

HIGH PRESSURE POU PURIFICATION OF CORROSIVE GASES

Effect On Gas Distribution Components

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Biography

Dr. Robert Torres is a principal research scientist at Matheson Gas Products in the area of microcontamination and corrosion. Dr. Torres is involved in investigations involving live gas testing of components in gas distribution systems. Additional research includes development and testing of new materials to be used in corrosive environments. Other endeavors include development of new analytical instruments and sampling methods capable of detecting ultra-low level of impurities in corrosive matrices.

A member of the American Chemical Society, Electrochemical Society, and National Association of Corrosion Engineers, Dr. Torres holds degrees in chemistry from the University of Wyoming (B.S.), and University of Colorado (Ph.D.), and assumed a post-doctoral position at the National Renewable Energy Laboratory. General research interests include electrochemistry, surface analysis, and interfacial phenomena.

Abstract

Purification of high pressure corrosive gases has traditionally utilized a pressure regulator prior to introduction into the purifier. However, reported regulator failures have been shown to be a major disadvantage using this conventional configuration. Such failures are a great concern owing to the damage they may cause to downstream components such as pressure transducers, mass flow controllers, and on-line monitors. A high pressure source gas purifier installed before the regulator offers a solution to this common problem.

Recently, a new purifier for corrosive gases has been developed by Matheson R&D personnel, that can be used up to full cylinder pressure for source gas purification. This new purifier can also be used at lower pressure for point of use (POU) purification at the wafer tool. The new purifier reduces moisture concentrations in high or low pressure corrosive gases to ppb levels. Being a second generation of Matheson's corrosive gas purifiers, this new purifier has remarkably higher moisture capacity than the first generation products. The new purifier was shown to have a capacity of 17 liters

of moisture per one liter of purifier (L/L) in 100% N₂, 20 L/L in >99% HCl and 23 L/L in >99% HBr. Such extended capacities ensure long uninterrupted source gas or POU operation. Additionally, all metal emissions from this purifier are below ICP-MS detection limits.

The effect of the high pressure purifier was investigated under severely corrosive gas conditions. HBr was doped with moisture at concentrations from 1 to 5,500 and sent through the experimental gas manifold at pressures of 320 psig (HBr cylinder pressure) and 30 psig. Several line valves and gas sticks were placed upstream and downstream of the purification media. After 18 days of exposure to the corrosive environment, the test components were measured for particles, leak rates, and visual corrosion. The results of this investigation are presented to illustrate the effect of the high pressure purifier on gas distribution components.

Introduction:

Source gas and point of use (POU) purification of process gases is necessary to meet the stringent demands of the semiconductor industry. Gas purity levels are tested in the cylinder at the production site and sometimes at the Fab prior to connecting the cylinder to the gas distribution system. However, this only ensures that the gas coming out of the cylinder meets the designated purity specifications of the cylinder. Unfortunately, many contaminants can be added to the process gas prior to reaching the tool or point of use. It has

been reported that the gas distribution system is responsible for 68% of contamination in a CMOS process¹. Impurity molecules can be introduced into the gas distribution system during cylinder changing, inadequate purge procedures, or exposure to contaminated equipment. It should be noted that although the gas from the cylinder is changed and analyzed frequently, the gas distribution components are continuously exposed to the process gas, and gradually become contamination sources for the wafer tool. This effect is enhanced when using corrosive process gases.

Source gas and POU purifiers will improve and maintain the purity of a gas or gas mixture by removing impurity molecules. Improvement in the gas quality translates into an economic enhancement in the manufacturing process, more reliable and consistent equipment performance, and a reduction in equipment maintenance or replacement.

The use of corrosive process gases causes an increase in contamination issues associated with gas distribution systems. Furthermore, the effect of moisture, within the corrosive gas or on the surface of the gas distribution system, has a large impact on contamination contribution. Previous research has shown that adsorbed water is extremely difficult to remove from a metallic surface². Matheson researchers have shown that once moisture is adsorbed onto a metallic surface, it is held quite strongly relative to other gas phase molecules due to hydrogen bonding³. In a simple experiment, Matheson researchers showed that moisture cannot be effectively removed by purging with a dry inert gas over the course of several hours. Mild heating or

addition of any hygroscopic material (such as anhydrous HCl) will cause a significant increase in the moisture concentration³ due, in part, to desorption from surfaces. In addition to desorbed moisture, it has been shown that HCl will react with iron oxides to produce iron chlorides and water⁴. Although this reaction subsides as the iron oxides are converted to iron chlorides, this provides an *in situ* mechanism for producing moisture within a completely dry gas distribution system.

It is well known that corrosion (accelerated by moisture) is a major problem in the semiconductor industry, and causes replacement of gas distribution systems on a regular basis. Metal halides and metallic particulate contamination (caused by corrosion) have a direct impact on the wafer yield. Corrosion is due to moisture reacting with the anhydrous acid gas to produce by-products that are much more corrosive than the anhydrous starting material. It has been reported that 0.1 ppm moisture in HCl does not attack 316L SS, while 200 ppm moisture in HCl can significantly corrode the 316L SS surface⁴. Corrosion data for HBr revealed that at levels of 0.5 ppm moisture, the electropolished 316L SS showed little reaction over the course of 10 days⁵. At 100 ppm moisture in HBr, results showed the electropolished 316L SS was pitted and covered in a dense metal bromide scale.

Corrosion in regulators has been demonstrated to be the single largest component failure within a gas distribution system⁶. The root cause for failure in the regulators was attributed to condensation of moisture and HBr during adiabatic expansion when the gas passes through the poppet of the regulator. Such pressure failures are a

great concern due to the damage this may cause to downstream pressure sensitive equipment.

Matheson's Nanochem[®] series of purifiers offer an effective solution for micro-electronic gas purification in its ability to deal with various gases and many types of impurities. While organometallic Nanochem[®] purifiers are very efficient for inert gases, inorganic materials are used for corrosive acid gas purification. Matheson's DPX purifier is known for its high performance under severely corrosive gas conditions, but is limited to relatively low pressures (<100 psig). HPX can handle full cylinder pressures from HBr and HCl, but suffers from a relatively lower moisture capacity than DPX.

Matheson's second generation corrosive gas purifiers are based on activated inorganic materials. These new materials are capable of handling cylinder pressures while offering capacities dramatically higher than those of HPX and even substantially higher than those of DPX. Furthermore, this new material operates at similar or better efficiencies as previous purifiers, and emits practically no metal impurities. A source gas purifier for high pressure corrosive gases has a distinct advantage. It is able to protect all components (MFC's, regulators, on-line monitors, etc.) and piping in the gas distribution system immediately after the cylinder valve.

The contents of this paper focus on measuring the effect of purification on typical gas distribution components and piping. A literature review in this area reveals much work accomplished on the effect of moisture on gas distribution piping and components, but the end results are typically photographs, SEM and XPS analysis. Although these

techniques are quite useful to obtain a qualitative sense of the degree of corrosion, they cannot provide quantitative analytical data. By contrast, the research contained herein shows direct quantitative information on the value of using a source gas or POU purifier. This was accomplished by conducting particle counts and leak rate measurements of components before and after exposure to HBr, upstream and downstream of the purifier.

Experimental:

Materials:

ULSI 5.0 purity (99.999%) grade Hydrogen Bromide (HBr) was obtained from Matheson Gas Products in Newark, California. Two different source gases were used during the course of the experiments. The first HBr cylinder was used by mixing the effluent from the cylinder with a moisture doped nitrogen stream. By using the HBr in this manner, it was possible to vary the moisture level from 1 ppm to >5000 ppm. The second HBr cylinder was intentionally doped with moisture such that the resultant concentration in the cylinder was 100 ppm. Over the course of the experiment, 46 lbs. (216.2 ft³) of HBr was directed through the test samples and purifier.

Dry nitrogen mixed with moisture was used as the matrix gas for moisture dilutions and was used to dope the HBr gas. Dry nitrogen was also used as the purge gas for the manifold before and after the HBr introduction. Nitrogen was obtained from a cryogenic source and was purified using a Nanochem[®]

OMX Bloc Purifier[™]. Use of the Nanochem[®] OMX Bloc Purifier reduced the moisture level in the nitrogen gas to <1 ppb. Unpurified cryogenic nitrogen was used during particle count measurements. The moisture level was determined to be approximately 1 ppm.

Procedures:

Manifold Purge and Drydown

The manifold was purged with Nanochem[®] purified nitrogen at a flow rate of 5 L/min overnight. While the nitrogen was still flowing, the manifold was wrapped in heat tape and heated for 24 hours at a temperature of 60-70°C. During this portion of the purge sequence the air actuated valves were being cycled in pairs for 30 minute intervals to purge out each leg of the manifold. Heating of the manifold was terminated and the manifold was allowed to cool. The purified nitrogen was turned off and all the valves were opened. HBr was then introduced into the manifold for 10 minutes. The manifold was then purged with purified nitrogen at a flow rate of 1 L/min for 90 hours. This method is used to ensure that moisture desorbing from the surface, and moisture being produced from the reaction of HBr with iron oxide is swept away and not responsible for corrosion in the test samples.

Handling of Test Components

The gas piping samples (also referred to as gas sticks) and valves were particle counted prior to exposure of HBr gas to record a baseline value for each component as well as after the exposure.

Since the gas sticks and valves were handled in a non-cleanroom environment, before and after exposure to HBr, there were some concerns of particle contamination that could have occurred during transfer to the particle counting apparatus. To minimize this type of airborne contamination, each gas stick and valve was purged for 30 minutes in the particle counting sample apparatus prior to collection of any data.

Precautions were also taken to ensure that corrosion of lines upstream of the test samples did not deposit corrosion products into the valves or gas sticks. 60 µm VCR[®] filter gaskets were installed upstream of each valve and gas stick prior to HBr exposure. Figure 1 represents the schematic of the manifold, including the purifier and test components. The gaskets were normal, ¼" VCR[®] style, nickel gaskets that had 316L SS filters pressed into the gasket body. The small surface area of the gasket trapped particles larger than 60 µm, while not trapping significant amounts of moisture.

Particle Counting of Gas Sticks

The gas sticks were subjected to three different forms of particle counting: Static, Dynamic and Impact particle counting. All particle counting tests were conducted based on ASTM Method F 1394-92: Standard Test Method for Determination of Particle Contribution from Gas Distribution Systems. All particle counts were conducted in nitrogen.

Static particle counting was performed on the gas stick by flowing 14 L/min through the test sample and 14 L/min through a bypass leg for a total flow of 28 L/min to the particle counter. Particle counts were recorded once per

minute for 20 minutes. The value reported for each individual sample was the average of the particles collected over this 20 minute time period.

Dynamic particle counts were performed similarly with respect to flow rates. However, upstream of the particle filter, on the sample leg, an air-actuated valve was cycled open for ten seconds and closed for two seconds. The valve was cycled five times during a one-minute period for 20 minutes. This action would cause a pressure drop and turbulent airflow within the test sample, causing particles to become dislodged from the test component. The value reported for each individual sample was the average of the particles collected over this 20 minute time period.

Impact particle counting was conducted by flowing 14 L/min through both the sample component and by-pass leg while striking the test component with a 50 gram steel ball that is attached to a string. The steel ball and string was drawn back to a 45° angle and then released. An apparatus was built to ensure that the impact test could be reproducibly repeated. The steel ball falls in an arc like a pendulum, striking the gas stick and dislodging particles on the internal surface. The impact on the sample was completed once per minute, for ten minutes. The value reported for each individual sample was the average of the particles collected over this 10 minute time period.

Particle Counting of Line Valves

Throughout the duration of the experiment, the valves were cycled nearly 1000 times. This included the cycles necessary to purge the manifold before and after HBr introduction.

The test valves were subjected to two different forms of particle counting: Static, and Dynamic particle counting.

All particle counting tests were conducted based on ASTM Method F 1394-92: Standard Test Method for Determination of Particle Contribution from Gas Distribution Systems. All particle counts were conducted in nitrogen.

After a 30-minute purge to achieve a baseline value, the test valves were subjected to a static particle count. Static particle counting was performed by flowing 14 L/min through the sample valve and 14 L/min through a bypass leg, for a total flow of 28 L/min to the particle counter. Particle counts were recorded once per minute for 20 minutes.

The test valves were then subjected to dynamic particle counting. During dynamic particle counts, the total flow rate (28L/min) into the particle counter remained the same throughout the experiment. The test valve was cycled open for ten seconds and closed for two seconds (five times during a one-minute period), for 20 minutes. By actuating the valve this would cause particles to become dislodged from the test component.

Helium Leak Rates

The test valves were subjected to two different forms of leak rate testing: “Inboard” with the valve open and closed, and “Across-the-Seat”. For inboard leak testing a cap was used to seal the inlet side of the valve and the leak detector was connected to the downstream side of the valve.

During the inboard leak rate procedure, the valve was placed in an environment comprised of Helium. While the valve was closed, a vacuum was established inside of the valve and a leak rate recorded. After recording the

leak rate with the valve closed, the valve was opened and the leak rate was recorded again with the valve open.

Across-the-seat leak checks were conducted by connecting a 5 psig helium source to the inlet side of the valve. The valve was kept closed, while a vacuum was established on the outlet side of the valve. The valve remained in this state for a period of 15 minutes with the leak rate information being recorded every minute. The data reported herein were collected at the tenth minute.

Moisture Doping

Experiments using full cylinder pressures were conducted by using the aforementioned, intentionally doped, cylinder (100 ppm moisture), and a standard ULSI grade HBr cylinder (≤ 1 ppm). Experiments using low pressure HBr were conducted by using an in-house, custom built, dynamic gas dilution manifolds and moisture permeation devices. Use of this equipment allowed the variation in moisture levels from parts per billion to parts per thousand.

Moisture in HBr Exposure Conditions

The test components (gas sticks and line valves) and purifier were exposed to various experimental conditions. The purpose for the variation was to simulate different conditions that an actual gas distribution system could encounter during normal operations. This included HBr pressure exposures of 30 psig to 320 psig (full cylinder pressure), moisture levels from 1 ppm to 5,500 ppm, and flow variations from 160 ml/min to 500 ml/min. In all cases, as illustrated by the data, the components downstream of the purifier were

essentially unaffected by the exposure to the HBr. The components upstream of the purifier encountered severe degradation in performance, and were visibly observed to exhibit severe corrosion. Table 1 presents the experimental parameters used during this study.

Table 1

H ₂ O Conc. (ppm)	HBr Expose Time	HBr Press (psig)	HBr Used lbs.	HBr Used Vol. ft ³
1	7 days	320	18.5 lbs	87 ft ³
100	3	320	14	66
225	7	30	12	57
5,500	1	30	1.5	7
Total	18		46 lbs.	217 ft³

Data and Results:

Particle Concentrations

Particle Counting of Gas Piping Samples

The gas piping samples (gas sticks) were placed upstream of the purifier and revealed a dramatic difference in static particle measurements compared to the downstream gas sticks. The average particle count for the upstream gas sticks after exposure to moisture laden HBr was 275 particles/ft³. The average particle count for the downstream gas sticks after exposure to moisture laden HBr was 2 particles/ft³. Figure 2 exhibits individual particle levels for each sample tested. This represents over two orders of magnitude difference in the amount of particle contamination for the purified versus non purified gas sticks.

The dynamic particle count results revealed similar results. The average particle count for the upstream gas sticks after exposure to moisture laden HBr was 453 particles/ ft³. The average particle count for the downstream gas sticks after exposure to moisture laden HBr was 2 particles/ ft³. Figure 3 depicts individual particle levels for each sample tested.

The impact particle count results were the most dramatic. The average particle count for the upstream gas sticks after exposure to moisture laden HBr was 3,493 particles/ ft³. The average particle count for the downstream gas sticks after exposure to moisture laden HBr was 6 particles/ ft³. Figure 4 represents individual particle levels for each sample tested. The trend of increasing particle levels when comparing static, dynamic, and impact tests, was an expected result. This result was due to the fact that each test was more aggressive in dislodging particles than the previous test. The results from the impact particle count tests represent a difference of over 500 times in the amount of particle contamination for the purified versus non purified gas sticks. What is even more impressive is the fact that the particle levels for the downstream samples are near zero. This indicates that the purifier makes a large impact on control of particulate contamination (due to corrosion). Furthermore, use of the source gas purifier allows the gas sticks to operate in a nearly particle free environment.

The gas sticks were cross-sectioned and examined visually. The upstream gas sticks exhibited much more discoloration, and appeared to have a thin scale or film present on the surface. The reddish-brown discoloration was thought to be due to

iron bromide formation on the surface. High magnification photographs showed the surface morphology to be rough with deposits and debris present. The downstream samples appeared to be slightly dulled or turbid compared to new electropolished tubing. There was no visible evidence of corrosion, discoloration, scale, nor debris present for the downstream samples. Although the upstream and downstream samples exhibited significant differences in appearance, the degree of corrosion of each sample remained a subjective evaluation. Particle counting and leak rate measurements offer objective quantitative data on the degree of corrosion. The visual results obtained demonstrate the need for quantitative methods of analysis, such as particle counting. As pointed out earlier, SEM and XPS analysis are useful techniques, but they are not able to ascertain quantitative information on the degree of corrosion. The fact that a gas line can undergo an impact test with a 50 gram steel ball, while not emitting particles, is unrefutable evidence that minimal corrosion has occurred.

Particle Counting of Line Valves

The diaphragm line valves that were placed upstream of the purifier revealed a dramatic difference in static particle measurements compared to the downstream valves. The average particle count for the upstream valves after exposure to moisture laden HBr was 2,114 particles/ ft³. The average particle count for the downstream valves after exposure to moisture laden HBr was <1 particle/ ft³. Figure 5 shows the

individual particle levels for each sample tested. This represents over three orders of magnitude difference in the amount of particle contamination between the purified versus non purified valves.

The dynamic particle count results revealed a similar trend. The average particle count for the upstream valves after exposure to moisture laden HBr was 4,790 particles/ ft³. The average particle count for the downstream valves after exposure to moisture laden HBr was 5 particles/ ft³. Figure 6 depicts the individual particle levels for each sample tested.

Trends observed for static and dynamic particle levels show the expected result of higher particle levels for the dynamic tests. The rationale for this behavior was mentioned previously. The results from the static and dynamic particle count tests represent a difference in the amount of particle contamination for the purified versus non purified valves of >2000, and ≈1000 times, respectively. This result is even more surprising than the results from the gas sticks. The increase of total particles in the valves compared to the gas sticks was an expected result. However, the near zero particle levels in the downstream valves was quite unexpected. This data indicates, again, that the purifier makes a large impact on control of particulate contamination (due to corrosion), and clearly demonstrates the value of using POU and source gas purifiers within gas distribution systems.

The valves were disassembled and examined visually. The results of this examination revealed that the upstream samples suffered severe corrosion. The upstream valves appeared highly discolored on the seat and diaphragm, and exhibited significant amounts of corrosion products within the

valve cavity. The downstream samples appeared slightly dulled, but showed no signs of corrosion products in the valve cavity.

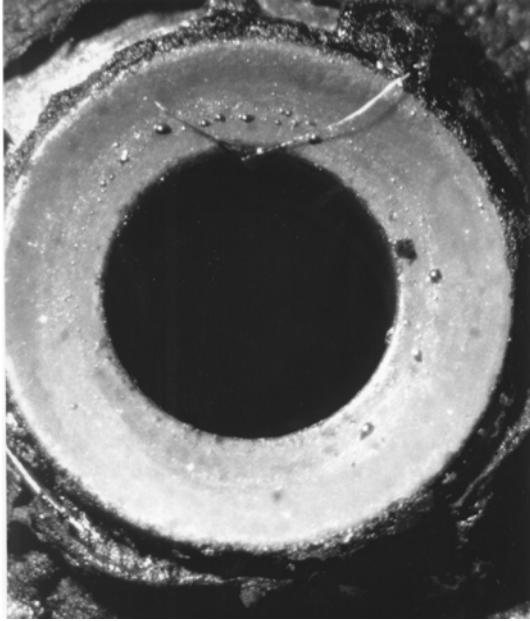
Size distribution of the generated particles, for both the gas sticks and valves, revealed that the majority of the particles generated were 0.3 microns. Figures 7 and 8 exhibit the particle size distribution for six different sizes. This size distribution is somewhat unusual. Typical particle tests on polymer seated valves show the smallest particle size will be the most abundant, and the largest the least abundant. It was unexpected that the 0.3 micron particle was the most abundant particle size for these samples. Although the particle counter used during these experiments was recently calibrated by the manufacturer, several independent tests were completed to ensure that the instrument was operating properly. The results of the tests demonstrated that the particle counter was operating normally, and the data collected for the gas sticks and valves were not effected by systematic or determinate errors.

Helium Leak Data

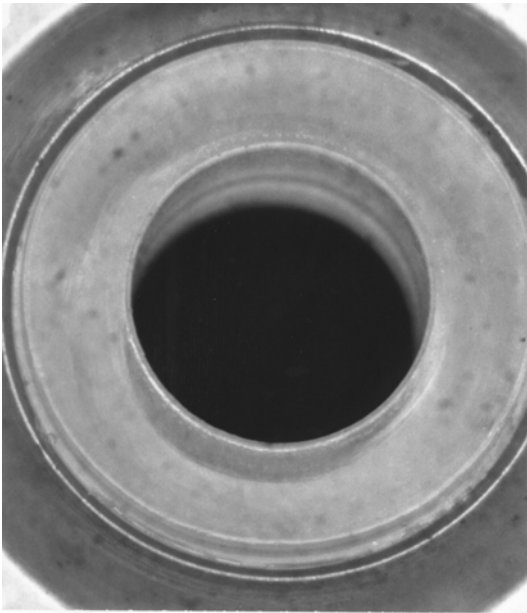
Inboard and across the seat helium leak rates were measured for each of the valves as previously described in the Experimental section of this paper. The average inboard helium leak rate for the upstream valves after exposure to HBr (in the closed position) was found to be 1.8×10^{-10} cc/sec. The average inboard helium leak rate for the downstream valves after HBr exposure (in the closed position) was found to be 1.9×10^{-10} cc/sec. The inboard leak rates for the sample valves did not change significantly with the valves in the open position. The results from the inboard leak rate data (before and after exposure

to wet HBr) reveal that the HBr did not have an effect on this parameter.

The average across-the-seat helium leak rate for the upstream valves before HBr exposure was found to be 2.7×10^{-8} cc/sec. The average across-the-seat helium leak rate for the upstream valves after HBr exposure was found to be 8.9×10^{-6} cc/sec. This corresponds to an increase in the leak rate of over 300 times for the valves placed upstream of the purifier. The average across-the-seat helium leak rate for the downstream valves before HBr exposure was found to be 3.3×10^{-8} cc/sec. The average across-the-seat helium leak rate for the downstream valves after HBr exposure was found to be 3.7×10^{-8} cc/sec. This corresponds to an insignificant increase in the leak rate for the valves placed downstream of the purifier. Figure 9 gives individual leak rate values for each sample tested. The large increase in the leak rates for the upstream valves was attributed to corrosion material being deposited between the valve body and polymer seat. Examination of the upstream valves revealed the sealing area to be highly contaminated with what was presumed to be iron bromide. Examination of the downstream valves revealed the sealing area to be free from any debris with minimal discoloration. Photographs 1 and 2 visually illustrate the observable differences between the upstream and downstream valves.



Photograph 1: Polymer seat of a line valve, after exposure to HBr, placed upstream of the purifier.



Photograph 2: Polymer seat of a line valve, after exposure to HBr, placed downstream of the purifier.

Conclusions:

The research contained herein shows direct quantitative information on the value of using a POU or source gas purifier. This was accomplished by

conducting particle counts and leak rate measurements of components before and after exposure to HBr, and upstream and downstream of the purifier. The effect of the high pressure purifier was investigated under severely corrosive gas conditions.

The test components (gas sticks and line valves) and purifier were exposed to various experimental conditions. The purpose for the variation was to simulate different conditions that an actual gas distribution system could encounter during normal operations. In all cases, as illustrated by the data, the components downstream of the purifier were essentially unaffected by the exposure to the HBr. The components upstream of the purifier encountered severe degradation in performance, and were visibly observed to exhibit severe corrosion.

The gas piping samples (also referred to as gas sticks) that were placed upstream of the purifier revealed a dramatic difference in static, dynamic, and impact particle measurements compared to the downstream gas sticks. This result was due to the fact that each test was more aggressive in dislodging particles than the previous test. The results from the static, dynamic, and impact particle count tests represent a difference in the amount of particle contamination for the purified versus non purified gas sticks of >100, >200, and >500 times, respectively. What is even more impressive is the fact that the particle levels for the downstream samples are near zero. This indicates that the purifier makes a large impact on control of particulate contamination (due to corrosion). The fact that a gas line can undergo an impact test with a 50 gram steel ball, while not emitting

particles, is unrefutable evidence that minimal corrosion has occurred.

Particle levels for the valves revealed similar trends. Data observed for static and dynamic particle levels show the expected result of higher particle levels for the dynamic tests. The results from the static and dynamic particle count tests represent a difference in the amount of particle contamination for the purified versus non purified valves of >2000, and \approx 1000 times, respectively. This result is even more surprising than the results from the gas sticks. The increase, of total particles, in the valves compared to the gas sticks was an expected result. However, the near zero particle levels in the downstream valves was quite surprising

Helium leak rates increased (across-the-seat) over 300 times for the valves placed upstream of the purifier compared to downstream valves. The large increase in the leak rates for the upstream valves was attributed to corrosion material being deposited between the valve body and polymer seat. Examination of the upstream valves revealed the sealing area to be highly contaminated with what was presumed to be iron bromide. Examination of the downstream valves revealed the sealing area to be free from any debris with minimal discoloration.

This data conclusively indicates, that the purifier makes a large impact on control of particulate contamination (due to corrosion), and clearly demonstrates the value of using POU and source gas purifiers within gas distribution systems.

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Acknowledgments:

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welding and assembly of the manifold and test samples in a very expeditious manner, and Steve Crochiere of the Maxtor Corporation for providing the photographs of the valve seats.

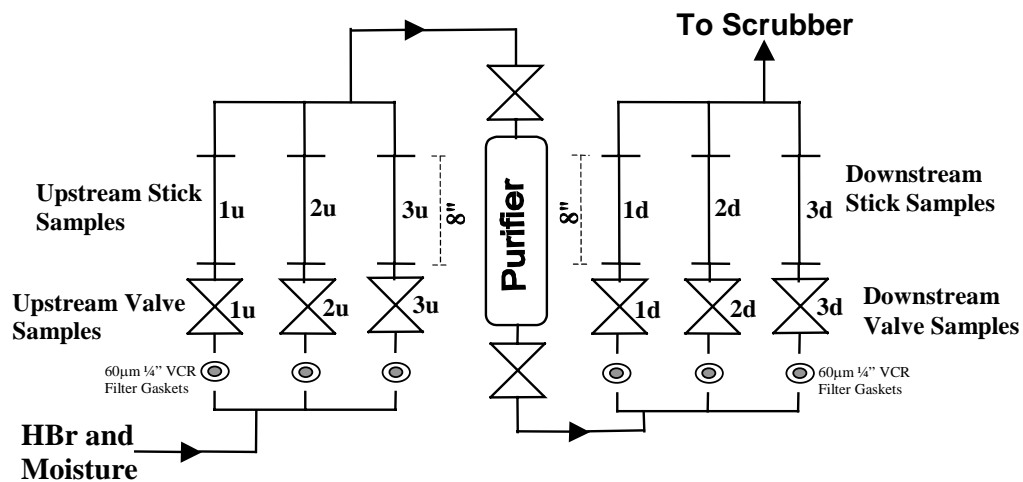


Figure 1: Schematic of the manifold used to conduct experiments.

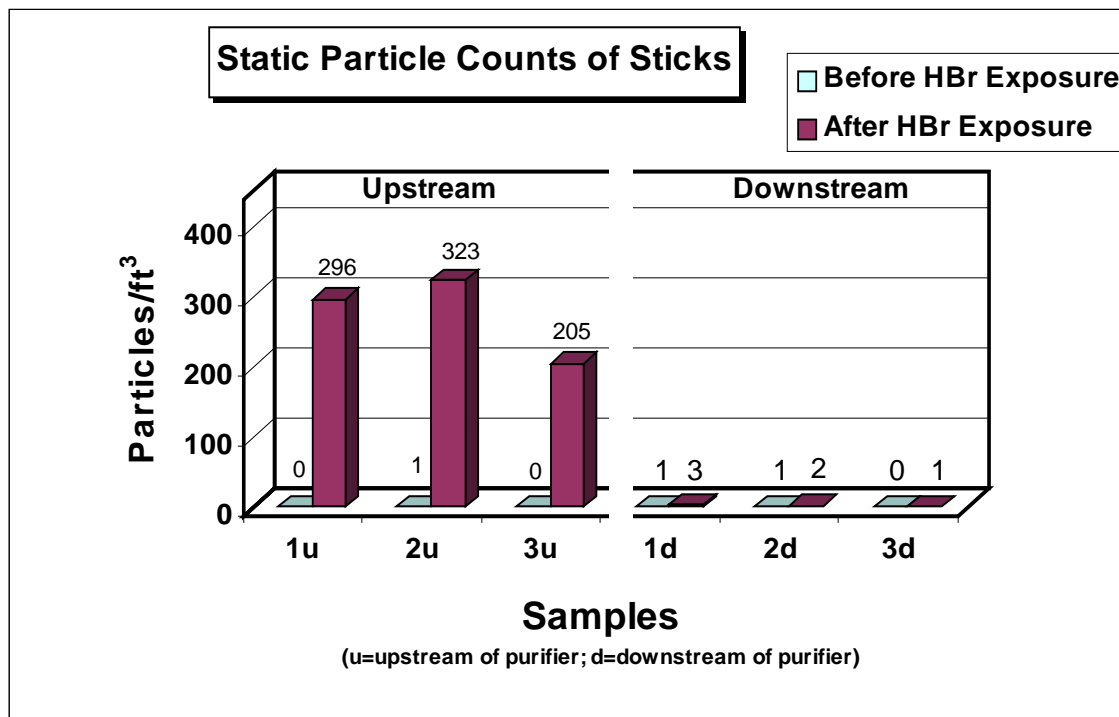


Figure 2: Average static particle counts of gas sticks before and after exposure to HBr.

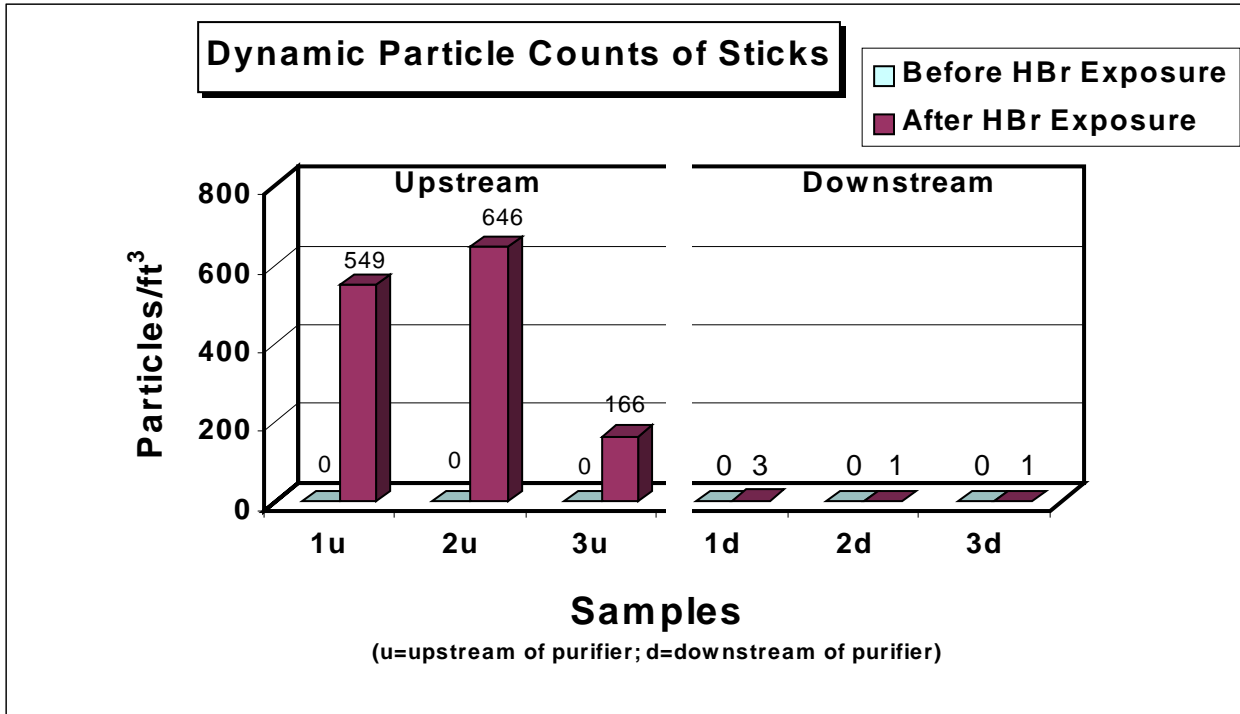


Figure 3: Average dynamic particle counts of gas sticks before and after exposure to HBr.

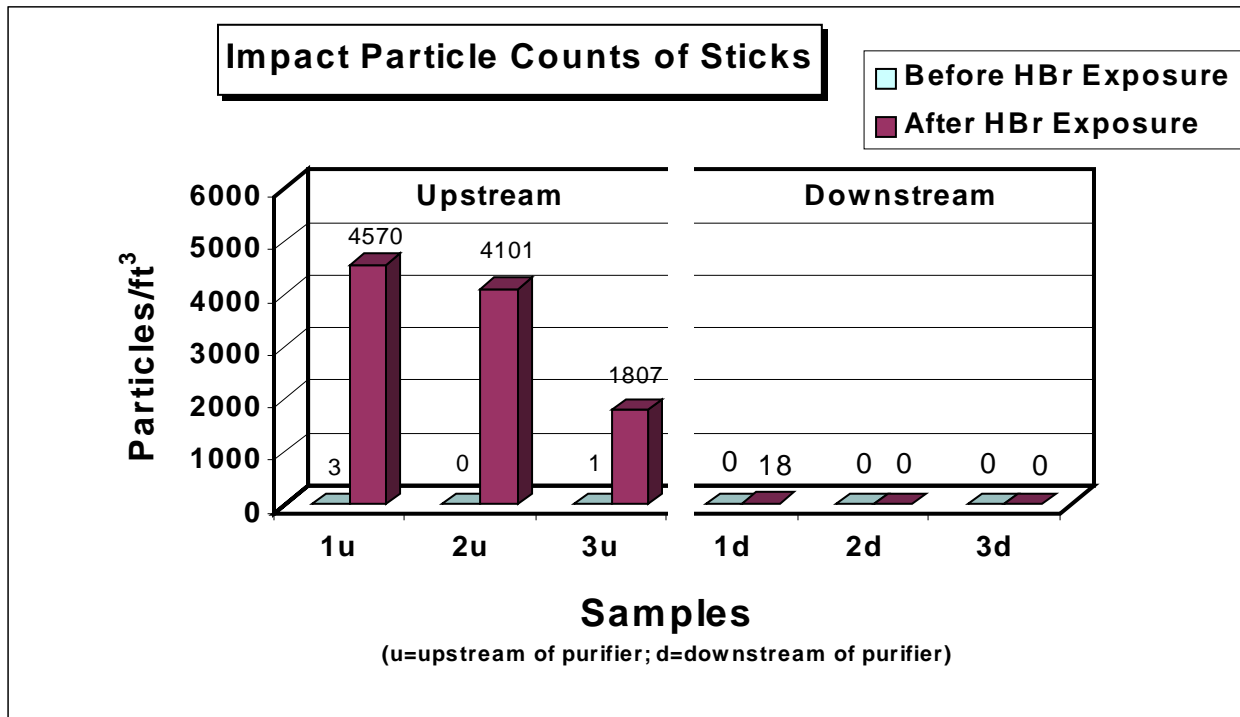


Figure 4: Average impact particle counts of gas sticks before and after exposure to HBr.

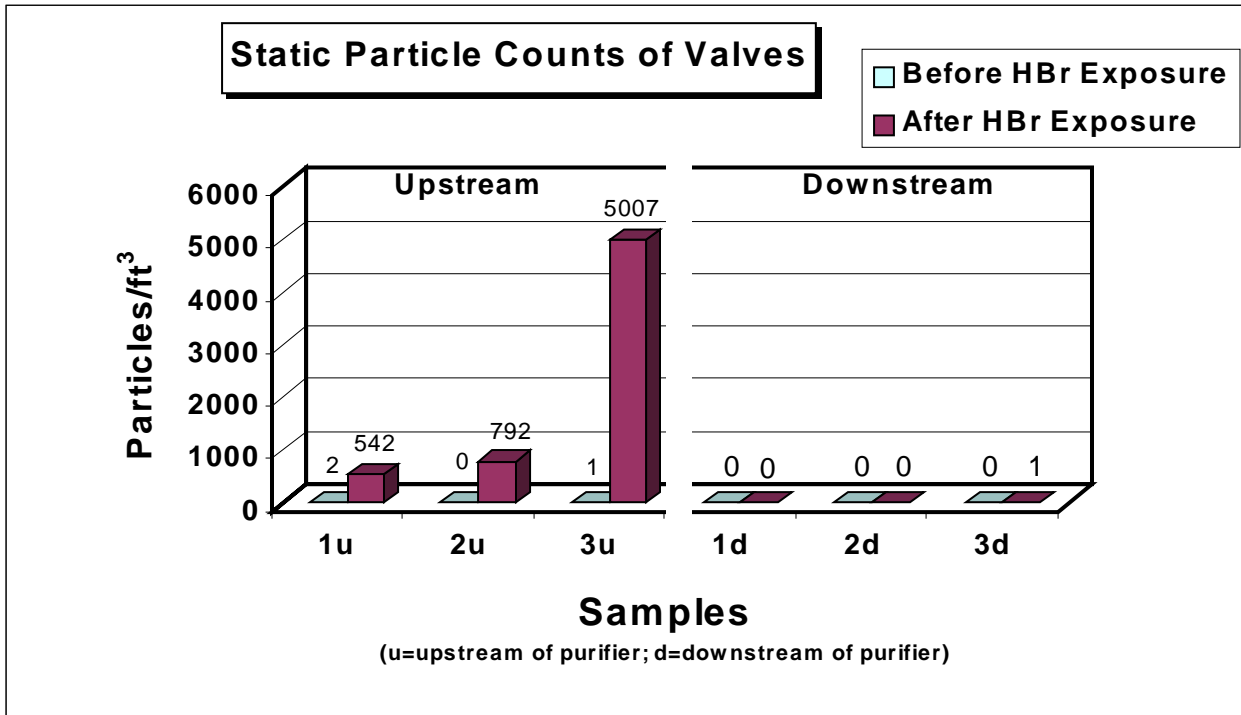


Figure 5: Average static particle counts of valves before and after exposure to HBr.

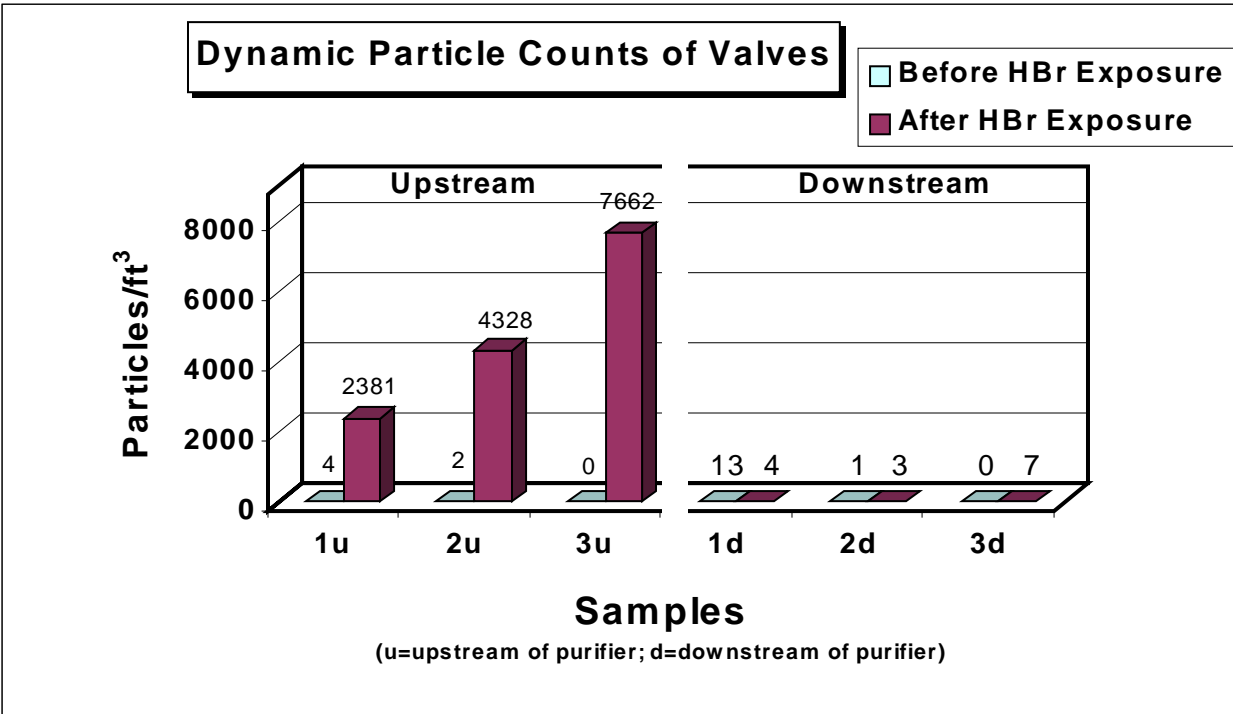


Figure 6: Average dynamic particle counts of valves before and after exposure to HBr.

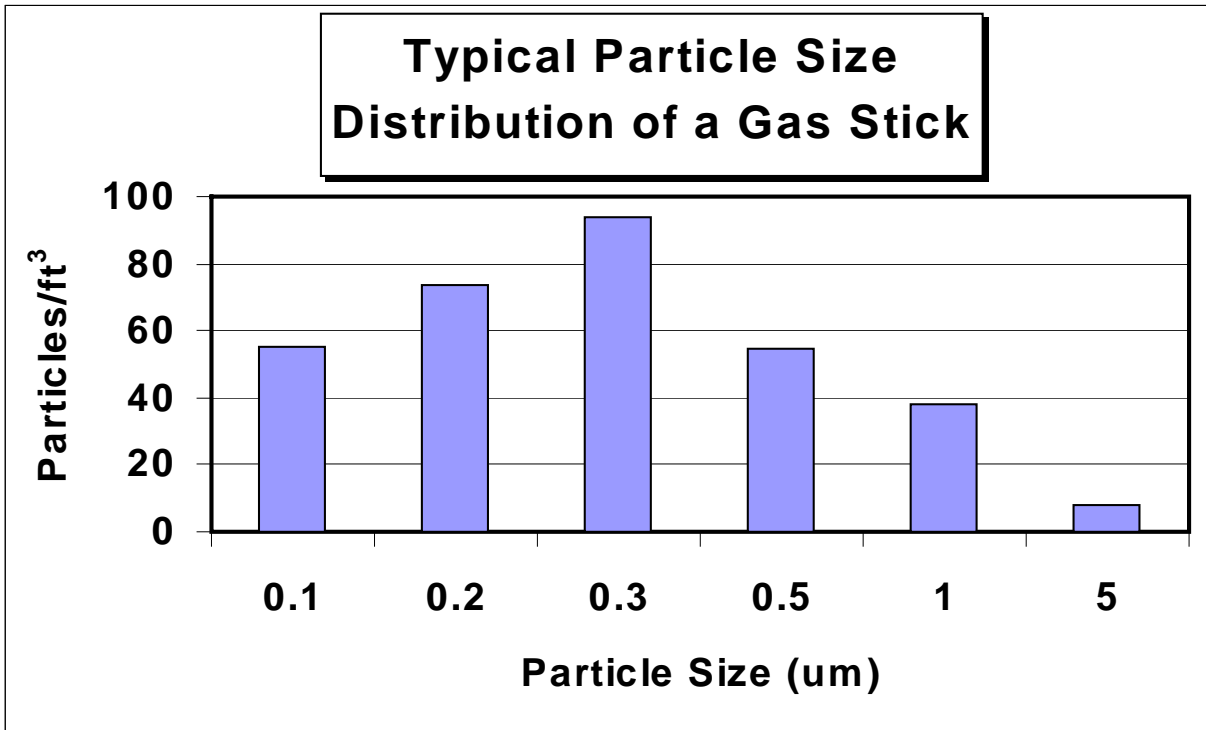


Figure 7: Average static particle counts of gas stick 1u after exposure to HBr.

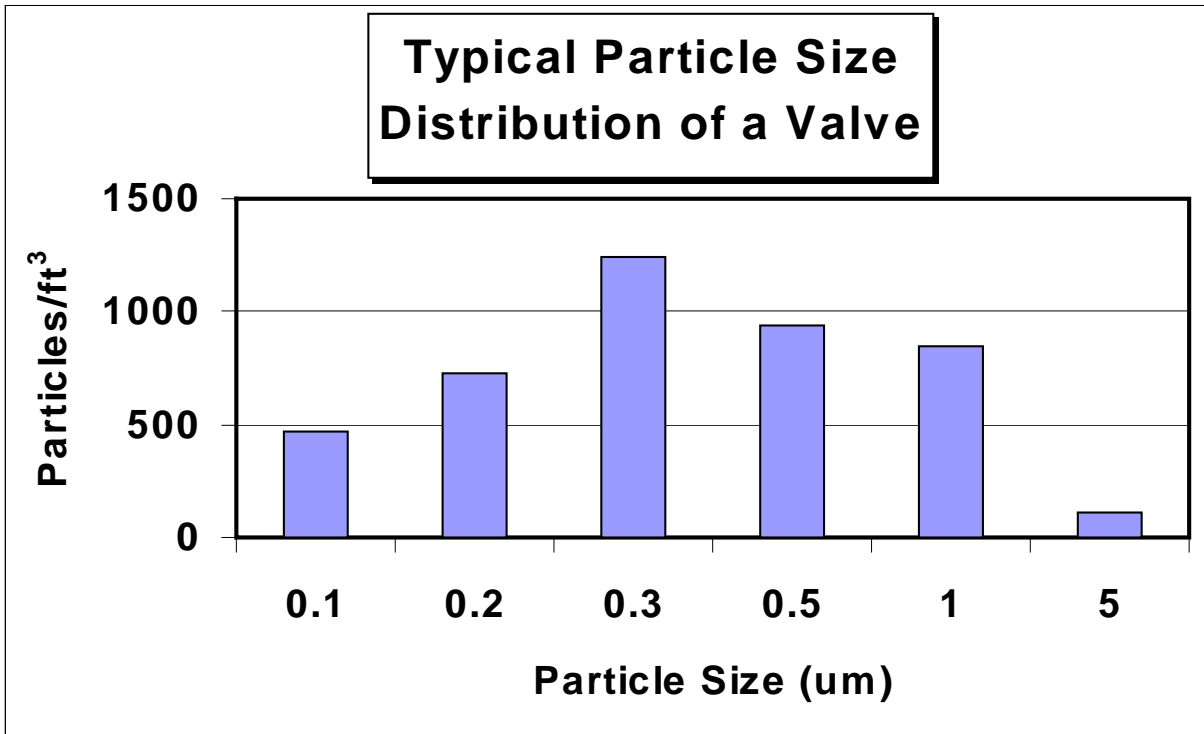


Figure 8: Average dynamic particle counts of valve 2u after exposure to HBr.

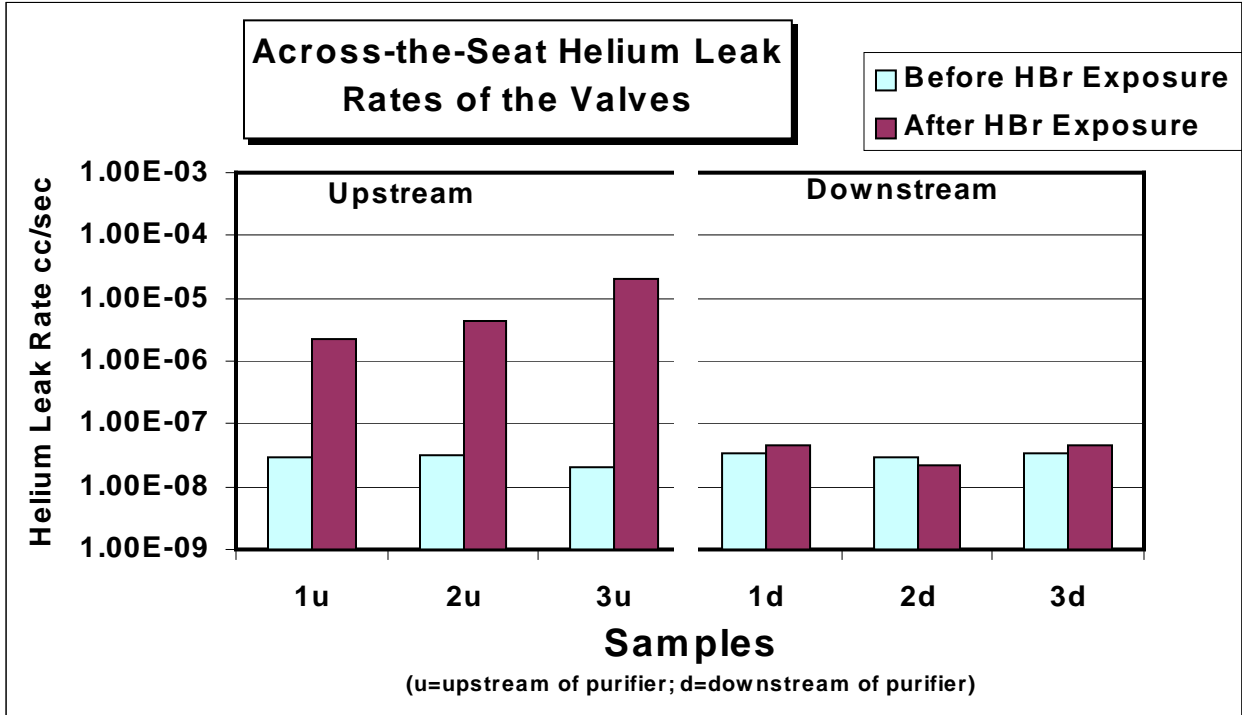


Figure 9: Across-the-seat Helium leak rates of the valves before and after exposure to HBr.