



# FINAL PUBLISHABLE REPORT

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**TABLE OF CONTENTS**

|   |                           |   |
|---|---------------------------|---|
| 1 | Overview .....            | 3 |
| 2 | Needs.....                | 3 |
| 3 | Objectives .....          | 3 |
| 4 | Results .....             | 4 |
| 5 | Impact .....              | 7 |
| 6 | List of publications..... | 8 |
| 7 | Contact details .....     | 9 |

## 1 Overview

Currently, in Europe, more than 60 % of processes used by the manufacturing industry and common equipment depend on accurate measurements of temperature and thermal properties of the materials. Thermal metrology plays a key role in improving the efficiency and environmental impact of these industrial processes.

Therefore this project allowed:

- Upgrading and implementing traceable calibration facilities, new measurement procedures and knowledge transfer to emerging NMIs and DIs in Europe.
- Increasing the availability of high-level facilities in the field of thermal metrology, useful for the Industry and for the society of these emerging countries.
- Bringing up new services for the industrial development and the commercial trading of these countries within the international competitiveness.

## 2 Needs

In the field of thermal measurements (Contact Thermometry, Radiation Thermometry and Thermal properties of materials), there is a growing need in Bosnia and Herzegovina, the Czech Republic, Croatia, Hungary, Ireland and the Republic of Serbia to review their existing capabilities, identify and prioritise their traceability needs and strengthen or upgrade their regional metrology systems.

Therefore, there was practically a need for:

- o Establishing measurement capabilities according to the needs of industries working in the temperature range from 50 °C to 2000 °C. These measurements should be traceable and accepted on both local and international markets.
- o Developing procedures of thermophysical properties measurements and improving the traceability scheme to SI. This provided comparable measurements in Europe and strengthened European and international trading.

The traceability chain for any thermal sensor from NMIs to industries has to be implemented via different levels of calibrations. NMIs calibrate reference sensors at the highest level providing the most precise thermal measurements with the lowest uncertainties. These reference sensors (contact or non-contact thermometers) are then used in secondary and lower level calibration laboratories. There was a need to complete, upgrade and implement regional metrological infrastructure in the field of thermal measurements in the beneficiary countries and to manage the metrological features and the traceability of thermal instrumentation used for contact or non-contact temperature measurements.

In the field of thermophysical properties of materials, the project focused on the characterisation of transport properties of materials (thermal conductivity). The support was based on an improvement of the corresponding metrological infrastructure and on a cross collaboration of participating laboratories with relevant experience in providing mutual research activities and appropriate knowledge transfer in thermal conductivity measurements using the guarded hot plate method. Application was useful for saving energy and particularly for improving the thermal efficiency of buildings and thermal insulation of industrial processes.

## 3 Objectives

The overall objective of the project is to provide emerging NMIs with access to facilities in the field of thermal metrology (e.g. high temperature contact thermometry, non-contact thermometry and thermo-physical properties characterisation). The project addresses the following scientific and technical objectives:

1. To improve the accuracy of high temperature measurements by contact thermometry in the range of 960 °C to 1084 °C in participating NMIs with limited metrology research capacity (FSB, IMBiH, BFKH and MoE) - WP1.

2. To compare current radiation thermometry calibration techniques and calibrate transfer standard pyrometers for radiation thermometry in the range of 50 °C to 2000 °C in participating NMIs seeking to establish a research capability in this field (FSB, BFKH, MoE and NSAI) - WP2.

3. To consolidate the traceability and capabilities for thermal conductivity measurements by Guarded Hot Plate (GHP) in emerging NMIs and DIs (CMI, BFKH and VINCA) from room temperature to 800°C. CMI, BFKH and VINCA will each implement thermal conductivity measurements by the GHP method; and the outcomes will help these NMIs/DIs to participate in an inter-laboratory comparison in this field - WP3.

4. To enhance the availability of thermal metrology facilities for contact thermometry, non-contact thermometry and thermophysical property characterisation for each emerging NMI and DI (FSB, IMBiH, BFKH, MoE, NSAI and VINCA). An individual strategy for the long-term development of their research capability and the ability to offer calibration and advice services to industries at a regional level will also be developed – WP4.

5. For the emerging NMIs and DIs to contribute to or extend their declared measurement capabilities, quality schemes and accreditations (such as CMC entries and ISO/IEC 17025 accreditations) - WP4.

## **4 Results**

- 1) The aim of this project was to transfer scientific and technical knowledge from the scientific and technical experts of NMIs/DIs which have significant experience in the field of thermal measurements to the laboratories which are developing, improving, or upgrading their own metrological infrastructure. The final objective was that they can exploit their new or existing facilities for the benefit of regional or local Industry. Improvement of the accuracy of high temperature measurements by contact thermometry in the range 960 °C to 1084 °C

First of all, TUBITAK identified the best practice methods and techniques in contact thermometry and then organized a training course focusing on knowledge transfer that would enable the partners from less experienced NMIs/DIs to improve their current measurement capabilities.

Through the various lecturers, the best practice methods and hands-on work in the laboratory, related to all aspects of high-temperature measurements, were shared. The training was organized as work on the physical construction of the silver fixed-point cell and also included all the aspects of the ITS-90 temperature scale realization.

The later covered the use of high-temperature resistance thermometers (HT-SPRTs) as interpolating instruments as well as calibration of Au/Pt and S type thermocouples as the instruments which are used most frequently for temperature measurements in industry and calibration laboratories. The additional objective of this collaborative work was to perform a practical assessment of the capabilities of the project partners in this field.

During the workshop MoE, FSB and IMBiH learned about design, handling, and repairs of the equipment existing in their respective laboratories. The knowledge gained allowed them to efficiently use the facilities and perform high-temperature calibrations with improved uncertainties for calibrations in fixed-point cells.

Finally one inter-laboratory comparison with High Temperature Platinum Resistance Thermometers (HT-PRTs) was performed. Special attention was given to overcome the difficulties, due to the stability of the thermometers, at high temperatures. Additional comparisons of one Gold/Platinum (Au/Pt) and S type thermocouples have been also performed. Versatile home-made software was developed by FSB in cooperation with TUBITAK in order to monitor the phase transition interface. Besides traceability, this exercise has provided the opportunity for demonstrating and implementing the procedures and establishing uncertainty budgets.

The outcomes resulted in the following achievements:

- The novel fixed-point cell was designed and installed in the new system at IMBiH for ITS-90 realizations. The primary calibration range had been extended from 660 °C to 961 °C allowing for the calibration of High-Temperature Standard Platinum Resistance Thermometers (HT-SPRTs) and Thermocouples (TCs) in the range from the Triple Point of Water to the Silver Fixed Point (Ag FP),
- Improvement of uncertainty components and evaluation of the overall uncertainty budgets,
- Skills for practical implementation of a procedure for realization of ITS-90 measurements at FSB, IMBiH and MoE thus providing partners to use the existing equipment efficiently,

- With the installation of the system for realization of the Ag FP temperature and calibration of thermocouples, MoE has extended their previous capabilities in this range. After publishing the final report of the comparison, the document supporting new CMCs will be submitted to the chair of TC-T for review in inter RMO.

The Inter-laboratory comparison of the temperature scale realizations in contact thermometry was conducted among the partners. The main objective of this collaborative work was to assess the partner's improved capabilities. The inter-laboratory comparison results will be used for identifying opportunities for further improvements in participants' capabilities and methods.

Moreover, the comparison provided a practical mean for assessment of partners' uncertainty budgets and their validation. It should be pointed out that for some participants this was the first exercise of this kind and therefore it is considered as very structuring and fruitful.

- 2) To compare current radiation thermometry calibration techniques and calibrate transfer standard pyrometers for radiation thermometry in the range of 50 °C to 2000 °C in participating NMIs seeking to establish a research capability in this field (FSB, BFKH, MoE and NSAI):

Partners worked collaboratively in this category by sharing knowledge and experience and having an inter-laboratory comparison of radiation thermometer calibration over a large temperature range from -20 °C to 2000 °C. The objective of this comparison was to help the participants to establish their uncertainty budgets and improve their calibration methods and procedures. Technical visits with a specific focus on research in radiation thermometry, especially the realisation of the international temperature scale above the freezing point of silver (961.78 °C) and improving calibration techniques were also conducted.

During the first year of the project, training was held at CMI in March 2016 and was organised by CMI, CNAM and UL towards staff from NSAI, MoE, FSB and VINCA. The training was intended to share knowledge about the bases of radiation thermometry through theory and practice; it lasted four days and included two days of lectures on radiation thermometry and thermal imaging and two days of hands-on experiments in the laboratory. Following this training a best-practice guide gathering the main concepts and measurement techniques studied during the training was prepared by UL and shared with the partners for further improvements with the aim of using it in the future for the new calibration service under development in NSAI, MoE, VINCA and FSB.

The second important part of the project was an inter-laboratory comparison of radiation thermometer calibrations intended to probe the uncertainty budgets of the participants and to pave the way for further improvements and it will possibly justify new CMCs in the field of radiation thermometry. This comparison was launched in October 2016 and was completed in October 2017 after the completion of the measurements in the eight participating partner laboratories. The circulation of the two pyrometers (devoted to the temperature ranges -20 °C to 950 °C and 950 °C to 2000 °C) was completed without noticing any breakage but with a few months of delay due to some unexpected difficulties in the transportation of the instruments and the adaptation of the schedules of the participants. The results of the comparison were agreed among the participants and presented at an international conference.

Finally, technical visits were performed at NSAI, FSB, MoE and VINCA to help identify the possibilities of improvement as well as the assessment of the existing methods and techniques.

Radiation thermometry activities were developed to help four NMIs; (FSB, MoE, NSAI, and VINCA) to launch or improve activities of research and calibration. Three additional participants (CNAM, CMI and UL) joined their efforts to share their experience.

Three types of activities were implemented:

- A 4-day training mixing lectures from CNAM, CMI and UL staff and hands-on experiments was hosted by CMI. This training allowed the participants to learn the basics of radiation thermometry and to perform practical experimental work with radiation thermometers and thermal imagers.
- An inter-laboratory comparison of radiation thermometer calibrations in the range from -20 °C to 2000 °C allowed the participants to test their methods, scrutinise their uncertainty budgets and improve their procedures. The comparison lasted a whole year and yielded a lot of information to the participants.

- Technical visits from experts of UL, CMI and CNAM which allowed the participants to tackle specific measurement issues and to discuss and improve measurement techniques paving the way to further developments and cooperation after the project.

The overall outcome was very structuring and fruitful for all the participants which achieved almost all the objectives assigned in the beginning of the project. All the objectives were fulfilled except the extension of the capabilities of MoE to 2000 °C: it was limited to 1800 °C due to the breakage of their high-temperature furnace.

Moreover, all the participants agreed on the fact that the inter-laboratory comparison organised on such a large temperature range was very useful for the validation of their methods and the preparation of new CMC entries in the future.

To summarize, the following capabilities of radiation thermometry in the range from 50 °C to 2000 °C for VINCA, MoE, NSAI and FSB have been improved:

- FSB has developed capabilities in the field of radiation thermometry in the temperature range 50 °C to 800 °C, with target uncertainties of 2 K.
  - MoE has improved temperature references in the high temperature range (up to 1800 °C) and assessed the corresponding uncertainty budget with target uncertainties of 2 K.
  - NSAI to has developed radiation thermometry capabilities in the range of 50 °C to 500 °C with target uncertainties of 2 K.
  - VINCA has improved traceability in the temperature range from 900 °C to 2000 °C.
- 3) To consolidate the traceability and capabilities for thermal conductivity measurements by Guarded Hot Plate (GHP) in emerging NMIs and DIs (CMI, BFKH and VINCA) from room temperature to 800°C. CMI, BFKH and VINCA will each implement thermal conductivity measurements by the GHP method; and the outcomes will help these NMIs/DIs to participate in an inter-laboratory comparison in this field:

In a first stage of the project a review of GHP facilities and methods available in each partner laboratory has been performed. Detailed technical information on these GHP apparatus have been provided by the partners to CMI for drawing up the state-of-art of the measurement capabilities of the partners in that field at the beginning of the project. The developments performed by BFKH, CMI, and VINCA aimed at improving their respective GHP facilities devoted to the thermal conductivity measurement.

In order to validate their new capabilities, partners participated in an inter-laboratory comparison on thermal conductivity measurement by GHP piloted by LNE. During the preparation phase of the sample materials, an RMG from VINCA has learnt at LNE about the inter-laboratory comparison procedure and characterisation tests to be performed before the launching. In details, as outcomes of this comparison, BFKH has reached the objectives concerning in particular a better control of the thermal source uncertainties by developing a standard heat flow source for heat flow meters, and a decrease of the temperature difference between the hot plate and cold plate of the HT-GHP. Direct traceability to the SI has been achieved by BFKH through the development of in situ calibration procedures performed from their own standard heat flow source.

At the beginning of the project, CMI could perform thermal conductivity measurements by GHP method in the temperature range 50 to 800 °C. Development of the new SGHP apparatus enables CMI to extend their measurement capabilities down to 20 °C, in agreement with the aims of the project.

After having improved the 2S-GHP apparatus, VINCA can now perform thermal conductivity measurements by GHP method in the temperature range [10 – 50 °C] with an expanded uncertainty close to 2 % (2.1 % in the case of the certified reference material IRMM440). These results agree with the objectives of VINCA in this project.

Several activities have been carried out by CMI, BFKH, and VINCA with the support of LNE in order to improve their measurement capabilities of thermal conductivity by GHP method.

The results are as follows:

- CMI extended the temperature range of their GHP facility down to 20 °C.
- BFKH achieved better control of the thermal sources of uncertainties by the development of a standard heat flow source for HFM, decreasing the temperature difference between the hot plate and cold plate of the HT-GHP; and achieving direct traceability to the SI.
- VINCA achieved a 2 % uncertainty ( $k=2$ ) from 10 °C up to 50 °C for their GHP facility and will be able to provide direct traceability to the SI.

## 5 Impact

### ***Impact on industrial and other user communities:***

An online stakeholder survey was circulated among each NMI/DI's contacts, with the intention to determine the needs of a core group of stakeholders. The stakeholders have been emailed copies of the annual newsletter and links to the project website, which keeps them informed of the latest news of the project.

Training course materials (slides, plans etc.) 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. Workshops for stakeholders and partners were organized in FSB and IMBiH. The topics were: fundamentals and calibration of thermocouples with demonstration of good practices and uncertainty calculations and presentation of the Eura-Thermal Project. Furthermore, the needs of stakeholders were discussed. Until now FSB has established collaborations with laboratories. Some of the laboratories were visited and others were provided with information upon requests via telephone and e-mail. Laboratories mainly asked for advice related to calibration uncertainties, but also for help with the practical procedures on site.

IMBiH strengthened its relationship with customers, industry and academic society in Bosnia and Herzegovina. During the project, a market survey took place and additional information about calibration needs in high temperature measurement have been learned. A separate survey that was conducted from by IMBiH, covering for all areas of metrology in Bosnia and Herzegovina brought even more information, and subsequently IMBiH have arranged and hold several meetings with their potential customers and partners and has investigated opportunities for cooperation.

### ***Impact on the metrology and scientific communities:***

Accepted presentations were made in several conferences. The latest were at CAFMET 2018 in Morocco and the 2018 Symposium on Thermophysical Properties in Boulder, Colorado, USA, a paper has also been submitted and abstract accepted for the IMEKO 2018 conference in Belfast. .

As a result of knowledge acquired through the project, a researcher in NSAI produced an MPhil thesis (<http://arrow.dit.ie/engmas/53/>) and published a paper in Elsevier *Measurement* journal ([10.1016/j.measurement.2017.03.023](https://doi.org/10.1016/j.measurement.2017.03.023)) in the field of radiation thermometry. Also, a researcher in VINCA submitted a PhD thesis to the University of Belgrade, School of Electrical Engineering, in the field of thermophysical properties measurements. Defence of the thesis is due on September 2018.

The consortium also presented an update of the progress of the project at the EURAMET TC-T annual meetings.

The objective of each talk, presentation or paper for the project was to promote the project and enhance the availability of facilities in the field of thermal metrology (e.g. high temperature contact thermometry, non-contact thermometry and thermo-physical properties of materials characterisation) in European emerging National Metrology Institutes (NMIs) and Designated Institutes (DIs), where access to these types of facilities is currently

limited.

***Impact on relevant standards:***

Technical committees CEN TC 89, ISO TC 106 and ISO TC 121 were contacted in order to disseminate the aims of the project. Each of the committees mentioned above were thought to best capture the topics covered by the project. The secretaries made their respective communities aware of the project and its aims, and forwarded any expressions of interest from potential stakeholders.

Forwarding the aims of the project to the relevant communities may serve to generate awareness of the project, and to establish further co-operation with technical committees. As a result of contact established between the consortium and TC 121, an invitation was received to attend and possibly contribute to the joint working group meeting in December 2017. This was an opportunity to demonstrate that the project outputs will have a direct impact on the standardisation activity of the working group.

***Longer-term economic, social and environmental impacts***

Many international studies have underlined the importance of reliable measurement infrastructures for supporting the development of industry and society in a more sustainable, safe and secure manner. The reduction of emissions and pollutants and the efficient use of energy and raw resources are important issues for all new emerging countries.

As temperature is one of the most widely measured parameters in a power plant and irrespective of the type of plant, accurate and reliable temperature measurement is essential for operational excellence.

Furthermore, as another example, power plants can generate extremely high temperatures that often cause measurement problems. For example, in energy-from-waste plants, furnace temperature is a critical measurement. Burning the waste at high temperatures minimises the release of harmful emissions, and therefore high accuracy and reliability in these measurements remains also critical.

According to these challenges, this project has contributed to improve availability and capability of metrology services. As a longer term economic impact all these services will indirectly allow Industry to save Energy and increase their competitiveness.

## **6 List of publications**

1. M.M. Terzić, N.D. Milošević, N.M. Stepanić, S.J. Petričević, "Development of a single-sided guarded hot plate apparatus", *Thermal Science*, Vol. 20, Suppl. 1, p. S321-S329
2. S. Boles, I. Pusnik, D. Mac Lochlainn, D. Fleming, I. Naydenova, S. Martin, "Development and Characterisation of a Bath-Based Vertical Blackbody Cavity Calibration Source for the Range -30 °C to 150 °C", *Measurement*, Vol 106, pp 121-127
3. S. Boles, "Development of Traceable Capabilities in Non-Contact Thermal Metrology", A thesis submitted to the Dublin Institute of Technology for the degree of Master of Philosophy, November 2016
4. N. Stepanić, M. Terzić, D. Radivojević, D. Raković, "An in situ temperature calibration of a guarded hot plate apparatus", *Thermal Science International Scientific Journal*, 2018, article published online: <https://doi.org/10.2298/TSCI180425176S>

## 7 Contact details

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| WP Leader (Thermal Conductivity)          | Guillaume Failleau      | LNE       |
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Courtesy of LNE