

Evaluation of Johnson Matthey Inert Gas Purifier for the removal of Carbon Dioxide, Oxygen and Moisture from Nitrogen Using Atmospheric Pressure Ionization Mass Spectrometry

Alan R. Bandy¹
Chemistry Department
Drexel University
Philadelphia, PA 19104

Test Instrumentation

This experiment tested the Johnson Matthey purifier Model IG-500XL (S/N 20111002-51924) for the removal of carbon dioxide, oxygen and water vapor in a matrix of nitrogen. The tests were performed at room temperature that was approximately 20°C.

The manifold used is shown in Figure 1. It was constructed using ultra smooth stainless steel tubing and VCR fittings. Liquid nitrogen boil-off was used as the matrix gas and had a moisture level of about 200 ppbv and carbon dioxide and oxygen levels of about 300 ppbv. Moisture, oxygen and carbon dioxide were reduced to levels of 0.5, 0.04 and 0.06 ppbv respectively using a Johnson Matthey prepurifier (Figure 1). Flow rates were measured with mass flow controllers calibrated with a flow calibrator (Model DC-2M, BIOS International Corp. (traceable to NIST). Moisture, oxygen and carbon dioxide were monitored with a Model ATTO SPEC 1 Extrel atmospheric pressure ionization mass spectrometer (APIMS) using a corona discharge ionization source and operated in positive ion mode. Oxygen and carbon dioxide test atmospheres were prepared by diluting a cylinder of nitrogen containing 100 ppmv of oxygen and carbon dioxide. Test atmospheres containing moisture were prepared by diluting the output of a permeation tube having a permeation rate of 3740 nL min⁻¹ (VICI Metronics Inc.). The permeation rate of this tube was obtained by monitoring the mass of the tube using a Model AP290D OHAUS Inc. electronic balance having an accuracy of $\pm 10.5^{-5}$ grams.

The manifold was first checked for leaks by pressurizing it with He to 60 psi through a 20mL/min mass flow meter. The maximum leak rate was ± 0.03 mL/min. A more sensitive leak check was performed by evacuating the manifold and then spraying each joint with Freon. Freon leaking into the system was monitored at 69 amu by the mass spectrometer operated in electron impact mode.

The manifold was dried initially by purging it with nitrogen for 4 days. A flow rate of about 3L/min was maintained in the manifold. Manifold pressure was maintained at 10 psi and the manifold temperature was maintained at 30°C. The amount of H₂O vapor or calibration gas added to the manifold was controlled by regulating the fraction of the analyte introduced into the manifold by varying the flow rate through a flow controller attached to a pump. This approach eliminated dead volume in the manifold when no calibration analyte was being added. The sensitivity of the APIMS was determined by adding a known amount of analyte downstream of the purifiers.

¹ President, MicroAnalytics, Inc.

Results

The purifier was challenged first with 340 pbv of carbon dioxide and oxygen. The purifier was tested for two hours at flow rates of 6, 10, and 13 L min⁻¹. The results are shown in Figures 2-7.

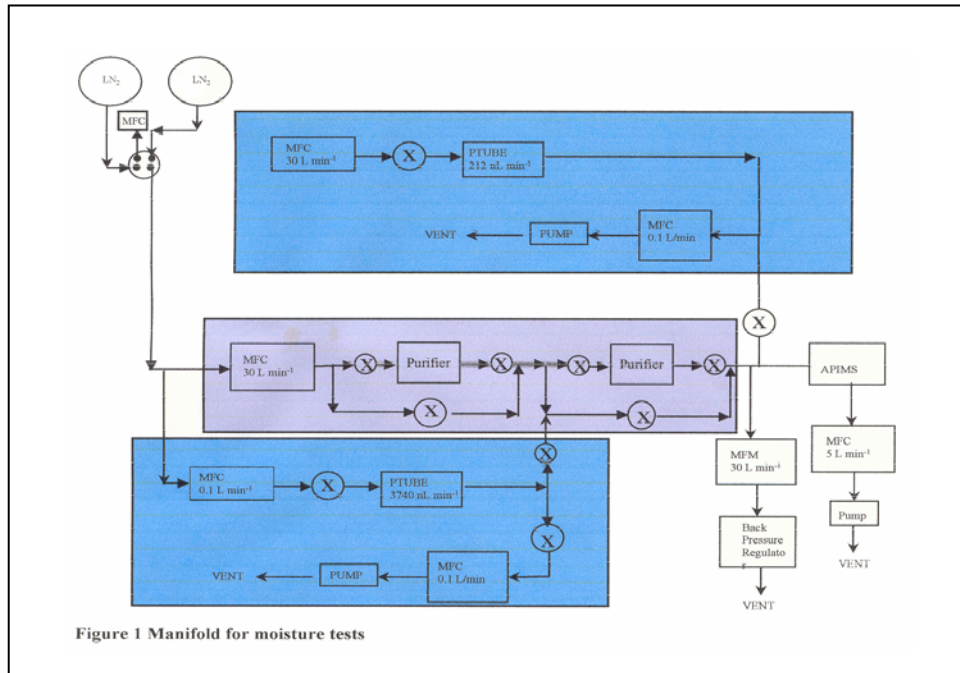
Inspection of Figures 2-7 reveals that the purifiers removed carbon dioxide and oxygen to levels well below 0.1 ppbv for each of the flow rates during the test periods.

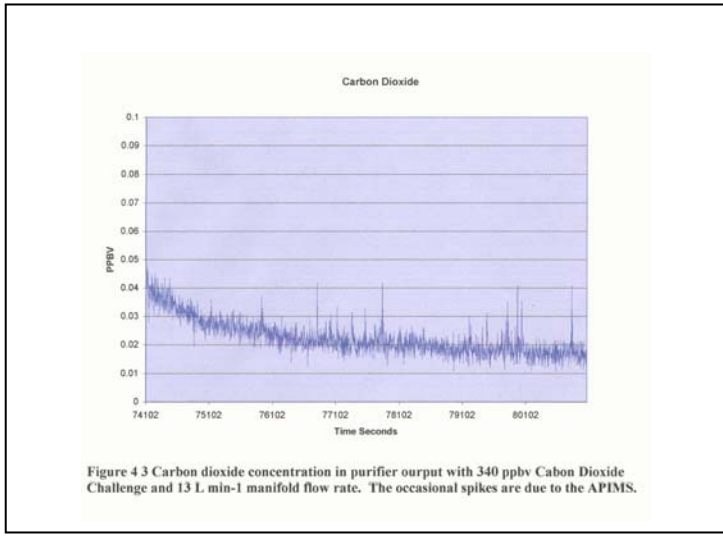
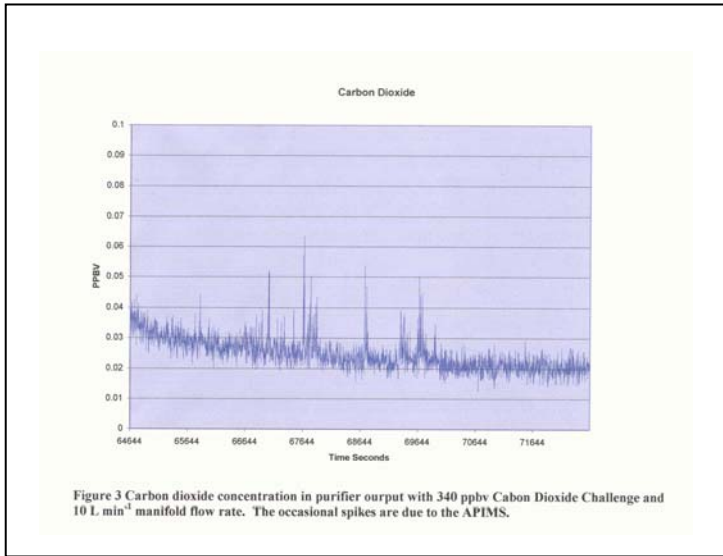
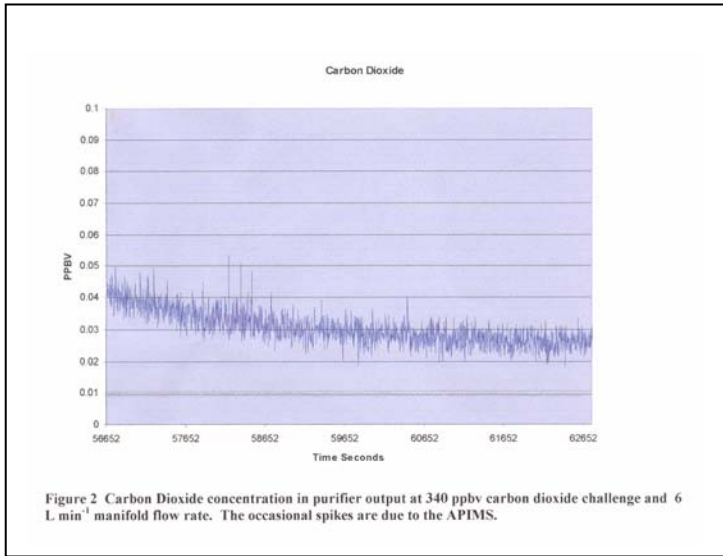
The purifier then was challenged with 320 pbv of moisture. These data for manifold flow rates of 3, and 10 and L min⁻¹ are shown in Figures 8 and 9.

Inspection of Figures 8 and 9 reveals that the moisture in the purifier output during challenge at each of the flow rates was always below 0.4 ppbv.

Conclusions

The Johnson Matthey purifier Model IG-500XL (S/N 20111002-51924) removed oxygen and carbon dioxide to levels below 0.2 ppb for two hours at flow rates of 3, 6, 10 and 12 L min⁻¹. The purifier removed moisture to below 0.4 ppbv for two hours at flow rates of 6 and 10 L min⁻¹.





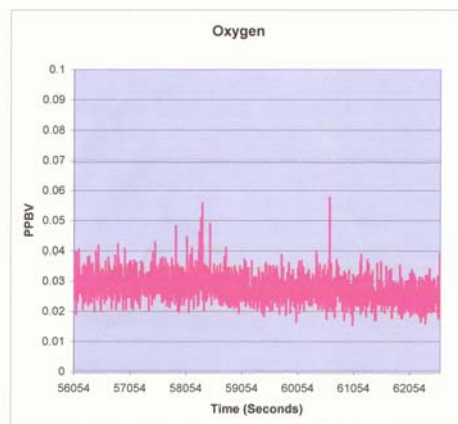


Figure 5 Oxygen concentration in purifier output at 340 ppbv oxygen challenge and 6 L min-1 manifold flow rate. The occasional spikes are due to the APIMS.

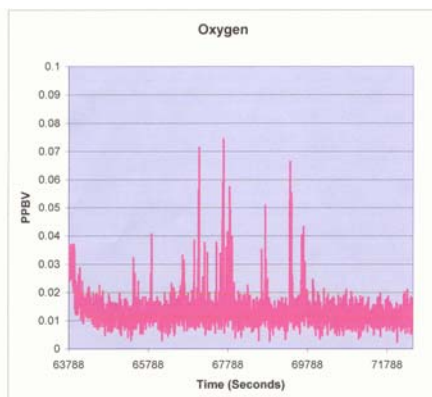


Figure 6 Oxygen concentration in purifier output at 340 ppbv oxygen challenge and 10 L min-1 manifold flow rate. The occasional spikes are due to the APIMS.

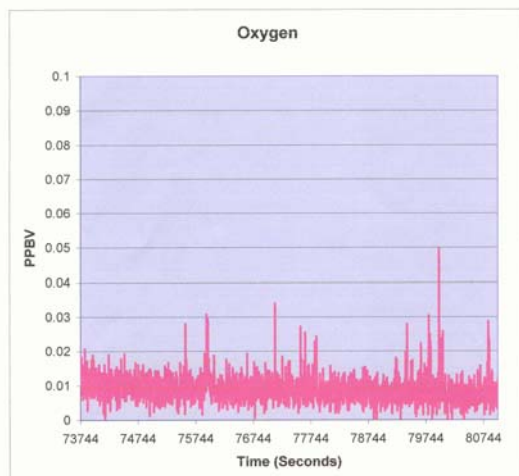


Figure 7 Oxygen concentration in purifier output at 340 ppbv oxygen challenge and 13 L min-1 manifold flow rate. The occasional spikes are due to the APIMS.

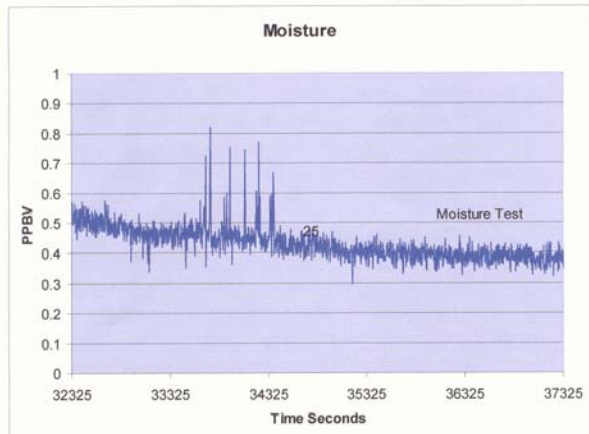


Figure 8 Moisture in purifier output at 340 ppbv moisture challenge and 3 L min⁻¹ manifold flow rate. The occasional spikes are due to the APIMS.