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Measurement Uncertainty of Silicone Oil Leakage Testing

W. E. Holland

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Allied-Signal Aerospace Company
Kansas City Division

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Technical Communications
Kansas City Division

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MASTER

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Prepared by W. E. Holland

An evaluation has been performed to determine the uncertainty of silicone tracer fluid leakage measurements for an environmental sensing device. The units are tested with an instrument which can measure silicone tracer fluid vapor by the gas chromatography method. An analysis has shown that the measurement uncertainty can be maintained at $\pm 20\%$ when the volumes used in the procedure are held to a given set of tolerances.

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SUMMARY

A mathematical simulation of the silicone tracer fluid leak testing procedures for an environmental sensing device showed that the leak rate criterion could be modeled by a simple equation involving five volumes that required control (certification). An uncertainty source evaluation was implemented to list and quantify random and systematic uncertainty sources for each volume. Where sufficient numbers of a type of volume were available, a standard deviation of the population was calculated to allow for setting a tolerance which would encompass most replacement items. A set of practical tolerances for the volumes was recommended, and the total uncertainty of the measurement process (using these tolerances) was calculated.

DISCUSSION

SCOPE AND PURPOSE

Metrology at Allied-Signal Inc., Kansas City Division (KCD) has been requested to evaluate the uncertainty involved in silicone oil leak testing.

Silicone oil is added to an environmental sensing device (ESD) as a damping material. It is necessary to test for leakage because leaking oil could contaminate other components. To speed the testing, low viscosity silicone oil (0.65 cs tracer fluid) is mixed with the damping material. The tracer fluid will diffuse through a leak more readily than the high viscosity oil so that leakage can be detected more rapidly.

ACTIVITY

The Testing Process

The ESD units are loaded into containers called "test fixtures," and the test fixtures are then heated to 75°C. The 0.65 cs tracer fluid boils at 60°C so that any fluid leaking from the units should diffuse readily into the test fixtures. After the test fixture has been heated for 2 hours minimum, a small sample of gas is removed and tested for tracer fluid using a gas chromatograph.

Calibration of the gas chromatograph is accomplished by preparing a "known" sample of the tracer fluid vapor in a test fixture and comparing the response of the gas chromatograph when injecting the known amount and the test sample. The test fixtures used for preparing the known samples are called "reference" fixtures.

The known sample is prepared in several steps as shown in Figure 1. First, 3 μ L of silicone tracer fluid are measured with a 5- μ L syringe and injected into a clean nitrogen-filled 21-L flask. The flask is then agitated and allowed to stabilize. The flask contains 15 solid, 3/8-in.-diameter, Teflon balls which help to distribute the fluid vapor uniformly in the flask when it is agitated. Next, approximately 1 mL of silicone tracer fluid vapor is withdrawn from the flask using a gastight 1-mL syringe. The plunger of the syringe is retracted to the stop so that a repeatable amount can be extracted. The vapor in the syringe is injected into a sealed, evacuated reference fixture containing control samples, and then the reference fixture is filled with air zero up to atmospheric pressure and stored at 75°C for a minimum of two hours. The known sample is now ready to be introduced into the sampler of the gas chromatograph.

Figure 1. Silicone Tracer Fluid Measurement Volumes

The sampler of the gas chromatograph is evacuated before a sample is drawn from a fixture containing either the known sample or the parts to be tested. After the fixture is connected, the pressures in the fixture and the sampler are allowed to equilibrate, and then the fixture is removed. However, only a portion of the amount drawn into the sampler--the amount in the sample loop--is flushed into the gas chromatograph.

Mathematical Simulation

Symbols used in the mathematical analysis of uncertainty are defined below. Refer to Figure 1 for additional explanation.

V1 = Volume dispensed by the 5.0- μ L syringe from the 3.0- μ L mark.

V2 = Volume of the 21-L flask minus volume of balls.

V3 = Volume of the 1-mL syringe at the stop (approximately 1.07 mL).

V4 = Volume of the reference fixture minus volume of the control samples (approximately 100 mL net).

W4 = Volume of the test fixture minus volume of the switches.

V5 = Volume of the sample loop.

V6 = Volume in evacuated lines (exclusive of sample loop).

Then the concentration of silicone tracer fluid in the flask is

$$C2 = \text{Concentration in flask} = V1/V2,$$

and the concentration in the reference fixture is

$$C4 = \text{Concentration in reference fixture} = V3 (C2)/V4$$

$$= V3 (V1/V2)/V4$$

$$= \frac{V1 \cdot V3}{V2 \cdot V4}$$

The volume of silicone tracer fluid A introduced into the sample loop from the reference fixture is

$$A = V5 \left(\frac{V1 \cdot V3}{V2 (V4 + V5 + V6)} \right).$$

Here the term $V4 + V5 + V6$ has been used in the denominator because the concentration is changed slightly when the volume $V5 + V6$ is added to the volume containing the silicone tracer fluid while the sample loop is being filled.

Next, it is necessary to find an equation for the volume S of silicone tracer fluid in the sample loop when a volume X of silicone tracer fluid is leaked into the test fixture $W4$. By comparison to the above equation, the volume S is

$$S = V5 \left(\frac{X}{W4 + V5 + V6} \right).$$

where,

X = Volume of silicone tracer fluid leaked from switches into test fixture

$$= \text{leak rate} \times \text{time} = R \cdot T,$$

or

$$S = V5 \frac{X}{(W4 + V5 + V6)} = V5 \frac{R \cdot T}{(W4 + V5 + V6)}.$$

The criterion for switch acceptance is $S \leq A$

or

$$\frac{V5 \cdot R \cdot T}{(W4 + V5 + V6)} \leq \frac{V1 \cdot V3 \cdot V5}{V2 (V4 + V5 + V6)}.$$

This criterion can be expressed in terms of the leak rate as

$$R \leq \frac{V1 \cdot V3 \cdot V5}{V2 (V4 + V5 + V6)} \frac{(W4 + V5 + V6)}{V5 \cdot T} = \frac{V1 \cdot V3}{V2 \cdot T} \frac{(W4 + V5 + V6)}{(V4 + V5 + V6)}.$$

The uncertainty in the leak rate measurement can be specified if the uncertainty in $V1$, $V2$, $V3$, $V4$, $W4$, and T can be determined. It is not necessary to calibrate volumes $V5$ and $V6$ because they are small compared to $V4$ and $W4$ and have constant values; an initial measurement will suffice.

The uncertainties of the variables imply a minimum and maximum value that each variable can attain. Likewise, the measured value of R can attain a range of values resulting from varying values of the variables. The equation for R can be used to determine this range. The minimum value of R is achieved when each term in the numerator has its minimum value and each term

in the denominator has its maximum value. Likewise, the maximum value of R is achieved when each term in the numerator assumes its maximum value and each term in the denominator reaches its minimum value.

The uncertainties of the variables V1, V2, V3, V4, and W4 have been analyzed, and a summary of uncertainty sources and amounts is shown in Appendix A. Statistical data to support this analysis is given in Appendix B. An evaluation of the uncertainty involved in certifying the volume of the control samples is included in Appendix C, and an evaluation of the difference in volume between control samples and switches is presented in Appendix D. Measurements of V5 and V6 are described in Appendix E.

Proposed Tolerances

Tolerances for the volumes based on the uncertainty analysis are proposed below.

<u>Volume</u>	<u>Proposed Tolerance</u>	<u>Minimum (mL)</u>	<u>Maximum (mL)</u>
V1	$(3.12 \pm 0.20) \times 10^{-3}$ mL	2.92×10^{-3}	3.32×10^{-3}
V2	21000 ± 200 mL	20800	21200
V3	1.0655 ± 0.01 mL	1.0555	1.0755
V4	98.1 ± 5 mL	93.1	103.1
W4	100.1 ± 5 mL	95.1	105.1

Total Uncertainty of the Process

The resulting minimum, maximum, and nominal values of the leak rate simulated by the calibration process for a one-hour test are given below.

$$R = \frac{2.92 \times 10^{-3} \times 1.0555 (95.1 + 5.24)}{21200 \times 3600 (103.1 + 5.24)} \text{ mL/s} = 3.74 \times 10^{-11} \text{ mL/s (min.)}$$

$$R = \frac{3.32 \times 10^{-3} \times 1.0755 (105.1 + 5.24)}{20800 \times 3600 (93.1 + 5.24)} \text{ mL/s} = 5.35 \times 10^{-11} \text{ mL/s (max.)}$$

$$R = \frac{3.12 \times 10^{-3} \times 1.0655 (100.1 + 5.24)}{21000 \times 3600 (98.1 + 5.24)} \text{ mL/s} = 4.48 \times 10^{-11} \text{ mL/s (nom.)}$$

These values yield an uncertainty range on the leak rate measurement of $\pm 20\%$.

To determine the leak rate for longer tests, divide the above values by the number of hours of testing. For example, for a two-hour test, the leak rate simulated by the calibration process would be 2.24×10^{-11} mL/s.

The portion of the uncertainty resulting from the uncertainty in each volume is given below:

<u>Volume</u>	<u>Percentage of Total Uncertainty</u>
V1	33%
V2	5%
V3	5%
V4	28%
W4	28%

Because V1, V4, and W4 provide the largest contributions to the total uncertainty, any attempts to reduce the uncertainty further should be focused on these variables.

ACCOMPLISHMENTS

The measurement uncertainty of silicone tracer fluid leakage testing has been quantified for the first time. Results of the evaluation show that an uncertainty of $\pm 20\%$ can be achieved at leak rates as small as 2.24×10^{-11} mL/s for a two-hour test.

Appendix A
UNCERTAINTY SUMMARY

UNCERTAINTY SUMMARY

MEASUREMENT UNC. SOURCES	MEAS. UNC. AMOUNT	PROPOSED TOLERANCE
<u>5.0 μL Syringe (V1)</u>		0.00312 $\pm$ 0.00020 mL
Systematic		
Error at 3.0 μ L	+0.000123, +0.000115, +0.000039, +0.000208 mL	
Random		
Dispensing Variations (16 samples, 3SD)	$\pm$ 0.000083 mL	
Balance Uncertainty	<u>$\pm$0.00004</u> mL	
Total	$\pm$ 0.000123 mL	
<u>21 L Reference Flask (V2)</u>		21000 $\pm$ 200 mL
Systematic		
Variation in Construction (3 samples) + 168, - 69, +152 mL		
Random		
Balance Uncertainty (Full, 24713g)	$\pm$ 2 mL	
Balance Uncertainty (Empty, 3603g)	$\pm$ 2 mL	
Filling Variation	<u>$\pm$1 mL</u> *	
Total	$\pm$ 5 mL	
<u>1 mL Syringe (V3)</u>		1.0655 $\pm$ 0.01 mL
Systematic		
Difference from 1.0000 mL (4 samples, avg.)	0.0670 mL	
Random		
Air Bubbles on Teflon	$\pm$ 0.0010 mL *	
Variation from Avg. Volume (1.0670 mL, 3SD)	$\pm$ 0.0047 mL	
Dispensing Variation (16 samples, 3SD)	$\pm$ 0.0006 mL	
Balance Uncertainty (Full, 3.36g)	$\pm$ 0.0001 mL	
Balance Uncertainty (Empty, 2.28g)	<u>$\pm$0.0001</u> mL	
Total	$\pm$ 0.0065 mL	
		* Estimated

UNCERTAINTY SUMMARY (Continued)

MEASUREMENT UNC. SOURCES	MEAS. UNC. AMOUNT	PROPOSED TOLERANCE
<u>Reference Fixture (V4) and Test Fixture (W4)</u>		
	Reference (V4)	98.1 $\pm$ 5.0 mL
	Test (W4)	100.1 $\pm$ 5.0 mL

Container

Random

Balance Uncertainty (Full, 830g)	$\pm$ 0.00299 mL
Balance Uncertainty (Empty, 680g)	$\pm$ 0.00254 mL
Filling Error (bubbles, etc.)	$\pm$ 0.01 mL *
Part-to-Part Variation (39 samples, 3SD)	<u>$\pm$5.34 mL</u>
Total	$\pm$ 5.36 mL

Control Samples

Systematic

Vol. Difference (Controls vs. Switches)	+1.952 mL
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Random

Uncertainty in Density and Weighing	$\pm$ 0.249 mL
Part-to-Part Variation (8 samples, 3SD)	<u>$\pm$0.60 mL</u>
Total	$\pm$ 0.849 mL

* Estimated

Appendix B
STATISTICAL DATA

STATISTICAL DATA

Fifty volumes were calibrated for the leak testing program. Each was given a Calibration Control Number (CCN).

The syringes, flasks, reference fixtures, and test fixtures were calibrated with water. The 5.0- μ L syringes were calibrated by weighing the amount of water dispensed from the 3.0- μ L mark, and the 1.0-mL syringes were calibrated by weighing the amount of water dispensed when the syringes were filled to the stop and emptied. The flasks, reference fixtures, and test fixtures were calibrated by weighing them empty and filled with water. Calibration of the control samples is described in Appendix C. Data from the calibrations are given below.

5.0 μ L Syringe (V1)

<u>CCN 20997</u>			<u>CCN 20998</u>	
Trial	Volume (10^{-3} mL)	Dev. from Avg. (10^{-3} mL)	Volume (10^{-3} mL)	Dev. from Avg. (10^{-3} mL)
1	3.123	0.000	3.099	-0.016
2	3.107	-0.016	3.132	0.017
3	3.134	0.011	3.050	-0.065
4	<u>3.129</u>	0.006	<u>3.179</u>	0.064
Avg.	3.123		3.115	

<u>CCN 20999</u>			<u>CCN 21000</u>	
Trial	Volume (10^{-3} mL)	Dev. from Avg. (10^{-3} mL)	Volume (10^{-3} mL)	Dev. from Avg. (10^{-3} mL)
1	3.030	-0.009	3.180	-0.028
2	3.016	-0.023	3.225	0.017
3	3.072	0.033	3.213	0.005
4	<u>3.037</u>	-0.002	<u>3.214</u>	0.006
Avg.	3.039		3.208	

<u>CCN</u>	<u>Deviation from 3.000×10^{-3} mL</u> <u>(10^{-3} mL)</u>	<u>Dev. from Avg.</u> <u>(10^{-3} mL)</u>
20997	3.123	0.002
20998	3.115	-0.006
20999	3.039	-0.082
21000	3.208	0.087

Standard Deviation (16 trials) = 0.028×10^{-3} mL
 3 Standard Deviations = 0.083×10^{-3} mL

21 L Reference Flask (V2)

<u>CCN</u>	<u>Volume (mL) @ 20°C</u>	<u>Dev. from 21 L (mL)</u>
20943	21168	+168
20944	20931	-69
21089	21152	+152

1 mL Syringe (V3)

<u>CCN 21051</u>			<u>CCN 21052</u>	
<u>Trial</u>	<u>Volume at</u> <u>Stop (mL)</u>	<u>Dev. from Avg.</u> <u>(mL)</u>	<u>Volume at</u> <u>Stop (mL)</u>	<u>Dev. from Avg.</u> <u>(mL)</u>
1	1.0679	-0.0001	1.0652	0.0001
2	1.0680	0.0000	1.0651	0.0000
3	1.0679	-0.0001	1.0647	-0.0004
4	<u>1.0681</u>	0.0001	<u>1.0655</u>	0.0004
Avg.	1.0680		1.0651	

<u>CCN 21053</u>			<u>CCN 20982</u>		
Trial	Volume at Stop (mL)	Dev. from Avg. (mL)	Volume at Stop (mL)	Dev. from Avg. (mL)	
1	1.0658	-0.0001	1.0691	0.0001	
2	1.0659	0.0000	1.0688	-0.0002	
3	1.0659	0.0000	1.0689	-0.0001	
4	<u>1.0659</u>	0.0000	<u>1.0691</u>	<u>-0.0001</u>	
Avg.	1.0659		1.0690		

<u>CCN</u>	Volume at Stop (mL)	Dev. from Avg. (mL)
21051	1.0680	0.0010
21052	1.0651	-0.0019
21053	1.0659	-0.0011
20982	1.0690	0.0020

Standard Deviation (16 trials) = 0.0002 mL
 3 Standard Deviations = 0.0006 mL

Reference Fixture (V4) and Test Fixture (W4)

<u>CCN</u>	<u>Volume (mL)</u>	<u>Dev. from 141.8 mL (mL)</u>	<u>Dev. from Avg. (mL)</u>
20927	144.63	2.81	3.08
20928	140.44	-1.38	-1.11
20929	141.74	-0.08	0.19
20930	137.98	-3.84	-3.57
20931	140.90	-0.92	-0.65
20932	141.91	0.09	0.36
20933, # 1	142.05	0.23	0.50
20933, # 2	139.94	-1.88	-1.61
20933, # 3	140.09	-1.73	-1.46
20933, # 4	144.75	2.93	3.20
20933, # 5	142.79	0.97	1.24
20933, # 6	142.54	0.72	0.99
20933, # 7	140.65	-1.17	-0.90
20933, # 8	140.22	-1.60	-1.33
20933, # 9	143.38	1.56	1.83
20933, #10	145.36	3.54	3.81
20933, #11	139.40	-2.42	-2.15
20933, #12	140.52	-1.30	-1.03
20933, #13	143.11	1.29	1.56
20933, #14	138.29	-3.53	-3.26
20933, #15	140.26	-1.56	-1.29
20933, #16	140.42	-1.40	-1.13
20933, #17	140.31	-1.51	-1.24
20933, #18	143.39	1.57	1.84
20933, #19	141.99	0.17	0.44
20933, #20	141.53	-0.29	-0.02
20933, #21	141.92	0.10	0.37
20933, #22	142.52	0.70	0.97
20933, #23	140.83	-0.99	-0.72
20933, #24	142.02	0.20	0.47
20933, #25	139.51	-2.31	-2.04
20933, #26	140.99	-0.83	-0.56
20933, #28	138.64	-3.18	-2.91
20933, #29	144.47	2.65	2.92
20933, #30	140.55	-1.27	-1.00
20934	143.27	1.45	1.72
20935	143.18	1.36	1.63
20936	140.41	-1.41	-1.14
20937	<u>143.48</u>	1.66	1.93

Avg. 141.55

Standard Deviation (39 samples) = 1.78 mL
3 Standard Deviations = 5.34 mL

Control Samples

<u>CCN</u>	<u>Volume of Control Samples (mL)</u>	<u>Dev. from Avg. (mL)</u>
20927	43.33	-0.20
20928	43.37	-0.16
20930	43.33	-0.20
20931	43.32	-0.21
20934	43.66	0.13
20935	43.74	0.21
20936	43.78	0.25
20937	<u>43.71</u>	0.18
Avg.	43.53	

Standard Deviation of Control Samples (8 samples) = 0.20 mL
3 Standard Deviations = 0.60 mL

Net * of Average Volumes = 141.55 mL - 43.53 mL = 98.02 mL

*Average volume of fixture less average volume of control samples.

Appendix C

UNCERTAINTY IN CONTROL SAMPLE VOLUME MEASUREMENTS

UNCERTAINTY IN CONTROL SAMPLE VOLUME MEASUREMENTS

Because some of the control samples have irregular shapes (threads, etc.), the best way to determine their volume is to measure their mass and density and then to calculate their volume using the equation $V = \text{mass}/\text{density}$. This will also ease the task of recalibration because the volume can be determined regardless of surface damage to the parts.

Density of control samples was determined by measuring the mass m and volume v of four specially selected blocks and then calculating the density using the formula $d = m/v$. The blocks were selected for uniformity and parallelism. Volume of the selected blocks was determined by measuring the length, width, and height of the blocks.

Uncertainty in the length, width and height measurements was ± 0.003 cm, and this yielded an uncertainty of ± 0.029 cm³ in the block volume measurements, as calculated by the equation:

$$\begin{aligned} v &= l \cdot w \cdot h = (3.187 \pm 0.003) \times (1.266 \pm 0.003) \times \\ &\quad (1.266 \pm 0.003) \text{ cm}^3 \\ &= 5.108 \pm 0.029 \text{ cm}^3. \end{aligned}$$

The uncertainty in the weighing of a block was 0.000519 g, so that the uncertainty in the density was ± 0.044 g/cm³ as calculated by the formula:

$$d = \frac{m}{v} = \frac{39.528 \pm 0.0005 \text{ g}}{5.108 \pm 0.029 \text{ cm}^3} = 7.7386 \pm 0.044 \text{ g/cm}^3.$$

Data for the blocks used for the density determinations are presented below.

Dimensions

<u>Length (in.)</u>	<u>Width (in.)</u>	<u>Height (in.)</u>	<u>Volume (cm³)</u>	<u>Weight (g)</u>	<u>Density (g/cm³)</u>
1.2550	0.5000	0.4997	5.13836	39.73183	7.7324
1.2575	0.4975	0.4983	5.10850	39.51283	7.7347
1.2515	0.4975	0.4988	5.08922	39.36226	7.7344
1.2550	0.4976	0.4990	5.10653	39.59088	<u>7.7530</u>
Average					7.7386

The uncertainty in the control samples in a reference fixture (average weight 338 g, average density 7.7386 g/cm³) was calculated to be $\pm 0.249 \text{ cm}^3$ from the formula:

$$V = \frac{\text{mass}}{\text{density}} = \frac{338 \pm 0.0015 \text{ g}}{7.7386 \pm 0.044 \text{ g/cm}^3} = 43.677 \pm 0.249 \text{ g/cm}^3.$$

Appendix D

DIFFERENCE IN VOLUME BETWEEN CONTROL SAMPLES AND SWITCHES

APPENDIX D

DIFFERENCE IN VOLUME BETWEEN CONTROL SAMPLES AND SWITCHES

The difference in volume between control samples and switches has been determined. Representative dimensions for the control samples were taken from the average dimensions of four control samples. The value at the midpoint of the tolerance band was used for switches.

Control Sample

$$l = 3.187 \text{ cm}$$

$$w = 1.266 \text{ cm}$$

$$h = 1.266 \text{ cm}$$

$$V = l \times w \times h = 3.187 \times 1.266 \times 1.266 \text{ cm}^3$$

$$= 5.108 \text{ cm}^3$$

Ends 1.261 cm dia x 0.230 cm length (2 places)

$$\text{Body} = 2.732 \times 1.260 \times 1.260 \text{ cm}^3 = 4.337 \text{ cm}^3$$

$$\text{Ends} = 2\pi r^2 h$$

$$= 2\pi \left(\frac{1.261}{2} \right)^2 0.230 \text{ cm}^3 = 0.574 \text{ cm}^3$$

$$\text{Grooves} = 2 (\pi (R_2^2 - R_1^2) h)$$

$$= 2\pi \left(\left(\frac{1.053}{2} \right)^2 - \left(\frac{0.908}{2} \right)^2 \right) 0.105 \text{ cm}^3$$

$$= 0.047 \text{ cm}^3$$

$$\text{Bevel} = 2 \left(\frac{1}{2} \right) \pi \left(\left(\frac{1.140}{2} \right)^2 - \left(\frac{1.053}{2} \right)^2 \right) 0.087 \text{ cm}^3$$

$$\text{Pins} = 6 \left(\pi \left(\frac{R}{2} \right)^2 l \right)$$

$$= 6\pi \left(\frac{0.102}{2} \right)^2 0.259 \text{ cm}^3$$

$$= 0.013 \text{ cm}^3$$

$$\text{Switch Volume} = \text{Body} + \text{Ends} - \text{Grooves} - \text{Bevel} + \text{Pins}$$

$$= 4.337 \text{ cm}^3 + 0.574 \text{ cm}^3 - 0.047 \text{ cm}^3 - 0.013 \text{ cm}^3 + 0.013 \text{ cm}^3$$

$$= 4.864 \text{ cm}^3$$

Difference

$$\text{Difference} = \text{Control Volume} - \text{Switch Volume}$$

$$= 5.108 \text{ cm}^3 - 4.864 \text{ cm}^3$$

$$= 0.244 \text{ cm}^3$$

For each test fixture we have 8 switches.

Hence for each test fixture the difference in volume is:

$$\text{Difference} \times 8 = 1.952 \text{ cm}^3$$

Appendix E

MEASUREMENT OF SAMPLE VOLUME AND EVACUATED TUBE VOLUME

MEASUREMENT OF SAMPLE VOLUME AND EVACUATED TUBE VOLUME

The volume of the sample loop was measured prior to installing it in the sampler by filling the sample loop tubing with water and weighing. The weight of the filled tube was subtracted from the dry weight to determine the weight of the water in the tube. This was multiplied by the appropriate density factor to determine the volume, 1.0074 mL.

The volume of the evacuated portion of the sampler was measured with a volumeter and was found to be 5.24 mL. Hence, the volume of the evacuated portion of the sampler exclusive of the sample loop was 4.23 mL.

END

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