The North American 1 Ω Inter-laboratory Comparison (2012-2014)

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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.

National Research Council Canada
Conseil national de recherches Canada
NRC·CNRC
Canada

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)

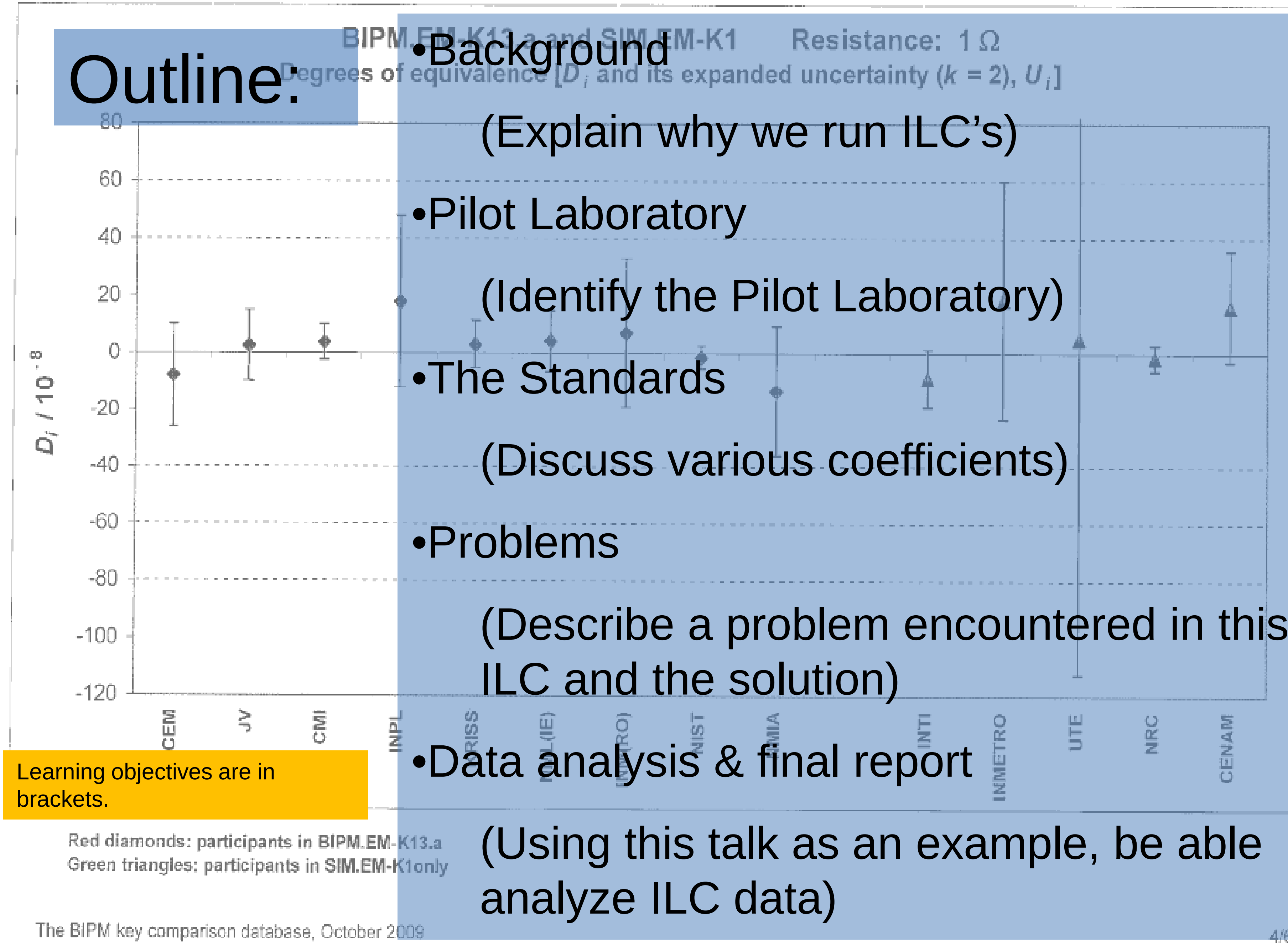
- Problems

    (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)

Learning objectives are in brackets.



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

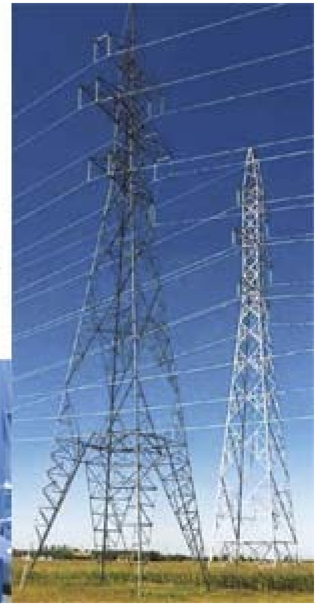
Laboratory	Relative Deviation
Australia	- 3.5 p.p.m.
Canada	- 4.2
East Germany	- 2.4
France	- 8.5
Great Britain	- 3.4
Japan	- 0.3
Russia	- 0.7
United States	- 0.4
West Germany	+ 3.8
International Bureau	0.0

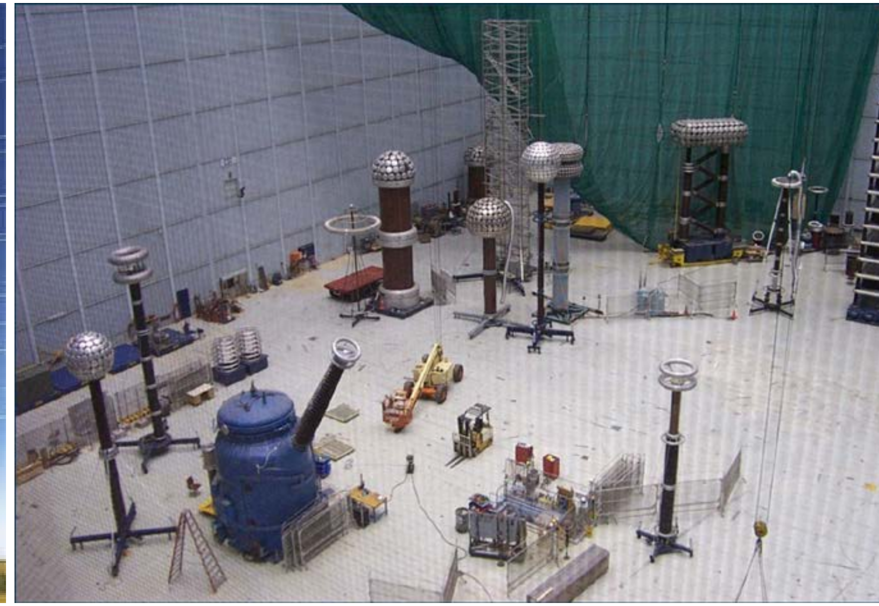
35

Canadian Calibration Laboratory as Pilot Laboratory

NRC acting as mentor

Hydro Québec
Institut de recherche





Hydro Québec

Institut de recherche

André Langlois Sylvain Bérubé

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

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

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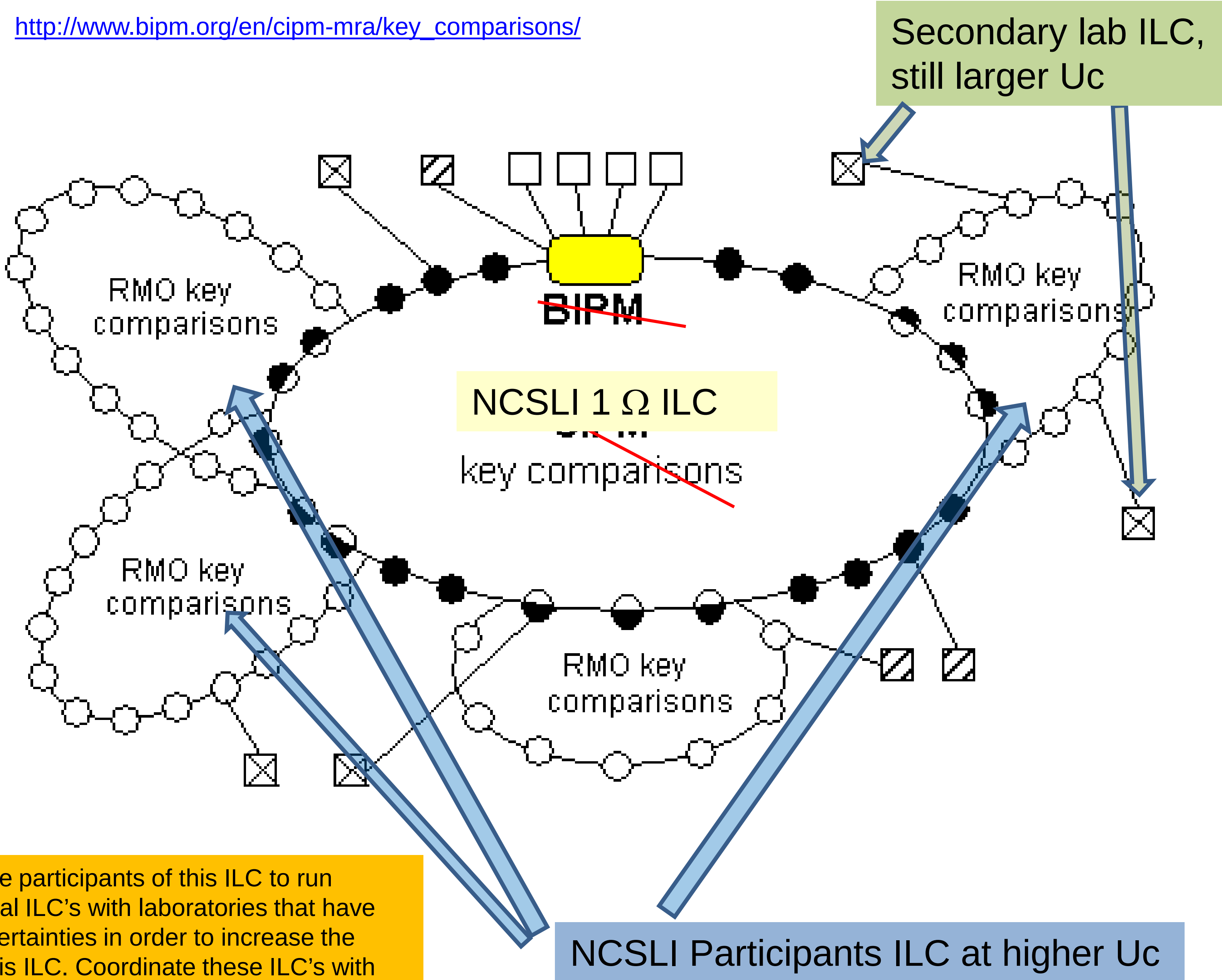
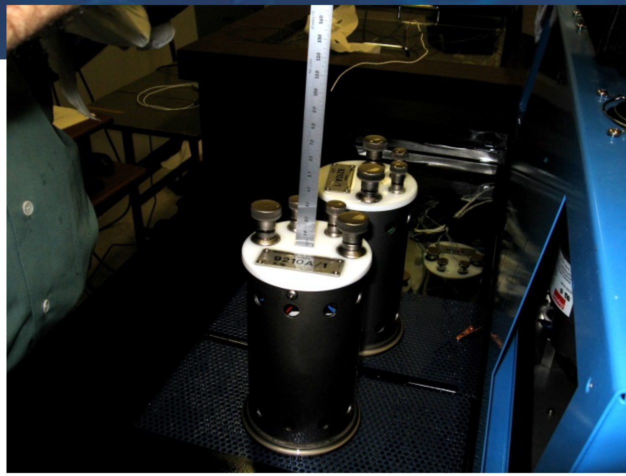
http://www.bipm.org/en/cipm-mra/key_comparisons/



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)

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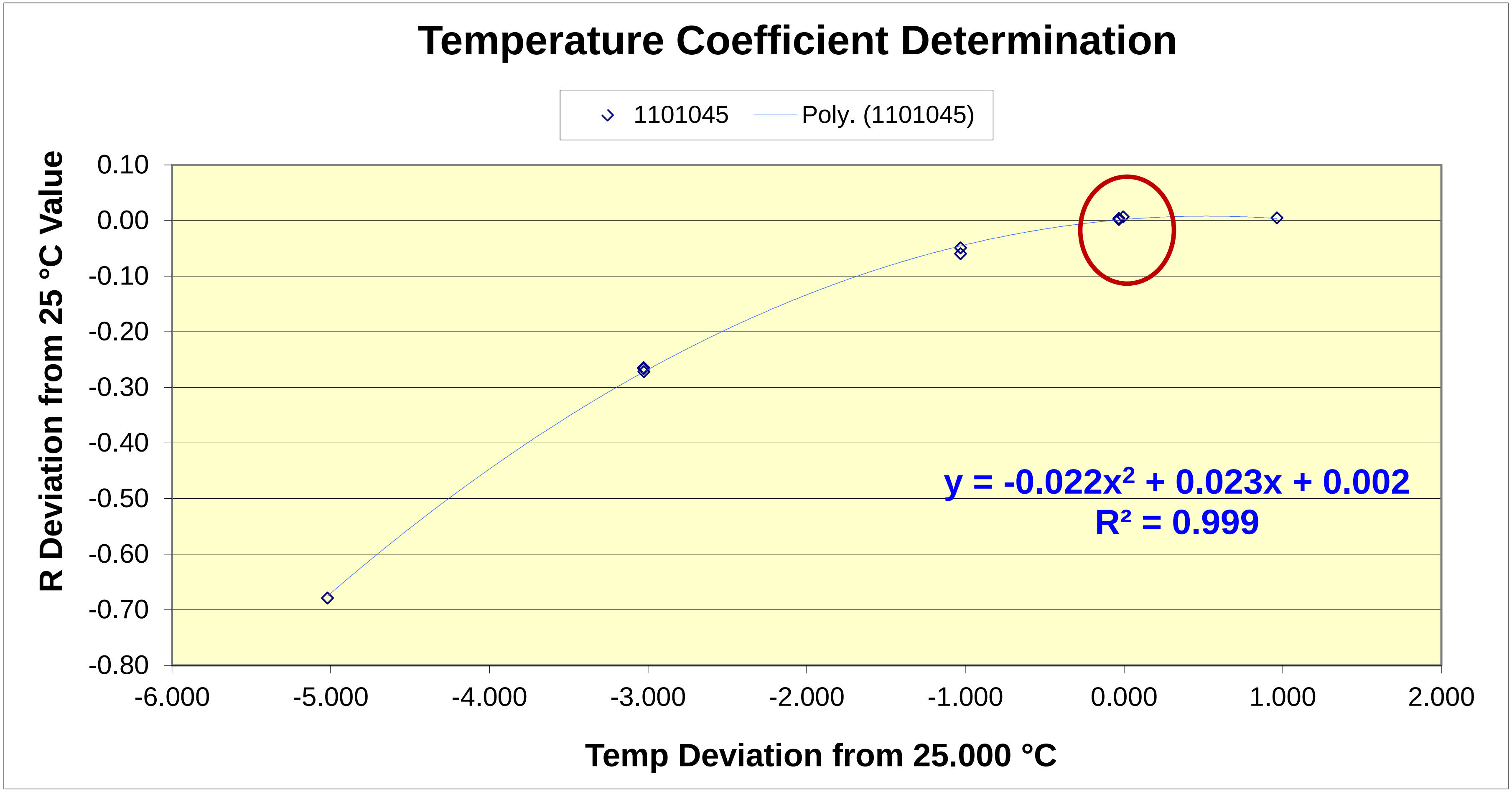
The Resistance Standards

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

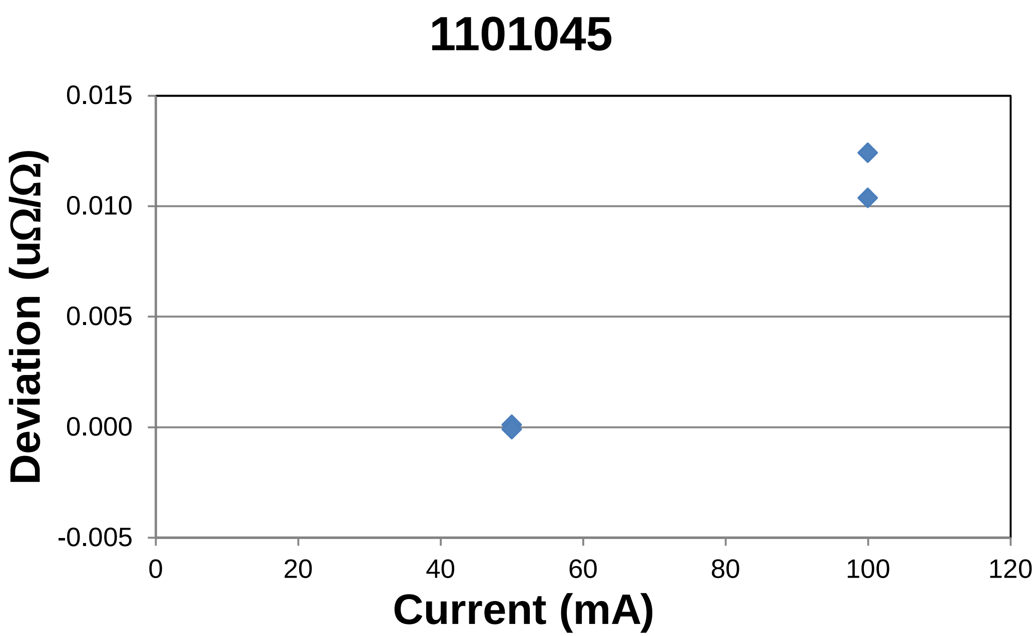


1101040: α = 0.03 ppm/deg β = -0.02 ppm/deg^2

1101045: α= 0.02 ppm/deg β= -0.02 ppm/deg^2

Note: All participating laboratories bath temperatures are within 50 mK of 25 °C

Power Coeficient



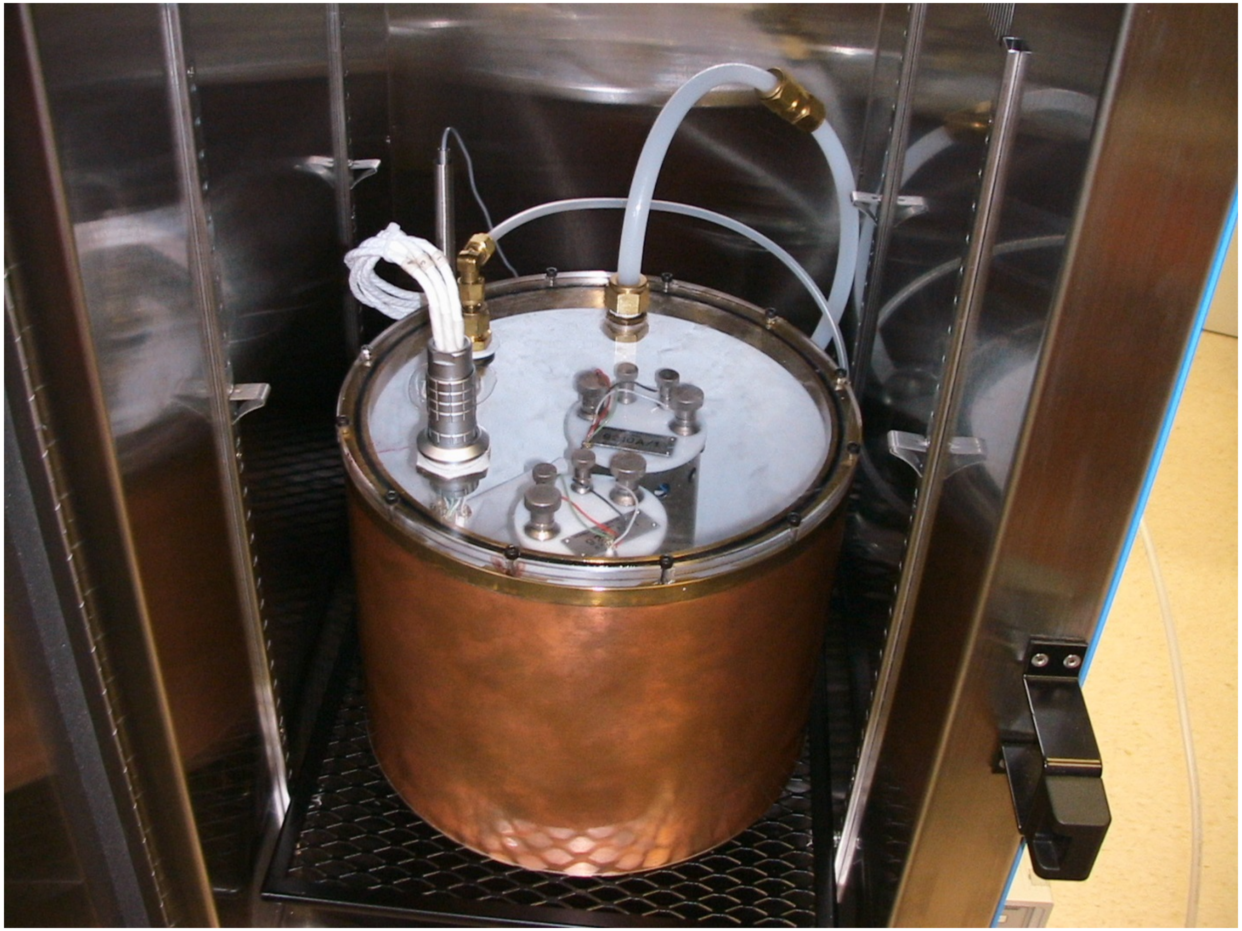
Well characterized 100 Ω resistor (1 mA and 0.5 mA)

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

1101040   0.00 ppm

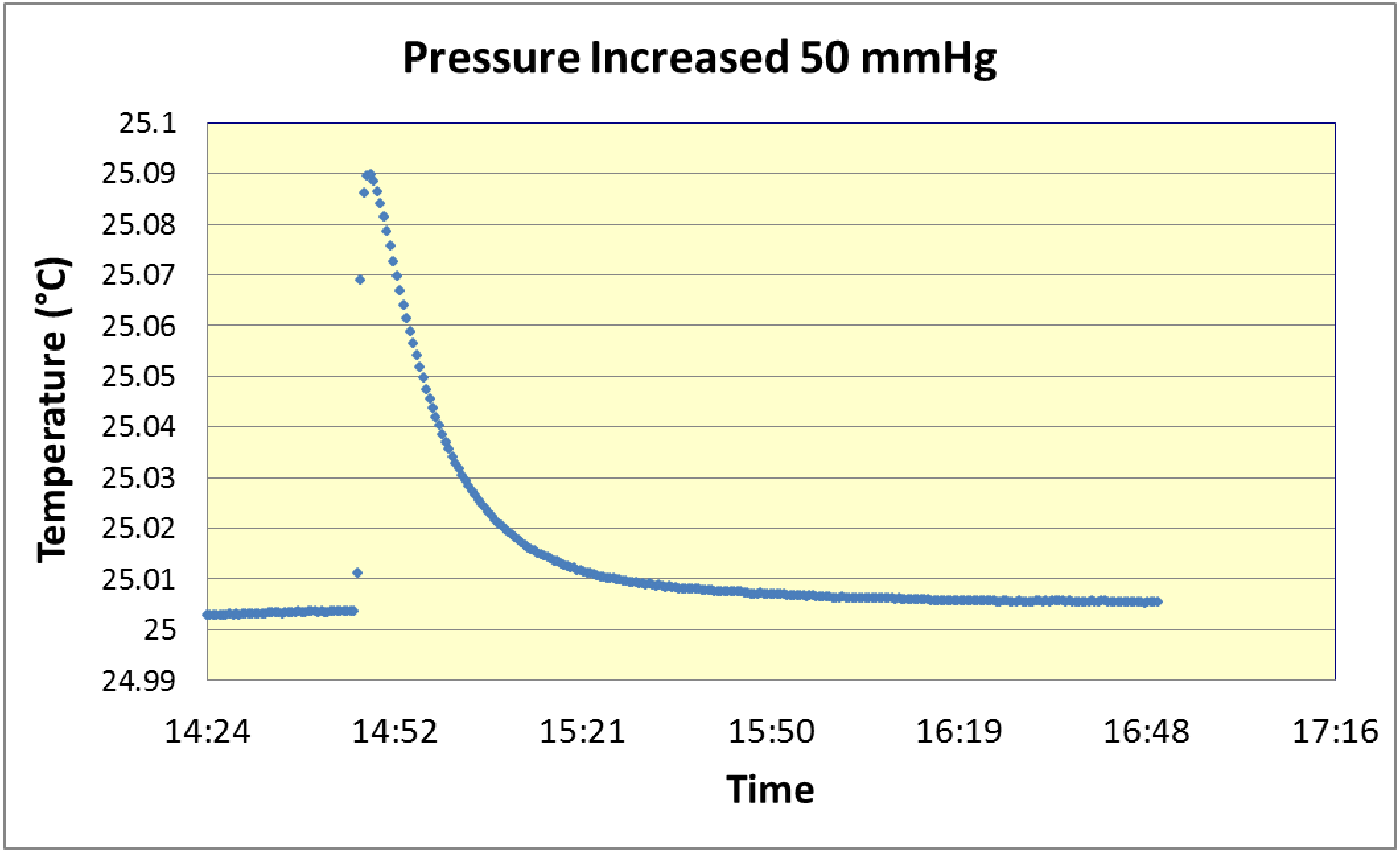
1101045 +0.011 ppm

Pressure Coefficients



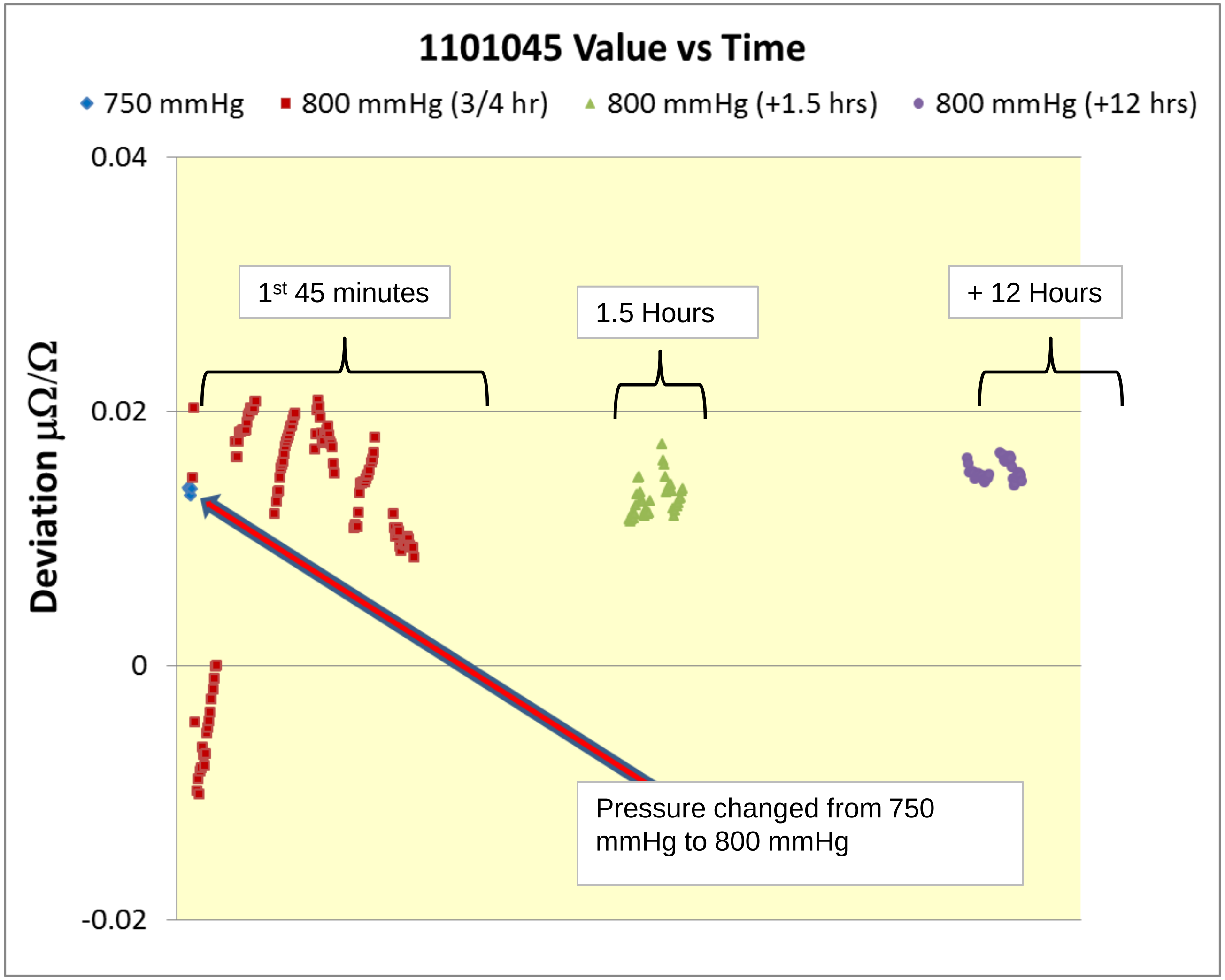
- Resistors in a pressure vessel
- Pressure vessel inside an air bath

Pressure Coefficients



- Changing the pressure changes the temperature ± 100 mK

Pressure Coefficients

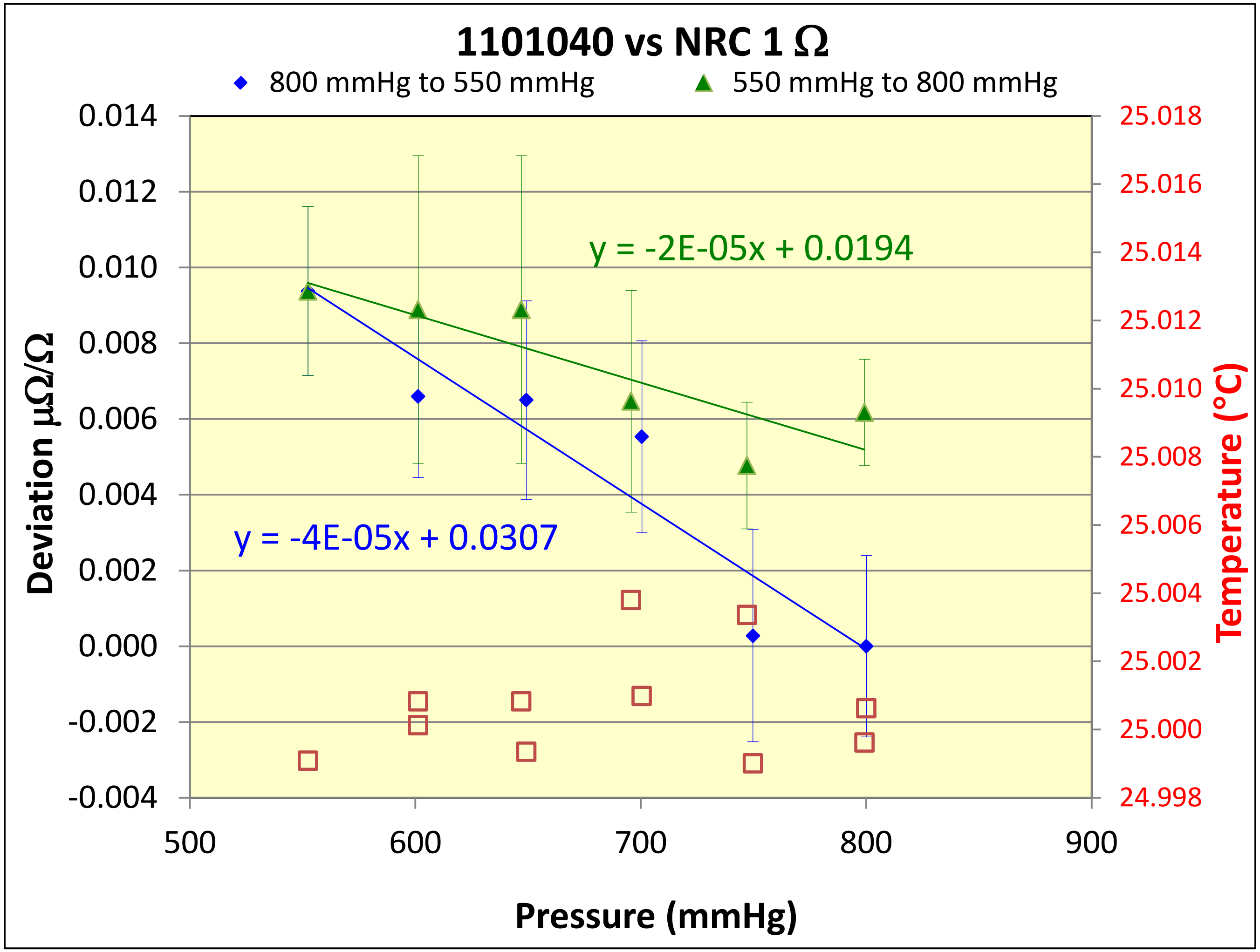


Measurement temperature was within 6 mK

Wait time varied between 2.5 hrs and 48 hrs.

Graph of 1 resistor

Pressure Coefficients



1101040: 0.01 μΩ/Ω

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

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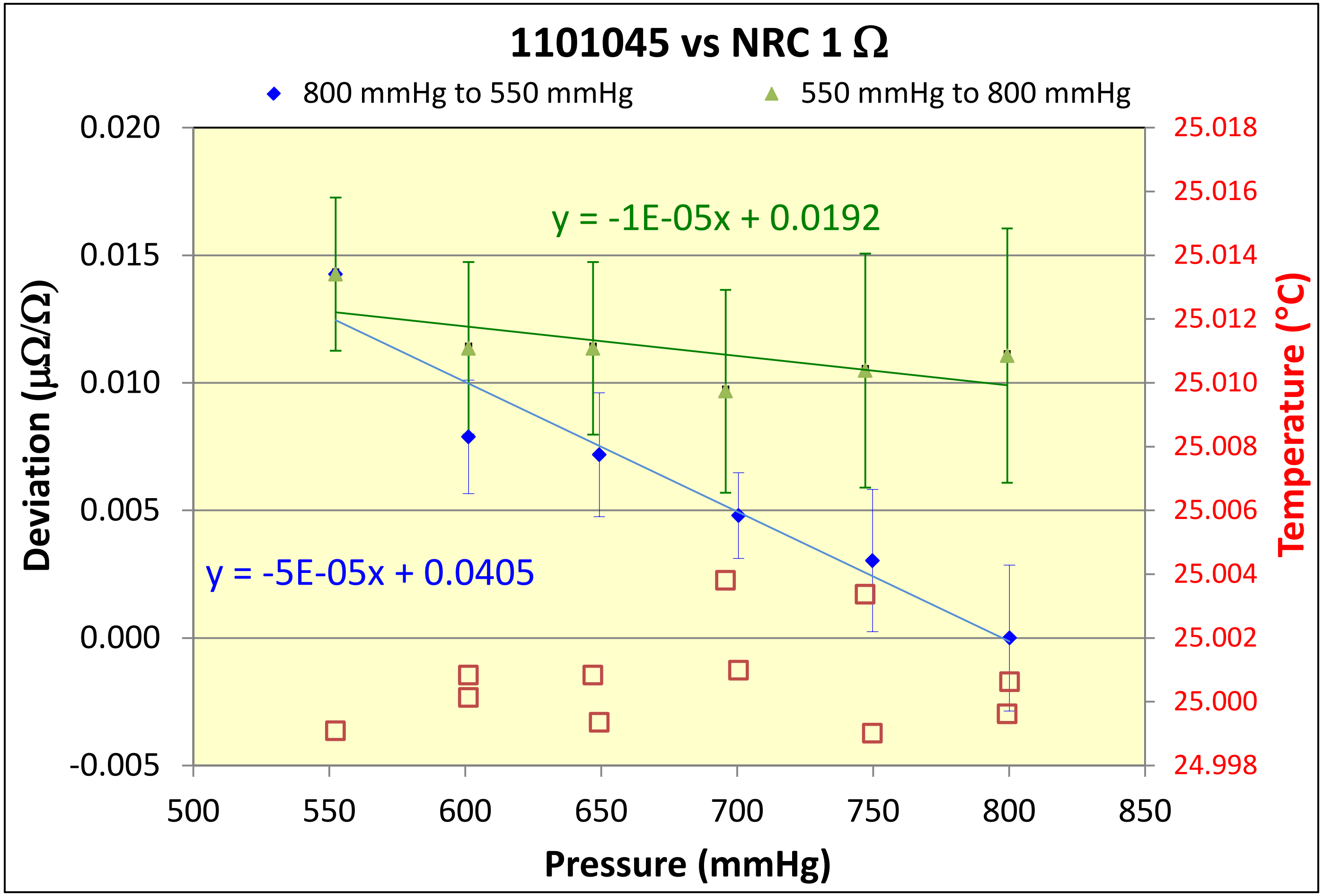
Pressure Coefficients

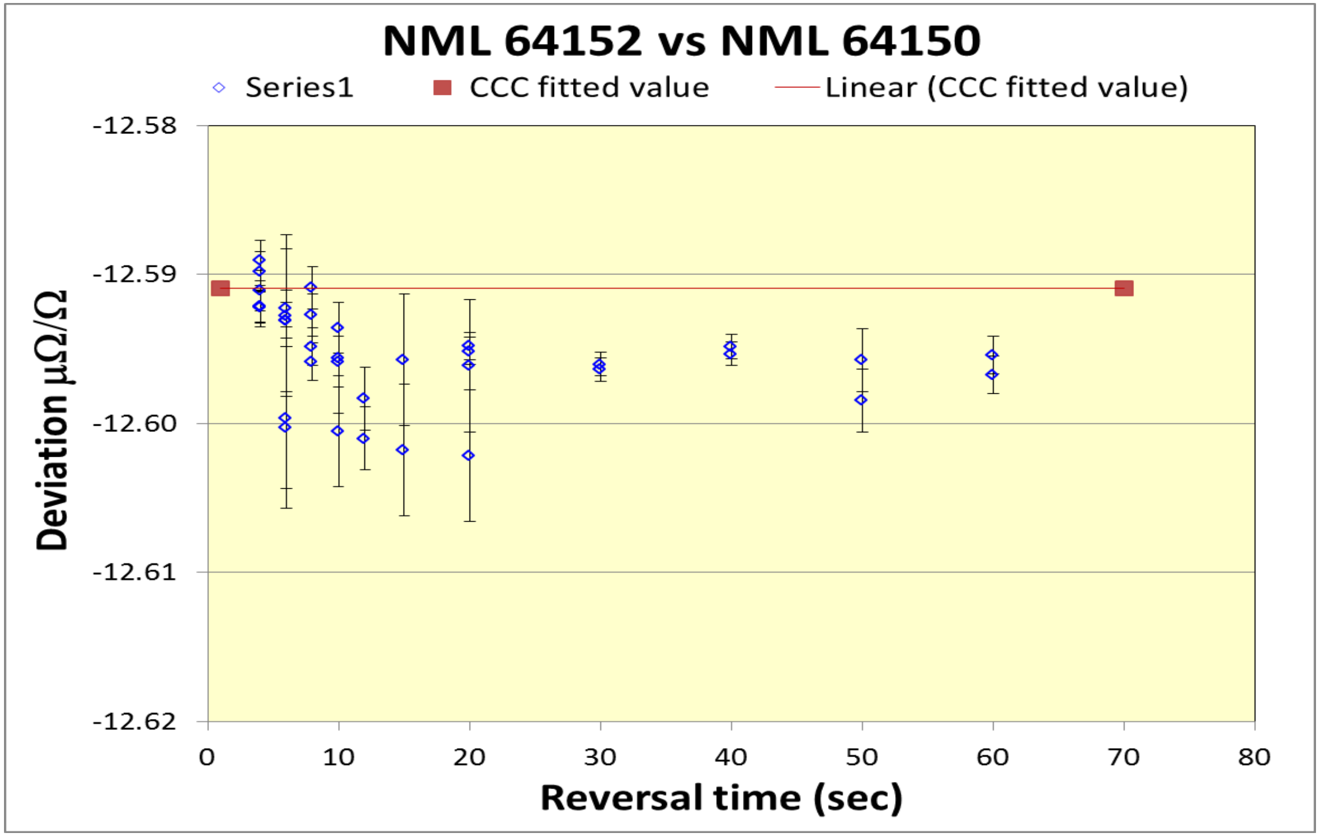


1101045: 0.014 µΩ/Ω

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

Resistance Standards

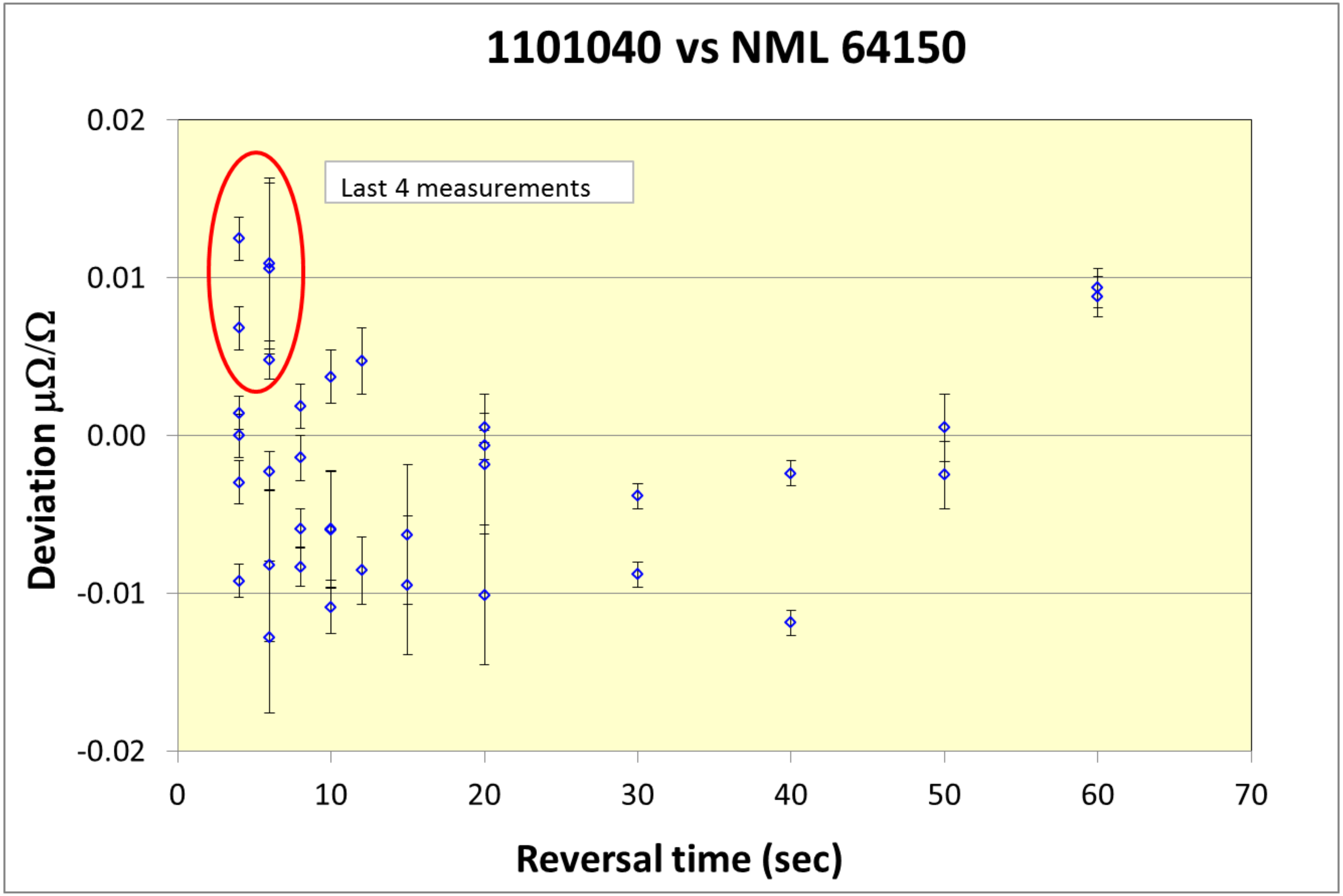
Measurement Reversal Time



- 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

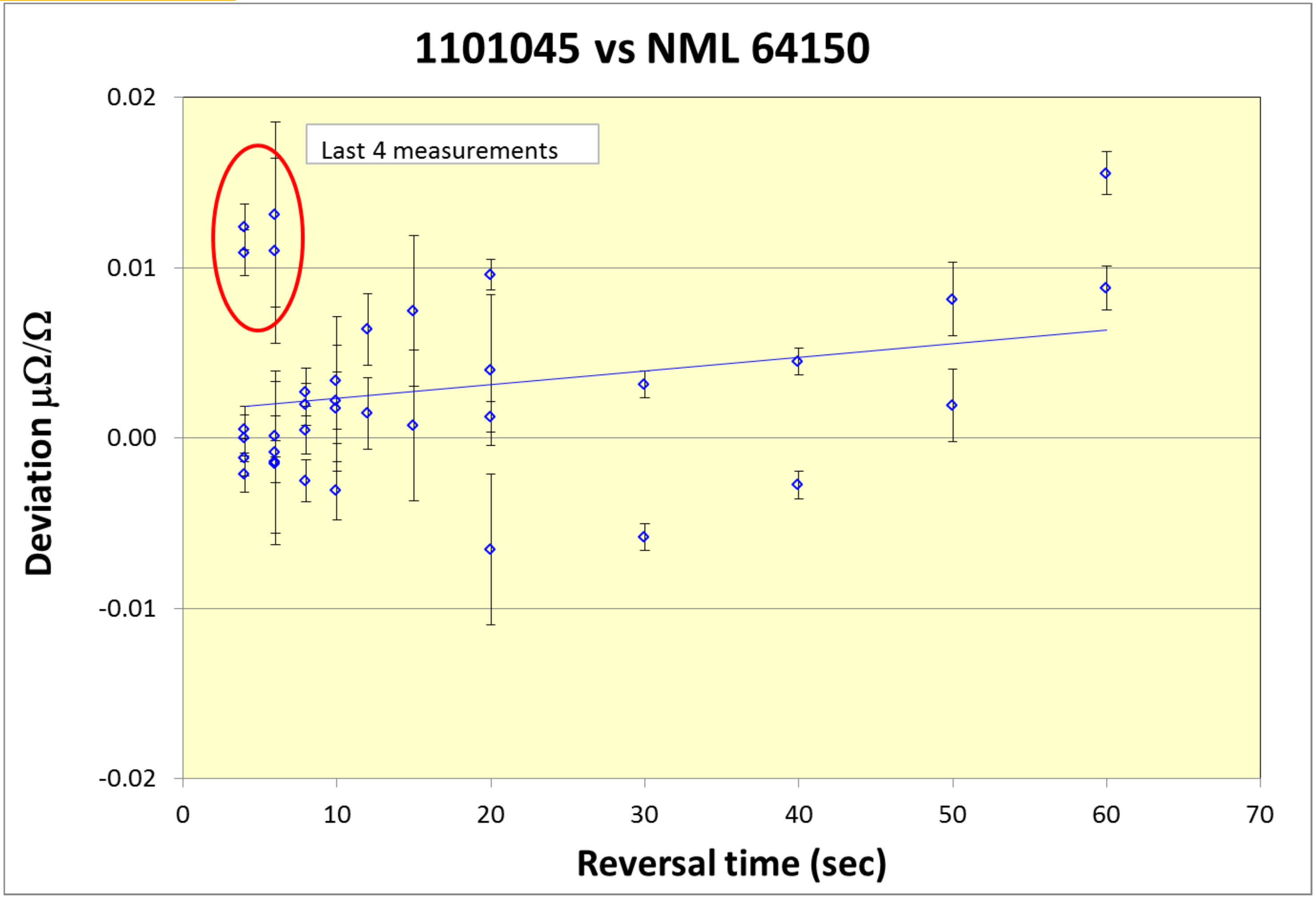


- These measurements were made over 5 days

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.

Resistance Standards

Measurement Reversal Time



Conclusion: The use of different reversal rates is not a significant factor with these resistors.

Sometimes Things go Wrong!

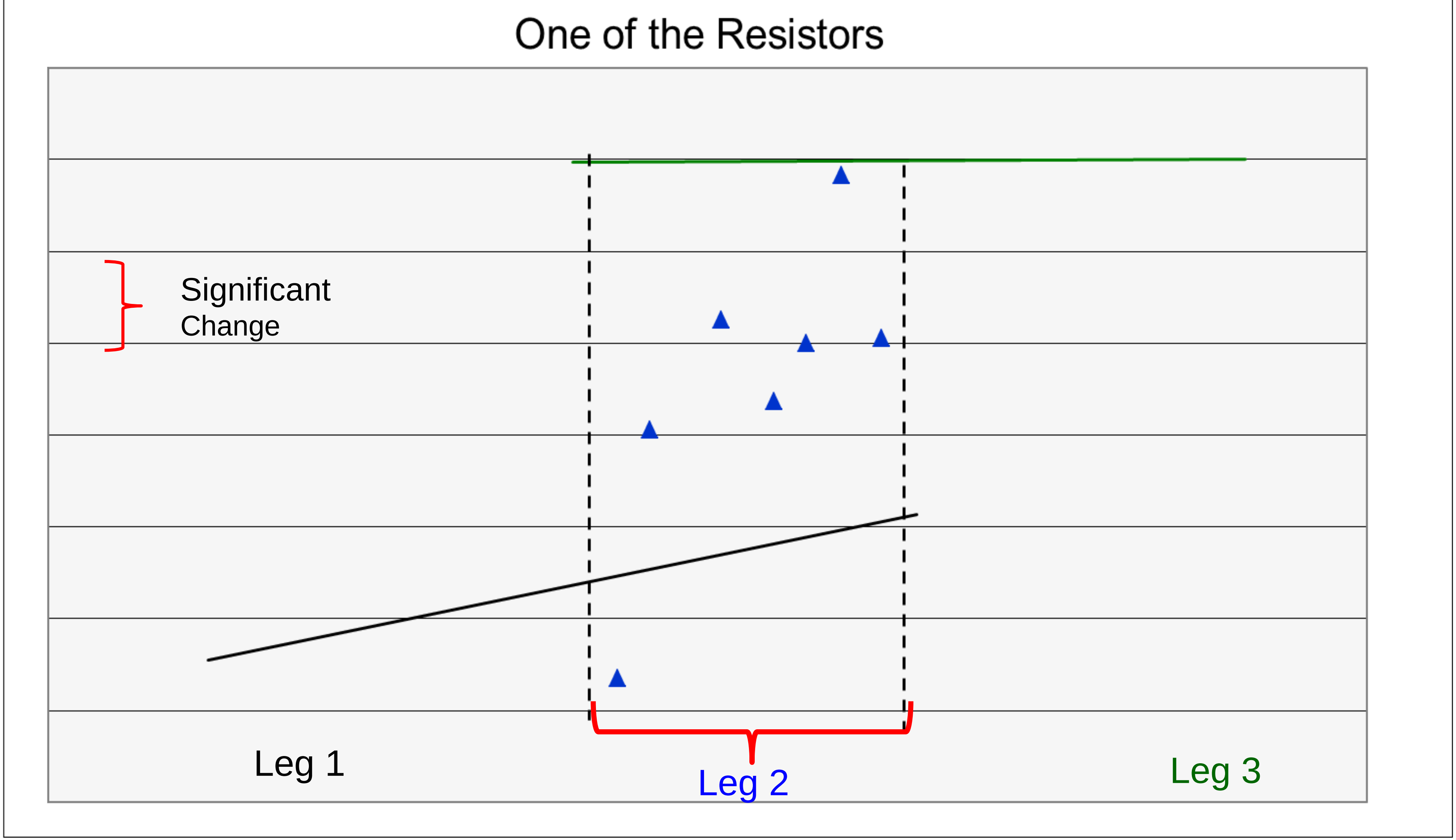
Something happened in Leg 2 and the results cannot be used

- Likely a shipping issue

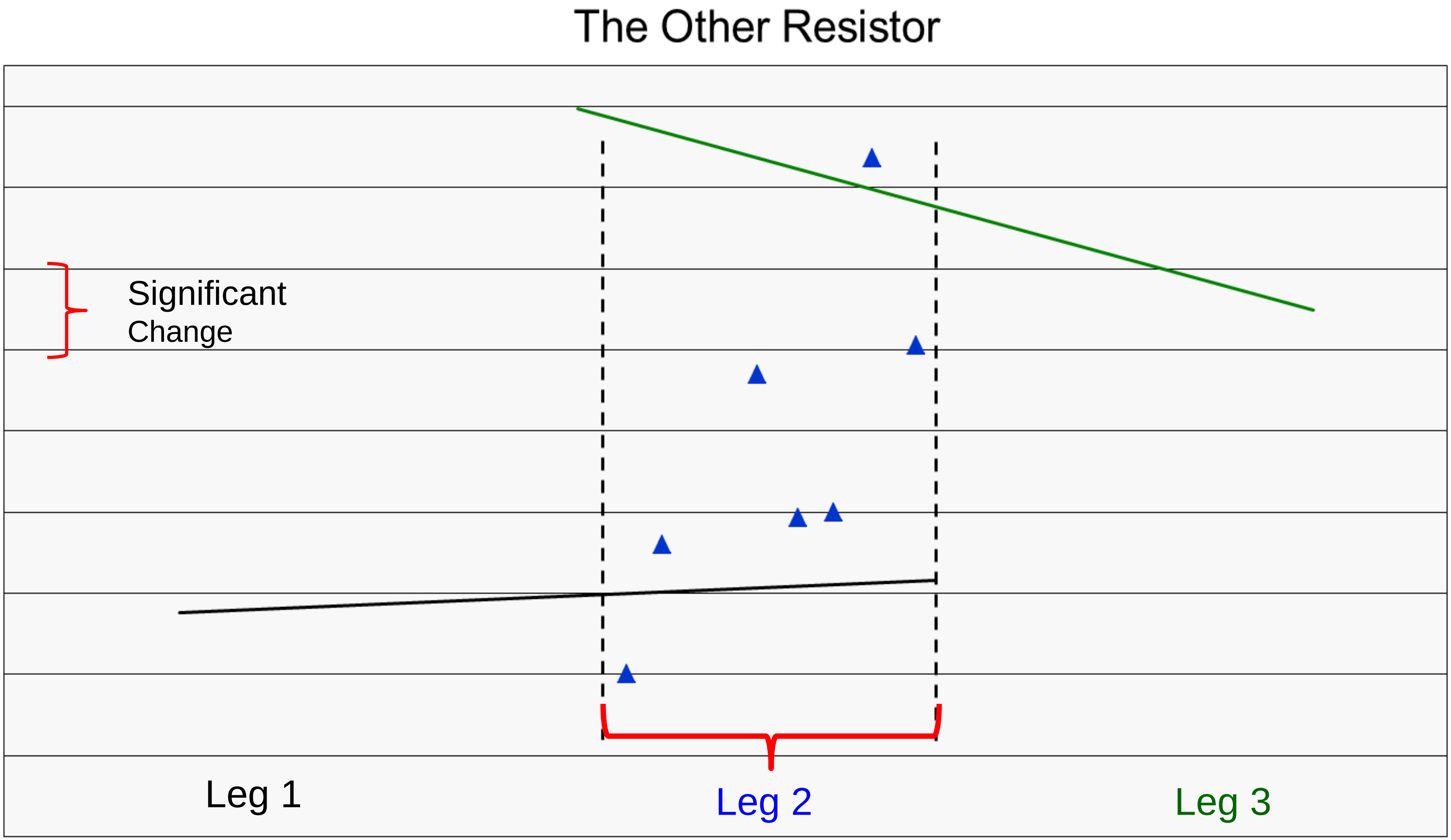
Solution – Make the shipping container bullet proof

Lesson: Shipping can be HARD on standards

Chronological View of Leg 2

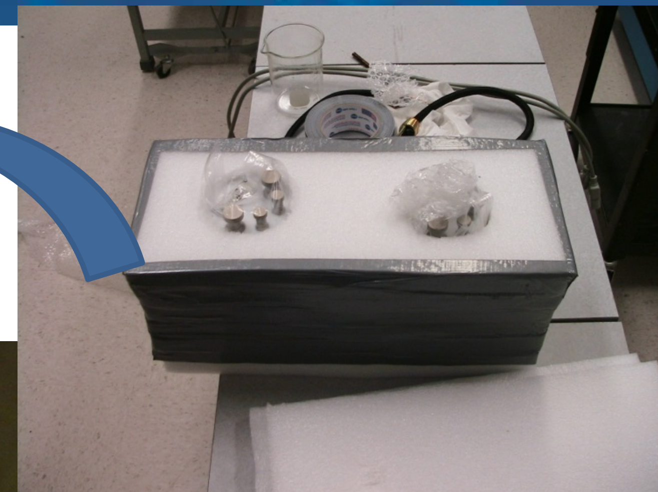


Chronological View of Leg 2



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.

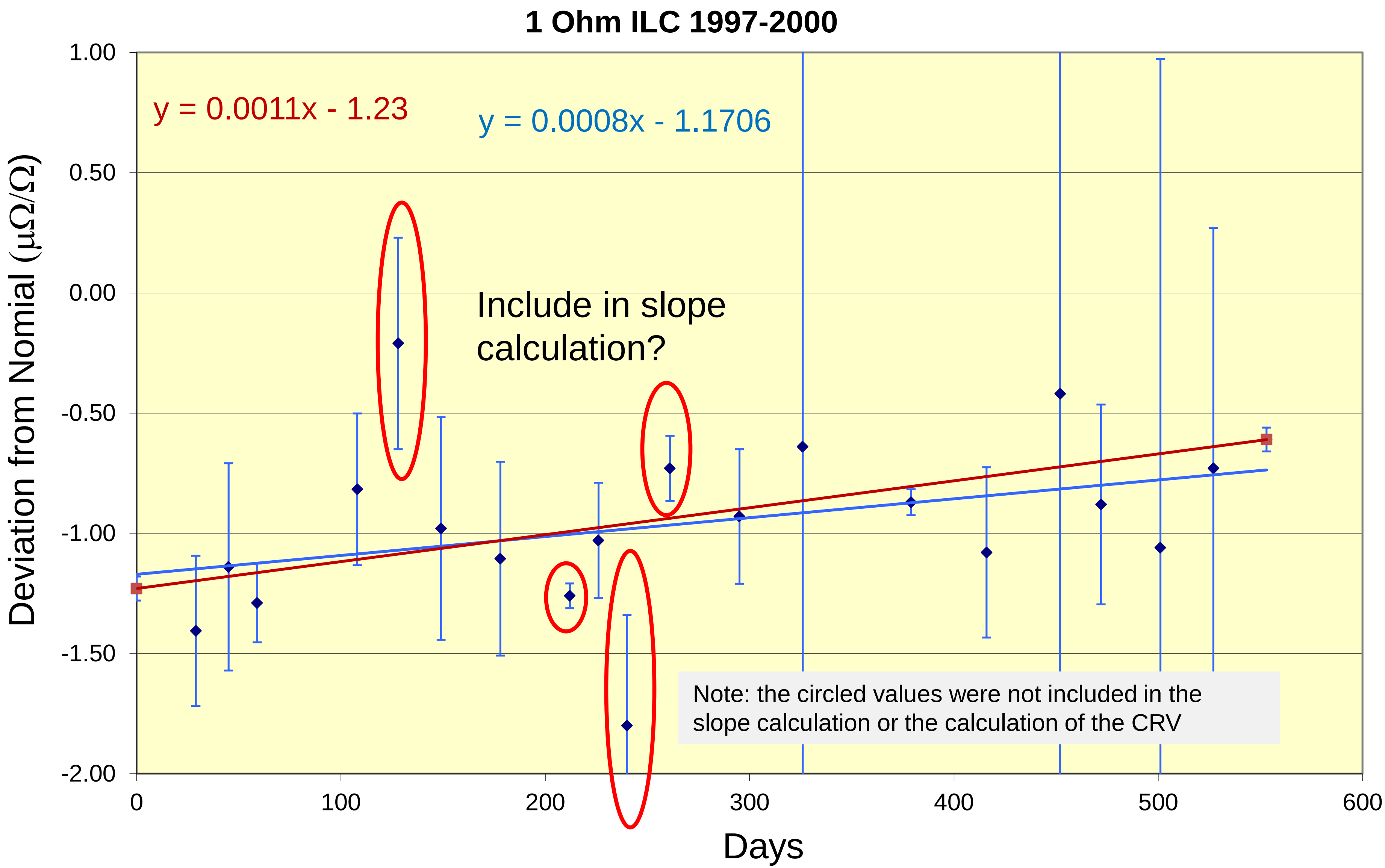
-Long Delays in providing the report

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



Weighted slope "The Easy Way"

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

	Lab	Time (days)	Date	Measured Value	Dev (ppm)	Uncertainty ppm (k=2)
1	NIST	0	Sep/21/1998	0.999 998 77	-1.230	0.05
2	1	29	Oct/20/1998	0.999 998 59	-1.406	0.312
3	2	45	Nov/05/1998	0.999 998 86	-1.140	0.431
4	3	59	Nov/19/1998	0.999 998 71	-1.290	0.164
5	4	108	Jan/07/1999	0.999 999 18	-0.817	0.316
7	6	149	Feb/17/1999	0.999 999 02	-0.980	0.463
8	7	178	Mar/18/1999	0.999 998 89	-1.106	0.403
10	9	226	May/05/1999	0.999 998 97	-1.030	0.24
13	12	295	Jul/13/1999	0.999 999 07	-0.930	0.28
14	13	326	Aug/13/1999	0.999 999 36	-0.640	2.04
15	14	379	Oct/05/1999	0.999 999 13	-0.871	0.054
16	15	416	Nov/11/1999	0.999 998 92	-1.080	0.354
17	16	452	Dec/17/1999	0.999 999 58	-0.420	4.319
18	17	472	Jan/06/2000	0.999 999 12	-0.880	0.416
19	18	501	Feb/04/2000	0.999 998 94	-1.060	2.033
20	19	527	Mar/01/2000	0.999 999 27	-0.730	1
21	NIST	553	Mar/27/2000	0.999 999 39	-0.610	0.05

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

LINEST calculation is based on the "Time (days)" and "Dev (ppm)"

0.000930173	-1.212103744
slope	intercept

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

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

Lab	Time (days)	Date	Measured Value	Dev (ppm)	Uncertainty ppm (k=2)	Fit Intercept + (days*slope)	Residuals Dev - Fit	Weighted Residuals Residual / Uc^2
NIST	0	Sep/21/1998	0.999 998 77	-1.230	0.05	-1.242	-0.018	-7.158502347
1	29	Oct/20/1998	0.999 998 59	-1.406	0.312	-1.210	-0.221	-2.268976622
2	45	Nov/05/1998	0.999 998 86	-1.140	0.431	-1.192	0.030	0.162822006
3	59	Nov/19/1998	0.999 998 71	-1.290	0.164	-1.177	-0.133	-4.936661115
4	108	Jan/07/1999	0.999 999 18	-0.817	0.316	-1.123	0.295	2.950700078
6	149	Feb/17/1999	0.999 999 02	-0.980	0.463	-1.078	0.094	0.436201278
7	178	Mar/18/1999	0.999 998 89	-1.106	0.403	-1.046	-0.059	-0.366155649
9	226	May/05/1999	0.999 998 97	-1.030	0.24	-0.993	-0.028	-0.48811208
12	295	Jul/13/1999	0.999 999 07	-0.930	0.28	-0.917	0.008	0.098250473
13	326	Aug/13/1999	0.999 999 36	-0.640	2.04	-0.883	0.269	0.064606759
14	379	Oct/05/1999	0.999 999 13	-0.871	0.054	-0.825	-0.011	-3.92032185
15	416	Nov/11/1999	0.999 998 92	-1.080	0.354	-0.784	-0.255	-2.033643297
16	452	Dec/17/1999	0.999 999 58	-0.420	4.319	-0.745	0.372	0.01992444
17	472	Jan/06/2000	0.999 999 12	-0.880	0.416	-0.723	-0.107	-0.61793701
18	501	Feb/04/2000	0.999 998 94	-1.060	2.033	-0.691	-0.314	-0.075951113
19	527	Mar/01/2000	0.999 999 27	-0.730	1	-0.662	-0.008	-0.008097198
NIST	553	Mar/27/2000	0.999 999 39	-0.610	0.05	-0.633	0.088	35.08732601

Lowest value here = BEST SLOPE

Intercept	-1.212103744
Slope	0.000930173

1341.086

Sum Square

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

5) Run Solver in Excel
Settings: Min value for Sum Sqr of Weighted Residuals by changing Slope and Intercept

	Lab	Time (days)	Date	Measured Value	Dev (ppm)	Uncertainty ppm (k=2)	Fit Intercept + (days*slope)	Residuals Dev - Fit	Weighted Residuals Residual / Uc^2
1	NIST	0	Sep/21/1998	0.999 998 77	-1.230	0.05	-1.242	0.012	4.637659332
2	1	29	Oct/20/1998	0.999 998 59	-1.406	0.312	-1.210	-0.196	-2.016516139
3	2	45	Nov/05/1998	0.999 998 86	-1.140	0.431	-1.192	0.052	0.28052082
4	3	59	Nov/19/1998	0.999 998 71	-1.290	0.164	-1.177	-0.113	-4.211975537
5	4	108	Jan/07/1999	0.999 999 18	-0.817	0.316	-1.123	0.306	3.062728122
7	6	149	Feb/17/1999	0.999 999 02	-0.980	0.463	-1.078	0.098	0.455971084
8	7	178	Mar/18/1999	0.999 998 89	-1.106	0.403	-1.046	-0.060	-0.370323259
10	9	226	May/05/1999	0.999 998 97	-1.030	0.24	-0.993	-0.037	-0.641095576
13	12	295	Jul/13/1999	0.999 999 07	-0.930	0.28	-0.917	-0.013	-0.163304377
14	13	326	Aug/13/1999	0.999 999 36	-0.640	2.04	-0.883	0.243	0.058416892
15	14	379	Oct/05/1999	0.999 999 13	-0.871	0.054	-0.825	-0.046	-15.83463461
16	15	416	Nov/11/1999	0.999 998 92	-1.080	0.354	-0.784	-0.296	-2.360918816
17	16	452	Dec/17/1999	0.999 999 58	-0.420	4.319	-0.745	0.325	0.017398724
18	17	472	Jan/06/2000	0.999 999 12	-0.880	0.416	-0.723	-0.157	-0.909771345
19	18	501	Feb/04/2000	0.999 998 94	-1.060	2.033	-0.691	-0.369	-0.089359626
20	19	527	Mar/01/2000	0.999 999 27	-0.730	1	-0.662	-0.068	-0.067922226
21	NIST	553	Mar/27/2000	0.999 999 39	-0.610	0.05	-0.633	0.023	9.394733351

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.

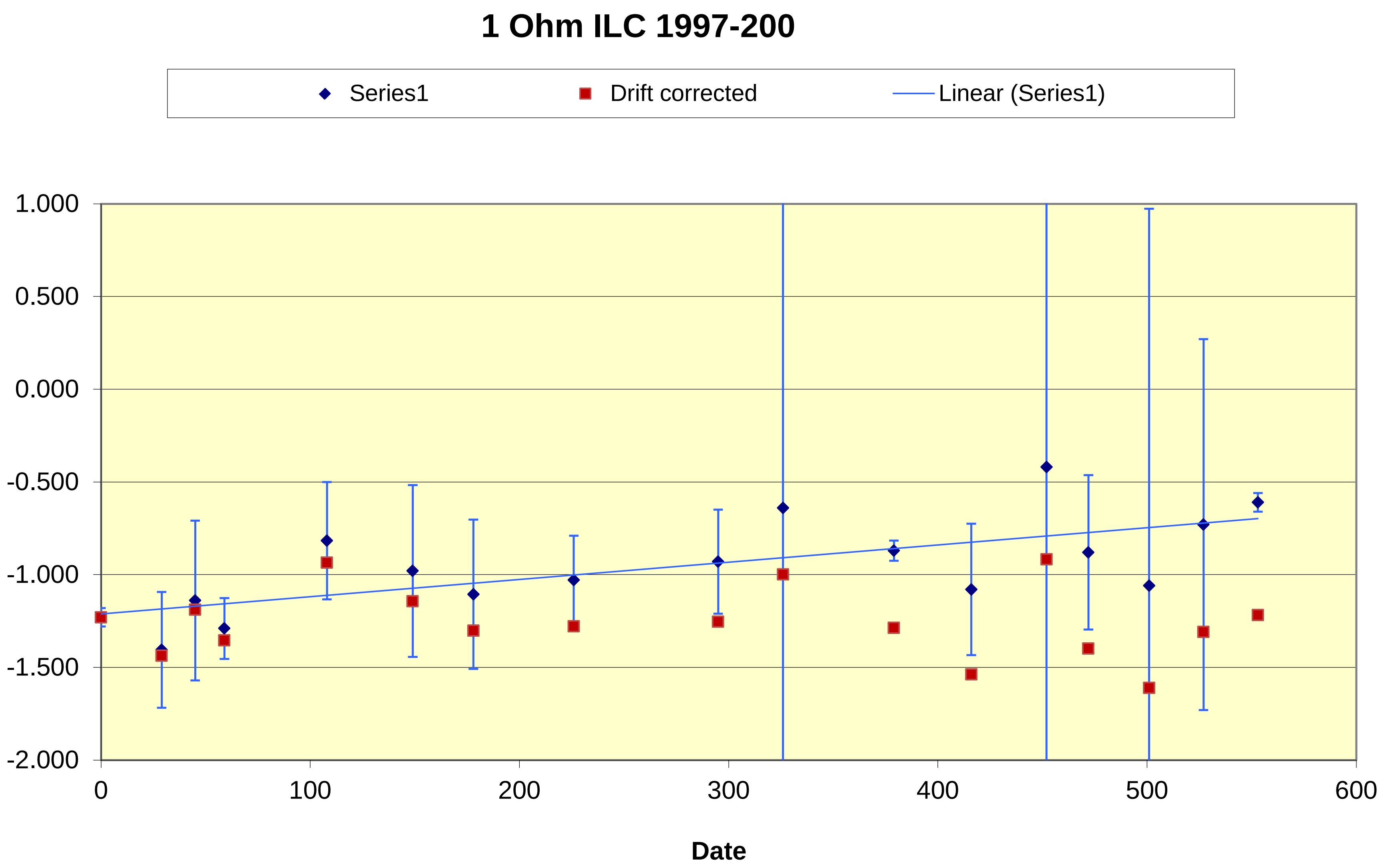
Intercept	-1.241594148
Slope	0.001099652

398.971
Sum Square

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.

Lowest value here = BEST SLOPE

Using the slope value, Remove the drift from each measurement



Calculate the Comparison Reference Value (CRV)

	Drift Correted Value	σ	σ^2	xi/σ^2	1/σ^2
NIST	-1.230	0.03	0.000625	-984.000	800.000
	-1.438	0.16	0.024336	-29.542	20.546
	-1.189	0.22	0.046440	-12.807	10.767
	-1.355	0.08	0.006724	-100.750	74.360
	-0.936	0.16	0.024964	-18.742	20.029
	-1.144	0.23	0.053592	-10.672	9.330
	-1.302	0.20	0.040602	-16.030	12.315
	-1.279	0.12	0.014400	-44.393	34.722
	-1.254	0.14	0.019600	-32.000	25.510
	-0.998	1.02	1.040400	-0.480	0.481
	-1.288	0.03	0.000729	-883.243	685.871
	-1.537	0.18	0.031329	-24.537	15.960
	-0.917	2.16	4.663440	-0.098	0.107
	-1.399	0.21	0.043264	-16.169	11.557
	-1.611	1.02	1.033272	-0.780	0.484
	-1.310	0.50	0.250000	-2.619	2.000
NIST	-1.218	0.03	0.000625	-974.486	800.000

Σ(xi/σ^2)/Σ(1/σ^2)	-1.249
σ(wm)=sqrt(1/Σ(1/σ^2)	0.02
2σ=	0.05

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

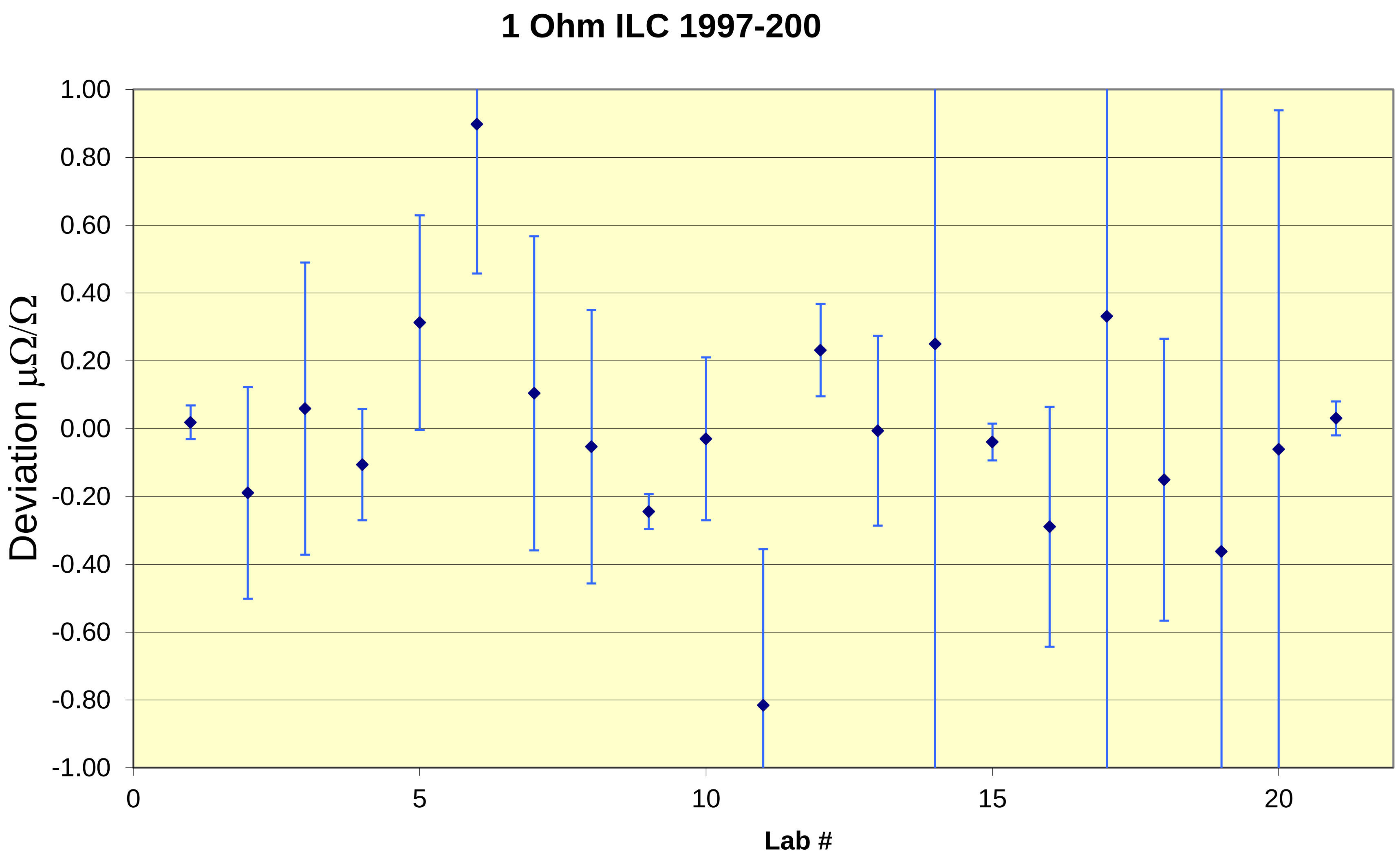
Note: As this is a comparison of different laboratories each laboratory should only contribute once to the CRV. The CRV is a reference value for the artifact against which all the laboratories measurements will be compared. All the participants who are not outliers contribute to this value, the laboratories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



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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

$$En = \frac{x - X}{\text{sqrt}(Uref^2 + Ulab^2)}$$

En = Normalized error

x = participants results

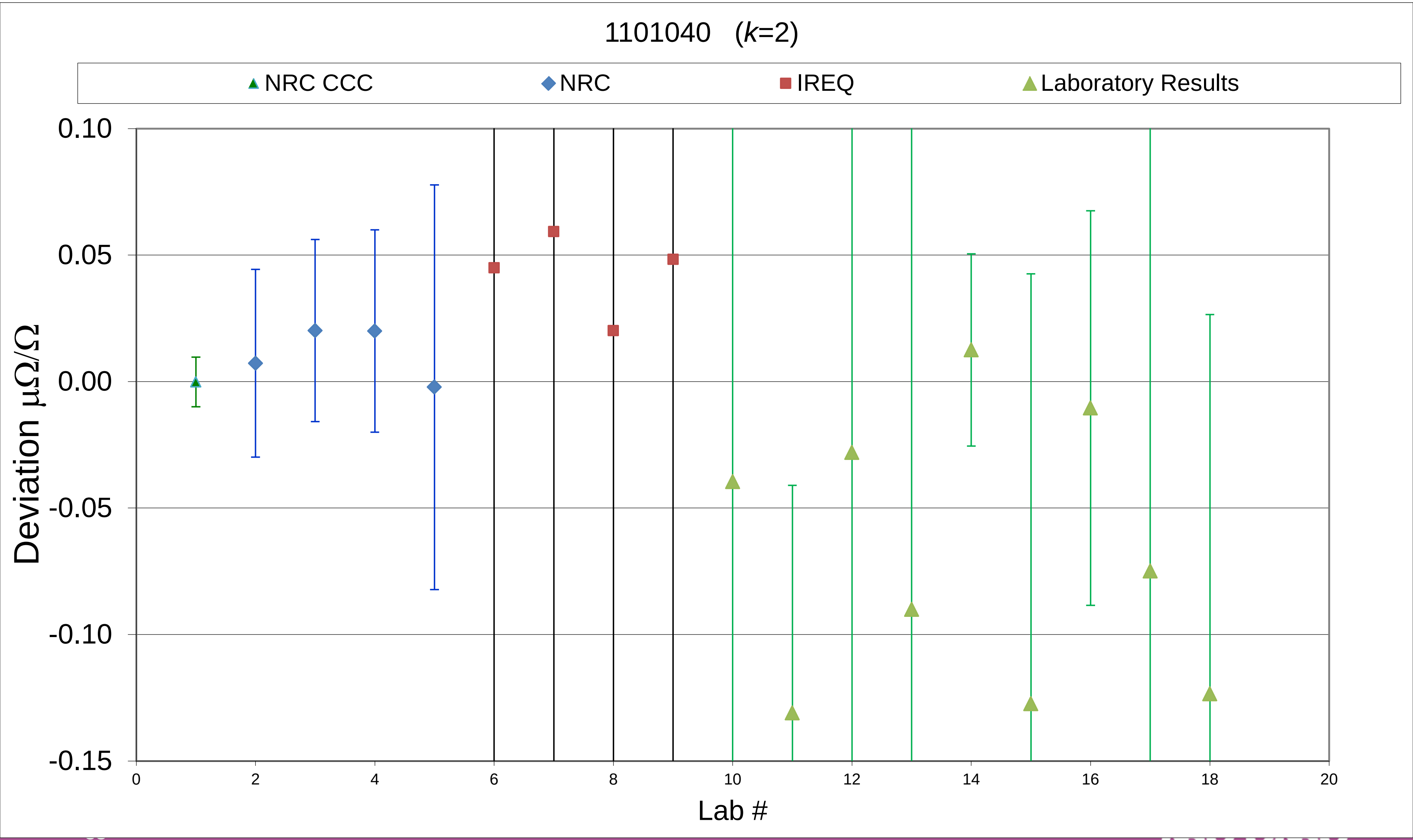
X = Reference value

Ulab = participants uncertainty (k=2)

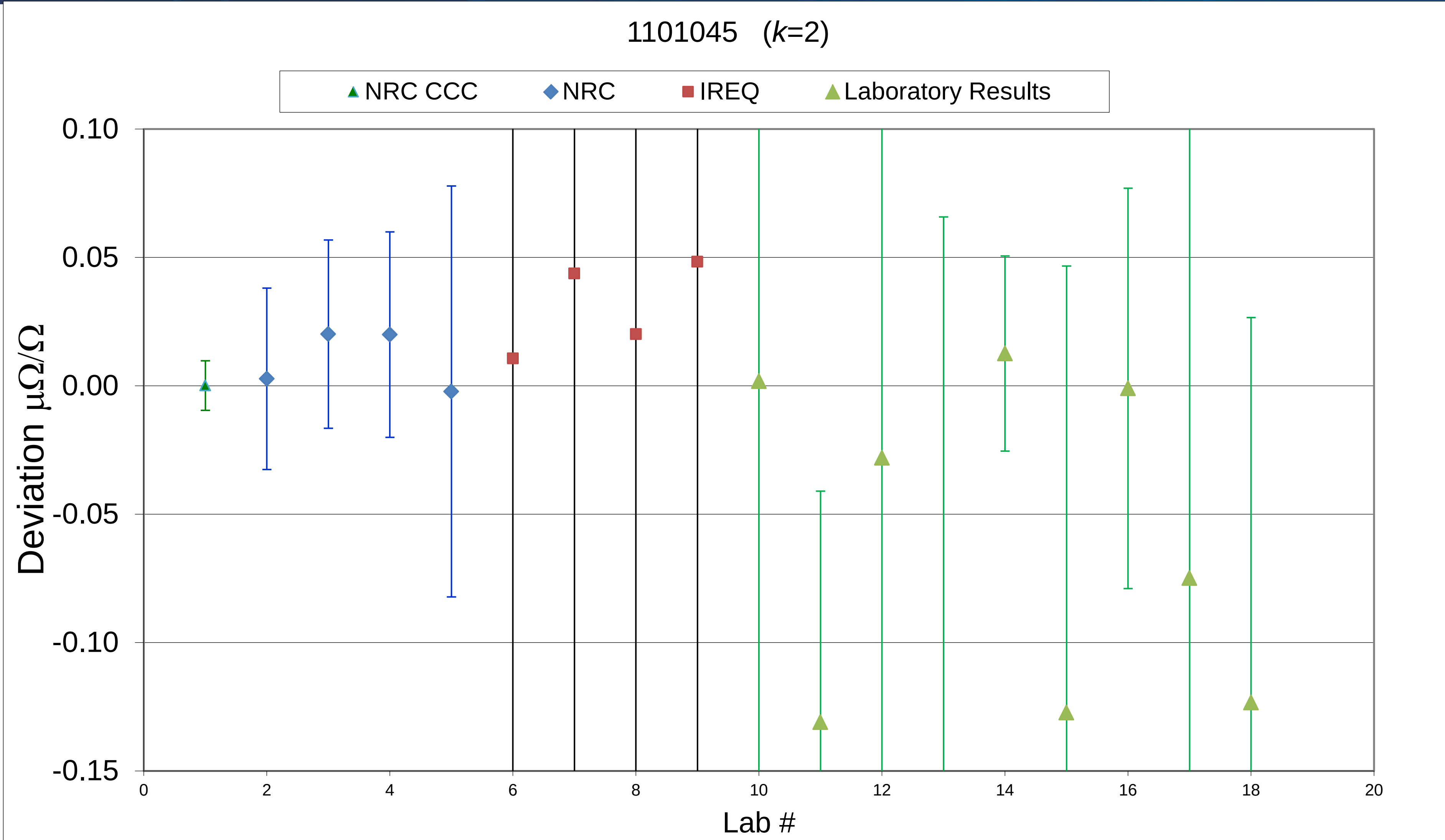
Uref = Reference value uncertainty (k=2)

En > 1 is not satisfactory

Results so far 1101040



Results so far 1101045



NRC·CNRC

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?

Many Thanks

NRC
Dave Inglis
Carlos Sanchez
Marcel Côté

IREQ
Syvain Bérubé
André Langlois
Benoit Buchard

NCSLI
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