LIQUEFIED SAMPLING VALVE

USER MANUAL

214.4.464.017UM

Edition 21.11.2024

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1 Introduction

This User Manual (UM) is intended to familiarize you with the liquefied sampling valve (hereinafter referred to as the LSV) and ensure its proper operation.

Before commissioning the LSV, you should carefully read the contents of this UM and the operating documentation for the chromatograph (bundle) with which you intend to use it.

When analyzing liquefied gases (using samplers and needle valves), you should also learn the operating documentation for these products.

The relevant sections of the UM contain instructions that must be followed when operating and servicing the LSV.

Instructions marked with this box must be followed to avoid injury when operating the chromatograph or damage to the equipment.

2 Description and operation

2.1 Purpose

The Liquefied sampling valve (hereinafter, the LSV or the valve) is designed for manual sampling and injecting liquid and gas samples under excess pressure of up to 3 MPa into the inlet of gas chromatographs of the Chromatec-Crystal series (hereinafter, the GC).

Depending on the internal volume of the sample, the LSV is available in the following design versions:

- 214.4.464.017-05 – 1.0 µL;
- 214.4.464.017-06 – 0.25 µL;
- 214.4.464.017-07 – 0.1 µL.

The corresponding symbol ("4.464.017-05", "4.464.017-06" or "4.464.017-07") is applied to the body of the LSV, and the internal volume of the sample is applied to the cover (Figure 1).

As for climate performance, the LSV is referred to cold-temperate category 4.2 according to GOST 15150-69. The operating conditions of the LSV comply with the requirements of GOST 26703-93.

2.2 Technical specifications

Technical specifications and delivery set is according to Passport of the LSV 214.4.464.017PS.

2.3 Construction and operation

The appearance of the LSV is shown in Figure 1 (the protective cover of the needle is removed).

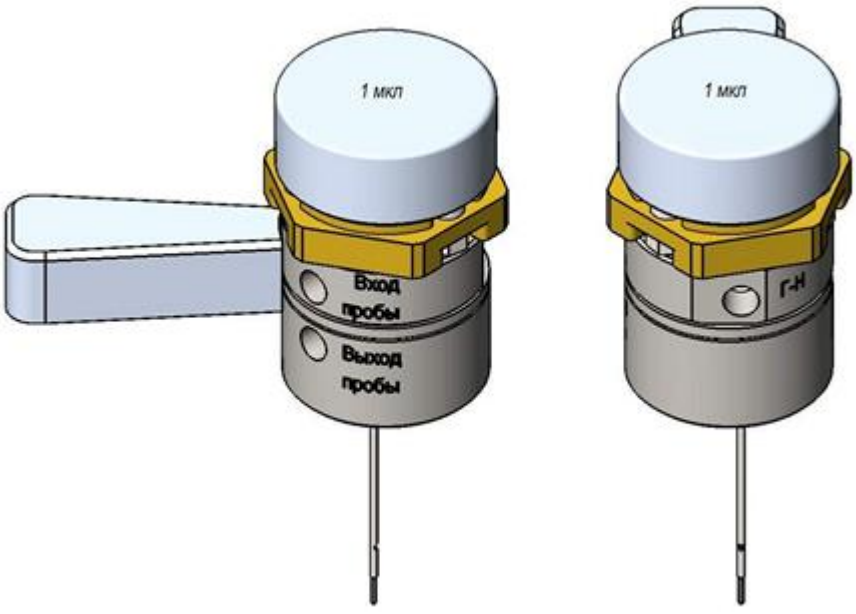
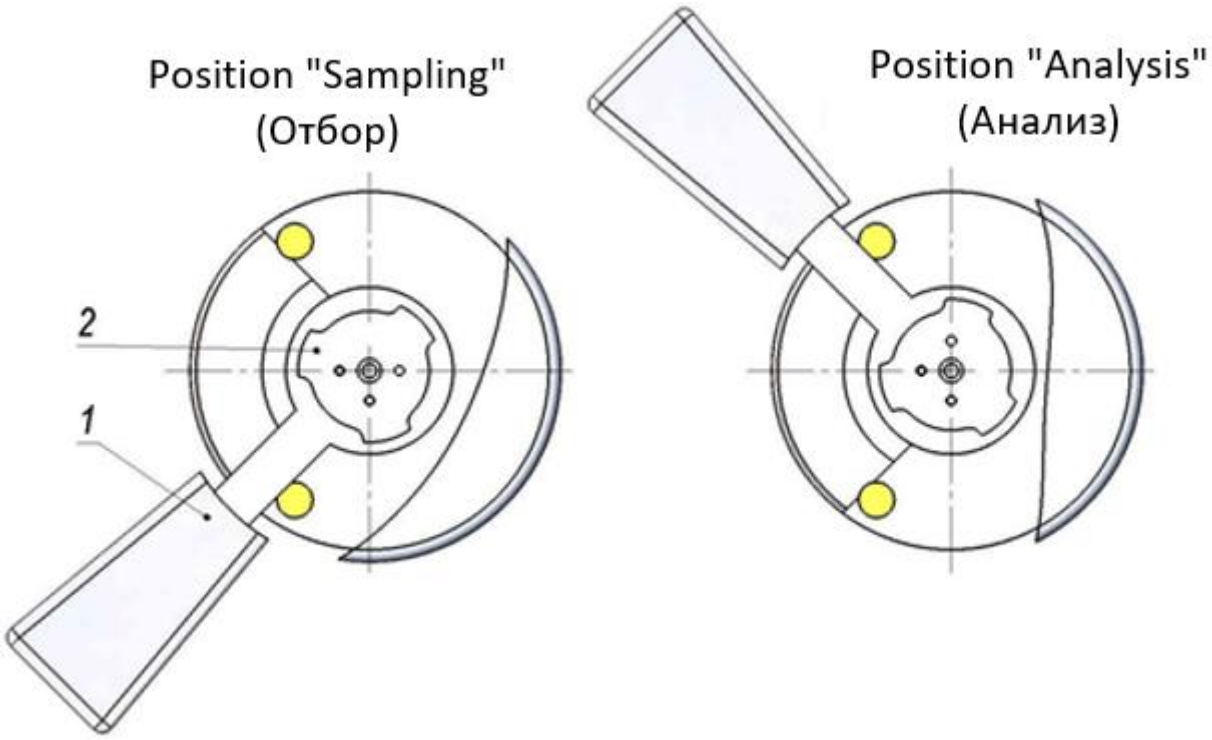


Figure 1 – Liquefied sampling valve. Overview

The body of the LSV has markings for the sample inlet and outlet fittings and for connecting the carrier gas. The cap has markings for the sample internal volume.

The sample internal volume of the LSV is the hole in disk 2. Changing the positions of the LSV ("Sampling" or "Analysis") is done by moving the arm 1 by its handle (see Fig. 2).

When changing the position of the LSV, the arm should be moved by its handle until it stops.



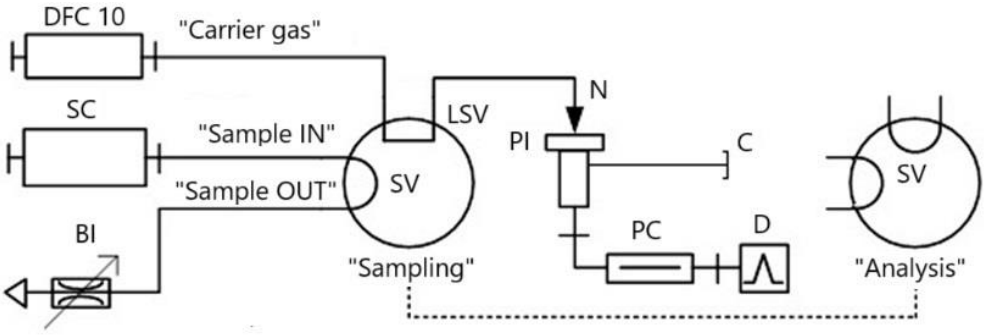
1 – the arm with a handle; 2 – disc with internal sample volume

Figure 2 – Liquefied sampling valve. Position "Sampling" – "Analysis"

In position "Sampling" ("Отбор") the internal volume of the valve is filled with a sample. In the "Analysis" ("Анализ") position, the internal volume with a portion of sample is included in the carrier gas line, while flow in the sampling line is not interrupted.

The signs "Sampling" ("Отбор") and "Analysis" ("Анализ") are engraved on the valve body.

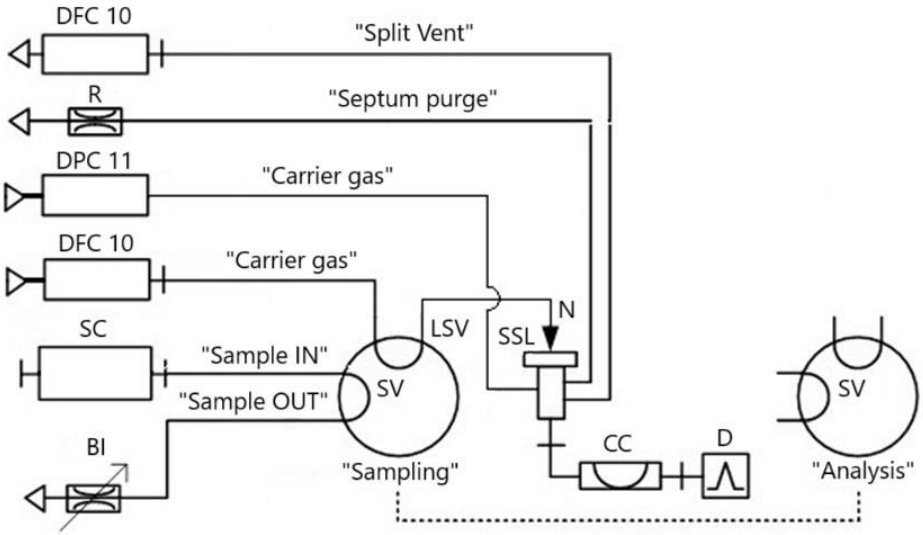
The flow diagram of the LSV when working with packed columns is shown in Figure 3.



D – Detector; N – needle; PI – packed inlet; C – cap 8.632.037; LSV – liquefied sampling valve; SV – sample volume; PC – packed column; SC – sampling cylinder (PU, PGO, PP type); DFC10 – digital flow controller; BI – bubble indicator 5.183.011 (with needle valve).

Figure 3 – Flow diagram of the LSV when working with packed columns

The flow diagram of the LSV when working with capillary column in split mode is shown in Figure 4.



D – Detector; N – needle; SSL – split splitless inlet; LSV – liquefied sampling valve; SV – sample volume; CC – capillary column; SC – sampling cylinder (PU, PGO, PP type); BI – bubble indicator 5.183.011 (with needle valve); DFC10 – digital flow controller; DPC11 – digital pressure controller.

Figure 4 – Flow diagram of the LSV when working with capillary column in split mode

3 Usage

3.1 Safety measures

Safety measures when working with the LSV are similar to those given in the UM for the GC.

Only persons who have studied the UM for the GC, this manual, the UM for the corresponding sampling cylinder, GOST 14921-78 "Liquefied hydrocarbon gases. Methods of sampling" (in terms of working with samplers), and who have passed the test of work skills at the workplace are allowed to conduct analyses using the LSV.

When working with flammable, harmful and aggressive gases, fire safety measures must be observed according to GOST 12.1.004-91, as well as measures provided for in special instructions developed by the consumer (in accordance with the specifics of the substances used) based on GOST 12.1.007-76

3.2 Preparation for operation and operation procedures

3.2.1 Protective casing installation

The LSV is supplied with a needle protected by a casing (see Figure 5). Before using the LSV, the protective casing is removed.

The needle installed in the LSV, in order to ensure safety and prevent damage during transportation or storage, is protected by a casing in accordance with Figure 5.

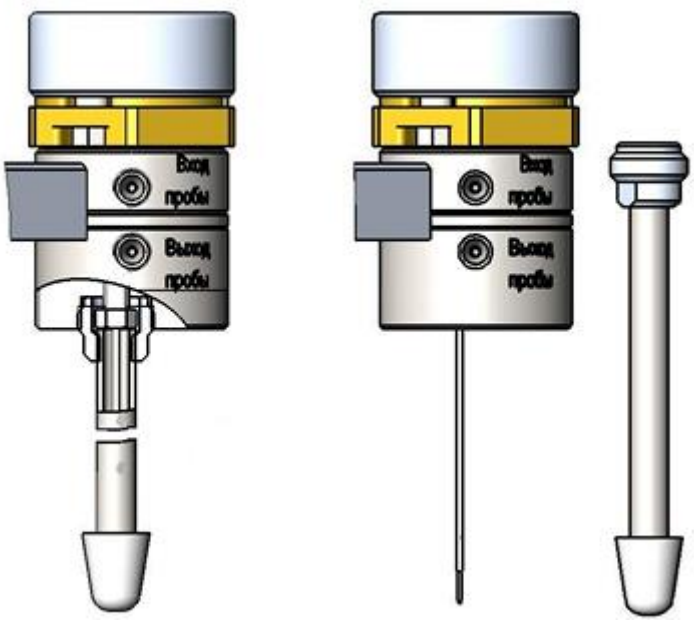


Figure 5 – Installation of protective casing of the needle

The protective cover should be screwed into the valve body until it stops, using force "by hand".

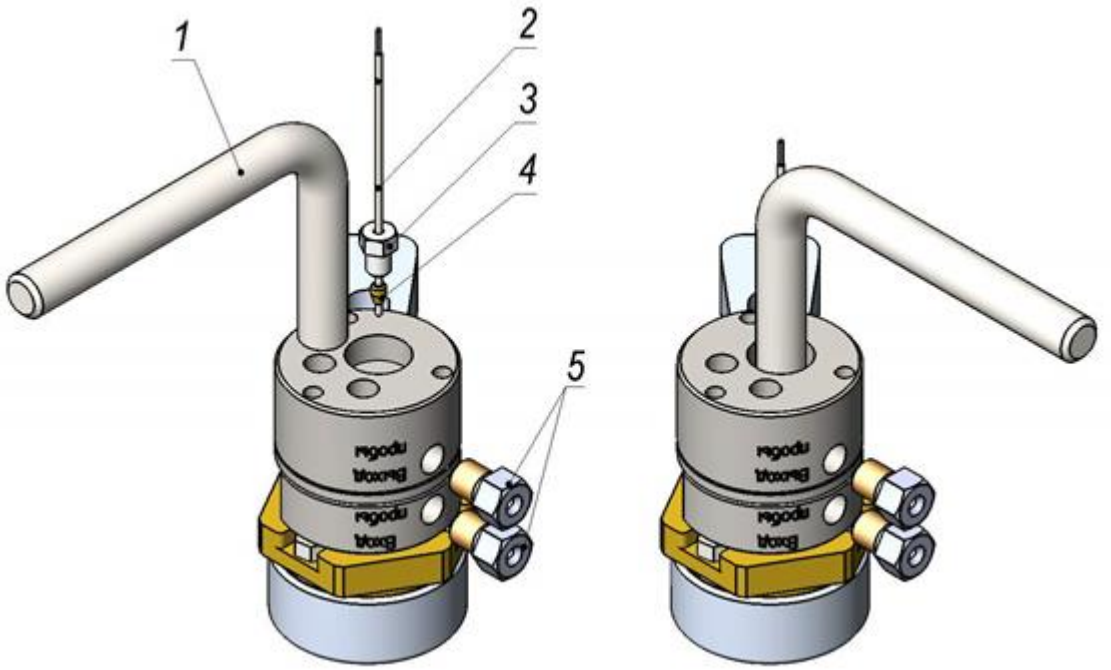
3.2.2 Needle installation

Before installing the needle (for example, when replacing it), it is recommended to temporarily screw nuts 8.930.286 into the valve body (see Figure 6). When tightening the needle, the nuts will act as a stop.

Insert the needle all the way into the valve body, tighten nut 8.930.111-02 with force "by hand" to fix the needle in the valve body, then use wrench 8.392.002 to tighten the nut by (35-45) degrees. After installing the needle, nuts 8.930.286 should be unscrewed from the valve body.

Needle 6.054.006-01, wrench 8.392.002 and fastening parts (ferrule 8.658.017, nut 8.930.111-02) are included in the shipping kit.

When tightening the needle, do not apply excessive force to the valve handle.



1 – wrench 8.392.002; 2 – needle 6.054.006-01; 3 – nut 8.930.111-02; 4 – ferrule 8.658.017; 5 – nut 8.930.286.

Figure 6 – Needle installation

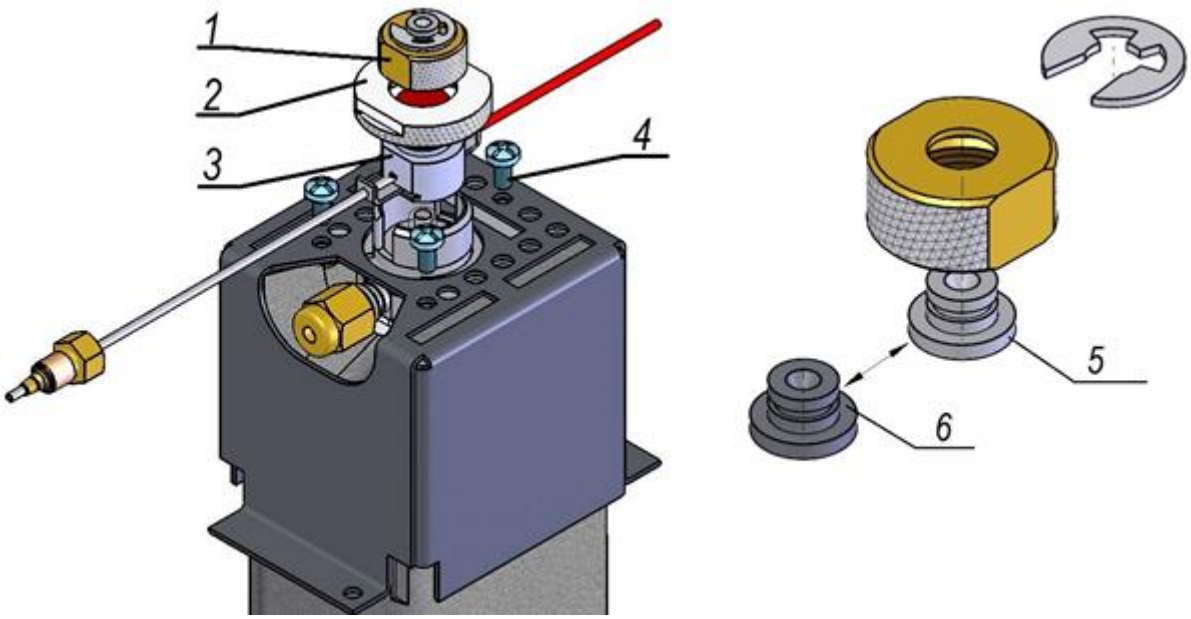
3.2.3 Mounting the LSV on the inlet

To install the tap on the GC inlet, follow procedure:

Unscrew nuts pos. 1 and 2 from the inlet. Check whether the valve needle passes through the hole in the needle guide pos. 5 (Figure 7). If necessary, replace the needle guide pos. 5 with another one with a hole diameter of 1.1 mm, pos. 6 (Figure 7), from the inlet shipping kit.

Take out adapter 3 from the inlet;

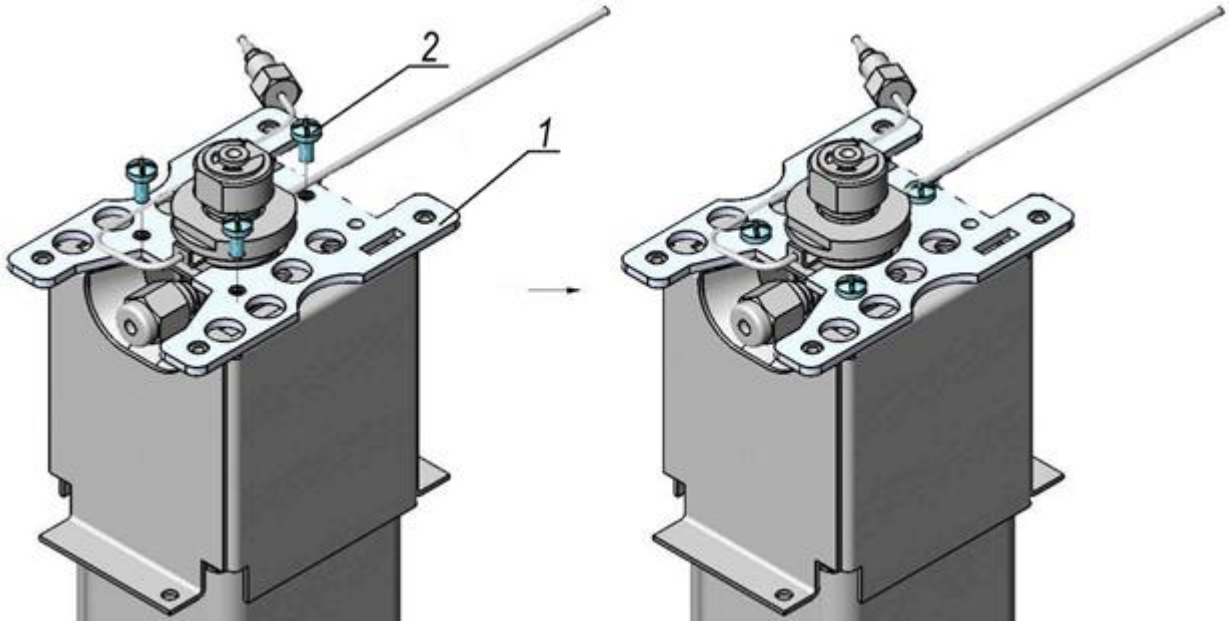
Unscrew screws pos. 4 securing the inlet casing;



1 – nut 6.482.013; 2 – nut 8.930.222; 3 – adapter 5.889.016; 4 – screws M3x6; 5 – needle guide 8.220.184; 6 – needle guide 8.220.184-01.

Figure 7 – Inlet preparation

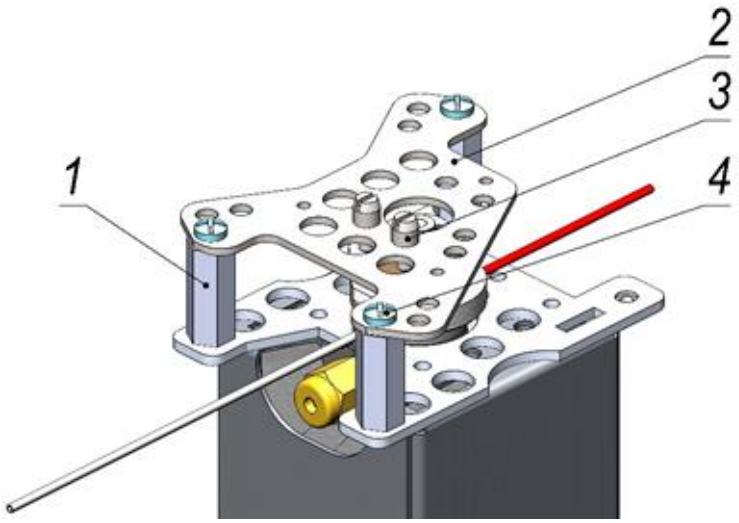
Install the plate 6.420.091 from the shipping kit of the LSV on the inlet casing and secure it with screws, as shown in Figure 8.;



1 – plate 6.420.091; 2 – screws M3x6 (3 pcs.).

Figure 8 – Installation of the plate fixing the LSV

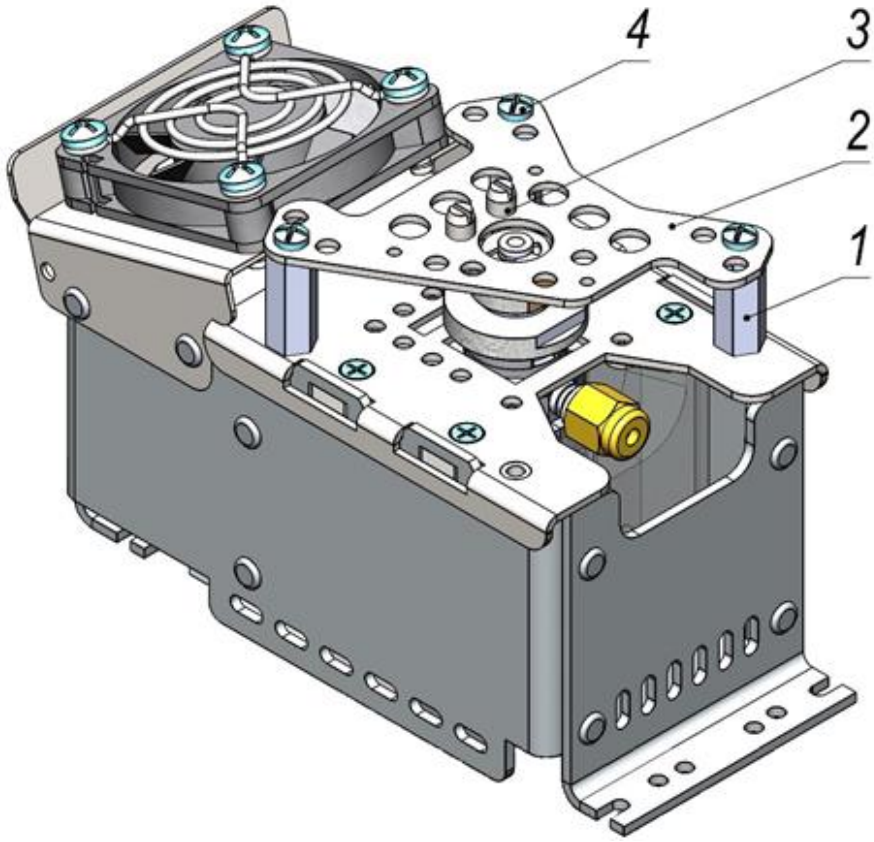
Screw the stands pos. 1 into the plate 6.420.091, as shown in Figure 9. Install the plate pos. 2 from the valve shipping kit onto the stands and secure with the screws pos. 4. Then screw the mounting pins pos. 3 into the plate (Figure 9).



1 – stand 8.120.189 (3 pcs); 2 – plate 8.612.945; 3 – pin 8.907.025 (2 pcs); 4 – screws M4x6 (3 pcs).

Figure 9 – Installation of mounting plate

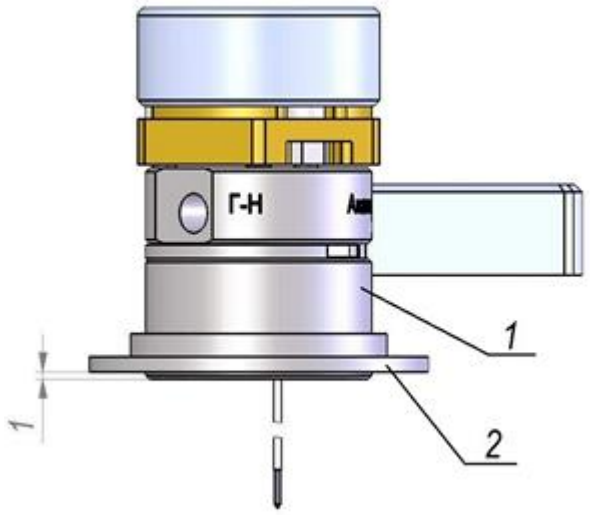
If the GC is equipped with an inlet with another casing configuration, then plate 6.420.091 is not installed, and plate 8.612.945 with stands is attached directly to the inlet casing (Figure 10);



1 – stand 8.120.189 (3 pcs); 2 – plate 8.612.945; 3 – pin 8.907.025 (2 pcs); 4 – screws M4x6 (3 pcs).

Figure 10 – Installation of mounting plate

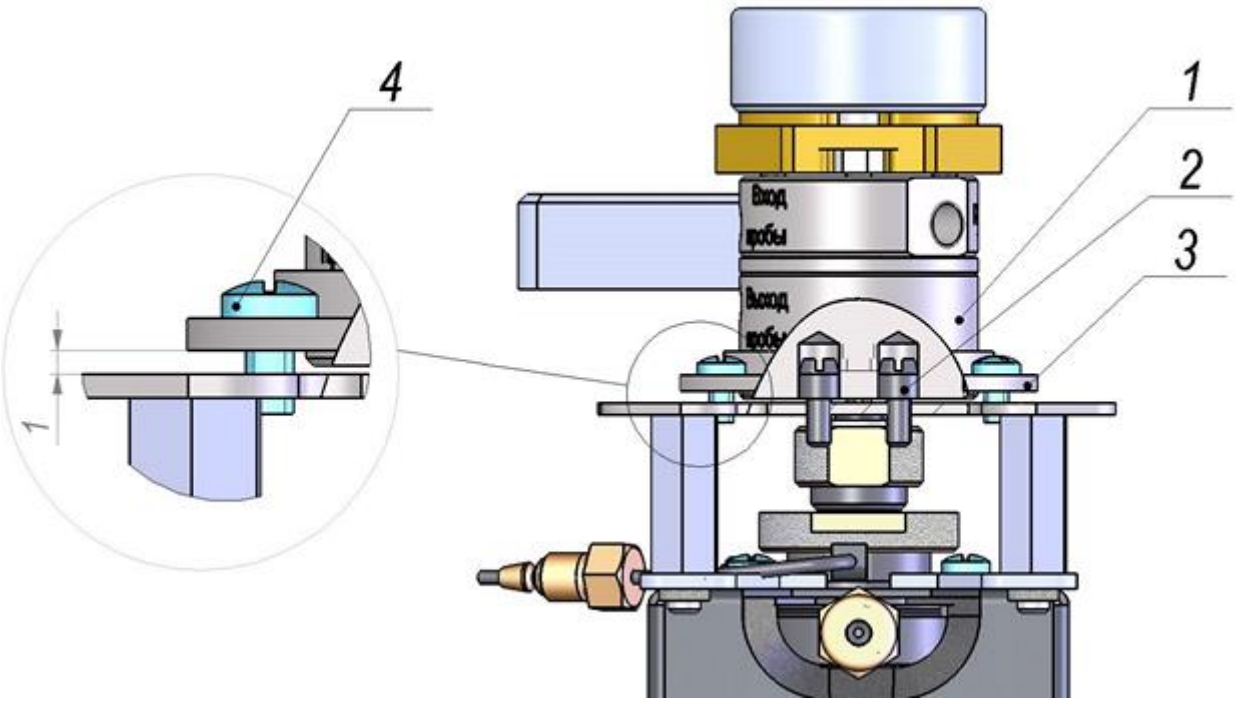
Screw a mounting ring pos.2 from the shipping kit onto the valve body (Figure 11);



1 – liquefied sampling valve 4.464.017-05 (-06; -07); 2 – Mounting ring 8.241.144.

Figure 10 – Mounting ringe installation

Install the valve on the inlet, aligning the holes in the body pos. 1 of the valve and the mounting pins pos. 2 on the plate. Screw up the mounting ring pos. 3 so that the mounting holes on the ring and plate are aligned, and there is a gap of approximately 1 mm between the ring and the plate, as shown in Figure 12. Secure the ring to the plate with the screws pos. 4;

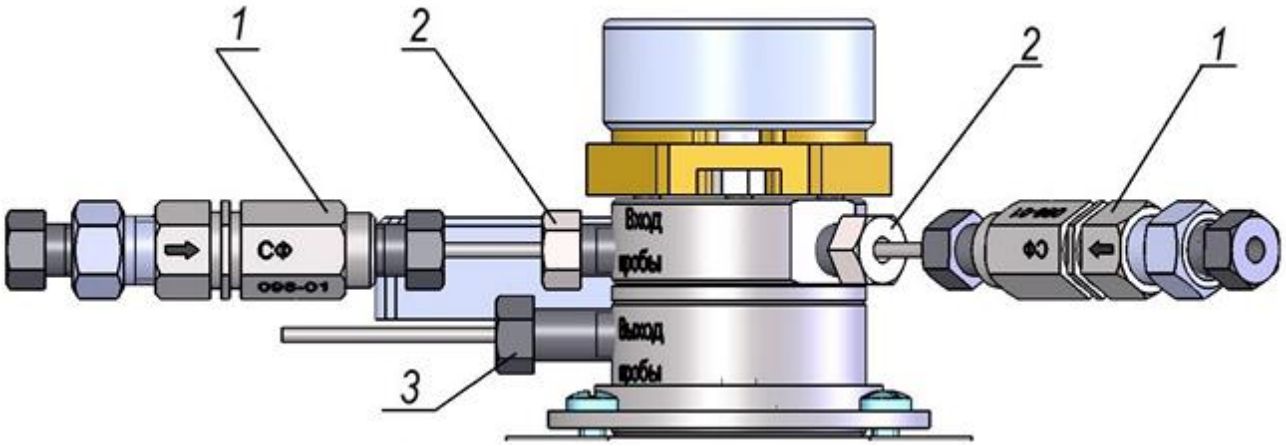


1 – valve body; 2 – pin 8.907.025; 3 – mounting ring 8.241.144; 4 – screws M3x6 (3 pcs).

Figure 11 – Securing valve on the inlet

Connect and tighten the external tubes for the carrier gas and sample using the nuts pos. 2 and 3 with special coating from the shipping kit. It is mandatory to install filters 5.884.098-01 removing

solid particles at the sample inlet (Вход пробы) and the carrier gas (Г-Н) channel, as shown in Figure 13.



1 – filter 5.884.098-01; 2 – nut 8.930.286; 3 – nut 8.930.263

Figure 12 – The LSV with connected filters

It is prohibited to use brass nuts without a special coating for connecting tubes to the valve.

3.2.4 Connecting sample cylinders to the LSV

When analyzing liquefied gases, sample cylinders of the PU, PGO or PP type are used to load the sample into the LSV.

Sample cylinders of PU and PGO types (constant volume vessels) should be positioned vertically during analysis, and the outlet fitting of the vessel should be above the level of the LSV, and keep this position during all the sequence of analyses.

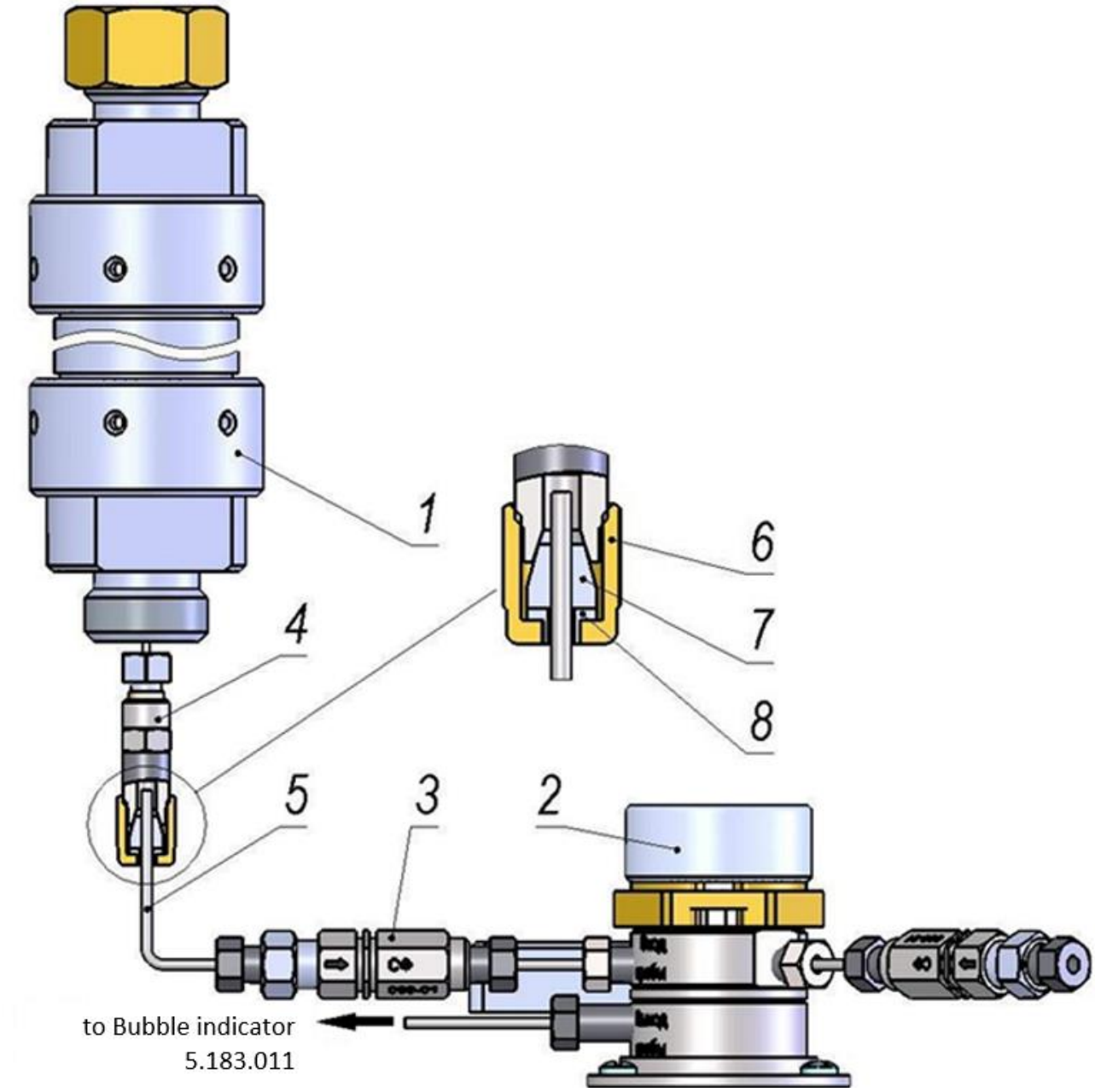
For convenience, it is recommended to fix PU, PGO cylinder of these types on a holder for sample cylinders 214.6.156.004 manufactured by Chromatec, all the necessary elements for fastening and connection are included in the holder delivery set. The procedure for working with the holder is described in the instructions 214.6.156.004I.

A laboratory stand can also be used to secure the sample cylinder, for this case follow connection in accordance with Figures 14 and 15.

Connection of PU type samplers to the LSV is performed in accordance with Figure 14. Adapter 6.454.102 is included in the shipping kit. To prevent from ingress of solid particles that may be contained in liquefied gas, the adapter has a mesh filter (8 µm cell).

Carry out preparation for connection as follows:

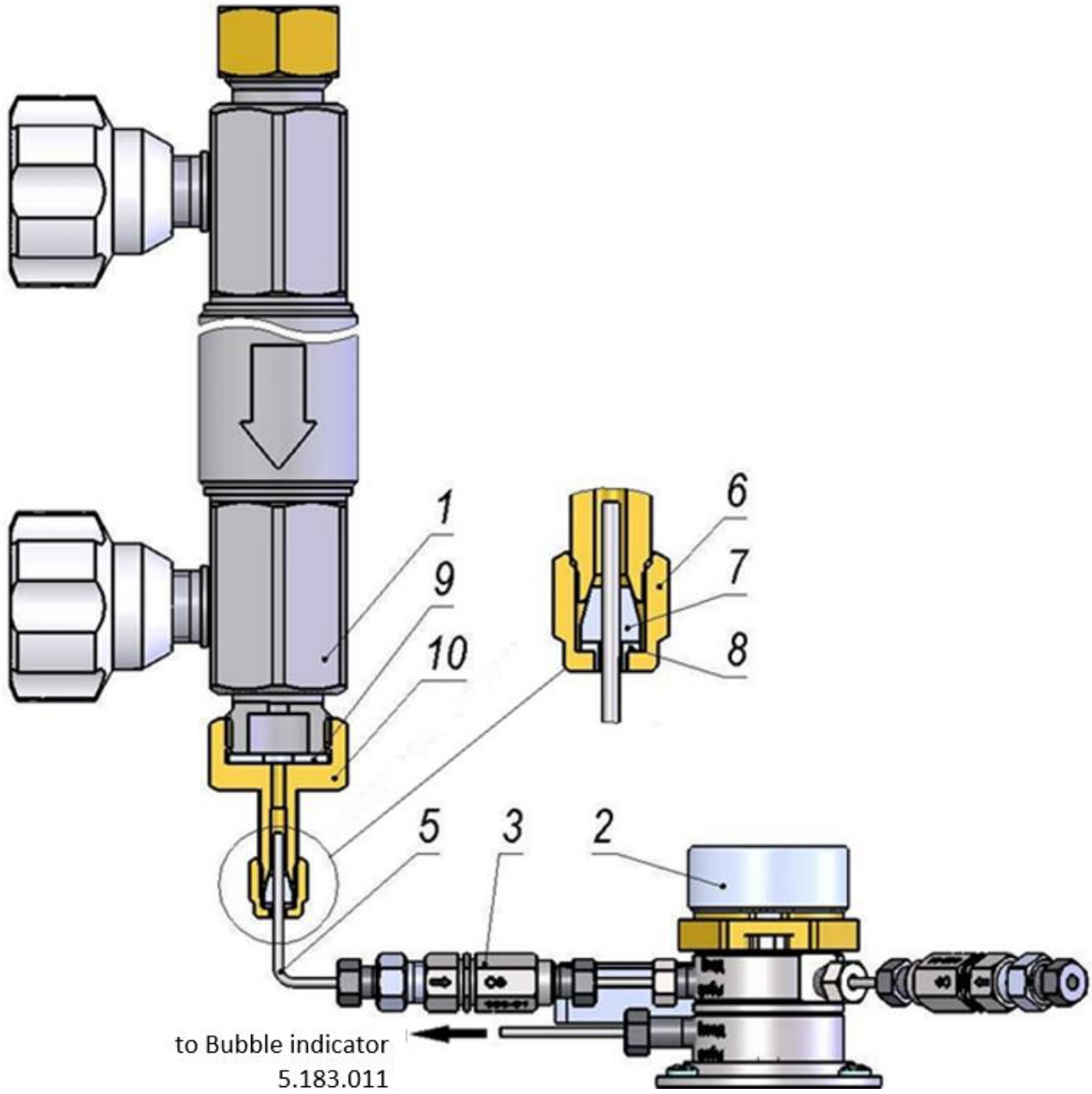
- Connect bubble indicator 5.183.011 (see section 3) to the sample outlet (Выход);
- Screw in a handle of the needle valve clockwise up to the stop;
- Connect tube pos. 5 and then adapter pos.4 to the filter pos.3;



1 – PU sample cylinder; 2 – the LSV; 3 – filter 5.884.098-01; 4 – adapter 6.454.102; 5 – tube 1.6 mm (1/16"); 6 – nut 8.930.167; 7 – ferrule 8.658.052; 8 – insert 8.223.429.

Figure 13 – Connecting the LSV to PU sample cylinder

When using PGO sample cylinder, then connection is performed in accordance with Figure 14.



1 – PGO sample cylinder; 2 – the LSV; 3 – filter 5.884.098-01; 5 – tube 1.6 mm (1/16"); 6 – nut 8.930.167; 7 – ferrule 8.658.052; 8 – insert 8.223.429; 9 – rubber gasket (disk 7.858.010-08); 10 – adapter 8.652.153.

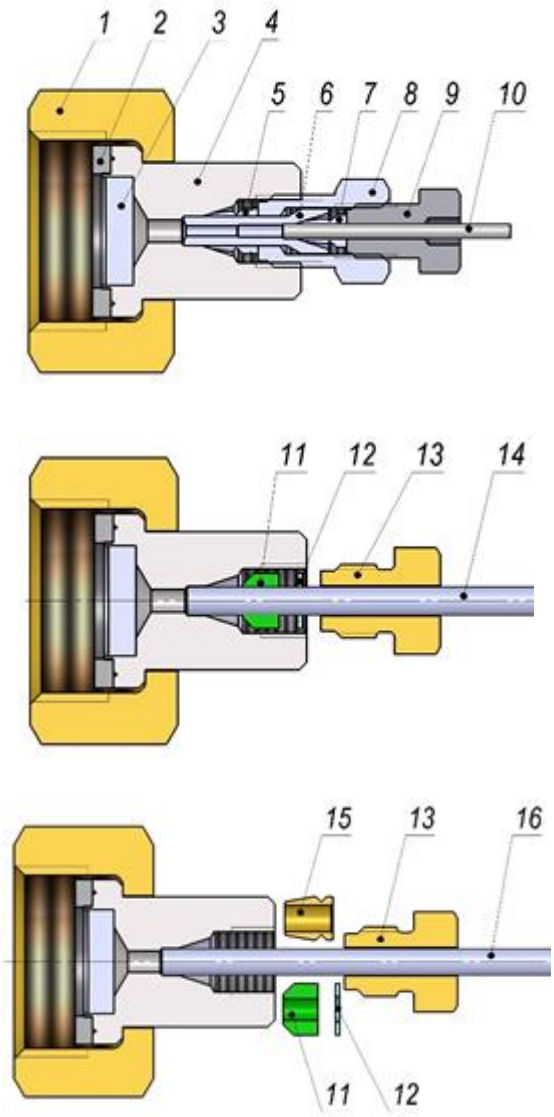
Figure 14 – Connecting the LSV to PGO sample cylinder

If necessary to filter the sample, use filter 5.884.056 (frit 8 µm) from shipping kit instead of adapter 8.652.153. The design of the filter, options for connecting tubes to the filter are shown in Figures 16 a, b, c. The necessary connection and sealing elements are available in the shipping kit.

a)

b)

c)



1 – nut 8.930.046-02

2 – gasket 8.685.162

3 – filter 7.062.035 (frit)

4 – housing 8.035.238

5 – ferrule 8.658.038-03

6 – tube 8.626.995

7 – ferrule 8.658.039-01

8 – nut 8.930.359

9 – nut 8.930.286

10 – metal tube OD 1.6 mm (1/16")

11 – ferrule 8.220.272-01

12 – washer 3

13 – nut 8.930.114

14 – PTFE tube 3 mm OD.

15 – ferrule 8.658.038 (for 3.0 mm tube) or ferrule 8.658.038-02 (for 3.2 mm (1/8") tube)

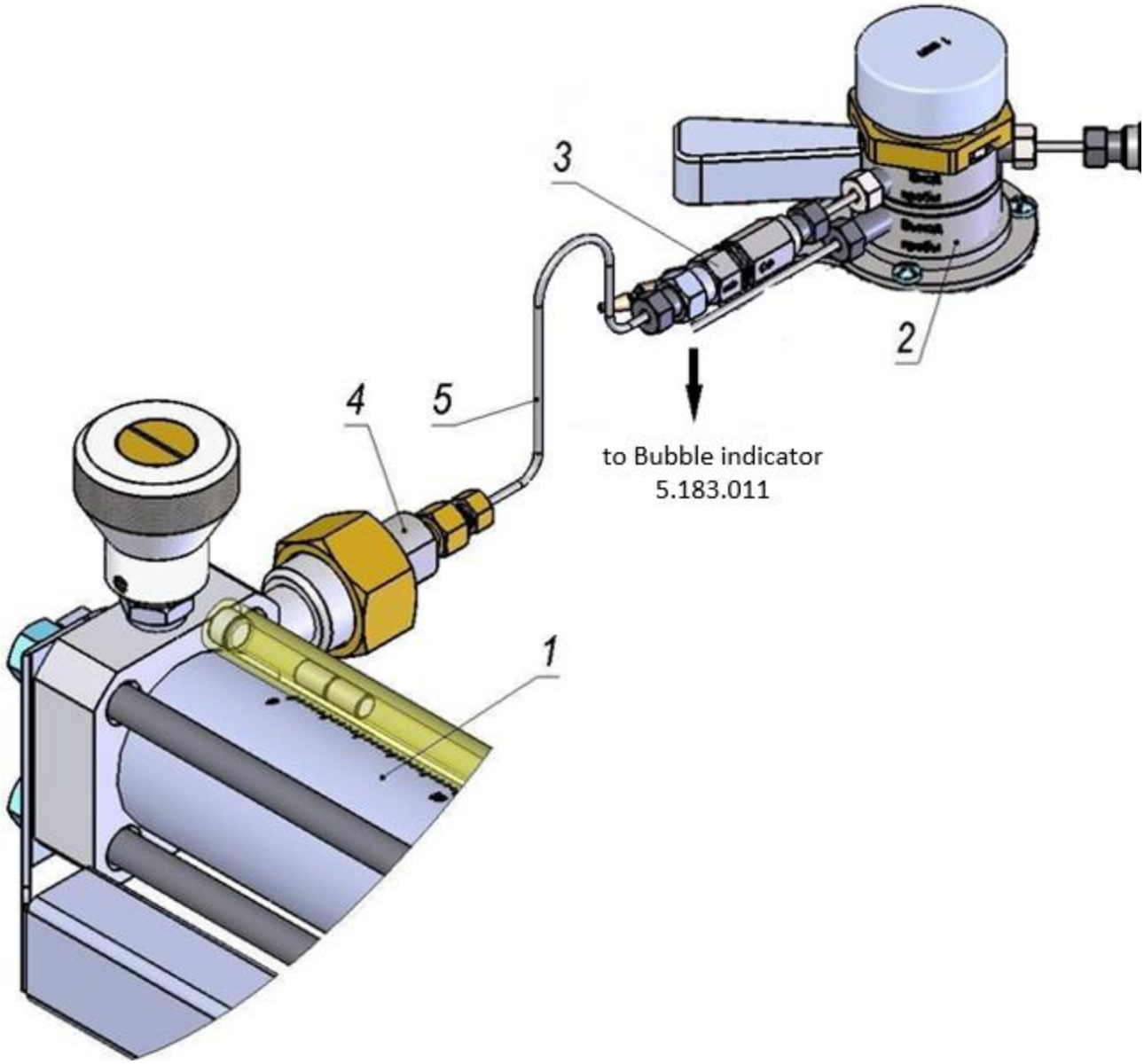
16 – metal tube OD 3.0 or 3.2 mm (1/8").

Figure 15 – Filter 5.884.056 and options for tube connection

Filter 5.884.056 (Figure 15a) is supplied by default with the necessary parts for connecting a metal tube with a diameter of 1.6 mm (1/16"). When connecting PTFE tube of OD 3 mm (Figure 15b) or metal tube of OD 3 – 3.2 mm (Figure 15c), it is necessary to remove parts pos. 5 – 9 (Figure 15a) and use the sealing elements from the GC shipping kit.

To replace the mesh filter (pos. 3, Figure 16a), first disconnect the tubes, then remove the filter from the housing by pushing it out of the housing (to remove the filter, it is recommended to use a metal rod with 2 – 2.5 mm OD). Replace the filter with a new one, inserting it into place with a little force. If necessary, replace the sealing gasket (pos. 2, Figure 16a). All the necessary elements are included in the shipping kit.

Connection of the PP-150 sample cylinder (with floating piston) is shown in Figure 17. The vessel should be placed horizontally on the workplace, in a place convenient for work and control.

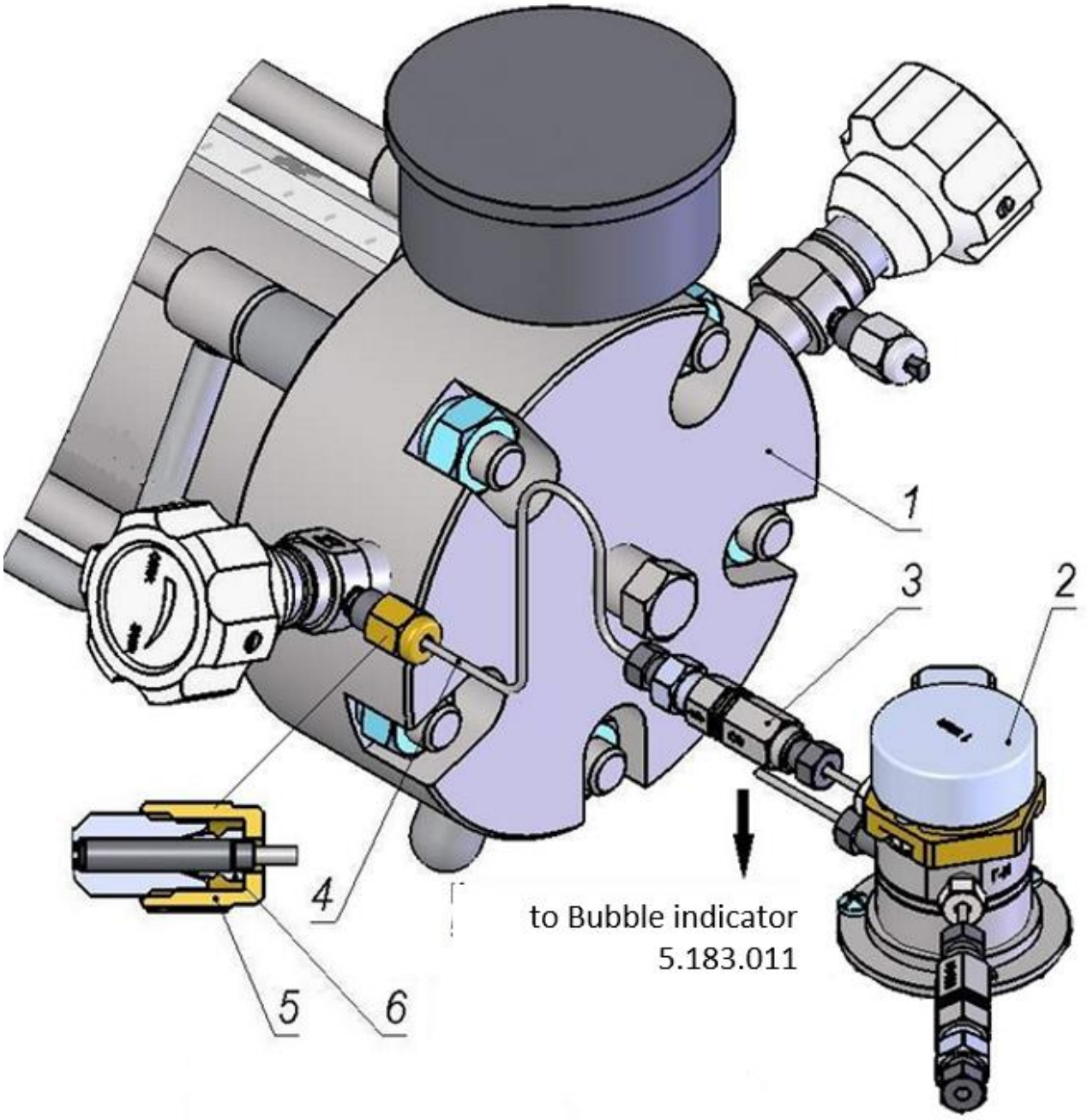


1 – floating piston sample cylinder PP-150; 2 – the LSV; 3 – filter 5.884.098-01; 4 – filter 5.884.056; 5 – tube 1.6mm (1/16") OD.

Figure 16 – Connecting the LSV to PP-150 sample cylinder

Connection of the PP-1000 (PP-2000) sample cylinder (with floating piston) is shown in figure 17.

The tube pos. 4 and sealing elements pos. 5 and 6 are included in the shipping kit of the sample cylinder.



1 – sample cylinder PP-1000 (PP-2000); 2 – the LSV; 3 – filter 5.884.098-01; 4 – tube 6.457.069-04; 5 – nut 8.930.284; 6 – ferrule 8.658.038-01.

Figure 17 – Connecting the LSV to PP-1000 (PP-2000) sample cylinder

The design of sample cylinders and the operating procedure are given in GOST 14921-78 and in the User Manuals: 214.4.056.000UM (for PU, PGO type); 214.4.056.013UM (for PP-150); 214.4.056.019UM (for PP-1000; PP-2000).

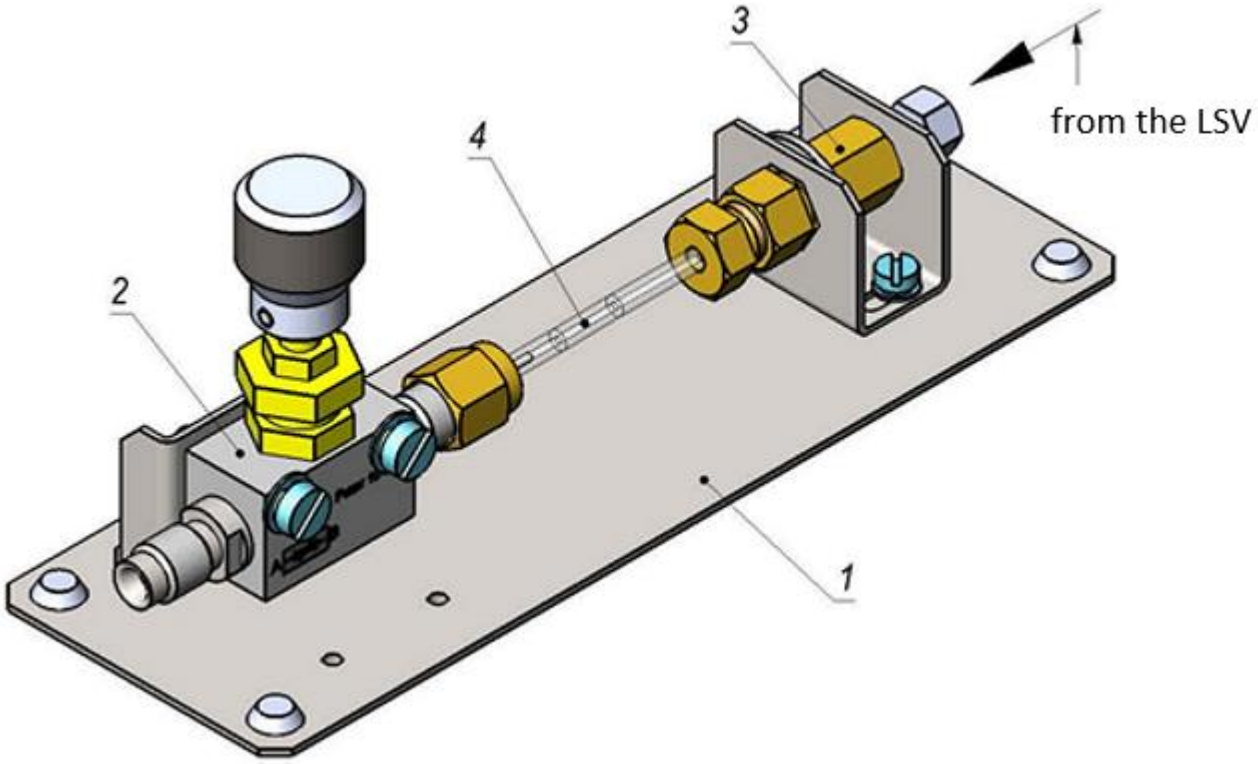
It is possible to use other similar vessels; in this case, you should get familiar with the relevant operating documentation.

3.2.5 Sample loading control

To control the sample loading, it is recommended to use the Bubble indicator 5.183.011 (hereinafter referred to as the indicator) from the shipping kit.

The indicator should be placed in a place convenient for work and observation.

The appearance of the indicator is shown in Figure 18.



1 – base; 2 – needle valve 5.150.018; 3 – union 6.115.168; 4 – indicator tube

Figure 18 – Bubble indicator. Overview

Work with the LSV and indicator during a series of analyses is carried out as follows:

Connect the sample outlet tube from the LSV to the union pos. 3, as shown in Figure 18.

If necessary, connect the tube from the LSV shipping kit to the outlet of the needle valve pos. 2 and lead it into a container with water or under the hood.

By smoothly rotating the needle valve handle counterclockwise, set the required sample flow rate and blow through the entire system for a certain time until a pure liquid sample flow is obtained.

Visually monitor the sample flow, there should be no bubbles of the degassed phase in the indicator tube pos. 4 (Figure 18).

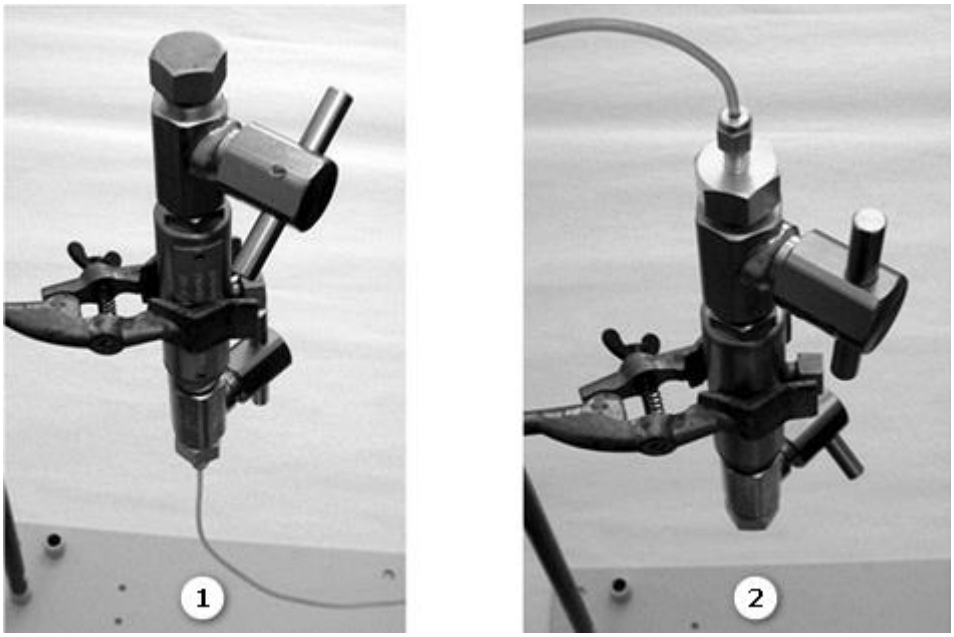
After injecting the sample (moving the valve handle to the "Analysis" position), close the needle valve by rotating the handle clockwise.

3.2.6 Blowing sampling line tubes after series of analyses

Once sequence of analyses is completed, the sample lines should be carefully blown to remove the rest of liquefied sample. The procedure below describes blowing on the example of PGO-50 sample cylinder.

Before disconnecting the sample cylinder, conduct a procedure:

- turn the vessel upside down with the fitting connected to the LSV moved upward (see Figure 20, right);
- switch the LSV to the "Sampling" position;
- blow the sampling lines with the gas phase;
- close the sampler valves;
- disconnect the sample cylinder.



1 – sample cylinder position when analysis of liquefied sample running, 2 – sample cylinder position when blowing of sample lines conducted

Figure 19 – Sample cylinder position

3.3 Operability test

Operability test is performed using the LSV as part of the GC operating as a sample injecting device. During the check, the repeatability of the results of two runs in series is monitored for all hydrocarbons of the analyzed liquefied gas sample (LPG) in accordance with the analysis test method according to GOST 10679-2019 "Liquefied hydrocarbon gases. Method for determining hydrocarbon composition". The permissible rates of repeatability are specified in Table 5 of GOST 10679-2019.

The operating mode of the chromatograph and the chromatographic columns should comply with GOST 10679-2019. It is permitted to use other chromatographic columns (including capillary columns) and measurement conditions that ensure separation of the sample into individual components. When using the LSV for other measurements, it is permitted to test it in accordance with other measurement methods approved in the established manner.

3.4 Troubleshooting

Apart from eliminating possible leaks of carrier gas or sample (see Maintenance section), the user should not attempt to fix any possible faults on their own. If any are detected, the user should contact the manufacturer or its regional representative.

4 Maintenance

The LSV does not require complex maintenance. During operation, it is necessary to monitor the tightness of the gas lines.

Check for leaks is carried out by the manometric control method according to GOST 25136-82. Leaks are eliminated by tightening the sealing elements, replacing, if necessary, the corresponding sealing ferrules.

It is permissible to check leaks using the gas flow regulator of the GC. The check should be performed in the following sequence:

- Remove the chromatographic column. Seal the released inlet fitting in the column oven with a suitable plug. When working with TCD detector, you can install a plug at the outlet of the corresponding TCD detector cell without removing the column (for example, plug 8.632.095 with ferrule 8.658.053). The selection of suitable sealing elements depending on the type of column, TCD outlet fitting is performed in accordance with the User Manual of the GC used.
- set the carrier gas flow rate in the line being tested to 90 ml/min;
- after a 5-minute hold, check the flow rate (leakage) of the carrier gas in this line.

Permissible leakage 1 ml/min with voltage on the flow regulator valve of at least 9 V.

When checking for leaks, measures should be taken to prevent the failure of the TCD filaments.

These measures consist of disabling the heating mode of the TCD filaments and monitoring the temperature of the filaments, which should be equal to the temperature of the detector.

If necessary (appearance of ghost peaks, increase in noise level), the gas lines and sample loop are washed with a solvent (technical ethyl alcohol GOST 17299-78), followed by purging with a carrier gas for at least 2 hours.

5 Manufacturer warranty

The manufacturer guarantees that the valve meets the technical specifications if the user complies with the operating conditions.

The warranty period is 12 months from the date of commissioning, but not more than 18 months from the date of acceptance.

The valve is subject to removal from warranty service if the manufacturer's seal is broken.

The seal of the valve is shown in Figure 21. The serial number and year of manufacture are engraved on the valve body.

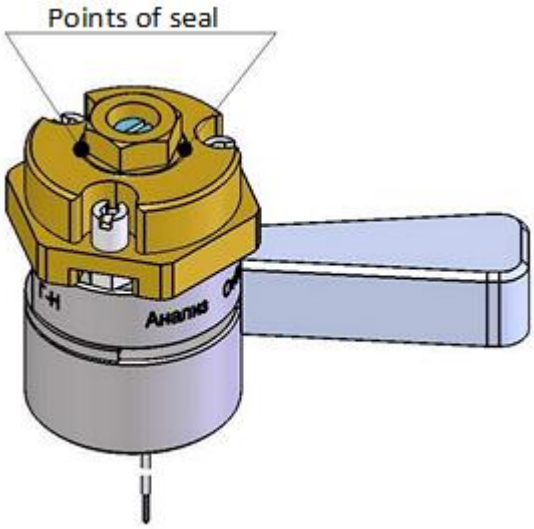


Figure 20 – Sealing the valve (a top cover removed)

6 Claims

In case of the valve failure within warranty period it is necessary to:

- draw up a technically reasoned act of defects concerning its incompliance with technical specifications, specified in the Passport;
- make abstracts from section "Acceptance Certificate";
- specify date and the organization that performed putting into operation;
- specify performed maintenance procedures.

It’s allowable sending copies of sections "Acceptance Certificate" and "Maintenance record" witnessed by head of customer’s organization.

The claim should be sent to the address:

Special Design Office “Chromatec”, 94 Stroiteley Str., Yoshkar-Ola, Russian Federation 424000; or at Correspondence address: Central post office, P.O. box 84, Yoshkar-Ola, Mari-El republic, Russian Federation 424000.

424000 г. Йошкар-Ола, ул. Строителей 94, СКБ "Хроматэк" или 424000, г.Йошкар-Ола, Главпочтамп, а/я 84.

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