Mass-spectrometric leak testing of big objects via test substance leakage monitoring

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Abstract. Mass spectrometer leak monitoring of big objects by means of the test substance leakage test is carried out on the special leak detection plants which include a test object, a leak detector or a partial pressure analyzer, check valves, vacuum meters, pumping equipment and calibrated leaks. The comparison of the flow of the test substance from the test object with the flow from calibrated leaks by means of a comparator allows detecting the leak rate and its measurement error. The article considers the characteristics of these plants and the ways how they can be determined. An example of such a plant intended for the leak detection of electronic equipment is considered.

1. Introduction

Leak monitoring of small objects (less than 5 liters) is carried out on mass-spectrometric leak detectors [1–3]. The characteristics of leak detectors are determined using a measuring chamber with the volume of 5 liters. It allows comparing the quality of leak detectors produced by various manufacturers. During leak monitoring of big objects, which usually have significant technological gas emission, the pumping system of a comparator (a leak detector or a partial pressure analyzer) cannot build up the right pressure needed for its work and an additional pumping system has to be used to reduce pressure [4, 5]. The test substance used for leak monitoring is evacuated not only by the comparator but also by the vacuum system in this case. Increasing technological gas emission worsens the minimum working pressure and the threshold of sensitivity of the control. Increasing the pumping speed of the object using the additional vacuum system during testing enhances the minimum and maximum working pressure but it worsens the testing sensitivity. Using pumps with selective pumping out in the additional vacuum system may increase the threshold of sensitivity of the control. The current standard for determining the characteristics of mass-spectrometric control was enforced quite a while ago and needs to be revised. This paper provides some proposals on how it can be improved.

2. The design of the leak detection plant

The vacuum leak detection plant by means of the leakage test (figure 1) consists of a mass-spectrometric comparator (a leak detector or a partial pressure analyzer), a vacuum chamber, vacuum meters, vacuum pumps, valves, and connecting pipelines. When detecting the characteristics of the
measuring vacuum chamber must be leak-proof within the limits of sensitivity of the mass-spectrometric comparator to be used. The measuring vacuum chamber may include the test object.

**Figure 1.** Scheme of vacuum leak detection plant by means of the leakage test of the test substance:
1 – calibrated leak; 2 – valve; 3 – measuring vacuum chamber; 4 – transmitter for measuring of low pressure; 5 – convector for measuring high pressure; 6 – leak valve; 7 – comparator; 8 – valve; 9 – valve, 10 – additional vacuum system; 11 – vacuum pump; 12 – valve.

The possibilities of the leak detection plant are determined by the following characteristics: the sensitivity to the flow; the minimum flow of the test substance (the threshold of sensitivity to the flow); the maximum flow of the test substance; the response time; the minimum working pressure in the test substance; the maximum working pressure in the test object.

3. Determining the characteristics of the leak detection plant

3.1. Determining the sensitivity of the test substance to flow

In the initial state of the vacuum system (figure 1) all valves are closed. Calibrated leak 1 is set. The value of calibrated leak 1 should be chosen with the value close to the rejection criteria \( Q_n \) of the test object. Valve 12 gets open and within no less than 15 minutes the inner cavity of the pipeline between calibrated leak 1 and valve 2 is pumped out by means of vacuum pump 11 to the residual pressure of maximum \( 10^{-1} \) Pa. Vacuum chamber 3 is pumped out to the minimum residual pressure by means of additional vacuum system 10 through valve 9. The value of background \( U_b \) is determined by means of the stable value of comparator 7. Valve 2 opens and the control object is pumped out from calibrated leak 1 into vacuum chamber 3, and the value of signal \( U_c \) is determined according to stable value 7.

Sensitivity \( K \) \( Y/A \) is calculated by the formula

\[
K = \frac{U_c - U_b}{Q_d} ,
\]

where \( U_c \) is the output signal with the calibrated leak being connected, \( Y \) \( Y \) is the arbitrary unit that is proportional to ion current of the comparator; \( U_b \) is the background signal of the comparator connected to the vacuum chamber when the calibrated leak is disconnected, \( Y \); \( Q_d \) is the flow of the test substance from the calibrated leak at a test temperature, \( A:A \) is the unit of measurement of flow (the following units are recommended: kmol/s, m\(^3\)/Pa/s at 298 K, ncm\(^3\)/s).

The value of leak \( Q \) in the test object is calculated by the formula

\[
Q = \frac{U}{K} ,
\]

where \( U \) is the output signal of the comparator caused by the leak in the test object, \( Y \);

3.2. Determining the minimum flow of the test substance

The change of an approximated background signal (calculated by means of the least-square method) relative to 10 minutes is taken as the value of drift \( U_d \). If there is rubber or other polymeric material in
valve 2 or at the point of the connection of leak, the value of the background signal of the vacuum system is determined without the calibrated leak \( I \) when the plug is set at its place and the valve 2 opens. If there is no rubber or other polymeric material, the value of the background signal is determined when there is calibrated leak and valve 2 is closed.

The square root of the dispersion of the background signal measured 10 times every 60 seconds is taken as the value of noise \( U_n \). A single overshoot of the background signal registered by the comparator while measuring is ignored. If repeated overshoots occur, the conducted measurements are stopped and new measurements are carried out.

The minimum registered signal \( U_{\text{min}} \), \( Y \), is calculated by the formula

\[
U_{\text{min}} = 2(U_n + U_d),
\]

where \( U_n \) is the noise of the signal, \( Y \); \( U_d \) is the drift of the signal, \( Y \).

The minimum flow of the test substance \( Q_{\text{min}} \) \( A \) is calculated by the formula

\[
Q_{\text{min}} = \frac{U_{\text{min}}}{K},
\]

3.3. Determining the maximum flow of the test substance

The test substance is supplied to the input of leak valve 6. Leak valve 6 must have the micrometrical graduation of regulated conductivity. A set of calibrated leaks \( I \) (minimum three) equally located across the entire measurement range of flows may be used instead of leak valve 6. The dependence of sensitivity on the value of flow of the test substance through the leak valve is calculated by the formula (1). According to the type of the dependence, the deviation from the linearity of the output signal of the comparator is determined given on the value of the flow of the control object. The maximum flow is taken as the value at which sensitivity decreases from its value at the rejection criterion by the magnitude of error of the measurement of flow.

3.4. Determining the response time

When valve 2 is closed, calibrated leak \( I \) is connected to vacuum chamber 3. Valve \( I2 \) is opened and within no less than 15 minutes the inner cavity of pipeline between calibrated leak \( I \) and valve 2 is pumped out by means of vacuum pump \( I1 \) to reach the residual pressure of no more than \( 10^{-4} \text{Pa} \). When the preset pressure is reached, the pumping is stopped and valve \( I2 \) is closed.

![Leakage curve](image)

**Figure 2. Leakage curve.**

Vacuum chamber 3 is evacuated to the ultimate pressure through valve 9 by additional vacuum system \( I0 \) when valves 2 and 8 are opened. Valve 2 is closed. Valve 2 gets opened when output signal \( 7 \) is set. The leakage curve (i.e. the dependence of the output signal of the comparator on time) is determined (fig. 2). The time of output signal of comparator \( Y_n \) is recorded and is taken as the time of leak tracing \( t_r \). Time constant \( \tau \) (figure 2), corresponding to the increase of the output signal by 63 \% of the maximum steady state value, is taken as the response time in the measurement mode of leak.

3.5. Determining the minimal working pressure

Vacuum chamber 3 is evacuated by means of additional vacuum system \( I0 \) through valve 9 to the minimum residual pressure, with valve 9 being closed. During leak monitoring the steady pressure is
taken as the minimum working pressure. For small vacuum chambers evacuating may be carried out by means of built-in facilities of comparator 7 instead of additional vacuum system 10.

3.6. Determining the maximum working pressure
Pumping dry air into vacuum chamber 3 by means of leak valve 6, different values of the air pressure are estimated in the vacuum chamber (at least three values for each pressure range). For each pressure, the threshold response to the flow \( Q_{\text{min}} \) is calculated by the formula (3) and the dependence of the threshold sensitivity on the pressure is estimated. The value of pressure at which the threshold sensitivity becomes smaller than the rejection criterion is obtained. This pressure is taken as the maximum working pressure.

4. Metrological characteristics
The uncertainty of the sensitivity \( K \) (1) is calculated by the following formula

\[
\delta_{\text{rel}}(K) = \sqrt{\delta_{\text{rel}}^2(Q_{\text{c,m}}) + \delta_{\text{rel}}^2(U_{\text{m}})}
\]

where \( \delta_{\text{rel}}(K) \) is the relative standard deviation of the sensitivity \( K \); \( \delta_{\text{rel}}(Q_{\text{c,m}}) \) is the relative standard deviation of the value of the calibrated leak \( Q_{\text{c,m}} \); \( \delta_{\text{rel}}(U_{\text{m}}) \) is the relative standard deviation of the value of nonlinearity for \( U \) measurement. The relative standard deviation of leak measurement results, which are considerably higher than the threshold sensitivity, is calculated by the formula

\[
\delta_{\text{rel}}(Q) = \sqrt{\delta_{\text{rel}}^2(K) + \delta_{\text{rel}}^2(U_{\text{m}})},
\]

where \( \delta_{\text{rel}}(K) \) is the relative standard deviation of the sensitivity \( K \); \( \delta_{\text{rel}}(U_{\text{m}}) \) is the relative standard deviation of the value of the nonlinearity for \( U \) measurement.

The total relative deviation of the leak measurement value in the test object with allowance for the deviation occurring while leakage measuring close to \( Q_{\text{min}} \), is calculated in % as follows:

\[
\delta_{\text{rel}} = (\delta_{\text{rel}}(Q) + \frac{Q_{\text{min}}}{Q}) 100.
\]

The value of \( Q \) in (7) may set equal to the rejection criterion \( Q_{\text{rc}} \).

5. Leak detection plant for electronic equipment
The plant (figure 3) is designed according to the scheme (figure 1), it has the working chamber with the volume of 55 liters, and the quadrupole mass-spectrometer XT300-M is used as the comparator. Evacuating the chamber is carried out by means of the turbomolecular pump nEXT300 and the scroll pump nXDS35i. The electronic equipment tested for leak detection is filled with helium. It may be set both outside and inside the vacuum chamber.

![Figure 3. Picture of the leak detection plant.](image)

The rejection sign \( Q_{\text{rc}} \) was equal to \( 10^{-9} \text{m}^3/\text{Pa}\text{s} \). The calibrated leak was determined \( Q_{\text{cal}} = 3.68 \times 10^{-10} \text{m}^3/\text{Pa}\text{s} \). The background pressure of helium \( U_b \) amounted to \( 9 \times 10^7 \text{Pa} \). The output signal of the analyzer \( U_c \) was equal \( 10^7 \text{Pa} \). After accumulation within 90 seconds the output signal was equal to \( 10^6 \text{Pa} \). According to the formula (1), the sensitivity \( K \) without accumulation was equal to \( 247 \text{ Pa}/(\text{m}^3/\text{Pa}\text{s}) \) and it was equal to \( 2692 \text{ Pa}/(\text{m}^3/\text{Pa}\text{s}) \) with accumulation.
The minimum registered signal \( U_{\text{min}} \) is equal to \( 1.8 \times 10^{-8} \) Pa. Using the formula (4) we obtain the threshold sensitivity without accumulation \( Q_{\text{min}} \), which is equal to \( 7.3 \times 10^{-11} \) m\(^3\)/Pa/s and it is equal to \( 6.7 \times 10^{-12} \) m\(^3\)/Pa/s with accumulation \( Q_{\text{min}} \).

The maximum measured helium flow \( Q_{\text{max}} \) is equal to \( 3 \times 10^{-2} \) m\(^3\)/Pa/s. The nonlinearity of measurement is equal to \( 10^{-11} \) Pa. The response time is 5 seconds.

The relative standard deviation of the value of the calibrated leak is equal to 20%. The relative standard deviation of the value of the nonlinearity of measurement is equal to 2.7%. The relative standard deviation of the value of the measured flow of leak \( Q = Q_r \) according to the formula (12) is equal to 21%.

6. Conclusion
The authors considered proposals on some improvement of techniques aimed at determining characteristics of mass-spectrometric leak monitoring for big objects via test substance leakage method. The present paper describes the structure and design of leak detection plants, which include a tested object, a leak detector or a partial pressure analyzer, check valves, vacuum meters, pumping equipment and calibrated leaks. The plants are exemplified by the one intended for leak detection of electronic equipment with the relative deviation of flow leak testing equal to the rejection criterion of 21%.

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