FINAL PUBLISHABLE REPORT

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Internal Funded Partners: 1 TÜBITAK, Türkiye 2 BFKH, Hungary 3 BIM, Bulgaria 4 BRML, Romania 5 CMI, Czech Republic 6 FSB, Croatia 7 IMBiH, Bosnia and Herzegovina 8 JV, Norway	External Funded Partners: 9. INM, Moldova 10. ME, Montenegro	Unfunded Partners:
RMG1: IMBiH, Bosnia and Herzegovina (Employing organisation); TÜBITAK, Turkey (Guestworking organisation) RMG2: DPM, Albania (Employing organisation); TUBITAK, Turkey (Guestworking organisation)		

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1 Overview

The aims of this project were to develop novel scientific and technical capabilities to provide both the dissemination of the International Temperature Scale of 1990 (ITS-90), which is an important concept in both developed and emerging NMIs/DIs, and, in addition, accurate low uncertainty temperature measurements using a thermocouple, which is the most frequently used temperature sensor. These objectives were achieved by developing novel practical methods and devices for monitoring thermocouple performance. The new concept of dual-type thermometers developed enables in-situ monitoring of thermocouple drift, whilst our small fixed point cells can be relatively easily attached to existing thermocouple probes, also enabling validation of thermocouple performances in situ. Two novel devices for testing thermocouple inhomogeneity and drift were also developed that utilize the Curie-point technique for maintaining known and stable temperatures.

2 Need

Knowledge of the lifetime and drift of thermocouples in industrial applications is not very well known and usually, thermocouples are periodically replaced to ensure continuity in process control quality. However, it can often be very difficult for the user to detect inadequate thermocouple performance. There are no standardised traceable methods that would enable thermocouple performance verification in-situ. The stability, homogeneity, and drift of thermocouples have been investigated throughout several EMRP and EMPIR projects: 14RPT05 Eura-Thermal, 14IND04 EMPRESS, SIB10 NOTED, etc. However, more comprehensive work on this topic is still required.

This project developed novel methods and techniques, traceable to the ITS-90, that can significantly simplify and improve testing of thermocouple drift and inhomogeneity in calibration laboratories as well as in other measurement sights. These techniques are beneficial to users as they enable increase of confidence in results of thermocouple performances tests as well as decrease in the uncertainty of temperature measurement by thermocouples. As a result, the efficiency of industrial processes utilizing thermocouples for temperature measurement and control can be increased.

Through collaboration on the design and validation of new temperature measurement methods and equipment all the project participants gained new knowledge and experience. It can be concluded that the project has helped to promote greater consistency in temperature metrology at the European level by enhancing capabilities of emerging European NMIs in the field of temperature measurement, improving their readiness for active participation in the future research projects.

3 Objectives

The overall objective of the project was to develop novel methods and techniques that significantly improve knowledge and facilities to provide confidence in the verification of thermocouple performance and improvements in temperature measurement and control capability. The project has contributed to establishing low measurement uncertainty and reproducible process control in Europe.
The specific objectives are:

1. To develop and test novel methods and devices for the monitoring of thermocouple drift in-situ in the temperature range up to 1100 °C. These methods have to be suitable for implementation in critical industrial processes in order to assist the users in maintenance and replacement decisions.
2. To develop and test easy-to-operate methods and instruments for the assessment of inhomogeneities of thermocouples for secondary calibration laboratories in the temperature range from 230 °C to 1100 °C.
3. To design and construct novel measurement facilities that can provide confidence in the verification of thermocouple performance and to identify and quantify the range of drift of the thermocouples. The new facilities, targeting primary calibration laboratories, should have the ability to measure the physical changes and behaviour of thermocouples under typical conditions of production and distribution processes with a target uncertainty of less than 1.5 °C.

For each participant, to develop an individual strategy for the long-term operation of the capacity developed, including regulatory support, research collaborations, quality schemes and accreditation. They will also develop a strategy for offering calibration services from the established facilities to their own country and neighbouring countries. The individual strategies will be discussed within the consortium, with other EURAMET NMIs/DIs and with a broad spectrum of stakeholders, through questionnaires and workshops organised in the local language. The individual strategies will lead to an overall strategy document to be presented to the EURAMET TC-T, to ensure that a coordinated and optimised approach to the development of traceability in this field is developed for Europe as a whole.

4 Results

The results of this project provide new temperature sensing techniques for better control of high-value manufacturing and other processes.

Objective 1 Development of novel dual-type temperature sensors and methods for in-situ testing of thermocouple drift

All the project partners contributed to the successful accomplishment of the Objective 1.

The new method for in-situ determination of thermocouple drift was developed and its performances were investigated. The basic idea behind the concept is that thermocouple drift can be continuously monitored in-situ by placing an additional and different temperature sensor in proximity. The essential requirement for this additional sensor is that it experiences a drift that is lower than the drift of the monitored thermocouple. A drift of a thermocouple can be determined by an algorithm that monitors, and over time compares, the differences in its temperature readings and the readings of an additional (reference) sensor. The preliminary algorithm was proposed by FSB, and it was then improved through collaboration of all project partners. All project partners collaborated on elaborating different designs of dual-type temperature sensors that include a monitored thermocouple and a reference temperature sensor accommodated within a common protective sheath. Altogether, five versions of different dual-type temperature probe designs were made. For each design FSB created 3D models and technical drawings required for manufacturing of probes. Detailed technical documentation was created for each of three types of dual-type temperature probes:

- dual-type temperature probe utilizing platinum resistance temperature sensors as a reference, which was intended for use in a temperature range between -50 °C and 650 °C
- dual-type temperature probes utilizing noble metal thermocouples (with a lower rate of drift than monitored thermocouple) as reference sensors, which were intended for use in a temperature range between 0 °C and 1084 °C
- dual-type temperature probes utilizing fibre-optic temperature sensor, which was intended for use in a temperature range between 550 °C and 1000 °C

To determine the precision of drift tracking, when dual-type thermometers are used for the purpose, all laboratories assembled at least one type of the probe and performed experimental tests. The tests were carried out by performing calibration of reference and monitored temperature sensors, which were then exposed to thermal cycling and calibrated again. The sequence was repeated until moderate or significant drift of monitored and/or reference sensor was determined by subsequent calibrations. The results obtained through experiments indicate that the new method enables in-situ determination of thermocouple drift with approximate accuracy of ±1.0 °C for the dual-type temperature probes utilizing platinum resistance sensor as a reference and ±1.8 °C for dual-type temperature probes utilizing thermocouples as the reference sensors. It was also observed that the new method is sensitive to sources of measurement errors other than drift. In some cases, drift was changing the direction during the experiments, which would be hard to determine by calibrations. Both characteristics could be highly valuable for the users of the new method.

Considering that the new method is aimed at industrial users that often rely on tolerances stated for the particular class of the thermocouple, an accuracy of ±1.8 °C (or better) could be sufficient in many cases. The stated drift determination accuracy is comparable with the tolerances of class 1 thermocouple types E, J, K, and N, according to ISO/IEC 60584-2:1993.

Investigations on the dual-type temperature probes utilizing fibre-optic temperature sensors were performed by JV. Investigations revealed drift of the fibre-optical part in the range of 3 °C, which was roughly comparable to industrial thermocouples. Likely, drift and noise during the measurements can partly be assigned to insufficient temperature control of the used GaInAs detector which was located over the fixed-point cell and thus exposed to the heat emitted from the furnace and cell. Future work on dual-type temperature probes utilizing fibre-optical temperature sensors is required for gaining better results. It should aim at better temperature control of the pyroelectric detector, investigation of lower-cost fibres, e.g., hollow-core silica fibres, and ease the practical handling of the probe by connecting the “detection” fibre to a longer and flexible silica fibre which transmits the radiation to filters/detector located elsewhere.

A manuscript containing detailed descriptions of the methods for in-situ determination of thermocouple drift by using dual-type temperature sensors was submitted to a peer-reviewed journal.

The RMG2 work was performed by a researcher from the National Metrology Institute of Albania, in the Temperature laboratory of TUBITAK UME. Project Coordinator guided the RMG2 researcher during the joint practical work on the construction process, as well as the development of experimental prototype dual-type thermometers with three thermocouple wires. While dual-type thermometers with two separate temperature sensors were investigated within the project, the RMG research was oriented towards an investigation of thermocouples consisting of three wires connected in one tip for in-situ determination of drift. The validation was performed in a comparison between the drift of the type K thermocouple wires, determined using the third wire made of type S, and the same drift was determined through calibrations of the type K thermocouple wires in a fixed-point cell. The accuracy of the drift determination using the novel 3-wire thermocouples, developed within this RMG, reached ± 2.5 °C.

Based on the results of the work related to the Objective 1, project participants have concluded that the Objective is successfully accomplished.

Objective 2 Development of traceable novel devices and methods for characterisation of thermocouple inhomogeneity for secondary calibration laboratories

Work related to the successful accomplishment of the Objective 2 was performed by: TUBITAK, FSB, CMI, IMBiH, MER, and BIM.

A new design for miniature cells enabled the mounting of removable miniature fixed-point cells around the hot junctions of the thermocouples and suitable for ITS-90 contact thermometry in the temperature range from the Sn FP (232 °C) to Cu FP (1084 °C), and standardized methods for their testing were developed. TUBITAK and FSB successfully designed the miniature fixed-point cells and created related technical documentation for their production. One design has immersion depth of approx. 100 mm (slim cells) and the other of 17 mm (mini cells). Special attention had been paid to the needs of the secondary laboratories as well as to the flexibility of the design that gives the possibility for these cells to be used in-situ and in the furnaces already existing in laboratories.

<u>TUBITAK UME</u>

1. In the course of development and construction of the mini cells among several designs four designs for miniature cells intended for use in contact thermometry in the temperature range from Sn FP (232 °C) to Cu FP (1084 °C) were successfully outlined and completed.
2. The Tin, Zinc, Aluminum and Copper fixed-point cells were successfully constructed at TUBITAK and ÇMI.

TUBITAK UME constructed a set of slim mini cells of Tin, Zinc and Copper fixed point cells, then characterised and successfully validated these in a three-zone furnace in the secondary calibration laboratory of TUBITAK UME.
TUBITAK UME validated the home-made “slim” cells by analysing their melting and freezing curves obtained by reference SPRT. In validation study, direct comparison between the home-made tin cell and the reference cell was carried out and the difference was found to be -15 mK with an associated comparison uncertainty of 2 mK. The results show that this method can be used for validation of slim cells. Additionally, TUBITAK UME home-made mini cells were successfully validated by analysing their melting and freezing curves obtained by associated reference thermocouples. The results have shown high reproducibility and repeatability of the mini cells. Calculated average drift value of Sn melting plateau after one month usage of mini-cell -0,016 °C. The technique is applied successfully to ITS-90 contact thermometry fixed-point cells in the temperature range from the Sn FP (232 °C) to Cu FP (1084 °C). The home-made cells were successfully validated.

After finishing the measurements at TUBITAK UME the Sn mini cell was prepared for measurements in dry block furnace in ME (Montenegro) secondary laboratory. Calculated average drift value of Sn melting plateau at different temperature profile after two-month usage of mini-cell was max 0.28 °C at first measurements and fell down to 0,22 at the end of the cycle.

Another Sn mini cell constructed at TUBITAK UME was prepared for measurements in dry block furnace in the emerging NMIs BIM, and ME secondary laboratories under typical conditions of use. The results have shown high reproducibility and repeatability of the mini cells.

CMI

CMI successfully constructed the set of Tin, Zinc and Aluminium slim fixed-point cells based on the novel developed design.

IMBIH

The main goal was to test whether the new Al cell constructed by CMI could improve the reliability of measurements for in-situ conditions, and to compare test measurement results. Three melting plateaus and three freezing plateau were measured with the aim of checking the reproducibility of measurements under different realization conditions and using different equipment. The maximum difference between all plateaus measured by TC S type was 1 µV.

IMBIH used an OBERON heat-pipe furnace IMBIH secondary laboratory under typical conditions of use. The IMBIH and CMI measurements for Al fixed point temperature value agreed within stated uncertainties, for different setups and equipment used. It was shown that "slim" fixed points can be used to estimate the inhomogeneity and drift of thermocouples in situ for most industrial purpose, especially on melting plateaus because they are stable enough and can be obtained quickly enough even outside laboratory conditions (e.g. in the small heat-pipe OBERON furnace).

The RMG1 Researcher from Institute of Metrology of Bosnia and Herzegovina spent 3 months on the joint research work at TUBITAK UME in the Temperature laboratory. The Project Coordinator from TUBITAK UME guided the RMG1 researcher to during the construction, characterization, and validation of high quality two slim silver (Ag) FP cells. Moreover, continuous joint work during the construction, validation, comparison of fixed points and calculation of uncertainty for a new design of fixed-point cells gave an additional value of 3-month practical work and training in the laboratory. The construction and validation of the miniature cells were performed using TUBITAKs facilities and procedures with IMBiH supplied materials (high purity silver metal (5N and 6 N), quartz and graphite parts). Additional to the RMGs planned construction of one AgFP cell (6N silver metal) a second was constructed using the same design but filled with a different purity of silver metal (5N sliver metal). The 6N Ag FP miniature cell was at first characterized and then compared with TUBITAK AgFP standard cell (Ag 189) according to TUBITAK's procedure for comparison of ITS-90 fixed-point cells. The calculated measurement uncertainty was 0,005 °C. The second 5N Ag FP was also tested at TUBITAK.

The knowledge transfer in high temperature measurements received by the IMBiH's RMG1 Researcher from TUBITAK, consisted of hands-on training in the construction, validation, comparison of fixed points and calculation of uncertainty for a new design of fixed-point cells.

The RMG1 Researcher finished the planned activities and shared the RMG results at the different workshops: online workshop at IMBIH, workshop at BFKH and JV workshop organized together at the final meeting.

Based on the results of the work related to the Objective 2, project participants have concluded that the Objective is successfully accomplished.

Objective 3 Design and construction of novel measurement facilities suitable for thermocouple performance investigation in primary calibration laboratories

Work related to the successful accomplishment of the Objective 3 was performed by: TUBITAK, BFKH, FSB, BRML, BIM, CMI, IMBiH, INM and MER.

Two facilities have been designed and constructed by BFKH in order to improve the evaluation of thermocouple performance. The principle of the developed technique is based on the Curie-point, which is a certain temperature at which the ferromagnetic material loses its magnetic properties, resulting in a constant temperature. The concept behind the development of thermocouple inhomogeneity monitoring facility (TIMF) is to characterize the thermocouple performance by moving a miniature Curie-point furnace along the thermo element. The miniature furnace can provide a temperature distribution of 0.1 °C and a temperature stability of 0.03°C. Keeping the two junctions of the sheathed thermocouple at the same ambient temperature, the electrical voltage given by the thermocouple is expected to be neglectable. Therefore, the variation in electrical voltage appertained to the section of the thermo-element under test gives the magnitude of its inhomogeneity. The TDMF facility, due to its long-lasting temperature plateau, is suitable for determination of thermocouples drift. Different temperatures can be realised by changing the type of the ferromagnetic material of the metal block. This technique is characterized by a fast-heating period of 12 minutes and temperature stability of the furnace of 0.02 °C. Specific measurements with the DTTs developed in WP1 have been performed at temperatures of 260°C and 360°C, determining the inhomogeneity and drift of these S and K type thermocouples. Due to the self-adjustment property of the furnace, the Curie-point furnace has fast heating period, stabilization without overheating, uniform temperature distribution and high temperature stability.

During the technical visit organised by BFKH, the partners participating in Objective 3 had the possibility to work with the TIMF and TDMF devices. A manuscript containing detailed description of the method for determination of thermocouple inhomogeneity and measurement results with S and K type thermocouples was published in a peer-reviewed journal.

One of the advantages of the developed method is of meeting wider range of users as the fixed-point method, since fixed-point cells are not required for its operation. Another advantage is providing end-less temperature plateau. Furthermore, the material remains solid, hence it is less sensible to pollution and pressure changes. The importance of these methods is related to a relatively cheap and simple solution for monitoring thermocouple performance.

FSB

A novel device for testing thermocouple inhomogeneity by using a set of heaters was developed. Resistive coil heaters are positioned around the tested thermocouple probe and the device heats different portions of the probe, while the hot junction is kept at a constant temperature. Inhomogeneity can be then determined by analysing voltages generated by the tested thermocouple. Compared to the common methods for thermocouple inhomogeneity testing, this device is unique since it has no moving components. Users can simply put the thermocouple into the device, run the testing sequence and collect the measurement results when a testing sequence is finished. The device also allows for simultaneous heating of different portions of the thermocouple probes, even at different temperatures and with different heating/cooling rates for each separate heater module, which enables much greater testing flexibility. Since all the components of the modules can be made of metal or ceramic, which are tolerant to high temperatures, the modules can be installed in the environments where tested thermocouples are used e.g., inside the furnaces.

Based on the results of the work related to the Objective 3, project participants have concluded that the Objective is successfully accomplished.

Objective 4 To develop individual strategies for the long-term operation of developed capabilities to support SI traceability and accreditation services

All the project partners contributed to the successful accomplishment of the Objective 4. In cooperation with all the project partners, MER, BIM, INM, BRML and FSB have updated their strategy documents and sent these to IMBiH, who prepared the overall strategy of the consortium for the long-term development of research capabilities in temperature. The overall strategy document aims to coordinate the strategies of the partners to ensure maximum awareness of each partner's plans, thereby reducing duplication of efforts and enhancing cooperation on common goals.

Based on the results of the work related to the Objective 4, project participants have concluded that the Objective is successfully accomplished.

5 Impact

To promote the uptake of the temperature calibration services, and to share insights generated throughout the project, results are being shared broadly with scientific and industrial end-users.

To date, two papers have been published in international journals, including an open-access paper in the Proceeding of 19th International Congress of Metrology.

Presentations were made at conferences, including the 50th European TEMPMEKO & TEMPBEIJING 2019 International Conference, in 2019. The project partners have given presentations at:

- CIM 2019, International Congress of Metrology, 24-26 September, Paris, France, and the main project objectives have been published in the Proceeding by EDP Sciences.

- TEMPMEKO & TEMPBEIJING 2019 International Conference, 10-14 June 2019 Chengdu, China.
- Symposium and Exhibition of Measurement Science, 20-22 November 2019,İZMİR, Turkey
- 15th Laboratory Competence Conference (CROLAB 2019), Cavtat, Croatia.
- "2020 XXX International Scientific Symposium Metrology and Metrology Assurance" held on 07-11 September 2020, Sozopol, Bulgaria
- International Metrology Congress (CIM 2021), Lyon, France
- 16th Laboratory Competence Conference (CROLAB 2021), Brijuni, Croatia

The Project's website has been regularly updated with new information and project progress: http://metfortc-empir.org. Workshops for stakeholders and partners were organized by: FSB in February 2020, BIM (online) in June 2021, IMBIH (online) in March 2022, BFKH in September 2022 and JV in May 2023.

Workshop presentations provided attendees with the basic principles of temperature measurements when using thermocouples, uncertainties associated with thermocouple calibrations, project objectives and activities as well as information on the project designs for dual-type thermometers. During the workshops a discussion about the existing and future customer needs in the area of temperature measurement was initiated. A visit to the national measurement standards for temperature, and humidity was also included in the workshops at FSB, BFKH and JV.

The stakeholders were representatives from accredited and non-accredited laboratories that are already working, or are planning to start working, in the field of temperature measurements.

During the workshops the RMG researchers presented their work related to:

- RMG1 Researcher in design of temperature fixed-point cells (presentations at the IMBIH, BFKH and JV organized workshop)
- RMG2 Design of a novel type of dual thermometer (presentation at the IMBiH organized workshop)

A technical collaboration was organised by BFKH after the workshop, from 6th to 7th September, on order to make common measurements using the novel facilities for verification of thermocouple performance, developed by BFKH.

Researchers from the Institute of Metrology of Bosnia and Herzegovina (IMBIH) and the Norwegian National Metrology Institute (JV) had worked together on evaluating a newly designed triple-type thermometer (TTT) consisting of two thermocouples (type S and N) and an optical fiber used as a sensor of the drift assessment device for one week in the laboratories of the JV in Kjeller, from 28th November to 1st December 2022.

At the second technical visit, in April 2023, researchers from the IMBIH and the JV worked together in the IMBIH laboratories in Sarajevo. Measurements were carried out on the newly designed DTT thermometers, which consist of two thermocouples in a common housing, designed by FSB, Croatia.

Impact on industrial and other user communities

This project's activities have been designed to meet the needs of secondary calibration laboratories, manufacturers of temperature sensors and accreditation agencies in participating emerging countries.The organized workshops were of great importance for the partner labs, as well as for the stakeholders, as a way to initiate networking and cooperation in the field of temperature measurement. Workshop presentation materials for industrial stakeholders have been placed on the website. These materials are available to all partners for download should they wish to run a similar training course.

Impact on the metrology and scientific communities

The project supported active participation and influencing in key European temperature related committees such as the EURAMET Technical Committee on Thermometry TC-T and COOMET TC1.10.

The technical committee EURAMET TC-T was contacted in 2021 in the mid-time of the project and one presentation including objectives, developed novel devices designs and early outputs and impact of the MetForTC project were presented.

The project activities and results were presented during the COOMET TC1.10 meeting in November 2021 by INM. Due to the nature of the work carried out in the MetForTC project, it is only at the end of the project that sufficient results were obtained and analyzed, which could be of interest to the standards committees identified

There are a high number of smaller NMIs in the consortium and their participation in this project substantially contributed to capacity building, particularly in the area of thermocouple calibration and the facilities and skills required to assess thermocouple performance.

Impact on relevant standards

The participations were built on activities already established by the consortium, who are highly influential in national and international metrology and standards committees and were used to facilitate greater awareness of the projects outcomes.

The project aims and results were presented to EURAMET TC-T contact persons on many occasions, two times during the annual meetings of the EURAMET Technical Committee for Thermometry, during the conferences as well as through private conversations. The participants of the Working group, responsible for writing guidelines, are aware of the project and they are free to consider the project results when writing or updating the related guidelines.

Longer-term economic, social and environmental impacts

Knowledge transfer, from experienced NMIs (TUBITAK, BKHF, CMI, and JV) to those less experienced (IMBiH, FSB, INM, BRML, BIM and MER), in how to use and develop new capabilities in temperature field was very beneficial. The beneficiary countries have obtained the skills and knowledge necessary to improve their capabilities in the field of temperature measurement. Improved capabilities are beneficial to stakeholders that use the services provided by laboratories that participated in this project.

The project strengthened the collaboration between European NMIs, and increased their competitiveness with NMIs outside Europe. It also increased partners readiness for the active participation in future research projects. Another impact was an increase in cooperation and liaisons anticipated with the scientific community, users' associations, manufacturers, and other stakeholders, in order to provide guidance to traceability and good practice in secondary thermometry.

6 List of publications

1. N. Arifovic, et al *A new EMPIR Project "MetForTC" for Developing Traceable Measurement Capabilities for Monitoring Thermocouple Performance,* Proceedings 19th CIM 2019, 18006 https://doi.org/10.1051/metrology/201918006
2. N. Arifovic, et al "TÜBİTAK UME'de Yeni EMPIR Projesi: Isılçift Performansının Gözlenmesi için İzlenebilir Ölçüm Yeteneklerinin Geliştirilmesi", Proceedings Symposium and Exhibition of Measurement Science, 2019, IZMIR/TURKİYA http://www.olcumbilim.org/wp-content/uploads/bildiriler/2019-15.pdf
3. Nedialkov, S., Spasova, S. and Aldev, K. 18RPT03 MetForTC Traceable Measurement Capabilities for Monitoring Thermocouple Performance, Metrology and Metrology Assurance 2020–Proceedings http://metrology-bg.org/fulltextpapers/Proceedings_MMO_2020.pdf
4. E. Turzo-Andras et al, Determination of Thermocouple Inhomogeneity Using Miniature Curie-point Furnace, JP Journal of Heat and Mass Transfer, Volume 34, 2023, http://dx.doi.org/10.17654/0973576323034

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