TÜBİTAK UME vacuum laboratory capabilities and activities

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Abstract. TÜBİTAK National Metrology Institute (TÜBİTAK UME) is a research and development institute that operates under the Scientific and Technological Research Council of Turkey (TÜBİTAK). TÜBİTAK UME Vacuum laboratory takes part in under the mechanics group among the groups of physics, mechanics and chemistry group laboratories. TÜBİTAK UME Vacuum Laboratory aims to develop and maintain national measurement standards, to establish vacuum pressure traceability to SI units and to disseminate this to industry, science and research in Turkey. The laboratory was founded in 1996 and started to provide secondary level calibration services with Vacuum Gauge Calibration System (VGCS-200). Primary vacuum standard called Multi-Stage Static Expansion System (MSSE1) was established in 2000. Commercially available secondary vacuum gauge calibration system was improved with the addition of automation under the name Vacuum Gauge Metrology System (VGMS) in 2010. The main activities of the Vacuum Laboratory are conducted in the following working fields: Primary and secondary vacuum measurements and calibrations, secondary calibrations of leak standards, primary constant pressure flowmeter (CPFM) standard establishment works, renewal works of existing primary static expansion system, European Metrology Research and Innovation Projects, Quadrupole Mass Spectrometer (QMS) characterization measurements on Dynamic Vacuum System (DVS), training and consultancy services in the field of vacuum metrology. In this paper, the Vacuum Laboratory capabilities and activities will be briefly introduced.

1. Introduction
TÜBİTAK National Metrology Institute (TÜBİTAK UME) is a research and development institute that operates under the Scientific and Technological Research Council of Turkey (TÜBİTAK) [1]. The institute aims to ensure the reliability of all measurements conducted in Turkey, to integrate of these measurements into the international system and to develop existing and new measurement technologies by establishing national measurement system. In accordance with this purpose, TÜBİTAK UME Vacuum Laboratory aims to develop and maintain national measurement standards, to establish vacuum pressure traceability to SI units and to disseminate this to industry, science and research in Turkey.

The laboratory takes part with two main measurement quantities in the BIPM (Bureau International des Poids and Measures)/CMC (Calibration and Measurement Capability) tables of mass and related quantities about the vacuum pressure and it has been accredited in 2005 by TÜRKAK (Turkish Accreditation Agency) in the following scope, given in table 1. The calibration and measurement capabilities are capacitance manometers (CDG), pirani gauges, thermal conductivity vacuum gauges, hot and cold cathode ionization gauges and spinning rotor gauges (SRG).
The laboratory participated in 3-year joint research project, ended in 2014, EMRP IND12 "Vacuum metrology for production environments" funded by the European Metrology Research Program, in the areas such as stability measurements of leak detectors, characterization of quadrupole mass spectrometers and investigation of the effects of various parameters on the sensitivity of the QMS [2]. Goal of this project is to open new measurement capabilities for vacuum and to help industry to characterize vacuum in industrial environments. In the studies of the characterization of QMS study, the laboratory has taken an active role as a task leader [3]. Further, the laboratory continues his contributions to the European Metrology Programme for Research and Innovation project named EMPIR 14IND06 “Industrial standards in the intermediate pressure-to-vacuum range”, which will end in 2018 [4]. The laboratory gives consultancy services and trainings to his customers from industry and military regarding vacuum metrology, calibration methods, and measurement techniques. The primary standard has been involved in key comparisons that were organized in the frame of EUROMET.M.P-K1 (442 a/b) and other international comparisons are still ongoing EURAMET.M.P-K15.1, COOMET.M.P-K15 and COOMET.M.P-S2.

2. The capabilities and activities of the laboratory

Vacuum gauge calibrations are performed on VGMS between $1 \times 10^{-4}$ Pa to $1 \times 10^{5}$ Pa with the relative uncertainty of $8.3 \times 10^{-2}$ to $7.9 \times 10^{-3}$. Figure 1 shows a simplified view of the test chamber with test ports at the top and test gas entering on the side and being ported to the top of the test chamber that is designed in accordance with ISO/DIS 3567. The main and rough pumps are at the bottom of the chamber. The comparison method is used for the calibration of vacuum gauges, where the device under calibration is compared to a calibrated reference standard. The readings of the two vacuum gauges can be compared only if both are exposed to the same pressure. The main part of the system is vacuum chamber with eight ports to which reference gauges and test gauges to be calibrated are connected. The vacuum chamber and the position of the test flanges have been designed to fulfill this condition. The system provides automated control of vacuum levels from nominally $1.3 \times 10^{-4}$ Pa to $1.3 \times 10^{-3}$ Pa dynamic pumping and up to 130 kPa static fill. The system uses a turbo pump with integrated electronic drive unit. A diaphragm pump backs the turbo and a dry scroll provides rough vacuum for the test chamber and gas feed tank on the system, with which ultimate vacuum values of $10^{-6}$ Pa are obtained. A nude ion gauge is used as feedback transducer to monitor the chambers high vacuum inlet pressure. Traceable pressure measurements are made by either a SRG or one of three high-accuracy CGSs, depending on the pressure range. In the low vacuum mode, one of the capacitance manometers with the ranges of 130 Pa, 1.3 kPa and 130 kPa is used as reference standard; whereas in the high vacuum mode spinning rotor gauge with the range of $10^{-4}$ Pa to 1 Pa is used. The system is controlled either by manually or by computer in combination of controllers of transfer

| Table 1. The laboratory scope by Turkish Accreditation Agency (TÜRKAK). |
|-------------------------------------------------|
| Calibration scope and calibrated gauges | Measurement Range, in Pascal | Best Measurement Capability (Expressed as an expanded uncertainty) ($k=2$), p in Pascal |
| Absolute Pressure / Pa / Pirani and Thermocouple vacuum gauges | $1 \times 10^{-1} \leq p \leq 1 \times 10^{5}$ | $4.1 \times 10^{-2} \times p$ to $2.0 \times 10^{-2} \times p$ |
| Absolute Pressure / Pa / Capacitance Diaphragm Manometers (CDG) | $1 \times 10^{-1} \leq p \leq 1 \times 10^{5}$ | $3.5 \times 10^{-2} \times p$ to $7.9 \times 10^{-3} \times p$ |
| Absolute Pressure / Pa / Hot and Cold Cathode Ionisation vacuum gauges | $1 \times 10^{-4} \leq p \leq 1 \times 10^{1}$ | $1.1 \times 10^{-3} \times p$ to $4.0 \times 10^{-3} \times p$ |
| Absolute Pressure / Pa / Spinning Rotor Gauges (SRG) | $1 \times 10^{-4} \leq p \leq 1 \times 10^{1}$ | $9.0 \times 10^{-2} \times p$ to $3.5 \times 10^{-2} \times p$ |
standards. Feedback transducer is ion gauge or capacitance manometer in low pressure mode and in the high pressure mode, respectively.

**Figure 1.** Vacuum Gauge Metrology System (VGMS).

**Figure 2.** TÜBİTAK UME Primary Vacuum System (MSSE1).

The reference standards of VGMS are traceable to the primary multi-stage static expansion system, MSSE1, shown in figure 2. The pressure range of the primary system is between 0.9 mPa to 1000 Pa with the relative uncertainty between $2.1 \times 10^{-3}$ to $9.5 \times 10^{-4}$ [5]. The expansion method of generating known low pressures was proposed by Knudsen (1910) and has subsequently been developed by a number of workers as a wide-range and accurate method of vacuum gauge calibration. Two methods have been developed of applying this principle to the generation of the known low pressures for calibration purposes. The principle of the method is to expand a small volume of suitable gas at a measured (relatively high) pressure into a pre-evacuated, larger volume, thereby effecting a pressure reduction which is calculable from the known ratio of the systems’ volumes. The generated pressure is directly calculable from the initial pressure and the volume ratio. To generate lowest pressures, a multi-stage system in which the large overall volume ratio is effectively achieved by successive expansions through a series of volume pairs, each of modest volume ratios. MSSE1 static expansion system has three stages each of volume ratio about $10^2$. This is known as the ‘Series Expansion’ method. The lower range of the system may be extended by one decade by using cascaded expansion method. The cascaded expansion method is basically based on the reduction of the pressure value by pumping the expansion volume after the first step with the adjacent transfer volume isolated and then once again expanding the gas from the transfer volume to the calibration volume. Work is continuing with measurements regarding this method. The traceability chain is schematically given in figure 3. Quadruple Mass Spectrometer measurements such as determination of gas impurities and monitoring the vacuum conditions in vacuum chambers in the mass range of 1 to 200 amu can be performed in the
laboratory. Needed measurements with different gasses such as $\text{N}_2$, $\text{Ar}$, He and $\text{CO}_2$ were performed at the laboratory in the scope of EMRP IND12 project.

![Diagram of experimental setup](image)

**Figure 3.** TÜBİTAK UME Vacuum Laboratory SI Traceability Chain; QBS: Quartz Bourdon Tube Sensor.

Under the scope of the project, the laboratory initiated to work in the fields such as leak standard measurements, stability measurements of QMS and Mass Spectrometer Leak Detector (MSLD), and the investigation of the effects of various parameters on the sensitivity of the spectrometer. These kinds of measurements have been conducted on the “Dynamic Vacuum System (DVS)” established in 2012, shown in figure 4. A simple and reliable dynamic vacuum system for in situ calibration of extractor gauges (EXG) and QMS’s was realized at TÜBİTAK UME in collaboration with colleagues from national metrology institute of Korea, KRISS. The system can also be used for QMS tests and characterization of dry vacuum pumps. The system consists of two chamber units. In the single chamber unit, throughput method is used with the throughput range higher than $10^{-3}$ mbar·l/s. In the double chamber unit, orifice method is used with the throughput range higher than $10^{-10}$ mbar·l/s. A simple way is used for pressure generation and control in this case. The gas admission from the gas regulator into the upper chamber is maintained through the UHV leak valve and through the 10 mm-orifice for the lower chamber. The double chamber unit can be operated by means of 4 replaceable orifices with different sizes (from 10 to 30 mm). The base pressure in the lower chamber is approximately less than $5\times10^{-7}$ Pa and can be reached within 12 hours by continuous evacuation of the system.

The system was modified with the integration of SCE (Standard Conductance Element) manufactured by National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (NMIJ/AIST) in 2014, figure 5. SCE serves to introduce a test gas with known flow rate to a vacuum chamber, which is evacuated at the pressure of less than 1 mPa. The SCE is made of a stainless-steel sintered filter with the pore size of less than 1 µm. Since the gas flow through the SCE satisfies the molecular flow condition even at the pressure up to 10 kPa, some useful characteristics of molecular flow are available. Molecular conductance is between $1\times10^{-10}$ m³/s to $2\times10^{-9}$ m³/s of the SCE. Calibrations for various gas species are available only with this single leak element because the conductance is easily compensated by the molecular mass of gas species.
The laboratory continues its activity in the field of gas flow rate measurements realizing a measurement station to meet the national requirements on the leak standard calibration in the range from $2 \times 10^8$ mbar·l/s to $2 \times 10^5$ mbar·l/s with permeation type helium leak standards having different leak rates, figure 6. The comparison method is used, where unknown leak is compared with two known standard leaks. Two standard leaks with known leakage rates normally lying on either side of the unknown leakage rate is used for comparison. The UME calibration apparatus works starting from reference leaks calibrated against primary flowmeter available in other NMIs. The simplicity and efficiency of comparison calibration method make it suitable for the calibration of leaks in industrial level. For this reason it can be considered as a secondary standard.

The temperature and moisture parameters are saved by PC automatically in the laboratory. The values of and their tolerances and calibration/test list are given in tables 2 and 3, respectively.

The use of primary flowmeter both to generate and measure gas flows is essential in continuous expansion systems used as primary vacuum standards and for standard leak calibration, and many metrological laboratories carry out gas flow determination studies [6–9]. Therefore, primary constant pressure flowmeter (CPFM) standard establishment project is continuing under the scope of TÜBİTAK UME Research Infrastructure Renewal and Development Projects funded by Republic of Turkey Ministry of Development, 2017-2019. The other project is also in progress: Renewal of existing primary static expansion system. The static expansion system has been used for many years and needs to be renewed. Targeted gas flow operating range regarding the CPFM is between $4 \times 10^{-12}$ mol/s to $4 \times 10^{-7}$ mol/s. The system design, procurement and manufacturing processes are continuing. With the primary gas flow meter installation and integration to the dynamic vacuum system, it will be
the primary level standard named continuous expansion system with the estimated range between $10^{-7}$ Pa to $10^{-3}$ Pa. In addition, traceability of gas flow rate measurements and standard leak calibrations will also be provided in this way. This works will extend the pressure measurement range at the primary level down to $10^{-7}$ Pa.

Table 2. The ambient conditions values.

| Ambient Parameters | Required Values | Tolerance |
|--------------------|-----------------|-----------|
| Temperature        | 20 ºC (23 ºC optional) | ± 0.5 ºC |
| Moisture           | % 45            | ± 15      |

Table 3. Vacuum Laboratory Calibration/Test List.

| Service code | Name |
|--------------|------|
| G2VK-1100    | Pirani, Thermal Conductivity, Thermocouple Type Vacuum Gauges |
| G2VK-1200    | Capacitance Diaphragm Type Vacuum Gauges (CDGs) |
| G2VK-2100    | Cold and Hot Cathode Type Vacuum Gauges |
| G2VK-2200    | Spinning Rotor Type Vacuum Gauges (SRGs) |
| G2VK-3100    | Full Range Vacuum Gauge Calibration |
| G2VK-5100    | Helium leak rate detection in vacuum ($2\times10^{-5}$ mbar-l/s – $2\times10^{-6}$ mbar-l/s) |
| G2VK-5200    | Helium leak rate detection in vacuum ($2\times10^{-6}$ mbar-l/s – $2\times10^{-8}$ mbar-l/s) |
| G2VK-9900    | Special Calibration / Test |

3. Conclusion
TÜBİTAK UME Vacuum Laboratory is responsible for supply of reference standards in the area of vacuum metrology in Turkey. The main aim is to provide traceability to National Standards based on SI units, Pa, for industry and government departments. The laboratory continues to keep its place in the national and international metrology network through both with the standards it has and its activities. The laboratory served under the Pressure Laboratory of TÜBİTAK UME until 2015 and it was separated from the pressure laboratory at the beginning of 2015. The studies mentioned above are conducted with three researchers who all have over 20 years of metrology experience.

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