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# Evaluation of Johnson Matthey Argon Purifier Using Atmospheric Pressure Ionization Mass Spectrometry

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## Abstract

The efficiency of the Johnson Matthey PureGuard Model IG-1000 purifier was evaluated for the removal of methane, oxygen, carbon monoxide, carbon dioxide and moisture using atmospheric pressure ionization mass spectrometry (APIMS). When operated at 25°C and 400°C and challenged with 420 parts per billion (ppbv) of carbon monoxide, carbon dioxide and 400 ppbv moisture, the purifier effluent contained less than 1 part per billion by volume of these species. When operated at 400°C and challenged with 420 ppbv of methane the purifier effluent contained less than 1 ppbv methane.

## Introduction

This experiment tested the transmission characteristics of a Johnson Matthey purifier for methane, carbon monoxide, carbon dioxide, oxygen and water vapor in a matrix of argon. The tests were performed with the purifier at 25°C and at 450°C.

## Experimental

Manifold systems for these experiments are shown in Figures 1 and 2 for water vapor and the other gases respectively. The manifold was constructed from 0.25-inch o.d. ultra smooth stainless steel tubing and VCR fittings manufactured by CAJON<sup>®</sup> Co. Liquid argon boil-off having a moisture level of about 100 ppbv was used as a source of dry argon. The argon was further dried to approximately 3.1 ppbv using another Johnson Matthey purifier. Flow rates were measured with either mass flow controllers or mass flow meters calibrated with a Model DC-2M flow calibrator manufactured by BIOS International Corp. (traceable to NIST). The moisture level was monitored with a Model ATTO SPEC 1 Extrel atmospheric pressure ionization mass spectrometer (APIMS) using a corona discharge ionization source. Two water permeation tubes were purchased from VICI Metronics Inc. The masses of these tubes were monitored as a function of time with a Model AP290D OHAUS Inc. electronic balance having an accuracy of  $\pm 10^{-5}$  grams. The permeation tubes were maintained at 41.0°C and had permeation rates of 215 nL/min 245 nL/min respectively.

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  $\pm 0.03$  mL/min. A more sensitive leak check of the section after the purifiers was performed by evacuating

this portion 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 by purging it with dry argon for 2 days. A flow rate of about 2L/min was maintained in the manifold. The manifold pressure was maintained at about 5 psi and the manifold temperature was maintained at 30°C.

The amount of H<sub>2</sub>O 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. From studies of analyte added downstream of the purifiers the sensitivity of the APIMS was determined. Although the sensitivity varied as much as 20% from species to species and from one moisture concentration to another the mean sensitivity for all species was 10<sup>4</sup> counts per second ppbv<sup>-1</sup>. The root mean noise of the APIMS was 10<sup>2</sup> counts per second ppbv<sup>-1</sup>, therefore, the lower limit of detection for all species was determined by blank fluctuations.

## Results

With a 400 ppbv challenge no detectable moisture passed through the purifier. We could easily detect an increase of 0.5 ppbv but the amount of moisture transmitted was probably substantially below that value. This also was true when the purifier was operated at room temperature and at 450°C.

For a 420 ppbv challenge of oxygen we observed slightly less than 1 ppbv passing through the purifier when it was cold and no measurable amount when the purifier was warm. When faced with the same challenge of carbon dioxide or carbon monoxide no observable amount of these species penetrated the purifier when the purifier was either hot or cold. The lower limit of detection for these species was less than 0.5 ppbv.

At the same challenge level with methane after about one hour with the purifier heated to 400°C, the methane signal was nearly zero.

