

● DRY BOX CONSTRUCTION

ROWLAND E. JOHNSON

Florida State University, Tallahassee, Florida

IN CONNECTION with some recent work on phosphorous halides, we found it necessary to construct a dry box. An exhaustive search of the literature at the time gave no references; more recently, Tyree¹ has given a description of a dry box which is well suited to produce a low-moisture atmosphere. Since our box incorporates many other features which we have found desirable, such as an air lock and internal drying system, we should like to report on it here in the hope that it will save future workers some of the troubles we experienced.

Our box was constructed of $\frac{1}{8}$ -in. steel sheet by a local machine shop. It is in the general shape of Tyree's box (see Figure 1) with angle iron brackets at the ends to give rigidity. The addition of another window at the top with a fluorescent fixture outside the box was used to light the interior. Any small holes in the metalwork were first painted with Glyptal, then a good grade of enamel was used over all surfaces for a finish. The windows are $\frac{1}{4}$ -in. Plexiglas, bolted to the metal window frames with $\frac{1}{4}$ -in. bolts on 3-in. centers. A putty-like tape, #590.5 Seam Sealing Tape, from Presstite Engineering Company, insures a gas tight seal between the plastic and metal. Sponge rubber proved quite unsatisfactory as a seal against water vapor.

¹ TYREE, S. Y., *J. CHEM. EDUC.*, **31**, 603 (1954).

To move materials rapidly into and out of the box, we use an air lock which can be evacuated. It is constructed from 8-in. cast iron pipe, 18 in. long, with hinged doors at each end. Rubber O-rings are fitted into grooves on the doors and seat on flanges welded onto the pipe ends. Another larger flange welded near one end of the lock bolts onto the side of the box, seating against a rubber gasket. The completed air lock weighs about 50 lb., but there is no appreciable deformation of the side of the box due to this weight. Before evacuation, each door is held shut by a C-shaped clamp which hooks behind the end flange and tightens by a hand screw through the center of the clamp. Because of the lock volume, a moderately fast pump is required. We use a Welsh Duoseal, Model 1403, which has a free air displacement of 100 liters per minute. Our cycle of operation on the air lock is evacuate, flush, re-evacuate and fill; it takes less than 5 minutes.

To produce and keep a low moisture level in our dry box atmosphere, we circulate the gas continuously through a drying tank containing about 5 lb. of activated alumina, $\frac{1}{4}$ -8 mesh, Grade F-1, from the Aluminum Company of America. This tank is a piece of 6-in. cast iron pipe, 6 in. long, mounted vertically. One end is a removable flat plate, sealed by an O-ring when bolted into place; inlet and outlet pipes are at opposite ends of the tank. The alumina charge is held in a

snugly fitting canister with a 16-mesh screen bottom, overlaid with a triple thickness of glass fiber window curtain fabric to prevent dust from entering the system. The gas enters the tank near the top, passes down through the alumina in the canister and out the bottom. When saturated with water, the alumina is replaced by removing the end plate, taking out the canister and inserting another with a fresh charge. The alumina is rejuvenated by heating to 250°C. while still in the canister and cooling in a vacuum desiccator. When interchanging canisters, the drying tank section of the circulating system is isolated by two packless valves; the tank may then be evacuated and refilled with gas from the dry box. This prevents air from entering the box which normally contains a nitrogen atmosphere.

The gas circulating pump is a Model 2520 from the Sutorbilt Corporation and has a capacity of at least 10 cu. ft. per minute in our system. Standard 1-in. hard drawn copper pipe and fittings were used in the circulating system; flexible copper tubing (refrigeration vibration eliminator) was used to connect to the circulating pump. The pump originally had a bad leak around the bearings on the drive shaft; we replaced the bearing cover with a brass plate and holder for an O-ring. The O-ring fits snugly around the shaft and lasts for about 100 hours operating time using a petroleum jelly lubricant. A more elaborate system could be devised in which the motor and pump are sealed inside a box which is part of the circulating system and no moving seals would be necessary.

Since we ordinarily have periods of a week or more when the box is not used, we run the circulator only when needed. Twenty minutes running time decreases the humidity to a dew point of less than -65°C . The humidity slowly increases with time after halting operation; apparently diffusion of water vapor through the gloves is one of the most important modes of entry.² We use relatively thin neoprene gloves sold by Erskine-Healey, Incorporated, Rochester, New York, and, in addition, wear surgeon's gloves when working in the dry box gloves. This second barrier helps decrease the moisture in contact with the dry box gloves. A time meter keeps track of the circulator operation time. Depending on the use of the box and air lock, 5 lb. of the desiccant will last about 30 hours operating time. (If we were to rebuild the box, we would increase the capacity of the desiccant. This is the operation requiring most frequent attention. The pressure drop through the alumina is quite small; the addition of 10 or 15 lb. more alumina would not decrease the circulating pump capacity appreciably.)

The entire circulating system is diagrammed in Figure 2. Electrical switches at knee level activate the magnetic valves, one leading from a nitrogen cylinder to the box, the other from the box to the evacuating pump. The nitrogen cylinder is connected so that entering nitrogen must pass through the desiccant before entering the box. While some may consider these magnetic valves dispensable, we find nothing more aggravating than to have too high or too low pressure in the box and no easy way to regulate it. The outlet tube from the dry box proper develops quite a low pressure due to the action of the circulating pump; we have used this suc-

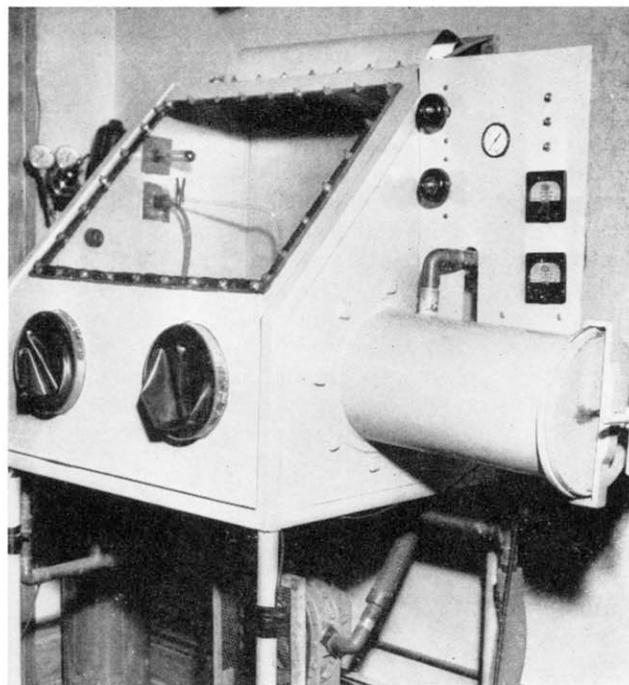


Figure 1. Dry Box Construction

tion to operate a vacuum-type sweeper inside the dry box.

When completed, the box was filled with Freon-12 by first displacing the internal volume with a rubber weather balloon and then admitting Freon to the box. A slight internal positive pressure of Freon in the box allowed us to detect leaks. The presence of Freon outside the box was detected in the following manner: A small probe, connected to the intake side of an auxiliary blower was run over the outside of the box and all fittings. The air from the blower outlet (which would contain Freon if there were a leak near the probe) was mixed with gas, and burned. The burner flame heated a copper tube; Freon showed its presence by an intensely colored flame. When no further leaks could be found, the internal volume was again displaced by the balloon and the box then filled with nitrogen.

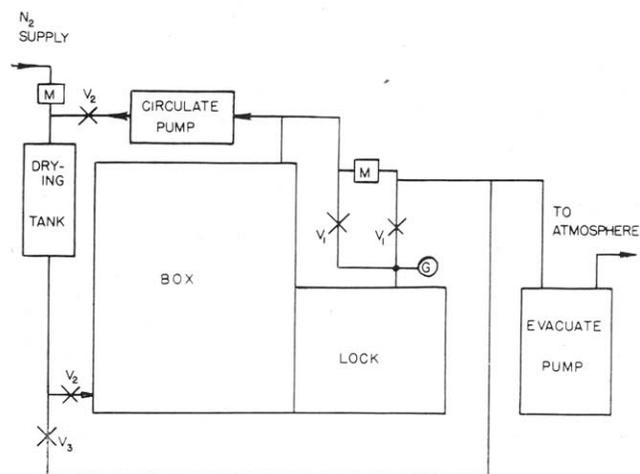


Figure 2. Circulation System

M, M, magnetic valves. V_1, V_1 , airlock control valves. V_2, V_2 , valves to isolate the drying tank (see text); these are normally open. V_3 , valve to evacuate drying tank; normally closed. G, vacuum gauge for airlock.

² ROWAN, J. H., Report Y-1075, Y-12 Plant, Oak Ridge, Tennessee, 1954.

Although more than a year of use has shown the box to give good service, some improvements might be made. As mentioned above, the desiccant capacity is too small. Also no simple direct reading humidimeter has been developed for the range of dew points from -30°C . to about -75°C . We use a silvered surface dew point apparatus which is tedious and quite subjective. We have tried measuring the electrical conductivity of a variety of materials such as filter paper and pottery saturated with salt solutions, solid salts themselves, glue and gelatin coatings containing hygroscopic materials and others. Nothing we have tested gives reproducible results; usually the electrical conductance in this range of humidities is too low to measure easily. The alumina gives essentially the same low dew point as long as it has not become satura-

ted. Once saturated, the dew point rises rapidly so we would be satisfied with an instrument which would indicate whether the dew point is less than or greater than -50°C . Perhaps an instrument similar to that of Minter³ which works on the difference of thermal conductivity of wet and dry air could be adapted to our purpose.

So many people collaborated with good ideas on this dry box that it is impossible to thank them all. However, we should especially like to acknowledge the assistance and ideas contributed by Al Florin of the Los Alamos Scientific Laboratory. This work was performed under contract AT-(40-1)-1317 with the Atomic Energy Commission.

³ MINTER, C. C., NRL Report 4437, Naval Research Laboratory, Washington, D. C., 1954.

