

ASSEMBLY & OPERATING INSTRUCTIONS FOR 9331-9333 CONCENTRIC TUBE FRACTIONATING UNIT

BILL OF MATERIALS (9333):

1. Vacuum jacketed section — Trubore® with double expansion coil to eliminate chocking.
2. Centerless ground precision center tube with distribution funnel and lower centering dimple which co-acts with upper centering knobs to maintain alignment.

In Addition to the Above (9331):

3. Distilling Head — partially vacuum jacketed supplied with a thermocouple/thermometer well and thermometer, an iron-core takeoff plunger, solenoid coil, coil support (nylon) and spherical joint clamps (7666).
4. Distilling Receiver, Catalog No. 9375
5. Graduated Cylinder (2), Catalog No. 9396
6. Distilling Receiver, Catalog No. 9397
7. Instatherm® Distilling Flask, 50 mL, with Clamp No. 9695-10 and Cord, No. 9698
8. Thermometer for above, Catalog No. 8285-16, 0-260°C
9. Three taper-joint clips for pot receiver and cylinder, Cat. No. 7600-15

ASSEMBLY:

1. **Caution!! The Silvered Column Jacket is evacuated. If broken, it will implode violently. When clamping in position, it is advisable to use a spring clamp or spring loaded chain clamp to avoid overtightening.**

Use extra care in unpacking the center tube, as the funnel stem is fragile. The head plunger must also be handled gently.

2. Flush the silvered section with solvent and dry. Wipe the center tube with a lint-free cloth dampened with solvent, or water with a little detergent added, to remove dirt. Be careful of the funnel end — do not wipe the funnel.

3. When dried, place the jacket at an angle of about 30° to the horizontal and insert the dry center tube gently, holding it well below the funnel (dimpled end goes in first) and adjusting the angle so that the center tube barely slides in. Do not release the Center Tube if you can feel much its weight with two fingers grasping it. Slide the Center Tube in and tap the Column gently to be sure that the dimple is firmly seated on the internal stem before bringing the Column to a full upright position. When the Center Tube is in the proper position, the top of the funnel will be approximately in the center of the upper bulb and the position of the dimple will be visible through the viewing stripe with the aid of a strong light (this is not already reliable).

Note! Do not assemble the parts when wet. Surface tension tends to hold the parts together and the tube will not slide in smoothly.

4. Clamp the Column in position and gently insert the head joint to be sure its drip tip does not touch the funnel. If this occurs, re-seat the Center Tube in the Column and try again. If the situation persists, return your Column to ACE for replacement or have us send another Center Tube. Center Tubes are not exactly interchangeable because the drip tip should be in close proximity to the funnel. The Columns are assembled and tested in our lab for proper operation prior to shipping.
5. Assuming the Head fits properly, align the Column vertically with a plumb bob which can be made from a string and weight. The column MUST be plumb for efficient operation. If it is not plumb, channeling will occur because of the influence of gravity.
6. Assemble the Head. First attach the bypass tube by means of the spherical joint clamps. Then insert the Plunger after cleaning the ball and dipping the ball in solvent allowing some to adhere. When the ball seats, twist it gently to see if you feel any grit in the joint; it should turn smoothly. If dirt is present, flush it out with solvent and gentle agitation of the plunger. Be careful when manipulating the plunger so that you do not break the upper drip tip that is positioned so that returning liquid will bridge over to the plunger.

Place the Head on the Column and then prepare the Coil Assembly. Tighten the split ring coil support over the Plunger Cap so that is snug enough to support the coil but still movable. Place the Cap Assembly over the Plunger, lightly greasing the joint. Place the Coil in position resting on the support and move the support so that the top of the Plunger can be just seen. Then plug the Coil into 120V source while adjusting the Coil support so the coil force just lifts the Plunger (not more than 1 mm). Unplug the coil wire. The Coil is made for a 75% duty cycle without overheating. It should never be left activated. Plug the Coil into a repeat cycle timer, the OFF-ON ratio of which will determine the reflux ratio in operation. The reflux ratio is thus fixed on a time basis and is independent of throughput rate. The Coil can be adjusted so that the Plunger will split drops in short "ON" times.

7. Support the Receiver when attaching it to the Head. A spring clamp is best used here so that the column is not forced out of line. Bring it up to rest under the side arm. Use one of the spring clips to secure the joint, placing the open ends under the ridge of the female member.
8. The Cylinder is attached to the Receiver and secured by a clip only. For non-vacuum operation, a slight twist will “lock” an ungreased joint; untwisting will release it. Use the second clip as a safety measure.
9. When ready, attach the Instatherm Flask to bottom of Column. Use glycerine in the thermowell to provide thermal conductivity.

OPERATION:

1. After assembly, check the operation of the Unit by distilling a few mL of Xylene or other material boiling in the range of 130°–150°C, This will give an indication of the heat loss from the Column and that the reflux is returning properly.

With Xylene refluxing, practically no heat should be felt emanating from the center of the jacket. The top and bottom will be uncomfortably warm at the shoulder and slightly warm one inch from it.

At about 180°C, radiation losses from all vacuum jackets make it necessary to provide additional heat or insulation, particularly at the top.

Even though the Column is sealed into the jacket, using a lathe, the inner tube is not necessarily exactly parallel with the outer jacket and slight corrections in posture may be required by moving one clamp to obtain uniform concentricity of the reflux.

2. It is understood that the operator will lubricate the joints. In order to minimize wash-out, it is good practice to grease only the outer portion of the joint. Additionally, special lubricants are available which are insoluble in hydrocarbons. If Teflon sleeves are used, trim to size with a razor blade after applying.
3. Calibrate the drip tips so that reflux rates can be determined. Most efficient operation is obtained with boil-up rates between 20 and 130 mL/min.

This is easily accomplished by removing the Plunger Cap and introducing a measured volume, drop by drop, onto the Plunger counting the drops from each tip, after wetting the Column and allowing it to drain until drops stop.

4. Do not operate the Column in a drafty location. The small volumes and heat inputs are significantly affected by low velocity air flow. In this regard, it should be noted that Instatherm response is more rapid than that of the temperature sensors. Use change in reflux rates as the sensitive indicator.

5. Be certain the system is open to the atmosphere or a pressure regulator before starting operation. Sudden surges of "air" may cause the Center Tube to come loose.
6. Follow enclosed instructions for using the Instatherm Flask. The element has a positive coefficient of resistance with temperature increase; the increasing resistance will decrease current flow and it sometimes happens that the voltage is increased to compensate. However, upon cooling, the resistance will again lower and if the controller setting is not cut back or the unit merely switched off, the current may be excessive when restarting. The Flask is intended for a maximum temperature of 200°C. It is possible to drive the temperature above this value, but at the expense of the silicone rubber insulation. It is normal for the rubber to slowly turn white and "chalk off". An R.T.V. rubber repair kit is available to recover damaged areas.
7. The Column characteristics below are based on a boil-up of 80 cc/hr.

Operating Hold-up, with Head, 1.6 mL, maximum

Static Hold-up, with Head, less than 0.3 mL

Static Hold-up of Head, 0.2 mL

Operating pressure drop, less than one mm Hg

Maximum throughput, 270 cc/hr



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