Introducing the MicroFD® with LyoSim™ & LyoPAT®

The MicroFD® with groundbreaking LyoSim® and LyoPAT®

This is what this freeze dryer can do with as few as 7 vials, in addition to the normal events you see in your current freeze dryer—

Understand and Optimize your Freezing—The Foundation of Freeze Drying:
- The only system to offer freezing analysis, control and optimization.
- Controlled nucleation on demand.
- See crystallization events not seen with thermocouples.
- Monitor all of the stages of freezing.
- Control crystal growth.
- Determine when the product is fully frozen.

Understand and Optimize Primary Drying:
- Calculates and reports heat flow over time.
- Measures and reports vial heat transfer coefficient - Kv.
- Determines and reports mass flow over time – grams/hour.
- Determines and reports cake resistance over time – Rp.
- End of primary drying using capacitance manometer pirani convergence.
- Shorten primary drying with maximized product temperature profile.

Understand and Optimize Secondary Drying:
- Determine end of secondary drying utilizing heat flow.
LyoPAT includes
FreezeBooster® Controlled Nucleation
AccuFlux® Heat Flow Measurement & Control
AutoDry Primary Drying Optimization

Redefining How
You Freeze Dry

And the MicroFD Does Even More

- Minimize your valuable API use during R&D.
- Save time. Set up freeze drying runs in minutes, not hours.
- Provides the information needed to optimize and transfer protocols.
- Designed and engineered to study center or edge vials.
- Product temperature prediction without thermocouples.
The story of a revolution in innovative freeze drying.

_In the beginning…_

Developing new products can be really expensive.

Loading a research and development lyophilizer with 100+ vials could cost a fortune and using less than a full tray may not give good representative results.

How can we invent an R&D and Cycle Development freeze dryer that uses less product?

It started with a dream to make a freeze dryer that uses less product.

So we made a small chamber freeze dryer—only 19 ten ml vials.

There was large variation in how the vials freeze dried.

To be able to transfer a cycle we need to have all of the vials behave like center vials – the last place to freeze dry. Popular theory said-eliminate radiant energy from the freeze dryer wall to eliminate edge effect by cooling the chamber wall….so we did. We cooled the walls.
Cooling the chamber wall does not work!

So radiation is **NOT** the only factor contributing to vial variation.

Elimination of radiant energy by cooling the chamber wall did not eliminate “edge effect”. Some other factor was creating vial sublimation variation on the edge vials.

But what is it?

Our theory was that product vials that are touching one another are affected by the thermal events in the vials surrounding them. The vial in the center of the array is touched and affected by 6 vials. The vials on the outside of the array are only touched by 3 vials so energy transfer between vials is **NOT** the same.

“Sublimating vials are heat sinks”

This vial-to-vial energy transfer has a greater impact on freeze drying than previously suspected and creates sublimation variations based on where the vial is in the vial array and how many vials are touching the subject vial(s).
Introducing LyoSim

The LyoSim ring system eliminates edge effect and promotes a more homogenous vial-to-vial freeze drying. LyoSim creates an environment for the outside edge vials in the array that is the same as the environment for the inside vials, thus eliminate edge effect.

Because of LyoSim technology, the MicroFD can be utilized just like any other R&D freeze dryer but with fewer vials!
The MicroFD with LyoSim and LyoPAT can be used in place of your standard laboratory freeze dryer for research, development and cycle transfer needs. How deep you dive into critical process parameters and the nuances of freeze drying is up to you.
“It takes me about 1 ½ hour to defrost, fill and load my one shelf tray on my R&D freeze dryer. With the MicroFD I can defrost, fill and load in less than 15 minutes.” MicroFD user

Use the MicroFD just as you would any other tray-type freeze dryer. Just do it with less product and less effort.

The MicroFD with LyoSim and LyoPAT has everything you need for your freeze drying processes.

Basic Specifications

Product Chamber:
- Top loading shelf, 6 inches in diameter.
- Shelf temperature control from -60 to +65C.
- Shelf uniformity of +/-0.30C.
- Shelf surface temperature and inlet temperature.
- Temperature controlled radiant top plate, height adjustable.
- Automatic stoppering.
- Controlled temperature LyoSim ring from -60 to +65C.
- Includes one set of LyoSim blocks for vial size of your choice.
- 7 “T” type product thermocouples.
- 7 thermocouple holder/placement for 20 mm vial neck sizes.
- 7 thermocouple holder/placement for 13mm vial neck sizes.
- Capacitance manometer and Pirani gauges in chamber.
- Proportional vacuum control 25 to 1500 mT +/-1 mT
- Temperature ramp rates of up to 3C/min.
- Gas back-fill.

Condenser:
- Fully visible
- Condenser temperature to -75C.
- Condenser capacity of 0.5L/24 hours.
- On demand isolation valve between chamber and condenser.
- Fast hot-gas defrost.
- 65 lpm vacuum pump
Easy to use with new intuitive controller interface and batch reports

The MicroFD utilizes the same PC/PLC platform that is used in other tray dryers - from research to production but in a new more intuitive interface.

- Full Manual Control
- 10 freezing steps
- 16 drying steps
- Set capacitance manometer vs pirani convergence for end of primary drying indication
- End of drying & final hold conditions
- User set alarm selection
- Add controlled nucleation if required
- Dashboard
- Defrost
- System Test
- Data Logging – alpha numeric displayed every minute
- Batch Records
- Graphing of all functions
- Calibration
- Lyosights for background information, useful studies, white papers, manuals
- On board system instructions
- Print function
- Optimization
- Batch reporting
Easy recipe and analyze mode.

At a glance dashboard.
The MicroFD LyoPAT controls are designed to be intuitive and take you step-by-step through the programming requirements. We want your freeze drying experience to be easy so that you may concentrate on the complexities of your research, formulation development and cycle transfer.
Millrock Reporter – Allows you to search by Batch ID or date.

Batch data screen allows users to call up the parameters they want to see.
Batch graphing screen—built with the data you want to see.

Temperature set points and read-outs.

Heat flow vs product temperature.
Temperature set points vs read-outs for LyoSim & Shelf Inlet and Surface

Expanded view of product temperatures and heat flow.
MicroFD with LyoSim & LyoPAT

All The Tools You Will Need to Learn Even More About Freeze Drying.

LyoPAT - Smarter Freeze Drying

FreezeBooster Controlled Nucleation
AccuFlux Heat Flow Measurement & Control
AutoDry Primary Drying Optimization
Learn more during the freezing process. Increase the amplitude of freezing events while creating greater uniformity across the batch and from batch-to-batch making freezing events easier to study. FreezeBooster® Controlled Nucleation can be used to force synchronized crystallization of the free water in the product to create uniformity.

Minimize freezing time using AccuFlux® to indicate the end of the freezing event as indicated by a decrease in heat flow to the product. No more guessing on when the product is fully frozen.

Identify primary and secondary crystallization events, in situ, using AccuFlux®.

Study the effect of free ice crystal size on your product. Using AccuFlux® you can control post nucleation heat flow thereby forcing smaller or larger ice crystal structure in your product thus effecting cake resistance.

Understand more about how your product freezes. See AccuFlux® heat flow during the phases of freezing including super cooling, nucleation, crystallization of free water and crystallization of the maximum freeze concentrate.

“The importance of the freezing step is almost obvious since it is the first process step, and because the characteristics of the frozen matrix (or “cake”) after water has been removed) determine how the rest of the process will run”

Pikal, Rambhatia, Ramot

To know more about the freezing process helps put you in control…

…and you have more control then you think with LyoPAT®.
LyoPAT Delivers a New Tool for Monitoring and Controlling Freeze Drying & Cycle Transfer

See freezing like you never have before.

Product heterogeneity due to uncontrolled nucleation.

More uniform ice matrix and product homogeneity - a result of controlled nucleation.

AccuFlux Heat flow profile during freezing.
AccuFlux Provides Unsurpassed Tools During Primary Drying Including All Critical Process Parameters for Easy Scale-Up

- Eliminate the need to do gravimetric tests with direct measurement of Kv in a single run.
- Standard pressure measurements – capacitance manometer and pirani vacuum gauges.
- Temperature parameters including shelf surface, shelf inlet, condenser, LyoSim, and all product temperature probes.
- Determination of M - mass flow.
- Determination of Rp - cake resistance
- Monitors all product thermocouples and thermocouple average to not exceed user indicated product safety temperature when using AutoDry.
- Determines end of primary drying with capacitance manometer & pirani convergence
- Graphing of all functions
- Alpha numeric display of all data points (1 minute intervals)

The unique and innovative LyoSim system allows researchers to study fewer vials and create thermal behavior that imitates center vials or edge vials so scientists can study either.
The Freeze Drying Protocol Developed Now Will Most Likely Be Utilized Forever.

Optimization Could Save You Tremendous Time and Money Over the Product’s Commercial Life.

Maximize product temperature and heat input without exceeding the critical melting point of the product thereby providing you with the shortest possible primary drying cycle.

Total Elapsed time: 54.1 hrs
- Freezing: 6.6 hours
- Primary Drying: 37 hours
- Secondary Drying: 10.5 hours
- Reduction in Process Time: 64 hours
- % Reduction in time: 55%
- Cake structure visually acceptable
- Residual Moisture: 0.61 +/- 0.04%

Blind study – protein product optimization using LyoPAT®.
Transfer Your Optimized Freeze Drying Cycles from the Lab to Production with Relative Ease.

The MicroFD with LyoSim and LyoPAT provides all of the necessary information for scale-up to production dryers in less time and with less product.
Additional Resources on MicroFD with LyoSim & LyoPAT

http://www.millrocktech.com/lyosights/

Developing Transferable Freeze Drying Protocols Using AccuFlux® and MicroFD®. TN Thompson, Qiming Wang, Cynthia Reiter

Integrated heat flux measurements as a non-invasive monitoring technique for freeze drying. Ilona Konrad, Victoria Pauli, Wolfgang Friess, Andrea Hawe, Gerhard Winter, Angelika Freitag

Innovative approaches for lyophilization process, equipment and drug product characterization. Ahmad M. Abdul-Fattah

Heat flux measurements as a new PAT Tool in freeze drying. Ilona Konrad, Victoria Pauli, Wolfgang Friess, Andrea Hawe, Gerhard Winter

Real Time Temperature and Heat Flux Measurements for Lyophilization Process Design and Monitoring. Nicholas Huls, Evan Liechty, Andrew Strognrich, Alina Alexeenko, Nithin Raghunathan, Xiafon Jiang, Dimitrious Peroulis

The Evolution and Studies Leading to the Design of the MicroFD. Millrock Technology

Monitoring secondary (solute+water) crystallization by DSC, synchrotron X-ray diffraction, and in vials using heat flux transducer. Qiming Wang, Michael Sztucki, T Narayanan, Brian Ivin, Adrian Marley, Kieran Joyce, Prem Mohanty, Evgenyi Shalaev
The LyoSight section of our website provides the freeze-drying community with
- Papers and Presentations
- Tech Notes
- Webinars
- Events
- Courses
- Tools

We take pride in matching the correct freeze dryer to your needs. Call us today so we can help you make the right choice in equipment.

Millrock Technology offers LyoRevival™. This program can be used to trade in your old freeze dryer or to revive one for continued use. Contact us about the details of this program.

If the MicroFD does not process enough product at one time for your particular needs take a look at the LyoPro with LyoPAT – Research and Development Freeze Dryer for people who need to do more product during R&D.

We offer a full line of freeze dryers, from manifold type BenchTop freeze dryers to the Production Quanta Steam Sterilized Lyophilizers.

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LyoSim, AccuFlux, AutoDry and FreezeBooster are patented technologies.

Millrock Technology, Inc.  39 Kieffer Lane  Kingston, NY 12401
+1.845.339.5700  www.millrocktech.com  info@millrocktech.com  sales@millrocktech.com
Performance Freeze Dryers for Any Application

Featuring:
- FreezeBooster®
- AccuFlux®
- LyoSim®
- AutoDry
- LyoPAT™

Freeze Dryers by Millrock Technology