10 TΩ and 100 TΩ High Resistance Measurements at NIST

Dean G. Jarrett[*] and Marlin E. Kraft[*]

[*]National Institute of Standards and Technology[1]
Dean.Jarrett@nist.gov

***Abstract* — The measurement techniques, standards, and bridges used to calibrate standard resistors in the 10 TΩ to 100 TΩ range at NIST are described. Standard resistors, guarded Hamon transfer standards, and 10:1 and 100:1 bridge ratios, were used to provide multiple paths from 1 TΩ to the higher levels of resistance. Settling times, voltage coefficients, drift rates, and temperature coefficients of the resistance standards were determined to evaluate the extension of high resistance measurements at NIST beyond the 1 TΩ level of resistance.**

Keywords — standard resistor, guarded Hamon transfer standard, settling time, scaling, measurements.

I. Introduction

The measurement of high resistance standards at NIST has evolved over the past two decades from manual and semi-automated measurement systems to several fully automated dual-source bridges with automated guarded scanners for connecting standard resistors to the measurement systems [1]. The standard resistors and guarded Hamon transfer standards have also been improved during this same period of time by designing and building resistors with improved stability that are less susceptible to variations in environmental and test conditions than standards that were used at NIST two decades ago [2-3]. Other advances have included the development of a high resistance cryogenic current comparator (HR-CCC) for scaling over the range 1 MΩ to 1 GΩ and the expanded use of temperature controlled chambers for high resistance standards [4]. NIST routinely provides high resistance measurement services over the range 10 MΩ to 1 TΩ. Measurements above 1 TΩ are more susceptible to leakage currents to ground, large RC time constants, and resistance standards with larger temperature coefficients of resistance (TCR) on the order of 100×10^{-6}/°C.

In response to customer needs and requests, NIST has resumed its efforts to make high resistance measurements in the 10 TΩ to 100 TΩ range. A dual-source bridge with coaxial automated switching was used for these measurements along with guarded Hamon transfer standards as well as single-element resistance standards. Evaluation of the bridge, the standards, and the scaling path from lower values of resistance was done to verify the results. Bridge ratios and guarded Hamon transfer standards were used to scale to the 10 TΩ level and multiple bridge ratios were used to scale to the 100 TΩ level. Settling time and voltage coefficient measurements of all standards were evaluated to determine short-term steady state measurement conditions. Control charts were used to evaluate the long-term stability of the transfer and check standards used at these levels.

II. METHODS

A. Build-up to 1 TΩ

The process of scaling or building-up to 1 TΩ from the quantum Hall resistance (QHR) $i = 2$ plateau (12.9 kΩ) is a long process involving multiple measurement systems, standards, check standards, and transfer standards. The hundreds of standard resistors calibrated each year at NIST make it impractical to repeat this process more often than two to three times a year. Control charts are maintained on

[1] Quantum Measurement Division, Gaithersburg, MD. NIST is part of the U.S. Department of Commerce. Official contribution of the National Institute of Standards and Technology; not subject to copyright in the United States.

multiple standards and check standards at each nominal value of resistance. A linear regression for each resistor is used to establish a drift rate which defines a predicted value as a function of time. The predicted values for standards and check standards are used during the time-interval after a scaling to disseminate the NIST ohm at each resistance level.

The first step in the scaling process from the QHR is done using the HR-CCC to scale directly from the QHR to 100 kΩ and 1 MΩ using 310:40 and 3100:40 winding ratios, respectively. The HR-CCC also has a 31 turn and a second 310 turn winding allowing winding ratios of 1:1, 10:1, and 100:1 that can then be used throughout the high resistance range of 1 MΩ to 1 GΩ as shown in Fig. 1. The HR-CCC range overlaps the lower decades of the dual source bridge's range of 1 MΩ to 100 TΩ. Both systems are used from 1 MΩ to 1 GΩ to provide the best balance of traceability, throughput, and robustness. Guarded Hamon transfer standards are also used in this range and up to 1 TΩ for scaling and as primary standards for the dual source bridge.

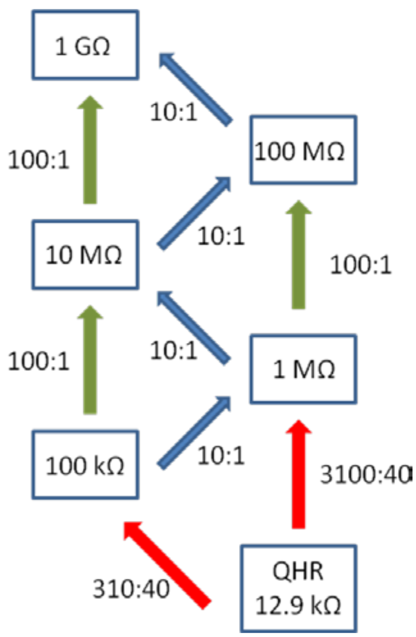

Fig.1. Scaling paths from the quantum Hall resistance (QHR) to the start of the high resistance range. High resistance cryogenic current comparator (HR-CCC) used to transfer from the *i = 2* plateau of 12.906 kΩ to high resistance standards at 1 MΩ to 1 GΩ. Red arrows show scaling paths from the QHR to decade resistance levels of 100 kΩ and 1 MΩ using 310:40 and 3100:40 winding ratios, respectively. Blue and green arrows show scaling paths to 1 MΩ through 1 GΩ decades of resistance using the 10:1 and 100:1 HR-CCC ratios.

Once high resistance standards are calibrated by the HR-CCC, the dual source bridge and guarded Hamon transfer standards are used to complete the next step of the scaling from 1 MΩ to 1 TΩ as shown in Fig. 2. A guarded Hamon transfer standard has ten resistance elements of the same nominal value that can be connected in three configurations: parallel, series-parallel, and series. The three configurations are at three sequential nominal values (i.e. a 10 x 10 MΩ transfer standard can be configured as 1 MΩ, 10 MΩ, and 100 MΩ) providing the 10:1 and 100:1 ratios to scale to higher ranges. NIST has at least one guarded Hamon transfer standard with a series configuration at each nominal value up to 10 TΩ that can be used during high resistance scaling. The redundancy of multiple paths with multiple standards provides confidence in the build-up to 1 TΩ. At each decade of resistance, three guarded Hamon transfer standards of sequential nominal values and different configurations are used, that is, at least one in series configuration, at least one in series-parallel configuration, and at least one in parallel configuration. The substitution technique is used to assign resistance values to the parallel configured guarded Hamon transfer standard from the series and series-parallel configured guarded Hamon transfer standards. This technique allows standard and test resistors of the same nominal value to be indirectly compared by substituting the standards into the same arm of a bridge circuit, which helps cancel errors due to ratio non-linearity, leakage currents, and contact resistances.

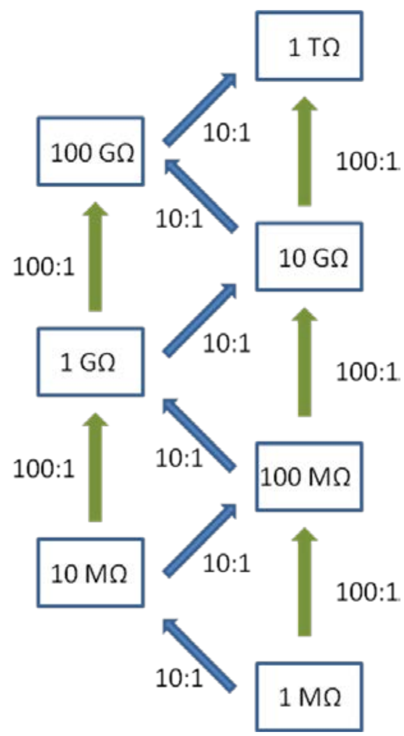

Fig.2. Scaling paths for high resistance measurements ranging from 1 MΩ to 1 TΩ. Guarded Hamon transfer standards used with dual source bridge to provide multiple paths to each higher decade of resistance. Blue arrows are 10:1 ratio of guarded Hamon transfer standard from series-parallel configuration to series configuration and green arrows are 100:1 ratio of guarded Hamon transfer standard from parallel configuration to series configuration.

Once a build-up is complete, control charts on the guarded Hamon transfer standards and check standards are updated and used until the next scaling to assign time-dependent predicted values based on the linear regression of the scaling measurements. Figure 3 shows the regression for one of the 1 GΩ guarded Hamon transfer standards. The regression line is the average of both parallel and series-parallel measurements (●) made at 10 MΩ and 100 MΩ, respectively. Error bars for the average of both parallel and series-parallel measurements are the standard deviation for both configurations over several days. For clarity of the plot, error bars for the parallel (◆) and series-parallel (■) measurements are not shown.

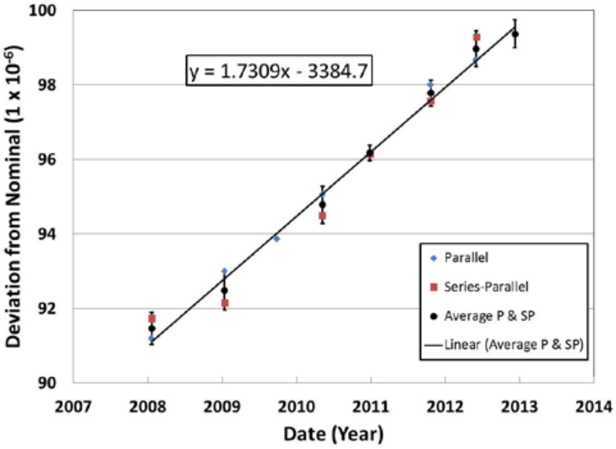

Fig.3. Control chart for one of the guarded Hamon transfer standards. A least-squares regression for data from most recent scaling measurements is used to determine a predicted value for the standards and check standards between each build-up to 1 TΩ.

B. Build-up to 10 TΩ and 100 TΩ

High resistance measurements beyond 1 TΩ have been worked on at NIST over the past decade. Building standard resistors and guarded Hamon transfer standards has been done at NIST for series resistances up to 100 TΩ. As resistance increases, so does the RC time constant, requiring longer settling times for the resistance standard to reach a steady state. Guarded Hamon transfer standards have been built at NIST to be used at 10 TΩ and 100 TΩ. The settling times of these standards have been evaluated by measurements made in the three configurations and by measuring the ten individual resistance elements that make up these guarded Hamon transfer standards. Recent analyses of both the 10 TΩ and 100 TΩ guarded Hamon transfer standards have been used to evaluate their suitability for scaling from 1 TΩ to 100 TΩ [3].

Since 2006, the 10 TΩ guarded Hamon transfer standard has been routinely measured in parallel (100 GΩ) and series-parallel (1 TΩ) modes to assign a value to the series (10 TΩ) resistance. The drift rate for this transfer standard is less than 80 x 10^{-6}/year. Measurement of the ten individual 1 TΩ resistance elements that are in the guarded Hamon transfer standard yielded a value that agreed within 50 x 10^{-6} of the mean value from the parallel and series-parallel modes. Both of these results are quite acceptable for a transfer standard to be used at 10 TΩ, however, an analysis of the settling time for the series configuration of this guarded Hamon transfer standard revealed that at least 700 s was required for this transfer standard to reach a steady state. By contrast, single element resistance standards at 10 TΩ can reach steady-state in 300 s. Figure 4 shows the resistance as a function of time for the 10 TΩ guarded Hamon transfer standard and a single element 10 TΩ check standard using a 700 s settling time. Four measurements of alternating polarity are shown for each resistor.

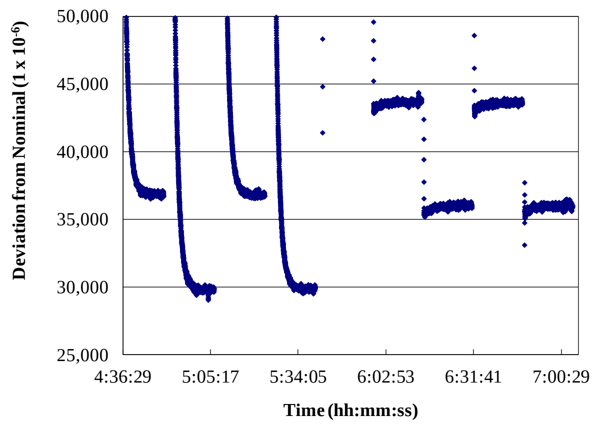

Fig.4. Settling times for two 10 TΩ resistors with a 700 s settling time. The guarded Hamon transfer standard measurements are on the left and measurements of a single-element check standard are on the right. A sequence of four measurements of alternating polarity are shown for each resistor.

Even with an automated bridge and an automated XY positioning system to connect resistors to the bridge, settling times beyond 300 s inhibit the capacity to make repeated measurements of standards, check standards, and unknown resistors at multiple voltages and test conditions by extending a set of measurements beyond 24 h. The long settling time also makes such guarded Hamon transfer standards unsuitable for use in the lower resistance arm of the dual source bridge. The solution to balancing throughput and stability was to only use the 10 TΩ guarded Hamon transfer standard during the scaling to calibrate single-element standard resistors as shown at the left of Fig. 5. In 300 s, the single-element transfer standard reaches a steady-state value that is within 80 x 10^{-6} of the steady-state value at 700 s so it can be calibrated against the 10 TΩ guarded Hamon transfer standard with a voltage applied for 700 s and then used to calibrate other 10 TΩ and 100 TΩ standards using a 300 s settling time.

A recent analysis of both 100 TΩ guarded Hamon transfer standards at NIST found that neither standard reached a steady-state, even after the test voltage was applied for up to 1800 s. The short-term drift was 150 x 10^{-6}/s. Measurement of the individual 10 TΩ resistance elements found that each guarded Hamon transfer standard had several individual resistors that would not reach a steady state, even with an 1800 s settling time, so those guarded Hamon transfer standards were rejected for scaling to 100 TΩ. To reach the 100 TΩ level without a 100 TΩ series-configured guarded Hamon transfer standard would require reliance on the dual source bridge ratios of 100:1 and 10:1 to scale from 1 TΩ and 10 TΩ, respectively. Figure 5 shows the two paths to 100 TΩ which use single-element transfer standards calibrated by the 1 TΩ and 10 TΩ guarded Hamon transfer standards to complete the build-up.

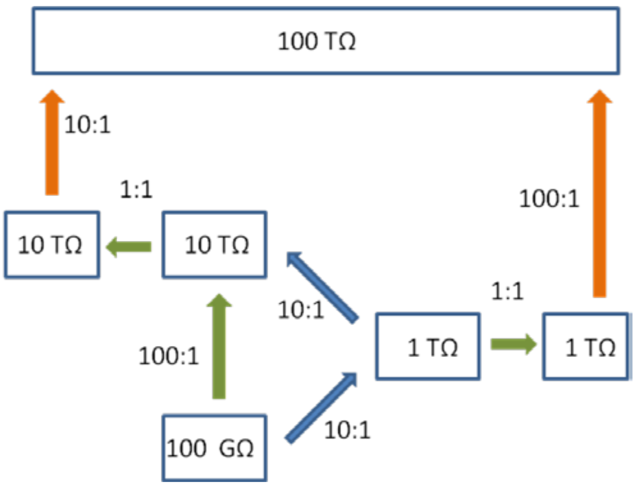

Fig.5. Extension of high resistance scaling from 1 TΩ to 10 TΩ and 100 TΩ. A 1 TΩ per step guarded Hamon transfer standard was used to build-up to 10 TΩ after characterization for settling-time and voltage dependence. 10:1 and 100:1 bridge ratios were used to build-up to 100 TΩ from 10 TΩ and 1 TΩ single-element resistance standards, respectively, providing two paths to the 100 TΩ decade of resistance.

III. RESULTS

Since late 2011, there have been three build-ups to 10 TΩ from 100 GΩ and 1 TΩ using the guarded Hamon transfer standards as show in Fig. 5. These sets of measurements have shown that the 10 TΩ guarded Hamon transfer standard has a drift rate of 80 x 10^{-6}/year but the values assigned from the parallel and series-parallel configurations of this transfer standard have consistently differed by 150 x 10^{-6}. Further investigation will be necessary to determine if the difference in resistance assigned from the parallel and series-parallel configurations can be reduced.

The build-up to 100 TΩ using bridge ratios of 100:1 and 10:1 is shown in Fig. 6 for the 200 V and 250 V measurements. Additional measurements at 500 V were also made but are not shown on the graph since the broader scale would obscure the details. At a given voltage in the range 200 V to 500 V, the resistance assigned to the 100 TΩ standard using the two different measurement paths (100:1 and 10:1 bridge ratios) differed by 300 x 10^{-6}, which was within the standard deviation of multiple measurements over several days.

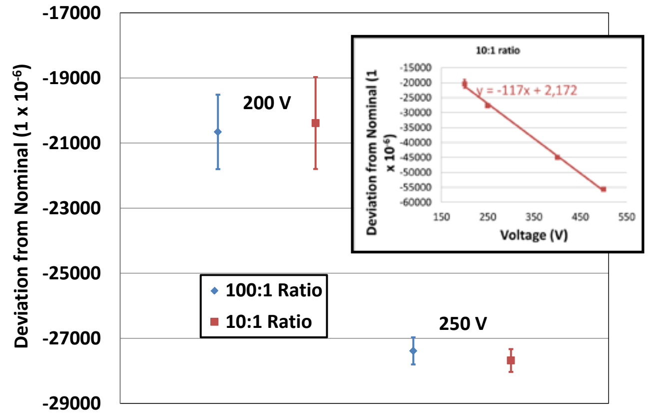

Fig.6. Comparative results for measurements of a 100 TΩ standard resistor. The 100:1 and 10:1 bridge ratios are made using single-element resistance standards of 1 TΩ and 10 TΩ, respectively, in the lower arm of the dual source bridge. Error bars are the standard deviation of multiple measurements over several days. Resistor under test has a voltage coefficient of -117 x 10^{-6}/V (as shown on inset plot) which accounts for the large difference between the 200 V and 250 V measurements.

IV. CONCLUSIONS

Scaling techniques to extend high resistance measurements to 10 TΩ and 100 TΩ are reported which provide multiple scaling paths to each higher decade of resistance beyond 1 TΩ. Guarded Hamon transfer standards, single-element transfer standards, and multiple bridge ratios (10:1 and 100:1) have been used to extend the high resistance range at NIST. Measurement of the resistance standards' settling times, voltage coefficients, and drift rates have been determined. At the time of preparation of this manuscript, the uncertainty analysis has not been completed but based on the measurements described here, the expanded uncertainties ($k = 2$) are estimated to range from 0.03 % to 0.05 % at 10 TΩ and 0.05 % to 0.1 % at 100 TΩ. Further results will be reported at the conference.

REFERENCES

[1] D. G. Jarrett, "Automated guarded bridge for calibration of multi-megohm standard resistors from 10 MΩ to 1 TΩ," *IEEE Trans. Instrum. Meas.*, vol. 46, no. 2, pp. 325-328, April 1997.

[2] R. F. Dziuba, D. G. Jarrett, L. L. Scott, A. J. Secula, "Fabrication of high-value standard resistors," *IEEE Trans. Instrum. Meas.*, vol. 48., no. 2, pp. 333-337, Apr. 1999.

[3] D. G. Jarrett, A. J. Dupree, "Next generation guarded Hamon transfer standards for high resistance," *CPEM 2010 Conference Digest*, June 13-18, 2010, Daejeon, South Korea, pp. 571-572.

[4] F. Hernandez, R. E. Elmquist, B. Rodriguez, "Cryogenic current comparator for the measurement of electrical resistance in the range of 10 kΩ to 1 GΩ," *Symposio de Metrogia 2010*, Queretaro, Mexico, October 27-29, 2010.