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LABORATOIRE NATIONAL DE MÉTROLOGIE ET D'ESSAIS

INRiM
ISTITUTO NAZIONALE DI RICERCA METROLOGICA

CALIBRATION OF VERY HIGH VALUE RESISTORS

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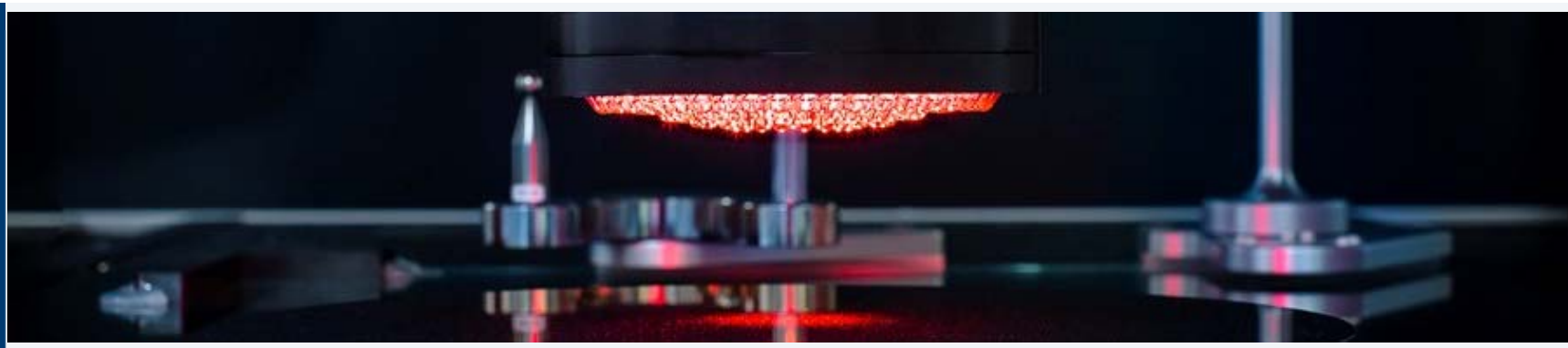
23th September 2024

CONTENT

Context of collaboration « High value resistor measurements »



LNE setup running since **2005** and validated by comparisons

LNE 2011: EM-S32 comparison of resistance standards at 1 TΩ and 100 TΩ

LNE 2018: EM-S44 comparison of Ultra Low DC current sources

➡ **Improvements are identified and necessary!**

LNE 2022: MF project proposal – not presented

LNE 2023: SRT n°i07 MUSICA project proposal by Luca Callegaro (INRiM), not accepted for European funding.

NEEDS REMAIN!

➡ **2024: 1 month LNE-INRIM collaboration.**

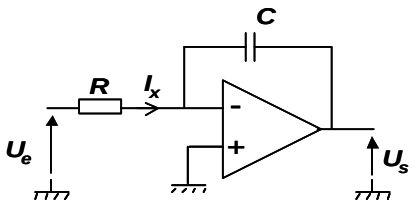
INRiM 2022: Metrological triangulation rules in ratio measurements of high standard resistance bridges, May 2022 DOI: 10.13140/RG.2.2.12240.58888 T.R. 19/2022

INRiM 2023: Low–frequency noise analyses in measurements of high standard resistance bridges November 2023, DOI:10.13140/RG.2.2.15636.88964, T.R. 30/2023

CONTEXT OF COLLABORATION: RESISTOR > 100 GΩ

LNE APPROACH

Measure the **A**bsolute value of a resistor using the **C**apacitor **C**harging method (CCA).



$$I_x = -C \cdot \frac{dU_s}{dt}$$

$$R_x = \frac{U_e + e_i}{I_x}$$

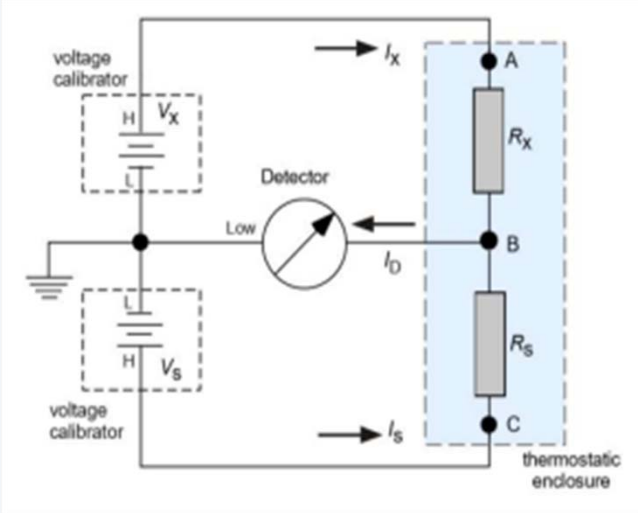
LNE-INRIM COLLABORATION

Compare the resistor value measured with LNE setup and the calculated value of the same resistor using the ratio from the INRIM setup.

$$R_x^{LNE} \leftrightarrow R_{x,calc}^{INRIM} = r_{INRiM} \cdot R_s^{LNE}$$

INRIM APPROACH

Measure the ratio of 2 resistors using the modified Wheatstone bridge (CBR – Commercial Bridge Ratio).



$$r = \frac{R_x}{R_s} = \left| -\frac{V_x}{V_s} \right|$$

$$I_D = I_s + I_x = \frac{V_s}{R_s} + \frac{V_x}{R_x}$$

INRIM HIGH VALUE RESISTORS

Summary of resistors and accessories

1. Standard resistor 1 PΩ GUILDLINE
   MOD 9337-1P
   SN 72587
   INRIM 20-001707

2. Standard resistor 100 TΩ MI
   MOD 9331G-100T
   SN 1101170 (IENMEZHR015)
   INRIM 20-001706 (264.09)

3. Standard resistor 100 TΩ GUILDLINE
   MOD 9337-100T
   SN 69640
   INRIM 23-000701

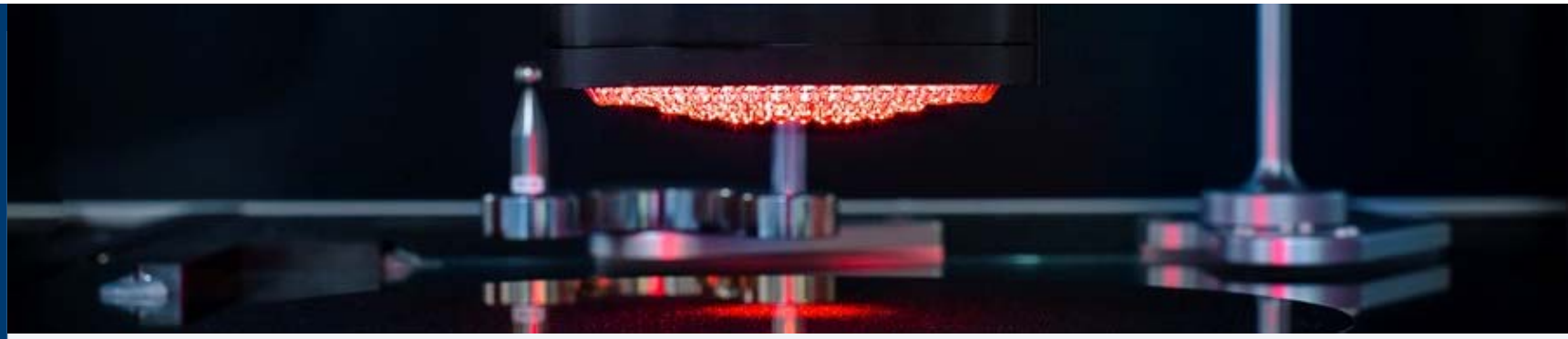
4. Standard resistor 10 TΩ MI
   MOD 9331G-10T
   SN 1101167 (IENMEZHR014)
   INRIM 20-001708 (263.09)

5. Standard resistor 1 TΩ GUILDLINE
   MOD 9337-1T
   SN 64486
   INRIM 23-001703 (IENMEZHR008)

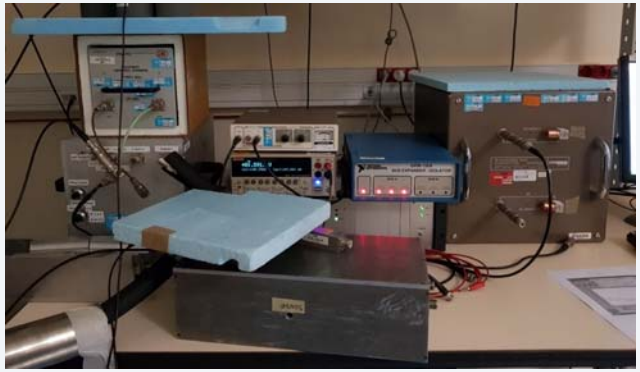
6. Accessories
   4 connectors
   2 cables coax (black) with N male connectors + 2 cables triax (yellow) with N male connectors
   4 cables coax with N male connectors

PHOTO

Schedule



WEEKS & RELEVANT ACTIVITIES



Week 1 (26/08 – 30/09)

- Welcome Iulian
- Presentations of Electrical Metrology team
- Visit of calibration laboratories
- Calibration of LNE CCA components

Week 2 (02/09 – 06/09)

- 100 TΩ: ± 100 V; ± 500 V; ± 1000 V;
- 1 PΩ: ± 100 V; ± 500 V; ± 1000 V;
- Measurements done with the setup in the initial configuration
- Some issues with the noise and external influences

➢ Re-arrangement of the setup components in a small place;
➢ Reduction of the thermal and electromagnetic influences;
➢ Remove the PC controls (therefore, the operators) away from the CCA setup.

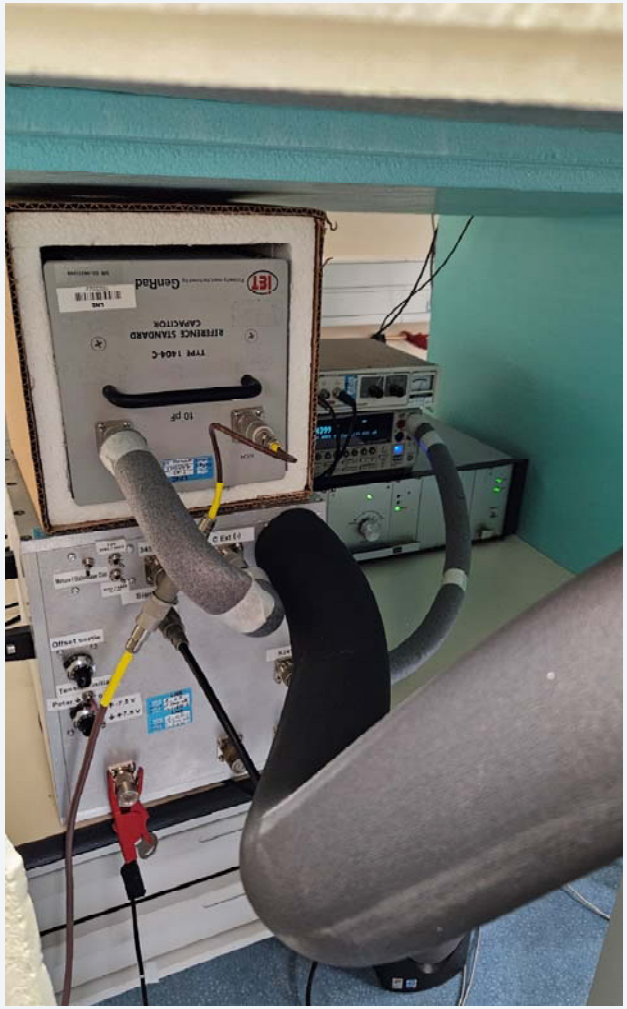
WEEKS & RELEVANT ACTIVITIES

Week 3 (09/09 – 13/09)

- 10 TΩ: ± 5 V; ± 10 V; ± 50 V; ± 100 V;
- Manufactured new low noise schielded cables
- The LNE setup was protected against air thermal flow
- Reduction of vibrations due to the door use (active laboratory)

Week 4 (16/09 – 20/09)

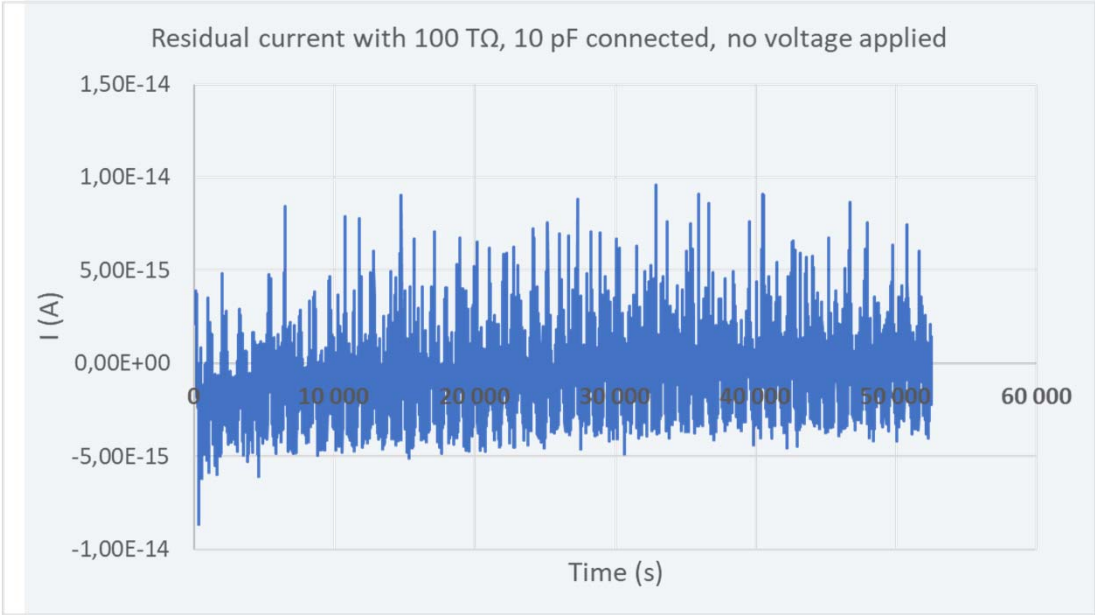
- CCA improved setup
  - 100 TΩ: ± 100 V; ± 500 V; ± 1000 V;
- Fempto amplifier based measurements (F. Piquemal)
  - 100 TΩ Guildline (network resistor): ± 1 V; ± 10 V;
  - 100 TΩ MI (bulk resistor): ± 1 V; ± 10 V;
  - 1 PΩ Guildline (network resistor): ± 1 V; ± 10 V;
- Processing measurement data & slides



2024 LNE – INRiM collaboration

ISSUES

- Many and high value spikes for residual current
- Significant discrepancy between results during polarity changes



Date	Test Voltage	Polarity	Mean value	Std of Mean	DoF
(-)	(V)	(-)	(TΩ)	(TΩ)	(-)
03-sept	100	Pos	100,0186	0,0105	21
03-sept	100	Neg	100,1902	0,0089	39

SOLUTIONS

To reduce the influence of **air thermal flow:**

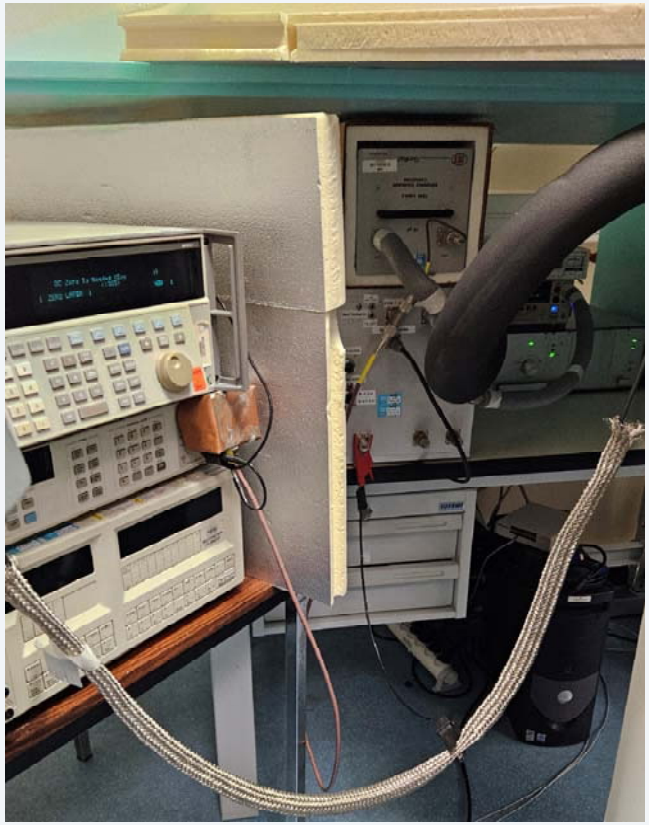
- ✓ Install thermal insulator plates to block direct flow of air conditionning at 20°C room and of the door opening.
- ✓ Setup arrangement to avoid air fluctuations by the fan of Kambic temperature chamber (hosting the high value resistors)
- ✓ All cables are covered with thick external thermal insulator and fixed to avoid their movements
- ✓ Wait for the temperature stabilization of the setup components when all were connected

To reduce the influence of **vibrations:**

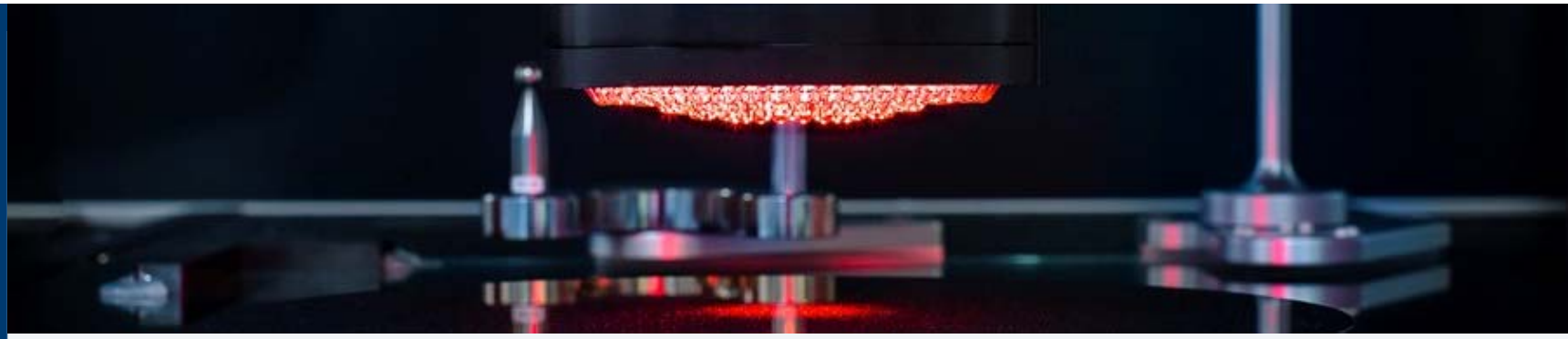
- ✓ Forbiden acces to the nearest door to the measurement room
- ✓ New cables with solid connection of the cables to connectors

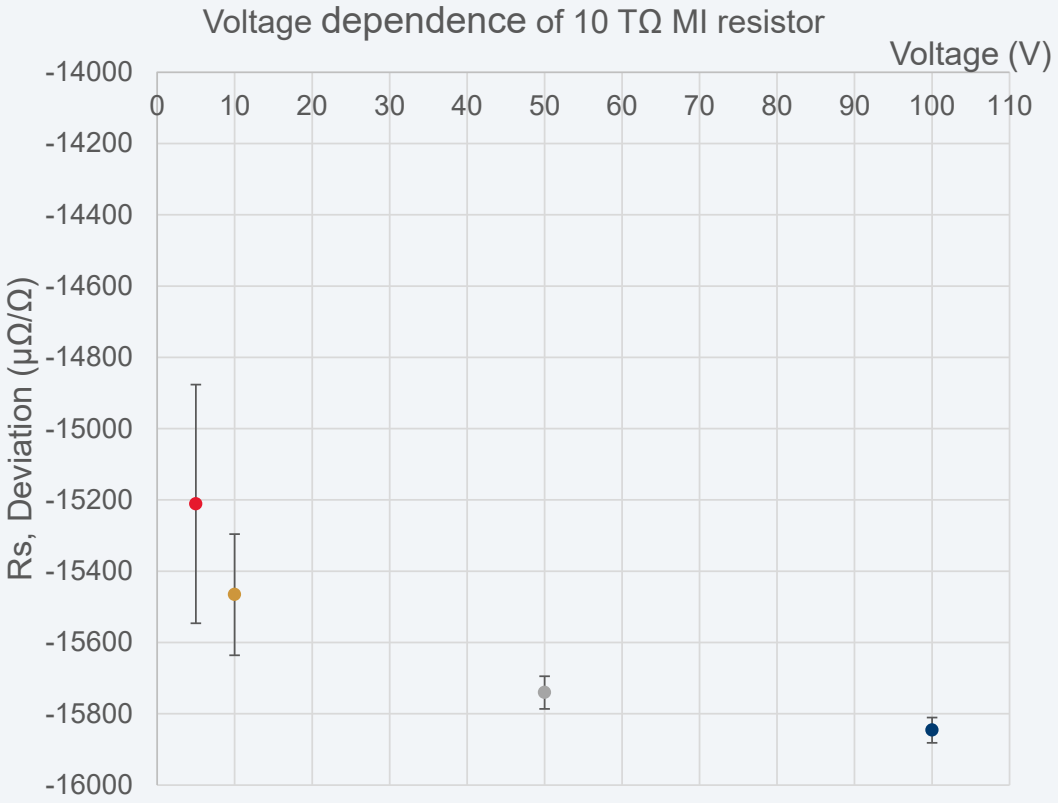
To reduce the **EMC influences**:

- ✓ Shortening the lenght of the cables
- ✓ Shielding cables with silver plated copper meshes
- ✓ Adding choke on the current path
- ✓ Connecting the shields to the common ground of the setup (to avoid ground loops)
- ✓ Using low noise schielded coaxial cables



Preliminary results





10 TΩ MI resistor

500 fA; 1 pA; 5 pA; 10 pA

Test Voltage	Rx mean of polarities	Deviation from nominal	Uncert. (k = 1)
(V)	(TΩ)	(µΩ/Ω)	(µΩ/Ω)
5	9,8479	-15211	335
10	9,8453	-15466	170
50	9,8426	-15741	46
100	9,8415	-15846	36

PRELIMINARY RESULTS

100 TΩ Guildline resistor

100 fA; 500 fA; 1 pA; 5 pA; 10 pA

Test Voltage (V)	Rx mean of polarities (TΩ)	Deviation from nominal (μΩ/Ω)	Uncertainty (k = 1) (μΩ/Ω)
10	100,0833	833	1650
50	100,1262	1262	400
100	100,1010	1010	280
500	100,1504	1504	225
1000	100,1504	1504	225

★ Measurements performed at 1 V and 10 V using Femto amplifier (F. Piquemal) – will be processed in the near future.

PRELIMINARY RESULTS

1 PΩ Guildline resistor

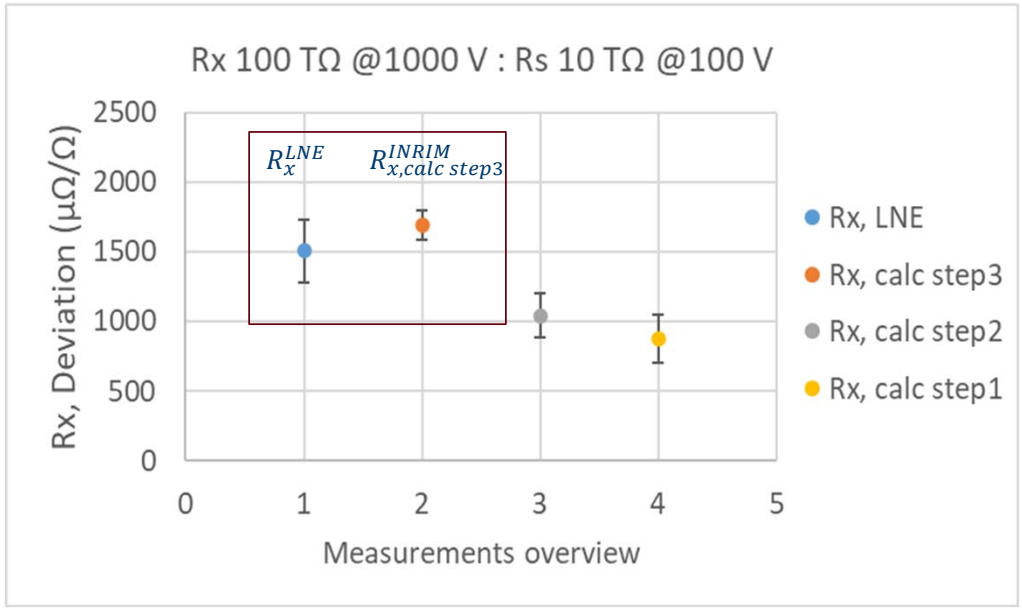
500 fA; 1 pA

Test Voltage	Rx mean of polarities	Deviation from nominal	Uncertainty (k = 1)
(V)	(PΩ)	(µΩ/Ω)	(µΩ/Ω)
☆ 500	1,00646	6459	2250
1000	1,00681	6811	2250

☆ This measurement is repeated with the improved setup this week.

★ Measurements performed at 1 V and 10 V using Femto amplifier (F. Piquemal) – will be processed in the near future.

PRELIMINARY RESULTS OF LNE – INRIM COMPARISON

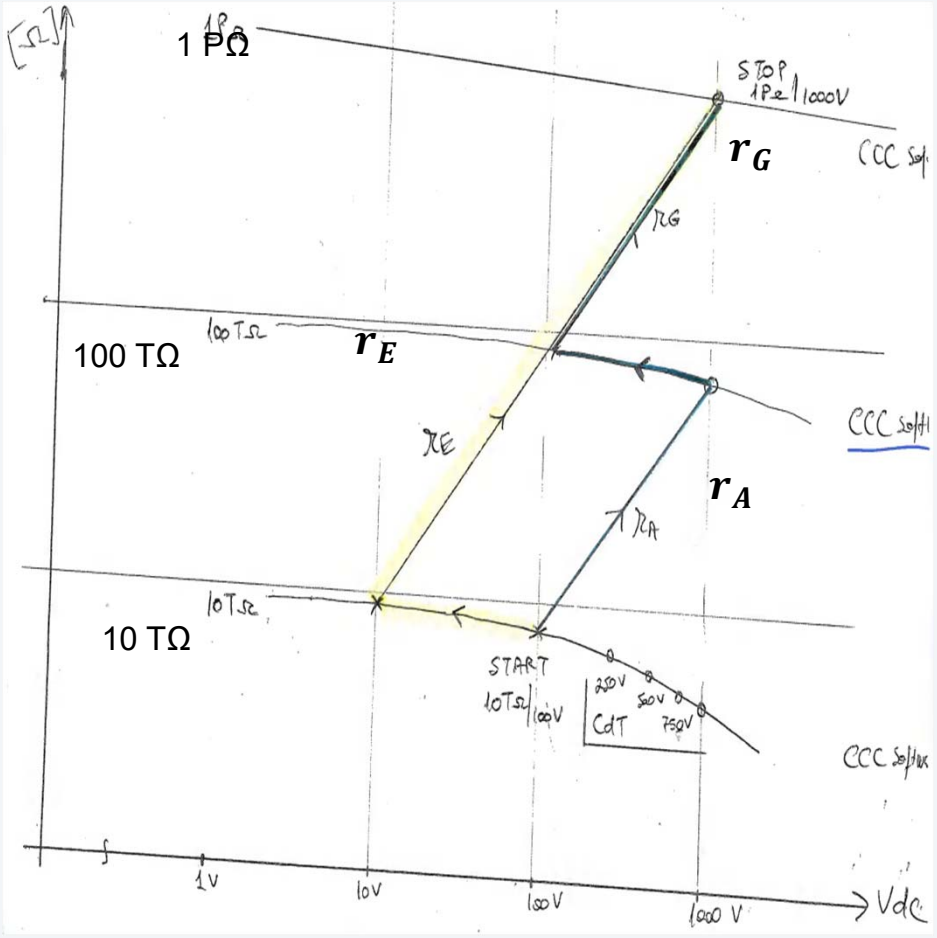


Note: The uncertainties are shown as the standard deviation of the mean.

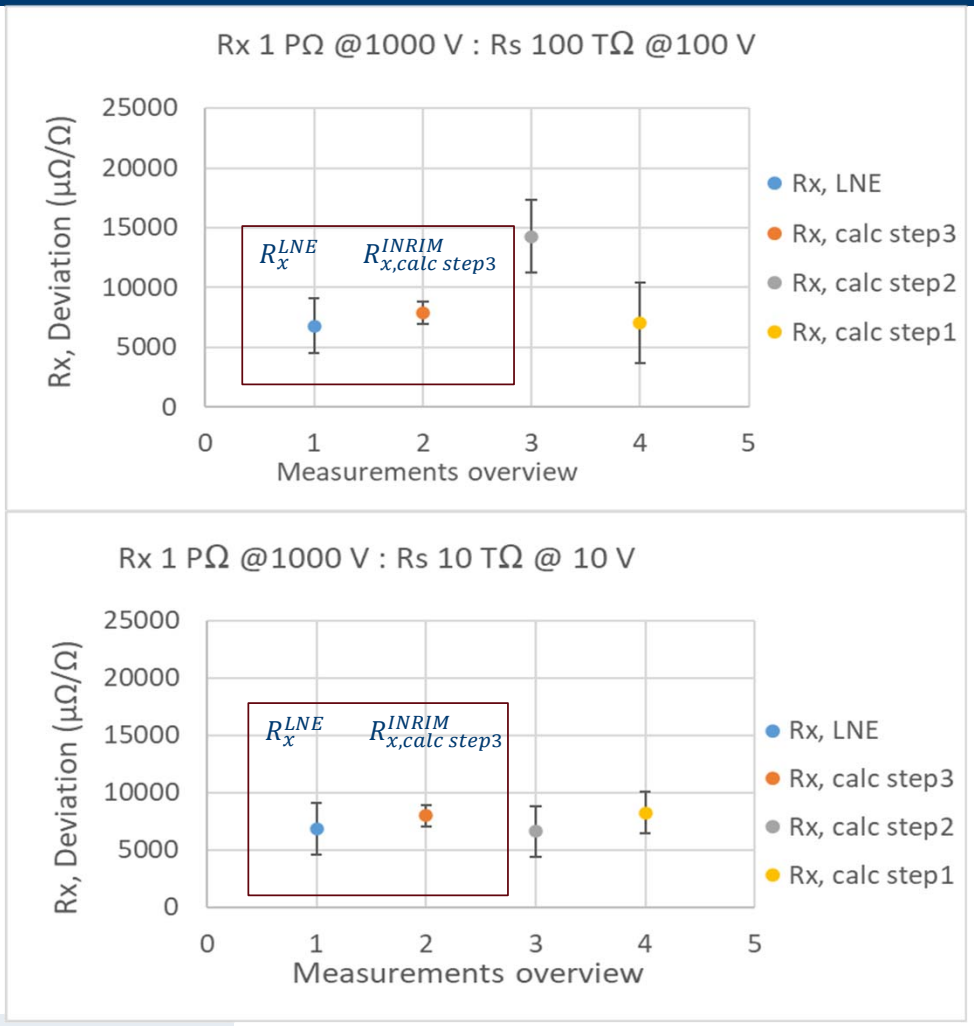
$$R_x^{LNE} \leftrightarrow R_{x,calc}^{INRIM} = r_A \cdot R_s^{LNE}$$

Rx, 100 TΩ Guildline – network resistor

Rs, 10 TΩ MI – bulk resitor



Preliminary results of LNE – INRiM comparison



$$R_x^{LNE} \leftrightarrow R_{x,calc}^{INRIM} = r_{INRiM} \cdot R_s^{LNE}$$

$$u_{Rx,Rs}^{LNE} \rightarrow CMCs$$

$$u_{Rx,calc}^{INRiM} = \sqrt{u_{rINRiM}^2 + u_{Rs}^2}$$

Rx, 1 PΩ Guildline – network resistor

Rs, 100 TΩ Guildline - network resistor

and

Rs, 10 TΩ MI – bulk resitor

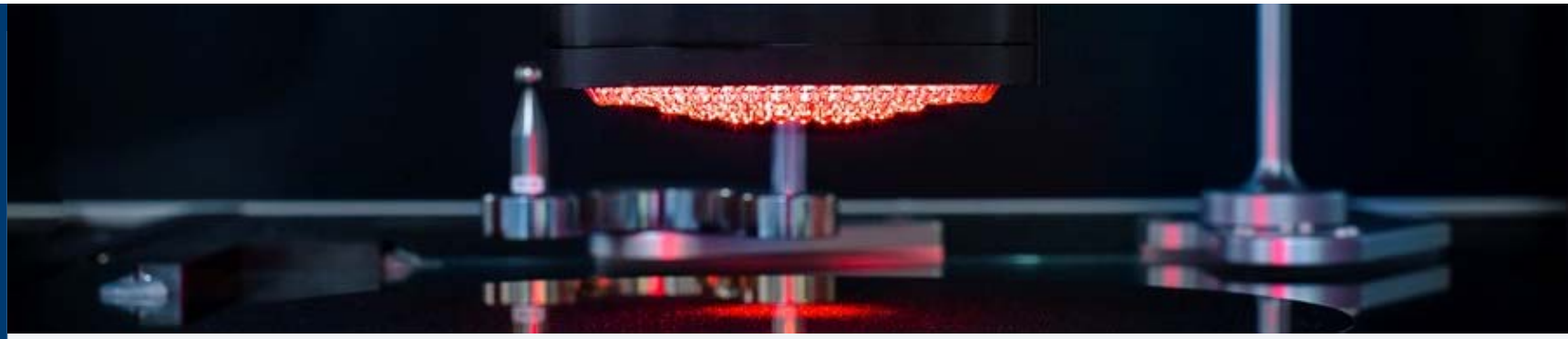
Value of r_{INRiM} comes from the TR 35/2023.

Condition of triangulation among 3 resistors:

$$abs\left(1 - \frac{\overline{r_A} \cdot \overline{r_G}}{\overline{r_E}}\right) \cdot 10^6 < \sqrt[2]{u_{rA}^2 + u_{rE}^2 + u_{rG}^2}$$

u_{rA}, u_{rE}, u_{rG} - relative standard deviation of the mean for the ratios rA, rE, rG (r_{INRiM})

Conclusions and future works



- Good preliminary results
- Further data processing
- The triangulation condition has been obtained for measurement at 1000 V and the resistors

  1 PΩ (Guildline) : 100 TΩ (Guildline): 10 TΩ (MI)

- In EURAMET EM-S45 ULCA comparison the LNE Capacitor Charging setup will be used with implemented improvements

Future works

- Commun technical report
- Obtention a robust method to calibrate HVR (High Value Resistors)
- Mitigate the risk of errors (extrapolation, systematic etc.) as required by ISO/IEC 17025
- Co-authored journal paper
- Measurements of LNE resistors by INRIM setup to validate the method of the triangulation among HVR
- Possible future common PRT
- LNE: new up-to-date setup development (PhD thesis)

Thank you for your attention.