



A company of the  
**GERRESHEIMER** group

**Instructions for  
Glass Oil Diffusion Pumps  
924770, 924780,  
924785, 924795**

Instruction Sheet Number 9247895

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### **Operating Characteristics**

Multi-stage fractionation type pumps provide low ultimate pressure through the use of alembics in the outlet tube just above the condensing zone for the vapor jet, and individual boilers for each stage. Fluid flows through the boilers in series starting nearest the pump outlet. The most volatile components delivered to the nozzle nearest the pump inlet. The final boiler (tar trap) is not connected to a jet and collects the nonvolatile residues.

### **System Assembly**

After unpacking pump, examine carefully for any physical damage incurred in shipping such as bump cracks, seal damage, broken heater coils, etc. Leak check vacuum system by pumping down with a mechanical pump and testing with a leak detector.

### **Filling Pump**

Clamp pump in place temporarily and fill boilers through the filling port in the outlet end with a long stem funnel, being careful to keep fluid out of the alembics. Adjust pump until all heater coils are covered evenly with about 3/16" of pump fluid. (For a pump already sealed to a system and provided with a cleanout tubulation, open tubulation and attach a length of clean rubber tubing with one end in container of fluid. Turn on mechanical pump until proper amount of fluid is drawn in. Close tubulation with rubber plug.

The pump should now be sealed directly to the vacuum system by a skilled glassblower in order to assure a leak free system capable of attaining the lowest ultimate pressure.

### **High Vacuum Side**

The inlet end of the pump is the large diameter tube angled slightly from the horizontal. For the maximum pumping speed, the system must be sealed in at this point. In most cases, maximum speed is not required, and for convenience, a smaller diameter vertical tube is inserted to connect to the system which is usually located above the diffusion pump. This right angle bend is usually enough to condense any backstreaming oil vapor and return it to the pump boilers. If necessary, forced air or water cooling can be provided.

### **Low Vacuum Side**

The outlet end is a vertical tube consisting of the following: several alembics for collecting the more volatile fractions of the pumping fluid, a vertical fill tube which can be closed off after filling or connected to a mechanical pump. This connection is best accomplished by making a connecting line of the same size glass tubing slipped inside a short length of rubber tubing at both ends to make butt joints between pumps. If pumping large amounts of water vapor or volatile solvents, provide a dry ice and acetone trap in the line to prevent excessive forepressure due to contamination of the pump fluid.

A mechanical pump with free air capacity of 10 to 50 liters per minute is adequate for backing almost any glass oil pump. Ultimate pressure should be less than 10 microns.

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### **Power**

Although listed wattage is obtained at line voltage (110-120 vac.), pumping speed and pressure will probably be obtained at some value less than 115 vac. A variable transformer such as 730900 must be provided to control heater input. Voltage input should be monitored with a voltmeter. Heater coils are wired in series and should be operated with boilers insulated as delivered from the factory. Variation of heater voltage affects pump speed, ultimate pressure, and forepressure breakdown.

### **Cooling**

For air cooled models, a small electric fan such as a muffin fan or a compressed air line can be directed at the area where oil vapor condenses after it leaves each jet.

Water cooling pumps are cooled with tap water below room temperature (20°C) entering the bottom of the jacket at the inlet end and flowing in series to the outlet end. Jackets are connected in series with rubber tubing.

### **Outgassing**

Outgassing the system is initiated reducing pressure below 200 microns with the mechanical pump. Turn on cooling and apply heat to degas pump fluid. As gas is evolved, forepressure will rise, but do not allow it to exceed 750 microns. Turn off heaters until pressure is below 500 microns. Condensate will rise up inside of vertical jet as fluid reaches operating temperature. Pump pressure will fall below forepressure as condensation takes place below the first alembic. If forepressure does not fall to near leak check pressure after continuous pumping, check for connection leaks or mechanical pump contamination due to moisture or pump fluid decomposition.

In order to obtain pressures below  $5 \times 10^{-7}$  torr, the system walls should be torched to 300°C when the pump begins operating. Pressures as low as  $1 \times 10^{-8}$  require torching as 500°C for an hour or more while using liquid nitrogen in the trap. Use extreme caution to heat the glass evenly, with system at reduced pressure, to avoid cracking or deforming glass.

### **Operating Technique**

Normally the lowest pressure attainable in the system at any given time is the indication that the pump is operating efficiently; therefore, the high vacuum gauge is the ultimate answer in determining correct heater input. Proper heater operation can be gauged quite accurately by observing the position and thickness of the condensate ring formed on the wall below the first alembic by the vapor expanded from the wall below the first alembic by the vapor expanded from the vertical jet. With proper cooling and forepressure, the ring should "break" about 20 to 30 mm above nozzle exit. If the ring breaks above this area, it indicates insufficient cooling. If the ring is heavy with large droplets, reduce heater input, while a difficult to define ring requires more heat. A low, fluctuating ring indicates high forepressure with the present heat input and heat should be increased.

To insure reaching ultimate pressure, check forepressure and be sure heater coils are covered with oil. It may take several hours to reach the lowest ultimate pressure (5 to 10 hours with 2 and 3 stage fractionating pumps).

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### Maintenance

When opening system to atmosphere, the glass above the boilers should be cool enough to touch to prevent decomposition of the pump fluid. Decomposed fluid should be removed if it has a burned odor and ultimate pressure is excessive.

After draining pump, it should be rinsed several times with acetone. To remove discoloration and deposits from burned fluid, use a hot chromic acid solution, being careful to keep heating coil exposure to the acid at a minimum. Torching the caked on deposits lightly will assist in their removal.

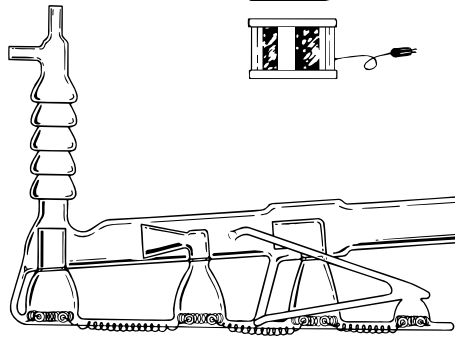
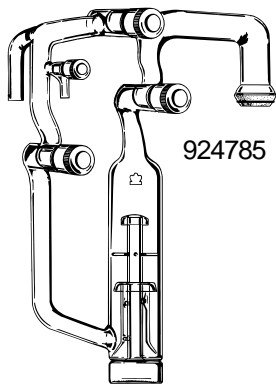
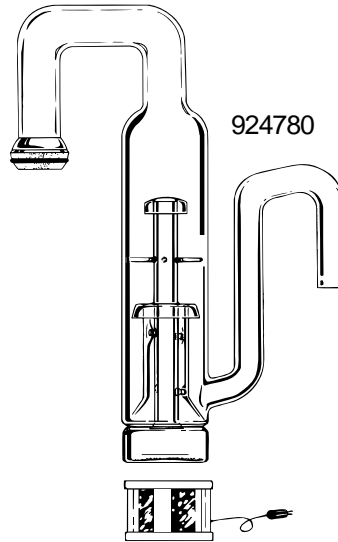
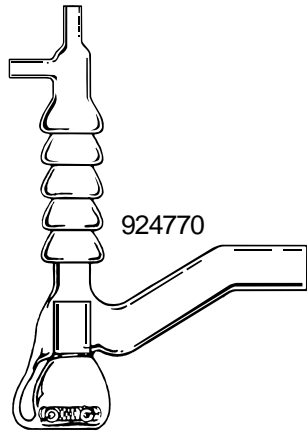
Light deposits may wash benzene with a final rinse with C.P. grade acetone. Vapors should be removed by purging with compressed air or a mechanical pump.

If deposits do not yield to above treatments, wash pumps with detergent and rinse for up to 5 minutes with dilute (10%) hydrofluoric acid. Flush with water and rinse with acetone. Dry with compressed air.

### OIL DEFFUSION PUMP SPECIFICATIONS

	924770	924780	924785	924795
Ultimate Vacuum	1 x 10 <sup>5</sup> mm or better	1 x 10 <sup>6</sup> mm or better	1 x 10 <sup>6</sup> mm or better	1 x 10 <sup>7</sup> mm or better
Pumping Speed	8 liters/sec. at 10 <sup>4</sup> mm	20 liters/sec. at 10 <sup>6</sup> mm	20 liters/sec at 10 <sup>6</sup> mm	30 liters/sec. at 10 <sup>4</sup> mm
Recommended Oil	923655 D.C. #705	923655 D.C. #705	923655 D.C. #705	923655 D.C. #705
Heater Rating (Max.)	50 w at	250 w at	60 vac	110 vac
Forepressure Required	40 Microns	50 Microns	50 Microns	75 Microns
Coolant	Air	Air	Air	Air
Height	14 ½" (37cm)	15" (38cm)	10 5/8" (27cm)	14 ½" (37cm)
Width	8 5/8" (22cm)	10" (25.5cm)	9 ¼" (23.5cm)	24" (61cm)
Forepump Connection	15 mm O.D.	16 mm O.D.	16 mm O.D.	15 mm O.D.
High-Vacuum	32 mm O.D.	Ts 35/25	Ts 35/25	32 mm O.D.

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### **Guarantee**

Kontes will promptly replace any product found to be defective or not in conformance with published specifications.

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