

# Single Sample Cylinder for RGA Correlation

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## Abstract

A large portion of the manufacturers of high reliability microelectronic devices must have those devices tested to be proven “dry”. A mass spectrographic technique, referred to as Residual Gas Analysis (RGA) has been the primary means of demonstrating that the devices contain less than the 5000 parts per million (ppm) of moisture. However, there are no standards for determining moisture levels of this nature when tested in this manner. Too often, the pass/fail criteria for a given production lot is exceeded and recourse becomes time consuming and expensive.

A small group of technologists has successfully fabricated and tested a Single Sample Cylinder (SSC) wherein moisture-laden air can be taken from one analytical facility to another for comparison analyses. The need for such a device is explained by a brief review of why “identical” microelectronic devices from the same production line have been found to contain different chemistries when analyzed via Residual Gas Analysis, per Test Method 1018 of the military standards.

The SSC is over two liters in volume; hence it can deliver hundreds of “air samples” that have the same chemical composition. It is maintained at 100° C during test but is kept at room ambient during non-analytical periods.

Several major problems had to be overcome in order to be able to continuously generate samples of the same chemistry. The primary two were moisture adsorption to all of the various exposed surfaces, and second was hydrogen diffusion from the materials of construction, with subsequent oxide reduction to form more water.

Some anomalies were experienced during initial preparation of the SSC and the early hours of equilibration. These again are believed to be adsorption/desorption related. Thereafter it was shown that the SSC could deliver samples that were within 2% of each other, or better, within a sample set. The level of precision between sample sets was +/- 2.5%.

This study was developed on a statistical basis, involving over 100 data points and was carried out over 1450 hours. The gas analyses were performed in a Defense Supply Center Columbus (DSCC) certified testing facility.

This study was conducted with the support of an additional analytical chemist and a statistician to assure that data generated met the goals of the stated objective.

## Keywords

Mil-Standard 883-Test Method 1018, Moisture Analysis, Residual Gas Analysis, Single Sample Cylinder.

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## Introduction

In the late 1970's, the U.S. DOD deemed it necessary to commence limiting the amount of moisture retained within hermetically sealed microelectronic devices. The limit was eventually established at 5000 parts per million (ppm) when tested at 100°C, and it was ‘published’ in Mil Std 883, as Test Method 1018 as in [1]. This test method requires that the device be heated to 100°C prior to and during the analysis. Gases within the cavity of the device are

“sampled” via a puncture and vacuum transfer mechanism within the Residual Gas Analysis (RGA) instrument as in [2].

Since the early beginning, many device manufacturers have found it difficult, at times, to stay below the limit as in [3]. In their endeavors to verify the ‘device failures’ at other facilities certified by the government to perform (RGA), it was quickly discovered that the certified laboratories often did not agree with each other when round robins or comparison studies were performed.

This problem was characterized in studies reported in 2000 at the Minnowbrook Microelectronics Conference at the Advanced Education Center of Syracuse University [4]. Thirty three lots of hermetic packages were analyzed in two different mass spectrometers both of which held DSCC suitability (Systems A & B). Both systems were calibrated and operated in accordance with Test Method 1018 requirements. About half of the sampled lots were from non-optimized processes still under development and for which internal moisture compliance was not necessarily anticipated. Sample sizes for each lot ranged from 8 to 30. One-half of each sample group ( $n =$  from 4 to 15) was measured in each system. Non-compliance to the water vapor limit was declared if two or more units per split-lot sample exceeded 5000ppmv, regardless of the number of samples in the split-lot. Highlights of the results follow.

Total lots analyzed.....33  
 Lots compliant in both systems.....23  
 Lots non-compliant in both systems.....4

Thus, in 27 of 33 cases, the two measurement systems **AGREED** on whether a given lot complied or did not comply to the limit.

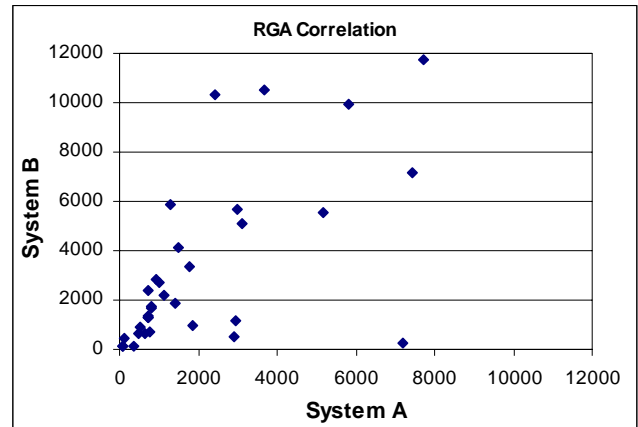
However, five lots were compliant in system A but not in system B. One lot was compliant in system B but not in system A.

Thus, in 6 of 33 cases (18%), the two measurement systems **DID NOT AGREE** on whether a given lot complied or did not comply to the limit.

As a further observation, of the 23 compliant lots and 4 non-compliant lots in both systems, 9 lots and 3 lots respectively had mean moisture values from the two measurement systems that were statistically equivalent (to a 95% confidence level). In all other cases (21 of the lots), the mean moisture values for each lot between measurement systems were statistically significantly different (regardless of whether lots were compliant or non-compliant to the limit).

A correlation plot (Figure 1) of mean moisture values for all the lots shows that correlation between systems (with a few exceptions) diverges dramatically above about 1000ppmv water vapor.

The divergence of means in the correlation plot and the relatively small number of lots with statistically equivalent means between measurement systems shed light on the matter of correlation between mass spectrometers. It underscored the need for a circulatable “consensus” standard for the internal water vapor measurement. Extensive further details of the study are available from the author of Ref. 4.



**Figure 1: Correlation Plot of mean moisture values.**

Next, in 2003, an informal presentation at the Minnowbrook Microelectronics Conference identified several different reasons as to why RGA units would probably never agree on individual, mass-produced samples as in [5]. Since a moisture standard does not exist for these test conditions, this investigator again provided insights in the 2004 Minnowbrook Microelectronics Conference, on how to minimize the effects of those factors. And, recognizing the fact that there are no primary standards, it was proposed that a consensus standard be fabricated by using a single, large volume cylinder, as the sole source of moisture laden air for all calibration/correlation studies as in [6].

Shortly thereafter, an independent group of technologists generated a database that substantiated the approach of generating hundreds of samples from the same source, i.e. a Single Sample (small volume) Cylinder (SSC). The National Aeronautics and Space Administration/ (NASA/GSFC) recognized the value in this approach and subsequently funded the next phase of the 1018 study as in [7].

This paper identifies several of those problematical factors and suggests how their elimination can bring greater precision to RGA data. The balance of this paper then describes how the SSC was fabricated, controlled and tested over an extended period of time, and after a short period of non-equilibrium, was found capable of delivering hundreds of samples of moisture laden air with essentially the same chemical composition.

## Contributing Factors

As indicated above, there have been various levels of anxiety in the electronics industries when required RGA data showed anomalies, and subsequent confirmatory

analyses by other certified RGA facilities provided even more deviant data [8]. The concern arises when the data is generated for lot acceptance purposes and weeks of production are dependent upon a few devices passing the RGA requirements. Several factors have been identified that contribute to the lack of correlation between the facilities. They are briefly discussed in this section.

#### A. No Primary Standards

The worlds of analytical chemistry and physics have advanced their respective sciences primarily through the use of reference points or standards. However, there are no standards available for identifying a known, exact amount of moisture under the conditions that exist either for microelectronic devices or for the analytical units performing the analyses. There have been attempts to remedy this problem, but none were found to be adequate.

#### B. Variations in Mechanical Tolerances

Wide variations in RGA data can result from electronic packages having significantly different internal volumes. A simple analysis of the minimum and maximum tolerances allowed on a given device per its respective print will readily show that the device cavity or sample size can vary significantly depending on how tightly one controls the tolerances. This may not be a major factor, but the differences can add to the variability in the data, especially with large aspect ratio devices. For example, a small, flat package of 0.02 cm<sup>3</sup> internal volume could have a “correction factor” somewhere between 0.5 and 2.

#### C. Expansion volumes or inlet chambers of the RGA

The physical means of sampling the devices and the transfer media differ within the various RGA instruments, and therefore so will the surface areas. Furthermore, no two certified RGA units are built the same. They have different design concepts and are constructed from different materials. Surface area and hence related chemical and physical phenomena, such as adsorption and absorption, will then contribute to variations in the RGA data.

#### D. Software Variations

Just as there are variations in analytical hardware, there are differences in the software that is used to generate and interpret the data. Various hardware and software factors enter into the equation that can readily alter the data. For instance, scan rates, pump out speeds, scan range, noise subtraction, detection limits, and sensitivity factors all have a significant contribution to make to the precision of the data. Yet, none of these factors is adequately controlled for all of the test equipment.

#### E. Sample History

Perhaps this is one of the most important variables that can go totally uncontrolled and even be unknown. Simple process controls such as FIFO (first in, first out) are often ignored when incoming lots of parts are mixed in the same supply bin “because they have the same Part Number.” Base material variations, plating lot differences, variations in times at temperature in ovens and storage, even time intervals between various steps in the plating processes have been shown to ultimately affect the final RGA data. Also, the commonly overlooked or ignored issue of absorbed/desorbed residual hydrogen in the base metals and platings and the subsequent reduction of surface oxides within the cavity of a microelectronic device by the hydrogen should not be minimized. For, even though a device is sealed in pure nitrogen, its chemistry does not remain the same. Subsequent processing and reliability stress testing allow the oxidation/reduction reactions to continue as demonstrated by Ellingham diagrams [10].

#### F. Residual “atmospheres”

Many RGA reports disclose the presence of argon in the analysis of the cavity gases of the device. All attempts to pump this gas out of the device prior to seal may fail as the argon may be carried into the device sealing process by the backfill gas, commonly “Ultra Pure” nitrogen. Yet, the argon content is often not included as a residual in the backfill gas analysis, but it is included in the RGA. Variations in the level of this inert gas can have different causes, some very condemning, such as insufficient pump down time during pre-seal in batch operations, or simply as a variable process residue from the gas supplier. Yet, the exact concentration of this gas becomes critical to proper RGA interpretation. Variations in argon values between devices should not be ignored, in fact, they must be explained.

#### G. Process Contamination/Residues

The analytical and trade journals are full of case studies where a given manufacturer found human contamination (dander, spittle, hair, etc.) in the ‘finished’ product; hence, the use of clean rooms for the manufacturing of some of the high reliability device [8]. However, there are still other sources of contamination that are more difficult to control. Process residues from cleaning solvents, improperly prepared adhesives where mix ratios differ and their precursor chemistries as well have been found to change the quality and character of devices being manufactured on a stable line [9]. These changes introduce new chemistries.

### H. Mechanics of Calibration and Sampling

The analytical community has attempted to generate secondary standards for moisture over the past several years. The three volume calibrator is an excellent example of one of the approaches. Moisture laden air (whose relative humidity is known via a National Institute of Standards and Technology (NIST) dew pointer is purged through three different pseudo-devices or chambers which are then individually injected into the RGA unit. The “known” moisture that the RGA detects is then plotted as a calibration point.

1) It has been shown that the “calibrator” can be manually or mechanically influenced, with significant changes in the subsequent calibration point.

2) A second point of concern in this area is the effect of gas flow dynamics from a static sample through a sample (puncture) hole of varying dimensions in comparison to the calibration that was done under different sampling conditions in a dynamic state.

The above factors do not represent a complete list of problematic factors, and only a cursory discussion or insight has been provided. Yet, many of these factors can be controlled when only one sample of the same geometry, physics, chemistry and history is used for calibration of the RGA instruments.

The single sample cylinder (SSC) has been fabricated and tested with that goal in mind. The approach to design, loading and testing is discussed in the following section.

### Fabrication of the SSC

The SSC was fabricated from a 2.2 liter stainless steel cylinder with two Swage-lok valves in series on each end of the cylinder. The entrapped volumes or sample volumes, between the serial valves was found to be 4.0 cm<sup>3</sup> and 2.2cm<sup>3</sup>. The “outside valve” for the volume was a “quarter turn” or micro valve to facilitate the manual sampling sequence.

It was recognized early on that two problems could occur with this equipment, (1) moisture could strongly adsorb to the inside surfaces and (2) hydrogen entrapped in the base metal of the cylinder could diffuse to the surface with prolonged time at elevated temperatures, which could contribute to the moisture content of the SSC via metal oxide reduction.

To offset the problem of moisture adsorption, a special heating mantle was fabricated by Glas-Col. This heating

unit was capable of heating the 14 pound SSC to 100°C and maintaining that temperature for long periods of time as needed. The mass of the SSC itself was also used to provide thermal ballast for when power was temporarily shut off to the mantle. After the SSC had been fabricated, it was submitted for hermeticity check via Test Method 1014 of the military standards. The two welds of the four valves and main tank of the SSC were tested and found to have a leak rate of 1.4x10<sup>-9</sup> and 1.3x10<sup>-9</sup> atm cm<sup>3</sup>/s (He) respectively.

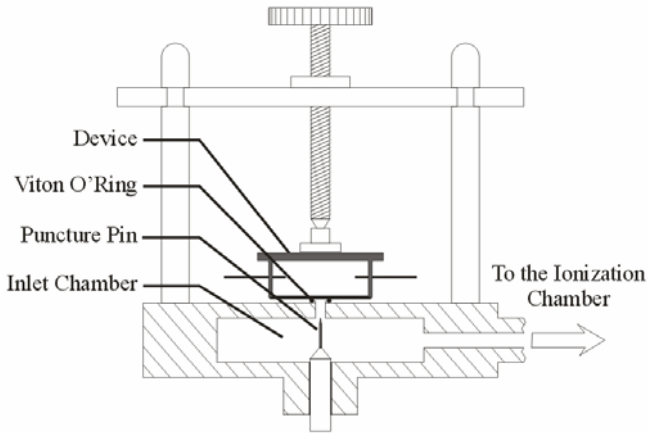
The second problem, diffusing hydrogen gas from the 304 stainless steel of the SSC, was addressed by attempting to generate a sufficiently thick oxide coating on the interior of the SSC. This was done by heating the SSC to 100°C with ambient air in contact with all metal surfaces. At first, a 3-day exposure to air with valves open at 100°C was done. This was then followed by an additional 14 days at 100°C with the valves closed. Follow-up RGA could not ascertain any differences in oxygen, hydrogen or moisture levels. Nevertheless, it was noted in the subsequent RGA studies done at 100°C, that an upward “drift” of moisture was discernable as a function of time. This problem was minimized by changing the storage temperature for the SSC to room ambient and keeping times at 100°C to a minimum.

### Preliminary RGA Studies

Historically, internal vapor content analysis of hermetic devices was developed for the purpose of detecting and monitoring moisture related failures of hybrids, ICs and relays used in military applications. Universally known as Residual Gas Analysis (RGA), its purpose was solely to quantify moisture with pass/fail criteria of 5000 ppmv. The associated procedure is documented in Mil-Std-883, Method-1018 “Internal Water Vapor Content”. Today, the general method is used to provide valuable information to evaluate, monitor and control all gases, vapors and solvents present within any cavity package whether it be military, medical, telecom, aerospace or other industry related package.

The test is performed using a specially designed Quadrupole Mass Spectrometer. This analytical technique involves the ionization and separation of gas molecules as they flow from the package cavity followed by a measurement of their relative abundance (ppmv) as a function of their mass-to-charge ratio (m/z).

Devices are loaded on the instrument one at a time. The inlet chamber (Figure 2) is then subjected to high vacuum before the pin punctures the device, thereby releasing the gaseous content into the system.



**Figure 2: Design of the inlet for RGA.**

By controlling the size of the transfer passage, the gases flow into the analyzer at a predetermined rate. Under the energy of an electron beam, gas molecules eject electrons, form ions, and fragment to some degree. Each substance has its own unique fracture pattern which is dependent on its chemical structure and instrument settings. As the ions enter the quadrupole mass filter, they are manipulated by electrical attraction and repulsion thus allowing a differentiation according to their mass-to-charge ratio ( $m/z$ ). The ions are then sequentially quantified over a given period of time.

Several preliminary RGA studies were performed on the SSC to verify its integrity and operator dependencies, if any, prior to performing the primary study.

The first study was a simple hermeticity check of the SSC via heating to 100°C and subsequent RGA. The data showed no indications of leakage in a short term of 14 days. The SSC was then cycled to room temperature and returned again to 100°C. Total elapsed time was approximately 16 days, again, no significant change in the RGA values was observed.

An operator dependency study was then conducted to ascertain if the manual switching of the SSC valves would have any effect on the RGA values. A statistician designed a twenty-sample experiment where two different operators generated the RGA data on a random basis. No differences between operators were discernable.

However, this ‘short study’ was performed at room temperature and a significant amount of heat was found to be conducted from the RGA heated sample mount to the body of the SSC. A thermal increase of several degrees to the value of the SSC was noted as the RGA inlet is maintained at 100°C or higher. Again, the rise of temperature was an indication that RGA should be performed at an elevated temperature.

## Preparation of the “Moisture Standard”

A compressed, certified dry air sample from Huan Welding Supply was used as the source for an “air” mixture. The “certification of analysis” for the gas was stated to be at a maximum moisture concentration of 1.6 ppm. Upon analysis the argon content was found to be in the 100-150ppm range. This reduction in argon content was deemed desirable as any leaks in the cylinder could be readily identified by the subsequent change in the argon content, argon having a normal atmospheric concentration of 9000 ppm.

The moisture content of the “standard” was produced by flowing the dry air through a NIST traceable dew point hygrometer set to 4600 ppm with a precision of ~202 ppm. This ‘mixture’ of moisture-laden air was then flowed through the SSC at 100°C for ~ 30 minutes. The heating of the SSC was done to minimize moisture adsorption on the SSC walls during preparation of the standard.

## RGA of the SSC as a Function of Time

Having prepared the SSC moisture standard at 100°C to minimize adsorption, it was then necessary to maintain this temperature whenever an analysis was to be performed. Hence, initially all analyses and storage of the SSC were done at 100°C minimum. The moisture contents as a function of time is provided in Figure 3 so one can observe the effects of time and other variables on the RGA of moisture content. The SSC was initially ‘charged’ with 4600ppm moisture and RGA data were generated on the SSC for over 1400 hours. Improvements in precision within data sets and between data sets were continuously observed as the environmental conditions of the SSC were gradually brought under control. A basic, statistical analysis of the RGA data generated is provided in Table 1 along with explanatory notes.

This table of data is only a portion of the statistical analyses and assessments that were generated. All of the RGA data and the associated analyses and reports of the independent statisticians are provided in the original report to NASA and are available upon request from the authors or NASA. Table 2 lists all RGA data, which is sequentially plotted in Figure 3. The states of non-equilibrium, equilibrium and sample “outliers” are readily depicted by the plot. A complete list of the moisture detected in the RGA data as a function of time is provided in Table 2.

**Table 1: RGA of the SSC as a function of time and temperature and indicated modifications.**

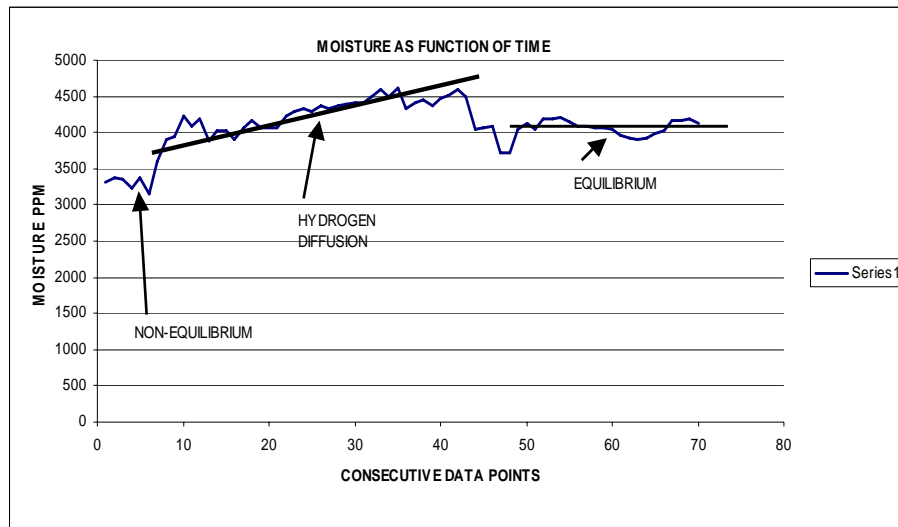
<u>Sample #<sup>d</sup></u>	<u>Time(Hrs)</u>	<u>Average RGA</u>	<u>Std Dev</u>	<u>Modifications</u>
Time 0 (1-3)	0	3345	36	Initial RGA reading
Time 1 (1-3)	20	3253	114	First “verification” RGA
Time 2 (1-3)	90	3811	192	Second “verification” RGA - new equilibrium
Time 3 (1-3)	160	4169	66	Continued RGA – noting increase in sample pressure
Time 4 (1-3)	250	3974	88	“ “ “ “ “ “ “
Time 5 (1-3)	350	4024	131	“ “ “ “ “ “ “
Time 6 (1-3)	520	4060	8	“ “ “ “ “ “ “
Time 7 (1-6)	570	4306	50	SSC transfer line insulated <sup>a</sup>
Time 8 (1-4)	610	4393	19	Continuous increase in sample pressure stopped
Time 9 (1-4)	615	4545	71	SSC at 110° C to ascertain if [H <sub>2</sub> O] increased
Time 10 (1-4)	660	4393	49	SSC at 100° C after 46 hours @ 23° C: equilibrium <sup>b</sup>
Time 11 (1-4)	750	4519	57	SSC now in temperature cycle; continued equilibrium
Time 12 (1-3)	820	4069	18	“ “ “
Time 13 (1-9)	1230	4046	190	SSC @ 23° C/ RGA @ 100° C: 5 min. dwell <sup>c</sup>
Time 13 (1-5)	1230	4067	20	“ “ “ “ 30 min. “
Time 14 (6-10)	1300	3937	34	“ “ “ “ “ “
Time 15 (11-15)	1450	4132	59	“ “ “ “ “ “

<sup>a</sup>A gradual increase in sample pressure was noted in each data set up to this point. It was found that the heated RGA sample mount and the SSC required additional thermal insulation. Thereafter, the anomaly was resolved.

<sup>b</sup>It was noted that the averages of the data sets were on a slight rise. Previous experience with hydrogen desorption from ferrous-based alloys led to the solution of this problem. ‘Storage’ temperature for the SSC was changed from 100°C to room ambient. Several additional data sets taken after this modification has shown the drift to have stabilized.

<sup>c</sup>A change in the manual valve switching sequence plus an increase in sample equilibration time were the final modifications that eliminated a significant amount of adsorption/desorption. The precision within the data sets improved to a nominal ±2%.

<sup>d</sup>The sample numbers in parenthesis correspond to reading numbers in table 2.



**Figure 3: RGA of Moisture as a Function of Time (from Table 2).**

**Table 2: RGA as a function of time and temperature.**

<u>Reading#</u>	<u>~Time in Hrs<sup>a</sup></u>	<u>[H<sub>2</sub>O]</u>	<u>Reading#</u>	<u>~Time in Hrs<sup>a</sup></u>	<u>[H<sub>2</sub>O]</u>
1	0	3303	36	660	4339
2	0	3371	37	660	4411
3	0	3361	38	660	4452
4	20	3231	39	660	4370
5	20	3377	40	750	4466
6	20	3151	41	750	4516
7	90	3591	42	750	4599
8	90	3901	43	750	4495
9	90	3943	44	850	4052
10	160	4220	45	850	4068
11	160	4094	46	850	4089
12	160	4195	47	1230	3728
13	250	3872	48	1230	3729
14	250	4027	49	1230	4049
15	250	4023	50	1230	4116
16	350	3903	51	1230	4038
17	350	4061	52	1230	4195
18	350	4164	53	1230	4197
19	520	4055	54	1230	4209
20	520	4070	55	1230	4153
21	520	4056	56	1230	4088
22	570	4229	57	1230	4081
23	570	4282	58	1230	4059
24	570	4334	59	1230	4071
25	570	4291	60	1230	4037
26	570	4375	61	1300	3961
27	570	4329	62	1300	3918
28	610	4373	63	1300	3905
29	610	4383	64	1300	3918
30	610	4403	65	1300	3986
31	610	4416	66	1450	4034
32	615	4484	67	1450	4159
33	615	4593	68	1450	4173
34	615	4484	69	1450	4178
35	615	4620	70	1450	4117

<sup>a</sup> Approximate number of hours on the SSC when the RGA data set was started.

As stated above, detailed discussions on standard deviations, t-tests, statistical significance, etc. are provided in the NASA report [7]. The comments and assessments in the reports of the co-investigators are their positions on the experiments and data generated in this effort. Our endeavor had been to obtain analytical and statistical inputs from one reviewer and a totally statistical assessment from the second reviewer, i.e. the statistician

was to review the data with as little knowledge of the ‘black boxes’, as well as the science and the associated problems, yet provide technical input. Their common conclusion was that the SSC was found to be suitable as a circulatable consensus standard to define the various RGA labs’ measurement reproducibility for water vapor.

## Conclusion

A portable, large volume cylinder with an associated set of sampling volumes and valves has been fabricated that has the potential to deliver hundreds of small volume samples, each with identical chemistries with no physical or historical variations. This cylinder can now be taken to various analytical facilities that perform moisture analyses in static samples to facilitate the calibration and cross correlation of the analytical instruments that are utilized for these analyses.

## Acknowledgment

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