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PROCEDURE FOR GENERATION OF BORON TRIFLUORIDE GAS

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Some of the materials used in the following operations are hazardous. Vendors should consult their local safety organizations for detailed safety instructions concerning the materials involved.

- A. Boron trifluoride (BF_3) - Avoid inhalation or ingestion of, or contact with this gas. This material is poisonous and decomposes upon contact with water vapor or organic substances into a dense white vapor containing hydrogen fluoride. All work with BF_3 should be performed in a well-ventilated area, preferably under a chemical hood. Care should be exercised to prevent any of the BF_3 from escaping into the air.
- B. Liquid nitrogen - Avoid contact with this material and with any object which has been in contact with it. Since the temperature of liquid nitrogen is -196°C , severe burns of the skin may result from contact with it. In addition, immersion of any object at room temperature in liquid nitrogen results in violent boiling and splashing, so extreme caution should be exercised when using this material for cooling purposes.

The purpose of this specification is to describe a procedure for preparation of pure BF_3 gas, suitable for use in BF_3 proportional neutron counters. Any deviation from this procedure may result in BF_3 which contains sufficient impurities to be useless as a filling gas for neutron counters. Impurity content as great as 0.2% may affect the operation of these instruments.

A. In preparation and handling of BF_3 gas an all-metal vacuum system is required since BF_3 may react with glass and with stopcock greases, present in a glass system, to form undesirable impurities in the gas. Copper and/or stainless steel tubing, standard copper or brass fittings and Hoke diaphragm vacuum valves are recommended. All joints should be silver soldered except at those points where it is desirable to have demountable sections, e.g. the BF_3 generator. At these points standard flare fittings may be used. All copper fittings should be annealed after flaring to obtain a better seal. A suitable system for generation and handling of the BF_3 is shown schematically in Figure 1. The BF_3 generator is a small stainless steel cylinder about two inches in diameter by one inch long. "TC" indicates a thermocouple vacuum

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gauge having a range from 1-1000 microns. Flare fittings may be soldered to the thermocouple gauges so that they may be replaced easily when they fail. The storage vessels are of approximately 500 cm³ capacity and the freeze out traps are half inch stainless steel tubes 12 inches long closed at the bottom end. These freeze out traps are useful in the purifying process as well as in transferring the BF₃ from one portion of the system to another. The pumps are two-stage fore pumps connected to the system through stainless steel U-tubes which are used as liquid nitrogen cold traps to isolate the system from the pumps. A center filled cold trap may be used in lieu of the U-tube.

- B. The BF₃ gas is generated from the thermal decomposition of CaF₂ · BF₃ (calcium fluoborate) complex. This material, enriched in the boron 10 isotope is obtained from the Oak Ridge National Laboratory, Isotopes Division, Oak Ridge, Tennessee, and must be requested on form AEC-100, "Stable Isotope Request". The amount of complex required may be determined from the fact that one liter of gas at standard pressure and temperature may be generated from approximately 6.5 gms of complex.

IV. PROCEDURE

- A. Before proceeding with the generation of the BF₃ gas, the vacuum system shall be free of leaks and chemically clean. In particular, all traces of soldering flux and organic substances shall be removed.
- B. Weigh out the required amount of BF₃ complex, allowing 20% extra for losses or incomplete generation. To compute the necessary weight (W) of complex to fill a total volume (V) the following formula may be used:

$$W = \frac{V}{1000} \times \frac{p}{76} \times 6.5 = 8.55 \times 10^{-5} p V \text{ grams}$$

where V is in cm³, and p is the filling pressure in centimeters of mercury. For example, to fill 14 counters each of 70 cm³ volume to 60 cm of mercury pressure would require $14 \times 70 \times 60 \times 8.55 \times 10^{-5} = 5$ gms of complex. Thus 6 gms of complex should be used. In addition, it is important to add, for the initial filling, the volume of the vacuum system to that of the counters being filled.

- C. Transfer the complex to the stainless steel generator. During the weighing and transfer, the complex shall not be allowed to come into contact with moisture and it shall be exposed to air as little as possible.
- D. Attach the generator to the vacuum system and open the valves to the pump. The valve connecting to the rest of the system should be closed at this time.
- E. Surround the generator with a small furnace whose temperature is adjustable, and set the temperature to 120°C ± 5°C. The generator and complex shall be held at this temperature for at least 16 hours while the pump is operating. During the last four hours the trap leading to the pump shall be cooled with liquid nitrogen. Before proceeding to the next step the pressure should be less than five microns as read by the thermocouple vacuum gauge. Pumping shall be continued until this pressure is attained. At intervals of approximately two hours, the tubulation of the vacuum system shall be flame-baked to approximately 150°C to enhance outgassing of the metal surfaces.

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- F. Increase the temperature of the furnace to 150°C and continue pumping for approximately two hours, the level of liquid nitrogen shall be maintained around the pump cold trap during this period.
- G. Allow the temperature of the furnace to increase gradually to 300°C. When the temperature reaches 180°C, or when the pressure, as indicated by the thermocouple gauge, starts to increase rapidly, the valve to the forepump shall be closed. The freeze out trap should be surrounded with liquid nitrogen to condense the BF_3 as it is evolved.
- H. Allow the temperature to remain between 300°C and 350°C for 30 minutes, or until the thermocouple gauge reaches a steady reading. Shut the valve to the generator and turn off the furnace.
- I. At this point, all of the evolved BF_3 should be condensed in the freeze out trap and the thermocouple gauge should indicate a pressure of no more than a few hundred microns. If the pressure is greater than this, the trap may be clogged with BF_3 condensed near the top. To remedy this condition, remove the liquid nitrogen and allow the trap to come to room temperature. Replace the liquid nitrogen so that it contacts only the bottom of the freeze out trap. When violent bubbling stops, gradually raise the level until the entire trap is cooled.
- J. When all the BF_3 is condensed in the freeze out trap, open the valve to the pump, making sure the pump cold trap is surrounded with liquid nitrogen. Continue pumping until the pressure has been reduced to less than ten microns.
- K. Shut the pump valve, remove the liquid nitrogen from the freeze out trap and allow the trap to warm to room temperature.
- L. Again condense the BF_3 in the freeze out trap by replacing the liquid nitrogen.
- M. Repeat steps, J, K and L at least six times, or until pumping effects no further reduction in the pressure observed while the BF_3 is condensed.
- N. The BF_3 may be stored in one of the storage vessels until ready for use. The storage vessel and all parts of the system which the BF_3 contacts shall have been previously cleaned by repeated flaming to 150°C while being evacuated by means of the pump.

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