July 21, 2004

DSCC INTERNAL WATER-VAPOR CONTENT CORRELATION PROGRAM

ROUND 3 RESULTS (1.0 cc)

DSCC and six laboratories with DSCC Laboratory Suitability to test method 1018 of MIL-STD-883 and/or MIL-STD-750 analyzed correlation samples as part of a continuation of the third round of DSCC's Internal Water-Vapor Content Correlation Program. The six suitable laboratories are Atlantic Analytical Laboratories, Mead Testing, Oneida Research Services (NY), Oneida Recherche Services (France), Pernicka Corporation, and Seal Laboratories. Other than DSCC, each Laboratory's identification has been made anonymous by substituting the name with a letter. All laboratories including DSCC tested four 1.0 cubic centimeter correlation samples. The results are summarized in this report.

It is our understanding that standards for measuring water on RGA equipment do not exist. Although we are able to increase or decrease the amount of moisture in the correlation samples to varying degrees, we are unable to state specifically how much water is in the samples or how much should be reported. This is in part due to the fact that samples are assembled in a glove box at approximately 22^{o}C and are tested at 100^{o}C in accordance with test method 1018. However, if the correlation samples are precise, then a valid correlation study can still be performed. In the following pages, we will discuss why we believe these correlation samples are precise and that this is a valid correlation study.

Results

1.0 Cubic Centimeter Correlation Samples

	Water	N_2	Ar	H_2	He	O_2	CO_2	FC	Avg Water	Std Dev	Diff (1)
DSCC	1585	99.8	112	0	0	114	4	0	1839	193	-1458
	2054	99.8	110	0	0	122	6	0			
	1864	99.7	135	0	0	564	6	0			
	1852	99.8	110	0	0	135	5	0			
Lab A	1645	99.8	96	34	ND	597	11	ND	1882	280	-1415
	1708	99.7	119	81	ND	674	15	ND			
	2268	99.7	143	59	ND	599	21	ND			
	1905	99.7	114	49	ND	630	14	ND			
Lab B	2995	99.7	122	133	ND	158	40	ND	2898	228	-399
	3161	99.6	126	105	ND	223	39	ND			
	2639	99.7	120	155	ND	102	32	ND			
	2795	99.7	122	173	ND	139	32	ND			
Lab C	5160	99.4	133	181	ND	<100	ND	ND	4607	383	+1310
	4478	99.5	108	190	ND	114	ND	ND			
	4510	99.5	104	130	ND	116	ND	ND			
	4278	99.5	121	243	ND	<100	ND	ND			
Lab D	3610	99.6	180	ND	ND	ND	ND	ND	3990	280	+693
	4210	99.5	70	ND	ND	ND	ND	ND			
	3950	99.5	250	ND	ND	ND	ND	ND			
	4190	99.5	100	ND	ND	ND	ND	ND			
Lab E	3589	99.6	123	53	ND	118	ND	ND	4090	448	+793
	4207	99.6	159	28	ND	127	13	ND			
	3919	99.6	143	31	ND	113	21	ND			
	4646	99.5	118	38	ND	111	26	ND			
Lab F	3724	99.6	113	117	ND	117	<100	ND	3777	42	+480
	3784	99.6	125	133	ND	118	<100	ND			
	3774	99.6	120	131	ND	117	<100	ND			
	3827	99.6	115	146	ND	128	<100	ND			
	3297	Average of all parts									

(1) difference of each laboratory's average water from 3297 (average water of all of the parts)

ND = none detected or less than 10 ppm. For some laboratories, results below 10 ppm have been truncated and are shown as ND to help protect anonymity.

FC = fluorocarbons

All results are in parts per million (ppm) except for nitrogen which is expressed as a percentage. 10,000 ppm = 1%.

Correlation Samples

Although we may not be able to report the accuracy (i.e., an exact value of water) for these correlations samples, one of our goals was to provide precise (i.e., repeatable) samples in order to have a valid correlation study. One quick method of measuring precision is to look at the standard deviation relative to the mean. In this study, the standard deviation from each laboratory divided by its mean value is typically about 10% and sometimes considerably less. Given the nature of this test, a ratio of approximately 10% is generally considered more than adequate. To be more objective, we also performed statistical tests. Three variance checks (Cochran's C test, Bartlett's test, and Hartely's test) were run to check that the variances of all laboratories were essentially equal. At the 5.0% significance level (alpha = 0.05), these tests all concluded that there was not a statistically significant difference (SSD) among the standard deviations. Therefore, the correlation samples are precise and performing further analysis of the means (i.e., a correlation study) is acceptable.

Analysis of Results

When looking only at the average water, the results show a fairly wide range. DSCC and Lab A were fairly close (1839 and 1882, respectively), whereas Lab C was highest at 4607 ppm. Labs D, E and F were fairly close to each other in the 4000 ppm range. Lab B (2898 ppm) was close to the overall average of 3297 ppm.

Given the results, do the labs correlate? Or, are they statistically significantly different (SSD)? To answer these questions, various statistical tests were run, including the Student's t Distribution, Fisher's Least Significant Difference, and Tukey's Honestly Significant Difference. All of these tests statistically compare the means of the various labs to each other by forming pairs of laboratories. Since there are seven laboratories to compare to each other, there are 21 lab pairs. For example, we compared DSCC to Lab A, then DSCC to Lab B, DSCC to Lab C … all the way to comparing Lab D to Lab F, and finally Lab E to Lab F. Ideally, we would like all 21 lab pairs to correlate. That is, there should be no statistically significant difference for all 21 lab pairs in order to say that all of the laboratories correlate with each other. The below data will show that relatively few lab pairs had no statistically significant difference. That is, the laboratories generally are not correlating with each other for the 1.0 cc volume.

For brevity purposes, we will only show the results of Tukey's Honestly Significant Difference (HSD) test, although all of the tests gave nearly similar results. Using 95% confidence level, we got the following.

SSD: 15 lab pairs

Not SSD: 6 lab pairs (DSCC – Lab A, Lab C – Lab D, Lab C – Lab E, Lab D – Lab E, Lab D – Lab F, Lab E- Lab F)

SSD = statistically significantly different

With a few exceptions, the results are generally more favorable when looking at gases other than water. The laboratories all reported similar numbers for nitrogen, argon, helium, carbon dioxide, and fluorocarbons. For oxygen, most labs reported similar numbers, except for Lab A, who averaged about 600 ppm and Lab D, who reported ND in all four samples. Hydrogen was a bit more widespread with DSCC and Lab D reporting zero and ND, respectively in all four samples; Lab A and Lab E both averaging close to 50 ppm; and Labs B, C, and E averaging close to 150 ppm.

Comparison of 0.1 cc and 1.0 cc

The following table displays how each laboratory performed relative to the overall average for water for 0.1 cc (reported previously) and 1.0 cc. This table can be used to determine, for example, if a particular laboratory reports higher or lower water results than its peers. For example, DSCC's results were below the average for both volumes, whereas Lab D's and Lab E's results were above the average for both volumes. Labs B and F were fairly close to the average for both volumes. Of course, this table was generated based on just two sets of data. More correlation rounds are necessary before we can establish trends.

Volume	Overall average	DSCC	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F
0.1 cc	4639	Below (-823)	Above (+294)	Above (+94)	Below (-774)	Above (+318)	Above (+544)	Below (-17)
1.0 cc	3297	Below (-1458)	Below (-1415)	Below (-399)	Above (+1310)	Above (+693)	Above (+793)	Above (+480)

DSCC Actions

Because the correlation results for 1.0 cc were less than desirable, another correlation study on this volume will be repeated soon. Also, since the 0.1 cc results are based on only three samples per laboratory, another round of the 0.1 cc samples will be repeated as soon as possible. Additional studies will help to validate this data, identify trends, and may help to identify where improvements can be made. However, at the time of writing, DSCC's RGA machine is not operating due to calibration of equipment and a necessary repair. When the machine is operating again, we will continue to build and test correlation samples. We would also like to ask the laboratories and industry experts to provide us with any ideas that they may have to improve correlation. In addition, we intend to have the correlation samples re-passivated. Doing so should provide us with 35 reliable samples in each volume.

DSCC would like to thank each laboratory for participating in this study. They performed the tests, sent us the results, and returned the correlations samples in a very timely manner. We understand and appreciate that, due to the size and configuration of

these 1.0 cc samples, at least one laboratory needed to take extra time to test these. We look forward to continuing to actively work with the laboratories to improve correlation.

If you have any questions, please contact Mr. Jim Eschmeyer at (614) 692-0591 or james.eschmeyer@dla.mil or Mike Yates at (614) 692-9887 or michael.yates@dla.mil.