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Progress towards in-situ traceability and digitalization of temperature measurements

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ABSTRACT
Autonomous control systems rely on input from sensors, so it is crucial that the sensor input is validated to ensure that it is 'right' and that the measurements are traceable to the International System of Units. The measurement and control of temperature is widespread, and its reliable measurement is key to maximising product quality, optimising efficiency, reducing waste and minimizing emissions such as CO_2 and other harmful pollutants. Degradation of temperature sensors in harsh environments such as high temperature, contamination, vibration and ionising radiation causes a progressive loss of accuracy that is not apparent. Here we describe some new developments to overcome the problem of 'calibration drift', including self-validating thermocouples and embedded phase-change cells which self-calibrate *in situ* by means of a built-in temperature reference and practical primary thermometers such as the Johnson noise thermometer which measure temperature directly and do not suffer from calibration drift. All these developments will provide measurement assurance which is an essential part of digitalisation to ensure that sensor output is always 'right', as well as providing essential 'points of truth' in a sensor network. Some progress in digitalisation of calibrations to make them available to end-users via a website and/or an Application Programming Interface is also described.

Section: RESEARCH PAPER

Keywords: temperature; thermometry; traceability; primary thermometry; process control; digitalization

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1. INTRODUCTION

The control and monitoring of temperature is a key part of almost every technological process. The thermodynamic temperature of a system is related to the average kinetic energy of the constituent particles of the system. However, this cannot be measured directly, so another parameter which varies with temperature, such as the speed of sound in a gas, must be measured and then related to the temperature through well understood physics. In general, such an approach to temperature measurement is very complicated, time consuming and expensive, and is not currently well suited to practical thermometry.

Most thermometry therefore makes use of practical sensors such as thermocouples and resistance thermometers. These yield a temperature dependent property such as voltage or resistance, which must then be related to temperature by comparison with a set of known temperatures, i.e. a calibration. The global framework for approximating the SI unit of temperature, the kelvin, is the International Temperature Scale of 1990 (ITS-90) [1]. The measurement infrastructure that makes this possible is maintained by National Metrology Institutes (NMIs), who perform periodic global comparisons of their own standards to ensure the equivalence of thermometry worldwide. These standards are then used to provide calibrations to end-users which are traceable to the ITS-90, and hence the SI kelvin. In this way an end-user may be confident that their temperature measurements are globally equivalent.

A key drawback of this empirical approach to thermometry is that when the sensing region of the thermometer is degraded in use, for example by exposure to high temperatures, contamination, vibration, ionising radiation and other factors. the relationship between the thermometer output and its temperature changes in an unknown way. This is referred to as 'calibration drift', and it is insidious because there is no indication in process that it is occurring. This is a big problem for applications where temperature monitoring and control is critical, such as in long-term monitoring (e.g. nuclear waste storage), or where processes need to operate within a narrow

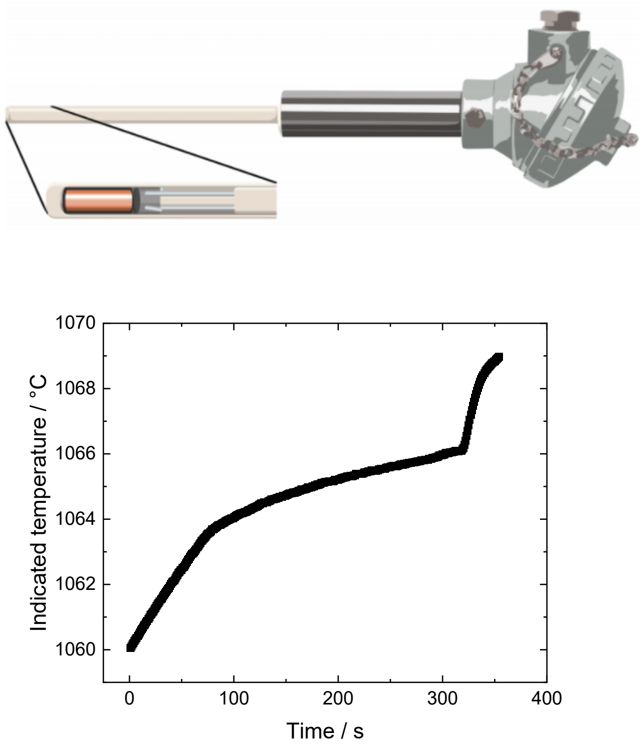

Figure 1. Top: self-validating thermocouple with protective sheath. Image courtesy of CCPI Europe. Bottom: melting curve observed during the recalibration of the INSEVA self-validating thermocouple, here using a gold ingot (melting temperature 1064.18 °C).

temperature window (e.g. aerospace heat treatment). The result is often reduced safety margins, sub-optimal processing, lower efficiency, increased emissions, and higher product waste or rejection.

In this article some new developments led by the UK's National Physical Laboratory (NPL), in collaboration with industry partners, are described to overcome the problem of calibration drift and to provide assurance that temperature sensor output is valid – a key part of increasingly widespread digitalisation to ensure sensor output is 'right' by providing *in-situ* validation. These include self-validation and in-process calibration which provide traceability to the SI kelvin at the point of measurement, and practical primary thermometry which measures temperature directly, has no need for calibration, and does not suffer from calibration drift.

Additionally, other promising practical primary thermometry techniques have been outlined, namely Doppler Broadening Thermometry, Ring Resonator thermometry, and Whispering Gallery Mode thermometry. These are collectively referred to as 'photonic thermometers' due to their use of electromagnetic radiation. Acoustic thermometry is also briefly discussed. Various groups worldwide, including NPL, are working on elevating the technological readiness of these techniques.

Finally, some developments in the digitalisation of calibrations are described, including automation, web-based access, and steps towards implementation of a standardised Digital Calibration Certificate. These will substantially reduce the amount of paperwork and opportunities for operator error and will facilitate digital transfer of calibrations and traceability for paperless audit trails.

2. SELF-VALIDATION

Thermocouples are very mature and well established, and are widely used in industry. However, they are particularly susceptible to drift of the calibration in harsh environments, whereby the relationship between emf and temperature changes in an unpredictable manner. This gives rise to a progressive, and unknown, temperature measurement error which in turn causes degradation in process monitoring and control. This can be monitored *in situ* by using a miniature phase-change cell (fixed-point) in close proximity to the measurement junction (tip) of the thermocouple [2]. The fixed point is a very small crucible containing an ingot of metal (or metal-carbon alloy [3] or organic material [4]) with a known temperature. The latest devices developed by NPL are able to accommodate the entire thermocouple and fixed-point assembly within a protective sheath of outer diameter 7 mm; the cell is typically about 4 mm in diameter and 10 mm in length. Importantly, this means that the self-validating thermocouple presents the same external form factor and appearance as a regular process control thermocouple. It is also of course fully compatible with existing connections and electronics. A self-validating thermocouple is shown in Figure 1.

In use, when the process temperature being monitored passes through the melting temperature of the ingot, the thermocouple output exhibits a 'plateau' during melting, due to the heat of fusion of the ingot restraining further temperature rise by ensuring incoming heat from the surroundings is absorbed, driving the phase change. Once the ingot is completely melted the indicated temperature resumes its upward trend. As the melting temperature of the ingot is known, having been traceably calibrated *a priori*, the thermocouple can be recalibrated *in situ*.

The question of the stability of the melting temperature of the miniature fixed point is important to consider, since that has the potential to inadvertently introduce further calibration drift. In fact, this is inherently stable, and it has been shown experimentally during the course of development of the devices that in typical applications the drift of the fixed point itself is negligible in the context of thermocouple measurement uncertainties. Contamination is by far the most likely cause of drift. As a general rule of thumb, 1 part per million of contamination by impurities gives rise to about 0.001 °C change in the melting temperature. So far, no evidence of measurable drift of the miniature fixed points has been found, even in quite harsh environments such as aerospace heat treatment processes. Calculations indicate that contamination by transmutation in ionising radiation environments is even less important in most situations, although the extreme case of operation in the core of a nuclear reactor may cause significant drift [5].

A typical output of a self-validating thermocouple during the recalibration process is shown in the lower panel of Figure 1. This device has been extensively characterised [6] and has been licensed by NPL to UK thermocouple manufacturer CCPI Europe, under the tradename INSEVA [7], who are conducting a series of trials in high value manufacturing industries at several plants in the UK and in Europe. Typical fixed-point materials for these applications include Ag (962 °C), Au (1064 °C), Cu (1084 °C), Fe-C (1153 °C) and Co-C (1324 °C).

A similar concept has been employed for an application in space-borne instrumentation, where the phase-change cell is part of the system whose temperature is to be measured. Such an embedded fixed-point has been demonstrated by NPL in collaboration with RAL Space on a prototype blackbody calibrator designed for operation as part of a spacecraft-borne

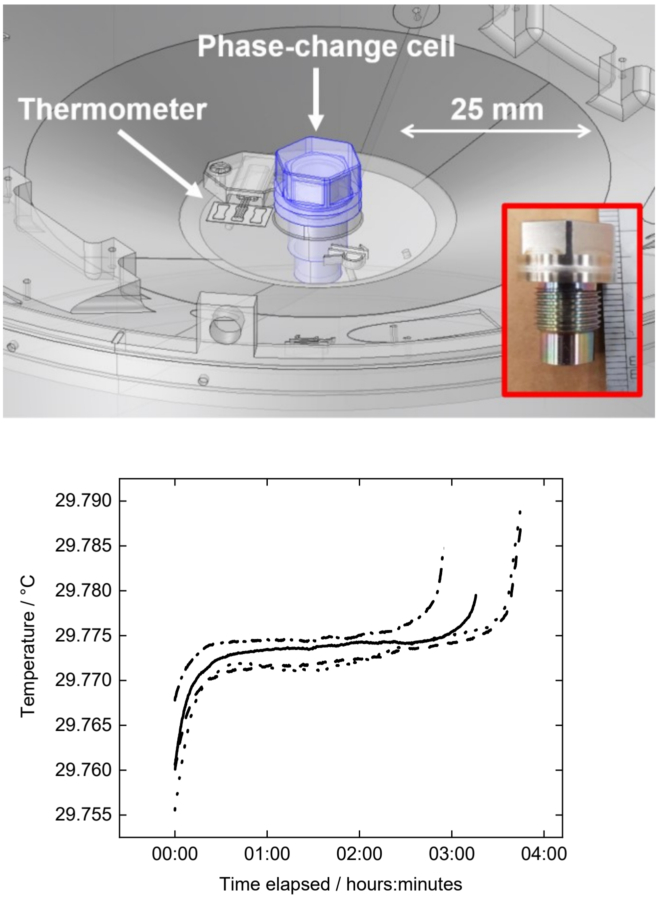

Figure 2. Top: phase-change cell embedded in the blackbody calibrator base; the adjacent PRT is to the left; inset shows a photograph of the phase-change cell. Bottom: melting curves observed during the *in-situ* calibration of the PRT using the miniature embedded phase-change cell, showing the narrow melting range and excellent reproducibility.

earth observation instrument suite [5]. The phase-change cell, containing approximately 2 g of gallium (melting point 29.7646 °C), is embedded in the aluminium blackbody calibrator base, close to an embedded platinum resistance thermometer (PRT). This enables the *in-situ* recalibration of the PRT in orbit.

In this application, some key developments included a mechanism to promote reliable freezing of the gallium without necessitating a large supercool (gallium is prone to cooling several degrees below its freezing temperature before nucleation is triggered), and a mechanism for preventing mechanical contact between the gallium ingot and the stainless steel cell wall, thereby avoiding the possibility of long-term contamination of the ingot and hence a change of its melting temperature. The ingot is shown in Figure 2.

It can be seen in the lower panel of Figure 2 that the remotely located PRT is able to indicate clearly defined melting curves with a useful duration of several hours, and a melting temperature range of less than 0.01 °C. By calibrating the phase-change cell against NPL's reference standard gallium cell, it is possible to perform *in-situ* traceable calibrations of the PRT on board the spacecraft with an expanded uncertainty of less than 0.01 °C.

For both the self-validating thermocouples and the embedded phase-change cell techniques, vigorous efforts are ongoing to automate the detection of the melting plateau, and, once detected, to characterise the 'fixed point' representing the invariant part of the melting curve. This is challenging to implement algorithmically in a manner sufficiently robust against noise and spurious artefacts in the data, but it is essential for autonomous *in-situ* recalibration.

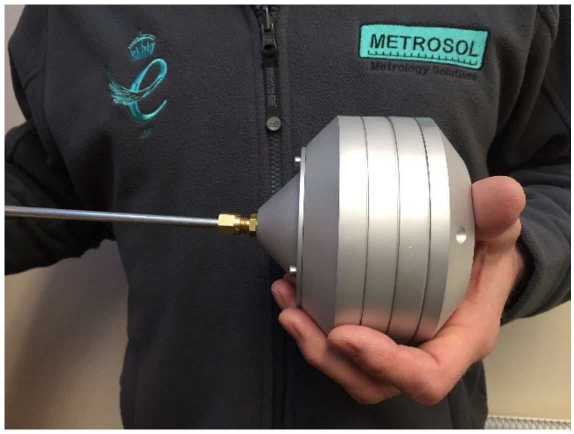

Figure 3. Prototype practical Johnson noise thermometer developed by Metrosol in collaboration with NPL. The sensing electronics are housed in the container to the right; the probe extends to the left.

NPL has had some success with a supervised learning approach (machine learning) on training data obtained from an industrial trial of self-validating thermocouples in a heat treatment application which yielded a large, high quality data set. This algorithm was then shown to work well on data that was not part of the training set, yielding typical expanded uncertainties in the melting point determination of about 0.5 °C (gold fixed point) and 1.0 °C (silver fixed point). Here and in the following, expanded uncertainties are taken to correspond to a coverage factor k = 2, i.e. a coverage probability of 95 %. Note that it is unlikely these uncertainties will be further reduced by improvements to the algorithm because they are dominated by experimental considerations associated with the physical measurement setup. Instead, algorithm development should focus on reliability and ability to characterise the plateau under adverse conditions such as noise, spurious artefacts, and faint signals. In other words, a demonstrable 'all weather' capability is needed.

Hence, while the new machine learning algorithm shows promise, it will need to be tested on a diverse set of data to demonstrate its universal applicability. The detection of characteristic shapes such as melting curves is essentially a pattern recognition problem. This is easy for humans because of the extraordinary sophistication of the visual cortex but it is not practical for conventional computer programming approaches, and in general machine learning or other artificial intelligence applications are needed to tackle this problem, together with good quality data for development and validation of the techniques.

3. PRACTICAL PRIMARY THERMOMETRY

The limitations of conventional temperature sensors which rely on calibration prior to use, and hence are prone to calibration drift, has led to renewed interest in practical primary thermometry. Primary thermometers measure some property that can be related to temperature directly through well-understood physics, and do not require a temperature scale or calibration. In addition, if all parameters needed to infer the temperature are measured simultaneously, the sensor is not subjected to calibration drift, since any change in the sensor material is accounted for in the measurement. Examples include acoustic thermometry (measuring the speed of sound) and Johnson noise thermometry (measuring the temperature-dependent voltage arising from the thermal motion of charge carriers in a resistor).

To turn one of these into a practical, commercially available reality, NPL has been collaborating with Metrosol Limited to develop a practical Johnson noise thermometer [8]-[10]. The Johnson noise voltage is related to temperature, T, by Nyquist's relation:

$$\langle V_T^2 \rangle = 4\, k\, T\, R\, \Delta f \,, \tag{1}$$

where $\langle V_T^2 \rangle$ is the mean squared Johnson noise voltage, k is the Boltzmann constant, R is the sensor resistance and Δf is the frequency bandwidth which is a function of the sensing electronics and cables. Importantly, if R is measured at the same time as the Johnson noise voltage, then all relevant properties of the sensing resistor are measured and so even if the sensor is degraded the thermodynamic temperature is always known.

The Johnson noise voltage is miniscule, and measuring it requires robust immunity to electromagnetic interference and electronic interference, arising from both external and internal influences. This is achievable by good design. A key challenge is the need for very high amplification of the noise signal. Its measurement in the presence of the inevitable electrical noise generated by the pre-amplifiers can be done with the use of correlation, whereby the signal is split into two different channels. Only the measured signal which is the same on both channels (i.e. the Johnson noise) is 'let through'. A drawback of this approach with conventional designs is that this results in excessively long correlation times of minutes to hours depending on the required uncertainty. On the other hand, industrial measurements typically require timescales of a few seconds.

Using the Nyquist equation (1) requires a knowledge of the bandwidth, which in reality is unknowable. The equation is generally used in ratio form at two different temperatures: the sensor temperature to be determined, and a known reference temperature. The Nyquist equation can hence be expressed as:

$$T = T_0 \left(\frac{V}{V_0}\right)^2 \frac{R}{R_0} \,, \tag{2}$$

where T_0, V_0 and R_0 are the reference temperature, Johnson noise voltage and resistance respectively. In general, it is very inconvenient to maintain a known reference temperature because it is impossible to match the frequency response of the two measurement circuits, and the resulting mismatch causes excessive measurement errors over the frequency range required for the current fast response time application. Various approaches have been employed to overcome this including the use of a synthesized noise signal from, for example, a Josephson array. While extremely accurate, this approach is also not feasible as it requires complicated low temperature equipment.

The NPL/Metrosol collaboration makes use of a quasi-random synthetic reference signal which is generated *a priori*. This reference signal is then superimposed on the measurement signal so that they both experience the same frequency response of the measurement electronics. The composite signal (superposition of Johnson noise and calibration 'tones') can then be decomposed with signal processing in the frequency domain, and their ratio determined in order to deduce the temperature of the sensor resistor. A further advantage of this mechanism is its high tolerance to highly non-flat, non-linear frequency response, and so a much higher bandwidth (up to 1 MHz) can be employed than in previous systems. This translates directly to shorter measurement times and hence faster response times, since more signal can be averaged in the same amount of time.

Johnson noise thermometry has until recently been the preserve of large national laboratories due to the extreme difficulty of isolating the miniscule Johnson noise voltage from the far larger external noise sources and the internal noise generated by the electronic components [11]. The development of a practical thermometer has been elusive and so far none have reached market, but the current NPL/Metrosol collaboration has now developed a working thermometer with unprecedented immunity to external electrical interference. The current prototype is shown in Figure 3. It has now passed the most stringent electrical immunity standard test, IEC 61000-4-3 [12]. The accuracy depends on the measurement duration; for an averaging period of about 5 s the expanded measurement uncertainty is ± 0.5 °C. The most obvious application is as a replacement for thermocouples where appreciable long-term drift is unacceptable. Efforts are now focused on increasing the maximum temperature range beyond about 150 °C and improving the electronics and signal processing.

Further developments in the pipeline include demonstration of the feasibility of photonic-based 'lab on a chip' thermometry approaches for *in-situ* traceability to the kelvin. Three approaches in various stages of investigation by NPL and its collaborators to facilitate direct *in-situ* traceability are Doppler Broadening, Ring-Resonator, and Whispering Gallery thermometry [13].

Doppler Broadening Thermometry (DBT) is based on the measurement of the Doppler profile of a molecular or atomic absorption line of a gas in thermodynamic equilibrium. The absorption line shape is dominated at low pressure by Doppler broadening and has a Gaussian profile corresponding to the Maxwell-Boltzmann distribution of velocities of gas particles along a laser beam axis. In practice various physical effects such as collisions distort the beam profile somewhat, but the theory of this is well understood and the absorption line shape may be fitted by a parameterised model. The Doppler half-width at half-maximum, Δv_D, is related to the temperature T by:

$$\Delta v_D = \frac{v_0}{c} \sqrt{2 \ln 2 \,\frac{k\, T}{M}} \,, \tag{3}$$

where v_0 is the line-centre frequency, c is the speed of light, and M is the absorber mass. Two key challenges currently being addressed are a) reducing the amount of ancillary equipment needed for implementing the technique and b) miniaturisation of the sensing element.

Ring-Resonator (RR) thermometry essentially utilises a closed-loop optical waveguide which is optically coupled to a second, adjacent, non-closed waveguide (separated by an air gap) via evanescence. The 'ring' or 'loop' enables propagation of circular electromagnetic waves with a characteristic resonance at a wavelength, λ_m, given by:

$$m \cdot \lambda_\mathrm{m} = n_\mathrm{eff} \cdot L, \tag{4}$$

where the integer m represents the resonance mode, n_eff is an index characteristic of the waveguide and L is the round-trip length of the loop. The temperature dependence of the refractive index and physical dimensions of the ring enable the use of the device as a thermometer by measuring the temperature-dependent shift in the wavelength given in (4). In practice, the change in refractive index per unit temperature is a factor of approximately 100 larger than the thermal expansion coefficients of the materials involved, so the latter may be ignored. The technique is readily miniaturised and has good resistance to chemical contamination. It also offers some of the lowest uncertainties of all the practical primary thermometry techniques, although great care is required during the fabrication process to avoid imperfections.

Whispering Gallery Mode (WGM) thermometers trace their ancestry to precision clock oscillators. In essence they are stable microwave resonators arranged such that a symmetric dielectric medium such as a cylinder or disk is suspended in the centre of a metal cavity. The electromagnetic field in the microwave region is coupled to an external waveguide to excite the resonant frequencies. The frequencies of these resonant 'whispering gallery' modes exhibit temperature dependence and may be related to temperature through an understanding of the associated physics, which enables the use of the device for thermometry.

Acoustic gas thermometry is also a candidate for practical primary thermometry. The speed of sound in a gas depends on the temperature and may be related to the gas temperature through well understood physics. By using an acoustic resonator which 'rings' like a bell when excited appropriately with loudspeakers, and by characterizing the resulting changes in the geometry of the device using microwaves to understand the resonant modes, an extremely accurate thermometer can be constructed. Such a device was used to determine the Boltzmann constant with unmatched accuracy as part of the global endeavour to redefine the kelvin in terms of fundamental constants [14].

4. DIGITALIZATION OF CALIBRATIONS

For many years the result of thermometer calibrations have been printed on paper and issued to the customer. Recently, however, there is a trend towards digitalisation of the calibrations so that the results are available online or in electronic files. This is very important functionality for many users; for example, in aerospace organisations where measurements are subject to significant regulatory compliance and demonstration thereof under frameworks such as AMS2750 which regulates heat treatment of metallic materials [15], it is very difficult to work with paper certificates. One successful approach has been that of CCPI Europe, who have fully automated certification with their PyroTag™ system [16].

Digitalisation of calibrations has numerous benefits including the reduction in operator errors (e.g. through manual data entry), removed need for paper-based processes and transactions, and easier management for asset managers, calibration managers, and technical staff. It will result in reduction of time and cost arising from a paperless system, offers secure storage and retrieval of information, and is audit ready to demonstrate traceability compliance.

This presents some infrastructural challenges, including the way the data is presented, the internal mechanisms in the calibration laboratory for enabling digitalisation, and security of the information required to ensure that only the intended recipients have access.

NPL has embarked on a programme to automate, as far as possible, its thermometer calibrations and the generation of calibration certificates, and to make them, and the associated data and metadata available online via a secure website. The certificates will be machine readable (XML). The results will also be available through an Application Programming Interface (API), allowing integration with customers' own software. A key aim is ultimately to integrate this capability with the international Digital Calibration Certificate (DCC), whose format is currently undergoing development [17]. Importantly, the calibration history will also be available to the user.

Importantly, the DCC offers the possibility of facilitating autonomous updating of calibration data. This could be exploited by the techniques described in this paper, particularly self-validating techniques which provide a live update of the calibration *in situ*. Updating one point in the calibration generally has an effect not only at the temperature at which the self-calibration is performed, but on the interpolating function over a wider temperature range. The updated calibration could be passed to the associated DCC which could then be updated to include the new parameters and correct the interpolating function over the wider temperature range of use. Clearly such a mechanism does not yet exist, but this is a functionality that should be considered in the formulation of the DCC format and its implementation. This approach may also be applicable to practical primary thermometry, though in that case the role of calibration certificates more broadly, and even the role of National Metrology Institutes in providing traceability in this regime, is currently not well defined.

5. CONCLUSIONS

Some new developments in temperature measurement have been presented which support digitalisation in various respects. Self-validation techniques using miniature temperature fixed points based on 'phase change cells' to provide *in-situ* traceability at the point of measurement will provide assurance that temperature sensor output is 'always right'. Practical primary thermometry measures temperature directly, rather than requiring calibration and the risk of consequent calibration drift in harsh environments, so ensures long-term reliable measurements; examples outlined here include Johnson noise thermometry, Doppler Broadening thermometry, Ring Resonator thermometry, Whispering Gallery Mode thermometry and acoustic thermometry. For conventional sensors, digitalisation of calibrations at NPL is becoming a practical reality with a web-based interface, associated API to enable end-users to access calibration data programmatically, and steps towards a standardised Digital Calibration Certificate format. These developments all support digitalisation of metrology and will increase the reliability of measurements, improving process efficiency and product yield, with a consequent reduction in harmful emissions. Future work will focus on elevating the technical readiness and bringing the innovations to market.

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