Features of technique for leak test of gas-filled dischargers

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Abstract. The paper gives an overview of the issues related to the technique for leak test of miniature gas-filled dischargers developed and manufactured by VNIIA.

Creating and maintaining tightness in vacuum and gas-filled devices is one of the main conditions for maintaining operational parameters that determine their operability and guaranteed shelf life. The high requirements for the standard of leak tightness \(1.4 \times 10^{-13} \text{ Pa} \cdot \text{m}^3/\text{s}\) of one of the lines of gas-filled devices produced by VNIIA led to the need to develop a special measuring procedure and to build an automated prototype of a vacuum machine for the final leak test (MFLT) to implement this procedure [1]. The technique for estimating the degree of leakage of dischargers is based on the mass spectrometric method, which is used as the basis for applying the accumulation in the development.

The partial pressure of the working gas on the MFLT is measured with QMG 220 PrismaPlus M2 quadrupole mass spectrometer. Under low pressure, its ion source can operate for a relatively long period of time without the need for any intervention in terms of routine maintenance. Nevertheless, after four years of operation of the mass spectrometer (~10 000 h), signs began to show indicating that the failure of the filament was a matter of just a few months.

Negative trends in the work of the mass spectrometer were not immediately apparent, but appeared gradually; typically after the discharge (transfer) of the accumulated portion, the dynamics of the change in the controlled response of the intensity at a given mass number deviated from the typical relation. This subsequently manifested in the form of significant distortions of the “zero” line for practically the entire group of recorded mass numbers from 1 to 45 (see figure 1). Previously almost absent, i.e. not typical, changes of pressure over time after the transfer of a gas portion in the form of a so-called “potential well” with a level of the trough extending to values below the background ones, began to be observed from one discharge to another, regardless of the accumulation time. As a result of indirect measurements, a minus sign appeared in the value of the measured leakage flux \(Q_i\).

So that the received mass-spectrometric information, which has undergone non-typical changes, was not lost and was not useless given no opportunity to operatively replace the cathode, it became necessary to try to solve this problem in the future, since the probability of its repetition could not potentially be excluded during operation in future. The appearance of the above-mentioned problem required additional studies, mathematical modeling, and debugging certain points of the measurement technique. A mathematical model was developed in the form of equation that approximately describes the process of the gas portion transfer and pressure change during its expansion in the volume of the measuring system (in current terms), using elements of regression analysis.
The function of the output current signal of the mass analyzer $I = f(t)$ can be represented as the following equation:

$$I = \Delta I_0 \left[ 1 - \exp\left( -\frac{S_{ef} t_{[i]}}{V_z + V_{ch}} \right) \right] \exp\left( -\frac{S_{ef} t_{[i]}}{V_z + V_{ch}} \right) + \frac{Q S_{f[i]}}{V_z + V_{ch}},$$

(1)

where $\Delta I_0$ – the increment of ion current of 20 M/e, [A]; $S_{ef}$ – “pseudo pumping” rate of 20 M/e ($S_{ef} = \text{Varia}$), [m$^3$/s]; $Q$ – flow of leakage from device [Pa·m$^3$/s]; $S$ – coefficient of absolute sensitivity of mass spectrometer, [A/Pa]; $V_z$ – total volume of measuring system, [m$^3$]; $V_{ch}$ – volume of accumulation chamber, [m$^3$]; $t_{[i]}$ – interval (1st) of time (1.7...2 s), determines pressure equalization time in two volumes ($V_z$ and $V_{ch}$) after gas portion is transferred, [s]; $t_{[i]}$ – current time from transfer to end of recording during measurements (before opening of the turbomolecular pump valve), [s].

**Figure 1.** Dynamics of changes in mass numbers during leak test (two gas portions were consistently “dumped”) with characteristic distortions – “potential wells”.

It should be considered that during the first seconds of the discharge of gas portion, the surge in pressure is dominant due to the lack of complete isotropy (uniformity of distribution) of pressure when it is redistributed in the volume and due to the tribological factor (friction of the valve-saddle surfaces). The process of “pseudo-pumping”, described by the expression

$$\Delta I_0 \left[ 1 - \exp\left( -\frac{S_{ef} t_{[i]}}{V_z + V_{ch}} \right) \right] \exp\left( -\frac{S_{ef} t_{[i]}}{V_z + V_{ch}} \right),$$

and the linear function (gas flow from the leak), described by the expression, also take place (due to the continuity of the flow), but their influence during the first few seconds is relatively small. It should be specially noted that the last expression most accurately describes the process of increasing the partial pressure of the working gas in the system due to the flow through the leak, and the desired value of the estimate of the leakage flow, $Q$, is the more accurate the closer to the “tail” part the sample is taken from the points used to determine it.
The principal nature of the studied function is known in advance based on the construction of the physical model, therefore, it is required to obtain only some numerical parameters that are part of the expression of the function. These include the flow, \( Q \), and \( S_{\text{ef}} \) and \( \Delta I_0 \) parameters, which are auxiliary and do not require high accuracy; the acceptable format of the result is “X.X”.

The problem of determining the required parameters \((Q, S_{\text{ef}}, \Delta I_0)\) can be tried to solve in different ways, for example, using the nonlinear least-squares method through construction of a system of normal equations. However, in order to avoid the “inconvenience” in solving the smoothing problem using (1), in which some of the parameters are nonlinear, the problem can be solved by setting a number of initial values of \( Q, S_{\text{ef}}, \Delta I_0 \) parameters [2]. For each of them, the sum of the squared deviations of \( Y_k \) function from the initial measured values \( I_k \) with the search for the minimal discrepancy \(|Y_k - I_k|^2\) is found.

Since for estimating the flow \( Q \) based on (1), the value of the background \( Q_b \) (determined from the change in the current signal of the analyzer during the time interval \( tt_1, tt_2 \), see figure 2) is not included explicitly in the variables involved in the calculation of the leakage flux, the value of the background flux is necessary for the final computations of the estimate of the degree of leakage \( Q' \), determined from the expression:

\[
Q' = (Q - Q_b), \text{ [Pa} \cdot \text{m}^{3/\text{s}}].
\]

The value of \( Q_b \) is calculated from the known equation of the following form:

\[
Q_b = \frac{\Delta I_0 V_t}{\tau}, \text{ [Pa} \cdot \text{m}^{3/\text{s}}],
\]

where (with other equal parameters, previously given), \( \Delta I_0 \) – difference between signals of mass spectrometer \( I_{n2} \) and \( I_{n1} \) at points of time \( tt_1 \) and \( tt_2 \), [A]; \( \tau \) – accumulation time for background \((tt_2 - tt_1)\), [s]. The results obtained in this case can have a “negative” value, but not by current increment, but already by the estimation of leakage flow \( Q' \).

Because of the peculiarities in the operation of the ion source of the mass spectrometer, as well as the “contamination” with water vapor, as previously noted, the pumping process is delayed (3 to 8 h). In view of this, it was necessary to provide another way of obtaining useful information, similar to the case when the background characteristic \( Q_b \) exceeds the permissible values \((Q')\).

As practice has shown, the most effective way to “not lose” the information received was the process of combining the operations of accumulation and measurement, i.e. the supply of test gas from the monitored device is not limited to the volume of the accumulation chamber. The gas, flowing “directly” through the leak with the open valves of the accumulation chambers ensuring the integration of the test object and the measuring volume, accumulates in the volume of the measuring system from the moment of shutting down the turbomolecular pump valve \((tt_1)\) for a predetermined time interval. At the same time, the nature of the current function does not change, and there are no manifestations of “distortion” that preceded the transfer of the accumulated portion to the volume of the measuring system. In this way, the operation for accumulating and transferring a portion of gas from the storage chamber is replaced by accumulation with registration in the combined volume, including the vacuum cavity of the measuring system and the storage cavity.

The leakage flow of the monitored device(s) \((n = 1...7)\) is determined by equation (4)

\[
Q = \frac{\Delta I(V_{n4} + nV_{ah})}{\tau} - Q_b, \quad (4)
\]

where \([\text{the terms of the equation are the same as the parameters in (3)}]\) \( \Delta I \) – difference of \( I_{n4} \) and \( I_{n3} \) signals at time \( tt_4 \) and \( tt_3 \) (more precisely, similar to \( tt_2 \) and \( tt_3 \)), since for this case, there will be only two time counts), [A]; \( \tau \) – accumulation time for background \((tt_4 - tt_3)\), [s].

Thus, in addition to the implemented software algorithm of data processing (“positive”, “zero” current increment), two more are added for the case with a “negative” increment:

1. Using (1) according to the above algorithm (without repeating the operation associated with the transfer of the accumulated portion, i.e. only resorting to analytical calculations);
2. Using (3) with additional procedures for separate registration of the background of the measuring system and the total leakage flow from the device along with the background of the system through accumulation in the combined volume.

![Graph-scan characterizing measuring process during implementation of “Express Analysis” project.](image)

**Figure 2.** Graph-scan characterizing measuring process during implementation of “Express Analysis” project.

In figure 2: \( t_1 \) – start time of sample background record; \( t_2 \) – end time of sample background record; \( t_3 \) – start time of sample measurement; \( t_4 \) – end time of sample measurement; \( t_{t1} \) – start time of background record of group of valves of accumulation chambers (or one); \( t_{t2} \) – end time of background record of group of valves of accumulation chambers (or one); \( t_{t3} \) – start time of measurement of group of valves in accumulation chambers (or one); \( t_{t4} \) – end time of measurement of group of valves of accumulation chambers (or one).

However, here it should be noted that the option using additional mathematical processing at the final stage of development of special software was still rejected because of its comparative complexity. Therefore, the method used and implemented by the software algorithm was the combination of volumes.

Speaking about the methodological improvements and their usefulness, it is obvious, however, that the further steps taken by the tester due to the emerging uncertainty regarding the time of further operation of the cathode should be aimed at replacing the cathode (cathode node) of the mass spectrometer in order not to aggravate the situation with measurements, since the accuracy of the received data will be low in future.

Thus, it is necessary to take into account the operational characteristics of the mass spectrometer during operation and it is a forced procedure for the operator controlling the leak testing process. Undoubtedly, the mentioned difficulties require certain skills of the operators when operating this analytical equipment, despite the availability of automation. Nevertheless, in case of cathode-related events close to failure, the technique for final leak test of the dischargers, as a separate option, there is
a possibility implemented by software to avoid “loss” of data obtained with hardware “distortions” by using an additional method of obtaining useful information, despite the above-mentioned problems.

References
[1] Bushin S A 2012 Vacuum Technique and Technology 2 98–9
[2] Wentzel E S 1964 Theory of Probability (Moscow: Science)