DRY BOX TECHNIQUE

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STUDIES of the chemical and physical properties of easily hydrolyzed materials have always been hampered by the experimental difficulties of preparing and transferring samples in the absence of water. In the case of such substances as zirconium and hafnium tetrachlorides, even momentary exposure to air results in appreciable changes in composition. The most precise work on such material has always involved indirect sampling techniques.[1, 2, 3] Considerable time and effort went into the design and construction of a "dry box" by means of which sampling of easily hydrolyzed materials could be made to approach the "ordinary" techniques. The box in use was made to our specifications by the Pickard Roofing Co. of Durham, North Carolina, at a cost to us of $616.00. Design details are the work of Mr. A. R. Bennett, of the departmental staff.

Scale details of the box are shown in Figure 1. One-sixteenth-in. stainless steel was used. All joints are welded and soldered over to insure airtightness. The bottom and back are braced by riveting to angle iron (not shown in drawing), and covering the rivets with solder. The bracing is necessary to prevent buckling of such large surfaces of steel. A double fluorescent tube bracket (containing additional 110 a.-c. outlet) is mounted on the inside of the top. The small chamber on the right end of the dry box is a predrying chamber.

[1] HONIGSCHMID, O., ET AL., *Z. anorg. Chem.*, **139**, 293 (1924).
[2] HONIGSCHMID, O., ET AL., *Ber.*, **58**, 453 (1925).
[3] VENABLE, F. P., AND J. M. BELL, *J. Am. Chem. Soc.*, **39**, 1598 (1917).

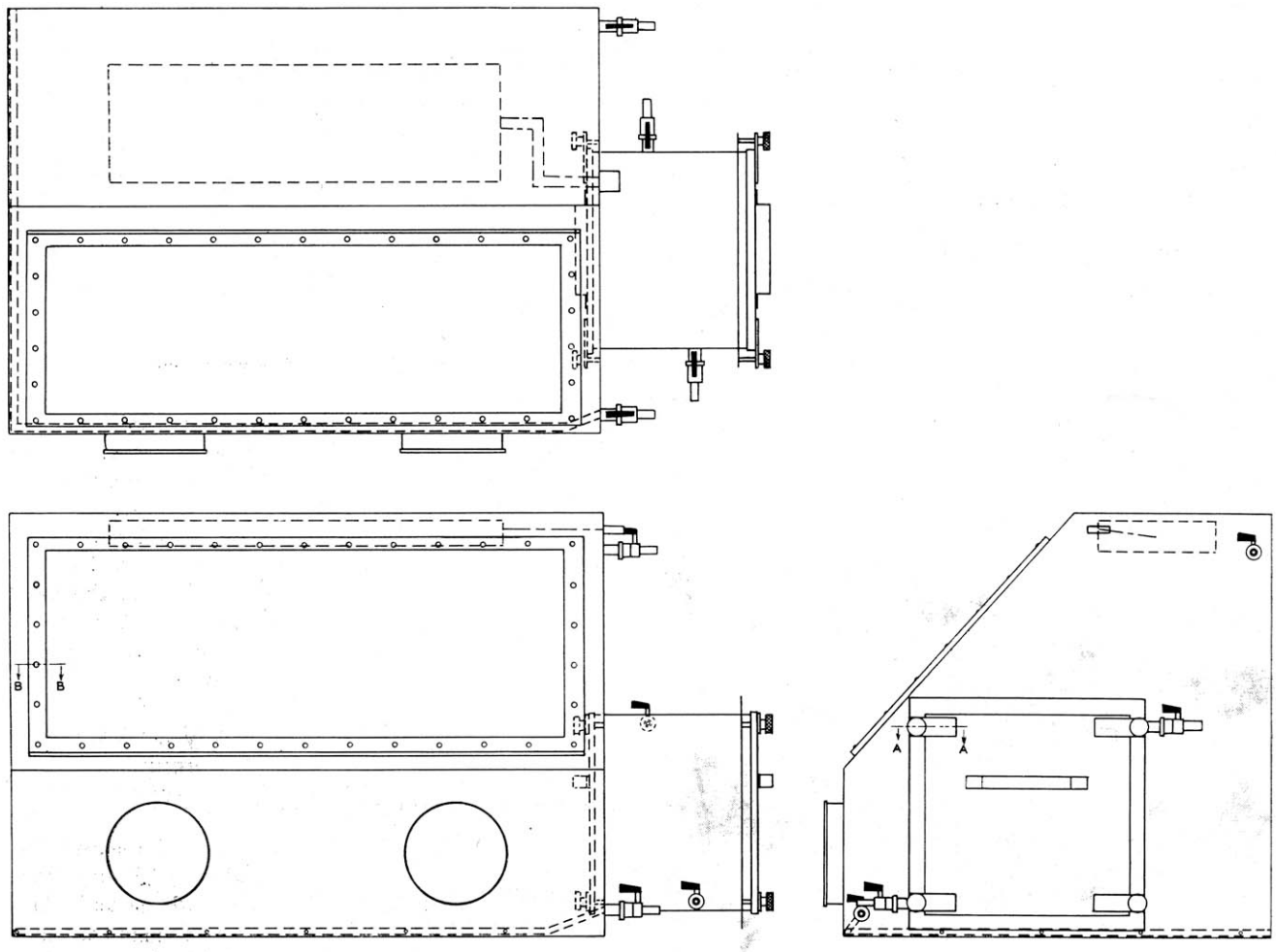

Figure 1. Dry Box

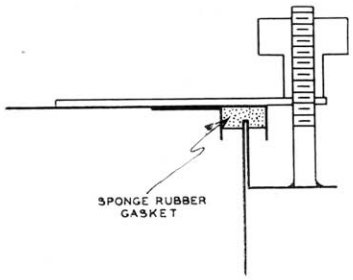

Figure 2. Detail of Predrying Chamber Seal

Provision is made for sweeping both chambers independently with some dry, indifferent gas. The outer and inner doors of the predrying chamber are interchangeable and the seal detail is shown in Figure 2. The interior of the box is viewed through a large safety-glass window, sealed as shown in Figure 3. Figures 2 and 3 are sectioned from Figure 1 as indicated. This window is removed only for major operations, whereas the predrying chamber doors are opened whenever materials are removed from or introduced into the main dry box. Special Neoprene dry-box gloves (obtainable from Safety Equipment Dist. Co., Knoxville, Tenn.), mounted on the ports by means of stainless steel slip

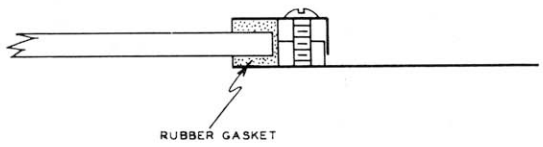

Figure 3. Detail of Window Seal

rings over rubber gaskets, are used to manipulate within the dry box. Figure 4 shows the dry box in operation. Nitrogen, dried by bubbling through concentrated sulfuric acid and then passing through two phosphorus pentoxide towers, is passed through the main chamber at all times. If the air in the box is to be dried by flushing in this matter, at least a week is necessary to establish equilibrium. Dishes of phosphorus pentoxide are kept in the box also. The nitrogen enters the box through perforated stainless-steel tubes along the bottom of the box and leaves via the valve in the upper corner. It is then passed through a dew-point meter, by means of which the humidity in the box is measured. After passing through the dew-point meter the gas is exhausted to the atmosphere through a tower of calcium chloride. It is desirable to maintain a dew point of about $-50°$ in the box. Under such conditions, samples of zirconium tetrachloride may be weighed on open weighing pans on the analytical balance in the box without detectable hydrolysis. It is of interest to note that

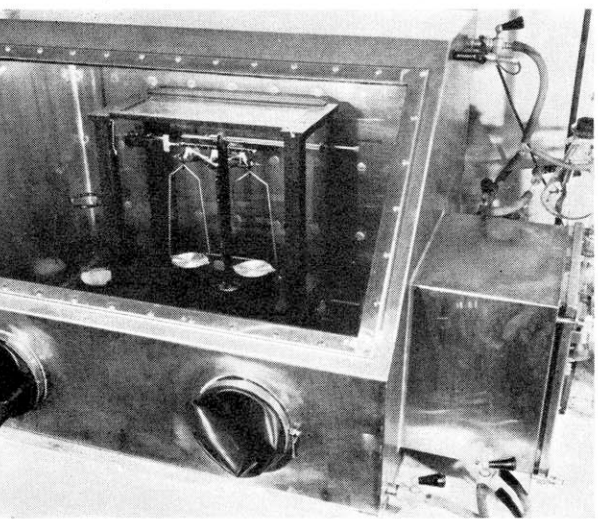

Figure 4. Dry Box Ready for Use

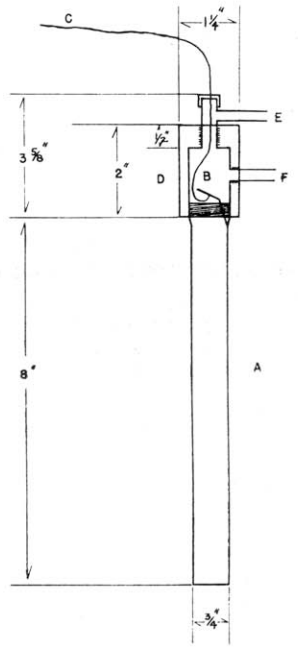

Figure 5. Diagram of Dew-point Meter

electrostatic charges persist on glassware and other insulators in this dry atmosphere and cause serious trouble in weighing accurately. Pyrex glassware is much worse in this respect than Kimble ware. The effect has been observed to render the balance unstable at times. The charge may be dissipated by means of a Reco Neutra-Stat, obtainable from Phipps and Bird, with the head containing the activity source located right over the balance pan. In addition the dry box and working parts of the balance should be grounded. The box is large enough to permit many operations. For example, a Fischer melting point apparatus may be introduced into the dry box for the purpose of determining melting points in an inert atmosphere. Also pressure filtrations can be carried out in the box.

In a typical operation the material to be sampled is placed in the predrying chamber, along with any other apparatus for sampling the material. The outside door is closed and the chamber is permitted to come to equilibrium. This is always hastened by including a dish of fresh phosphorus pentoxide. This equilibration may

take as much as two hours. The inner door is opened, working through the gloves, and the materials moved into the box. The inner door is then closed. Once safely in the dry box the material is transferred to ordinary weighing bottles, from which containers it may be sampled, using the analytical balance. Samples are removed from the box by opening the inner door, moving materials into the predrying chamber, closing the inner door and opening the outer door.

The dew-point meter is shown in Figure 5. The long copper tube A is immersed in a dry ice acetone Dewar and a polished copper surface B is silver-soldered to A. The temperature is recorded by a thermocouple C spotted under the center of the surface. The top D is of Lucite, threaded to fit the large copper tube and the two smaller copper tubes E and F. The exit gas from the dry box enters at F and leaves at E. The temperature at which dew is first observed on the polished copper surface is read as the dew point.

ACKNOWLEDGMENT

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