

*The ultimate value
in
Analysis & Control,
Protocol
Optimization,
&
Cycle Transfer*



MagnumPro



RevoPro

LyoPRO **Freeze Dryers**

Every LyoPro freeze dryer comes complete with the LyoPAT[®] analysis and control system delivering the ultimate freeze-drying process.

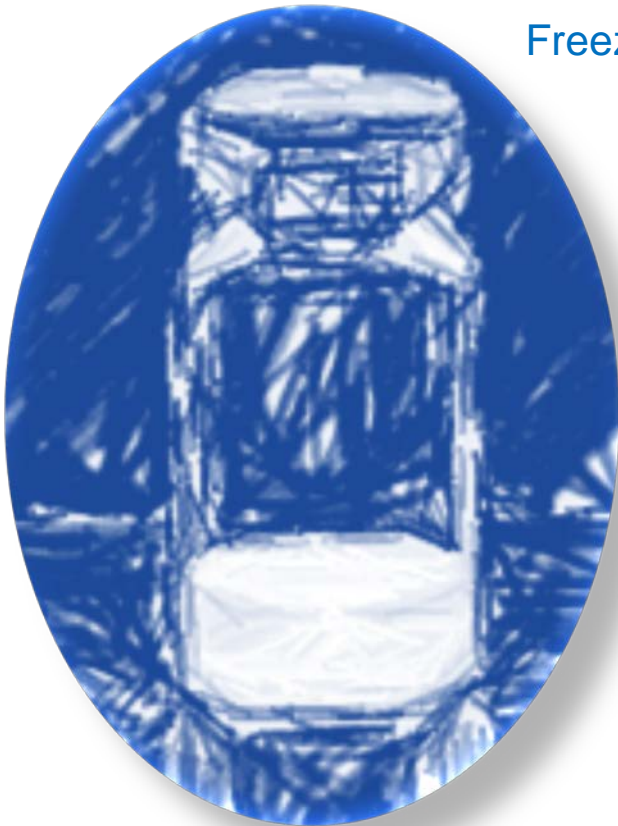
Revo[®] Pro with LyoPAT and Magnum[®] Pro with LyoPAT combine the best technology tools and innovations available in the market today for freeze drying analysis, optimization and cycle transfer. LyoPAT technology coupled with our robust and dependable freeze dryers provide researchers with both the innovative tools and the throughput they need for day-to-day operations.

LyoPAT is a unique combination of innovative freeze-drying technologies.

AccuFlux AccuFlux identifies and controls the critical process parameters in Freezing and Primary Drying.

AutoDry AutoDry optimizes primarying drying based on product critical temperatures and executes the freeze drying cycle without researcher assistance.

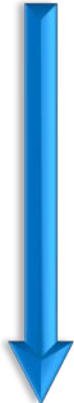
FreezeBooster Controlled Nucleation provides uniformity of crystallization in a batch and from batch-to-batch.



LyoPAT

Analyze ➡ **Optimize** ➡ **Transfer**

Analyze



AccuFlux presents researchers with all of the critical process parameters required to understand what is happening during the freeze-drying process.

Included are:

- Kv – Vial heat transfer coefficient
- Rp – Cake resistance – a relative indicator of cake porosity
- Mass Flow – The sublimation rate from the cake.
- % Dry

Convergence of the Pirani Vacuum Sensor and Capacitance Manometer signals the end of Primary Drying.

Millrock Reporter provides complete alpha-numeric data with graphing capabilities for data retrieval and review.

Optimize



Freezing Cycle Optimization: FreezeBooster Controlled Nucleation creates a homogenous ice matrix from batch to batch and within a batch during a freeze-drying run. Forcing a unified nucleation event creates amplified and clear thermal information thus allowing better product temperature control and repeatability of the freeze-drying process. Some studies indicate that controlled nucleation helps create a better ice matrix for optimizing primary drying.

Primary Drying Optimization: AutoDry increases the product temperature to its maximum safe temperature in as short a time as possible during primary drying. AutoDry controls the shelf temperature based on the product temperature and automatically adjusts to keep the product close to but not more than the product's critical temperature.

Transfer

LyoPAT delivers the measured Kv value at the end of the drying cycle which in turn is utilized to transfer the freeze-drying cycle successfully from one freeze dryer to another. When Kv of a recipient dryer is unknown the transferred cycle can simply be lengthened during Primary Drying in order to achieve a successful outcome.

Practical Application

Starting to use the LyoPro freeze dryers and their advance monitoring and controls is easy.

- First Run: Program and run your existing freeze-drying cycle for analysis.
- Second Run: Start the optimization process by using AutoDry.
- Third Run: Continue to optimize the cycle by utilizing controlled nucleation and AutoDry.
- Fourth Run: Optimize further by studying the effect of post nucleation AccuFlux control and AutoDry.

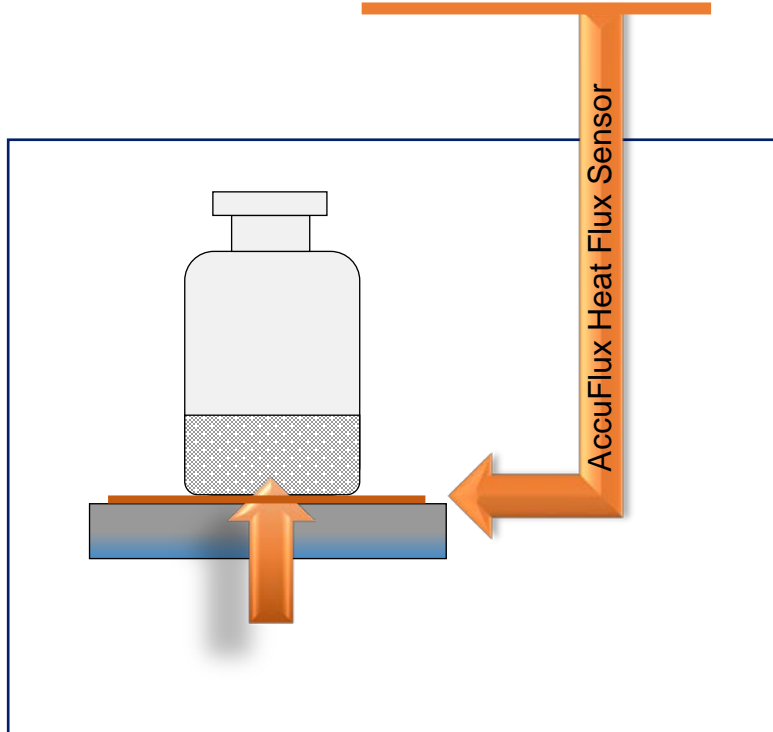
Heat Flux Monitoring with AccuFlux

AccuFlux Heat Flux Monitoring & Control provides direct measurement of K_v . This measurement is vital for cycle transfer. Additionally AccuFlux monitors mass flow and cake resistance for comparison against other thermal profiles for cycle development and optimization.

- Eliminate the need to do gravimetric tests with direct measurement of K_v – vial heat transfer coefficient - in a single run.
- Dynamic measurement of mass flow.
- Continuous measurement of R_p - cake resistance.
- Direct measurement of shelf surface temperature.
- Monitors all product thermocouples and thermocouple average to not exceed user indicated product safety temperature.
- Control post-nucleation events with heat flow control for larger or smaller crystal formation.

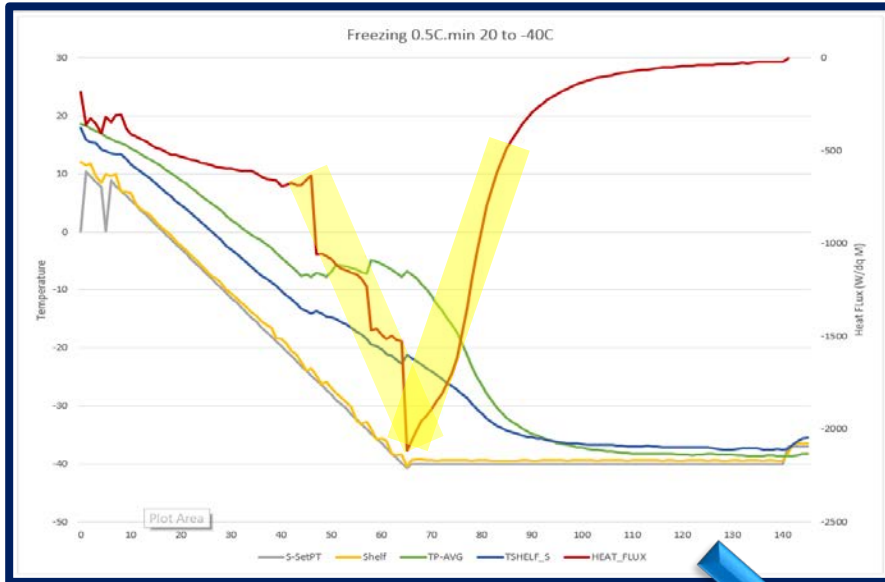
It all starts with K_v .

Based on **direct measurement** of heat flux.



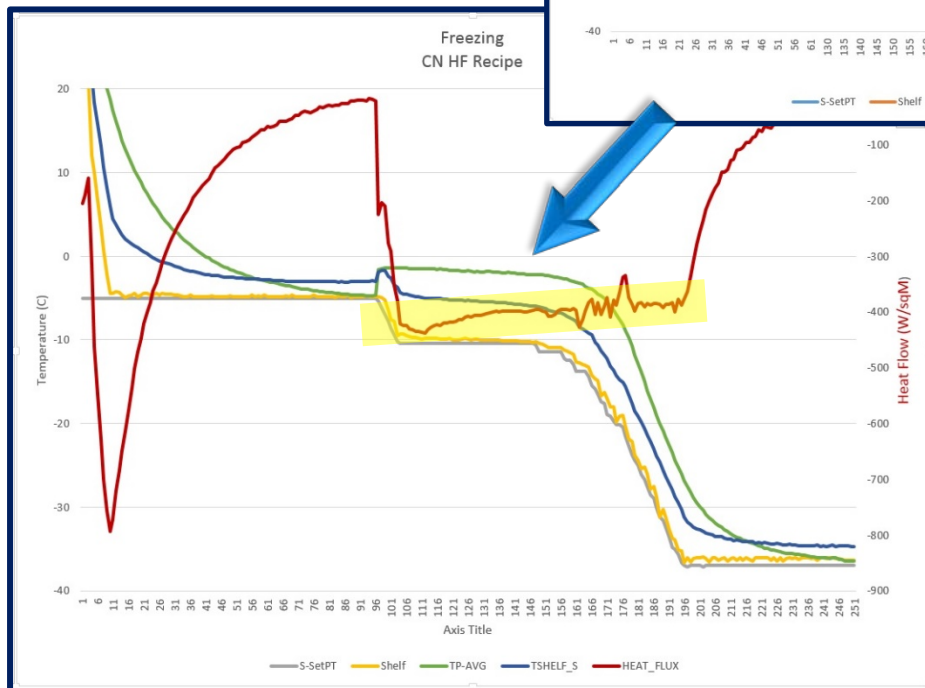
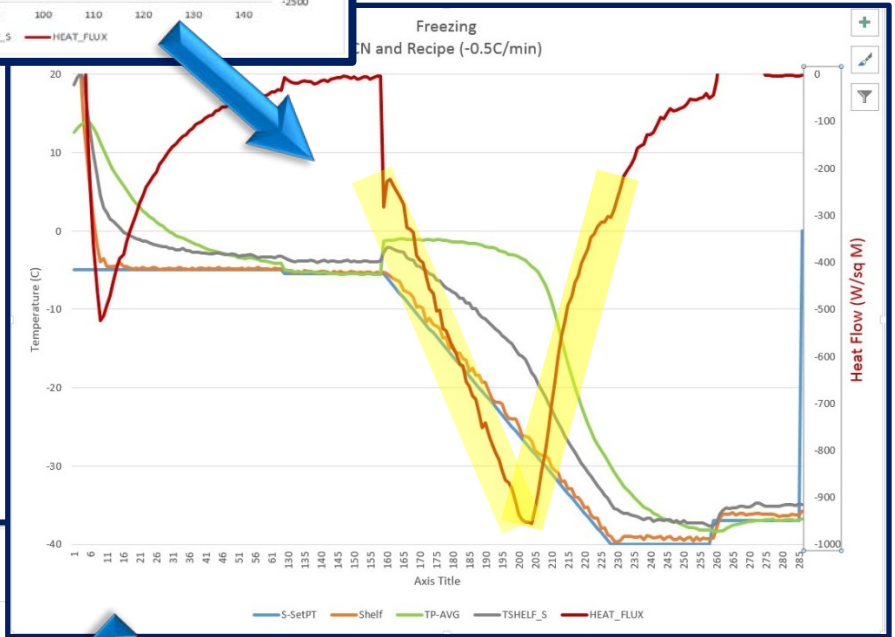
More information on K_v
determination with AccuFlux and
how it can work for you.

Optimization with Heat Flux Control with AccuFlux



No Controlled Nucleation with no post nucleation heat flow control. Illustrates product nucleating at different times and deepest V heat flux pattern and. Indicates heterogenous crystalline structure.

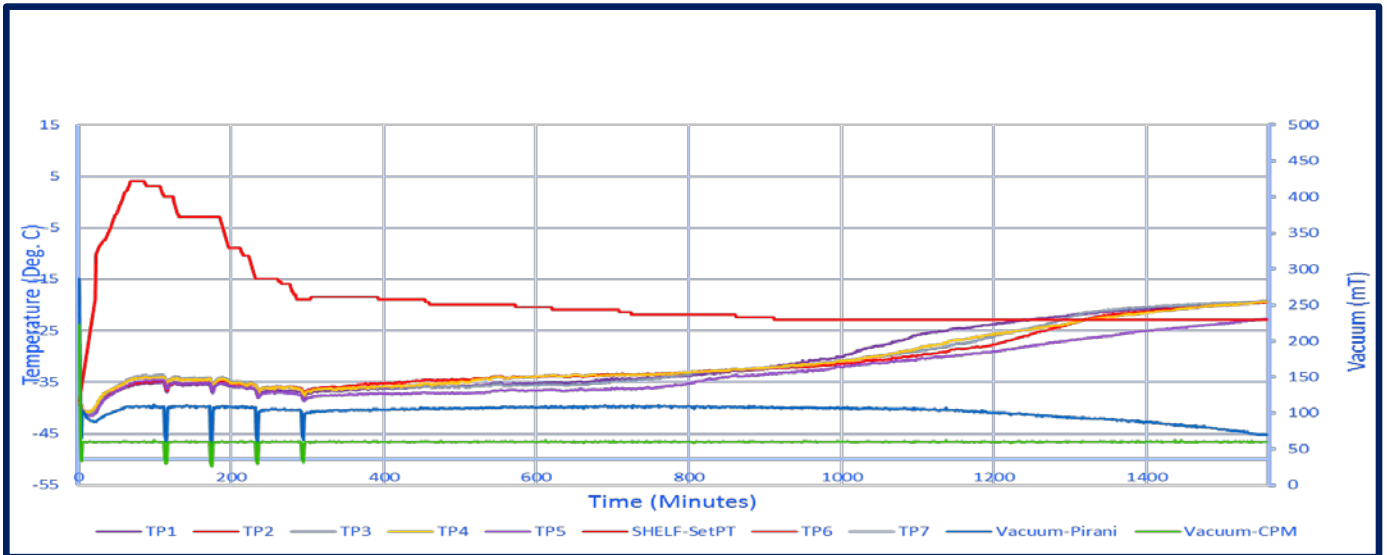
Controlled Nucleation with no post nucleation heat flow control. Illustrates product nucleating at the same time and a less deep V heat flux pattern. Indicates a more homogenous crystalline structure.



Controlled Nucleation with post nucleation heat flow control. Eliminates deep V pattern and promotes crystalline homogeneity within the product vial.

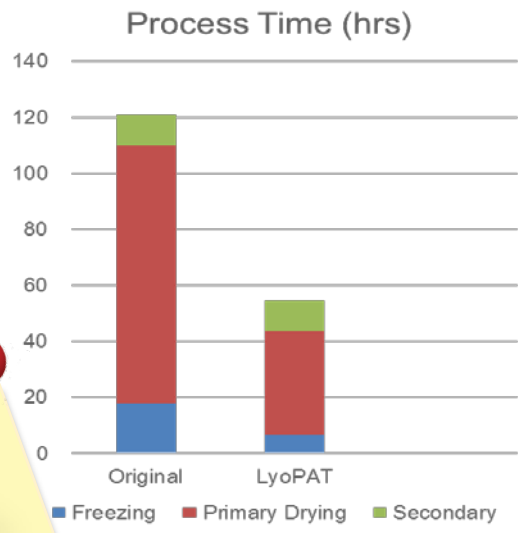
Primary Drying Cycle Optimization with AutoDry

Instead of guessing what shelf temperature to use during primary drying AutoDry maximizes shelf temperature without your constant supervision. Once the critical parameters are loaded into the system Auto-Dry will execute the entire freeze drying cycle without user intervention. Auto-Dry uses Millrocks' patented 'Critical Vial Method' direct measurement of the product temperature and controls from the warmest vial, thus providing the maximum shelf temperature while eliminating the possibility melt-back or collapse. Since the method is not based on batch calculations Auto-Dry is active throughout the entire primary drying cycle.



Blind study – protein product optimization using LyoPAT®.

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%



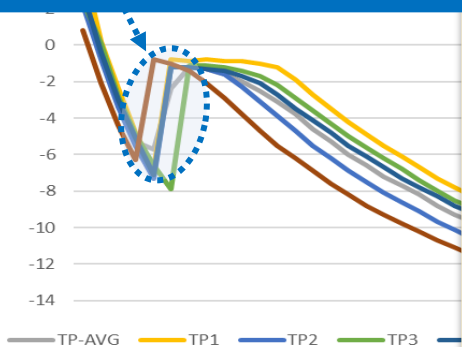
Optimizing the freeze-drying cycle now could save thousands or millions of dollars over the commercial product life cycle. Once your cycle protocol is developed it will probably never be altered.

Controlled Nucleation with FreezeBooster

RevoPro and Magnum Pro are configured to allow the connection of the FreezeBooster through a side port in the tray chamber. This configuration allows the operator the option of adding a sample extractor to the front door if required and allows unimpaired visual access to the chamber through the front door of the freeze dryer.

FreezeBooster comes complete with its own height adjustable table that allows easy installation and connection of the FreezeBooster port to the freeze dryer port. FreezeBooster can be simply disconnected and moved to another dryer on demand.

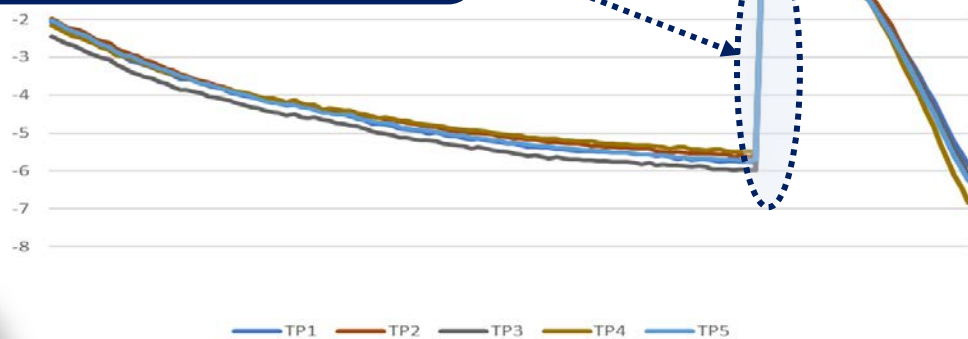
Illustration of uncontrolled nucleation with different degrees of super-cooling



Controlled Nucleation provides even nucleation across the batch and from batch-to-batch creating batch and cycle uniformity. During this process no measurable water is added to the product. In some instances, controlled nucleation may also help optimize the freezing cycle thereby saving time and money.



Illustration of controlled nucleation with same degree of super-cooling for all products



"We find that the ice fog technique used with reduced pressure in the chamber achieves rapid ice nucleation and uniform ice crystal structure in all the vials within the batch." Patel, Bhugra, Pikal.
Reduced Pressure Ice Fog Technique for Controlled Ice Nucleation during Frreeze-Drying. AAPS Pharm Sci Tech 2009



Click here for information on FreezeBooster and the controlled nucleation process

New Intuitive LyoPAT Interface

Take Control in a Different Way

Millrock Technology, Inc's goal has been to create a freeze dryer controller that is totally intuitive and as easy to use as possible. The software is constructed for a logical progression from analyzing your existing protocols to optimizing freezing and primary drying to protocol transfer to another freeze dryer.

The screenshot displays the OptiDry Enterprise 2018 software interface. At the top, it shows cycle parameters: Cycle (min), Phase (min), Step# (min), Time (13:17:02), Date (12/28/2017), Shelf Setpoint (0.0 °C), Shelf Temp (##.# °C), Condenser Temp (##.# °C), Product Avg (0.0 °C), Pressure Setpoint (0), and Shelf Target Temp (0.0 °C). It also includes fields for Batch ID, Recipe Name, Product Name, System ID, Product Number, and Product Description.

The main interface is divided into several sections:

- FREEZE DRYING:** Includes a 'START CYCLE' button, a 'PROCEED' button, and a grid for step enablement and temperature/pressure control across 10 steps.
- PRIMARY DRYING:** Similar to the freeze drying section, with a grid for 16 steps and controls for product temperature, pressure convergence, and hold time.
- ALARMS:** A table showing the status of various probes (PROBE1 to PROBE16) and system conditions like Condenser Overload, Vacuum Overload, and Power Outage.
- STORAGE SET POINT:** Controls for Shelf Temperature and Pressure.
- ALARMED STATE:** Similar to the Alarms section, showing current status for probes and system conditions.
- Graphs:** A line graph showing temperature and pressure over time, with a 'SHELF' label.

The MANUAL control panel is organized into several sections:

- TEMPERATURE CONTROL:** Includes a 'STOP CYCLE' button at the bottom.
- SHELF CONTROL:** Features a 'SHELF CONTROL' toggle (OFF/ON), Shelf Temperature (0.0 °C), Ramp By Time (0 min), and Ramp By Rate (10.0 °C/min).
- CONDENSER CONTROL:** Includes a 'CONDENSER CONTROL' toggle (OFF/ON).
- PRESSURE CONTROL:** Includes a 'VACUUM CONTROL' toggle (OFF/ON), 'PRESSURE' (0), and 'PRESSURE RELEASE' toggle (OFF/ON).
- VALVES:** Includes an 'ISOLATION VALVE' toggle (CLOSE/OPEN).

The LEAK RATE control panel displays:

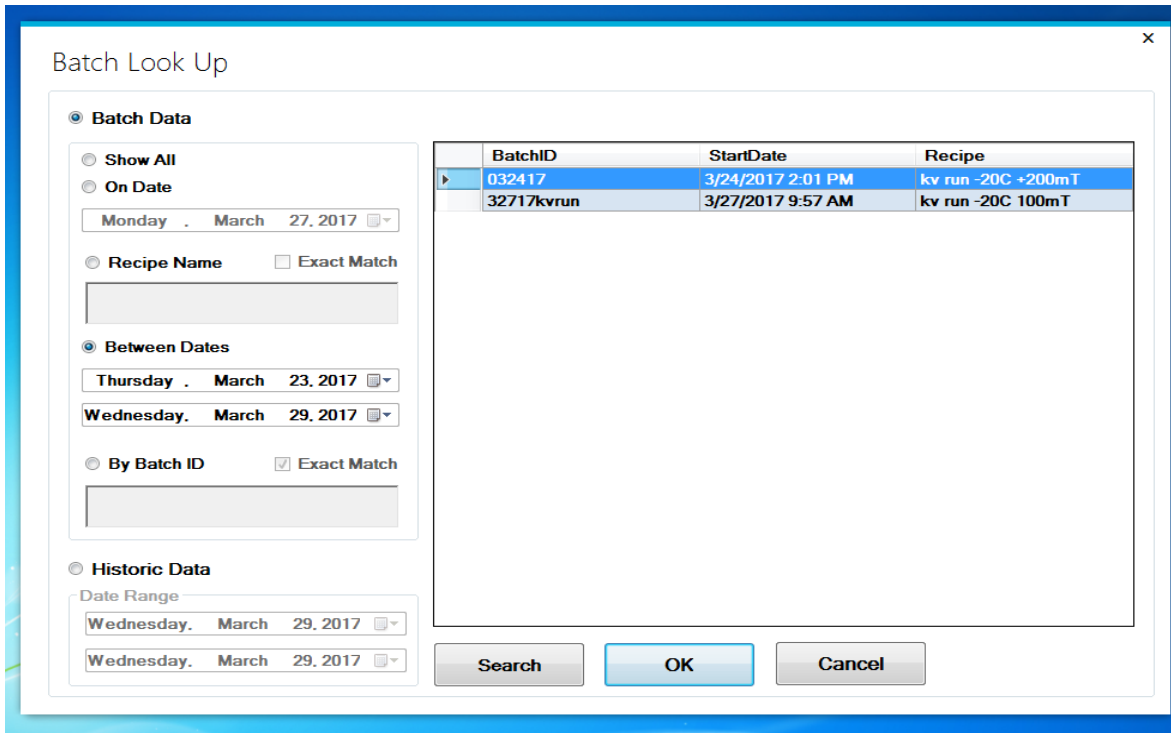
- Test Set Points:** Test Duration (0 min), Pressure Set Point (0), and Condenser Set Point (-85 °C).
- Test Results:** Starting Pressure (0), Ending Pressure (0), and Leak Rate (0 /Hr).
- A prominent red 'STOP CYCLE' button is located at the bottom.

The HOT GAS DEFOST control panel displays:

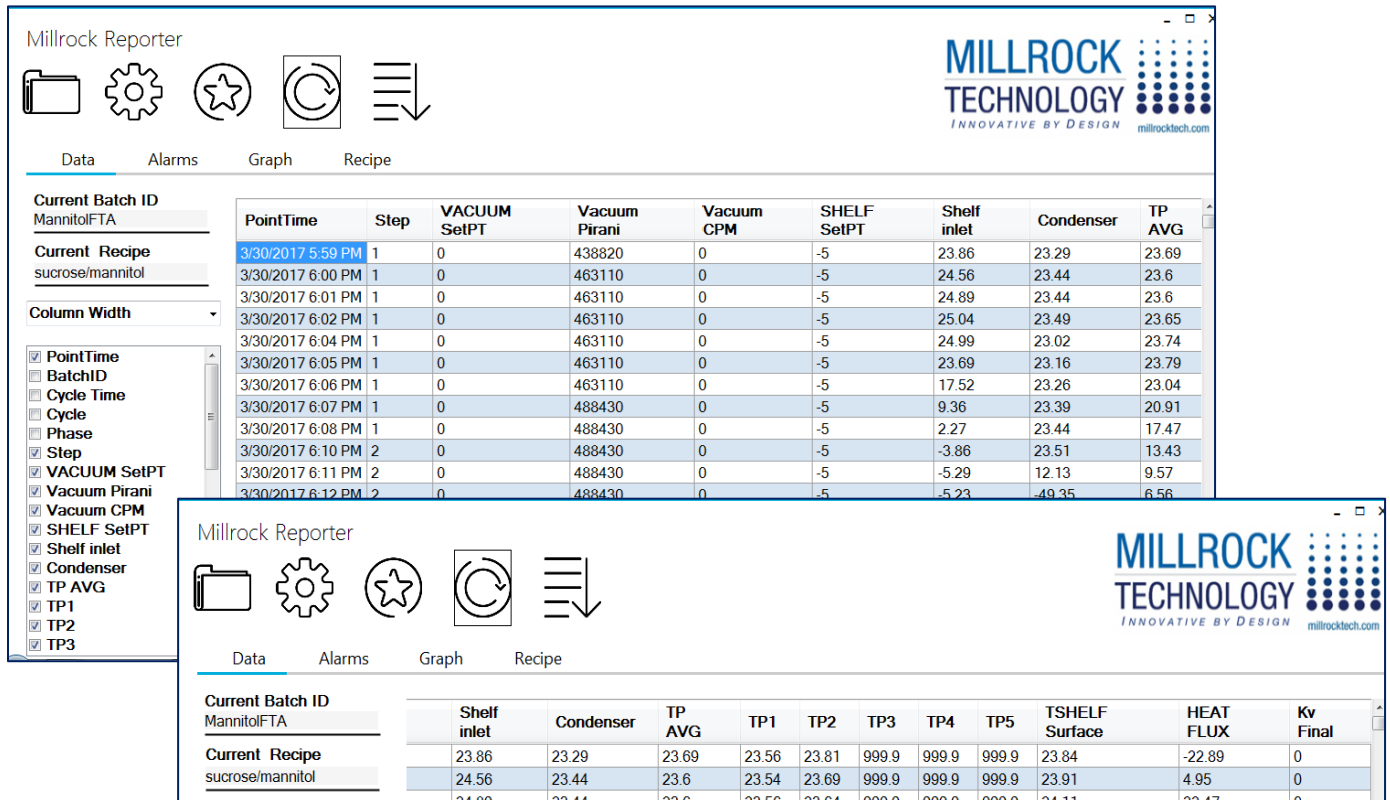
- Defrost Time (min)
- Time Elapsed (min)
- Time Remaining (min)
- Condenser Temp (##.# °C)
- A prominent red 'STOP CYCLE' button is located at the bottom.

Millrock Batch Reporter

Millrock Reporter – Allows you to search by Batch ID or date.

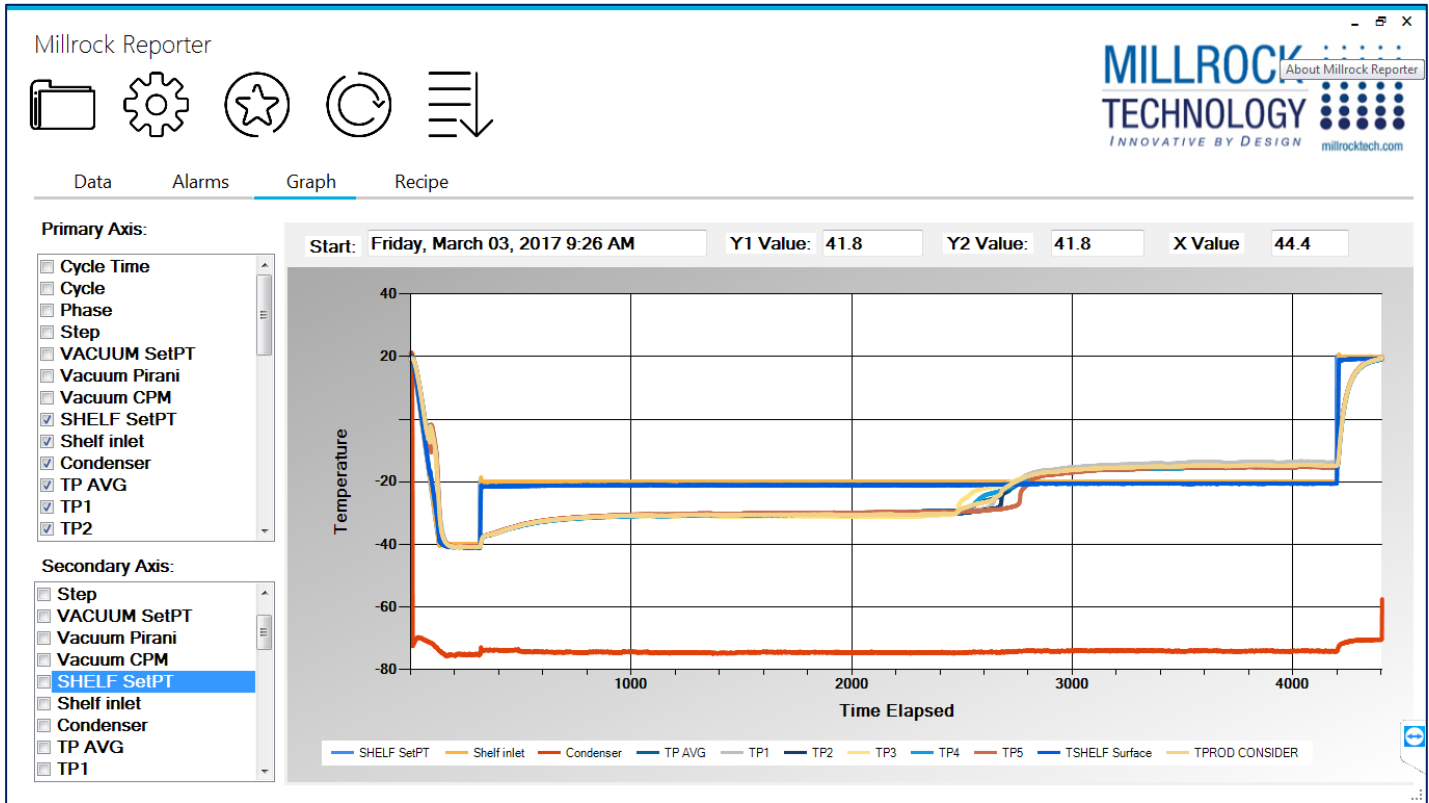


Batch data screen allows you to call up the parameters you want to see.

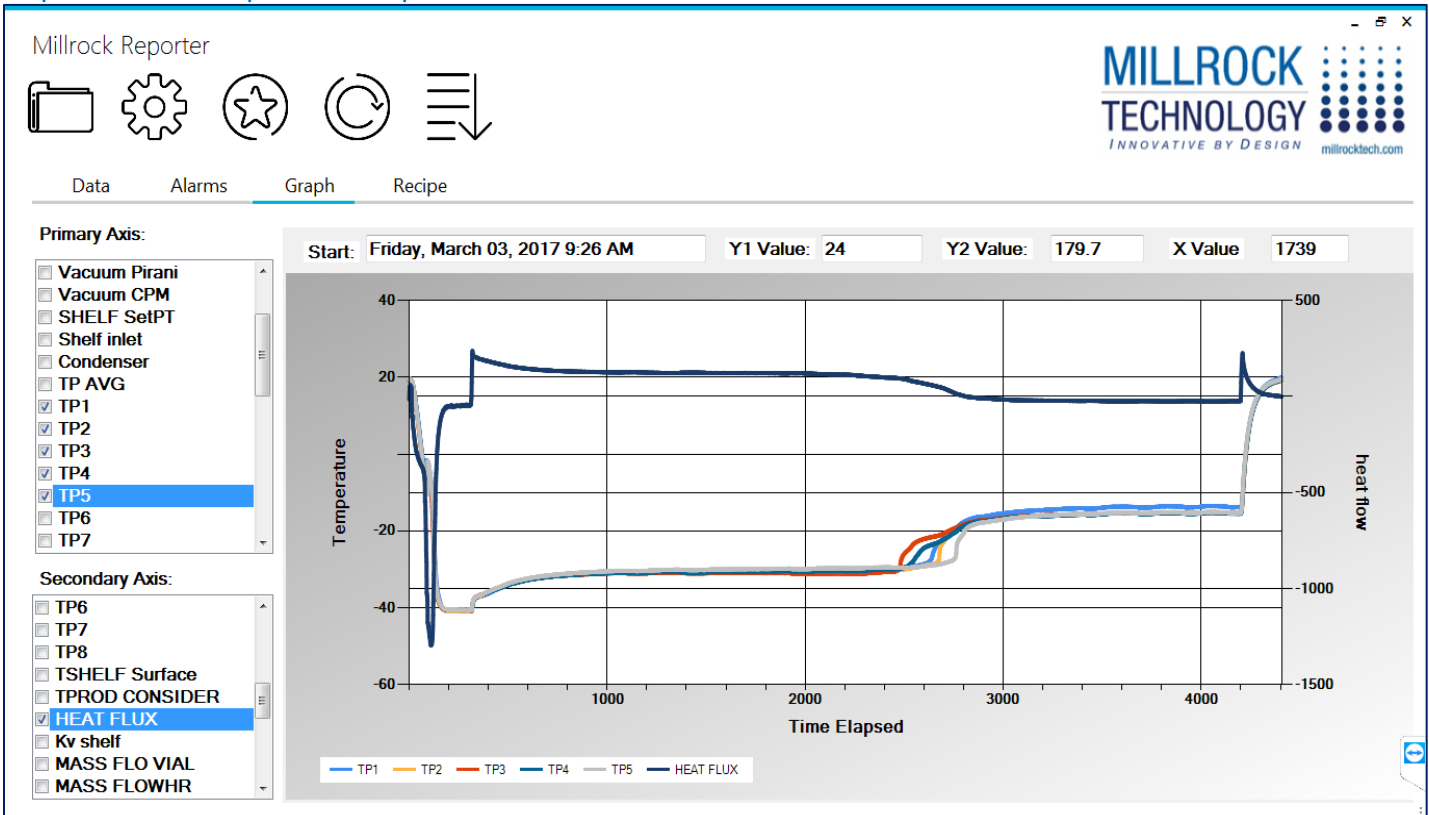


Batch graphing screen—built with the data you want to see.

Temperature set points and read-outs

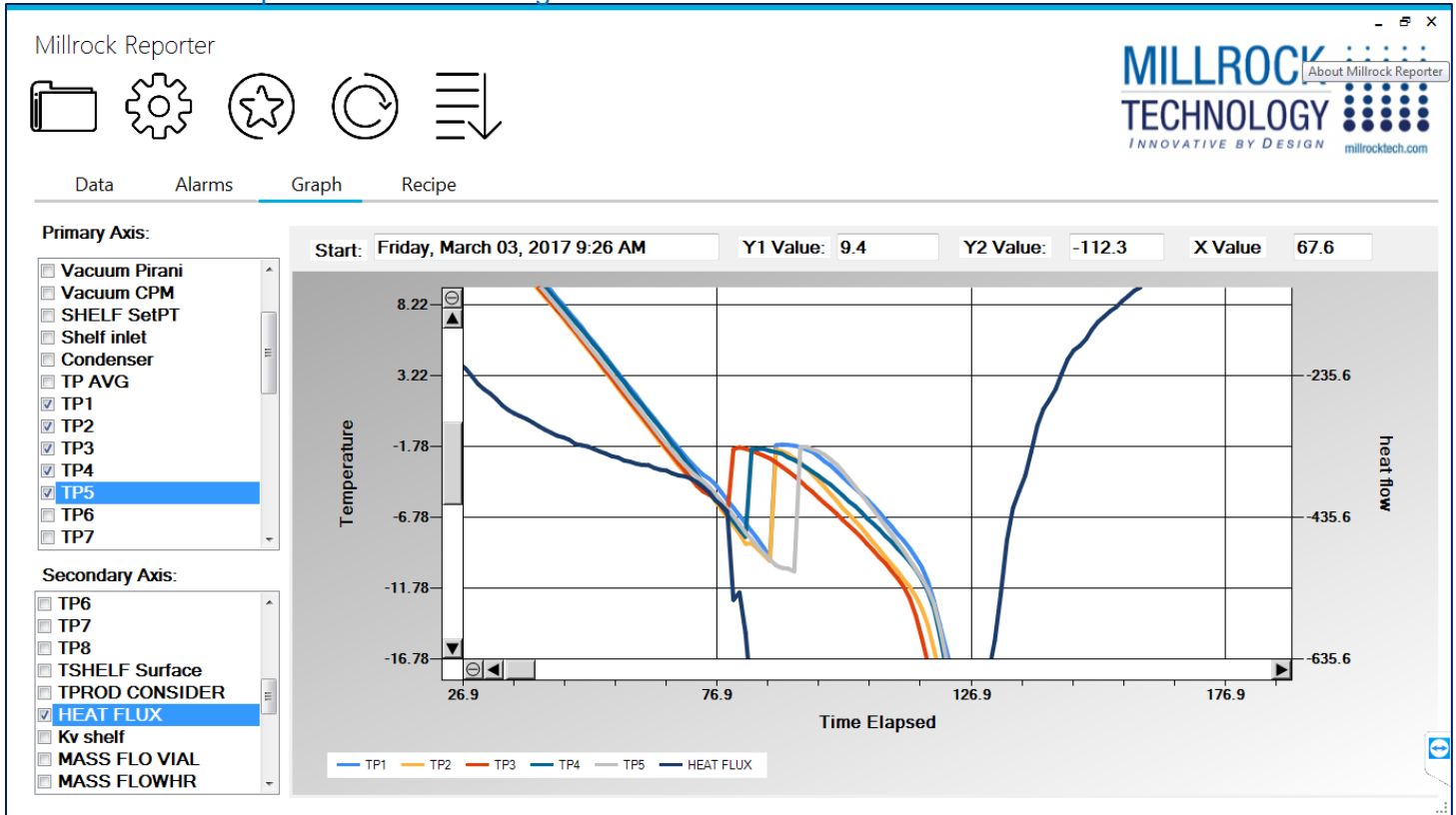


Expanded view of product temperatures and heat flow.



Expanded view allows a better view of the details.

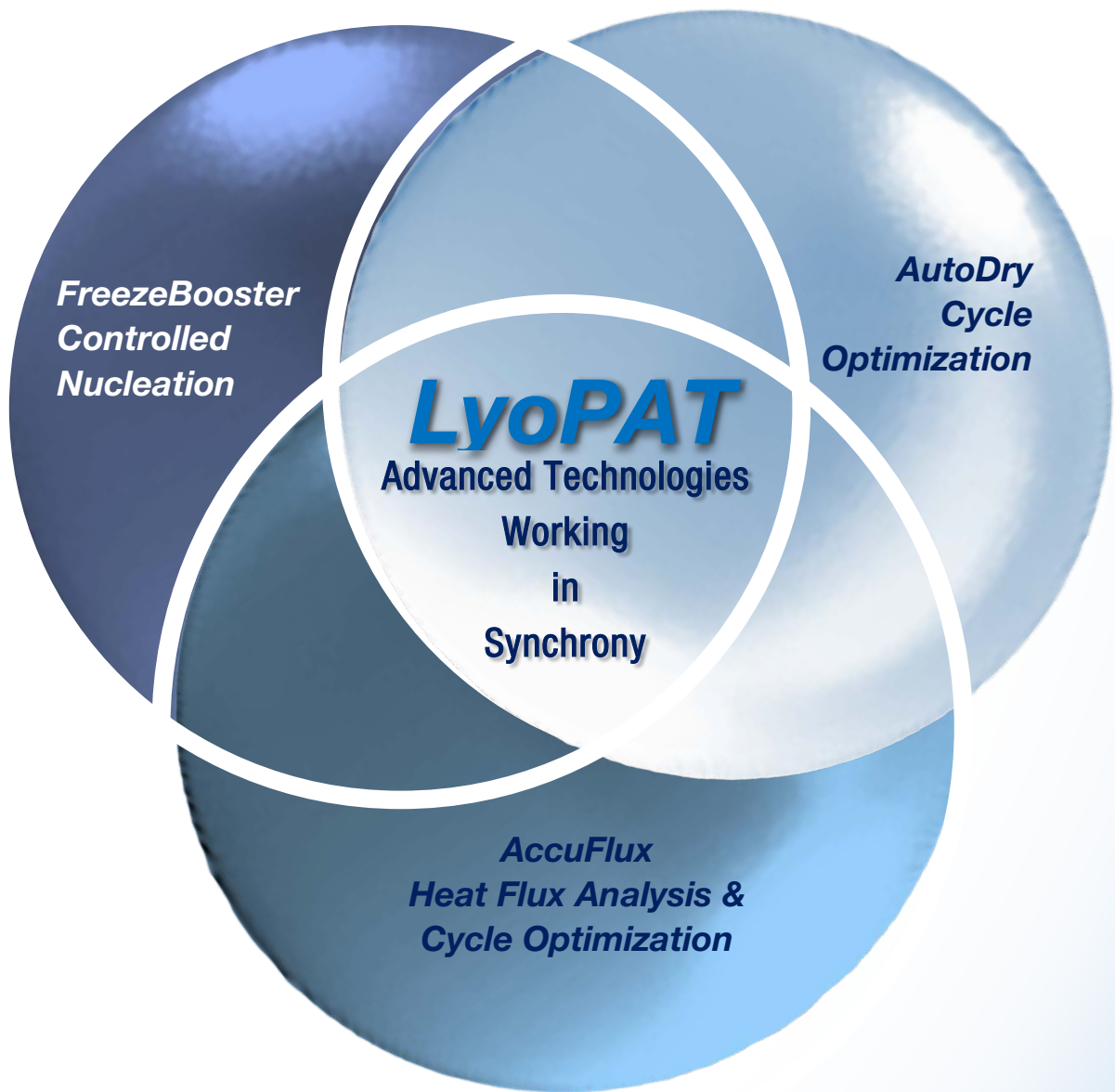
Product thermocouples & heat flux during nucleation



Detailed alpha-numeric display and print out.

The table displays detailed data for the current batch and recipe. The columns represent various process parameters and their final values. The data is organized into rows for the current batch ID (Mannitol/FTA) and the current recipe (sucrose/mannitol). The table includes a 'Column Width' dropdown menu and a list of parameters to be displayed, such as PointTime, BatchID, Cycle Time, Cycle, Phase, Step, VACUUM SetPT, Vacuum Pirani, Vacuum CPM, SHELF SetPT, Shelf inlet, Condenser, TP AVG, TP1, TP2, and TP3.

Current Batch ID	Shelf inlet	Condenser	TP AVG	TP1	TP2	TP3	TP4	TP5	TSHELF Surface	HEAT FLUX	Kv Final
Mannitol/FTA	23.86	23.29	23.69	23.56	23.81	999.9	999.9	999.9	23.84	-22.89	0
Current Recipe	24.56	23.44	23.6	23.54	23.69	999.9	999.9	999.9	23.91	4.95	0
sucrose/mannitol	24.89	23.44	23.6	23.56	23.64	999.9	999.9	999.9	24.11	22.47	0
Column Width	25.04	23.49	23.65	23.61	23.69	999.9	999.9	999.9	24.31	32.65	0
	24.99	23.02	23.74	23.69	23.79	999.9	999.9	999.9	24.36	26.82	0
	23.69	23.16	23.79	23.71	23.84	999.9	999.9	999.9	24.01	-18.04	0
	17.52	23.26	23.04	23.14	22.94	999.9	999.9	999.9	20.14	-324.27	0
	9.36	23.39	20.91	21.22	20.54	999.9	999.9	999.9	14.05	-670.08	0
	2.27	23.44	17.47	18.05	16.83	999.9	999.9	999.9	7.69	-876.55	0
	-3.86	23.51	13.43	14.25	12.58	999.9	999.9	999.9	1.72	-972.04	0
	-5.29	12.13	9.57	10.51	8.64	999.9	999.9	999.9	-1.68	-838.71	0
	-5.23	-49.35	6.56	7.48	5.61	999.9	999.9	999.9	-3	-647.65	0
	-5	-70.73	4.36	5.17	3.46	999.9	999.9	999.9	-3.47	-497.64	0
	-4.95	-71.51	2.77	3.49	2.06	999.9	999.9	999.9	-3.81	-402.88	0
	-4.97	-71.57	1.6	2.21	0.99	999.9	999.9	999.9	-4.07	-336.05	0
	-5.03	-71.61	0.67	1.2	0.14	999.9	999.9	999.9	-4.33	-293.65	0
	-5.05	-71.57	-0.07	0.4	-0.54	999.9	999.9	999.9	-4.53	-258	0
	-5.05	-71.42	-0.71	-0.3	-1.11	999.9	999.9	999.9	-4.66	-225.46	0



LyoPro Freeze Dryers

We traded in one of our freeze dryers for a Revo SL with LyoPAT and we were amazed at how versatile the freeze dryer was and how helpful it was in our lab.

Revo Pro & Magnum Pro

Shelf System

Construction: 316L Stainless Steel.
Shelf finish 20 Ra or better.
Chamber finish 20 Ra or better.
Platinum cured silicone gasket
Pre-seal vacuum check.
Hydraulic stoppering ram, bottom up.
Sloped to drain tray chamber
Increased stoppering pressure for hard to stopper vials.
Partially open side panels to allow more uniform vapor flow.
6 inch (15.24 cm) vapor port to condenser
Throughput of XX
Pneumatic butterfly type Isolation valve.
2.5 KF sanitary fitting for residual gas analyzer connection on vapor port
Extra 2.5KF sanitary fitting on top of chamber
Capacitance manometer and Pirani gauge on chamber
Temperature of -70C to +65C (-60 to +65C outside of US)
16 Product sensors – “T” type thermocouples
One set of product thermocouple holders for 20 mm vials
One set of product thermocouple holders for 13 mm vials
Side dome with viewing port for nucleation station attachment.
Temperature stability: resolution:
Shelf temperature uniformity of
Shelf pulldown from +20 to -40C in 30 minutes
Leak rate of less than XmT/hour/volume
1 removable bottom tray per shelf

Condenser

Construction: 316L Stainless Steel.
Sloped to drain.
Exposed coil with direct refrigeration.
Temperature reading from coil surface inside chamber.
Pirani vacuum sensor.
Condenser capacity of 30L
Maximum condenser rate of 20L/24 hours.
Platinum cured silicone gasket
6 inch (15.24 cm) vapor port to chamber
Extra 2.5KF sanitary fitting on top of chamber
Temperature dry and empty of -85C
Automatic hot-gas defrost



Refrigeration System

Highly reliable scroll compressors.
Oversized compressors for enhanced performance.
CFC free non-proprietary refrigerants.



*the most
dependable and
robust
cooling
&
heating
system
available
in a
freeze dryer
today*

Vacuum System

375 lpm corrosion resistant vacuum pump
Oil mist eliminator on vacuum pump outlet
2 stage rotary drive oil sealed pump standard
Optional 300 lpm dry pump

Revo Pro Shelf Surface Area

# of Shelves	Shelf Size In (cm)	Total Shelf Area ft ² (m ²)	Distance Between Shelves In (cm)
1	12 x 24" (30.48 x 60.96 cm)	2 ft ² (0.186 m ²)	14.5 in (36.8 cm)
2		4 ft ² (.0373 m ²)	7.0 in (17.7 cm)
3		6 ft ² (0.557 m ²)	4.5 in (11.4 cm)
4		8 ft ² (0.743 m ²)	3.25 in (8.2 cm)
5		10 ft ² (0.929 m ²)	2.5 in (6.3 cm)



Revo Pro Vial Capacity

Vial ml	Dia mm	Height mm	Number of shelves				
			1	2	3	4	5
2	16	41	774	1548	2322	3096	3870
5	22	48	403	806	1209	1612	2015
10	24	58	322	644	966	1288	
20	29	74	218	436	654		
50	43	81	96	192			
100	52	92	65	130			

Ask about our shelf latching kits if you need to change the distance between shelves.

Magnum Pro Shelf Surface Area

# of Shelves	Shelf Size In (cm)	Total Shelf Area ft ² (m ²)	Distance Between Shelves In (cm)
5	12 x 24" (30.48 x 60.96 cm)	10 ft ² (0.93 m ²)	5.5 in (139 cm)
6		12 ft ² (1.10m ²)	4.5 in (114 cm)
7		14 ft ² (1.30 m ²)	3.75 in (95 cm)
8		16 ft ² (1.48 m ²)	3.25 in (82 cm)
9		18 ft ² (1.67 m ²)	2.8 in (71 cm)
10		20 ft ² (1.86 m ²)	2.5in (63.5 cm)

Magnum Pro Vial Capacity

Vial ml	Dia mm	Height mm	Number of shelves					
			5	6	7	8	9	10
2	16	41	3870	4644	5418	6192	6966	7740
5	22	48	2015	2418	2821	3224	3627	
10	24	58	1610	1932	2254	1744	2898	
20	29	74	1090	1308	1526	768		
50	43	81	480	576	672			
100	52	92	325	390	455			



Options for Revo Pro & Magnum Pro

Sample extractor – allows removal of vials for residual water studies. Front door mounted.

Water Cooled Compressors – for labs and facilities that do not have enough air conditioning for proper air cooling of the equipment.

Clean room or isolator configuration.

Validation Documentation

IQ/OQ Workbook

Factory Acceptance Test and/or Site Acceptance Test

Liquid Nitrogen Trap for light non aqueous solvent loads such as ethanol or tfa

Clean-In-Place

Hydrogen Peroxide Sterilizer connections

We offer a full line of freeze dryers, from BenchTop freeze dryers to the Production Steam-Sterilized Lyophilizers



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.

The LyoSight section of our website provides the freeze drying community with

- Technical Papers and Presentation
- Webinars on Topics of Interest to scientists who freeze dry
- 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, Inc. 39 Kieffer Lane Kingston, NY 12401

+1.845.339.5700 www.millrocktech.com info@millrocktech.com sales@millrocktech.com