Measurement Uncertainty of Silicone Fluid Leakage Testing for Rolamites

Federal Manufacturing & Technologies

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MEASUREMENT UNCERTAINTY OF SILICONE FLUID LEAKAGE TESTING FOR ROLAMITES

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Contents

Illustration

Table

Abstract

An evaluation has been performed to determine the uncertainty of silicone fluid leakage measurements for two rolamites. The units are tested with a gas chromatograph, an instrument which can measure silicone fluid vapor by the gas chromatography method. An analysis has shown that the portion of the measurement uncertainty resulting from the uncertainties of the volumes used in the procedure can be maintained at ±18% when the volumes are held to a given set of tolerances.

Summary

A mathematical simulation of the silicone fluid leak testing procedures for two rolamites 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. A set of practical tolerances for the volumes was recommended, and the portion of the measurement uncertainty resulting from the uncertainties of the volumes was calculated.

Discussion

Scope and Purpose

Metrology at AlliedSignal Federal Manufacturing & Technologies (FM&T) has been requested to evaluate the uncertainty involved in silicone oil leak testing. This was a portion of the work performed by a Total Quality team organized to identify the volumes required for the testing, to determine the leak rate criteria for the testing process, and to reduce the process uncertainty to a minimum.

Silicone oil is added to two rolamites as a damping material. It is necessary to test for leakage because leaking oil could contaminate other weapon components.

The silicone oil is a mixture of 20.0 cS silicone fluid, 1.0 cS silicone fluid, and triethylene glycol dimethyl ether. Silicone oil in rolamite A contains 25.6% by weight of the 1.0 cS silicone fluid, and the oil in rolamite B contains 41.4% by weight of the 1.0 cS silicone fluid. Only the 1.0 cS fluid is measured by the leak testing process, so that the leak rate specification refers only to the 1.0 cS silicone fluid.

Activity

The Testing Process

Leak testing procedures for two units are described in internal procedures. The units are loaded into containers called "test fixtures," and the test fixtures are then heated to 75°C. After the test fixture has been heated for 2 hours minimum, a small sample of gas is removed and tested for 1.0 cS fluid vapor using a gas chromatograph.

Standardization of the gas chromatograph is accomplished by preparing a "known" sample of the 1.0 cS silicone fluid vapor in a test chamber assembly and comparing the response of the gas chromatograph when injecting the known amount and the test sample. The test chamber assemblies 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 μL of 1.0 cS silicone fluid are measured with a 5-μL syringe and injected into a clean nitrogen-filled 21410-mL flask. The flask is then agitated to vaporize the fluid, and the vapor is allowed to stabilize. Next, approximately 1 mL of silicone fluid vapor is withdrawn from the flask using a gas-tight 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, and then the reference fixture is filled with air zero up to atmospheric pressure and stored at 75°C for a minimum of one hour. The known sample is now ready to be introduced into the sampler of the gas chromatograph.

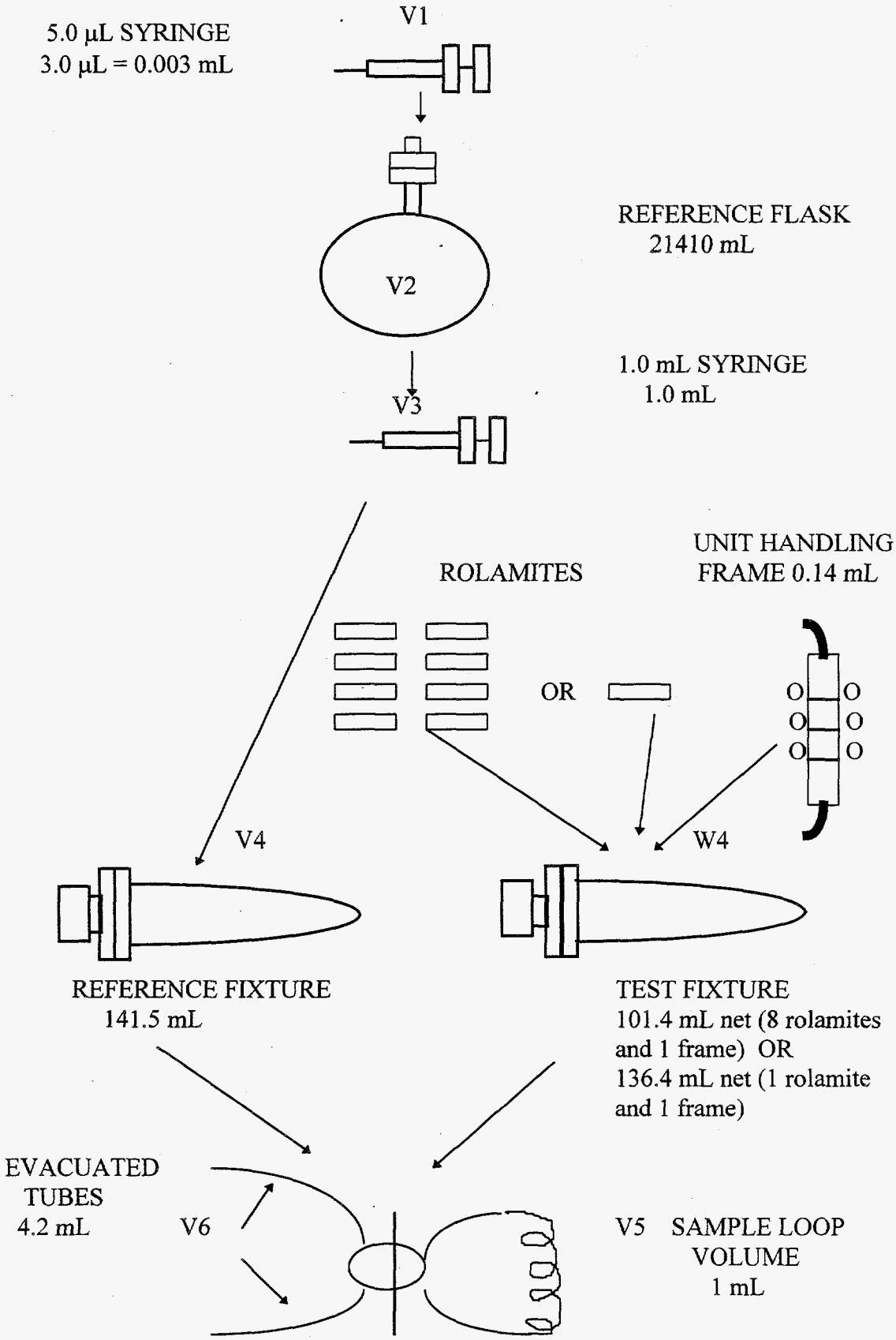

Figure 1. Silicone Fluid Measurement Volumes

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-μL syringe from the 3.0-μL mark.

V2 = Volume of the 21410-mL flask minus volume of 96 beads (6.35mm dia.).

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

V4 = Volume of the reference fixture.

W4 = Volume of the test fixture (volume of the test chamber assembly minus volume of the unit handling frame and the rolamites.)

V5 = Volume of the sample loop.

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

Then the concentration of silicone fluid in the flask is

C2 = Concentration in flask = V1/V2,

and the concentration in the reference fixture is

C4 = Concentration in reference fixture = V3 (C2)/V4

= V3 (V1/V2)/V4

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

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

$$A = V5 \frac{(V1 \bullet V3)}{V2(V4 + V5 + V6)}$$

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 fluid while the sample loop is being filled.

Next, it is necessary to find an equation for the volume S of silicone fluid in the sample loop when a volume X of 1.0 cS silicone 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 1.0 cS silicone fluid leaked from rolamites into test fixture

= leak rate x time = R • T,

or

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

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

or

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

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

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

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. Details of the calculation of the rolamite external volume and volume uncertainty are included in Appendix C, along with details of the determination of the volume of the unit handling frame. Measurements of V5 and V6 are described in Appendix D.

Proposed Tolerances

Tolerances for the volumes based on the uncertainty analysis are proposed below. These values are for tests involving 8 rolamites per test fixture.

Table 1. Proposed Tolerances for Measurement Volumes

Volume	Proposed Tolerance	Minimum (mL)	Maximum (mL)
V1	$(3.13 \pm 0.20) \times 10^{-3}$ mL	2.93×10^{-3}	3.33×10^{-3}
V2	21410 ±50 mL	21360	21460
V3	1.0667 ±0.01 mL	1.0567	1.0767
V4	141.5 ±5.0 mL	136.5	146.5
W4	101.4 ±6.2 mL	95.2	107.6

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.

$$R = \frac{2.93 \times 10^{-3} \times 1.0567\ (95.2 + 5.24)\ \text{mL/s}}{21460 \times 3600\ (146.5 + 5.24)} = 2.65 \times 10^{-11}\ \text{mL/s (min.)}$$

$$R = \frac{3.33 \times 10^{-3} \times 1.0767\ (107.6 + 5.24)\ \text{mL/s}}{21360 \times 3600\ (136.5 + 5.24)} = 3.71 \times 10^{-11}\ \text{mL/s (max.)}$$

$$R = \frac{3.13 \times 10^{-3} \times 1.0667\ (101.4 + 5.24)\ \text{mL/s}}{21410 \times 3600\ (141.5 + 5.24)} = 3.15 \times 10^{-11}\ \text{mL/s (nom.)}$$

When only one rolamite is tested per test fixture the tolerance of W4 is changed to 136.4 ± 5.2 mL. In this case the minimum, maximum, and nominal values are as shown below.

$$R = \frac{2.93 \times 10^{-3} \times 1.0567\ (131.2 + 5.24)\ \text{mL/s}}{21460 \times 3600\ (146.5 + 5.24)} = 3.60 \times 10^{-11}\ \text{mL/s (min.)}$$

$$R = \frac{3.33 \times 10^{-3} \times 1.0767\ (141.6 + 5.24)\ \text{mL/s}}{21360 \times 3600\ (136.5 + 5.24)} = 4.83 \times 10^{-11}\ \text{mL/s (max.)}$$

$$R = \frac{3.13 \times 10^{-3} \times 1.0667\ (136.4 + 5.24)\ \text{mL/s}}{21410 \times 3600\ (141.5 + 5.24)} = 4.18 \times 10^{-11}\ \text{mL/s (nom.)}$$

These values yield an uncertainty range on the leak test measurement of ±18%.

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 involving 8 rolamites per test fixture, the leak rate simulated by the calibration process would be 1.58×10^{-11} mL/s.

Accomplishments

The portion of the measurement uncertainty resulting from the uncertainties of the volumes used for silicone fluid leakage testing has been quantified. Results of the evaluation show that an uncertainty of ±18% can be achieved at leak rates as small as 1.58×10^{-11} mL/s during a two-hour test.

Appendix A

Uncertainty Summary

UNCERTAINTY SUMMARY

MEASUREMENT UNC. SOURCES	MEAS. UNC. AMOUNT	PROPOSED TOLERANCE
5.0 µL Syringe (V1)		0.00313 ±0.00020 mL
Systematic		
Difference from 3.13 µL	-0.000015, -0.000030	
Line at 3.0 µL	+0.000027, -0.000008	
Random		
Dispensing Variations (11 samples, 3SD)	±0.000093 mL	
Balance Uncertainty	±0.00004 mL	
Total	±0.000133 mL	
21410 mL Reference Flask (V2)		21410 ±50 mL
Systematic		
Variation in Construction (2 samples)	+31, -31 mL	
Random		
Balance Uncertainty (Full, 24713 g)	±2 mL	
Balance Uncertainty (Empty, 3603 g)	±2 mL	
Filling Variation	±1 mL *	
Total	±5 mL	
1 mL Syringe (V3)		1.0667 ±0.01 mL
Systematic		
Difference from 1.0667 mL (4 samples)	+0.0016, -0.0028, -0.0017, +0.0028 mL	
Random		
Air Bubbles on Teflon	±0.0010 mL*	
Dispensing Variation (14 samples, 3SD)	±0.0017 mL	
Balance Uncertainty (Full, 3.36 g)	±0.0001 mL	
Balance Uncertainty (Empty, 2.28 g)	±0.0001 mL	
Total	±0.0029 mL	

*Estimated

Reference Fixture (V4), Test Chamber Assembly, and Test Fixture (W4)

Reference (V4)	141.5 ±5.0 mL
Test Chamber	141.5 ±5.0 mL
Test (1 Rolamite) (W4)	136.4 ±5.2 mL
Test (8 Rolamites) (W4)	101.4 ±6.2 mL

Test Chamber Assembly

Systematic

Variation in Construction (36 samples, max.)	±3.84 mL

Random

Balance Uncertainty (Full, 830 g)	±0.00299 mL
Balance Uncertainty (Empty, 680 g)	±0.00254 mL
Filling Error (bubbles, etc.)	±0.01 mL *
Total	±0.01553 mL

One Rolamite

Random

Variation in Dimensions	±0.147 mL

8 Rolamites

Random

Variation in Dimensions	±1.176 mL

Unit Handling Frame

Random

Uncertainty in Density and Weighing	±0.0152 mL
Part-to-Part Variation (5 samples, 3SD)	±0.0045 mL
Total	±0.0197 mL

*Estimated

Appendix B

Statistical Data

STATISTICAL DATA

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

The syringes, flasks, reference fixtures, and test chamber assemblies were calibrated with water. The 5.0-µL syringes were calibrated by weighing the amount of water dispensed from the 3.0-µL 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 chamber assemblies were calibrated by weighing them empty and filled with water. Measurement of the unit handling frame volumes is described in Appendix C. Data from the calibrations are given below.

5.0 µL Syringe (V1)

Trial	Volume from 3.0 µL Line (10^{-3} mL)				Dev. from Avg. (10^{-3} mL)			
	CCN 20997	CCN 20999	CCN 21000	CCN 00872	CCN 20997	CCN 20999	CCN 21000	CCN 00872
1	3.1230	3.0386	3.2082	3.1217	0.0079	-0.0617	0.0517	0.0000
2	3.1230	3.1050	3.1303		0.0079	0.0047	-0.0262	
3	3.1270	3.1220	3.1310		0.0119	0.0217	-0.0255	
4	3.0872	3.1355			-0.0279	0.0352		
Avg.	3.1151	3.1003	3.1565	3.1217				

CCN	Volume from 3.0 µL Line (10^{-3} mL)	Dev. from Avg. (10^{-3} mL)
20997	3.115	-0.009
20999	3.100	-0.024
21000	3.157	0.033
00872	3.122	-0.002

Standard Deviation (11 trials) = 0.031 x 10^{-3} mL
3 Standard Deviations = 0.093 x 10^{-3} mL

21410 mL Reference Flask (V2)

CCN	Volume (mL) @ 20°C	Dev. from 21410 mL (mL)
00829	21441	+31
00830	21379	-31

1 mL Syringe (V3)

Trial	Volume at Stop (mL)				Dev. from Avg. (mL)			
	CCN 21051	CCN 21052	CCN 21053	CCN 20982	CCN 21051	CCN 21052	CCN 21053	CCN 20982
1	1.06797	1.06511	1.06588	1.06898	-0.00035	0.00126	0.00090	-0.00056
2	1.06850	1.06341	1.06500	1.06968	0.00026	-0.00044	0.00002	0.00014
3	1.06848	1.06319	1.06407	1.06997	0.00016	-0.00066	-0.00091	0.00043
4	1.06823	1.06368			-0.00009	-0.00017		
Avg.	1.06832	1.06385	1.06498	1.06954				

CCN	Volume at Stop (mL)	Dev. from Avg. (mL)
21051	1.0683	0.0016
21052	1.0639	-0.0028
21053	1.0650	-0.0017
20982	1.0695	0.0028
Avg.	1.0667	

Standard Deviation (14 trials) = 0.0006 mL
3 Standard Deviations = 0.0017 mL

Reference Fixture (V4) and Test Chamber Assembly

CCN	Volume (mL)	Dev. from Avg. (mL)
20927	144.63	3.11
20928	140.44	-1.08
20930	137.98	-3.54
20931	140.90	-0.62
20933, #2	139.94	-1.58
20933, #3	140.09	-1.43
20933, #4	144.75	3.23
20933, #5	142.79	1.27
20933, #6	142.54	1.02
20933, #7	140.65	-0.87
20933, #8	140.22	-1.30
20933, #9	143.38	1.86
20933, #10	145.36	3.84
20933, #11	139.40	-2.12
20933, #12	140.52	-1.00
20933, #13	143.11	1.59
20933, #14	138.29	-3.23
20933, #15	140.26	-1.26
20933, #16	140.42	-1.10
20933, #17	140.31	-1.21
20933, #18	143.39	1.87
20933, #19	141.99	0.47
20933, #20	141.53	0.01
20933, #21	141.92	0.40
20933, #22	142.52	1.00
20933, #23	140.83	-0.69
20933, #24	142.02	0.50
20933, #25	139.51	-2.01
20933, #26	140.99	-0.53
20933, #28	138.64	-2.88
20933, #29	144.47	2.95
20933, #30	140.55	-0.97
20934	143.27	1.75
20935	143.18	1.66
20936	140.41	-1.11
20937	143.48	1.96
Avg.	141.52	

Standard Deviation (36 samples) = 1.85 mL
3 Standard Deviations = 5.55 mL

Unit Handling Frames

No.	Volume of Unit Handling Frame (mL)	Dev. from Avg. (mL)
1	0.1578	0.0000
2	0.1592	0.0014
3	0.1555	-0.0023
4	0.1591	0.0013
5	0.1572	-0.0006
Avg.	0.1578	

Standard Deviation of Unit Handling Frames (5 samples) = 0.0015 mL
3 Standard Deviations = 0.0045 mL

Appendix C

Rolamite and Unit Handling

Frames Volumes and Their Uncertainties

ROLAMITE AND UNIT HANDLING FRAME VOLUMES AND THEIR UNCERTAINTIES

Dimensions and dimensional tolerances for the rolamite components were taken from drawings and were used to calculate the external volume and the external volume tolerance of the assembled rolamites. The two rolamites have the same external dimensions.

Component	Nom. Volume	Max. Volume	Min. Volume
Foot Case	0.267272 $in.^3$	0.275161 $in.^3$	0.260345 $in.^3$
Reset Cap	0.003379	0.003680	0.003060
Actuate Cap	0.033292	0.034434	0.032168
Actuate Pin	0.000248	0.000273	0.000273
Actuate Pin	0.000248	0.000273	0.000273
Ball	0.000052	0.000056	0.000048
Rolamite	0.304895 $in.^3$	0.313877 $in.^3$	0.296167 $in.^3$
Rolamite	4.9963 mL	5.1435 mL	4.8533 mL

The volume and volume tolerance of the rolamites is, therefore, 4.996 mL ± 0.147 mL.

The average volume of the unit handling frame was determined by averaging the volumes of five frames. The volume of the frame was determined by dividing the weight by the density of the wire material. The uncertainty in the weighing of the frame was ±0.4037 mg. The density of the wire material was determined by measuring the mass, length, and diameter of a small section of the wire and calculating the density using the formula $d=m/\pi\,(d/2)^2 l$, where m is the mass, d is the diameter and l is the length. The density of the wire was determined to be 7.822 g/cm^3 and the uncertainty in the density was ±0.688 g/cm^3 so that the uncertainty of the frame volume (weight 1.2341 g) was calculated to be ±0.0135 cm^3 from the formula

$$V=\frac{\text{mass}}{\text{density}}=\frac{1.2341\ g \pm 0.000404\ g}{7.822\ g/cm^3 \pm 0.688\ g/cm^3}$$

$$=0.1578\ cm^3 \pm 0.0152\ cm^3$$

The volume of one rolamite is 4.996 mL. Hence, the internal volume of the average test fixture containing one rolamite and one unit handling frame is 141.52 mL minus 4.996 mL minus 0.1578 mL, or 136.37 mL.

Eight rolamites displace a volume of 39.97 mL. Thus, the internal volume of the average test fixture containing eight rolamites and one frame is 141.52 mL minus 39.97 mL minus 0.1578 mL, or 101.39 mL.

Appendix D

Measurement of Sample Loop Volume and Evacuated Tube Volume

MEASUREMENT OF SAMPLE LOOP 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.