

April 5, 2004
REVISED May 27, 2004
See page 4 for updates

DSCC INTERNAL WATER-VAPOR CONTENT CORRELATION PROGRAM

ROUND 3 RESULTS (0.1 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 for the third round of DSCC's Internal Water-Vapor Content Correlation Program. The six laboratories were 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.

The correlation samples were sealed in a glove box containing nitrogen and moisture. The glove box is connected to DSCC's RGA equipment and also to a second hydrocomputer. This enables the moisture content in the glove box to be easily checked. Also, the exact concentrations of the gases can be obtained by running a sample through the mass spectrometer. Using our mass spectrometer, we analyzed the concentrations of gases in the glove box twice at the time of sealing the correlation samples. The moisture averaged about 3000 ppm; oxygen averaged 110 ppm; and argon averaged 338 ppm. After prebaking DSCC's correlation sample for 16 to 24 hours at 100°C in accordance with test method 1018, we obtained 3816 ppm moisture, 90 ppm oxygen, and 334 ppm argon. From our experience in working with these correlation samples, we commonly see that the moisture from test units is several hundred parts per million more than what we had in the glove box at the time of sealing. We believe that the reason for this difference is due to the temperature differences between the glove box and the test units. The glove box is kept at room temperature and units are tested at 100°C, i.e., almost 80°C difference. For this reason, we cannot give an exact value of what moisture should be reported. On the other hand, oxygen and argon concentrations are typically very steady when comparing results from the glove box to the test units. Because the results from our correlation sample were what we anticipated, we sent the remaining samples to the other laboratories.

Each laboratory tested three 0.1 cubic centimeter correlation samples and DSCC tested one 0.1 cc sample. The reason why we used just 19 correlation samples for this study rather than the 35 that we have used in the past is due to a problem that we have experienced with some of our correlation samples. Possibly due to a cleaning process that we had been using, we believe that the passivation layer has been degraded on many of our samples. Of the samples whose passivation layer is believed to be degraded, we have seen higher moisture readings than expected.

Laboratory E reported one correlation sample containing 12,900 ppm of water. At this point, we do not know if this was a laboratory anomaly or if there was a problem with the correlation sample. We will reseal this particular sample and analyze it on our RGA equipment. If this part's passivation layer has degraded, then we will also get a higher than anticipated moisture result. We will update this report after we analyze this particular sample. At this point, we will provide calculations with and without this particular sample.

Results

0.1 Cubic Centimeter Correlation Samples

	<u>Water</u>	<u>N₂</u>	<u>Ar</u>	<u>H₂</u>	<u>He</u>	<u>O₂</u>	<u>CO₂</u>	<u>FC</u>	<u>Avg Water</u>	<u>Std Dev</u>	<u>Diff (1)</u>
DSCC	3816	99.539	334	283	0	90	9	0	3816		823
Lab A	5060	99.3	221	203	ND	1159	20	ND	4933	128	294
	4935	99.4	255	194	33	850	32	ND			
	4804	99.4	301	228	ND	580	27	ND			
Lab B	4085	99.5	351	67	0	75	23	0	4733	755	94
	5562	99.4	351	56	0	61	23	0			
	4551	99.5	352	58	0	61	30	0			
Lab C	3677	99.6	345	229	ND	ND	<100	ND	3865	258	774
	3760	99.6	365	148	ND	<100	<100	ND			
	4159	99.5	355	197	ND	157	112	ND			
Lab D	4470	99.5	430	ND	ND	ND	40	ND	4957	480	318
	5430	99.4	410	ND	ND	ND	30	ND			
	4970	99.4	430	ND	ND	ND	30	ND			
Lab E	12900	98.6	342	204	0	40	47	0	7755	4460	3116
	4983	99.4	349	153	33	75	47	0	5183**	282**	544**
	5382	99.4	358	190	0	303	42	0			
Lab F	4897	99.4	327	279	ND	ND	<100	ND	4622	321	17
	4700	99.5	350	251	ND	ND	<100	ND			
	4269	99.5	346	239	ND	ND	<100	ND			
	4639**	Average of all parts without the 12,900 sample									
	5074	Average of all parts									

(1) absolute value of the difference of each laboratory's average water from **4639** (average water of all parts**)

ND = none detected

FC = fluorocarbons

** Results reported without the first sample from Lab E.

All results are in parts per million (ppm) except for nitrogen which is expressed as a percentage.

Analysis of Results

When looking only at the water, the results appear to be bimodal. DSCC and Lab C reported average moisture around 3800 ppm. All of the other laboratories' averages range from 4622 ppm to 5183** ppm. Concerning the other gases, the laboratories correlate fairly well, with a couple of exceptions. Laboratory B and especially Laboratory D reported less hydrogen than the other laboratories. Laboratories A and E both reported 33 ppm helium in just one part, whereas all other parts had zero or ND. The results for oxygen were the most widespread. Laboratory A reported significantly more oxygen than the other laboratories, whereas laboratories D and F did not detect any oxygen.

Although there were some differences among the laboratories, there were also areas of correlation. Considering the average moisture in all samples was 4639 ppm, every part** tested was within +/- 1000 ppm of 4639 ppm. Comparing the average moisture from each suitable laboratory to the overall average of 4639, the difference was fairly tight, ranging from 17 ppm to 774 ppm. All laboratories reported zero or none detected for fluorocarbons. Nitrogen and argon correlated fairly well. On the other hand, we realize that only three parts were tested at just one volume. More correlation studies are needed, at this volume and other volumes.

DSCC Actions

These results will be discussed individually with the laboratories, especially where differences were noted. We are continuing our work with the 1.0 cc and 0.01 cc volumes. We are presently evaluating the 12,900 ppm moisture sample from Lab E. We also feel that another round of 0.1 cc samples should be sent out soon to validate this study and to look for any trends. We also intend to have the correlation samples re-passivated. In doing so, we hope to obtain 35 samples that give us consistent results.

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 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.

May 27, 2004

Update Concerning Part From Lab E

Laboratory E reported one part with 12,900 ppm water. We felt that it was important to determine if this high reading was because of an anomalous correlation sample or because of an error from Lab E. Therefore, using several samples from the Round 3 correlation study plus the 12,900 ppm sample, we assembled a new batch of parts.

The first part of the experiment was for DSCC to test four “good” samples from this new batch in addition to the sample that had previously read 12,900 ppm. The results are listed below in ppm.

Samples tested by DSCC	<u>Water</u>	<u>O₂</u>	<u>Ar</u>
1	3574	77	166
2	2935	61	168
3	3230	60	166
4	3238	49	166
5 (Previously 12900 ppm from Lab E)	6041	70	168

The average water from the first four “good” parts was 3244 ppm. These parts also had a fairly tight standard deviation (261), giving us confidence that the batch was well-made. The fifth part (the part in question) was then tested and had 6041 ppm water, almost twice as much water as the average of the previous four. These results support the theory that this particular correlation sample’s passivation layer had degraded.

The second part of the experiment was to send four additional “good” samples from this same batch to Lab E. The results are listed below in ppm.

Samples tested by Lab E	<u>Water</u>	<u>O₂</u>	<u>Ar</u>
1	3052	135	166
2	3517	ND	159
3	3264	47	166
4	3900	18	120

The average water for Lab E was 3433 ppm, comparing favorably with DSCC’s results (average 3244 ppm). Also, Lab E had no outliers this time. Based on these results, it appears that the 12,900 ppm sample from Lab E was due to an anomaly with the sample itself and not laboratory error.