

THERMAL DESORBER TDS-1

MODEL CHROMATEC TDA

USER MANUAL

214.2.393.004-03UM

REVISION B

PRODUCT P/N 400-1201

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Table of contents

Important notes 8

1 Safety provisions 9

2 Description 10

2.1 Introduction 10

2.2 Technical specifications 11

2.3 Delivery set 11

2.4 Thermal desorber overview 12

2.5 Placing sample tubes in the tray 15

2.6 Flow diagram 16

2.7 High temperature operation 18

3 Operation modes 19

3.1 Analysis 19

3.1.1 Pre run 19
3.1.2 Ready 19
3.1.3 Leak test 19
3.1.4 Dry purge 20
3.1.5 Desorption 20
3.1.6 Analysis 20

3.2 Tube conditioning 20

3.3 Trap clean 20

3.4 Trap test 21

4 Installation 22

4.1 Preliminary requirements 22

4.1.1 Operating conditions 22
4.1.2 Workplace requirements 22
4.1.3 Electrical requirements 23
4.1.4 Gas supply requirements 23
4.1.5 Computer requirements 24
4.1.6 GC requirements 24

4.2 Unpacking the thermal desorber 25

4.3 Thermal desorber placement 25

4.4 Manipulator installation 26

4.5 Gas connections 28
4.6 Sample disposal arrangement 29
4.7 Electrical connections 29
4.8 Ethernet Connection 29
4.9 Connection to the GC 30
4.10 Trap installation 31
4.11 GC preparation 31
4.12 Connecting transfer line to the GC 32
4.13 Software configuration 32
4.13.1 Software Installation 32
4.13.2 Setting the computer's network address 37
4.13.3 Configuration setting 41
4.14 Manipulator setting 43
4.14.1 Tray coordinates setting 43
4.14.2 Decap position coordinates setting 44
4.14.3 Decapper setting 45
4.14.4 Tube oven position setting 46
4.14.5 Heater mechanism setting 47
4.14.6 Adjusting the tube sealing mechanism 48
5 Operation 50
5.1 Turning on 50
5.2 Using display 50
5.3 Viewing and setting of system parameters 51
5.3.1 System parameters setting 51
5.3.2 Time and date setting 51
5.3.3 Firmware version view 52
5.3.4 Ethernet connection setting 52
5.3.5 Eco Settings 53
5.4 Creating a method 54
5.4.1 Analysis mode 56
5.4.2 Conditioning mode 58
5.4.3 Trap clean mode 59
5.4.4 Trap test mode 59
5.5 Method editing 60
5.6 Method deleting 60

5.7 Working with sequence 61

5.7.1 Creating a sequence 62
5.7.2 Starting a sequence 63
5.7.3 Stopping a sequence 63
5.7.4 Pause of sequence 63

5.8 TDA Control via Control Panel software 64

5.8.1 Status 64
5.8.2 Sequence 64
5.8.3 Configuration 65
5.8.4 Sequence operation 71

5.9 Checkout 74

5.9.1 Sorbent tube conditioning 74
5.9.2 Trap cleaning 74
5.9.3 Thermal desorber checkout 75

5.10 Turning off 77

6 Method development 78

6.1 Thermal desorption basics 78

6.2 Thermal desorption applications 79

6.3 Method development objectives 79

6.4 Sorbent selection 79

6.5 Packing sorbent tubes 81

6.6 Conditioning sorbent tubes 83

6.7 Packing traps 84

6.8 Trap cleaning 85

6.9 Trap test 86

6.10 Sampling 86

6.11 Analysis of solid samples 91

6.12 Sample tube storage 92

6.13 Thermal desorber operation parameters 93

6.13.1 Desorption temperature 93
6.13.2 Purge gas flow rate 94
6.13.3 Desorption time 94
6.13.4 Trap low temperature 95
6.13.5 Trap high temperature 95
6.13.6 Heating rate 95

6.13.7 Trap heating time 95
6.13.8 Dry purge 95
6.13.9 Valve temperature 96
6.13.10 Transfer line temperature 96
6.13.11 Carrier gas flow rate 96
6.13.12 Split 96
6.13.13 Re-collection 96
6.13.14 Leak test 97

6.14 Calibration 97

6.14.1 Calibration with standard gas mixtures 97
6.14.2 Calibration with liquid mixtures 98

6.15 General recommendations for analysis performance 100

7 Maintenance 102

7.1 Maintenance types 102

7.2 Routine maintenance 102

7.2.1 Cleaning of thermal desorber surfaces 102
7.2.2 Decapper cleaning 103
7.2.3 Checking cleanness of transfer line needle hole 103

7.3 Periodic maintenance 103

7.3.1 Inlet septum replacement 104
7.3.2 Leak testing 104
7.3.3 Packing or replacement of sorbent tubes 105
7.3.4 Replacing of sealing O-rings 105
7.3.5 Replacing a vent trap 108
7.3.6 Replacing a trap and sealing O-rings of the trap 109
7.3.7 Replacing transfer line needle 112
7.3.8 Replacing fuses 113

8 Troubleshooting 115

8.1 Poor repeatability 115

8.2 Broadening or tailing peaks 116

8.3 Right-angled triangle shaped peaks 117

8.4 Splitting peaks 117

8.5 Ghost peaks 117

8.6 No peaks on chromatogram 119

8.7 Hardware troubles 119

8.8 Error messages 122

9 Transportation and storage 124
10 Manufacturer warranty 124
11 Claims 125
12 Annex 126

Important notes

This User manual (hereinafter – the manual) contains the overview of the thermal desorber TDS-1, model Chromatec TDA (hereinafter – the thermal desorber, or TDA) and the instructions for its correct operation.

Prior to the commissioning of the thermal desorber, thoroughly read the manual and the operational documents on the chromatograph (the GC) to be used together with the thermal desorber.

The instructions to be observed when operating and maintaining the thermal desorber are given in the respective sections of the manual.

Notes marked with this symbol should be followed to prevent injury when operating the thermal desorber.



Follow the instruction marked with such symbol to prevent burns when touching the heated surface.



Follow the instruction marked with this symbol to prevent the explosion hazard.

1 Safety provisions

The thermal desorber can be used by person familiar with the present manual and having adequate skills and knowledge to operate this device.

The thermal desorber should be grounded. For grounding, use power plug and grounding terminal pro-vided on the rear side of sampler. For thermal desorber 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 thermal desorber with removed covers**!**

Do not touch the moving parts of the thermal desorber!

Avoid getting sleeves, clothing items and other objects in the moving parts of the device!



Thermal desorber contains heated zones (sample tube thermostat, rotary valve, trap), which may have a high temperature. Allow these units to cool down to room temperature before maintenance.

Before technical maintenance disconnect the power cord from power mains and close 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 working with gases, which are usually contained in cylinders under pressure up to 15 MPa (150 bar), you should follow the "Rules for the Construction and Safe Operation of Pressure Vessels" PB 03-576-03.

When the thermal desorber will not be used for a long time, disconnect it from power supply and close gas line.

2 Description

2.1 Introduction

The Thermal desorber is intended for thermal desorption of volatile compounds that are pre-concentrated in sample tubes packed with sorbents. With additional concentration of sample in cold trap, you can focus it before sample goes into chromatographic column.

The Thermal desorber has a tray, in which sample tubes are installed. The manipulator provides automatic transferring tubes from the tray into tube oven and reverse handling.

The Thermal desorber can be supplied with an additional Re-collection option. This option allows dividing the flow during the desorption of the sample from the trap.

TDA operation 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:

- Ambient temperature from 15 to 32 °C;
- Relative humidity not more than 80 % (non-condensing);
- Atmospheric pressure from 84 to 107 kPa (from 630 to 800 mm Hg);
- Free of dust, smoke, organic vapors, corrosive fumes indoor air.

The Thermal desorber is designed to operate under following power supply conditions: AC 220±10% V, 50/60 ±1 Hz.

The Thermal desorber meets safety requirements IEC 61010-1-2014, IEC 61010-2-010-2013, IEC 61010-2-081-2013.

There 2 terms referred to the tubes in this document: *Sample tube* and *Sorbent tube*. In many ways these are considered as synonyms but have some difference in the meaning.

Sorbent tube is a tube packed with sorbent through which analyzed air is aspirated and volatile organic compounds are concentrated in the sorbent.

Sample tube is a tube packed with a sorbent (named as Sorbent tube for this case), or packed with a piece sample (polymers, soil, other samples). So the term *Sample tube* brings wider meaning than the term *Sorbent tube*.

2.2 Technical specifications

Table 2.1 - Technical characteristics of a thermal desorber

Parameter	**Value**
Sample tube size (O.D. × length), inch (mm)	1/4 ×3.5 (6.35 × 89)
Sample tube tray capacity	50
Tube oven temperature, °C	from (t_{amb}*+10) to 425
Cold trap temperature, °C	from minus 20 to 425
Trap heating rate	500; 1000; 1500; 2000; 2500; 3000
Valve temperature, °C	150 to 350
Transfer line temperature, °C	40 to 350
Carrier gas flow rate, mL/min	0 to 200
Purge gas flow rate, mL/min	0 to 200
Relative standard deviation (RSD) of output signal (areas and retention times) when using thermal desorber, %, max **	2.0
Dimensions (width, depth, height), mm, max	345, 550, 630
Weight, kg, max.	30
Power consumption, W, max.	700

* t_{amb} – ambient temperature

** RSD test is carried out following Section 5.10

2.3 Delivery set

The thermal desorber contains the items and documents listed in the Passport.

Sample tubes, traps, caps and other materials that may be needed when performing analyzes are not included in the delivery kit of the thermal desorber. These materials are delivered by separate order and their set depends on the analytical application being solved.

2.4 Thermal desorber overview

The thermal desorber may contain parts slightly modified due to recent developments or may be subject to slight reworks having no impact on specifications and safety requirements and not mentioned herein.

The key components of the thermal desorber:

- Robotic arm;
- Tray;
- Sample tube oven;
- Trap;
- Rotary valve;
- Transfer line;
- Digital flow regulators;
- Solenoid valves;
- Power source;
- Controller;
- Display.

The display is located on the front panel.

The thermal desorber is equipped with the tray and the robotic arm to move the sample tubes.

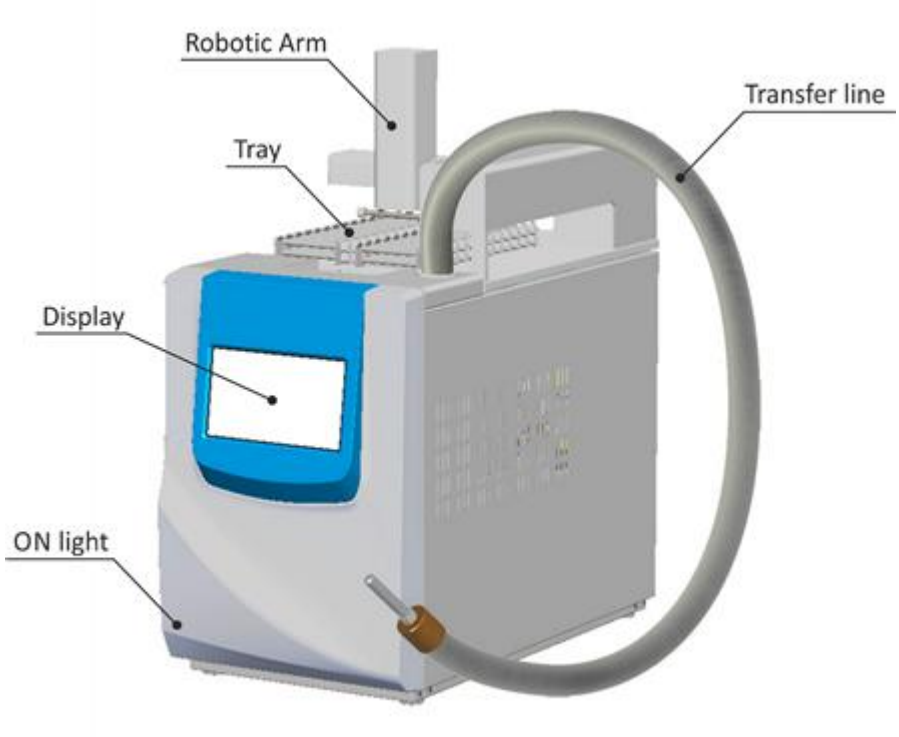


Figure 2.1 – Thermal desorber. Overall view

The thermal desorber also has the following mechanical units located under the top cover (Figure 2.2):

Decapper;

Shifter;

Sealer;

Tube oven.

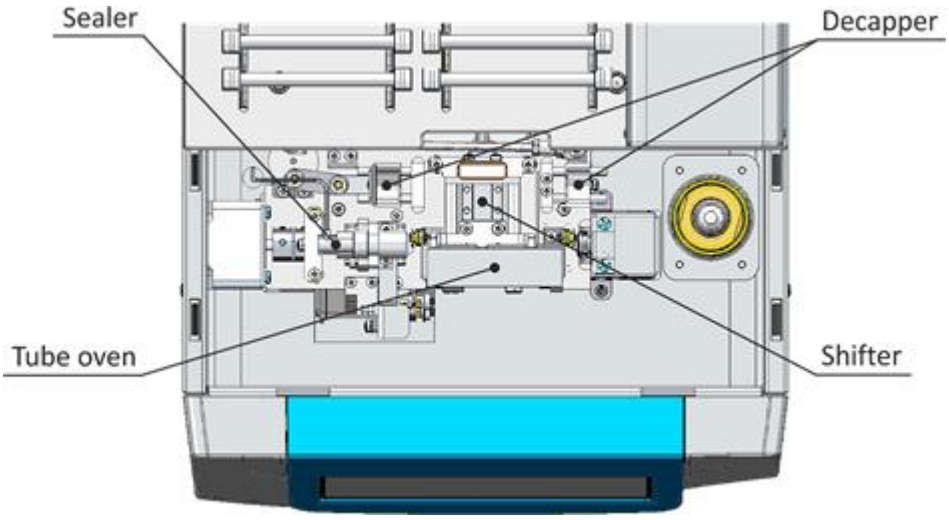


Figure 2.2 – Mechanical units

The following connections are provided in the back of the thermal desorber: mains plug, mains switch, RS232 interface to connect with a chromatograph, Start plug, Ethernet plug, and gas fittings.

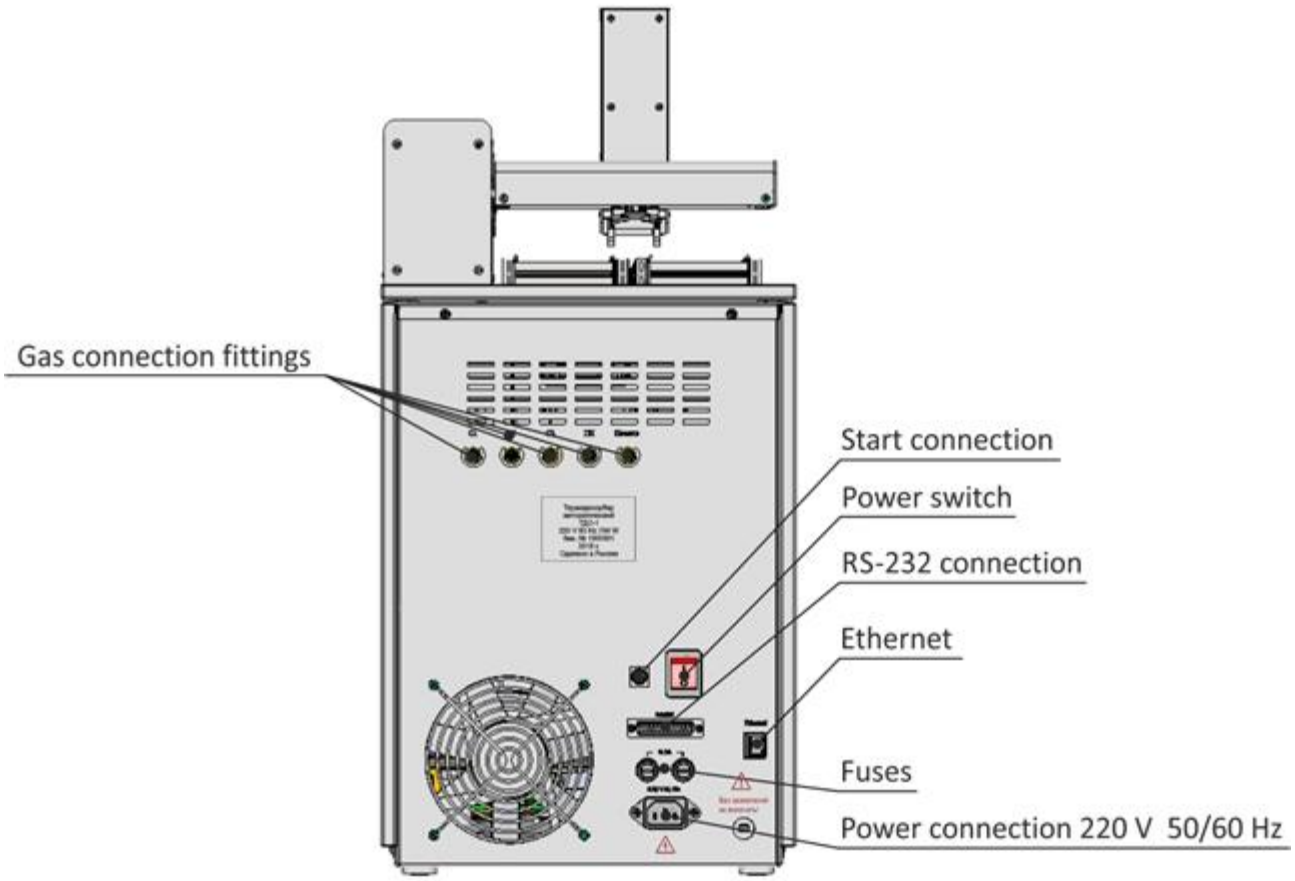


Figure 2.3 – Thermal desorber. Rear view

Tray is used to hold sample tubes. The capacity of the tray is 50 tubes. The placement of sample tubes in the tray is described in Section 2.5.

Robotic arm moves sample tubes from the tray to the decapper.

Decapper is used to remove caps from the sample tube before installing it into the oven.

Shifter moves the sample tube from the decapper into the sealer.

Sealer installs the sample tube in the purge gas line.

Sample tube oven is used to desorb analytes from the sample tube. The oven comprises a heated housing into which the sample tube is inserted.

The oven temperature is set in range (t_{amb} +10) to 425 °C and held constant during the specified time.

Trap is used to concentrate analytes. The trap is a glass tube containing a sorbent. During desorption of the components coming from the sample tube, the trap is cooled with Peltier elements to the temperature below ambient. During sample injection, the trap is heated quickly to transfer the analyzed components to the column.

During sample concentration, the trap temperature is set in range -20 to 35 °C; during insertion of the sample into the column the trap temperature is set in range 35 to 425 °C.

Rotary valve. The two-position 6-port rotary valve with a temperature control is used to switch the carrier and the purge gases.

The rotary valve temperature is set in range 150 to 350 °C and held constant during all steps of the thermal desorber operation.

The rotary valve is switched by the electric actuator. The rotary valve position (Desorption / Analysis) is sensor controlled.

Transfer line transfers the sample from the trap into the column. To prevent condensation of the analyzed components, the temperature of the transfer line is held constant in range 40 to 350 °C. At the end of the line a needle is located to punch the inlet septa.

Carrier gas flow controller controls carrier gas flow in the transfer line. The carrier gas flow is set in range 0 to 200 mL/min and held constant during all steps of the thermal desorber operation.

Purge gas flow controller controls the flow of the purge gas moving through the sorbent tube and the trap. The purge gas flow is set in range 0 to 200 mL/min automatically at the following stages: purge, desorption, sorbent tube conditioning, and trap cleaning.

Solenoid valves switch gas flows. The valves are normally closed.

Power supply generates the required supply voltages for the thermal desorber components.

Main board stores operation algorithms and methods parameters; to set and hold temperature of the sorbent tube thermostat, the trap, the rotary valve, transfer line, and gas flow; to control the electric actuator of the rotary valve and valves, as well as to connect the thermal desorber with the chromatograph.

Display is used to control and set the thermal desorber operation parameters.

2.5 Placing sample tubes in the tray

When working with the thermal desorber, sorbent tubes with length 3.5 in (89 mm) and O.D. ¼ in (6.35 mm) are used. Sorbent tubes made of glass or stainless steel can be used.

To use the manipulator correctly, the outer surface of sorbent tubes should be free from any contaminants. All media, such as stickers, markings, duct tape etc, should be completely removed from the surface of sorbent tubes prior to their insertion into the tray.

Before placing the sorbent tube in the tray, take it out from the container or remove its storage plugs (Section 6.12). Then, insert the caps 6.433.075 (Figure 2.) into sample tube ends.

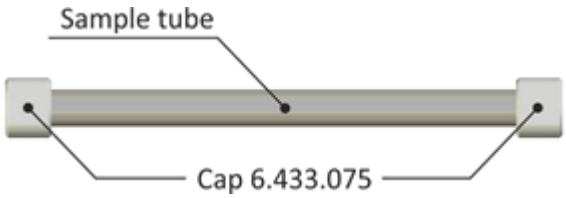


Figure 2.4 – Caps on the sample tube

Placing of the sorbent tubes in the tray should be performed prior to setting the sequence and loading the operation mode to the thermal desorber.

Do not place and do not remove sorbent tubes in/from the tray during the manipulator operation!

Each sorbent tube has a marking or an arrow with flow direction. Sorbent tubes should be placed in the tray so that arrow marking was located on the right side of the tube and the arrow pointed to the left. Sorbent tubes are installed in two rows, i.e. the lower tube has the lesser number than the upper one (Figure 2.).

The position **P** in the tray should not be occupied when placing the sorbent tubes! Instead, it is used for displacing the tubes at manipulator working.

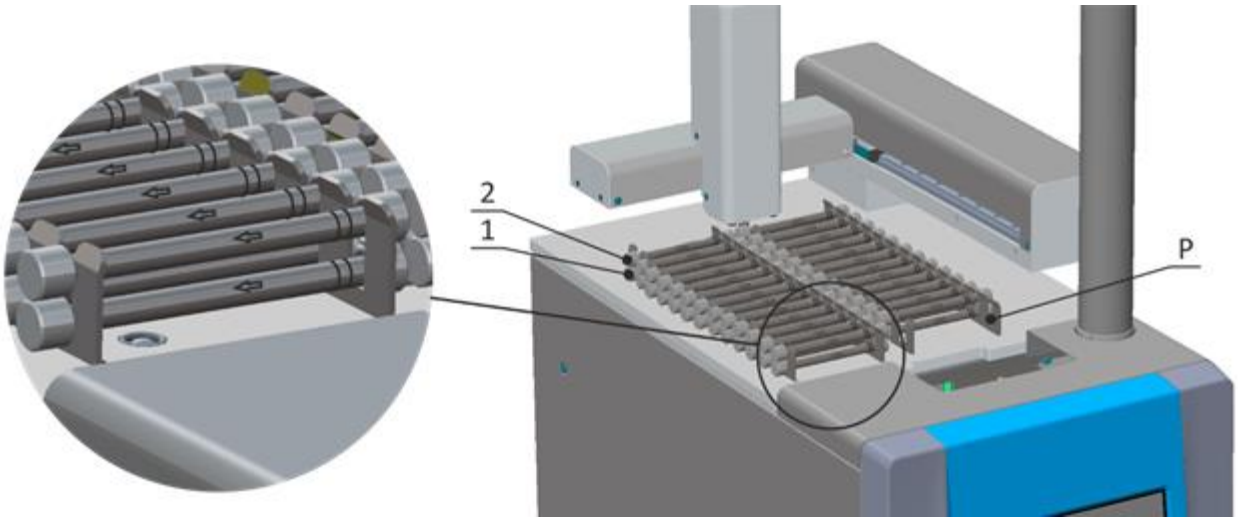


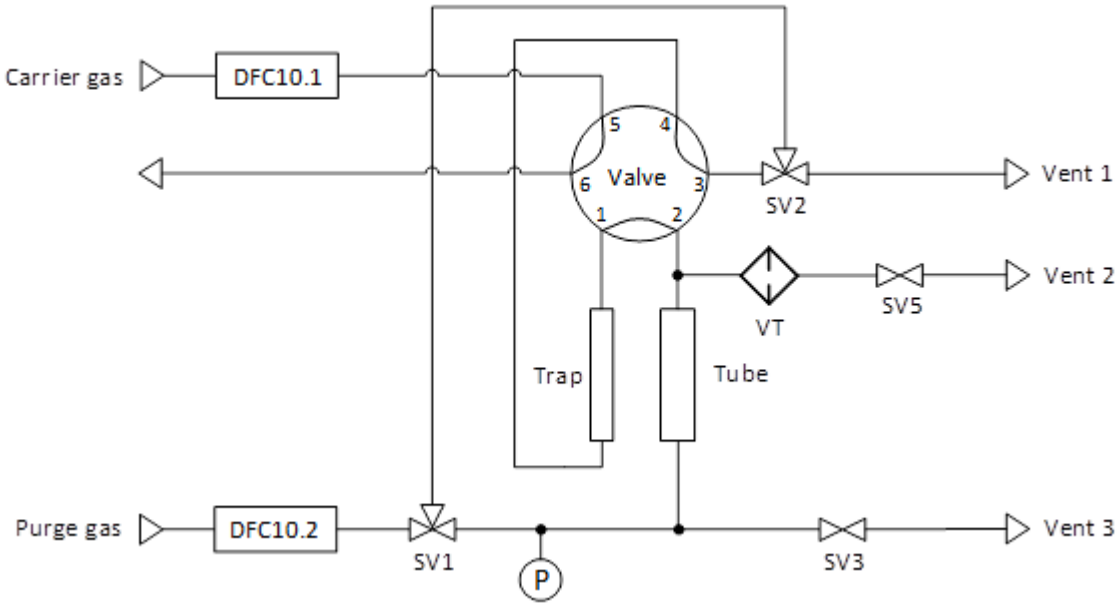
Figure 2.5 – Placing sorbent tubes in the tray



After analysis, the tubes are returned to the tray in a heated condition. Do not touch the tubes until they cool to room temperature.

2.6 Flow diagram

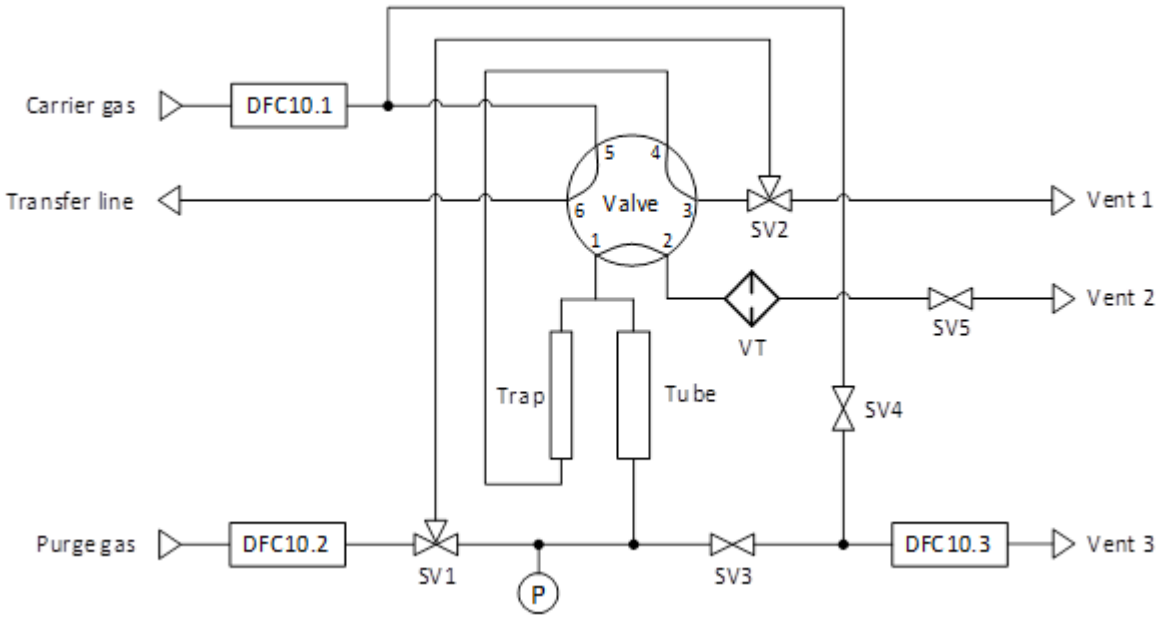
The thermal desorber gas flow diagram without Re-collection option is given below:



SV1 – SV5 – Solenoid Valves; Valve – rotary valve; Trap – Trap; DFC10.1 – Digital Flow Controller for carrier gas; DFC10.2 – Digital Flow Controller for purge gas; DFC10.3 – Digital Flow Controller for split vent; Tube – Sample tube oven; VT – Vent trap.

Figure 2.6 – Flow diagram

The thermal desorber gas flow diagram with Re-collection option is given below:



SV1 – SV5 – Solenoid Valves; Valve – rotary valve; Trap – Trap; DFC10.1 – Digital Flow Controller for carrier gas; DFC10.2 – Digital Flow Controller for purge gas; DFC10.3 – Digital Flow Controller for split vent; Tube – Sample tube oven; VT – Vent trap.

Figure 2.7 – Flow diagram

2.7 High temperature operation

Sealing of sample tubes is carried out using rubber O-rings. O-rings 8.685.176 and 8.685.177 are included in the Shipping kit. They have a maximum operating temperature of 330 °C. It should be noted that at temperatures above 300 °C, the lifetime of these O-rings is significantly reduced. Therefore, in the case of setting the tube oven temperature or the valve temperature above 300 °C, it is recommended to use high-temperature O-rings (8.685.176-01 and 8.685.177-01).

3 Operation modes

The thermal desorber allows selecting one of the following operation modes: Analysis, Conditioning, Trap clean, and Trap test.

The **Analysis** mode is used to desorb the analyzed components coming from the sorbent tube, concentrate the sample in the pre-cooled trap, and, subsequently, inject the components into the GC. In the Analysis mode, the thermal desorber runs through the steps as follows:

- Pre run,
- Ready,
- Leak test,
- Dry purge,
- Desorption,
- Analysis.

The **Conditioning** mode is used to clean sorbent tubes by heating them with the purge gas.

The **Trap clean** mode is used to remove contaminants from the trap.

The **Trap test** mode is used to prove the trap purity. In the Trap test mode, the carrier gas is moved into GC through the pre-heated trap.

3.1 Analysis

3.1.1 Pre run

During **Pre run** step, predetermined parameters of carrier gas flow rate, valve temperature, transfer line temperature and trap low temperature are established. Purge gas does not pass through the sorbent tube. The rotary valve stays in the Desorption position. The tube oven is cooled down to room temperature after heating at previous analysis run.

3.1.2 Ready

The thermal desorber enters the **Ready** step only after the tube oven is cooled to the dry purge temperature. For example, if the method is set to a dry purge temperature of 20 °C and an ambient temperature of 25 °C, then the thermal desorber will not be able to go to Ready.

At Ready step, a sorbent tube is to be placed to the oven and the analysis runs.

3.1.3 Leak test

After installation, the sorbent tube is checked for leaks by measuring pressure drop in the purge gas line. If the pressure drop is more than the set allowable value (default is 1 kPa or 0.2 psi), the error message “Leak test failed” is displayed, the current sample tube shall be withdrawn to the tray and the next tube is to be analyzed in the sequence.

3.1.4 Dry purge

Dry purge step is applied to remove air and moisture from the sorbent tube prior to desorption. The flow of purge gas is directed in such a way that air and moisture are blown to the vent, bypassing the trap.

3.1.5 Desorption

After dry purge, the purge gas flow rate and the tube oven temperature are set. The set values are hold for desorption time.

At this stage, the analytes are transferred by the purge gas from the sorbent tube into a cooled trap in which they are concentrated.

3.1.6 Analysis

The valve switches into Analysis position, the trap is heated up to high temperature and this will be maintained during a specified time interval.

At the **Analysis** step, compounds are desorbed from the trap and moved by carrier gas via transfer line into the GC column.

When working with split, the flow transferring the sample is divided at the output of the trap and part of the flow is directed through the tube to the vent.

Splitting the flow can be used to capture part of the sample on a current or a new sorbent tube for repeat analysis.

3.2 Tube conditioning

Tube conditioning is used to remove volatile contaminants from a sorbent. After conditioning, tube is ready for next sampling.

In this mode, the leak test of the sorbent tube is occurred first, then the specified purge gas flow rate and the temperature of the sorbent tube thermostat are set. The set values are maintained during the tube cleaning time.

The maximum duration of conditioning of each sorbent tube, specified in the program is 24 hours.

3.3 Trap clean

Trap clean allows to remove volatile impurities from the sorbent. This mode is recommended for a new trap being installed or the thermal desorber being in downtime for a long time.

In this mode, the purge gas flow rate and trap temperature are set. The set values are maintained during the trap cleaning time.

The maximum duration of the trap cleaning is 15 minutes.

3.4 Trap test

Trap test is used after cleaning the trap to confirm its purity. It is also recommended to conduct a trap test prior to running a sequence of analyzes to ensure that the analytical system is clean.

In the Trap Test mode, the specified flow rate of the carrier gas is set, the valve switches to the Analysis position, the target trap temperature is set and maintained for a certain time (blank analysis runs).

4 Installation

Installation and setup of the thermal desorber is usually carried out by a Chromatec service engineer or a trained and qualified person. The information provided in this section is intended for experienced users.

Tools required

- Screwdriver PH 1 (cross)
- Screwdriver SL 4 (flat-blade)
- Wrench 8 mm
- Wrench 10 mm
- Cutter 6.899.004 (for metal tubing)

4.1 Preliminary requirements

4.1.1 Operating conditions

Thermal desorber operation is carried out in closed laboratory rooms at ambient air temperature from 15 to 32 °C, relative humidity not more than 80 %, atmospheric pressure from 84 to 107 kPa (from 630 to 800 mm Hg).

Thermal desorption is a highly sensitive method of analysis. The thermal desorber shall be operated in a laboratory room with no background organic vapors in the ambient air.

4.1.2 Workplace requirements

Ensure that the table surface is stable. Since the thermal desorber uses automatically moving components, make sure that there are no vibrations generated by equipment near the location of the thermal desorber.

Table 4.1 –Dimensions and weight of the thermal desorber

Width, mm	345
Depth, mm	550
Height, mm	630
Weight, kg	30

The distance between the rear wall of the thermal desorber and the room wall should be at least 30 cm. It is recommended to have a passageway at the back of the thermal desorber for connection of gas lines and maintenance.

Ensure that the installation location allows for easy disconnection of the thermal desorber and disconnection of the power cord in case of emergency.

4.1.3 Electrical requirements

Electrical power supply of the Thermal Desorber is carried out from AC mains with voltage (187-242)V, frequency (50/60±1) Hz. Maximum power consumption of the thermal desorber is 700 W.

The thermal desorber must be grounded. Grounding is carried out by means of the mains plug and additional grounding. The "⊥" contacts of the mains sockets must be earthed (connected to the external earthing circuit) using copper wires with a cross-section of at least 1.5 mm^2.

Do not connect the Thermal Desorber to an electrical network to which a refrigerator, electric motors and other equipment capable of generating electrical interference is connected.

4.1.4 Gas supply requirements

The thermal desorber has two independent gas channels - carrier gas and purge gas. The carrier gas must be an inert gas (helium, nitrogen, hydrogen, argon) that is connected to the gas chromatograph. The purge gas may be different from the carrier gas. Note that hydrogen is not allowed as a purge gas.

The connected gases must meet the following requirements:

- Purity of at least 99.999 %, hydrocarbon content not exceeding 0.00005 %.
- Inlet pressure from 0.40 to 0.62 MPa.
- When working with gases in pressurized cylinders, use a two-stage reducer with a metal diaphragm, e.g. RDB-2-0.6 (214.5.882.003).
- Connect the gases to the thermal desorber using copper or steel tubes with an outside diameter of 3.2 mm (1/8'').
- The distance from the gas source to the thermal desorber should be kept as short as possible. Avoid using a large number of adapters to connect piping.
- If the gas source is located at a significant distance from the thermal desorber, and if multiple instruments are to be powered, use 6.35 mm (1/4'') diameter steel piping to bring the gas to the lab bench where the thermal desorber will be placed.

To avoid trapping impurities contained in the inert gas, it is recommended to install filters to trap moisture, hydrocarbons and oxygen at the inlet to the thermal desorber on the purge gas line.

4.1.5 Computer requirements

The software runs on computers running the Windows operating system. The following are the minimum computer requirements.

Table 4.2 – Minimum computer requirements

Windows	7, 8.x, 10
RAM, GB, not less than	4
Intel or AMD processor, year of release not earlier than	2012
Monitor resolution, not less than	1024 x 768
Free hard disk space, GB, not less than	100
Free Ethernet port	+
Additional equipment	Keyboard and mouse

4.1.6 GC requirements

If the thermal desorber is connected to a Chromatec-Crystal chromatograph, the firmware version must be 03.21.16.691 or higher.

The transfer line of the thermal desorber is connected to the evaporator of the gas chromatograph. Accordingly, the chromatograph must have a Split/Splitless (SSL) inlet. A nut for connecting the transfer line to the Chromatec-Crystal chromatograph is included in the shipping kit. To connect the transfer line to third-party chromatographs, nuts are required, supplied separately by order (Table 4.3).

Table 4.3 – Nuts securing the transfer line to the inlet

Chromatograph Model	**Description**	**Catalog Number**
Chromatec-Crystal 2000M, 5000, 9000	Nut	8.936.107 (Chromatec)
Agilent 6890, 7820, 7890, 8860, 8890	Nut	8.936.121 (Chromatec)
Thermo Trace 1300 and 1310	Nut	8.936.114 (Chromatec)

In order to optimize sample injection into the inlet, a liner with a narrow inner diameter is installed. The catalog numbers of the liners are given in the table below).

Table 4.4 – Recommended liners

Chromatograph Model	**Description**	**Catalog Number**
Chromatec-Crystal 2000M, 5000, 9000	Inlet liner, 1.2 mm ID	7.352.066 (Chromatec)
Agilent 6890, 7820, 7890, 8860, 8890	Inlet liner, direct, 1.5 mm ID	18740-80200 (Agilent)
Thermo Trace 1300 and 1310	Direct straight liner, 1.2 mm ID	453A1335 (Thermo)

To synchronize sample injection and start of analysis, the chromatograph must have a free external start connector. If no ports are available, use splitters or Y-cables (Table 4.5).

Table 4.5 – Splitters and external start Y-cables

Chromatograph Model	Description	Catalog Number
Chromatec-Crystal 2000M, 5000, 9000	Adapter (connecting TDA and MSD to the DETECTOR connector)	5.104.036 (Chromatec)
Agilent 6890, 7820, 7890, 8860, 8890	Y remote start cable (connecting two devices to the REMOTE connector)	G1530-61200 (Agilent)
Thermo Trace 1300 and 1310	Y shape cable (connecting two devices to the AUTOSAMPLER HANDSHAKE connector)	23043623 (Thermo)

The external start cable to the Chromatec-Crystal chromatograph is included in the shipping kit. In case of connection of the thermal desorber to third-party chromatographs, optional cables are required (Table 4.6).

Table 4.6 – External start cables

Chromatograph Model	Description	Catalog Number
Chromatec-Crystal 2000M, 5000, 9000	Cable	6.644.146 (Chromatec)
Agilent 6890, 7820, 7890, 8860, 8890	Cable	6.644.777-04 (Chromatec)
Thermo Trace 1300 and 1310	Cable	6.644.777-05 (Chromatec)

4.2 Unpacking the thermal desorber

Unpack the thermal desorber as well as the optional accessories and check the conformity with the packing list.

In case of any damage, contact the Chromatec customer service. Claims for damage during transportation must be submitted immediately to the carrier.

4.3 Thermal desorber placement

The thermal desorber shall be installed on the laboratory table to the left of the gas chromatograph provided that the transfer line does not hinder the manipulator movements. The laboratory table shall withstand the weight of the TDA, GC, and any other equipment installed on the table.



For efficient cooling of the electronic components, the minimum distance between the thermal desorber and the chromatograph should be at least 5 cm. The length of the transfer line, which is 1 m, must be taken into account.

To avoid the risk of fire, no flammable or combustible substances or materials should be placed within 30 cm of the thermal desorber.

4.4 Manipulator installation

- Place manipulator on the top part of the thermal desorber.

At proper placing, the pins on the thermal top surface coincide with the holes in the manipulator base, and the RS-232 connector in the lower part of the manipulator corresponds to the counter connector on the thermal desorber case.

- Fix the manipulator with two screws M4x14 DIN 965 (Figure 4.1).

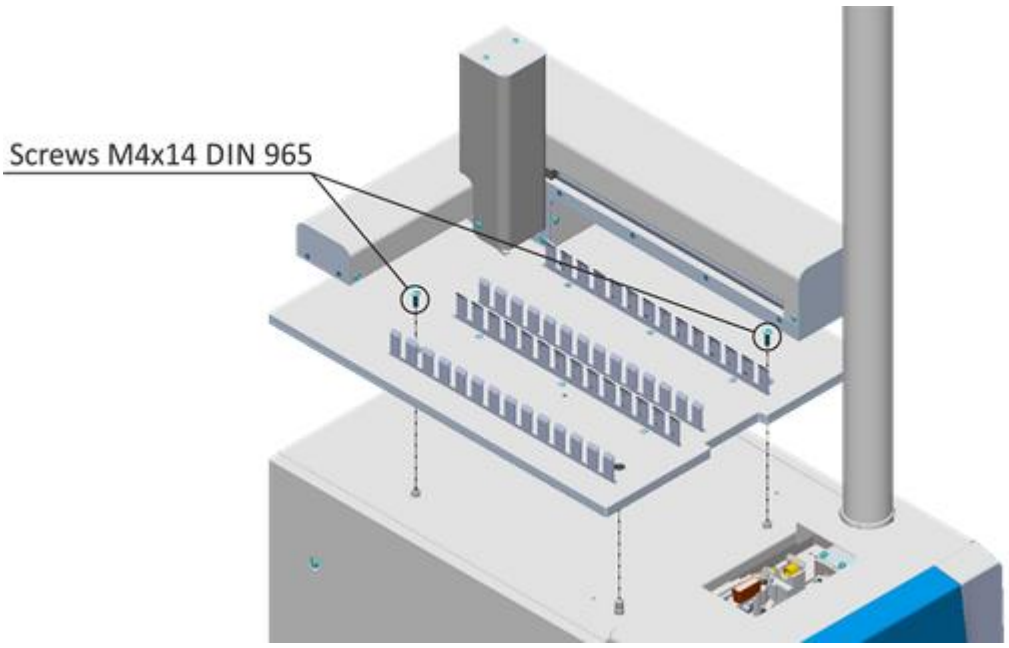


Figure 4.1 – Manipulator installation

Install clamp 8.668.406 on the manipulator and fix it with screws M3x10 DIN 912 (Figure 4.2).



Figure 4.2 – Clamp installation

4.5 Gas connections

Maximum permissible pressure of gas at the thermal desorber inlet is 0.62 MPa (90 psi).

When connecting gas lines to the thermal desorber, use clean tubes of copper or steel. To remove any contaminations from metal tubes, heat them in the thermostat and then purge with clean inert gas.

There should be no scratches, dents or cracks on the outer surface of the tubes. The ends of the connected tubes should be flat.



Do not use hydrogen as a purge gas!

When using a carrier gas in a hydrogen tank, a flame arrestor should be installed on gas line between the pressure reducer valve and the thermal desorber.

When using hydrogen as a carrier gas, hydrogenation of reactive and unsaturated compounds may occur at high temperatures.

The thermal desorber is equipped with separate fittings for connection of carrier and purge gas lines. This allows using different gases for desorption of components coming from the sorbent tube and the trap.

Connect the gas lines to the fittings of the thermal desorber located at the rear wall (Figure 4.).



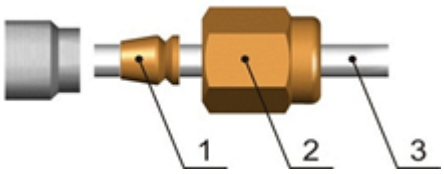
Figure 4.3 – Gas fittings

- Connect carrier gas to the fitting Carrier.

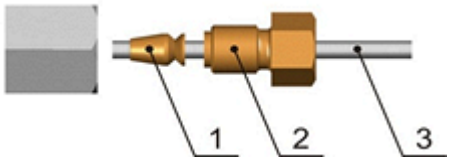
Use the same type of carrier gas as in the GC.

- Connect the purge gas to the Purge fitting, which will be used during desorption from the sample tube. The type of purge gas may differ from the carrier gas.
- Unscrew the caps from the Vent1, Vent2 and Vent3 fittings and leave them free.

Tubing connections are made using brass or stainless-steel ferrules (Figure 4.2). After tightening the nut by hand until it stops, it is tightened with a wrench at ¾ turn (270 °). When re-sealing the nut is tightened by hand until it stops and tightened with the wrench at 60 °.



1 – Ferrule 8.658.038-02 (brass) or 8.658.038-03 (stainless steel), 2 – Nut 8.930.167 (brass, M8 thread), 3 – Tube O.D. 1/8” (3.2 mm)



1 – Ferrule 8.658.039-01 (stainless steel), 2 – Nut 8.930.161 (brass, M6 thread) or 8.930.286 (stainless steel, M6 thread), 3 – Tube O.D. 1/16” (1.6 mm)

Figure 4.2 – Tubes connection

After connecting the gas lines to the thermal desorber, check them for leaks (see Section 7.3).

4.6 Sample disposal arrangement

When working with toxic and harmful substances during desorption, cleaning tubes or traps, these compounds can be released through the sample vent channels. In this case, tubes should be connected to the sample vent ports (Vent1, Vent2, Vent 3) in the rear part of the thermal desorber and conducted to an exhaust hood.

4.7 Electrical connections

The TDA is operated under following power supply conditions: 220±10% AC, 50/60±1 Hz.

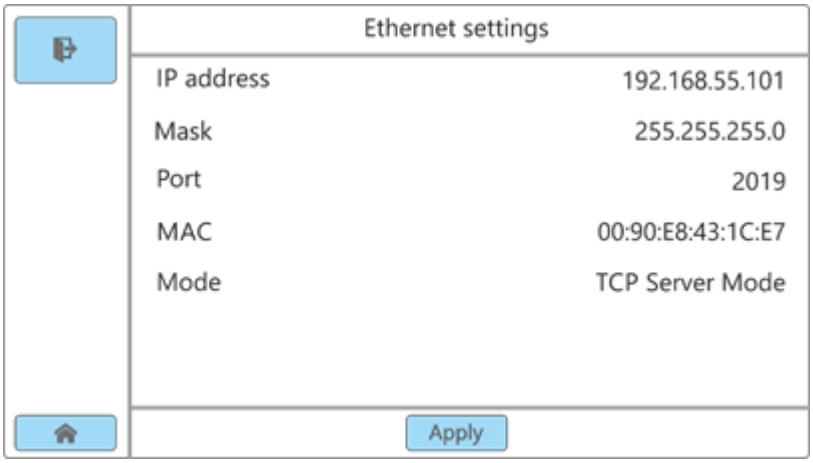
Use power cord SCZ-1 to connect the thermal desorber to power supply.

4.8 Ethernet Connection

To control of the TDA from a PC, it is necessary to connect the thermal desorber and the PC to the same local network. For connection, a patch cord UTP cat.5e cable is used.

Connect the patch cord to the Ethernet connector of the TDA.

Click icon **System** in the Main menu.



IP address – IP address. Thermal Desorber is shipped with a default IP address of 192.168.55.XXX. When connecting Thermal Desorber to a computer via a switch, the last number XXX must be unique to each computer and must not match the network addresses of the chromatograph, Thermal Desorber and other network devices. If a router is used, the IP address is assigned by the network administrator.

Mask – Subnet mask, specify 255.255.255.255.0.

Port – Port number. When connecting through a switch, specify port 2019. When using a router, the port number is assigned by the network administrator.

MAC – Network interface ID (MAC-address). It is assigned by manufacturer and cannot be changed.

Mode – To connect the thermal desorber to the local network, select **TCP Server Mode**.

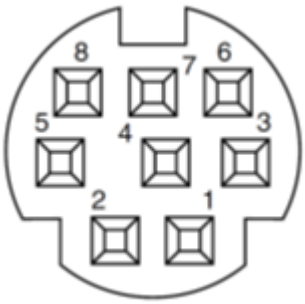
Click **Apply**.

4.9 Connection to the GC

The thermal desorber is connected to the Chromatec-Crystal GC via the START connector located on the rear panel of the instrument.

Fit the cable 6.644.146 to the connector START of the thermal desorber and to the connector SAMPLER of the GC.

Connection of the thermal desorber to the third-party chromatographs is carried out with the cable 6.644.777 (not included in the shipping kit), which at one end has a connector MDN-8M. This connector is connected to the START connector on the rear panel of the thermal desorber. The wires on the other side of the cable are connected to the corresponding wires of the START IN cable of the GC.

Pin	Wire color	Description	Scheme
1	(red)	GC Start (Output)	
2	(green)	TDA ready (Output)	
3	(black)	Common	
4	(orange)	Not used	
5	(blue)	GC Ready (Input)	
6	(brown)	Not used	
7		Not used	
8		Not used	

GC Start – signal from the TDA to the GC to start analysis (obligatory).

TDA ready – signal from TDA to the GC that thermal desorber is ready (not obligatory).

GC ready – signal from the GC to the TDA that the GC is ready (obligatory). If the GC does not send a ready signal, then contacts 3 and 5 should be closed.

4.10 Trap installation

The TDA is supplied with a Tenax TA trap installed. If it is necessary to use a trap with another sorbent, replace it according to Section 7.3.6.

4.11 GC preparation

The procedure Chromatec-Crystal GC preparation is described below. If the TDA is connected to third-party chromatographs, the accessories are different.



Cool the GC inlet to prevent burns.

- Install the liner 7.352.066 into GC inlet.
- Replace the inlet nut 6.482.013 with the nut 8.936.107.

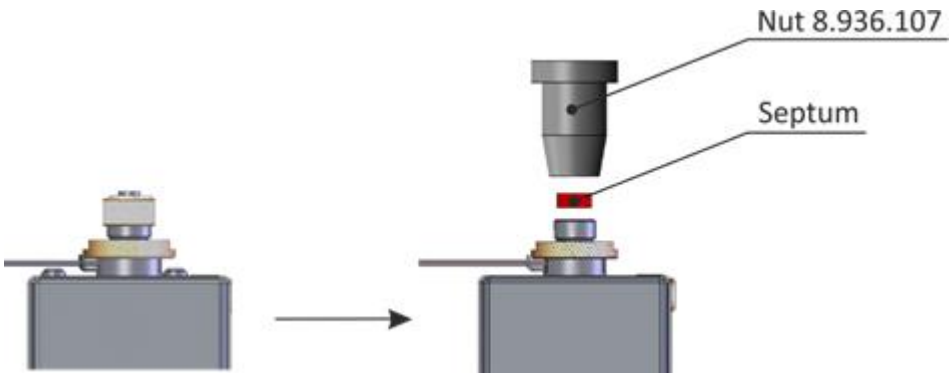


Figure 4.5 – GC inlet preparation

4.12 Connecting transfer line to the GC

Before connecting the transfer line to the GC, check that needle hole is clean (Section 7.2.3).

Connect transfer line to the GC as follows:

- Release GC inlet nut 8.936.107.
- Insert transfer line needle into the nut 8.936.107.
- Tighten the nut 8.936.107 by hand.

4.13 Software configuration

To operate of the thermal desorber from a PC, the Control Panel software is used.

The version of the Control Panel should be 2.0.1908.2000 or higher.

4.13.1 Software Installation

Connect a USB drive with "Chromatec Analytic" software to your computer (if the software is supplied on a DVD, insert the distribution disc into the appropriate drive on your computer).

In Windows Explorer select the USB drive (DVD drive) and run the **setup.exe**.

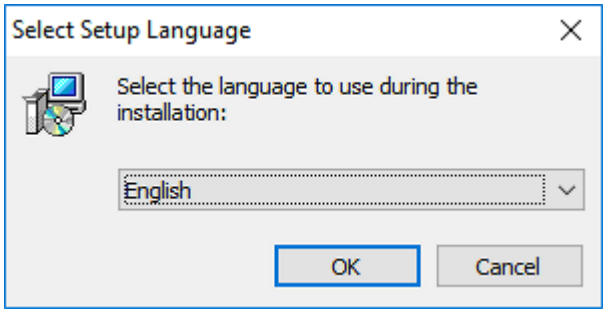
- Select the language to use during the installation. Press **OK**.
- Press Next.
- Accept the agreement.
- Press Next.
- If "Chromatec Analytic 3" has not been previously installed on this computer, you are prompted to specify the location of the data folder.
- Press **Next**.
- Select components to install

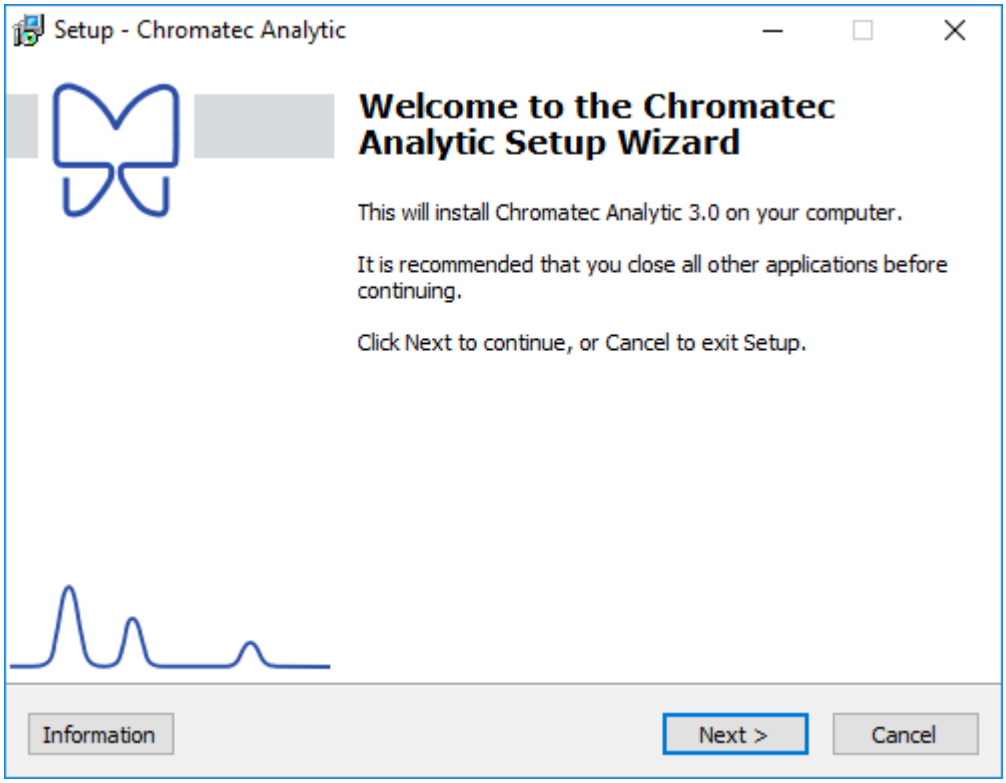
Checked and blocked components are installed always. This is a main program.

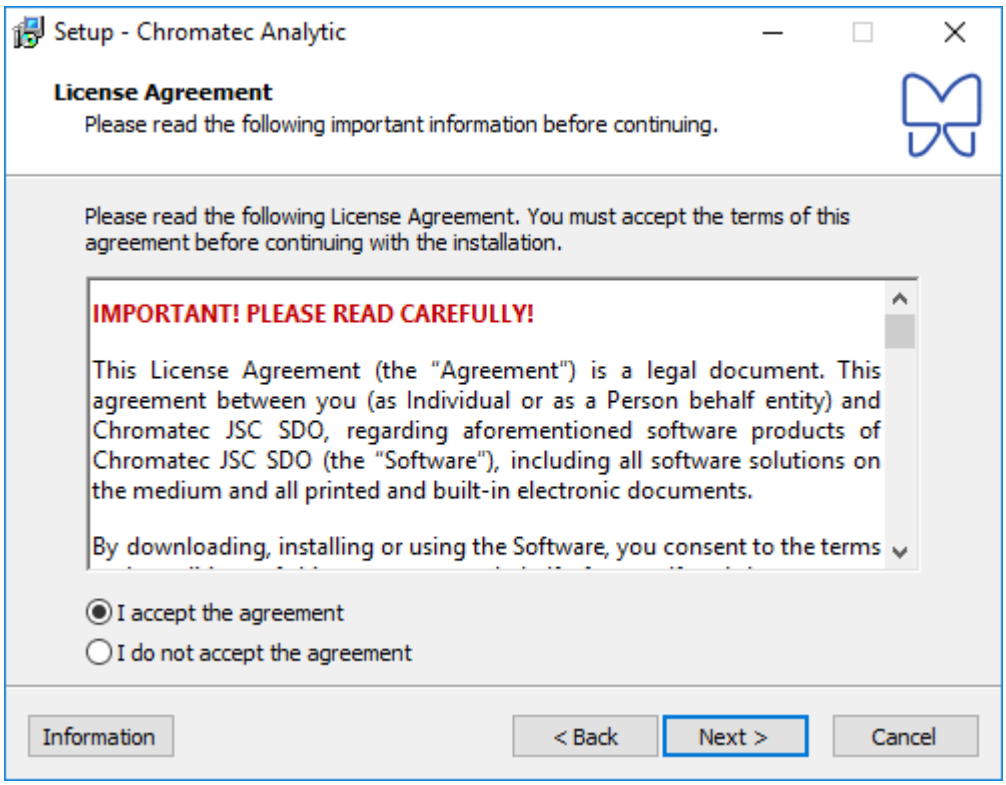
Description of the other components included in the software is given in the table below. As you can see this description if you click on the **Information** button.

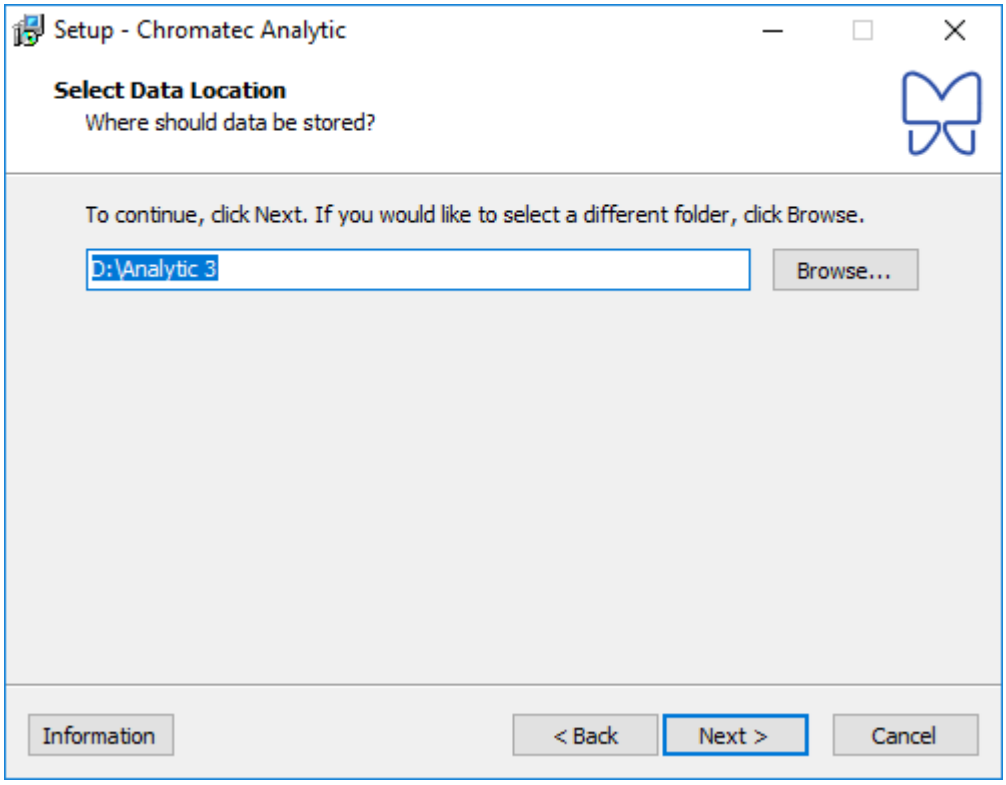
Press **Next** on the current and the next page.

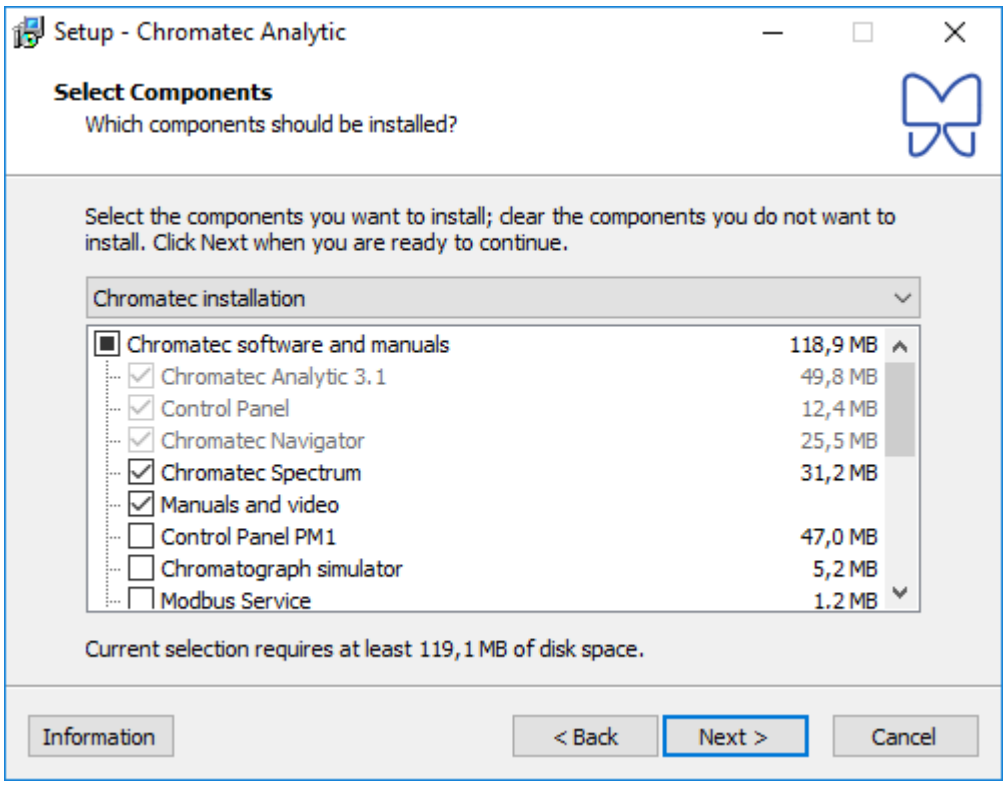
Wait for the installation.

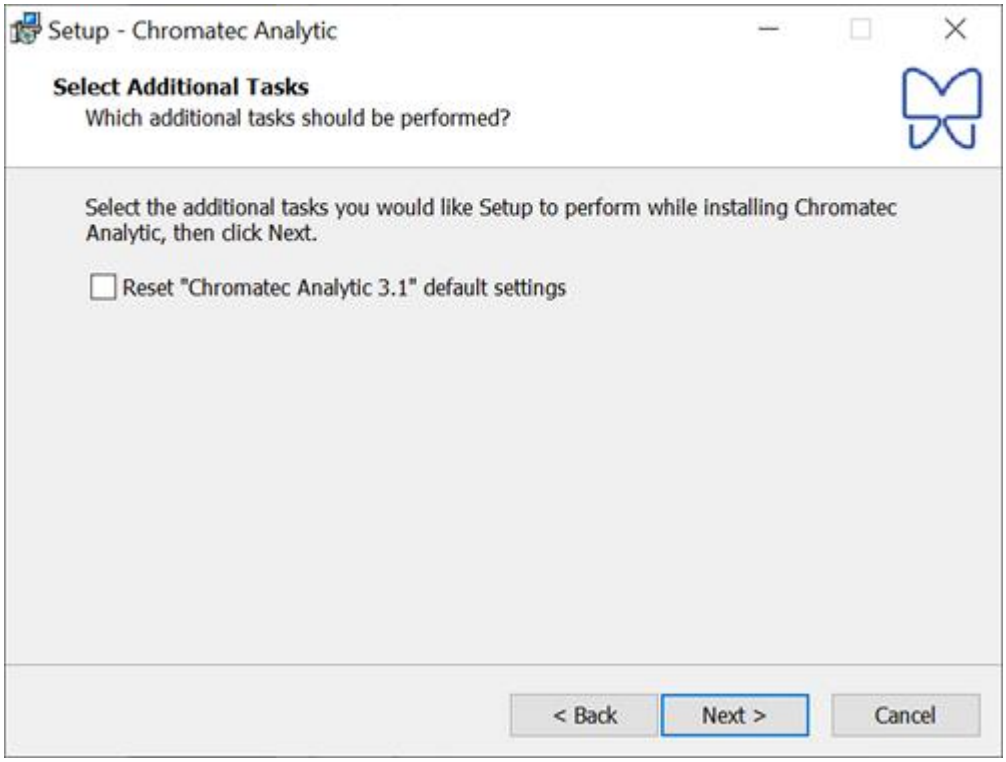




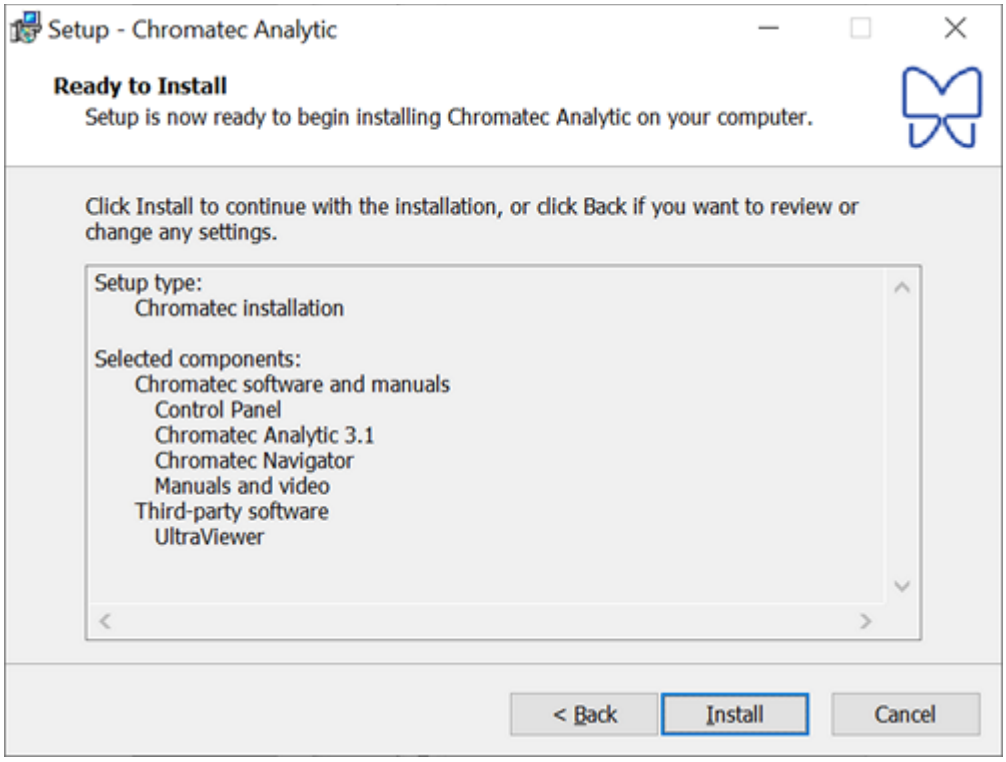

Setup - Chromatec Analytic
License Agreement
Please read the following important information before continuing.
Please read the following License Agreement. You must accept the terms of this agreement before continuing with the installation.
IMPORTANT! PLEASE READ CAREFULLY!
This License Agreement (the "Agreement") is a legal document. This agreement between you (as Individual or as a Person behalf entity) and Chromatec JSC SDO, regarding aforementioned software products of Chromatec JSC SDO (the "Software"), including all software solutions on the medium and all printed and built-in electronic documents.
By downloading, installing or using the Software, you consent to the terms
I accept the agreement
I do not accept the agreement
Information
< Back
Next >
Cancel


Setup - Chromatec Analytic
Select Data Location
Where should data be stored?
To continue, click Next. If you would like to select a different folder, click Browse.
D:\Analytic 3
Browse...
Information
< Back
Next >
Cancel

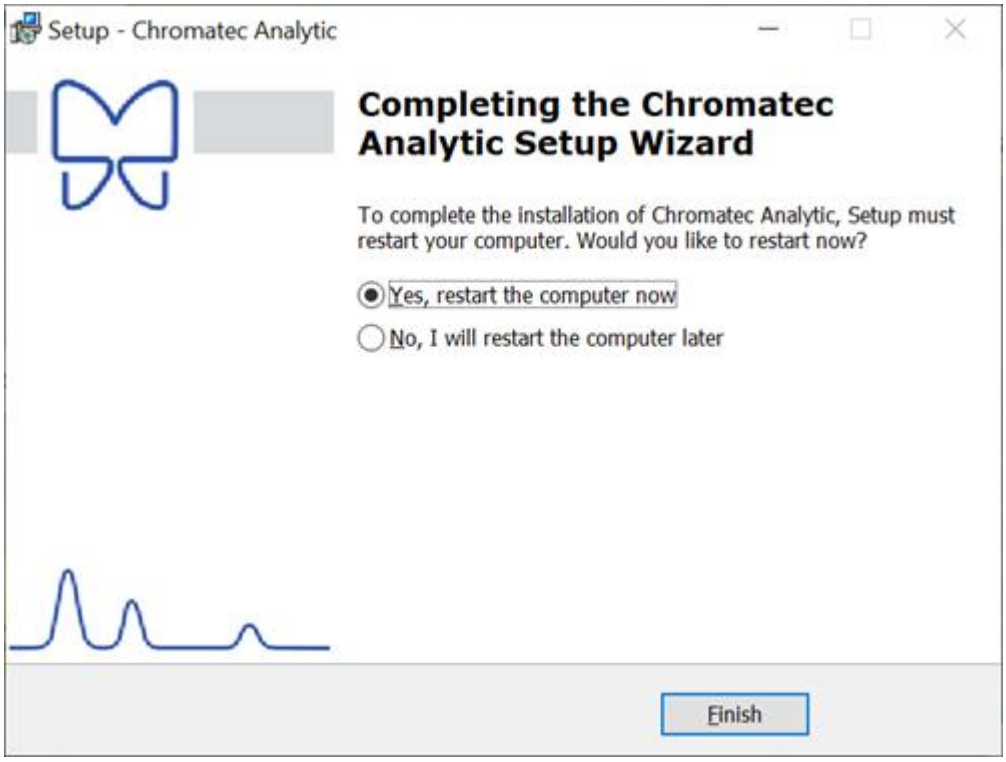

Setup - Chromatec Analytic
Select Components
Which components should be installed?
Select the components you want to install; clear the components you do not want to install. Click Next when you are ready to continue.
Chromatec installation
Chromatec software and manuals 118,9 MB
Chromatec Analytic 3.1 49,8 MB
Control Panel 12,4 MB
Chromatec Navigator 25,5 MB
Chromatec Spectrum 31,2 MB
Manuals and video
Control Panel PM1 47,0 MB
Chromatograph simulator 5,2 MB
Modbus Service 1.2 MB
Current selection requires at least 119,1 MB of disk space.
Information
< Back
Next >
Cancel


Setup - Chromatec Analytic
Select Additional Tasks
Which additional tasks should be performed?
Select the additional tasks you would like Setup to perform while installing Chromatec Analytic, then click Next.
Reset "Chromatec Analytic 3.1" default settings
< Back
Next >
Cancel

Click Install to continue.



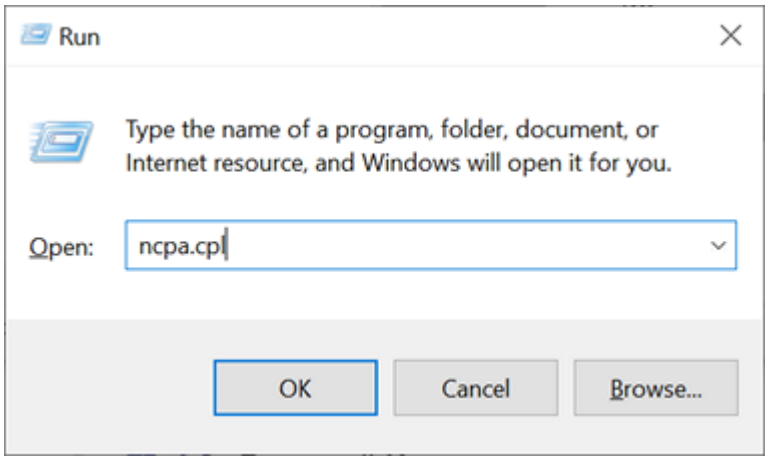
Wait for the installation to complete and click Finish.



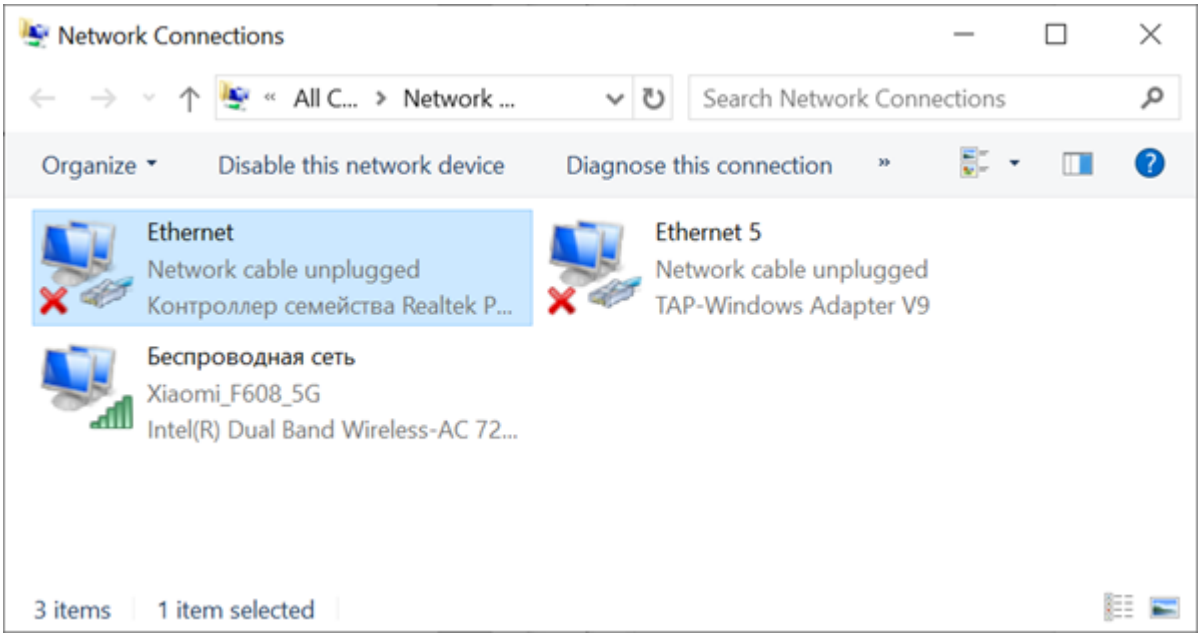
4.13.2 Setting the computer's network address

On your keyboard, press the Win + R key combination.

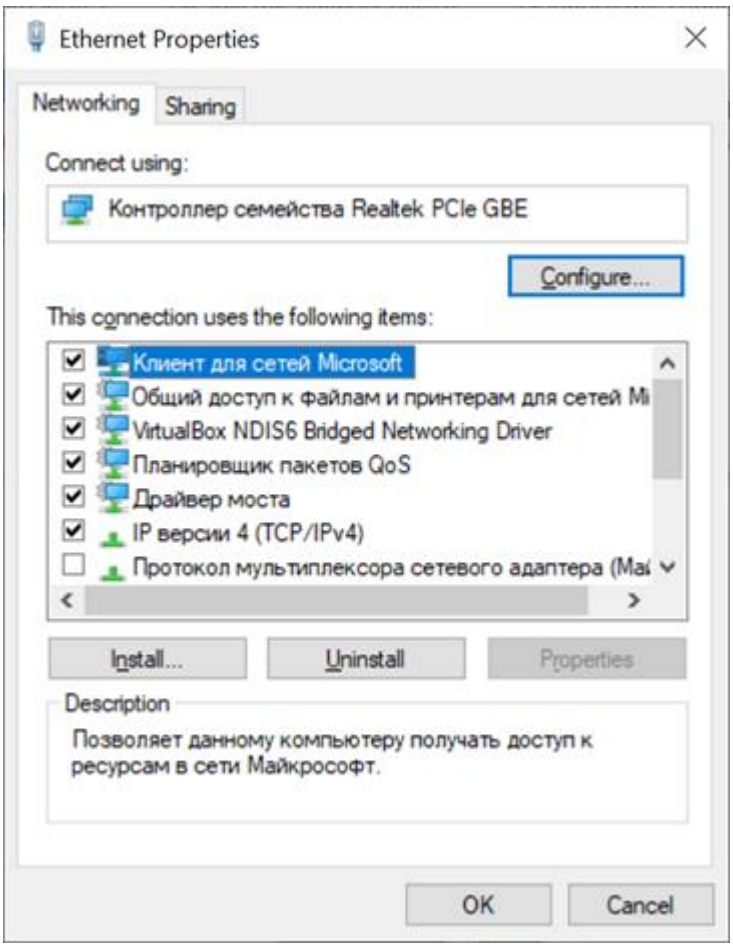
In the window that opens, enter the command ncpa.cpl and click OK.



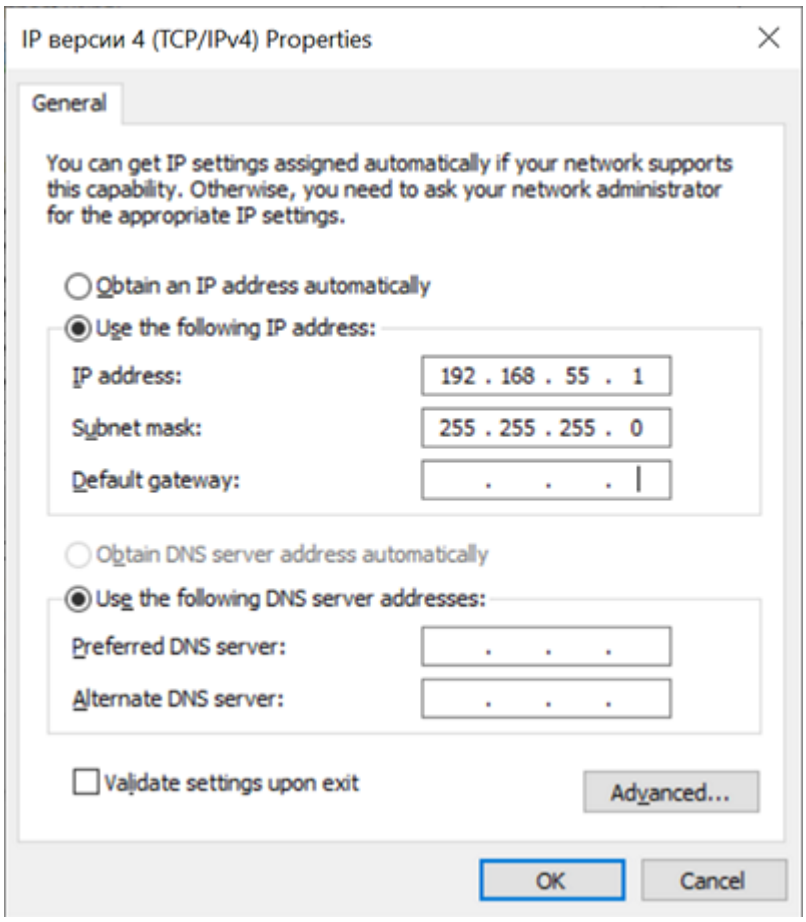
In the Network Connections window, right-click on an available connection. From the menu, select Properties.



Select Internet Protocol version 4 (TCP/IPv4) and click Properties.



Enter the IP address and subnet mask. Click OK.



When connecting the thermal desorber to a computer via a switch, the IP address of the computer must be 192.168.55.XXX. The last number XXX must be unique to each computer and must not match the network addresses of the chromatograph, thermal desorber, or other network devices.

If a router is used, the IP address, subnet mask, and default gateway are assigned by the network administator.

Connection setup

Open the **Control Panel**.

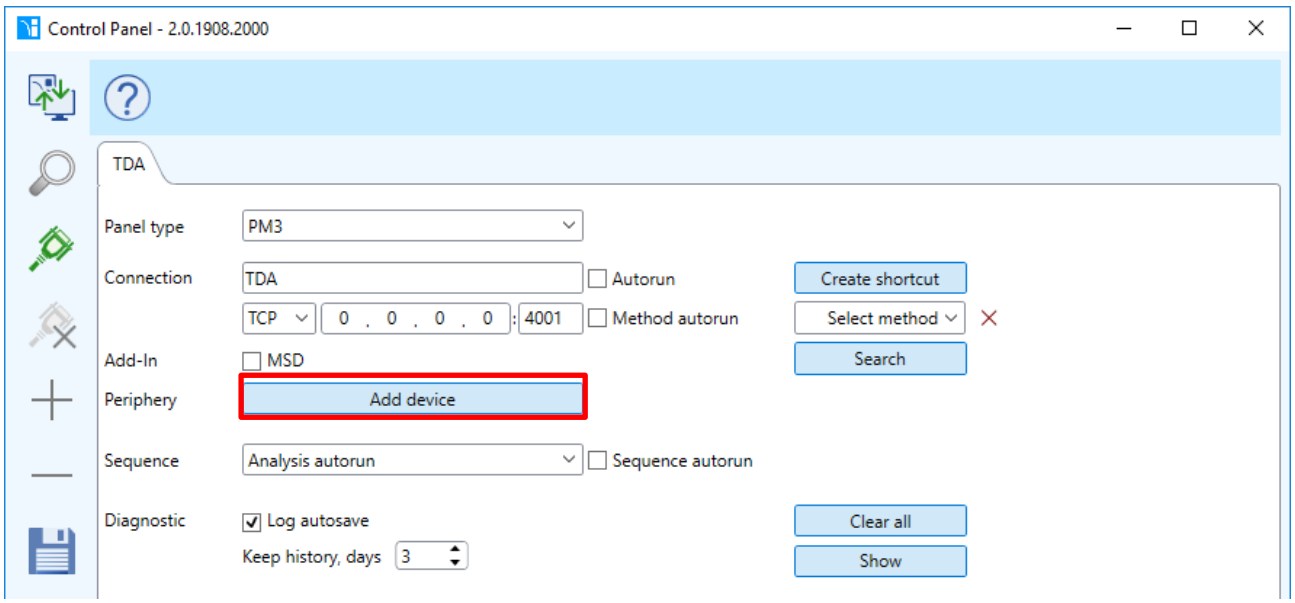
Open the **Connections** window.

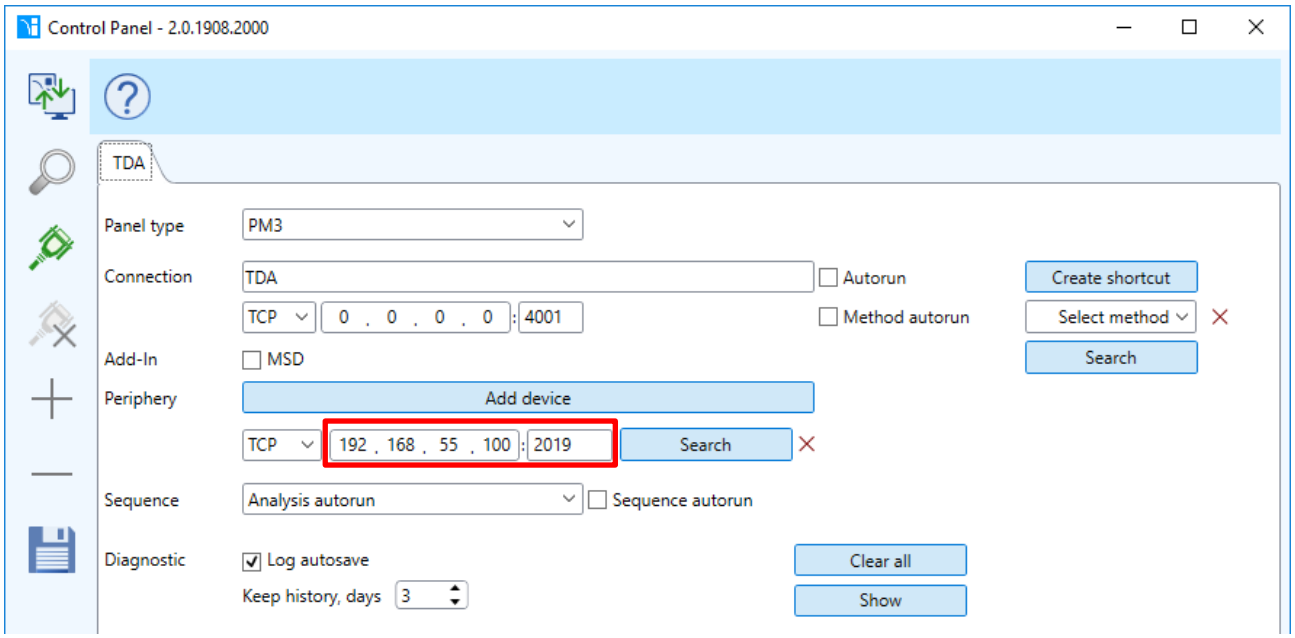
Click **Add connection**.

In the Periphery line, click Add device.

Enter the IP address. If the IP address is unknown, click Search and select the TDA from the list. In addition, the IP address can be found or change in the TDA display menu System / Ethernet.

Enter the port number. The default port number assigned to the thermal desorber is 2019.

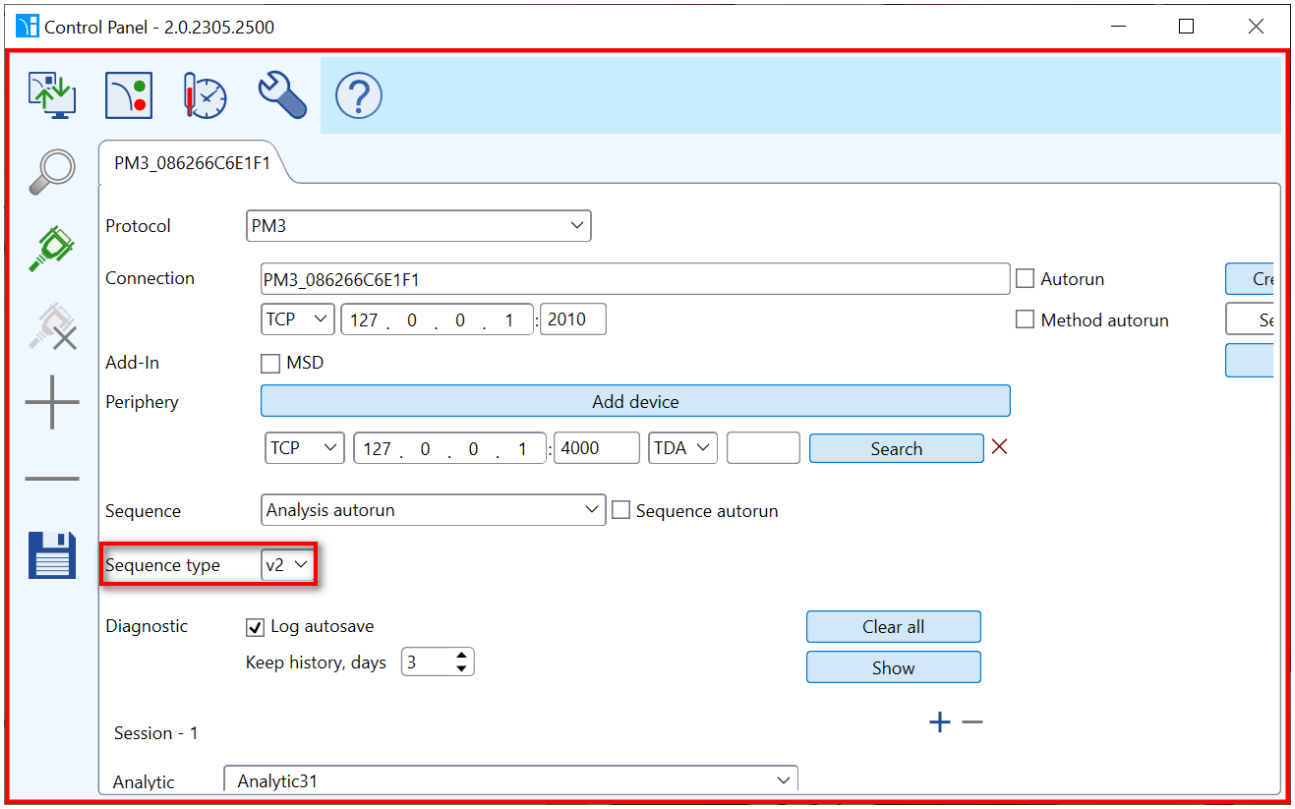




Click Connect to instrument.

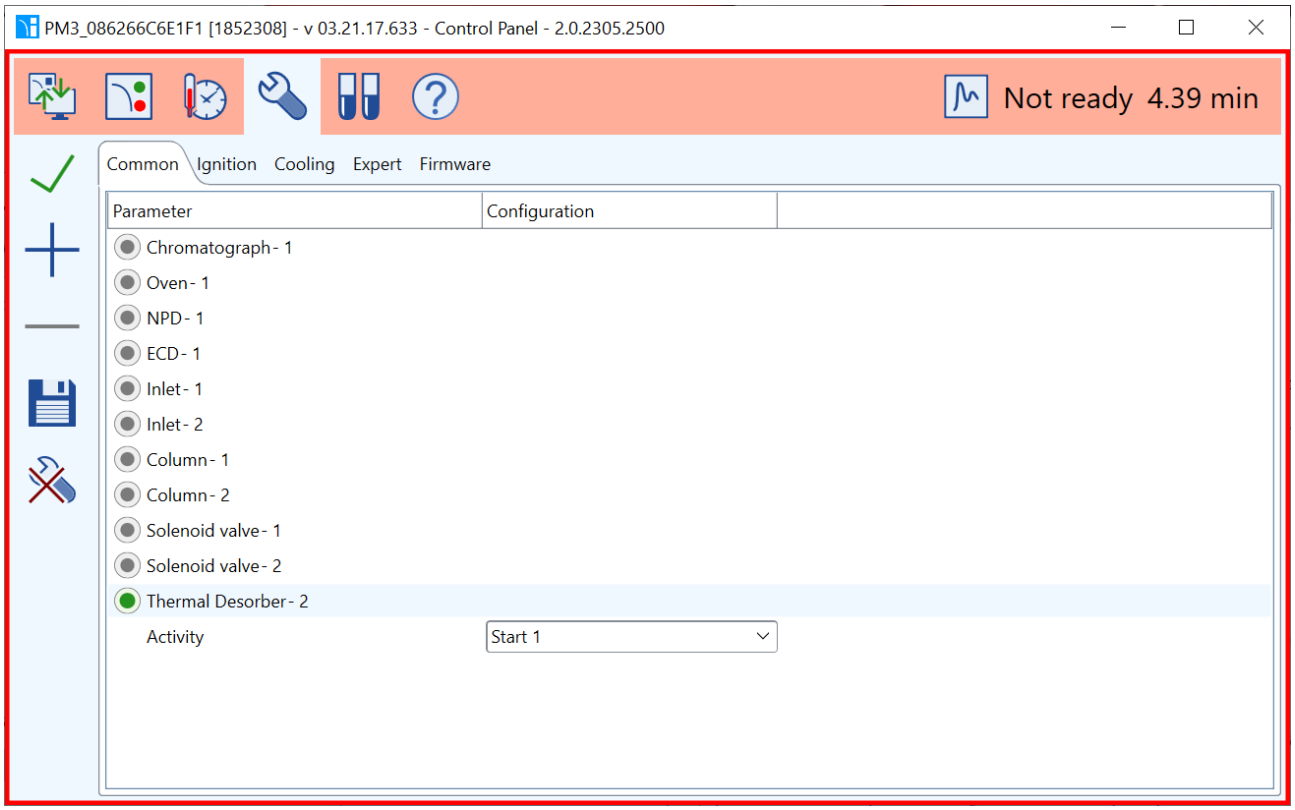
4.13.3 Configuration setting

Go to the Connection Settings tab and select **Scheduler v2**.



When connecting a thermal desorber to a Chromatec-Crystal chromatograph, an external device is added to the configuration.

Go to the **Configuration** page.



Add an **External Device**.

Select the following parameters:

Device Type - TDS.

Channel - Start 1.

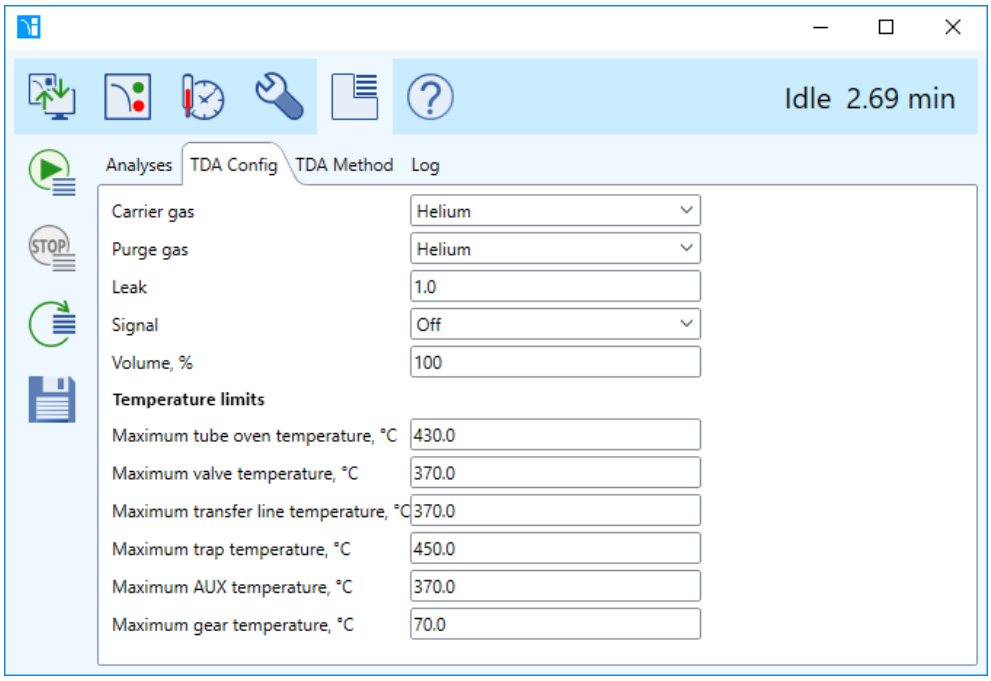
Connection Port - Port 2 (Corresponds to the SAMPLER connector).

Click **Apply**.

Click **Save**.

Go to the **Scheduler** page.

Select **TDA Config** tab.



Carrier gas – Select type of carrier gas: helium, nitrogen, argon, hydrogen.

Purge gas – Select type of purge gas that is used for desorption of compounds from sample tube: helium, nitrogen, argon.

Leak – Set the allowable value of pressure drop in the purge gas line, which is used for leak check at the installation of the sorbent tube. The recommended value is 1 kPa (0.2 psi). Value range from 0 to 10 kPa (from 0 to 2 psi).

Signal – Warning sound activation.

Volume, % – The volume of warning sounds.

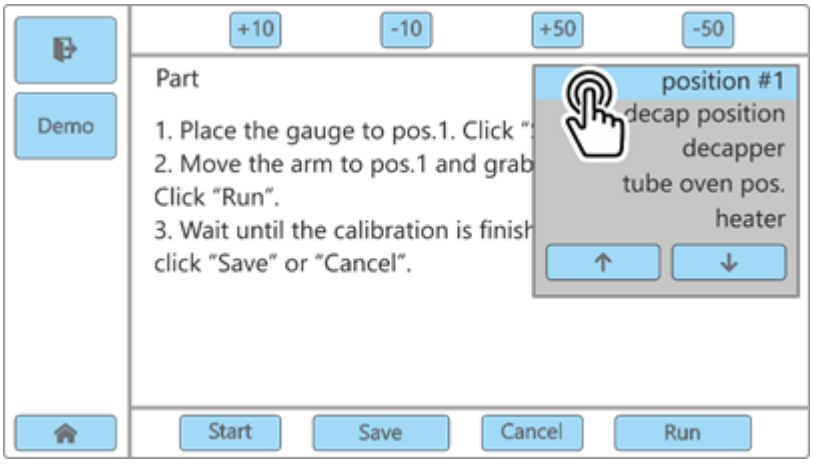
In the group **Temperature limits** set the maximum temperature for each heater. To protect the sorbent in the tube and trap from overheating, set the appropriate maximum operating temperatures for the parameters "Maximum tube temperature" and "Maximum trap temperature". These values depend on the type of sorbent used (section 6.4). It is recommended to use the default values for other units.

4.14 Manipulator setting

- Select the section **Service** in the Main menu.
- Select the section **Arm**.

4.14.1 Tray coordinates setting

- Select **position #1** in the drop list.



- Put the caps 6.433.075 on the gauge 8.236.753-01 and place it in the position #1 of the tray.

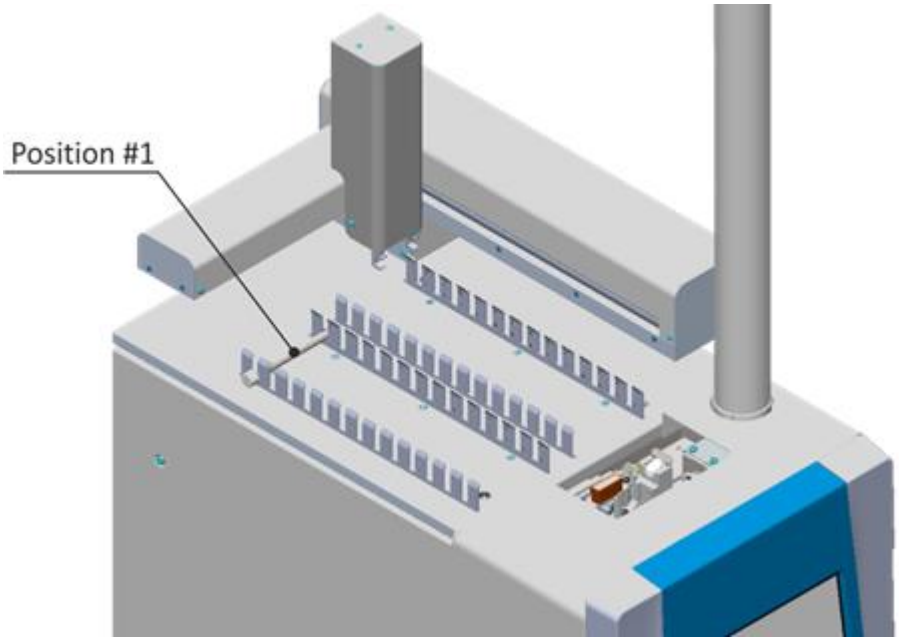


Figure 4.6 – Location of position #1 in the tray

- Click **Start**.
- Manually move the robotic arm at the position #1 of the tray.
- Grab the gauge by robotic arm so that gripper's location corresponds to the lines on the gauge.

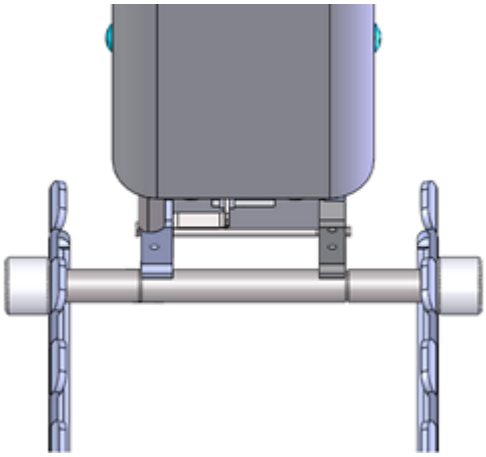
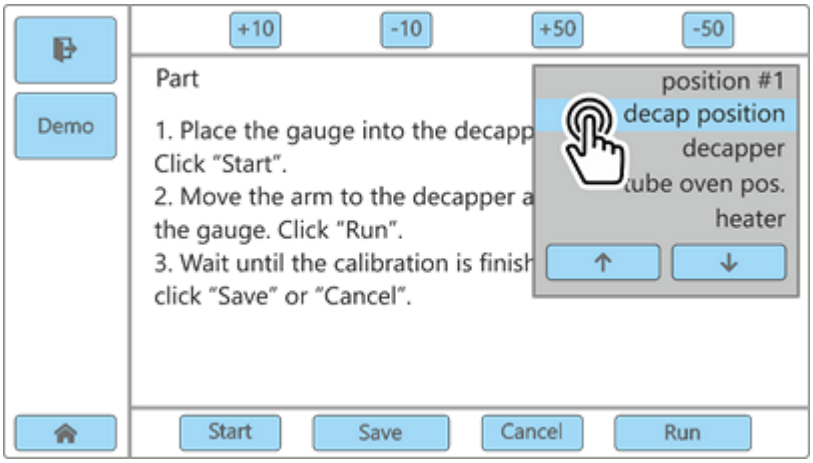


Figure 4.7 – Calibration of position #1 of the tray

- Fit the gauge in the gripper tightly.
- Click **Run**.
- Wait until the manipulator completed calibration and placed the gauge back into position #1.
- Click **Save**.

4.14.2 Decap position coordinates setting

- Select **decap position** in the drop list menu.



- Put the caps 6.433.075 on the gauge 8.236.753-01 and place it in the decap position.

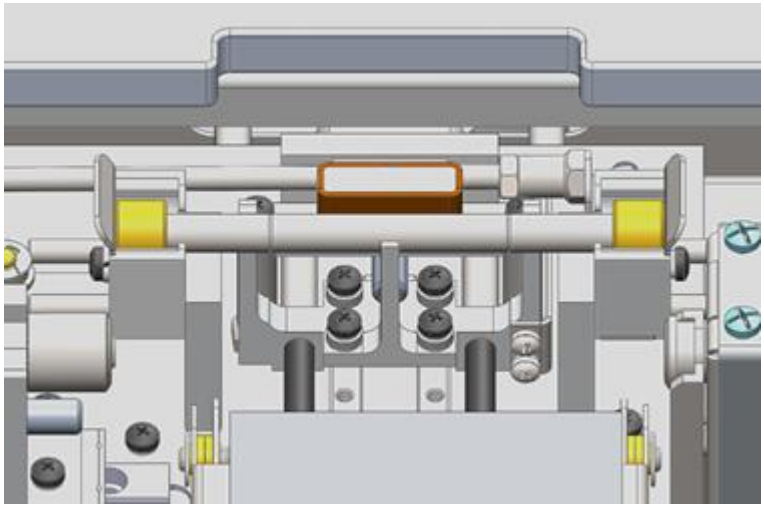


Figure 4.8 – The gauge in the decap position

- Click **Start**.
- Manually move the robotic arm at the decap position.
- Grab the gauge by robotic arm so that gripper's location corresponds to the lines on the gauge
- Fit theip gauge in the gripper tightly.

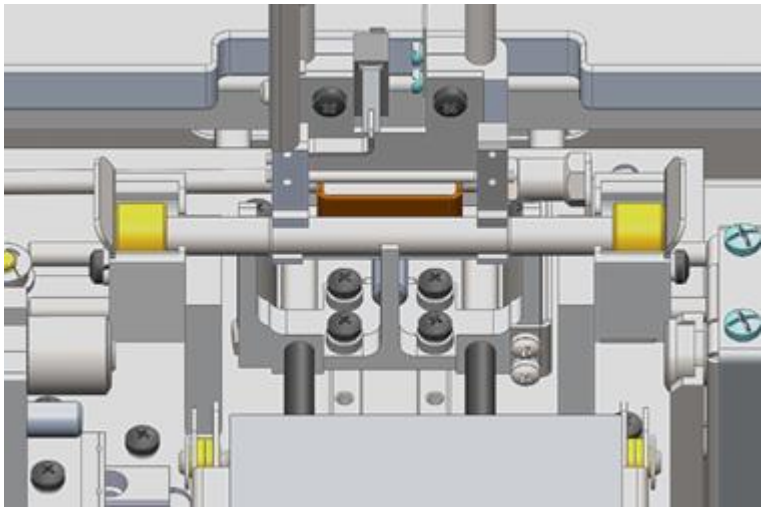
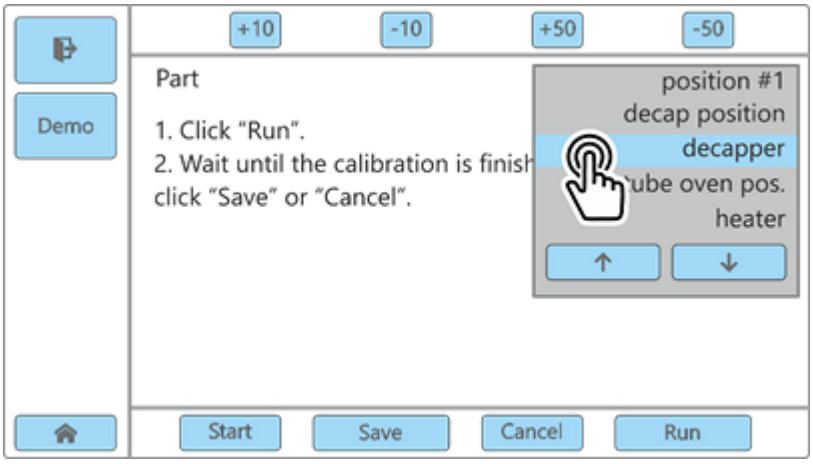


Figure 4.9 – Calibration of the decap position

- Click **Run**.
- Wait until the manipulator completed calibration and placed the gauge back into position #1.
- Click **Save**.

4.14.3 Decapper setting

- Select **decapper** in the drop list menu.

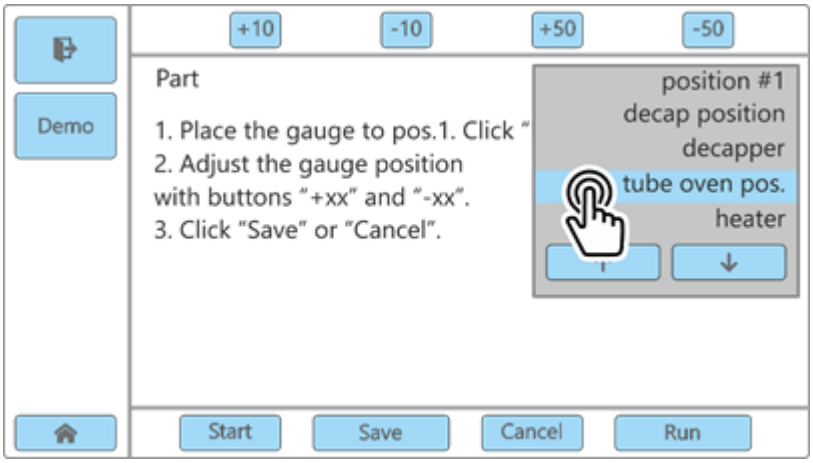


- Click **Run**.
- Wait until the calibration is completed.
- Click **Save**.

4.14.4 Tube oven position setting

Before setting of tube in the oven, execute setting of tray coordinates (Section 4.14.1), decap position coordinates (Section 4.14.2), and decapper setting (Section 4.14.3).

- Select **tube oven pos** in the drop list menu.



- Put the caps 6.433.075 on the gauge 8.236.753-01 and place it in the position #1 of the tray.

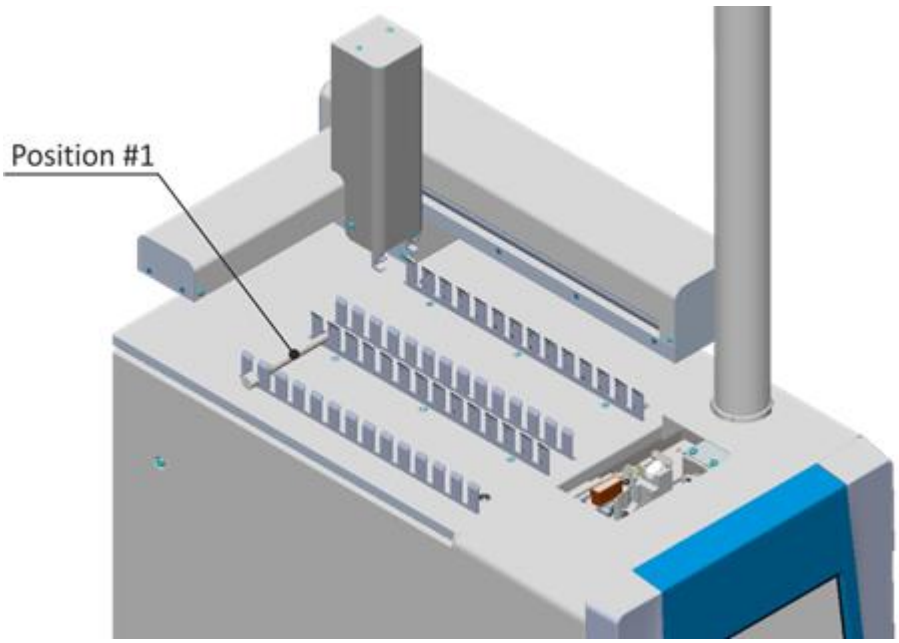


Figure 4.10 – Location of position #1 in the tray

- Click **Run**.
- Wait until the robotic arm transferred the gauge into the decap position, removed the caps and placed the gauge into the oven position.
- Using buttons "+50" and "-50" roughly adjust position of the gauge so that it was located on the same axis as right and left grabbing nozzles.
- Using buttons "+10" and "-10" adjust position of the gauge finely.

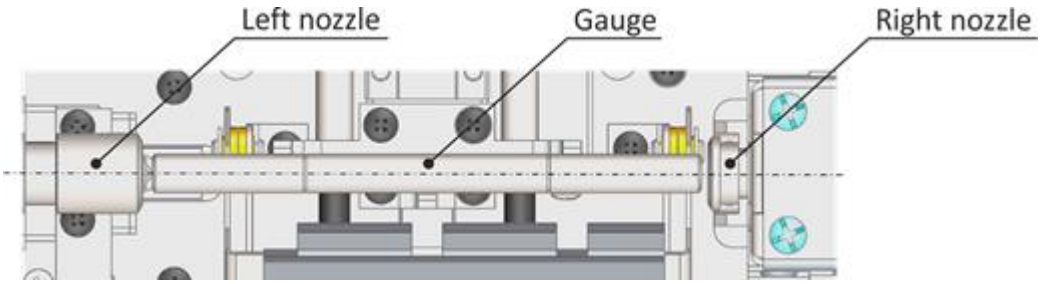
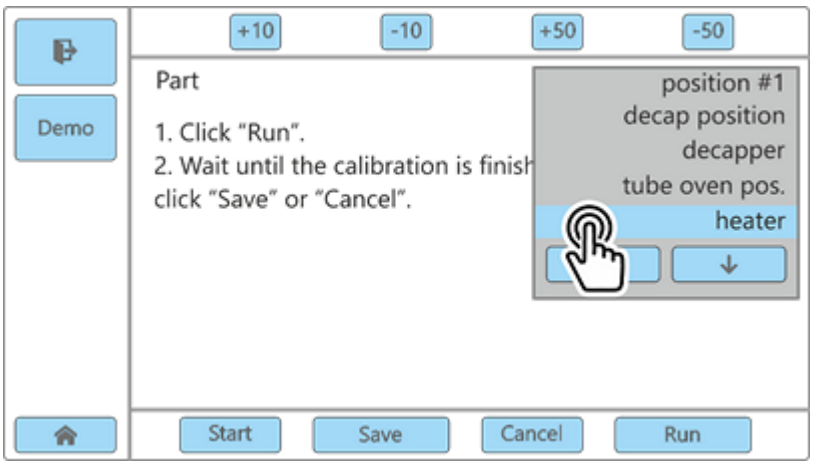


Figure 4.11 – Calibration of oven position

- Select **Save**.

4.14.5 Heater mechanism setting

- Select **heater** in the drop list menu.



- Select **Run**.
- Wait until the calibration is completed.
- Click **Save**.

4.14.6 Adjusting the tube sealing mechanism

The sealing sealer mechanism setting is available for TDA thermal desorber software version v2.02.002:0006 (14.02.2020), TDA handler module v01.01.15 and higher.

Before setting the tube sealer mechanism, set the tray coordinates (section 4.14.1), decap position coordinates (section 4.14.2), Decapper setting (section 4.14.3), and set Tube oven position (section 4.14.4).

The TDA thermal desorber has two options for setting the tube sealing mechanism, automatic and manual.

In the automatic setting, the clamping mechanism sets an initial value of 1000 steps and then checks for leaks. In case of leakage, the mechanism adds an additional 10 steps and the leakage is checked again. The maximum value of the number of steps is 1500. If leakage is observed at the maximum value of steps, the calibration is stopped and leakage of the system must be corrected (section 7.3.2). Automatic adjustment is carried out within 3-15 min and is recommended for initial system setup.

If the number of steps of the tube sealing mechanism is known, manual adjustment can be performed using buttons "+xx" and "-xx". In this case, a visual check of the tube seal is made by the degree of deformation of the ring in the right flange. Manual adjustment saves time, but some experience is required. The recommended number of steps for manual adjustment is 1050.

After manual adjustment of the tube sealing mechanism it is recommended to check the tightness according to section 7.3.2.

From the drop-down menu, select - sealing.



- Install the 6.433.075 caps on the pattern 8.236.753-01 and place it in the #1 position of the tray.
- Press the Start button.
- Press the Run button to start the automatic adjustment.
- Use the +xx and -xx buttons to perform the manual setup. Use the +50 and -50 buttons to approximately adjust the clamp with the O-ring gauge. Use the +10 and -10 buttons to make the fine adjustment.
- Press the Save button.

5 Operation

5.1 Turning on

Turn on the TDA and accompanied equipment as follows:

- Turn on the GC, the personal computer and other required equipment.
- Set up pressure of carrier gas and purge gas at the inlet of the thermal desorber in the range from 0.4 to 0.6 MPa (60 – 85 psig).
- Turn on the power switch on the rear panel of the TDA.

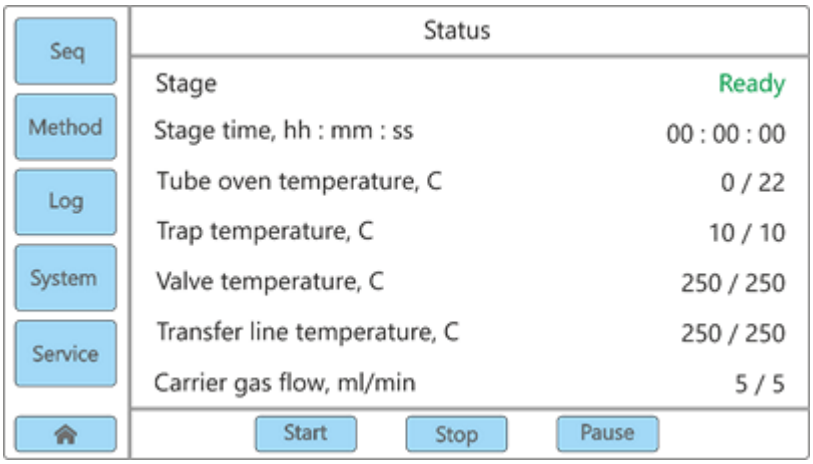
TDA initialization occurs within 10 seconds. After switching on, the thermal desorber goes to the zero stage. At the zero stage, there is no heating of any zones, and no gas flow rates set.

5.2 Using display

Use display located on the front panel to view and set the parameters of thermal desorber. Selecting commands and entering values is made by lightly touching the screen with your finger or stylus, designed to work with a capacitive screen.

Never use sharp and metal tools, such as pens, pencils, screwdrivers, when working with the display.

Main menu screen of the instrument is shown below.



The main menu displays information on the state of thermal desorber, as well as set and measured values of temperatures and gas flow rates.

- **Sequence** section (**Seq**) is used to create a sequence for performing a series of analyses.
- **Method** section allows you to create and edit methods.
- **Log** section contains information about the past analyses. This section also contains error messages.
- **System** section is used to set and view the system parameters of the device.

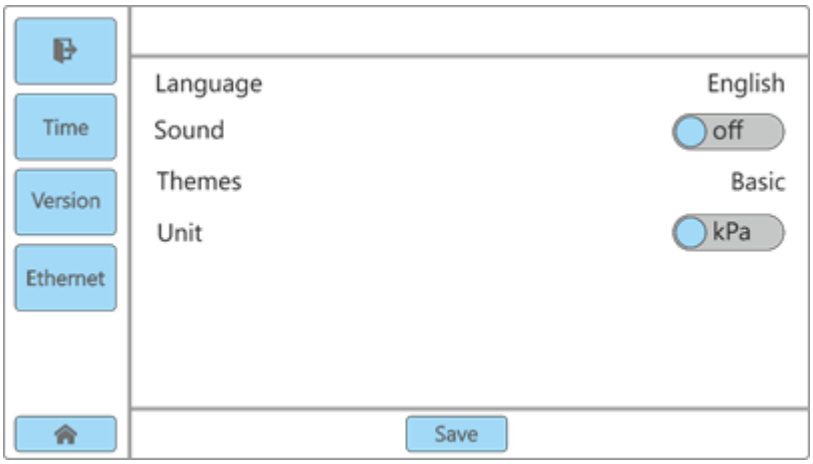
- **Service** section is intended for diagnostics and configuration of the thermal desorber. The settings of any parameters in this section are carried out by a Chromatec service engineer or trained and qualified person.
- Return to the Main menu .

5.3 Viewing and setting of system parameters

System parameters are usually set at the factory. You can use the default parameters. If necessary, the system parameters can be changed by the user.

5.3.1 System parameters setting

Click icon **System** in the Main menu.



Language – Select a language of the menu (English or Russian). After selecting the language, the menu and all messages will be displayed in this language.

Sound – Enable sound when you press the display to select and view parameters, as well as an audible alert before the manipulator moves and when an error occurs in operation.

Themes – Set color background for menu.

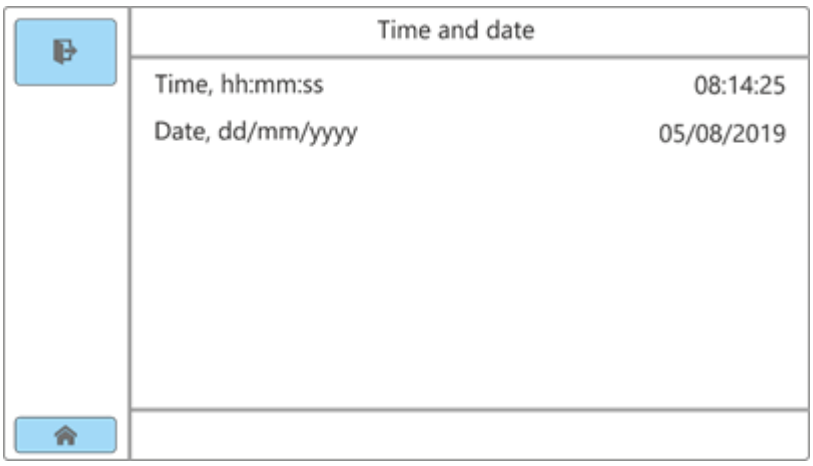
Unit – Selection of pressure units (kPa or psi).

GC ready level – Setting the GC logical level of ready (low or high). With Chromatec Crystal GC select **low** level.

Click **Save** to save parameters.

5.3.2 Time and date setting

Click icon **Time** to set date and time.



Time – Time setting (hh:mm:ss) in 24-hour format.

Date – Date setting (dd.mm.yyyy).

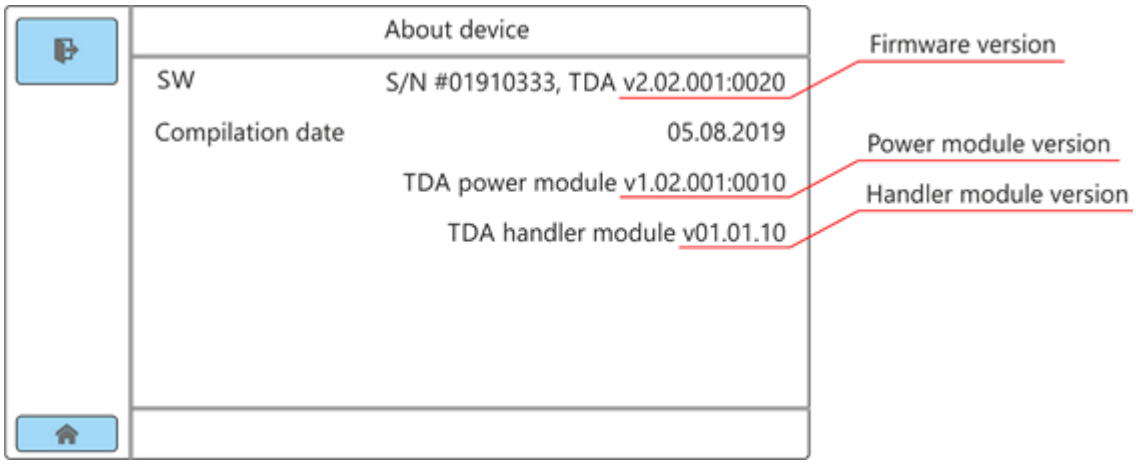
Click icon to return to the previous menu.

Click **Save** to save parameters.

5.3.3 Firmware version view

Viewing firmware version is helpful when contacting the manufacturer's services.

Click icon **Version** in the section **System** to view the firmware version.



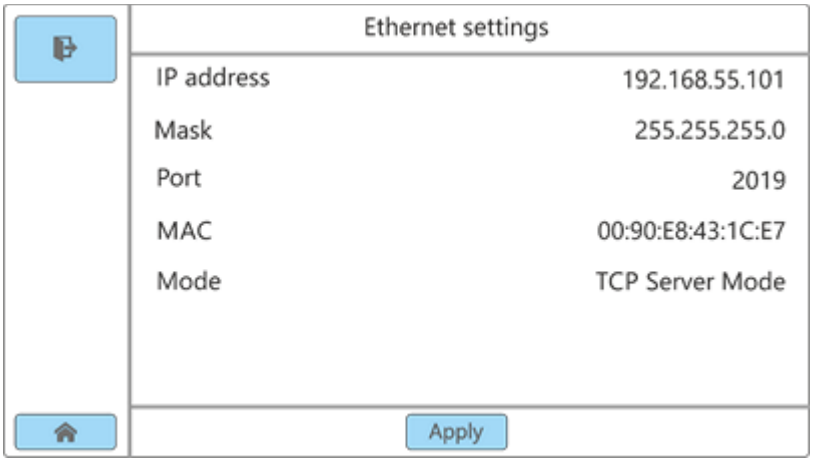
Click icon to return to the previous menu.

5.3.4 Ethernet connection setting

Local network connection is used when connection of thermal desorber to a computer with handling through Control Panel software required.

The TDA is supplied with a default IP address that may not match your local network. Contact your network administrator to assign an IP address, subnet mask, and connection port.

Click **Ethernet** in the section **System**.



IP address – IP address assigned by administrator.

Mask – subnet mask, assigned by network administrator.

Port – port number, assigned by network administrator.

MAC – Network interface ID (MAC-address). It is assigned by manufacturer and cannot be changed.

Mode – To connect the thermal desorber to the local network, select **TCP Server Mode**.

Click **Apply**.

Click icon to return to the previous menu.

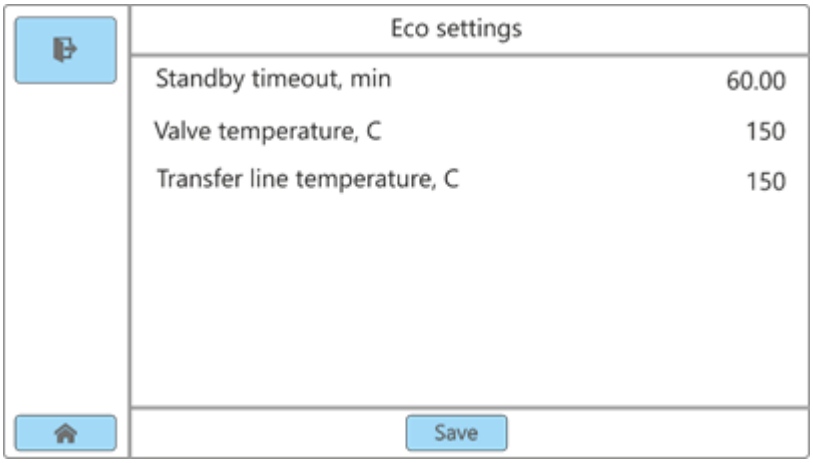
5.3.5 Eco Settings

Eco settings is available for TDA thermal desorber software version v2.02.002:000D (02.07.2020) and higher.

Eco standby mode is used to reduce power consumption when the thermal desorber is idle for an extended period of time. In this mode, the temperature of the tap, transition line, and trap cooling is reduced.

Exit eco standby mode when you transmit a method or start a sequence.

- Click the Eco icon in the System section.



Standby timeout - Specify the time the thermal desorber is idle. After the set time value has elapsed, the unit enters the energy saving mode. The range of values is from 0 to 120 min. If 0 is set, the energy saving mode will not be used. To avoid entering the power saving mode when performing a sequence of analyses, the waiting time should be longer than the duration of the chromatographic analysis. The recommended wait time is 60 min.

Valve Temperature - The temperature of the valve in power save mode. The range of values is 150 °C to 350 °C. The recommended valve temperature is 150 °C.

Transfer Line temperature - Transition line temperature in energy saving mode. The range of values is 40 to 350 °C. The recommended transition line temperature is 150 °C.

- Press the Save button.
- Press icon to return to the previous menu.

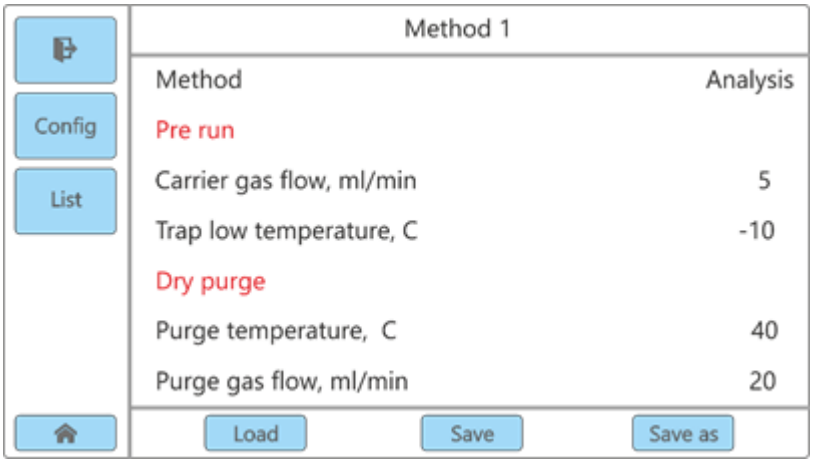
5.4 Creating a method

The method is a mode of operation of the thermal desorber containing the parameters of temperatures and flow rates of gases set during the analysis.

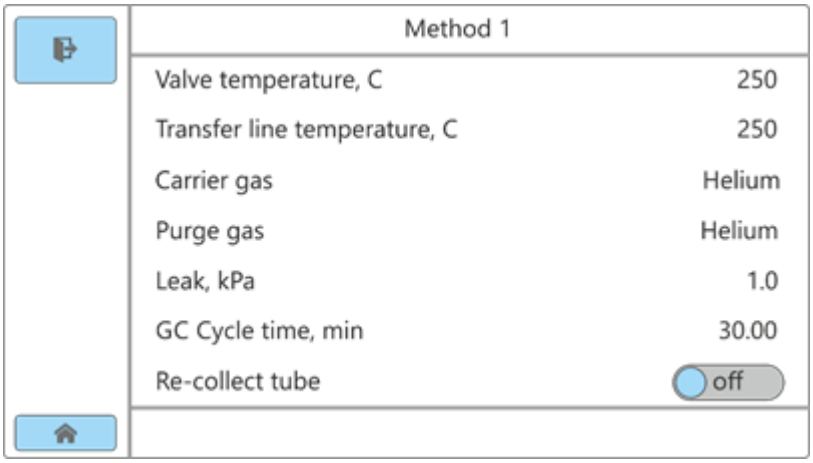
The parameters of the method depend on the components to be determined, the sorbent used in the tube and the trap. In any case, a specific method is created for each type of analysis.

Up to 10 different methods can be stored in the memory of the thermal desorber. If you need more methods, using Control Panel software allows storing unlimited number of methods.

Select section **Method** in the Main menu.



Set **Configuration** parameters in this section.



Valve temperature – Heating the valve is necessary to prevent condensation of analytes. The recommended temperature of the valve in the analysis of volatile organic compounds from 200 to 250 °C. Operating temperature range from 150 to 350 °C.

Transfer line temperature – The heated transfer line is designed to transfer the analytes from the trap to the chromatographic column. Typically, the transfer line temperature is set at 5 to 10 °C above valve temperature. Operating temperature range from 40 to 350 °C.

Carrier gas – Select type of carrier gas: helium, nitrogen, argon, hydrogen.

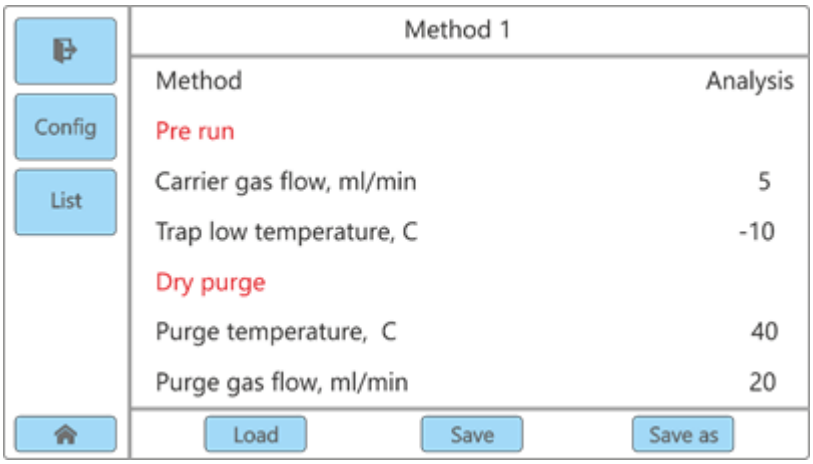
Purge gas – Select type of purge gas that is used for desorption of compounds from sample tube: helium, nitrogen, argon.

Leak – Set the allowable value of pressure drop in the purge gas line, which is used for leak check at the installation of the sorbent tube. The recommended value is 1 kPa (0.2 psi). Value range from 0 to 10 kPa (from 0 to 2 psi).

GC Cycle time – This is the cycle time of GC run from start to start, including the cooling time and time required to come to Ready state. This parameter allows desorption of the next tube during the current analysis in the chromatograph. The thermal desorber calculates the start time for the desorption of the next tube in such a way that it is completed to the readiness of the GC.

Re-collect tube –This option is used to collect a part of the sample on the sorbent tube during desorption of components from the trap.

Return to the previous menu **Method** to set operating parameters.



Select operating mode of the thermal desorber – Analysis, Conditioning, Trap clean, Trap test.

Analysis mode is used for desorption of the analyzed substances from the sample tube, followed by focusing of the components in the trap and further transfer of analytes into the GC.

Conditioning mode is to be chosen to remove contaminants from the sorbent tubes, for example, before sampling.

Trap clean mode allows you to remove volatile contaminants from trap sorbent, for example, when installing a new trap.

Trap test mode checks the purity of the trap by heating it and transfer the gas flow to the GC for blank analysis.

A detailed description of the modes of operation is given in Section 3.

5.4.1 Analysis mode

Section **Pre run**



Carrier gas flow – The recommended value is from 5 to 15 mL/min. The flow rate of the carrier gas must not exceed the flow rate of the split vent gas in the GC. Value ranges from 0 to 200 mL/min.

Trap low temperature – It is installed during the desorption of components from the sorbent tube to concentrate analytes in a cooled trap. The recommended value is minus 10 °C. During desorption of samples with high humidity, the temperature of the trap is to be set at 4 °C. Temperature ranges from minus 20 to 35 °C.

Section **Dry purge**

Dry purge	
Purge temperature, C	0
Gas flow, ml/min	20
Purge time, min	2.00

Purge temperature – Recommended value is from 40 to 50 °C. For stable operation, the purge temperature must be at least 5 °C above the ambient temperature. Value ranges from 0 to 425 °C. A setpoint of 0 °C means purging at room temperature.

Purge gas flow – Recommended value from 20 to 30 mL/min. Value ranges from 0 to 200 mL/min.

Purge time – Recommended value from 1 to 2 minutes. Value ranges from 0 to 15 minutes. If time is 0, the tube will not be purged.

Section **Desorption**

Desorption	
Desorb temperature, C	280
Gas flow, ml/min	30
Desorb time, min	5.00

Desorb temperature – Set for desorption of the analyzed substances from the sorbent tube. The desorption temperature depends on the components to be analyzed and the type of sorbent, it is generally recommended in test method procedures. Temperature ranges from 35 to 425 °C.

Desorb gas flow – For optimal desorption efficiency, it is recommended to set the purge gas flow rate from 20 to 50 mL/min. Flow ranges from 0 to 200 mL/min.

Desorb time – Time is set in such a way as to ensure the most complete desorption of the analyzed components from the sorbent and their transfer into the trap. The countdown starts from the moment of heating. When choosing the desorption time, it is necessary to consider the time of heating the thermostat to the set temperature. For example, the heating time of the sorbent tube thermostat to 300 °C is about 3 minutes. Value ranges from 0 to 99 minutes.

Section **Analysis**

Analysis	
Split	0
Trap temperature, C	280
Heating rate, C/min	2000
Analysis time, min	2.00

Split – It is used with Re-collection option. During the desorption from the trap part of the sample is sent to the vent (Section 6.13.12) or to a re-collect tube (Section 6.13.13). When working with the re-collect tube, the recommended value is 1, which means a 1:1 flow split. If split flow is 0, the sample is not divided, and full sample volume sent to the column. Value ranges from 0 to 100.

Trap high temperature – The temperature of the trap depends on the sorbent type. Temperature ranges from 35 to 425 °C.

Heating rate – Select the values: 500; 1000; 1500; 2000; 2500; 3000 °C/min. When the trap heats up quickly, volatile components pass into the column in a few seconds. Slow heating is recommended for determination of thermolabile components.

Analysis time – Sets the time during which the trap will heat up. Recommended value is 1 to 2 minutes. Value ranges from 0 to 15 minutes.

Section **Conditioning**

Conditioning	
Tube oven temperature, C	300
Gas flow, ml/min	50
Conditioning time, min	10.00

Tube oven temperature – Setting temperature of the thermostat during cleaning of the sorbent tube. The cleaning temperature of the tube depends on the maximum operating temperature of the sorbent. Usually, the cleaning temperature is set at 10-20 °C above the temperature of desorption. Temperature ranges from 35 to 425 °C.

Purge gas flow – To reduce the tube cleaning time, it is recommended to set the purge gas flow rate from 50 to 100 mL/min. Flow ranges from 0 to 200 mL/min.

Conditioning time – The recommended tube cleaning time after analysis is 10 to 15 minutes. Value ranges from 0 to 1440 minutes. When setting time is 0, the tube will not be cleaned.

5.4.2 Conditioning mode

Tube oven temperature, C	300
Gas flow, ml/min	50
Conditioning time, min	20.00

Tube oven temperature – The temperature of the conditioning tube depends on the maximum operating temperature of the sorbent. See Section 6.6 for details about tubes conditioning. Temperature ranges from 35 to 425 °C.

Purge gas flow – The recommended purge gas flow rate is from 50 to 100 mL/min. Flow ranges from 0 to 200 mL/min.

Conditioning time – The recommended tube cleaning time is 15 to 30 minutes. Value ranges from 0 to 1440 minutes.

5.4.3 Trap clean mode

Trap temperature, C	280
Gas flow, ml/min	30
Conditioning time, min	5.00

Trap high temperature – The cleaning temperature of the trap depends on the maximum operating temperature of the sorbent, which is given in the passport of the trap. See Section 6.6 for details about tubes conditioning. Temperature ranges from 35 to 425 °C.

Purge gas flow – The recommended purge gas flow rate is from 30 to 50 mL/min. Flow ranges from 0 to 200 mL/min.

Conditioning time – The recommended tube cleaning time is 10 to 15 minutes. Value ranges from 0 to 15 minutes.

5.4.4 Trap test mode

Carrier gas flow, ml/min	10
Trap temperature, C	280
Heating rate, C/min	2000
Split	0
Analysis time, min	2.00

Carrier gas flow – The recommended value is from 5 to 15 mL/min. The flow rate of the carrier gas must not exceed the flow rate of the split vent gas in the GC. Value ranges from 0 to 200 mL/min.

Trap high temperature – The temperature of the trap depends on the sorbent type. Temperature ranges from 35 to 425 °C.

Heating rate – Select the values: 500; 1000; 1500; 2000; 2500; 3000 °C/min.

Split – It is used to send a part of the sample to the vent during the desorption of components from the trap. If split flow is 0, the sample is not divided, and full sample volume sent to the column. Value ranges from 0 to 100.

Analysis time – Sets the time during which the trap will heat up. Recommended value is 1 to 2 minutes. Value ranges from 0 to 15 minutes.

Once the method developed, click **Save as**, enter a name of the method and click ✔.

5.5 Method editing

Enter the section **Method** in the Main menu.

Click **Load** and select a method saved in the past.

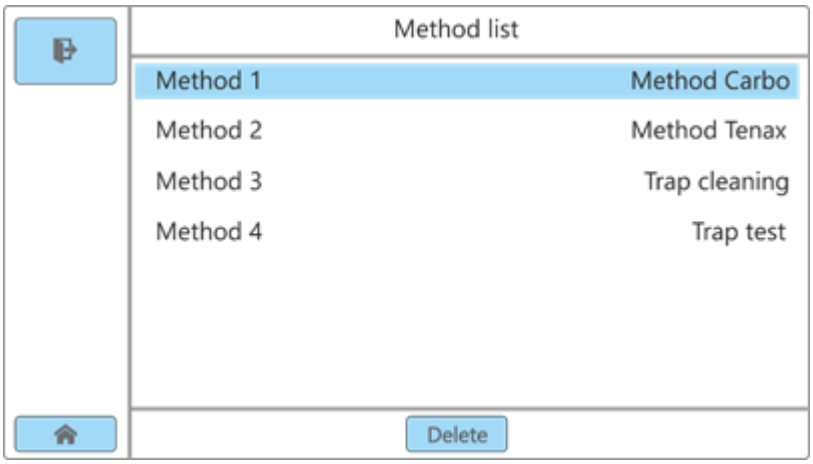
Make the necessary changes or edit the required method parameters according to Section 0.

Click **Save** to apply changes in the method.

5.6 Method deleting

Enter the section **Method** in the Main menu.

Click **List** to get access to the list of saved methods.

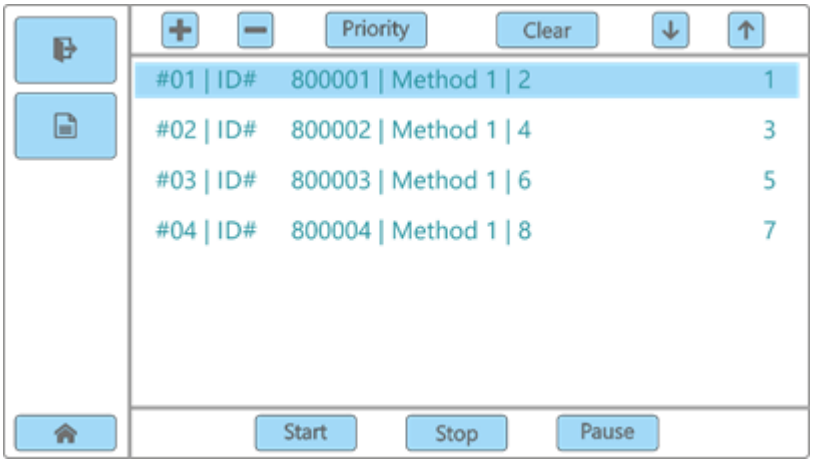


Highlight the line with the method you want to delete and press **Delete**.

5.7 Working with sequence

A sequence is a series of analyses performed according to a given plan.

Enter the section **Sequence** (**Seq**) in the main menu.



Below is a description of the buttons and icons when working with a sequence.

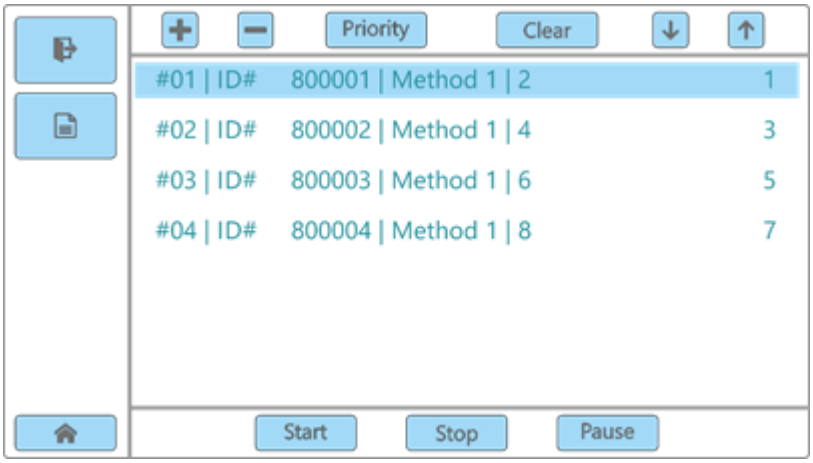
Return to a previous menu.

Editing of highlighted analysis.

- Adding a new analysis.
- Delete selected analysis.
- Set a priority for the selected analysis in the sequence. Priority analysis will be conducted immediately after the end of the current analysis.
- Clear the sequence.
- Move the selected analysis down one position in the list.
- Move the selected analysis up one position in the list.
- Start running the sequence.
- Stopping running the sequence, also stops the execution of the current analysis.
- Temporary termination of the sequence, the current analysis will be completed to the end. To resume the sequence, you must press **Pause** again.

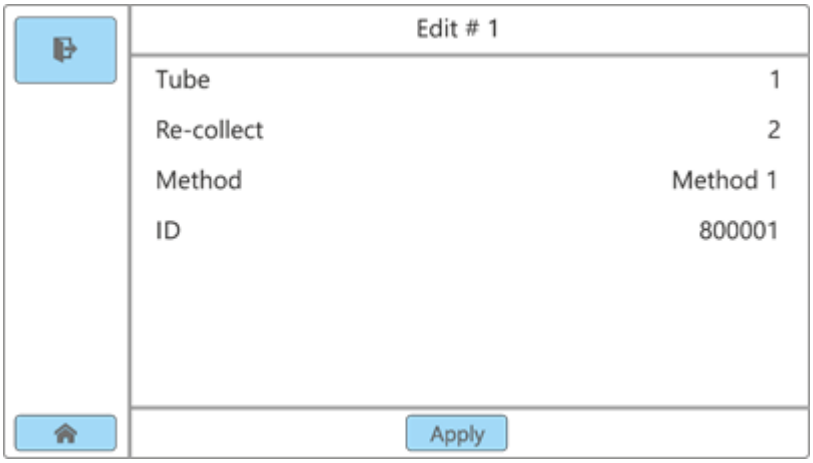
5.7.1 Creating a sequence

Click the **Sequence** section in the Main menu.



Add a new line in the sequence table.

Click the icon to edit selected analysis.



Fill out the following lines:

Tube – Sample tube position in the tray.

Re-collect – The position of the re-collect tube in the tray, it should be indicated when using the re-collect tube. You can use a new tube or the same, sample was desorbed from.

Method – Select analysis method.

ID – Sample tube ID (bar code), indicated when applicable.

Click **Apply**.

Add and edit the required number of lines (analyses) in the sequence in the same way.

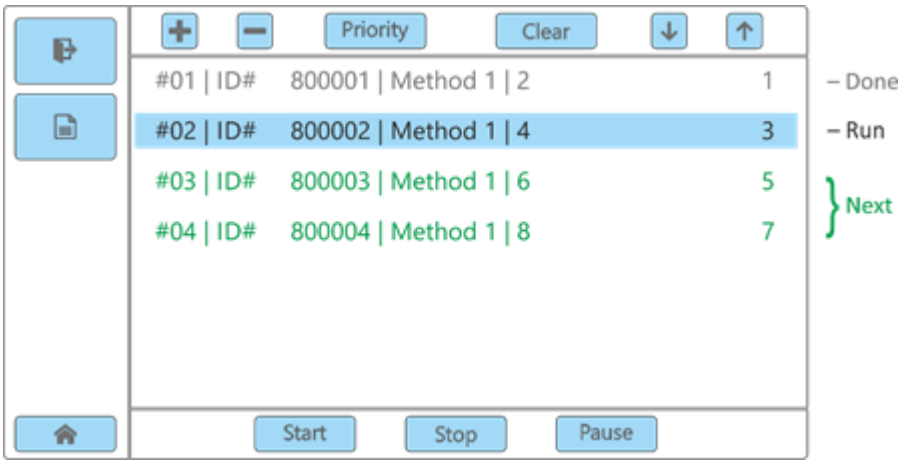
Using the internal software, you can create a sequence containing no more than 90 analyses. If you need more analysis, use Control Panel software that allows to create an unlimited number of analysis (Section 0).

5.7.2 Starting a sequence

Enter the section **Sequence** (**Seq**) in the Main menu.

Click **Start**.

After starting the sequence, the name of the current analysis will turn from blue-green to black, and the waiting analyzes in the sequence will be colored green. The completed analyses are displayed in grey.



5.7.3 Stopping a sequence

If you interrupt the analysis sequence, the current analysis also stops.

Enter the section **Sequence** (**Seq**) in the Main menu.

Click **Stop**.

If the analysis was stopped during the desorption and some components enter the trap, then the trap must be cleaned before starting the next analysis.

5.7.4 Pause of sequence

Pause of the sequence, while the current analysis will be completed until the end.

Enter the section **Sequence** (**Seq**) in the Main menu.

Click **Pause**. To resume the sequence, you should press **Pause** again.

5.8 TDA Control via Control Panel software

The setting of the parameters of the thermal desorber is performed before the start of the analysis.

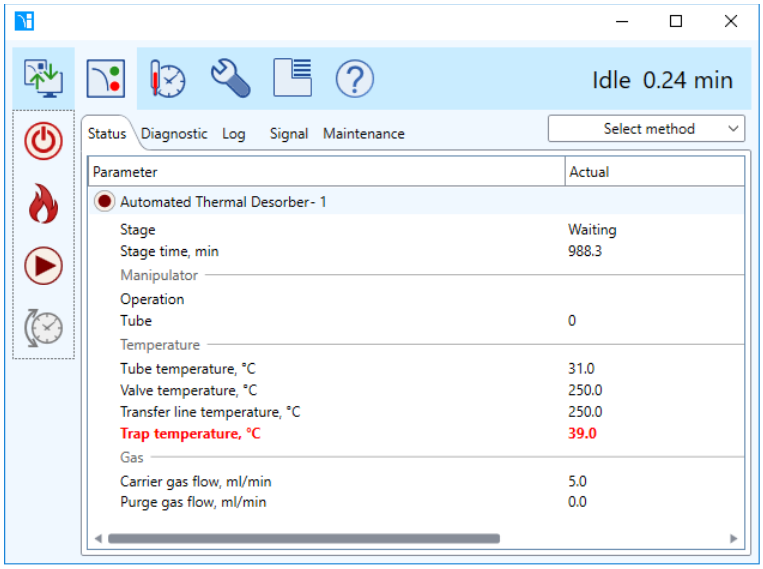
Start the connection using **Control Panel** icon.

5.8.1 Status

Go to the **Status** tab.

The Status tab displays the set and actual values, as well as the stage of TDA operation.

The parameters that have reached their set point are displayed in black. If any parameter is not ready, it is displayed in red.



5.8.2 Sequence

Go to the **Sequence** tab.

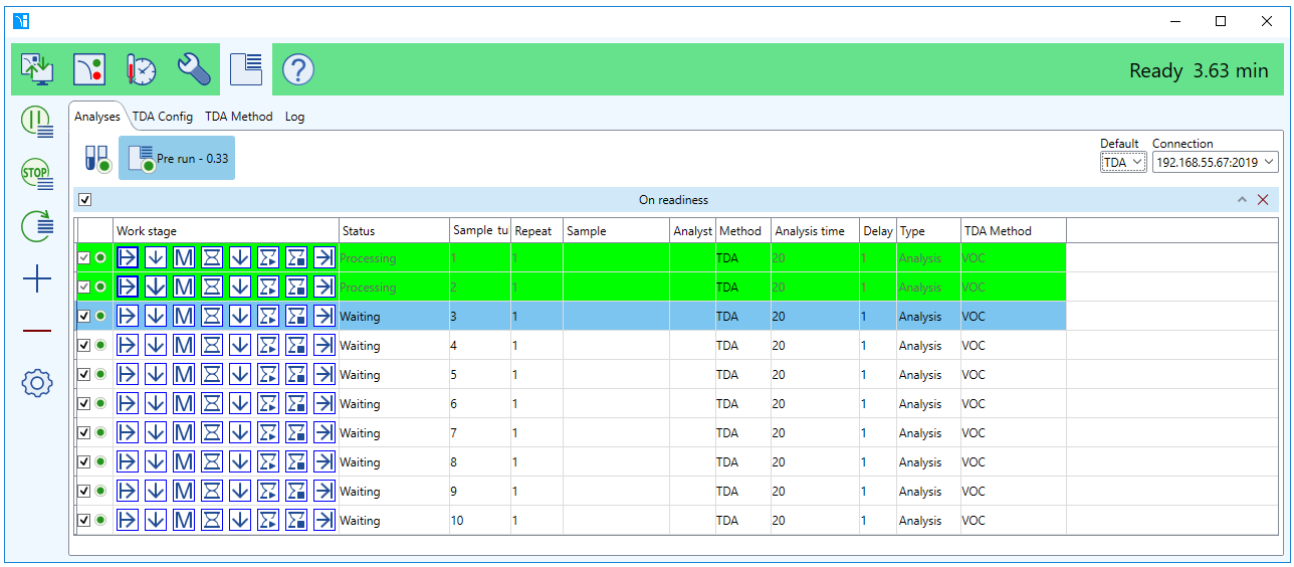
There are following tabs:

Analysers. A list of scheduled analyses (sequence plan).

TDA Config. Instrument settings.

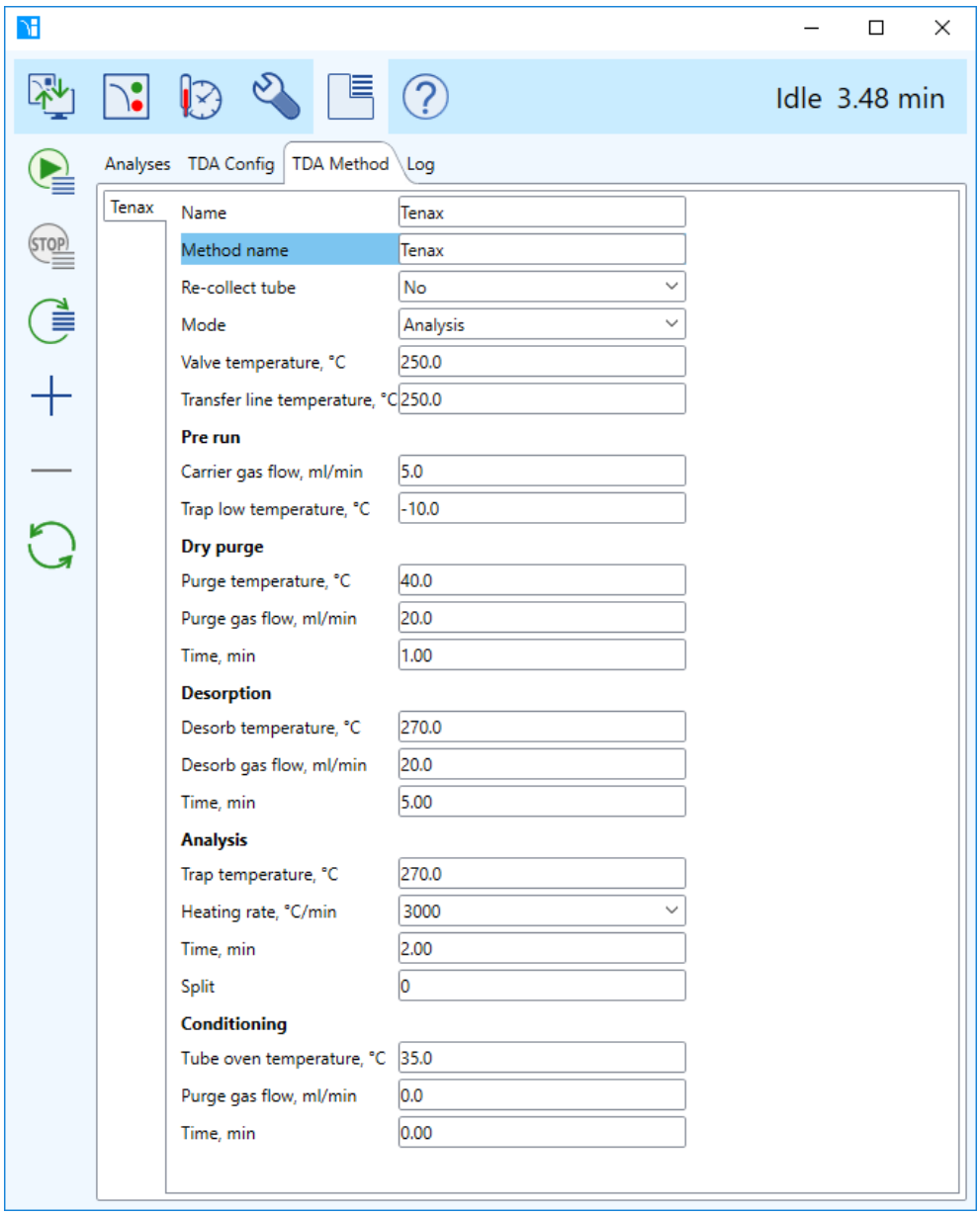
TDA Method. Operating method.

Log. Review of performed operations and errors occurred during sequence running.



5.8.3 Configuration

Select **TDA Method** tab.



Add a new Method.

In the field **Method name** enter a name of the method.

Re-collect tube – This option is used to collect a part of the sample on the sorbent tube during desorption of components from the trap.

Select operating mode of the thermal desorber – Analysis, Conditioning, Trap cleaning, Trap test.

Analysis mode is used for desorption of the analyzed substances from the sample tube, followed by focusing of the components in the trap and further transfer of analytes into the GC.

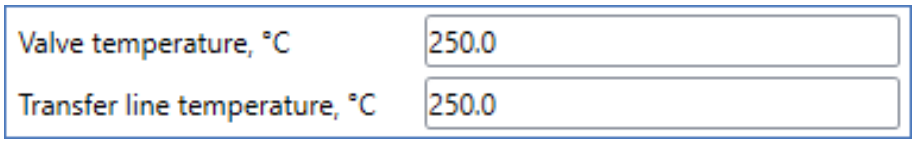
Conditioning mode is to be chosen to remove contaminants from the sorbent tubes, for example, before sampling.

Trap clean mode allows you to remove volatile contaminants from trap sorbent, for example, when installing a new trap.

Trap test mode checks the purity of the trap by heating it and transfer the gas flow to the GC for blank analysis.

A detailed description of the modes of operation is given in Section 3.

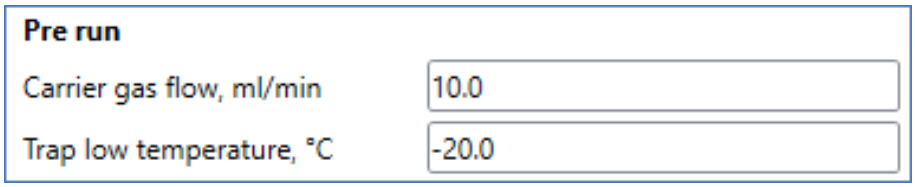
Valve temperature – Heating the valve is necessary to prevent condensation of analytes. The recommended temperature of the valve in the analysis of volatile organic compounds from 200 to 250 °C. Operating temperature range from 150 to 350 °C.



Transfer line temperature – The heated transfer line is designed to transfer the analytes from the trap to the chromatographic column. Typically, the transfer line temperature is set at 5 to 10 °C above valve temperature. Operating temperature range from 40 to 350 °C.

Analysis mode

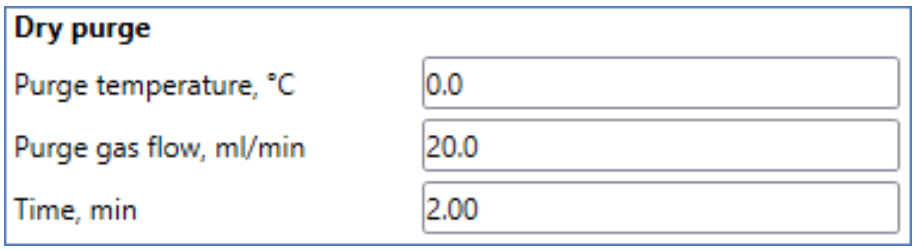
Section **Pre run**



Carrier gas flow – The recommended value is from 5 to 15 mL/min. The flow rate of the carrier gas must not exceed the flow rate of the split vent gas in the GC. Value ranges from 0 to 200 mL/min.

Trap low temperature – It is installed during the desorption of components from the sorbent tube to concentrate analytes in a cooled trap. The recommended value is minus 10 °C. During desorption of samples with high humidity, the temperature of the trap is to be set at 4 °C. Temperature ranges from minus 20 to 35 °C.

Section **Dry purge**

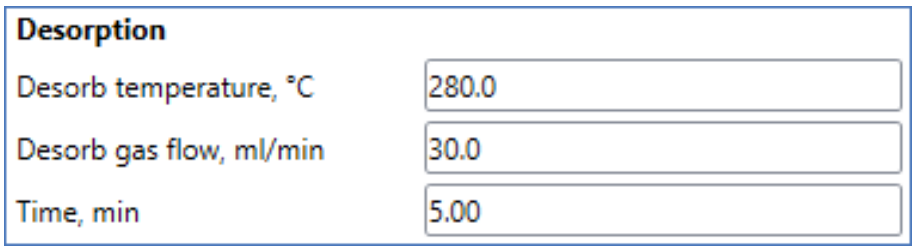


Purge temperature – Recommended value is from 40 to 50 °C. For stable operation, the purge temperature must be at least 5 °C above the ambient temperature. Value ranges from 0 to 425 °C. A setpoint of 0 °C means purging at room temperature.

Purge gas flow – Recommended value from 20 to 30 mL/min. Value ranges from 0 to 200 mL/min.

Purge time – Recommended value from 1 to 2 minutes. Value ranges from 0 to 15 minutes. If time is 0, the tube will not be purged.

Section **Desorption**

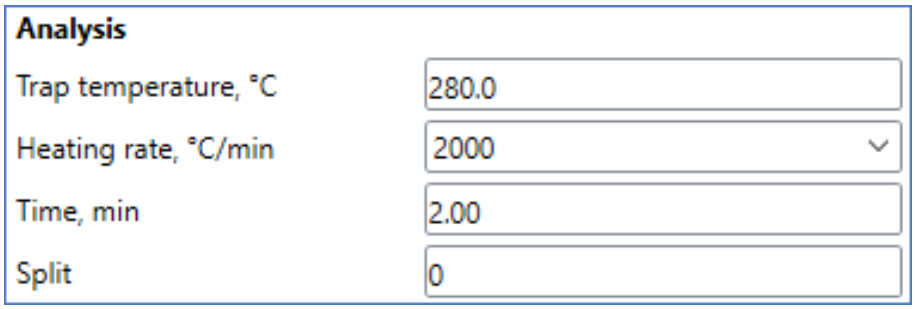


Desorb temperature – Set for desorption of the analyzed substances from the sorbent tube. The desorption temperature depends on the components to be analyzed and the type of sorbent, it is generally recommended in test method procedures. Temperature ranges from 35 to 425 °C.

Desorb gas flow – For optimal desorption efficiency, it is recommended to set the purge gas flow rate from 20 to 50 mL/min. Flow ranges from 0 to 200 mL/min.

Desorb time – Time is set in such a way as to ensure the most complete desorption of the analyzed components from the sorbent and their transfer into the trap. The countdown starts from the moment of heating. When choosing the desorption time, it is necessary to consider the time of heating the thermostat to the set temperature. For example, the heating time of the sorbent tube thermostat to 300 °C is about 3 minutes. Value ranges from 0 to 99 minutes.

Section **Analysis**



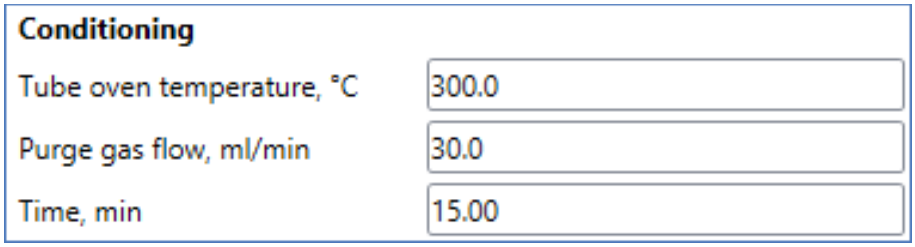
Trap high temperature – The temperature of the trap depends on the sorbent type. Temperature ranges from 35 to 425 °C.

Heating rate – Select the values: 500; 1000; 1500; 2000; 2500; 3000 °C/min. When the trap heats up quickly, volatile components pass into the column in a few seconds. Slow heating is recommended for determination of thermolabile components.

Analysis time – Sets the time during which the trap will heat up. Recommended value is 1 to 2 minutes. Value ranges from 0 to 15 minutes.

Split – It is used with Re-collection option. During the desorption from the trap part of the sample is sent to the vent (Section 6.13.12) or to a re-collect tube (Section 6.13.13). When working with the re-collect tube, the recommended value is 1, which means a 1:1 flow split. If split flow is 0, the sample is not divided, and full sample volume sent to the column. Value ranges from 0 to 100.

Section **Conditioning**

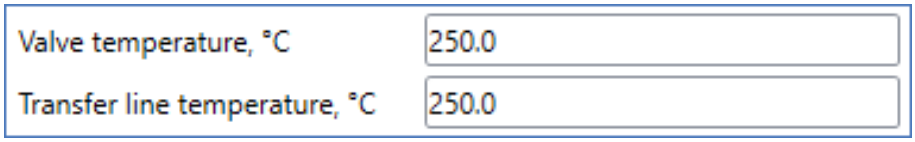


Tube oven temperature – Setting temperature of the thermostat during cleaning of the sorbent tube. The cleaning temperature of the tube depends on the maximum operating temperature of the sorbent. Usually, the cleaning temperature is set at 10-20 °C above the temperature of desorption. Temperature ranges from 35 to 425 °C.

Purge gas flow – To reduce the tube cleaning time, it is recommended to set the purge gas flow rate from 50 to 100 mL/min. Flow ranges from 0 to 200 mL/min.

Conditioning time – The recommended tube cleaning time after analysis is 10 to 15 minutes. Value ranges from 0 to 1440 minutes. When setting time is 0, the tube will not be cleaned.

Conditioning mode

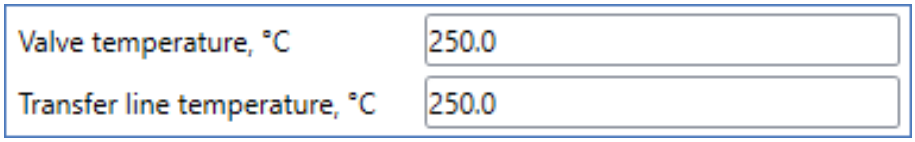


Tube oven temperature – The temperature of the conditioning tube depends on the maximum operating temperature of the sorbent. See Section 6.6 for details about tubes conditioning. Temperature ranges from 35 to 425 °C.

Purge gas flow – The recommended purge gas flow rate is from 50 to 100 mL/min. Flow ranges from 0 to 200 mL/min.

Conditioning time – The recommended tube cleaning time is 15 to 30 minutes. Value ranges from 0 to 1440 minutes.

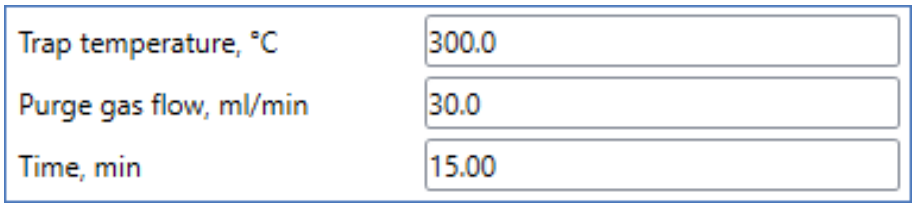
Trap cleaning mode



Valve temperature - Heating of the valve is necessary to prevent condensation of components. The recommended valve temperature for trap cleaning is 250 to 300°C. The operating temperature range is 150 to 350°C.

Transfer Line Temperature - When cleaning the trap, contaminants do not enter the transfer line and chromatograph. However, to accelerate instrument availability before and after trap cleaning,

it is recommended to set the transfer line temperature similar to the analysis. The operating temperature range is from 40 to 350°C.

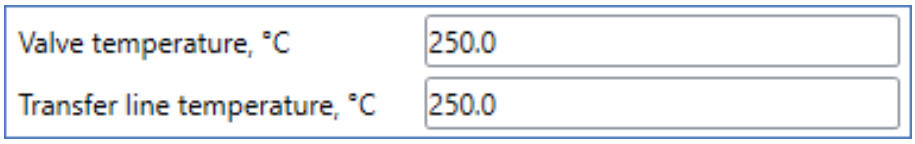


Trap temperature – The cleaning temperature of the trap depends on the maximum operating temperature of the sorbent, which is given in the passport of the trap. See Section 6.6 for details about tubes conditioning. Temperature ranges from 35 to 425 °C.

Purge gas flow – The recommended purge gas flow rate is from 30 to 50 mL/min. Flow ranges from 0 to 200 mL/min.

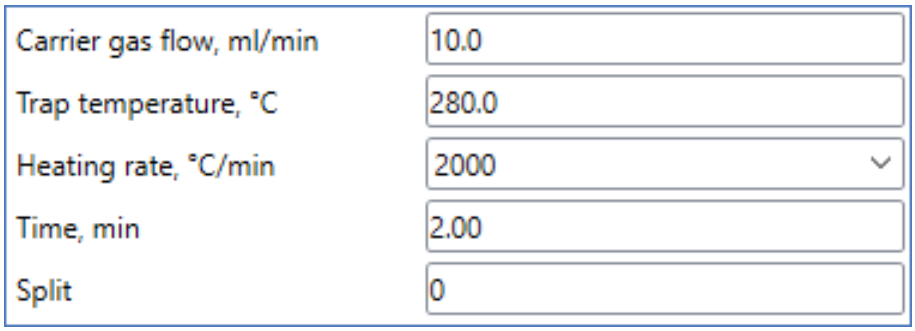
Cleaning time – The recommended trap cleaning time is 10 to 15 minutes. Value ranges from 0 to 15 minutes.

Trap test mode



Valve temperature - Heating of the valve is necessary to prevent condensation of components. The recommended valve temperature when analyzing volatile organic components is 200 to 250°C. The operating temperature range is 150 to 350 °C.

Transfer Line Temperature - The heated transfer line is designed to transfer the analyzed components from the trap to the chromatographic column. Typically, the transfer line temperature is set equal to the valve temperature or 10 °C higher. The operating temperature range is from 40 to 350°C.



Carrier gas flow – The recommended value is from 5 to 15 mL/min. The flow rate of the carrier gas must not exceed the flow rate of the split vent gas in the GC. Value ranges from 0 to 200 mL/min.

Trap temperature – The temperature of the trap depends on the sorbent type. Temperature ranges from 35 to 425 °C.

Heating rate – Select the values: 500; 1000; 1500; 2000; 2500; 3000 °C/min.

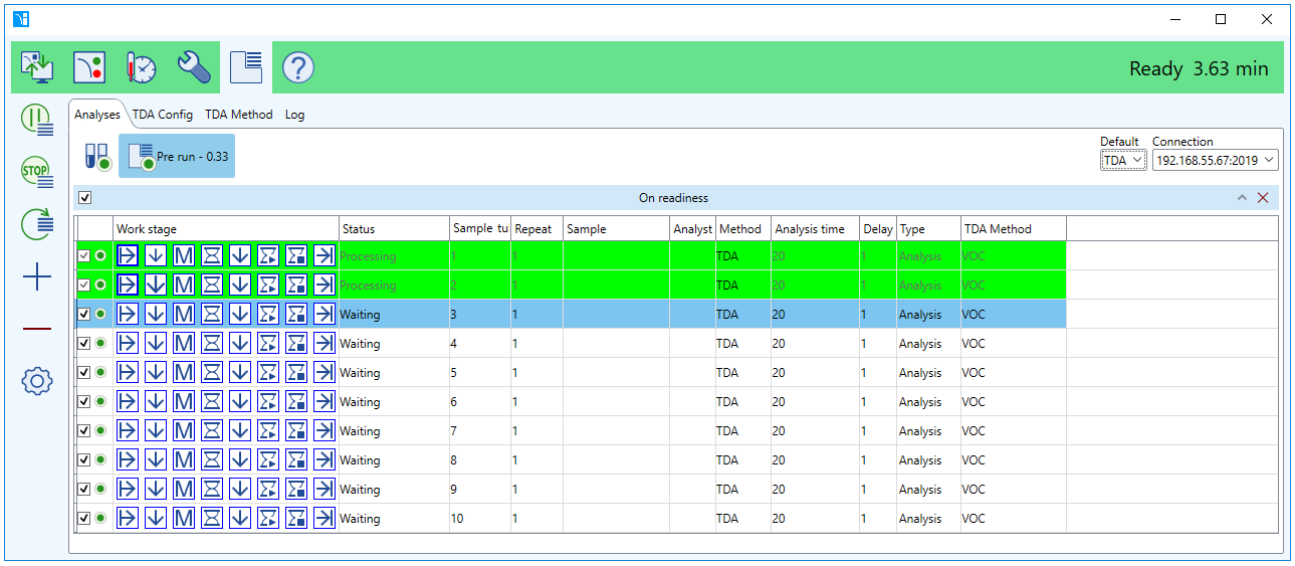
Analysis time – Sets the time during which the trap will heat up. Recommended value is 1 to 2 minutes. Value ranges from 0 to 15 minutes.

Split – It is used to send a part of the sample to the vent during the desorption of components from the trap. If split flow is 0, the sample is not divided, and full sample volume sent to the column. Value ranges from 0 to 100.

5.8.4 Sequence operation

Go to the **Analyses** tab.

Creating a Sequence



Add a new line in the list of analyses.

Fill out the following data:

- **Sample tube** – The number of sample tube position in the tray.
- **Re-collect tube** – The position of the backup tube in the tray (if applicable).
- **TDA Method** – Method (operating mode) of the thermal desorber.
- **Project** – Select the project to save chromatogram.
- **Method** – GC method.
- **Sample** – Sample name (to be saved in Sample info of a chromatogram).
- **Type** – Chromatogram type.
- **Analysis time** – If the TDA is connected to the Chromatec-Crystal GC, the analysis time is specified in the GC method. If TDA is used with third-party GCs, the analysis time is specified manually. This parameter enables overlapping tube desorption. Desorption is completed simultaneously when the GC is ready to maximize throughput.

Delay – Time to GC readiness after analysis. This is the time required to cool the column to the initial temperature. The cooling rate of the column depends on the ambient temperature. Therefore, the delay time should be set sufficiently long to include such possible variations in the cooling rate.

If necessary, fill out the fields **Notes**, **Analyst** etc.

In the same way you can add and fill out the lines for the next analyses.

Starting a sequence



Click **Start** on the sidebar.

Pause of sequence



Click **Pause** on the sidebar.

Pause of the sequence, while the current analysis will be completed until the end. The next analyses will not resume until you click **Start**.

Stopping a sequence



Click **Stop** on the sidebar.

If you interrupt the analysis sequence, the current analysis will be completed until the end, but other analyses will not start.

Stop running the current analysis



Go to the page **Status**.



Click **Stop** on the sidebar.

If the analysis was stopped during the Desorption stage and some components enter the trap, then the trap must be cleaned before starting the next analysis.

Adding a sample to a running sequence



Click the **+** icon in the sidebar. Fill in the line that appears, specify the tube number, method, etc.

Changing the order of analysis in the sequence



Hold down the left mouse button and drag the line to the required position.

To avoid accidental changes in the cell, drag the row by selecting the **Work stages** or **Status** cells.

Moving the active and following analysis is not possible.

Skipping the sample in the sequence

In the Activity column, uncheck the analysis you want to skip. After that, its status will change to Not Active.

Deleting a sample from the sequence



Left-click on the sample you want to delete.

Click the **–** icon in the sidebar. In the window that appears, confirm the operation.

Recycling a sequence



Click the **Recycle** icon in the sidebar.

The sequence will continue recycling indefinitely until the **Stop** icon is pressed.

Saving a sequence



Click **Settings** icon.



Select **Save sequence**. Enter the name and click **Save** button.

Opening a sequence



Click **Settings** icon.



Click **Open sequence**. Select the required file and click **Open** button.

5.9 Checkout

Checkout includes control of relative standard deviation (RSD) of GC signal (time and area).

Materials required

- Checkout mix – toluene in methanol (or acetone), concentration from 0.1 to 1.0 mg/mL
- Sorbent tube Tenax® TA 6.452.065
- Injection unit for thermal desorption tubes, P/N 400-1931
- Syringe 10 µL (e.g. SGE-Chromatec-02)
- Capillary column BPX-Volatile 60 m × 0.32 mm × 1.8 µm (alternatively you can use the columns with other phase, length and other parameters, if analytes separated).

5.9.1 Sorbent tube conditioning

Before starting the checkout, the sorbent tube must be conditioned.

Table 5.1 – Parameters for Tenax TA tube conditioning

Parameter	Value
Valve temperature	250 °C
Transfer line temperature	250 °C
Leak test	1 kPa (0.2 psi)
Tube temperature	300 °C
Purge gas flow	50 mL/min
Desorption time	20 min

5.9.2 Trap cleaning

The trap is cleaned according to the parameters below.

Table 5.2 – Parameters for Tenax TA trap cleaning

Parameter	Value
Valve temperature	250 °C
Transfer line temperature	250 °C
Leak test	1 kPa (0.2 psi)
Trap temperature	300 °C
Purge gas flow	30 mL/min
Heating time	15 min

5.9.3 Thermal desorber checkout

Set the following operating parameters of thermal desorber and the GC.

Table 5.3 – Thermal desorber operating parameters

Parameter	Value
Configuration	
Valve temperature	200 °C
Transfer line temperature	200 °C
Carrier gas	helium
Purge gas	helium
Leak test	1 kPa (0.2 psi)
Pre run	
Trap temperature (low)	minus 10 °C
Carrier gas flow	5 mL/min
Dry Purge	
Tube temperature at purge	0 °C
Purge gas flow	20 mL/min
Purge time	2 min
Desorption	
Tube temperature at desorption	270 °C
Purge gas flow	30 mL/min
Desorption time	5 min
Analysis	
Trap temperature (high)	270 °C
Trap heating rate	2000 °C/min
Heating time	2 min

Table 5.4 – GC operating mode (SSL inlet and FID detector)

Parameter	Value
Column oven temperature	150 °C
Inlet temperature	200 °C
Carrier gas pressure (helium)	120 kPa*
Split	1:20
FID temperature	250 °C
Hydrogen flow	25 mL/min
Air flow	250 mL/min

* The column oven temperature and carrier gas pressure values are referred to BPX-Volatile column 60 m × 0.32 mm × 1.8 µm. When using another column, the analysis parameters may be different and chosen in such a way as to ensure the separation of toluene from the solvent.

The analysis is performed in the following order:

- Connect sorbent tube to the injection unit (P/N 400-1931).
- Inject 1 µL of the checkout mix into the sorbent tube.
- Purge the sorbent tube with inert gas in the direction of sampling. The volume of the gas being purged depends on the solvent in the checkout mix (Table 6.7).
- Install the sorbent tube into the thermal desorber and run analysis.
- Repeat the sequence of 5 to 10 analyses for repeatability test.
- Calculate Relative standard deviation (RSD) in Chromatec Analytic software by area and retention time. The RSD value shouldn't exceed 2.0 %.

Typical chromatogram of checkout mix analysis is shown below (Figure 5.1).

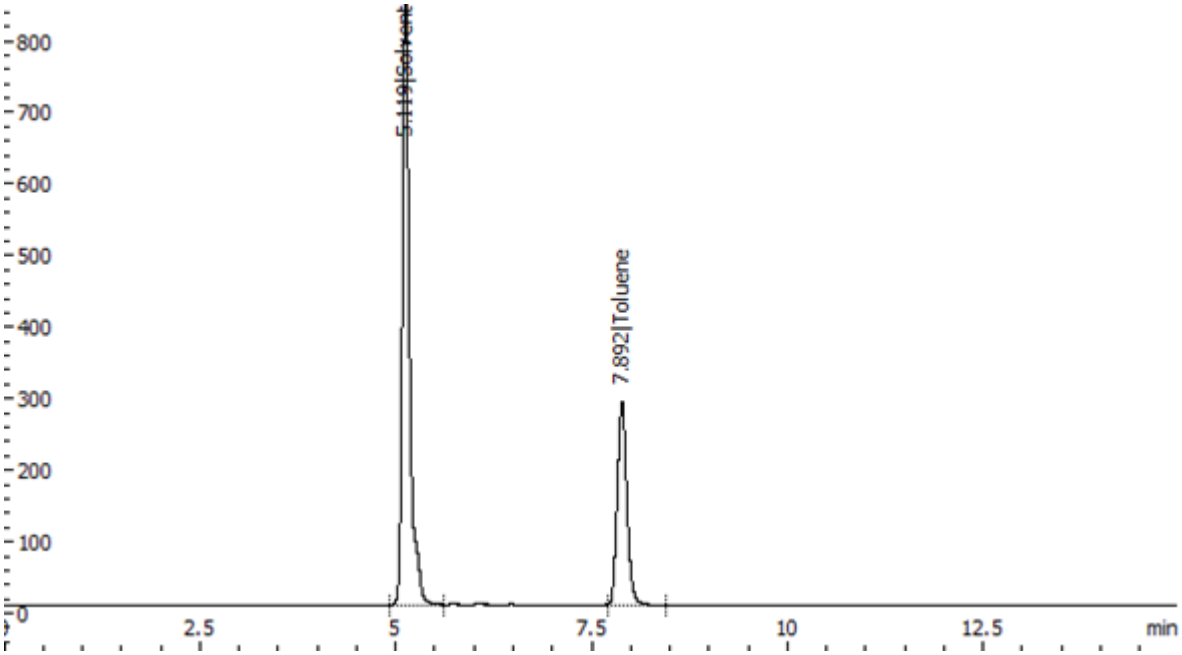


Figure 5.1 – Chromatogram of checkout mix

For checkout it is allowed to use other detectors, GC columns, set other operating parameters of the thermal desorber and the GC, and other measurement conditions stipulated by available and validated test method. In this case, the requirements for the repeatability test (control of RSD) are also stipulated by this test method procedure.

5.10 Turning off

Thermal desorber can be turned off without waiting for all components to cool down (valve, transfer line, tube oven).

- In the case of sequence running, press the **Stop** button and wait until the tube is transferred to the tray.
- On the rear panel of the thermal desorber switch the **Power** button to the off position.

6 Method development

6.1 Thermal desorption basics

The thermal desorption method is based on extracting volatile compounds from a sorbent at heating. Then these compounds are purged by inert gas flow and transferred to the GC.

The simplest version refers to a single stage thermal desorption (Figure 6.1). In this case compounds are moved directly from sample tube into chromatographic column. As sorbent content in tube is relatively large, it is impossible to ensure rapidly desorption of compounds. This results to of broadening peaks and deterioration of chromatographic separation.



Figure 6.1 - Single stage thermal desorption

To ensure a narrow band of analytes in chromatographic column, two stage thermal desorption is used. During two stage thermal desorption components refocused onto trap, from which they are transferred in a narrow band to a chromatographic column (Figure 6.2). Two focusing methods are available:

- cryofocusing in a capillary;
- cold trapping.

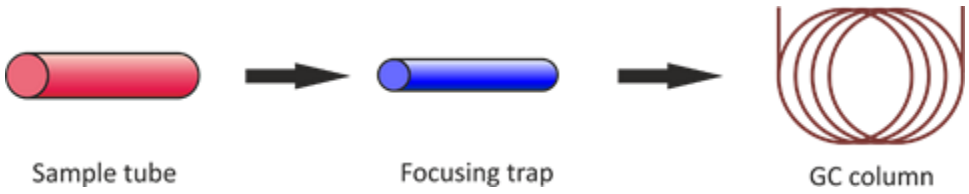


Figure 6.2 – Two stage thermal desorption

Cryofocusing occurs in quartz capillary with inner diameter between 0.18 and 0.53 mm being immersed into liquid nitrogen. Cryofocusing is a preferable method for collecting very light compounds. However, this method has certain disadvantages. At first, this method leads to significant consumption of liquid nitrogen. At second, moisture contained in the sample tube may lead to capillary clogging.

Alternative method to focusing is trapping in a cooled trap. The trap is packed with sorbent and cooled by Peltier elements. The sorbent allows concentrating even very volatile compounds without using liquid nitrogen. At the same time, lower sorbent volume allows to ensure rapid desorption and transferring sample into chromatographic column as a narrow band.

6.2 Thermal desorption applications

The main application of thermal desorption is analysis of volatile and semi-volatile organic compounds in ambient, workplace and indoor air. Also, it is used to test emissions of building materials and consumer products.

The thermal desorption also is used to analyze volatile organic compounds (VOC) in solid samples, for example, to determine residual solvents in pharmaceutical products, packing materials, monomers in polymers, odors and flavors in food products, etc.

The thermal desorption may be applied to analysis of different components that can be determined by gas chromatography. Range of analytes is limited by volatility and compounds with boiling temperature above 400 °C will be determined non-quantitatively. The thermal desorption cannot be used to analysis compounds with decomposition temperature below desorption temperature.

6.3 Method development objectives

The method development assumes the following objectives to be achieved:

- Sampling from an air to a sorbent without breakthrough.
- Completely desorption analytes from a sorbent tube.
- Completely transfer and collect analytes in a cold trap.
- Quantitatively transfer compounds to chromatographic column as narrow band as possible.
- Avoid influence of artifacts during sampling and analysis.

These objectives can be achieved by selecting a proper sorbent, sampling conditions and thermal desorber operation parameters.

6.4 Sorbent selection

During sampling sorbent should retain analytes while during desorption it should easily allow desorption. Depending on analytes, various sorbents can be used. See below recommendations for sorbent selection.

Depending on components sorption capability, sorbents are relatively divided to weak, medium and strong. Weak sorbents are well in retaining semi-volatile compounds, medium sorbents are suitable to volatile compounds, and strong sorbents are suitable to highly volatile compounds.

If sample represents a mixture of components with wide boiling temperature range, it is recommended to use tubes packed with several layers of different sorbents. For multi-bed tubes select sorbents having close desorption temperatures (difference should not exceed 50 °C). Otherwise, you cannot condition more heat-resistant sorbent without destruction that having less resistance to temperature.

If sample mixture is unknown, it is recommended to use multiple beds sorbent tubes packed with following sorbents: Carbopack C, Carbopack B and Carbosieve S-III. Such sample tubes can collect almost all compounds.

Table 6.1 – Sorbent selection recommendations

Sorbent	Range of analytes volatility	Maximum operating temperature, °C	Specific surface area, m^2/g	Analyte examples
Tenax® TA	boiling point 100 to 400 °C. n-C_6 to n-C_{26}	350	35	Aromatic non-polar compounds (with boiling point above 100 °C) and less volatile polar compounds (with boiling point above 150 °C)
Tenax® GR	boiling point 100 to 450 °C. n-C_7 to n-C_{30}	350	35	Alkylbenzenes, aromatic polycyclic hydrocarbons and polychlorinated biphenyl, also compounds specified for Tenax® TA
Porapak™ Q	boiling point 50 to 200 °C. n-C_5 to n-C_{12}	250	550	Different VOCs, including oxygen containing compounds
Porapak™ N	boiling point 50 to 150 °C. n-C_5 to n-C_8	180	300	Specific adsorbent for volatile nitriles: acrylonitrile, acetonitrile, propionitrile, also suitable for pyridine, volatile alcohols from ethanol, methyl ethyl ketone, etc.
Chromosorb™ 102	boiling point 50 to 200 °C	250	350	Different VOCs, including oxygen containing compounds and volatile halogenated compounds to methylene chloride
Chromosorb™ 106	boiling point 50 to 200 °C	250	750	Different VOCs, including hydrocarbons n-C_5 to n-C_{12} and oxygen containing compounds
Carbopack™ X	n-C_3 to n-C_5	400	240	Volatile hydrocarbons, aromatics
Carbopack™ B	n-C_5 to n-C_{14}	400	100	Different volatile organic compounds, including ketones, alcohols, aldehyde (with boiling point above 75 °C), all non-polar compounds within specified volatility range and perfluorocarbon tracer gases
Carbopack™ C	n-C_8 to n-C_{20}	400	12	Alkylbenzenes and aliphatic compounds
Carbosieve® S-III	boiling point - 60 to 80 °C. n-C_2 to n-C_5	400	800	Ultra volatile compounds from ethane, volatile halogen-containing compounds and freons

Sorbent	Range of analytes volatility	Maximum operating temperature, °C	Specific surface area, m^2/g	Analyte examples
Carboxen® 1000	boiling point - 60 to 80 °C. n-C_2 to n-C_4	400	1200	Ultra-volatile hydrocarbons, volatile halogen-containing compounds and freons

6.5 Packing sorbent tubes

The sorbent tubes with O.D. ¼ in (6.35 mm) and length 3.5 in (89 mm) are suitable for TDA. Sorbent tubes are made of glass, stainless steel or other inert material. For labile analytes, such as sulfur-containing compounds, use glass tubes.

Typically, tubes are packed with 200-1000 mg of a sorbent depending on its density, eg, approx. 250 mg for porous polymers (Tenax, Porapak) or 500 mg for carbon sorbents (Carbopack, Carbosieve). The recommended sorbent size is 60/80 mesh.

The length of sorbent layer should not exceed heating zone, but should be at least 14 mm away from either tube end to minimize inaccuracy resulted by diffusion at low pumping rate. To hold sorbent in a glass tube, glass wool or quartz wool is used. Length of glass wool layer should be approx. 5 mm. To hold sorbent in a stainless-steel tube, gauze, glass wool or quartz wool is used (Figure 6.3).

For analysis of labile components, you can secure sorbent layer in a tube with silanized glass wool. In this case, maximum tube temperature must not exceed 275 °C.

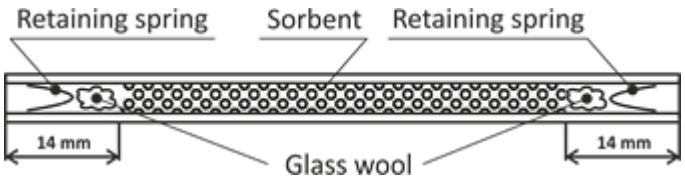


Figure 6.3 – Packing tube with sorbent

If the tube is packed with several sorbents, they should be located in ascending order regarding their strength and separated between each other with a glass wool. So, a sample will enter in direction from weak sorbent towards strong one in such a manner as semi-volatile compounds will be retained by weak sorbent while highly volatile components by strong one. The direction of sampling flow is marked by arrow. Desorption occurs in reverse direction and semi-volatile compounds do not contact with strong sorbent as it could lead to their irreversible sorption (Figure 6.4).

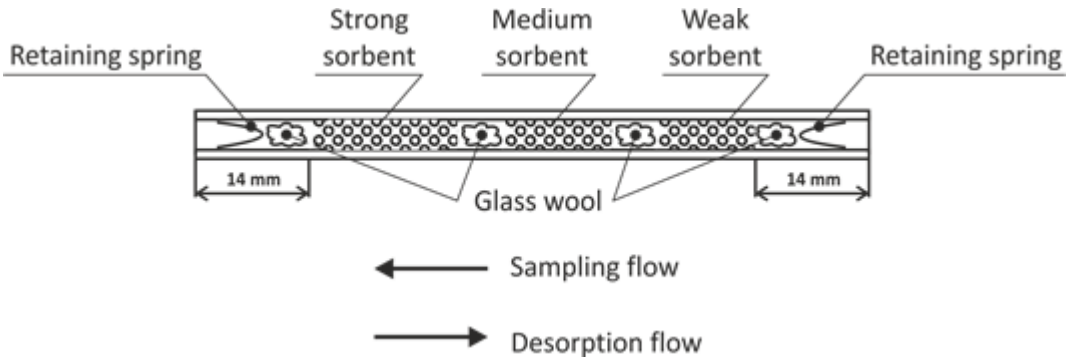


Figure 6.4 – Packing multibed tube with several sorbents

Examples of components and combinations for multibed tubes are listed in table below.

Table 6.2 – Multibed sample tubes

First layer	**Second layer**	**Third layer**
Tenax® TA	Carbopack™ B	–
Tenax® GR	Carbopack™ B	–
Tenax® TA	Carboxen® 1000	–
Tenax® TA	Carbosieve® S-III	–
Tenax® GR	Carboxen® 1000	–
Tenax® GR	Carbosieve® S-III	–
Carbopack™ B	Carbosieve® S-III	–
Carbopack™ B	Carboxen® 1000	–
Tenax® TA	Carbopack™ B	Carboxen® 1000
Tenax® TA	Carbopack™ B	Carbosieve® S-III
Tenax® GR	Carbopack™ B	Carboxen® 1000
Tenax® GR	Carbopack™ B	Carbosieve® S-III
Carbopack™ B	Carbosieve® S-III	Carboxen® 1000
Carbopack™ C	Carbopack™ B	Carboxen® 1000
Carbopack™ C	Carbopack™ B	Carbosieve® S-III

It is recommended to re-pack the tubes with a new sorbent each year or after 100 analyses.

6.6 Conditioning sorbent tubes

Sorbent tube conditioning is used to remove volatile contaminants from a sorbent. The conditioning may be carried out directly in the thermal desorber or in a desorber P/N 400-1903 (special device for conditioning sorbent tubes).

When proceeding with conditioning for the first time, it would be preferable to smoothly increase temperature to maximum in each three or four steps. For final conditioning, temperature should be set by 20-30 °C above desorption temperature. To ensure quick removal of contaminants, apply higher inert gas flow rate (100 ml/min for single-layer tubes or 50 ml/min for combination tubes). For a new tube, conditioning time depends on sorbent type and temperature, but anyway it should be not less than 1 hour.

For the next tube conditioning, you can set temperature by 10-20 °C above desorption temperature for a time between 15 and 30 minutes.

Conditioning temperature must not exceed maximum operating temperature of sorbent (Table 6.3).

For recommended maximum temperatures for tube conditioning depending on sorbent, see Table 6.1. For multiple beds tubes, conditioning temperature must be selected for a least thermally stable sorbent. For example, maximum conditioning temperature for tubes packed with Tenax GR, Carbopack B and Carbosieve S-III must be 330 °C.

Table 6.3 – Recommended maximum conditioning temperatures

Sorbent	**Maximum conditioning temperature, °C**
Tenax® TA	330
Tenax® GR	330
Porapak™ Q	250
Porapak™ N	180
Chromosorb™ 102	250
Chromosorb™ 106	250
Carbopack™ X	350
Carbopack™ B	350
Carbopack™ C	350
Carbosieve® S-III	350
Carboxen® 1000	350

For tube conditioning in the thermal desorber:

To ensure quick cleaning, apply purge gas flow rate (approx. 100 mL/min), for multibed tubes apply 50 mL/min.

Increase valve temperature higher than 250 °C to avoid condensation of contaminants.

After conditioning, check the tube by blank analysis. If any contaminant is revealed, repeat conditioning once more.

Pre-conditioned tubes should be stored being sealed with storage plugs or in a sealed container. See Section 6.12 for detailed description of sorbent tubes storage.

6.7 Packing traps

Typically, the trap is filled with the same sorbent as used in the sorbent tube. Since the trap concentrates substances at low temperature and a small volume of gas is passed through it, it can be filled with a weaker sorbent than the one used in the tube (Table 6.4).

Table 6.4 – Sorbent recommendation for packing traps

Tube sorbent	**Trap sorbent**
Tenax® TA	Tenax® TA
Tenax® GR	Tenax® GR
Porapak™ Q	Porapak™ Q or two-bed trap (Carbopack™ B & Carbosieve® S-III)
Porapak™ N	Porapak™ N or two-bed trap (Carbopack™ B & Carbosieve® S-III)
Chromosorb™ 102	Chromosorb™ 102 or two-bed trap (Carbopack™ B & Carbosieve® S-III)
Chromosorb™ 106	Chromosorb™ 106 or two-bed trap (Carbopack™ B & Carbosieve® S-III)
Carbopack™ X	Carbopack™ X
Carbopack™ B	Carbopack™ B or Tenax® GR or Tenax® TA
Carbopack™ C	Carbopack™ C or Tenax® TA
Carbosieve® S-III	Carbosieve® S-III or two-bed trap (Carbopack™ B & Carbosieve® S-III)
Carboxen® 1000	Carboxen® 1000 or two-bed trap (Carbopack™ B & Carboxen® 1000)
Tenax® TA / Carbopack™ B	Tenax® TA or two-bed trap (Tenax® TA & Carbopack™ B)
Tenax® TA / Carbosieve® S-III	Two-bed trap (Tenax® TA & Carbosieve® S-III)
Tenax® TA / Carboxen® 1000	Two-bed trap (Tenax® TA & Carboxen® 1000)
Tenax® GR / Carbopack™ B	Tenax® GR or two-bed trap (Tenax® GR & Carbopack™ B)
Tenax® GR / Carbosieve® S-III	Two-bed trap (Tenax® GR & Carbosieve® S-III)
Tenax® TA / Carboxen® 1000	Two-bed trap (Tenax® GR & Carboxen® 1000)
Carbopack™ B / Carbosieve® S-III	Two-bed trap (Carbopack™ B & Carbosieve® S-III)
Carbopack™ B / Carboxen® 1000	Two-bed trap (Carbopack™ B & Carboxen® 1000)
Tenax® TA / Carbopack™ B / Carbosieve® S-III	Two-bed trap (Tenax® TA & Carbosieve® S-III)

Tube sorbent	Trap sorbent
Tenax® TA / Carbopack™ B / Carboxen® 1000	Two-bed trap (Tenax® TA & Carboxen® 1000)
Tenax® GR / Carbopack™ B / Carbosieve® S-III	Two-bed trap (Tenax® GR & Carbosieve® S-III)
Tenax® GR / Carbopack™ B / Carboxen® 1000	Two-bed trap (Tenax® GR & Carboxen® 1000)
Carbopack™ B / Carbosieve® S-III / Carboxen® 1000	Two-bed trap (Carbopack™ B & Carbosieve® S-III)
Carbopack™ C / Carbopack™ B / Carbosieve® S-III	Two-bed trap (Carbopack™ C & Carbopack™ B)
Carbopack™ C / Carbopack™ B / Carboxen® 1000	Two-bed trap (Carbopack™ C & Carbopack™ B)

The trap is filled with a sorbent in the same way as a sorbent tube. The length of the sorbent layer should be 40 mm. When filling the trap with several sorbents, the weaker one should be located on the tapered side (Figure 6.5).

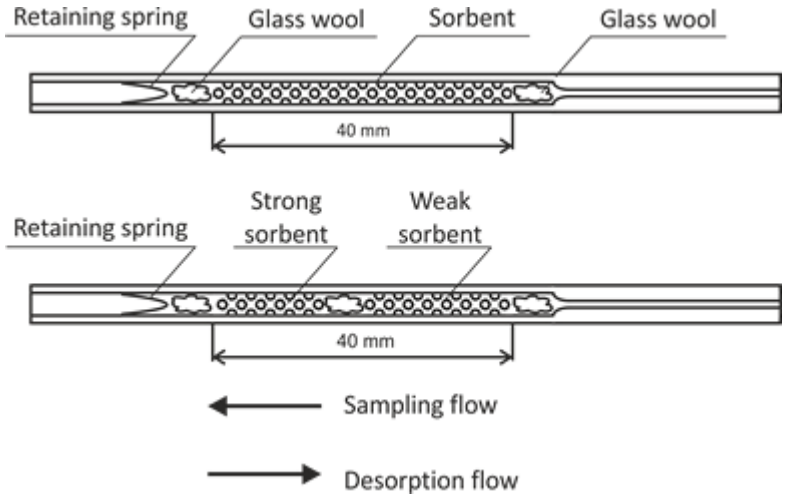


Figure 6.5 – Packing trap with sorbent

During the desorption of substances from the trap, the gas flow is directed in the opposite direction than at trapping. Different directions of the gas flow prevent contact of low volatile substances with a strong sorbent, which could lead to their irreversible sorption.

After filling the trap should be cleaned (Section 6.8).

6.8 Trap cleaning

Trap cleaning procedure is used to remove contaminants from it before analysis.

For cleaning the trap, an empty tube should be installed in the thermostat of sorbent tube.

Increase valve temperature higher than 250 °C to avoid condensation of contaminants.

Cleaning temperature is usually set at 10 to 20 °C above the upper temperature of the trap heating. However, it should not exceed the maximum operating temperature of the sorbent (Table 6.3).

The recommended purge gas flow while cleaning the trap is 30-50 mL/min.

Usually, it takes about 10 to 15 minutes to clean the trap.

6.9 Trap test

Trap test is used when you need to check that the trap has no contaminants after cleaning.

Prior to trap test install an empty tube into sorbent tube thermostat.

Operating method for trap test generally is the same as working operating method.

6.10 Sampling

Sampling should be carried out into pre-conditioned sorbent tubes. During storage for a long time, sorbent tubes can sorb organic contaminants from ambient air, even if they were stored plugged in a sealed container.

The following sampling methods can be used for air analysis:

- active;
- passive.

Active sampling

Sampling is carried out by pumping air through a sorbent tube using a sampling pump at a constant flow rate for a certain time.

The sampling pump should provide a maximum air flow rate of 200 mL/min at backpressure of 5 kPa.

The sorbent tubes are connected to the sampling pump using fittings 8.652.506 or 8.652.507, depending on the model of the aspirator (Figure 6.6). The arrow on the tube indicates the direction of sampling, and the labeled end of the metal tubes indicates the sample inlet. Thus, when using multibed tubes, the sample must first pass through a weak sorbent, and then through a strong sorbent.

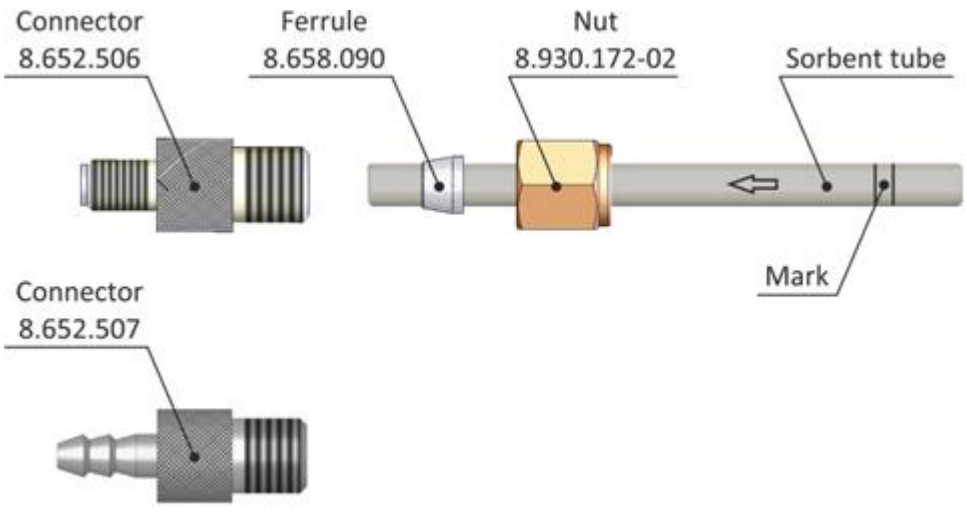


Figure 6.6 – Connection of sorbent tube to a sampling pump

To connect several sorbent tubes in series, use fitting 8.652.613 (Figure 6.7).

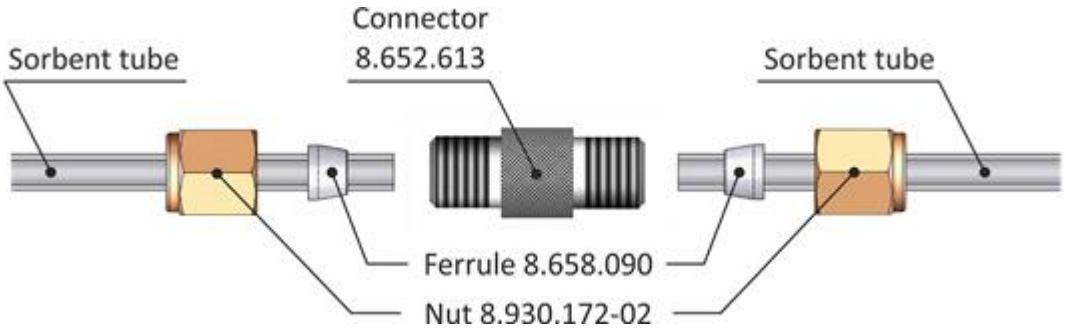


Figure 6.7 – Series connection of sorbent tubes

The recommended air flow rate through the sample tube is between 50 and 200 mL/min. The specified flow rate range is limited by the following criteria: at a lower flow rate, diffusion of analytes leads to a significant error while at a higher flow rate the analytes could be break through the tube.

Volume of air to be pumped through the tube depends on a specific analysis method. Minimum sample volume depends on detector sensitivity while maximum depends on adsorption capability.

The Table illustrates estimated masses of analytes and retained by adsorbent depending on sample type and pumped air volume (Table 6.5).

Table 6.5 – Mass of analytes retained by adsorbent depending on sample type

Sample type	**Expected concentration, µg/m3**	**Mass of analytes, ng, for a air volume**		
		1 L	2 L	10 L
Polluted urban area	10 – 1000	10 – 1000	20 – 2000	100 – 10000
Indoor environment	5 – 500	5 – 500	10 – 1000	50 – 5000
Normal urban area	5 – 50	5 – 50	10 – 100	50 – 500
Normal rural area	0.5 – 5	0.5 – 5	1 – 10	5 – 50
Forest	1 – 10	1 – 10	2 – 20	10 – 100
Cleaned air	0.1 – 20	0.1 – 20	0.2 – 40	1 – 200

Adsorption capability describes breakthrough volume and retention volume.

Breakthrough volume – volume of test atmosphere that can be passed through a sorbent tube before the concentration of eluting vapor reaches 5 % of the concentration in the initial mixture.

The breakthrough volume can be determined using calibration gas mixtures with concentrations between 1 and 10 mg/m^3. In this case you need a system comprising of calibration gas mixture source, needle valve, flow meter, sorbent tube and FID or another detector (Figure 6.8).

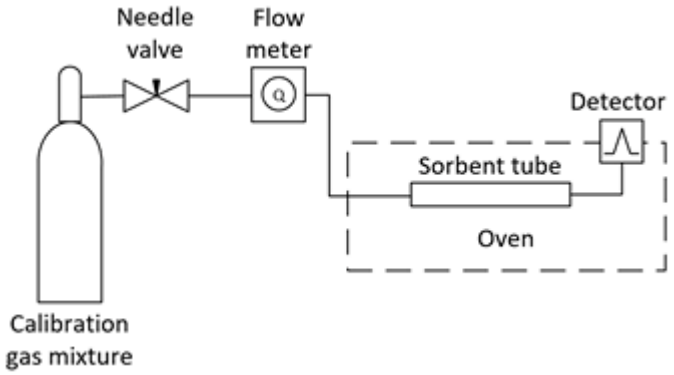


Figure 6.8 – Diagram for breakthrough volume measuring system

Calibration gas mixture passes through a sorbent tube with a constant flow rate between 20 and 200 mL/min. Flow rate should be selected in this range appropriate for the sample rate intended. Note the time when gas initiate entering the system. After passing through the sorbent tube, analytes goes to the detector and a signal is generated. Measurements should be continued until plateau appears on the chromatogram. Determine time that was need to get 5 % of maximum signal.

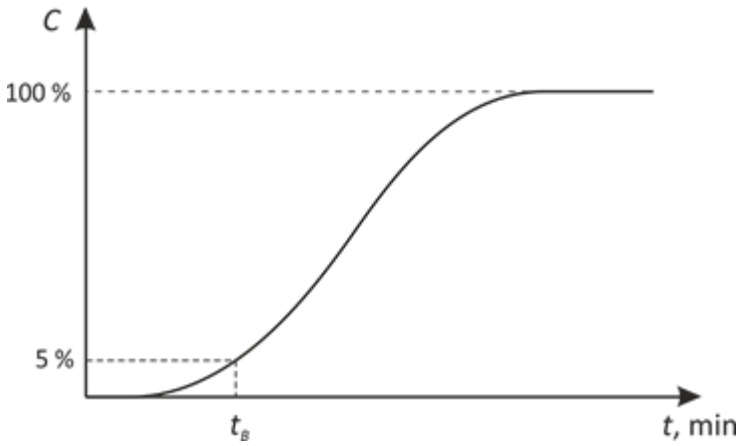


Figure 6.9 – Determination of breakthrough volume

Calculate breakthrough volume V_B (L) according to the following formula:

$$V_B = Ft_B$$

where F – gas flow rate, L/min; t_B – time needed to get 5 % of maximum signal, min.

Typically, dead volume of sampling line is significantly less than breakthrough volume. If this is not the case, dead volume can be determined by repeated measurement using an empty sample tube. In this case breakthrough volume result needs to be corrected.

Retention volume – gas volume needed to get a maximum peak during elution of compound from the sample tube.

Diagram of system used to determine a retention volume is shown below (Figure 6.10).

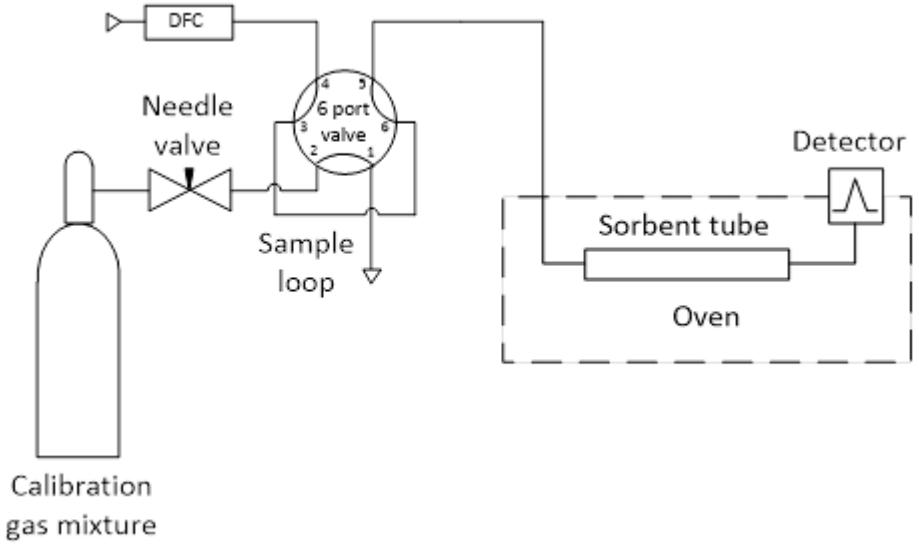


Figure 6.10 – Diagram for retention volume measuring system

The sorbent tube is connected between the sampling valve and the detector. The gas flow regulator ensures a constant carrier gas flow through the tube. To ensure quick analysis, the sorbent tube

should be placed into the oven. Using sampling valve, inject a certain volume of calibration gas mixture into the sample tube and determine time needed to get a maximum peak (Figure 6.11). Measurements should be carried out at different temperatures in such a manner as to get retention time between 2 and 20 minutes.

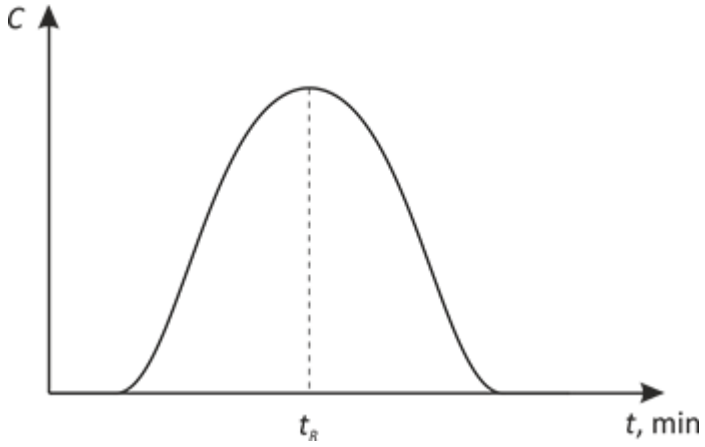


Figure 6.11 – Determination of retention volume

Calculate retention volume V_R (L) according to the following formula:

$$V_R = Ft_R$$

where F – carrier gas flow rate, L/min; t_R – retention time, min.

To get a retention volume for at room temperature, you need to draw a graph showing retention volume vs. temperature and extrapolate it to 20 °C (Figure 6.12).

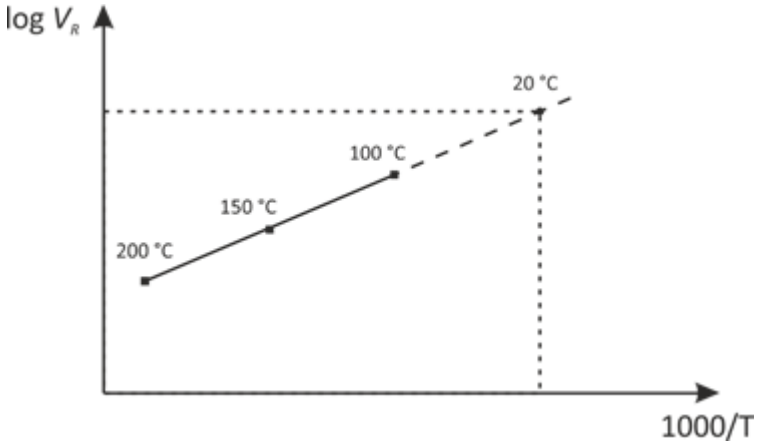


Figure 6.12 – Extrapolation of retention volume to room temperature

The breakthrough volume and the retention volume are unique for each sorbate-sorbent pair and depends on ambient temperature. Typically, the breakthrough volume and the retention volume decrease by 2 times when temperature increases by 10 °C. This should be considered for warm air sampling.

To have a certain margin in order to ensure reliable measurement, "Safe sampling volume" is used. This term refers to a recommended maximum volume of sample. The safe sampling volume must not exceed 70 % of breakthrough volume or 50 % of retention volume. The safe sampling volume is determined in liters (L) or liters of pumped air per gram of adsorbent (L/g). Safe sampling volume values for different organic compounds and certain adsorbents can be found in literature[1]. Before using reference values, it is recommended to check them.

[1]ISO 16000-6:2011 Indoor air – Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS or MS-FID

ASTM Method D 6196-15 Standard Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air

MHDS 72 UK Health and Safety Executive. Method for the Determination of Hazardous Substances. Volatile organic compounds in air – Laboratory method using pumped solid sorbent tubes, thermal desorption and gas chromatography

Certain adsorbents, for example Carbosieve® S-III or Carboxen® 1000, also may hold a certain amount of moisture. Therefore, when sampling in high relative humidity conditions (> 90 %), decrease sampling volume by 10 times.

6.11 Analysis of solid samples

The thermal desorption method can be used to analyze solid samples, for example, medical products, polymers, food products etc.

Complete extraction of volatile components from solid samples can be achieved for highly homogeneous systems only. In all other cases, the thermal desorption method will only give information about composition that can be used, for example, to compare several samples or get product profile (fingerprint).

The sample is placed into a central part of the sample tube and fixed with two glass wool plugs. At avoid moving glass wool plugs it should be fixed with retaining springs (Figure 6.13).

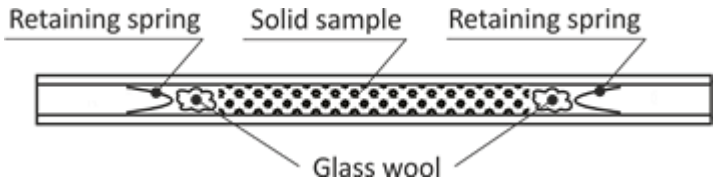


Figure 6.13- Solid sample in tube

When proceeding with analysis of solid samples containing highly boiling compounds (e.g., resins), place into tube a little amount of sample. This will help to avoid contamination of the system. Before placing the solid sample, you can place a small adsorbent bed that will pass volatile analytes but trap compounds such as resins or fats (Figure 6.14).

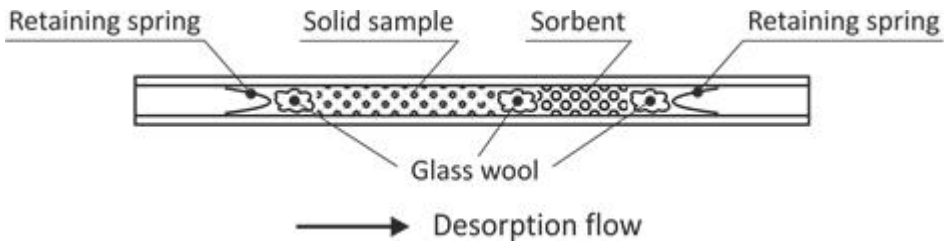


Figure 6.14 – Placing a sorbent after the soil sample

If you expose for thermal desorption soil samples or the samples containing a moisture, use hydrophobic sorbent, like Tenax TA.

6.12 Sample tube storage

For storage keep the sorbent tubes tightly sealed with caps assembly 6.433.091 (Figure 6.15).

Install the cap assemply 6.433.091 as follows:

- Place the ferrule 8.658.090 into plug 8.632.092.
- Screw the nut 8.930.172-02 on the plug 8.632.092, but do not tighten it.
- Insert the sorbent tube into the nut 8.930.172-02 up to the end.
- Tighten the nut 8.930.172-02 by hand until the tube is retained by the nut (not able to move freely).

Do not use wrenches to swage the nut at tube sealing. It may damage the sorbent tube.

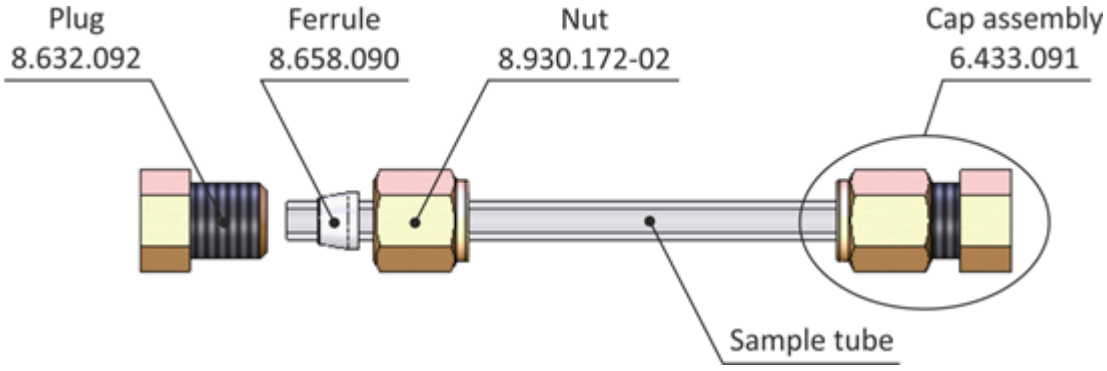


Figure 6.15 – Installation of cap on sorbent tube

Alternatively, a container 6.087.025 can be used for sample tubes storage instead of caps (Figure 6.16).

Place a sample tube into container 6.087.025 as follows:

- Unscrew a nut 6.432.056.
- Insert the sample tube into container.
- Screw the nut 6.432.056 by hand.

Do not use wrenches to swage the nut at tube sealing. It may damage the sorbent tube.

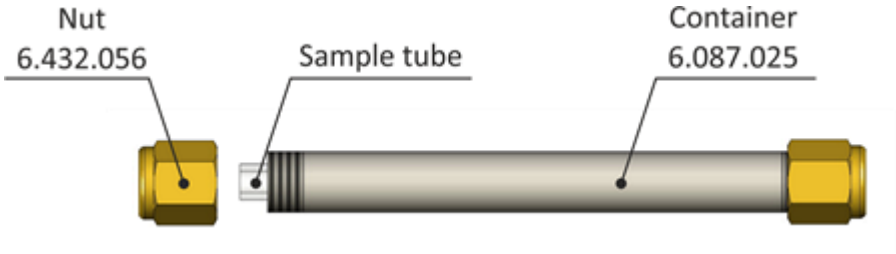


Figure 6.16 – Placing the sample tube into the container

If analysis will not be carried out within 8 hours after sampling, store sample tubes in clean sealed container made of glass or metal. To protect sample tube against pollution, place into container a clean white cotton bag packed with activated carbon.

The container with tubes may be stored in a refrigerator at temperature between 4 and 6 °C if there are no organic compounds in a refrigerator.

In case of temperature change, re-tighten caps of the sample tubes to ensure that they are sealed.

During long term storage of multiple beds tubes, semi-volatile compounds may migrate from the weak sorbent to the strong one. Therefore, during analysis this could lead to incomplete desorption of semi-volatile compounds.

6.13 Thermal desorber operation parameters

This section contains guidance on setting operation parameters of the thermal desorber. Parameter selection should be carried out in accordance with the method of analysis used.

6.13.1 Desorption temperature

Desorption temperature should be selected in such a manner as to ensure complete extraction of components from the adsorbent. On the one hand, the temperature should be as high as possible, and it is limited of adsorbent operating temperature (Table 6.1). However, when temperature is close to maximum operating temperature of the adsorbent, it begins to decompose, and different contaminants appear as a result. Therefore, it is recommended to avoid setting too high desorption temperature.

The recommended maximum desorption temperatures for different adsorbents are shown below (Table 6.6).

For desorption of compounds from sample tubes packed with different adsorbents, select desorption temperature considering the adsorbent with minimum operating temperature.

When choosing desorption temperature, also consider possible decomposition of analytes at that temperature.

Table 6.6 – Recommended maximum desorption temperatures

Sorbent	Maximum desorption temperature, °C
Tenax® TA	300
Tenax® GR	300
Porapak™ Q	225
Porapak™ N	180
Chromosorb™ 102	225
Chromosorb™ 106	225
Carbopack™ B	325
Carbopack™ C	325
Carbosieve® S-III	325
Carboxen® 1000	325

To check that compounds were desorbed from the adsorbent completely (desorption efficiency) carry out a subsequent analysis of the sample tube. After repeated desorption peak responses should not exceed level specified in the analysis standard. Another may to check a desorption efficiency is comparison of peak responses during a thermal desorption and a direct injection into the GC injector.

If components cannot be completely desorbed at maximum operating temperature of the adsorbent, increase desorption time or use another adsorbent which is weaker or has a higher operating temperature.

6.13.2 Purge gas flow rate

The purge gas flow rate should be selected that compounds are completely desorbed from the sample tube within a minimum time. On the other hand, too high purge gas flow rate may lead to breakthrough of compounds through the trap. So, optimum purge gas flow rate is between 20 and 50 mL/min.

6.13.3 Desorption time

The desorption time should be enough for the complete desorption of components. Depending on the desorption temperature and the purge gas flow rate, the desorption time may be selected between 5 and 10 minutes. It is necessary to set the minimum time to improve the productivity of the thermal desorber.

6.13.4 Trap low temperature

In most cases, to trap volatile organic compounds set the temperature of the trap minus 10 °C. When analyzing very humid samples, it is possible that the trap may be blocked with ice. In this case, the measured purge gas flow rate will be 0 mL/min. To prevent the trap from ice clogging when analyzing samples containing a significant amount of water, it is recommended to set the trap temperature above 4 °C.

The trap low temperature should be selected in such a manner as to prevent breakthrough analytes. You can check whether components could break through the trap in the following way. Inject a standard mixture directly into the GC injector and through the trap in the thermal desorber. If response during direct analysis exceeds that during analysis in the thermal desorber, this means that sample cannot be completely focus in the trap. In this case it is necessary to decrease the trap temperature. If decrease temperature down to –20 °C cannot solve the problem, use a stronger adsorbent in the trap.

6.13.5 Trap high temperature

The trap high temperature should be selected in such a manner as to ensure complete desorption. You can check desorption of compounds from trap in the same way as for the sample tube. If peak responses during next desorption are not negligible comparing to responses obtained during analysis, then increase the trap high temperature

The trap high temperature must not exceed maximum operating temperature of the sorbent (Table 6.1).

6.13.6 Heating rate

Heating rate should ensure rapid desorption of components from the sorbent and their transfer into a GC column. For analysis of volatile organic compounds is set higher heating rate.

For analysis of thermally labile analytes which may decompose at rapid temperature increase is set lower heating rate.

6.13.7 Trap heating time

Typically, for complete desorption of components from the trap needs 1-2 minutes. If this time is insufficient, for example, in case of low heating rate, increase heating time in such a way that during next analysis chromatogram shows no peaks remained from previous analysis.

6.13.8 Dry purge

The dry purge step prior to desorption is used to blow air from a sample tube. To remove air, the purge gas flow rate is set at 20 mL/min, the purge time is 1 min (for polymeric or carbon sorbents) or 2 min (for molecular sieves).

Dry purge step can also be used to remove moisture from the sorbent tube, which was concentrated during the sampling with high humidity. Moisture removal most effectively occurs at a temperature above room temperature; therefore, the purge temperature is usually set at 40–50 °C, and the flow rate of the purge gas is 20–30 mL/min. The time for blowing moisture depends on its quantity, as well as on the type of sorbent in the tube. For example, for Tenax and Carbopack sorbents, usually 1-2 minutes is enough, for Carbosieve sorbents 3-5 minutes required, Carboxen sorbents are hydrophilic and removing moisture from them without loss of the analyzed substances is difficult.

6.13.9 Valve temperature

Valve heating is needed to prevent condensation of analytes. The valve temperature depends on analytes and typically is set between 200 and 250 °C.

6.13.10 Transfer line temperature

The transfer line temperature should be selected as to prevent condensation of analytes. To get best repeatable results, the transfer line temperature should be set by 5-10 °C above valve temperature.

6.13.11 Carrier gas flow rate

To transfer sample from the thermal desorber to the GC, carrier gas flow rate should be set between 5 and 15 mL/min. For capillary columns with inner diameter less 0.32 mm and carrier gas flow rate through the column between 0.5 and 2 mL/min should be set minimum split in the injector (between 1:1 and 1:10)

6.13.12 Split

If Re-collection option is installed, TDA allows to split the sample flow at desorption from the focusing trap. The discharged sample is to be concentrated on the sorbent tube and sent for disposal.

Split can be applied when sample has a high concentration of the analytes.

When working with Split in the Analysis stage, a tube filled with activated carbon is to be installed in the thermostat of the sample tube.

Split rate should be set so that the vent gas flow didn’t exceed 500 mL/min.

6.13.13 Re-collection

This option is used to collect part of the sample during desorption from the trap at split of the sample. The current or new tube is used as a re-collect tube. If necessary, the re-collect tube can be analyzed again.

When working with a backup tube, the flow split is usually set to 1:1, so half of the sample is sent to the chromatograph for analysis, and the other half is transferred to the backup tube.

6.13.14 Leak test

The leak test value should be set in such a manner as to ensure that pressure drop in the sample tube during the leak test before desorption step will be checked. This value depends on adsorbent and packing density of the sample tube.

6.14 Calibration

The calibration must be as close as possible to real analysis conditions. To get accurate results, the standards must be injected in the sample tube and completely repeat all analysis steps.

Generally, calibration is carried out using standard gas mixtures or liquid mixtures.

6.14.1 Calibration with standard gas mixtures

Calibration gas mixtures should be prepared as specified in standards ISO 6141 and ISO 6145. Depending on component subject to analysis (workplace air, indoor air, ambient air etc.), concentration of analytes in calibration mixtures may be between 100 µg/m^3 and 10 mg/m^3. However, it should correspond to the expected average concentration in the samples.

For calibration, sorbent tubes should be prepared by sampling through them a known volume of standard gas mixture, for example 100 mL, 200 mL, 400 mL, 1L, 2 L, 4 L or 10 L. For sorbent tube connection diagram. The mixture volume must not exceed the breakthrough volume (Figure 6.17).

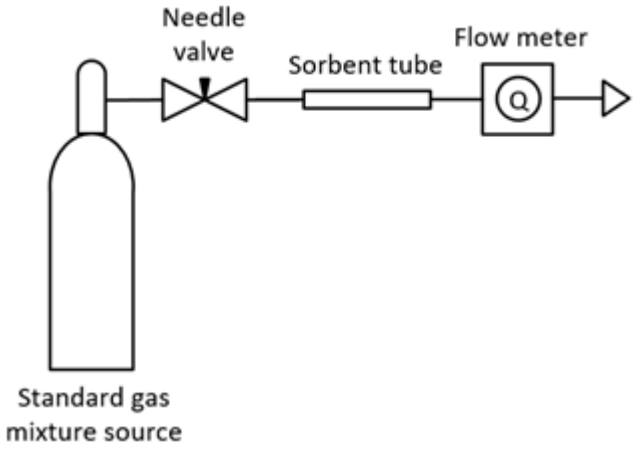


Figure 6.17 – Connection the sorbent tube to the standard gas mixture source

If concentration in the standard gas mixture significantly exceeds expected concentrations in the sample, you can use the sampling valve with the loop to inject into the sample tube gas mixture volume from 0.5 to 10 mL. The sample must be carried from the loop into the sample tube by inert

gas volume of which must exceed the loop volume more than 20 times. The inert gas supply may be provided using the restrictor 5.150.015-02, which provides flow rate approx. 20 mL/min at the pressure 400 kPa. For principal diagram of device used to carry sample to the sorbent tube using the sampling valve (Figure 6.18).

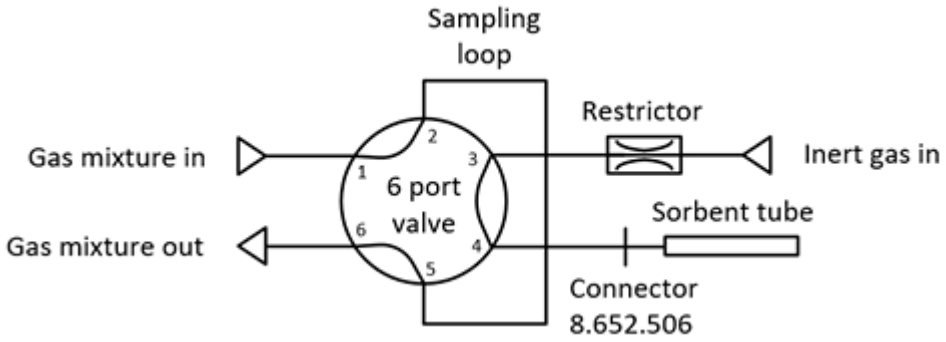


Figure 6.18 – Injection gas mixture in the sorbent tube

6.14.2 Calibration with liquid mixtures

For calibration with liquid mixtures, consider that RSD will be higher than for calibration with gas mixtures as injection sample with syringe adds a significant error.

Before calibration calculate concentration in the liquid calibration mixture. Mass of analytes must correspond to mass during sampling.

Here is the example of the calculation of compounds concentration in the calibration mixture.

- Expected concentration in air is 10 µg/m^3.
- During sampling, 1 liter air is pumped.
- 1 liter air contains 10 ng of the compound.
- During calibration, 1 µL of liquid mixture will be injected to the sample tube.
- In the calibration mixture concentration should be 10 ng/µL or 10 µg/mL.

At passive sampling, the concentration of the substance in the calibration liquid mix is calculated as follows:

If the rate of diffusion absorption and exposure time are known, calculate the mass of the substance *m* (µg) by the formula:

$$m = \frac{C q_V t}{10^6}$$

where *C* – expected concentration of the substance in analyzed air, µg/m^3;

q_v – diffusion absorption rate, cc/min (mL/min);

t – exposure time, min.

For example, for heptane, the rate of diffusion absorption on a tube filled with Tenax TA, under standard conditions (temperature 25 °C, pressure 101.3 kPa) is 0.43 CC/min

If the expected concentration of heptane in the air is 10 μg/m^3, and the exposure time is 8 hours (480 min), then the mass of the captured heptane will be 2 ng.

Suppose, when calibrating, 1 μL of the liquid mixture will be injected into the tube, so it is necessary to prepare a calibration mix with a concentration of 2 ng/μL (2 μg/mL).

To prepare liquid calibration mixtures, use high purity grade solvents (e.g. GC, ACS reagent or for analysis), that must be checked to ensure that there are no contaminants. Before selecting a solvent, ensure that it will not elute together with analytes. It is recommended to use more volatile solvent than analytes. In this case, before analysis it can be purge from the sorbent tube. The most preferable solvent is methanol.

Volume of inert gas for purging solvents out from a sorbent tube Tenax TA is shown in Table 6.7.

Table 6.7 – Volume of inert gas for purging solvents

Solvent	**Gas volume, L**
Acetone	1,2
Dichloromethane	0,9
1,1-Dichloroethylene	0,9
Methanol	0,1
n-Pentane	1,0
Ethanol	0,4

A calibration with liquid mixtures can be carried out in two ways: injection solution via injector or injection solution directly into the sorbent tube.

Injecting a calibration solution via GC inlet

This method of sample injection can be used for the calibration of components with a boiling point of not more than 200 °C.

Connect the sorbent tube with its sampling side (no tag on his side) to the GC inlet, and set inert gas through the inlet at a rate of 100 mL/min. If a capillary inlet is used, the split vent should be plugged or set 0 mL/min (Figure 6.19). Set temperature of the inlet at a value, slightly below than boiling point of the solvent. Using a syringe, inject a calibration solution in volume of 1 to 5 μL through inlet septa. Hold some predetermined time to blow out the solvent, then disconnect the tube and seal it tightly.

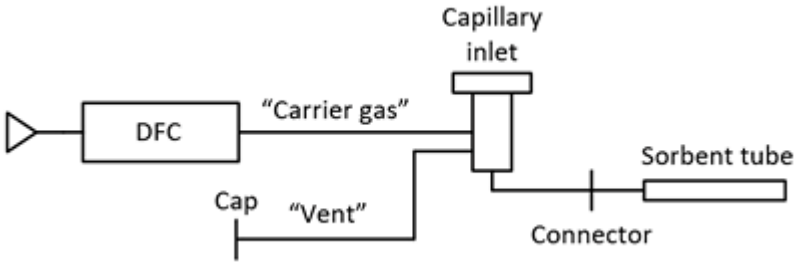


Figure 6.19 – Connection of the tube to the inlet for calibration

Injecting a calibration solution directly into the sorbent tube

Using a syringe, inject 1 - 5 µL of calibration mixture into the sorbent tube from the sampling side (no tag from this side). The liquid solution must be injected into the glass wool.

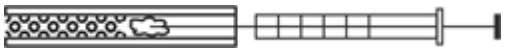


Figure 6.20 – Injecting a calibration mixture directly into the tube

After injecting the calibration solution, purge pure inert gas through the tube, so that during subsequent desorption the components pass through the sorbent and simulate the real conditions of the analysis as much as possible. The volume of the purged gas is determined by test method, it should not exceed the breakthrough volume of the analyzed compounds.

For injecting liquid mixtures into the sorbent tube and subsequent removing a solvent, Injection unit for thermal desorption tubes (P/N 400-1920) can be used.

If there is no available the Injection unit (P/N 400-1920), you can arrange an inert gas line (helium or nitrogen) to purge the tube after the calibration solution is injected. To do this, a tee and a pneumatic restrictor 5.150.015-02 are installed in the inert gas line before entering the GC. Use the fitting 8.652.506 to connect line to the sorbent tube. When the calibration is not carried out, the line can be closed with a plug (Figure 6.21).

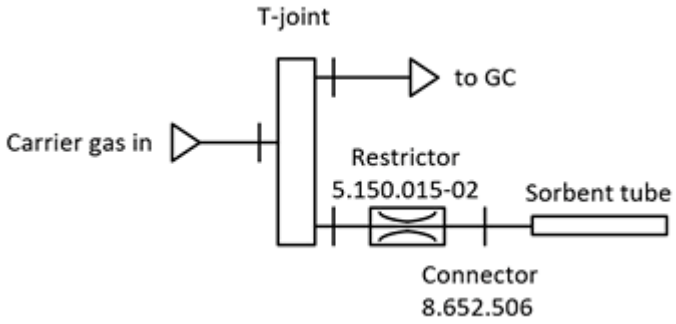


Figure 6.21 – Diagram for purging the tube with inert gas

6.15 General recommendations for analysis performance

For sample tube marking avoid using paints and markers containing solvents. Also, avoid using stickers.

New caps and containers for the storage of sorbent tubes may sorb organic impurities during transportation. Before first use, it is recommended to warm containers and plugs at 120 °C for 2 hours.

If not used, caps and containers for the storage of sample tubes should be stored in a clean sealed container (for example, in a desiccator). During long-term storage, caps or containers for sample tubes may sorb organic pollutants from ambient air. Also, after using caps and containers with sample tubes, there may rest a small number of impurities on the surface. For cleaning, place caps

in an ultrasonic bath with isopropanol for 10 to 15 minutes. After warming, thoroughly wash the caps with distilled water and dry at 60 °C for 20 minutes to remove residual solvent.

Before analysis, it is recommended to make two blank analyses: with laboratory blank tube and analysis blank tube. Laboratory blank tubes should be prepared and stored with sampling tubes. This analysis eliminates errors associated with conditioning tubes and their storage in the laboratory. Also, they can be used for monitoring adsorbent stability. Blank tubes analysis should be prepared and transferred to a sampling place together with sampling tubes. By sampling caps are removed from sorbent tubes and immediately put back. This procedure eliminates the errors associated with sample storage and transportation.

7 Maintenance

7.1 Maintenance types

The thermal desorber requires two types of maintenance:

- Routine maintenance.
- Periodic maintenance.

Current maintenance may be carried out by customer.

Periodic maintenance may be carried out by customer or service engineer.

The recommended time interval for both maintenance types should be considered based on 10–20 analyses per day.

Time intervals of maintenance may vary depending on:

- Intensity of thermal desorber usage.
- Working temperatures of the valve, the trap, the tube oven.
- Types of samples.

Maintenance procedures also should be carried out when at troubleshooting.

7.2 Routine maintenance

For routine maintenance procedures see table below.

Table 7.1 – Routine maintenance procedures

Procedure	**Interval**
Cleaning of thermal desorber external surfaces	Daily
Decapper cleaning	Monthly
Checking cleanness of transfer line needle hole	When transfer line is connected to the inlet

7.2.1 Cleaning of thermal desorber surfaces

Materials required

- Lint-free tissue
- Water

The external surfaces of the thermal desorber are wiped with a soft lint-free tissue moistened with water. It is allowed to add a small amount of detergent to the water.

Do not use abrasives or solvents to clean the external surfaces of the thermal desorber.

7.2.2 Decapper cleaning

Materials required

- Cotton applicators
- Solvent (Isopropanol)

Turn off the thermal desorber before cleaning the decapper.

Over time, dust and other small mechanical particles accumulate in the slots of the caps. Cleaning the slots from dust is carried out with cotton applicators moistened with a solvent (isopropanol).

Dust removal from the aluminum base, on which the cap removal elements are located, is carried out with a vacuum cleaner with a micro nozzle or a cotton applicator moistened with a solvent (isopropanol).

Do not use detergents or abrasive materials to clean the decapper, as they may damage the moving parts when leaked inside.

Do not use aggressive solvents (acetone, dichloromethane, etc.), because they can damage plastic parts.

7.2.3 Checking cleanness of transfer line needle hole

Cleanness of transfer line needle hole should be examined visually when the needle is installed into GC inlet. Possible dirt, for example pieces of rubber septum, can cause of carryover effect.

Procedures:

- When rubber residuals are revealed in the needle hole, accurately remove them with a needle with sharp end. Alternatively, you can use a thin wire.
- When rubber residuals entered inside the needle, remove it, accurately clean with thin wire, blow by inert gas and re-install as specified in Section 7.3.7.

7.3 Periodic maintenance

Periodic maintenance procedures are listed below.

Table 7.2 – Periodic maintenance procedures

Procedure	Interval
Inlet septum replacement	If leak appeared
Gas lines leak testing	Yearly or if leak appeared
Packing a sorbent tube	Yearly or after 100 run cycles
Replacing a sorbent tube	After 500 run cycles, or at troubleshooting
Replacing a vent trap	Yearly or after 1000 run cycles, as well as at troubleshooting

Replacing sealing O-rings of the trap	Yearly or if leak appeared
Replacing sealing O-rings of the sorbent tube	After 1000 run cycles or if leak appeared
Vent trap replacement	Yearly
Replacing the transfer line needle	On demand at troubleshooting
Replacing fuses	On demand at troubleshooting

7.3.1 Inlet septum replacement

Inlet septum is worn out at installation of transfer line in the injector, so if leaks appeared, the septum should be replaced.



Before septum replacing cool the inlet to avoid getting burned.

Procedures:

- Take out transfer line needle from the inlet.
- Unscrew a nut 8.936.107.
- Replace a septum.
- Screw the nut 8.936.107 back.
- Check that needle hole is clean.
- Install the needle of transfer line back into the inlet.

7.3.2 Leak testing

Materials required

- Cap 6.432.002
- Septum 7.010.019 (inlet septum)

Leak testing is performed for two channels: Carrier gas (Carrier) and Purge gas (Purge).

Leak testing for Carrier gas channel.

- Place a piece of rubber (e.g. septum) to the transfer line needle.
- Set carrier gas pressure between 0.4 and 0.44 MPa.
- Turn on the thermal desorber.
- Set carrier gas flow rate to 20 mL/min and save a method.
- Hold 1 minute, then check measured carrier gas flow rate. Acceptable value must be less than 1 mL/min. If this leakage value is exceeded, spot the leak point by a leak detector. When leak spot is revealed, eliminate leakage.

Leak testing for Purge gas channel:

- Place the cap 6.432.002 to Vent 1 fitting.
- Set at thermal desorber input gas line a pressure rate between 0.1 and 0.15 MPa.

- Turn on the thermal desorber.
- Place the sample tube into position #1.
- Enter the menu **Service** – **Leak test**.
- Set purge gas flow to 20 mL/min.
- Hold 2 minutes, then check measured carrier gas flow rate. Acceptable value must be less than 1 mL/min. If this leakage value is exceeded, spot the leak point by a leak detector. When leak spot is revealed, eliminate leakage.

To avoid contamination of the system, it is prohibited to use soap solutions for leak test!

When re-tightening fittings, avoid applying to much force exceeding values shown in Section 4.5.

If you cannot spot the leak by the leak detector, it can be determined by installing plugs to a certain portions of gas lines.

7.3.3 Packing or replacement of sorbent tubes

Packing procedure for sorbent tube is described in Section 6.5.

Replacement of the sorbent tube is made upon sorbent lifetime expiration.

7.3.4 Replacing of sealing O-rings

The thermal desorber has a reminder to replace O-rings. By default, the counter is set to 1000 samples. When the set value is exceeded, the warning message "Change O-rings" is displayed.

Replace sealing O-rings when the power is disconnected.



Replacing the O-rings is carried out after cooling the heated valve to room temperature.

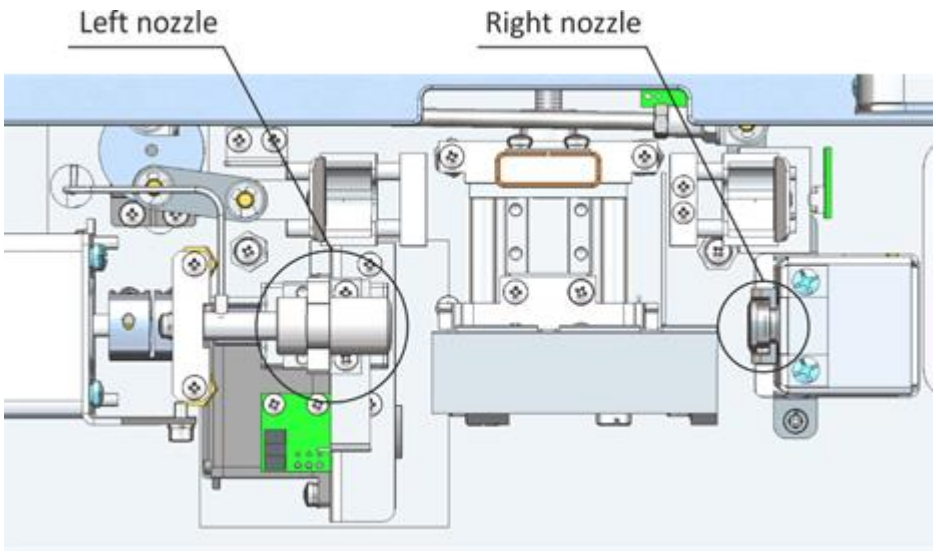
Tools required

- Screwdriver PH 1 (cross)
- Hook 6.461.001

Materials required

- Cotton applicators
- O-ring 8.685.176 – 2 pcs.
- O-ring 8.685.177 – 1 pc.

O-rings are located in the left and right nozzles.

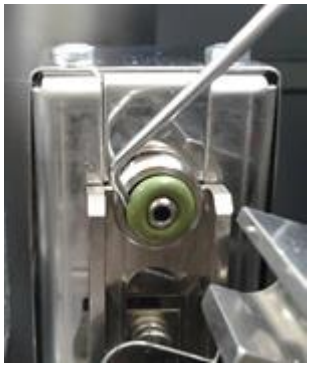


Replacing right O-ring

In the right nozzle O-ring 8.685.177 is installed.

Procedures:

- Pick up the O-ring with a hook 6.461.001 and remove it.



- Remove mechanical particles from the groove with a cotton applicator.
- Carefully place a new O-ring with your fingers into the groove.

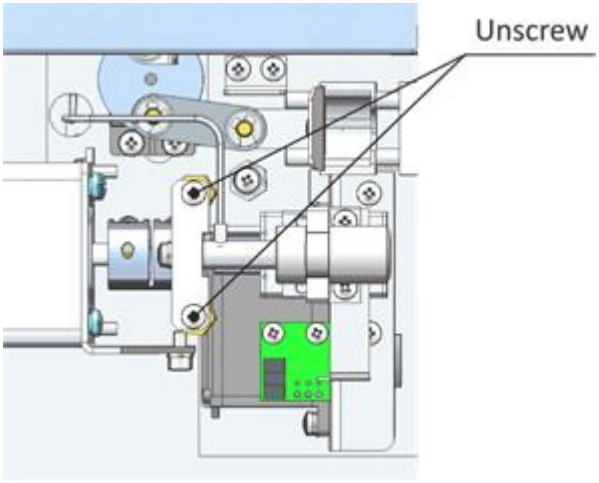
Use hook 6.461.001 to remove the rings only. Install new rings by hand, without using any tools.

Replacing left O-ring

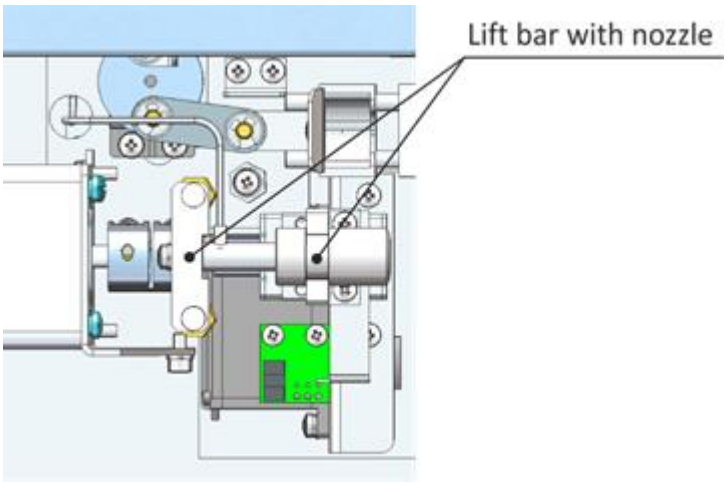
In the left nozzle two O-rings 8.685.176 are installed.

Procedures:

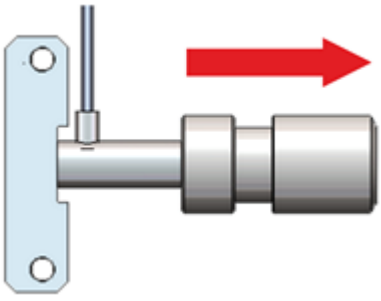
- Remove the screws that secure the bar.



- Lift the bar with the nozzle.



- Slide the nozzle off the guide.



- Pick up the O-ring with a hook 6.461.001 and remove it from the nozzle.



- Remove the ring on the other side of the nozzle in the same way.
- Remove mechanical particles from the groove with a cotton applicator.

- Carefully place new rings 8.685.176 on each side of the nozzle with your fingers.
- Install the flange in the guide and fix the bar with screws.

Apply the hook 6.461.001 only to remove O-rings. Install new rings with your fingers, without using any tools.

After replacing the O-rings, reset the maintenance counter. In the main menu, select **Service** - **Maint**. In the **O-ring changing** bar, press the counter, enter 0 and confirm with the button ✔.

7.3.5 Replacing a vent trap

The thermal desorber has a reminder to replace the vent trap. By default, the counter is set to 365 days. When the set value is exceeded, the warning message "Change filter" is displayed.

Replace a vent trap when the power is disconnected.

Tools required

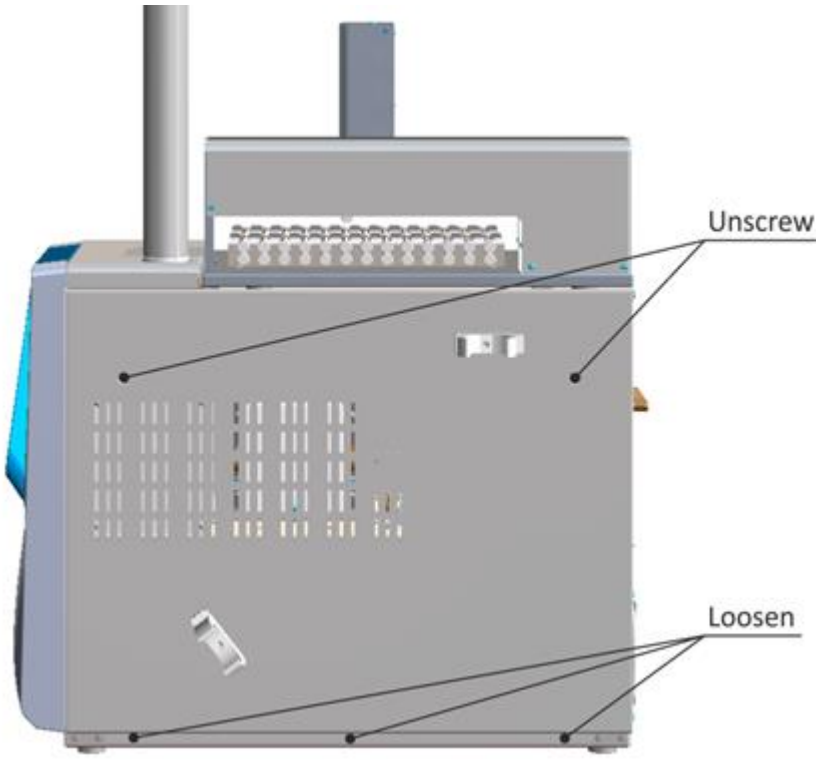
- Screwdriver PH 1 (cross)
- Wrench 8 mm
- Wrench 10 mm

Materials required

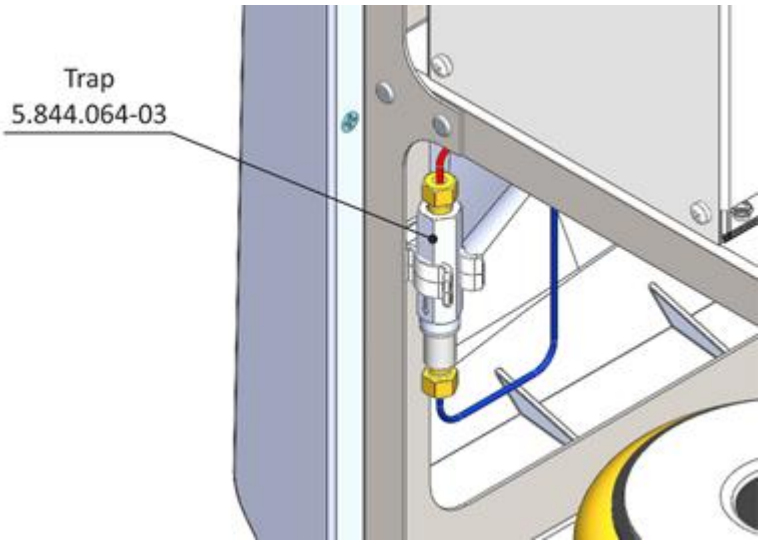
- Trap 5.884.064-03

Procedures:

- Remove the right cover.



- Unscrew the nuts sealing gas tubes in the upper and lower parts of the trap.



- Remove the trap from a bracket.
- Fit a new trap on the bracket, the arrow should be from top to bottom.
- Connect tubes to the trap.

Fit the side cover of the thermal desorber.

After replacing the vent trap, check leak of the purge gas line according to Section 7.3.2.

To reset the maintenance counter in the main menu, select **Service** - **Maint**. In the **Filter changing** bar, press the counter, enter 0 and confirm with the button ✔.

7.3.6 Replacing a trap and sealing O-rings of the trap

The trap is replaced when the type of sorbent should be changed or at expiration of trap lifetime.

Trap sealing O-rings are replaced when integrity of the O-rings appeared (cracks, dents, shape distortion) or when leaks in the trap occurs.

The thermal desorber has a reminder to replace the trap. By default, the counter is set to 1000 samples. When the set value is exceeded, the warning message "Change trap" is displayed.

Replace the trap and sealing rings when the power is disconnected.



Replacing the trap and sealing rings is carried out after cooling the trap and the thermostat of the valve to room temperature.

Tools required

- Screwdriver PH 1 (cross)

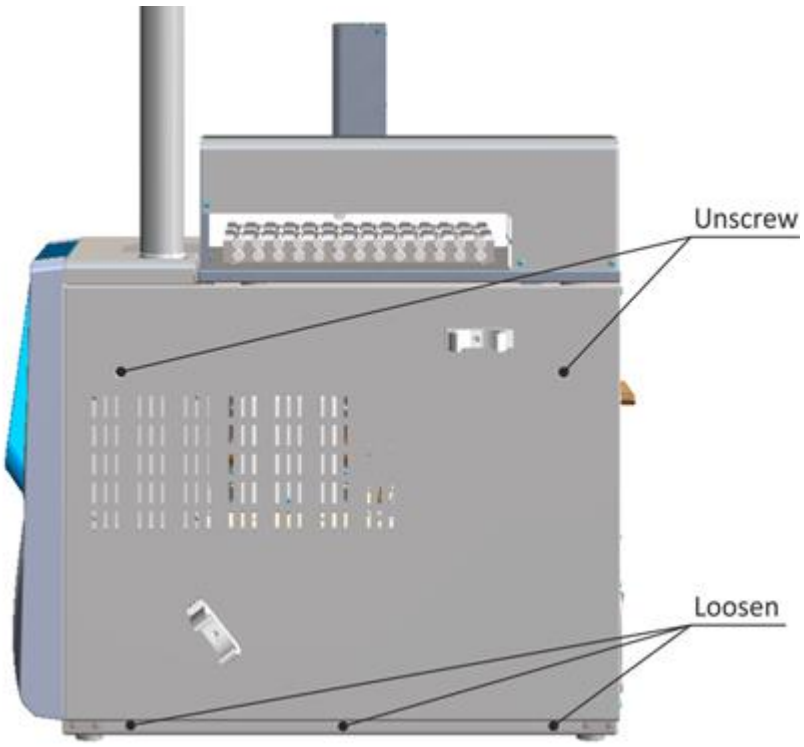
Materials required

- Trap 6.452.033 (type of sorbents depends on the analysis)

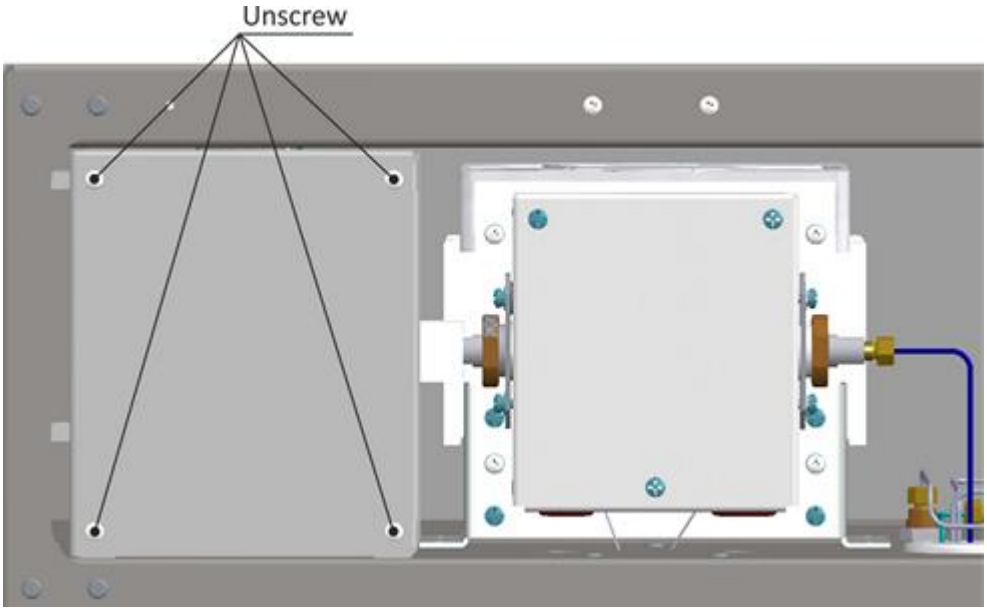
- O-ring 8.683.032 – 2 pcs.

Procedures:

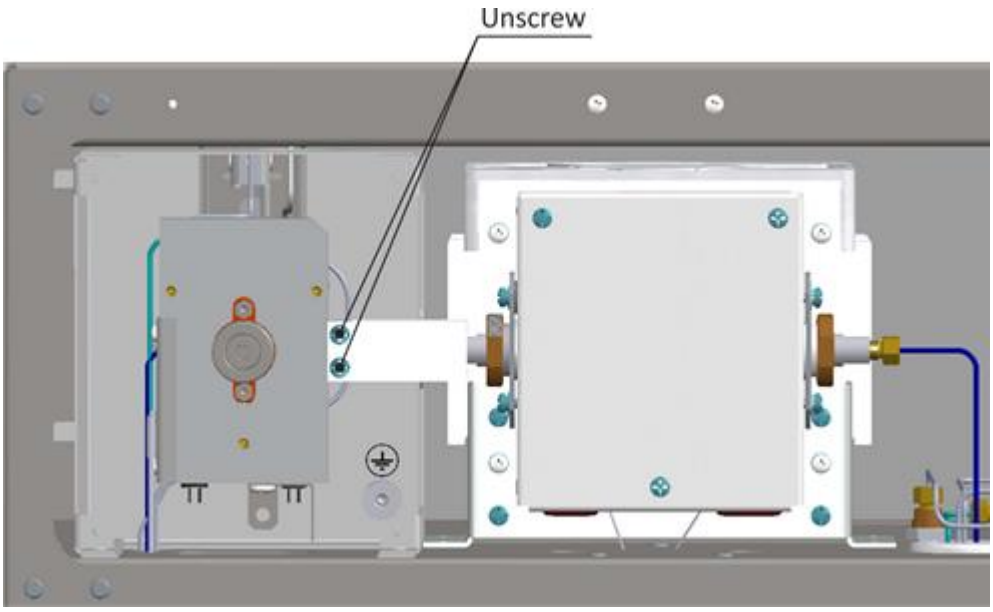
- Remove the right cover.



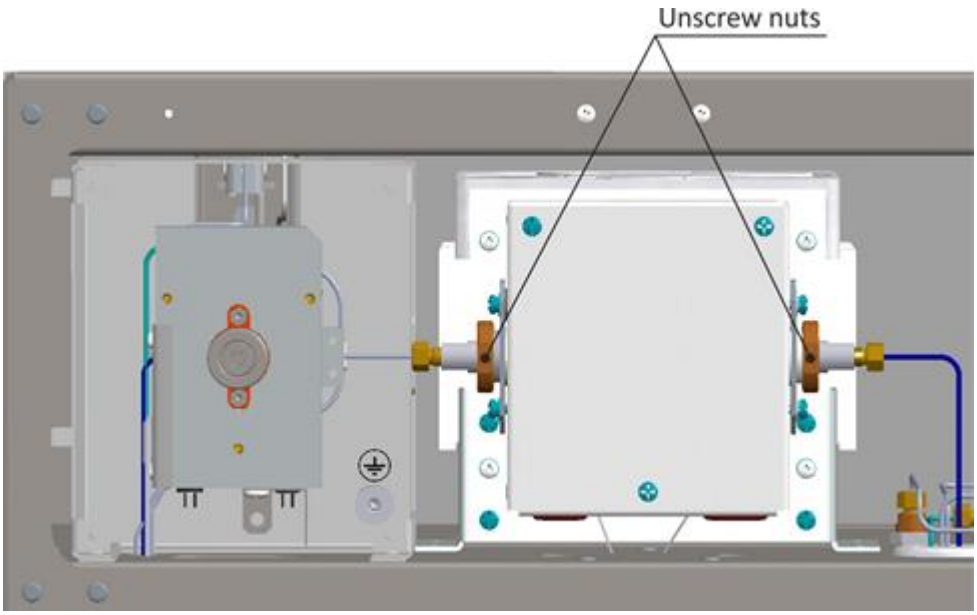
- Unscrew the screws on the valve oven cover.



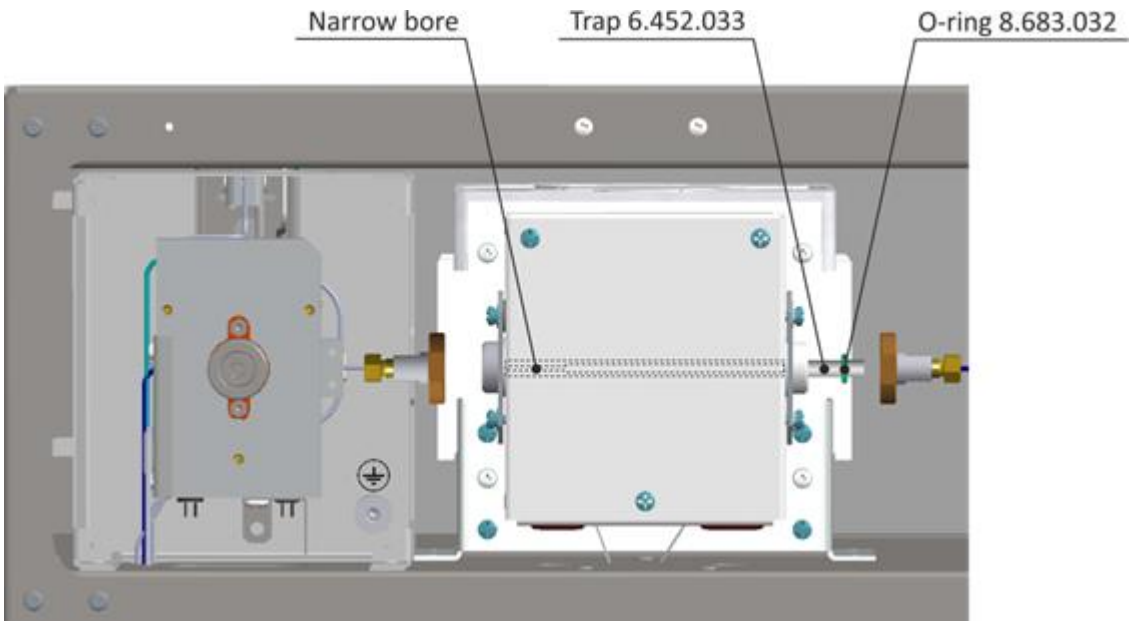
- Unscrew the screws of the plate.



- Unscrew the nuts fixing the trap.



- Take off O-rings 8.683.032 and extract the trap.



Insert a new trap with its tapered side in the direction of the valve and assemble the thermal desorber in reverse order.

Tighten the nuts of fixing the trap by fingers without using tools, as this can damage the trap.

After installing the trap, check the leak of the purge gas channel according to Section 7.3.2.

To reset the maintenance counter in the main menu, select **Service** - **Maint**. In the **Trap changing** bar, press the counter, enter 0 and confirm with the button ✔.

7.3.7 Replacing transfer line needle

The transfer line needle should be replaced when it becomes dirty, clogged or damaged.

Tools required

- Wrench 8 mm
- Wrench 5.5 mm

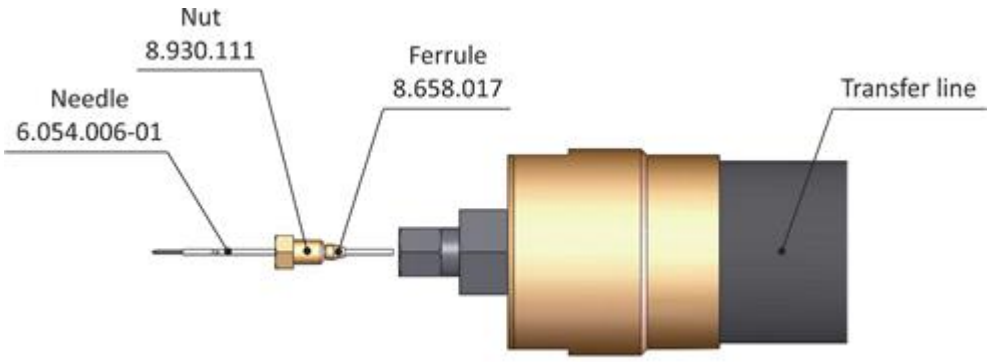
Materials required

- Needle 6.054.025

Needle 6.054.025 consists of a needle 6.054.006-01, a ferrule 8.658.017 and a nut 8.930.111.

Procedures:

- Unscrew a nut 8.930.111
- Remove old needle
- Insert a new needle 6.054.006-01
- Place a ferrule 8.658.017 onto a new needle
- Tighten the nut 8.930.111



After installing the needle, check the leak according to Section 7.3.2.

7.3.8 Replacing fuses

The failure of the fuses, as a rule, indicates problems with the external electrical supply of thermal desorber. Before replacing fuses, check the stability of the voltage in the electrical power supply.

To avoid electric shock, turn off the thermal desorber and unplug the power cord from the electrical outlet.

Tools required

- Screwdriver SL 4 (flat-blade)

Materials required

- Fuse 10 A/250 V – 2 pcs.
- Install only fuses 10 A/250 V into the thermal desorber.

Procedures:

- Unscrew the fuse holder with a screwdriver



- Take out the holder and extract the fuse from it.



- Install a new fuse in the holder.
- Close the holder with the screwdriver

Before switching on the thermal desorber, make sure that the electrical network is grounded. If there is no grounding in the electrical network, connect the thermal desorber to the external ground loop with a cable 6.644.056.

If after the installation of new fuses, they again burn out, contact manufacturer service support.

8 Troubleshooting

During the operation of thermal desorber, analytical troubles may occur, leading to unsatisfactory analysis results. As a rule, User himself can fix analytical troubles of the thermal desorber.

During operation following analytical troubles may be encountered:

- Poor repeatability of parallel measurement results.
- Broadening or tailing peaks.
- Splitting peaks.
- Right-angled triangle shaped peaks.
- Ghost peaks.
- No peaks on chromatogram.

If any analytical troubles arise, check the compliance of the thermal desorber operating method with the analytical application you are performing. A description of method development and the optimizing parameters are given in Section 6.

8.1 Poor repeatability

Repeatability test is carried out according to Section 5.9.

It should be noted that the value, regulated by the method of analysis should be considered for evaluation, rather than the value of standard deviation (RSD) specified in the technical specifications.

Possible trouble	**Action**
Poor repeatability cause at GC side	Disconnect the thermal desorber from the GC. Check the repeatability by injecting the control mixture into GC inlet
Sample volume is not constant	When aspirating liquid samples, check that there is no air bubbles in the syringe
	When aspirating air samples to obtain repeatable results, the concentration and volume of gas pumped through the tube should be constant from run to run
	When analyzing solid samples, the sample should be homogenized and its mass in the tube should be constant
Leak at carrier gas or purge gas channel	Test the leak according to Section 7.3.2

8.2 Broadening or tailing peaks

Broadening peaks

Possible trouble	Action
A liner in GC inlet has a wide inner diameter	Install liner 7.352.066 into GC inlet
Insufficient flow of carrier gas during the desorption of components from the trap	Set carrier gas flow rate as recommended in Section 6.13.11
The trap is filled with a too strong sorbent	Choose weaker sorbent as recommended in Section 6.7

Tailing peaks

Possible trouble	Action
GC column is not suitable	Replace the column
Deterioration of column performance	Replace the column
Sorbent lifetime in the trap expired	Replace the trap (Section 7.3.6)

Tailing or broadening of the first chromatographic peaks of volatile (in the beginning of chromatogram)

Possible trouble	Action
During desorption from the sorbent tube, a large amount of water or solvent is concentrated on the trap	Increase the temperature or time of Dry purge step prior to desorption
	Increase the lower temperature of the trap so that the water or solvent was concentrated at minimum level
	Set the heating rate from 500 to 1000 °C/min

8.3 Right-angled triangle shaped peaks

Possible trouble	Action
GC column overloaded	Increase split ratio in GC inlet

8.4 Splitting peaks

Possible trouble	Action
A sample with a large amount of solvent is introduced into the sorbent tube	Blow out the solvent from the sorbent tube before installing it in the thermal desorber
The sorbent tube contains a large amount of moisture	Optimize the tube dry purge parameters prior to desorption, for example, increase the dry purge time (Section 6.13.8)

8.5 Ghost peaks

If ghost peaks are detected, take into account that all sorbents give bleed impurities at desorption, the amount of which can reach ng level. Porous polymer sorbents like Chromosorb and Porapak have the highest bleed level (from 5 to 50 ng), then Tenax (from 0.1 to 1 ng). The lowest bleed is observed for carbon adsorbents like Carbopack, Carbosieve, Carboxen (from 5 to 50 pg).

Ghost peaks coinciding in retention time with the peaks of the analyzed components

Possible trouble	Action
Incomplete desorption of substances from the tube	Increase desorption temperature
	Increase purge gas flow at desorption
	Increase desorption time
	Use a weaker sorbent in the tube (see Section 6.4)
Incomplete desorption of substances from the trap	Increase high temperature of the trap
	Increase time of trap heating
	Use a weaker sorbent in the trap (see Section 6.4)

Ghost peaks having a retention time that is different from the analyzed components

Possible trouble	Action
Purge gas contains pollutants	Use purge gas with a minimum purity of 99.999 %
Filter at purge gas line contaminated	Replace the filter in purge gas line
The tube, connecting gases to the thermal desorber, contaminated	Disassemble connecting gas lines, heat the tubes in an oven at 150 °C purging by stream of pure inert gas or replace them with a clean line
Desorption temperature too high resulting in decomposition of the sorbent	Decrease temperature of desorption

To identify the source of contamination, the following series of analyzes is consistently carried out:

- Blank analysis on the GC with thermal desorber turned off. If there are ghost peaks, look for ghost peaks source in the GC.
- Blank analysis of an empty sample tube, with desorption time 2 min. If there are no ghost peaks on the chromatogram, the reason is incomplete desorption of the analyzed substances from the sorbent or insufficient cleaning of the sample tubes. In accordance with test method, increase the desorption temperature, the purge gas consumption and desorption time in such a way that complete desorption of substances takes place and there are no peaks during re-desorption. Carefully bake the sample tubes.
- Blank analysis of an empty sample tube, with desorption time 4 min. If on the chromatogram obtained, the responses of ghost peaks increased by 2 times compared with the previous step, then the reason is the insufficient purity of the purge gas. Use purge gas with a purity of not less than 99.999%, replace the filters in the purge gas line, and replace the tubes which supply the purge gas to the thermal desorber.
- Blank analysis of an empty trap. If there are no ghost peaks on the obtained chromatogram, the reason is incomplete desorption of the analyzed substances from the trap. In accordance with the test method, increase the temperature, the flow rate of the carrier gas and the heating time in such a way that complete desorption of substances takes place.

If a blank analysis with an empty sample tube and a trap shows the presence of contaminants, then the following causes are possible:

- Low temperature the valve or the transfer line. Increase the temperature of the valve and the transfer line.
- Contaminated needle of the transfer line. Clean the needle or replace according to Section 7.3.7.
- Contaminated internal channels of the valve. Thoroughly blow the channels with clean inert gas.
- Contaminated tube of the transfer line. Thoroughly blow the line with a clean inert gas. If it is difficult to remove contaminants in the line, it can be washed with perchloroethylene, toluene, ethanol or ethyl acetate.
- Contaminated tubes of a carrier gas or a purge gas line. Thoroughly purge the tubes with a clean inert gas. If it is difficult to remove contaminants in the tube, it can be washed with perchloroethylene, toluene, ethanol or ethyl acetate.

Before washing the tubes, contact manufacturer service representative for detailed instructions.

8.6 No peaks on chromatogram

Possible trouble	Action
Cause at GC side	Disconnect the thermal desorber from the GC. Inject the control mixture into GC inlet to check peaks availability
The transfer line needle is not inserted into GC inlet	Insert transfer line into GC inlet
No purge gas at the thermal desorber	Check gas supply at the input of Purge channel of the thermal desorber
Fitting Vent 1, Vent 2 or Vent 3 sealed	Remove caps from the fittings Vent 1, Vent 2 and Vent 3 and keep them free
Leak in the carrier gas or purge gas line	Check the leak according to Section 7.3.2

8.7 Hardware troubles

When operating thermal desorber, hardware troubles may occur, leading to various malfunctions. These malfunctions, depending on the level of complexity, are eliminated by the User working with the thermal desorber, the service personnel of the enterprise operating the thermal desorber, or a service engineer.

Thermal desorber does not turn on

Possible trouble	Action
Power cord is not connected	Check power cord connection
Fuses burnt out	Check fuses, replace if necessary (Section 7.3.8)

Thermal desorber does not go to the Ready

Possible trouble	Action
The temperature of the trap, valve, transfer line or flow of carrier gas cannot be set	See recommendations below depending on the type of fault
The set temperature at dry purge for sorbent tube below the ambient temperature	Set the purge temperature to at least 5 °C above the ambient temperature
GC analysis time is calculated and set incorrectly	Change the time of GC analysis in the method (Section 0)

The specified flow rate of carrier gas cannot be established

Possible trouble	Action
Incorrect carrier gas type selected in configuration	Check carrier gas type in configuration parameters
No carrier gas supplied to the thermal desorber	Check carrier gas supply at Carrier fitting
Insufficient carrier gas pressure	The pressure of the carrier gas in the gas line at the input to the thermal desorber should be more than 0.4 MPa
Leak in carrier gas channel	Check for leaks (Section 7.3.2)
Transfer line needle is clogged	Check that the needle is clean (Section 7.2.3)
Different types of carrier gas are used in the thermal desorber and in the GC	Connect the thermal desorber and the GC to the same carrier gas
Faulty gas flow controller (DFC)	Replace the DFC
The valve does not turn to the end position, blocking the gas channels	Contact manufacturer service representative

The specified flow rate of purge gas cannot be established

Possible trouble	Action
Incorrect purge gas type selected in configuration	Check purge gas type in configuration parameters
No purge gas supplied to the thermal desorber	Check purge gas supply at Carrier fitting
Leak in purge gas channel	Check for leaks (Section 7.3.2)
Fitting Vent 1, Vent 2 or Vent 3 sealed	Remove caps from the fittings Vent 1, Vent 2 and Vent 3 and keep them free
Gas flow controller (DFC) failure	Replace the DFC

The specified vent gas flow cannot be established

Possible trouble	Action
Fitting Vent 1, Vent 2 or Vent 3 sealed	Remove caps from the fittings Vent 1, Vent 2 and Vent 3 and keep them free
The transfer line needle is not inserted into GC inlet	Insert transfer line into GC inlet
Too high split flow rate	Reduce split flow rate

Low carrier gas pressure set in the GC	Set higher carrier gas pressure in the GC

The trap is cannot cool to the set low temperature

Possible trouble	Action
Too high ambient temperature	Make sure that the ambient temperature is below 32 °C
Thermal desorber is located close to the chromatograph or a room wall	Move the thermal desorber from the GC or the wall
Trap fan malfunction	Check the rotation of the trap fan, in case of malfunction, replace the fan
Fault of the fan on the rear panel	Check the rotation of the fan on the rear panel, in case of malfunction, replace the fan

Trap temperature rises during desorption from the sample tube

Possible trouble	Action
Purge gas flow during desorption is too large (more than 50 mL/min)	Reduce purge gas flow

The trap does not heat to the set temperature

Possible trouble	Action
The trap heater wires are not tightly screwed to the terminals	Tighten the heater fixing nuts to the terminals
Malfunction of power module board	Replace a power module board

Sample tube heater does not heat up

Possible trouble	Action
Heater malfunction	Replace the heater
Malfunction of power module board	Replace a power module board

The valve does not heat up

Possible trouble	Action

Heater malfunction	Replace the heater
Malfunction of power module board	Replace a power module board

Transfer line does not heat up

Possible trouble	**Action**
Heater malfunction	Replace the transfer line
Malfunction of power module board	Replace a power module board

8.8 Error messages

If an error occurs during operation of the thermal desorber, a corresponding message is displayed. This section describes a list of error messages, as well as possible causes and remedy actions.

When some errors occur, the thermal desorber stops the sequence and goes to the Waiting stage. In this case, after troubleshooting, in the **Service** menu, click the **Reset** button and run the method again.

In the event of the fatal error, the TDA stops operation and all heating zones are turned off. Further TDA operation is not possible.

Table 8.1 – Error messages

Error message	**Possible cause**	**Action**
Tube # Leak test is failed!	There is a leak in the purge gas channel	Check the leaks in purge gas line (Section 7.3.2)
	O-ring of AUX flange heater is worn out	Replace O-ring (Section 7.3.4)
	O-rings in sample tube sealer are worn out	Replace O-rings (Section 7.3.4)
	Trap is broken	Replace the trap (Section 7.3.6)
	Trap O-rings are damaged	Replace O-rings (Section 7.3.6)
	Setting of tube position in the thermostat is not correct	Re-set tube position in the oven (Section 4.14.4)
	Solenoid valve SV2 is damaged	Replace solenoid valve SV2
Method list is completed	No more than 10 methods can be stored in the internal memory	Delete previously saved and unused methods (Section 5.6)
Tube not found	There is no tube in the set position of the tray	Place the tube to this position of the tray or set the correct position of the tube

	Tray position calibration is not correct	Calibrate position # 1 of the tray (Section 4.14.1)
Could not load tube	In the position of the tray to move the tube in, there is another tube placed or there is some kind of obstacle	Release tray from obstacles. Reset error. Re-set method carrying out
Could not move arm	There is an obstacle when moving the manipulator	Release tray on its way of movement from obstacles. Reset error. Re-set method carrying out
Tube lost	When moving the manipulator, tube fell out of the gripper	Place the tube back in the tray. Reset error. Re-set method execution
Tube in gripper	There is a tube in the gripper	Remove the tube from the griper of the manipulator
Could not move tube in oven	There is an obstacle when transferring the tube from the oven	Release the sample tube shifter from the obstacle. Reset error. Re-set method carrying out
Sealer failed	There is an obstacle in the movement of the flange of the sorption tube seal assembly	Release the seal assembly from the obstacle. Reset error. Re-set method carrying out
Fatal error. Heater #	Malfunction of the temperature sensor of the corresponding channel	Contact manufacturer service representative

9 Transportation and storage

The thermal desorber in manufacturer 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.

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.

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

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

10 Manufacturer warranty

The manufacturer guarantees that the thermal desorber complies with the technical specifications provided the customer observes the conditions of transportation, storage, installation, operation, as well as with the implementation of recommendations on the time schedule of maintenance given in Section 7 of this manual.

The warranty period is 12 months from the date of commissioning, but not more than 18 months from the date of acceptance of the quality control department of the manufacturer, unless other conditions are stipulated in the purchase contract.

The warranty does not cover consumables: filters, septa, liners, needles sealing materials (couplings, rings, and other similar materials) that are suitable for delivery.

Warranty period for sorbent tubes and traps are determined by the accompanying documents for these products. If the accompanying documents do not contain instructions on the warranty period of operation, the warranty period of operation is 3 months from the date of commissioning but not exceeding 6 months from the date of shipment.

11 Claims

In case of thermal desorber 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 1.2.
- 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.

12 Annex

Sorbents types and their short marking graved on the sorbent tubes are listed in the table below.

Table 12.1 – Sorbents and their short marking

Sorbent	**Short marking**
Tenax® TA	TTA
Tenax® GR	TGR
Chromosorb™ 102	C102
Chromosorb™ 106	C106
Porapak™ N	PN
Porapak™ Q	PQ
Carbopack™ B	CB
Carbopack™ C	CC
Carbopack™ X	CX
Carbosieve® S-III	CSIII
Carboxen® 1000	C1000

The list of consumables required for the thermal desorber is given in the table below.

Table 12.2 – Consumables

Description	Cross ID	Order P/N (qty)
Sorbent tube, SS, 1/4" x 3.5" (empty, no caps)	-	653-2001 (10/pk)
Sorbent tube, glass, 1/4" x 3.5" (empty, no caps)	-	653-1001 (10/pk)
Glass wool, DMCS treated	-	635-0120 (50g/pk)
Sorbent tube, SS, preconditioned, with storage caps (Tenax® TA)	-	653-2012 (ea)
Sorbent tube, glass, preconditioned, with storage caps (Tenax® TA)	-	653-1012 (ea)
Sorbent tube, SS, with storage caps (Tenax® TA)	6.452.065	653-2010 (ea)
Sorbent tube, glass, with storage caps (Tenax® TA)	6.452.067	653-1010 (ea)
Sorbent tube, SS, preconditioned, with storage caps (Air Toxic)	-	653-2302 (ea)
Sorbent tube, glass, preconditioned, with storage caps (Air Toxic)	-	653-1302 (ea)
Sorbent tube, SS, with storage caps (Air Toxic)	-	653-2300 (ea)
Sorbent tube, glass, with storage caps (Air Toxic)	-	653-1300 (ea)
Sorbent tube, SS (activated charcoal)**	6.452.065-33	653-2504 (ea)
Trap (Tenax® TA)*	6.452.033	650-1001 (ea)
Vent trap	5.884.064-03	510-2134 (ea)
Transfer line needle (with ferrule and nut)	6.054.025	541-4212 (ea)
O-ring 006 (tube radial sealing), Tmax 300 °C	8.685.176	506-3062 (10/pk)
O-ring 003 (tube face sealing), Tmax 300 °C	8.685.177	506-3058 (10/pk)
O-ring 006HT (tube radial sealing), Tmax 425 °C	8.685.176-01	506-3063 (ea)
O-ring 003HT (tube face sealing), Tmax 425 °C	8.685.177-01	506-3059 (ea)
O-ring 005 (trap sealing)	8.683.032	631-9011 (ea)
Cap (sealing tube in the tray of the thermal desorber)	6.433.075	650-2024 (10/pk)
Storage cap (storage and transportation)	6.433.091	650-2026 (10/pk)
Container	6.087.025	650-2022 (10/pk)

* Depending on the method, the sorbent may be different. Specify the sorbent in the order

** Used in split mode (see Section 6.13.12 for details)