The Ace-Burlitch Inert Atmosphere System

ASSEMBLY & INSTRUCTIONS
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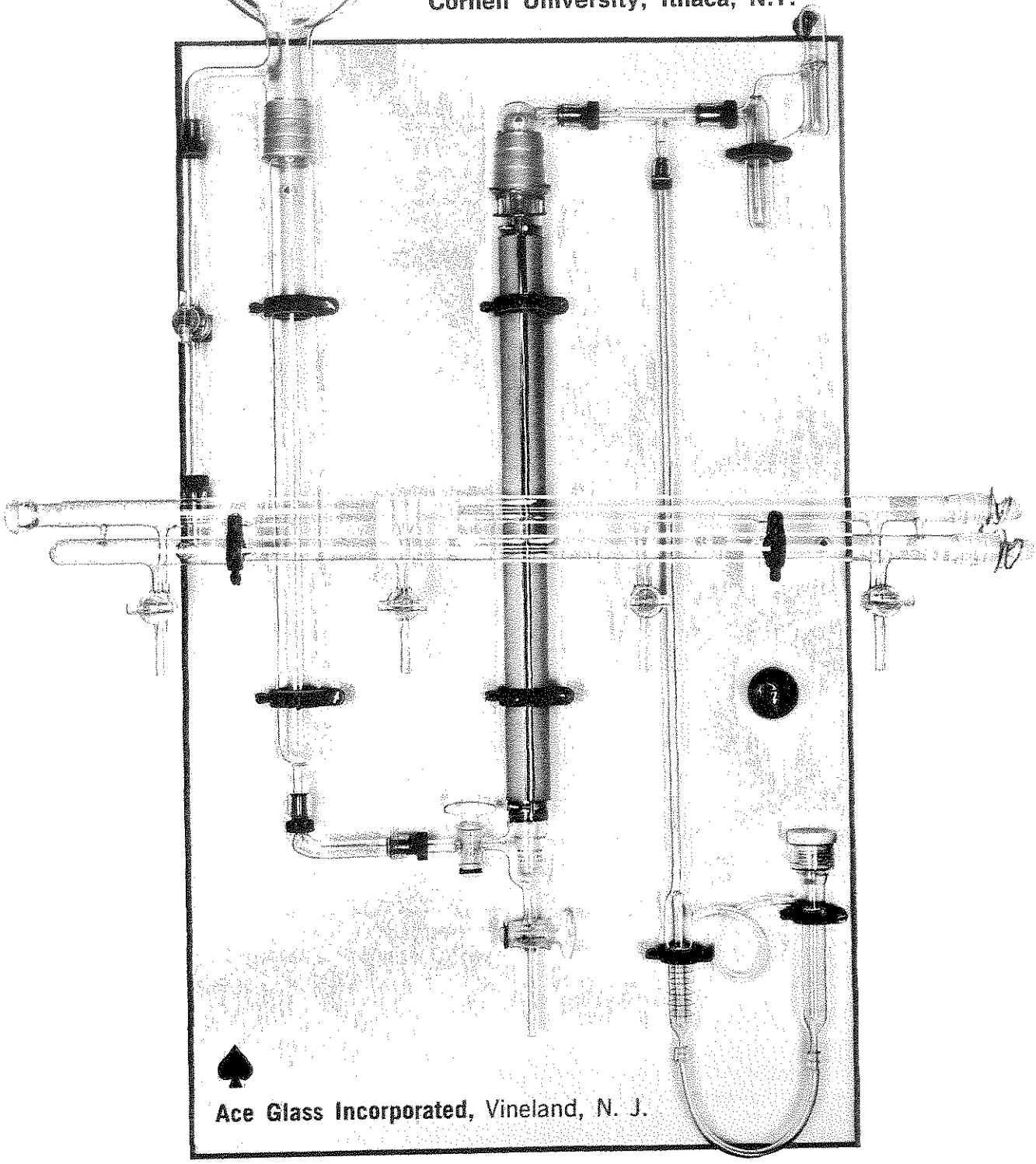

Ace Glass Incorporated, Vineland, N. J.

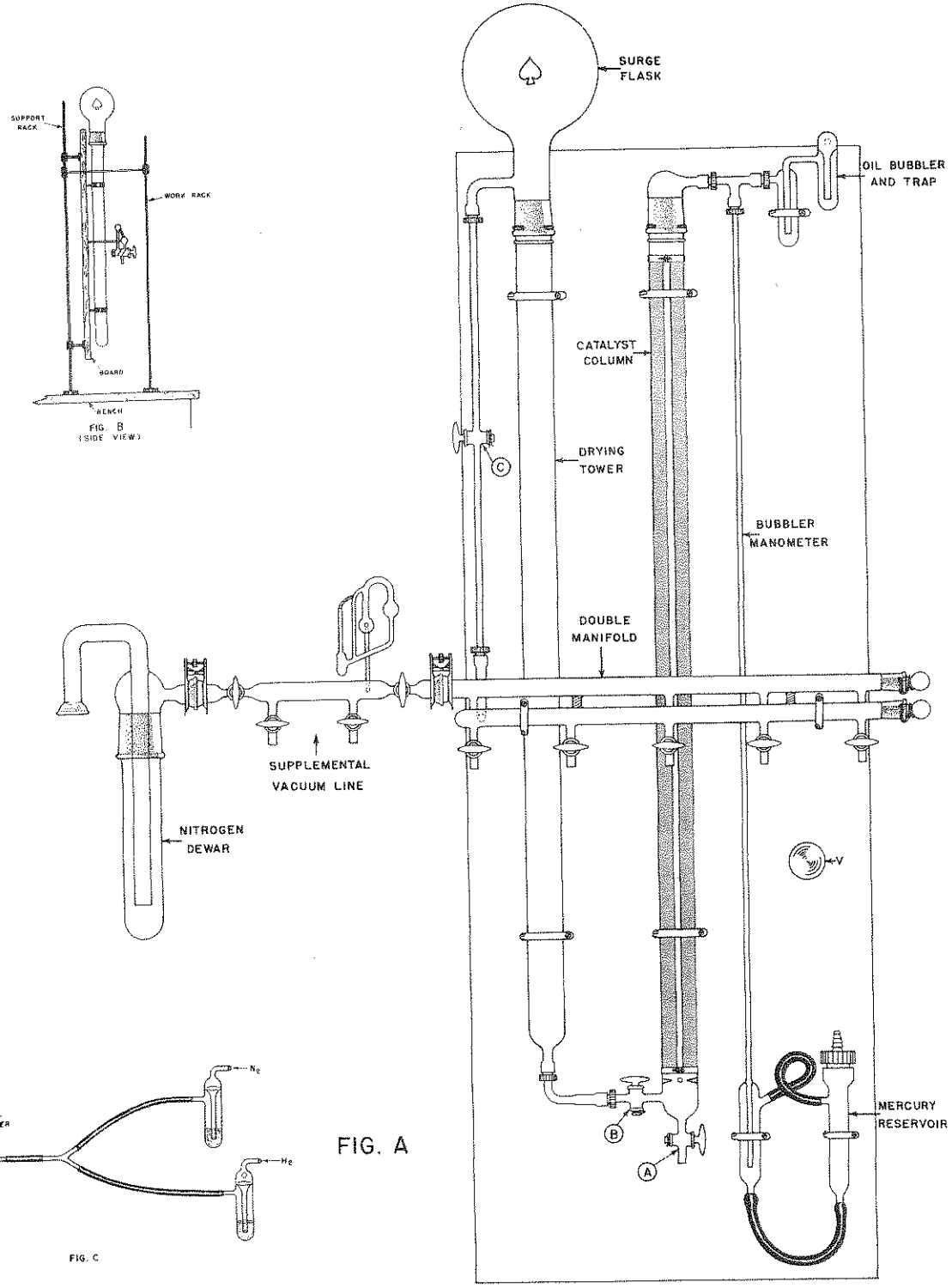

FIG. B (SIDE VIEW)

FIG. A

FIG. C

I. GENERAL COMMENTS:

The Ace-Burlitch Inert Atmosphere System is designed for maximum convenience in the handling of air and moisture sensitive compounds in conjunction with Ace No-Air Labware. The double manifold arrangement provides multiple sources of vacuum or purified inert gas from four two-way vacuum stopcocks. A very convenient arrangement results when the system is rack mounted above the benchtop with a second rack of vertical bars mounted in front (see figure B). On most benches this will leave eight to ten inches of bench space. Although this may at first appear to be a very small work area, it is usually quite adequate since most of the apparatus is clamped to the rack. This setup makes the vacuum stopcocks and inert gas flow control valve readily accessible just behind the experiment and the board provides a white background for the true observation of colors and changes.

II. ASSEMBLY AND LEAK TESTING:

1. Attach the two manifold clamps to the board with the long threaded bolts. The back of the clamp should be approximately 3⅜" from the board. Secure each clamp to the threaded bolt with a nut and secure the bolt to the board with a nut and a washer.
2. Mount the board on a solid rack at a convenient height. Typically the manifold clamps would be 30 inches above the bench top.
3. Slide the wire screen down to the indentations in the catalyst column and clamp the latter so that the arms of stopcock B (see figure A) are 11 inches from the bottom of the board and parallel to it. Lubricate stopcocks A and B with No. 8230-06 High Vacuum Silicone Stopcock Grease.
4. Loosely assemble the oil bubbler and trap, connecting tube, and catalyst column cap with the threaded fittings. **Note** that the O-rings and their seats must be free of all dust to insure a leak-free seal.
5. Install the cap on the catalyst column (lubricate with Hi-Vac Silicone grease) and adjust the threaded fittings so that the oil bubbler and trap may be clamped in place. Tighten the threaded fittings.
6. Connect the ¼" copper tube from the compression fitting on the side arm of the needle valve, V, to the threaded fitting on the oil bubbler. Use Teflon® tape to seal the threads of the compression fitting to the body of valve V. **Note** that care must be taken to see that the copper tube is not scratched in the area of the O-ring. Any scratches should be removed with very fine sandpaper followed by finishing or polishing paper.
7. Install the mercury bubbler-manometer and the mercury reservoir. Connect the bottom tubes with a 12-inch piece of heavy wall plastic tubing and the side arms with a 9 inch loop of medium wall tubing (see figure A). With the side arms at the same height, add clean mercury to the reservoir until the level in the bubbler-manometer is at the 2 cm. mark.
8. Clamp the drying tower loosely in place and install the L-shaped connector arm with the two threaded fittings. The top of the ground joint on the tower should be about even with that on the catalyst column. Tighten the clamps and the threaded fittings.
9. Mount the 3 liter pressure surge flask on the drying tower after greasing the ground joint. Install the clamps.
10. Clamp the double manifold into place (clamps fit on lower manifold) so that the threaded fitting on the manifold is directly under the fitting on the arm of the surge flask (see figure A). Slight adjustment of the lengths of the manifold clamps may be necessary to make the stopcock arms vertical.
11. Install stopcock, C, by rotating the surge flask a few degrees and inserting one arm of the stopcock to the **bottom** of the threaded fitting on the manifold (with the O-ring and nut loosely in place). Rotate the surge flask back into position and insert the upper arm of the stopcock into the threaded fitting. Tighten both fittings and lubricate stopcock C with Hi-Vac grease.
12. Install the matched vacuum stopcocks and the end plugs (these with No. 7600 clamps) in the double manifold. Connect the upper manifold to a vacuum system, i.e., supplemental vacuum line No. 7818-48 that contains a McLeod gauge which can be read to 0.001 mm. or less. In routine operation, a two-stage mechanical pump No. 14003 and a liquid nitrogen trap No. 7818-50 are employed. Sealing wax is preferred for the ball joint connections.
13. Connect two of the stopcocks on the double manifold with a short piece of heavy wall vacuum tubing then systematically check the various parts as follows:
(1) Vacuum manifold — all manifold stopcocks closed.
(2) Inert-gas manifold — two manifold stopcocks open and C closed.
(3) Drying tower and surge flask—C open and B closed.
(4) Complete system — C and B open A and V closed.

III. Catalyst Column Activation

1. After the system has been determined to be free of leaks, refill with air, lower the bubbler-manometer, disconnect the copper tube and remove the oil bubbler and the cap from the catalyst column.
2. Load BASF catalyst into the column with a wide funnel. When the column is ¾ full place a 150° thermometer in the center so that the 120° mark is just visible above the top of the Instatherm® coating and continue filling the catalyst up to this point. For the most stringent requirements the catalyst may be crushed to increase its surface area. If this is done
a) some uncrushed material should be placed at the bottom of the column (3 to 4" layer) to prevent the smaller particles from falling through the screen **and**
b) care must be taken to **not** include much of the very finely divided material as this will impair the flow.
3. Fill the oil bubbler with 1 in. of mineral oil or preferably low vapor pressure silicone oil (Dow Corning 710 or equivalent) and replace the bubbler, manometer and cap assembly. Secure with clamp.
4. Connect a cold trap of 100 ml. capacity and cooled with ice to stopcock A and run a tube from the exit side of this trap to a hood.
5. Set up two oil bubblers in a Y-arrangement (see figure C) to the inlet side of the needle valve.
6. With B closed and V and A open, start the nitrogen flow (2-3 bubbles per second) and heat the catalyst column gradually to 120° by applying 15 to 30 volts AC to the Instatherm jacket with a variable transformer. Some water will collect in the ice-cooled trap.
7. After the temperature has stabilized at 140° for **at least 30 min.**, introduce hydrogen at about 10-30 percent of the nitrogen flow (one bubble every 3-5 seconds). A temperature increase of a few degrees will be noticed as activation of the catalyst begins. An increase of 10-15° is normal; this is accompanied by a darkening of the catalyst at the top. **CAUTION** – The temperature should never exceed 230° or the efficiency of oxygen absorption will be greatly impaired.

If the initial temperature increase is too great, reduce the hydrogen flow, and vice versa. Once the temperature has stabilized the activation of the catalyst may proceed unattended. This requires several hours and is essentially complete when the color change from gray-green to jet black reaches the bottom of the heated zone.

8. When the color change is complete shut off the nitrogen and pass pure hydrogen over the catalyst for 1 hr.
9. Disconnect the cold trap, close valve V and quickly evacuate the heated column, through a tube connected to A, for 30 min. to remove residual water. **Note:** Degassing of the oil in the oil bubbler causes some temporary foaming. If this is excessive stop the evacuation briefly to permit the bubbles to subside.
10. Cool the column and connect the source of inert gas (nitrogen or argon) to the needle valve, V*. Close A and

fill the system slowly with inert gas. (***Note:** Also it is imperative that the inert gas tank be equipped with a regulator valve to avoid excessive forepressure).

*Copper or other non-porous, metal tubing should be used to connect V to the tank regulator. (***Note:** The use of rubber or particularly plastic tubing will shorten the life of the active catalyst considerably). Such copper tubing and all connections must be leak free as indicated either by evacuation or pressurization to 50 p.s.i. (valve V **closed**) and soap bubble testing. The nominal pressure from the tank will be 10 to 15 p.s.i.

IV. Preparation of the Drying Tower

1. Remove the surge flask by lowering stopcock C then push a **very loosely** packed piece of glass wool to the bottom of the column to form a layer ½ in. thick.
2. Fill the column with a phosphorous pentoxide based drying agent that is adsorbed on an inert, porous material that will permit even and rapid flow. Mallinckrodt "Aquasorb" is recommended for this purpose. WITHOUT PACKING fill to within two inches of the bottom of the 45/50 joint. It is essential that the drying agent be as loosely distributed as possible.
3. Quickly place a 2" plug of **loosely** packed glass wool on top of the drying agent, clean and regrease the joint and replace the surge flask and stopcock C.
4. Evacuate the drying tower via a stopcock on the double manifold. **At least** 24 hours is required to outgas the drying agent and to remove adsorbed water from the glass walls. The latter process may be facilitated by heating. This is conveniently accomplished by using two heating lamps on the surge flask and a hot air gun No. 2073 on the manifold. **Caution** when heating the double manifold, **both** manifolds must be heated evenly and simultaneously to minimize strain.
5. After the degassing process the stopcocks on the double manifold are closed and the system is filled with inert gas by **slowly** opening B then V.

V. Some Operational Notes

1. For nearly all applications the catalyst column is operated at room temperature. For greater efficiency it may be operated at 100-120°. As the catalyst is expended it will revert to the original gray-green color. When this band reaches the midpoint of the column, the catalyst should be regenerated.

The activated catalyst is pyrophoric in air. If it is necessary to expose it to the air it should first be "killed" slowly by passing a dilute mixture of air in the inert gas through the column until the color change to gray-green is complete. If this is done too rapidly the resulting temperature increase (over 150°) may ruin the catalyst for further use.

2. A pressure of 2 cm. of mercury (as set by the bubbler-manometer) provides adequate flow through the columns for most applications. For increased flow the pressure may be increased by raising the mercury reservoir, but the resulting internal pressure when the system is closed requires that all ground joints be secured very tightly.

The exit tube in the mercury reservoir should be led to a hood in areas of poor ventilation.

3. (a) Although almost any type of pressure-vacuum rubber tubing may be used to connect the manifold to the experiment, the heavy wall thickness of some types of tubing results in excessive weight and stiffness that makes manipulation difficult. The principal requirement is that the tubing be as resistant to gas diffusion as possible. Experience has shown that butyl rubber tubing is the best for this purpose. Because of the low permeability to gases, thinner wall sections may be used. Typically ¼ in. i.d. butyl rubber tubing with a ⅛" wall works well.* (The same size silicone rubber tubing is too permeable for many applications). ***Note:** It should be noted that phenol is often used in the manufacture of butyl rubber and residual quantities may be present. If this is detrimental to the experiment it can be largely removed by baking the tubing in vacuum at about 100° for several hours.

A coat of Kel-F or other fluorocarbon grease may be applied to the inside of the tubing to make it more resistant to corrosive vapors.

(b) When not in use the tubing should be kept closed under an inert atmosphere or vacuum to minimize the adsorption of atmospheric water vapor. A candy cane shaped piece of 10 mm. dia. glass rod serves excellently in this function; at the same time it provides a means of hanging the tube out of the way.

4. A Critical Test — In the absence of expensive oxygen and water analyzers, it may be worthwhile to apply a practical test to determine whether the system is functioning properly. One very critical test consists of exposing a very dilute solution of potassium-benzophenone ketyl in hexane to the inert atmosphere of the system. Due to the low solubility of the ketyl in hexane the very dilute, light blue solution is extremely sensitive to traces of oxygen and water.

A solution of the ketyl may be prepared by refluxing a mixture of 1 gram of potassium metal (in small pieces that are freshly cut) with 2 grams of benzophenone in 500 ml. of hexane, under the inert atmosphere, in a still that was previously flamed dry in vacuum. After development of the light blue color (several hours) the still is allowed to cool to ambient temperature while open to the inert gas system, and then closed. The blue color should remain indefinitely. If the color fades it may be due to traces of water in the still and the test should be repeated at least twice by simply refluxing the mixture until the blue color returns. Traces of phenol in butyl rubber tubing cause the test to fail repeatedly. Traces of reactive solvents such as dichloromethane also may cause the test to fail. If solvents are suspected, evacuate the entire system then slowly refill the inert gas and repeat the test.

5. The stopcocks on the double manifold should be regreased one at a time with a rapid flush of inert gas to keep atmospheric air and water out of the inert gas manifold. Then close stopcock C and evacuate this manifold and refill it slowly with inert gas by opening C.

6. In the event of an accident the double manifold may be removed for cleaning by closing stopcock C and sliding it upward until the lower arm is disengaged from the threaded fitting on the manifold. One way to clean the double manifold is to immerse it in a tall alcoholic potassium hydroxide bath, one half at a time, followed by a similar rinse in water and a dilute sulfuric acid solution. A few hours in the caustic solution is usually sufficient and will not harm the ground surfaces of the stopcocks. If a hydrocarbon-based grease was used on the stopcocks, a pre-treatment with trichloroethylene or similar grease solvent will be necessary.