Low Voltage Traceability and Uncertainty Evaluation for High Accuracy Thermocouple Calibration Utilizing a Modern Automated Potentiometer

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Abstract

The Hart Scientific Division of Fluke Corporation manufactures a family of high quality noble metal thermocouples intended for laboratory applications. The calibration laboratory is required to calibrate these thermocouples to very low levels of uncertainty. Noble metal thermocouples are calibrated by fixed point over the range of 0 °C to 1000 °C. The temperature uncertainty components that contribute to the total uncertainty are easily dealt with in this laboratory because the laboratory has the capability for low uncertainty calibration of SPRTs. The equipment and procedures used in the SPRT process were easily adapted to thermocouple calibration. However, the voltage measurement aspects of the calibration are not so simple. The voltages generated vary between 1.7 mV and 16 mV, and the sensitivities vary between 8 μV/°C and 24 μV/°C depending on thermocouple type and temperature. With expanded uncertainty requirements between 0.01 °C and 0.15 °C, nanovolt measurement accuracy is required. To further complicate matters, the Hart Scientific laboratory is an accredited laboratory with a high volume workload (relatively speaking) and strict quality control requirements; any solution proposed would have to be suitable for such an environment.

A system was developed using a 1.018 VDC – based manual seven-dial potentiometer, sensitive digital voltmeter, and low thermal switch. As the manual potentiometer aged and became unreliable, and the need for automation arose, it was replaced with a modern 10 VDC – based automated potentiometer. The change in potentiometers created challenges that could only be solved through introduction of new hardware and changes in system architecture.

This paper will present a description of the system utilizing the manual potentiometer and a description of the system as it now operates utilizing the automated potentiometer, highlighting the architectural differences. Additionally, a thorough uncertainty analysis will be presented emphasizing the voltage uncertainties and voltage traceability. Finally, the challenges and solutions in transferring the voltage measurement to the thermocouple calibration itself will be presented.

Introduction

Temperature calibrations comprise two distinct paths of traceability and uncertainty. First, there is the actual realization of temperature and degree of thermal equilibrium between the temperature reference and the measurand. Second, there is the electrical measurement of the sensor. Each aspect is distinct and presents different challenges. In our laboratory, the realization of temperature and achievement adequate thermal equilibrium is not generally a problem because we have years of experience in executing low uncertainty temperature measurements and have demonstrated this capability. [1] Applying these methods to thermocouple calibration, and understanding that the laboratory is set up to calibrate SPRTs and the thermocouples of interest are constructed very much like SPRTs, we simply modeled the SPRT process. Once it was demonstrated that this model proved appropriate through a series of tests and experiments, the results were reflected in the uncertainty analysis. [2] There is one significant exception in this analogy, and that is the issue of thermocouple inhomogeneity. This behavior required a series of difficult experiments and involves continuing evaluation. Unfortunately this topic is beyond the scope of this paper. The main challenge resided with the requirements of the electrical measurements necessary for low uncertainty thermocouple calibrations. Thermocouples are voltage generating devices. The discrete voltage values vary between 1.7 mV and 16 mV, and the sensitivities (Seebeck coefficients) vary between 8 uV/°C and 24 uV/°C depending on thermocouple type and temperature. [3, 4] Under inspection, it becomes clear that the voltage measurements can easily become the largest component in low uncertainty thermocouple calibrations. Additionally, the best equipment for making low uncertainty low voltage measurements does not lend itself to efficient high productivity laboratory operation.

Project Goals

The uncertainties listed on our NVLAP Scope of Accreditation [5] are shown in Table 1. These uncertainties represent essentially what we set out to achieve when we began this project.

Temperature	Best Uncertainty (k =2)	T/C Type
0.010 °C (TPW)	0.010 °C	Au/Pt
156.599 °C (FPIn)	0.020 °C	Au/Pt
231.928 °C (FPSn)	0.020 °C	Au/Pt
419.527 °C (FPZn)	0.020 °C	Au/Pt
660.323 °C (FPAl)	0.020 °C	Au/Pt
961.78 °C (FPAg)	0.020 °C	Au/Pt
1000 °C (extrapolated)	0.025 °C	Au/Pt
156.599 °C (FPIn)	0.150 °C	Type S & Type R
231.928 °C (FPSn)	0.150 °C	Type S & Type R
419.527 °C (FPZn)	0.150 °C	Type S & Type R
660.323 °C (FPAl)	0.150 °C	Type S & Type R
961.78 °C (FPAg)	0.150 °C	Type S & Type R

Table 1. Hart Scientific Thermocouple Accredited Uncertainties

The Au/Pt thermocouple uncertainties present more of a challenge than do those of the Type S and R thermocouples. This is only partially due to the voltage measurement. The Au/Pt thermocouple has a higher Seebeck coefficient than that of the others at any given temperature so the voltage measurement is a bit easier with the Au/Pt thermocouple than the others, somewhat offsetting the lower uncertainty demand. However, the main advantage the Au/Pt thermocouple has over the others is that it is less susceptible to traditional thermocouple problems such as preferential oxidation, inhomogeneity, and a component termed inherent instability. If we were able to remove these components from the Type S and R thermocouple analysis, the combined total would drop down to about 0.050 °C, placing the voltage measurement requirements for the Type S and R thermocouples almost on par with that of the Au/Pt thermocouple.

Taking a closer look at the voltage uncertainty requirements reveals the real challenge. Assuming a 50 – 50 split between temperature and voltage uncertainties, we see the Au/Pt thermocouple voltage uncertainties range from 0.25 uV to 0.50 uV. These values are the actual values needed when measuring the thermocouples themselves and must include all of the calibrations, transfers, and traceability leading up to the final measurements.

	Type S and Type R				Au/Pt			
	FPSn	FPZn	FPAl	FPAg	FPSn	FPZn	FPAl	FPAg
Seebeck Coefficient (uV/°C)	8.7	9.2	10.4	11.4	12.6	16.2	20.1	24.9
uV required for < 50% contribution	1.31	1.38	1.56	1.71	0.25	0.32	0.40	0.50

Table 2. Voltage Uncertainty Requirements

Manual DCC Potentiometer Solution

The traditional method for achieving low uncertainty low voltage measurements involves the use of a DC reference (saturated standard cell bank or zener diode stabilized DC reference) and seven or eight dial potentiometer. Manual potentiometers are available in resistive voltage divider (RVD) architecture and direct current comparator (DCC) architecture. Because of the technology and measurement methods employed, the DCC potentiometers are the superior of the two types and the type we chose to use. The most highly refined of these instruments is specified for accuracy at 5 x 10^{-7} of reading and linearity better than 5 x 10^{-8} of full scale. [6]

Figure 1. DCC Potentiometer with Light Beam Coupled Amplifier

The use of a (DCC) potentiometer has several advantages. First, two or more reference voltage devices can be used with one as the traceable reference and the other as the “battery” reference. The value from the traceable reference is transferred to the “battery” reference using the potentiometer itself. The battery reference is used to actually power the potentiometer. Thus, there is always a traceable reference to provide for uninterrupted use when the main reference is out for calibration. Additionally, these DC reference standards can be reversed to verify that the potentiometer is measuring correctly at the ratio of 1. Second, the linearity will be extremely high and "inherently" stable, providing very high accuracy and requiring very few recalibrations. [7] Third, the potentiometer, being a ratio device, requires no external standard for calibration and traceability. Consequently, this instrument can be properly maintained within the laboratory itself. Finally, if used properly, carefully, and with a degree of experience, the measurements results will be extremely good and difficult or impossible to match using any other approach.

However, as with all of our choices, this choice presents several challenges as well. First, a manual potentiometer is slow, tedious, and oftentimes frustrating to operate and requires considerable expertise. The learning curve is longer than with most modern instruments. Second, because of the time involved, multiple measurements are inconvenient and take too much time for meaningful statistics to be applied. Third, also because of the time involved, the voltage being measured must remain extremely stable over the duration of the measurement. Since we are looking at nanovolt resolution in electrically heated furnaces, this is quite difficult to achieve. Fourth, the potentiometer itself is a large delicate instrument and is not portable. It is best left to the corner of the lab out of traffic and away from sources of electrical interference, vibration, and temperature gradients. Fixed point cells are contained in furnaces that often occupy the wall of the laboratory and in areas that create temperature gradients and electrical interference. Additionally, all electrical connections must be made very carefully with low thermal wire; so long signal cable is not advised. Consequently, it is difficult to connect the thermocouple to the potentiometer. Fifth, the best types of potentiometer for this job require several hours of set–up and self measurement (standardization) before they are ready for accurate use. Finally, the light beam coupled amplifier is so sensitive to vibration that proper operation requires that it be physically removed from the potentiometer (it is designed with this feature) and placed in a box filled with sand to attenuate vibration. We found that in our laboratory housed within a factory environment the best results were achieved on the weekend when the majority of factory operations were quiet. Weekend – only calibrations cannot meet demand.

It would seem that the disadvantages outweigh the advantages. Moreover, the goals require both low uncertainties and high productivity. Under the best of circumstances the potentiometer directly could never satisfy the productivity demands, even if the other issues could be overcome. However, the DCC potentiometer has a feature that facilitates a straightforward solution. Not only does the potentiometer measure voltage with low uncertainty, it generates voltage with low uncertainty. Thus, the introduction of a stable high resolution digital voltmeter, characterized by the potentiometer and used for the actual thermocouple measurements, could solve the problem. The traceability and low uncertainties could be obtained through the potentiometer and the actual measurements of the thermocouple could be accomplished using the SDVM. If the correct SDVM was chosen, speed, robustness, statistical analysis, and thermal/electrical immunity could be added to the system. The resulting traceability diagram is shown in Figure 1.

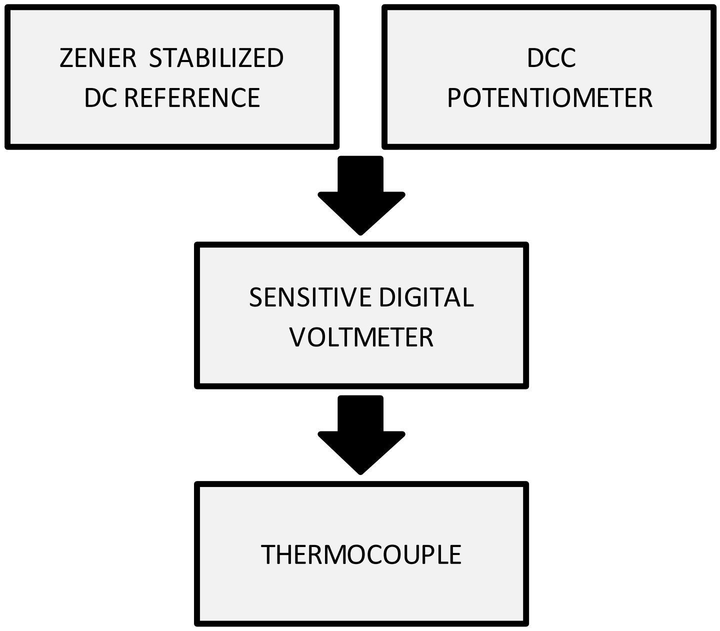

Figure 2. DCC System Traceability Diagram

The DCC potentiometer is standardized and then set to generate voltage on the x0.1 (200 mV) range. Use of the DCC 20 mV range was abandoned in our application for three reasons. First, the 200 mV range provided the accuracy and resolution we required, so spending the extra time to standardize an additional range did not seem like time well spent. Second, we had a second purpose for the sensitive DVM where calibration above 20 mV would be useful on the occasions where we chose to do so. Finally because of sensitivity to vibration and electrical interference, the 20 mV range was extremely difficult to standardize and use, so even on the occasions when we did endeavor to use the 20 mV range the results were only marginally better than the results obtained on the 200 mV range.

Once standardized, the sensitive digital voltmeter (SDVM) is connected to the potentiometer through an ultra low thermal switch using shielded low thermal cables. The switch is a five position push button switch with self wiping silver plated contacts. One of the positions is shorted with a copper wire to serve as the zero reference. Refer to Figure 2.

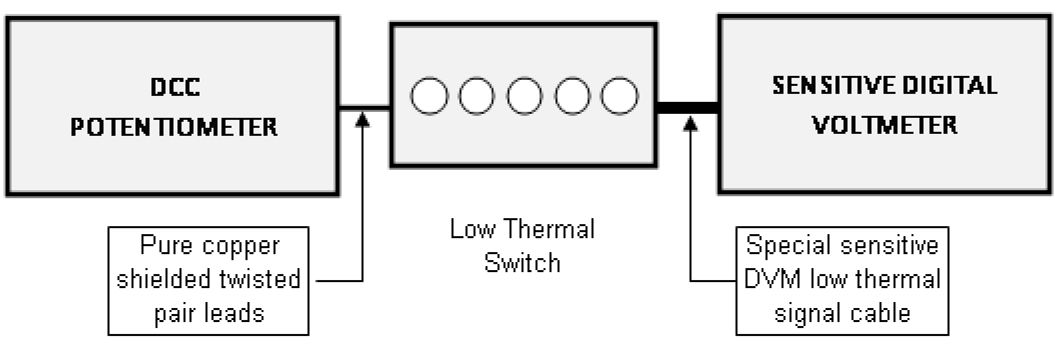

Figure 3. DVM Calibration Schematic

The SDVM can be characterized using two different approaches. Either the nominal (expected) voltage values for the thermocouples being calibrated can be tabulated directly or voltage values at equally spaced intervals can be measured with the results fitted to a best fit straight line (BFSL). The latter approach could cause a bit more uncertainty due to fitting errors if the linearity of the SDVM is less than “perfect.” (Our expectations were that the linearity would be far superior to the manufacturer’s specifications. However we could not quantify the actual linearity nor rely on this expectation until sufficient evidence could be collected.) In our application, we used the nominal voltage values for many calibrations until we understood the linearity performance of the SDVMs.

The characterization of the SDVM is simply a matter of zeroing the instrument, dialing in the discrete voltage values, taking the samples, and recording the averages and standard deviations. No adjustments to the SDVM are made. Once all of the data has been collected it is entered into a spreadsheet and corrections are calculated. These corrections are applied to subsequent measurements taken with the SDVM. Additionally, the change in indication from the previous characterization is calculated and the compared against a tolerance determined by the uncertainty budget and the stability characteristics of the SDVM. The linearity is also evaluated for the reasons described in the previous paragraph. Refer to Table 3 and Figure 4 for examples.

Thermocouple Type	Fixed Point Cell	Nominal Voltage (mV)	Measured Voltage (mV)	Correction (mV)	Fitting Residuals (mV)
Au/Pt	Ag	16.12049	16.120960	-0.000470	0.000022
Au/Pt	Al	9.32044	9.320717	-0.000277	0.000024
Au/Pt	Zn	4.94563	4.945782	-0.000152	0.000026
Au/Pt	Sn	2.23618	2.236252	-0.000072	0.000029
Type S	Ag	9.14838	9.148672	-0.000292	0.000004
Type S	Al	5.86013	5.860327	-0.000197	0.000006
Type S	Zn	3.44689	3.447012	-0.000122	0.000013
Type S	Sn	1.71500	1.715081	-0.000081	0.000006
Type R	Ag	10.00343	10.003804	-0.000374	-0.000054
Type R	Al	6.27709	6.277338	-0.000248	-0.000033
Type R	Zn	3.61130	3.611474	-0.000174	-0.000034
Type R	Sn	1.75623	1.756326	-0.000096	-0.000008

Table 3. Example of SDVM Characterization

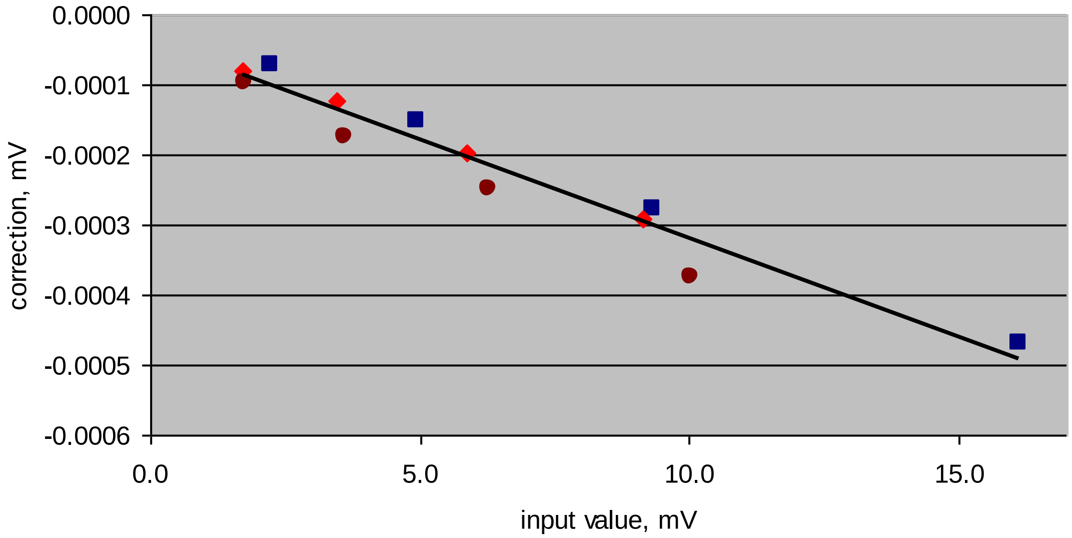

Figure 4. SDVM Characterization Linearity

Uncertainties

Although our expectations were quite high, the uncertainties achieved with this system actually turned out better than expected. The main reasons for this were that the SDVM performed better than anticipated both with regards to stability between calibrations and noise performance. Additionally, the specifications for the low thermal switch turned out to be extremely conservative. One would have to seriously neglect maintenance of the switch to approach the 0.1 uV specification. The uncertainty budget (early example) is shown in Table 4.

Uncertainty Evaluation	**Type S and Type R**				**Au/Pt**				
Type A Evaluation	**FPSn mK**	**FPZn mK**	**FPAl mK**	**FPAg mK**	**TPW mK**	**FPSn mK**	**FPZn mK**	**FPAl mK**	**FPAg mK**
Process variability	4.0	4.0	5.0	5.8	NA	4.0	4.0	5.0	5.8
Precision of measurement	0.1	0.1	0.1	0.1	NA	0.1	0.1	0.1	0.0
Least squares fit for DC transfer standard	0.0	0.0	0.0	0.0	NA	0.0	0.0	0.0	0.0
Reference function (estimated)	7.2	6.8	6.1	5.5	2.3	2.1	1.6	1.3	1.0
Total A	8.3	7.9	7.9	8.0	0.0	4.5	4.3	5.2	5.9
n	49	47	47	49	0	49	47	47	49
Type B Evaluation									
Fixed point value (reference cell certification)	0.3	0.5	0.9	2.0	0.0	0.3	0.5	0.9	2.0
Ice bath system	5.8	5.8	5.8	5.8	1.2	1.2	1.2	1.2	1.2
Hydrostatic head correction	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Non-ideal immersion profile	1.2	1.2	1.2	1.2	0.0	1.2	1.2	1.2	1.2
Inhomogeniety (estimated)	12.3	22.3	35.3	50.0	1.0	1.7	3.0	4.5	6.2
Low thermal switch thermal EMF (0.05μV)	3.3	3.1	2.8	2.5	0.0	2.3	1.8	1.4	1.2
SDVM long term stability (0.1μV)	6.6	6.3	5.6	5.1	0.0	4.6	3.6	2.9	2.3
SDVM calibration (≈ 0.05μV)	3.3	3.1	2.8	2.5	0.0	2.3	1.8	1.4	1.2
Over-determined quadratic fit for UUT	20.0	20.0	20.0	20.0	2.0	2.0	2.0	2.0	2.0
Total B (including fit)	25.5	31.4	41.5	54.6	2.5	6.4	5.9	6.3	7.6
Total Standard Uncertainty (including fit)	26.8	32.4	42.3	55.1	2.5	7.8	7.3	8.1	9.6
Total Expanded Uncertainty (including fit) (*k* =2)	**53.6**	**64.9**	**84.5**	**110.3**	**5.0**	**15.7**	**14.6**	**16.3**	**19.2**

Table 4. Thermocouple Calibration Uncertainty Budget

Troubles Begin

The DCC potentiometer in use was manufactured in the early 1980s and had been in service for about fifteen years when it was purchased by Hart. It had been used in a clean room and was in excellent condition. However it needed minor attention so it was partially reconditioned, aligned, and thoroughly tested by the author. After it was determined that the potentiometer was operating to specification it was placed into service in the thermocouple program. After about five years of continuous use it began to show signs of deterioration. The switches began to get noisy and the standardization process became more and more difficult to accomplish. Eventually it became clear that repairs had to be performed. After evaluation of the problems we came to the conclusion that we only had a few years of additional use before a major overhaul would be necessary. Although the author is qualified to operate and perform alignment and minor repairs on this instrument, major repair and overhaul is another matter entirely and beyond the author's talents. It was at this time that we discovered that there were very few individuals left in the instrument business qualified to perform such an extensive repair/overhaul operation, fewer still were interested in taking on the task Furthermore, none of them were sure if the necessary parts could even be obtained. It was clear that the potentiometer would have to be replaced.

Automated Potentiometer Solution

The only automated potentiometer commercially available at the time was the model 8000A binary voltage divider (BVD) potentiometer manufactured by Measurements International Limited. We were confident with MI instruments because we had experience with their excellent bridges and had a good working relationship with the company. Unfortunately there were two obstacles preventing us from plugging this instrument in as a direct replacement for our aging potentiometer. First, it was designed to work with zener stabilized DC reference voltage standards, not saturated standard cells or thermocouples and is 10 V referenced instrument rather than a 1.018 V referenced instrument Consequently it has resolution limitations when measuring absolute mV; a measurement requiring a large ratio. This is a characteristic of little concern for most applications. Binary voltage divider resolution is a function of the input voltage, range, and effective BVD bit count. When used with the proper detector, the 8000A BVD is a 25+ bit divider. Coupled with a 10 V input, the instrument has a range of 0 to 10 V. Resolution is computed as follows (25 and 26 bit counts shown):

$$resolution = \frac{10V}{2^{25}} = \frac{10V}{33554432} = 2.98 \times 10^{-7}\, V = 298\ nV \tag{1}$$

$$resolution = \frac{10V}{2^{26}} = \frac{10V}{67108864} = 1.49 \times 10^{-7}\, V = 149\ nV \tag{2}$$

The resolution realized would fall between 149 nV and 298 nV. Our uncertainties would require at the minimum an order of magnitude better resolution. The second obstacle in our application is that the 8000A does not generate voltage. If we wanted to go with this solution we would need an intermediate instrument as a voltage source for calibration of our SDVMs. However, the potentiometer was automated, multi–channel, and the specifications were better than the potentiometer our uncertainties were already based on. We were convinced that this potentiometer was the solution but we were unsure at this point how we would design the calibration station.

After several false starts, it became clear that to achieve the performance we required from the potentiometer, we would have to operate it at the voltage levels for which it was designed. Consequently, the introduction of an additional component, a precision voltage divider, would be necessary. A voltage divider with the requisite performance characteristics was not commercially available so one had to be designed and built. The design was developed in collaboration with engineers from the calibration laboratory of our parent company and it was constructed there. Since that time several similar dividers have been built for other projects. The divider is calibrated annually in voltage mode on the JJ system operated at the Fluke PSL. The divider was constructed to optimize long term stability and reduce temperature effects. The resulting traceability diagram is shown in Figure 5.

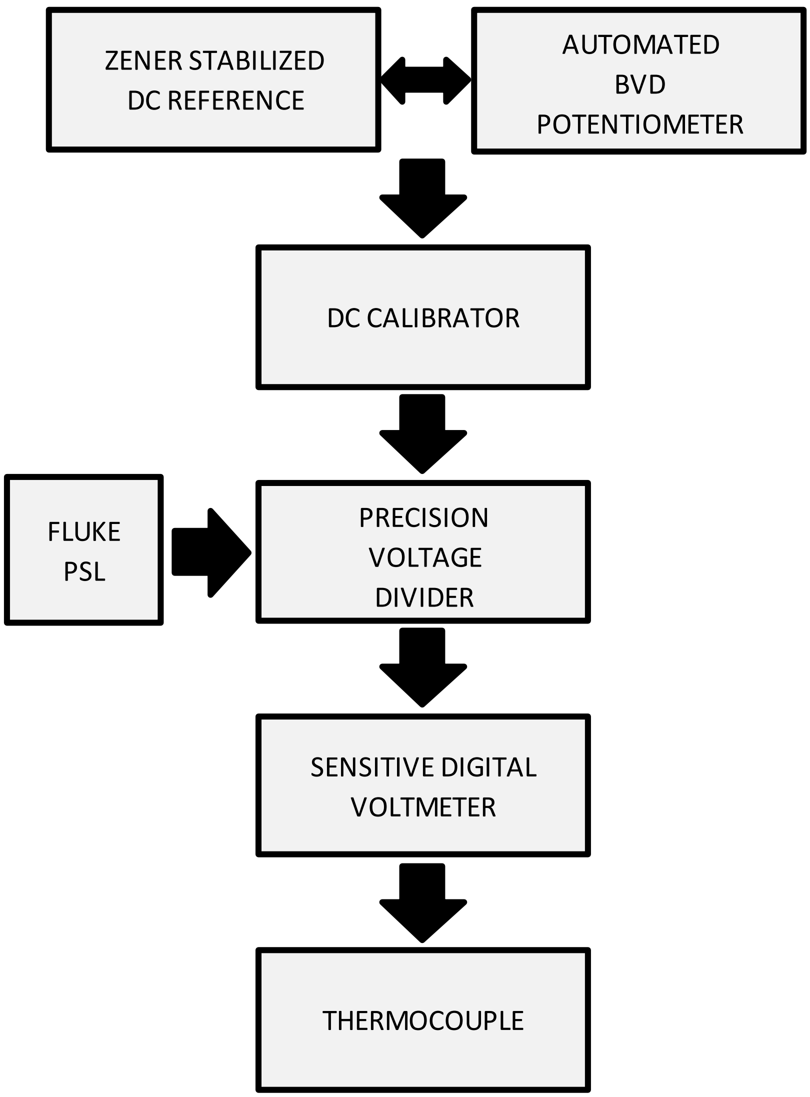

Figure 5. Automated System Traceability Diagram

Operation of the Automated System

Operation of the automated system is more complex than the DCC system because there are more instruments and more transfers. However, much of the operation is automated and software driven. Additionally, the instruments are of modern design and involve digital control. Consequently, the idiosyncrasies of the seven dial DCC potentiometer, along with the incompatibilities within our building have been eliminated. Therefore, the system is far more straightforward and less troublesome to operate.

The system is assembled as shown in Figure 6. The SDVM to be calibrated can be seen beneath the bench top on the left protected from drafts. The long scale DMM (HP3458) functions as the potentiometer detector. The potentiometer software is written to accept a variety of DMMs as detectors; however, the overall performance is affected directly by detector performance. Best results will be obtained when using a long scale DMM. The precision divider is encased in the foam box on the bench top (divider in precarious position for photo only).

Figure 6. Automated System Equipment Set–Up

The system is operated as follows: First, the potentiometer is connected to the external detector, DC source (often referred to as “battery” on older potentiometers), and DC reference according to the operating manual. For best results the instruments should be left to stabilize for 30 minutes to several hours to allow all thermals to settle. The detector is then zeroed using its internal zero routine. Once the zero is established the potentiometer software is programmed to perform the automated calibration/standardization process. This process is used to assign a value to the DC source and mathematically “align” the thirteen BVD stages to achieve the highest linearity possible. As was the case with the DCC potentiometer discussed previously, the BVD potentiometer employs two DC references, one to function as the traceable reference and the other as the stable source reference for the potentiometer circuit. After the standardization process, the traceable reference is disconnected until the next standardization process. The source is in use whenever the potentiometer is in use.

After the calibration/standardization process is complete the potentiometer is ready for use. However, we take the additional step of routinely verifying the results of this process by now using the freshly calibrated/standardized potentiometer to measure both the value of the 10 V output used as the traceable reference in the standardization process and the 1.018 volt output of the DC reference. Since the potentiometer has 20 input channels located on the rear panel, all of the connections were made at the beginning of the calibration/standardization process. Performing the verification sequence is simply a matter of running an additional program macro and evaluating the data. Control charts are kept of these verifications. If the results are outside of the control lines the process will be repeated and/or the cause investigated. Refer to Figure 7.

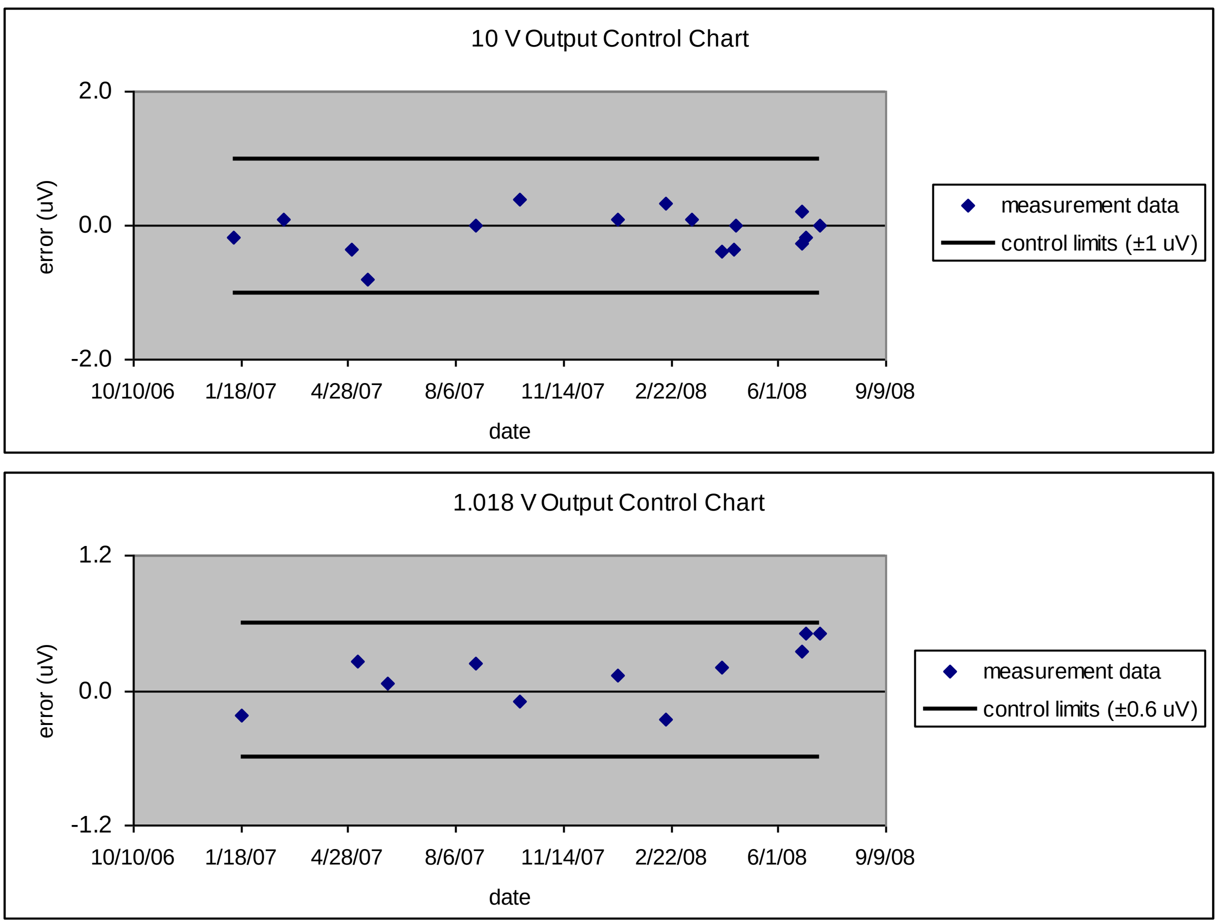

Figure 7. Potentiometer Calibration/Standardization Verification Control Charts

After the control charts indicate that the calibration/standardization process was successful, the process of characterizing the DC calibrator can begin. The DC calibrator is characterized from 0 to 2 VDC at 0.2 V intervals or 0 to 10 VDC at 1 V intervals, depending on the application. This process is semi automated in the respect that the potentiometer process is automated but the DC calibrator settings must be manually set through the front panel keypad or stepped through using the memory function. Consequently, the process can run automatically but the metrologist must remain in close proximity to keep it progressing. The potentiometer software records the measured data at each point for use later so there is no need for the metrologist to be bothered with data collection. The DC calibrator performance is evaluated in several ways. First, the linearity of the data itself is checked. The DC calibrator should have excellent linearity characteristics and the potentiometer linearity is well beyond the noise limits of the measurement. Consequently, any significant departure from linearity is a warning sign that a problem exists either with the calibration or one of the instruments. Second, the data is verified for short term stability by comparing the new data with the previous characterization data. This information reveals how the DC calibrator performs between characterizations and directly influences the uncertainty budget. Finally, the long term behavior of the DC calibrator is evaluated. As the data accumulates trends develop and the calibrator's behavior begins to become predictable. This information can be used to adjust calibration intervals and possibly (positively) impact the uncertainty budget. These evaluations do not include control limits because strict statistical limits have not been established. Rather, these evaluations are used to provide insight into the stability of the system and as an internal quality control measure. Refer to Figures 8 through 10 for examples of these types of evaluations.

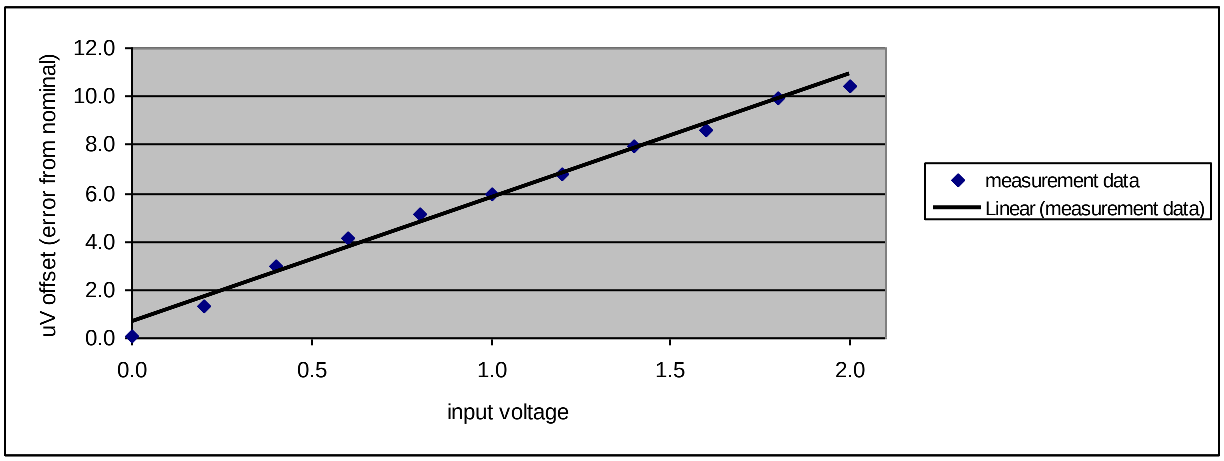

Figure 8. DC Calibrator Linearity Verification Example

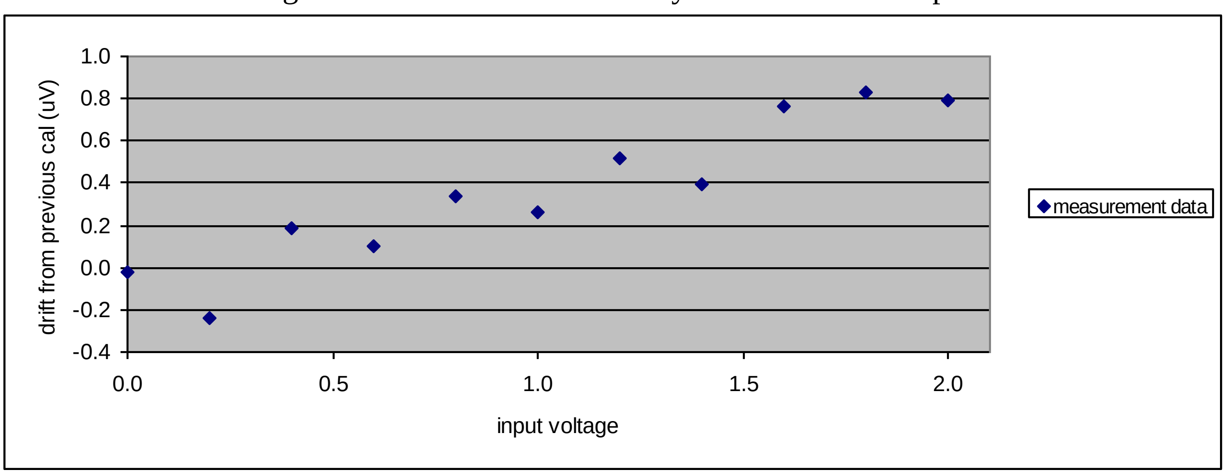

Figure 9. DC Calibrator Short Term Stability Verification Example

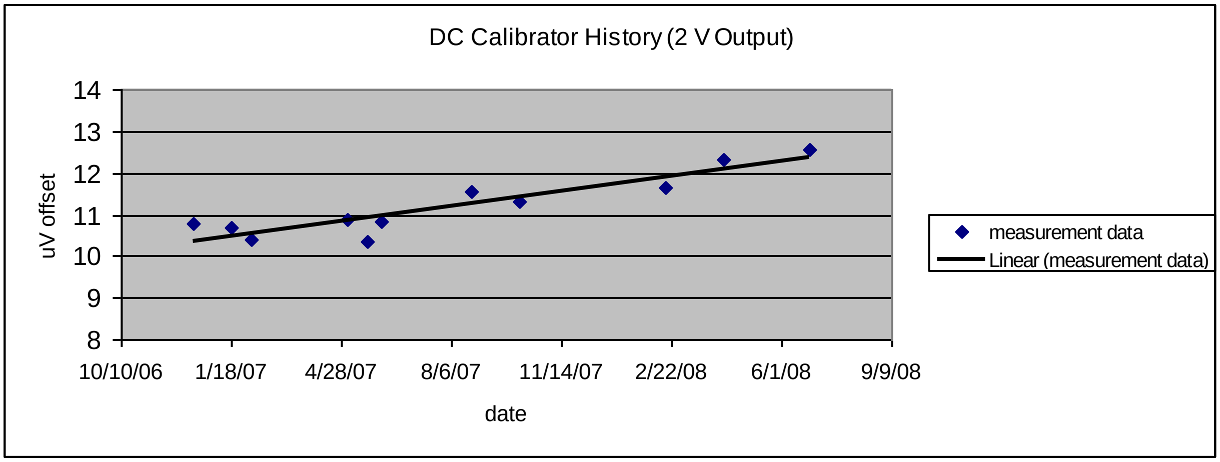

Figure 10. DC Calibrator Long Term Stability Verification Example

Once the DC calibrator has been characterized, it is disconnected from the potentiometer and then it can be used for up to five days before re-characterization is again necessary. When this is done it is prudent, but not absolutely necessary, to perform a closing characterization.

Calibration of the SDVM proceeds as follows: The DC calibrator is connected to the input of the precision voltage divider. The input of the SDVM is cleaned and connected to the output of the precision voltage divider using its special input cable. Finally, the precision voltage divider is placed inside the foam box along with the monitoring thermometer. The whole system is allowed to stabilize. Refer to Figure 11.

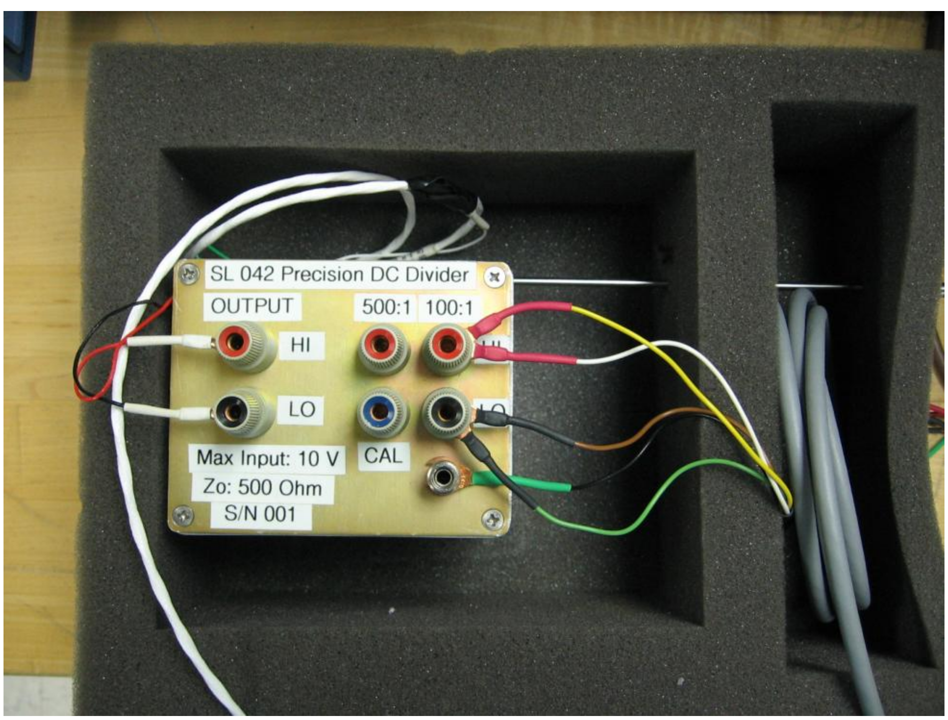

Figure 11. Connections to Precision Voltage Divider

The DC Calibrator is then set to four wire mode and programmed for 0.0 VDC. The SDVM is set to the proper measurement configuration and the zero measurement is allowed to stabilize. After stabilization, the SDVM is nulled and the eleven discrete voltage values are measured in sequence. Data and statistics are collected and entered directly into the spreadsheet. The spreadsheet is used at this stage to check the data for self consistency. If the data is acceptable the SDVM disconnected and the next SDVM can be calibrated. Once the calibrations are complete the data can be evaluated.

Data Evaluation

The SDVMs are used only on the 100 mV range, only for thermocouple applications, and never outside of a controlled laboratory environment. Consequently, they have proven to be far more stable than the specifications suggest. After they have been characterized, the data is evaluated for linearity and stability. Since these instruments are used directly in calibration processes, the drift evaluation has limits that are used to determine tolerance status like any other calibrated instrument. The limits are evaluated at the discrete voltage values representative of the thermocouple output voltage values at the fixed points. Refer to Table 5.

DATA						
TC Type	FP	Nominal emf (mV)	Previous emf (mV)	Computed emf (mV)	U (k=2) (nV)	Error (mV)
Au/Pt	Tin	2.23618	2.23616	2.23619	38	0.00001
	Zinc	4.94563	4.94561	4.94564	31	0.00001
	Aluminum	9.32044	9.32043	9.32045	24	0.00001
	Silver	16.12049	16.12049	16.12050	34	0.00001
Type S	Tin	1.71500	1.71498	1.71501	40	0.00001
	Zinc	3.44689	3.44687	3.44690	35	0.00001
	Aluminum	5.86013	5.86011	5.86014	29	0.00001
	Silver	9.14838	9.14837	9.14839	24	0.00001
	Copper	10.57480	10.57479	10.57481	24	0.00001
Type R	Tin	1.75623	1.75621	1.75624	40	0.00001
	Zinc	3.61130	3.61128	3.61131	34	0.00001
	Aluminum	6.27709	6.27707	6.27710	28	0.00001
	Silver	10.00343	10.00342	10.00344	24	0.00001
	Copper	11.64043	11.64042	11.64044	25	0.00001

Drift Evaluation					
TC Type	FP	Drift (uV)	Drift (°C)	Drift Tolerance (uV)	Tolerance Status
Au/Pt	Tin	0.029	0.0023	0.100	P
	Zinc	0.025	0.0015	0.100	P
	Aluminum	0.018	0.0009	0.100	P
	Silver	0.007	0.0003	0.100	P
Type S	Tin	0.030	0.0037	0.100	P
	Zinc	0.027	0.0029	0.100	P
	Aluminum	0.023	0.0022	0.100	P
	Silver	0.018	0.0016	0.100	P
	Copper	0.016	0.0013	0.100	P
Type R	Tin	0.030	0.0032	0.100	P
	Zinc	0.027	0.0025	0.100	P
	Aluminum	0.022	0.0020	0.100	P
	Silver	0.017	0.0014	0.100	P
	Copper	0.014	0.0010	0.100	P

Table 5. Example of SDVM Calibration Evaluation

In addition to the drift analysis and tolerance status evaluation, the linearity is evaluated for the same reasons as those discussed for the DC calibrator. An example is shown in Figure 12.

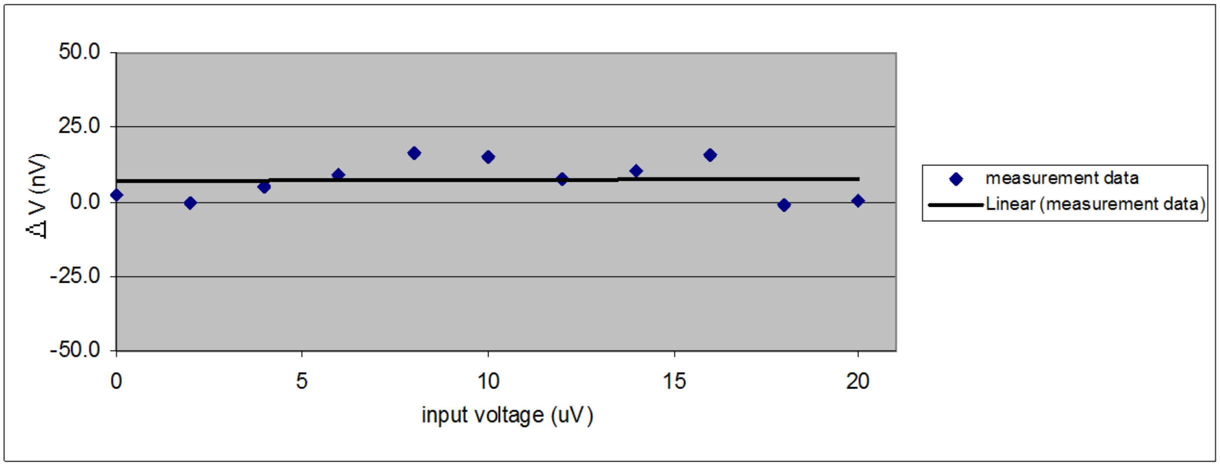

Figure 12. Example of SDVM 0 to 20 mV Linearity

Uncertainty Evaluation

Originally, calibration of the SDVM was part of the thermocouple calibration instruction and the uncertainty budget was contained within the thermocouple uncertainty budget. Now, due to both the complexity of the SDVM calibration and the number of SDVMs requiring calibration for other applications, this process now has a distinct calibration instruction an individual uncertainty budget. Refer to Table 6.

The thermocouple uncertainty budget has also evolved. Due to work by NIST and others, it is now understood that the reference function for the thermocouples addressed in the calibration does not perfectly fit the ITS-90. [8] This issue had to be addressed with the inclusion of an addition component of uncertainty.

Similarly, although the effects of thermocouple inhomogeneity are not new to the thermocouple community, precise methods for quantification that can be generalized to calibration procedures have only recently been published. As a result, supplementary tests have been added to our thermocouple calibration instruction to quantify these effects for each thermocouple being calibrated [9]. A line has been added to the uncertainty budget to account for this component.

Finally, a traditional interpretation of a GUM compliant uncertainty evaluation addresses uncertainties present during the calibration itself, not during subsequent use of the calibrated device. [10] This issue has been a source of contention among the precision thermometer community (SPRT, thermocouple, and thermistor) for some time. Type S and R thermocouples are understood to be somewhat unstable as they age and as they experience temperature cycling. Previously, this component has been left up to the user to apply during use of the thermocouple. However, some metrologists are of the opinion that the uncertainty budgets should be different for new "pristine" instruments and used instruments because the results will be different during use even if the results in the laboratory during calibration are identical. The thermocouple community as a whole is beginning to see the wisdom of this point of view. The component called "inherent instability is an attempt to quantify this behavior. The limits in the uncertainty budget were determined through analysis of the many examples of repeated calibrations of Type S and R thermocouples using both external customer owned and internal used devices. Consequently, the uncertainty budget now has two sets of totals detailing the calibration uncertainties. One set of totals omitting the effects of inhomogeneity and inherent instability and one set of totals including them. Refer to Table 7. Both totals along with explanatory information are reported on the report of calibration.

Voltage Applied (mVdc)	**B** (nVdc)	**C** (nVdc)	**D** (nVdc)	**E** (nVdc)	**F** (nVdc)	**G** (nVdc)	**H** (nVdc)	**I** (nVdc)	**J** (nVdc)	**K** (nVdc)	**L** (nVdc)	**M** (nVdc)	**Combined Uncertainty** (nVdc)	**Expanded Uncertainty** (nVdc)
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.42	17.32	2.89	17.62	35.23
2	0.10	0.87	0.12	1.15	4.00	0.37	0.60	1.15	0.17	1.42	17.32	2.89	18.17	36.35
4	0.20	1.73	0.23	2.31	7.99	0.74	1.20	2.31	0.35	1.42	17.32	2.89	19.75	39.50
6	0.30	2.60	0.35	3.46	11.99	1.11	1.80	3.46	0.52	1.42	17.32	2.89	22.13	44.26
8	0.40	3.46	0.46	4.62	15.98	1.49	2.40	4.62	0.69	1.42	17.32	2.89	25.08	50.17
10	0.50	4.33	0.58	5.77	19.98	1.86	3.00	5.77	0.87	1.42	17.32	2.89	28.44	56.87
12	0.60	5.20	0.69	6.93	23.97	2.23	3.60	6.93	1.04	1.42	17.32	2.89	32.06	64.12
14	0.70	6.06	0.81	8.08	27.97	2.60	4.20	8.08	1.21	1.42	17.32	2.89	35.87	71.75
16	0.80	6.93	0.92	9.24	31.96	2.97	4.80	9.24	1.39	1.42	17.32	2.89	39.82	79.65
18	0.90	7.79	1.04	10.39	35.96	3.34	5.40	10.39	1.56	1.42	17.32	2.89	43.87	87.74
20	1.00	8.66	1.15	11.55	39.95	3.71	6.00	11.55	1.73	1.42	17.32	2.89	47.99	95.99

All "ppm" uncertainites have been converted to voltages in "nVdc". The "Expanded Uncertainty" is k=2.

Column Descriptions

B Fluke Everett PSL calibration uncertainty on 732B. Identified on cal certificate as "0.1 ppm" and represented here as "0.05 ppm, (k=1)" (distribution: normal).

C Hart observed yearly drift over 10 year history. Identified as 0.75 ppm/yr, but due to rectangular nature, it is represented here as "0.75 ppm/yr/$(3)^{1/2}$, (k=1)" (distribution: presumed rectangular).

D MINTL 8000A "calibration/standardization" routine uncertainty". Identified as 0.1 ppm, and represented as "0.1 ppm/yr/$(3)^{1/2}$, (k=1)" (distribution: presumed rectangular).

E MINTL 8000A "software reported uncertainty" per measurement of the 5440B characterization. Identified as 1.0 ppm (worst case at 200 mVdc). It is represented here as "1.0 ppm/(3)1/2, (k=1)" (distribution: presumed rectangular).

F Procedural limit for drift allowed between opening and closing characterization values of the 5440B. Identified as 3.46 ppm (worst case at 200 mVdc). It is represented here as "3.46 ppm/(3)1/2, (k=1)" (distribution: presumed rectangular).

G Procedural limit for measurement precision during characterization of 5440B. Uncertainty value determined at worst case (200 mVdc), n=30 samples . "1.0 ppm/$(n-1)^{1/2}$, (k=1)" (distribution: normal).

H Fluke Everett PSL calibration uncertainty on the 100:1 Portion of the Fluke SL 042. Identified on cal certificate as "0.6 ppm" and represented here as "0.3 ppm, (k=1)" (distribution: normal).

I Hart Tolerance for yearly drift. This is 1.0 ppm/yr, but due to rectangular nature, it is represented here as "1.0 ppm/yr/$(3)^{1/2}$, (k=1)" (distribution: presumed rectangular).

J Divider temperature effects. Uncertainty of monitoring thermometer of ±0.1 °C applied to the divider's temperature compensation specification. The ±0.1 °C uncertainty yields a ±0.15 ppm uncertainty in the 100:1 divider ratio. Represented as "0.15 ppm/yr/(3)1/2, (k=1)" (distribution: presumed rectangular).

K Procedural limit for measurement precision of the 2182 SDVM (*n* = 200) during characterizarion of the 2182 SDVM. Identified as "20 nVdc/$(n-1)^{1/2}$, (k=1)" (distribution: normal).

L Procedural limit for "zero drift" of the 2182 SDVM during characterization. Based on observation and evaluated at worst case. Identified as "30 nVdc/$(3)^{1/2}$, (k=1)" (distribution: presumed rectangular).

M 2182 SDVM resolution uncertainty. The minimum 2182 resolution is 10 nVdc, rectangular in its effect, and equal to "10 nVdc/$(12)^{1/2}$, (k=1)" (distribution: presumed rectangular).

Table 6. SDVM Characterization Uncertainty Budget

Uncertainty Evaluation	Type S and Type R				Au/Pt				
Type A Evaluation	**FPSn mK**	**FPZn mK**	**FPAl mK**	**FPAg mK**	**TPW mK**	**FPSn mK**	**FPZn mK**	**FPAl mK**	**FPAg mK**
Process variability as observed by check standard	6.5	4.8	5.6	6.1	NA	6.5	4.8	5.6	6.1
Precision of measurement (procedure limit n = 200)	0.1	0.1	0.1	0.1	NA	0.1	0.1	0.1	0.1
Uncertainty in reference function (estimated)	7.2	6.8	6.1	5.5	2.3	2.1	1.6	1.3	1.0
Uncertainty due to inherent instability of Type S & R	35.0	35.0	35.0	35.0	NA	NA	NA	NA	NA
Total A (omitting inherent instability)	**9.7**	**8.4**	**8.3**	**8.2**	**2.3**	**6.8**	**5.1**	**5.7**	**6.2**
Total A' (including inherent instability)	**36.3**	**36.0**	**36.0**	**36.0**	**NA**	**NA**	**NA**	**NA**	**NA**
n	48	46	46	47	0	48	46	46	47
Type B Evaluation									
Fixed point value (reference cell certification)	0.3	0.5	0.9	2.0	0.0	0.3	0.5	0.9	2.0
Ice bath system	5.8	5.8	5.8	5.8	1.2	1.2	1.2	1.2	1.2
Hydrostatic head correction	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Non-ideal immersion profile	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Temperature Uncertainties	**5.9**	**5.9**	**6.0**	**6.2**	**1.6**	**1.7**	**1.7**	**1.8**	**2.6**
Low thermal switch thermal EMF (0.04μV)	2.7	2.5	2.2	2.0	0.0	1.8	1.4	1.1	0.9
Sensitive DVM long term stability (0.1μV)	6.6	6.3	5.6	5.1	0.0	4.6	3.6	2.9	2.3
Sensitive DVM calibration (≈ 0.05μV)	5.7	5.4	4.8	4.4	8.3	4.0	3.1	2.5	2.0
Over-determined quadratic fit for TC under test	20.0	20.0	20.0	20.0	2.0	2.0	2.0	2.0	2.0
Inhomogeneity	27.0	51.4	77.1	110.0	0.0	4.0	6.9	10.5	14.7
Total B (omitting homogeneity)	22.8	22.6	22.2	22.1	8.7	6.8	5.6	4.8	4.6
Total B' (including homogeneity)	35.3	56.1	80.3	112.2	8.7	7.9	8.9	11.6	15.4
U (omitting inherent stability and homogeneity)	24.8	24.1	23.7	23.6	9.0	9.7	7.5	7.5	7.7
U' (including inherent stability and homogeneity)	50.7	66.7	88.0	117.8	9.0	10.5	10.2	12.9	16.6
U (*k* =2) (omitting inherent stability and homogeneity)	**49.5**	**48.2**	**47.4**	**47.1**	**18.0**	**19.3**	**15.1**	**15.0**	**15.4**
U' (*k* =2) (including inherent stability and homogeneity)	**101.4**	**133.3**	**175.9**	**235.6**	**18.0**	**21.0**	**20.5**	**25.8**	**33.1**

Table 7. Thermocouple Calibration Uncertainty Budget

Laboratory Intercomparison

For verification that the measurement system was operating correctly and that the results were as expected, it was determined that a laboratory intercomparison would be necessary. Two SDVMs were used and two ranges were verified; 0 to 20 mV (the thermocouple range) and 0 to 100 mV (the maximum range of this system). The Fluke PSL staff had been extremely helpful throughout the development process and helped us several times when we needed support with our SDVM calibrations in the past. They graciously agreed to participate in this comparison; the system used for the calibration was the JJ system used as the basis for the Fluke volt. Although the JJ uncertainties are extremely low, the uncertainties for the SDVM calibration are only just comparable to our potentiometric system. The results of the comparison are shown in Tables 8 and 9.

Nominal Voltage (mV)	Hart Data □ nV	Hart U (k =2) nV	Fluke Data □ nV	Fluke U (k =2) nV	Difference (Hart – Fluke)	E_n
0	7.4	35.2	-11	80.0	18.4	0.210
2	4.7	36.1	-8	80.0	12.7	0.145
4	-4.1	38.6	-28	80.0	23.9	0.269
6	-4.7	42.4	-33	80.0	28.3	0.313
8	2.8	47.3	-20	80.0	22.8	0.246
10	-0.8	52.9	-18	80.0	17.2	0.179
12	-9.3	59.0	-8	80.0	1.3	0.013
14	-11.7	65.5	3	80.0	14.7	0.142
16	-12.6	72.3	2	80.0	14.6	0.136
18	-25.1	79.2	3	80.0	28.1	0.250
20	-19.1	86.4	17	80.0	36.1	0.307

Table 8. 20 mV Range Interlaboratory Comparison Results

Nominal Voltage (mV)	Hart Data □ nV	Hart U (k =2) nV	Fluke Data □ nV	Fluke U (k =2) nV	Difference (Hart – Fluke)	E_n
0	5.5	35	41	100.0	35.5	0.335
10	149.0	43	182	100.0	33.0	0.303
20	274.5	61	324	100.0	49.5	0.423
30	408.7	83	466	100.0	57.3	0.441
40	538.4	106	609	100.0	70.6	0.484
50	670.6	130	748	100.0	77.4	0.472
60	804.1	153	885	100.0	80.9	0.443
70	944.0	178	1030	100.0	86.0	0.421
80	1018.8	202	1170	100.0	151.2	0.671
90	1130.5	227	1314	100.0	183.5	0.740
100	1289.3	252	1441	100.0	151.7	0.559

Table 9. 100 mV Range Interlaboratory Comparison Results

The results of both comparison experiments show very good results and do not require further investigation. On the 0 to 20 mV range, the E_n values fall below 0.5 in all cases, indicating excellent agreement between the two laboratories. On the 0 to 100 mV range, the E_n values fall below 0.5 in all but three cases and never exceed the cutoff of 1.0. However, for reasons described elsewhere, it is the policy of this laboratory to investigate E_n results between 0.5 and 1.0. [11] Consequently, as time permits, the 0 to 100 mV range comparison range will be repeated. The overall excellent results indicate that this is not urgent.

Conclusion

The purpose of this paper has been to outline the development of the DC measurement system involved in the thermocouple calibration program at Hart Scientific. Through this work we have shown the evolution from the traditional DCC potentiometer based system through to the modern automated BVD potentiometer. Traceability diagrams have been presented which outline the traceability from our service supplier for DC voltage (our parent company, Fluke PSL) to our eventual calibration of thermocouples. Additionally, several uncertainty budgets have been shown which present, in detail, the uncertainties involved in the calibration process leading from our initial calibrations to the final measurements involved in thermocouple calibration. Finally, laboratory intercomparison results have been presented which show excellent agreement between the Fluke PSL JJ system and the Hart Scientific BVD potentiometer system.

Acknowledgements

The author would like to thank Neil Faulkner at Fluke PSL for his help with the design and construction of precision voltage divider and Tom Kolat for his work on the BVD potentiometer calibration system and uncertainty analysis.

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