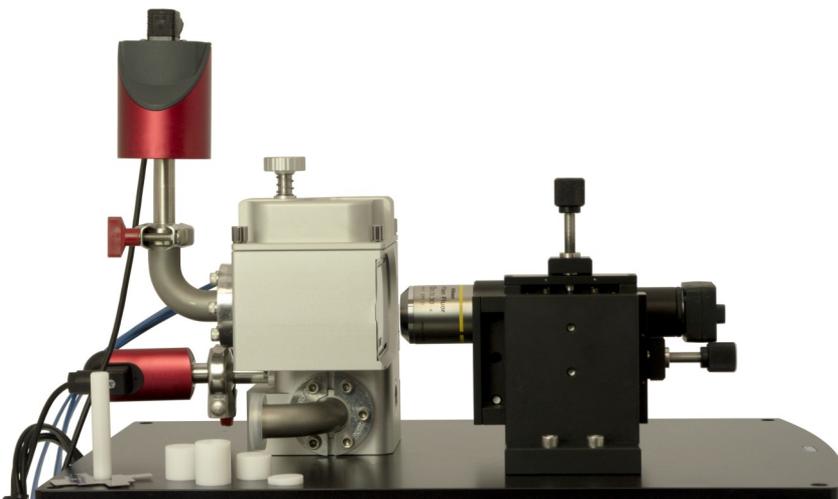
With the 10x Darkfield lens and control of the X, Y and Z axis, the system is optimised for capturing the freeze drying process. The arrangement of the optical set-up allows for structural changes to be seen as they occur in the lyophilisation vials.

### **MULTIPLE VIAL COMPATIBILITY**

Compatible with most standard freeze drying vials (2R (up to seven vials), 4R, 8R, 10R, 15R and 20R).

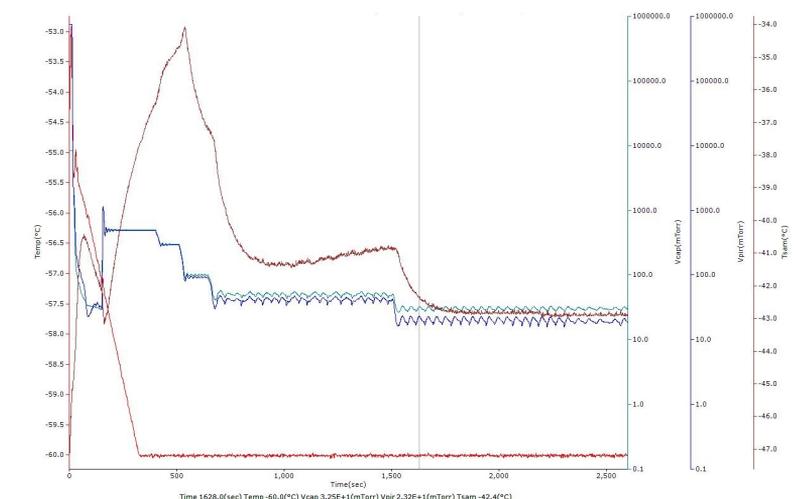
### **IN-VIAL THERMOCOUPLES**

The chamber can be fitted with a maximum of three thermocouples which allows the temperature to be recorded across different vials or as a gradient within one.

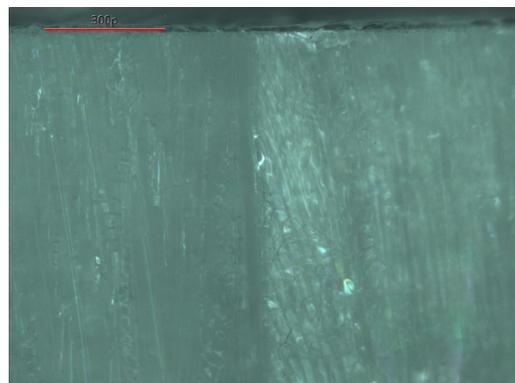


# Application Examples

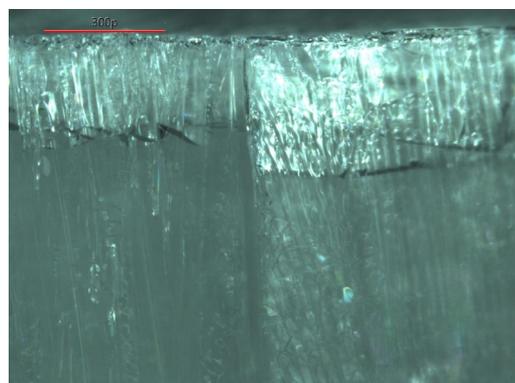
The FDVS allows full control over temperature and vacuum through customisable control profiles. Below is an example of a freeze-drying graph from the control software LINK. Two temperature readings are shown, one from within the lyophilisation vial and one of the stage shelf, together with the readings of both capacitive and pirani sensors.



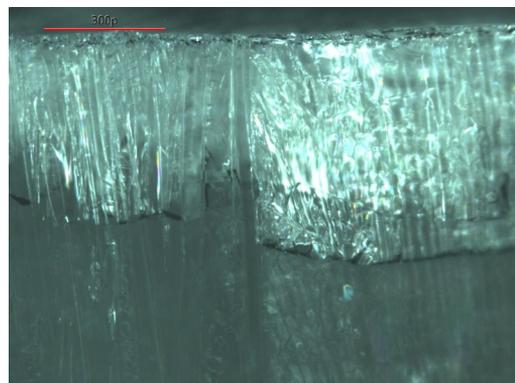
Below are still images of the drying front of a sucrose sample.



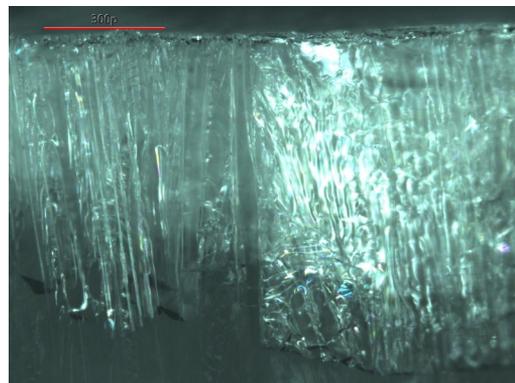
10:44:51 30 September 2016  
Temp -50.0°C Vcap 8.25e+03mT Vpir 7.29e+05mT Tsam -38.0°C Ramp Row 1 Rate 30°C/min Limit



10:48:11 30 September 2016  
Temp -50.0°C Vcap 3.69e+01mT Vpir 1.18e+01mT Tsam -37.7°C Ramp Row 1 Rate 30°C/min Limit



10:50:21 30 September 2016  
Temp -50.0°C Vcap 3.42e+01mT Vpir 6.77e+00mT Tsam -39.0°C Ramp Row 1 Rate 30°C/min Limit



10:54:21 30 September 2016  
Temp -50.0°C Vcap 9.17e+01mT Vpir 7.43e+01mT Tsam -38.0°C Ramp Row 1 Rate 30°C/min Limit

## Testimonial

**Dr Zixin Huang**  
*University of Erlangen, Research Group of Dr H. Giesler*

“The idea of designing the FDVS system is not just to make a mini-scale freeze dryer but also to create a versatile platform, with which one is able to quantitatively evaluate the freeze-drying process under controlled condition within a single vial. The purpose of this system is to facilitate the freeze-drying cycle design and process transfer.

Process parameters such as sublimation rate and resistance to vapour flow can be determined during the drying process. By this means, the freeze-drying cycle under given condition could be quantitatively assessed. With the optical unit, which is equipped with a darkfield lens, one is also able to observe the freezing and drying process, which would be helpful for better understanding the drying behaviour”.

## Technical Specification\*

<b>Temperature Range</b>	-100°C to 125°C
<b>Heating Rate</b>	0.01°C to 30°C
<b>Pressure Range</b>	40mTorr to atmospheric Pressure
<b>Compatible Vials</b>	2R (up to seven vials), 4R, 8R, 10R, 15R and 20R
<b>Pressure Sensors</b>	Pirani and Capacitive
<b>Temperature Monitoring</b>	Fitted with three thermocouples
<b>Camera</b>	1.3MP

\*subject to change

## Discover More...



### FDCS196

With a temperature range down to  $-196^{\circ}\text{C}$ , ultra low temperature eutectics can be investigated with the FDCS196 system. Chamber pressure is monitored by a Pirani vacuum gauge mounted directly on the stage. The XY manipulators can be used to follow the drying front moving across the sample. Pressure can be automatically controlled by the new Linkam MV196 motorized valve. A graph of temperature against time also shows the plot of the chamber pressure throughout the experiment.



### Optical DSC

The optical DSC (Differential Scanning Calorimeter) system has been optimised for those wishing to measure the transition temperatures and enthalpy changes of their samples. The design allows mounting of the stage on a microscope, granting image and time lapse recording of sample transitions with high detail. An optically sealed crucible is also available for those wishing to conduct closed experiments. The optical DSC enables the user to measure thermal and glass transitions of a wide range of substances whilst accurately controlling temperature from  $-180^{\circ}\text{C}$  to  $450^{\circ}\text{C}$ .



### Humidity

The RH95 Relative Humidity Controller provides environmental sample control to Linkam's range of temperature stages. It provides precise control in a compact, self-contained package with no requirement for dry air supply. The RH% is accurately controlled between 10%-90% (temperature range ambient to  $85^{\circ}\text{C}$ ).

## Contact Details

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