AUTOSAMPLER AS-2M (3D)

USER MANUAL

214.2.508.006-05UM

PRODUCT P/N: 400-0501

Rev. 20.12.2023

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Important notes

The present User Manual (UM) is intended to get familiar with Autosampler AS-2M (3D) P/N 400-0501 (hereinafter referred to as the Autosampler) and contains all necessary instructions to ensure its correct operation and maintenance.

Before using the Autosampler accurately read the present User Manual and relevant service documents included to chromatograph that is going to be used with.

During operation, it is recommended to read this manual periodically in order to recollect the characteristic features of the aurosampler.

Instructions marked with this box are necessary for carrying out in order to avoid injuries sustaining at working with Autosampler.

Two marking types of parts are used in this and accompanied documents:

XXX-XXXX – *Part number* or *Cat. Number* referred to ordering documents;

214.X.XXX.XXX(-XX) or simplified X.XXX.XXX(-XX) – *Cross ID* referred to internal manufacturer documents.

Both can be used for ordering parts.

The sampler can be equipped with:

- 0.5, 1.0, 5.0 and 10.0 µl microsyringes (used in Agilent 7673 and Agilent 7683 autosamplers) – complete with holder 214.6.152.012, necessary to limit the size of safe extension of the plungers of these microsyringes from their glass bodies;
- microsyringes of 100 and 250 µl volume – complete with the holder 214.6.152.013 (autasampler having an option of headspace injection into the inlet). In this case a special washing mode is realized – pumping their working cavities with air;
- syringe unit equipped with a 10 µl microsyringe with a flange for "top washing", which also has a possibility of washing "from vials" – complete with holder 214.6.152.011.

In the optional version (autasampler AS-2M (3D), is designed for automatic microsolid phase extraction (MEPS – Micro Extraction by Packed Sorbent).

Types of microsyringes, description of their designs, as well as methods of work with them by the operating personnel are described in this manual (p. 2.4.3, 6.1).

Specific type of microsyringe(s) or syringe unit, as well as their quantity shall be agreed with the customer when concluding the contract.

1 Safety provisions

The Autosampler represents a robot engineering, so be careful with movable parts.

The Autosampler can be used by person familiar with the present User Manual and having adequate skills and knowledge to operate this device.

The Autosampler should be grounded. For grounding, use power plug and grounding terminal provided on the rear side of sampler. For sampler connection, the "⊥" terminal of mains socket must be connected to external grounding loop by copper wire with cross-section of at least 1,5 mm^2; terminal must be connected to grounding loop using cable 6.644.056 supplied in shipping kit.

DO NOT USE sampler with removed covers**!**

Maintenance, replacement of vials, filling of the vial with flushing liquid may be performed only when the power cord is disconnected from the mains and the air (or gas) line is shut off.

When using flammable, hazardous or aggressive substances, including those dedicated for syringe washing, observe safety fire precautions specified in national standard GOST12.1.004-91. Also, observe precautions specified in relevant instructions prepared by User (according to substances used) based on national standard GOST 12.1.007-76.

When sampler will not be used for long time, disconnect it from power supply and close gas line.

2 Description

2.1 Introduction

The Autosampler AS-2M (3D) is intended for automatic sampling and injecting liquid samples into gas chromatographs "Chromatec-Crystal 5000", "Chromatec-Crystal 9000". AS-2M (3D) is controlled from the "Control Panel" program included in the "Chromatec Analytic" software package.

Operation of the Autosampler is carried out in closed laboratory premises, provided that combustible gases and highly inflammable liquids may be present in volume insufficient for creation of explosive– or fire-hazardous mix. Environmental conditions:

- Air temperature from 10 to 35 °C;
- Relative humidity not more than 80 %;
- Atmospheric pressure from 84 to 107 kPa (from 630 to 800 mm Hg);
- Impurity content in premises air shall be normal, regulated by GOST 12.1.005.

Autosampler climatic performance: TC category 4.2 in accordance with GOST 15150.

The autosampler is designed to operate under following power supply conditions: 50±1 Hz, 187-242V AC.

The autosampler meets electrical safety requirements per class 1, type H according to national standard GOST 12.2.025-76.

For correct operation of the autosampler the following facilities required:

- *Control panel* software (SW), 2.0.8.18 version or above;
- Central module PM3 (GC firmware) 3.20.16.560 version or above

Control panel SW is supplied in *Chromatec Analytic* SW pack. Required central module (firmware) is uploaded in GC by manufacturer. Besides, firmware file for uploading is available on CD with *Chromatec Analytic* SW.

2.2 Technical specifications

Refer to Passport 214.2.508.006-05PS for technical specifications. Refer to corresponding sections of Passport for Technical specifications of options.

Table 2.1 – Aspiration and injection rates depending on setting rate N

Syringe, µL	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
	Actual rate, µl /sec									
0,5	0,15	0,175	0,21	0,26	0,35	0,52	0,70	1,05	2,10	3,95
1	0,30	0,35	0,42	0,52	0,70	1,04	1,40	2,10	4,20	7,90
5	0,75	0,875	1,05	1,30	1,75	2,60	3,50	5,25	10,5	19,75
10	1,50	1,75	2,10	2,60	3,50	5,20	7,00	10,5	21,0	39,5
100	14,1	16,5	19,8	24,8	33,0	49,5	66,0	99,0	198	317
250	35,25	41,25	49,5	62,0	82,5	123,75	165,0	247,5	495	792,5

Table 2.2 – Injection (aspiration) time of 1 µl sample, msec

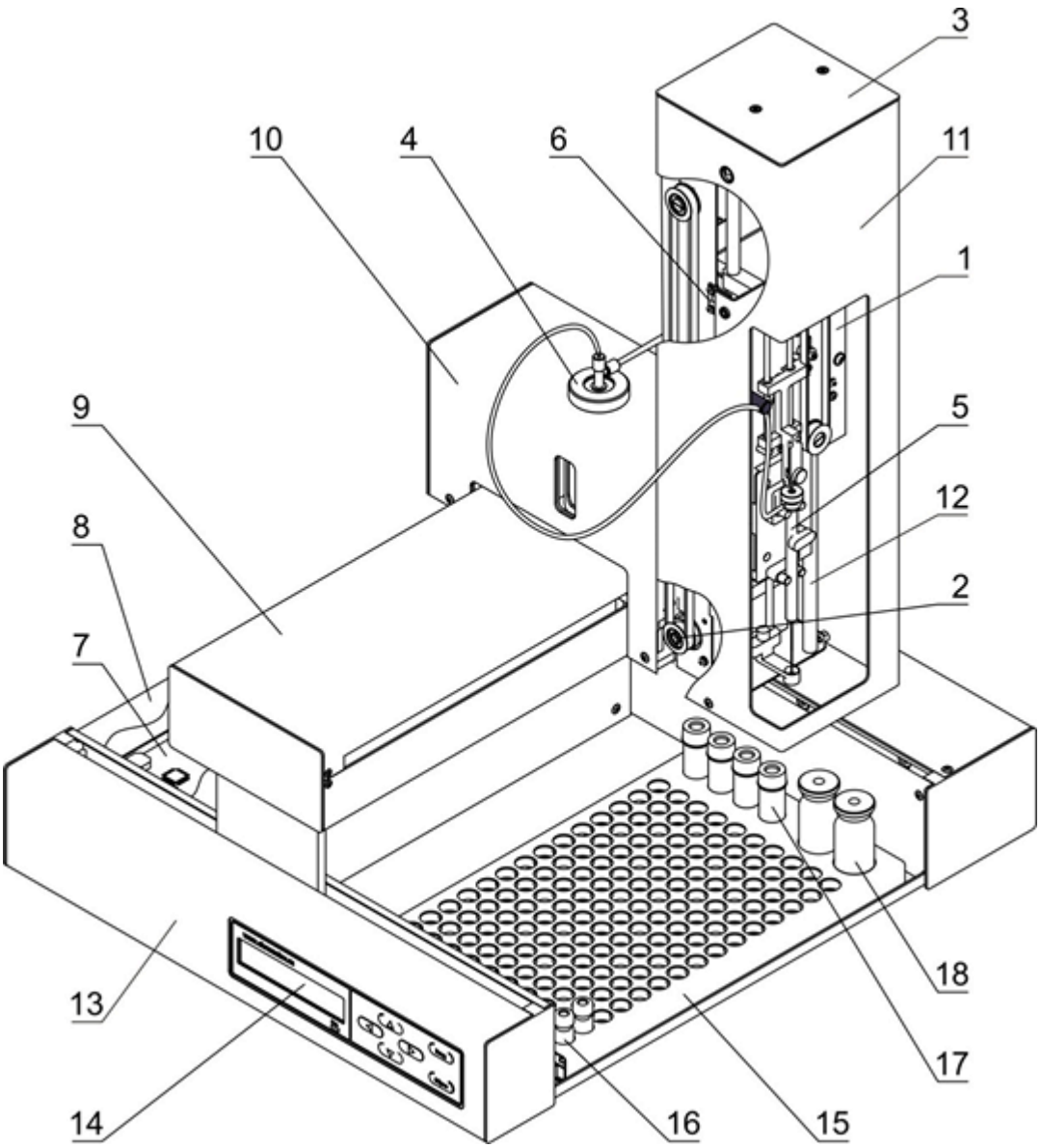
Syringe, µL	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
0,5	6670	5710	4760	3840	2860	1920	1430	950	480	250
1	3335	2855	2380	1920	1430	960	715	475	240	125
5	1334	1142	952	467	572	384	286	190	96	50
10	667	571	476	384	286	192	143	95	48	25
100	71	60	50	40	30	20	15	10	5	3
250	28,4	24	20	16	12	8	6	4	2	1,2

2.3 Delivery set

The Autosampler contains the items and documents listed in Passport (Section 6).

2.4 Construction

2.4.1 Overview



1 – syringe gear; 2 – syringe gear drive; 3 – Tower; 4 – washing unit; 5 – syringe unit (or syringe); 6 – optoelectronic sensor of syringe unit position; 7 – control unit; 8, 9, 10, 11 – covers; 12 – guide; 13 – front panel; 14 – display; 15 – replaceable tray; 16 – vial (2 ml); 17 – solvent vial (4 ml); 18 – waste vial (15 ml).

Figure 2.1 – Autosampler overview

The Autosampler contains the following components:

- syringe gear (in the tower);
- syringe gear drive (in the tower);
- basement with 2 drives providing movement of tower in horizontal axes by two orthogonally related directions;
- replaceable tray for sample vials, solvent vials and waste vials;
- washing unit;
- syringe unit (or syringe);
- control unit.

Syringe gear 1 (Figure 2.1) with mounted syringe unit 5 (or syringe) provides sampling and injection of set volume of the sample. At operation, syringe gear drive carries out set plunger movements.

The syringe gear 1 moves along cylindrical guide 12 by drive 2. It has three positions: upper, intermediate and low. These positions are controlled by optoelectronic sensor 6. The numerical values of intermediate and low positions are user-configurable. These positions can be set by user depending on operating conditions.

When syringe gear is in upper position, the tower 3 can move in horizontal axes drawn by 2 drives located on the basement.

In intermediate position of syringe gear, the Autosampler performs sampling operations, and:

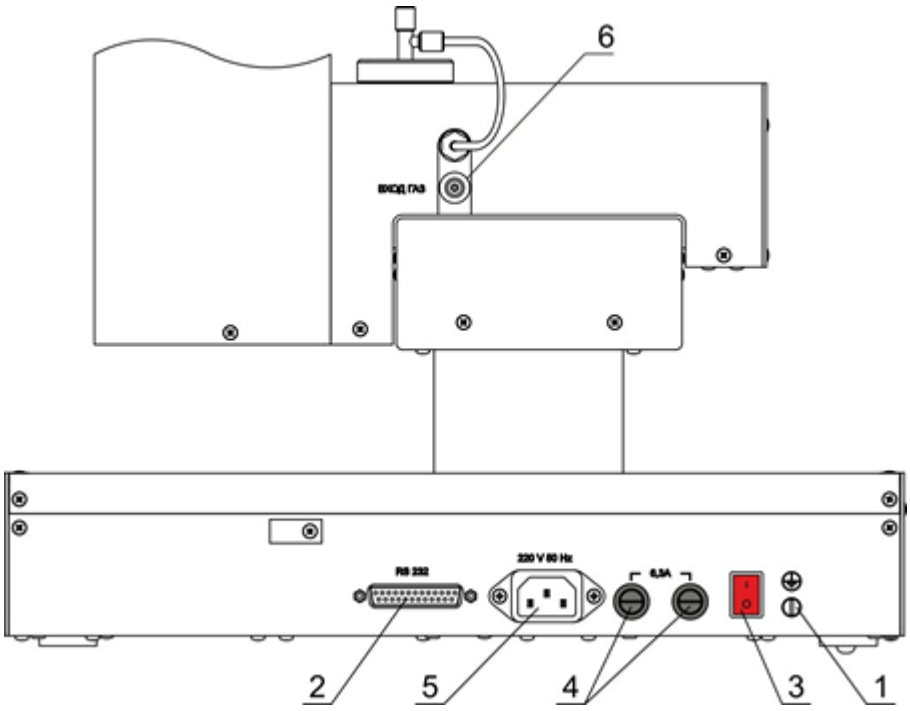
- "Top washing" mode, sampler provides plunger stroke (plunger moves upwards-downwards) or solvent drain;
- "From vials" washing mode, the autosampler allows for syringe washing.

In lower position, the Autosampler provides injection to inlet.

The control unit 7 is used to control sampler unit’s operation; it includes power unit and controller board.

The autosampler units are protected with covers 8, 9, 10 and 11.

The front panel 13 (Figure 2.1) contains display 14. The display shows current status, shows alarms and allows tuning coordinates at installation. Red LED is lighted whenever the Autosampler is on.



1 – grounding terminal; 2 – interface RS232; 3 – "POWER" toggle switch; 4 –fuses; 5 – "POWER 220V 50Hz" connector; 6 – "GAS IN" fitting.

Figure 2.2 – Autosampler rear view

The rear side of cover 11 features a screw, which is to be used for sampler transportation (Figure 2.3).

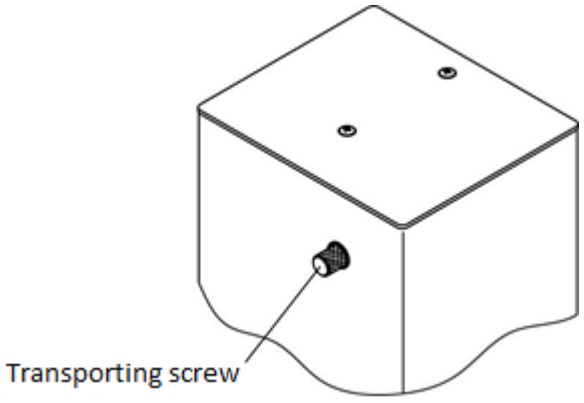
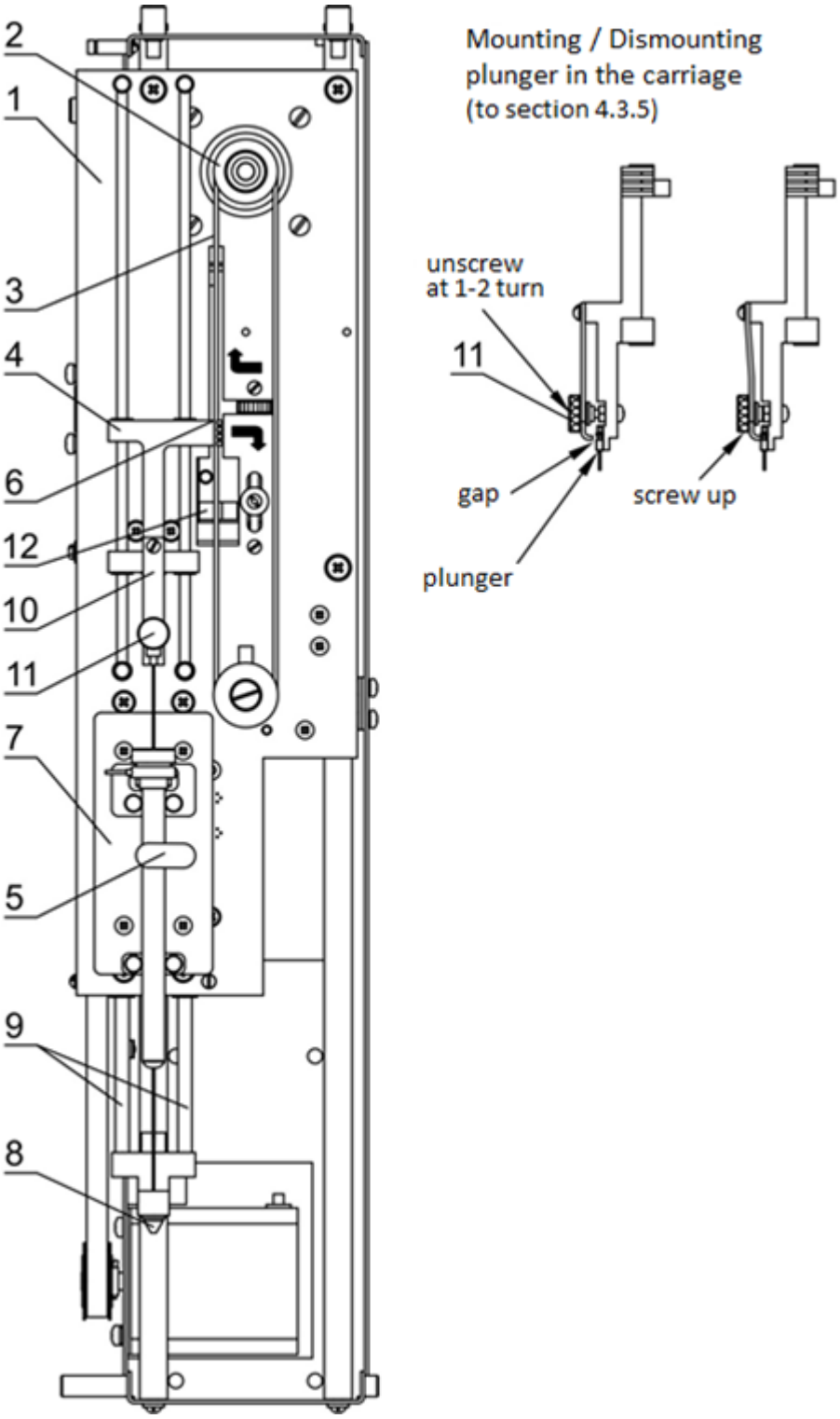


Figure 2.3 – Screw installed for transporting position of the Autosampler

Before the Autosampler transportation check that the screw is in transportation position to avoid negative movements of syringe gear along the guide.

2.4.2 Syringe gear



1 – tower basement; 2 – stepping motor pulley; 3 – flat toothed belt; 4 – carriage; 5 – latch; 6 – curtain; 7 – syringe adapter; 8 – needle adapter; 9 – guides; 10 – spring plate; 11 – screw; 12 – optoelectronic sensor.

Figure 2.4 – Syringe gear overview

The syringe gear (Figure 2.4) includes a base 1 with mounted software-controlled stepping electromotor. The motor shaft incorporates pulley 2 that drives flat toothed belt 3 transforming rotational motion of motor rotor to reciprocating movement of carriage 4. The carriage moves plunger of syringe. Syringe plunger fixed in the carriage 4 by a spring plate 10 and screw 11.

Syringe unit (or syringe) is fitted in a bed of syringe adapter 7 and fixed by latch 5.

Needle adapter 8 protects needle from mechanical damage and provides its aligning when working with vials and inlets.

The carriage 4 has curtain 6. The curtain and optical sensor of syringe plunger 12 are used to input data about current position of syringe plunger (this position corresponds to "zero-volume" sample in syringe) to control unit.

Do not apply excessive force when tightening screw 11.

2.4.3 Syringe unit and syringes

Type	Capacity, µL	Chromatec Cross ID	SGE P/N	Hamilton P/N
Syringe unit	10.0	4.464.114	-	-
Syringe	10.0	-	002821	80393
Syringe	5.0	-	001822	87993
Syringe	1.0	-	000610	80176
Syringe	0.5	-	000410	86276
Syringe	100.0	214.4.464.084	-	-
Syringe	250.0	214.4.464.102	-	-
Syringe	10.0*	214.4.464.119	-	-
Syringe	10.0**	214.4.464.128	-	-
Syringe	5.0*	214.4.464.123	-	-

* Needle length 50 (51) mm, needle outer diameter 0.47 mm.

** Gastight, needle length 50 (51) mm, needle outer diameter 0.47 mm.

Syringes with volumes of 0.5, 1.0, 5.0, 10.0 µl and needle lengths of 42-43 mm are used for injecting liquid samples. The syringe needle has a variable outer diameter of 23s/26s (0.63/0.47 mm) providing better reliability. These syringes are commercially available from manufacturers SGE or Hamilton. Only vial “washing from vials” is available for these syringes.

The 5.0 µl microsyringe has a thin and long plunger. This requires increased attention from the operator when using it, who must create conditions for quality washing of the pair "cylinder – plunger"; even slight rubbing of the plunger in the cylinder is inadmissible.

Syringe unit 4.464.114 with a volume of 10 µl is designed for liquid sample injection. The autosampler can be washed "from the vial" or "from the top". Top washing provides longer service life due to complete flushing of all syringe cavities.

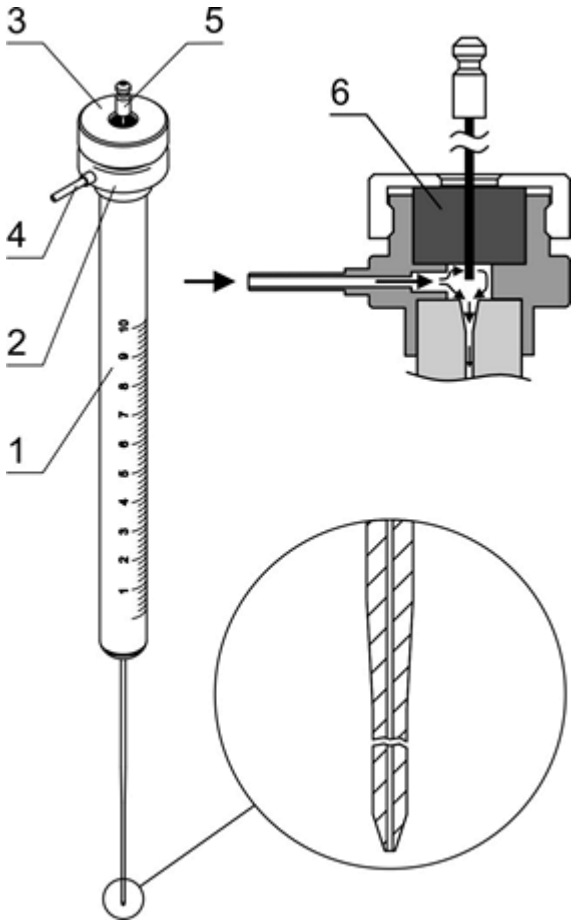
Syringe unit 4.464.114 (Figure 2.5) incorporates syringe 1 and assembly with a flange 2 in base. The assembly serves to deliver solvent through a side located fitting 4 into syringe barrel in top washing mode. Solvent flow direction is shown in in right fragment below. Teflon sealing 6 pressed by nut 3 provides tightness of assembly.

In case of difficulties with the syringe unit during its operation, contact the repair service of the manufacturer or its regional representative.

In the "top washing" mode, the microsyringe plunger is located in the sleeve 6 above the end face of the microsyringe body.

The microsyringe needle of the syringe unit 4.464.114 has a variable cross-section (Figure 2.5).

Refer to Section 4.8 for the installation procedure for the syringe units and microsyringes.



1 – syringe; 2 – flange; 3 – nut; 4 – fitting; 5 – plunger, 6 – teflon sealing.

Figure 2.5 – Syringe unit 4.464.114

The 5 and 10 µl microsyringes with 50 mm needle length and 0.47 mm diameter are recommended for direct sample injection into the column (on column), but can also be used for other sample

injection types into the inlet. Microsyringes are manufactured by Chromatec based on SGE syringes. The 214.4.464.128 microsyringes have a gas-tight Teflon plunger.

100 and 250 µl gas-tight microsyringes with interchangeable needle with side opening. They are manufactured at Chromatec on the basis of Hamilton syringes. Syringes are designed for use in the autosampler only in the headspace from the vial and its injection into the chromatograph inlet. For 0.5 and 1.0 µl microsyringes – this is the size H = 137 mm max, for 5 and 10 µl microsyringes H = 150 mm, for syringe unit 4.464.114 – H = 172.5 mm.

The types of some of the microsyringes used in the autosampler, their basic dimensions, and the limits of allowable piston outreach for each are listed below. For the Hamilton 7000.5 ASRN and Hamilton 7001 ASRN, this is dimension H = 137 max).

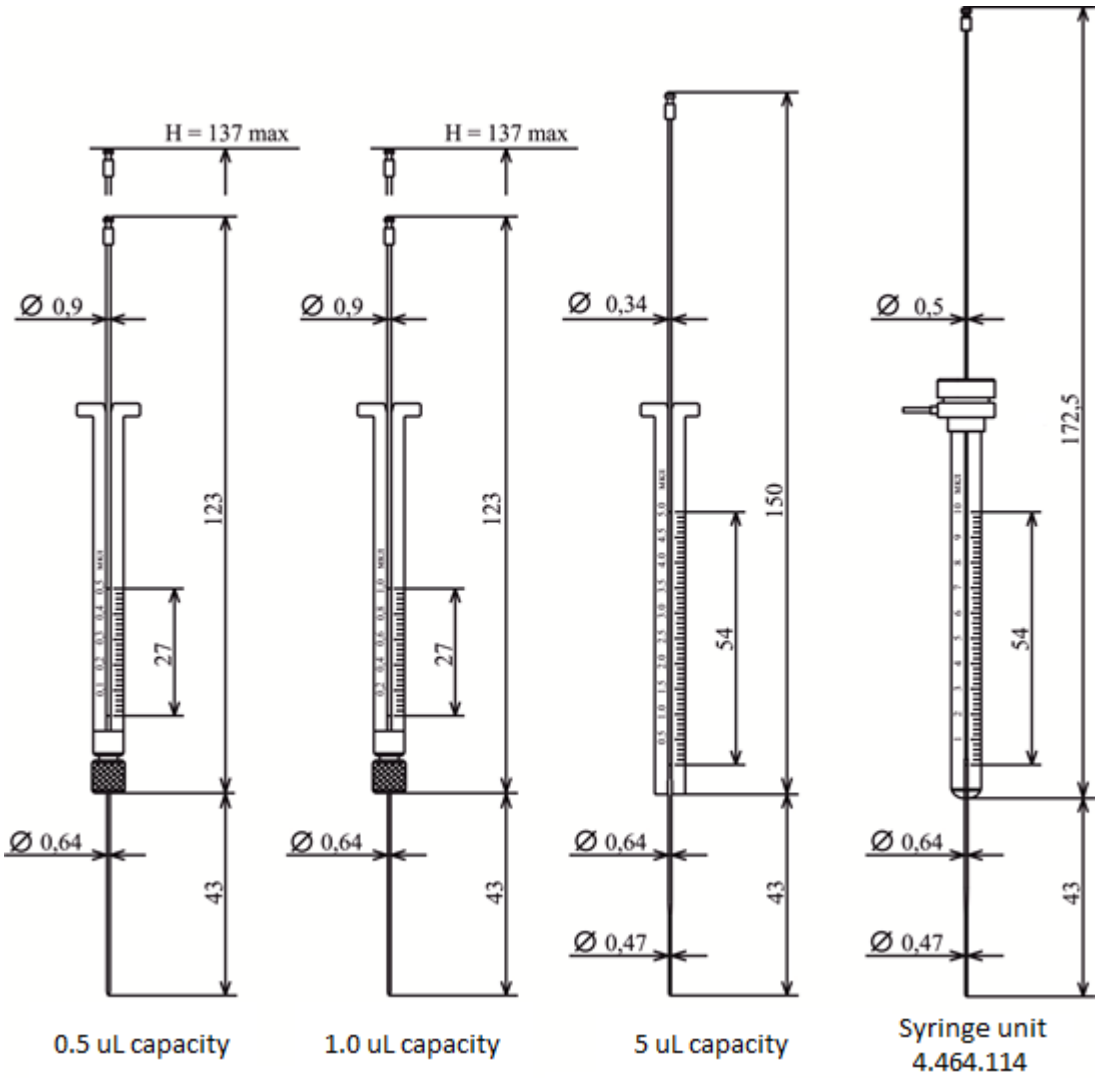


Figure 2.6 – Syringes types

2.4.4 Replaceable syringe unit and replaceable syringe adapters

The syringe unit and microsyringes are installed in special replaceable adapters. Each adapter corresponds to a specific type of guide (Figure 2.8).

There are four mounting holes and 2 magnets on the base of the syringe gear (Figure 2.7). When installed in the autosampler, the four pins of the holder (or thermostat) align with the mounting holes. The magnets secure the holder in the autosampler.

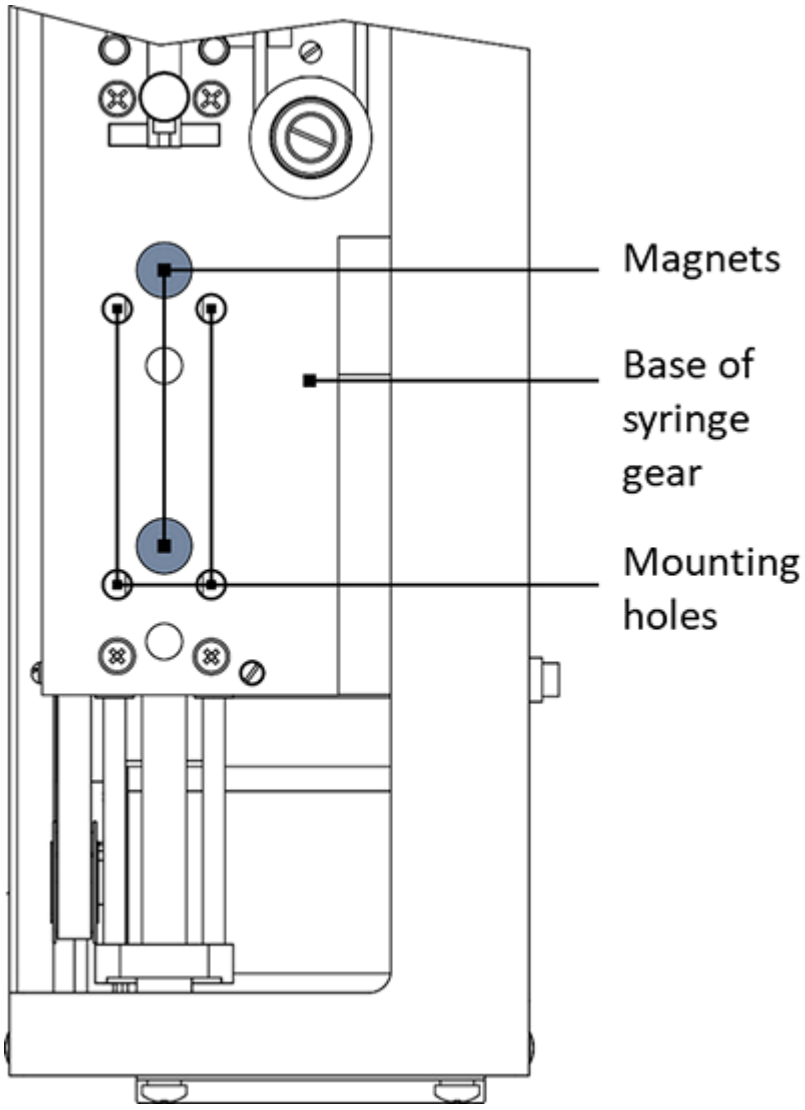


Figure 2.7 – Installation of adapters

Replaceable adapters serve for installation of various syringes into the autosampler.

4 syringe adapters are:

- Adapter 1 6.452.016 (for syringe unit 4.464.114;);
- Adapter 1 6.452.012 (for syringes 0.5; 1.0; 5.0; 10 µL, supplied in Liquid option set);
- Adapter 6.452.013 (for syringes 100 and 250 µL for ambient headspace applications, optional, not supplied on default);
- Adapter 6.452.017 (for SPME holder).

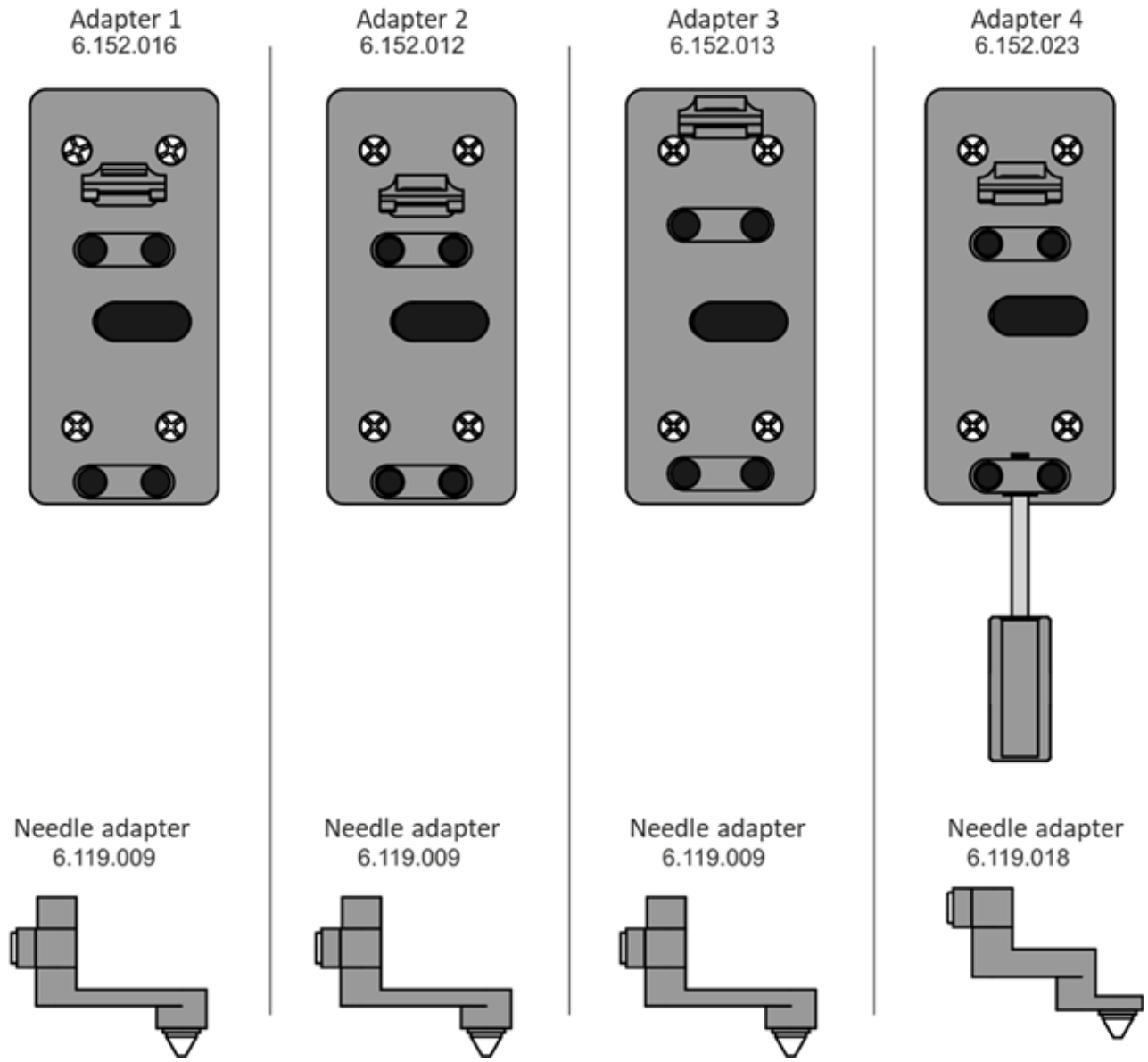
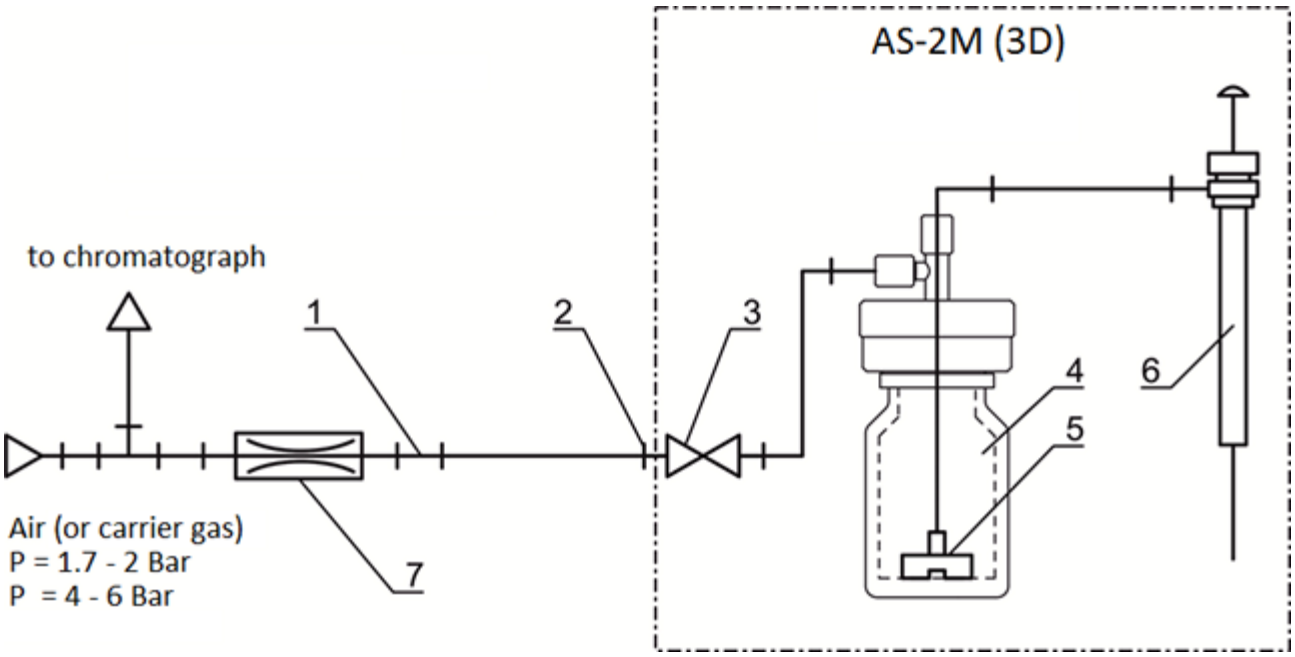


Figure 2.8 – Replaceable adapters

2.4.5 Washing unit

The washing unit is a 30ml glass bottle filled with solvent. In washing mode, compressed air or carrier gas flow passing solenoid valve delivers into bottle (see pneumatic diagram, Figure 2.8). Due to overpressure created in the vial (about 0.015 MPa), the solvent moves from bottle and goes over teflon tube to fitting 4 of syringe unit (Figure 2.5). If the end of the plunger 5 moves above top end of syringe barrel (plunger in washing mode position, valve switching on), the solvent goes through the channel of the syringe glass barrel and the needle discharging into waste vial. By this way plunger tip, syringe channel and needle channel are washed by solvent thoroughly. Insignificant volume of solvent that flows upward through the sealing, is not a failure attribute of syringe unit.



1 – tube 6.457.109 (assembly); 2 – "GAS IN" fitting; 3 – solenoid valve; 4 – solvent bottle; 5 – filter 5.884.087; 6 – syringe unit; restrictor 5.150.015 (for 1.7 – 2 bar) or 5.150-015-02 (for 4 – 6 Bar).

Figure 2.8 – Connection diagram for sampler and compressed gas source

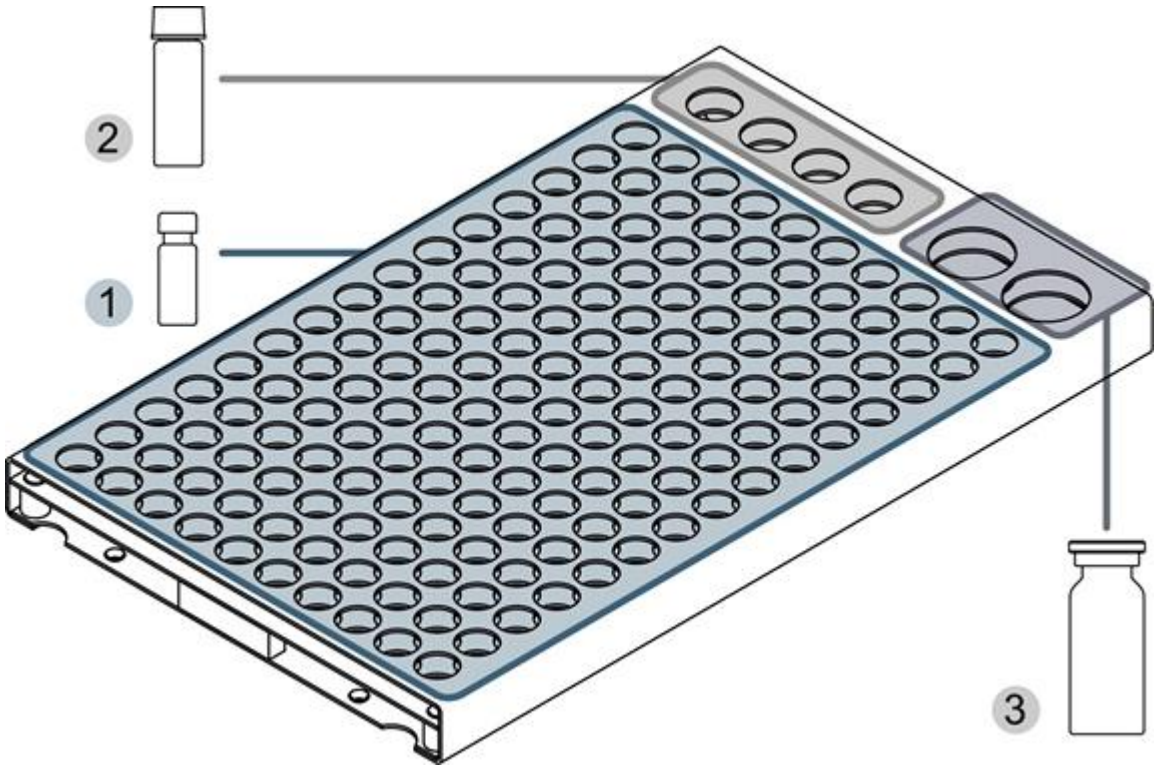
Tube 6.457.109 (assembly) contains the default pneumatic restrictor 5.150.015 (20 mL/min at 100 kPa, Nitrogen, labeled "015"). Use this assembly to connect gas lines at 0.17 to 0.2 MPa (e.g., from a Chromatec compressor). Replace the pneumatic restrictor in the assembly with 5.150.015-02 (20 mL/min at 400 kPa, Nitrogen, labeled "015-02") if the inlet pressure is 0.4 – 0.6 MPa.

2.4.6 Syringe gear drive

The syringe gear drive contains a stepper motor driving a flat-tooth belt connected to the syringe gear. Syringe gear position (upper, intermediate and lower) is controlled by optoelectronic sensor 6 (Figure 2.4).

2.4.7 Replaceable tray

Replaceable tray 15 (Figure 2.1) for vials is located on the basement, its positions is fixed by vertical stands. The tray contains numerical seats (1 – 150) for sample vials (2 ml), seats for solvent vials A, B, C, D (4 ml) and 2 widest seats for waste vials 15 ml (Figure 2.).



1 – sample vials (2 ml, up to 150 pcs.); 2 – solvent vials (4 ml, 4 pcs); 3 – waste vials (15 ml, 2 pcs.)

Figure 2.10 – Replaceable tray

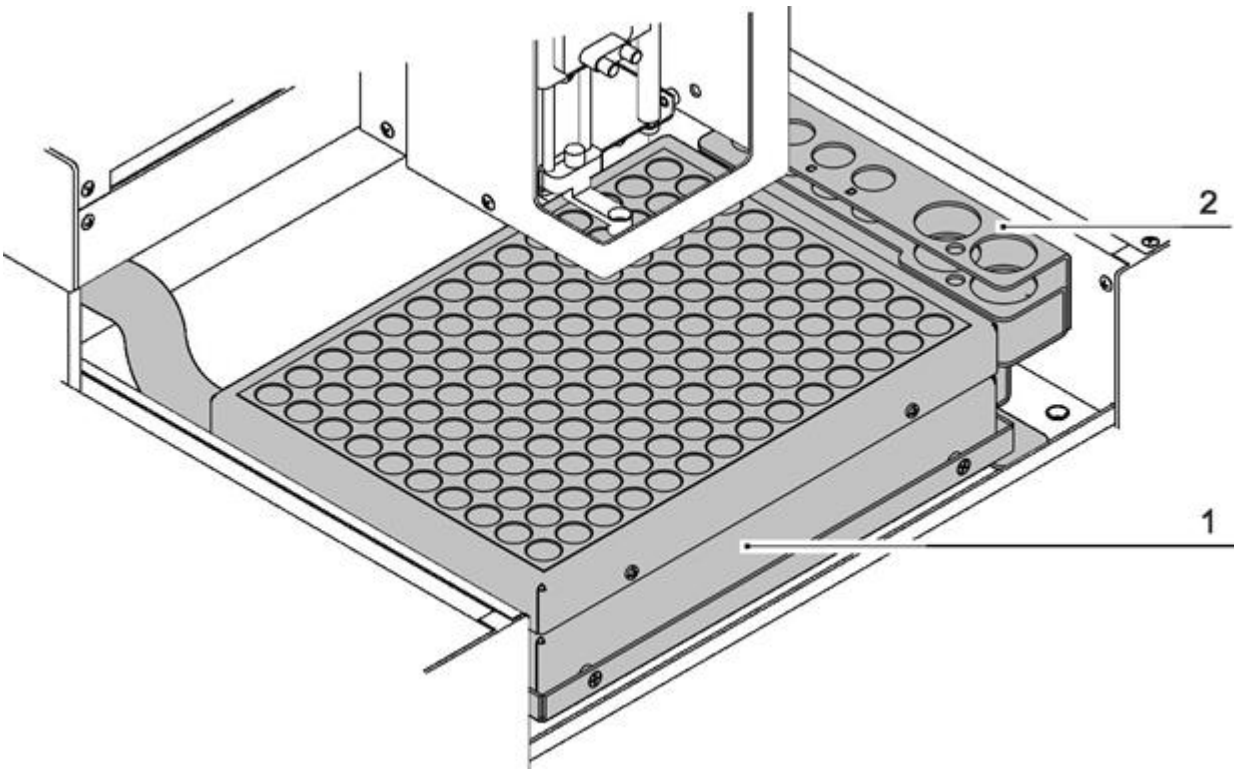
The syringe gear 1 with the syringe unit 5 (or microsyringe) installed therein is capable of being moved horizontally in two mutually perpendicular directions by two electromechanical drives.

In this case, the syringe gear 1 can occupy the following positions:

- positions of sample injection into the chromatograph inlets (up to three). The syringe gear can inject the sample into any of these inlets;
- washing position. This is one of six possible positions at which:
- one of the two flush vials is located under the microsyringe needle of the syringe unit;
- one of four vials (A, B, C, D) is positioned under the microsyringe needle of the syringe unit to allow the microsyringe to be washed "from the vial";
- sampling position. This is one of 150 possible positions where one of the sample vials is located under the microsyringe needle.

2.4.8 Temperature controlled tray

The temperature-controlled tray is designed for cooling/heating and subsequent temperature control of sample vials as part of the autosampler.



1 – Temperature controlled tray for vials with samples; 2 – Tray for vials with solvents and for draining the washing liquid

Figure 2.11 – 2.4.8 Temperature controlled tray 214.5.868.103

Technical specifications

- Number of sample vials: 140 pcs.
- Recommended refrigerant temperature: +4 to +60 °C.
- Maximum refrigerant temperature: +80 °C.
- Temperature gradient: max. 1 °C.
- Automatic tray recognition in the control panel.

Use only ethylene glycol or propylene glycol as refrigerant. It is permissible to use distilled water as a temporary substitute.

Do not use untreated tap water.

It is permissible to operate the temperature controlled tray at temperatures below +4 °C and at sub-zero temperatures using ethylene glycol or propylene glycol as the refrigerant.

As a cryothermostat it is recommended to use the device by LOIP FT 205-25. Technical specifications, device and operating conditions are available at https://loip.ru/catalog/laboratornoe-oborudovanie-proizvodstva-loip/laboratornye-termostaty-i-kriostaty/nizkotemperaturnyie-termostatyi-serii-loip-ft/kriotermostat-zhidkostnyy-ft-205-25/.

3 Operation modes

The autosampler operation mode represents a set of stages (commands) that are executed in series.

Depending on function, all commands in sequence are divided into following groups:

- Pre-injection syringe washing (this includes consequently washing by solvent, washing by sample, stroking by sample)
- Sample priming
- Sample injection
- Post injection syringe washing (syringe washing by solvent and liner washing by solvent can be applied optionally).

Steps responsible for syringe washing and liner washing can be included into sequence or canceled.

3.1 Pre-injection syringe washing

3.1.1 Solvent washing – "Top washing" mode

This mode is available for 10µL syringe unit only.

Only ethyl alcohol is recommended to use for top washing mode. Other solvents may damage solenoid valve in washing unit system. In case other solvents applied, "washing from vial" is recommended to use.

If any other solvents are required, the "from vial" washing mode must be used.

If the autosampler's sequence includes the "Simple sampling type and "Sandwich plus solvent" modes at the same time, it is not recommended to use the "top" washing mode.

In this washing mode the operator sets the washing time. When setting up a "Simple", "Sandwich" sampling types – the washing liquid can be discharged into any of the three vials. When setting up the sampling type "Sandwich plus Solvent", vials A and B are used, and the drain vial can be any of them as well.

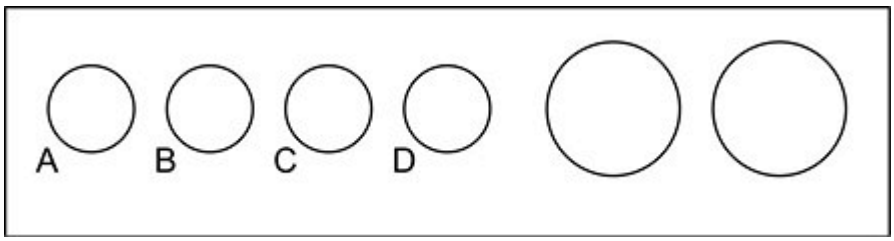


Figure 3.1 – Location of solvent vials (including internal standard) and flush vials for draining flushing fluids.

If the sampling mode is "simple" or "sandwich" when washing "top washing", the drainage is carried out sequentially into each of the service vials.

"Top washing" mode algorithm:

- Syringe gear moves to washing position (syringe needle is located right above any of waste vials);
- syringe needle moves down into waste vial;
- syringe plunger moves up to washing position (plunger tip locates above end of syringe barrel);
- solenoid valve opens channel of compressed air (or gas) to solvent bottle and simultaneously closes connecting channel between bottle and environment; solvent under pressure goes to syringe unit from above;
- after set washing time the solenoid valve stops gas flow, vial air space is connected to atmosphere; washing terminated;
- plunger moves downwards to zero position;
- syringe needle moves away from vial.

Recommended washing time is between 3 and 10 seconds.

3.1.2 Solvent washing – “From vial A” and “From vials A&B”

In this mode, the operator sets the number of drains.

Washing from vial A algorithm:

- syringe gear moves to position A;
- syringe needle enters solvent vial (vial A) to specified depth;
- syringe plunger moves to position corresponding to current solvent volume (minimum at 30% of syringe full capacity);
- syringe needle moves away from vial;
- syringe gear moves to waste position (1 or 2, figure 3.1);
- syringe needle enters drain vial;
- syringe plunger moves to lower position (solvent draining);
- syringe needle moves away from vial;
- steps 1-8 will be repeated according to user-selected washing cycles.

The "from vials A and B" mode involves washing the microsyringe with two different solvents.

Washing from vial A and B algorithm:

- syringe gear moves to position A;
- syringe needle enters solvent vial (vial A) to specified depth;
- syringe plunger moves to position corresponding to current sample volume (minimum at 30% of syringe full capacity);
- syringe needle moves away from vial;
- syringe gear moves to waste position (1 or 2, figure 3.1);
- syringe needle enters waste vial;
- syringe plunger moves to lower position (solvent draining);
- syringe needle moves away from vial;
- steps 1-8 will be repeated according to user-selected washing cycles;

- syringe gear moves to position B;
- syringe needle enters solvent vial (vial B) to specified depth;
- syringe plunger moves to position corresponding to current solvent volume (minimum at 30% of syringe full capacity);
- syringe needle moves away from vial;
- syringe gear moves to waste position (1 or 2, figure 3.1);
- syringe needle enters waste vial;
- syringe plunger moves to a lower position (solvent draining) at set flush speed;
- syringe needle moves away from vial;
- steps 1-8 will be repeated according to user-selected washing cycles.

Lower the needle into the drain vial to approximately ¼ of the vial height. The lowering level of the needle is chosen higher than during sampling to prevent the drain flushing liquid from getting on the needle.

The recommended minimal number of washing cycles is 3.

3.1.3 Air flushing mode for syringes 100 and 250 µl

Algorithm:

- syringe gear moves to sampling position for specified sample vial;
- syringe plunger moves up with specified speed to position corresponding to specified sample volume (If the specified volume is less than 50% of the microsyringe volume, then regardless of this, the sample volume to be washed will be equal to 50% of the total microsyringe volume.);
- plunger keeps set position within viscosity delay;
- syringe plunger moves down to lowest position;

Steps 2-4 will be repeated according to user-selected washing cycles.

3.2 Sample washing

Sample washing is not available for any "Sandwich", "Sandwich plus solvent" or "Sandwich with internal standard" injection mode specified

In this mode, analyst can select number of washing cycles and sample volume to be aspirated.

Sample washing algorithm:

- syringe gear moves to sampling position of selected sample vial;
- syringe needle enters sample vial to specified depth;
- syringe plunger moves with specified speed to position corresponding to specified sample volume (at least 50% of total syringe volume);

- syringe needle moves away from vial;
- syringe gear moves to position of a waste vial (1 or 2, figure 3.1);
- syringe needle enters a waste vial;
- syringe plunger moves to lower position (sample draining);
- syringe needle moves away from vial;
- steps 1 – 8 will be repeated according to user-selected washing cycles.

3.3 Sampling

3.3.1 Algorithm of “Simple” sampling mode.

- syringe gear moves to sampling position for specified sample vial;
- syringe needle enters sample vial to specified depth;
- syringe plunger moves to position corresponding to specified volume with specified aspiration speed;
- plunger keeps set position within viscosity delay;
- syringe plunger moves to lowest position;
- steps 3-5 repeat the user-configured number of plunger strokes;
- syringe plunger moves to position corresponding to specified volume with specified aspiration speed;
- plunger keeps set position within viscosity delay;
- syringe needle moves away from vial.

Recommended modes during sampling and sample injection.

Number of strokes during sampling from 7 to 10.

Sampling rate from 1 to 3.

Rate of sample injection into the inlet from 4 to 9.

Total sample volume for microsyringes:

- 0.5 µl volume, not more than, µl 0.25
- 1.0 µl volume, not more than, µl 0.5
- 5.0 µl volume, not more, µl 2.5
- 10.0 µl volume, not more, µl 10.0

3.3.2 Algorithm of "Sandwich" sampling mode:

In this mode **Top air plug** is air gap between plunger tip and sample, "**bottom air plug**" is air gap between sample and needle.

- syringe plunger moves to position corresponding to specified volume of Top air plug;
- syringe gear moves to sampling position for specified sample vial;
- syringe needle enters sample vial to specified depth;

- syringe plunger moves with aspiration specified speed to position corresponding to specified Sample volume + Top air plug volume;
- plunger keeps set position within viscosity delay;
- syringe needle moves away from vial;
- syringe plunger moves with specified aspiration speed to position corresponding to specified Top air plug volume + Sample volume + Bottom air plug volume.

3.3.3 Algorithm of "Sandwich with solvent" sampling mode:

In this mode **Top air plug** is air gap between plunger tip and solvent, **Middle air plug** is air gap between solvent and sample, **Bottom air plug** is air gap between sample and needle

When implementing the "solvent sandwich" sample injection mode, it is recommended:

- Aspiration speed is recommended to set 1 for this sampling mode;
- microsyringe to wash in "from vial" mode.

Algorithm:

- syringe plunger moves to position corresponding to specified volume of top air plug;
- syringe gear moves to solvent sampling position (specified vial A, B, C or D);
- syringe needle enters solvent vial to specified depth;
- syringe plunger moves with specified aspiration speed to position corresponding to specified solvent volume + top air volume;
- plunger keeps set position within viscosity delay;
- syringe needle moves away from vial;
- syringe plunger moves to position corresponding to specified top air volume + solvent volume + middle air volume;
- syringe gear moves to sampling position for specified sample vial;
- syringe needle enters sample vial to specified depth;
- syringe plunger moves with specified aspiration speed to position corresponding to specified top air volume + solvent volume + middle air volume + sample volume;
- syringe needle moves away from vial;
- syringe plunger moves with specified aspiration speed to position corresponding to specified top air volume + solvent volume + middle air volume + bottom air volume.

3.3.4 Algorithm of "Sandwich with internal standard" sampling mode:

In this mode **Top air plug** is air gap between plunger tip and internal standard, **Middle air plug** is air gap between internal standard and sample, **Bottom air plug** is air gap between sample and needle.

When implementing the "solvent sandwich" sample injection mode, it is recommended:

- Aspiration speed is recommended to set 1 for this sampling mode;
- microsyringe to wash in "from vial" mode.

Algorithm:

- syringe plunger moves to position corresponding to specified volume of top air plug;
- syringe gear moves to internal standard sampling position (dedicated vial with internal standard);
- syringe needle enters vial with internal standard to specified depth;
- syringe plunger moves with specified aspiration speed to position corresponding to specified solvent volume + top air volume;
- plunger keeps set position within viscosity delay;
- syringe needle moves away from vial;
- syringe plunger moves to position corresponding to specified top air volume + int.standard volume + middle air volume;
- syringe gear moves to sampling position for specified sample vial;
- syringe needle enters sample vial to specified depth;
- syringe plunger moves with specified aspiration speed to position corresponding to specified top air volume + int.standard volume + middle air volume + sample volume;
- syringe plunger moves with specified aspiration speed to position corresponding to specified top air volume + int.standard volume + middle air volume + bottom air volume
- syringe needle moves away from vial;

3.4 Sample injection into inlet

Sample injection algorithm:

- the tower moves to a position directly above selected inlet;
- syringe needle enters the inlet to specified depth;
- specified pre-injection delay takes place (if set above 0);
- syringe plunger moves to lower position (autosampler injects sample);
- specified post-injection delay takes place (if set above 0);
- syringe needle moves away from the inlet.

If Pre-injection delay and Post-injection delay is 0, the maximum time period of the needle being in inlet is not more than 0.2 sec

3.5 Post-injection solvent washing

This procedure is similar to that described in section 3.1.1

When analyzing alcohol-containing liquids (ACL), the liner can be washed with up to 5 µl bidistilled water after each working analysis.

4 Preparation before operation

Before proceeding with the autosampler preparation, make sure that autosampler and chromatograph are disconnected from power supply. Also check that gas lines are closed.

Table 4.1 – Main step of autosampler installation

Step	**Preparation procedure**
Step 1. Gas Chromatograph preparation	1. Installing nut and septum on GC inlet
	2. Check for leaks
Step 2. Autosampler mounting	3. Assembling and installation of the autosampler on support
	4. Cables connection
	5. Gas connection
Step 3. Vials loading	6. Tray installing
	7. Vials loading (solvent vials, waste vials, sample vials)
Step 4. Coordinate tuning	8. Inlet coordinate tuning
	9. Solvent vial coordinate tuning
	10. Sample vial coordinate tuning
	11. Waste vial coordinate tuning

Detailed procedure for each step is described below.

4.1 Installing nut and septum on GC inlet

Follow procedure:

- Remove nut from chromatograph inlet;
- Place new septa and screw special nut 6.482.013-03 (for liquid option, from AS shipping kit), on the inlet instead of standard nut, use new OV Grey Septa (or BTO equivalent) when nut replacing.

Use the following procedure when installing new nut and septa:

- install the nut as far as to eliminate any play of threaded connection;
- slightly tighten the nut by rotating it clockwise to 270 degrees. After that septum (silicone rubber) should significantly deform; if these instructions are followed, the "nut – septum" connection may be considered as sealed provided that other connections are sealed;
- for capillary inlet, set the carrier gas parameters (pressure, rate or flow) and split ratio parameters. Then, observe the measured values of carrier gas flow and gas flow over vent line on GC display to check the inlet line; numerical difference between specified flow rates may be considered as leakage indicator of inlet line with good septum;
- loosen the nut until play appears in threaded connection. Then, tighten nut-adapter on inlet to eliminate this play;
- pre-tighten the nut by rotating it clockwise to 60-90 degrees. After that septum must slightly deform;
- penetrate the septum with needle 6.054.005 (included to shipping kit);
- check the inlet line and observe the flow rate values of input carrier gas and vent flow; if numerical difference between specified flow rates is the same, this means that inlet line with penetrated septum is adequately sealed. If this difference exceeds 0.1ml/min or higher, this means leakage in septum (i.e. extra gas flows through the septum);
- slightly turn the nut to fix it in such a position to ensure minimal torque and no leakage.

4.2 Inlet leak check

To check the septum tightness, the difference of measured values of gas flow rates is evaluated in the "Control panel" program – the operator evaluates the value of the parameter **Total Flow** for the interested inlet. It should not exceed the value of the parameter allowable flow (Figure 4.1). Once new septa installed, analyst should check the difference.

The operator estimates the flows difference after the new septa is installed.

The inlet considered as leak-free, if flow rate difference between input gas and vent gas at "Ready" state does not exceed that fixed for new septa in the same GC operating conditions.

While operating, analyst shall periodically check the leakage in capillary inlet line (approx. each 30–40 septum punctures) according to above procedure.

If any leakage exceeding 0.1 ml/min is revealed, recover tightness by turning nut-adapter clockwise to approx. 30 degrees.

Perform these actions whenever leakage detected until turning nut-adapter against stop.

If all recommended instructions are observed, the septum service life may extend to 200 punctures (or several thousands in certain cases).

To avoid syringe needle damage and decreased septum service life, tighten the nut-adapter with minimal torque enough to ensure no leakage in the inlet.

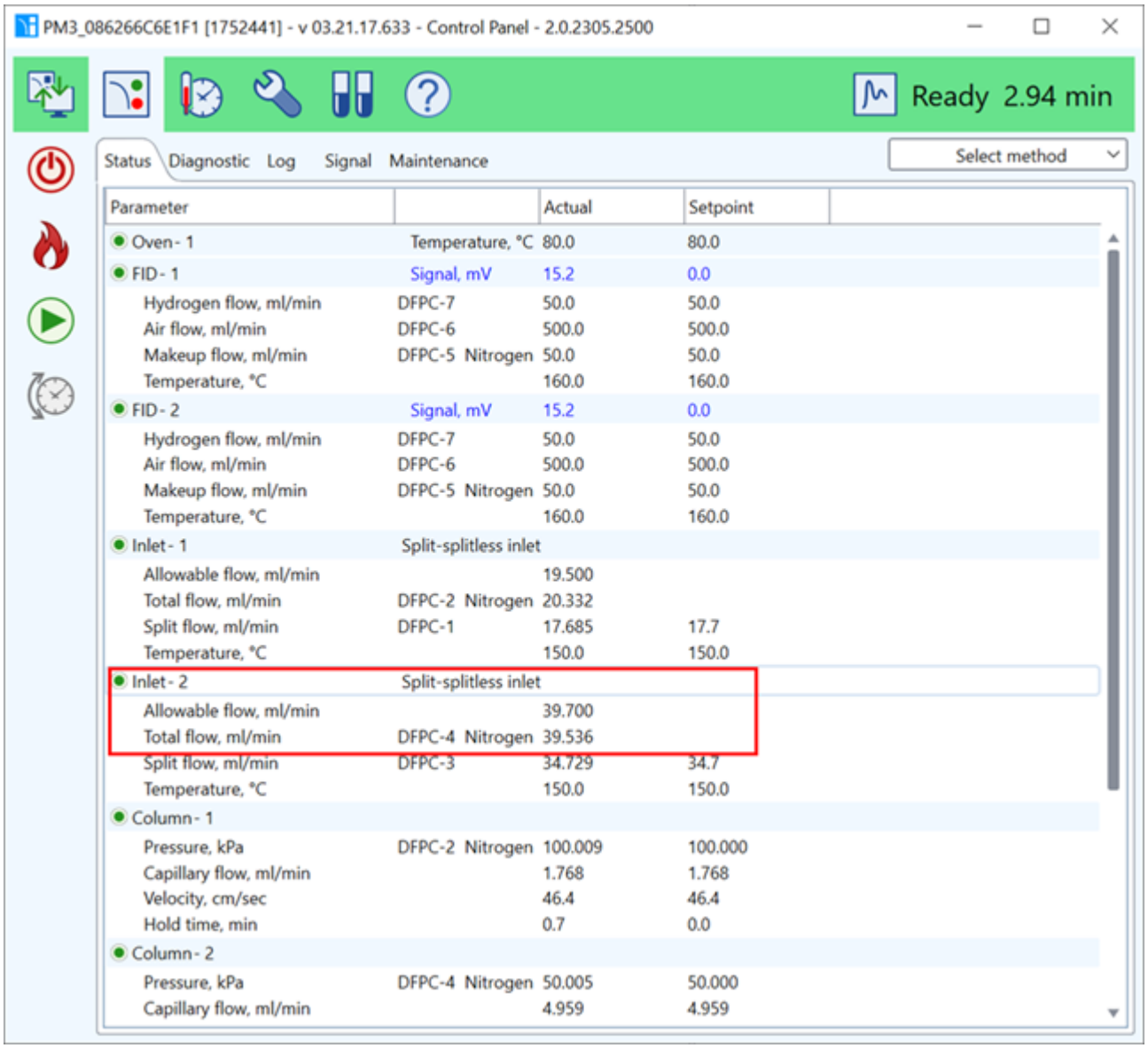


Figure 4.1 – Determination of inlet septum tightness in PM3

In case of excessive torque applied to nut-adapter (septum is compressed too much) or when using syringe with damaged needle, the septum service life may decrease to 5 punctures.

The inlet septum life can be increased if when replacing the liner, the adapter with the nut installed on it is removed (only the lower nut – adapter fixing is unscrewed), as this operation does not disturb the leak tightness regulation of the "adapter – septum" pair.

4.3 Autosampler installation

4.3.1 Support assembling and autosampler fitting to support

Refer to manual 6.410.021I for assembly, installation and adjustment of the support.

4.3.2 Cables connection

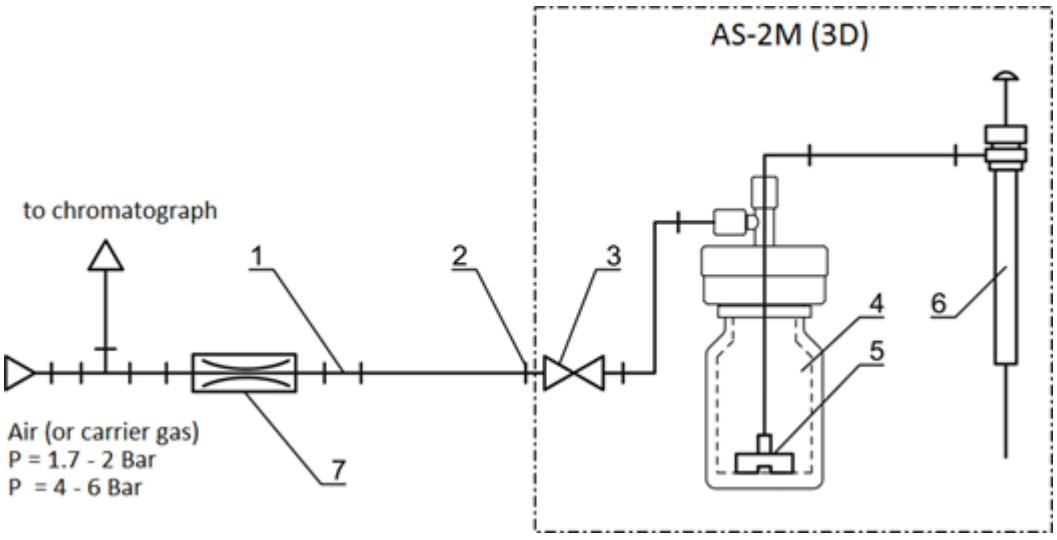
Cables required for connection are supplied in shipping kit.

- Use RS-232-25/25 SCB-139 cable from shipping kit to connect the autosampler to slot "AUX" of gas chromatograph.
- Connect the power cord SCZ-1 to power mains.

4.3.3 Gas connection to the autosampler

Connection of the autosampler to the gas source is only necessary when using the syringe unit in conjunction with top washing mode.

- Remove the plug from the washing unit connection .
- Connect to the compressed air (or gas) line using the tube 6.457.109 from the shipping kit – according to the diagram (Figure 4.2). When using a gas source with a pressure of 0.17 – 0.2 MPa (e.g., from a Chromatec compressor), pneumatic resistance 5.150.015 labeled "015" (20 ml/min at 100 kPa, Nitrogen) is used; if the inlet gas pressure is 0.4 – 0.6 MPa, pneumatic restrictor 5.150.015-02 labeled "015-02" (20 ml/min at 400 kPa, Nitrogen) is used. In this case, the end of the tube with a tee and pneumatic restrictor is connected to the compressed air (or gas-) line.



1 – tube 6.457.109 (assembly); 2 – "GAS IN" fitting; 3 – solenoid valve; 4 – solvent bottle; 5 – filter 5.884.087; 6 – syringe unit; restrictor 5.150.015 (for 1.7 – 2 bar) or 5.150-015-02 (for 4 – 6 Bar).

Figure 4.2 – Autosampler connection to gas source

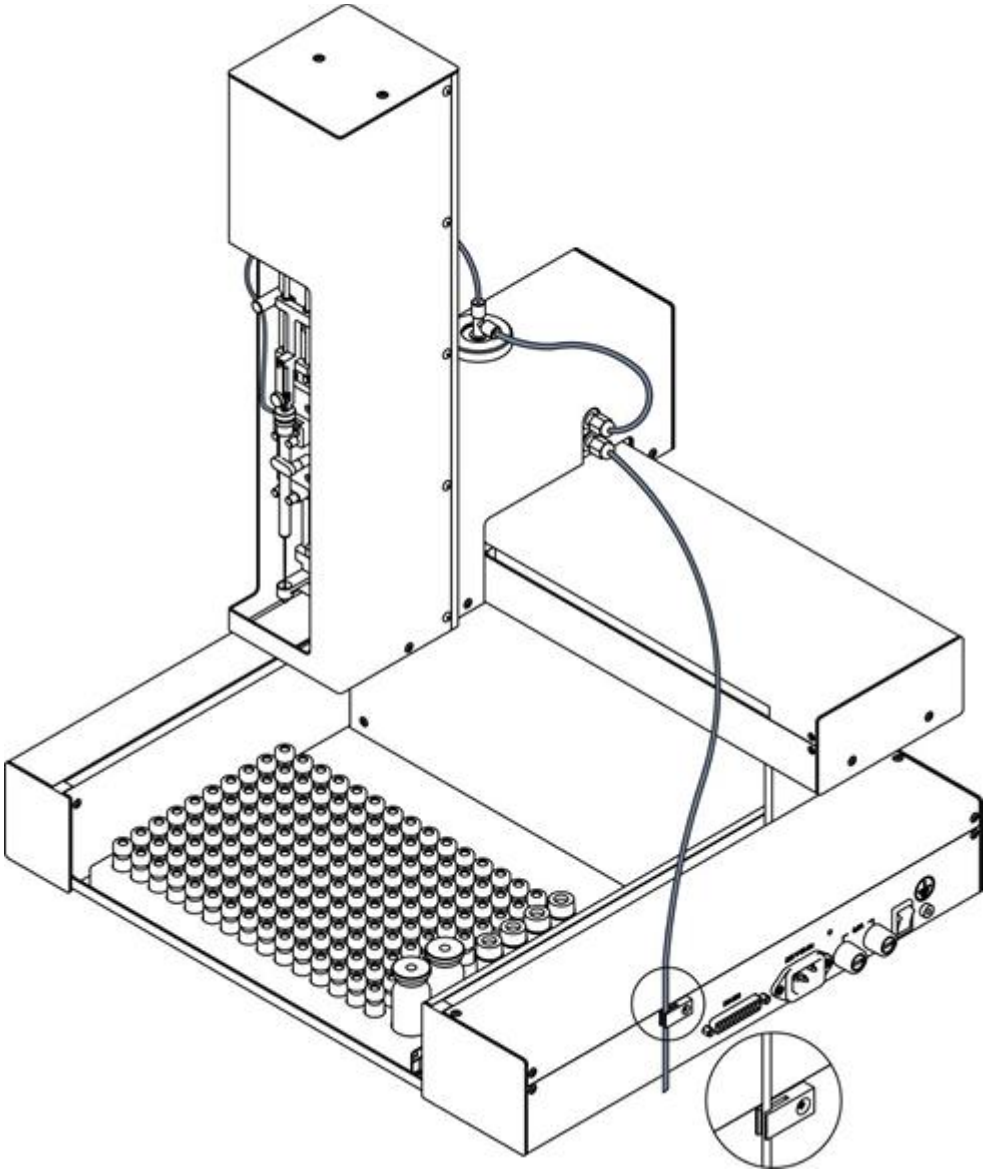


Figure 4.3 – Fixing the tube

Turn off the Autosampler before fixing the tube.

To fix the tube on the Autosampler wall follow:

- Move tower to a distant position as shown in figure above;
- Fix the tube so that it was loosened at a distant position of the tower;
- Shifting the tower to all extreme positions, make sure that tube doesn't touch movable parts of the autosampler, if necessary, change the fixed point of the tube.

4.3.4 Tray installation

To install the tray 214.6.150.308 it is necessary to align the slots on its wall with the pins in the autosampler base. Then gently lower the left part of the tray (Figure 4.4)

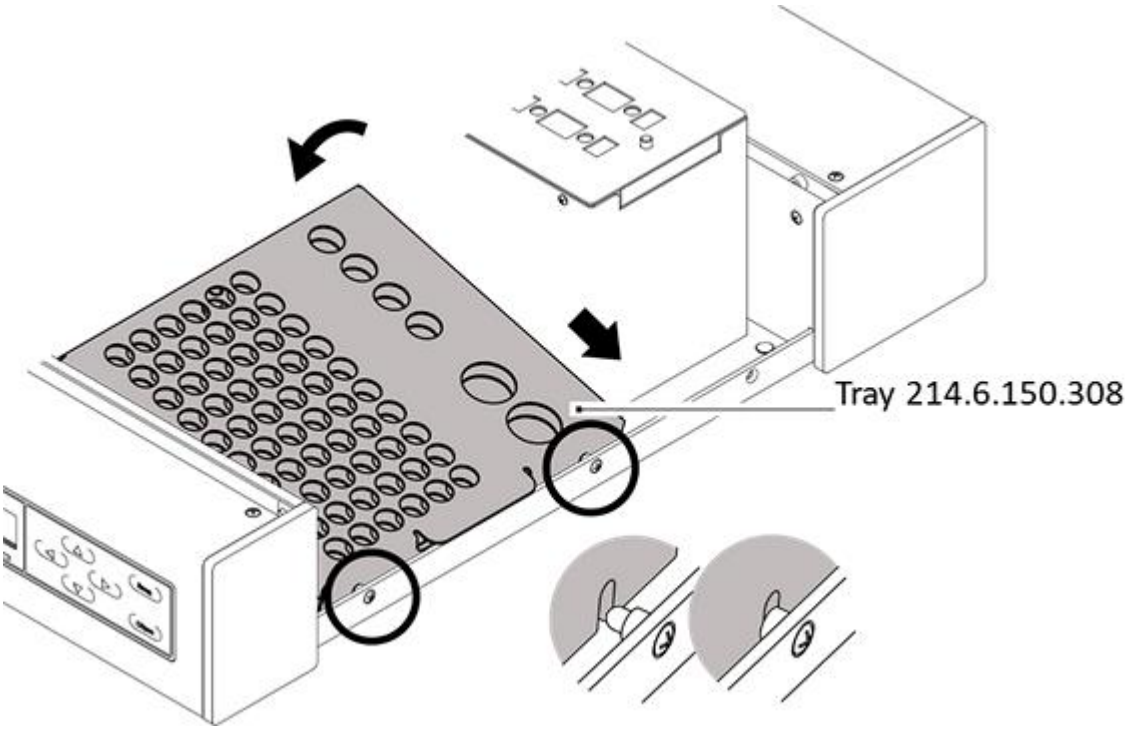


Figure 4.4 – Tray installation

4.3.5 Installation of a temperature controlled tray

Make all connections with the equipment unplugged.

Install the solvent and waste vial stand in the autosampler as shown in Figure 4.5.

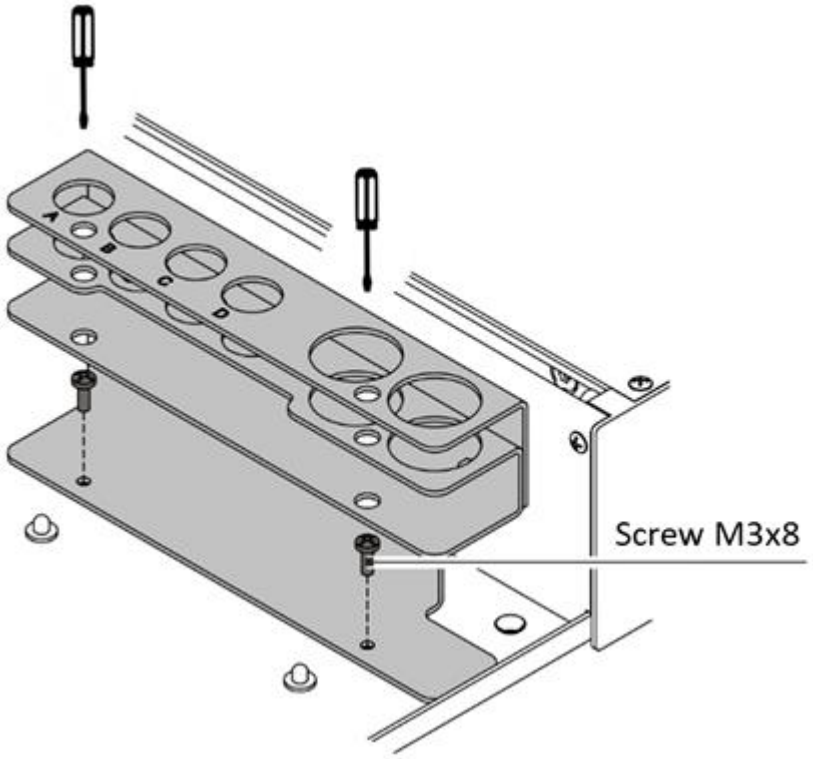


Figure 4.5 – Installing the stand for solvent and waste vials

Install the tray on the autosampler, positioning the refrigerant supply lines and drain tube as shown in Figure 4.6.

When moving the autosampler platform, make sure that the lines do not interfere with its movement.

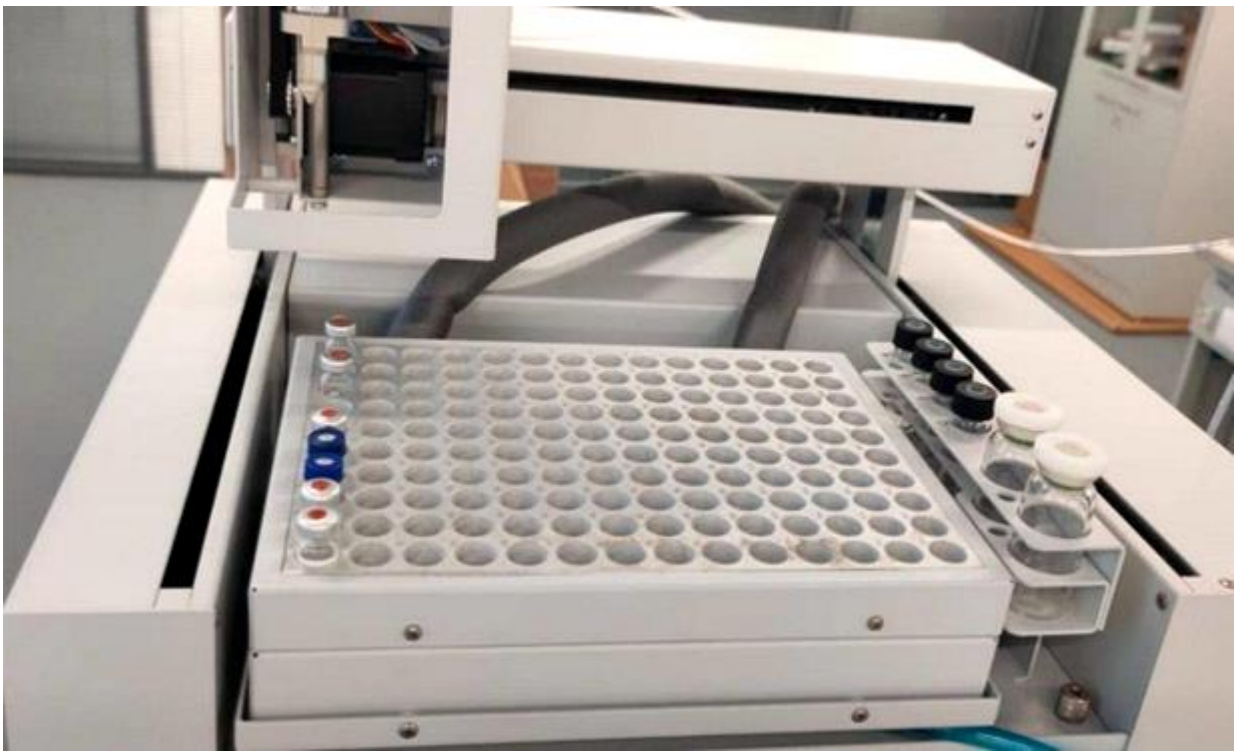


Figure 4.6 – Location of refrigerant lines and drain tube

Make sure the plastic tube is securely connected to the drain fitting at the back of the tray (Figure 4.7). The tube should allow easy drainage of condensate from the temperature controlled tray without restriction. Ensure the following:

- the tube is sloped toward the drain fitting;
- there are no bends that could obstruct the flow of water;
- the open end of the tube is not submerged in the collected water of the drainage tank.

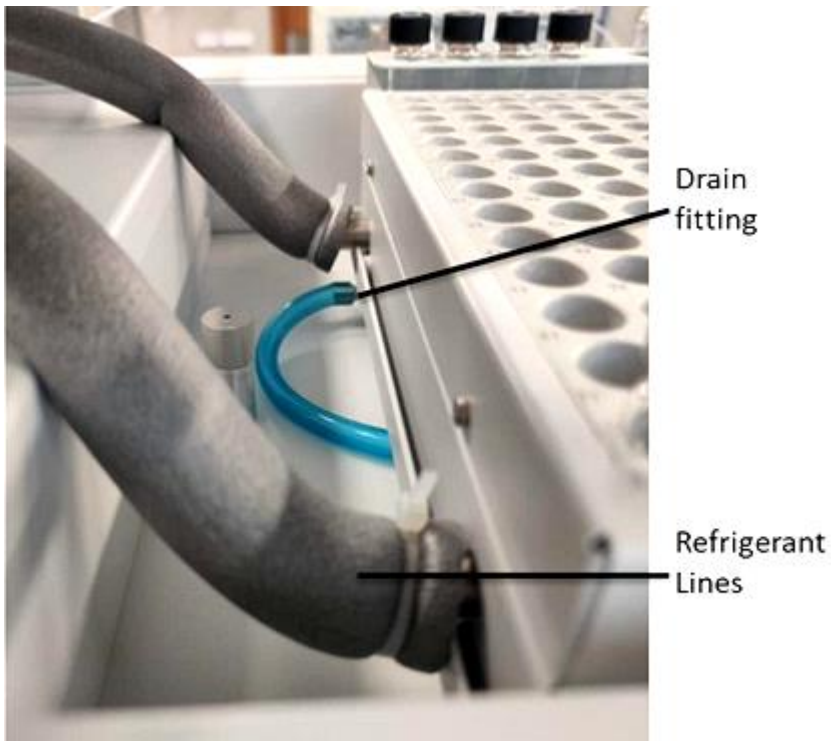


Figure 4.7 – Connection of drainage tube to fitting

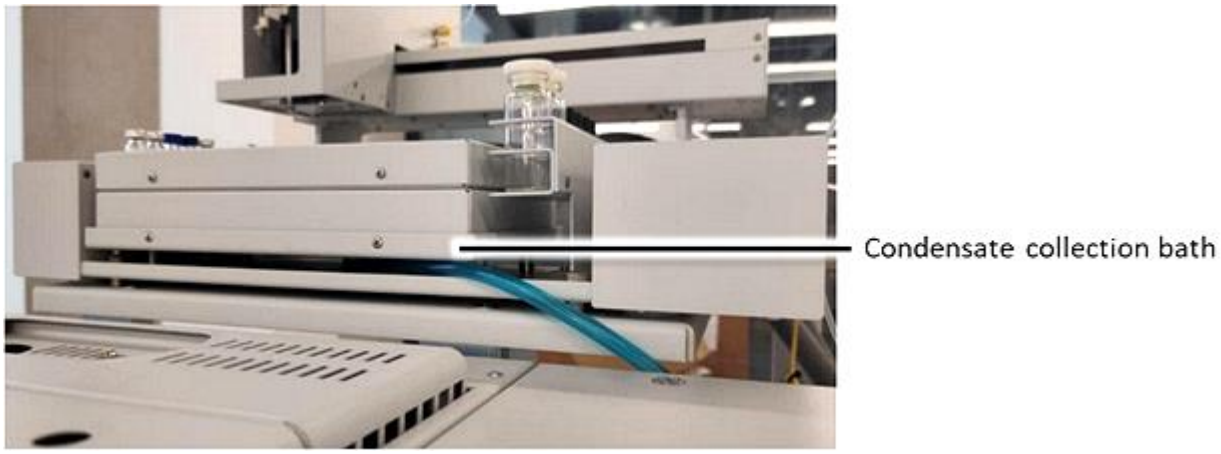


Figure 4.8 – Location of the drain tube

If possible, remove all equipment from under the autosampler with temperature-controlled tray or take measures to protect the equipment under the tray from possible leaks. If a low sample temperature is maintained in a high-temperature or high-humidity environment, water condensation from the temperature-controlled tray in the event of an accidental leak can damage the equipment under the tray.

Regularly check the tubes, drain tube and condensate collection bath for signs of leakage due to material fatigue (cracks). Check that there is no moisture under the tray on the surface of the metering unit.

Do not put the appliance into operation if there are signs of leaks (liquid under the bath or at the tubes connections).

4.3.6 Preparing to install a microsyringe or syringe unit

According to section 2.4.4 determine the type of adapter. Install the required adapter (1, 2 or 3) on the magnets in the syringe gear (Figure 4.9).

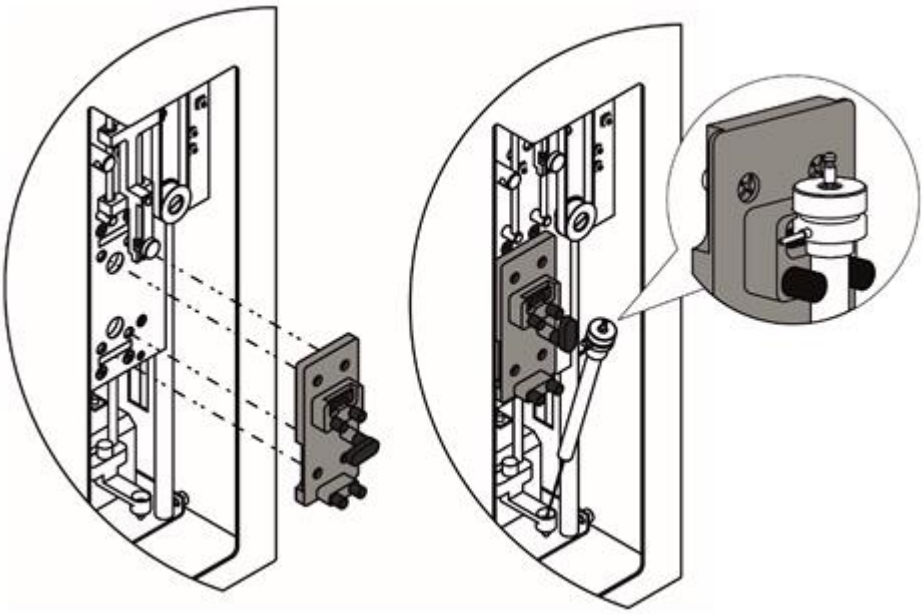


Figure 4.9 – Installing the adapter and syringe unit

Specifics of preparation of microsyringe installation with 51 mm needle length:

Install the adapter 5 (6.152.023) on the magnets in the syringe gear (Figure 4.10);

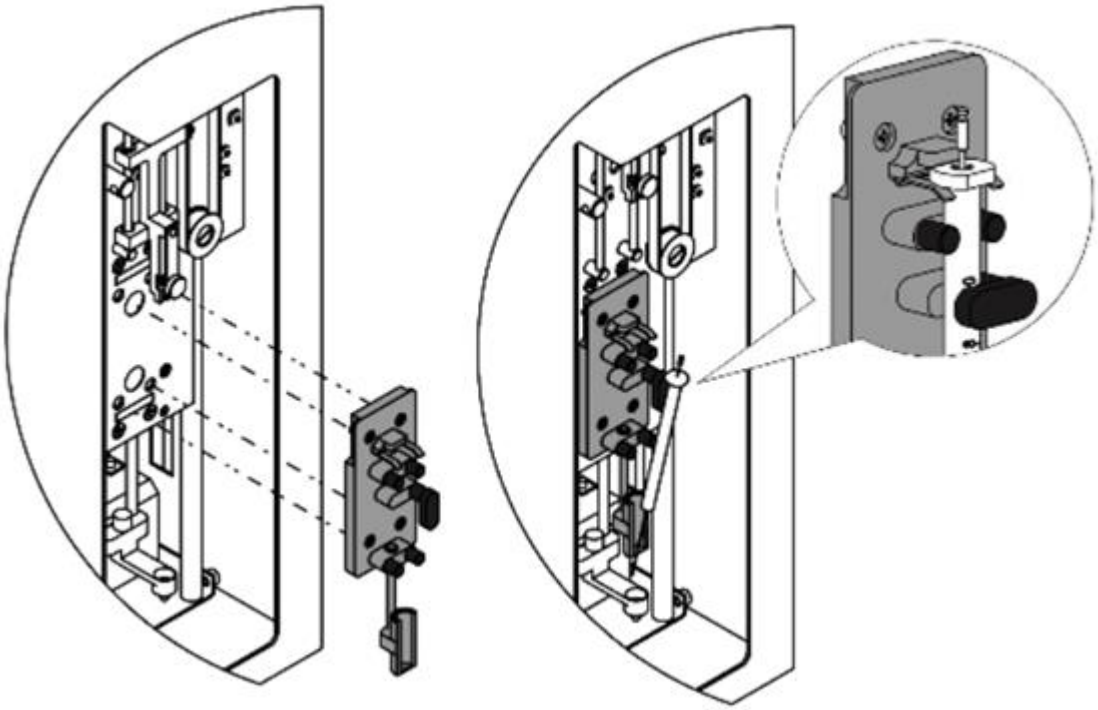


Figure 4.10 – Installing the microsyringe adapter 4.464.119 (or 4.464.128) and 4.464.123

Move the autosampler tower as far forward and towards yourself as possible; lower the syringe gear carriage to the lower position (Figure 4.11).

Remove needle adapter 6.119.009 by loosening two screws with an angle wrench (hexagon) from the shipping kit (Figure 4.12-a). Install needle adapter 6.119.018. When tightening the guide mounting screws, lift it with one finger by 1... 2 cm (Figure 4.12) and tighten it with a wrench without applying excessive force (use a short arm of the wrench).

Check the ease of sliding by pressing the guide 6.119.018 from below. If the needle adapter rubs or jams, loosen the retaining screws and repeat the installation procedure.

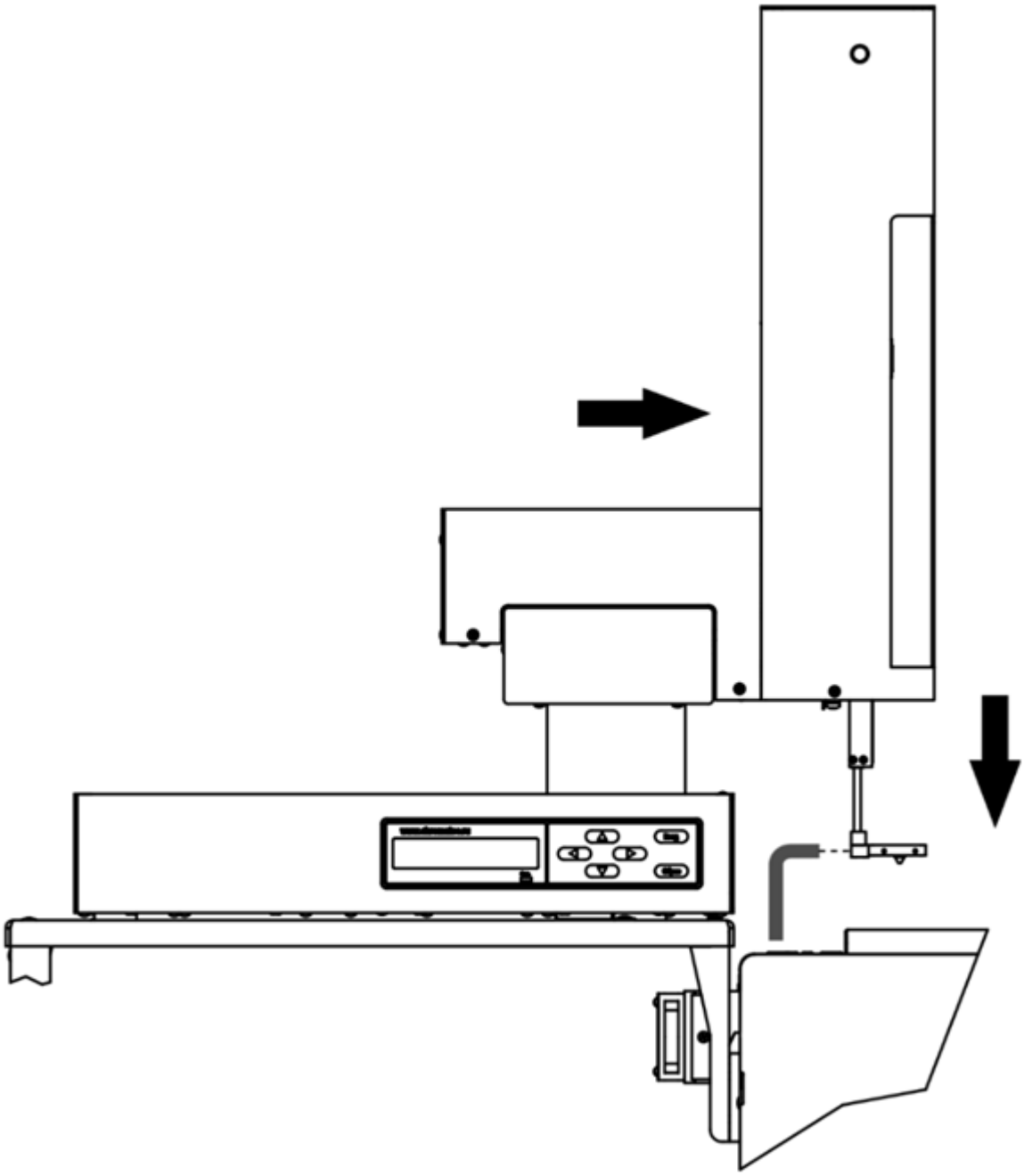


Figure 4.11 – Position of the autosampler "tower"

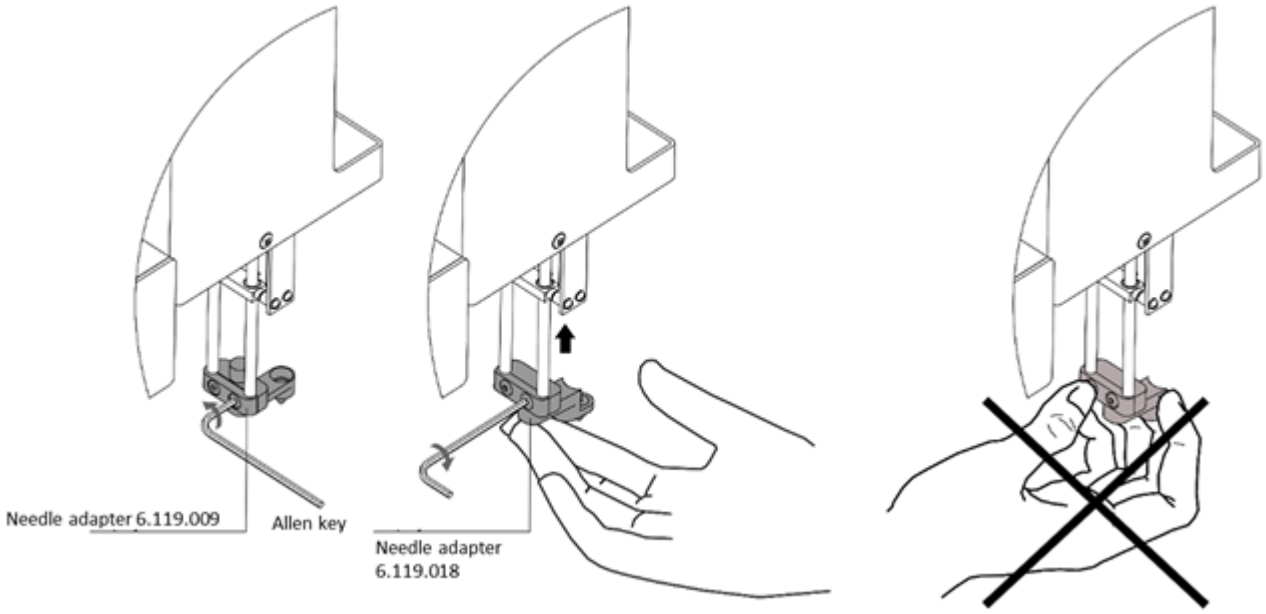


Figure 4.12 – Installing the syringe needle adapter

4.4 Inlet coordinate tuning

- Switch on the power supply to the autosampler and the chromatograph (can be switched on in any order). Operational information on the autosampler status is shown on the display. The autosampler and chromatograph must be connected with a data cable.
- Use the display and buttons on the front panel of the Autosampler to adjust coordinates. Using buttons ▼▲ pass to menu Setting.
- Select option Number of inlets and, using buttons ◀ ▶, specify number of GC inlets to inject in.
- Pass to menu Inlet 1 position.
- The message shall be displayed: Touch Inlet 1 nut.
- Move the tower to inlet position manually.

The tower must be moved lightly (drivers turned off). If movement is blocked by drivers, it means that program for setting coordinate was not applied. Turn the Autosampler off and then turn it on again in 5 seconds. The AS-2M (3D) parameter group should appear in the Chromatograph Status dialog box. Repeat the above steps.

- Manually move the syringe gear down so that needle adapter bottom tip inserted into cone of inlet nut. Fine tuning is not required on this step. Once needle adapter contacted the nut, remove your hands.
- In 3 seconds, the drivers of the Autosampler will turn on automatically and the carriage will raise slightly on 5 mm.
- Using buttons ◀ ▶▼▲ move the tower precisely at the inlet of the inlet nut.
- Set ENTER – apply.
- The Autosampler will provide computing this coordinate and checking accuracy of setting. As soon as the towers comes back to home position, Inlet 1 tuning is completed.
- Carry out tuning of all other inlets coordinates in the same way.

4.5 Sample vials coordinate tuning

Adjustment of sample vials coordinates is carried out by vial #1 position only.

- Place a caliber 8.126.085 (from shipping kit) into vial position #1 of the tray instead of sample vial.
- Using buttons ▼▲ pass to menu Setting.
- Select option Vial 1 position.
- The Autosampler will perform factory settings automatically. The tower must go down to contact the pattern.
- Using buttons ◀ ▶▼▲ match bottom tip of the needle adapter with a hole on the pattern.
- Set ENTER – apply.

It is unacceptable to use a vial with a deformed septum instead of a caliber when adjusting vial coordinates.

The caliber is installed in the tray with a smaller diameter hole upwards (Figure 4.13).

Incorrect calibration of vials leads to errors in autosampler operation.

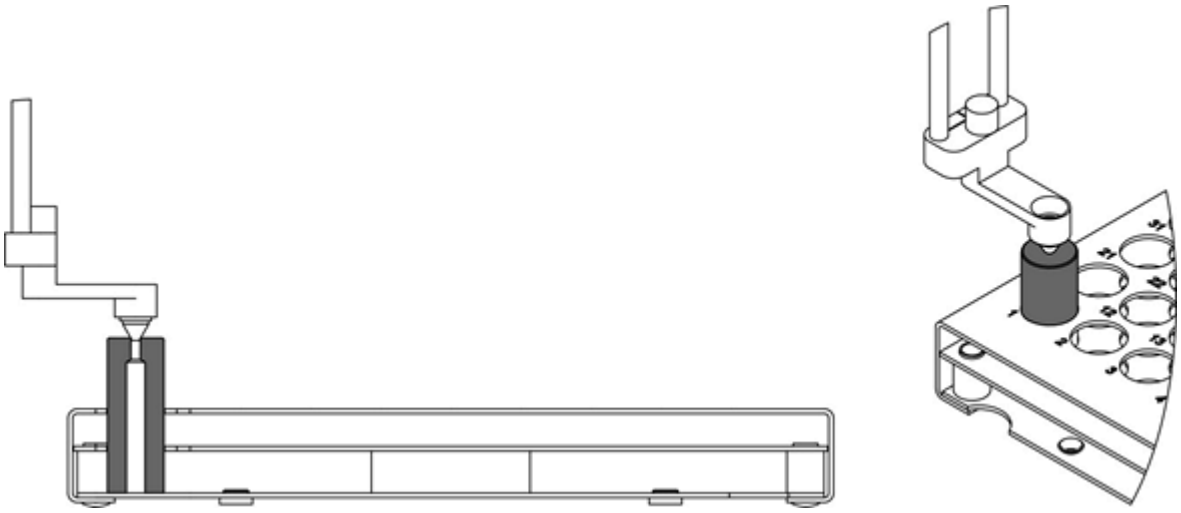


Figure 4.2 – Correct location of vial pattern

As soon as the tower comes back to home position, **sample vial** coordinate tuning is completed. **Remove the caliber from the tray after setting completed and place vial back.**

4.6 Solvent vials coordinate tuning

This tuning is required for some particular autosampler operating modes: washing "from vials" and sampling mode "sandwich with solvent".

- Turn on the autosampler and the GC. Autosampler's display reflects current status.
- Use the display and buttons on the front panel of the Autosampler to adjust coordinates. Using buttons ▼▲ pass to menu Setting.
- Select option Solvent A position.
- The Autosampler will perform factory settings automatically. The tower must go down to touch septa of solvent vial (A).
- Using buttons ◀ ▶▼▲ adjust coordinate of solvent aspiration point.
- Set ENTER – apply.

As soon as the tower comes back to home position, **solvent vial** coordinate tuning is completed.

4.7 Waste vials coordinate tuning

- Turn on the autosampler and the GC. Autosamplers display reflects current status.
- Use the display and buttons on the front panel of the Autosampler to adjust coordinates. Using buttons ▼▲ pass to menu Setting.
- Select option Waste position.
- The Autosampler will perform factory settings automatically. The tower must go down to touch socket of waste vial cap with bottom part of the needle adapter.
- Using buttons ◀ ▶▼▲ adjust coordinate of waste position.
- Set ENTER – apply.

As soon as the tower comes back to home position, **waste vial** coordinate tuning is completed.

4.8 Mounting/dismounting syringe units and microsyringes

Top washing Kit 214.4.069.056 includes syringe units 4.464.114

Mounting syringe unit or syringe:

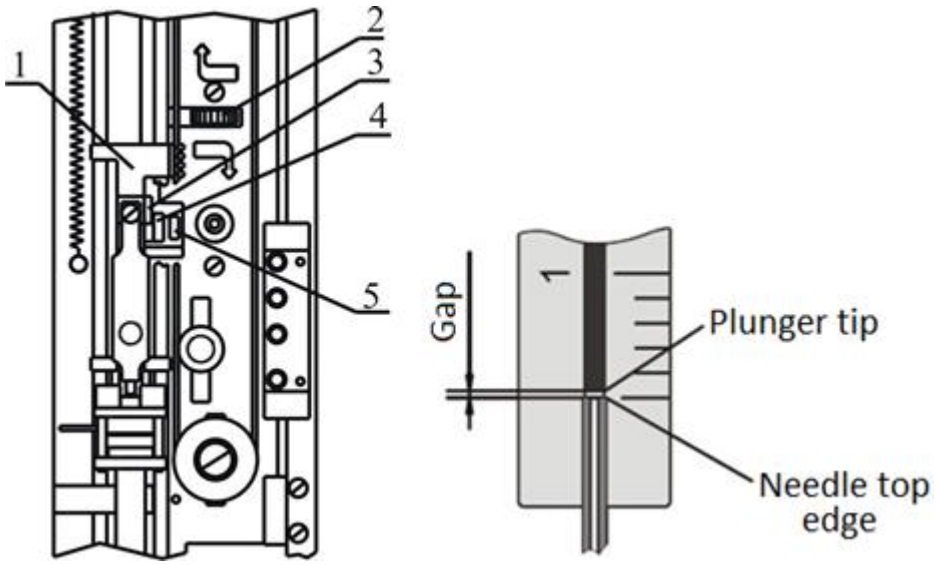
- Carriage 4 of syringe gear (Figure 2.4) should be shifted at 5 – 6 cm above its bottom position, and screw 11 released at 1–2 turns. Make sure that there is a gap between spring plate 10 and carriage body to insert bead of syringe plunger;
- Rotate a latch 5 of the syringe adapter 7 into vertical position;
- Fit syringe unit (or syringe) to adapter (Figure 2.4) and insert needle into hole of needle adapter. Then push glass barrel into seat up to the end, plunger should be inserted into glass barrel;
- Rotate the latch 5 into horizontal position to fix the syringe (Figure 2.4);
- Shift downward the carriage 4 until so that bead of the plunger went into gap between spring plate 10 and carriage body 4;
- Fix plunger bid by screw 11, do not make excessive force while rotating clockwise;

- For syringe unit: carefully connect PTFE capillary from solvent bottle to side located fitting of the syringe unit;
- with special care manually move the carriage 4 from lower position upwards against stop and return it to original position. This is required to check how light the movement of syringe plunger in glass body. If any evidence of blocking is revealed, remove the syringe unit from sampler and replace it with new one supplied in the shipping kit. The removed syringe unit should be repaired either by the user in accordance with the recommendations set forth in Section 6.1 of this manual or sent to the manufacturer for such work on a paid basis, regardless of the operating time of the autosampler.

The syringe unit is removed in reverse order.

When installing the microsyringe plunger, do not overtighten screw 11.

Once syringe unit (or syringe) is installed, proceed with adjustment of optoelectronic sensor of syringe plunger. Move the carriage 1 (Figure 4.3) with fixed syringe plunger head to lower position until syringe plunger tip affects needle top edge in the barrel. Then, slightly move the carriage 1 upwards to obtain the visual gap between needle top edge and plunger end. The gap value affects the repeatability and accuracy of analysis: more the gap, lower the repeatability and vice versa (Figure 4.3). Then, turn the screw 2 to move board with mounted optical encoder 4 slightly upwards and downwards to get a position where LED goes ON (left rotation of screw 2 corresponds to upward movement of board with encoder and vice versa).



1 – carriage; 2 – board adjustment screw; 3 – curtain; 4 – optical encoder; 5 – LED.

Figure 4.3 – Plunger position adjustment

4.9 Connecting washing tube to syringe unit

- Make sure that a tube from washing unit is threaded through a hole in guide 1 (Figure 4.4) of syringe gear. Spare length of the tube from the guide 1 to the end should be 75 – 78 mm.
- Fit end of the tube to syringe unit. The tube from rack 1 is directed vertically downward and has a small bend radius at the syringe unit connection. Avoid sharp bending of the tube while connecting.

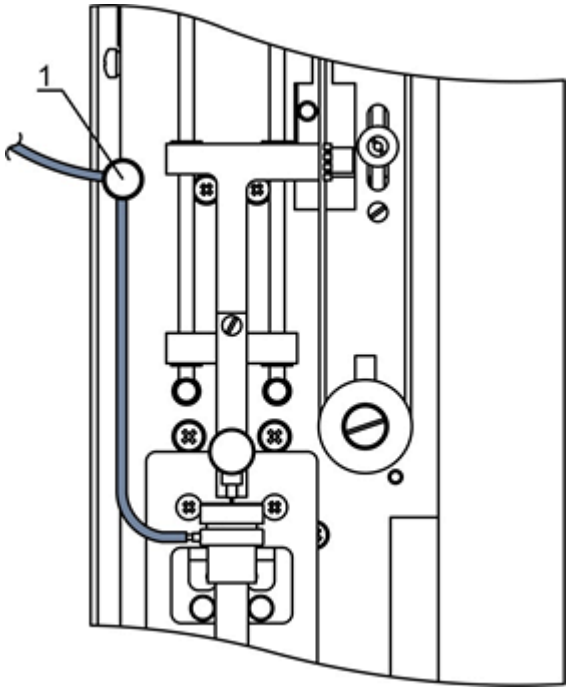


Figure 4.4 – Tube connection to the syringe unit

Do not allow excessive spare length of the tube (above 78 mm) as it will lead to catching of the tube in tower cover (Figure 4.5).

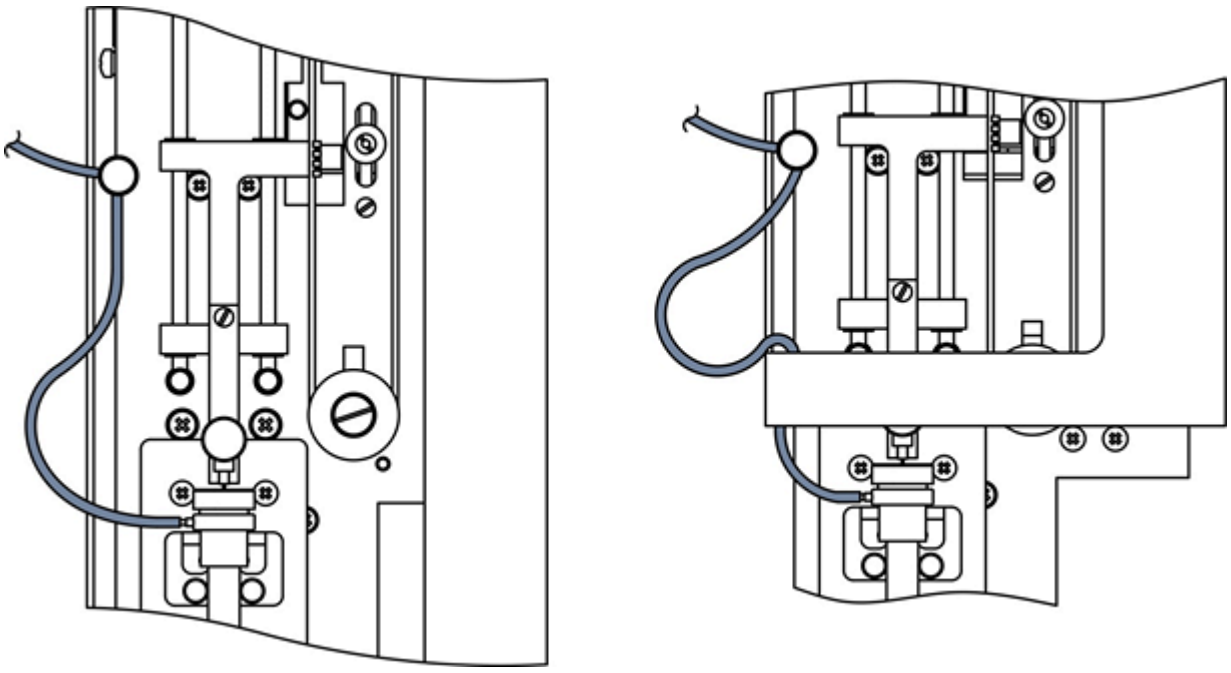


Figure 4.5 – Incorrect connection of washing tube to syringe unit

4.10 Completing the preparation for work

The passage of the microsyringe needle through the septum should be easy but not compromise the tightness of the inlet.

The vials and vial of the washing unit must be thoroughly washed and dried.

- Unscrew the plug (together with the connections) on the vial of the washing unit, pour the washing liquid (usually the solvent in which the sample is dissolved) into the vial, the vial is hermetically sealed with the plug;
- After that recheck the tightness of the septum and the inlet path as a whole according to the above described procedure;

The liquid sample injection into the inlet of gas chromatographs of " Crystal" series is carried out with the use of Hamilton or SGE microsyringes.

5 Autosampler operation

5.1 Recommendations for selecting the operating mode

Proper autosampler instrumental method allows for the best chromatographic analysis performance in terms of analytical accuracy and separation efficiency, while minimizing the influence of previous sample and syringe problems.

Typical recommended parameters are summarized below:

Table 5.1– Recommended parameters of the autosampler instrumental method

Operating parameter	**Value**
Solvent washing	
Washing time, sec ("top washing ")	4
Solvent volume, µl (washing mode "from vial")	3
Number of strokes (washing mode "from vial")	3
Sample washing	
Solvent volume, µl	3
Number of washing cycles	3
Sampling	
Aspiration speed	from 1 to 3
Number of strokes	from 7 to 10
Viscosity delay, sec	0 or 1
Sample volume, µl	0,2-2
Injection	
Pre injection delay, sec	0
Post injection delay, sec	0
Injection speed	From 4 to 9

Some details below may help to optimize autosampler operating parameters and improve chromatographic analysis results.

Sample volume
The volume of the sample to be injected is normally specified in the analysis procedure and should be selected so that the volume of sample vapor generated by heating in the inlet does not exceed the liner volume. For reliable operation of the piston (plunger), the maximum total volume drawn into the syringe:

- 0.5 µl volume, not more than, µl 0.5
- µl volume, not more than, µl 1.0
- 5.0 µl volume, not more, µl 2.5

- 10.0 µl volume, not more than, µl 7.0
- 100.0 µl volume, not more, µl 100.0
- 250.0 µl volume, not more, µl 250.0

Although the autosampler allows sampling volumes corresponding to the full 5 and 10 µl syringe scale, it is recommended to limit the maximum sample volume to the values above to avoid damage to the piston (plunger).

Influence of previous sample

If components from a previous sample appear in the chromatogram, increase the number of solvent wash cycles or the wash time.

When washing "from vial", the volume of solvent withdrawn during washing must exceed the volume of the sample to be injected.

Effect of viscosity and cavitation

When sampling in the syringe barrel, cavitation effects can occur under the piston (plunger), resulting in undesirable vapor bubbles in the syringe barrel. To reduce the likelihood of bubble formation, especially for light boiling liquids and viscous samples, reduce the Aspiration Speed and add a Viscosity Delay (for viscous samples).

Bubble Removal

Bubbles in the syringe barrel are "expelled" by stroking in combination with a low aspiration speed and a high injection speed. The number of strokes should be increased to remove bubbles.

Needle warm-up time before injection (post-injection)

Warming the needle prior to sample injection allows for more efficient vaporization of high-boiling sample components. Warming the needle after sample injection allows for more efficient removal of sample residue from the internal volume of the needle. Both options should be used in conjunction with the Sandwich or Solvent Sandwich sample injection technique, so that the air plug keeps the needle empty during the delay, this avoids fractionation of sample components.

Optimization

Different samples may require different optimal autosampler conditions. When selecting new autosampler parameters, various aspects of the new method should be evaluated: convergence of results in a series, influence of the previous sample, peak shape, possible fractionation of components, etc.

5.2 Using Scheduler

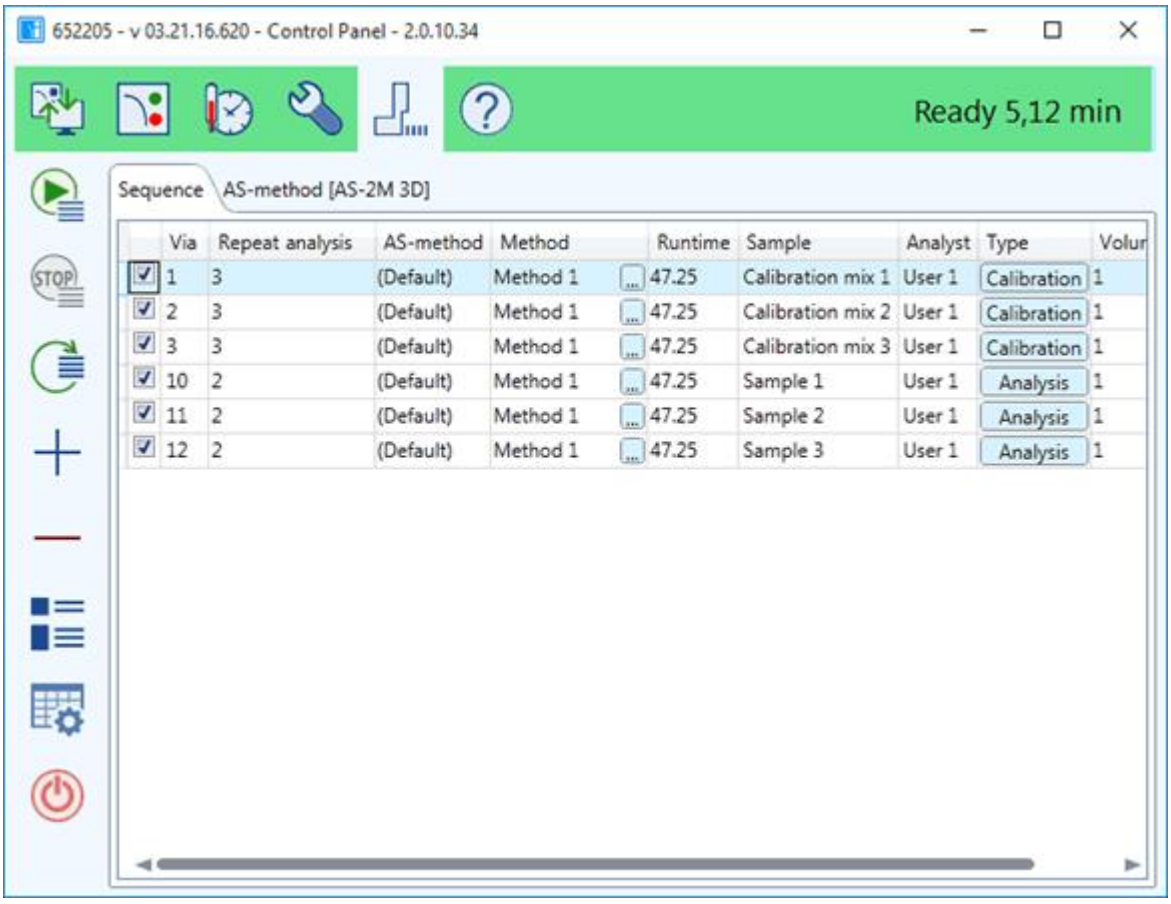
Autosampler operation is carried out via "Control panel 2.0" software. Before starting operation, it is obligatory to study "Chromatec Analytic" User Manual (214.00045-51I).

To control Autosampler operation and creating sequence select a tab **Sequence** in Control panel SW.

This section provides information on setting the autosampler instrumental method. Refer to Section 3.5 in the Chromatec Analytic User Manual for information on setting up the analysis sequence.

To add a new autosampler method:

- Click the Autosampler tab.
- In the list that appears, select – Create New.



Via	Repeat analysis	AS-method	Method	Runtime	Sample	Analyst	Type	Volur
1	3	(Default)	Method 1	47.25	Calibration mix 1	User 1	Calibration	1
2	3	(Default)	Method 1	47.25	Calibration mix 2	User 1	Calibration	1
3	3	(Default)	Method 1	47.25	Calibration mix 3	User 1	Calibration	1
10	2	(Default)	Method 1	47.25	Sample 1	User 1	Analysis	1
11	2	(Default)	Method 1	47.25	Sample 2	User 1	Analysis	1
12	2	(Default)	Method 1	47.25	Sample 3	User 1	Analysis	1

Figure 5.1 – Adding a autosampler method

Go to the tab **AS-method [AS-2M 3D].**

You can also create a new autosampler method on the **AS-method [AS-2M 3D]** tab using the corresponding button.

If you always work with the same autosampler method, you cannot create a new one, but go directly to the **AS-method [AS-2M 3D]** tab and set the default method. In this case in the **Method** cell, it will be written **(Default).**

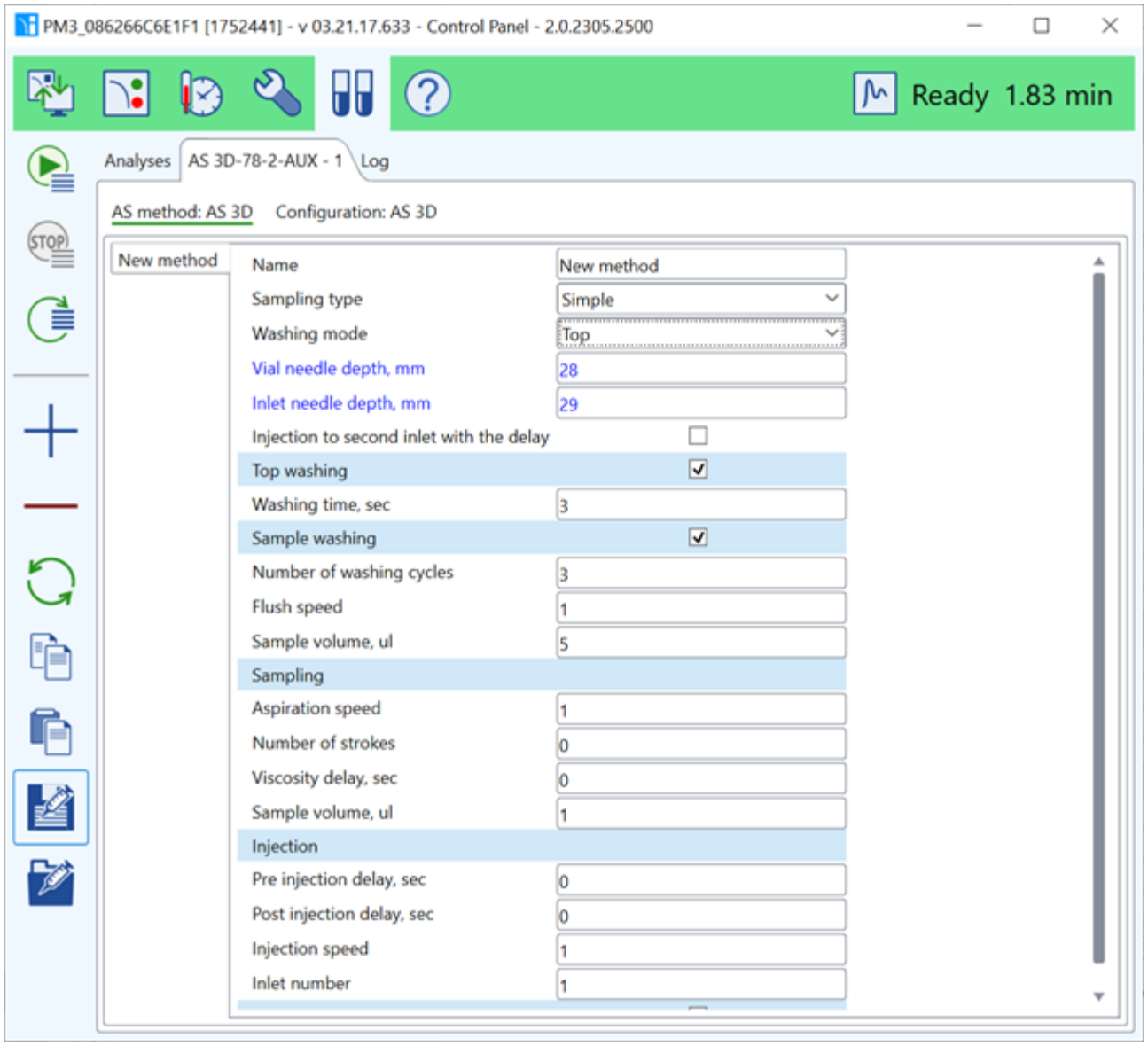


Figure 5.2 – AS method for AS-2M (3D)

In the **Configuration** parameter group, select **Syringe**.

Select Sampling type:

- Simple. Normal Sampling type. The number of strokes is available.
- Sandwich. The sample to be aspirates is enclosed in two air layers. The autosampler does not stroke during sampling.
- Solvent sandwich. First the autosampler draws air ("upper"), then solvent (from a given vial), then air ("middle"), then the sample again air ("lower"). The autosampler does not stroke during sampling.

- Sandwich with internal standard. First the autosampler takes air ("upper"), then the internal standard (from the specified vial), then air ("middle"), then the sample again air ("lower"). The autosampler does not stroke during sampling.
- Multiple simple injection. Normal sampling. The number of strokes can be set. The number of injections can be set. The autosampler injects the sample from the same vial several times and then gives the START command.

Select Washing Mode:

- None. The autosampler does not perform solvent washing.
- From Vial. The autosampler washes from service vials A, B, C, and D. The operator can use both vials with solvent or just one. Set the volume of solvent to be withdrawn and the number of washings.

Set the **Vial needle depth** (in millimeters). The default setting is 28 mm. This is the maximum immersion depth. If your sample contains sediment at the bottom of the vial, you can reduce the needle plunge depth to prevent entering sediment from the vial.

Set the **Inlet needle depth** (in millimeters). The default setting is 29 mm. This is the maximum immersion depth. This parameter depends on the specifics of the analysis methodology and the location of the inlet liner packing.

Configure the **Solvent Washing** parameters. If solvent washing is not required, uncheck the box.

Configure the **Sample Washing** parameters. If this washing is not required, uncheck the box.

Sample Washing is available if **Washing Mode = From Vial**.

Configure the **Sampling** parameters.

Configure the parameters of Sample injection into the inlet.

For AS-2M (3D) autosampler the option **Sample injection with 2 inlets delay is available**. If it is enabled, the parameters appear in the Injection parameter group:

- Delay of the second injection (min). It starts after the first injection. After the delay expires, the autosampler injects the sample from Vial 2 (it is set on the Sequence tab) into inlet.

If a solvent washing after injection is defined, it is performed after the first injection and after the second injection. In this case, the delay countdown starts after solvent washing has been performed.

- Inlet number 2.

The inlet may be specified the same for both inlets if necessary.

The sampling and injection speeds are set in units from 1 (lowest) to 10 (highest).

Correspondence of the speed number to the actual value (µl/sec) is given in the autosampler operation manual.

When aspiration a volume (total volume including sample, solvent and air layers) of more than 70% of the syringe scale, the plunger downward speed is limited to 4th.

If you are working with several AS-2M modes, set the **Method Name**.

The autosampler method is saved automatically.

5.3 Features of sequence setting

The operator can customize the sequence in a simplified way by specifying the range of vials from which sample injections will be made.

Specify the range of vials with a hyphen. You can add individual vials by separating them with a semicolon. For example – 1-5;10. Injections will be made from vials 1 through 5 and from vial 10.

In this case, all analyses corresponding to this line will have the same instrumental methods, processing method parameters and chromatogram passports.

If an injection with a delay of two inlets is required, set Vial 2 for the second injection.



If the Cycle or Vial 2 columns are hidden, display them using the menu of this button.

5.4 Troubleshooting

Below (Table 5.2) are given possible malfunctions of the autosampler, which can be eliminated by the user, their causes and methods of elimination. In case of other malfunctions contact the manufacturer or his regional representative.

Table 5.2 – Troubleshooting

Problem	**Possible cause**	**Remedy action**
Syringe unit microsyringe plunger jams during operation (after storage)	Sediments in the gap between the piston and the channel in the microsyringe housing due to drying of sample residue	Replace the syringe unit or microsyringe. When storing the autossampler, the microsyringe piston should be maximally extended out of the channel
Repeatability of analysis sequence out of standard limits (peak height and (or) area)	Leak in the inlet	Retighten or change septa
	Leak in vial	Fit the insert 8.214.061 into vial cap, replace vial septum
	Incorrect operating mode: aspiration and injection rate; plunger strokes; washing	Change operating mode of the autosampler
	Liner configuration is not suitable for this sample	Replace the liner
	Wearing of syringe or syringe of syringe unit	Replace the syringe unit or syringe
Carryover. Chromatogram contains peaks relating to previous sample	Washing is not sufficient / efficient	Increase number of washing strokes (or washing time) / consider changing solvent
	Insufficient or failed washing of syringe channel due to lack of solvent, leakage or clogged filter 5.884.087 in bottle	Check the solvent level in bottle. If solvent level is too low, fill the bottle. Eliminate any leakage. Replace the filter 5.884.087
Solvent leakage in syringe unit and tube connections	Sampler input pressure exceeds the limit.	Follow instructions specified in section 4.3.3 referred to gas tube connection.
	Leakage in tube connections	Eliminate leakage in pipeline connections
	Teflon sealing if syringe unit worn out	Replace the syringe unit

5.5 Error messages

Error messages may appear when malfunctions occur in the autosampler. Error messages, typical causes, and remedies are listed below.

Table 5.3 – Error messages

Error message	Possible cause	Remedy action
01. No sensor position	One of the X-axis or Y-axis turret position sensors is defective	Contact the manufacturer or authorized representative
	Failure of one of the X-axis or Y-axis drive motors	
	Damage to the flexible loop from the control unit to the "tower" or actuator position sensors in the "X" or "Y" axis	
02. syringe plunger movement error	The gap between the needle and plunger end is not set	Adjust the piston position, see step 4.8.
		Clean the syringe according to section 6.4. If cleaning fails, replace the syringe.
	Damage to the flexible cable	Contact the manufacturer or authorized representative
	Syringe plunger drive malfunction	
03. Carriage upper position setting error 04. Error setting the middle carriage position 05. Error setting the lower carriage position	Syringe gear sensor malfunction	Contact the manufacturer or authorized representative
	Damage to the flexible cable from the control unit to the sensor	
	Malfunction of syringe gear drive motor	
06. inlet nut not found	Incorrect installation of the autosampler on the GC	Check the correct installation of the autosampler, see point 4.3.
	Faulty microswitch	Check the function of the microswitch. Contact the manufacturer for replacement.

6 Maintenance

The maintenance is to be provided to keep good performance of the Autosampler and meet to its technical specification during operation. At maintenance it is necessary to observe safety measures according to section 1.

The most critical parts in the Autosampler are syringe and syringe unit. Depending on its operating conditions, autosampler performance is determined significantly. That's why maintenance of syringe and syringe unit shall be especially carefully provided.

6.1 Recommendations for the use of microsyringes

6.1.1 Hamilton recommendations

Hamilton microsyringe application recommendations in English are enclosed with each microsyringe.

General guidelines for using microsyringes to liquid sampling.

When using a microsyringe, squeeze only the microsyringe tip and plunger head. This will prevent variations in liquid sampling accuracy due to body heat. Pump the liquid to be sampled with the plunger while the needle of the microsyringe is submerged in the liquid. This will expel any air trapped in the needle and microsyringe. Minimize the use of a "dry" microsyringe.

When moving a plunger that has not been wetted with liquid, it is possible that the plunger may " jam" in the barrel of the microsyringe. Therefore, if forces are encountered when moving a "dry" microsyringe piston, immediately moisten the microsyringe with solvent.

Excessive force when moving the microsyringe plunger should also be avoided, as this may cause the plunger to bend.

Each microsyringe is matched to the plunger to ensure the highest dispensing accuracy. Microsyringe plunges are not interchangeable.

If the plunger accidentally comes completely out of the microsyringe barrel, gently wipe it with a lint-free cloth before reinserting it into the barrel. Do not touch the plunger, as grease from fingers often interferes with reliable piston operation.

When sampling, please note that the greater the ratio of the total volume of the microsyringe to the volume of liquid to be sampled, the greater the sampling error. It is recommended to choose this ratio not more than 5-10.

Microsyringe washing

The life of a microsyringe is directly related to its cleanliness. To clean the microsyringe, it is best to use solvents that are most effective and preferred for sample dissolution and do not contain alkali, phosphate or detergent.

The microsyringe can also be rinsed with high purity water, acetone. Water and acetone are satisfactory washing agents.

To clean the plunger, remove it from the microsyringe barrel, and gently wipe it with a lint-free cloth. Re-insert the plunger into the barrel and pump purified water or acetone through the needle and microsyringe. The microsyringe should be air dried for storage.

Solvent Compatibility

The adhesives used to bond needles in gas-tight microsyringes are chemically resistant. However, some solvents, particularly halogenated hydrocarbons, can corrode and damage these highly resistant adhesives with prolonged exposure. In cases where such solvents are used, the use of microsyringes with removable needles is recommended. No adhesives are used when connecting the needles to the barrel of these microsyringes. Avoid prolonged immersion of the microsyringe in any solvent during cleaning. After use, rinse the microsyringe thoroughly with purified water, acetone, or other solvent compatible with the sample. Air dry the microsyringe.

Pressure Parameters

Microsyringes can withstand pressures of 13.0 MPa for volumes ranging from 5-100 µL and 6.5 MPa for volumes of 250 and 500 µL.

Clogged needles can create counter-pressures on the plunger in the microsyringe that exceed recommended levels. Use only clean wire or dissolution methods to remove any needle plugging

Temperature parameters

The best microsyringe results are achieved at temperatures above 10 °C. Microsyringes with glued needles should not be heated above 50 °C. For applications requiring temperatures up to 115 °C, microsyringes with removable needles should be used.

Rapid temperature changes of the microsyringe should be avoided.

Usage

Grasp the syringe by the flange to reduce inaccuracy. Avoid variations in liquid measure due to body heat by grasping the syringe flange and plunger as you draw and dispense fluids. Fully prime the syringe by aspirating and dispensing sample to remove trapped air which can cause inaccuracies. Eliminate trapped air, which is compressible and affects syringe accuracy and precision, by completely priming the syringe with sample.

Always pull liquid into a syringe barrel to wet the interior surface. Avoid unnecessary moving or pumping of the plunger in a dry syringe. Excessive dry pumping increases plunger wear shortens syringe life expectancy and may lead to damage beyond repair.

Do not apply force to move a plunger. Too much pressure can irretrievably bend the plunger or crack the syringe glass barrel. If the plunger feels like it is binding or rough, it may be soiled or bent. Do not force the plunger. Try using an appropriate solvent and wiping with a lint-free cloth. Then follow the cleaning procedure to clean the barrel and try again.

The plungers on Microliter syringes are hand-fitted and only liquid-tight. The plungers are not replaceable for Microliter syringes.

Avoid touching the plunger with your fingers. Abrasions, scratches or oil due to handling the plunger with your fingers may interfere with proper plunger operation.

If the plunger is inadvertently removed from the syringe barrel, wipe it carefully with a lint-free tissue. Reinsert the plunger into the barrel and pump deionized water or acetone through the needle and syringe.

Cleaning and care

The life of your Hamilton syringe is directly related to its cleanliness and proper care. In general, solvents suitable for routine cleaning include methanol, acetonitrile and acetone. Use solvents of high purity grade. Halogenated hydrocarbons should not be used because they may damage some glue joints.

To clean Hamilton syringes, it is best to use solvents known to be effective in solvating the sample. Preferred cleaning agents are non-alkaline, non-phosphate and non-detergent based. A biodegradable, non-phosphate, organic Cleaning Solution Concentrate is available from Hamilton (p/n 18311).

Chemical Compatibility

The adhesive used to affix needles and hubs to Hamilton Microliter and Gastight syringes is the most chemically resistant available. However, with prolonged exposure, some solvents may attack and deteriorate this highly resistant adhesive. Caution should be exercised with solvents containing halogenated hydrocarbons such as dichloromethane (methylene chloride). For applications using these solvents, Removable Needle (RN) syringes are recommended because no adhesive is present in the fluid path. Be sure to rinse the syringe thoroughly after each use with a solvent that is known to solubilize your sample followed by a solvent such as acetone to ensure that the glue does not remain in contact with a potentially harmful solvent.

Cleaning Syringes

Rinse the syringe after use with an appropriate solvent or cleaning agent. Following the use of a cleaning agent, rinse the syringe with deionized water and finally acetone. Wipe the exterior surfaces of the syringe barrel and needle with a lint-free tissue. Make sure that there is no residual cleaning agent in the syringe before using or storing the syringe. Do not soak or submerse the entire syringe in any cleaning agent. Prolonged contact with cleaning agents may damage bonded parts.

Clogged needles

For a partially clogged needle, flush the syringe with an appropriate solvent to solubilize the clog. For a completely clogged needle, do not attempt to clean by forcing liquid or compressed air through the syringe. Excessive pressure will split the glass barrel. Alternatively, use the Hamilton Needle Cleaning Kit (p/n 76620A). Start by using the cleaning wires to dislodge any foreign material. Then flush with the Cleaning Solution Concentrate to further dissolve the clog. Once the clog is removed, rinse the syringe and needle thoroughly with deionized water. Wipe the exterior surfaces of the syringe barrel and needle dry with a lint-free tissue. Make sure that there is no residual cleaning agent in the syringe before using or storing the syringe.

Temperature

Syringes should be used at a constant temperature. Accuracy and reproducibility specifications are determined at 25 ºC. Avoid rapid temperature changes, which can lead to splitting of the syringe glass barrel.

Autoclaving cemented needle syringes is not recommended as glass and metal expands at different rates which would compromise the glued connection.

6.1.2 Features of syringes use in the autosampler

When starting use a new syringe, user may encounter two typical issues:

- Seizing plunger moistened with solvent or sample in glass barrel (blocking effect);
- needle channel become clogged with rubber due to puncturing of inlet septum.

When maintenance of syringe unit (or syringe) is not carried out timely (or maintenance is inadequate), needle channel may become completely clogged and plunger may hardly move and scratch the glass body of syringe. This may lead to fatal failure of syringe unit or syringe, i.e. plunger bending (Figure 6.1).

The Manufacturer is not responsible for failed syringe unit or syringes with bended needles or plungers as soon as these failures appear only as a result of improper use.

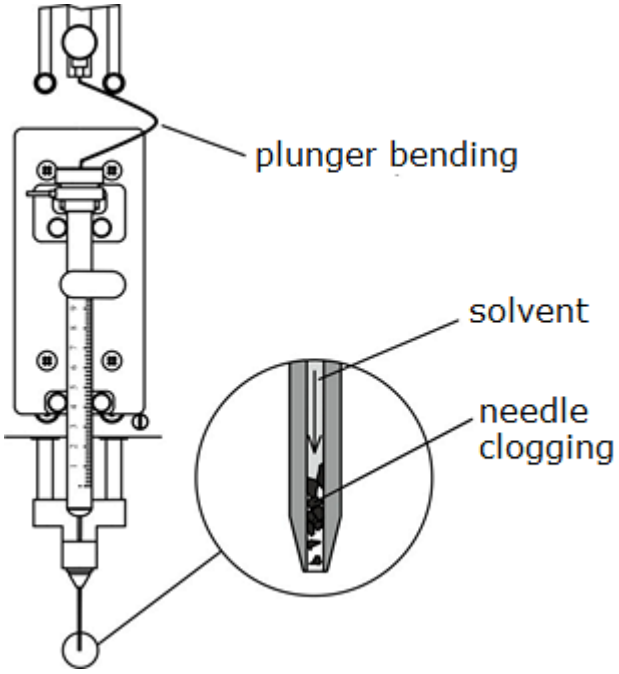


Figure 6.1 – Syringe plunger bending

Plunger seizing

Plunger seizing can be caused by two factors:

- plunger adaptation during which metal particles of plunger penetrate glass walls of syringe;
- solid deposits appear on syringe walls and plunger walls due to mechanical contact with sample and solvent during operation.

All solvents used for flushing should be of high purity grade. Poor grade solvents often contain impurities that remain in the syringe barrel and cause the plunger to seize or stick in the barrel.

Clogged needle

The microsyringe needle channel may be clogged by rubber particles resulting from the sharp and insufficiently curved needle tip passing through the inlet septum (Hamilton, SGEChromatec microsyringe needle type 2), or rubber particles smaller than 0.09 mm may enter the open channel in the Hamilton 701 10 µl (23S – 26S /1.71´´/HP) microsyringe needle. A typical pattern of channel clogging in these needle types (at high magnification) is shown below (Figure 6.2).

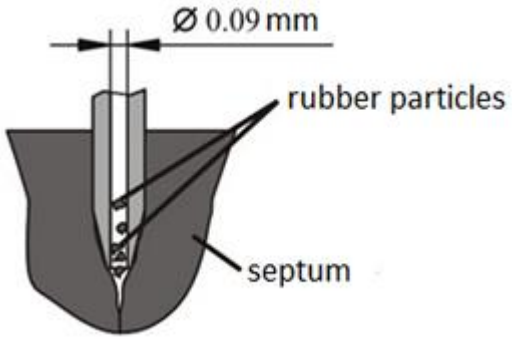


Figure 6.2 – Needle channel clogging

Besides at some circumstances this effect heightened due to following reasons:

- particles that constantly appear as a result of plunger wearing;
- solvent contains too many contaminants (bad purity);
- sample nature (disposition to polymerization, salts presence or other unwilling features);
- accumulative effect of above-mentioned reasons.

When considering term "bad purity" we mean the solid mechanical particles with size of several microns presented in solvent. These particles may penetrate syringe and needle channel and adhere to syringe walls or to rubber particles already presented in needle channel.

For typical picture of abovementioned effect (zoomed picture), see Figure 6.3.

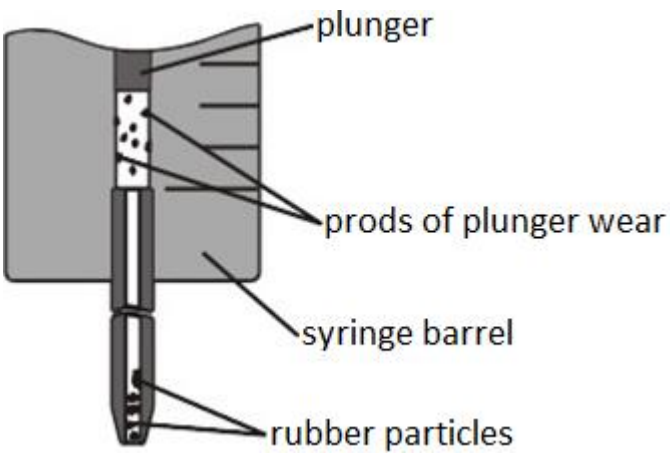


Figure 6.3 – Clogging syringe channel

If the syringe unit (or microsyringe) is not maintained during operation (or if insufficient or unqualified maintenance is performed), the needle channel may be hermetically clogged and the plunger may be rubbed when moving along the cylinder of the glass body. This results in a fatal syringe unit or microsyringe failure – plunger bending (Figure 6.1).

The manufacturer is not responsible for failed syringe units or microsyringes with bent needles and plungers, as these defects can only be caused by improper operating conditions.

Refer to Section 0 for a list of maintenance actions required to keep the syringe unit in good working order.

6.2 Types of maintenance

Two types of maintenance are foreseen for the Autosampler:

- Current maintenance. Generally, it's carried out by user.
- Periodic maintenance. It's carried out by service personnel trained for autosampler service.

6.3 Current maintenance

Current maintenance implies on daily observation for correct operation of autosampler machinery and conducting some simple service procedures, as follows:

- Check that Autosampler moving elements are not damaged or bent, properly working and driving smoothly according to planned algorithm.
- Maintain the availability of washing liquid on a daily basis, refill the vial (or vials) if necessary, ensure the cleanliness of the washing liquid and the entire washing path;
- Check inlet septum of the GC free of leaks. Whenever new septum placed, check that needle of autosampler syringe penetrates through the septum smoothly.
- Visually check gas and liquid lines for leaks. If leaks are detected, retighten seals, replacing them if necessary.

- Keep syringe internal channels and needle clean.
- Check that all screws and nuts of the autosampler are tightened reliably enough. Especially observe for movable parts of the autosampler, nuts and screws fixing syringe.
- For temperature-controlled tray – check tubings, drain tube and condensate pan for signs of leakage due to material fatigue (cracks).
- Constantly check and keep solvent vials full enough, as well as waste vials enough space for waste liquid. Add pure solvent into solvent vial if necessary. Do not allow arising any contaminants in solvent vials. Keep caps of solvent and waste vials clean and dry.
- For temperature-controlled tray, check that there is no moisture under the tray on the autosampler surface.

Check the lightness of movement of the plunger in the microsyringe barrel

The check is performed daily before the autosampler operation (the autosampler should be switched off). To check, move the carriage of the syringe gear up and down manually up to the stops. There should be no perceptible forces when moving the carriage.

If you find difficulties in the movement of the plunger should be washed syringe unit in accordance with the instructions of section 6.4.

Chromatograph inlet septum replacement

Do not reuse used septa. Doing so will usually cause the microsyringe needle to break.

The quality of liquid sample injection into the chromatograph inlet depends largely on the condition of the microsyringe, and the inlet rubber septum.

The procedure for replacing the inlet septumis described in the section.

After installing the inlet septum, **manually** check the passage of the autosampler microsyringe needle through the inlet septum. To do this, holding the stand 1 by hand, gently move the syringe gear until the microsyringe needle contacts the septum, carefully insert the microsyringe needle through the septum into the inlet, then move the syringe gear upwards.

The passage of the needle through the septum should be easy but not disturb the tightness of the inlet.

Monitoring the cleanliness of the syringe unit microsyringe channels

Visual control for cleanness of internal washing cannels, syringe barrel and needle. The control of 10 µL syringe unit can be provided at top washing mode by checking the solvent flow rate through body channel and syringe needle.

Every day observe the time needed for air bubble to move through glass body of syringe (this time must be within 3 seconds). If time exceeds the specified value, this indicates washing system failure. If this time exceeds 4 seconds, dot not use sampler.

When using syringes with "washing from vial" mode, check cleanness observing how air bubbles are eliminated from the syringe barrel at aspirating and flushing solvent in washing mode.

To recover sampler normal operation, replace the syringe with new one and proceed with cleaning of removed syringe.

6.4 Periodical maintenance

The main procedures of periodical maintenance are described below:

- Clean syringe unit or syringe if there any suspicion on carryover, plunger seizing or sticking, clogging washing channels or syringe needle.
- Periodically or once per month remove dirt from the enclosures and sampler units; blow the control unit with compressed air (do not remove enclosure and protect the fan impeller from rotation).
- Periodically or once per month wipe the guides of syringe gear and syringe gear drive with cotton cloth slightly moistened with liquid lubricant. Also apply 1-2 oil drops to each friction assembly.

WARNING: Special lubricant has been applied to rubbing parts and components. Do not use any lubricants of any kind.

All solvents used to wash the syringe must be of high purity. Insufficiently pure solvents usually contain impurities that remain in the syringe barrel and cause galling of the plunger.

The cleaning procedure consists of sequentially cleaning the wash channel, the microsyringe plunger, the inner channel, and the microsyringe needle.

Cleaning the washing path

The syringe unit must be installed in the autosampler. Cleaning is carried out with a solvent flow, with a washing time of at least 30 seconds.

Cleaning plunger and barrel channel of syringe

To perform this procedure, the syringe unit or microsyringe should be removed from the autosampler (see section 4.8).

To eliminate the possibility of plunger jamming in the cylinder of a 5 µl or 10 µl glass microsyringe barrel, remove the microsyringe from the autosampler:

- Remove plunger from syringe and moisten it with pure alcohol. Insert plunger into syringe and make a certain number of plunger strokes (pull the plunger out to half a length). If any evidence of plunger blocking is detected, immediately stop procedure.
- Remove plunger and polish it with lint-free cloth moistened with alcohol. Correct polishing means that you move cloth in direction from cap to end. Repeat the above-mentioned procedure until movement become light and smooth.
- Particles that appear as a result of friction should be removed from syringe surface by special cleaning brush representing a steel wire spring with dia. 0,3 mm having wound thin lint-free

thread with step of 3–5 mm. For barrel channel wash with solvent flow directed from needle to syringe cylinder (plunger must be removed).

Sometimes sticking plunger in the needle (or in the syringe barrel) may be eliminated by soaking syringe in distilled water within up to 72 hours. Then carefully stroke plunger using distilled water or acetone until plunger movement become light and smooth

Needle cleaning

For a partially clogged needle, flush the syringe with an appropriate solvent to solubilize the clog (e.g. Needle cleaning kit NCK, SGE Part.N.031782).

For a completely clogged needle, do not attempt to clean by forcing liquid or compressed air through the syringe. Excessive pressure will split the glass barrel. Clogged needle channel can be cleaned by mandrel (stainless steel wire of dia. 0.08mm). Once the clog is removed, rinse the syringe and needle thoroughly with deionized water.

7 Transportation and storage

The autosampler in manufacturers package can be shipped over any distance by any transport except for non-pressurized aircraft compartments and open decks of sea transport. Ensure adequate protection of shipping container from atmospheric precipitations.

Shipping requirements:

- ambient temperature between – 50 and 50 °C;
- maximum RH 98 % at 35 °C;
- avoid any dust or aggressive vapors in air.

For autosampler shipment, remove the syringe unit (or syringe) from autosampler and place it into Shipping kit package in order to protect it against damage.

Shipping containers should be placed into transport in such a way that prevents them from mechanical shocks. During loading operations, avoid any container drops or mechanical damage. Also, protect them against precipitations.

The packaged sampler should be stored in closed warehouse meeting the conditions 2 per GOST 15150-69:

- ambient temperature between – 30 and 40 °C;
- maximum RH 98 % at 25 °C.

Avoid any dust or aggressive vapors in air.

Samplers may be stored in stacks (three layers maximum).

Before using sampler taken from warehouse in cold periods, leave it at normal conditions for 4 hours minimum.

8 Manufacturer warranty

The manufacturer guarantees the autosampler meets the requirements of technical specifications if the buyer observes proper transportation, storage, installation and operational conditions.

Manufacturer assumes no claims associated with syringe failure or syringe unit failure (bended or clogged syringe needle, clogged washing channel) as soon as these problems occur only as a result of non-compliance with operation and maintenance instructions specified in this Operation Manual.

Actual autosampler design may slightly differ from that specified in this User Manual (this doesn't affect performance).

The warranty period is 12 months from entry into service (EIS), but not more than 18 months since acceptance.

Warranty is not valid, and claims cannot be accepted if instructions described in section 6 are not carried out timely and properly.

9 Claims

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

- draw up a technically reasoned act of defects concerning its incompliance with technical requirements, specified in section 2.2;
- make abstracts from section "Acceptance certificate";
- specify performed maintenance procedures.

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, ZIP code 424000.

In Russian: 424000 г. Йошкар-Ола, ул. Строителей 94, СКБ "Хроматэк"

Or 424000, г.Йошкар-Ола, Главпочтамп, а/я 84.

tel/fax: +7(8362) 68-59-16, E-mail: mail@chromatec.ru

Other Phones of manufacturers departments:

Service department, tel. +7(8362) 68-59-19, 68-59-32, fax.+7(8362) 68-59-87

E-mail: service@chromatec.ru

Sales department, tel. +7(8362) 68-59-68, 68-59-69, fax 68-59-70,

E-mail: sales@chromatec.ru