ENVIRONMENTAL STRESS TESTING OF THE SINGLE SAMPLE CYLINDER

A Proven Consensus Standard for Internal Gas Analysis (IGA) or Residual Gas Analysis (RGA)

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NASA GSFC
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PS 620

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EXECUTIVE SUMMARY:

In August 2008, Schuessler Consulting was contracted by NASA GSFC in support of the NASA Electronic Parts and Packaging (NEPP) program to perform two separate studies on moisture laden air in a stainless steel cylinder that had been designed to become a consensus standard for Test Method 1018[1]. This Test Method was originally released for hybrids under Mil. Std. 883 but was quickly utilized on other microelectronic devices under the auspice of Mil. Std. 750. The cylinder had subsequently been fabricated for the 750 community. It was back-filled with moist air and subsequently analyzed over a period of time under a previous NASA contract[2]. It had been shown that moisture in the 4000 – 5000 ppm range could be analyzed rather precisely with a mass spectrometer, commonly referred to as a Residual Gas Analyzer, RGA[3]. The scope of this study was to ascertain if the composition and precision varied as a function of thermal shock at sub-zero temperatures and whether there was consensus when the standard was submitted to other RGA units.

It was demonstrated and published that the consensus standard would yield precise RGA data for moisture within ± 1% when optimized for a given RGA unit[3]. It has been subsequently shown in this study at Oneida Research Services, that sub-zero storage did not affect that precision when a well-defined protocol for the analysis was followed. The consensus standard was taken to a second facility for analysis where it was found that moisture adsorption on the transfer lines caused precision to drop to ± 12%.

The Single Sample Cylinder (SSC) is a one liter stainless steel cylinder with associated sampling valves and has considerable weight and volume. But this considerable size allows for approximately 300 gas samples of the same composition to be delivered to any RGA unit. Lastly, a smaller cylinder, approximately 75 cc, of a second consensus standard was fabricated and tested with a different mix of fixed gases where moisture was kept in the 100 ppm range. This second standard has the potential of providing 30 gaseous samples and can be readily shipped to any analytical facility that desires to generate comparison RGA data. A series of comparison residual gas analyses was performed at the Honeywell Federal Manufacturing & Technologies facility in the National Nuclear Facility Administration's plant in Kansas City to complete this project.

It was shown that improvements in the precision of a given RGA unit can be done by controlling the cycle time for each analysis and increasing analysis temperatures to minimize moisture adsorption. It was also found that a "one time event" in the subzero storage of the large SSC did not effect the units ability to continuously supply precise samples of the same chemistry, however the "event" caused a permanent +8% shift in the reported value of the moisture content.

Lastly, a set of SSC RGA results was plotted on a common graph with DSCC "correlation study" RGA data. The result demonstrates the ability of the SSC to remove many of the individual variances that single, individual samples introduce.

The consensus standards are now in storage at Oneida Research Services, one of the DSCC certified houses that does RGA to Military Standards, where they await future studies.

The analytical data and the operational parameters of the instruments used are provided in the following discussion. Limitations and suggested means for improvement of both precision and accuracy are provided.

DISCUSSION:

This effort focused on resolving two issues; first, determine the effects of sub zero thermal shock and second, to ascertain the effects of transportation to and use on a second RGA system. Therefore this report is presented in two halves with the conclusions for each effort presented in that respective section. Only the RGA data for moisture and other detected gases are presented in the tables of this report. The complete RGA, as generated and submitted by the respective analytical facility are provided as attachments 1& 2.

EFFECT OF SUB-ZERO TEMPERATURES

Temperature had already been identified as a significant parameter that would effect the moisture concentrations determined for the Single Sample Cylinder (SSC). As reported in the first NASA funded study, it was shown that the hydrogen, still remnant within the stainless steel of the SSC, would diffuse to the internal surface at high temperature (~ 100 ^{o}C), reduce surface oxides and cause the moisture content of the SSC to increase. The question arose as to whether sub-zero exposure would cause irreversible adsorption/absorption as the water vapor was cooled to less than its dew point?

The SSC was subjected to – 10 ^{o}C for over 12 hours, returned to room-ambient, then analyzed at 100 ^{o}C via the protocol previously established for RGA. This environmental stress test was then repeated. In order to assure improvements in accuracy and precision of the moisture data, as part of this study, it was determined that it was imperative to identify as many mass spectrometer parameters as possible and to retain or reproduce those numerical values wherever possible. The RGA data that was generated as a result of subzero temperatures on the SSC are provided in figure 1. Note that there was a definitive shift in the “absolute” value for moisture content by ~8%, but the precision of the analyses has remained essentially the same at +/-1%. The operational parameters of the RGA for this part of the study are presented as attachments 3 & 4.

Note that the first set of RGA data (DAY1-1, 2, 3, and 4) had a wide range in values or poor precision Figures 1 and 2. This was corrected by introducing into the test protocol the requirement of a strict adherence to using the same time intervals for each step of the analysis.

The shift in the relatively constant value for moisture content may have been caused by various factors. Initially, the cause was believed to be a calibration issue. For instance, if upon calibration with a NIST traceable dew pointer with a $\underline{+}$ 100 ppm precision, a given mass spectrometer could be ‘set’ or “calibrated” to the high side of the precision or “range” of the dew pointer, then the subsequent readings of the SSC will also be high, but in reality have not changed at all. ROM estimates of the impact that this “off set” during calibration can have on the final data approximates an error of 2-3 %

FIGURE 1 - ORS RGA DATA OF SUBZERO STUDY

SAMPLE ID		DAY1-1	DAY1-2	DAY1-3	DAY1-4	DAY2-1	DAY2-2	DAY2-3	DAY2-4
INLET PRESS torr (mm)		220	221	221	232	226	230	231	231
NITROGEN	%v	79.3	79.3	79.2	79.3	79.4	79.4	79.3	79.3
OXYGEN	%v	20.2	20.1	20.2	20.2	20.1	20.1	20.2	20.2
ARGON	ppmv	167	157	167	158	164	164	166	163
CO2	ppmv	182	169	179	167	186	153	155	177
MOISTURE	ppmv	4521	5061	5070	4856	4518	4435	4657	4634

SAMPLE ID		DAY3-1	DAY3-2	DAY3-3	DAY3-4	DAY4-1	DAY4-2	DAY4-3	DAY4-4
INLET PRESS torr (mm)		233	236	235	234	233	234	234	234
NITROGEN	%v	79.4	79.5	79.3	79.3	79.5	79.6	79.6	79.6
OXYGEN	%v	20	20	20.1	20.2	20	19.9	19.9	19.9
ARGON	ppmv	162	160	163	154	160	158	157	159
CO2	ppmv	136	143	144	174	174	144	134	168
MOISTURE	ppmv	4940	4701	4949	4858	4723	4718	4693	4679

SAMPLE ID		DAY5-1	DAY5-2	DAY5-3	DAY6-1	DAY6-2	DAY6-3	DAY6-4	DAY7-1
INLET PRESS torr (mm)		227	227	228	235	238	238	237	228
NITROGEN	%v	79.5	79.5	79.6	79.7	79.6	79.6	795	79.7
OXYGEN	%v	20	19.9	19.8	19.7	19.9	19.9	20	19.7
ARGON	ppmv	166	160	157	170	164	171	151	169
CO2	ppmv	151	162	156	143	152	183	156	150
MOISTURE	ppmv	5179	5203	5130	5030	5149	5176	5157	5085

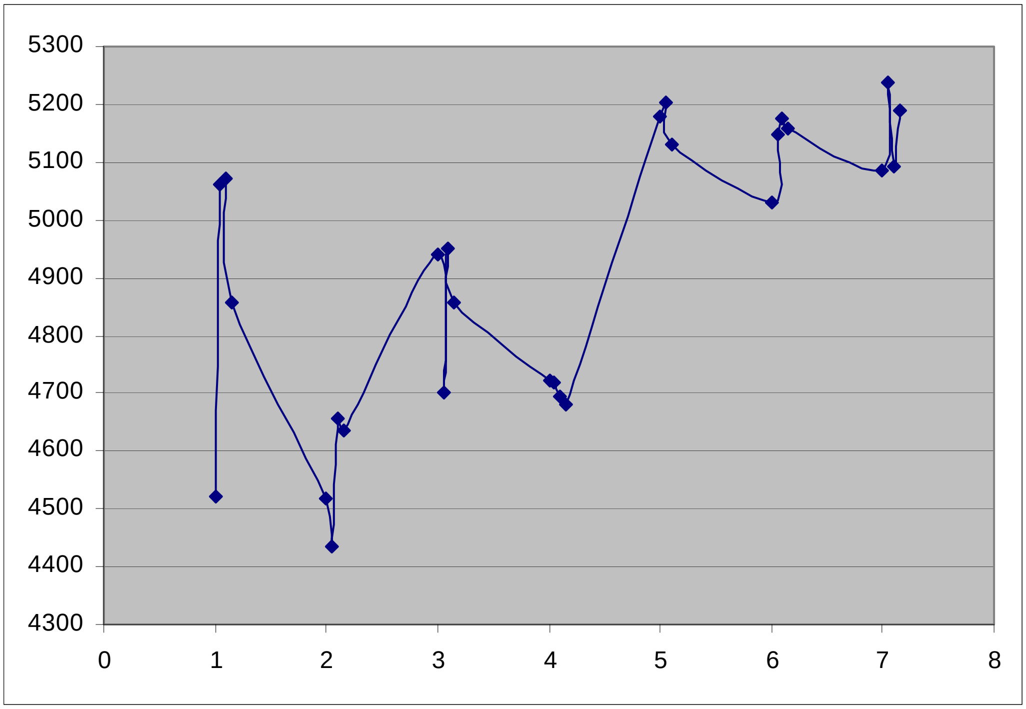
SAMPLE ID		DAY7-2	DAY7-3	DAY7-4
INLET PRESS torr (mm)		229	230	229
NITROGEN	%v	79.5	79.5	79.5
OXYGEN	%v	20	20	19.9
ARGON	ppmv	157	151	165
CO2	ppmv	162	182	178
MOISTURE	ppmv	5237	5091	5188

Sample ID DAY1-1,2,3,4:SSC was heated for>2hours at 105 C prior to analysis
Sample ID DAY2-1,2,3,4:SSC was heated for>2hours at 105 C prior to analysis
Sample ID DAY3-1,2,3,4:SSC was heated for>2hours at 105 C prior to analysis
Sample ID DAY4-1,2,3,4:SSC was heated for>2hours at 105 C prior to analysis
SSC was stored in laboratory room ambient conditions between Day 1,2,3&4
Sample ID DAY5-1,2,3: SSC was cooled to -10 C for a min. of 12 hours, heated for>2 hours @ 105 C prior to analysis
Sample ID DAY6-1,2,3,4: SSC was cooled to -10 C for a min. of 12 hours, heated for>2 hours @ 105 C prior to analysis
Sample ID DAY7-1,2,3,4:SSC was heated for>2hours at 105 C prior to analysis

Note: There are significant variations in the CO_2 data on an intra- and inter- laboratory basis. This may be due, in part, to the lack of calibration in the respective range. The question of variation has also been raised with RGA experts, but the exact cause is not known at this time.

A plot of the moisture analyses as a function of time is presented in Figure 2. The wide range in the data on Days 1 and 2 is more obvious as compared to the data on Days 6 and 7 when the timing for each analysis was tightly controlled.

FIGURE 2 - MOISTURE AS A FUNCTION OF TIME UNDER RANDOM VERSUS CONTROLLED CONDITIONS



An alternate theory for this shift in moisture has been proposed wherein micro cracking of the metal oxide passivation layer inside the SSC during the sub-zero soak has occurred or a micro-void in the welded valve joints has burst. This would allow a new path or source for H_2 from the base metal of the SSC to diffuse out into the sample and reduce surface oxides to add to the moisture content. But the question is: If it were oxide cracking, why is there just the one shift with continued stability thereafter when two test cycles had been performed? The exact cause for this shift still awaits determination.

At this time the former theory seems equally plausible and is being actively pursued via a tighter dew pointer range as a partial solution to the problem.

Regardless of the cause for the shift, this experiment indicates that the interior of the SSC experienced a one time change that increased the moisture content after cooling through the dew point. (Another question is now put before us: Can microelectronic packages behave the same way?) Nevertheless, it has been demonstrated that the SSC can withstand subzero temperatures and still deliver samples of constant chemistry to within +/-1% precision.

EFFECTS OF TRANSPORTATION

The second part of this study was to ascertain how well the SSC would provide repeatable samples after having been “shipped” to an alternate analytical facility. Since a learning curve was still being formed for this protocol, one guiding requirement had to be in effect, specifically:”eyes on”. It was deemed imperative that throughout this effort the SSC not leave the possession or “eye sight” of at least one person that was knowledgeable of its make up and proper handling during test and storage. This would essentially guarantee that if there were any changes in the data from the SSC, it could be assured that ‘someone’ was knowledgeable of the history of the SSC, and thereby rule out such factors as thermal/mechanical shock, improper sampling, abuse, etc. as root causes of the change. Throughout this part of the project, that responsibility was fulfilled by a co-contributor, Dan Rossiter of Oneida Research Services.

Several alternate analytical facilities were approached for this part of the study. Unfortunately, a variety of factors prevented all but one facility from participating in this effort in the given time frame.
The facilities considered were:

1. Atlantic Analytical Services, N.J. - originally interested, did not respond on follow-up.
2. IBM Analytical Services, NY - analyses are for internal use only.
3. Navy Crane Weapons Center, IN - equipment not functional
4. Matco Inc., PA - qualitative analysis only.
5. Fort Meade, MD - High Security Facility - difficult to access
6. DSCC. Columbus, OH - initial interest but withdrew support.
7. Honeywell Kansas City, Mo - performed comparative RGA on both cylinders
8. Pernicka, CO - not considered at this time for geographical reasons.
9. West Coast Analytical, CA - not considered for geographical reasons

The major reason for the geographical restrictions was that the ‘eyes on’ aspect of this portion of the effort did not allow for air transportation. All attempts to arrange to hand-carry either of the SSC cylinders aboard a commercial flight as checked luggage or freight were met with either a flat out negative response by the Transportation Safety Agency or no response at all.

Personnel at the Honeywell Federal Manufacturing & Technologies National Nuclear Administration’s Facility in Kansas City graciously volunteered their time and analytical support to perform a series of gas analyses on both of the cylinders. Having agreed upon a date for the support from the Honeywell F M & T facility, ORS personnel were able to ‘hand carry’ the SSC to this National Nuclear Security Administration facility for the comparative RGA. Their results are presented in figure 3.

FIGURE 3 - HONEYWELL RGA RESULTS FOR THE LARGE SSC

	Four Analyses of the Large SSC			
	Run 1	**Run 2**	**Run 3**	**Run 4**
Gases Analyzed				
Helium %	0.001	0.0011	0.0011	0.0011
Methane	0.0004	0.0004	0.0004	0.0004
Water	**0.2531**	**0.3076**	**0.3107**	**0.3381**
Nitrogen	81.1257	81.0392	80.9911	80.8761
Carbon Monoxide	0.0005	0.0004	0.0003	0.0004
Oxygen	18.5982	18.6307	18.6759	18.7635
Argon	0.0157	0.0155	0.0155	0.0156
Carbon Dioxide	0.005	0.0046	0.0045	0.0045
Total Hydrocarbon	0.0003	0.0003	0.0003	0.0003

0.0001% = 1ppm

An analysis of the data reveals an interesting trend. Apparently, moisture from the SSC “standard” is being adsorbed onto the transfer lines from the SSC and possibly within the RGA sample chamber. Note how the values trend upwards from 2530 ppm to 3381 ppm. It was observed that there are several “pieces” of instrumentation on the RGA which have the potential to add more surface area upon which adsorption can occur. It appears that upon pump down, more adsorbed moisture is desorbed the longer the surface is pumped. So if the pump down to a low noise level is a short time, less water is desorbed and subsequent analyses yield higher and higher moisture levels. Attempts to reduce adsorption effects by controlling the time intervals between and during analyses are only part of the answer, see attachment #6. Internal surface area variations between RGA units may require “personalized” pump down protocols to help reduce the adsorption problem.

EFFECTS OF COMMERCIAL /GROUND TRANSPORTATION

A second, smaller rendition of the SSC was fabricated and backfilled with a gas composition of lower moisture content. The primary goal here was to ascertain if shipping the SSC via ground transportation would have any effects on the final data. The moisture content of this SSC was also lowered; it tends to facilitate the detection of any changes in the composition that might occur from shipping. As in the previous effort, the Honeywell facility was utilized. This allowed the work to be done by personnel now familiar with the objectives of the effort as well as the mechanics of the analytical protocol.

The results for both laboratories are presented in figure 4 where it can be seen that the basic composition has not changed, but a significant shift in the moisture content has occurred. To test the theory that adsorption was once again taking place, the cycle time for the analysis was deliberately shortened for the final analysis. The RGA operator was instructed to use the established analytical protocol and operational parameters, such as acquiring sufficient pump down pressure, noise levels, etc. of the RGA as the decision factor for starting the analysis. Hence, the cycle time was reduced from 35 minutes to 9 minutes while all operational parameters/requirements were still in effect. It can be readily seen that this change in cycle time significantly affects the amount of moisture being detected, as a shift from an average value of 260 ppm (with a range of 68 ppm) to the single point value of 337 ppm is observed (increasing the range to 109 ppm) In other words, excluding the final data point, statistical analysis of the data set yields a 260 ppm average value but with the outlier (337 ppm) of ~30%. The overall shift of this data set from that originally generated on the smaller SSC by ORS is also very significant from a chemist's point of view – but arguably acceptable from a manufacturing engineer's point of view. This deviation (as well as the other differences in the data) is believed to be caused by the moisture (and the other gases) calibration technique and is discussed below.

FIGURE 4 - RGA ANALYSIS OF SMALL SSC

		ORS DATA			**HONYWELL DATA**				
		test #1	test #2	test # 3	Run 1	Run 2	Run 3	Run 4	Run 5
INLET PRESSURE	Torr (mm)	137	137	134	0.601	0.601	0.601	0.601	0.601
NITROGEN	%v	89.1	89.1	89.1	90.11	90.11	90.09	90.09	90.09
OXYGEN	ppm	867	814	824	85	99	97	96	86
ARGON	ppm	236	248	247	276	289	289	288	282
CO2	%v	1.02	1.02	1.01	0.636	0.633	0.635	0.636	0.635
WATER	**ppm**	**103**	**104**	**119**	**226**	**294**	**278**	**255**	**337**
HYDROGEN	ppm	1042	1016	1029	714	624	620	663	666
HELIUM	%v	9.66	9.62	9.62	9.11	9.11	9.13	9.13	9.13

The smaller SSC was subsequently shipped back to ORS for a final analysis to again determine if there was any change in the gas composition, figure 5. The data strongly suggests that adsorption of moisture is taking place and a more suitable set of "optimum operational parameters" should be developed for this instrument to minimize this phenomenon. This problem, adsorption, may be minimized by modifying the operational parameters and minimizing the amount of surface area within the Honeywell RGA unit that the sample gas can make contact with. The former is easier to address, but was substantiated by Maxine Pennington, Technology Leader at the Honeywell facility, in her comments on this analysis. Specifically, they feel that the effects of moisture adsorption in the analyses of the SSC would have been reduced if the Honeywell operator were given the opportunity to use their analytical protocol in conjunction with that developed for the SSC. [Point well taken] Overall, adsorption is a very complex phenomenon with many contributing factors that in themselves have a confounding variance. One can suggest that all surfaces be kept at a minimum of 115^{o} C for a start, but it would be prudent to have a study performed in an attempt to discover what other obstacles/problems would be introduced by the additional thermal input.

FIGURE 5 – SECOND ANALYSIS OF SMALL SSC – ORS DATA

		test A	test B	test C
INLET PRESSURE	torr (mm)	127	127	125
NITROGEN	%v	89.7	89.6	89.6
OXYGEN	ppmv	756	774	778
ARGON	ppmv	246	245	252
CO2	ppmv	1.05	1.05	1.05
MOISTURE	ppmv	215	180	185
HYDROGEN	ppmv	932	907	894
HELIUM	%v	9.07	9.13	9.09

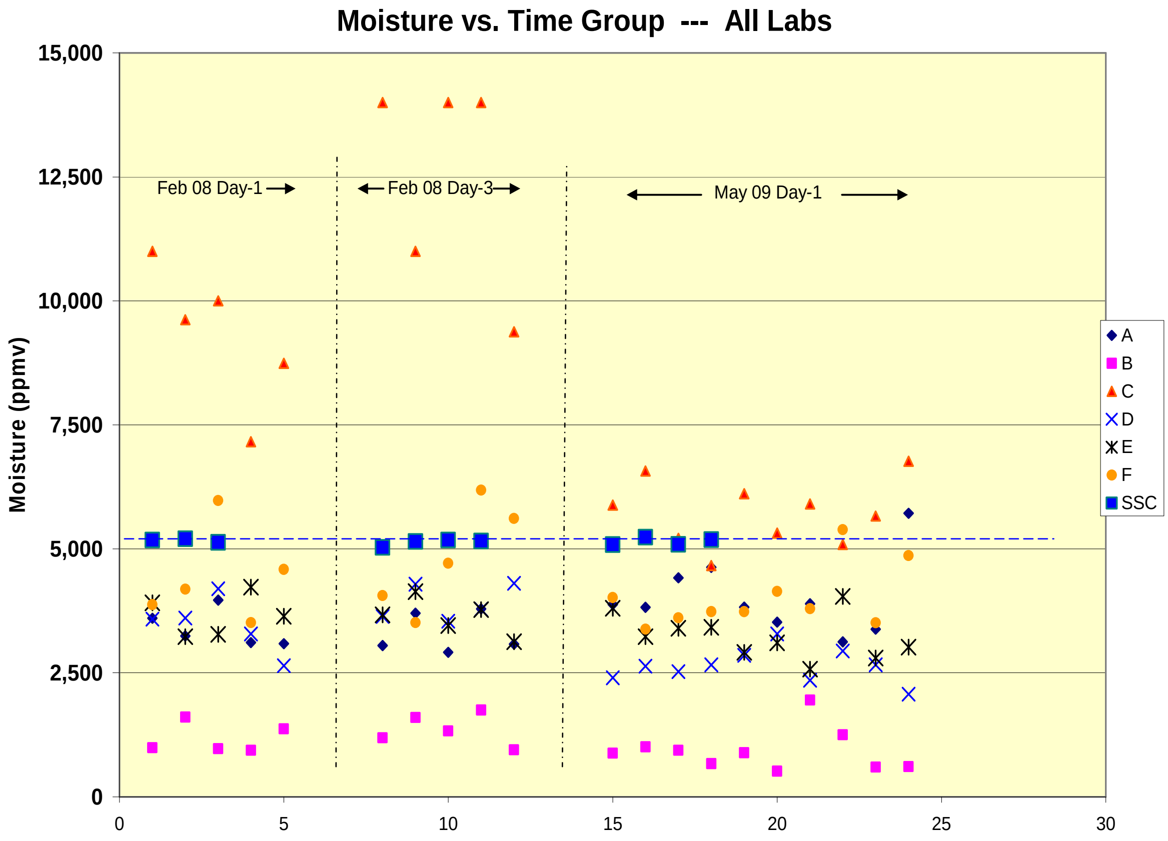
As noted in figure 4, the set of Honeywell RGA data from the small SSC showed much higher moisture content. The concern over this data is that it was off by a factor of 2X from what the previous ORS RGA unit had originally reported. Since it is not known what the absolute moisture content was, one cannot debate the data. The second set of ORS RGA data, figure 5, i.e. after return to ORS, showed a much better comparison differing by ~0.5X. However, it was finally realized that the published analytical protocol (Mil Std 883, Test Method 1018) for this analysis requires a <u>single point</u> calibration around 4000 ppm and from there <u>extrapolation</u> is utilized to generate data "at the bottom end" of the curve. Apparently, this is standard practice, but it does not provide an analytical chemist with any level of comfort, as it is the norm in chemistry to have at least three calibration points to cover the range of the data. Furthermore, as discussed earlier, the present means of calibration is with a NIST traceable dew pointer (a chilled mirror hygrocomputer). The dew point hygrometer, like any other piece of analytical equipment, will have a known precision. Apparently, one could utilize such calibration equipment with a wide precision and hence place a bigger bias on the RGA data that is subsequently generated. Therefore it is proposed that only the best calibration equipment is used for RGA calibration and that it have the tightest precision limits possible.

This issue becomes extremely important when one considers that "1018 data" is being utilized for more than the moisture content analyses. Process and Manufacturing Engineers are attempting to use all of the RGA data to optimize their processes, characterize materials, provide reliability predictions, etc. As such, the RGA data must be as precise and accurate as possible. It has also been reported that a 1000 ppmv pass/fail criteria is becoming a requirement for some high reliability houses[4]

Additionally, one final study was performed to verify the significance of varying the time lapses <u>during</u> an RGA analysis. The data is presented in the attachment #7 for the second set of RGA on the small SSC - tests D, E & F. The results were dramatic – the moisture value dropped from an average of 193 ppmv to 56 ppmv with a range of 23 ppm when the cycle time was left to the discretion of the operator.

Lastly, the basics of this report were presented at the Minnowbrook Microelectronics Conference held Oct. 6-9, ’09. In a discussion of the most recent DSCC “correlation data” from the various RGA laboratories[5], it was decided to overlay the SSC data from the subzero storage test onto the graphic depicting the results of the labs. The result is presented in figure 6 where the SSC is the blue square data. It should be noted that there were differences in test variables between the two studies, however those differences are not considered to be significant and the net result is very obvious.

FIGURE 6: SSC PLOTTED OVER DSCC DATA



The wide variation in the data from any one lab is the result of differences in the make up of the individual “samples” - albeit “they were from the same lot” and the complications that the individual RGA unit has introduced, i.e. those variations in time, temperature, etc.

SUMMATION

This study has not really uncovered any new scientific breakthrough - instead it has reminded us that there are few, if any, “original ideas”. Variations in data have been occurring because we have been ignoring some of the basic principles of our science. Four basic concepts have been trampled upon:

1) To reduce the effects of moisture adsorption - use high temperatures, e.g. >110 C
2) To reduce the effects of moisture adsorption - reduce surface area
3) Calibration can only be improved by using tighter standards
4) Calibration should be done with at least three points that cover the range of the data

There are many other areas of concern in standardized protocol and calibration, but they lie outside the scope of this study. However, there, is one very important additional point that should be stressed: Software should be written such that it is NOT trying to interpret the noise in the background of an analysis.

REFERENCES:

1) Military Standard 883 – Military Standard Test Methods and Procedures for Microelectronics – Test Method 1018

2) NASA MSFC Contract # NNGO5CCC95P, Nov. 28, 2005

3) Schuessler, Rossiter, Lowry & Sierzant, “Single Sample Cylinder for RGA Correlation”, JMEP, Vol4, No.2, 2nd Qtr.2007 (ISSN 1551-4897)

4) Private communication, Richard Kullberg, Minnowbrook Microelectronics Conference, Oct 6, 2009.

5) Private Communication, Daniel Rossiter, Minnowbrook Microelectronics Conference, Oct.6, 2009

ACKNOWLEDGEMENTS

The author would like to express his appreciation for the support of the NASA Electronics Parts and Packaging (NEPP) program, as well as that provided by Oneida Research Services personnel and the personnel at the Honeywell FM&T National Nuclear Facility in Kansas City. Their technical support, guidance and patience have allowed this technology to take another step forward towards a goal of generating more precise and accurate RGA data.

ATTACHMENT 1

ONEIDA
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TEST REPORT
INTERNAL VAPOR ANALYSIS

PHILIP SCHUESSLER
SCHUESSLER CONSULTING
P.O. BOX 188
GREENVILLE, N 12083
UNITED STATES

ORS LOT NO : 179344-001
DATE TESTED : 9/9/2008
QUANTITY TESTED : 8
PACKAGE TYPE : SINGLE SOURCE CYLINDER
MFG CODE : STUDY

PO: Schuessler
Rel. No:

SAMPLE ID		DAY1-1	DAY1-2	DAY1-3	DAY1-4	DAY2-1	DAY2-2	DAY2-3	DAY2-4
INLET PRESSURE	torr	220	221	221	232	226	230	231	231
NITROGEN	%v	79.3	79.3	79.2	79.3	79.4	79.4	79.3	79.3
OXYGEN	%v	20.2	20.1	20.2	20.2	20.1	20.1	20.2	20.2
ARGON	ppmv	167	157	167	158	164	164	166	162
CO2	ppmv	182	169	179	167	186	153	155	177
MOISTURE	ppmv	4,521	5,061	5,070	4,856	4,518	4,435	4,657	4,634
HYDROGEN	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
METHANE	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
AMMONIA	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
HELIUM	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
FLUORO-CARBONS	ppmv	ND	ND	ND	ND	ND	ND	ND	ND

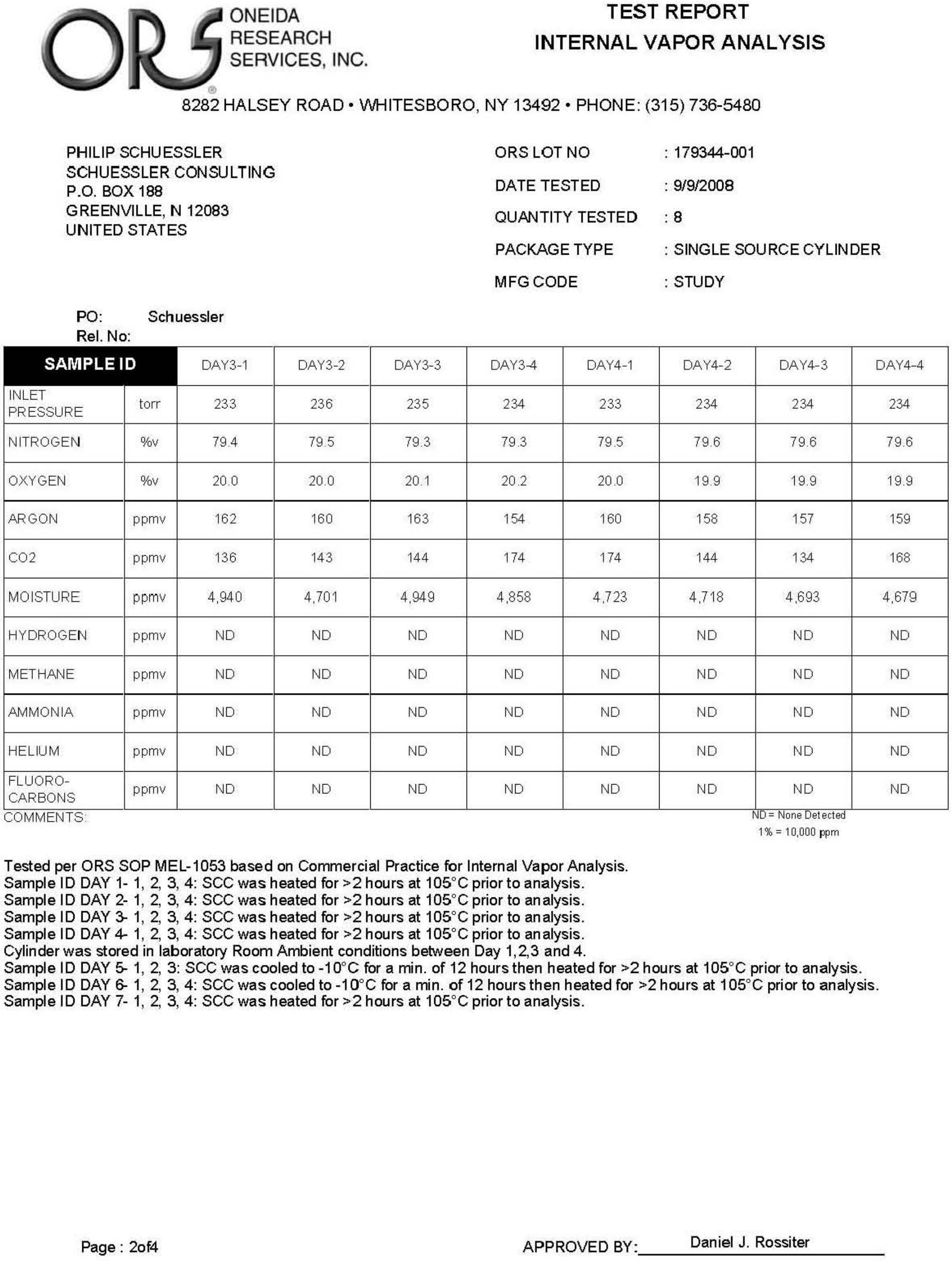
COMMENTS:

ND = None Detected
1% = 10,000 ppm

Tested per ORS SOP MEL-1053 based on Commercial Practice for Internal Vapor Analysis.
Sample ID DAY 1- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 2- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 3- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 4- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Cylinder was stored in laboratory Room Ambient conditions between Day 1,2,3 and 4.
Sample ID DAY 5- 1, 2, 3: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 6- 1, 2, 3, 4: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 7- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.

Page : 1of4

APPROVED BY: Daniel J. Rossiter



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SERVICES, INC.

TEST REPORT
INTERNAL VAPOR ANALYSIS

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PHILIP SCHUESSLER
SCHUESSLER CONSULTING
P.O. BOX 188
GREENVILLE, N 12083
UNITED STATES

ORS LOT NO : 179344-001
DATE TESTED : 9/9/2008
QUANTITY TESTED : 8
PACKAGE TYPE : SINGLE SOURCE CYLINDER
MFG CODE : STUDY

PO: Schuessler
Rel. No:

SAMPLE ID		DAY3-1	DAY3-2	DAY3-3	DAY3-4	DAY4-1	DAY4-2	DAY4-3	DAY4-4
INLET PRESSURE	torr	233	236	235	234	233	234	234	234
NITROGEN	%v	79.4	79.5	79.3	79.3	79.5	79.6	79.6	79.6
OXYGEN	%v	20.0	20.0	20.1	20.2	20.0	19.9	19.9	19.9
ARGON	ppmv	162	160	163	154	160	158	157	159
CO2	ppmv	136	143	144	174	174	144	134	168
MOISTURE	ppmv	4,940	4,701	4,949	4,858	4,723	4,718	4,693	4,679
HYDROGEN	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
METHANE	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
AMMONIA	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
HELIUM	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
FLUORO-CARBONS	ppmv	ND	ND	ND	ND	ND	ND	ND	ND

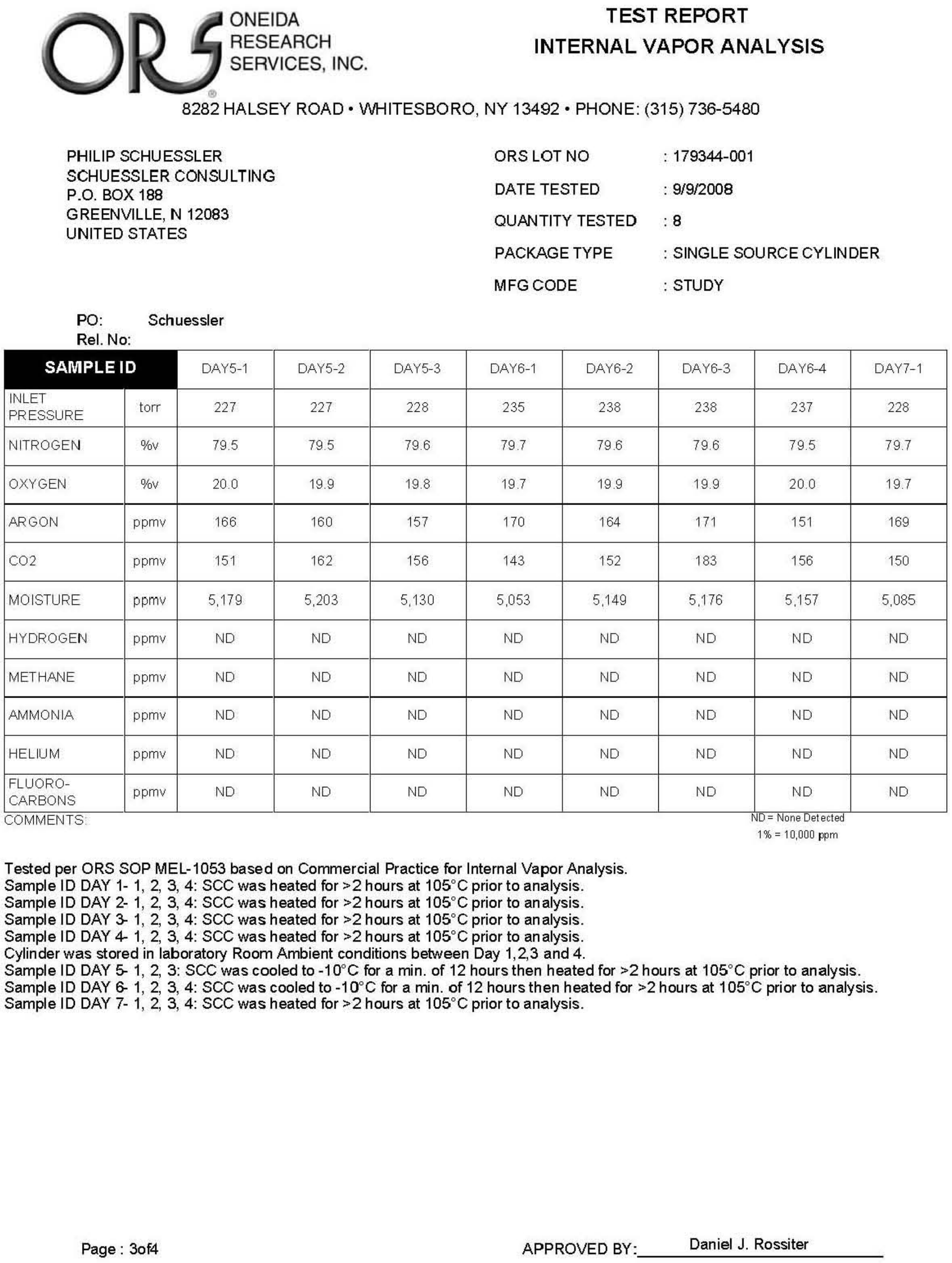
COMMENTS:

ND = None Detected
1% = 10,000 ppm

Tested per ORS SOP MEL-1053 based on Commercial Practice for Internal Vapor Analysis.
Sample ID DAY 1- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 2- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 3- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 4- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Cylinder was stored in laboratory Room Ambient conditions between Day 1,2,3 and 4.
Sample ID DAY 5- 1, 2, 3: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 6- 1, 2, 3, 4: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 7- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.

Page : 2of4

APPROVED BY: Daniel J. Rossiter



ORS ONEIDA RESEARCH SERVICES, INC.

TEST REPORT
INTERNAL VAPOR ANALYSIS

8282 HALSEY ROAD • WHITESBORO, NY 13492 • PHONE: (315) 736-5480

PHILIP SCHUESSLER
SCHUESSLER CONSULTING
P.O. BOX 188
GREENVILLE, N 12083
UNITED STATES

ORS LOT NO : 179344-001
DATE TESTED : 9/9/2008
QUANTITY TESTED : 8
PACKAGE TYPE : SINGLE SOURCE CYLINDER
MFG CODE : STUDY

PO: Schuessler
Rel. No:

SAMPLE ID		DAY5-1	DAY5-2	DAY5-3	DAY6-1	DAY6-2	DAY6-3	DAY6-4	DAY7-1
INLET PRESSURE	torr	227	227	228	235	238	238	237	228
NITROGEN	%v	79.5	79.5	79.6	79.7	79.6	79.6	79.5	79.7
OXYGEN	%v	20.0	19.9	19.8	19.7	19.9	19.9	20.0	19.7
ARGON	ppmv	166	160	157	170	164	171	151	169
CO2	ppmv	151	162	156	143	152	183	156	150
MOISTURE	ppmv	5,179	5,203	5,130	5,053	5,149	5,176	5,157	5,085
HYDROGEN	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
METHANE	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
AMMONIA	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
HELIUM	ppmv	ND	ND	ND	ND	ND	ND	ND	ND
FLUORO-CARBONS	ppmv	ND	ND	ND	ND	ND	ND	ND	ND

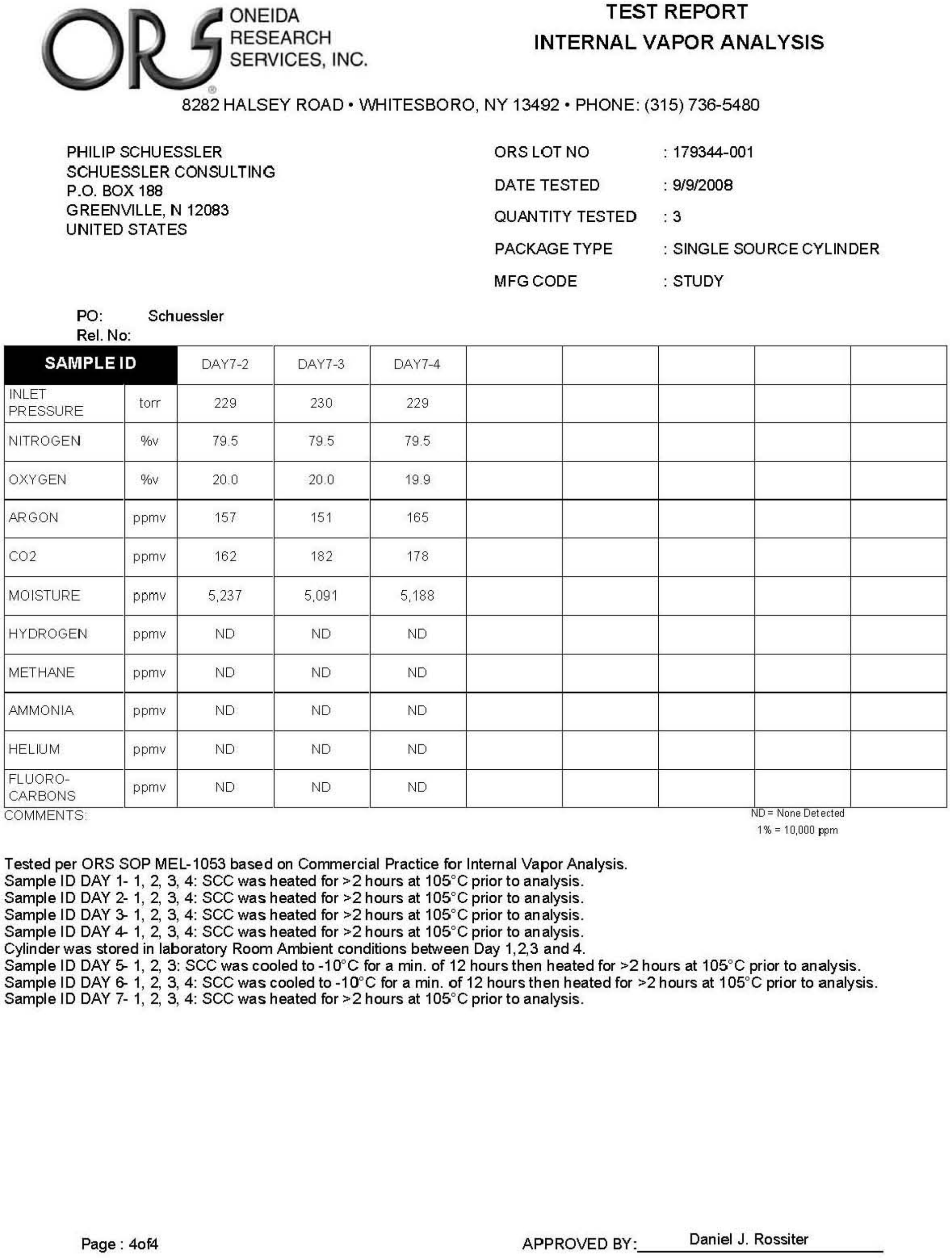
COMMENTS:

ND = None Detected
1% = 10,000 ppm

Tested per ORS SOP MEL-1053 based on Commercial Practice for Internal Vapor Analysis.
Sample ID DAY 1- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 2- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 3- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 4- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Cylinder was stored in laboratory Room Ambient conditions between Day 1,2,3 and 4.
Sample ID DAY 5- 1, 2, 3: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 6- 1, 2, 3, 4: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 7- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.

Page : 3of4

APPROVED BY: Daniel J. Rossiter



ORS ONEIDA RESEARCH SERVICES, INC.
8282 HALSEY ROAD • WHITESBORO, NY 13492 • PHONE: (315) 736-5480

TEST REPORT
INTERNAL VAPOR ANALYSIS

PHILIP SCHUESSLER
SCHUESSLER CONSULTING
P.O. BOX 188
GREENVILLE, N 12083
UNITED STATES

ORS LOT NO : 179344-001
DATE TESTED : 9/9/2008
QUANTITY TESTED : 3
PACKAGE TYPE : SINGLE SOURCE CYLINDER
MFG CODE : STUDY

PO: Schuessler
Rel. No:

SAMPLE ID		DAY7-2	DAY7-3	DAY7-4					
INLET PRESSURE	torr	229	230	229					
NITROGEN	%v	79.5	79.5	79.5					
OXYGEN	%v	20.0	20.0	19.9					
ARGON	ppmv	157	151	165					
CO2	ppmv	162	182	178					
MOISTURE	ppmv	5,237	5,091	5,188					
HYDROGEN	ppmv	ND	ND	ND					
METHANE	ppmv	ND	ND	ND					
AMMONIA	ppmv	ND	ND	ND					
HELIUM	ppmv	ND	ND	ND					
FLUORO-CARBONS	ppmv	ND	ND	ND					

COMMENTS:

ND = None Detected
1% = 10,000 ppm

Tested per ORS SOP MEL-1053 based on Commercial Practice for Internal Vapor Analysis.
Sample ID DAY 1- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 2- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 3- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 4- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.
Cylinder was stored in laboratory Room Ambient conditions between Day 1,2,3 and 4.
Sample ID DAY 5- 1, 2, 3: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 6- 1, 2, 3, 4: SCC was cooled to -10°C for a min. of 12 hours then heated for >2 hours at 105°C prior to analysis.
Sample ID DAY 7- 1, 2, 3, 4: SCC was heated for >2 hours at 105°C prior to analysis.

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APPROVED BY: Daniel J. Rossiter

ATTACHMENT 2: HONEYWELL SSC DATA

Honeywell FM&T 5/20/2009
Kansas City, MO
Analytical Science Laboratory

Volume % (1%=10,000 ppm)

	Zero Air Moisture Std			Four separate Analyses of Static Moisture Std			
	0.1 cc	**0.01 cc**	**0.001 cc**	**Run 1**	**Run 2**	**Run 3**	**Run 4**
Gases Analyzed							
Hydrogen	0	0	0	0	0	0	0
Helium	0.0004	0	0.0001	0.001	0.0011	0.0011	0.0011
Methane	0.0004	0.0004	0.0008	0.0004	0.0004	0.0004	0.0004
Water	0.3799	0.3776	0.4123	**0.2531**	**0.3076**	**0.3107**	**0.3381**
Neon	0.0006	0.0004	0.0004	0	0.0001	0.0001	0
Nitrogen	78.709	79.5035	79.0588	81.1257	81.0392	80.9911	80.8761
Carbon Monoxide	0.0004	0.0003	0.0003	0.0005	0.0004	0.0003	0.0004
Oxygen	20.0299	20.108	20.5168	18.5982	18.6307	18.6759	18.7635
Argon	0.0044	0.0046	0.0051	0.0157	0.0155	0.0155	0.0156
Carbon Dioxide	0.0047	0.0045	0.004	0.005	0.0046	0.0045	0.0045
Total Hydrocarbon	0.0003	0.0004	0.0006	0.0003	0.0003	0.0003	0.0003
Fluorocarbon	0.0001	0.0001	0.0002	0.0001	0.0001	0.0001	0.0001
Ammonia	0.0003	0	0.0001	0	0	0	0

ATTACHMENT 3

Variables of Internal Gas Analysis

Oneida Research Services, Inc.

Mass Spectrometer

Type (Quadrupole, Magnetic Sector, Time of Flight): Quadrupole
Manufacturer: Pfeiffer Vacuum
Mass Range Capability: 1-512
Scan Speed Capability: 0.5ms – 60sec / AMU
Dynamic Range of Peak Signal Detection: 10^{-12} - 10^{-5}
Type of Ion Source: Cross-Beam
Filament Type: Tungsten filament
Detector Type: Secondary Electron Multiplier
Is the Detector On-Axial or Off-Axial: Off-Axial

Inlet

Inlet Type (Batch or Single Sample): Single Sample Type
Transfer Volume Size/Length: 2.5cc volume / 6” Transfer
How do you control the quantity of gas in the ion source?: Orifice
How is the inlet heated, and to what temperature?: Direct heater elements to Stainless Steel Inlet.
What size samples can be tested? 0.01 to infinite volume.
Transfer efficiency – S.F. as a function of volume: ~1 to 3 SF from large to small volumes.

Tuning the Mass Spectrometer

Describe how spectra are collected, processed and stored.: Spectra is measured in “SCAN” move (1/64/mass steps). Scan data can be converted to “PEAK: mode with firmware to measure centroid height of each mass. Resolution is 1 amu. All raw data from tests are saved (background and sample test).
What procedures do you use to tune and control the mass spectrometer for best performance?: Balance raw peak height to width ratio at 10% of peak height through entire mass range. Ratio should be ~ 0.75 AMU. System is tuned daily.
How do you monitor the pressure in the ion source?: Cold Cathod Gauge 100 torr to 2.4X10-9 torr range.
How do you determine if the spectrometer produces linear response to gas pressure? An Air decay test can be run to demonstrate linearity over pressure range. Testing pressure should not exceed 10-4 torr or filament linearity is lost.

How do you calibrate the mass scale? Toluene or Xenon trace gas is used to calibrate the mass scale. Know mass assignment of the gas are used to align mass scale through the range. Mass Scale is checked daily.

How stable is the calibration of the mass scale? Quadupole systems are very stable. Unless a filament change is performed, the mass scale generally does not shift.

Standard Test Procedure

Describe how you acquire data

Scan speed: Typically 20ms/AMU

Mass range: 1-150 AMU

Timing sequence: Background spectra is acquired, sample is punctured, the pressure rise is detected and either the sample spectra is automatically started to acquire data or a manual method can be used. All raw data that is acquired is saved in the data file.

What Mode (Scan , Stair, Peak, Mid, etc) is used to acquire data? PEAK-FIR,

Describe the raw data (the fundamental peak signal that is measured) Raw spectra with 1/64 steps per AMU is smoothed using a FIR filter (Pfeiffer firmare). The smoothed raw peak is then used to find the centroid of the curve. That centroid value is used as the peak intensity value.

Describe signal processing that occurs on the raw data (if any): FIR filter.

Describe the data that is saved: Pressure data, mass spectra data, calibration data, time stamps, quantitated results, sample information and system parameters.

How is it archived? All raw data is saved to a secure server. Data is backed up daily and archived on a yearly basis.

How are primary peaks measured? All peaks from 2-150 amu are measured in the standard method. 512 capable.

How is moisture measured? Moisture is measure by detecting mass 18, 17 and 16 and analyzing their respective ratios with specified error bands to arrive at an intensity value (or mass 18).

Describe the data that is used for quantification: Primary peaks are used for quantitation once respective ratios are subtracted from the raw data.

Describe in detail how the test data is quantified: In general, the intensity of a primary peak (eg. 18) is divided by the total of all primary peaks found. (eg. Moisture -18 intensity/ total of all primary peak intensities)

Describe the rationale for deciding the presence and identity of trace level gases: Most substances require as a minimum, 1 secondary peak to identify the substance. In some cases, 2 or more. Identifying only the primary peak as evidence of a substance in the spectra can be mis-leading.

Calibration Procedure

How frequently is the spectrometer turned for mass scale and peak shape? Daily

How do you inject calibration gases vs. how do you inject the test sample gas? Calibration gas is injected at the same location as a sample.

What is your NIST traceable moisture standard? (mfg/model/age and how many) Edgetech, Vigilent model, 2 years old.

What is the accuracy for the NIST traceable reference? +/- 0.1%

How is it calibrated and by whom? Manufacturer (Edgetech).

How often is it calibrated? Yearly

How is it maintained? Cleaned per mfg. requirements weekly.

How do you calibrate for moisture? Using Dew Point Hygrometer.

How do you calibrate for primary gases (N_2, O_2, Ar, CO_2, He, H_2) and how often? Quarterly (method 1018 requires yearly).

Where is Calibrator located? At the start of the transfer passage – same location where a sample would be pierced and inlet into system.

How to insure consistent calibration (manual vs. automated)? Inlet of calibration gas is done by puncturing a 0.010” thick kovar lid. The lid is sealed to the inlet at the piercing location of a sample. The calibrators used are manual for large volumes (1cc and up) , and automated for volumes less than 1cc.

Location of Calibrators vs. test sample? Same location as samples for testing.

Cleanliness of mirror? Cleaned weekly.

ATTACHMENT 4

Variables of Internal Vapor Analysis
Participant: Charles Cook, NNSA's Kansas City Plant, Managed and Operated by Honeywell Federal Manufacturing and Technologies May 2009

Mass Spectrometer

Type (Quadrupole, Magnetic Sector, Time of Flight): Quadrupole
Manufacturer: Balzers QMG 422 incorporated into Pernicka
Mass Range Capability: 1-512
Scan Speed Capability: 0.5ms to 60 sec/u
Dynamic Range of Peak Signal Detection: < 10^{-15} millibar (seven decades)
Type of Ion Source: Cross Beam (axial)
Filament Type: Re (rhenium)
Detector Type: SEM
Is the Detector On-Axial or Off-Axial: 90 degrees off axis

Inlet (larger volumes that are attached in front of instrument)

Inlet Type (Batch or Single Sample): Single sample
Transfer Volume Size/Length: varies according to plumbing required to connect to external valve
How do you control the quantity of gas in the ion source? Controlled volume inlet through calibration valve to mass spec sample volume then vernier isolation valve meters to the source
How is the inlet heated, and to what temperature? Tape wrap with digital readout thermocouple to mass spectrometer
What size samples can be tested? 1cc to
Transfer efficiency – S.F. as a function of volume: varies

Inlet (smaller samples that can be press fitted to the puncture device by heated platform)

Inlet Type (Batch or Single Sample): Single sample
Transfer Volume Size/Length: less than 0.5cc
How do you control the quantity of gas in the ion source? Sample fully expanded into internal mass spec sample volume then isolation valve to the ion source controlled by vernier setting
How is the inlet heated, and to what temperature? Heated stage 103 C +/- 5
What size samples can be tested? 0.001cc to about 3cc
Transfer efficiency – S.F. as a function of volume: varies

Tuning the Mass Spectrometer

Describe how spectra are collected, processed and stored.: mass spectrometer scans from mass 1 to 150 (generally) spectra are stored as selected ion signals indicating gases of interest. Five scans of background are stored before sample introduction. Twenty sample scans are collected after sample introduction, the average background at each mass is subtracted from each sample spectrum and quantification is done based on response factors achieved in calibration of the instrument. One full spectrum is displayed after the final sample scan for visual qualitative analysis and printed, if desired. The full spectrum data is not stored

What procedures do you use to tune and control the mass spectrometer for best performance? The mass offset voltage, optimizing signal has not been adjusted on our mass spec since installation. It would be adjusted if there was any signal degradation.

How do you monitor the pressure in the ion source?: Digital readout from baratron

How do you determine if the spectrometer produces linear response to gas pressure? Calibration with response factors are done at four volumes (pressures). The calibration response factor for the total pressure of sample interest is automatically chosen as the basis of quantification. Some gases are not linear over a dynamic range of their own partial pressure

How do you calibrate the mass scale?: observe mass assignments and compare to calibration gas content. No adjustments have been required since installation (PFTBA used over the full mass range) but would be if any source work (maintenance, filament replacement, etc) were done

How stable is the calibration of the mass scale? Very stable. Has not been adjusted since installation checkout

Test Procedure

Describe how you acquire data

Scan speed: 20 data scans at 4 sec per scan

Mass range: 1-150

Timing sequence: scan 5 x background then 20 x sample

Describe the raw data (the fundamental peak signal that is measured) intensities at mass/charge ratio of 2,4,12, 14, 15, 16, 18, 19, 20, 28, 30, 32, 34, 40, 43, 44, 55, 57, 69, 84

Describe signal processing that occurs on the raw data (if any)

Describe the data that is saved: signals at each mass of selected interest for both background and sample

How is it archived? PC in excel format

How are primary peaks measured? Standard base peaks are used for all gases

How is moisture measured? Mass 18

Describe the data that is used for quantification : corrections are made for interfering components as well as isotopic abundances of interfering fragment peaks

Describe in detail how the test data is quantified each scan has the average background subtracted at each mass, then the resulting signal is adjusted for mass spectral interferences (isotopic and fragmentation). Then the calibration response factor is applied to translate the signal intensity to the corresponding gas concentration

Describe the rationale for deciding the presence and identity of trace level gases Signals less than x times the background signal are eliminated. A final full mass spectrum is displayed and interpreted for molecular identification of hydrocarbons or other impurities

Calibration Procedure

How frequently is the spectrometer tuned for mass scale and peak shape?
Visual of peak shapes and assignments only upon operator detecting

How do you inject calibration gases vs. how do you inject the test sample gas?
Calibration gases are inlet by flowing through a multi-volume calibrator valve, instantaneously trapping a selected quantity and then toggling a "burst" into the mass spec's expansion volume to simulate a package burst. Sample packages are tested by single puncture directly into the mass spectrometer's sample expansion volume

What is your NIST traceable moisture standard? (mfg/model/age and how many)
In Metrology department:
<u>On the Mass spec:</u>
chilled mirror Eastern/model sys/2003
General purpose humidity generator
MCM moisture monitor checked every 3 months to NIST traceable Metrology standard

What is the accuracy of the NIST traceable reference?
Metrology:

How is it calibrated and by whom? Metrology engineers perform calibration on MCM moisture monitor

How often is it calibrated? 3 months

How is it maintained? Return to manufacturer, if can't be calibrated

How do you calibrate for moisture? Flow standard nitrogen through the moisture generator, adjust pressures to achieve nominal 4,000 ppm +/- 400ppm on the chilled mirror and MCM moisture monitor. This is done daily when samples are going to be analyzed

How do you calibrate for primary gases (N_2, O_2, Ar, CO_2, He, H_2) and how often?
Commercial certified standards with certificates of analysis. Three levels of traces gases in nitrogen/helium. Three levels of trace gases are used: 8 ppm, 50 ppm, and 300 ppm hydrogen, neon, carbon dioxide, argon, carbon monoxide, and oxygen. Helium is present at 5-6% in the nitrogen matrix. Choose closest gas matrix mix and use as the daily performance calibration

Where is Calibrator located? Direct attachment to mass spec source's inlet expansion volume

How to insure consistent calibration (manual vs. automated)? Manual toggle for the burst of gas, all mass spectral acquisition is automatic

Location of Calibrators vs. test sample? Less than 1 inch, at right angles to each other, at 100C

Cleanliness of mirror? Clean, Double checked with correlation to in-line MCM moisture monitor

ATTACHMENT 5

ONEIDA
RESEARCH
SERVICES, INC.
8282 HALSEY ROAD • WHITESBORO, NY 13492 • PHONE: (315) 736-5480

TEST REPORT
INTERNAL VAPOR ANALYSIS

PHILIP SCHUESSLER
SCHUESSLER CONSULTING
P.O. BOX 188
12083 GREENVILLE NY
UNITED STATES

ORS LOT NO : 182157-001
DATE TESTED : 7/2/2009
QUANTITY TESTED : 3
PACKAGE TYPE : CYLINDER
MFG. CODE :

PO: Schuessler
Rel. No:

SAMPLE ID		TEST#1	TEST#2	TEST#3					
INLET PRESSURE	torr	137	137	134					
NITROGEN	%v	89.1	89.1	89.1					
OXYGEN	ppmv	867	814	824					
ARGON	ppmv	236	248	247					
CO2	%v	1.02	1.02	1.01					
MOISTURE	ppmv	103	104	119					
HYDROGEN	ppmv	1,042	1,016	1,029					
METHANE	ppmv	ND	ND	ND					
AMMONIA	ppmv	ND	ND	ND					
HELIUM	%v	9.66	9.62	9.62					
FLUORO-CARBONS	ppmv	ND	ND	ND					

COMMENTS:

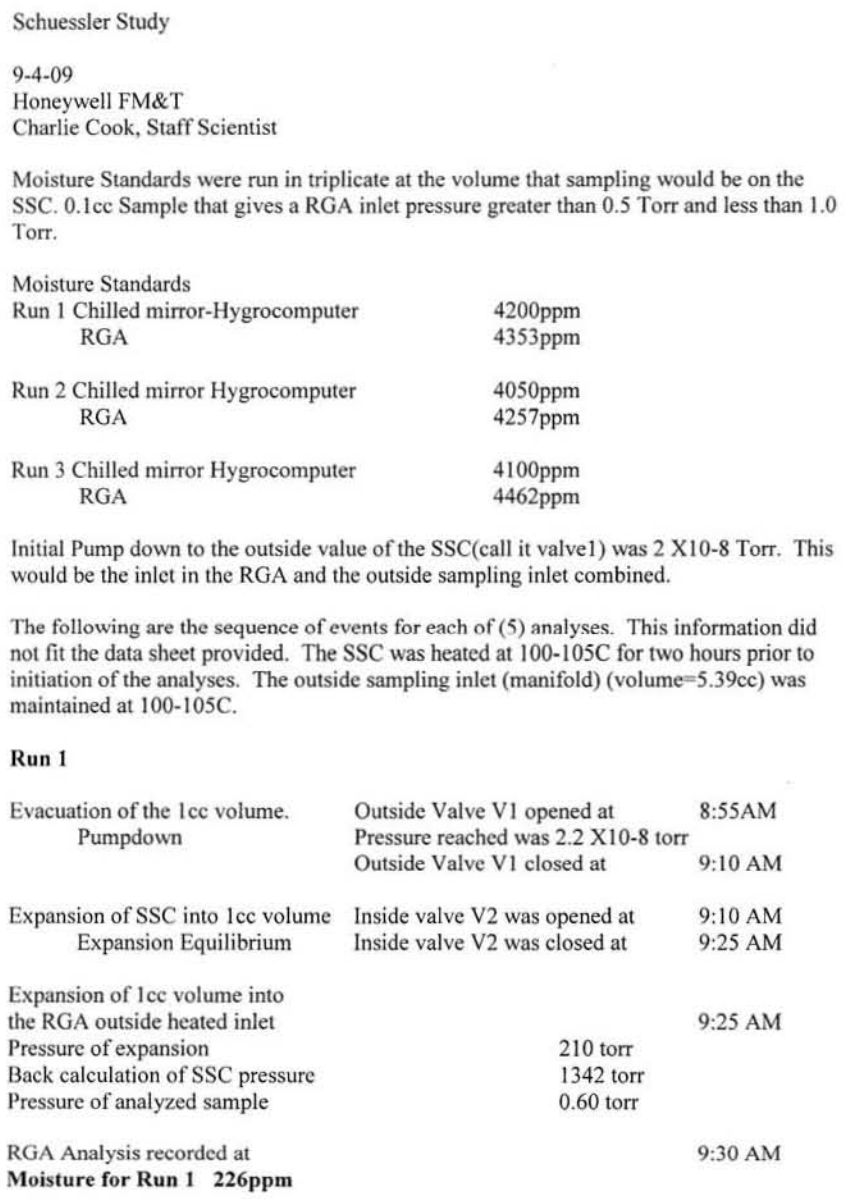
ND = None Detected
1% = 10,000 ppm

Tested per ORS SOP MEL-1053 based on Commercial Practice for Internal Vapor Analysis.
Cylinder was prebaked at 100C for 2 hours and tested at 100C.

Page : 1of1

APPROVED BY: Daniel J. Rossiter

ATTACHMENT 6



Schuessler Study

9-4-09
Honeywell FM&T
Charlie Cook, Staff Scientist

Moisture Standards were run in triplicate at the volume that sampling would be on the SSC. 0.1cc Sample that gives a RGA inlet pressure greater than 0.5 Torr and less than 1.0 Torr.

Moisture Standards	
Run 1 Chilled mirror-Hygrocomputer	4200ppm
RGA	4353ppm
Run 2 Chilled mirror Hygrocomputer	4050ppm
RGA	4257ppm
Run 3 Chilled mirror Hygrocomputer	4100ppm
RGA	4462ppm

Initial Pump down to the outside value of the SSC(call it valve1) was 2 X10-8 Torr. This would be the inlet in the RGA and the outside sampling inlet combined.

The following are the sequence of events for each of (5) analyses. This information did not fit the data sheet provided. The SSC was heated at 100-105C for two hours prior to initiation of the analyses. The outside sampling inlet (manifold) (volume=5.39cc) was maintained at 100-105C.

Run 1

Evacuation of the 1cc volume.	Outside Valve V1 opened at	8:55AM
Pumpdown	Pressure reached was 2.2 X10-8 torr	
	Outside Valve V1 closed at	9:10 AM
Expansion of SSC into 1cc volume	Inside valve V2 was opened at	9:10 AM
Expansion Equilibrium	Inside valve V2 was closed at	9:25 AM
Expansion of 1cc volume into the RGA outside heated inlet		9:25 AM
Pressure of expansion	210 torr	
Back calculation of SSC pressure	1342 torr	
Pressure of analyzed sample	0.60 torr	
RGA Analysis recorded at		9:30 AM

Moisture for Run 1 226ppm

Run 2

Evacuation of the 1cc volume	Outside Valve V1 opened at	9:30 AM
Pumpdown	Pressure reached was 2.5X10-8 torr	
	Outside Valve V1 closed at	9:45 AM
Expansion of SSC into 1cc volume	Inside Valve V2 opened at	9:45 AM
Expansion Equilibrium	Inside Valve V2 closed at	10:00 AM
Expansion of 1cc volume into the RGA outside inlet		10:01 AM
Pressure of expansion	206 torr	
Back calculation of SSC pressure	1316 torr	
Pressure of analyzed sample	0.53 torr	
RGA analysis recorded at		10:03 AM

Moisture for Run 2 294 ppm

Run 3

Evacuation of the 1cc volume	Outside Valve V1 opened at	10:05 AM
Pumpdown	Pressure reached was 2.2 X 10-8 torr	
	Outside valve V1 closed at	10:20 AM
Expansion of SSC into 1cc volume	Inside valve V2 opened at	10:20 AM
Expansion Equilibrium	Inside Valve V2 closed at	10:35 AM
Expansion of 1 cc volume into the RGA outside inlet		10:35 AM
Pressure of expansion	201 torr	
Back calculation of SSC pressure	1284 torr	
Pressure of analyzed sample	0.56 torr	
RGA analysis recorded at		10:37 AM

Moisture for Run 3 278ppm

Run 4

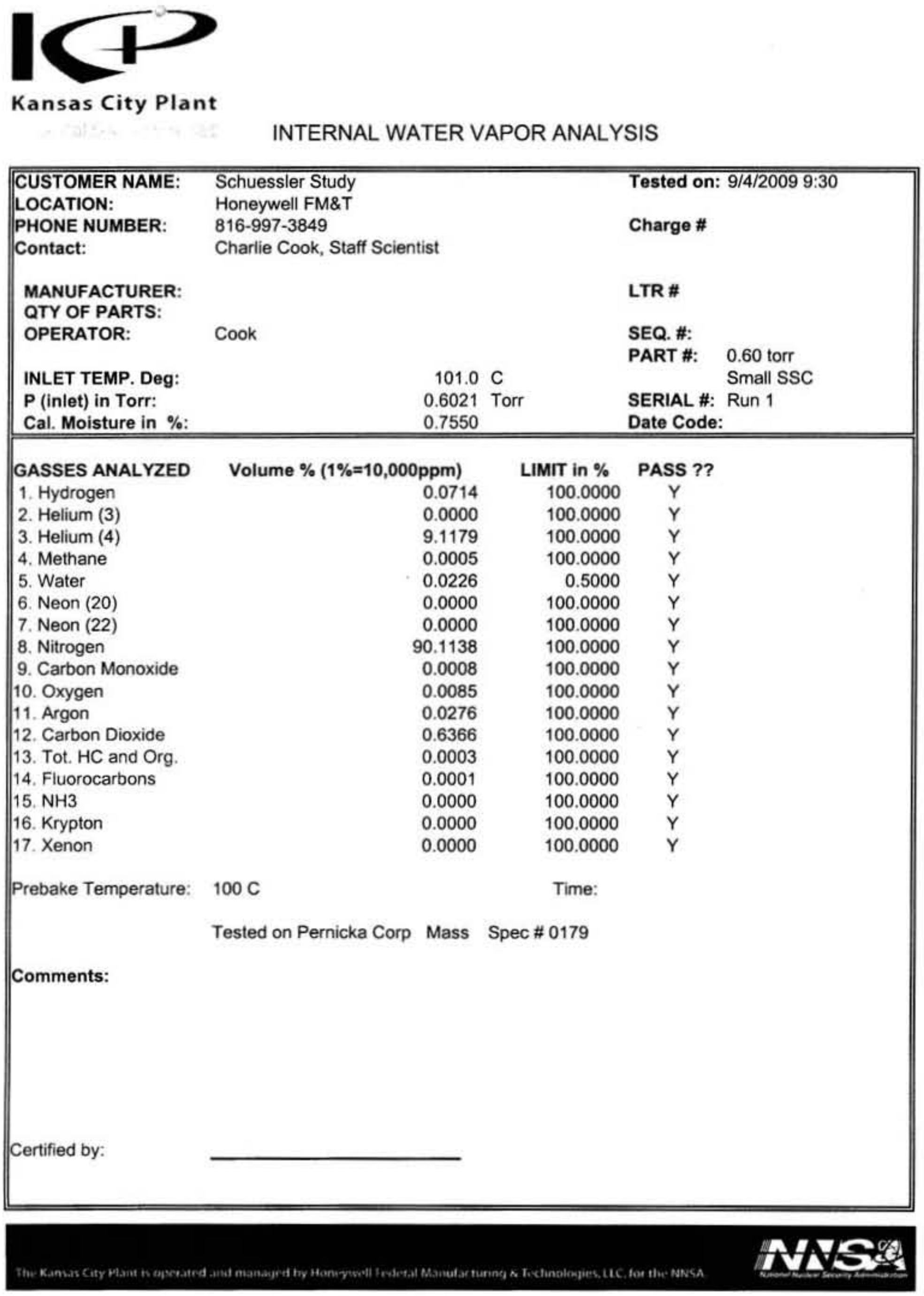
Evacuation of the 1cc volume	Outside Valve V1 opened at		10:40AM
Pumpdown	Pressure reached was 1.9 X10-8 torr		
	Outside Valve V1 closed at		10:55 AM
Expansion of SSC into 1cc volume	Inside Valve V2 was opened at		10:55 AM
Expansion Equilibrium	Inside Valve V2 was closed at		11:10 AM
Expansion of 1 cc volume into the RGA outside heated inlet			11:10 AM
Pressure of expansion		197 torr	
Back calculation of SSC pressure		1259 torr	
Pressure of analyzed sample		0.56 torr	
RGA analysis recorded at			11:12 AM

Moisture for Run 4 255 ppm

Run 5

Inlet and manifold pumpdown to	2.2 X10-8 torr		11:15 AM
Outside Valve V1 opened at			11:15 AM
1cc volume pumped down to 2.2 X10-8 torr			11:16 AM
Outside Valve V1 closed at			11:16 AM
Inside Valve V2 opened at			11:17 AM
Inside Valve V2 closed at			11:18 AM
1cc volume expanded into RGA outside manifold			11:18AM
Pressure of expansion		194 torr	
Back calculation of SSC pressure		1239 torr	
Pressure of analyzed sample		0.60 torr	
RGA analysis recorded at			11:24 AM
Total time of Run 5 analysis		9.0 minutes	

Moisture for Run 5 337 ppm



Kansas City Plant

INTERNAL WATER VAPOR ANALYSIS

CUSTOMER NAME: Schuessler Study

Tested on: 9/4/2009 9:30

LOCATION: Honeywell FM&T

PHONE NUMBER: 816-997-3849

Charge #

Contact: Charlie Cook, Staff Scientist

MANUFACTURER:

LTR #

QTY OF PARTS:

OPERATOR: Cook

SEQ. #:

PART #: 0.60 torr Small SSC

INLET TEMP. Deg: 101.0 C

P (inlet) in Torr: 0.6021 Torr

SERIAL #: Run 1

Cal. Moisture in %: 0.7550

Date Code:

GASSES ANALYZED	Volume % (1%=10,000ppm)	LIMIT in %	PASS ??
1. Hydrogen	0.0714	100.0000	Y
2. Helium (3)	0.0000	100.0000	Y
3. Helium (4)	9.1179	100.0000	Y
4. Methane	0.0005	100.0000	Y
5. Water	0.0226	0.5000	Y
6. Neon (20)	0.0000	100.0000	Y
7. Neon (22)	0.0000	100.0000	Y
8. Nitrogen	90.1138	100.0000	Y
9. Carbon Monoxide	0.0008	100.0000	Y
10. Oxygen	0.0085	100.0000	Y
11. Argon	0.0276	100.0000	Y
12. Carbon Dioxide	0.6366	100.0000	Y
13. Tot. HC and Org.	0.0003	100.0000	Y
14. Fluorocarbons	0.0001	100.0000	Y
15. NH3	0.0000	100.0000	Y
16. Krypton	0.0000	100.0000	Y
17. Xenon	0.0000	100.0000	Y

Prebake Temperature: 100 C

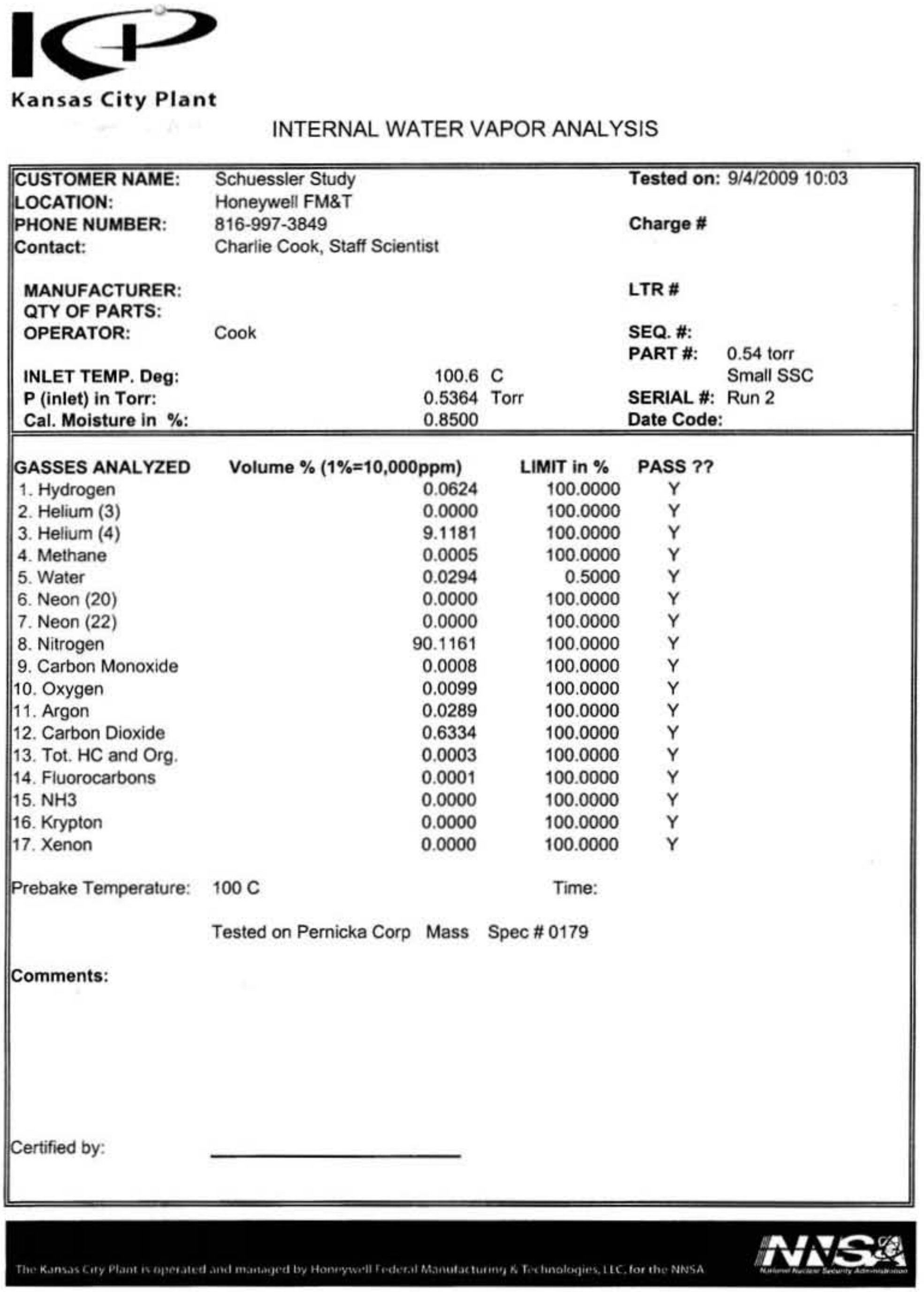
Time:

Tested on Pernicka Corp Mass Spec # 0179

Comments:

Certified by: ____________________

The Kansas City Plant is operated and managed by Honeywell Federal Manufacturing & Technologies, LLC, for the NNSA



Kansas City Plant

INTERNAL WATER VAPOR ANALYSIS

CUSTOMER NAME:	Schuessler Study		**Tested on:**	9/4/2009 10:03
LOCATION:	Honeywell FM&T			
PHONE NUMBER:	816-997-3849		**Charge #**	
Contact:	Charlie Cook, Staff Scientist			
MANUFACTURER:			**LTR #**	
QTY OF PARTS:				
OPERATOR:	Cook		**SEQ. #:**	
			PART #:	0.54 torr
INLET TEMP. Deg:		100.6 C		Small SSC
P (inlet) in Torr:		0.5364 Torr	**SERIAL #:**	Run 2
Cal. Moisture in %:		0.8500	**Date Code:**	

GASSES ANALYZED	**Volume % (1%=10,000ppm)**	**LIMIT in %**	**PASS ??**
1. Hydrogen	0.0624	100.0000	Y
2. Helium (3)	0.0000	100.0000	Y
3. Helium (4)	9.1181	100.0000	Y
4. Methane	0.0005	100.0000	Y
5. Water	0.0294	0.5000	Y
6. Neon (20)	0.0000	100.0000	Y
7. Neon (22)	0.0000	100.0000	Y
8. Nitrogen	90.1161	100.0000	Y
9. Carbon Monoxide	0.0008	100.0000	Y
10. Oxygen	0.0099	100.0000	Y
11. Argon	0.0289	100.0000	Y
12. Carbon Dioxide	0.6334	100.0000	Y
13. Tot. HC and Org.	0.0003	100.0000	Y
14. Fluorocarbons	0.0001	100.0000	Y
15. NH3	0.0000	100.0000	Y
16. Krypton	0.0000	100.0000	Y
17. Xenon	0.0000	100.0000	Y

Prebake Temperature: 100 C Time:

Tested on Pernicka Corp Mass Spec # 0179

Comments:

Certified by: ____________________

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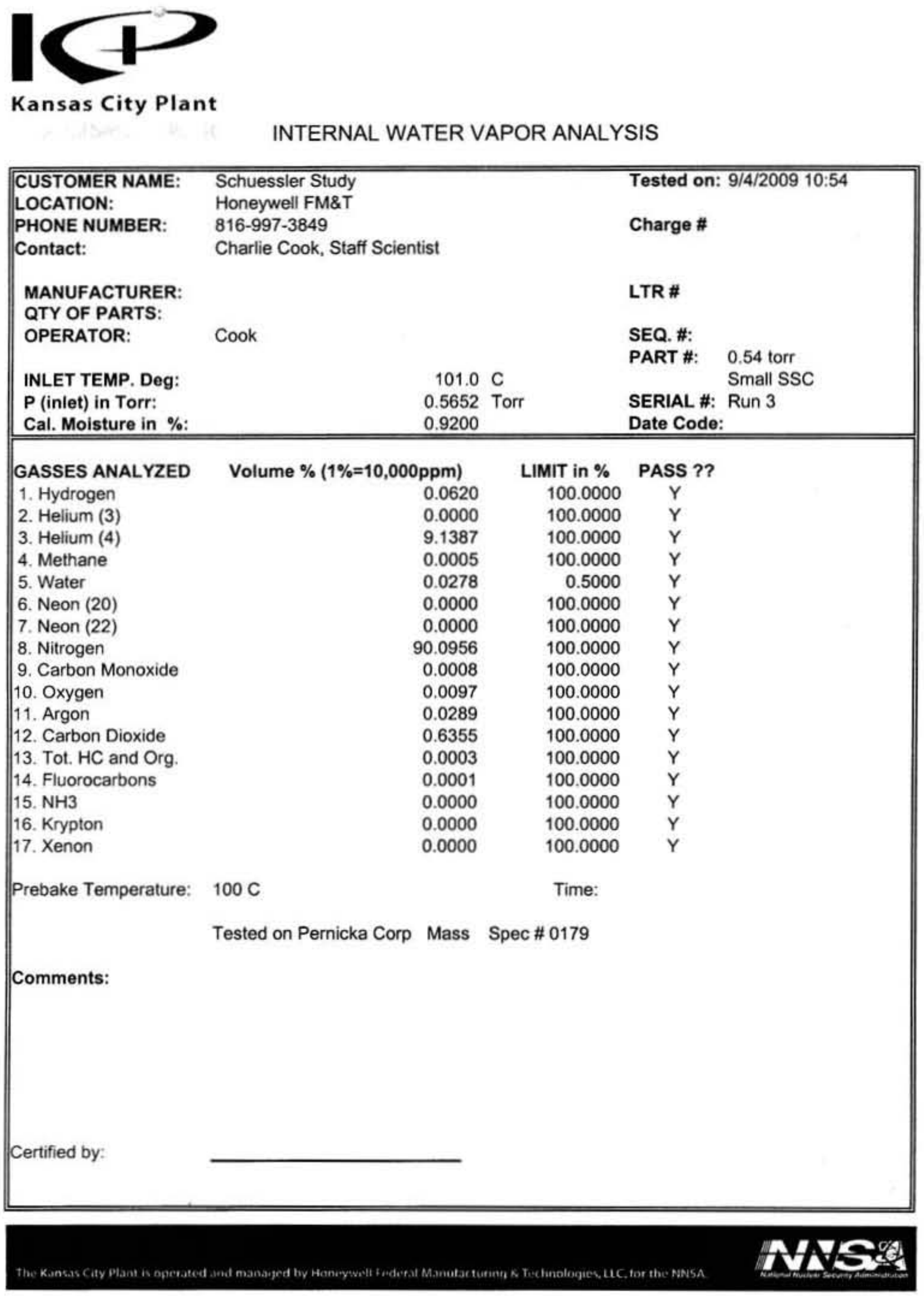

Kansas City Plant

INTERNAL WATER VAPOR ANALYSIS

CUSTOMER NAME: Schuessler Study
LOCATION: Honeywell FM&T
PHONE NUMBER: 816-997-3849
Contact: Charlie Cook, Staff Scientist

Tested on: 9/4/2009 10:54
Charge #

MANUFACTURER:
QTY OF PARTS:
OPERATOR: Cook

INLET TEMP. Deg: 101.0 C
P (inlet) in Torr: 0.5652 Torr
Cal. Moisture in %: 0.9200

LTR #
SEQ. #:
PART #: 0.54 torr Small SSC
SERIAL #: Run 3
Date Code:

GASSES ANALYZED	**Volume % (1%=10,000ppm)**	**LIMIT in %**	**PASS ??**
1. Hydrogen	0.0620	100.0000	Y
2. Helium (3)	0.0000	100.0000	Y
3. Helium (4)	9.1387	100.0000	Y
4. Methane	0.0005	100.0000	Y
5. Water	0.0278	0.5000	Y
6. Neon (20)	0.0000	100.0000	Y
7. Neon (22)	0.0000	100.0000	Y
8. Nitrogen	90.0956	100.0000	Y
9. Carbon Monoxide	0.0008	100.0000	Y
10. Oxygen	0.0097	100.0000	Y
11. Argon	0.0289	100.0000	Y
12. Carbon Dioxide	0.6355	100.0000	Y
13. Tot. HC and Org.	0.0003	100.0000	Y
14. Fluorocarbons	0.0001	100.0000	Y
15. NH3	0.0000	100.0000	Y
16. Krypton	0.0000	100.0000	Y
17. Xenon	0.0000	100.0000	Y

Prebake Temperature: 100 C Time:

Tested on Pernicka Corp Mass Spec # 0179

Comments:

Certified by: ____________________

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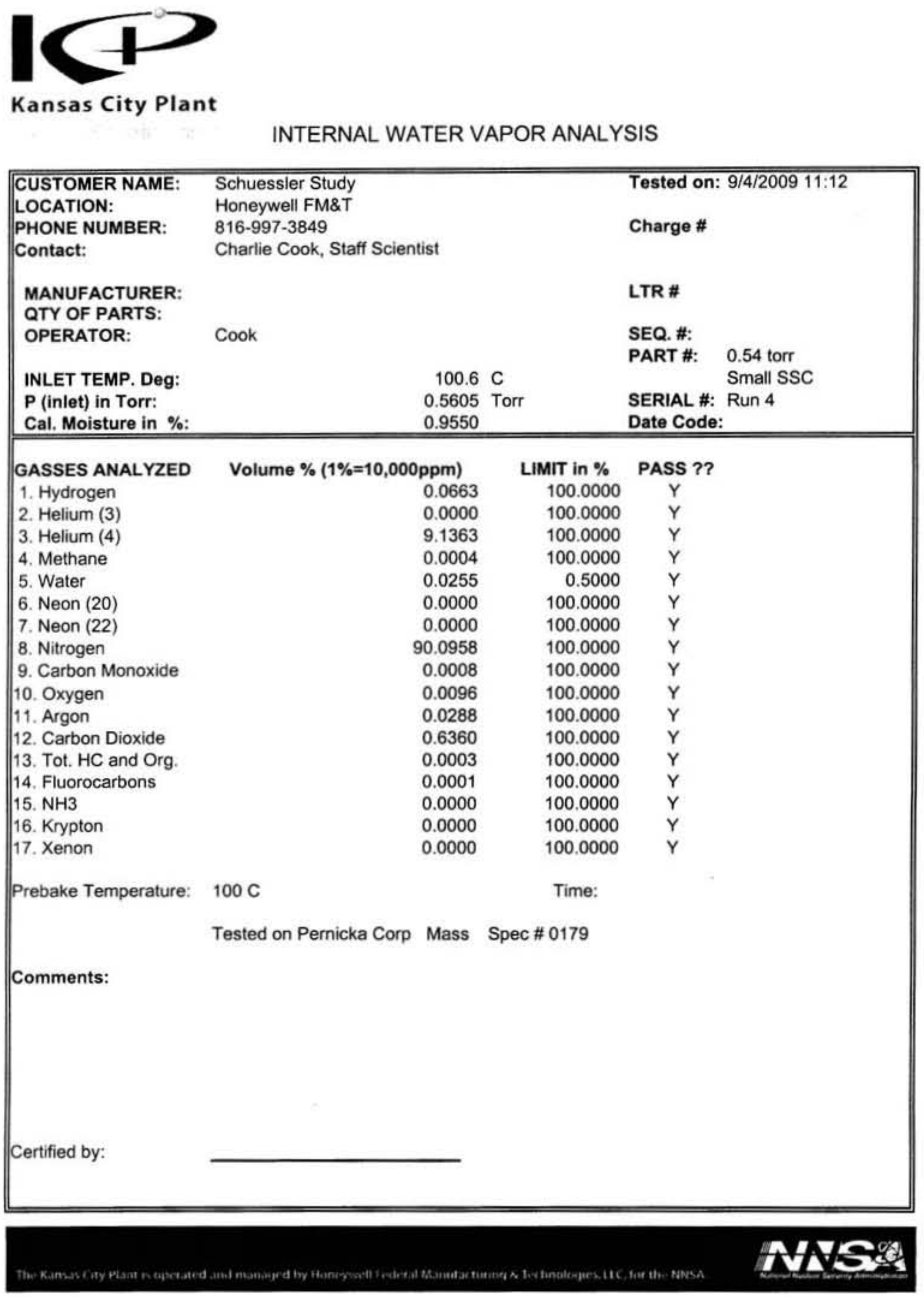

Kansas City Plant

INTERNAL WATER VAPOR ANALYSIS

CUSTOMER NAME:	Schuessler Study	**Tested on:**	9/4/2009 11:12
LOCATION:	Honeywell FM&T		
PHONE NUMBER:	816-997-3849	**Charge #**	
Contact:	Charlie Cook, Staff Scientist		
MANUFACTURER:		**LTR #**	
QTY OF PARTS:			
OPERATOR:	Cook	**SEQ. #:**	
		PART #:	0.54 torr Small SSC
INLET TEMP. Deg:	100.6 C		
P (inlet) in Torr:	0.5605 Torr	**SERIAL #:**	Run 4
Cal. Moisture in %:	0.9550	**Date Code:**	

GASSES ANALYZED	**Volume % (1%=10,000ppm)**	**LIMIT in %**	**PASS ??**
1. Hydrogen	0.0663	100.0000	Y
2. Helium (3)	0.0000	100.0000	Y
3. Helium (4)	9.1363	100.0000	Y
4. Methane	0.0004	100.0000	Y
5. Water	0.0255	0.5000	Y
6. Neon (20)	0.0000	100.0000	Y
7. Neon (22)	0.0000	100.0000	Y
8. Nitrogen	90.0958	100.0000	Y
9. Carbon Monoxide	0.0008	100.0000	Y
10. Oxygen	0.0096	100.0000	Y
11. Argon	0.0288	100.0000	Y
12. Carbon Dioxide	0.6360	100.0000	Y
13. Tot. HC and Org.	0.0003	100.0000	Y
14. Fluorocarbons	0.0001	100.0000	Y
15. NH3	0.0000	100.0000	Y
16. Krypton	0.0000	100.0000	Y
17. Xenon	0.0000	100.0000	Y

Prebake Temperature: 100 C Time:

Tested on Pernicka Corp Mass Spec # 0179

Comments:

Certified by: ____________________

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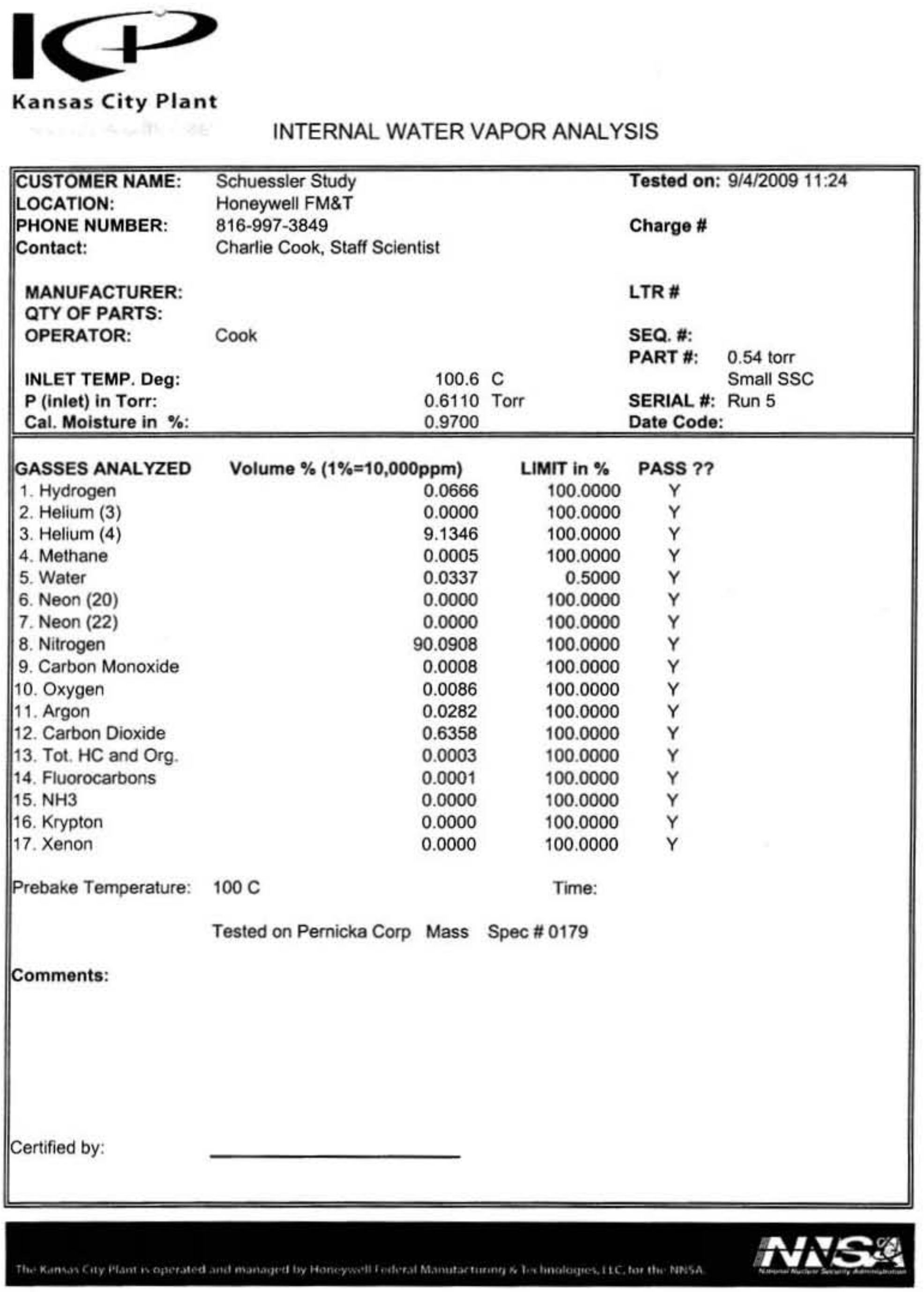

Kansas City Plant

INTERNAL WATER VAPOR ANALYSIS

CUSTOMER NAME:	Schuessler Study	**Tested on:**	9/4/2009 11:24
LOCATION:	Honeywell FM&T		
PHONE NUMBER:	816-997-3849	**Charge #**	
Contact:	Charlie Cook, Staff Scientist		
MANUFACTURER:		**LTR #**	
QTY OF PARTS:			
OPERATOR:	Cook	**SEQ. #:**	
		PART #:	0.54 torr
INLET TEMP. Deg:	100.6 C		Small SSC
P (inlet) in Torr:	0.6110 Torr	**SERIAL #:**	Run 5
Cal. Moisture in %:	0.9700	**Date Code:**	

GASSES ANALYZED	**Volume % (1%=10,000ppm)**	**LIMIT in %**	**PASS ??**
1. Hydrogen	0.0666	100.0000	Y
2. Helium (3)	0.0000	100.0000	Y
3. Helium (4)	9.1346	100.0000	Y
4. Methane	0.0005	100.0000	Y
5. Water	0.0337	0.5000	Y
6. Neon (20)	0.0000	100.0000	Y
7. Neon (22)	0.0000	100.0000	Y
8. Nitrogen	90.0908	100.0000	Y
9. Carbon Monoxide	0.0008	100.0000	Y
10. Oxygen	0.0086	100.0000	Y
11. Argon	0.0282	100.0000	Y
12. Carbon Dioxide	0.6358	100.0000	Y
13. Tot. HC and Org.	0.0003	100.0000	Y
14. Fluorocarbons	0.0001	100.0000	Y
15. NH3	0.0000	100.0000	Y
16. Krypton	0.0000	100.0000	Y
17. Xenon	0.0000	100.0000	Y

Prebake Temperature: 100 C Time:

Tested on Pernicka Corp Mass Spec # 0179

Comments:

Certified by: ____________________

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ATTACHMENT 7

ONEIDA
RESEARCH
SERVICES, INC.

TEST REPORT
INTERNAL VAPOR ANALYSIS

8282 HALSEY ROAD • WHITESBORO, NY 13492 • PHONE: (315) 736-5480

PHILIP SCHUESSLER
SCHUESSLER CONSULTING
P.O. BOX 188
12083 GREENVILLE NY
UNITED STATES

ORS JOB NO. : 183249-001
DATE TESTED : 10/1/2009
QUANTITY TESTED : 6
PACKAGE TYPE : CYLINDERS
MFG. CODE : SMALL SSC

PO: Schuessler
Rel. No:

SAMPLE ID		A	B	C	D	E	F		
INLET PRESSURE	torr	127	127	125	122	120	119		
NITROGEN	%v	89.7	89.6	89.6	89.4	89.4	89.4		
OXYGEN	ppmv	756	774	778	765	745	724		
ARGON	ppmv	246	245	252	245	237	240		
CO2	%v	1.05	1.05	1.05	1.04	1.05	1.05		
MOISTURE	ppmv	215	180	185	71	48	50		
HYDROGEN	ppmv	932	907	894	917	927	906		
METHANE	ppmv	ND	ND	ND	ND	ND	ND		
AMMONIA	ppmv	ND	ND	ND	ND	ND	ND		
HELIUM	%v	9.07	9.13	9.09	9.32	9.37	9.34		
FLUORO-CARBONS	ppmv	ND	ND	ND	ND	ND	ND		

COMMENTS:

ND = None Detected
1% = 10,000 ppm

Tested per ORS SOP MEL-1053 based on Commercial Practice for Internal Vapor Analysis.
Sample ID A, B, C: Tested according to study schedule (30 minutes between tests).
Sample ID D, E, F: Tested at operators descrestion (~10 minutes between tests).

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APPROVED BY: Daniel J. Rossiter