

AUTOSAMPLER AS-2M (3D) HEADSPACE

USER MANUAL

214.2.508.006-06UM

Rev. 22.12.2023

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1 Important notes

The present User Manual (UM) is intended to get familiar with Autosampler AS-2M (3D) Headspace (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.

The relevant sections of this User Manual (the UM) describe directions and procedures recommended for operation and maintenance of the Autosampler. Take attention to symbols below to make your operation more efficient.

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.

2 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 Operation 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 included to Shipping kit.

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

Before to proceed with technical maintenance, vial replacement or solvent bottle charging, disconnect the power cord from power mains and close air (or gas) source.

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.

3 Description

3.1 Introduction

Autosampler AS-2M (3D) Headspace is intended for automatic aspirating and injecting headspace samples into gas chromatographs "Chromatec-Crystal 5000", "Chromatec-Crystal 9000", for determination of volatile organic compounds by static headspace method.

The autosampler is manufactured in 2 models: P/N 400-0601 supplied with 1ml syringe unit, and P/N 400-0602 supplied with 2.5 ml syringe unit.

Autosampler features can be extended by option for liquid injection and option for SPME (Solid Phase Micro-Extraction).

 # Liquid option for 3D Headspace, P/N 400-0691 is applicable for liquid injection and ambient headspace injection. Syringes for liquid injection of 10 µL capacity supplied in option set, other syringes 0.5, 1.0, 5.0 µL can be supplied by order. Gastight syringes 100 and 250 µL and holder 214.6.152.013 required for ambient headspace injection are supplied by order.

 # SPME option for 3D Headspace, P/N 400-0692 is applicable for Solid phase micro-extraction (SPME) technique.

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 $\pm$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.

3.2 Technical specifications

Refer to Passport 214.2.508.006-06PS for Technical specifications. Refer to corresponding sections of Passport for Technical specifications of options.

Table 3.1 – 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 3.2 – Injection (aspiration) time of 1 µl sample, msec

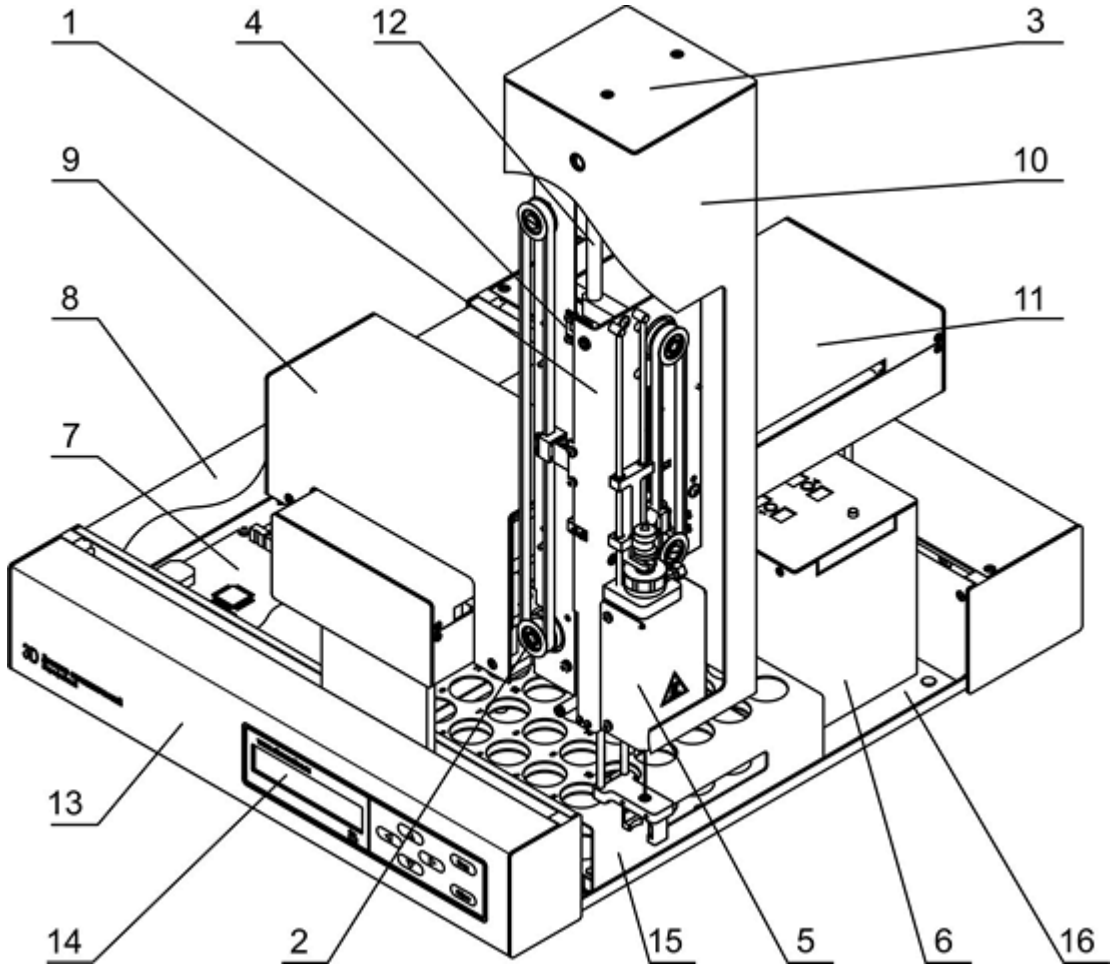
Syringe, µL	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
0,5	6670	5710	4760	3840	2860	1920	1430	950	480	250
1	3335	2855	2380	1920	1430	960	715	475	240	125
5	1334	1142	952	467	572	384	286	190	96	50
10	667	571	476	384	286	192	143	95	48	25
100	71	60	50	40	30	20	15	10	5	3
250	28,4	24	20	16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

3.3 Delivery set

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

3.4 Construction

3.4.1 Overview



1 – syringe gear; 2 – syringe gear drive; 3 – Tower; 4 – optoelectronic sensor of syringe unit position; 5 – syringe thermostat; 6 – sample vial incubator (thermostat); 7 – control unit; 8, 9, 10, 11 – covers; 12 – guide; 13 – front panel; 14 – display; 15 – removable tray; 16 – basement.

Figure 3.1 – Autosampler overview

The autosampler configuration includes the following functional units:

- 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;
- syringe thermostat;
- sample vial incubator with orbital shaker;
- fiber bakeout thermostat (available for SPME option only);
- removable sample tray;
- control unit.

Syringe gear 1 (Figure 3.1) containing syringe unit in its thermostat, performs aspirating and injecting headspace sample at programmable conditions (volume, temperature, etc.). Syringe unit is the

syringe with fitted adapter for top purging. Syringe gear carries out plunger movement by set program.

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 analysis method.

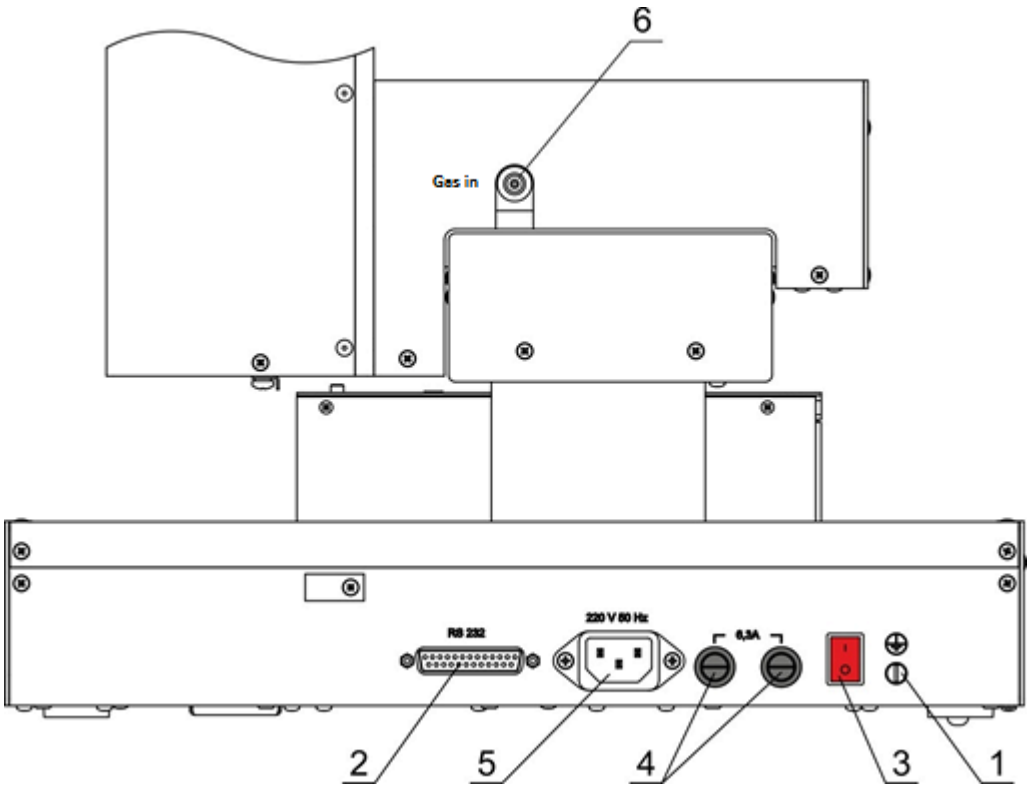
When syringe gear is in upper position, the tower 3 can move in horizontal axes.

In intermediate position, sampler performs sampling operations.

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 front panel 13 (Figure 3.1) 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 3.2 – Autosampler rear view

The rear side of cover 10 features a screw, which is to be used for sampler transportation (Figure 3.3).

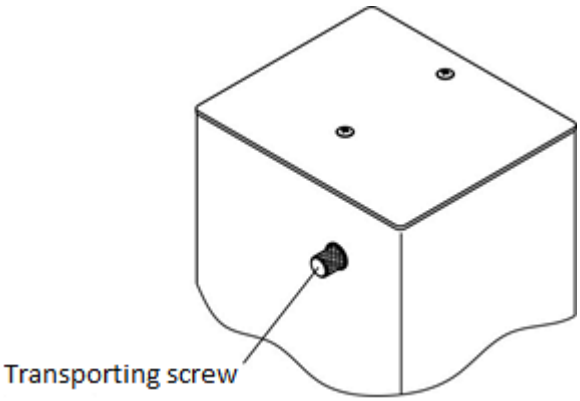
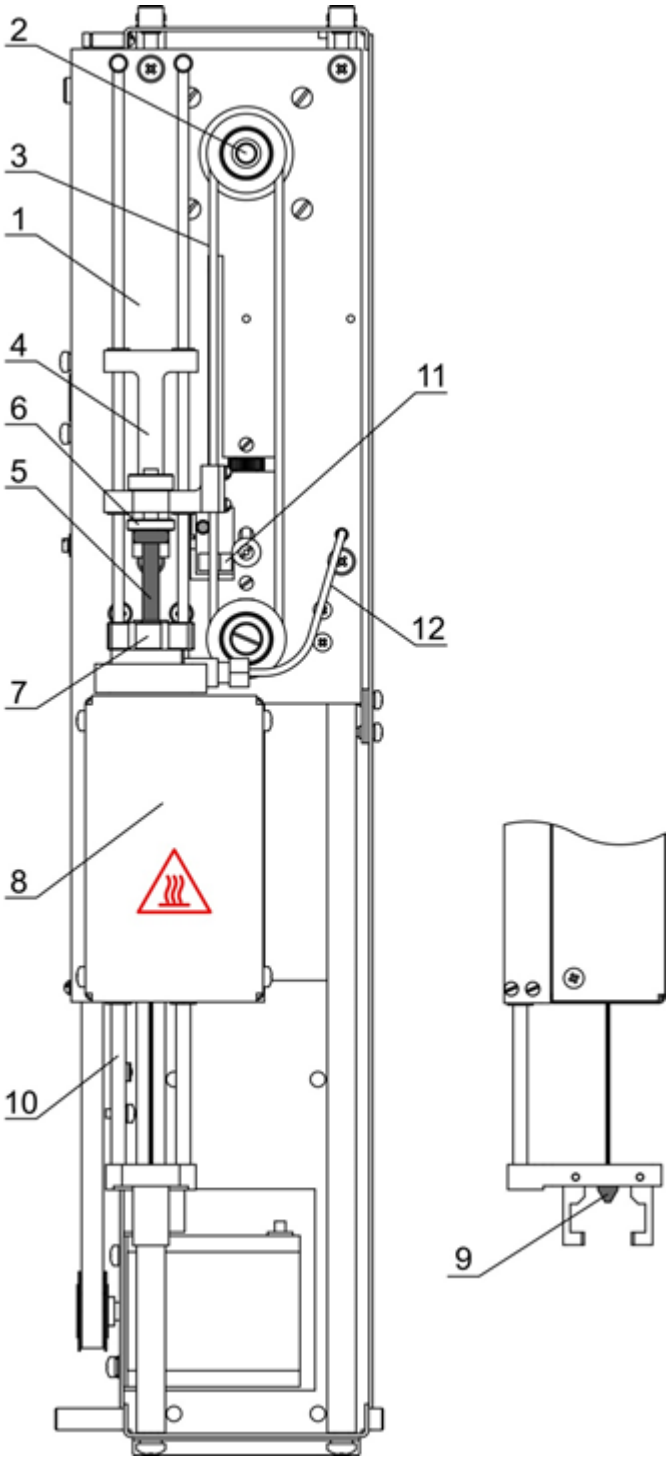


Figure 3.3 – Screw installed for transporting position of the Autosampler

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

3.4.2 Syringe gear



1 – tower basement; 2 – stepping motor pulley; 3 – flat toothed belt; 4 – carriage; 5 – syringe unit 214.4.464.103 (or 214.4.464.104); 6 – screw for plunger fitting; 7 – nut for syringe unit fitting; 8 – syringe thermostat; 9 – needle adapter; 10 – guide; 11 – optoelectronic sensor; 12 – syringe purging tube.

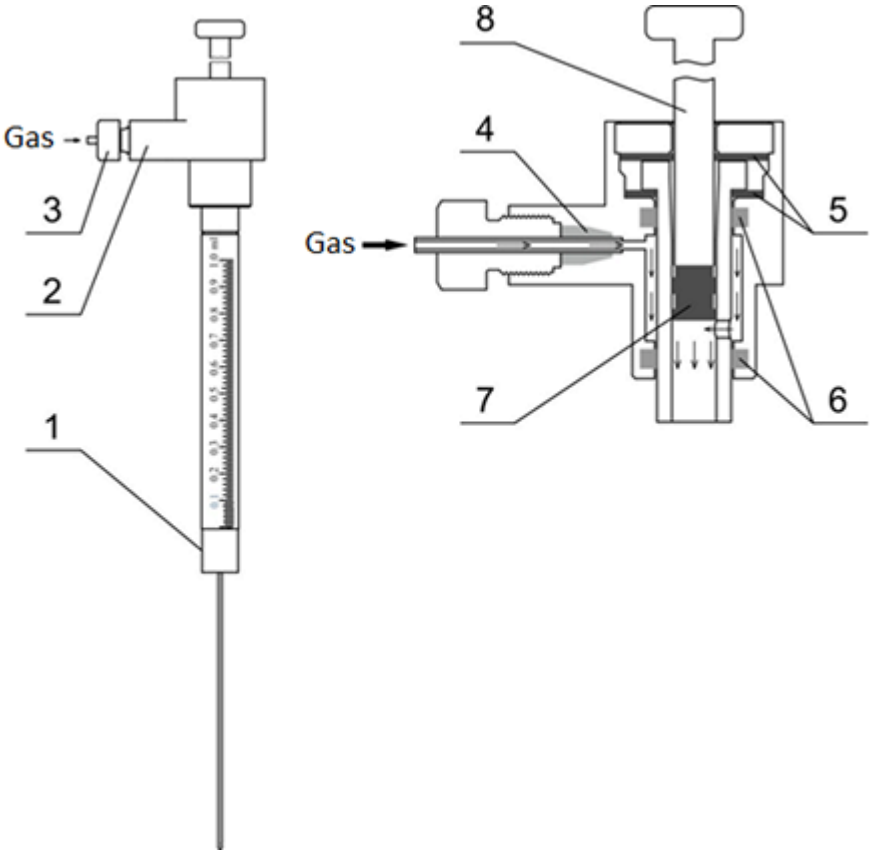
Figure 3.4 – Syringe gear overview

The syringe gear (Figure 3.4) 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 5.

The thermostat 8, fitted on the base 1, contains syringe unit 5 duly installed. Needle of the syringe passes through the needle adapter 9, which fixed on the guides 10. Needle adapter 9 serves for protection needle tip and aligning needle with vials and inlet nut while sampling and injecting. Nut 7 fixes syringe unit 5 in the thermostat 8. Screw 6 fixes plunger of the syringe in the carriage 4.

The carriage 4 has a curtain. The curtain and optical sensor of syringe plunger 11 are used to input data about start position of syringe plunger (this position corresponds to "zero-volume" sample in syringe) to control unit.

3.4.3 Syringe unit and syringes



1 – syringe SGE-1ml (or 2.5ml); 2 – flange; 3 – fitting; 4 – ferrule; 5 – washers; 6 – O-ring; 7 – plunger; 8 – plunger rod.

Figure 3.5 – Syringe unit design

Syringe unit 4.464.103 (Figure 3.5) is intended for autosamplers performing static headspace method. The core of syringe unit is a gastight syringe **SGE-1.0 ml** (P/N 008131) equipped with adapter for purging its internal barrel and needle volume from the remnants of previous headspace sample. Syringe unit 4.464.104 is the same in design but containing another syringe – **SGE-2.5 ml** (P/N 008631).

In case of any problems with the syringe unit during its operation, contact the customer service department of the manufacturer or its regional representative.

3.4.4 Replaceable syringe thermostat and replaceable syringe adapters

Headspace syringe unit is installed into syringe thermostat. Syringe for liquid and SPME holder are installed into corresponding replaceable adapters.

The base of syringe gear has 4 holes and 2 magnets for adapters fitting (Figure 3.6). To fit syringe thermostat or adapters on the autosampler, just match pins of adapter with holes on the base; magnets provide jointing.

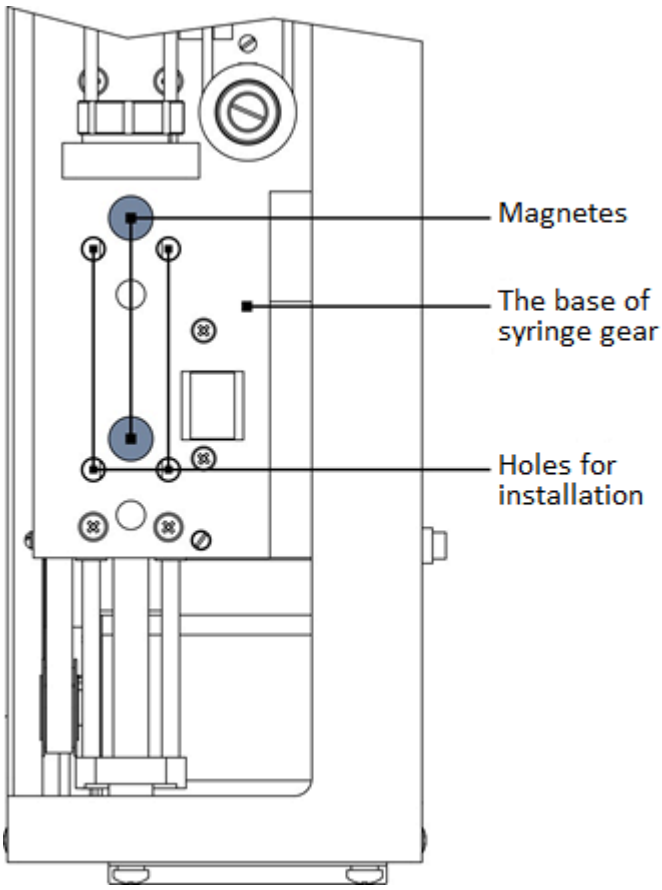
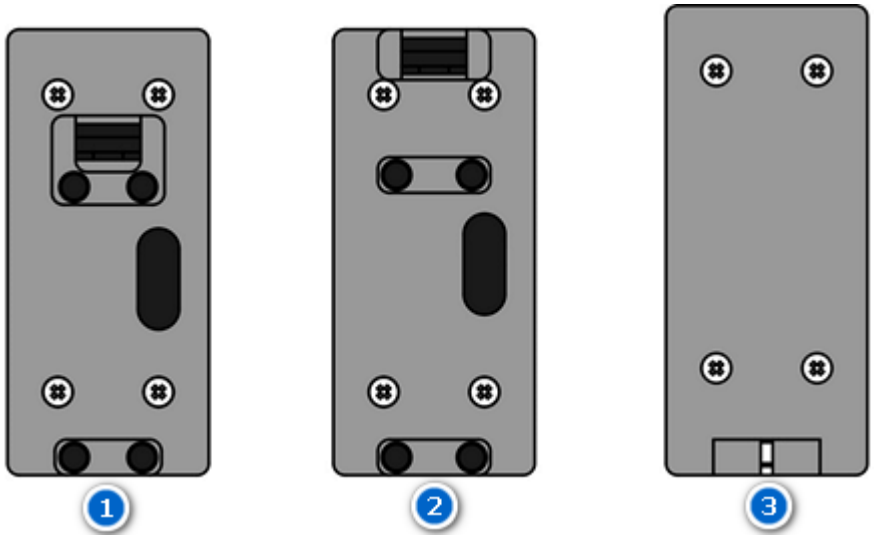


Figure 3.6 – Installation of syringe thermostat and adapters

Replaceable thermostat is a heating unit which keeps temperature of syringe unit in headspace applications. Settable temperature range is from 40 to 150 ˚C (40 ˚C at ambient 30 ˚C or below).

Replaceable adapters serve for installation of various syringes into the autosampler. 3 syringe adapters are available (Figure 3.7):

- Adapter 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).



1 – Adapter 6.152.012 (Liquid), 2 – Adapter 6.152.013 (100, 250 µL, amb. HS), 3 – Adapter 6.152.017 (SPME)

Figure 3.7 – Replaceable adapters

3.4.5 Incubator (vials thermostat)

Sample vial incubator is a heating unit installed on moving basement. Incubator capacity is 4 vials. The incubator provides headspace equilibration in the vials for headspace and SPME applications. Settable temperature range is from 40 (for Headspace) or 30 (for SPME) to 170 ˚C. Incubator incorporates orbital shaker, shaking mode programmable.

3.4.6 Fiber heater

Fiber heater supplied as a part of tray for "SPME" option.

The heater carries out bakeout of fiber before sampling to eliminate background and ghost peaks. The heater has purging channel to blow fiber with inert gas while heating. Temperature of heater is programmable, recommended value depends on fiber type, may vary from 50 to 350°C. The heating channel is connected from the gas chromatograph (the GC).

3.4.7 Basement and drives

Basement is a moving platform (XY-robot). The platform incorporates drives which provide moving tower by horizontal axes (X and Y).

3.4.8 Replaceable trays

Replaceable trays 15 for sample vials are located on the basement 16 (Figure 3.1). Individual tray is required for each option.

Replaceable tray for Headspace applications has 30 vials capacity for vials of 20 ml or 10 ml.

Replaceable tray for liquid samples has 60 vials capacity for 2 ml sample vials, 4 solvent vials capacity (4 ml) and 2 waste vials capacity (20 ml).

Replaceable tray for SPME applications contains 26 vials capacity for 20 ml sample vials and fiber heater. The fiber heater is shown on Figure 5..

4 Operation modes

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

4.1 Static headspace mode

Operation procedure of headspace option involves performing analytical cycle of preparation and injection of each sample step by step. Analytical cycle for each sample vial corresponds to one line in Analysis Tab of Scheduler Chromatec Analytic Software. Analysis runs are combined in sequences. Once Scheduler operation starts, all active sequences shall be fulfilled.

Headspace mode contains the following states in cycle:

- Rest mode.
- Heating and keeping temperature.
- Sample vial loading.
- Sample vial Incubation.
- Syringe purge before sampling.
- Sampling.
- Injection.
- Syringe purge after injection.
- Sample vial unloading from incubator.

4.1.1 Rest mode

The autosampler keeps Rest mode whenever it does not run sequence. The rest mode starts immediately after setting GC mode from Control Panel. In the Rest mode all temperature zones keep operating temperature.

4.1.2 Heating and keeping temperature

This state starts once Scheduler set a new active analyses sequence. If current temperatures are the same as newly set, this state is jumped. If set temperatures are different from current values, these can be changed for new sequence.

4.1.3 Sample vial loading

The syringe gear grabs vial with its cramp on the bottom part of needle adapter and brings it into vacant socket of the incubator. While loading, shaker does not work. Sample vial loading occurs so that incubation time prior to scheduled injection time.

4.1.4 Sample vial Incubation

Within Incubation state sample vial is kept in the incubator at permanent temperature for a set incubation time (equilibrium time).

At incubation state sample can be agitated by set mode. Shaking mode parameters:

- speed can be set in range from 300 to 600 rpm.
- shaking time and delay time.

4.1.5 Syringe purge before sampling

Syringe purging procedure is synchronized with sampling procedure, it is terminated 1 minute prior to beginning of sampling. Parameters: Pre-Sampling purging time and Pre-filled inert gas volume shall be set for this procedure.

Purging is carried out by inert gas (typically carrier gas). Flow rate is limited by restrictor (approximately 20 ml/min), which is installed in tube. If volume pre-filled by inert gas is over 0, once purging terminated, specified volume shall is set and gas from the syringe is flushed into sample vial before sampling.

Syringe purging algorithm

- Plunger is moved upward into purging position so that plunger tip is located above side hole in syringe barrel;
- Solenoid valve opens inert gas flow;
- After purging time passed, solenoid valve stops inert gas flow (purging is finished).

4.1.6 Sampling

Sampling procedure starts after syringe purged and vial incubation time terminated.

Sampling algorithm

- Shaking takes a pause (if active).
- Syringe gear (tower) moves to the incubator.
- Syringe needle is immersed into vial through a cover of the incubator at set depth.
- Syringe plunger moves down with set velocity to set volume.
- Plunger delay takes place in set point within set delay time to align pressure between vial and syringe.
- If number of strokes (sample homogenization) is above 0, these will be performed.

4.1.7 Injection

Injection algorithm

- Syringe, with headspace sample taken in the barrel, moves to a position right over inlet to be injected in;

- Syringe needle enters inlet at set depth;
- Pre-injection delay occurs (needle is heated before injection);
- Plunger moves down with set velocity (injection speed), sample is injected into inlet;
- Post-injection delay occurs (needle is heated after injection);
- Needle is taken out from the inlet.

4.1.8 Syringe purge after injection

Post-injection purge starts after injection of the sample, as soon as syringe needle went out of the inlet. This procedure may overlap with sample vial loading and unloading procedures.

Post injection purge algorithm

- Plunger is moved upward into purging position so that plunger tip is located above side hole in syringe barrel;
- Solenoid valve opens inert gas flow;
- After purging time passed, solenoid valve stops inert gas flow (purging is finished).

4.1.9 Vial unloading

Sample vial is unloaded from the incubator and lifted into the tray. While unloading, shaker does not work.

4.2 Liquid injection mode

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

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
- Sampling
- 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.

4.2.1 Pre-injection solvent washing

In this mode analyst can select type of the solvent (A, B, C, D), volume of solvent for priming and number of washing cycles. Single or double washing algorithm are available.

Single washing algorithm – Washing with single solvent:

- the tower moves to position of selected solvent vial (A, B, C or D);
- syringe needle enters solvent vial to a specified depth;
- syringe plunger moves to position corresponding to current priming volume (If the specified sample volume to be injected is less than 30% of the microsyringe volume, the volume of solvent aspirated will be equal to 30% of the total volume of the microsyringe regardless.);
- syringe needle moves away from vial;
- syringe gear moves to position of a waste vial (any);
- syringe needle enters a 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.

The "two solvent" mode involves washing the microsyringe with two different solvents.

Double washing algorithm – Washing with 2 different solvents:

- the tower moves to position of selected first solvent vial (A, B, C or D);
- syringe needle enters first solvent vial to a specified depth;
- syringe plunger moves to position corresponding to current priming volume (minimum at 30% of syringe full volume);
- syringe needle moves away from vial;
- tower moves to position of a waste vial (any);
- syringe needle enters a 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;
- the tower moves to position of a selected second solvent vial (A, B, C or D);
- syringe needle enters second solvent vial to a specified depth;
- syringe plunger moves to position corresponding to current priming volume (minimum at 30% of syringe full volume);
- syringe needle moves away from vial;
- carousel moves to position of a waste vial (any);
- syringe needle enters a waste vial;
- syringe plunger moves to lower position (solvent draining);
- syringe needle moves away from vial;
- steps 10 – 17 will be repeated according to user-selected washing cycles.

Level of needle immersion into waste vial for flushing is appr. ¼ of vial height. It is set on default intentionally higher than at sampling in order to avoid waste solvent ingress to a needle tip.

The recommended minimal number of washing cycles is 3.

4.2.2 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 priming volume.

Sample washing algorithm:

- tower 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;
- tower moves to position of a waste vial (any);
- 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.

4.2.3 Sampling

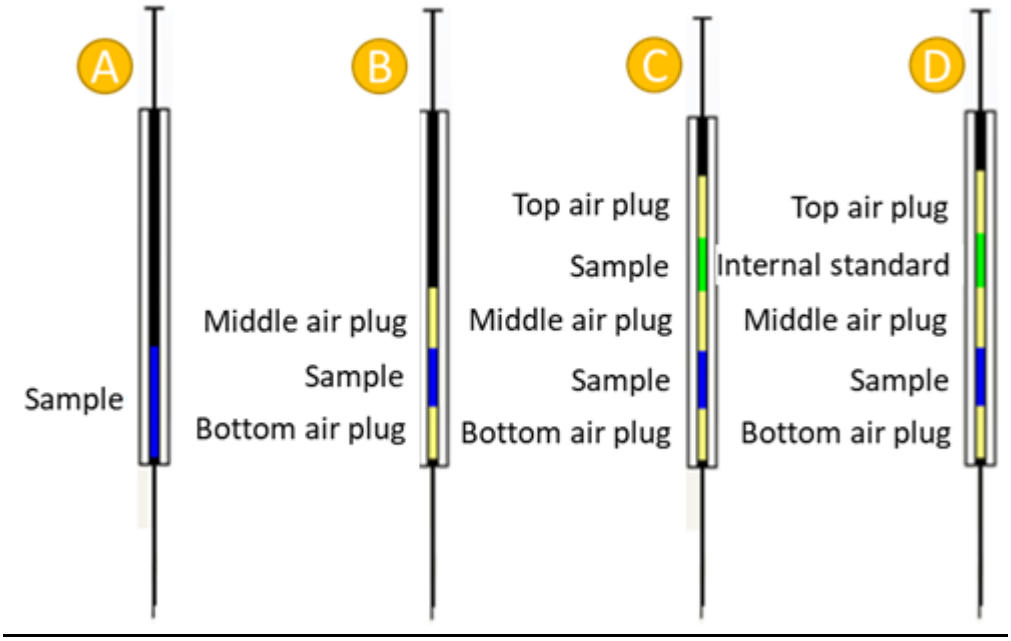


Figure 4.1 – Sampling modes A – Simple; B – Sandwich; C – Solvent sandwich; D – Internal standard sandwich

"Simple" injection mode:

- tower 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.

Table 4.1– Recommended parameters of the autosampler instrumental method

Operating parameter	**Value**
Number of strokes	from 7 to 10
Aspiration speed	From 1 to 3
Injection speed	From 4 to 9
The total volume of samples to be aspirated 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 than, µl	2.5
10.0 µl volume, not more than, µl	10.0

"Sandwich" sampling algorithm

In this mode "Middle 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 middle air plug;
- tower 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 + middle air 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 average air volume + sample volume + low air volume.

"Sandwich plus solvent" sampling algorithm

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

- 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;
- tower 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;

- tower 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.

"**Sandwich with internal standard" sampling algorithm**

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.

- 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;
- tower 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;
- tower 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 needle moves away from vial;
- syringe plunger moves with specified aspiration speed to position corresponding to specified top air volume + int. standard volume + middle air volume + bottom air volume.

4.2.4 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 0.1 sec.

4.2.5 Post-injection solvent washing

This procedure is like that described in section 4.2.1.

When using syringes 100 and 250 µL, air stroking is carried out on default on this stage, it cannot be omitted.

4.2.6 Liner washing

At GC operation heavy compounds of sample may settle on liner glass wool in the inlet, gradually contaminate liner and making an influence on measurement results. This contaminating effect can be minimized by using liner washing function. Liner glass wool lifetime typically extends in this case.

Liner washing may take place after each run (at analysis termination) or after determined number of runs.

Liner washing algorithm:

- The tower moves to solvent vial position (specified vial A, B, C or D);
- syringe needle enters solvent vial to specified depth;
- syringe plunger moves to position corresponding to specified solvent volume;
- plunger keeps set position within viscosity delay;
- syringe needle moves away from vial;
- tower moves to a position of waste vial;
- syringe needle enters waste vial;
- syringe plunger moves to lower position (solvent draining);
- syringe needle moves away from vial;
- tower moves to solvent vial position (specified vial A, B, C or D);
- syringe needle enters solvent vial to specified depth;
- syringe plunger moves to position corresponding to specified solvent volume;
- plunger keeps set position within viscosity delay;
- syringe needle moves away from vial;
- tower moves to zero position;
- syringe needle enters inlet to specified depth into inlet;
- syringe plunger moves to lower position (autosampler proceeds with solvent injection);
- syringe needle moves away from the inlet.

When alcohol-contained samples analyses take place, using bidistillate water injections up to 5 µl for liner washing is recommended after each run.

4.3 Ambient headspace mode

4.3.1 Sampling for syringes 100 and 250 µL

Syringes 100 µL and 250 µL are intended for sampling and injecting headspace sample from 2 ml vials at ambient temperature. Volume of liquid sample in 2 ml vial must not exceed 1 ml.

Sampling algorithm:

- the tower moves to a position of selected sample vial;
- syringe needle enters sample vial to specified depth (depth range is limited from 4 to 14 mm from vial cap, to avoid liquid sample ingress);
- syringe plunger moves upward with specified speed to position corresponding to specified sample volume (at least 50% of total syringe volume);
- specified delay takes place;
- syringe plunger moves downward to zero position;
- steps 3 – 5 will be repeated according to user-selected number of strokes (for sample homogenization);
- syringe plunger moves upward to specified sample volume;

4.3.2 Sample injection by syringes 100 and 250 µL

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.

4.3.3 Air purging for syringes 100 and 250 µL

This algorithm allows cleaning syringes by ambient air.

Algorithm:

- the tower moves to "home" position;
- syringe plunger moves upward with specified speed to position corresponding to specified air volume (at least 50% of total syringe volume);
- specified delay takes place;
- syringe plunger moves downward to zero position;
- steps 2 – 4 will be repeated according to user-selected number of strokes.

4.4 SPME mode

Autosampler procedures for SPME mode involve the following stages:

- Preparation
- Vial incubation
- Extraction
- Desorption
- Fiber cleaning (bakeout)

4.4.1 Preparation

Temperatures of fiber heater and vial incubator are established in this stage.

4.4.2 Sample vial incubation

The syringe gear grabs vial with its cramp on the bottom part of needle adapter and brings it into vacant socket of the incubator. Within Incubation state sample vial is kept in the incubator at permanent temperature for a set incubation time (equilibrium time).

At incubation state sample can be agitated, shaking speed is in the range from 300 to 600 rpm.

4.4.3 Extraction

Syringe needle is immersed into vial and fiber draws out from the needle. Components are trapped on the sorbent. Depth of fiber immersion into vial can be set within range from 22 to 31 mm from the septum top surface.

Vial shaking is limited as low as 250 rpm on this stage.

4.4.4 Desorption

Needle with the fiber is withdrawn from the vial, the tower moves to the inlet. Then needle is injected into inlet and fiber draws out from the needle at set depth (35 – 50 mm from top surface of septum). Components desorb from the fiber in hot inlet. After exposure of the fiber within desorption time the needle is withdrawn from the inlet.

Once desorption finished, the vial is unloaded from incubator into tray.

4.4.5 Fiber cleaning (bakeout)

The tower moves to fiber heater, needle is immersed into thermostat, fiber is drawn out from the needle, solenoid valve opens inert gas flow to the heater. Fiber is baked out in inert gas flow within set time. At termination the needle with the fiber is withdrawn from the heater.

5 Preparation before operation

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

5.1 Main stages

Depending on the option to be installed ("Headspace", "Liquid injection" or "SPME") autosampler preparation has some features.

Table 5.1 – Stages of autosampler preparation for operation

Autosampler option	**Sequence of steps to prepare for operation**
Headspace injection	1. Installation of the chromatograph inlet nut and septum
	2. Checking the tightness of the inlet
	3. Assembly and installation of the autosampler on the support
	4. Connecting the compressed gas source
	5. Connecting the cables
	6. Installing the Static Headspace Analysis option
	7. Loading the vials
	8. Setting the coordinates of inlets
	9. Setting the coordinates of vials in the position "in the tray"
	10. Setting the coordinates of the vials in the "in incubator" position
	11. setting the coordinates of the sampling position
Liquid sample injection	1. Installation of the chromatograph inlet nut and septum
	2. Checking the tightness of the inlet
	3. Assembly and installation of the autosampler on the support
	4. Connecting the cables
	5. Installing the "Liquid Sample Injection" option
	6. Loading the vials
	7. Setting the coordinates of the inlets
	8. Setting the coordinates of sample vials

	9. Setting the coordinates of Solvent vials
	10. Setting the coordinates of the Solvent vials Waste vials positions
SPME	1. Installation of the chromatograph inlet nut and septum
	2. Checking the tightness of the inlet
	3. Assembly and installation of the autosampler on the support
	4. Connecting the compressed gas source
	5. Connecting the cables
	6. Installing the SPME option
	7. Loading the vials
	8. Setting the coordinates of the inlets
	9. Setting the coordinates of vials in the position "in the tray"
	10. Setting the coordinates of the vials in the "in incubator" position
	11. setting the coordinates of the fiber bakeout position

Detailed procedure for each step is described below.

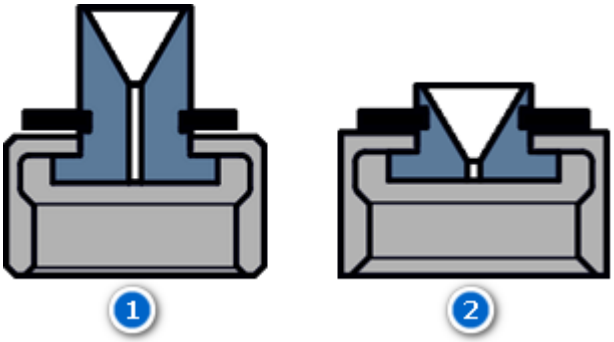
5.2 Gas chromatograph preparation

5.2.1 Installing nut and septum on GC inlet

This procedure also applicable for replacing septum and inlet leak check.

Follow procedure (for any option):

- Remove nut from chromatograph inlet;
- Place new septa and screw special nut 6.482.024 (for Headspace and SPME options, from shipping kit) or 6.482.013-03 (for liquid option, from shipping kit), on the inlet instead of standard nut, use new **OV Grey Septa** (or equivalent) when nut replacing.



1 – Nut 6.482.024 for Headspace and SPME options, 2 – Nut 6.482.013-03 for liquid option

Figure 5.1 – GC inlets nuts for the Autosampler

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.

5.2.2 Inlet leak check

Check the septum leakage through the "Control panel" software. Evaluate difference between measured flow rates of referred to the inlet: **Total gas flow** minus **Split/Vent flow**.

Once new septa installed, analyst should check the difference.

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.

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.

To increase the septum service life, while liner replacement, remove adapter with mounted nut fitting septa (remove only lower nut fastening adapter).

5.3 Autosampler installation

5.3.1 Support assembling and autosampler fitting to support

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

5.3.2 Power connection and connection to the GC

Cables required for connection are supplied in shipping kit.

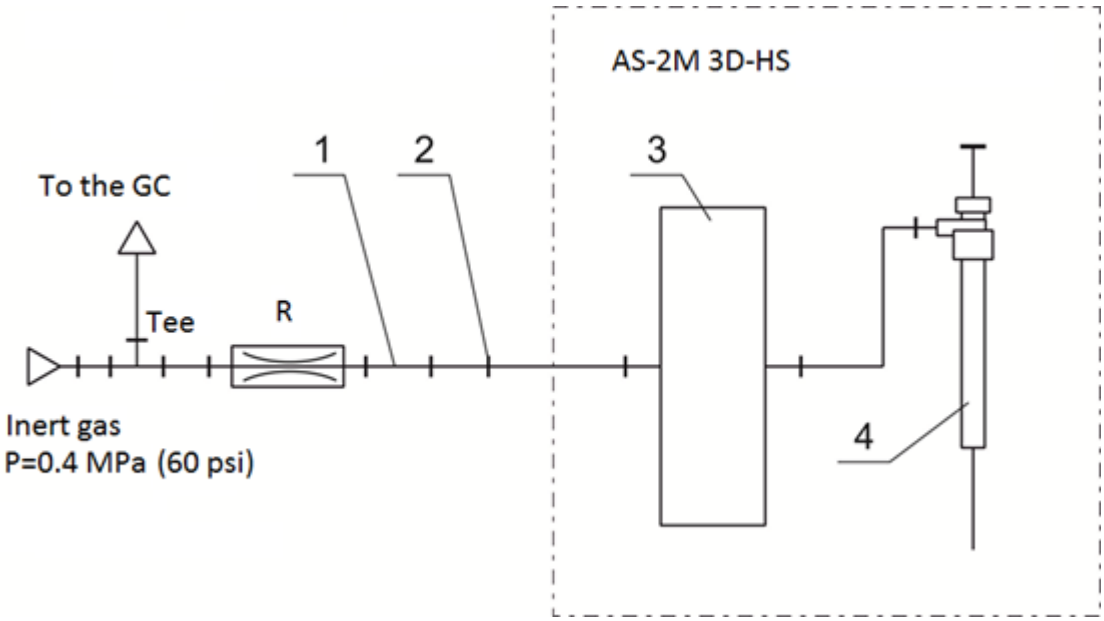
- Use RS-232-25/25 SCB-139 cable from shipping kit to connect the autosampler to slot "AUX" of gas chromatograph.
- Connect the power cord SCZ-1 to power mainsGas connection to the autosampler

5.3.3 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.

Gas connection for Headspace option

- Remove the plug from the washing unit connection .
- Connect the Autosampler to inert gas flow using a tube 6.457.109-01 (from shipping kit), see diagram below (Figure 5.2). The end of tube assembly with Tee joint and a restrictor R is to be connected to gas source. Then fix the tube on the wall of the Autosampler.



1 – tube 6.457.109-01; 2 – fitting "Gas in"; 3 – solenoid valve; 4 – syringe unit, R – pneumatic restrictor (frit).

Figure 5.2 – Autosampler connection to gas source, HS option

Do not connect gas line to the Autosampler without pneumatic restrictor.

Use only high purity inert gas as a gas source for autosampler as gas flow purge analytical syringe.

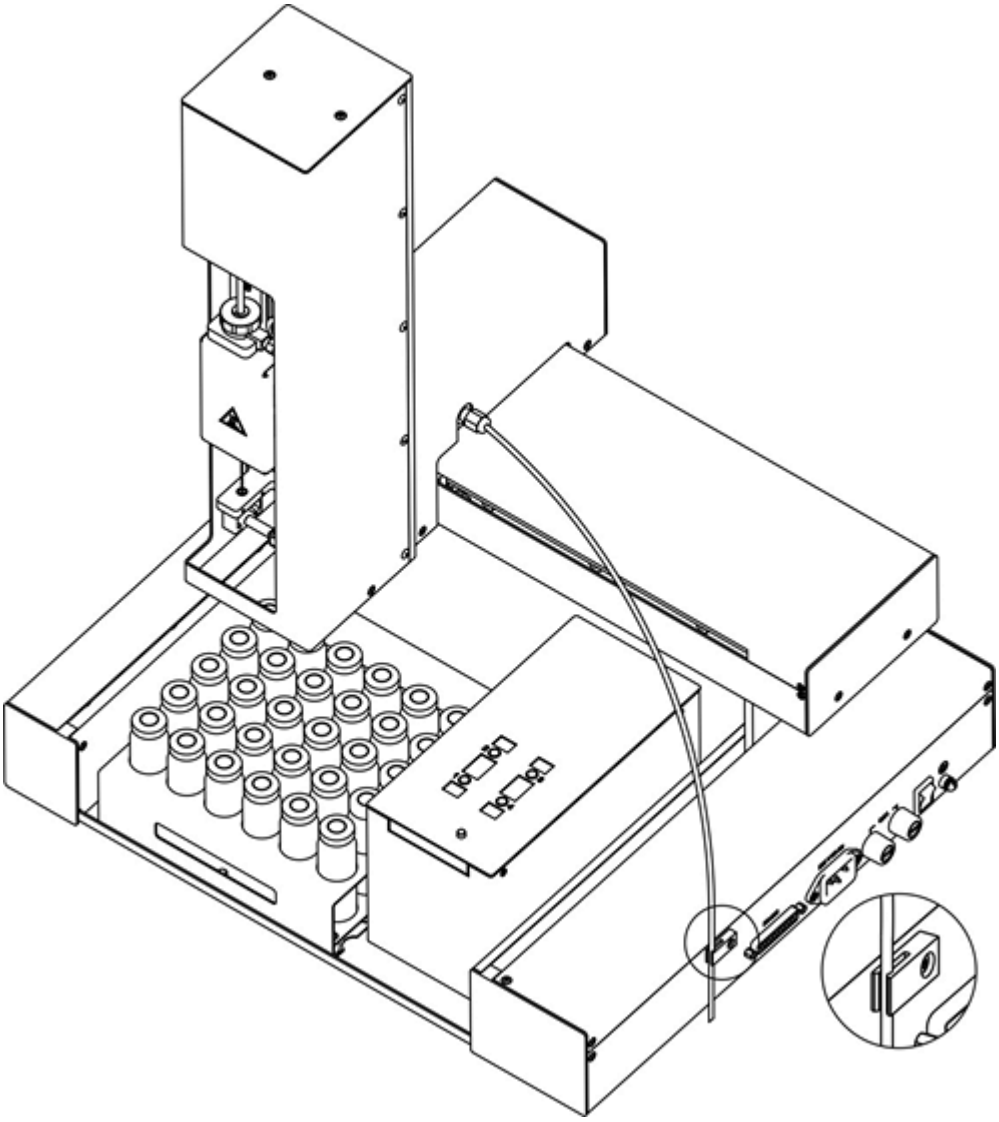


Figure 5.3 – Fixing the tube

Turn off the Autosampler before fixing the tube

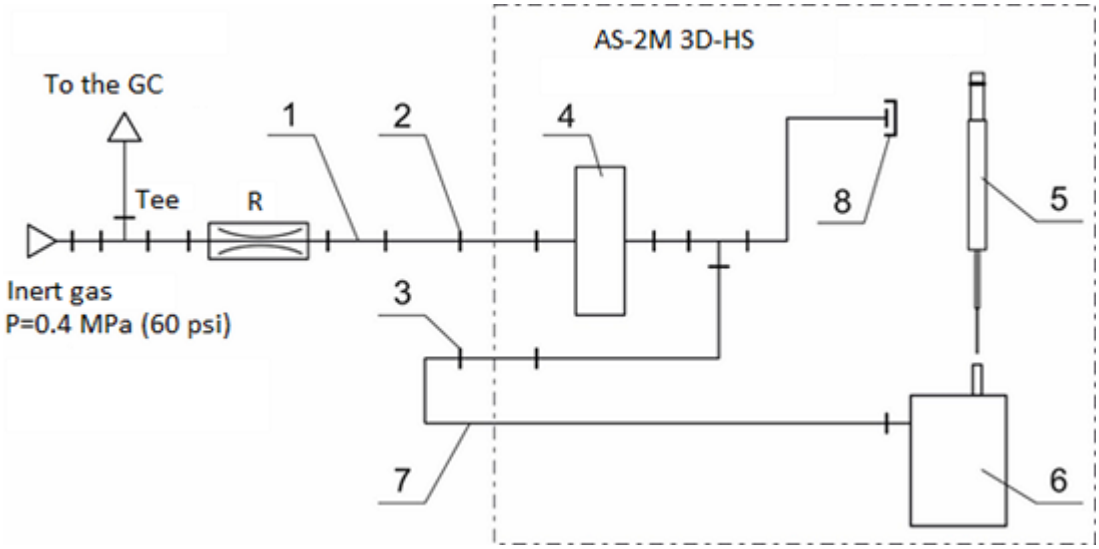
To fix the tube on the Autosampler wall follow:

- Move tower to a distant position as shown on Figure 5.;
- 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 Figure 5.3.

Gas connection for SPME option

Connect the Autosampler to inert gas flow using a tube 6.457.109-01 (from shipping kit), see diagram below (

Figure 5.4). The end of tube assembly with Tee-joint and a restrictor R is to be connected to gas source. Fit the cap 8.



1 – tube 6.457.109-01; 2 – fitting "Gas in"; 3 – Fitting "SPME"; 4 – solenoid valve; 5 – SPME holder; 6 – fiber heater; 7 – tube 6.457.263 connection to "Gas in" fitting; 8 – cap

Figure 5.4 – Autosampler connection to gas source, SPME option

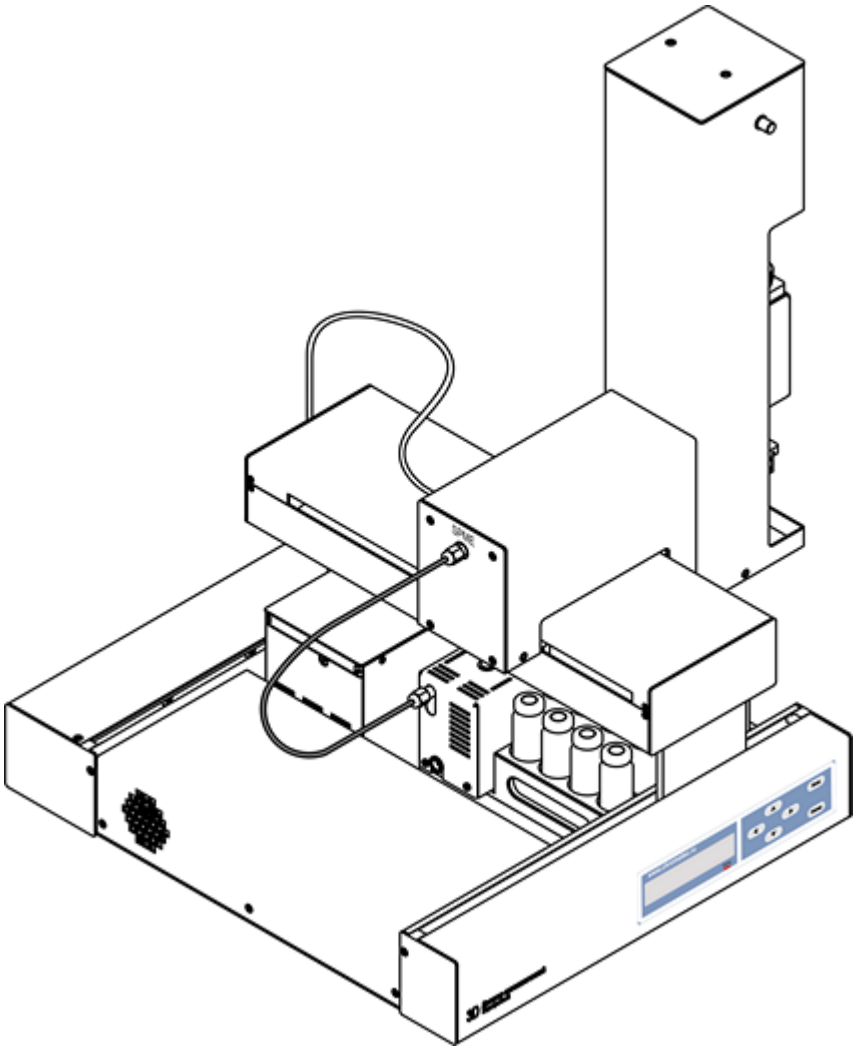


Figure 5.5 –Connecting tube 6.457.263

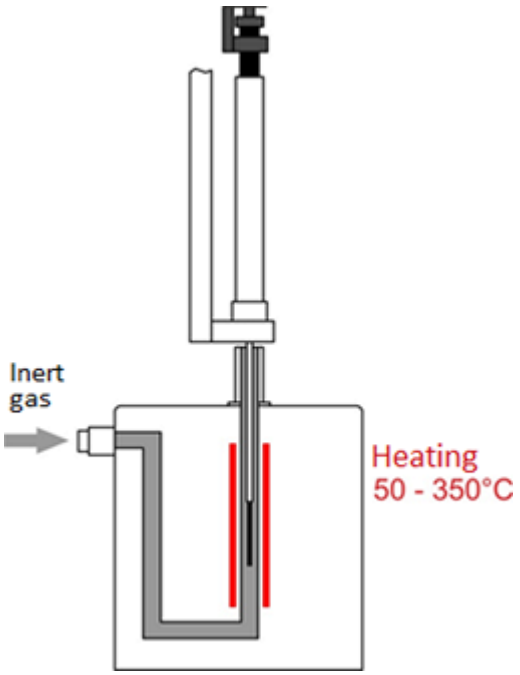


Figure 5.6 – Fiber bakeout diagram

5.4 Headspace option installation

The autosampler is supplied with Headspace option installed on default. Thus, at first installation procedures of section 5.4.1 (needle adapter and vial cramp replacement) and 5.4.2 (syringe thermostat installation) are not required.

Headspace option installation is required when autosampler is re-configured from other options to Headspace.

Headspace option installation involves the following steps:

- Vial cramp mounting;
- Install of replaceable syringe thermostat;
- Install syringe unit and adjust plunger position;
- Place vial tray.

5.4.1 Mounting vial cramp 6.275.038

This procedure is to be carried out when re-configuring the autosampler from Liquid option to Headspace option. Procedure of mounting / dismounting vial cramp 6.275.038 is the same as described in section 5.5.1.

5.4.2 Installation of replaceable syringe thermostat

This procedure is to be carried out when re-configuring the autosampler from other options to Headspace option. Procedure of mounting / dismounting replaceable syringe thermostat is the same as described in section 5.5.2.

5.4.3 Installation of tray 8.120.539

Tray 8.120.639 (30 vials capacity) is intended for using with Headspace option only.

Tray is to be placed on 4 pins located on autosampler basis.

To install the tray, 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 5.7)

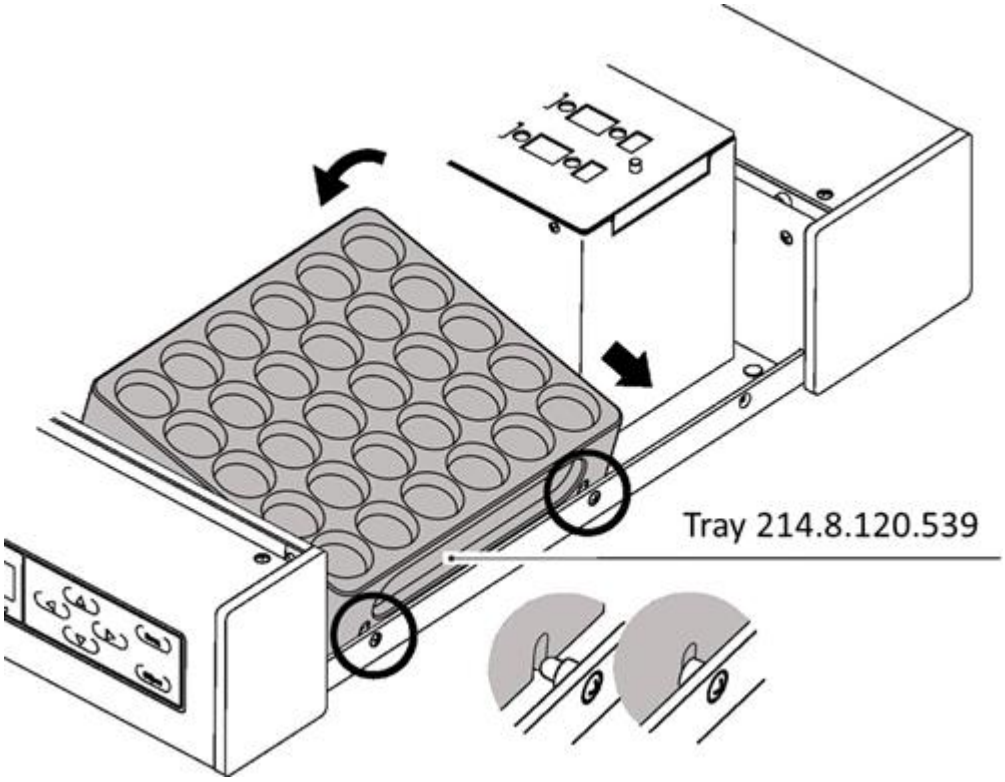


Figure 5.7 – Tray installation

Once tray installed, the Autosampler identifies its type automatically and switch to proper operating mode (Headspace).

5.4.4 Installing syringe unit and plunger adjustment

Turn off the Autosampler before starting this procedure.

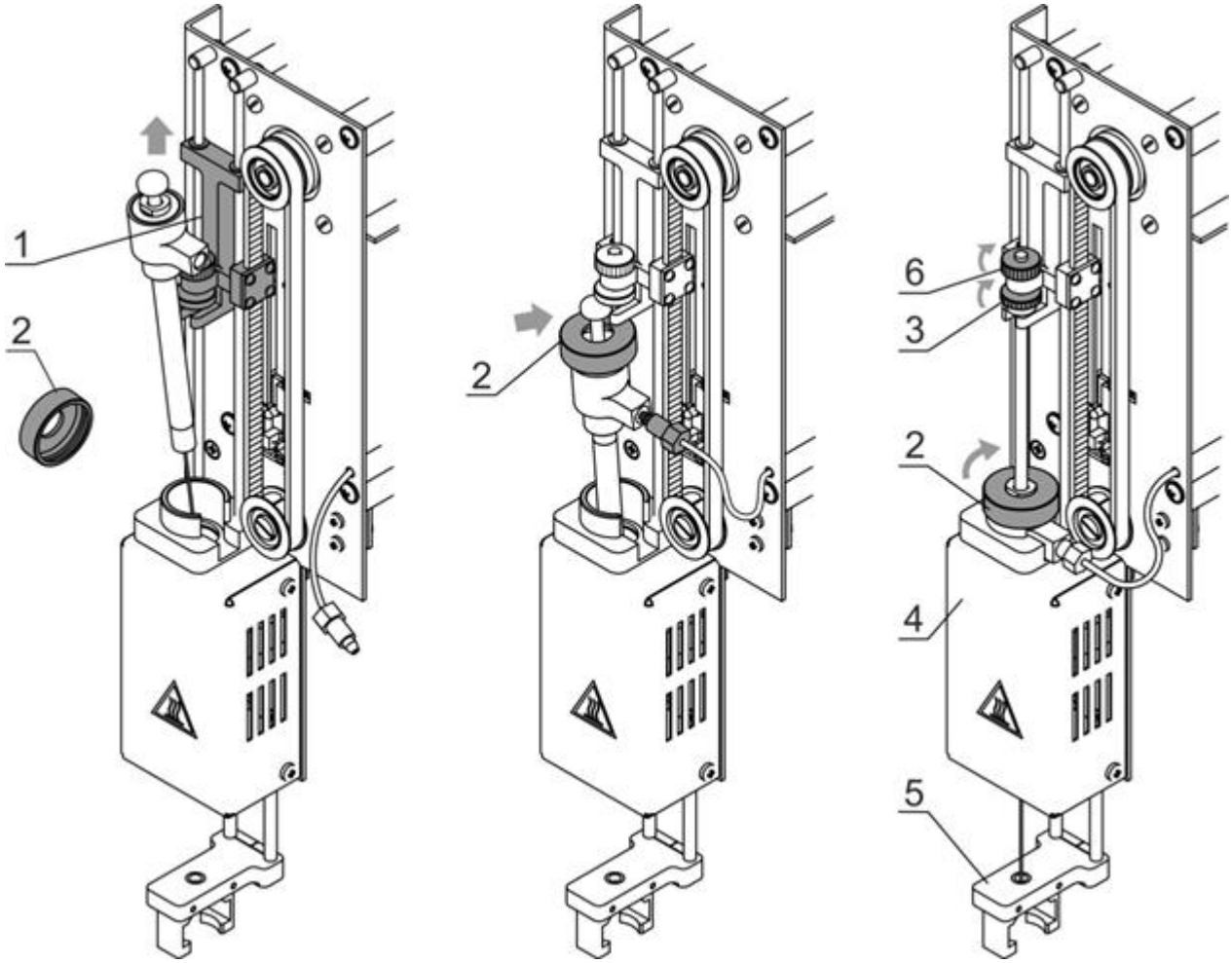


Figure 5.8 – Syringe unit installation

- Remove nut 2 fixing the syringe unit (Figure 5.).
- Shift the carriage 1 upward to the stop.
- Put on the nut 2 on the plunger of syringe unit;
- Connect tube for syringe purge;
- Insert syringe unit into thermostat 4, placing plunger into so-called "folk" on the carriage and moving syringe unit together with carriage smoothly down so that needle of the syringe is inserted into hole of needle adapter 5.
- Screw nut 2 fixing the syringe unit.
- Shift the carriage 1 with syringe plunger downward to the stop.
- Fix the plunger in the carriage with fixing screw 3.
- Fix the lock-nut 6.

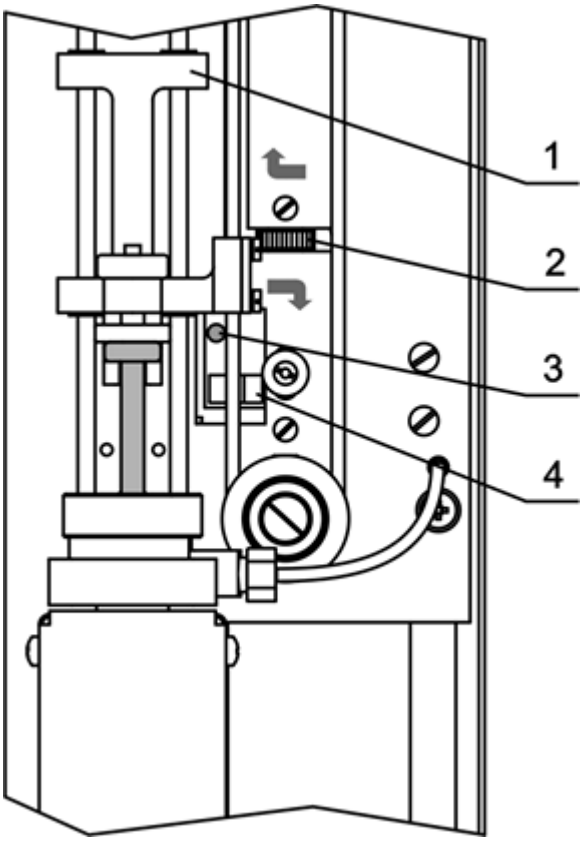
Plunger position adjustment

Whenever new syringe unit installed, proceed with adjustment of optoelectronic sensor 4 of syringe plunger position.

Zero position of syringe plunger refers to Zero scale division of autosampler firmware control. Optimal position of the plunger is that plunger tip located slightly higher of the lowest point (by stop). Correct adjustment of the plunger improves repeatability and diminishes carryover.

Take attention that plunger adjustment procedure is different for different options (Headspace, Liquid or SPME). Follow instructions in relevant sections of User Manual for proper adjustment.

- The Autosampler is turned off before adjustment.
- Rotate the screw 2 to the right to move the board with mounted optical encoder 4 downward up to the stop (down arrow).
- Turn on the Autosampler. Once turned on the Autosampler will try moving the plunger downward to Zero position but fail. The LED 3 is not light; sound of plunger striking against syringe barrel (interrupted crack) is heard.
- Then rotate adjusting screw 2 to the left (up arrow), shift the board with optical encoder 4 upward until the LED 3 gets light permanently. This point corresponds to Zero position of plunger. Then rotate the screw 2 in the same direction (left) at 2 turns (the LED 3 must keep light).



1 – carriage; 2 – adjusting screw to shift board; 3 – LED; 4 – optoelectronic encoder

Figure 5.9 – Plunger position adjustment

Two situations may arise during the above-mentioned adjustment:

- Encoder is located above too much (gap is too big). As a result, the analysis repeatability will be worse;
- Encoder is located too low (LED 3 is not light, 1 – carriage; 2 – adjusting screw to shift board; 3 – LED; 4 – optoelectronic encoder

- Figure 5.), shutter on the carriage cannot overlay encoder, so the stepping motor 2 (1 – tower basement; 2 – stepping motor pulley; 3 – flat toothed belt; 4 – carriage; 5 – syringe unit 214.4.464.103 (or 214.4.464.104); 6 – screw for plunger fitting; 7 – nut for syringe unit fitting; 8 – syringe thermostat; 9 – needle adapter; 10 – guide; 11 – optoelectronic sensor; 12 – syringe purging tube.
- Figure 3.4) doesn't stop. As a result, autosampler operation is not possible, the carriage vibrates with acoustic noise; the screen of the Autosampler displays message "Fatal error. Plunger sensor".

Inlet septum penetration check

Once syringe installed, check manually how needle of the syringe penetrates inlet septum:

- Turn the Autosampler off;
- Move the tower into position above the inlet;
- Gradually move syringe gear downward until needle tip touches septa (your hand is on syringe thermostat);
- Then carefully penetrate septum, move syringe needle down to the stop and return to upper position.

Passage of the needle though the septa must be smooth but not causing leak of the inlet.

5.5 Liquid option installation

Before starting installation of Liquid option, make sure that procedures described in sections 5.2 and 5.3 provided carefully. Check that nut 6.482.013-03 is installed on GC inlet. If not, come back to section 5.2.

Follow procedures:

- Replace needle adapter;
- Fit syringe adapter 6.152.012 (if syringes for liquid injection to be installed, 0.5 … 10 µL) or syringe adapter 6.152.013 (if syringes for ambient headspace injection to be installed, 100 or 250 µL);
- Fit plunger adapter;
- Install syringe;
- Place vial tray for liquid injection.

5.5.1 Installation of needle adapter 6.119.009

Move the tower at the end right & front position (approx. above front inlet); decrease carriage of syringe gear at lower level (Figure 5.).

Loosen 2 screws by hex key and remove installed needle adapter (Figure 5. – a).

Install the adapter 6.119.009. When tightening the screws of the adapter, lift it with one finger by 1 ... 2 cm (Figure 5. – b) and tighten screws with a key without applying excessive force (use the short key shoulder).

Check for ease of sliding by pressing the bottom adapter 6.119.009. If friction or / and jamming of the rails occurs, loosen the fastening screws and repeat the installation step.

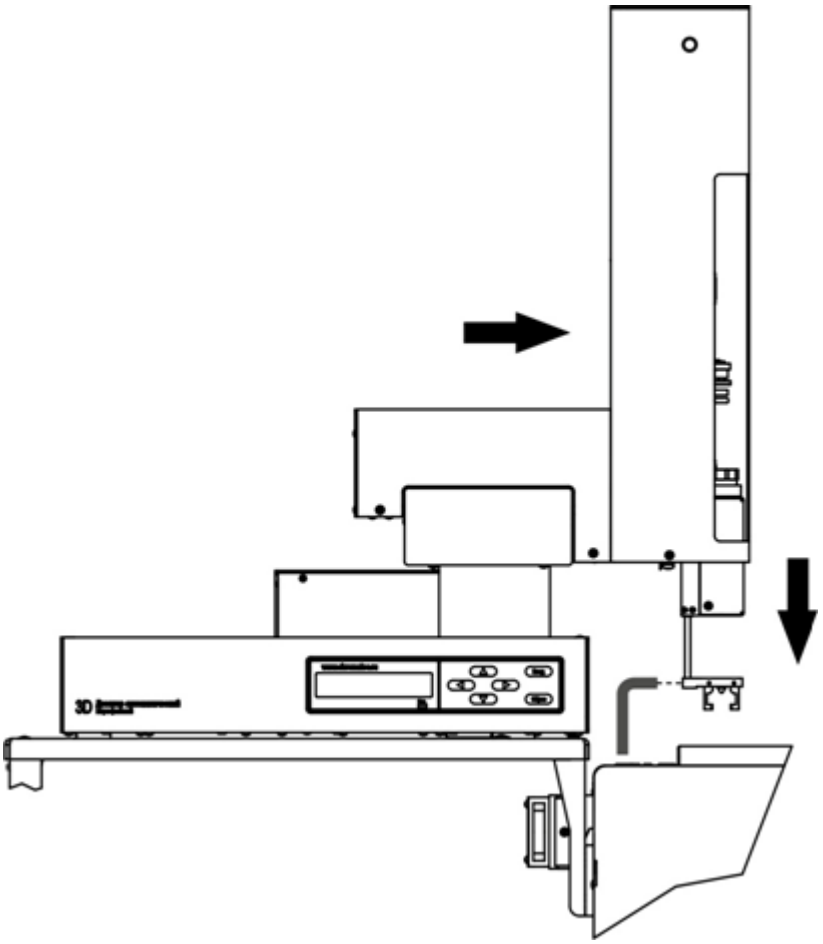


Figure 5.10 – Tower position for needle adapter replacement

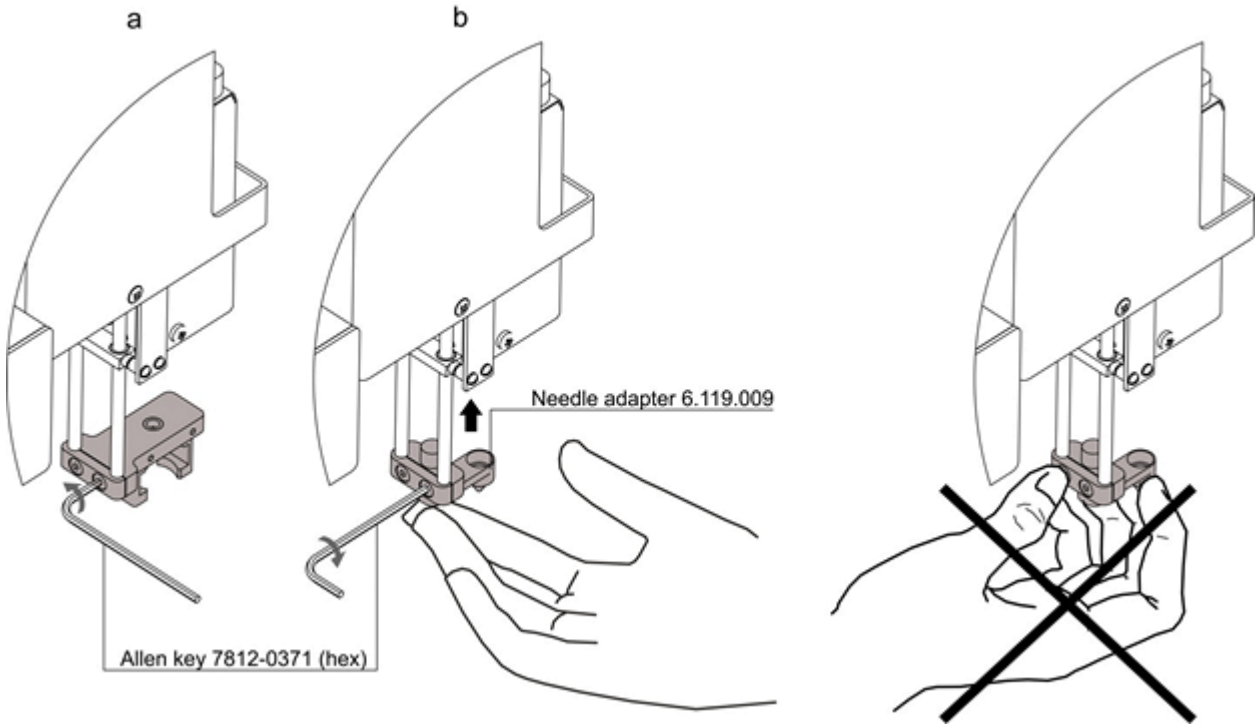


Figure 5.11 – Needle adapter replacement

5.5.2 Installation of Syringe adapter

Before starting this procedure, make sure that Autosampler is turned off and Headspace Syringe unit is removed from the thermostat.

Disconnect removable syringe thermostat as follows:

1) Seize the thermostat by hand; 2) rotating the thermostat, "unstick it from the carriage", overcoming magnets attraction (Figure 5.2 – a).

Fit new syringe adapter on magnets (Figure 5.2 – b).

Once proper syringe adapter installed, the Autosampler identifies its type automatically and switch to relevant operating mode (Liquid injection mode).

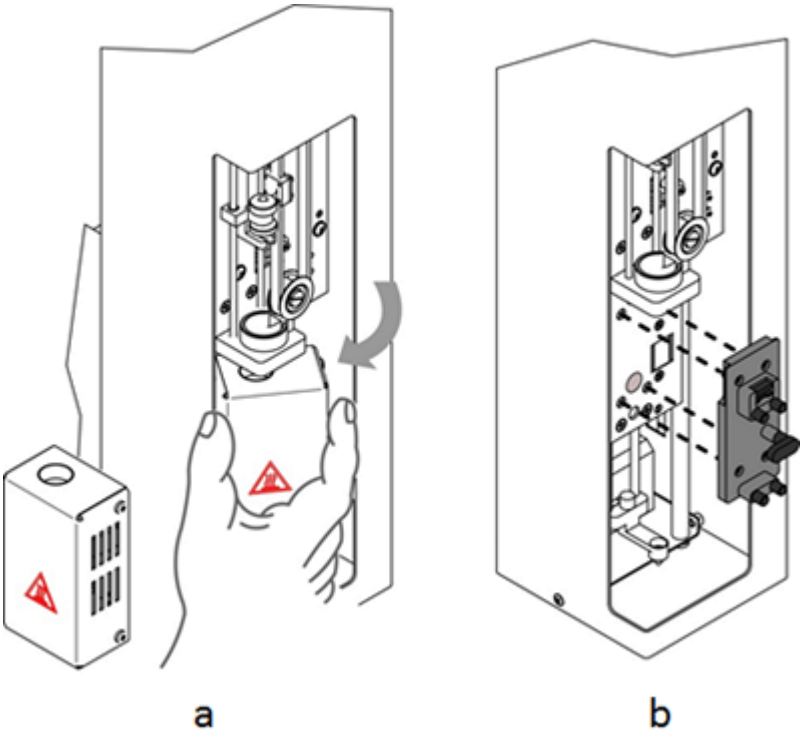
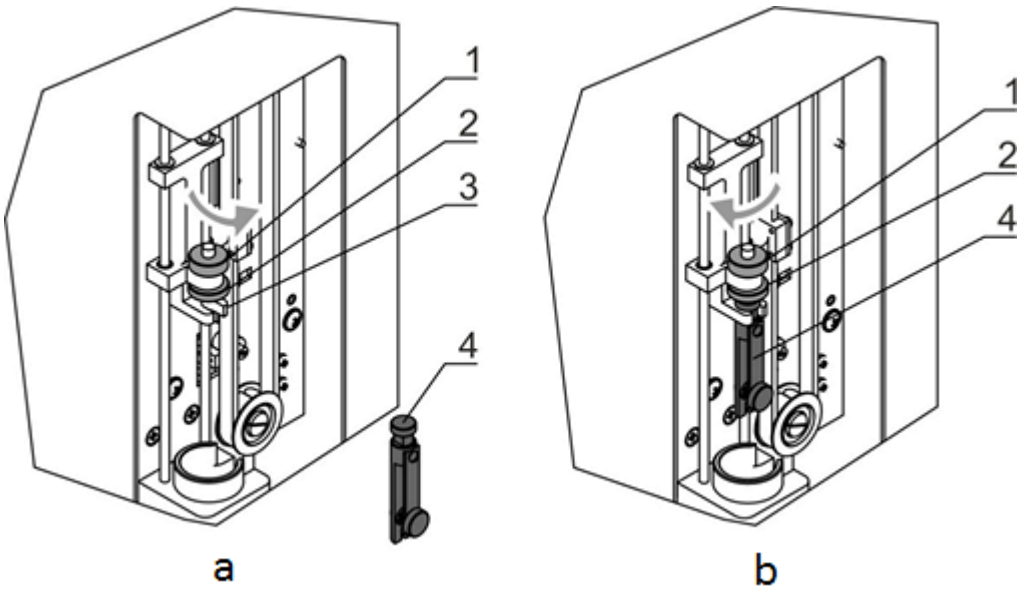


Figure 5.2 – Syringe adapter replacement

5.5.3 Installation of plunger adapter 6.152.015

Loosen lock-nut 1 and rotate screw 2 as shown on the figure below (a). Allow gap between bottom part of the screw 2 and folk of the carriage 3 suitable to place head of plunger adapter 4. Move the carriage 3 upward at 6-7 cm from its lower position (Figure 5.3 – a).

Insert plunger adapter with its head into gap so that head was located under the screw 2. Fix plunger adapter in the carriage by screw 2 and lock-nut 1 (Figure 5.3 – b).



1 – lock-nut; 2 – fixing screw; 3 – carriage; 4 – plunger adapter 6.152.015.

Figure 5.3 – Installation of plunger adapter

5.5.4 Installation of tray 6.150.309

To work with liquid samples it is necessary to install the tray 6.150.309 (take it from the shipping kit).

The tray is placed on the 4 pins of the autosampler base.

To install the tray it is necessary to align the slots on its wall with the pins in the autosampler base. Then smoothly lower the left part of the tray (Figure 5.14).

Once tray installed, the Autosampler identifies its type automatically and switch to proper operating mode.

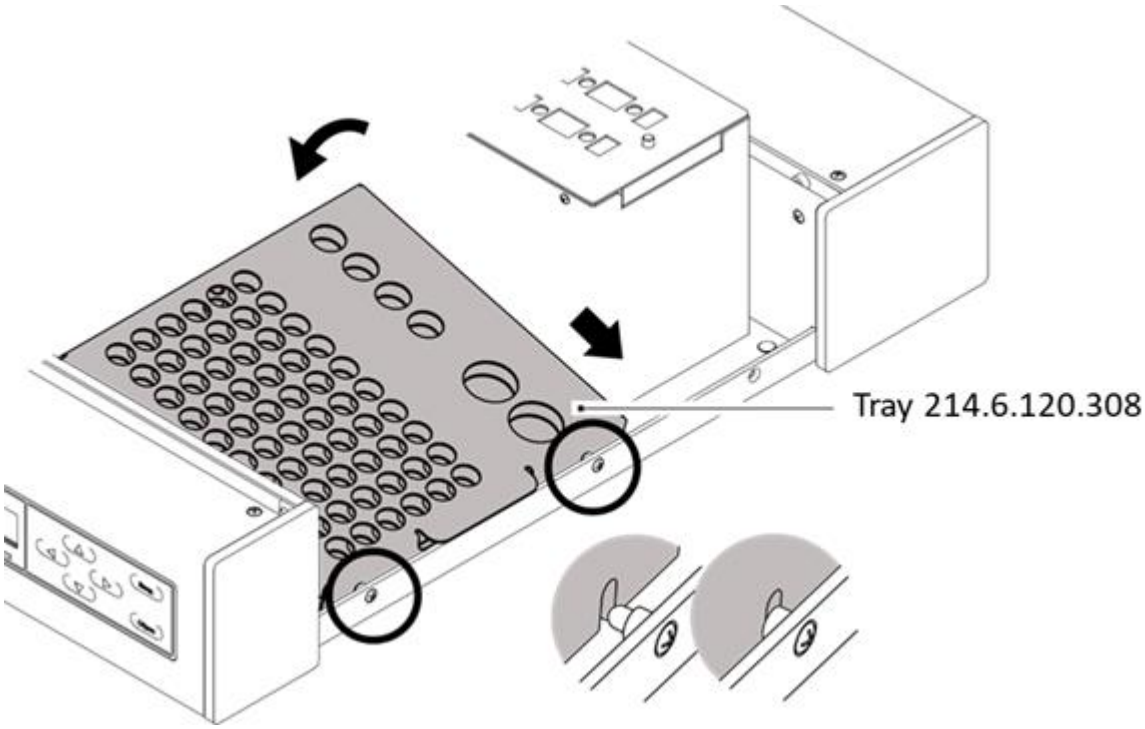


Figure 5.14 – Tray installation

5.5.5 Syringes mounting / dismounting. Plunger adjustment

Turn off the Autosampler before starting this procedure.

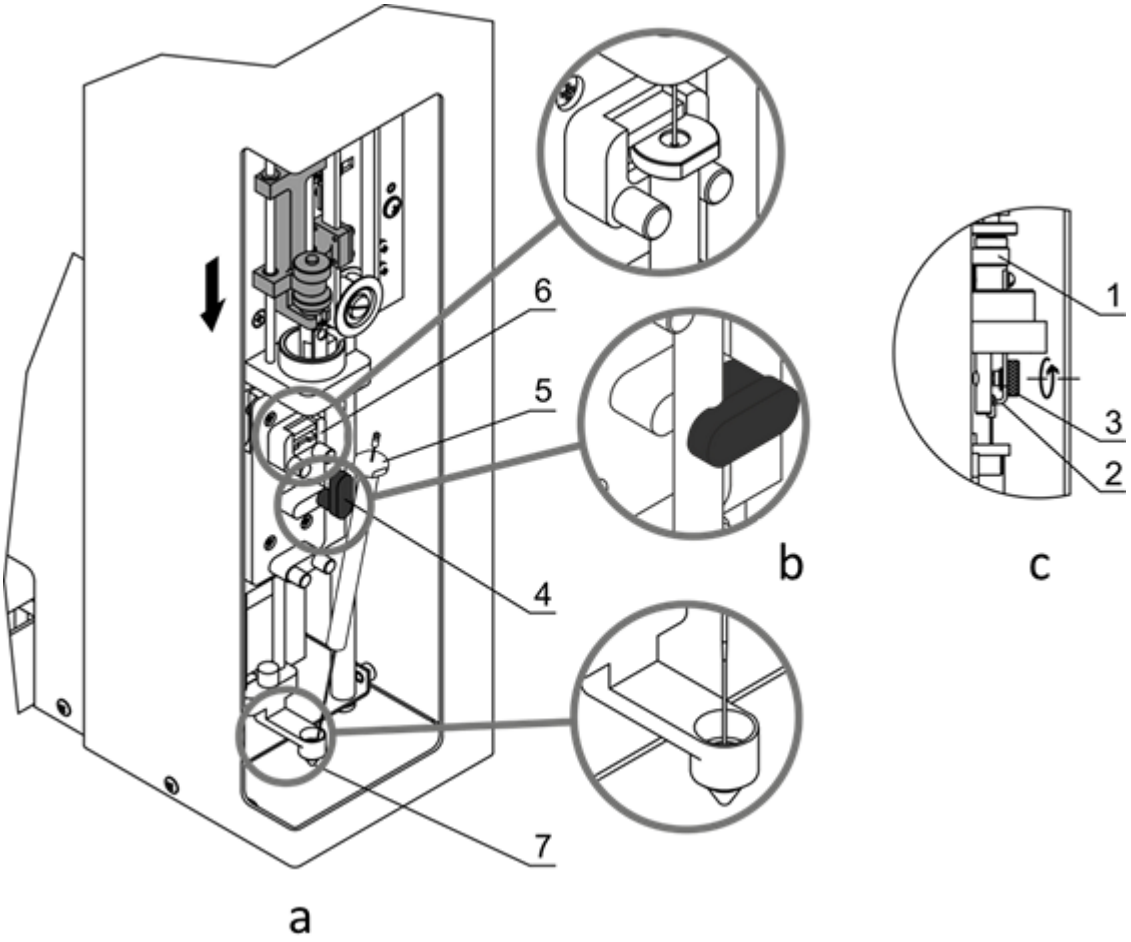
Make sure that suitable syringe adapter has been installed. Follow instruction in section 3.4.4 to select syringe adapter.

Liquid option of the Autosampler is supplied with 10 µL syringes in shipping kit.

To install syringe, follow procedure below:

- Rotate a latch 4 of syringe adapter 6 into vertical position (Figure 5.4 – a);
- Place syringe 5 into seat, insert needle into hole of needle adapter 7 at the same time, push glass barrel into seat up to the end (Figure 5.4 – a);
- Rotate the latch 4 into horizontal position to fix the syringe 5 (Figure 5.4 – b);

- Shift the carriage 1 with plunger adapter downward so that bead of the plunger went into gap between spring plate 2 and carriage body 1 (the gap can be widened by rotating screw 3 anticlockwise, Figure 5.4 – b, c);
- Fix plunger bid by screw 3, do not make excessive force while rotating clockwise (Figure 5.4 – c);



1 – carriage; 2 – spring plate; 3 – screw; 4 – latch; 5 – syringe; 6 – syringe adapter; 7 – needle adapter

Figure 5.4 – Syringe installation

When installing the microsyringe plunger, do not overtighten screw 3 (Figure 5.15 – c).

Syringe unit (or syringe) dismounting can be carried out in reversed order.

Plunger position adjustment

Whenever new syringe is installed, proceed with adjustment of optoelectronic sensor of syringe plunger.

Zero position of syringe plunger refers to Zero scale division of autosampler firmware control. Optimal position of the plunger is that plunger tip located slightly higher of the lowest point (by stop). Correct adjustment of the plunger improves repeatability and diminishes carryover.

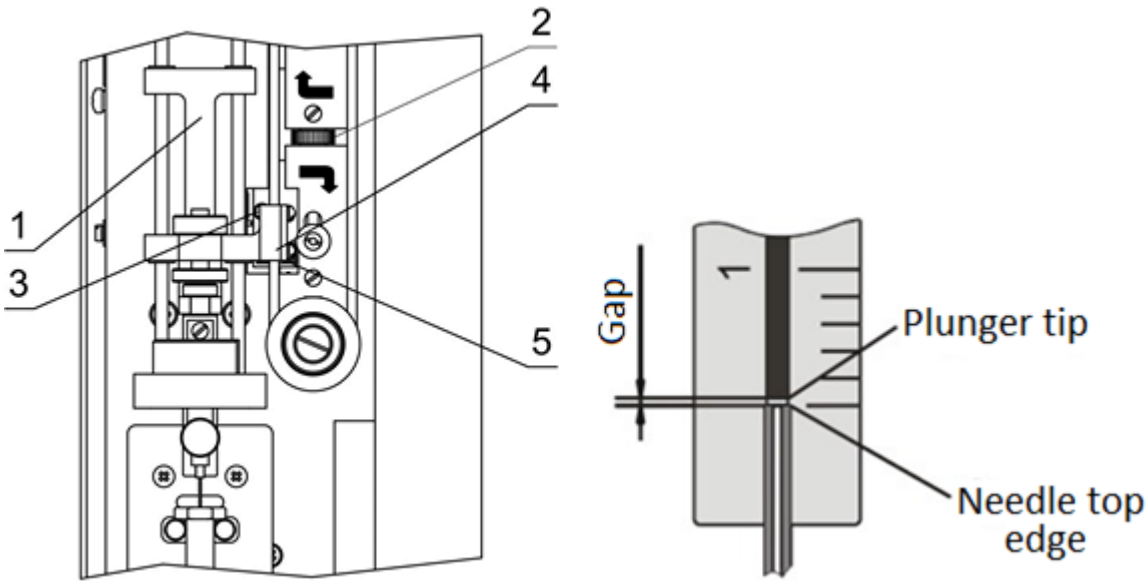
Take attention that plunger adjustment procedure is different for different options (Headspace, Liquid or SPME). Follow instructions in relevant sections of User Manual for proper adjustment. Using incorrect adjustment procedure may damage plunger.

Follow procedure:

- The Autosampler is turned off before adjustment.
- Then, rotate the screw 2 **to the left** to move the board with mounted optical encoder 5 upward up to the stop (up arrow).
- Turn on the Autosampler.
- On the Autosampler display, using buttons ▼▲ pass to menu **Setting**.
- Select option Plunger sensor pos.
- Then rotate adjusting screw 2 to the right (down arrow) shifting the board with optical encoder 5 downward. Press Enter periodically, in 1-3 turns, to update Zero position of the plunger. One full turn corresponds to 0.4mm of plunger shifting.
- Keep on rotating as described above until minimum gap (less than ½ of division scale) between plunger tip and needle top edge is obtained (Figure 5.5).
- Once adjustment completed, press **Enter**.

Carry out a check test to be assured that Autosampler keeps adjustment correctly.

- Turn the Autosampler off.
- Move the carriage upward manually on 2-3 cm.
- Turn the Autosampler on. Once turned on, the Autosampler must move the carriage back to Zero position so the gap is the same as had been adjusted.



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

Figure 5.5 – Plunger position adjustment

Two situations may arise during the above-mentioned adjustment:

- Too big gap corresponding to 0.2-0.5 µl (for syringe 10 µl) leads to the fact that sensor is installed too high. As a result, the analysis repeatability will be worse;

- No gap. This means that optoelectronic sensor is positioned too low (LED is OFF). As a result, sampler cannot operate and software shows "Fatal error. Plunger sensor" message.

Checking the needle passage through the inlet septum

Once syringe installed, check manually how needle of the syringe penetrates inlet septum:

- Turn the Autosampler off;
- Move the tower into position above the inlet;
- Gradually move syringe gear downward until needle tip touches septa;
- Then carefully penetrate septum, move syringe needle down to the stop and return to upper position.

Passage of the needle though the septa must be smooth but not causing leak of the inlet.

5.6 SPME option installation

Before starting installation of Liquid option, make sure that procedures described in sections 5.2 and 5.3 provided carefully. Check that nut 6.482.024 is installed on GC inlet. If not, come back to section 5.2.

Follow procedures:

- Fit vials cramp;
- Install SPME holder adapter;
- Install SPME holder and adjust fiber position sensor;
- Place tray for SPME option.

5.6.1 Mounting vial cramp 6.275.038

This procedure is to be carried out when re-configuring the autosampler from Liquid option to Headspace option. Procedure of mounting / dismounting vial cramp 6.275.038 is the same as described in section 5.5.1.

5.6.2 Installation of SPME holder adapter

SPME holder adapter 6.152.017 is to be fitted on magnets instead of syringe adapter of Liquid option or Syringe thermostat of Headspace option. The procedure of replacement is described in section 5.5.2.

5.6.3 Installation of tray 6.150.233

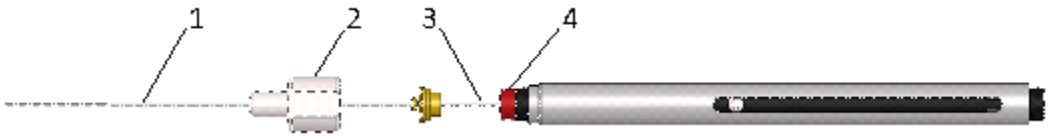
Tray 6.150.314 (30 vials capacity) is intended for using with SPME option only

Installation of the tray is performed in the same way as described in clause 5.4.3

Once tray installed, the Autosampler identifies its type automatically and switch to proper operating mode (SPME).

5.6.4 SPME holder preparation

Before SPME holder installation, make sure that SPME fiber is suitable for operation. The average lifetime of SPME fiber is up to 100 cycles, depending on samples character. Used fibers should be replaced. Replacement procedure is described below.



1 – needle of SPME holder; 2 – nut; 3 – fiber; 4 – nut

Figure 5.6 – SPME holder

Remove fiber from SPME holder as follows:

- Unscrew nut 2.
- Take out needle 1 from nut 2.
- Unscrew nut 4.

Install new fiber into SPME holder as follows:

- Screw the nut 4.
- Insert needle 1 into nut 2.
- Screw nut 2.

5.6.5 SPME holder mounting / dismounting. Fiber position adjustment

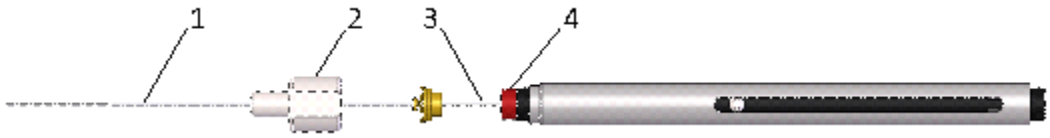
Turn off the Autosampler before starting this procedure.

Make sure that suitable syringe adapter has been installed. Follow instructions in section 3.4.4 to select proper syringe adapter.

Follow procedure below to install SPME holder:

- Move plunger into upper position, fiber must be hidden inside needle of the holder (Figure 5.7 – a);
- Put the insert 2 on the syringe 1 (Figure 5.7 – a);
- Move the carriage 3 at maximum upper position (Figure 5.7 – b);
- Install syringe 1 with insert 2 into seat (insert needle into hole of needle adapter 4 at the same time) (Figure 5.7 – b);

- Fit the nut 5, join it with insert 2 on the syringe; place the plunger into so-called "folk" on the carriage, then move the holder together with the carriage smoothly down so that needle is inserted into hole of needle adapter 4 (Figure 5.7 – c).



1 – SPME holder; 2 – insert 7.840.056; 3 – carriage; 4 – needle adapter; 5 – nut; 6 – fixing screw; 7 – locknut.

Figure 5.7 – SPME holder installation

Fiber position adjustment

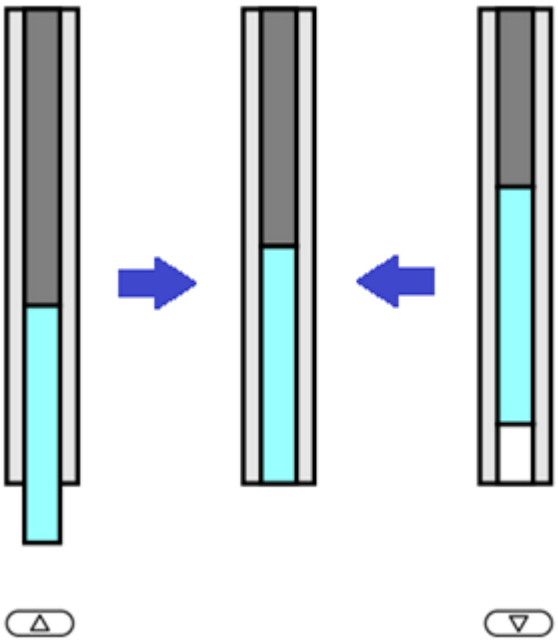
Once SPME holder is installed, proceed with adjustment of optoelectronic sensor of fiber position.

Optimal position (Zero position) of the fiber is that fiber tip located directly on the same level as needle tip. Correct adjustment of fiber position prevents it from damage and diminishes carryover.

Take attention that plunger (fiber) adjustment procedure is different for different options (Headspace, Liquid or SPME). Follow instructions in relevant sections of User Manual for proper adjustment.

Follow procedure:

- Turn on the Autosampler. Once turned on, the Autosampler mover the carriage upward to prevent fiber from damage.
- On the Autosampler display, using buttons ▼▲ pass to menu **Setting**.
- Select option **Plunger sensor pos.** Press **Enter**. The carriage with plunger will move to factory setting of Zero position.
- Then using buttons ▼▲ adjust fiber position in such a level that its tip is located on the same level as the needle tip (Figure 5.8 – in the middle). For observation convenience raise the needle adapter 4 slightly. (Figure 5.7 – c).
- Once the adjustment is completed, press **Enter.**



Incorrect – Correct – Incorrect

Figure 5.8 – SPME fiber position adjustment

Carry out a check test to be assured that Autosampler keeps adjustment correctly.

- Turn the Autosampler off.
- Move the carriage upward manually on 2-3 cm.
- Turn the Autosampler on. Once turned on, the Autosampler must move the carriage back to Zero position so the fiber does not protrude from the needle.

5.7 Loading vials

Sample vials and solvent vials (or solvent bottle) must be carefully washed and dried.

For liquid option: to improve repeatability in series of analyses from one vial and reduce sample loss due to vaporization from vial, the insert 8.214.061 (supplied in shipping kit) is recommended to fit into vial cap above septa.

- Fill solvent vials with suitable solvent (for liquid option only).
- Fill sample vials with samples to be analyzed;
- Install the vials into tray.

5.8 Coordinates tuning

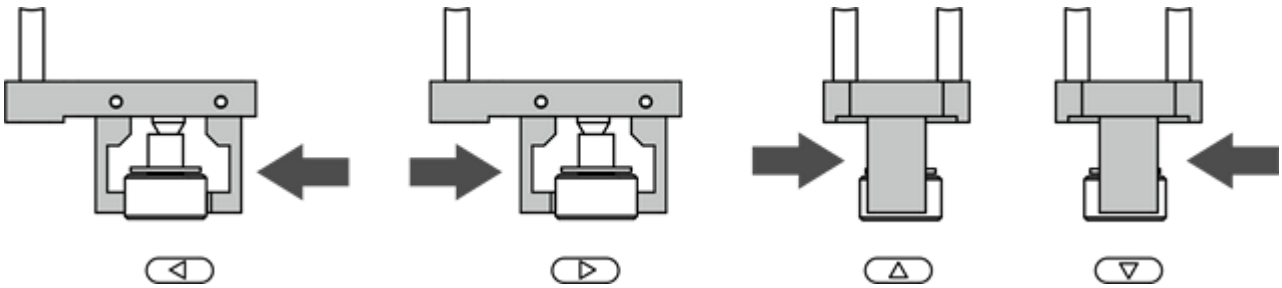
5.8.1 Coordinate tuning for Headspace option

Inlet coordinate tuning.

- Turn the autosampler on. Autosampler's display reflects current status.
- Use the display and buttons on the front panel of the Autosampler to tune 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** (generally inlet 1 is front).
- 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. 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 2 mm.
- Visually estimate how precisely needle adapter is located above center of the inlet. Use buttons ◀ ▶▲▼ to move the tower so that needle adapter cone was located finely above inlet hole, on the same vertical axes (Figure 5.).



Front view – Right view

Figure 5.20 – Fine adjustment of needle adapter position

- 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.

Sample vial "in tray" coordinate adjustment.

- Place a pattern 8.126.091 (from shipping kit) into vial position #1 of the tray instead of sample vial.

Correct location of the pattern is shown on Figure 5. *(left), and incorrect location (right).*

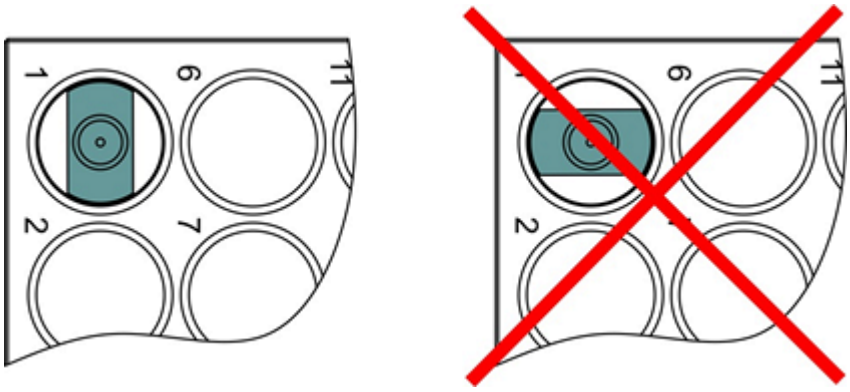


Figure 5.21 – Pattern location on the tray

- Turn the autosampler on. Autosampler's display reflects current status.
- 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 mark on the pattern (Figure 5.9).

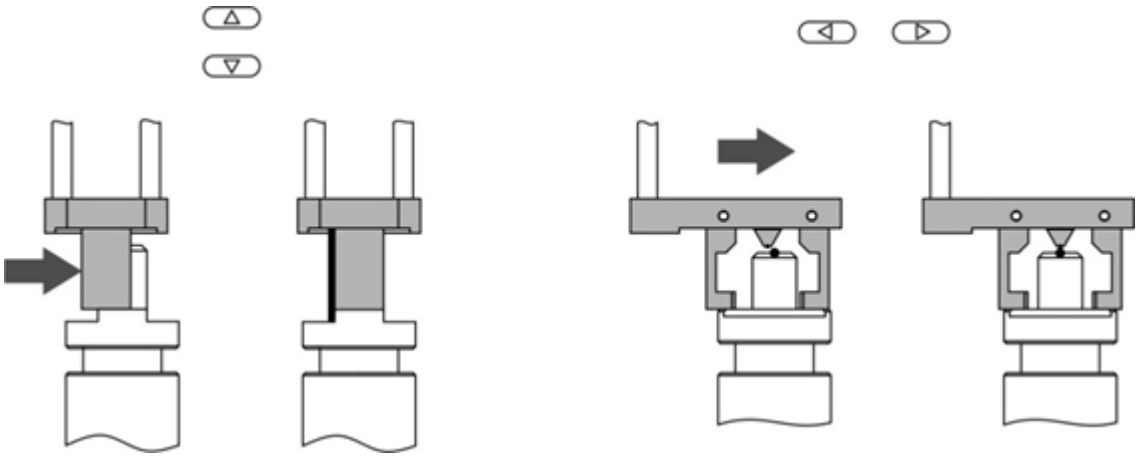


Figure 5.9 – Vial coordinate adjustment

- Set ENTER – apply.

Never use sample vial instead of pattern when vial coordinate adjusting.

The Autosampler will provide computing this coordinate and checking accuracy of setting. As soon as the towers comes back to home position, **Vial in tray** coordinate adjustment is completed.

Remove the pattern from the tray after setting completed.

Sample vial "in incubator" coordinate adjustment.

- Slide back incubators cover by hand. Place a pattern 8.126.091 (from shipping kit) into vial position #1 of the incubator.

Correct location of the pattern is shown on Figure 5.10 *(left), and incorrect location (right).*

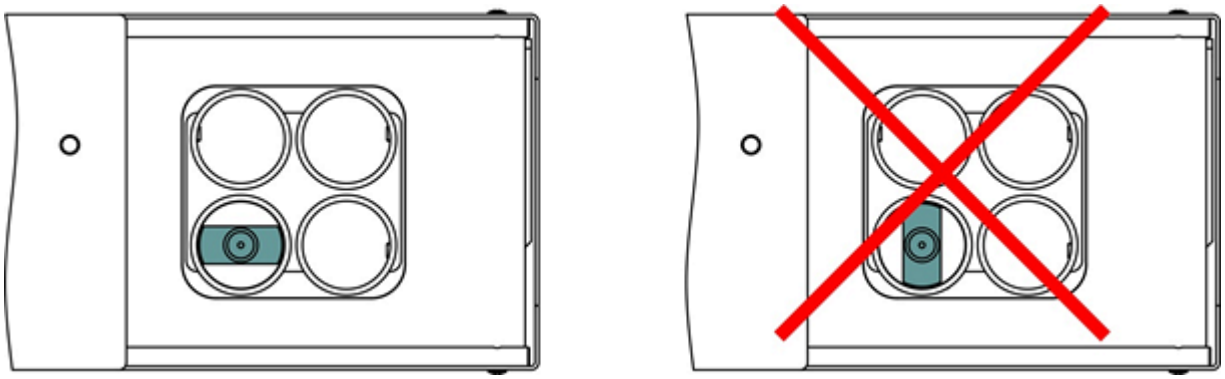


Figure 5.10 – Pattern location on the incubator

- Using buttons ▼▲ pass to menu **Setting**.
- Select option Incubator 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 mark on the pattern (Figure 5.9).
- Set ENTER – apply.

Never use sample vial with septa instead of the pattern. This will cause errors in autosampler operation.

As soon as the tower comes back to home position, **Vial in incubator** coordinate tuning is completed.

Remove the pattern from the incubator after setting completed.

Sampling position coordinate adjustment.

- Using buttons ▼▲ on the front panel, pass to menu **Setting**.
- Select option Sampling position.
- The Autosampler will perform factory settings automatically. The tower must go down to contact the pattern.

This tuning is required to confirm their coordinate (height) when sampling.

5.8.2 Coordinate tuning for Liquid option

Inlet coordinate tuning.

- Turn the autosampler on. 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 **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. 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 2 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.

Sample vials coordinate adjustment

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

- Place a pattern 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.

Never use sample vial with septa instead of the pattern. This will cause errors in autosampler operation.

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

Remove the pattern from the tray after setting completed and place vial back.

Solvent vials coordinate adjustment.

- 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.

Waste vials coordinate adjustment

- 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.

5.8.3 Coordinate tuning for SPME option

Inlet coordinate tuning

- Turn the autosampler on. 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 **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 Inlet1 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. 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 2 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.

Sample vial "in tray" coordinate adjustment

- Place a pattern 8.126.091 (from shipping kit) into vial position #1 of the tray instead of sample vial.

Correct location of the pattern is shown on Figure 5. *(left), and incorrect location (right).*

- Turn the autosampler on. Autosampler's display reflects current status.
- 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 mark on the pattern (Figure 5.9).
- Set ENTER – apply.

Never use sample vial instead of pattern when vial coordinate adjusting.

As soon as the towers comes back to home position, **Vial in tray** coordinate tuning is completed.

Remove the pattern from the tray after setting completed.

Sample vial "in incubator" coordinate adjustment.

- Slide back incubators cover by hand. Place a pattern 8.126.091 (from shipping kit) into vial position #1 of the incubator.

Correct location of the pattern is shown on Figure 5.10 *(left), and incorrect location (right).*

- Using buttons ▼▲ pass to menu **Setting**.
- Select option Incubator 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 mark on the pattern (Figure 5.9).
- Set ENTER – apply.

Never use sample vial with septa instead of the pattern. This will cause errors in autosampler operation.

As soon as the tower comes back to home position, **Vial in incubator** coordinate tuning is completed.

Remove the pattern from the incubator after setting completed.

Fiber bakeout coordinate tuning

- Using buttons ▼▲ on the front panel, pass to menu **Setting**.
- Select option Fiber heater pos.
- The Autosampler will perform factory settings automatically. The tower must go down to contact the pattern.
- Using buttons ◄►▼▲ move the tower precisely at the hole of fiber heater (Figure 5.11).

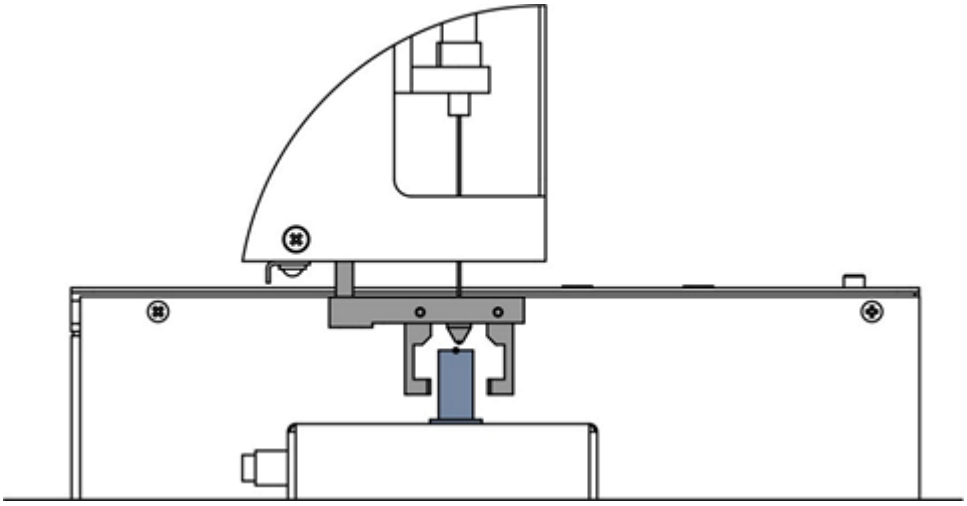


Figure 5.11 – Fiber bakeout coordinate tuning

- Set ENTER – apply.

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

Sampling position coordinate adjustment

- Using buttons ▼▲ on the front panel, pass to menu **Setting**.
- Select option Sampling position.
- The Autosampler will perform factory settings automatically. The tower must go down to contact the pattern.

This tuning is required to confirm their coordinate (height) when sampling.

6 Autosampler operation

Autosampler operation is carried out via "Control panel 2.0" software.



Work with the "Control Panel" is described in the " Chromatec Analytic" User Manual.

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.

6.1 Instrumental method for headspace injection

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

Set parameters:

Syringe temperature. Set in the range of 40 to 150°C. The recommended temperature value is equal to or slightly above the sample temperature.

Incubator temperature. Set temperature of sample incubation within a range from 40 to 170°C. Maximum temperature for aqueous samples must not exceed 95°C. Recommended value is 80°C.

Shaker control

This parameter includes:

- Shaking, sec,
- Delay, sec,
- Frequency, rpm.

If Shaking time is set as 0, shaker doesn’t work. If delay time is set as 0, shaker keeps on constantly except time when vial loading / unloading, and sampling occurs. Other set values allow flexible setting of shaking intervals.

Frequency (in rotations per minute – rpm) is the speed of shaker rotation.

Syringe purge before sampling

- Pre-sampling purge time. This parameter can be determined in practice; it depends on sample carryover character. Recommended purge time at least 1 minute.
- Volume pre-fill. If this volume of inert gas is set above zero, the set volume will remain in the syringe barrel after purging and it to be transferred into vial once needle penetrated vial septum before aspiration of sample.

Sampling

- Volume, ml. Depending on the syringe capacity the volume can be set up to 1 or 2.5 ml.
- Sampling speed, ml/min. The speed of plunger aspiration. Generally Sampling speed and Injection speed are set at the same level.
- Number of strokes. Set required number of strokes (sample homogenization), generally, not more than 10.

- Plunger delay, sec. Recommended value is 10 seconds. At delay, plunger is kept in upper position to even pressure in syringe barrel and sample vial.

Injection

- Pre-injection delay, sec.
- Post injection delay, sec. Recommended value 5 seconds or more.
- Injection speed. Set at 5 or 10 ml/min less than carrier gas flow rate in the inlet.
- Inlet needle depth, mm

Syringe purge after injection. Recommended value is from 3 to 10 minutes.

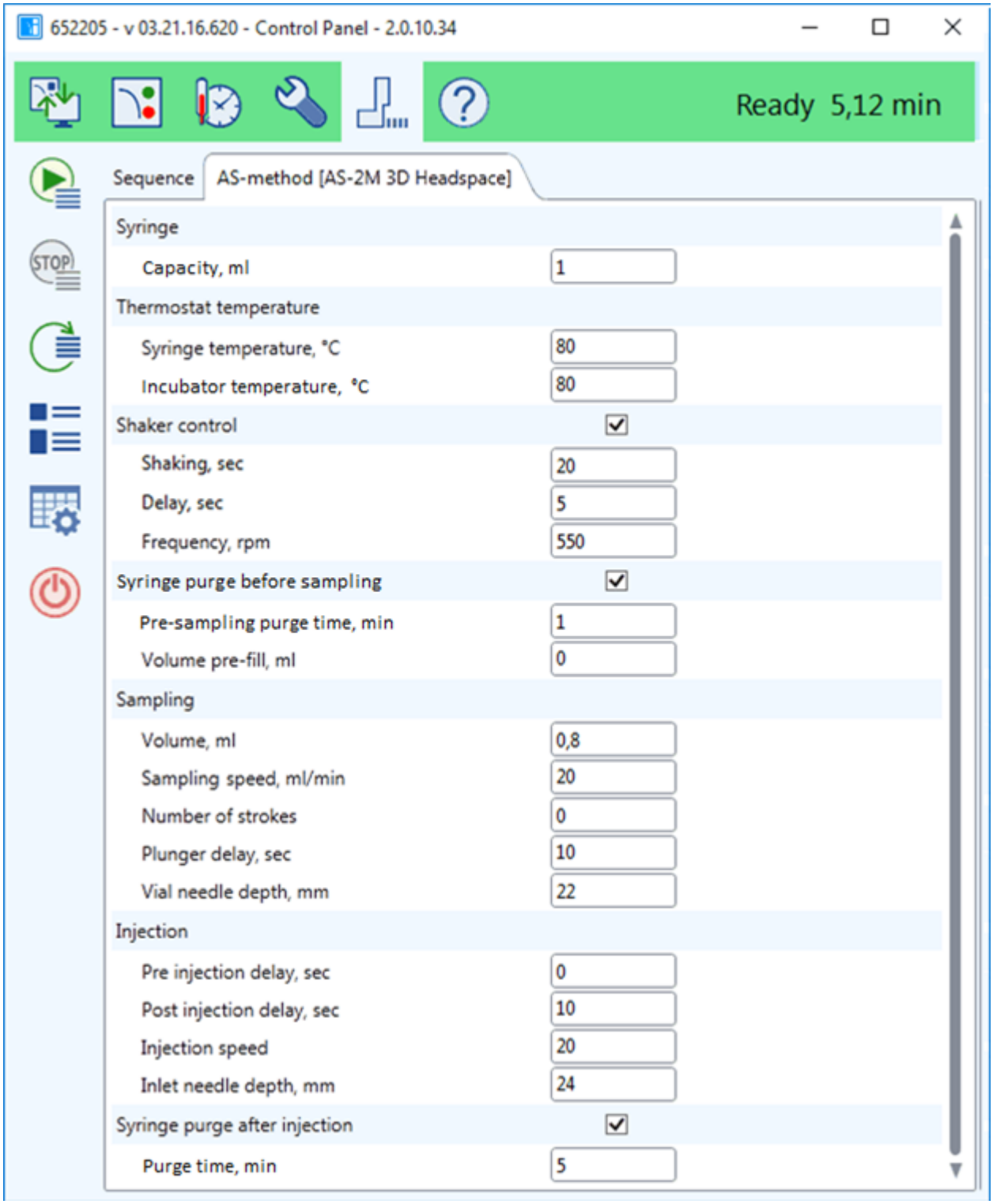


Figure 6.1 – AS-2M (3D) headspace operating mode

For AS-2M liquid autosamplers, the autosampler mode can be set individually for each analysis in the sequence.

Operating mode for **AS-2M (3D) Headspace** is the same for all analyses in sequence. This is intentional feature as "Control panel" applies "overlapping for sample preparation time" to reduce total time for sequence fulfillment. Loading of the next sample vial into incubator occurs while previous run is going on (not completed).

The autosampler loads the next sample vial into its incubator without waiting for the previous analysis to complete. The "Control panel" determines the sequence of operations to be performed by the autosamplers to avoid unnecessary downtime.

The **Incubation** and **Dwell time** parameters are set on the Analyses tab individually for each analysis.

The parameter in the "**Delay, min**" column should be greater than the time taken by the chromatograph to prepare for the next analysis.

Autosampler operation steps can be monitored on diagram of Sequence tab.

Table 6.1 – Color symbols on diagram

Color	AS activity
	Autosampler carries out mechanical movements within this time (loading or unloading vials, syringe purging). This time is not specified by User, it is determined by autosampler design and operating mode.
	Vial incubation time. Set **Incubation time** on the **Sequence** tab individually for each analysis run.
	Autosampler carries out sample aspiration. This time is not specified by User, it is determined by autosampler design and operating mode.
	Mark of analysis run start
	Mark of second injection (in the same run cycle).
	GC analysis run time
	Dwell time after analysis. Set **Dwell time** on the **Sequence** tab individually for each analysis run.
	Analysis run completed.

6.2 Instrumental method for headspace injection

The setting is made when the "Liquid Sample Injection" option is installed

Unlike the "Headspace Injection" case, when working with liquid samples, the operator can create an individual autosampler instrumental method for each analysis.

To add new AS method for analysis:

- Click on cell AS-method.
- In the dropped menu select – **Create new method**.

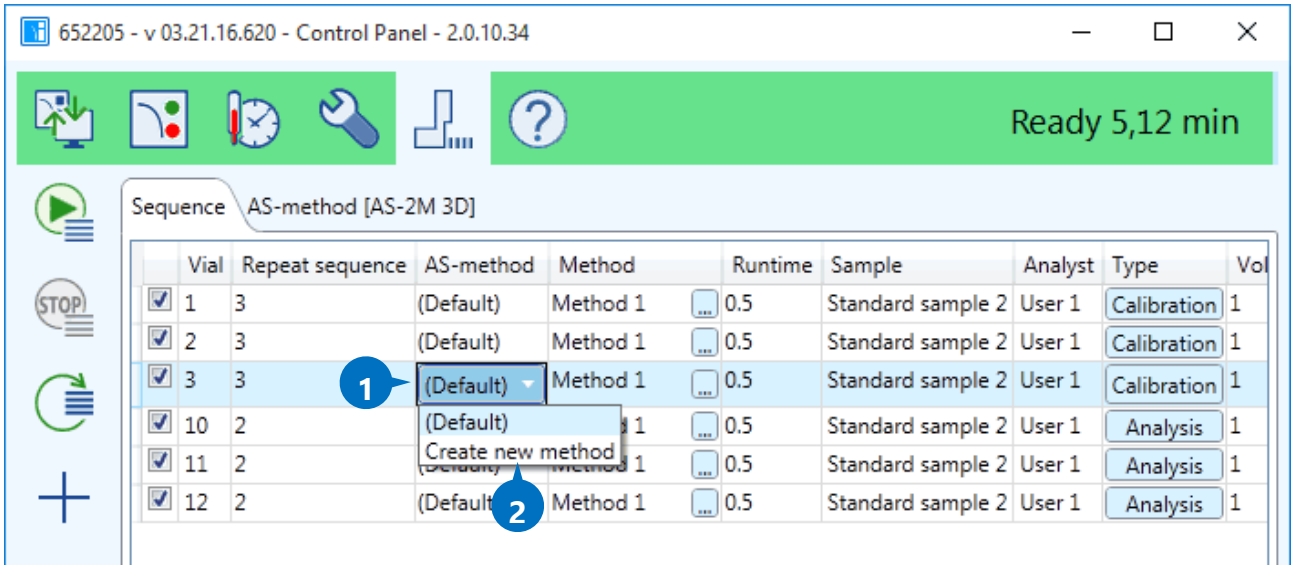


Figure 6.2 – Adding AS method for analysis

Pass into the tab **AS-method [AS-2M 3D]**.

Also new AS method can be created on the tab **AS-method [AS-2M 3D]** using appropriate button.

If you will always use the same AS method, you may skip creating new method, just go the tab **AS-method [AS-2M 3D]** and set method on default. In this case in the cell **AS-method** shall be indicated **(Default)**.

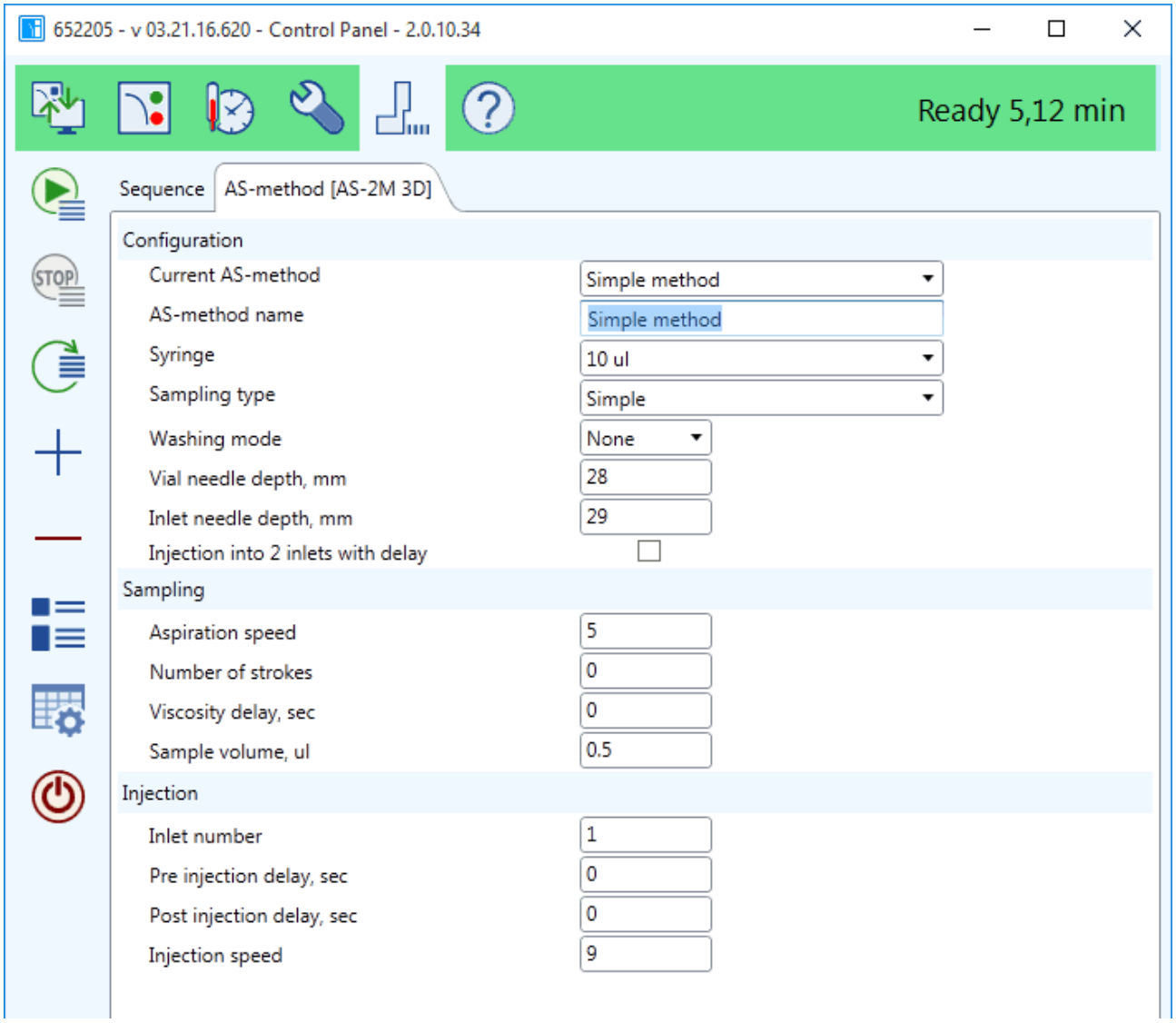


Figure 6.3 – AS method for AS-2M (3D) liquid

Set Configuration parameters.

Select **Syringe**. Specify capacity depending on the syringe installed

Select **Sampling type**:

- **Simple**. For Simple injection technique (the whole volume of injection is filled with sample). Parameter **Number of strokes** is available.
- **Sandwich**. Injected sample is located between 2 air plugs. Strokes are not available.
- **Sandwich with solvent**. Five layers included when sampling: first layer is top air plug ("top"), then solvent (from specified vial), then middle air plug ("middle"), then sample from target vial, and finally bottom air plug ("bottom"). Volume for each layer can be set. Strokes are not available for this mode.
- **Sandwich with internal standard**. Five layers included when sampling: first layer is top air plug ("top"), then internal standard (from specified vial), then middle air plug ("middle"), then sample from target vial, and finally bottom air plug ("bottom"). Volume for each layer can be set. Strokes are not available for this mode.

- **Multiple injection**. For Simple injection technique. Parameter **Number of strokes** is available. Parameter **Number of injections** is available. Autosampler injects sample from the same vial several times, then give a commend START to the GC.

Set Washing mode:

- **None**. No solvent washing applied.
- **From vials**. Autosampler carries out washing by solvent using solvent vials A, B, C and D. Either using single solvent or 2 solvents are available. Set **Solvent volume** to be aspirated and **Number of washing cycles**.

Set **Vial needle depth** (mm). On default 28 mm depth is set. It is maximum depth for vial. If the sample contains sediment on the bottom or you need to analyze top fraction liquid after extraction, reduce vial needle depth.

Set **Inlet needle depth** (mm). On default 29 mm depth is set. It is maximum depth for inlet injection. Changing this parameter depends on features of test method, sample character and location of glass wool in the liner of inlet.

Configure the **Solvent Washing** options. If solvent washing is does not need to be performed, uncheck the box.

Set parameters **Sample washing**. Is sample washing is not required, remove a tick.

Sample washing is available if **Washing mode = From vials** only.

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.

6.3 Features of sequence setting for liquid samples

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.

AS method for SPME option

This setting can be carried out when SPME option have been installed.

To set method for autosampler, click to the tab **AS-Method [AS-2M 3D SPME]**.

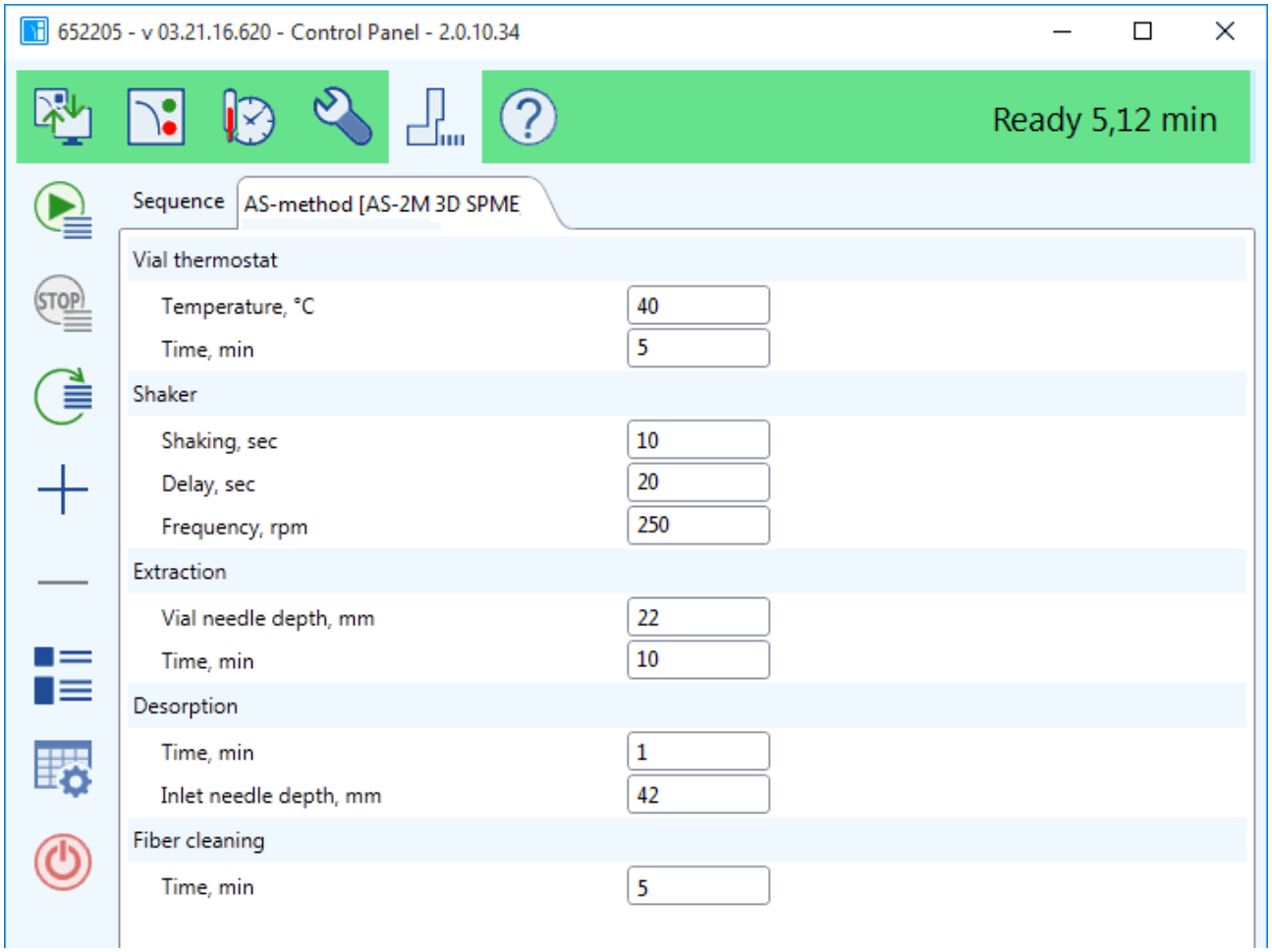


Figure 6.4 – AS method for AS-2M (3D) SPME

Set parameters:

Vial thermostat

Temperature. Set temperature of sample incubation within a range from 30 to 170 °C. Recommended temperature for aqueous samples is from 40 to 70 °C. If the temperature is above 70 °C, the analytes may desorb from the fiber. If you want to carry out extraction at room temperature (without heating) set to 0 °C.

Time, min.

Set incubation time, recommended value 5 minutes if shaker is on. You may skip the incubation step, if setting 0 minutes.

Shaker

- Shaking, sec.
- Delay, sec.
- Frequency, rpm.

If Shaking time is set as 0, shaker doesn't work. If delay time is set as 0, shaker keeps on constantly except time when vial loading / unloading, and sampling occurs. Other set values allow flexible setting of shaking intervals.

Frequency (in rotations per minute – rpm) is the speed of shaker rotation.

Extraction

Vial needle depth. Set this parameter within a range from 22 to 31 mm from septum level. The value 22 mm is set on default. For this level, use 10 – 15 ml of liquid sample volume for SPME extraction from headspace phase, and 18 ml for SPME extraction from liquid phase.

If sample volume is different from recommended values, set vial needle depth so that fibers tip was located at least 3 mm above upper edge of meniscus of liquid in vial for SPME extraction from headspace phase. In case of SPME extraction from liquid phase, fibers tip must be immersed into liquid sample at 9 mm from the bottom edge of meniscus.

Time, min. Recommended value is 20-30 minutes. Range of possible values is from 0.1 to 999 min.

Desorption

Inlet needle depth. Set this parameter within a range from 35 to 50 mm from septum level. The value 42 mm is set on default.

Time, min. Recommended value is 1-2 minutes. Range of possible values is from 0.1 to 999 min.

Fiber bakeout

Time, min. Recommended value is 5 minutes. Range of possible values is from 0 to 999 min. You may skip the fiber bakeout, if you set 0 min.

6.4 Troubleshooting

The following are possible malfunctions of the autosampler that can be eliminated by the user, their causes and methods of elimination. In case of other malfunctions contact the manufacturer or his regional representative.

To prevent liquid sample ingress into the syringe, make sure that there are no drops of liquid under septa of vial before placing it into tray.

Drops of liquid under septa may appear due to excessive shaking speed of vial. Some liquid may ingress into syringe needle while penetrating septum, this resulted in poor repeatability and carryover. Carefully wash and purge the syringe to fix this problem.

Table 6.2 – Troubleshooting for headspace option

Type of failure	Possible cause	Remedy action
The repeatability within sequence of analyses goes beyond the acceptable relative RSD value as to peak heights and/or areas	GC inlet is not leak-free.	Check the air-tightness of GC inlet septa and gas line connections. Tighten or replace the septa if necessary.
	The following operating modes are not optimized: sample injection speed; Incubation time; vial temperature.	Optimize Autosampler operating mode.

	Liquid sample ingress into syringe from vial.	Fit the syringe into Autosampler. Dry it with carrier gas in purging mode.
	Syringe plunger wear or needle plugged with septum particles.	Replace the syringe unit. Clean the needle if possible.
Carryover. Chromatogram contains peaks relating to previous sample	No purging or insufficient purge time of the syringe or its needle due to short purging time or low temperature.	Increase the syringe temperature and purge time.
	Liquid sample ingress into syringe from vial.	Fit the syringe into Autosampler. Dry it with carrier gas in purging mode.

6.4.1 Troubleshooting for Liquid option

Table 6.3 – Troubleshooting for Liquid option

Problem	**Possible cause**	**Remedy action**
Seizing or blocking of plunger in syringe of syringe unit during operation (especially after storage)	Deposits presented in gap between plunger and channel in syringe barrel due to dried sample residuals	Follow cleaning procedure described in section 7.2.2. If cleaning doesn't help, take new one.
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 method of the autosampler
	Liner configuration is not suitable for this sample	Change liner
	Wearing of syringe or syringe of syringe unit	Replace the syringe unit or syringe
Carryover. Chromatogram contains peaks relating to previous sample	Insufficient washing of the syringe	Increase number of strokes when solvent washing.

6.4.2 Troubleshooting for SPME option

Table 6.4 – Troubleshooting for SPME option

Problem	Possible cause	Remedy action
No analytes peaks or poor peaks response	Inlet septa leakage	Properly tighten the septum nut or replace the inlet septa.
	Vial septa leakage	Check vial cap sealing, adjust the crimper.
	Extraction conditions is incorrect	Change the temperature, the extraction time, the depth of fiber immersion, sample volume, etc.
	Fiber coating is damaged, or life time is ended	Replace the fiber.
	Incorrect SPME fiber	Choose the appropriate fiber for your application.
Ghost peaks on chromatogram	Fiber is not conditioned	Condition the fiber.
	Carryover from previous analysis on the fiber	Increase temperature and/or desorption time.
Poor reproducibility	The extraction is carried out at room temperature	Set the extraction temperature, e.g. 40 °C.
	Analytes are not fully extracted from solid sample	Grind the solid sample or dissolve it in a suitable solvent
	Not enough time to reach the equilibrium	Increase the extraction time.
	Competing compound displaces analytes	Reduce the extraction time.
Needle bends during injection into GC inlet	Inlet septa is too hard	Use soft silicone septa with partially predrilled hole, e.g. BTO (SGE), AG3 (CRS).
	Inlet position is not adjusted	Check inlet position adjustment.
Needle bends during injection into sample vial	Vial septa in too thick	Use vial septa with thickness 1 mm, e.g. SU860106 (Supelco).
	Vial oven position is not adjusted	Check vial oven position adjustment.
Fiber breaks during injection into GC inlet	Inlet liner is packed with glass wool.	Use liners without glass wool, e.g. liner 7.352.066.
	The end of the needle is plugged with a piece of septum.	Septum nut is overtightened. Slightly loosen the septum nut.

6.5 Error messages

At Autosampler operation 2 types of errors may display on the autosampler panel: Error and Fatal Error. List of *Errors* and *Fatal errors* messages, as well as relevant possible cause and remedy actions are described below (Table 6.5, Table 6.6).

Error is related to troubles in Autosampler operating algorithm. Error doesn't interrupt Autosampler operation through a Schedule, after an error cause has been fixed; the Autosampler goes on running by a Schedule. For example, User may take out a waste vial or solvent vial to handle with it (to empty waste vial or load solvent vial). If the autosampler didn't find a vial, it waits until User place it back and press Enter, then the Autosampler continues a Schedule.

If sample vial is not found, the Autosampler will send a message to "Control Panel" software and jump to the next vial. This error also doesn't interrupt a Schedule.

Fatal error is a kind of errors, which cannot be fixed by simple tuning. Generally, it is a damage of actuating units or their parts (malfunction of drive, sensor or connecting cable).

Table 6.5 – Error messages

Error message	**Possible cause**	**Remedy action**
No sample, vial #X	Vial #X not found	Check the vial in tray, pos. #X
No solvent		
No waste vial		
Inlet pos. calibr.	Level of inlet nut has been changed at maintenance	Tune inlet coordinates (section 5.8).
Trouble of vial	A trouble found on the way of movable parts to vial position (sample/solvent/ waste vial), or vial placed incorrectly	Remove any troubles, check that vial seats straightly in its place, check coordinates (section 5.8).
Trouble of solvent		
Trouble of waste pos.		
Trouble of inlet	A trouble found on the way of movable parts to inlet position	Remove any troubles, check coordinates (section 5.8).

Table 6.6 – Fatal error messages

Fatal error message	Possible cause	Remedy action
Syringe gear pos.	Malfunction of optoelectronic sensors related to "X" or "Y" axes	Refer to official service representative
	Malfunction of stepping motor related to "X" or "Y" axes	
	Damage of flexible cable from control board to optoelectronic sensors of tower or "X" or "Y" motor	
Plunger sensor	No gap between plunger and needle edge	Adjust plunger position, see section 5.4.4 (for Headspace option), section 5.5.5 (for Liquid option), section 5.6.5 (for SPME option).
	Syringe plunger seized or blocked	Follow cleaning procedure described in section 7.3. If cleaning doesn't help, take a new one.
	Cable is damaged	Refer to official service representative
	Plunger stepping motor malfunction	
Top sensor	Damage of optoelectronic sensors related to syringe gear	Refer to official service representative
	Damage of flexible cable from control unit to optoelectronic sensor	
	Damage of stepping motor related to syringe gear	
Placing error	Autosampler is installed on the support incorrectly	Follow installation procedure carefully. See section 5.3.1
Inlet pos. calibr.*	Level of inlet nut has been changed	Tune inlet coordinates (section 5.8).
Touch sensor	Incorrect mounting of the inlet adapter 6.119.009. Error of Touch sensor	Follow section 5.5.1 to re-install the needle adapter. Refer to official service representative

* – A *Fatal* error "Inlet pos. calibr." displays if the simple Error "Inlet pos. calibr." has been displayed 3 times followed by 3 failed attempts to re-tune inlet coordinate. A Schedule cannot be continued in this case.

7 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 2.

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

7.1 Types of maintenance

Two types of maintenance are foreseen for Autosampler:

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

Frequency of maintenance procedures depends on the following criteria:

- Intensity of Autosampler operation,
- Operating mode of the chromatograph and the autosampler,
- Samples character.

Maintenance procedures also shall be provided to fix some autosampler malfunctions.

7.2 Current maintenance

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

For all options:

- Check that Autosampler moving elements are not damaged or bent, properly working and driving smoothly according to planned algorithm.
- Check inlet septum of the GC free of leaks. Whenever new septum placed, check that needle of autosampler syringe penetrates through the septum smoothly. See sections 5.2 and 7.2.3 for details.
- Keep syringe internal channels and needle clean. See section 7.2.2.
- 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 liquid option only:

- Every morning before autosampler turning on, check that plunger moves in syringe barrel easily and smoothly. See section 7.2.1 for details.

- 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 Headspace and SMPE options:

- Visually check gas and connections for leaks. If any leak appears, retighten fittings and replace sealing elements if required.

For SPME option

- Check fiber condition and properties. Depending on samples character the fibers should be replaced in 50 – 100 cycles of sorption / desorption. Refer to section 5.6.4 for details.

7.2.1 Check for lightness of plunger movement

This check shall be carried out for liquid option only.

Observation is to be conducted every day before autosampler turning on. Autosampler should be turned off when checking. Move the plunger by carriage upward and downward to the stop. There should be no perceptible excessive force when plunger moving.

If any excessive effort appears, follow syringe cleaning procedure.

7.2.2 Check for syringe channels cleanness

Follow this procedure if you have poor sensitivity, no peaks on chromatogram or carryover. Possible contaminations (rubber particles, liquid sample drops) in the needle may cause carryover or even clog it.

Visual check for cleanness of internal washing cannels, syringe barrel and needle. Take out syringe from the autosampler. Manually aspirate air into syringe, then dip needle tip into clean water and flush air observing that bubbles go out from the needle. If bubble elimination is poor or no bubbles observed, check the needle hole carefully. Wipe needle tip with pileless cloth as soon as procedure completed.

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

7.2.3 Septum replacement and Inlet leak check

Refer to section 5.2 for septum replacement and inlet leak check.

Check inlet for leaks whenever you inject sample into inlet. Tighten inlet nut slightly to eliminate leak if observed.

Do not apply excessive force when tightening nut, it may lead to damage of inlet thread! Once you reached the stop at nut tightening, follow with replacing septum.

7.2.4 Check for proper fixing syringe unit

Syringe unit should be fixed in the thermostat (or needle adapter) and plunger should be reliably tightened in the carriage or plunger adapter. Check that nut 2, screw 3 and lock-nut 6 are tightened properly (Figure 5.8).

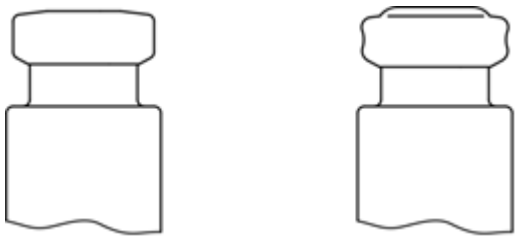
7.2.5 Sample vials preparation

Fill vial with sample up to half of its capacity or less. Before usage vials should be carefully washed and dried.

When tightening vial caps, the crimper should be adjusted so that following conditions were observed:

– crimped cap seal vial with sample tightly (no leaks acceptable),

– top surface of the cap should be flat (no protuberance or dents acceptable).



Correct – Incorrect

Figure 7.1 – Caps crimping

7.3 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).

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

Syringe cleaning

Syringe cleaning agents will usually depend on the contaminating material. Methanol, methylene chloride, acetonitrile and acetone are commonly used solvents for cleaning.

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.

Depending on the syringe type refer to original syringe manufacturer instructions for cleaning.

To carry out cleaning remove syringe from the Autosampler.

Cleaning gastight syringes (for Headspace option syringes)

- Rinse the syringe barrel and plunger with an appropriate solvent.
- Flush a poor solvent through a needle to clean it.
- Clean externally by wiping with a tissue.
- After syringe has been cleaned dry it in ambient air or autoclave.

Autoclave: Remove plunger from the barrel prior to autoclaving. Set autoclave to a maximum temperature of 70 °C for fixed needle. Expose the syringe barrel and plunger heating for at least 30 minutes. Allow syringes to come to room temperature before inserting the plunger back into the barrel.

- After cleaning, install the syringe into the Autosampler and perform a blank analysis to check that no solvent peaks response interfering on chromatogram.

Cleaning plunger and barrel channel of 5 & 10 µl syringe

Proceed with following to avoid plunger sticking in glass body:

- 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.

8 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.

9 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 7 are not carried out timely and properly.

10 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 3.2;
- 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:

R&D department	+7(8362) 68-59-35
Marketing department	+7(8362) 68-59-18
Service department	+7(8362) 68-59-19, 68-59-32
Sales department	+7(8362) 68-59-69, 68-59-70