Publishable Summary for 14RPT05 Eura-Thermal
Developing traceable capabilities in thermal metrology

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.

Need

In the field of thermal measurements (Contact Thermometry, Radiation Thermometry and Thermal properties of materials), there was 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:

- 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.
- 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 was 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.

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)

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)

3. To consolidate the traceability and capabilities for thermal conductivity measurements by Guarded Hot Plate (GHP) in emerging NMIs and DIs (CMI, MKEH 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

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

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)

Progress beyond the state of the art

Contact Thermometry in the range 960 °C to 1084 °C:

TUBITAK supported MoE, FSB and IMBiH to learn how to build fixed point cells thus allowing them to perform high temperature calibrations. This supported industry in those regions and improved achievable uncertainties for calibration with fixed point cells. As an outcome of training organized by TUBITAK in contact thermometry, partners used the acquired knowledge for the novel cell design of IMBIH cells, improvement of uncertainty components and implementation of a procedure for realization of measurements at FSB and MoE.

Moreover MoE repaired its broken cell and characterised it against reference silver fixed point acquiring the new facility for the calibration of the thermocouples. TUBITAK constructed the two Silver fixed point cells for IMBIH and the IMBIH's staff participated to the final measurements on fixed point cells. After that the cells were transported to IMBIH and used further in domestic system designed for open cell measurement. Finally the fixed point cells were used for the inter-laboratory comparison measurements.

IMBiH took the first steps in working with open fixed point and had the opportunity to learn about the construction and use of open cells and in future to establish a primary system of fixed points. Furthermore, new necessary equipment, for providing New Services in contact thermometry at Ag FP was acquired through this project.

Improvement of uncertainty components and implementation of procedures for realization of measurements at partners' laboratories further have been demonstrated in the frame of the on-going inter-laboratory comparison of High Temperature Platinum Resistance Thermometers (HT-PRTs) and Reference thermocouples (Au/Pt and S type). The inter-laboratory comparison was completed and therefore provides the traceability to partners. Furthermore, FSB in cooperation with TUBITAK developed a software application that supported data acquisition and data analysis during fixed point calibration, and which was very versatile and compatible with many other laboratory instruments.

Radiation Thermometry in the range - 20 °C to 2000 °C:

The provision of expertise and assistance in obtaining traceability by CNAM, UL and CMI has allowed MoE, FSB, NSAI and VINCA to improve their knowledge in the field of radiation thermometry, as well as improve their measurement capabilities and traceability.

A comparison of radiation thermometer calibration performed over the range -20 °C to 2000 °C was conducted and has shown an overall good agreement among the participants. The results of this comparison triggered a series of actions in the participating laboratories aiming for the improvement of the capabilities. Technical visits from experts of the consortium have contributed to the identification of the best options for future developments.

This work has led to the facilitation of access to traceable measurements within the respective regions of the participants.

Thermal conductivity measurements by GHP from room temperature to 800 °C:
LNE provided expertise and assistance to CMI, BFKH and VINCA on how to obtain traceability to the SI in the field of GHP measurements. This facilitated access to high level traceability schemes for industry in their respective regions.

Availability of thermal metrology facilities for contact thermometry, non-contact thermometry and thermo-physical property characterisation for each emerging NMI and DI

This project has ensured that for each emerging NMI and DI, new or upgraded metrological thermal capacity, skills and expertise was available and suitable to support regional industry development and hence to sustain economic competitiveness of the European member states. For achieving this objective, links with the wider European community and effective trainings helped ensuring that adequate support was available to industrial stakeholders within those countries.

Emerging NMIs and DIs to contribute to, or extend, their declared measurement capabilities, quality schemes and accreditations

For achieving this goal, a regional high temperature metrology service was be established, where a timely calibration service was provided to industry in countries where the domestic NMIs/DIs did not have the required capabilities. For that, in each country the needs of industries weremet to the required level of uncertainty without unnecessary duplication of calibration services within the consortium countries. The final objectives were to get updated or new CMCs approved or accreditations as well and promote them to the customers.

Results

Contact Thermometry in the range 960 °C to 1084 °C:
TUBİTAK purchased the necessary materials, provided training to MoE, FSB and IMBiH and finalised the design of the fixed point cells.

A draft protocol of the inter-laboratory comparison with High Temperature Platinum Resistance Thermometers (HT-PRTs) was prepared. Special attention was given to overcome the difficulties, due to the stability of the thermometers, at high temperatures. Due to the problems with stability of thermometers at high temperatures it was agreed with partners to use one additional (HT-PRT); this was provided by IMBIH and brought to TUBITAK for circulation during the inter-laboratory comparison. It was also discussed and agreed that additional comparisons of one Gold/Platinum (Au/Pt) and S type thermocouples should be done.

Partners used the acquired knowledge for designing a novel cell, preparing technical documentation for building the cells, including 3D models and drawings. MoE is repairing and characterizing their domestic Ag fixed point cell. All materials for constructing silver cells in IMBiH are now available and will be used for manufacturing soon.

Software for facilitating the inter-laboratory comparison process is currently under finalisation;

Home made software developed by FSB in coorporation with TUBITAK can monitor the phase transition interface, and since partner NMIs are using a different equipment, the software will also enable them to select different instruments to be logged. The software is now fully functional for temperature measurements using an ASL resistance bridge F18, F700 and F900 with or without scanner. The software which can be used for general temperature measurements, including monitoring of the phase transition interface during the ITS-90 temperature scale realization is currently in use in TUBITAK, IMBIH, MoE and FSB. The software will provide a tool for standardizing the method of measurement and uncertainty evaluation.

The inter-laboratory comparison on HT-SPRTs and Reference Thermocouples was performed. Besides traceability, this now provide the opportunity for demonstrating and implementing the procedures and establishing uncertainty budgets.

Radiation Thermometry in the range -20 °C to 2000 °C:
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. This comparison will 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 is 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.

Thermal conductivity measurements by GHP from room temperature to 800 °C:
Each partner has reviewed the current state of the art of their GHP facilities and produced a technical report suggesting further improvements. An inter-comparison protocol was prepared and subsequently agreed by all involved partners, i.e. by LNE, CMI, BFKH and VINCA. The protocol implies thermal conductivity measurements in the laboratories of all related partners and on specimens of selected materials.

A first set of materials was characterized (in terms of thickness and density) at LNE, with the support of N. Stepanic (VINCA) by a Research Mobility Grant of two months (April-May 2017). Once conditioned, the material specimens have been disseminated toward all the partners. The measurements have been started at LNE and were independently performed by each partner. Measurements have been completed and analysed by LNE at the beginning of 2018. This inter-laboratory comparison enabled to validate the improvements of the GHP facilities engaged by each partners and to define a new reference material now available within each laboratory.

Relationship between the partner's laboratories has been strengthened during this project, since CMI hosted a visit from a researcher of BFKH in order to share knowledge on thermal conductivity measurements using the GHP method. A similar technical visit by a researcher from VINCA was previously performed during a three weeks stay at LNE.

Availability of thermal metrology facilities for contact thermometry, non-contact thermometry and thermo-physical property characterisation for each emerging NMI and DI
In contact Thermometry, FSB has now acquired knowledge in design and construction of the metal fixed point cells, as it can be demonstrated through the 3D models and technical drawings. The knowledge gained was also used for disassembly and cleaning of the FSB's fixed point of Zinc. MoE worked on the repair and

characterization of their domestic Ag fixed point cell. IMBiH made the first step in working with open fixed point cells. All IMBIH fixed points at the moment are closed and the silver fixed point will be first open cell. This will enable the possibility to take into account the pressure component into the uncertainty budget. Since the CCT recommendations were that the open cells should be used as reference cells and closed cells should be calibrated by comparison with the open cell. IMBIH got the opportunity to learn about the construction and use of the open cells and will be able in future to establish a primary system of fixed points. Furthermore, new equipment such as a furnace for fixed-point realizations and a turbo molecular pump, necessary for this work was acquired in this period. Collecting experience in design of the system for operation of open cells was valuable for acquiring of additional equipment. IMBiH should at one's disposal a system for realising an Ag open fixed point ready by the end of the year. Further improvements are needed to be undertaken on analysis and calculation of uncertainty components.

In radiation Thermometry, several technical visits and staff exchanges have been undertaken between the participants. These technical visits have helped NSAI, MoE, FSB and VINCA to improve their facilities and methods for the calibration of radiation thermometers and for research purposes in the field of radiation thermometry and thermal imaging.

In thermal conductivity, CMI, BFKH, and VINCA have worked on their facilities in order to upgrade their capabilities in terms of thermal conductivity measurements by GHP method traceable to the SI and to reduce the measurement uncertainties. For example, VINCA developed and built a new GHP apparatus for measurements of poorly conductive materials such as ceramics and glasses and this work was published in a scientific journal. The existing GHP apparatus at VINCA for measurements of insulation materials was upgraded with new cold plates and a home-made control and data acquisition software. Temperature and thermal conductivity ranges were upgraded down to 10 °C and 30 mW/(mK), respectively. A new in situ temperature calibration of the upgraded GHP apparatus has been established and performed. CMI have developed a new small low temperature GHP with an extended temperature range down to 20 °C (instead of 50 °C at the beginning of the project). BFKH has developed a standard heat flow source and improved the control of the temperature balance between the cold and hot plates in order to achieve direct traceability to the SI (implementation of new calibration procedures on the thermal instrumentation) and to control the thermal sources of uncertainties.

Emerging NMIs and DIs to contribute to, or extend, their declared measurement capabilities, quality schemes and accreditations
EURAMET Technical Committee for Thermometry (TC-T) working group for calibration and measurement capabilities has been contacted in order to get information about rules for submitting CMCs especially for a new field like thermal conductivity. The process is under progress in order to respect the rules accordingly. Furthermore through the manufacture of a Silver fixed-point cell, IMBiH will be soon provided with a standard and be able to provide calibration services at the primary level.Further analysis will be carried out on the results to identify potential collaborations and service requirements.

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 attended 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 presentation was made 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) 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 meeting in Boras, Sweden in April 2018. 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.

List of Publications

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

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

S. Boles, “Development of Traceable Capabilities in Non-Contact Thermal Metrology”, Thesis submitted to the Dublin Institute of Technology for the degree of Master of Philosophy, November 2016

N. Stepanić, M. Terzić, D. Radivojević, D. Raković, “An in situ temperature calibration of a guarded hot plate apparatus", Thermal Science International Scientific Journal, June 2018

<table>
<tr><td colspan="2">Project start date and duration:</td><td>01 June 2015, 36 months</td></tr>
<tr><td colspan="3">Coordinator: Dr Jean-Remy Filtz, LNE, Tel: +33 130 691 215 E-mail: jean-remy.filtz@lne.fr
Project website address: www.eura-thermal.org</td></tr>
<tr><td>Internal Funded Partners:
1 LNE, France
2 CMI, Czech Republic
3 CNAM, France
4 FSB, Croatia
5 IMBiH, Bosnia and Herzegovina
6 BFKH, Hungary
7 MoE, Republic of Serbia
8 NSAI, Ireland
9 TUBITAK, Turkey
10 UL, Slovenia</td><td>External Funded Partners:
11 VINCA, Republic of Serbia</td><td>Unfunded Partners:
-</td></tr>
<tr><td colspan="3">RMG1: VINS, Serbia (Employing organisation); LNE, France (Guestworking organisation)</td></tr>
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