HIGH-PRECISION EQUIPMENT COMPLEX FOR THE
CERTIFICATION AND CHECKING OF DISPENSERS

E. A. Khatskevich

We consider a high-precision equipment complex for the certification and checking of the dispensers of gas
analysis instruments, for example, those of gas chromatographs and chromatographic analyzers. The dispensers
are used for the withdrawal, measuring off, and delivery of a given volume of the dispensed material, in
gaseous, vapor, or liquid state.

Before 1988, there was in force, in our country, a standard [1], which was revoked in July 1988, and replaced by a
set of technical conditions. Nevertheless, in these documents, depending on the standard of accuracy in measuring the batch
volume, this being expressed, for example, by the relative mean square deviation (MSD), characterizing the random error
component, and the maximal deviation from the nominal value of an actual batch volume, all dispensers are classified as
standard, working, or technical. The most accurate of these, the reference standard dispensers, must be measured with a relative
random error not exceeding 0.6% for nonthermostatic gas dispensers, not more than 2.0% for nonthermostatic liquid
dispensers, and not more than 4.0% and 1.5%, respectively, for thermostatic gas and liquid dispensers.

These normative documents do, however, have the following shortcomings:

the relative error of a batch volume measurement is interpreted without taking the systematic error component into
account;

the random error component, characterized by the MSD value, is standardized for a very narrow macrobatch volume
range (0.1-1.0 cm$^3$);

it is not indicated how many concurrent observations were made, and for which confidence level the relative MSD
values standardized in these documents are established;

The first work on establishing the adsorption-weight method of measuring the batch volume of dispensers was carried
out at VNIIM in 1975 [2], and a device was proposed in the following year for implementing this method [3].

The problems associated with the measurement of batch volumes and the certification of dispensers were also studied
by other organizations. Thus, in 1980, a method was developed at VNIIMS for the certification of microdispenser batch
volumes [4]. Two methods of measuring the actual microbatch volumes were considered: the adsorption-weight [2], and gas
chromatographic.

Deficiencies in the method of [4] are the omission of accuracy standards in the measurement of the batch volume and
the rather limited spread of values — the measurement of actual microdispenser batch volumes is in the range from $0.5 \cdot 10^{-3}$
to $20 \cdot 0.10^{-3}$ cm$^3$. Furthermore, in implementing the adsorption-weight procedure in this method, the mass of the adsorber
is weighed on one arm, which introduces additional unforeseen systematic errors, associated with aerostatic forces, from the
volume difference of the two bodies balanced against each other i.e., the adsorber volume on one side, and balancing weights,
on the other [5].

In 1986, a method was devised at the VNII of Chromatography (VNIIKhROM) for the certification of working
microdispensers, with batch volumes from $0.2 \cdot 10^{-3}$ to $70.0 \cdot 10^{-3}$ cm$^3$. The gas chromatographic method proposed is based
on using a gas chromatograph for comparing the chromatograph signal on introducing a test batch of unknown (variable)
volume, by the working or technical dispenser to be standardized.

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TABLE 1

<table>
<thead>
<tr>
<th>Number and type of VNIIM dispenser</th>
<th>Batch number</th>
<th>Actual value of batch volume, cm$^3$</th>
<th>Absolute measurement error, cm$^3$</th>
<th>Relative measurement error, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>BK 'S'</td>
<td>1</td>
<td>0.2762</td>
<td>±0.0005</td>
<td>±0.18</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.5770</td>
<td>±0.0007</td>
<td>±0.12</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1.085</td>
<td>±0.002</td>
<td>±0.19</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>2.569</td>
<td>±0.004</td>
<td>±0.16</td>
</tr>
<tr>
<td>MK No. 9</td>
<td>2</td>
<td>2.393·10$^{-1}$</td>
<td>±0.014·10$^{-3}$</td>
<td>±0.6</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>7.836·10$^{-2}$</td>
<td>±0.043·10$^{-2}$</td>
<td>±0.6</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>16.639·10$^{-2}$</td>
<td>±0.043·10$^{-3}$</td>
<td>±0.3</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>64.91·10$^{-3}$</td>
<td>±0.16·10$^{-3}$</td>
<td>±0.3</td>
</tr>
</tbody>
</table>

TABLE 2

<table>
<thead>
<tr>
<th>Number and type of dispenser</th>
<th>Nominal batch volume, cm$^3$</th>
<th>Actual batch volume, cm$^3$</th>
<th>Absolute measurement error, cm$^3$</th>
<th>Relative measurement error, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>No. 1 (NPO Khimavtomatika, Dzerzhinsk)</td>
<td>0.5</td>
<td>0.350</td>
<td>±0.0013</td>
<td>±0.25</td>
</tr>
<tr>
<td>No. 1 D-46 (VNIKA-NEFTEGAZ, Moscow)</td>
<td>0.8·10$^{-2}$</td>
<td>0.825·10$^{-2}$</td>
<td>±0.008·10$^{-1}$</td>
<td>±1.0</td>
</tr>
</tbody>
</table>

TABLE 3

<table>
<thead>
<tr>
<th>Number and type of VNIIM dispenser</th>
<th>Date of measurement</th>
<th>Batch number</th>
<th>Type of gas chromatograph</th>
<th>Actual batch volume, cm$^3$</th>
<th>Relative error, %</th>
</tr>
</thead>
</table>
| No 1                               | June 1984           | 1            | Model 5730 (Hewlett Packard, USA) | 0.936·10$^{-3}$  
|                                   |                     | 3            |                           | 2.475·10$^{-3}$  
|                                   |                     | 5            |                           | 4.78·10$^{-3}$   
|                                   |                     | 7            |                           | 10.94·10$^{-3}$  |
|                                   | June 1989           | 1            | Model Tsvet-530 (NPO Khimavtomatika, Dzerzhinsk) | 0.942·10$^{-3}$  
|                                   |                     | 3            |                           | 2.470·10$^{-3}$  
|                                   |                     | 5            |                           | 4.75·10$^{-3}$   
|                                   |                     | 7            |                           | 10.96·10$^{-3}$  |

The overall relative error standardized in this method is ±3.2%, which is 1.6 times larger than the value of the relative error standardized for the gas chromatographic measuring device incorporated in the high-precision equipment of the D. I. Mendeleev VNIIM Scientific Production Department [6].

Specialists at VNIKhRROM have developed a method for the certification of dispensers with changeable batches in the volume range from 0.125 to 2.0 cm$^3$. The overall relative error in volume measurement standardized in this method is 3.7%, which is approximately seven times larger than the error of the gas chromatographic measuring device incorporated in the high precision VNIIM equipment for the certification and checking of dispensers [7].

The UGD-3 high-precision equipment was certified in 1991 (UVT78-A-92). It was designed for reproducing the unit of the volume being dispensed. At the highest precision in the country, measurements can be made of the batch volumes of gas and liquid dispensers having batch volumes in the range 0.1·10$^{-3}$ to 5.0 cm$^3$. The equipment has been used for the certification and checking of reference standard dispensers. Detailed information of the constructional features and the results of measuring actual batch volumes with this equipment are given in [8].

For the helium delivery in the sorbent regeneration unit which is a constituent part of the UGD-3, a novel device, described in detail in [9], is used for admitting the gases.