A Gas Converter for Preparing Nitrogen Samples for Mass Spectrometric Analysis

The preparation of nitrogen gas for mass spectrometric analysis usually involves oxidation of ammonium nitrogen by alkaline hypobromite in an evacuated system. The apparatus proposed by Rittenberg has been commonly used. Essentially this involves the use of a rotary pump as well as a Toepler pump for evacuation and a mercury manometer to indicate when a suitable pressure has been reached. This method was used by the senior author and found to be very time consuming and resulted in samples with considerable air contamination owing to the low sensitivity of the mercury manometer and possibly to the presence of air in the mercury. This note describes a gas converter which is essentially a refinement of the original Rittenberg apparatus and facilitates the preparation of replicate samples in a relatively short time with very little air contamination.

The sample containing N in ammonium form is boiled vigorously to expel dissolved air, concentrated to about 3 ml and transferred with an eye dropper while still hot to the reaction flask B (Fig. 1). The alkaline hypobromite is placed in flask A which has a protruding lip designed to prevent the hypobromite from coming into contact with the vacuum grease. The entire system is evacuated with a good rotary pump.

As the pressure in the system decreases the solution in B boils and then freezes and the hypobromite in A degases. Evacuation is allowed to proceed until a steady reading is indicated on the thermocouple gauge G with stopcock E shut off (all stopcocks should be high vacuum). At this time stopcock D is shut off thereby disconnecting the pump from the left side of the system. Evacuation of the right side is continued and flask A is rotated carefully 180° to introduce the hypobromite into the reaction flask. This flask is then warmed gently with a micro-burner to speed up the reaction, which is allowed to proceed until the visible evolution of bubbles has ceased. A final check of the pressure in the right side of the system is made with stopcock E shut off. Stopcock D is then opened and the nitrogen gas allowed to fill the system. After approximately 0.5 min. stopcocks D and H are closed and break-seal tube I is sealed off above H in a flame, appropriately labelled and stored until analysed. Before attaching this tube to the spectrometer a metal pellet is carefully inserted into the sleeve and allowed to rest on the tip. Following evacuation the tip is broken by raising the pellet with a magnet and then...
allowing it to drop. Since the largest volume is to the left of stopcock D, most of the gas sample remains in this part of the system. By putting other break-seals in place and evacuating as before, replicate gas samples can be collected.

The ethanol-dry ice trap C is designed to remove any water vapour present. After attachment of the break-seal tube to the spectrometer, CO$_2$ and other impurities are removed by immersing the sealed portion of the tube in liquid air. The thermo-couple F, powered by a dry cell, is incorporated into the voltmeter-galvanometer complex G and used as the vacuum gauge. Calibration indicated that pressures approaching $10^{-4}$ mm Hg can be obtained with this apparatus. Air contamination could be further reduced by using a diffusion pump in addition to the rotary pump and by eliminating some of the stopcocks and ground joints.

Prior to introduction of another sample of ammonium nitrogen to the reaction flask, air is allowed to fill the entire system in order to minimize memory effects. Following this, items A, B and C are disconnected, washed with hot water and rinsed with distilled water.

The time required for preparing a sample for spectrometric analysis was approximately 20 minutes. The range of air contamination was 0–0.4%.

**Summary**

Apparatus and a procedure are described for preparing nitrogen samples for spectrometric analysis. The apparatus is a modification of Rittenberg's with major changes being a break-seal for collecting the sample and a volt-