Metrological support of test leak in vacuum

D M Fomin
Saint-Petersburg, D.I. Mendeleev Institute for Metrology, Moskovskiy avenue, 19

E-mail: vacuum@vniim.ru

Abstract. The article discusses work principles of test leaks in vacuum, metrological support and main problems arising during operation.

Test leaks are widely used in various industry fields for calibration of equipment used to control leak tightness and emission of gas in vacuum systems and produced items. For example, parameters of mass spectrometer helium leak detector are adjusted by means of test leak (helium leak) installed inside the device (or in the tested vacuum system).

The following types of test leaks (helium leaks) are currently approved as measurement tools and are included in the Federal informational fund on uniform measurements provision (State Registry of Measurements Tools). See Table 1.

| N  | Name of Measurement Tools | Reproduced flow rate range, Pa·m³/s | Maximum permissible relative error of flow reproduction, % |
|----|--------------------------|-------------------------------------|----------------------------------------------------------|
| 1. | Gelit 1                  | 7·10⁻¹⁰ - 2·10⁻⁸                   | ±15                                                      |
| 2. | Gelit 2                  | 3·10⁻¹¹ - 7·10⁻¹⁰                  | ±20                                                      |
| 3. | 10xxxx                   | 1·10⁻⁶ - 9·10⁻⁶                    | ±15                                                      |
| 4. | Fx4xxx                   | 1·10⁻¹⁰ - 3·10⁻⁵                   | ±15                                                      |

Test leaks allow adjusting leak detector in order to get correct quantitative characteristics of leak tightness. Before mass spectrometer helium leak detectors were included into Federal informational fund on uniform measurements provision, qualitative characteristics of leak detectors (including maximum permissible relative error) were determined with their help.

The main equation used in mass spectrometer helium leak detectors to calculate the rate of inleakage $Q$, has the form [1]

$$Q = Q_{3T} \cdot \frac{\alpha_{\text{изм,эт.}} - \alpha_{\Phi}}{\alpha_{\text{изм,эт.}} - \alpha_{\Phi}} \tag{1}$$

where $Q_{3T}$ – the amount of flow of helium test leak, installed inside the leak detector or in the vacuum system, specified in the certificate on verification (calibration) of leak;

$\alpha_{\text{изм.}}$ – the rate of tracer gas flow in vacuum system or in produced item, measured by leak detector in real time;

$\alpha_{\text{изм,эт.}}$ – the rate of tracer gas flow from the leak, installed inside the leak detector (or in vacuum system), measured by leak detector;
\( \alpha \phi \) – background signal.

As can be seen from the formula, the test leak makes a significant contribution in accuracy and reliability of test results, therefore metrological support of this equipment is essential to improve the accuracy of test results.

The most common test leaks are diffusion and capillary ones. Operation principle of diffusion test leaks is better to be considered by the example of test leak (helium leak) Gelit 1 and Gelit 2. The difference in construction modifications is in the diverse material of permeable element – quartz glass (Gelit 1) and molybdenum glass (Gelit 1).

Diffusive test leak (picture 1) is a hermetically sealed metallic tank (1) with a pipe (3) on one side for the connection with the tested vacuum system or vacuum system of leak detector. Inside the tank a tube is connected with the permeable element being a ball-shaped flask (2) made of molybdenum or quartz glass. The walls of the tank, tubes and permeable element create enclosed volume filled with helium up to the pressure from 6 to 100 kPa. Diffusion of helium through the flask walls of permeable element takes place during the operation. Wall thickness, surface area of permeable element as well as helium pressure in the tank define the range of produced flow.

Capillary test leak has similar construction but instead of permeable element, a capillary made of different materials (glass, fluoroplastic, metal, etc.) is installed. Tracer gas is filled in under the pressure of 0.1 to 1 mPa, in some cases pressure reaches 10 mPa. The major advantage of capillary test leaks is the possibility of filling them with different types of gas, which do not pollute a capillary and do not react with its surface.

In Russian Federation at present time the check gauge of the highest accuracy for test leak verification (calibration) is the State reference (operational) check gauge of the unit of gas flow in the vacuum in the range of 1,0\( \times \)10\(^{-12}\)…1,00 Pa\( \cdot \)m\(^3\)/s (GVET 49-2-2006). Total inaccuracy of gauge measurements is characterized by the sigma estimate of the result of measurements (\( \Sigma 0 \)): in the range of 1\( \times \)10\(^{-12}\) to 1\( \times \)10\(^{-9}\) Pa\( \cdot \)m\(^3\)/s not more than 0,1\( \times \)0,015; in the range of 1\( \times \)10\(^{-9}\) to 1 Pa\( \cdot \)m\(^3\)/s not more than 0,015. The check gauge has three measurement units with different operational principles – liquid-mechanical measurement unit with the range of measures 1\( \times \)10\(^{-7}\) to 1 Pa\( \cdot \)m\(^3\)/s; cumulative measurement unit with the range of measures 1\( \times \)10\(^{-9}\) to 1\( \times \)10\(^{-4}\) Pa\( \cdot \)m\(^3\)/s; reduction gear-box measurement unit with the range of measures 1\( \times \)10\(^{-12}\) to 1\( \times \)10\(^{-5}\) Pa\( \cdot \)m\(^3\)/s. The check gauge is kept and used in D.I. Mendeleev Institute for Metrology.

The main problem in the usage of test leaks is the difference of the temperature of the measured flow and the temperature of test leak itself. The necessity of temperature correction application in the flow rate produced by test leaks is caused by the fact that, as a rule, the temperature, at which test leak is operated, does not coincide with the temperature of its calibration. [2]

In the research [2] the temperature correction is proposed as follows:

\[
Q_T = Q_K \frac{T}{T_K} e^{-\frac{-\frac{E}{R T}}{1 + \frac{1}{T_K}}}
\]

where \( Q_T \) – flow produced by test leak at the temperature \( T \); \( Q_K \) – flow produced by test leak at the temperature of its verification (calibration) \( T_K \).
To specify corrections with the help of this formula it is necessary to identify the rate of activation for permeable elements of test leak (for example, quartz or molybdenum glass and fluoroplastic).

Currently manufacturers most often indicate average calculated value of temperature correction for specific test leak model. As a rule, the value of temperature correction is 3%/K. Manufacturers also indicate the flow rate in measuring units and the temperature of verification (calibration). To recalculate the flow the following formula should be used:

\[ Q_T = Q_K \left(1 + \frac{K_T}{100}\right)^{(T-T_K)} \]

where \( Q_T \) – flow produced by test leak at the temperature \( T \), \( mL/min \);
\( Q_K \) – flow produced by test leak at the temperature of its verification (calibration) \( T_K \), \( mL/min \);
\( K_T \) – temperature correction indicated by manufacturer;
\( T \) – operating temperature of test leak, K;
\( T_K \) – temperature of verification (calibration) of test leak, K.

However, the temperature can be disregarded if the flow rate is set as a molecular consumption, i.e. penetration of amount of substance (mole) per unit of time. In our country it is not common yet to delineate flow in measuring units mole/s, however, world vacuum community has taken a decision for gradual transition to measuring flow (especially for standard equipment) namely in mole/s measuring units, which allows to neutralize temperature dependence while measuring.

To measure precisely with the help of test leak it is necessary to use device for temperature stabilization (aerial or aquatic thermostat, heat shield etc.). It is experimentally proven in the article [2], that for diffusion test leak type Gelit 1 and Gelit 2 15 minutes exposure under the newly set temperature is enough for temperature stabilization of the measured (reproduced) flow.

The next important problem during the operation of the test leak is the impact of background flows. Background flows have significant impact on the accuracy and reliability of measuring results. These flows are caused by two main reasons – background flows of measuring system (of the leak detector, vacuum system itself and so on) and background flows, which come from connection pipe walls of the test leak. The first type of flows can be successfully dealt with, for example, through system heating, cleanup, prolonged unloading, and also “zeroing” of the background signal with the help of modern devices of leak testing.

The second type of background signal arises due to improper storage of test leak – long-time storage in the air with the opened connection pipe, the impact is even more in case of long time storage of the test leak with connection pipe closed by vacuum valve. In this case tracer gas is accumulated in permeable element and the flow rate for some indefinite time can significantly rise from 30% to 100% in comparison with assigned rate. Unfortunately, modern leak detectors as a rule produce rather low vacuum at the input, that does not allow promptly to pump out tracer gas accumulated in permeable element, which creates additional error when measuring by means of this test leak.

It is experimentally proven that the longer is the unloading of test leak connection pipe, the higher is the accuracy of measured flows and the lower is the measuring error.

Proceeding from facts mentioned above it is very important to comply with correct terms of test leak storage, and especially before verification (calibration), as improper storage can significantly raise high the flow rate for indefinite time, as a result the operator can get incorrect nominal rate of the flow.

Another serious problem is elaboration and serial domestic production of test leaks with an option to be filled with various gases (hydrogen, nitrogen, argon, Freon, etc.). These test leaks are required for calibration of sniffer leak detectors. It is also necessary to conduct state testing for the approval of the type for determination of metrological characteristics of test leaks data. For the current moment
there are no test leaks with the option of filling by another than He4 gas approved as SI in RF State Register.

In the year 2019 OJSC “Vaktron” on demand of D.I. Mendeleev Institute for Metrology elaborated and produced new capillary test leaks – helium (nitrogen) leak VAKTRON VKT with the option of filling both by helium and nitrogen.

![Picture 2. External view of VKT leak and the diagram of flow dependence on the pressure inside the leak tank](image)

The main advantage of new test leaks is an opportunity to regulate the flow by increasing or reducing test gas pressure inside the tank. Discharge of test leak can occur as to vacuum, so to the atmosphere. It is also worth mentioning about the simplicity of filling the tank with tracer gas individually. Preliminary tests confirmed characteristics indicated by manufacturer.

Timely metrological service and also compliance with operating and storage rules for test leaks in vacuum will allow to significantly enhance accuracy of measurements with the help of this equipment, and therefore improve the quality of produced items, reduce the probability to make wrong decision concerning tested objects.

**References**

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