Technical Overview

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Calibrated Helium Leaks: Uncertainty, Tolerances, and Test Uncertainty Ratios

Introduction

Helium leak detection is used in a diverse range of industries and an equally wide variety of test methods. Common to nearly all, is a need to understand the relative accuracy of a given measurement.

Ensuring measurement accuracy requires calibration to a known value. This is provided by a calibrated leak installed in the instrument. Agilent Helium Leak Detectors (HLDs) have an internal reference leak that is traceable to a primary reference standard provided by the United States National Institute of Standards and Technology (NIST). These internal leaks contain unpressurized helium and have a nominal leak rate of 1.5×10^{-7} atm·cm^3/s. Alternatively, or in addition to the internal leak, customers may also use an external calibrated helium leak. External calibrated leaks are typically pressurized, and a stable leak rate is achieved by helium permeating through a specific material or escaping through a small capillary. All calibrated helium leaks should have a certificate of calibration which states the standards to which the leak was tested and the tolerance range for which that leak is certified.

The purpose of this technical overview is to aid the reader in understanding the key characteristics of a calibrated leak and the meaning of the terminology used on the certificate, such as uncertainty, tolerances, and test uncertainty ratios.

How the internal calibrated leak works on the Agilent HLD

The Agilent internal calibrated leak (Figure 1) is an unpressurized helium leak that flows through a permeable membrane. A linear relationship of flow rate of helium through a diffuse membrane is explained by Darcy's Law, a special case of the Navier-Stokes equations. The leak includes a temperature monitoring circuit and is connected to the instrument's valve block (Figure 2) and valved into the mass spectrometer vacuum system as needed for calibration.

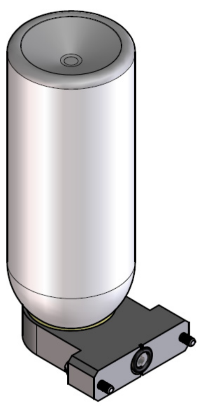

Figure 1. Internal calibrated leak assembly.

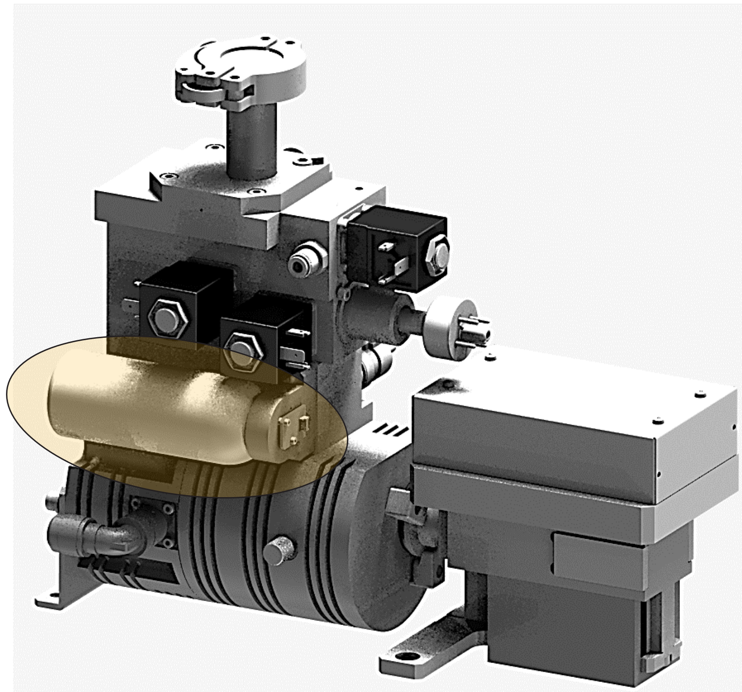

Figure 2. Calibrated leak installed on HLD vacuum system.

During manufacturing, the leak is calibrated using a NIST traceable primary reference standard. This calibration is performed at a single temperature at a single point in time. The Agilent HLD software uses an experimentally determined polynomial curve fit expression to compensate the leak rate for the actual temperature of the calibrated leak during operation. Simply stated, when a calibrated leak is warmer, it will leak at a faster rate than when it is cooler. The helium leak rate is further compensated based on an experimentally determined helium depletion rate, as a function of time. The helium leak rate decreases by a small percentage every year based on a decreased differential helium partial pressure between the calibrated leak volume and the outside atmosphere. The certification on a calibrated leak is good for at least one year.

Understanding the internal calibrated leak label

The label of the internal calibrated leak on the Agilent HLD (Figure 3) lists the following information:

- Agilent part number
- Serial number
- Leak rate measured using a NIST primary reference standard, in units of atm·cm^3/s.
- Temperature at which the leak rate was measured
- The date the leak was calibrated
- Factor is a temperature correction factor used by the leak detector. While the temperature at the time of

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Helium Calibrated Leak

Part No.	R2015301		
Serial No.	XXXXXXXXXX	Temp	XX.X °C
Leak Rate	X.XE-07	Std. C.C./Sec.	
Cal. Date	DD – MMM – YYYY	Factor	X.X °C
Due Date	DD – MMM – YYYY+1	Cal. By	X.X

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Figure 3. Sample internal calibrated leak label.

calibration is measured using NIST traceable instrumentation, the actual temperature measurement chip on the calibrated leak is not as accurate. The factor is a temperature offset value that shows the difference between the measurement of the NIST traceable factory calibration temperature and the value read by the chip. The HLD software uses the measured temperature with the correction offset factor to make temperature-related leak rate compensation.

– The date the calibration expires.

– The initials of the operator who performed the calibration.

Statement of calibration and understanding the statistics

The following is the statement of calibration issued with Agilent internal calibrated leaks:

The present estimate of the expanded uncertainty in the measured leak rate of this artifact at 23°C at the time of calibration is ±5.7%, with a 95% confidence level, coverage factor, k = 2. With a tolerance of 20%, this equates to a TUR of 3.5:1. (For a TUR of 4:1, the confidence level is 92%, coverage factor, k = 1.76)

To understand what this means in terms of the accuracy of the instrument, a bit of explanation might be helpful. Tolerance refers to the product's manufacturing specification limits. Agilent's process requires the ability to test accurately and precisely, within a reasonable range. While the manufacturing process will nominally produce a 1.5×10^{-7} atm·cm^3/s helium leak, understand that it will be certified and labeled with the exact measured value of helium which it is leaking.

Since no instrument is infinitely precise, every measurement should be reported with an estimated uncertainty, a range of how far from exact that measurement may be. Expanded uncertainty is the measurement uncertainty with a safety factor.

Illustrated here is a safety factor of k = 1 and k = 2.The 20% tolerance is a description of the upper and lower specification limits (USL and LSL). For example, if the calibrated leak value is 1.5×10^{-7} atm·cm^3/s, the USL is $(1.20) \times (1.5 \times 10^{-7}$ atm·cm^3/s), and the LSL is $(0.80) \times (1.5 \times 10^{-7}$ atm·cm^3/s). This is shown graphically in Figure 4.

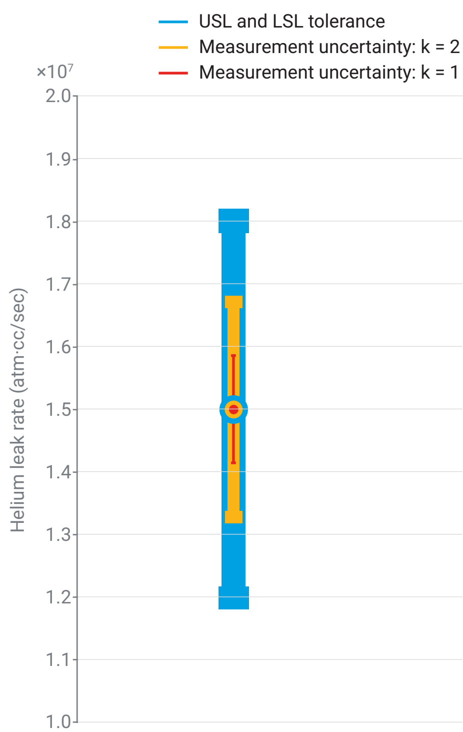


Figure 4. Calibrated leak tolerance and measurement uncertainty.

Test uncertainty ratio (TUR) is the ratio of the specification limit gap (USL – LSL) and uncertainty in the measurement of that part when declaring the calibration. TUR is defined in Equation 1.

$$TUR = \frac{USL - LSL}{ku}$$

Equation 1.

where:

– USL is the upper specification limit of the calibrated leak

– LSL is the lower specification limit of the calibrated leak

– k is the correction factor. A k value of 2 indicates 95% confidence.

– u is measurement uncertainty (note that any bias, if included, would add to the denominator, not scale with k.)

NIST has adopted standard use of k = 2 for a correction factor.[1] The gold standard for a TUR value is 4.[2] If a predefined USL and LSL are provided, a goal for TUR is set, and an uncertainty level is given, k can be solved for, as shown in Equation 2.

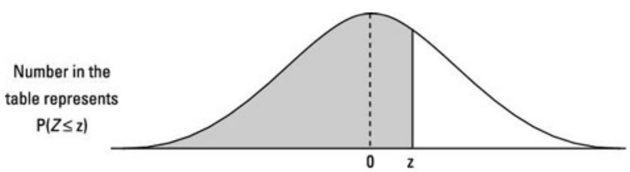
$$k = \frac{USL - LSL}{TUR \times u}$$

Equation 2.

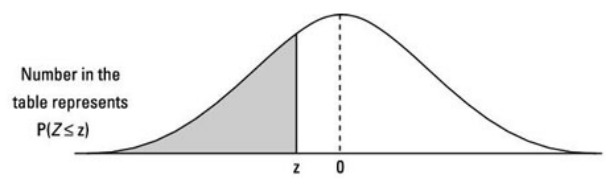
Solving for k, the confidence level can be solved for using a Z-table. For example, if USL and LSL are defined as ±20% of 1.5 E-7 atm·cc/sec, and TUR is set for 4, and u is given as 5.7% of 1.5×10^{-7} atm·cm^3/s, then k would solve to be 1.75. Using the Z-table (Figure 5), this corresponds to 0.9599 – 0.0401 = 0.92 confidence, or equivalently, an alpha risk (α) of 8% (green highlight).

α is the complementary probability of the confidence, 1 – confidence. For reference, 5% alpha risk is a standard value used for statistical control of manufacturing operations. 8% risk is an elevated "risk" statement at the expense of being able to demonstrate a higher test uncertainty ratio.

If a laboratory required TUR of 10, with ±20% USL and LSL tolerances, k would solve to be 0.70. Using the Z-table, this corresponds to 0.758 – 0.242 = 0.52 confidence, or, equivalently, an alpha risk of 48% (purple highlight).



z	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
0.0	.5000	.5040	.5080	.5120	.5160	.5199	.5239	.5279	.5319	.5359
0.1	.5398	.5438	.5478	.5517	.5557	.5596	.5636	.5675	.5714	.5753
0.2	.5793	.5832	.5871	.5910	.5948	.5987	.6026	.6064	.6103	.6141
0.3	.6179	.6217	.6255	.6293	.6331	.6368	.6406	.6443	.6480	.6517
0.4	.6554	.6591	.6628	.6664	.6700	.6736	.6772	.6808	.6844	.6879
0.5	.6915	.6950	.6985	.7019	.7054	.7088	.7123	.7157	.7190	.7224
0.6	.7257	.7291	.7324	.7357	.7389	.7422	.7454	.7486	.7517	.7549
0.7	.7580	.7611	.7642	.7673	.7704	.7734	.7764	.7794	.7823	.7852
0.8	.7881	.7910	.7939	.7967	.7995	.8023	.8051	.8078	.8106	.8133
0.9	.8159	.8186	.8212	.8238	.8264	.8289	.8315	.8340	.8365	.8389
1.0	.8413	.8438	.8461	.8485	.8508	.8531	.8554	.8577	.8599	.8621
1.1	.8643	.8665	.8686	.8708	.8729	.8749	.8770	.8790	.8810	.8830
1.2	.8849	.8869	.8888	.8907	.8925	.8944	.8962	.8980	.8997	.9015
1.3	.9032	.9049	.9066	.9082	.9099	.9115	.9131	.9147	.9162	.9177
1.4	.9192	.9207	.9222	.9236	.9251	.9265	.9279	.9292	.9306	.9319
1.5	.9332	.9345	.9357	.9370	.9382	.9394	.9406	.9418	.9429	.9441
1.6	.9452	.9463	.9474	.9484	.9495	.9505	.9515	.9525	.9535	.9545
1.7	.9554	.9564	.9573	.9582	.9591	.9599	.9608	.9616	.9625	.9633
1.8	.9641	.9649	.9656	.9664	.9671	.9678	.9686	.9693	.9699	.9706
1.9	.9713	.9719	.9726	.9732	.9738	.9744	.9750	.9756	.9761	.9767
2.0	.9772	.9778	.9783	.9788	.9793	.9798	.9803	.9808	.9812	.9817
2.1	.9821	.9826	.9830	.9834	.9838	.9842	.9846	.9850	.9854	.9857
2.2	.9861	.9864	.9868	.9871	.9875	.9878	.9881	.9884	.9887	.9890
2.3	.9893	.9896	.9898	.9901	.9904	.9906	.9909	.9911	.9913	.9916
2.4	.9918	.9920	.9922	.9925	.9927	.9929	.9931	.9932	.9934	.9936
2.5	.9938	.9940	.9941	.9943	.9945	.9946	.9948	.9949	.9951	.9952
2.6	.9953	.9955	.9956	.9957	.9959	.9960	.9961	.9962	.9963	.9964
2.7	.9965	.9966	.9967	.9968	.9969	.9970	.9971	.9972	.9973	.9974
2.8	.9974	.9975	.9976	.9977	.9977	.9978	.9979	.9979	.9980	.9981
2.9	.9981	.9982	.9982	.9983	.9984	.9984	.9985	.9985	.9986	.9986
3.0	.9987	.9987	.9987	.9988	.9988	.9989	.9989	.9989	.9990	.9990
3.1	.9990	.9991	.9991	.9991	.9992	.9992	.9992	.9992	.9993	.9993
3.2	.9993	.9993	.9994	.9994	.9994	.9994	.9994	.9995	.9995	.9995
3.3	.9995	.9995	.9995	.9996	.9996	.9996	.9996	.9996	.9996	.9997
3.4	.9997	.9997	.9997	.9997	.9997	.9997	.9997	.9997	.9997	.9998
3.5	.9998	.9998	.9998	.9998	.9998	.9998	.9998	.9998	.9998	.9998
3.6	.9998	.9998	.9999	.9999	.9999	.9999	.9999	.9999	.9999	.9999



z	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
−3.6	.0002	.0002	.0001	.0001	.0001	.0001	.0001	.0001	.0001	.0001
−3.5	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002	.0002
−3.4	.0003	.0003	.0003	.0003	.0003	.0003	.0003	.0003	.0003	.0002
−3.3	.0005	.0005	.0005	.0004	.0004	.0004	.0004	.0004	.0004	.0003
−3.2	.0007	.0007	.0006	.0006	.0006	.0006	.0006	.0005	.0005	.0005
−3.1	.0010	.0009	.0009	.0009	.0008	.0008	.0008	.0008	.0007	.0007
−3.0	.0013	.0013	.0013	.0012	.0012	.0011	.0011	.0011	.0010	.0010
−2.9	.0019	.0018	.0018	.0017	.0016	.0016	.0015	.0015	.0014	.0014
−2.8	.0026	.0025	.0024	.0023	.0023	.0022	.0021	.0021	.0020	.0019
−2.7	.0035	.0034	.0033	.0032	.0031	.0030	.0029	.0028	.0027	.0026
−2.6	.0047	.0045	.0044	.0043	.0041	.0040	.0039	.0038	.0037	.0036
−2.5	.0062	.0060	.0059	.0057	.0055	.0054	.0052	.0051	.0049	.0048
−2.4	.0082	.0080	.0078	.0075	.0073	.0071	.0069	.0068	.0066	.0064
−2.3	.0107	.0104	.0102	.0099	.0096	.0094	.0091	.0089	.0087	.0084
−2.2	.0139	.0136	.0132	.0129	.0125	.0122	.0119	.0116	.0113	.0110
−2.1	.0179	.0174	.0170	.0166	.0162	.0158	.0154	.0150	.0146	.0143
−2.0	.0228	.0222	.0217	.0212	.0207	.0202	.0197	.0192	.0188	.0183
−1.9	.0287	.0281	.0274	.0268	.0262	.0256	.0250	.0244	.0239	.0233
−1.8	.0359	.0351	.0344	.0336	.0329	.0322	.0314	.0307	.0301	.0294
−1.7	.0446	.0436	.0427	.0418	.0409	.0401	.0392	.0384	.0375	.0367
−1.6	.0548	.0537	.0526	.0516	.0505	.0495	.0485	.0475	.0465	.0455
−1.5	.0668	.0655	.0643	.0630	.0618	.0606	.0594	.0582	.0571	.0559
−1.4	.0808	.0793	.0778	.0764	.0749	.0735	.0721	.0708	.0694	.0681
−1.3	.0968	.0951	.0934	.0918	.0901	.0885	.0869	.0853	.0838	.0823
−1.2	.1151	.1131	.1112	.1093	.1075	.1056	.1038	.1020	.1003	.0985
−1.1	.1357	.1335	.1314	.1292	.1271	.1251	.1230	.1210	.1190	.1170
−1.0	.1587	.1562	.1539	.1515	.1492	.1469	.1446	.1423	.1401	.1379
−0.9	.1841	.1814	.1788	.1762	.1736	.1711	.1685	.1660	.1635	.1611
−0.8	.2119	.2090	.2061	.2033	.2005	.1977	.1949	.1922	.1894	.1867
−0.7	.2420	.2389	.2358	.2327	.2296	.2266	.2236	.2206	.2177	.2148
−0.6	.2743	.2709	.2676	.2643	.2611	.2578	.2546	.2514	.2483	.2451
−0.5	.3085	.3050	.3015	.2981	.2946	.2912	.2877	.2843	.2810	.2776
−0.4	.3446	.3409	.3372	.3336	.3300	.3264	.3228	.3192	.3156	.3121
−0.3	.3821	.3783	.3745	.3707	.3669	.3632	.3594	.3557	.3520	.3483
−0.2	.4207	.4168	.4129	.4090	.4052	.4013	.3974	.3936	.3897	.3859
−0.1	.4602	.4562	.4522	.4483	.4443	.4404	.4364	.4325	.4286	.4247
−0.0	.5000	.4960	.4920	.4880	.4840	.4801	.4761	.4721	.4681	.4641

Figure 5. Using a Z-table to determine confidence level.

Table 1 illustrates a list of TUR options, solved using the equations provided. Each entry uses k = 2 and u = 5.7% of a leak rate of 1.5×10^{-7} atm·cm^3/s.

Table 1. TUR options.

Tolerance	k	TUR	Confidence
±20%	2	3.5:1	95%
±20%	2	4:1	92%
±20%	2	10:1	52%

Final words

The above applies only to the accuracy of the supplied internal calibrated leak and the accuracy of the electronics in the instrument. Accuracy of a leak test system (including any fixtures or apparatus connected to the leak detector) can only be determined by performing a proper gauge repeatability and reproducibility analysis.

Terminology and specifications used to describe external calibrated leaks may differ from those used here.

References

1. Jun-Feng, S. The Guidelines for Expressing Measurement Uncertainties and the 4:1 Test Uncertainty Ratio. *NIST*, July **1997**.
2. Khanaman, S.; Morse, E. Test Uncertainty Ratio (TUR). *American Society for Precision Engineering* **2009**.

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