ACCREDITED CALIBRATION LABORATORY SERVICE AS A SUBJECT OF AN INTEGRAL QA SYSTEM AT EARS

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1. Introduction

Environmental Agency of the Republic of Slovenia (EARS) is a part of Ministry of the Environment and Spatial Planning. After integration of former Hydrometeorological Institute into EARS in year 2000, Monitoring Office was established with the intention of operating a network of automatic stations for meteorology, hydrology, ambient air-quality, water quality and ionizing radiation. At present time, there are about 60 automatic stations in our measuring networks, sending data in real time to the central database of EARS, which are quite different regarding measuring parameters and purpose. Strategic issue of EARS is quality of measured data in meteorological, hydrological and ecological network. The most important goal of Monitoring Office is to build the common integral quality assurance system for different measuring networks, which will base on ISO/IEC 17025 standard. New legislation that covers environmental monitoring, especially modern European directives in the field of air and water quality, requires that quality systems has to be established to assure technical competence, quality and international comparability of data. Meteorology, on the other hand, is bound mainly to WMO guides and recommendations, ICAO and other relevant documents. Calibration Laboratory (CL) is an important subject in an integral QA system in terms of periodical calibrations of field measuring instruments. CL perform accredited calibrations of measuring instruments in the field of temperature and air pressure. Accreditation, based on ISO/IEC 17025 standard, was achieved in 1999 in the field of temperature calibrations by joined assessment of French accreditation service - COFRAC (Comitee Francais d'Accreditation) and Slovenian Accreditation - SA. Scope of accreditation was extended in the field of air pressure in 2002. Calibration Laboratory is now preparing for accreditation assessment in the field of relative humidity and air quality quantities: carbon monoxide, sulphur dioxide, ozone, nitrogen monoxide and nitrogen dioxide.

Each accredited laboratory establishes and maintains its traceability to the national or international level. Recognition and analysis of uncertainty sources is most important subject in calibration procedure development and uncertainty evaluation. Traceability schemes and calibration procedures are further presented in the area of calibration in Calibration Laboratory.

2. Temperature

Calibration Laboratory performs calibrations of different type thermometers: classic liquid-in-glass thermometers, platinum resistance thermometers, self-indicated thermometers, thermistors and thermographs in a temperature range from -40°C to 50°C. We use Pt25 as a reference temperature standard which is periodically calibrated in slovenian national laboratory (LMK) with fixed point method. This external traceability links reference standard to the International temperature scale ITS-90. Internal checks of reference standard in two fixed points (water triple point and gallium melting point) are regularly performed to ensure drift and metrological stability. Intercomparison of our two fixed points assure redundant traceability to the ITS-90 in reduced temperature interval. Standard platinum resistance thermometer Pt25 is strictly used for Pt100 working standard comparison calibration in a state-of-art temperature controlled liquid bath. At present time there is not any accepted standard procedure for the metrological evaluation of the calibration baths.

Accredited laboratories have to set up their own procedures to evaluate time instability and spatial unhomohenity of calibration baths according to available equipment of the laboratory. In the field of temperature comparison calibrations, uncertainty of reproduced quantity represents major contribution to the total uncertainty. Therefore the correct and precise characterization of calibration baths is very important.

Calibration laboratory developed its own procedures to estimate liquid bath and climatic chamber metrological characteristics. Other equipment used in calibration process (standard resistors, multimeters) are traceable to the Slovenian Institute for Quality and Metrology (SiQ) and National Research Center in Canada. Traceability scheme is shown figure 1.

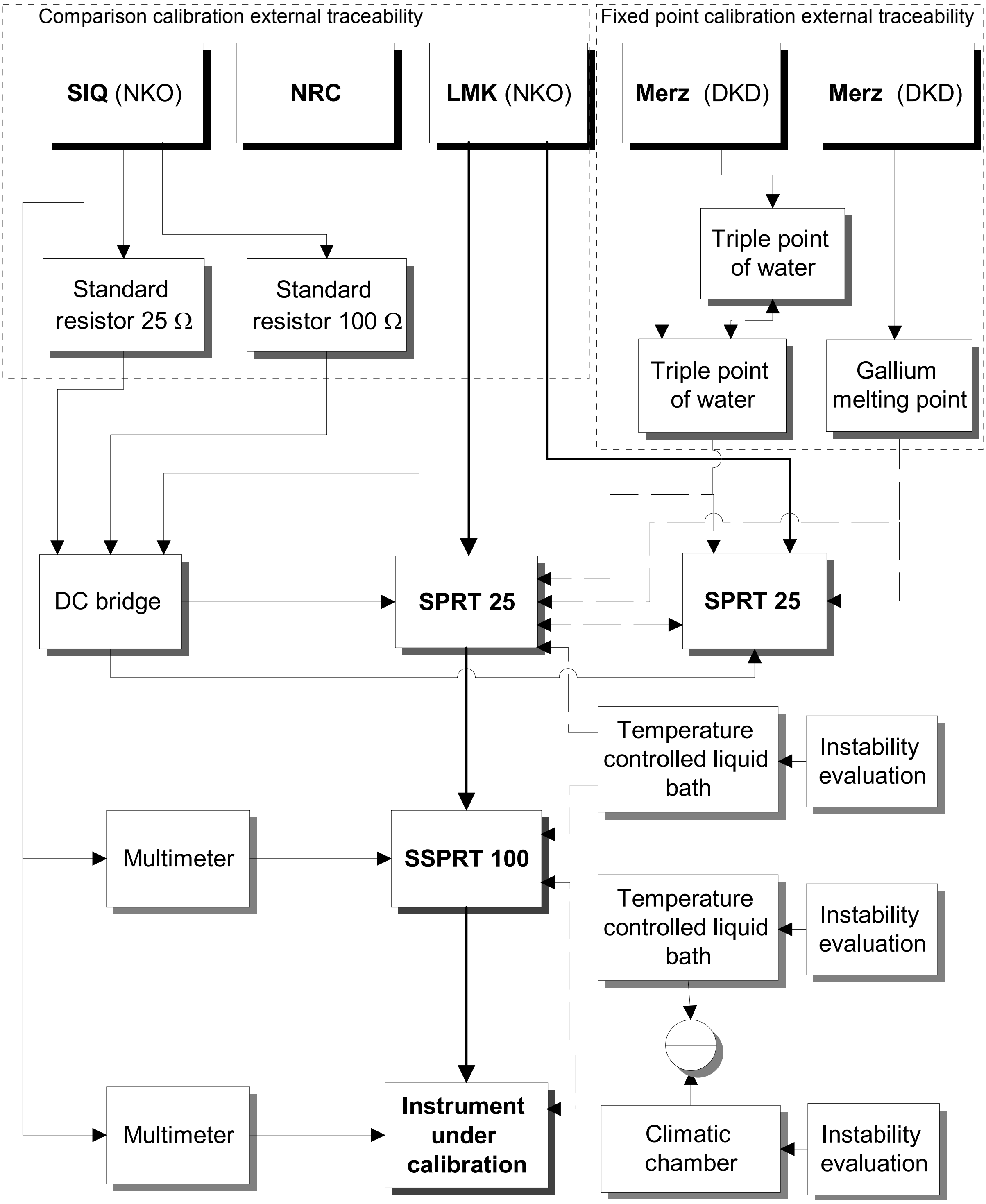

Figure 1: Traceability of temperature calibrations at EARS

Best measurement capability (BMC) for resistance thermometer comparison calibration, evaluated from uncertainty budget and accepted in accreditation assessment is 15 mK. For liquid-in-glass thermometers and other instruments under calibration the BMC is 70 mK.

3. Air pressure

Most instruments calibrated in Calibration Laboratory in the field of air pressure are electronic barometers, barographs and mechanical barometers in the range from 600 to 1200 hPa. For barographs and mechanical barometers the Theodore Friedrichs pressure chamber is commonly used. Degranges et Heut dead weight gauge is a pressure reference standard. Highly pure nitrogen (N5) combined with pressure regulator (DH PPC1) is used as a pressure media as shown in figure 2. Dynamometer of reference standard with piston-cylinder assembly transforms pressure into force. Special precisely defined set of masses are used to check and recalibrate dynamometer on a daily bases.

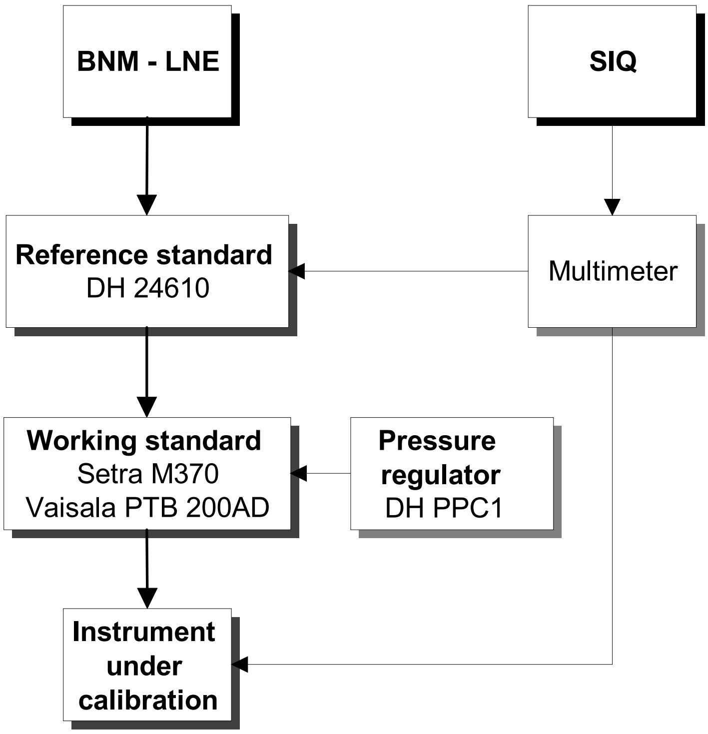

Figure 2: Traceability of calibrations of air pressure instruments

Reference standard is traceable to French BNM-LNE laboratory with expanded uncertainty 6 Pa.
Two high precision barometers Setra M370 and Vaisala PTB200AD are working standards with uncertainty estimation of 17 Pa. For high precision pressure calibration reference standard can be used instead of working standards.

4. Relative humidity

Traceability of relative humidity instrument calibrations is maintained in the range from 10% to 95% in temperature range from -20°C to 40°C. Dew-point hygrometer Mitchell S4000 as a reference standard was primarily calibrated in WYKO Calibration Service in Great Britain as shown in figure 3. In future reference standard will be calibrated in CL using temperature standards and capabilities. Recently CL bought Thunder Scientific 2500 two pressure humidity generator to improve efficiency and metrological capabilities. Humidity generator is nowadays in a testing and implementing mode and it is expected to improve best measurement capabilities to 1% level.

Operating principle of dew-point reference standard is based on a plated copper mirror and Peltier thermoelectric device as commonly known. At a temperature determined by the moisture content of sample air, dew forms on the mirror surface as Peltier device cools the mirror. This formation of dew causes reduction in reflected light intensity from red LED light source. The control loop maintains the mirror surface at the exact dew-point temperature which is accurately measured by an embedded platinum resistance thermometer. The measurement uncertainty arises from several effects on the mirror surface (Raout and Kelvin effect, uncertainty of determining dew/frost point, uncertainty of sampling system) and uncertainty of dew-point temperature measurement.
The most important contribution to the overall uncertainty of dew-point hygrometer is uncertainty of measurement of ambient air temperature and dew-point temperature. Expanded uncertainty 50% relative humidity and air temperature 40°C of Mitchell S4000 is 0.7%.

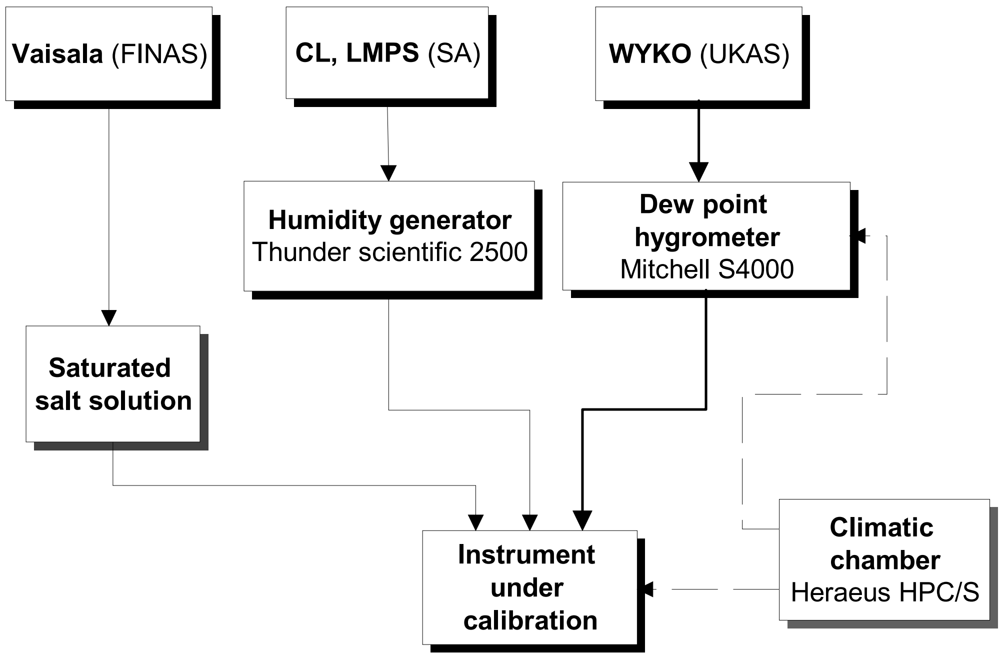

Figure 3: Traceability of calibrations of relative humidity instruments at EARS

Saturated salt solutions are used for adjustments of capacitive humidity instruments only. Climatic chamber is the main uncertainty source in comparison calibrations of field instruments (capacitive sensors, hygrographs). Reduction of time instability and spatial unhomogenity is possible if reduced volume of climatic chamber is used. Expanded calibration uncertainty of field instruments is at present time 4%.

5. Air quality quantities

Calibration Laboratory is now preparing for accreditation assessment of air quality parameters: concentration of carbon monoxide, sulphur dioxide, ozone, nitrogen monoxide and nitrogen dioxide at ambient air concentration levels. CL uses certified reference standards (material measures held in cylinder containers or for the case of O3 a TEI 49C PS O3 calibrator) traceable to Czech national standards (CHMI). Our working instruments are calibrated in two points (zero air and CRM), as shown in figure 4. For customers we offer calibrations of instruments and material measures (cylinders or sample generators). In the case of instrument calibrations we offer two point calibration by zero air and CRM and multipoint calibration by comparison measurement to our working instrument. In comparison measurements between two instruments a stable but not traceable sample generator is used. In case of calibrations of material measures we offer calibrations of gas cylinders and multipoint calibrations of sample generators. For the future we plan to become an independent calibration laboratory by accrediting procedures for preparation of gas mixtures containing above stated substances (accept ozone). Gas mixtures will be prepared by dilution of reference materials manufactured by NMI using static mixing chamber.

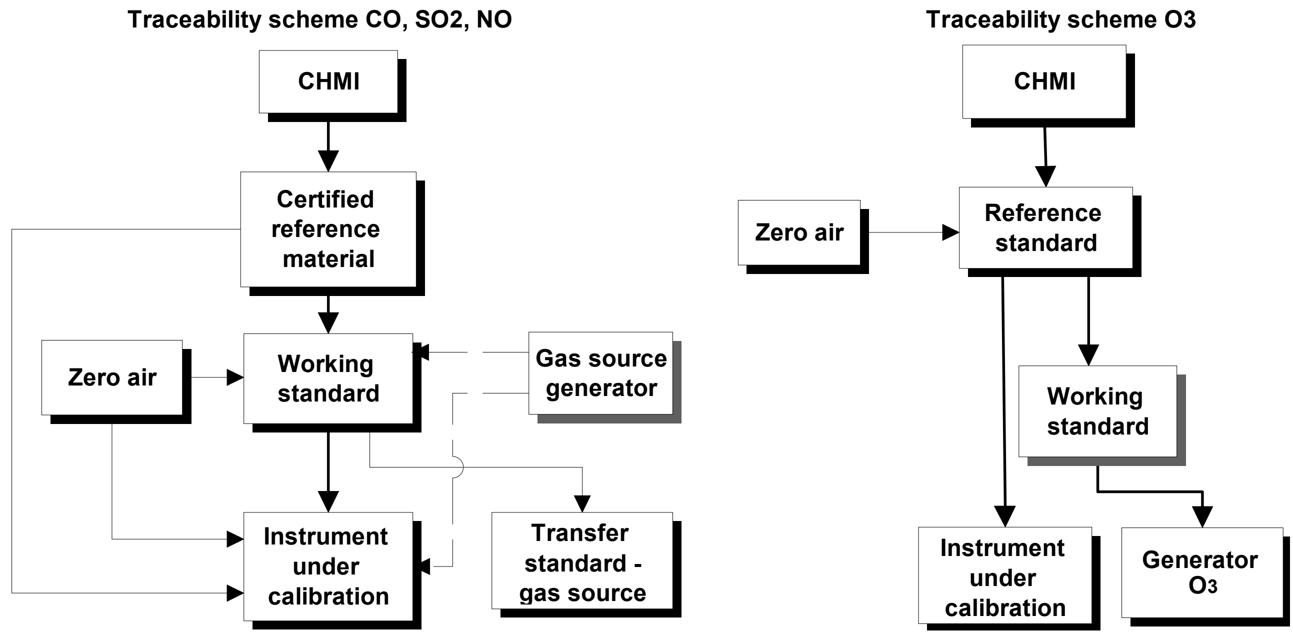

Figure 4: Traceability of calibrations of air quality quantities

6. Conclusion

Best measurement capabilities is a measure to compare laboratories between themselves and represent the lowest uncertainty level of calibration. BMC are shown on table 1.

Measured quantities	Range	Best measurement capability (k=2)
Temperature		
Resistance thermometers	-40 °C ÷ 50 °C	15 mK
Liguid-in-glass thermometers	-20 °C ÷ 50 °C	70 mK
Thermographs	-20 °C ÷ 0 °C 0 °C ÷ 50 °C	0.3 °C 0.2 °C
Thermophysical properties		
Water triple point	0.01 °C	1 mK
Gallium melting point	29.7646 °C	1.5 mK
Relative humidity	10 % ÷ 95 %	1% *
Air pressure	600 hPa ÷ 1200 hPa	$1.3 \cdot 10^{-5} \cdot p + 5$ Pa
Air quality quantities		
CO concentration	0 -15 ppmv	0.07 ppm + $0.016 \cdot c_{CO}$ *
SO_2 concentration	0 - 500 ppbv	0.8 ppb + $0.021 \cdot c_{SO2}$ *
NO concentration	0 - 500 ppbv	0.9 ppb + $0.031 \cdot c_{NO}$ *
NO_2 concentration	0 - 500 ppbv	1.5 ppb + $0.02 \cdot c_{NO2}$ *
O_3 concentration	0 - 500 ppbv	2.6 ppb + $0.024 \cdot c_{O3}$ *

* Expected expanded measurement uncertainty

Table 1: Best measurement capabilities of Calibration Laboratory

Other capabilities of CL include field calibration of solar radiation instruments using Sun as a light source, wind speed and wind direction instruments testing, precipitation instrument testing, calibration of water level sensors and calibration of analog inputs of automated stations.

According to the quality system of EARS Calibration Laboratory covers almost all calibration needs of our meteorological, hydrological and ecological network.

References

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