The North American $1 \, \Omega$ Inter-laboratory Comparison (2012-2014)

Kai Wendler
Orlando, Florida 2014
NCSLI Conference and Symposium

I have added some extra comments in this version of the talk in order make certain slides easier to understand.
Before we Begin

“Knowledge not shared, is wasted.” - Clan Jacobs.

- Certain commercial equipment, instruments or material are identified in this paper to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Research Council Canada, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.
Outline:

• Background
  (Explain why we run ILC’s)

• Pilot Laboratory
  (Identify the Pilot Laboratory)

• The Standards
  (Discuss various coefficients)

• Problems
  (Describe a problem encountered in this ILC and the solution)

• Data analysis & final report
  (Using this talk as an example, be able analyze ILC data)
Purpose of ILC

The purpose of an ILC is to demonstrate that different laboratories measuring the same artifact should obtain measurements that agree within the experimental uncertainty.

.....or maybe they don’t agree, and if not what is going on?
Some of NRC’s Goals

1 Ω is a key value in resistance and hasn’t been run since 1998-2000. Measurement systems have improved a great deal in that time.

- NRC as mentor
- Canadian Laboratory as the pilot laboratory
- Robust protocol
- Robust, but not overly complex data analysis
- Serve as an excellent example
## 50 Years Ago

### International Comparison of Units of Resistance

**January, 1961**

<table>
<thead>
<tr>
<th>Laboratory</th>
<th>Relative Deviation</th>
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<tbody>
<tr>
<td>Australia</td>
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<tr>
<td>Canada</td>
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<td>- 2.4</td>
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<td>- 8.5</td>
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<td>Great Britain</td>
<td>- 3.4</td>
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<td>Japan</td>
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<tr>
<td>Russia</td>
<td>- 0.7</td>
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<tr>
<td>United States</td>
<td>- 0.4</td>
</tr>
<tr>
<td>West Germany</td>
<td>+ 3.8</td>
</tr>
<tr>
<td>International Bureau</td>
<td>0.0</td>
</tr>
</tbody>
</table>
Canadian Calibration Laboratory as Pilot Laboratory
NRC acting as mentor

Hydro Québec
Institut de recherche

Cal Lab is here

Host of Canadian NCSLI Oct 2014
Accredited for 0.7 ppm uncertainty in resistance at 1 Ω

NCSLI Member
Accredited by CLAS
Large scope
90% internal clients

Accredited for 0.7 ppm uncertainty in resistance at 1 Ω

Medium sized laboratory

9 calibration lab
4 repair

André Langlois    Sylvain Bérubé
Diversity

- Geographical Diversity

- 3 Accreditation Bodies:
  - CLAS, A2LA & Navlap

- 5 separate paths to the SI:
  - 2 NMI’s
  - 3 Independent QHR systems

- Many different measurement systems

- Hand Carry & Shipped legs

- Government & Private Industry
I encourage participants of this ILC to run smaller local ILC’s with laboratories that have higher uncertainties in order to increase the reach of this ILC. Coordinate these ILC’s with Mike Cadenhead, head of committee 132 or myself.

Some details

• Two Measurements International 9210A EvenOhm 1 Ω Resistors

• 3 Legs (now 4)
  • Canadian hand carry
  • US labs
  • QHR and CCC (+ 2 Repeats)
  • (US lab repeat)
The Resistance Standards

Resistance Standards Change with time, their environment and measurement settings.

- Temperature Coefficients
- Power Coefficients
- Pressure Coefficients
- Reversal time
- Drift
Temperature Coefficients

- Measured using a programmable oil bath

Temperature Coefficient Determination

\[ y = -0.022x^2 + 0.023x + 0.002 \]

\[ R^2 = 0.999 \]

1101040: \( \alpha = 0.03 \text{ ppm/deg} \) \( \beta = -0.02 \text{ ppm/deg}^2 \)

1101045: \( \alpha = 0.02 \text{ ppm/deg} \) \( \beta = -0.02 \text{ ppm/deg}^2 \)

Note: All participating laboratories bath temperatures are within 50 mK of 25 °C
Well characterized 100 Ω resistor (1 mA and 0.5 mA)

Calibrated 1 Ω through a 10 Ω resistor, using DCC bridge

1101040  0.00 ppm

1101045  +0.011 ppm
Pressure Coefficients

- Resistors in a pressure vessel
- Pressure vessel inside an air bath
Pressure Coefficients

• Changing the pressure changes the temperature ± 100 mK
Pressure Coefficients

Measurement temperature was within 6 mK
Wait time varied between 2.5 hrs and 48 hrs.

Graph of 1 resistor
Pressure Coefficients

1101040 vs NRC 1 Ω

- 800 mmHg to 550 mmHg
- 550 mmHg to 800 mmHg

\[ y = -2E-05x + 0.0194 \]
\[ y = -4E-05x + 0.0307 \]

This value will be added in quadrature to each laboratories stated measurement uncertainty.

1101040: 0.01 μΩ/Ω
This values will be added in quadrature to each laboratories stated measurement uncertainty.
Resistance Standards
Measurement Reversal Time

- Resistors measured against NML 1 Ω, reversal rates from 4 seconds to 60 seconds
- Measurements made by Nick Fletcher at BIPM using a low frequency ac bridge show NML resistors are least affected by reversal times.
Resistance Standards
Measurement Reversal Time

1101040 vs NML 64150

- Last 4 measurements

- These measurements were made over 5 days
Measurement Reversal Time

Resistance Standards

Note: I likely will add an Uc of 0.01 to 0.02 ppm in quadrature to the $k=1$ uncertainty of each laboratory to deal with this problem.

Conclusion: The use of different reversal rates is not a significant factor with these resistors.
Sometimes Things go Wrong!

Something happened in Leg 2 and the results cannot be used.

- Likely a shipping issue

Solution – Make the shipping container bullet proof

Lesson: Shipping can be HARD on standards
Chronological View of Leg 2

One of the Resistors

Significant Change

Leg 1

Leg 2

Leg 3
Chronological View of Leg 2

The Other Resistor

Leg 1  Leg 2  Leg 3

Significant Change
What Could Have Caused This Change?

• Perhaps a shipping issue?

• A new hard foam container
• Resistors in centre of the container
• Original Shipping container inside a larger crate
Sometimes Things go Wrong

- Unfamiliarity with Uncertainty the protocols uncertainty sheet.
  Mistake in reported value, problem spotted by laboratory, new report submitted.
- Long Delays in providing the report

The Devil is in the Details!
Resistance Standards
Slope (Drift)

• It is well known that for a standard of resistance, the measurements typically show a trend in time, which we assume can be modeled as a linear trend. (Sim EM k1,k2,s1)

• The drift of the resistor will be determined from the measurement data………………..But how???
  • NMI opening and closing values
  • Pilot lab data
  • All data
  • Weighted slope
What is the real slope

Include in slope calculation?

Note: the circled values were not included in the slope calculation or the calculation of the CRV
# Weighted slope “The Easy Way”

1) Remove Outliers   2) Use LINEST to determine Initial Slope

<table>
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<tr>
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<td>1</td>
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Note: Every measurement that is not an outlier was used in the determination of the slope, since the purpose of this calculation is to determine the actual linear drift of the resistor.

1. LINEST used to determine initial slope, paste values here

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<thead>
<tr>
<th>slope</th>
<th>intercept</th>
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<tbody>
<tr>
<td>0.000930173</td>
<td>-1.212103744</td>
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</table>

Note: LINEST can be run as an array. This provides both the slope and the intercept as well as the uncertainties for both these calculations (uncertainties not shown here).
3) From slope and intercept calculate the fit
4) Calculate Weighted Residuals

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</tbody>
</table>

Note: When you use the linest calculation, the best slope is determined by calculating the lowest value for the sum of the squares of the residuals, this provides the same weight to each data point.

Intercept: -1.212103744
Slope: 0.000930173

Lowest value here = BEST SLOPE

1341.086
Sum Square
5) Run Solver in Excel

Settings: Min value for Sum Sqr of Weighted Residuals by changing Slope and Intercept

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<td>0.023</td>
<td>9.394733351</td>
</tr>
</tbody>
</table>

Note: An uncertainty component for this calculation still needs to be determined. This uncertainty will likely be added in quadrature to the drift corrected values uncertainty for each laboratory.

Intercept: -1.241594148
Slope: 0.001099652

Lowest value here = BEST SLOPE

Note: For this to work the slope and intercept need to be values, not the LINEST formula. The Fit, Residual, Weighted Residual and Sum Square of the WR must be formulas. These values will all change when solver alters the value of the slope and intercept.
Using the slope value, Remove the drift from each measurement

1 Ohm ILC 1997-200

- Series1
- Drift corrected
- Linear (Series1)
Calculate the Comparison Reference Value (CRV)

<table>
<thead>
<tr>
<th>Drift Corrected Value</th>
<th>σ</th>
<th>σ^2</th>
<th>xi/σ^2</th>
<th>1/σ^2</th>
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<tbody>
<tr>
<td>NIST</td>
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<tr>
<td>-1.230</td>
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<td>0.000625</td>
<td>-984.000</td>
<td>800.000</td>
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<td>10.767</td>
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<td>0.03</td>
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</tbody>
</table>

Each laboratory can only contribute once to the WM, when two values are present, weighting is multiplied by 0.5

Note: As this is a comparison of different laboratories each laboratory should only contribute once to the CRV. The CRV is a reference value for the artifact against which all the laboratories measurements will be compared. All the participants who are not outliers contribute to this value, the laboratories contribution is weighted according to its measurement uncertainty.

Different approaches can be used. I have not tried it, but I would guess there would be very little difference in the value of the CRV between all these methods.

- Only one value from a lab that measured more than once could be used (ie only use one value from the pilot lab).
- An average value and date could be used (ie average both NIST measurements into one value).
- Reduce the weighted value by the number of measurements, in this case multiply xi/σ^2*0.5 & 1/σ^2*0.5.
Determine each laboratories deviation from the CRV

Note: This is the difference from the value corrected for drift vs the CRV.
Data Analysis

- Adjust uncertainties to account for the pressure coefficient
- Correct value to 50 mA
- Calculate slope, remove drift
- Calculate CRV
- Calculate the Deviation from CRV
Data Analysis

- Calculate En
- Each participant will receive a report about their measurements

\[
En = \frac{x - X}{\sqrt{U_{\text{ref}}^2 + U_{\text{lab}}^2}}
\]

- En = Normalized error
- x = participants results
- X = Reference value
- U_{\text{lab}} = participants uncertainty (k=2)
- U_{\text{ref}} = Reference value uncertainty (k=2)

En > 1 is not satisfactory
Results so far 1101040

1101040 (k=2)

- NRC CCC
- NRC
- IREQ
- Laboratory Results

Deviation $\mu\Omega/\Omega$ vs. Lab #
Results so far  1101045

1101045  \((k=2)\)

- Deviation  \(\mu\Omega/\Omega\)
- Lab #

Graph showing deviation of 1101045 for 20 labs with symbols for NRC CCC, NRC, IREQ, and Laboratory Results.
Final Report

• Once all the data has been collected a final report will be written and sent to all the participants.

• The final report will be published, including an appendix with the protocol.
Questions?

Kai Wendler  
Technical Officer  
Tel: 613-990-7624  
Kai.Wendler@nrc-cnrc.gc.ca  
www.nrc-cnrc.gc.ca

Many Thanks

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Benoit Buchard

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Ryan Brown