LAB-3

CENTRIFUGAL MOLECULAR
DISTILLATION SYSTEM

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INTRODUCTION

Myers Vacuum is pleased to present this proposal for the LAB-3 centrifugal distillation system for your consideration.

Short Path Molecular distillation is a purification technique which has gained wide acceptance in the chemical, food processing, pharmaceutical and petroleum industries as well as the specialty chemicals industry.

The principle behind the Myers Vacuum Molecular Stills is low pressure (Vacuum) and extremely short residence time. Once the feed material has been degassed, it flows onto the center of the spinning heated disc. Evaporation of material takes place in less than 1 second. As the material moves out on the radius of the disc, the surface area increases exponentially increasing the area per volume exposing more material to surface for evaporation. The residue is then caught within a gutter surrounding the rotor and flows down to the collecting vessel below. The distillate condenses on the outer shell of the still and flows by gravity to another collecting vessel also below the still.

The major advantages that can be expected from the Lab-3 Molecular Still are:

1) High product percentage yield. Close to 1 theoretical plate.
2) Elimination of color bodies.
3) Elimination of odor fractions.
4) Reduction of acid values in ester distillation.
5) Removal of excess reactants.
6) Elimination of residual metal catalysts.
7) Minimized thermal hazard (a necessity for heat sensitive compounds).

The still itself may be purchased separately or with controls and pumping system.

The Lab-3 has a 3 inch diameter rotor with a throughput capability of 0 - 2 lbs/hour. The Vacuum pumping system maintaining pressure around 1 X 10^{-3} Torr pressure.

FEATURES

There are several important design features on the Lab-3 that make it an outstanding tool in the laboratory:

1) Simple to clean. - Mostly stainless steel so there is little glass to break. No gear pumps to clean or leak.
2) Modular design allows for easy set up and moving location.
3) Sits on a lab bench and takes up very little space.
4) Sight ports on both Degass and main chambers so one can observe the process as it occurs.
5) Feed material flows from a degasser feed chamber to the still below, where distillation takes place, and then, to collection vessels directly beneath the still. The material flows by gravity in a vertical path.
6) Can easily be heat traced for higher melting point products.
7) Information learned on the Lab-3 may be used in scaling up to larger sized equipment.
8) Distills batches as small as 50 ml or up to 2 lbs/hr.

OPERATING OBJECTIVES

The Lab-3 is designed to distill thermally sensitive organic and silicone compounds in the molecular weight range of 150+ AMU at vacuums from 1 X 10^{-3} to 700 Torr. Some examples of distillation from the Lab-3 are as follows:

1) Fatty acid amides
2) Dimer acids
3) Epoxy resins
4) Drying oils
5) Monesters
6) Di-octyl phthalate
7) Vitamin A
8) Tocopherols (Vitamin E)

The following pages contain a detailed description of the various sections of the Lab-3 system.

MATERIAL HANDLING

The Lab-3 high vacuum still is designed as a batch / continuous distillation unit. Figure 1 illustrates the flow of the process material through the still. Green designates Feed material, Blue Residue and Yellow distillate.

The material flows by gravity from the Degasser reservoir: where the material is also degassed by removing small amounts of low boiling materials and trapped gases to enable more efficient vacuum distillation at the next stage. The material then flows via the rotor feed valve (17) on to the center of the heated, spinning rotor (5) in the high vacuum distillation chamber. On the rotor, the material spreads out into a thin film. As the material spreads across the rotor, a certain portion (the distillate) evaporates. The fraction which distills is selected by the operator by setting the temperature of the rotor . Once set, this remains constant until the run is completed or until new conditions are selected.

The distillate vapor condenses on the condenser in the vacuum chamber and flows from the system via the distillate collect valve (15) into the distillate collector (14).

The portion which does not distill (the residue) moves off the edge of the rotor to be trapped by the gutter and flows from the still via the residue collect valve (16) into the residue collector (13). The residue may be recycled by opening the recycle valve (18).

Depending upon the feedstock material or process requirements, either the distillate or the residue may be your final product. In many cases both may be of value.

Process Flow Valves

Numbers in parenthesis ( ) refer to item numbers in Figure 1.

Rotor Feed Valve(17)	Controls flow rate of feedstock from reservoir to rotor.
Distillate Collect Valve(15)	Controls flow of distilled material from chamber to collector vessel.
Residue Collect Valve(16)	Controls flow of residue material from chamber to collector vessel.
Residue Recycle (18)	Controls recycling of residue to feed stock reservoir.
Degasser Feed (19)	Feeds material into degasser and increases pressure in degasser.

VACUUM SYSTEM

The Lab-3 is designed to distill materials in the pressure range from 1 X 10^{-3} Torr to atmosphere. The lower the distillation pressure, the lower the heat required to effect evaporation, the lower the chance of damaging the product and the lower the energy costs of the purification process.

The Lab-3 includes a complete, fully valved vacuum system (See Figure 1 Purple lines). This fully valved system allows the operator to isolate certain parts of the system while making desired adjustments, thus eliminating the need to completely shut the system down. All manifolds are stainless steel with standard vacuum fittings for easy disassembly and reassembly for cleaning.

The mechanical pump is a direct drive rotary vane vacuum pump. It is used to pump the foreline (exhaust) of the diffusion pump (High vacuum), and to rough pump the degasser, the distillation, the distillate and the residue chambers.

The High Vacuum pump is a rugged 2" diffusion pump enabling the distillation chamber to operate under high vacuum. An automatic temperature controller monitors the output water temperature of the diffusion pump and meters the flow of cooling water for maximum pump efficiency and minimal amount of water usage.

The trap valve removes easily condensable-vapors, or low boiling volatiles so as not to contaminate the oils in the mechanical and diffusion pumps and to prevent oil in those pumps from back streaming into the chamber.

Vacuum Pumping System Assembly;

Numbers in parenthesis ( ) refer to item numbers in Figure 1.

CHAMBER TRAP (9)	Liquid Nitrogen trap to Isolate/protect the Vacuum pumps from any product vapor
TRAP VALVE (9)	Isolates vacuum pumping system (including trap from Still assembly).
HI-VAC VALVE (8)	Part of a three position valve. Isolates diffusion pump from Trap Assembly.
FORELINE VALVE(7)	Part of a three position valve. Isolates diffusion pump from mechanical vacuum pump.
ROUGHING & TRAP VALVE (20)	Allows pressure in trap and still assembly to be "rough pumped" (reduced by

mechanical vacuum pump).

DEGASSER ISOLATION VALVE (10) Isolates degasser from vacuum system

DISTILLATE COLLECTOR 3 POSITION VALVE (12) Controls rough pumping and venting of Distillate collector.

RESIDUE COLLECTOR 3 POSITION VALVE (11) Controls rough pumping and venting of residue collector.

Other Valves (on front of Pumping System Assembly)

CONDENSER WATER VALVE Controls flow of cooling water to distillation chamber condenser.

DIFF PUMP COOLING Turns on/off cooling water to diffusion pump. Control of water flow is automatically controlled for most efficient operation

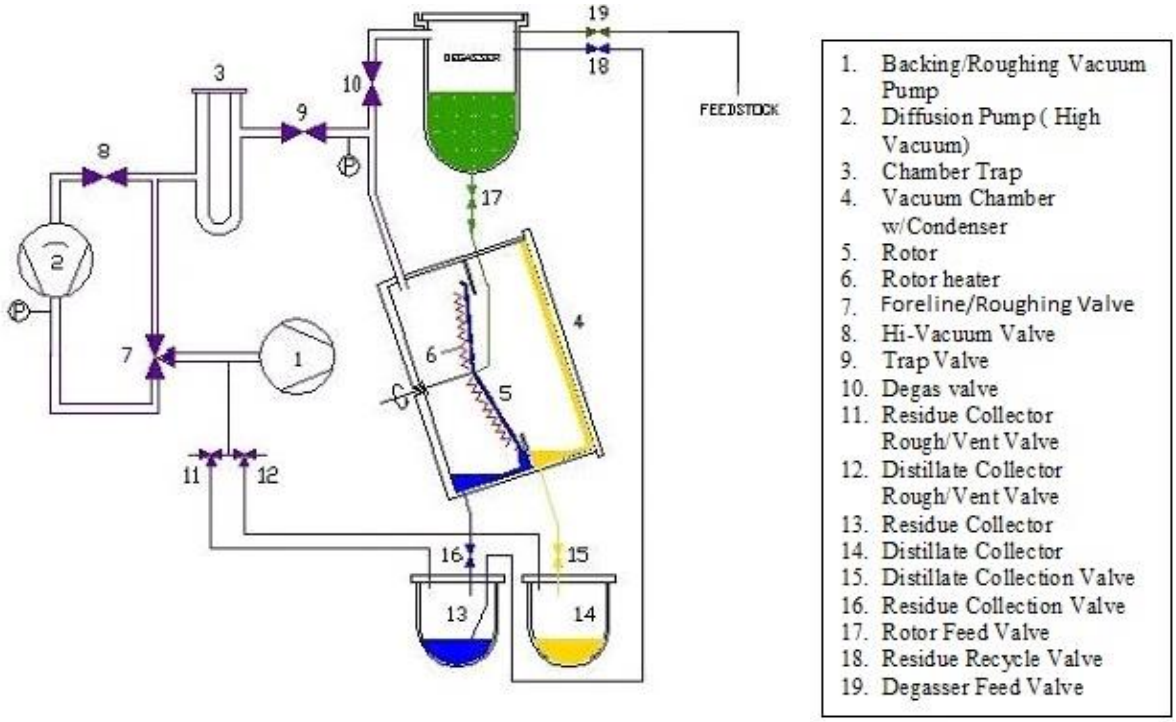


Figure 1

CONTROL CENTER

The Lab-3 control center contains all of the controls and readouts required for operation:

Vacuum Readout: There is a two (2) - station pressure monitoring section with leak detect mode. This enables the operator to get accurate pressure measurements in the distillation chamber and on the foreline. Plus leak detect function simplifies system check out after reassembly.

Temperature Readout: There is an SCR temperature controller which enables the operator to set and control the temperature of the rotor. The thermocouple is located inside the rotor for true film temperature measurement.

The control center also provides a variable power switch for the addition of heat tapes if desired.

CONTROL SWITCHES AND THEIR FUNCTION

The following list identifies each control and explains its function. Capital letters identify the how control is labeled on the still.

Switches on front of instrument module;

MECH. PUMP (1)	Power to motor of mechanical vacuum pump.
DIFFUSION PUMP (2)	Power to diffusion pump heaters. Interlocked to mechanical pump so heaters will not be energized unless MECH PUMP switch is on.
ROTOR MOTOR	Power to rotor motor.
ROTOR HEATER (6)	Power to rotor heater through controller that regulates the rotor temperature.
AUX. POWER	Power from variable controller for use by customer. (Resistance Loads only)

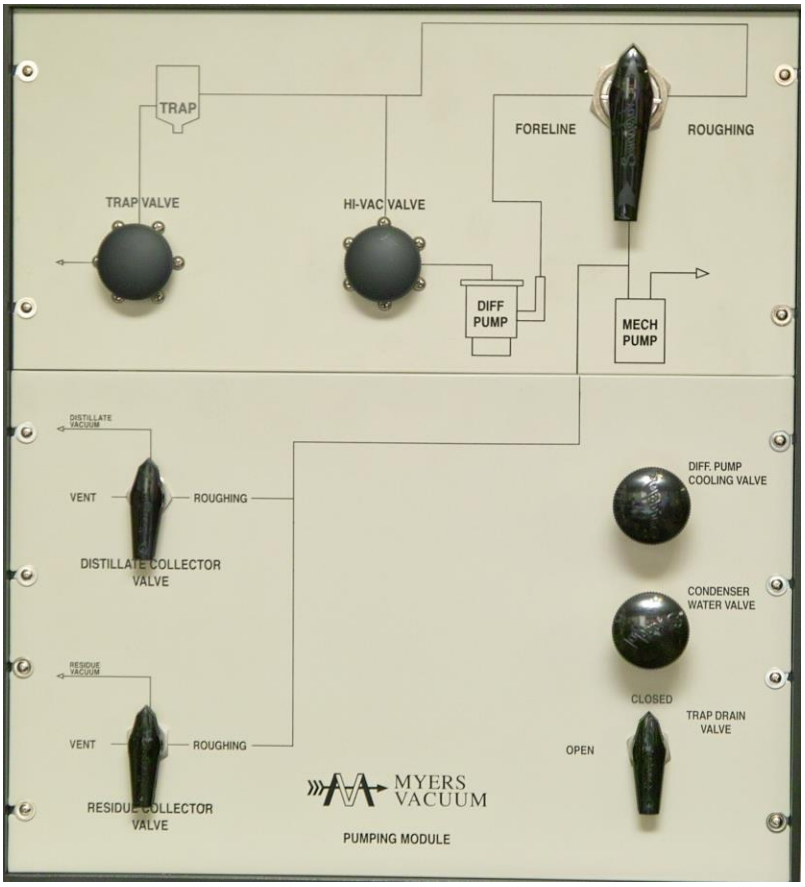
Digital temperature display on front of instrument module

TEMPERATURE CONTROLLER	Monitors temperature and adjusts power as required to maintain temperature of rotor/product.

COMPLETE LAB - 3 SYSTEM STANDARD BENCH MODEL

STILL ASSEMBLY: Includes degassing feed chamber (volume .42 Liters maximum),
3" diameter heated rotor, water-cooled condensing surface, feed valve, distillate / residue valves, and distillate / residue collecting vessels (volume .48 Liters maximum each). The residue collecting vessel is equipped with a recycle line returning to the degassing chamber for multiple pass operation. The still components are mounted on a stand and are easily accessible for cleaning and maintenance. Wetted components are stainless steel, viton seals and a minimum of glassware.

PUMPING SYSTEM: Includes 2" diffusion high vacuum pump, mechanical pump, vacuum cold trap, vacuum manifold with valves, and water manifold with valves. It is mounted in a cabinet which connects to the Still Assembly via flexible hoses.



CONTROL CENTER: Includes thermistor vacuum gauges, digital temperature controller, individual switches for heater, Rotor rotation, vacuum pumps (mechanical and High Vacuum) and a switched variable power outlet for use with the addition of heat tapes.

PERFORMANCE SPECIFICATIONS

Throughput Capacity:	0 - 2 lbs/ hr (.9 kg/hr)
Design:	Batch/ Continuous

PUMPING SYSTEM:

Ultimate Pressure	$< 1 \times 10^{-3}$ Torr
Operating Pressure	$1\text{-}10 \times 10^{-3}$ Torr to Atmosphere
Mechanical Forepump	3.4 CFM
High Vacuum Pump	285 liters/sec

UTILITIES:

Power (60 cycles)	115 Volts, 50/60 Hz, 3 Wire 1 Phase, 20 Amps

Available for 230 volt, 50 hertz service.

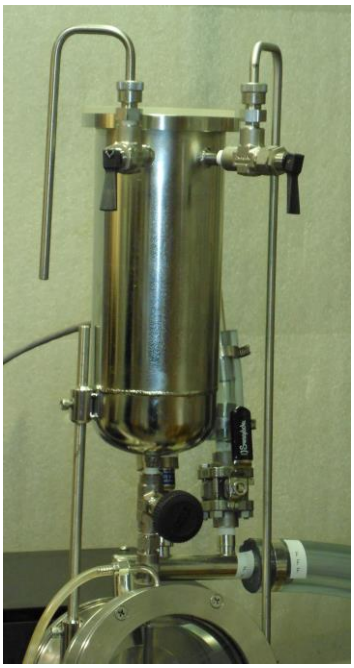
Power Consumption	1.0 KW/H
Water (25°C)	0.5 gal / min. (1.9 liters/min)

DIMENSIONS: For Still Assembly

STILL ASSEMBLY	Height	25 in. (64 cm)
	Width	11in. (28 cm)
	Depth	16in. (41 cm)
	Weight	50 lbs. (23 kg)
	Rotor Size	3 in. (7.6 cm) Diameter
CONTROL ASSEMBLY	Height	11 in. (28 cm)
	Width	10.5in. (28 cm)
	Depth	11in. (27 cm)
	Weight	25 lbs. (11 kg)
PUMP MODULE	Height	22 in. (56 cm)
	Width	20 in. (51 cm)
	Depth	18in. (58 cm)
	Weight	82 lbs. (37 kg)

OPTIONS

- Step up transformer for operation at Voltages other than 115 VAC
- Heat Tape to wrap feed tank, Distillate and Residue collectors – With adapter to plug into control unit for operation
- Extra Trap can be added to the vacuum line in series with the main trap and Vacuum pumps This will allow operation with cooling recirculator at specific temperature to make a separate cut of vapor material



Larger Feed/Degasser Tank to hold 1 Liter Material

Larger Distillate and Residue collectors to hold 1 liter material each