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A New Impedance Metrology Infrastructure at GUM

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***Abstract* – Collaboration of the Polish National Metrology Institute (GUM, Warsaw) with the Silesian University of Technology (SUT, Gliwice) and joint participation in international projects focused on the development of digital sources and digital impedance bridges contributed to the development of the impedance metrology infrastructure at GUM. The article presents current work related to expanding the current infrastructure with a new 4TP, mostly thermostated impedance standards terminated with MUSA connectors. Design solutions, results of thermal stability measurements and results of the comparison of new impedance standards made with the currently implemented digital impedance bridge are presented in the paper.**

I. INTRODUCTION

For almost 60 years, it has been known that a four-terminal pair (4TP) definition achieves the highest accuracy in impedance measurement [1]. The 4TP definition requires more complex measurement setups than simpler impedance definitions, but on the other hand, it reduces to a minimum the dependence of the measurement outcome on the effects of electrical connections and circuit layout details [2-6]. Consequently, a 4TP definition allows the best accuracy in impedance measurement to be achieved. Unfortunately, although some commercial LCR measuring instruments like HP 4284, GenRad 1693, or Agilent E4980A are designed for 4TP measurements, manufacturers of impedance standards rarely equip them with four coaxial connectors. This prompts national metrology institutions (NMIs) to develop and construct their own solutions or modify the present standards to fit them to the 4TP definition. With the beginning of the present century, many NMIs started to replace their impedance standards with BPO MUSA connectors or construct new ones [7-11]. At the NMI level, the accuracy and frequency range are steadily increasing; hence, the role of the 4TP definition will be very important.

The newly available technology of digital impedance bridges, developed in the framework of recent joint research projects (in particular SIB53 AimQuTE [12] and 17RPT04 VersICaL [13]), has shown the potential to realize SI impedance units and scales at GUM at a reduced cost, and thus provide new primary-level electrical impedance calibration services for industry and calibration centres. Due to the implementation at GUM of a new 4TP sampling-based digital impedance bridge, it was necessary to adapt the current infrastructure to the new measuring system. This is especially important in terms of impedance standards. Therefore, thermostated sets of RLC standards were designed and constructed, which are described in detail in Section 2. When designing the standards, it was considered that such standards usually work in a laboratory with a temperature variable within the limits of 23±1ºC. The target impedance comparison uncertainty is at the level of few ppm, therefore, taking into account the temperature coefficients of the available *R* and *C* components of the standards (the maximum values are up to 30 ppm/ºC), thermoregulation of these objects is necessary. In such a case, thermoregulation at the level of hundredths of a degree causes that the influence of the standard value fluctuations caused by the temperature change may be neglected in the final uncertainty calculation. The assumed operating frequency of the standards covers the range from 20 Hz to 20 kHz.

II. 4TP IMPEDANCE STANDARDS

A. Capacitors

In 2018-2021, three sets of thermostated capacitance standards were built successively at GUM: 1) KC0G-10nF, 2) KC0G-1XF L52-1/20; and 3) KC0G-1XF L52-2/21 (see Fig. 1). KC0G-1XF L52-1 and L52-2 consist of five capacitance standards: 1 nF, 10 nF, 100 nF, 1 µF and 10 µF. All standards use SMD 1206 GMR31 series C0G capacitors from Murata, Japan [14]. The C0G (NP0) dielectric is the most popular formulation of the “temperature-compensating,” EIA Class I ceramic materials. Modern C0G formulations contain neodymium, samarium, and other rare earth oxides. C0G ceramics offer one of the most stable capacitor dielectrics available. The change in capacitance with temperature is 0 ± 30ppm/°C. Capacitance drift or hysteresis for C0G (NP0) ceramics is less than ± 0.05% versus up to ± 2% for films. The typical capacitance change with life is less than ± 0.1% for C0G (NP0), one-fifth that shown by most other dielectrics. Moreover, C0G (NP0) formulations show no aging characteristics.

Stages of the construction of the capacitance standards are presented in Fig. 2. The SMD capacitors are first embedded in natural pure wax, which has a positive effect on the long-term stability of the parameters of the standard. Then each single capacitor is inserted into the shielded housing visible in the upper left photo in Fig.2. After that, the capacitors are placed in an enclosure with heating foil and insulated with an aerogel mat. The aerogel used has low density and extremely low thermal conductivity 0.014 W/(m·K). Aerogels are usually made based on silica, although recently carbon nanotubes and graphene have also been utilized. There are also aerogels based on zeolites and aluminoxanes, but silica is by far the most widely used.

The temperature controller stabilizing temperature of the capacitors uses an NTC temperature sensor. An additional Pt100 sensor serves as temperature monitor. Both sensors are mounted inside the case. The controller is a simple and rugged on/off electronic controller, mounted within the thermal insulation (see Fig. 1), and adjusted to operate at approximately 26.5ºC with 0.01ºC hysteresis. This temperature results from the fact that, according to the specifications of the manufacturers of multilayer ceramic capacitors (MLCC) [14], the smallest changes in capacitance along with temperature changes can be expected around 26-27ºC. The temperature controller of the set of standards is supplied from a +12 V DC power supply through a 4-pin socket. It is recommended to use a battery during calibration. However, a power supply can be used to charge the battery. The internal temperature of the enclosure is measured using resistance thermometer Pt100 that can be accessed through two BNC connectors indicated with Pt100. The standards are equipped with MUSA Metrology Grade silver-plated connectors manufactured by Canford, UK. The connectors are indicated with H_{CUR} and L_{CUR} for the current high and current low, respectively, and H_{POT} and L_{POT} for the potential high and potential low, respectively.

Fig. 1. 4TP resistance and capacitance standards used at GUM and SUT (two sets of thermostated capacitors and one set of thermostated Vishay resistors – at the bottom from the left, and 10 nF thermostated capacitors with battery – at the top.

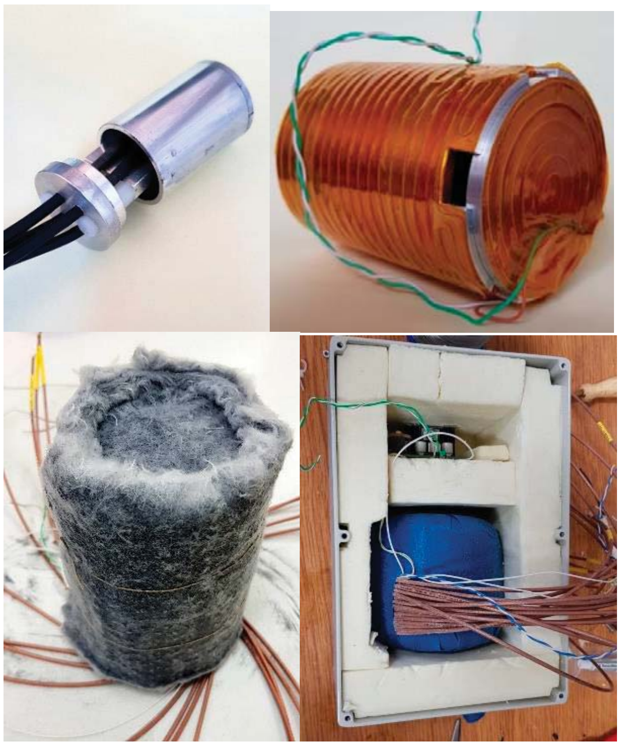

Fig. 2. Stages of construction of the thermostated set of standard capacitors (shielded housing with a single capacitor – top left, enclosure with heating foil – top right, set of five capacitors mounted in the heating enclosure and insulated with aerogel – bottom left, complete set without cover – bottom right)

B. Resistors

A set of five 4TP resistance standards was manufactured at SUT in 2021 (Fig. 3). The standards have nominal values of: 10 Ω, 100 Ω, 1 kΩ, 10 kΩ, and 100 kΩ. Ultra-high precision Vishay HZ-series (Z-foil) resistors were used to fabricate these standards [15]. Since the temperature coefficient of resistance is ± 0.2 ppm/°C there is no need to mount them in the thermostat.

Another set of resistance standards was manufactured at GUM (see the rightmost standard in Fig. 1). High precision Vishay H-series bulk metal foil resistors were used to fabricate these standards [16]. Since the resistance temperature coefficient for this type of resistor is ± 2 ppm/°C, the temperature of the standards is controlled in the same way as described for the capacitance standards in Chapter II.A. The standard was indicated as Vishay-1XΩ, Nr L52-1/21.

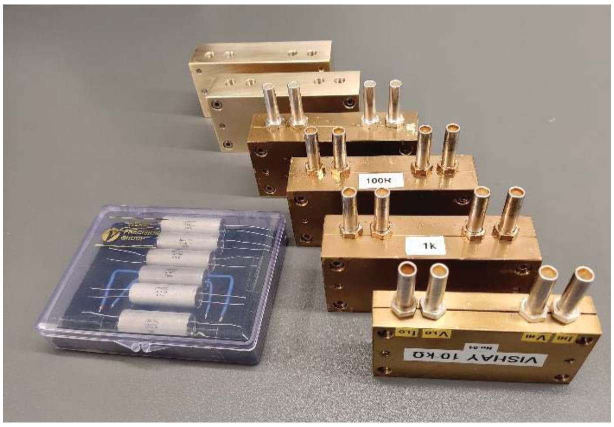

Fig. 3. 4TP resistance standards manufactured at SUT (Vishay HZ resistors with brass body, terminated with MUSA connectors)

C. Inductors

Calibration laboratories maintain traceability to national standards using 2T or 3T inductance standards. When such standards are used to calibrate a 4TP impedance meter, the standard must be reconverted from 2T or 3T to 4TP with suitable adapters [9] that are not commercially available, what reduces the calibration accuracy. It is therefore advisable to replace the three original binding post terminals with a 4TP coaxial connectors (e.g. BPO MUSA [7, 8]). Currently at GUM all inductance standards are 3T. Some of them were modified to be thermostated (see Fig. 4). The thermostat stabilizes the internal temperature within 0.01°C for ambient temperature within 18-25°C. However, it was decided to manufacture custom MUSA adapters and tees (see the lower right corner of Fig. 4) that enable one to meet the 4TP definition.

Fig. 4. Thermostated standard inductor 1482-L. Modified by Ukrainian NMI. The MUSA adapter and the MUSA tees are visible in the small window at the bottom right

D. Temperature stability of the standards

Temperature stability of the capacitance and resistance standards presented in Fig. 1 was examined at ambient temperature of 23.5±1.0ºC using a four-wire resistance measurement of the Pt100 sensor built into the standards. Measurements were made using an Agilent 3458A precision multimeter and software made in the LabView environment. The temperature of each of the standards was monitored in two seconds intervals from the moment the power was turned on (that correspond to the ambient temperature) until the steady state temperature was reached. The measurement results are presented in the Fig. 5 and Fig. 6 for capacitance standards and for resistance standard, respectively.

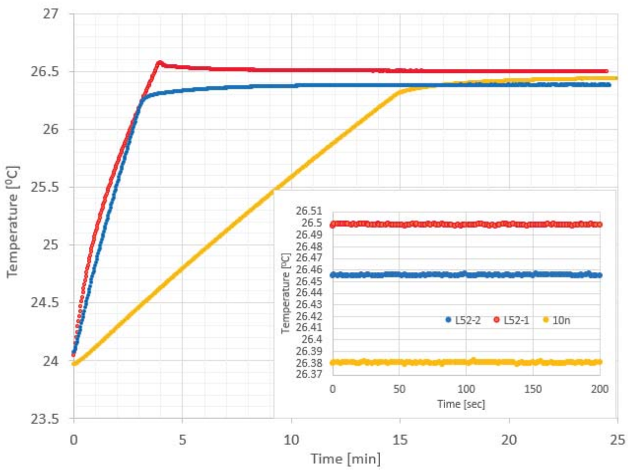

Fig. 5. Temperature stability of capacitance standards KC0G-10nF, KC0G-1XF L52-1/20 and KC0G-1XF L52-2/21

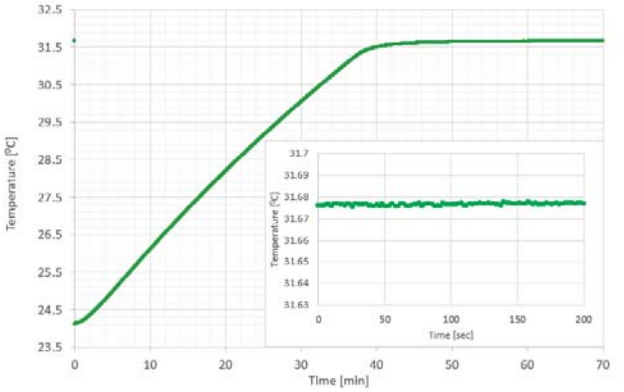

Fig. 6. Temperature stability of resistance standard Vishay-1XΩ, No. L52-1/21.

The last 100 measurement results obtained in the steady state are shown in the small windows inside the graphs. It is visible that temperature changes in the steady state do not exceed 0.01°C.

III. 4TP BRIDGE

A 4TP sampling-based digital impedance bridge developed at SUT is currently being implemented at GUM. Fig. 7 shows the schematic of the sampling-based GUM bridge and Fig. 8 shows the photograph of the bridge. Detailed description of the bridge and the high-performance source used to supply the bridge are given in [15] and [16], respectively.

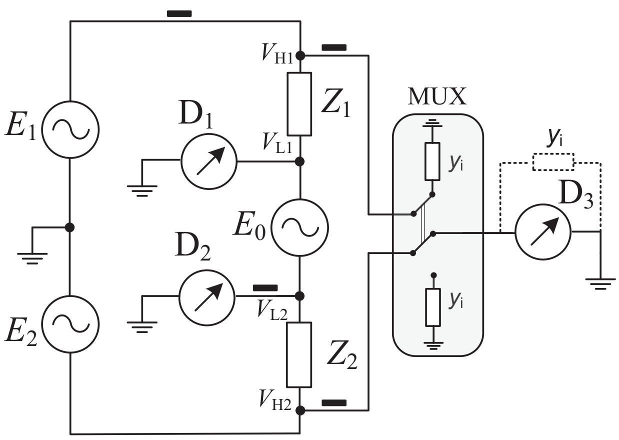

Fig. 7. Basic schematic of the GUM 4TP bridge. Solid black rectangles represent coaxial current equalizers (chokes) and y_i represents input admittance of the digitizer D_3

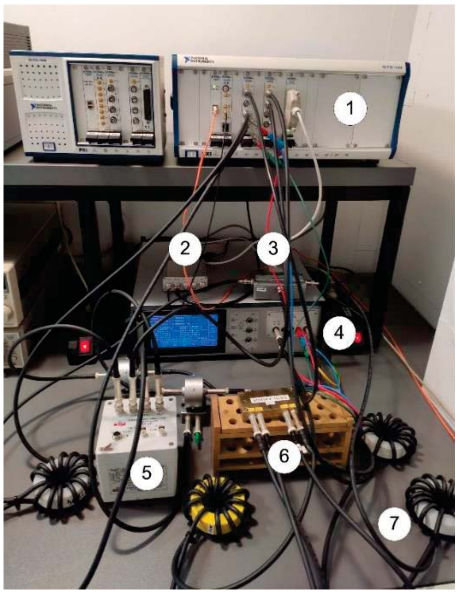

Fig. 8. Photograph of the bridge: 1- NI PXI sampling system, 2-opto/TTL converter, 3- multiplexer, 4- DSS_2CH two-channel AC voltage source, 5- thermostated 4TP capacitance standard KC0G-10nF , 6- 4TP standard Vishay resistor, 7-current equalizer.

IV. COMPARISONS

The construction of appropriately stable impedance standards encouraged us to perform further validation tests of the implemented bridge. Initially, two new sets of capacitance standards (KC0G-1XF L52-1 and L52-2) were calibrated at GUM using the ultra-precision capacitance bridge Andeen-Hagerling AH2700A to determine their reference values. In turn, the third new capacitor KC0G-10nF was calibrated at INRiM using the three-voltmeter method. Next, four 4TP Vishay standard resistors (100 Ω, 1 kΩ, 10 kΩ, and 100 kΩ) presented in Fig. 3 were calibrated through R-C comparison using the new 4TP sampling-based bridge developed at SUT.

V. CONCLUSIONS

The article presents new design solutions of impedance standards, which have been recently implemented at GUM. Only initial tests have been presented for some newly developed standards. No disturbing behavior was observed during the comparison for any of the thermostated capacity standards. The monitoring of the standards temperature with the use of the built-in Pt100 sensor indicates a relatively quick (less than 30 minutes) achievement of the steady state of the capacitance standards and shown that the temperature fluctuations are within 0.01 K. Thus, the component of the uncertainty of comparison related to the instability of the standard can be ignored in this case. The *R-C* comparison performed with the use of the 4TP sampling-based bridge has shown

Table. 1. R-C Comparison results (the value of the unknown standard resistors was calculated from the R-C ratio measured by the new 4TP digital impedance bridge at GUM)

Standards compared	Reference value			DUT		Difference
	C, nF	$u_{rel}(C)$, µF/F	tgδ, ×10^{-5}	R, kΩ	τ, ×10^{-5}	ΔR, µΩ/Ω
100 Ω vs. 1 µF (L52-1/20)	1000.035	70	65	0.09999938	11	
100 Ω vs. 1 µF (L52-2/21)	1000.020	70	75	0.10000004	10	6.6
1 kΩ vs. 100 nF (L52-1/20)	99.99634	35	8.6	1.0000170	0.93	
1 kΩ vs. 100 nF (L52-2/21)	100.00397	35	8.6	1.0000193	0.75	2.3
10 kΩ vs. 10 nF (L52-1/20)	10.000157	2	1.9	10.000153	0.67	
10 kΩ vs. 10 nF (L52-2/21)	10.000531	2	5.7	10.000106	0.95	4.7
10 kΩ vs. 10 nF (L52-1/20) *	10.000115	2	2.4	9.9999898	0.66	
10 kΩ vs. 10 nF (KC0G-10n) *	9.9999453	2	4.3	9.9998810	0.7	1.0

high consistency of the results obtained for a standard resistor compared with two different capacitors. In addition to the high consistency of the active components, a good agreement of the reactive components (τ) was also observed. The results of the comparison presented in the article, which are only a part of the tests performed to validate the new 4TP digital bridge, demonstrate the high accuracy and usefulness of the bridge for standard comparison at GUM. Currently, GUM and SUT are working on the inclusion of the new bridge in the national system of measures, which is associated with the development of an uncertainty budget for all new comparisons and the declaration of a new CMC.

REFERENCES

[1] R. D. Cutkosky, "Four-terminal-pair networks as precision admittance and impedance standards," Commun. Electron., vol. 70, pp. 19–22, Jan. 1964.

[2] S. Awan, B. Kibble, and J. Schurr, Coaxial Electrical Circuits for Interference-Free measurements (Electrical Measurement). Edison, NJ, USA: IET, 2010.

[3] L. Callegaro, "Electrical Impedance: Principles, Measurement, and Applications", CRC Press, 1st edition, 2012.

[4] AR. Calhoun, "Four terminal pair concepts," Cal Lab, pp. 38–41, Nov./Dec. 1996.

[5] B. P. Kibble, "Four terminal-pair to anything else!" in Proc. IEE Colloq. Interconnections From DC Microw. (Ref. No. 1999/019), London, U.K., Feb. 18, 1999, pp. 6/1–6/6.

[6] B.P. Kibble, "Proposals for Extanding Traceable Impedance Measurements to Higher Frequencies", Metrologia, vol. 35, No. 1, pp. 17-20, 1998.

[7] L. Callegaro, V. D'Elia, F. Francone, and D. Serazio, "100 mH travelling standard for the EUROMET 607 pilot intercomparison," in CPEM Tech. Dig., Ottawa, ON, Canada, Jun. 16–21, 2002, pp. 352–353.

[8] L. Callegaro, V.D'Elia and D. Serazio, "10-nF Capacitance Transfer Standard", IEEE Trans. Instr. Meas., Vol. 54, No. 5, pp. 1869-1872, 2005.

[9] E. Dierikx, J. Melcher, L. Callegaro, "Final report on the supplementary comparison EURAMET.EMS26: inductance measurements of 100 mH at 1 kHz" (EURAMET project 816), Metrologia 49 (1A), January 2012.

[10] L. Callegaro, V. D'Elia and J. Bohacek, "Four-Terminal-Pair Inductance Comparison Between INRIM and CTU," in IEEE Transactions on Instrumentation and Measurement, vol. 58, no. 1, pp. 87-93, Jan. 2009

[11] L. Callegaro, V. D'Elia, F. Francone and D. Serazio, "100 mH travelling standard for the EUROMET 607 pilot intercomparison," Conference Digest Conference on Precision Electromagnetic Measurements, 2002, pp. 352-353.

[12] SIB 53 AIM QuTE final publishable JRP Report "Automated Impedance Metrology extending the quantum toolbox for electricity", Euramet, June 2017

[13] 17RPT04 VersICaL final publishable JRP Report "A versatile electrical impedance calibration laboratory based on digital impedance bridges", Euramet, 2020

[14] https://www.murata.com, specification of Murata GRM31C5C1H104JA01#

[15] www.foilresistors.com, Specification of Vishay HZ Series, Document Number: 63120, March 2010

[16] www.foilresistors.com, Specification of Vishay H Series, Document Number: 63006, March 2010

[17] K. Musioł, "A new sampling-based four-terminal-pair digital impedance bridge", Measurement-Sensors, Volume 18, December 2021.

[18] M. Kampik, K. Musioł, "Investigations of the high-performance source of digitally synthesized sinusoidal voltage for primary impedance metrology", Measurement, Volume 168, 2021.