LABORATORY GAS CHROMATOGRAPHS

214.2.840.039UM "Chromatec-Crystal 5000"

214.2.840.075UM "Chromatec-Crystal 9000"

USER MANUAL

PART 1

DESCRIPTION AND OPERATION. GENERAL INFORMATION

Edition 10.01.2025

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1 USER MANUAL STRUCTURE

User manual supplied with gas chromatographs "Chromatec-Crystal 5000" and "Chromatec-Crystal 9000" consists of the following parts:

- **Installation instructions**. Instructions on GC location and installation
- **Part 1. Description and operation. General information**. General data about construction, operation and maintenance of the GC.
- **Part 2. Inlets, Gas valves, detectors**. Data about construction, operation features and maintenance of inlets, valves and detectors.

2 INTRODUCTION

Instructions marked by this symbol are necessary for carrying out in order to avoid injuries sustaining at working with chromatograph.

Instructions marked by this symbol are necessary for carrying out in order to avoid chromatograph damage.



Instructions marked by this symbol are to be carried out when there is electron-capture detector (ECD) in chromatograph, which containing radioactive source Ni63.



This symbol makes warning about danger of burn. It is painted on:

Top cover of the gas chromatograph (hereinafter the GC) (warning about hot surfaces of injectors and detectors).

Rear cover of the GC indicating: "**Hot air**" (warning about danger of burn at oven heating and cooling cycles.



Instruction marked by this symbol is warning about explosion risk.

There is a sign in the oven door:

Attention! Before GC turning on make sure that hydrogen lines are sealed or connected to the column.

The chromatograph is intended for analysis of liquid, gaseous and solid samples of various organic and some inorganic compounds.

This manual contains information about construction and operation principles of gas chromatographs referred to models:

- "Chromatec-Crystal 5000" Modification 1. 214.2.840.039 (further C5000.1)
- "Chromatec-Crystal 5000" Modification 2. 214.2.840.0039-01 (further C5000.2)

Hardware and software bundle for medical researches on the basis of chromatograph "Chromatec-Crystal 5000" Manufactured according to Technical Specifications (TS) 9443-004-12908609-99

- "Chromatec-Crystal 9000" (further C9000). 214.2.840.075 Manufactured according to according to TS 4381-010-12908609-08

This user manual (UM) is intended for persons working with gas chromatograph "Chromatec-Crystal" and personnel maintaining it. The manual serves for acquaintance with chromatograph construction and its components, providing correct chromatograph operation and keeping the GC in constant operation availability.

For operation and maintenance, the GC, personnel shall have knowledge of computer operation at beginner level.

Information on chromatograph construction and operating principle are focused on specialists acquainted with foundations of gas chromatography and principles of gas chromatographic devices construction.

SDO Chromatec products may have some modified parts manufactured according to new design or may be undergone inessential improvements which are not described in current document.

This UM reflects principal data about product in order to provide efficient operation and maintenance.

As for repairing, it recommended to use service documents or instructions on setting and control for corresponding products or its component.

2.1 Safety precautions

Warning! Don’t start operating chromatograph without being acquainted with safety precautions stated in Section 2.1 of **GC. Installation instructions.**

Don’t start operating chromatograph without being acquainted with installation instructions, GC User Manual and user manuals on auxiliary equipment.

2.2 Operation conditions

Warning! Don't start operating chromatograph without being acquainted Operating conditions stated in section 2.2 **GC. Installation instructions.**

Don't start operating chromatograph without being acquainted with installation instructions, GC User Manual and user manuals on auxiliary equipment.

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

2.3 Technical specifications

Check out technical specifications controlled at GC manufacturing or repairing are specified in logbooks:

- 214.2.840.043LB (Chromatec-Crystal 5000).
- 214.2.840.073LB (Chromatec-Crystal 9000).

Full technical specifications are specified in the product specification data sheet. Basic specifications on the GC mainframe are specified below (Table 2.1).

The number of detectors is given without taking into account the mass spectrometric detector (MSD), since it is structurally an external device in relation to the chromatograph and is installed next to it.

Data transmission from the chromatograph:

• Digital via Ethernet interface;

• analog with a programmable electronic attenuator (chart scale 10 mV) (separate version on request)

Table 2.1 – Chromatograph basic technical specifications

Parameter	**Value**
Gas Chromatograph mainframe overall dimensions: (width, depth, height), mm	460, 590, 480
Weight, kg	49
Electric supply	Alternating current network 220V±10%, 50-60Hz
Power consumption, W	maximum 2500 average 700
General data	
Instrument keyboard and display:	Touch screen LCD for control and monitoring (optional).

Parameter	Value
	External keyboard with 4-line LCD display (optional).
Full electronic flow and pressure control	Yes
Max number of heating zones	13
GC communication interface	LAN Ethernet
Inlets available	
Packed Inlet	
Split/Splitless Inlet:	
Programmable Split/Splitless Inlet	
Detectors available	
Flame Ionization Detector (FID)	
Flame Photometric Detector (FPD)	
Thermal conductivity detector (TCD)	
Micro Thermal conductivity detector (μ-TCD)	
Electron-capture detector (ECD)	
Micro Electron-capture detector (μ-ECD)	
Nitrogen phosphorous detector (NPD)	
Catalytic combustion detector (CCD)	
Photoionization detector (PID)	
Pulsed discharge detector (PDD, PDHID)	
Mass Spectrometry Detector (MSD)	
Inlets available	
Packed Inlet	
Split/Splitless Inlet:	
Programmable Split/Splitless Inlet	
GC capability	
Max Number of inlets	up to 3
Max Number of detectors	up to 4
Max Number of valves	up to 6

The operating temperature ranges of the flow rates and gas pressures, the maximum power consumption can be limited individually in each chromatograph to optimize and improve the convenience and safety of the operating conditions of the device. These settings are performed in user and expert configuration parameters.

Technical specifications such as: general requirements, reliability, electromagnetic compatibility, composition of the set, marking, package, safety precautions, acceptance instructions are specified:

- in the Bundle User manual 214.2.849.043UM for the GC "Chromatec-Crystal 5000".
- in Chromatograph User manual 214.2.849.073UM for the GC "Chromatec-Crystal 9000".

2.4 Delivery set

Composition of the set is described in the documents specified below (Table 2.2):

Table 2.2 – Documents containing information about composition of the set

Product	**Document containing data about composition of the set**
Hardware and software Bundle for medical researches on the basis of chromatograph "Chromatec-Crystal 5000"	Logbook 214.2.840.043LB
Chromatograph "Chromatec-Crystal 9000"	Logbook 214.2.840.073LB

Chromatograph is supplied to customer in accordance with purchase contract specification and GC pneumatic diagram for particular customer application.

GC pneumatic diagram and corresponding shipping kit are specified in attachment B of current User manual.

3 CHROMATOGRAPH CONSTRUCTION

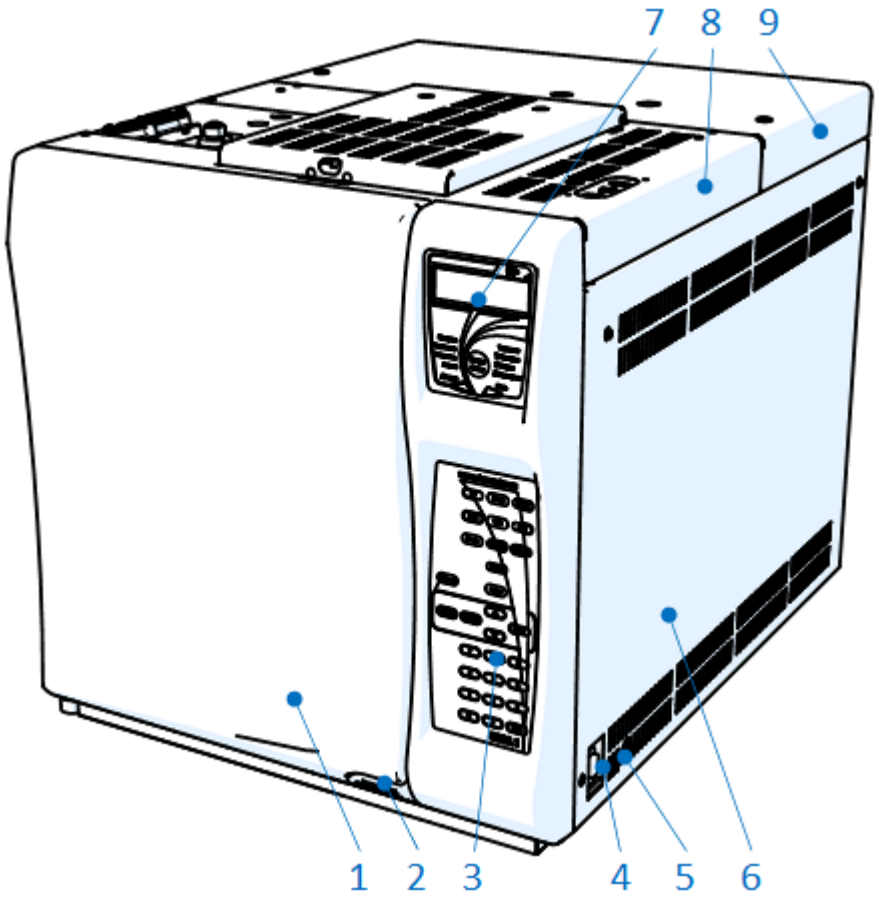
Chromatograph is a tabletop device, consists of functional assemblies disposed in single frame. Built-in detectors, inlets and valves are located on analytical platform under top front cover of the GC. Gas controllers and solenoid valves are located on analytical platform under top rear cover. Electronic boards are located mainly under right side cover of the GC.

Mass-spectrometric detector is a separate device; it can work either in tandem with the GC or independently from GC.

The general view of chromatographs "Chromatec-Crystal 5000", versions 1 and 2, as well as ("Chromatec-Crystal 9000" is shown in the figures below.

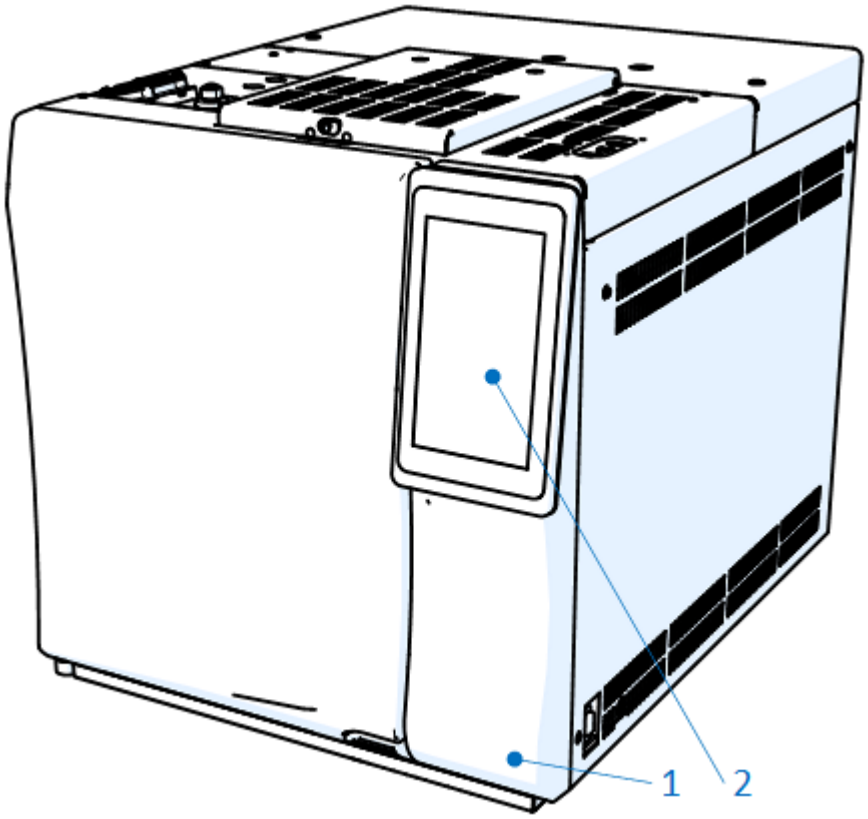
In accordance with the wishes of the customer chromatograph "Chromatec-Crystal 5000.1" can be equipped with a control panel with a push-button keyboard and a text four-line display or a touch screen.

The GC overview is shown below.



1 – oven door; 2 – oven door latch (push to open); 3 – built-in keyboard (for C5000.1 only); 4 – on/off button; 5 – slot for connection external keyboard (for C5000.2, C9000 only); 6 – right cover (electronic side); 7 – Status board; 8 – top front cover; 9 – top rear cover.

Figure 3.1 – Chromatograph Chromatec-Crystal 5000.1 (9000). Overview



1 – “Power supply” indicator, 2 – touch screen.

Figure 3.2– Chromatograph "Chromatec-Crystal 5000.1" with built-in touch screen. Overview

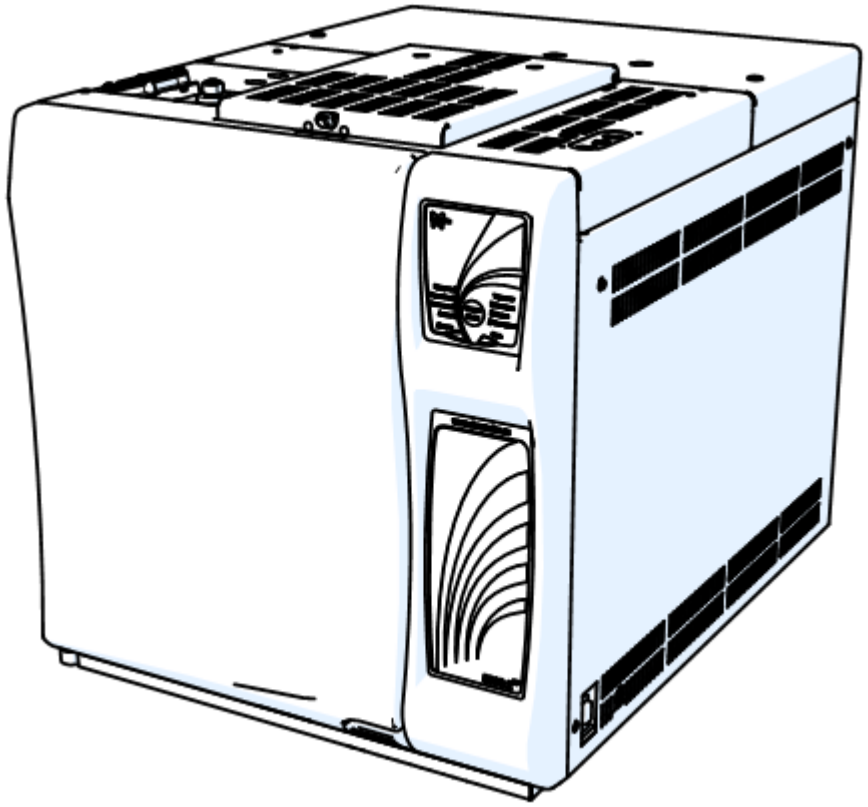
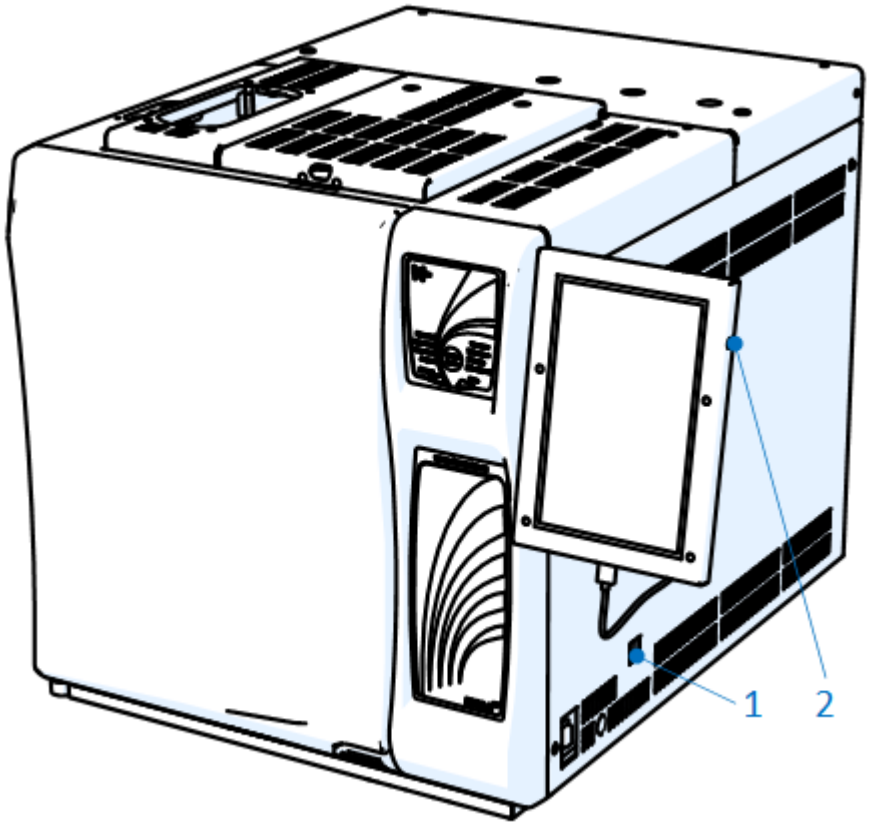


Figure 3.3– Chromatograph "Chromatec-Crystal 5000.2"

On a separate order, the chromatograph can be equipped with an additional Touch Screen Control Display P/N 150-6112.

Unlike the built-in touch display (Figure 1.2), this Control Display is implemented removable, based on a tablet computer with Windows operating system

The Control Display is mounted on the right-side cover of the chromatograph using the holder 1E-8181.



1 – USB slot for power connection; 2 – Standby button

Figure 3.4 – Chromatograph "Chromatec-Crystal 5000.2" additional Touch Screen Control Display

The tablet is connected using a USB cable to the corresponding slot on the right cover of GC.

Control Panel from the Chromatec Analytic software package is installed on the tablet.

Control Panel software is connected to the chromatograph wirelessly via Wi-Fi using a router included with the optional control panel.

Connecting the router to the chromatograph and the computer is described in section 3.8.6 of the chromatograph installation instructions.

The version of the Control Panel software installed on a tablet is similar to the PC version. The configuration of the router is described in section 2.2.4, the specifics of the operation are described in section 7.7 of the Chromatec Analytic user manual.

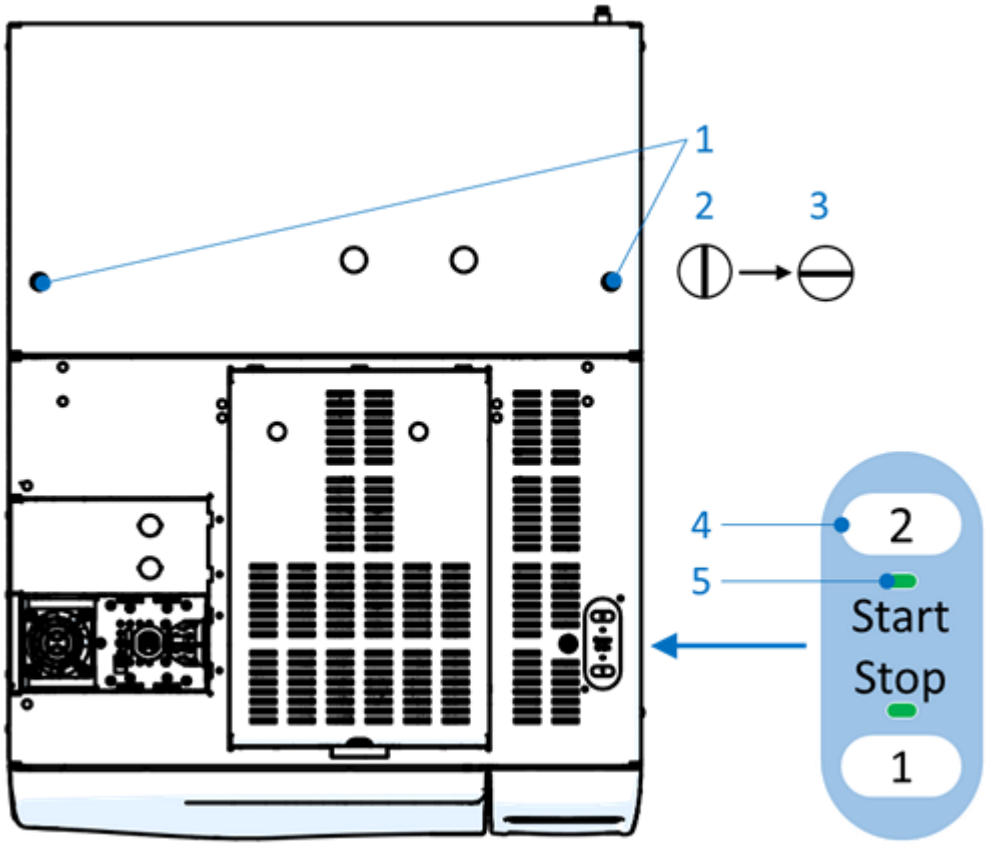
Control Display is turned on autonomously. Control Display off automatically when the chromatograph is turned off.

Above features relate only to the external removable Touch Screen Control Display (Figure 1.4) and do not apply to the built-in display (Figure 3.2). It does not require a Wi-Fi router.

If there is an external or built-in touch display, the operator can use the following functions:

- Setting the instrumental method and configuration of GC and MSD.
- Monitoring of GC status, monitoring of diagnostic parameters.
- Viewing and recording of detector signals and diagnostic signals (graphs of temperature changes, gas flow rates and pressures, voltages, etc.).
- Ensuring the operation of the chromatograph, MSD and external devices without a personal computer

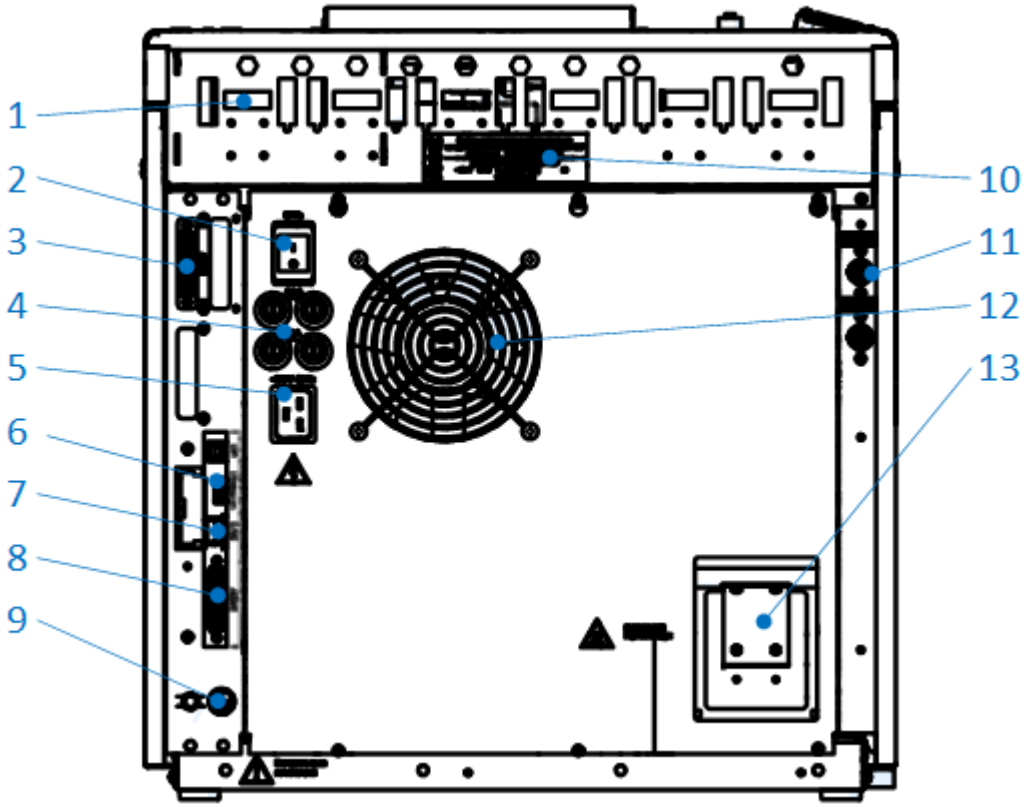
There is a life-threatening voltage in the right and rear compartments of the chromatograph. It is forbidden to remove the compartment covers if the plug is connected to the mains.



1 – locks, 2 – "closed" position, 3 – "opened" position, 4 – additional buttons for Start and Stop of analysis, 5 – additional “Ready” indicator.

Figure 3.5 – Chromatograph. Top view

Turn the lock and raise top front cover by sides to obtain an access to detectors and inlets. Turn the locks and remove top rear cover to obtain an access to gas controllers and solenoid valves.



1 – gas connection fittings; 2 – power on/off switcher; 3 – RS-232 (to PC); 4 – fuses; 5 – 220V (to power cord); 6 – USB-flash slot; 7 – Ethernet (LAN) slot to PC; 8 – "Chromatec" peripheral device slot; 9 – grounding terminal; 10 – serial number tag; 11 – DETECTOR slot (to MSD and 3rd party detectors “Ready out; 12 – ventilator of rear compartment”); 13 – oven exhaust.

Figure 3.6 – Chromatograph. Rear view

Data about construction, usage and maintenance of detectors, inlets, valves are specified in Part 2 of current UM.

If any device is connected to inlet (e.g. autosampler), access to detectors is carried out at opening detectors top cover.

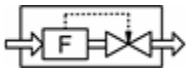
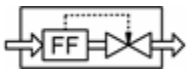
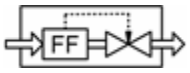
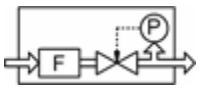
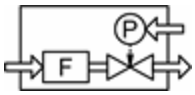
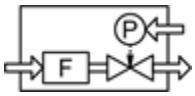
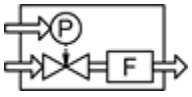
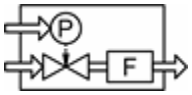
During heating/cooling cycles of oven hot air blast is blowing out of the exhausts (Figure 1.6, p.14), which may cause burns. Do not place any subjects closer, than on (40-50) cm to the exhausts.

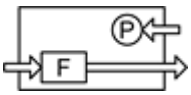
3.1 Gas flow and pressure controllers

3.1.1 General information

Digital flow controllers (DFC) and digital pressure controllers (DPC) intended for carrier gas, hydrogen and air flow and pressure control operating with different units (Inlets, Detectors, Auxiliary units). Types and features of DFPC controllers are shown in table below.

Table 3.1 – DFPC types

EPC controller type and marking	**Pressure, kPa**	**Operation principle**	**Function**	**Working range**
DFC 10-02	360-1250		Flow	10 – 500 ml/min
DFC 10D-02 Digital flow controller, high-speed	360-1250		Flow	1 – 500 ml/min
DFC 10H-03 Digital flow controller, high-speed	360-1250		Flow	5 – 1250 ml/min
DPC 11-02 Digital pressure controller	360-700		Pressure Flow	5 – 450 kPa 10 – 500 ml/min
DPC 11H-02 Digital pressure controller	360-1250		Pressure Flow	20 – 1050 kPa 10 – 500 ml/min
DPC 11H-03 Digital pressure controller	360-1250		Pressure Flow	20 – 1050 kPa 5 – 1250 ml/min
DPC-11A2-02 Digital pressure controller	360-700		Pressure Flow	5 – 450 kPa 10 – 500 ml/min
DPC-11H2-03 Digital pressure controller	360-1250		Pressure Flow	20 – 1050 kPa 5 – 1250 ml/min
DPC-11U-02 Digital pressure controller “to itself"	20–600		Pressure Flow	20 – 450 kPa 10 – 500 ml/min
DPC-11U-03 Digital pressure controller "to itself "	20–1250		Pressure Flow	20 – 1050 kPa 5 – 1250 ml/min

Gas flow and pressure meter (Indicator)	20-1250 (flow sensor) 20-400 (pressure sensor)		Pressure (absolute)	20 – 400 kPa
			Flow (Indicator)	10 – 500 ml/min
Gas flow meter			Flow (Flowmeter)	20 – 450 ml/min (γ=$\pm$ 4%)*
Mechanical pressure controller	10-1000		Flow	(1-5) ml/min

*Main reduced measurement error for flow

DFPC type is automatically recognized in the GC. Application of the DFPCs follows the GC flow diagram; configuring is carried out in the Control Panel of the Chromatec Analytic software.

The inlet gas pressure must be sufficient to ensure the specified flow rates (pressures) of the gas, taking into account the pneumatic resistance of the pneumatic diagrams at the outlets of the regulators.

Due to the scatter of the parameters of purchased elements of regulators, the upper measurement limits may differ slightly from those indicated.

Pressure rate can be set in kPa (on default) or psi units for all types DPC11. Setpoint resolution is varied from 0.1 to 0.001, e.g., to 0.001 psi. Setpoint resolution is adjusted in expert setting options.

Each gas channel has independent setting options by type of gas, functionality, control parameters, set point resolution and operating range. The settings can be carried out in User and Expert parameters options.

3.1.2 Digital flow and pressure controllers

Digital gas flow controllers DFC10, digital gas pressure controllers DPC-11and upstream pressure controllers DPC-11U.

Marking of DFPC shows coding basic specifications and design features:

DFC-10 – measurement and control of gas mass flow
DPC-11 – measurement and control of gas pressure
D – measurement and control of gas mass flow extended to low level to 1 ml/min.
H – high pressure sensor up to 1050 kPa.
U – upstream pressure control and measurement
P – DFPC case is purged with inert gas
“2” in the controllers 11A2, 11H2 – is the second pressure sensor to measure and control a pressure level in the flow diagram, outside of the DFPC.
“-02” ending – flow transducer, measuring up to 500 ml/min
“-03” ending – flow transducer, measuring up to 1250 ml/min

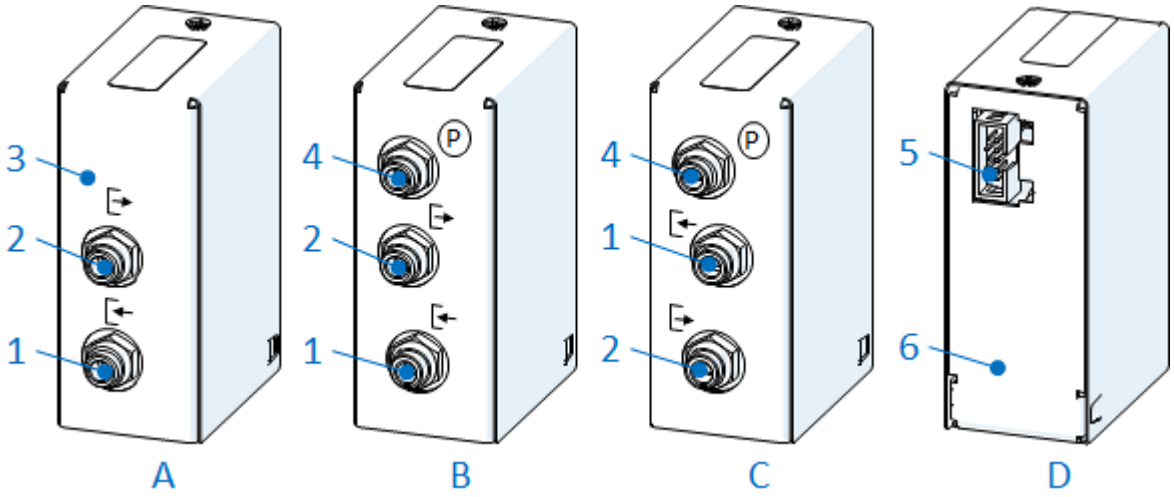
Gas controller DFPC contains the controlling electromagnetic analogue valve and transducer of gas flow in voltage, connected among each other with tubing.

Flow transducer measures gas flow by thermal principle. The corresponding voltage is transmitted on solenoid valve in order to control a required gas flow.

The solenoid valve works in voltage range from 0 to 10 V: the least value corresponds to closed valve position, the greatest – maximum opened. Valve work is most resistant at voltage from 3,5 to 6,0 V.

DFC 10-02 and DFC 10D-02 gas controllers provide constant mass gas flow at output pressure from 0 to 350 kPa and at input pressure from 360 to 700 kPa. For stable flow control it is recommended, that the pressure difference between input and output was at least 50 kPa.

6-th gas channel of DFPC module has extended by 2 times range of flow level – from 10 to 800 ml/min. Typically it is appointed for air flow to flame detectors.



A – DFC10-02, DFC10D-02, DFC10H-03, DPC11-02, DPC11H-02, DPC11H-03;

B – DPC11A2-02, DPC11H2-03;

C – DPC11U-02, DPC11U-03;

D – Rear view.

1 – input fitting; 2 – output fitting; 3 – casing; 4 – fitting to pressure sensor; 5 – base; 6 – IDC slot (to mainboard).

Figure 3.7 – Digital flow and pressure controllers

Gas flow and pressure meter (Indicator)– measures the mass flow rate of gas according to the thermal principle. It also has an absolute pressure sensor. Indicator is designed to monitor changes in the flow rate and pressure without evaluating their values with normalized accuracy.

Gas flow meter has a similar design to the Gas flow and pressure meter. It can be used to measure the flow rate of the analyzed gas with a normalized metrological characteristic – the main reduced error of measuring gas flow.

Calibration and primary verification of gas flow is carried out on nitrogen gas by the manufacturer.

To measure the flow of other gases, it is necessary to recalculate the values in accordance with the table.

Table 3.1 – Gas coefficients

Gas	Formula	Relative coefficient at 20°C and 1 bar.
Nitogen*	N2	1,00
Argon *	Ar	1,40
Hydrogen*	H2	1,01
Air	Air	1,00
Helium*	He	1,41
Oxigen	O2	0,98
Methane	CH4	0,76

* For the marked gases, the correction is entered into the software automatically when the gas type is specified, for the remaining gases, a "manual" recalculation is required relative to the specified gas type "nitrogen".

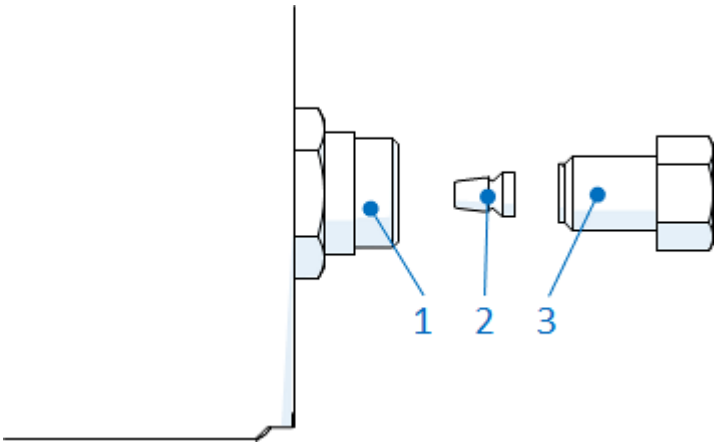
DFPC fittings have marking, it is important to follow them when connecting tubes:

– Gas input

– Gas output

– Pressure sensor fitting

– Purge line to blow DFPC casing with inert gas



1 – DFC fitting, 2 – ferrule 8.658.039-01 (stanless steel), nut 8.930.161

Figure 3.8 – Connection of tube to DFC fittings

DPC 11 controllers additionally have a pressure sensor and maintain the gas pressure at the outlet, and also measures its flow rate. In DPC-11-02 sensor monitors the gas pressure in the outlet fitting of the regulator.

In DPC-11A2-02, DPC-11H2-03, DPC-11U-02, DPC-11U-03 pressure sensor has its own fitting placed on the housing of the DPC, and can control the pressure at a given point in the pneumatic diagram of the chromatograph.

Gas controllers DPC11-02, DPC11H2-03 can work either in mode of gas pressure control or in mode of control of gas flow, speed, cap.flow (gas flow through capillary column).

For using convenience at working with capillary column in the software "Chromatec Analytic" is implemented the calculator allowing the user to operate these or other parameters of work (pressure, flow, peripheral gas speed through capillary column). The gas calculator is implemented in composition of the program "Control panel", and also as separate program.

Controllers can be used as an electronic gas flow meter. For this purpose, they should set the maximum value of the controlled parameter (flow rate or pressure).

DFC-10 DPC-11 (Except DPC11U) can be used as flow meter, for this one need to set maximum flow or pressure level (500 ml/min or 500 kPa).

Purged regulators (marked P) DPC11H-02 have a sealed housing. Helium or other gas through a separate fitting constantly blows through the internal cavities of the regulator to prevent the diffusion of air components into the regulator line.

Like the rest of the DPC, the purged regulators are equipped with a flow sensor. Flow sensors do not participate in the control processes (feedback comes from pressure sensors) and serve only to assess the condition and operating mode of the chromatograph.

It should be borne in mind that in a helium atmosphere the signal of the flow sensors is shifted – the flow value may differ from 0 when the valves are closed. The offset value depends on the individual features of the flow sensor and the pressure of the gas supplied to the device (the purge flow is limited by the pneumatic resistance built into the fitting).

Zero of the flow sensors should be taken as its reading after at least an hour of purge, when the air inside the regulator housing is replaced by helium.

Chromatec Analytic software allows you to set the controller parameters for each gas channel separately.

3.1.3 Gas flow rate checkup

Digital flow controllers DFC-10 and DPC-11 accommodate built-inflow gas meter, adjusted at manufacturing. The manufacturer does not standardize absolute flow accuracy for DFPCs.

At DFPC checkup the following parameters are observed:

- DFPC check for leaks
- Compliance of measured flow (pressure) rate with set value.

Test for compliance of real (absolute) flow rate with set value is not obligatory, nevertheless it can be provided (follow proceure described below).

In case of detection of a discrepancy between the "electric zero" and the actual flow rate set, it is possible to calibrate the gas flow controller according to the procedure described in the user manual

for Chromatec Analytic, section 7.5. Service operations. Calibration of the flow sensor. This function is supported for the firmware version of the central module 03.21.16.625, the Control Panel program 2.0.12.0 and higher).

When disconnecting DPC from gas lines, make sure that you set minimum temperatures of detectors, inlets and columns to prevent them from breakage.

DFC check for leaks:

- There should be no pressure in gas line before DFC. Switch GC on, do not set any mode on DFC.
- In 10 minutes, monitor DFC flow rate indication.

DFC flow rate indication shouldn't exceed 0.3ml/min (with no gas set). This indication meets to "electrical zero" of DFC. Registered rate of "electrical zero" is to be subtracted from leaks indications obtained in future leaks checking.

Take into account that if "electrical zero" was registered as 0 ml/min, the true "electrical zero" may be in negative side. Therefore, using such regulator for leaks check may understate real leaks.

- Disconnect tube from the outlet of tested DFPC, then seal the outlet (use plug 8.632.053 with ferrule 8.658.053 for DFPC-10(11)-02;
- Set working pressure on gas cylinder (0,4±0,04) MPa.
- Set flow 100 ml/min on DFC (or 350 kPa on DPC.
- Keep it within 10 min, then register flow rate indication on DFC.

DFPC flow rate indication should not exceed "electrical zero" rate for DFC. Excess indicates leaks in DFPC.

Checkup of compliance of measured flow rate with set flow

- Remove top covers of the GC;
- For DFC-10, disconnect any tubes from the outlet of checked DFC; for DPC-11 – leave tube connected, if the following pneumatics provides restriction of the flow in operating flow range, not less than 20 – 50 ml/min;
- Set gas flow value for tested DFC-10 or pressure value for DPC-11;

Stabilization of the flow (pressure) may take from several seconds to 1 minute. Gas controller is considered tested successfully if in 1 minute measured flow (pressure) rate meets to its set value, LED Check prm by this parameter doesn't light.

Test for compliance of real (absolute) flow rate with set value

This test shall be performed after leak check.

Measurement of gas flow rate (for carrier gas, hydrogen, air) shall be carried out with the help of burette (or soap-film flowmeter) and stop-watch by measuring speed of passing soap film through

determined burette volume. When using soap-film flowmeter, measured parameters shell be recalculated to normal conditions of temperature and ambient pressure (25°C, 101.3 kPa).

For carrier gas DFPC checkup is to be carried out in three points: lower, middle, higher points of flow rate range.

- Open top covers of the GC;
- disconnect tube from output of tested DFPC, connect flowmeter to this output instead;
- set gas flow rate for tested channel DFPC;
- check compliance of real flow rate with the set one in 10 minutes after setting.

For flame feeding gases (air, hydrogen) checkup shall be carried out at working flow rates, transferring the GC into "Test mode" in advance.

Sometimes it's necessary to use DFPC as a flow sensor, to measure flow rate in output of units (TCD detector, MPC and EPC in flow supporting mode etc.). For this purpose, set maximum flow rate (DFC) or pressure (DPC) rate (500 ml/min or 500 kPa) so valve was completely opened, the mass gas to be measured through it.

3.1.4 Gas pressure rate checkup

Checkup of real pressure rates compliance with set pressure rate.

Measurement procedure is carried out with the help manometer (accuracy class 0.15).

- Open top covers of the GC;
- disconnect tube from output of tested DPC, connect T-joint 6.453.022-02 to this output instead;
- attach restrictor 5.150.010 or 5.150.015 to one fitting of T-joint, connect gauge to another one;
- turn on the GC, transfer it into "Test mode" and set gas pressure at tested DPC;
- check compliance of real pressure rate with the set one in 10 minutes after setting;
- if measured flow pressure differs from the set figure more than ±2 kPa, despite it's free of leaks and input pressure level is within (0,4±0,04) MPa (admissible hydrogen pressure is 0,14 MPa), it points at malfunction of the DFC. Please contact manufacturer or its regional representative.

Checkup is to be carried out in three points: lower, middle, higher points of pressure rate range.

When disconnecting DPC from gas lines, make sure that you set minimum temperatures of detectors, inlets and columns to prevent them from damage.

If pressure value of electrical "null" and some set pressure value varies from measured, these may be corrected for each DFPC in Expert tab of "Control Panel" SW In the same way as for flow correction. Specify measured electrical "null" and calibration factor in corresponding parameters "Pressure. Zero setting" and "Pressure. Calibration factor" (for Central module firmware version 03.09.16.431 or higher).

3.2 Traps (filters)

Traps used with the GC are classified to their purpose:

- Cleaning of carrier gases and auxiliary gases from moisture, hydrocarbons and carbon dioxide at GC inlet;
- Additional cleaning gases after gas controllers;
- Cleaning vent gas from sample components;
- Cleaning analyzed gas of undesirable sample components, mechanical particles.

Table 3.3 – Types of traps installed in input gas lines before GC (outside GC)

Filter	**5.886.001(-05)**	**5.886.001-01**
Material	Stainless steel	Stainless steel
Max pressure	1.25 MPa	1.25 MPa
Max volume	35 ml	35 ml
Sorbent	MolSieve CaA	Active charcoal
Fraction	1-2 mm	1-2 mm
Connection thread (input and output)	Male M8×1	Male M8×1
Max diameter of connected tubing	3 mm*	3 mm*
Purpose	Carrier gas, hydrogen lines	Air lines

* Either 3mm or 1/8" (3.18mm) OD tubing is suitable for connection.

Traps of 3rd party manufacturers may be applied at GC inlet to provide deeper cleaning of gases when operating high-sensitive detectors like MSD, PDD, μ-TCD, μ-ECD, CCD.

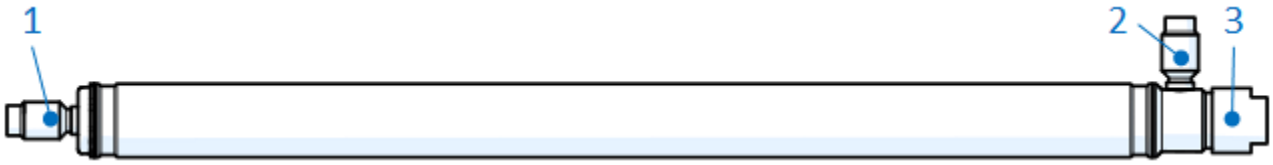
Table 3.4 – Types of traps disposed inside the GC

Filter	**5.884.048**	**5.884.064**	**5.884.065**	**6.112.008-01**	**5.844.015**
Material	Stainless steel	Stainless steel	Stainless steel	Stainless steel quartz	Brass, glass
Max pressure	1 MPa	1 MPa	1 MPa	0.4 MPa	0.4 MPa
Max volume	20 ml	1 ml	2 ml	1.5 ml	0.5 ml
Sorbent	MolSieve CaA	MolSieve CaA	Empty	Glass wool	Glass wool
Fraction	1-2 mm	0.2-0.4 mm			1-2 mm
Connection thread (input and output)	Male M8×1	Female M8×1	Female M6×0,75	Male M8×1	Female M8×1
Max diameter of connected tubing (input)	3mm or 1,6 mm	3 mm	1,6 mm	3 mm	1,6 mm
Max diameter of connected tubing (output)	1,6 mm	1,6 mm	1,6 mm	1,6 mm	3 mm
Purpose	Split vent	Carrier gas, hydrogen lines at DFPC output	H2 NPD, ref. gas TCD, make up CCD	Sample path line	Sample path line

3.2.1 Trap 5.886.001

The trap (Figure 3.9) is positioned in carrier gas lines and auxiliary gas lines outside the GC. Trap 5.886.001(-05) (filled with MolSieve) is intended for trapping moisture, carbon dioxide and hydrocarbons. Trap 5.886.001-01 intended for trapping hydrocarbons only.

Operational position of the trap is vertical or slightly inclined (up to 10°). This prevents from passing gas aside of sorbent. Trap has a stainless-steel frit (5 µm) at its outlet. Cap has a sealing teflon disk 8.260.100 inside. Glass wool tampon is inserted at inlet, in order to prevent from pouring sorbent.



1 – Output, 2 – Input, 3 – Cap.

Figure 3.9 – Trap 5.886.001. Overview

The trap is used separately or as a part of block of traps 5.884.012. For the best gas purification, it's recommended to connect consequently in gas line 2 traps 5.886.001-01 (active charcoal) and 5.886.001 (MolSieve).

Input of the trap shall be connected with tube to gas source. Traps output connect with the GC gas input (Figure 3.10).

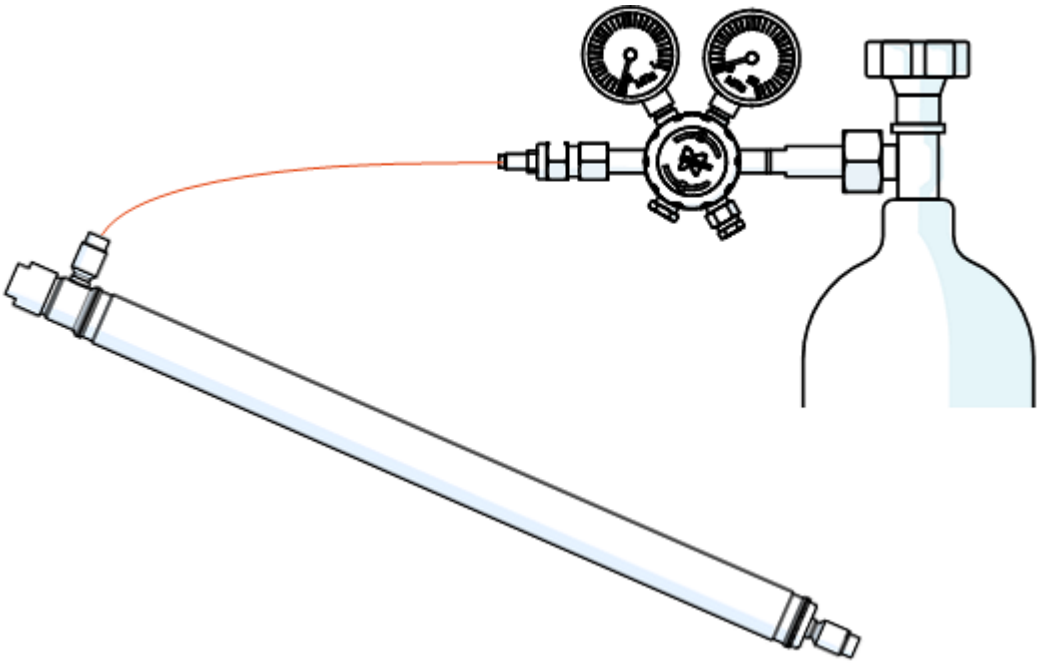
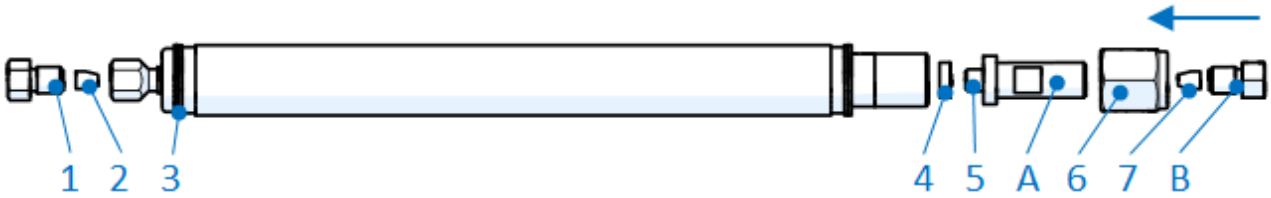


Figure 3.10 – Trap 5.886.001. Connection to the GC

3.2.2 Trap 5.884.048

Trap (Figure 3.11) is mounted in split vent gas line connected to split/splitless Inlet before DFC. Trap is intended for trapping solvent vapours.

Working position of the trap is vertical.



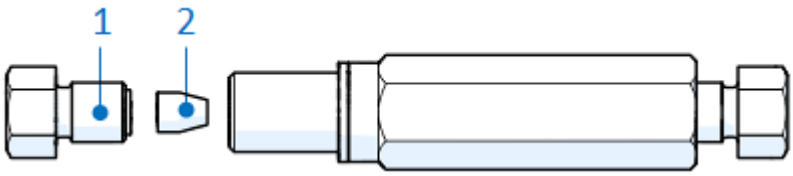
1 – Nut 8.930.167-02, 2 – Ferrule 8.658.053, 3 – Frit 5 um, 4 – O-ring 8.683.032-02, 5 – Gauze 4 um, 6 – Nut 8.930.172.

Figure 3.11 – Trap 5.884.048

Modification	**Max diameter of connected tube**	**Replaceable parts**	
		A	B
5.884.048	3 mm	Fitting 6.454.111	Nut 8.930.167
5.884.048-01	1.6 mm	Fitting 6.454.111-01	Nut 8.930.167-02

3.2.3 Trap 5.884.064

Trap 5.884.064 (Figure 3.12) is positioned in carrier gas or hydrogen gas line at DFPS output.



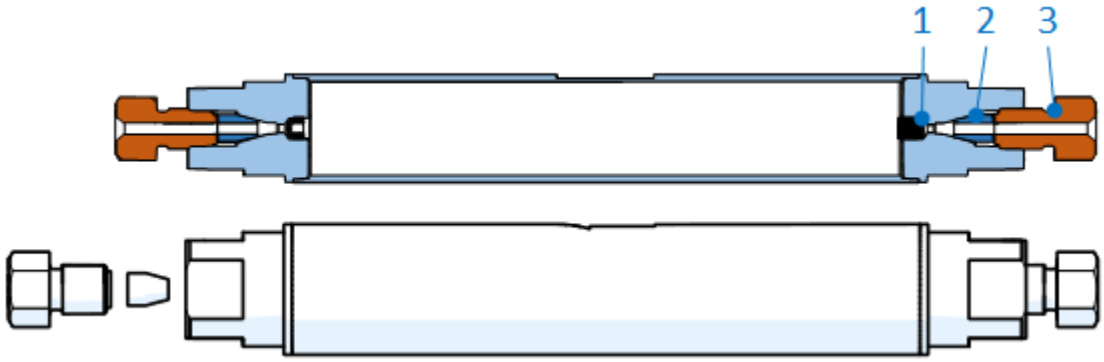
1 – Nut 8.930.161, 2 – Ferrule

Figure 3.12 – Traps 5.884.064

Model	**Ferrule**
5.884.064	8.658.053 (rubber)
5.884.064-01	8.658.039 (brass)

3.2.4 Trap 5.884.065

Trap 5.884.065 (Figure 3.13) is positioned at DFPC output in gas lines of TCD reference cell flow, by hydrogen line to NPD, by reactant gas line to CCD. Trap has a frit at its input to reduce deviations of gas flow level.

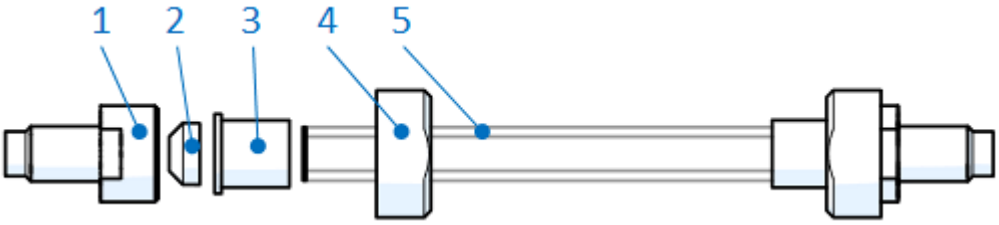


1 – Frit, 2 – ferrule, 3 – nut 8.930.161

Figure 3.13 – Trap 5.884.065

3.2.5 Trap 6.112.008

Trap (Figure 3.14) is positioned in sampling gas line. It is intended for cleaning analyzed gas from aerosols, acid gases or any undesirable contaminants. Depending on modification trap may have different stuff. Trap has a permanent body (glass tube).



1 – Fitting 8.652.126, 2 – ferrule 8.220.380, 3 – sleeve 8.223.183, 4 – nut 8.930.078, 5 – tube 7.352.024 (glass)

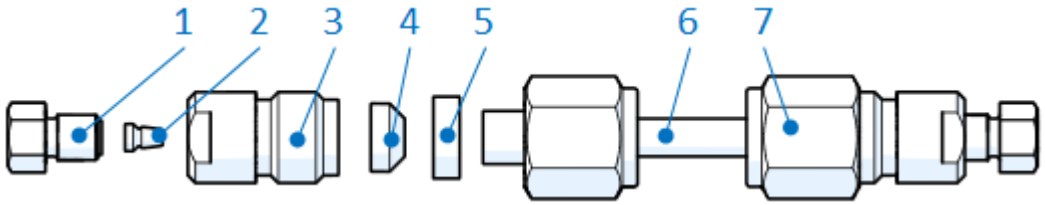
Figure 3.14 – Trap 6.112.008

Modification	Stuff	Purpose
6.112.008	Calcium chloride	Removing moisture
6.112.008-01	Glass wool	Removing solid particles
6.112.008-02	Silica gel / CoCl2	Moisture indicator
6.112.008-03	Empty	Depending on stuff in use

3.2.6 Trap 5.884.015

Trap is positioned in sampling gas line. It is intended for cleaning analyzed gas from aerosols and for protection gas lines from liquid sample ingress when it is used for determination of dissolved gases in electrical insulating oil. When being filled with special sorbents it may be used for trapping target chemicals (aromatics or olefins in ambient air analysis).

Unlike trap 6.112.008, it has smaller internal volume (0,7 ml) and can be applied when volume of gas sample is limited (less than 10 ml). Usually, it is fixed on bracket for filling syringes with carrier gas.



1 – Nut 8.930.161, 2 – ferrule 8.658.039, 3 – casing 8.034.406, 4 – sleeve 8.220.380 (rubber), 5 – sleeve 8.223.013, 6 – tube 7.352.050, 7 – nut 8.930.172

Figure 3.15 – Trap 5.884.015

3.2.7 Traps maintenance

General information on traps regeneration

Technical maintenance for the traps described above consists in regeneration of sorbents in traps by baking or replacement of sorbent. Traps of other manufacturers may be used in consist of the GC. These traps may not be regenerated, but replaced when their lifetime is expired according to supplied manual instructions.

Traps shall be baked completely, for example when traps in hydrogen gas line are regenerated, it's recommended to bake all the traps in this line (hydrogen generator traps, traps at DFPC output and others if available).

When regenerating traps in air gas line, compressors traps are also recommended to bake.

When regenerating traps in carrier gas line at the GC input, traps located at DFPCs output at carrier gas lines are also recommended to bake (if available).

Do not mix sorbents from different traps. It concerns not only sorbents of the same nature and fraction, but also sorbents of the same nature and fraction extracted from different traps. Each trap is better to fill with the same sorbent and to connect into the same place in pneumatic scheme where it was before.

Traps are to be sealed tightly sealed (with caps or plugs).

After carrier gas and hydrogen traps baking, Instrument calibration is recommended to carry out not earlier than in (10–12) hours of GC running.

Regeneration or replacement of the sorbent in input gas traps.

Regeneration or replacement of the sorbent in traps 5.886.001 is carried out:

- For MolSieve traps – in each (300–500) hours of running;
- For charcoal traps – in each (2000–3000) hours.

Recommendations on baking trap 5.886.001 (suitable for hydrogen generator and compressor traps as well):

- unscrew cap of the trap using wrench;
- take out the glass cloth tampon (for trap 5.886.001);
- empty trap adsorbent into metal or china ware;
- bake trap adsorbent for (2–4) hours depending on baking temperature and pollution degree;
- fill adsorbent back to trap periodically packing it by tapping. Sorbent is better to be filled hot;
- on the top the glass cloth tampon should be placed (for trap 5.886.001);
- screw in the cap with the sealing teflon disk inside (use wrench 10 for trap 5.886.001). Tightening stress should be enough to avoid leaks. Before screwing make sure that there is no any glass cloth fiber on the sealing edge of traps body;
- install trap on the place it was before, connect it to gas lines;
- blow the trap from air.

Trap adsorbent baking temperatures:

- charcoal: (140–165) °C (it is very important not to exceed temperature top limit);
- MolSieve CaA: (250–400) °C (for deeper cleaning of sorbent it is recommended to set temperature at its top limit);
- silica gel: (200–250) °C.

Once traps installed, check for leaks should be taken according to section 3.4.4.

Traps which are set on the carrier gas and hydrogen line after baking need blowing from air remains.

Regeneration or replacement of the sorbent in sample split vent trap 5.884.048

Regeneration or replacement of the sorbent in the vent trap (filled with MolSieve) is to be carried out in each (300–500) hours of operation, and also if ghost peaks appear in the beginning of chromatogram, which indicates at ingress of volatile compounds from vent channel back into inlet.

When analyzing high boiling point samples (e.g. crude oil), washing split vent tube (connecting inlet and vent trap) is strictly recommended at the same time with traps baking.

Regeneration procedure for vent trap is the same as for trap 5.886.001.

Regeneration of traps 5.884.064, 5.884.065

Design of the traps 5.884.064 and 5.884.065 is not supposed for sorbent replacement. To regenerate the trap, disconnect these from gas lines, the connect to its output tube 1.6 mm, fix it using nut 8.930.161 and brass ferrule 8.658.039. Then tube 1.6mm is to be connected to inert gas source. Set gas flow through the trap (15-25 ml/min) and keep trap at high temperature for 1-2 hours.

Traps on hydrogen channel

When working with hydrogen generator after all traps assembled and gas lines properly connected, switch hydrogen generator on, wait until pressure reaches its maximum, then in GC, unscrew the nut connected to hydrogen DFPC input fitting for (15-20) minutes. During this time the traps are blown out with hydrogen flow from air. Then screw back the nut on input DFPC fitting. The chromatograph during hydrogen traps blowing can be switched off.

When working with hydrogen gas cylinder, the hydrogen working pressure should be given, then bleed hydrogen by depressurization of its line on input of DFPC for hydrogen during (40–60) sec. Then screw fitting tight. While blowing traps at hydrogen line, the chromatograph can be switched off.

Traps on the carrier gas line

Take attention that oxygen remains in traps can harm GC column, therefore never exceed 30 °C in the oven until gas lines of carrier gas and hydrogen are carefully blown from oxygen remnants.

When working with capillary column after connection of all gas lines, open gas cylinder with carrier gas, set chromatograph working mode and unscrew the nut on fitting in sample vent line for (2-3) minutes. Then screw the fitting.

When working with packed columns in working mode, blow GC traps within (1-2) hours with general flow gas through gas flow controllers not less than 100 ml/minutes in total. When working with TCD detector (helium or argon as a carrier gas) blowing time should be not less than 2 hours.

3.3 Thermostats

3.3.1 Temperature zones of the GC

The GC has 13 temperature channels (maximum):

- Column oven;
- 3 channels for inlet;
- 3 channels for detectors;
- 4 channels for valves, piston injector;
- 2 auxiliary temperature channels. It may control: methanizer, auxiliary column thermostat, piston injector.

Described distribution between temperature channels is prearranged. Sometimes it may be different in real GC. Distribution of temperature channels in particular GC is specified in gas diagram supplied with the GC. At turning on the GC determines availability of temperature zones automatically. Setting temperature channels to corresponding thermostats of built-in units is performed in GC configuration (see section 3.7.4, power board setting).

When GC delivered in simple configuration, the power board is equipped with limited number of temperature zones (7 or 5). When the power board has 7 temperature zones, these are distributed in following way: 1 for the oven, 2 for detectors, 2 for inlets, 2 auxiliary zones.

Thermostats of the GC (except column oven) and their parts (heater assembly, heating element and temperature sensor) are described in the table below.

Table 3.2 – Heated units and their components

Temperature zone	**Heater assy**	**Heating element**	**Temperature sensor**
Detectors TCD and CCD	214.5.863.171	214.5.863.165	214.5.182.027-02
Single detector FID, PID, NPD, FPD, ECD	214.5.863.170	214.5.863.165	214.5.182.027-02
Two detectors FID, PID, NPD, FPD, ECD coupled on a heater	214.5.863.185	214.5.863.164-01	214.5.182.027
Inlet 7.2 or 7.3	no	214.5.863.164	214.5.182.027
Heated valve located from the left on bracket	214.5.863.193	214.5.863.166	214.5.182.027
Heated valve located on platform	214.5.863.167	214.5.863.166	214.5.182.027-01
Piston injector	214.5.863.198	214.5.863.164	214.5.182.027-01
Additional column thermostat	no	214.5.863.164	214.5.182.027-01
Methanizer	no	214.5.863.166	214.5.182.027

Each heater is an assembly including heating element, radiator, temperature sensor, brackets for fixing. Plug of heating element and temperature sensor of each temperature zone is connected into

corresponding slot of the power board. When connecting, plugs of heating element and temperature sensor must be attached to the same temperature channel.

Detectors TCD and CCD (up to three) are located on a single heater.

Detectors (FID, PID, NPD, FPD, ECD) are unified by installation socket, two detectors can be disposed on one heater.

3.3.2 Column oven

Below (Figure 3.16) main parts of the oven are shown.

At mode of temperature supporting (isotherm) oven shutters are closed. Regulation of shutters angle position at oven cooling is single step, performed by actuator (7).

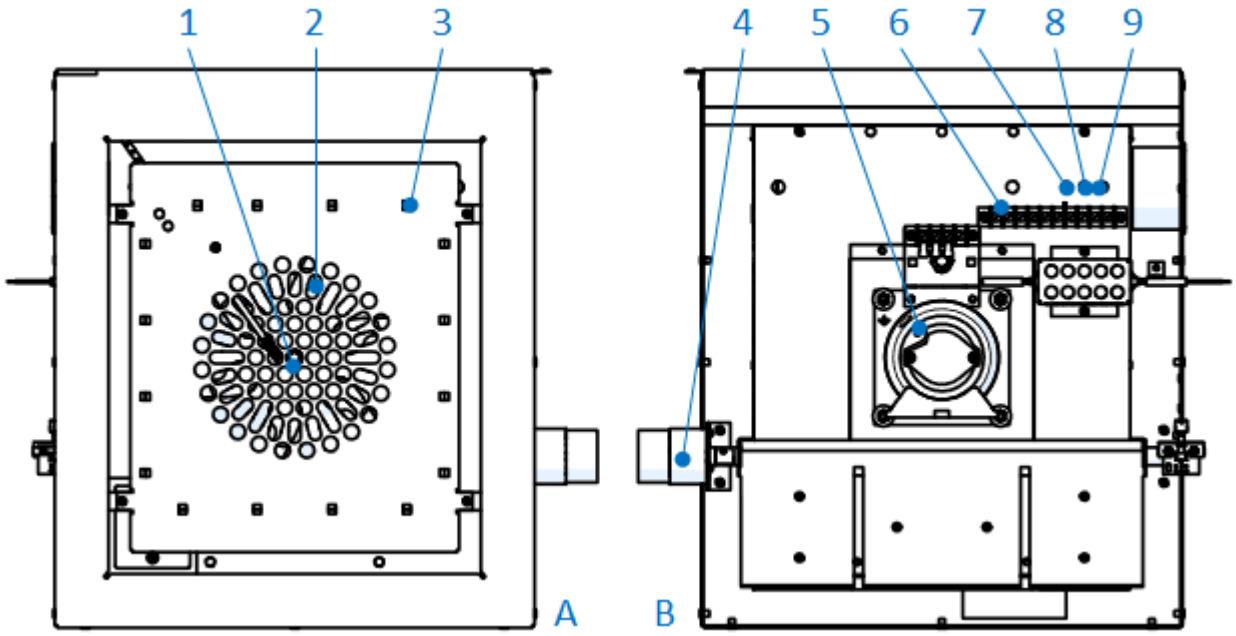


Figure 3.16 – Column oven

Table 3.3 – Main elements of the oven

№	Part number	Name	Purpose
1	5.132.016*	Temperature sensor	Temperature measurement in the oven
2	6.393.009	Vane	Air mixing in the oven
3	5.863.092	Heater	Oven heating
4	6.330.067	Actuator	Air mixing in the oven
5	МКРц 2х/k 4,6×0,9	Tube	Isolator of the heater
6	ТВ–41–10	Terminal	Heater and temperature sensor wires connections
7	6.330.052	Actuator	Operating with oven flaps

6	8.626.021	Tube-isolator	Isolator of temperature sensor wire
8	8.626.397	Tube-isolator	Isolator of power heater wire
9	5.132.086	Temperature sensor	Temperature sensor of emergency shutdown

* Included in heater 5.863.092 (assembly)



During oven heating/cooling cycles, hot air flow is blowing out of the exhausts which may cause burns. Be careful of it. Do not place any subjects closer, than on (40-50) cm to the exhausts.

When oven door opened, avoid touching heated surfaces of oven and door.

Input ventilating exhaust is located in the bottom side of the GC. Be careful, do not place any objects or paper sheets under the GC in order to keep free input ventilating exhaust of the oven.

The stability of maintaining the temperature of the thermostat columns is 0.02 °C.

The minimum temperature programming speed is 0.1 °C. The discreteness of setting the temperature values of the thermostat and programming is 0.1 °C.

The maximum programming speed of the column thermostat according to the linear law is 125 ° C/min (for the temperature range 50 – 70 ° C).

The maximum achievable programming speed of the column thermostat is 250 °C/min (for the temperature range 40 – 45 °C).

The chromatograph provides automatic cooling of the column thermostat to protect the columns when the carrier gas supply stops.

Column Thermostat Options

The operating temperature range and dynamic characteristics of the column thermostat can be expanded with additional options.

The thermostat ventilation device 214.5.883.050 provides the lower limit of the operating temperature from T (approx. c.) +3 ° C, as well as the cooling rate of the column thermostat from 450 to 50 ° C in 3.3 minutes (at room temperature 22 ° C).

Oven cooling system allows you to maintain the lower limit of temperature control -10 ° C or -15 ° C, depending on the model of the device.

The option of cooling oven with carbon dioxide allows you to maintain the lower limit of temperature control from -40 ° C, the option of cooling with liquid nitrogen – from -100 ° C.

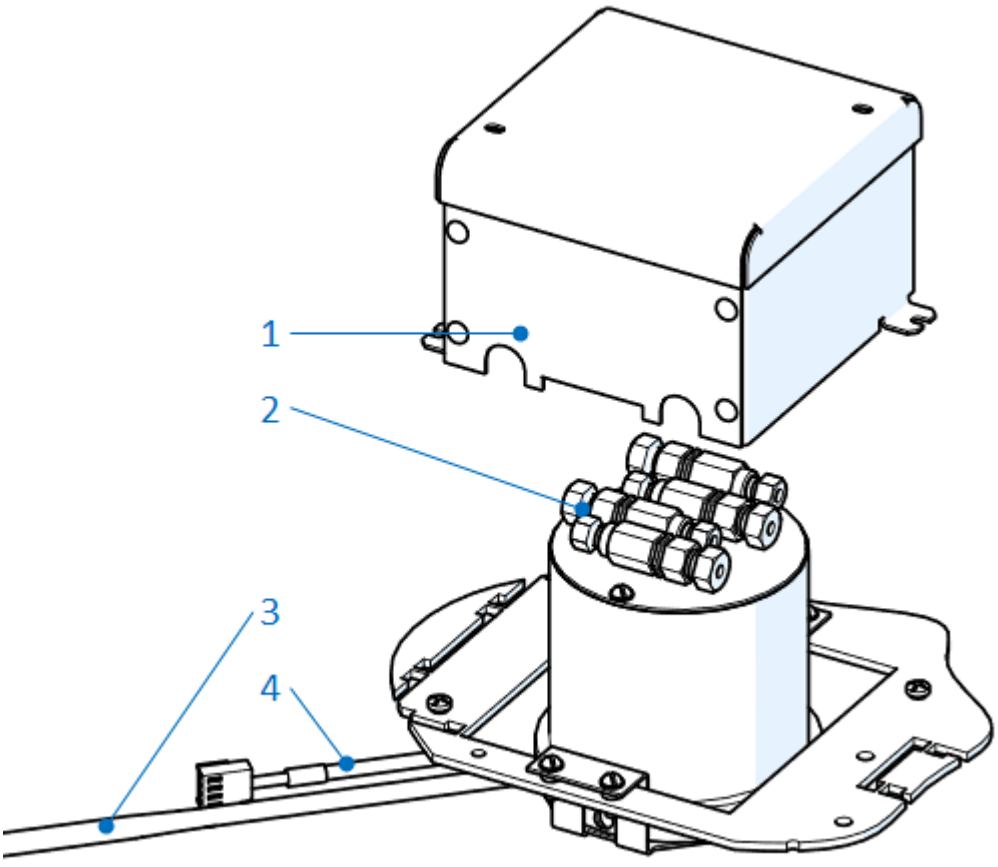
3.3.3 Auxiliary column thermostat

Auxiliary column thermostat (assembly named as montage kit 4.075.124 (further – the thermostat) is intended for location of a packed column in a temperature zone independently of the oven.

Table 3.4 – Technical specifications of the thermostat

Parameter	**Value**
Temperature, °C	from 50 to 150
Thermostat temperature mode	isotherm
Column's configuration:	
– total length (OD of columns)	3m (1/8")
– diameter of column spiral, mm	70
– outer diameter of the columns, mm	3

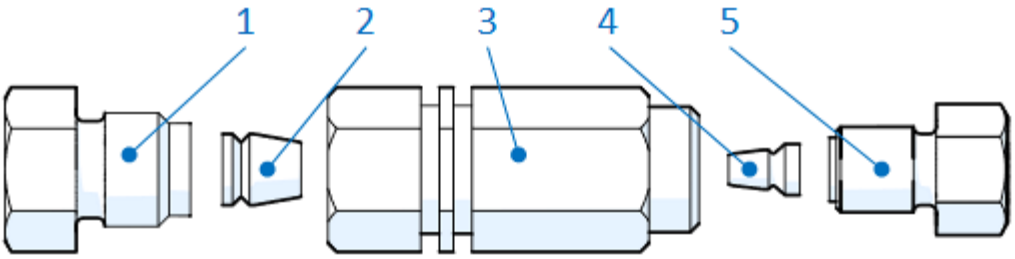
Thermostat is a chamber founded by two thermal protecting covers (Figure 3.17).



1 – Cover 6.430.0377, 2 – connectors 6.115.168-01, 3 – Heating element 5.863.164, temperature sensor 5.182.027-01

Figure 3.17 – Auxiliary column thermostat (Montage kit 4.075.124)

The design of connector 6.115.168-01 of the thermostat and the connection of the column and tube to it are shown below (Figure 3.18).



1 – Nut 8.930.114 (M8×1, brass); 2 – ferrule 8.658.038 (brass, Ø3 mm); 3 – casing 8.034.473-01 (12X18H10T); 4 – ferrule 8.658.039-01 (stanless steel, Ø1,6 mm); 5 – nut 8.930.161 (M6×0,75, brass).

Figure 3.18 – Connector 6.115.168-01

1/8" Stainless steel packed columns located in the thermostat have special configuration in spiral (type 7 by manufacturer classification). Column contains nuts 1 and ferrules 2 at delivery.

When columns are replaced, check for leak shall be carried out.

Recommendations for the correct sealing of the tubes and the ends of the column are given in section 3.4.4. UM – Installation instructions.

To avoid peaks tailing in connection points, insert tube into connector up to the stop. Hold the tube in this position at tightening.

3.3.4 Methanizer

Methanizer is intended for reduction of carbon oxide and dioxide into methane with its subsequent detecting by flame ionization detector of gas chromatograph. Reduction (or conversion) of carbon oxides into methane is carried out on the nickel-based catalyst in hydrogen flow under high temperature.

Methanizer allows determining carbon oxides at their minimal contents to 1×10^{-4} %vol. at 1 ml sample volume.

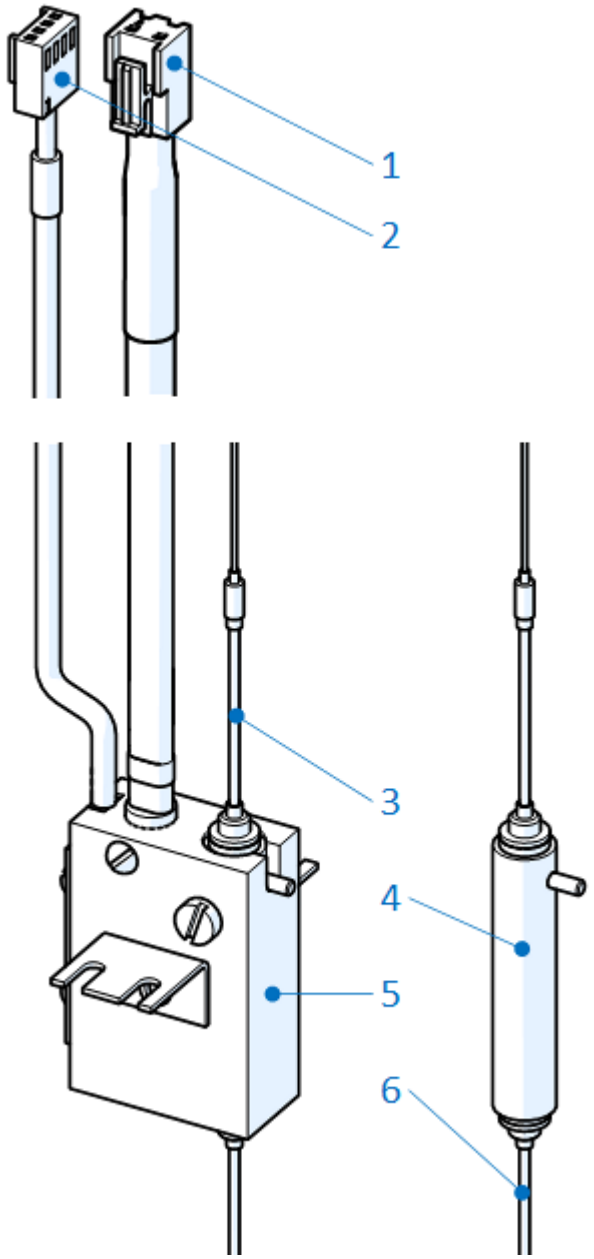
Methanizer represents heated metal tube filled with the catalyst.

Heating element and temperature sensor of methanizer are connected to power board, located in right compartment of the GC.

Methanizer 5.886.048 includes an all-welded non-disassembled reactor 5.884.109 filled with a catalyst. The carrier gas and hydrogen are mixed in a tee located in front of the metanator.

If two methanizers are built in the GC, each of them has its own heater. Arrangement of methanizer and channels of temperature regulation are defined according to chromatograph gas diagram.

Methanizer is connected to GC column outlet in flow before FID detector. Operation conditions for catalyst in methanizer are permanent hydrogen flow and keeping high temperature. Hydrogen flow shall be about (20-40) ml/min (optimal for FID). Detector's hydrogen input tube shall be sealed.



1 – heating element 5.863.166, 2 –temperature sensor 5.182.027, 3 – tube 6.457.116-01, 4 – reactor 5.884.109, 5 – casing 8.034.889, 6 – carrier-gas+hydrogen

Figure 3.19 – Methanizer 5.886.048 construction

Below (Figure 3.20) shows dependence of degree of carbon oxides reduction (conversion) from methanizer temperature. For simultaneous carbon oxides conversion, the temperature 325 °C is optimum.

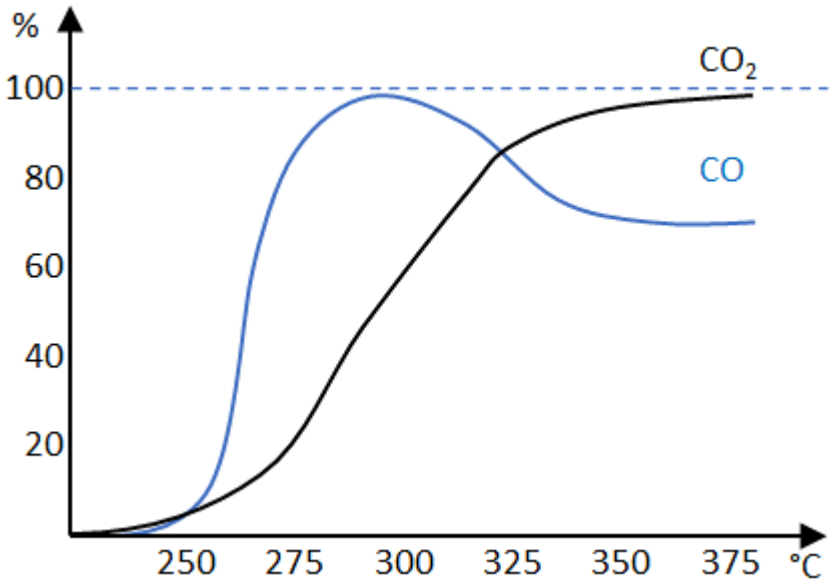


Figure 3.20 – Conversion – methanizer temperature diagram

High content of heavy hydrocarbons (exceeding C2), S-content components and other undesirable impurities in sample flow could lead to catalyst activity deterioration and to increasing of FID signal.

Catalyst regeneration

If significant FID signal (higher 300 – 400 mV) is observed and sensitivity by carbon oxide and carbon dioxide has been decreased, catalyst in methanizer should be regenerated. Regeneration procedure consists of two consequent steps: first oxidation in air flow, then reduction in hydrogen flow.

Catalyst oxidation is carried out as follows:

- Reduce carrier gas flow through the column down to 10 ml/min;
- To side tube of methanizer connect air source from DFC instead of hydrogen source, set 50 – 80 ml/min of air flow;
- Increase methanizer temperature up to (395–400) ˚C, keep for 2-3 hours;

Oxidation is carried out in "Test mode", so FID flame is not lighted.

Catalyst reduction is carried out as follows:

- Reconnect hydrogen gas line instead of air, set working flow rate for hydrogen;

Set temperature (395–400) ˚C in methanizer and keep for 4-6 hours until FID signal becomes even;

If described procedure of methanizer doesn’t help, the catalyst is must be changed. Methanizer with new catalyst should be regenerated as described above.

High FID signal with a methanizer can be observed in the presence of wet hydrogen.

3.4 Gas lines and flow splitters

In this section "passive" (non-functioned) pneumatic elements are viewed: tubes, collectors, tee-joints (tees), flow splitters, using which internal units are connected between each other and operate in the GC.

Gas lines are formed by tubes; flow splitters, tee-joints and collectors serve for splitting and junction of gas flows.

3.4.1 Tubes and gas line connections

External gas lines are described in section 3.4 of User manual. Part "Installation instructions".

Internal gas lines are formed mainly by stainless steel tubing of 1.02 mm ID, 1.6 mm OD (1/16").

If necessary to make gas line longer or to joint tubes with different diameter, proper connectors are available (see logbook and part 3 of User Manual).

For special applications, the sample gas line can be made of inert material or stainless steel with an inert inner lining. The tube material is selected by the manufacturer depending on the analytical task.

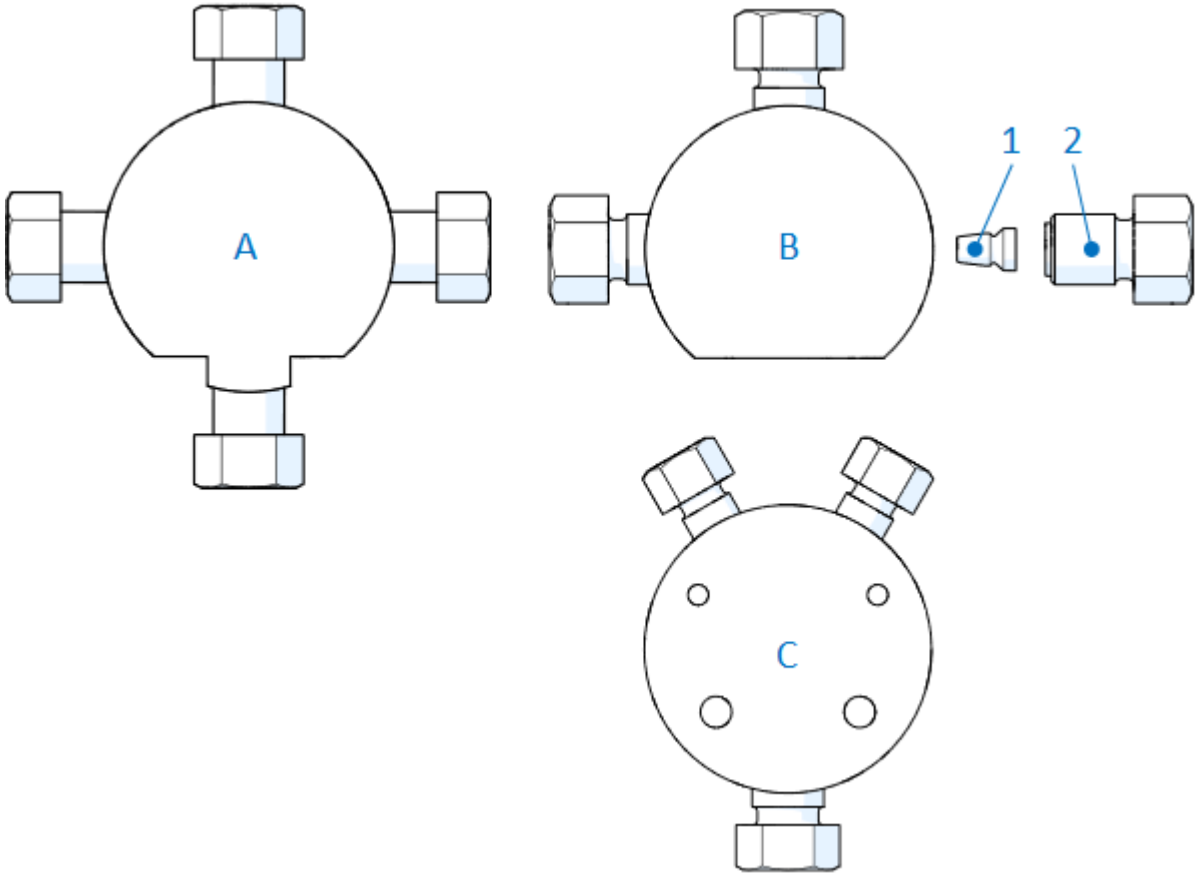
If it is necessary to extend the tube or to connect tubes of different diameters in the pipeline, appropriate adapters are used (supplied in the spare parts kit or on request).

See section 3.4.4 for proper sealing of tubing and column User manual. Part "Installation instructions".

3.4.2 Collectors and tee-joints

Collectors and tee-joints serve for splitting of external input gas flow, for splitting and junction of gas flows inside the GC.

Collectors used in the GC are described below:



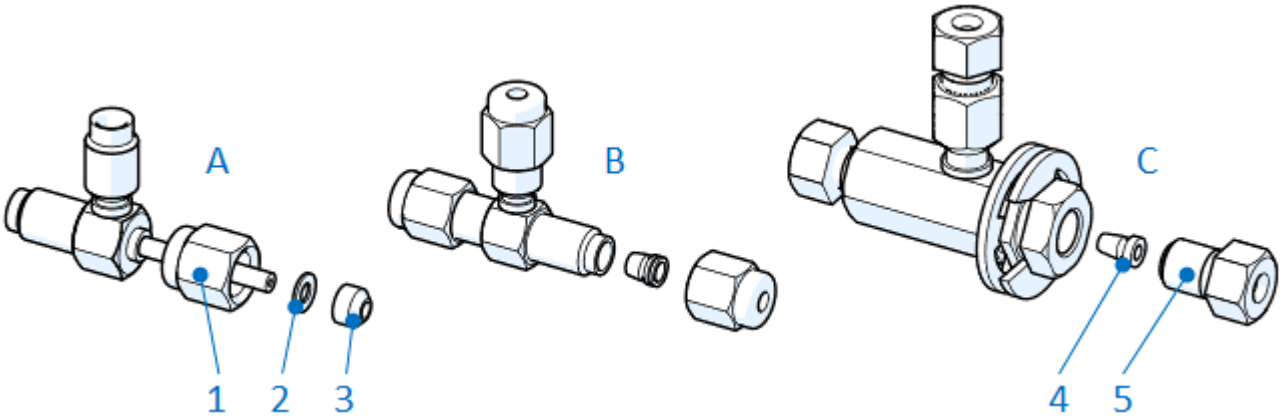
Collectors: A – 6.617.013, B – 6.617.014, C – 6.617.015; 1 – ferrule 8.658.039 (brass); 2 – nut 8.930.161

Figure 3.21 – Collectors

Collectors 6.617.015 are attached in rear cover of the GC with the help of 2 screws M3×16 and 2 sleeves 8.223.070-02.

Table 3.5 – Collectors. Description

Part Number	Ports	Body material	Thread, port ID	Nuts, ferrules
6.617.013	4	brass	M6x0.75	Nut 8.930.161 Ferrule 8.658.039 (brass)
6.617.013-01	4	stainless steel	M6x0.75	
6.617.014	3	brass	M6x0.75	
6.617.014-01	3	stainless steel	M6x0.75	
6.617.015	3	brass	Input (1port): M8x1 (3mm) Output (2ports): M6x0.75 (1,6mm)	Input: Nut 8.930.114, Ferrule 8.220.272-01 (rubber)
6.617.015-01	5	brass	Input (1port): M8x1 (3mm) Output (4ports): M6x0.75 (1,6mm)	Output: Nut 8.930.161, Ferrule 8.658.053 (rubber)
6.617.015-02	3	brass	Input (1port): M8x1 (3mm) Output (2ports): M6x0.75 (1,6mm)	Input: Nut 8.930.114, Ferrule 8.658.038-02 (brass)
6.617.015-03	5	brass	Input (1): M8x1 (3mm) Output (4): M6x0.75 (1,6mm)	Output: Nut 8.930.161, Ferrule 8.658.039 (brass)



A) 6.453.148, B) 6.454.022, C) 6.453.094

1 – Nut 8.930.167, 2 – Washer 3, 3 – Ferrule 8.220.272-01, 4 – Ferrule 8.658.038, 5 – Nut 8.930.161

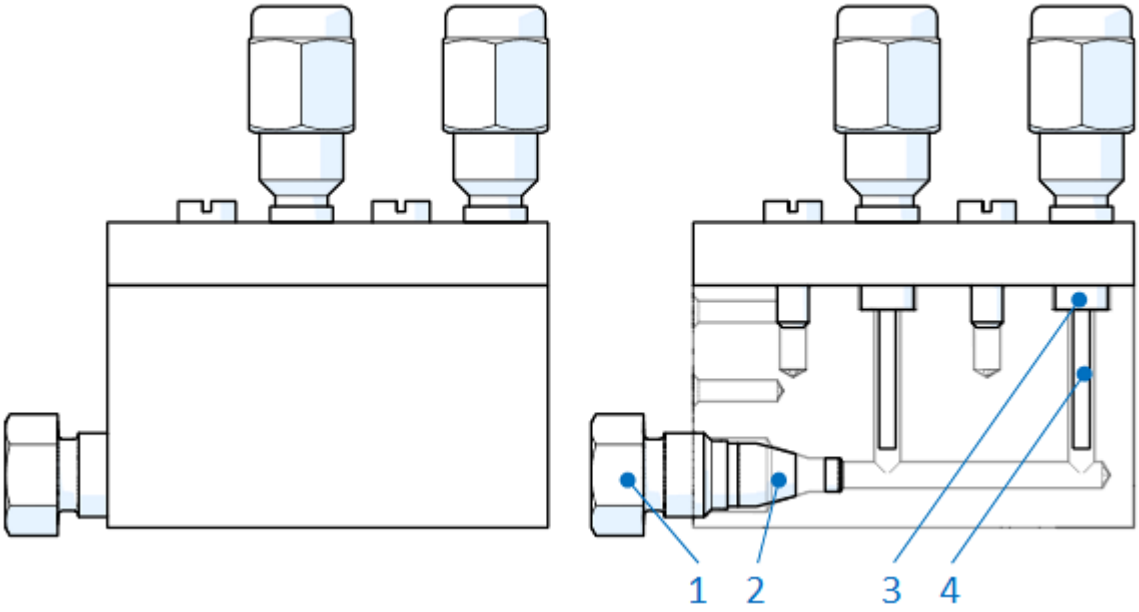
Figure 3.22 – Tee-joints

Tee-joints 6.454.022 and 6.453.148 are used mainly for outside gas lines connection (3 mm OD tubing).

Tee-joint 6.453.094 has female ports, thread M6x0,75 for tubing 1.6mm OD. Tubes connection is carried out using nuts 8.930.161 and ferrules 8.658.039 (brass) or 8.658.053 (rubber). Tee-joint 6.454.094-01 has opposite female ports, thread M8x1 for tubing 3mm OD (connection with nuts 8.930.114 and ferrules 8.658.038 or 8.220.272-01) and one side female port M6x0.75 for tubing 1,6mm OD. Nuts and ferrules are not supplied on default (available by order).

3.4.3 Flow splitters

Flow splitters are used in the GC for simultaneous feeding two flame detectors (FID-FID or FID-NPD) with fuel gases from single hydrogen DFC or air DFC.



a) Flow splitter 5.885.010-01 (air), 5.885.010 (hydrogen) 5.885.010-02 (make up) has the same design. b) Flow splitter 5.885.016 (hydrogen).

1 – nut 8.930.161; 2 – ferrule 8.568.039-01; 3 – tube 7.352.013-01 (capillary for air flow splitter) or 7.352.013 (capillary for hydrogen flow splitter); 4 – ferrule 8.220.009.

Figure 3.23 – Flow splitters

For flow splitters 5.885.010(-01) gas flow ratio in each outlet is equal (1:1).

For flow splitter 5.885.016 gas flow ratio is 2:1, outlet with two capillaries have double flow rate comparatively to another outlet. Such flow splitter is used for simultaneous feeding tandem FID and NPD detectors.

Flow splitter outputs are connected to 1.6mm OD tubes with the help of nuts 8.930.167-02, washers and ferrules 8.658.053.

3.4.4 Gas lines connections check for leaks

Leak test of outside gas lines is described in current User Manual, Installation Instructions part, section 3.6 "Feeding gas lines check for leaks". This section describes how to make leak test for gas lines inside the GC.

The column oven temperature must be reduced to 40 °C (or lower) before starting the check.

Signs of leaks appearance are:

- Excessive carrier gas consumption;
- Component's retention time increasing;
- Poor repeatability of analyses results.

While working, the main cause of leaks is worn septum of inlet.

Gas lines may be classified as follows:

• Lines that are designed for excessive gas pressure and can be plugged. For example, the output of TCD. This output can be plugged and the tightness of the system can be checked from the DFC to the detector output.

• Lines that are under gas pressure close to atmospheric. For example, the output of the column to FID detector

First type lines can be checked for leaks with the help of digital gas flow controllers (DFC). Before checking it is necessary to make sure that both gas cylinder regulator and digital flow controller intended for checking are correct in work.

Using DFC for leaks check is preferable to the other methods because it avoids soap liquids which can flow in fitting joints and then contaminate gas lines. Besides, such way allows evaluating leakage rate quantitatively.

When using MSD, PDD, CCD detectors, even insignificant leak may cause detrimental increasing of detectors signal and features deterioration due to oxygen diffusion from ambient into gas lines. Such leaks are impossible to be revealed using DFPCs, commercial leak detectors are recommended for this purpose instead (for example, Agilent Flow Tracker 2000, Leak Mode, Restek Leak Detective II™ Electronic Leak Detector and similar).

The point of leak can be revealed, by rearranging plugs and caps, sequentially excluding certain gas lines sections, or tightening sealing of gas lines in connections where leak is possible, and simultaneous controlling of DFC indication.

Small leaks can be detected by dripping ethyl alcohol onto the joints – the likely places of leakage.If point od leak has been found, it shall be eliminated by retightening proper connections and, if necessary, by replacement of sealing elements. Detailed information about gas lines connections is given in section 3.4.1.

When tightening connections, do not put force exceeding recommended value, specified in corresponding UM sections.

While leaks check is carried out, it is recommended to increase pressure in gas lines up to working values (0.4-0.64) MPa.

Regulator checking:

The procedure is described in section 3.4.2. User Manual, Installation Instructions part. Regulator on the cylinder should not leak when its regulating spring is released (usually when the adjusting screw of the reducer is fully unscrewed counterclockwise).

DFC checking

- There should be no pressure in gas line before DFC. Switch GC on, do not set any mode on DFC. In 10 minutes, monitor DFC flow rate indication, it shouldn't exceed 0,3ml/min (with no gas set). This indication meets to "electrical zero" of DFC. Registered rate of "electrical zero" is to be subtracted from leaks indications obtained in future leaks checking. Take into account that if "electrical zero" was registered as 0 ml/min, the true "electrical zero" may be in negative side. Therefore, using such regulator for leaks check may understate real leaks;
- Seal DFC output with cap 6.432.002 from shipping kit;
- Set working pressure on gas cylinder (0,4±0,04) MPa;
- set flow 100 ml/min on DFC;
- In 10 min register flow rate indication on DFC. It should not exceed "electrical zero" rate for DFC. Excessive floe rate indicates leaks in DFC.

Gas diagram for check (Figure 3.24):

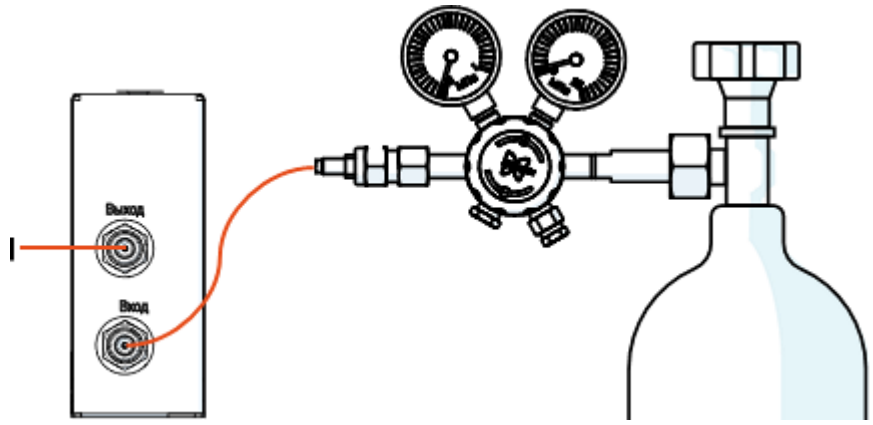


Figure 3.24 – DFC checking diagram

Leaks check using DFC

- Set the plug on output of gas line or device;
- Control gas pressure on the regulator pressure gage. Carrier gas pressure should match nominal for GC (0,4±0,04) MPa;
- Set the flow on the flow controller not less than 30 ml/min. After gas inleakage into closed capacity has been got, the DFC flow indication should be no more than (0.5-0.8) ml/min. When exceeding it is recommended to tighten possible leak points, or consequently exclude them and define the cause.

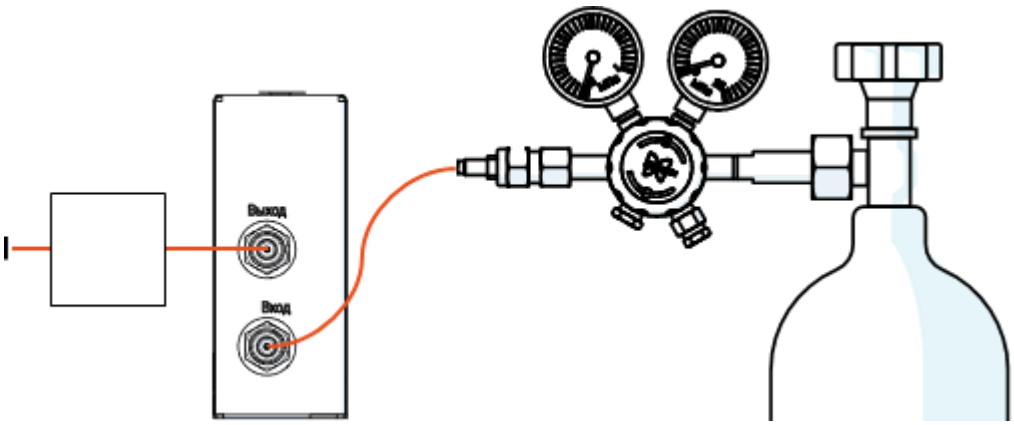


Figure 3.25 – Leaks check using DFC

When providing described procedures, take attention at following points:

- If there is significant volume of checked lines, traps or columns filled with Mol.Sieve, the time of gas inleakage can reach as long as 30 minutes.
- Using helium as a carrier gas can increase inleakage time comparing to the other gases.
- The inleakage time is possible to reduce when disconnecting the carrier gas after gas cylinder regulator with additional valve.

Using DFC check for leaks can be carried out for inlets, sampling and switching valves, as well as for external samplers not containing DFC.

TCD detector lines can be checked for leaks together with connected columns, when output sealed with plugs.

When TCD checking for leaks it is necessary to switch filaments into state "Off".

When using capillary columns with TCD, it's inadmissible to check leaks by sealing TCD outputs with plugs. High pressure may damage quartz column.

Lines which are under gas pressure close to atmospheric can be checked only at special devices availability. In some cases is inexpedient to put in "absolutely leak proof" seals which considerably complicate construction and increase overall dimensions.

Leaks in such lines and devices are excluded by constructive decisions: by corresponding pipelines passage section, by sealing joints shape.

Leakproofness of such lines is offered to be sized up on background signal and noise level of detectors on chromatogram.

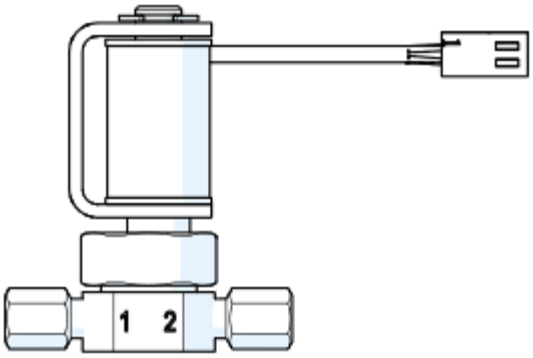
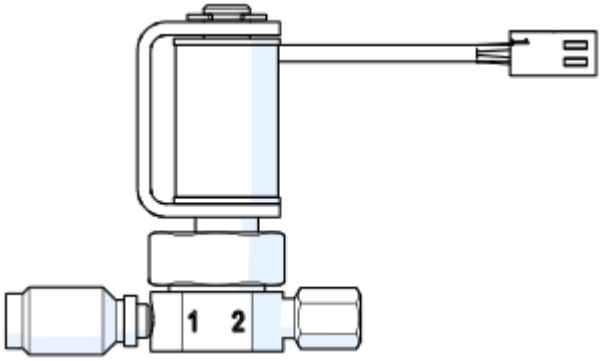
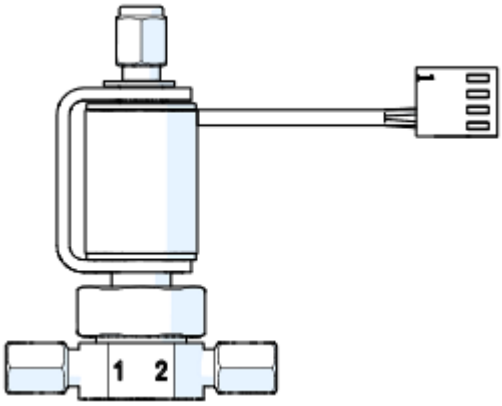
3.5 Solenoid valves

Solenoid valves are used in the GC for flow switching:

- at backflushing of for-column or inlet;
- at operating Dean switcher.

Connection of gas lines is provided according to particular gas diagram of the GC. Types and part numbers of solenoid valves are given below.

Table 3.6 – Solenoid valves. Description

Overview	Part.N	Number of ports	Ports features
	5.890.042-03	2	input (1) / output (2): Thread Female M6×0.75 For tube 1.6mm
	5.890.042-04	2	input (1): Thread Male M8×1 For tube 3mm output (2): Thread Female M6×0.75 For tube 1.6mm
	5.890.043-03	3	input (1)/ output (2, 3): Thread Female M6×0.75 For tube 1.6mm

Specified solenoid valves are being used in the GC with PM3 electronics and not suitable for previous PM2 electronics. Valves may work as in "normally-open" and in "normally-closed" position.

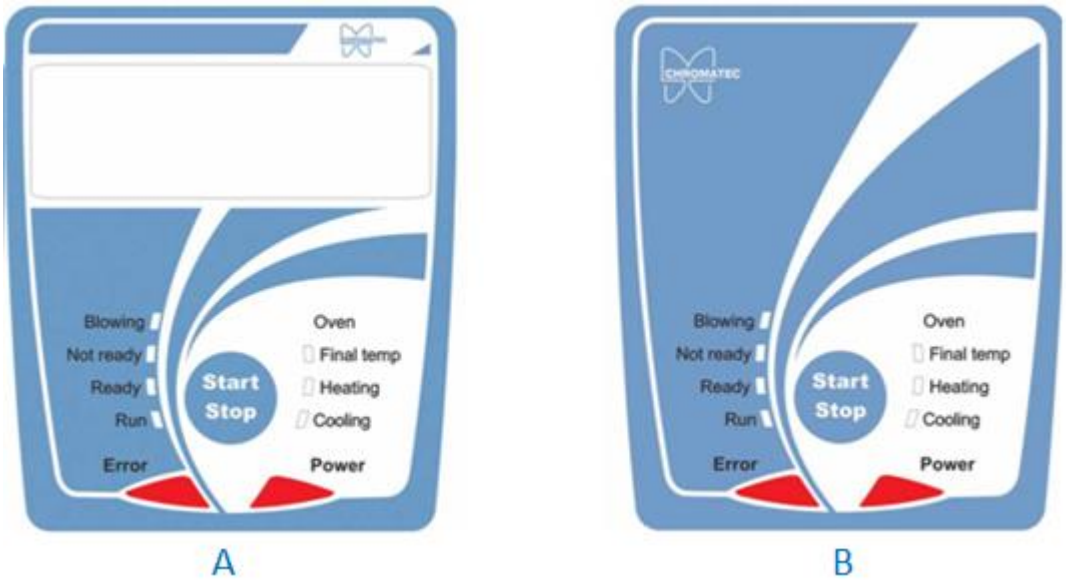
The maximum operating pressure of the valves is 300 kPa.

3.6 GC control panel

Control panels of GC C5000 both 1 and 2 versions have some common elements of control and status, located on status board:

- LED indicators;
- START/STOP button.

LED indicators of status board (Figure 3.26) show current state of the GC. GC C5000.1 also has a four-line text display.



a) C5000.1 status board. b) C5000.2, C9000 status board.

Figure 3.26 – Display and LED status board

All the models have **Start/Stop** button on status board, which initials or interrupts analyses.

Besides, additional buttons Start 1 and Start 2 are located on the top cover of the GC next to inlets (Figure 3.5). These intended for Start/Stop analyses when independent channels 1 and 2 are in force. Additional Start 1 button is absolutely equal to status board Start/Stop by function.

3.7 PM3 electronics

Processor module PM3 is a basis of GC electronics. GC electronics provides complete operation of the instrument, units built-in and programming interaction with external devices. The main features are full electronic control of operating parameters, Control of temperature zones, electronic (digital) control of gas flow and pressure, diagnostics and control of paramteters and failures, reserve chromatogram storage, programming operation with PC and connected external devices.

3.7.1 Board's location

Table 3.7 – Boards supplied with the controller in the GC

Part N	**Board or unit**	**Supplied with PM3 controller**	**Location**
214.2.087.030	Power unit	On default	In rear compartment
214.3.030.014	Microprocessor controller	On default	In right compartment
214.5.428.009	Power board	On default	In rear compartment
214.5.081.070	Module DFPC 1.x	Depending on configuration	In right compartment
214.5.081.058	Module DFPC 2.x	Depending on configuration	In right compartment
214.5.123.014-01	Stabilizer 200 V	Depending on configuration	In right compartment
214.5.123.015-01	Stabilizer 800 V	Depending on configuration	In right compartment
214.5.123.013-01	Stabilizer 3 mA	Depending on configuration	In right compartment
	Amplifiers	Depending on configuration	In right compartment
214.6.730.157	Variator	On default	In rear compartment

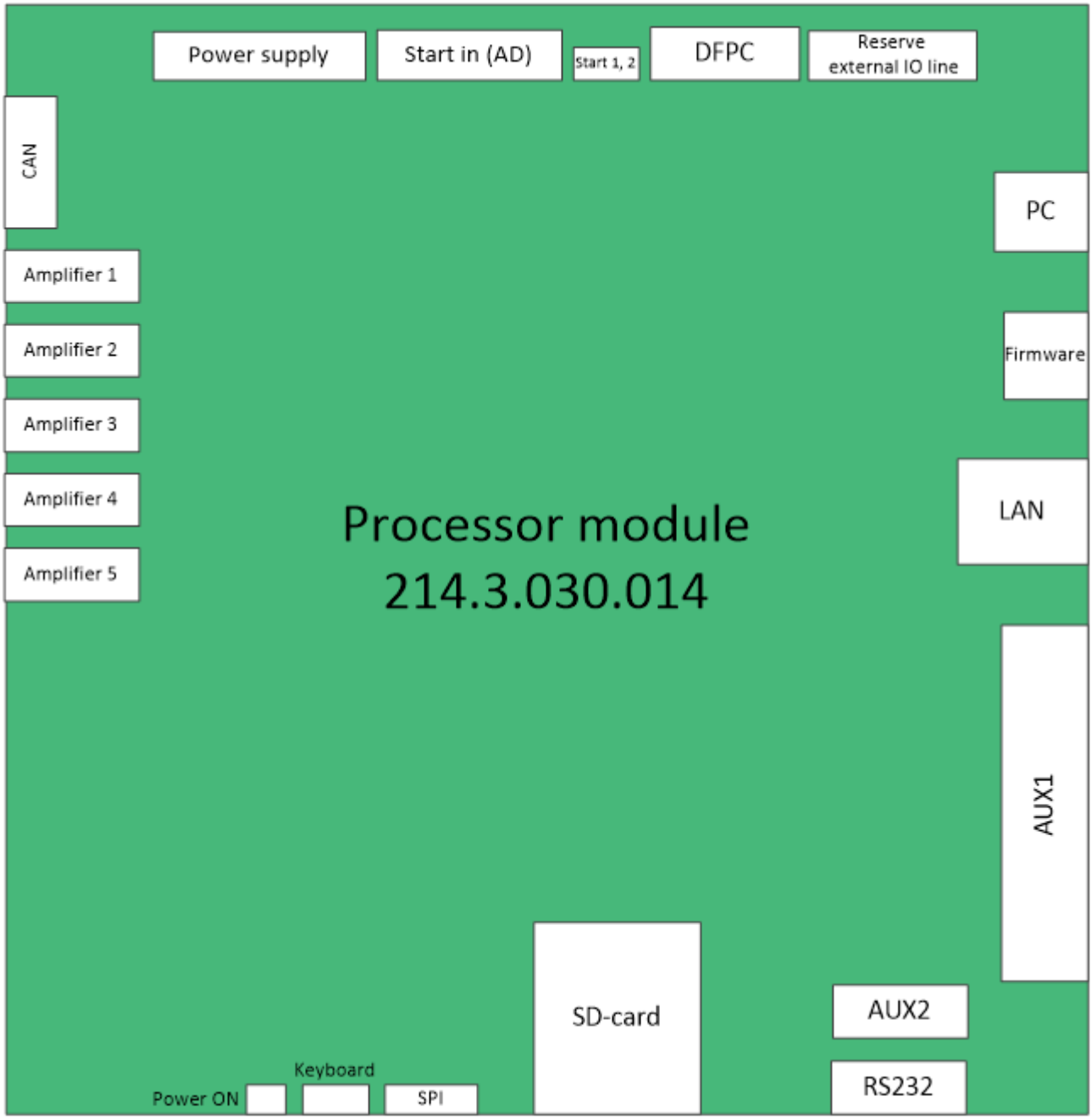


Figure 3.27 – Location of slots on Processor controller

Table 3.8 – Microprocessor controller slots description

Marking	Description
Power board	Connection to power board. Counterpart in power board is marked as CAN bus, see Figure 3.28.
Power	Controller's power supply.
AD	Slot for connection Start In/Ready out channel to external device (3rd party manufactured detectors and samplers). Connected to outside slots: Detector (Ready out), Sampler (Start in)
Start 1,2	Connection of auxiliary start buttons.
DFPC	DFPC board connection. If there are several DFPC boards, theses shall be connected by means of common cable with several counterparts
Reserve internal I/O lines	Auxiliary digital inputs/outputs
Firmware	Connection USB-flash (flash-memory) for updating GC firmware
LAN	Connection GC to computer by Ethernet interface.
AUX1, AUX2	Connections to Chromatec-made peripherals to the GC
RS-232	Connection GC to computer by RS232 interface
SD-card	SD memory card. Is used for storing GC configuration and chromatogram backup copies
SPI	Standby interface
Keyboard	GC keyboard connection
Power on	Slot for connection on/off button
Amplifier	Amplifiers connection

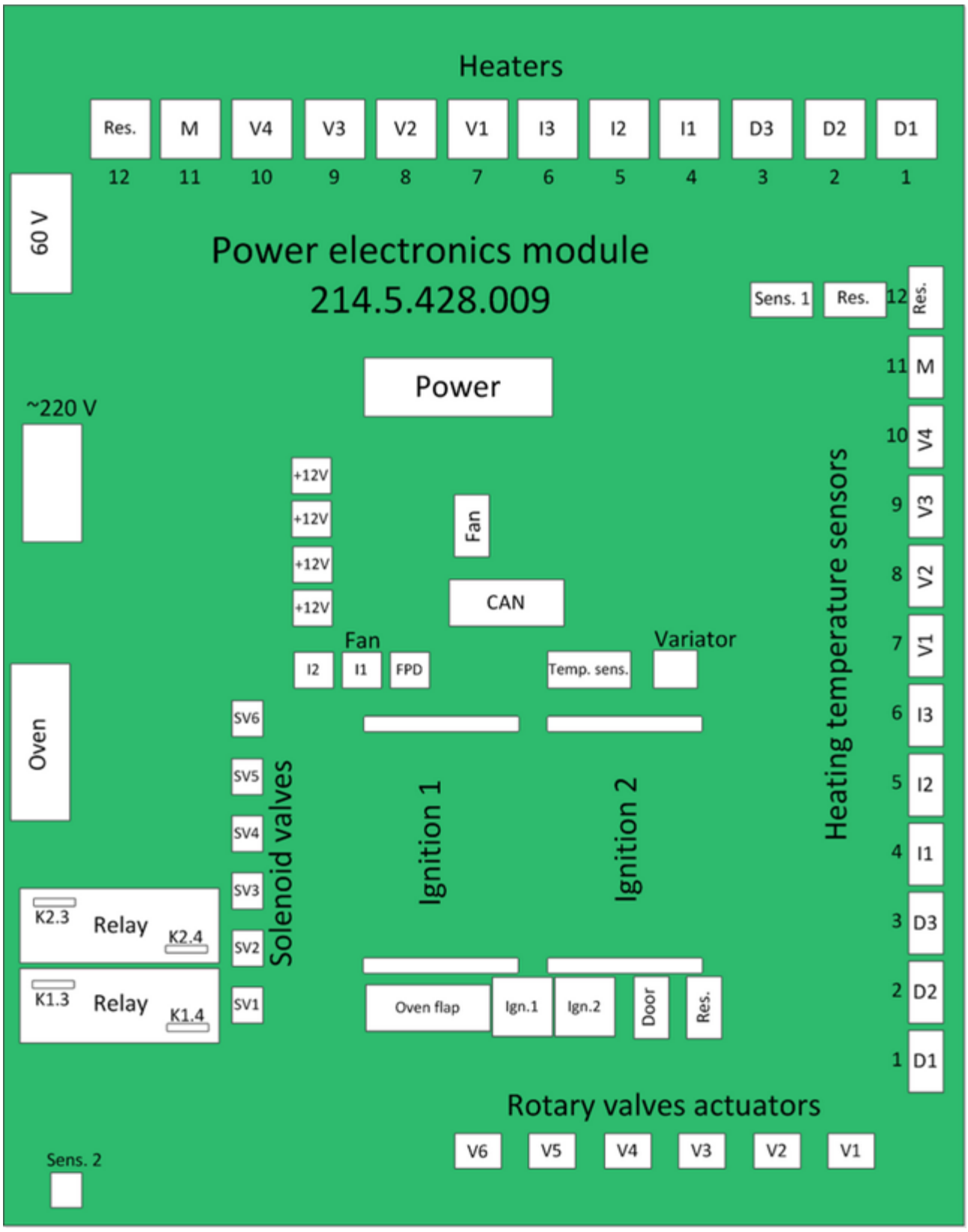


Figure 3.28 – Location of slots on power board

Table 3.9 – Power board slots description

Marking	Description
Oven	Oven heating element connection
220 V	Input voltage 220V for oven heater
60 V	Input voltage 60V for other heating elements
Heating elements	Slots for heating elements and corresponding temperature sensors. Purpose of slots on default: 1, 2, 3 (D1, D2, D3) –detectors thermostats 4, 5, 6 (I1, I2, I3) – inlets thermostats 7, 8, 9, 10 (V1, V2, V3, V4) –valves thermostats 11 (M) – controlling methanizer thermostats 12 (Res.) – auxiliary thermostat, like 2nd methanizer, etc. Res.Sens – auxiliary (reserve) temperature sensor (without corresponding heating element) 14 (Sens.1) – oven temperature sensor
V1…6	Rotary valves actuators connection
SV1…SV6	Solenoid valves connection
Oven flap	Oven flaps actuator connection
Ign.1, Ign.2	Connecting ignition wires for flame detectors
Door	Oven door opening sensor
Res.	Auxiliary sensor
Ignition1, Ignition2	Ignition micro-boards connection
Variator	Variator board connection
Amb.temp	Ambient temperature sensor
PWR. Fan	Rear compartment Fan
Fan I1, I2	PTV Inlet cooling fan connection
Fan FPD	FPD fan connection
CAN	CAN bus to PM3 processor controller connection

12 heating channels are actually multi-purpose. If any unit not stipulated for connection into intended slots is necessary to connect (methanizer 2, for example), it can be connected to any vacant heater (e.g. connect to H10 if there is no Valve 4 in the GC). Then rename heating element and corresponding temperature sensor in software (see section 0, subsection **Power board setting**).

Slots numeration determines number of hardware object. For example, of valve actuator is connected into slot **Кр3** (Valve3), thus in hardware object **Valve 3** will be created, even though there is no Valves 1 and 2.

There are possible 2 types of gas controllers operating with PM3 controller:

- Functional gas controllers.

- Unified DFC-10 and DPC-11 (compatible with PM1 and PM2 as it is).

The type of controller connected to the PM31 controller is determined automatically. All DFPC are connected to the corresponding gas module.

The MPXA6115A6U atmospheric pressure sensor is installed on the module, which is used to compensate for pressure when calculating the flow and velocity of the carrier gas in the capillary column, as well as when calculating the volume of the gas sample when entering the metering valve.

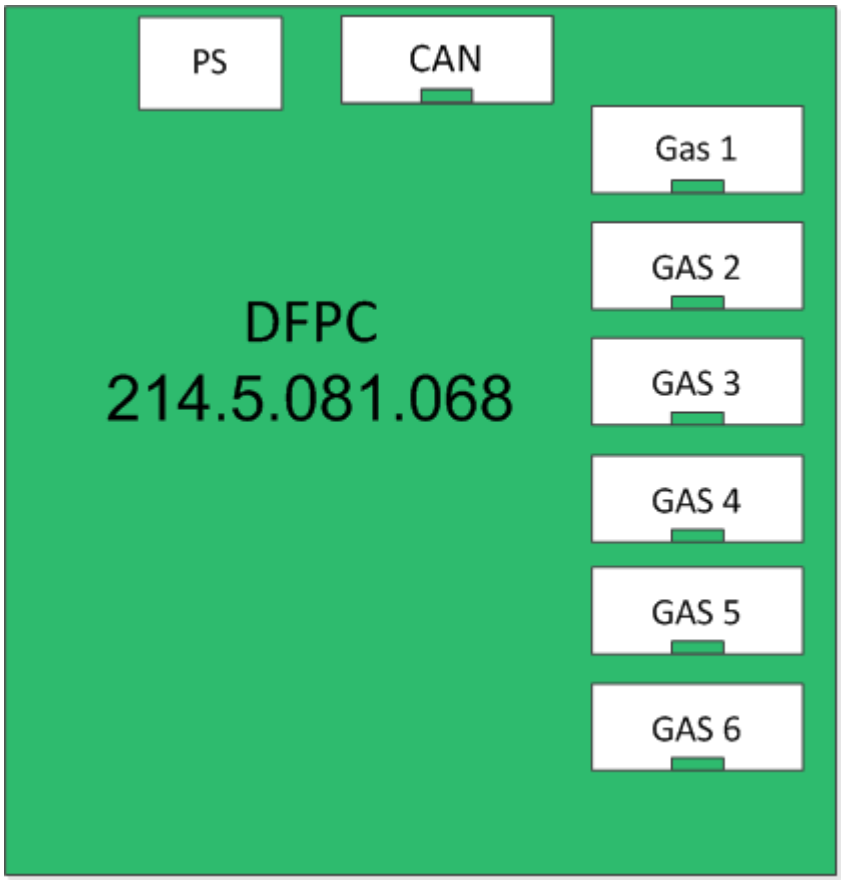


Figure 3.29 – Location of slots on DFPC board

Table 3.10 Description of slots marking

Marking	**Description**
Power	Power connection
CAN	connection to microprocessor controller
DFPC-1..DFPC6	Slots for DFPC connection

DFPC boards intended for functional and unified gas controllers are different. Functional gas controller cannot be connected into gas unified DFPC board and vice versa. Gas controllers of different types are different in size

If additional gas controllers are required to be connected in the GC and all the channels of DFPC controller are occupied, additional DFPC board can be installed into the GC. Both gas controllers are connected to microprocessor controller by common cable.

For the second DFPC board connected, gas channels Gas-1…Gas-6 shall be re-numerated as Gas-7…Gas-12 by software (see section 0, subsection **DFPC board setting**).

Depending on the task, up to 4 gas gas modules can be installed in the chromatograph. The total number of DFPC s in this case can reach 24. At the same time, 10 DFPC are installed on the analytical platform inside the chromatograph, and the rest can be mounted on an external bracket.

3.7.2 LED indicators

LED indicators are located on status board – see Figure 3.26.

Table 3.11 – LEDs indicate states of GC operation
● light is on, ○ light is off, [+/-] light is blinking

	Conditioning	**Not ready**	**Ready**	**Analysis**	**1**	**2**
No mode	○	○	○	○	○	○
Not ready	○	●	○	○	○	○
Waiting	○	◐	◐	○	◐	◐
Ignition	○	◐	○	○	○	○
Ready	○	○	●	○	●	●
Prep-conditioning	◐	◐	○	○	○	○
Conditionning	●	○	○	○	○	○
Gas saving	○	○	◐	○	○	○
Cooling	○	●	○	○	○	○
Analysis	○	○	○	●	○	○

LEDs 1 and 2, located next to Start 1/Start 2 buttons on the top cover, show state of corresponding channel. LEDs on GC status board show state of the channel 1.

LED Power lit whenever GC is on.

LED Check prm is on, if any parameter went out of admissible value range in any channel, any time except states "Initialization" and "No mode".

LED Heating is on, when oven temperature is going up.

LED Cooling is on, when oven temperature is going down.

LED Isotherm lit in state "Analysis", when oven temperature is constant.

If all LEDs blink simultaneously, this means internal hardware has been damaged at updating firmware (for example due to cutting-off while updating). In this case updating shall be carried out again (see section 0).

Brief description of the states of the chromatograph

Idle (no method). The state begins immediately after switching on the device and lasts until the method is transferred to the device in automatic or manual mode.

Not Ready. At the **Not Ready** state, the chromatograph performs the specified parameters (sets temperatures, flows, pressures).

Waiting. **Waiting** state occurs after **Not Ready** before entering the **Ready** stage, the duration of the stage is determined by the operator. The **Waiting** stage is either forced by the operator, or determined by the sequence of operation of programmable devices (valves, taps, column thermostat). To switch from **Waiting** to **Ready** when manually injecting a sample, press the Start button.

Gas savings. During the **Not Ready** and **Waiting** phase, the gas saving function is performed, the gas flows are automatically reduced or turned off. Carrier gas savings are performed for the sample waste channel (split), for the carrier gas channel (when working with a packed column), as well as when completing the inlets with the option of automatic septum purging). Gas saving parameters settings are set in the chromatograph instrumental mode for input devices.

Ignition. Ignite state ignites the flame detectors (FID, TID, FPD). At the stage of ignition, a special program of gas flows is carried out, corresponding to the optimal mode of ignition of the flame.

Ready. **Ready** state corresponds to the readiness of the instrument for analysis. The operator or automatic sampler injects the sample.

Analysis. At the **Analysis** state, the sample is injected into the chromatograph, separation and detection of the sample components are performed.

Prep Blowing. At this state, all instrument parameters are prepared for the next stage, **Blowing**.

Blowing. **Blowing** step conditions, the column. There are several circumstances under which the **Blowing** step is performed:

- **Blowing** before the start of work (comes automatically after turning on the device immediately after reaching the **Ready** stage);
- **Blowing** after analysis (occurs at the end of each analysis).

Cooling. The temperatures of all zones are reduced, while maintaining the gas flows set in the corresponding "Cooling" mode. This step is performed at the end of work before turning off the device.

Sleep mode. At the **Sleep mode** state, all temperature zones and gas flows are turned off. The transition from the **Sleep mode** state to work (**Not Ready** state) is carried out at the time specified by the operator.

3.7.3 Keyboard overview

- To scroll menu options on display use Arrow keys.
- To select parameters from suggested list use key SELECT.
- To input numerical values, use corresponding numeric part of keyboard.
- To come into menu marked with cursor or accept changed parameter, use key ENTER.
- To go out from dialogue to upper menu level use key BACK. Except from this key, there is a command with the same name in dialogues which has similar action, as well as command **Next**, which allows going to the next dialogue.

The following keys are intended for quick viewing and editing parameters:

- SIG – detectors signals
- DET – detectors parameters.
- INL – inlets parameters.
- TMP – thermostats and gases.
- AUX – parameters of auxiliary (external) devices, such as autosamplers
- VALVE – parameters of rotary valves and solenoid valves.

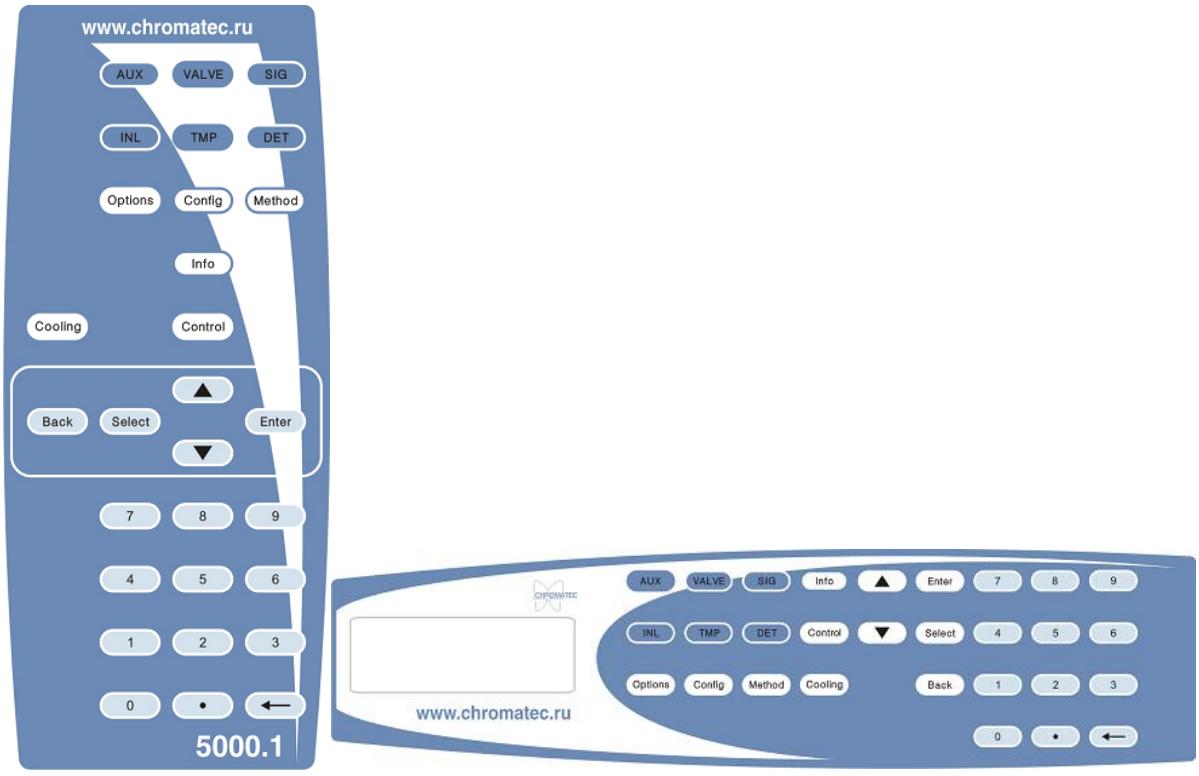


Figure 3.30 – Built-in keyboard (C5000.1) and external keyboard (C5000.2, C9000)

Detail description is in section 3.7.9.

The GCs C5000.2 and C9000 may be supplied with external keyboard, connected to slot next to on/off button.

3.7.4 CONTROL key menu

CONTROL key is a basic menu to access to all parts of GC firmware as well as auxiliary functions. User can find the following data:

Channels Channel 1 Not ready State time 12:25	Current status of channels of the GC.
Method	Alternative entry into METHOD key menu, see section 3.7.5 for details.
Parameters view	Entry into menu to review parameters of all devices.
Thermostats	Entry into menu to check thermostats current state.
FID-1 150.0/150.0 TCD-1 160.0/155.5 Inl-1 100.0/76.6 Column 50.0/50.0	State is shown as: "set" / "measured".
Gases	Entry into menu to check gas controller's current state.
DFPC 1:0 20.0/20.0 DFPC 1:1 200.0/200.0 DFPC 1:2 20.0/19,3 DFPC 7 30.0/30.0 DFPC 8 25.5/24.1	State is shown as: "set" / "measured". For multichannel gas controllers after colon number if its channel displayed.
Signal	Alternative entry into SIG key (Signal) to review detectors signals.
Configuration	Entry into KONFIG key – (Configuration), see section 3.7.6 for details.
Updating	Entry into menu of GC firmware updating.
Center GM 00930001 FID 00130030 TCD 00145201 PB 00130021 MDFPC 00300013	Operator may update firmware for these modules. Center – basic firmware of the GC. GM – gas module. PB – power board module. MDFPC – multi-channel DFPC.

Other devices in the list are detectors, for amplifiers setting.

Number specified after each device is its serial number.

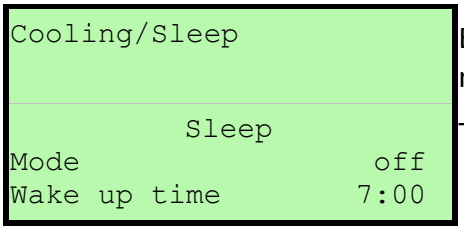
To update firmware, load a file of new version into removable device USB-flash. Disconnect USB-flash from PC and connect it to "Firmware" slot on the rear wall of the GC. Then select appropriate command in this menu.

File of the basic firmware (Center) is named as **Image.pm3**.

Once updating completed, the GC will restart automatically.

If electric supply was broken while updating is in process, after power supply restoration reserved firmware will automatically run. At this, all LEDs will blink simultaneously.

After reserved firmware running, user can repeat updating firmware. GC configuration, being set in advance, will be saved.

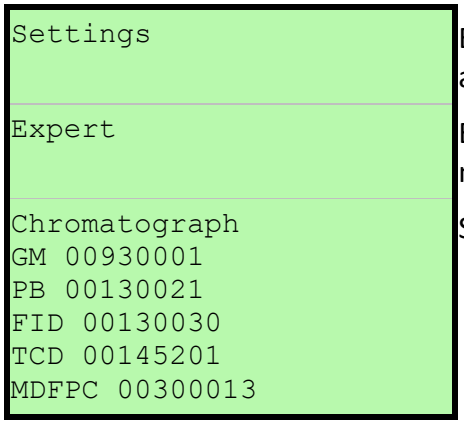


Entry into COOLING key to set and start "Cooling" and "Sleep" mode.

To start "Sleep" mode in the GC, turn on appropriate mode.

The GC will come to low power consuming condition. Gas control and heating thermostats shall be turned off. In fact, the power will be consumed for current time tracing. At setting Wake up time the GC shall perform mode specified in the menu OPTIONS (Autoload method, see section 3.7.7).

"Sleep" mode is convenient to use, if the GC must be ready for operation (being in "Ready" state) by the beginning of working day. The GC shall be not turned off in this case.

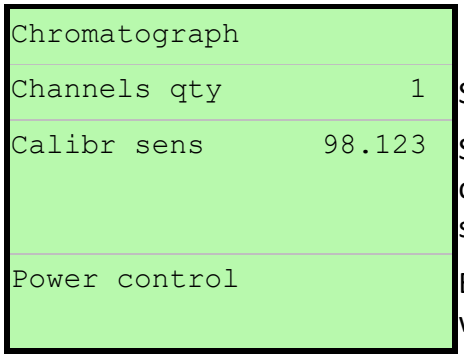


Entry into "Settings" menu, can be carried out by OPTIONS key as well, see section 3.7.7 for details.

Entry into menu for Expert settings, typically performed at manufacturing.

Select device in the list to set its parameters.

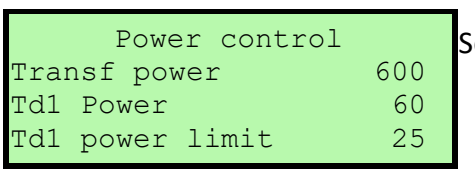
GC settings:



See section 3.7.6 for details about channels

Specify here ambient pressure value (kPa) measured by certified barometer. Indication of pressure sensor of the GC shall be corrected according to specified value.

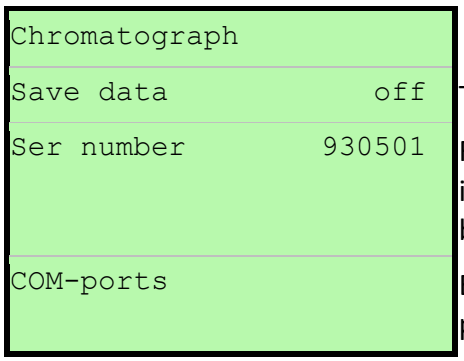
Entry into menu for limitation of heaters power (operation with UPS of limited power).



Set power of transformer in the GC.

Nominal power (W) and power limit (W) can be set for all listed heaters.

Power limit allows reducing power consumption of the GC and increase heaters lifetime. Heating rate of thermostats will be decreased respectively.



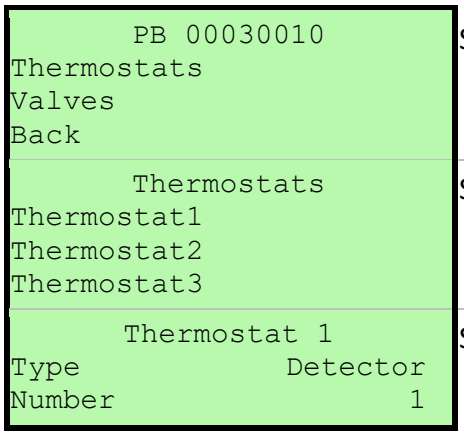
This option allows saving chromatograms in GC memory.

Firmware allows changing Serial number of the main board, if it's different from the GC serial number (required at main board replacement).

Entry into menu of external ports control responsible for peripherals connection.

Power board setting:

Pass to menu PB xxxxxxxx (here xxxxxxxx is Power board serial number) and press ENTER.



Select **Thermostats** dialogue.

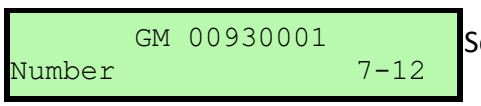
Select Thermostat to set it, e.g., **Thermostat 1**.

Set type and number for thermostat.

User can re-determine temperature zones of the GC, for example, to create temperature zone for 4th detector. This setting depends on particular configuration of temperature zones and never changed in practice. For most cases configuration on default is suitable.

DFPC board setting:

Select the menu GM xxxxxxxx (here xxxxxxxx – is serial number of Gas Module board) and press ENTER.



Select numbers of DFPCs connected to this board.

Possible variants:

- 1-6
- 7-12

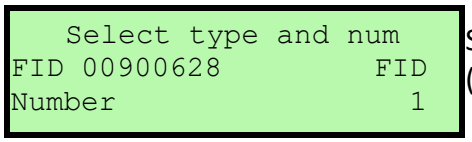
For example, if the GC has DFPCs in quantity of 11, while DFPCs from 1 to 6 are connected to Gas module 0930001, and DFPCs from 7 to 11 are connected to Gas module 0930003. For this case the following configuration shall be selected:

- GM 0930001 1-6
- GM 0930003 7-12

When service engineer connects DFPC cables to jacks on the gas module board, he must observe for board serial number (painted on the board).

Amplifiers setting:

Select menu of the detector, which amplifiers shall be set, for example FID xxxxxxxx (here xxxxxxxx – is serial number of amplifier) and press ENTER.

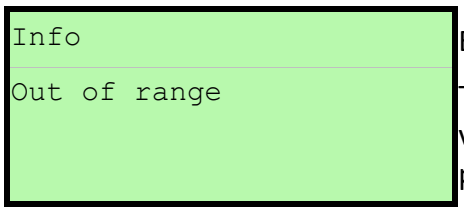


Select type of amplifier (FID, NPD, PID) and its order number (FID-1, FID-2 etc).

The order number can be specified for any other amplifier, for example – FPD XXXXXX.

Amplifier type can be set only for detectors FID, NPD and PID as soon as unified amplifier in design is used for these three types of detectors.

Amplifier order number determines detector channel in Chromatec Analytic SW.



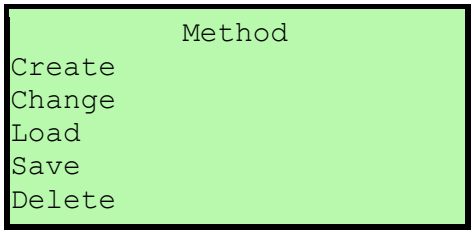
Entry into menu of INFO key, see section 3.7.8 for details.

This menu displays parameters which doesn't meet to set value. Until there is any parameter "out of range", red Check prm LED indicator lights on the status board.

3.7.5 METHOD key menu

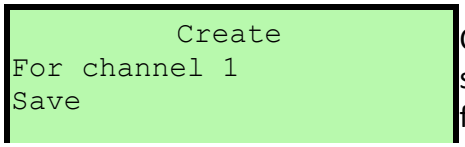
In the context words "mode" and "method" are synonyms.

To work with GC mode, use key METHOD.



Mode (method) creation:

Use the **Create** option and press key ENTER.



Choose a channel for the method to be created. If there are several channels in the GC, individual method can be created for each channel.

```
 Start 1. Create method
Analysis, min          10
Waiting, min            0
Conditioning          off
```

Set Analysis time, waiting time and set the Conditioning on if it's required after each analysis.

```
FID-1
TCD-1
Inlet-1
Oven-1
```

List of devices in the GC is displayed below.

Waiting time determines how long the state "Waiting" lasts. This state starts after the state "Not ready", when all parameters meet to set value provided that waiting time is not nil. This state can be used as a delay before the state "Ready".

Waiting time is helpful to set if there is inadmissibly high signal drift by the state "Ready". Set waiting time more than time required for signal stabilization, in this case the state "Ready" starts with appropriate signal drift. Signal stabilization time can be determined by experiment.

When **Preprun** option is on, the state "Waiting" is not limited by time, though waiting time may be specified. In this case the state "Waiting" will last until user or scheduler performs **Preprun**.

Option **Conditioning** allows additional oven temperature programming once analysis terminated. If Conditioning after analysis is required, set this option on, then set temperature programming parameters.

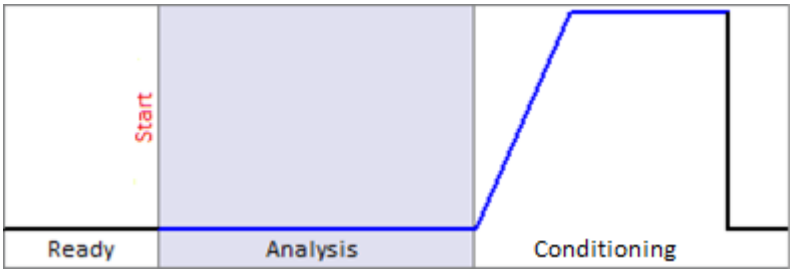


Figure 3.31 – Conditioning after analysis

Further modes of units are suggested to set.

Oven mode:

```
          Oven-1
Isotherm count          2
Flow iso 1           60.0
Time iso 1,min        5.0
Speed 1/2            10.0
Flow iso 2          150.0
Next
Back
```

Set desired number of isotherm (on default – 1) and press ENTER key (or just transfer cursor), then parameters **Time iso** and **Speed x/y** will be formed in dialogue. Number of parameters at 1 less than isotherm count. If only one isotherm is used, no programming parameters available.

Final isotherm will last up to analysis termination.

Press ENTER on the **Next** line to pass to the next device.

Detector's mode:

In detector mode temperature and flow of feeding gases (air, hydrogen, make up gas, reference gas etc.) are set according to required operating mode.

```
          FID-1
Temperature        200.0
Air                200.0
Hydrogen            20.0
Make up             15.0
Next
```

Here set detector temperature, gas flow rates for air, hydrogen and make-up (list of gases varies for different detectors).

Press ENTER on the **Next** line to pass to the next device.

Inlet's mode:

Temperature parameters are set in inlet mode. For gas parameters see below Column mode.

```
         Inlet-1
Isotherm count         2
Temper iso 1        35.0
Time iso 1,min         0
Speed 1/2          300.0
Temper iso 2       150.0
Next
Back
```

This example shows temperature programming mode (2 steps). Such mode can be applied for programmable inlet 8.1.

Set number of isotherms, temperature values at isotherm and time and speed rate. The final isotherm lasts up to analysis termination (no need to specify).

Column mode:

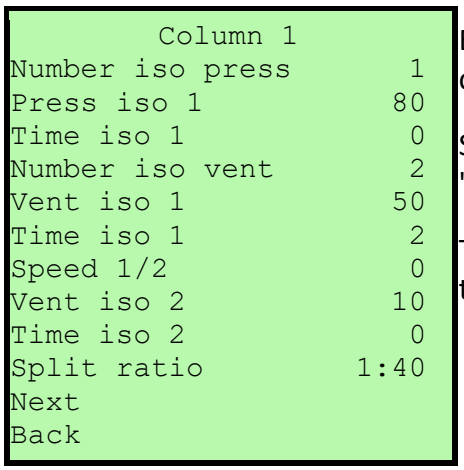
In column mode the following parameters can are set:

- Carrier gas parameters (flow, pressure, speed or cap.flow (flow through capillary column)), feeding column, connected to this inlet.
- Vent gas parameters (for capillary column).

```
        Column-1
Number iso flow        1
Flow                30.0
Next
Back
```

Example for packed column mode with digital flow controller.

When number of iso-line is more than 1, flow programming parameters display, like example below.



Example for capillary column mode with digital pressure controller.

Split vent gas programming is set (Number of iso-lines is – 2). "Save mode" can be set in this manner

Time of the last iso-line and Speed for vent gas are not set, these shall be equal to null.

In Sample vent option **Split ratio** and **Flow iso1** values are interdependent. At setting **Flow iso1** and confirming by ENTER, **Split ratio** recalculates automatically.

Carrier gas saving at operation with capillary columns.

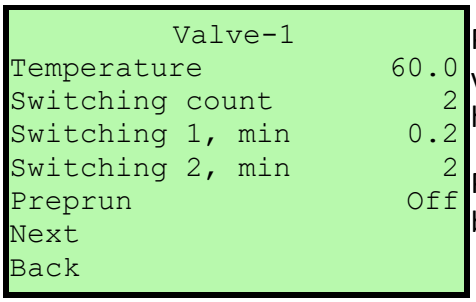
If number of iso-lines is over 1, the last value (usually reduced flow at split vent) will be kept within "Not ready" state. Once all parameters come to norm, the GC passes to the state "Waiting" (LEDs READY and NOT READY blink in turn on GC status board). Press START to go to the state "Ready" and setting first iso-line values. When working with automatic peripherals transfer to "Ready" state is provided automatically.

Valve mode:

In valve mode the following parameters can be set:

- temperature (for heated valve),
- Switching parameters (for automatic valve).

For gas parameters see above Column mode.



Parameter Temperature displays if thermostat is specified in valve configuration, otherwise valve is considered to be non-heated.

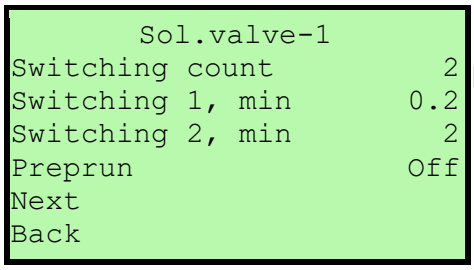
Parameters **Switching N, min** display once **Switching count** has been selected and key ENTER pressed to confirm.

When the **PrepRun** option is enabled, as soon as all the parameters of the chromatograph come within tolerance, it goes to the "Waiting" stage. When the operator, without injection a sample, presses the START key, the valve is switched (it is not indicated in the mode) and the chromatograph goes to the "Not Ready" state, and then "Ready". The operator can then inject the sample and press START again. The analysis starts and the valve executes the switching program set in the mode.

This behavior is used in chromatographs, the gas scheme of which involves backflushing of the pre-column. A feature of such schemes is that the initial flow direction is reversed (to prevent heavy sample components from entering the column from the inlet between analyzes). To switch flow from reverse to direct, a PrepRun is performed just before sample injection.

Solenoid valve mode

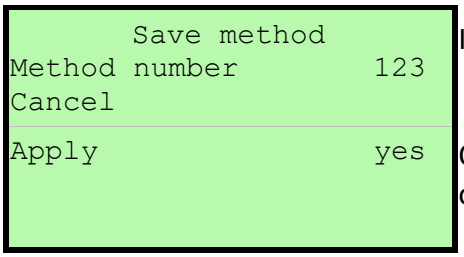
For solenoid valve its switching parameters are set.



Parameters **Switching N, min** display once **Switching count** has been chosen and key ENTER pressed to confirm.

Operation of the solenoid valve when the PrepRun option is enabled is similar to the operation of the valve described above

As soon as all steps of method creation has been fulfilled, the dialogue of method saving displays:

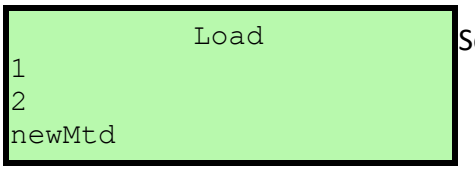


Input method name (numerical only) and press ENTER.

Once method saved, user can apply it immediately or skip in order to create another method.

Loading mode (method):

To load on of previously created methods choose option **Load** in menu of key METHOD.

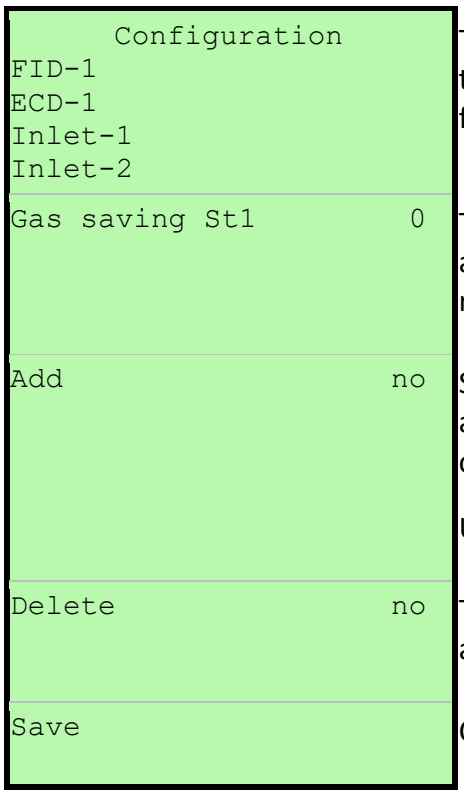


Select required method in the list and press ENTER.

3.7.6 CONFIG key menu

This menu contains GC configuration setting. Commonly, configuration setting is carried out once and is not required in daily work. Anyway, in some cases some parameters may be needed to change.

Press CONFIG key.



The list of automatically identified units appears on the display: the list of units is determined by GC configuration, described I flow diagram.

This parameter specifies time of standstill in the "Ready" state, after which the GC goes to "Gas saving" state reducing gas flow rates.

Some units, such as "column" cannot be identified automatically, these shall be added manually into configuration.

Using SELECT key specify the added units and press ENTER.

To delete manually added unit find it in the list of manually added units using SELECT key, then press ENTER.

Choose this command to save configuration.

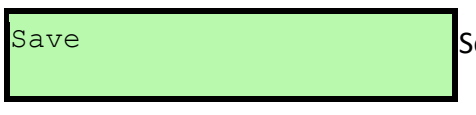
According to flow diagram of the GC add units, which are not identified automatically. These units are:

- Valve-M Manual valve.
- ED External device (3rd party manufactured devices as well as some Chromatec devises can be added, e.g. 1-stage thermal desorber).
- Gas meter Digital flow gas meter.
- Press reg Pressure regulator.
- Column Chromatographic column.

Once units are added, choose first one to set it, press ENTER.

When all parameters for the first unit have been set, command **Next** displays. Press ENTER to go to the next unit. Set all units in the same way.

As soon as all units have been set, option **Configuration** option will appear on the display.



Select **Save** command and press ENTER.

Channel is common property for all units.

Channel is a sign by which all units are grouped in the GC. Such grouping allows running several independent analyses in the GC at the same time. By productivity, the GC with 2 independent channels is two GCs in one.

Commonly term "2-, 3-channel GC" means that there are installed 2 or 3 detectors in GC. For GCs with PM3 controller sense of this term is slightly different. 2-channel GC may have even 3 or 4 detectors, as well as 1-channel GC

Property Channel may have values:

- **Start-1**, **Start-2** means that unit, for which this value is appointed, will take a part in the analysis started by corresponding key.
- **No** – unit is not connected to any channel. The GC shall not observe its condition and control parameters.

Column oven is common for all channels in the GC, therefore its Start channel is not specified.

On default all the GCs are adjusted at one channel. Setting additional channels is carried out in expert configuration parameters (see section 3.7.7).

Description of the parameters is given below.

Detectors:

Channel	St1	Select start channel **St1**, **St2** or **no**.
Thermostat	Td1	Select detectors thermostatic zone. Here **Td1** means that detector is mounted on the forward heating platform, **Td2** means back heating platform. Several detectors can be mounted on the heating platform, 3 detectors TCD for example.
Single DFPC	off	Select this option depending on the type of DFPC controllers connected to the detector: single channel DFPC (option is on), or multi-channel DFPC (off). Then depending on DFPC type, additional setting parameters appear.
MDFPC #	no	Select number of MDFPC channel connected to the detector if available.
Make up DFPC	1	Make up DFPC number shall be specified for detectors **FID, NPD, TCD, CCD, PID, ECD**. This parameter is available if **Single DFPC** is on.
Air DFPC	2	Air DFPC number shall be specified for air flow of **FID, NPD, FPD**. This parameter is available if **Single DFPC** is on.
H2 DFPC	3	H2 DFPC number shall be specified for hydrogen flow of detectors **FID, NPD, FPD**. This parameter is available if **Single DFPC** is on.
Frequency, Hz	25	Frequency of data acquisition for detectors signal. The more frequency value, the narrower peaks can be detected, but nose level increases at the same time. 25 Hz set value is optimal for most applications.
Max temper	350	Maximum allowable temperature for detectors thermostat. At exceeding this value (due to incorrect setting or any accident)

	the GC starts emergency cooling. Max temperature value depend on detectors type. The following levels recommended: FID, NPD, ECD – 450 °C; FPD – 400 °C; PID – 250 °C; TCD – 300 °C; CCD 5.184.023-01.00 – 200 °C; CCD 5.184.023-02.00 – 80 °C. If several detectors are located in the same thermostat, minimal value shall be set.
PMT supply on	Photoelectron multiplier power on/off for **FPD**.
PMT zero level 0.0	Due to some difference of PMT parameters, some **FPD** have zero signal above null until being ignited. In this case GC may identify ignition falsely. To avoid it, set PMT zero level value the same as measured signal value at the moment when detector temperature reached its set level.
Shift 800	Set baseline shift value. Amplifier balances baseline level at set shift value automatically. Recommended value for **TCD** and **CCD** – (800-1000) mV.
Inversion off	Set this option on if peaks on chromatogram are negative.
Power 100%	Set filament heating power level depending on carrier gas type being used with **TCD**. Power 100% is admissible to set for helium or hydrogen carrier gas only. Otherwise 25% power should be set. For **CCD** 80% power level is recommended, in case of poor sensitivity 100% power admissible.
Filament TCD on	If option set on, **TCD** supports automatic mode of filament overheating protection. During operation filament option shall be set on. Switch off filaments heating for maintenance or column conditioning.
Sensor CCD on	If option set on, **CCD** supports automatic mode of sensor overheating protection. During operation sensor option shall be set on. Switch off sensors heating for maintenance or column conditioning.
Lamp PID on	**PID** Lamp switching on/off.
Current ECD 100	This option corresponds to level of ionizing current passing through ECD detector cell. **ECD** current is set in relative units from 0 (0 nA) to 350 (4.1 nA), value on default – 100, apprx. equals to 1.17 nA current level. Set value depends on activity of radioactive source.
Make up gas Nitrogen	Select make up gas type: nitrogen, helium, argon
Ignition threshold 2.0	This parameter is for **FID**, **NPD**, **FPD**. At ignition, signal of detector increases. To determine that ignition was successful,

	signal should exceed set threshold value. Recommended figures for detectors: • FID – 2 mV; • NPD – 4 mV; • FPD – 0.5 mV.
H2 increment 5	This parameter is for **FID**, **NPD**, **FPD**. Increment value at which hydrogen flow increases in further ignition attempt (if the previous was failure).
Ignition board # 1	This parameter is for **FID**, **NPD**, **FPD**. It indicates number of ignition board to control ignition wire of the detector.
Flame control on	This parameter is for **FID**, **NPD**, **FPD**. If this option is off, the GC shall go the state "Ready" ignoring flame control.
Air flow cool 15 H2 flow cool 15 Make up flow cool 10	This parameter is for **FID**, **NPD**, **FPD**. Air, hydrogen and make up gas flow rates for "Cooling" state.
Air flow ign 250 H2 flow ign 40	This parameter is for **FID**, **NPD**, **FPD**. Air and hydrogen flow rates for "Ignition" state.
Allow signal 0.0	Allowable detector signal. If this level is exceeded, the message to carry out maintenance displays. "0.0" value turns off this option.
DAC out off	Turning on detectors signal to analogue output (for plotter connection).
Traping off	This option turns on/off traping of the signal on-the-fly.

Columns:

Single DFPC off	Select this option depending on the type of DFPC controllers connected to the column: single channel DFPC (option is on), or multi-channel DFPC (off). Then depending on DFPC type, additional setting parameters appear.
MDFPC # no	Select number of MDFPC channel connected to the column if available.
Input DFPC 1	Select number of single DFPC at the column input.
Vent DFPC 2	Select number of single DFPC at the split vent.
Pressure in to 0.4 MPa	Maximum adjusted pressure at the column input. Depending on DPC-11 the following values can be selected the following: • to 0.4 MPa – for all DPC-11 except DPC11.h • to 1.0 MPa – for DPC11.h Incorrect setting will result in incorrect operation of gas controller.

Gas type helium	Select carrier gas type.
Mode pressure	DPC operating mode for capillary column: pressure, speed, flow or cap.flow (flow through capillary column).
Column length 30.0	Length of capillary column, m.
Column diameter 0.53	Internal diameter of capillary column, mm.
Leak check off	This option turns on automatic leak check for the inlet.
Leak value 5	This option adjusts allowable leak value, ml/min. Automatic leak check control principle as follows. If: Flow Inp.DFPC$_{MEASURED}$ > (Flow Vent$_{SET}$ + Cap.flow$_{CALCULATED}$ + Allowable leak value$_{SET}$) Then Leak error displays, the GC will not go the "Ready" state.
Column out amb.press	This option allows to select pressure measuring **at the column outlet**: • amb.press (ambient pressure measured by built-in pressure sensor). • vacuum (pressure equals zero). • rel.press (Relative pressure, value set by User in option "Pressure out", see below). • abs.press (Absolute pressure, value set by User in option "Pressure out", see below).
Pressure out 101.325	Pressure value at column outlet. "Relative" or "absolute" pressure depends on above parameter.
Pressure cool 15	Pressure value at the column input at "Cooling" state.
Pressure save 15	Pressure value at the column input at "Gas saving" state.
Vent fl. cool 15	The split vent flow at "Cooling" state.

Inlets:

Channel St1	Select start channel **St1**, **St2** or **no**.
Max temper 350	Maximum allowable temperature. At exceeding this value (due to incorrect setting or any accident) the GC starts emergency cooling. Recommended temperature value 400 °C.

Valves:

Channel St1	Select start channel **St1**, **St2** or **no**.

Thermostat Tv1	Select temperature zone for valve. Several valves can be located in one thermostat.
Start position A	Initial position according to flow diagram.
DFPC 1 off DFPC 2 off	Supplementary gas controllers connected to the valve.
Max temper 150	Maximum allowable temperature. At exceeding this value (due to incorrect setting or any accident) the GC starts emergency cooling. Recommended temperature value 150 °C for Chromatec-made valve.
Not return off	If option is on, the valve will not return to initial position after analysis cycle. Example of this algorithm: 10-port valve sampling into column at any switching (forward or back).
Gas type 1(2) helium	If supplementary DFPC is available for the valve, gas type shall be selected.
Pressure cool 1(2) 15 Press ignit 1(2) 15	If supplementary gas controller is available for the valve, specify pressure (or flow) value for the states "Cooling" and "Ignition".

Solenoid valves:

Channel St1	Select start channel **St1**, **St2** or **no**.
Start position A	Initial position according to flow diagram.
DFPC no	Supplementary gas controllers connected to the solenoid valve.
Gas type 1(2) nitrogen	If supplementary DFPC is available for the valve, gas type shall be selected.

Oven:

Max temper 250	Maximum allowable temperature. At exceeding this value (due to incorrect setting or any accident) the GC starts emergency cooling. Recommended temperature value 10 °C below maximum allowable temperature for column according to its passport.
T oven < T det on	If this option is on, Oven temperature shall never exceed detectors measured value.
Heating type normal	Select heating type –normal or double. Double heating type must be selected for correct operation with Uninterruptable power supply (UPS).
Choke normal	Select mode for chokes (flaps) control – normal or slow. For slow mode algorithm of hoke rotating changes to slower

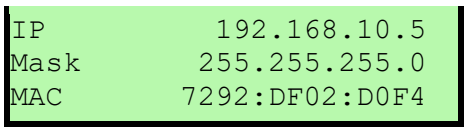
	manner, optimal for operation in oven temperature close to ambient.
Preprun off	Preprun for oven, if option is on, after analysis termination the oven will keep the last isotherm, one preprun performed (press START in the "Waiting" state), the GC goes to "Not ready" state passing to first isotherm.

3.7.7 OPTIONS key menu

Most parameters under this key are set once at first installation.

Press OPTIONS key.

Setting Activation	Turning on/off DEMO period. Press ENTER to set activation parameters for DEMO period
Do not activate Defence off Activation needed Defence on	Turn on **Defence** option if required. Additional setting parameters appear.
Exp date 14/08/14	Expiration date for DEMO period, after that limited mode of operation shall be activated. **Limited mode doesn't allow displaying detectors signals.**
Code	The code shall be entered to transfer the GC into fully functional operation mode.
Autoload method no	Select operating method in this option if automatic loading is needed at GC turning on or setting "Sleep" mode. Method should be created in advance.
Condition method no	Prep-conditioning (column conditioning in the beginning of working day) shall be automatically carried out according to specified method, then operating method specified in previous option shall be loaded. Method should be created in advance.
Sound on	
Keyboard off Ready off Power on on	In this subsection, options of sound beeping can be adjusted.
Connection	Here adjust options of connection the GC to PC.
Ethernet IP 192.168.55.100 Mask 255.255.255.0 MAC 00D0:9300:05E9	Input IP-address of the GC in this option. If the GC is connected into a computer network, IP-address should be specified by system administrator.
USB	No longer used



Procedure of connection creation using "Control panel" SW is described in paragraph 2.5 of "Chromatec Analytic" User Manual.

USB connection in PM3 electronics is transformed into net-connection by means of special driver. Therefore, when USB connection is to be used, IP-address and subnet mask are to be input in the same way as with Ethernet connection.

If there are no any other devices connected to the PC through Ethernet, any IP-address can be set (or suggested on default).

Example of connection:

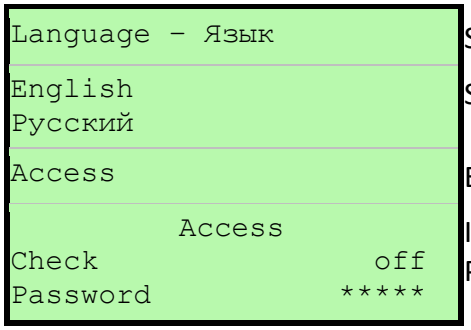
Two gas chromatographs are connected to the PC through Ethernet. PC is not connected to computer network.

The GCs may have the same IP-addresses at delivery, contradiction of IP-addresses occurs in this case. To avoid it, turn on one GC first then change IP-address as described above. For instance:

IP-address on default: 192.168.55.100.

IP address after changing: 192.168.55.10.

Once IP-address was changed, the settings should be saved. To save settings press BACK key until **Setting** menu then select **Save** command and press ENTER.



Select language of the interface.

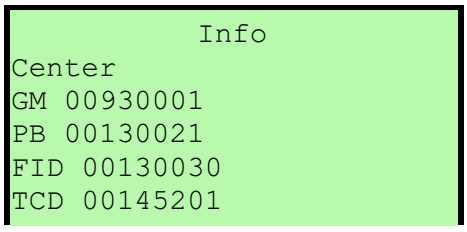
Select language for menu.

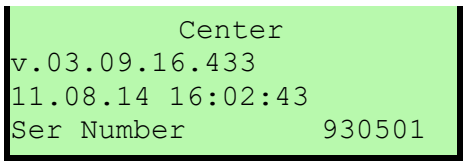
Enter into menu to adjust access to settings.

If you need to limit access to settings, specify "Yes" and set Password.

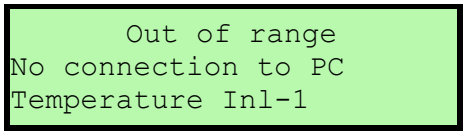
3.7.8 INFO key menu

Info key menu contains data of firmware versions for GC modules:





Double clicking to Info key opens list of parameters being out of range (errors):

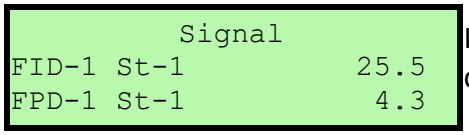


3.7.9 SIG, DET, TMP, INL, VALVE, AUX keys menu

The following keys are intended for quick viewing and editing parameters:

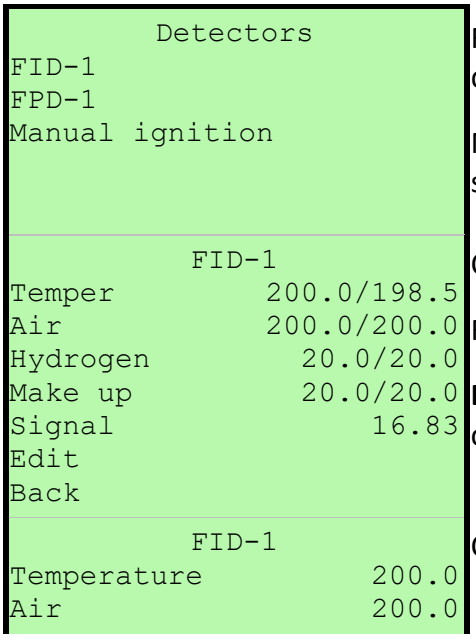
- SIG – detectors signal viewing
- DET – detectors parameters
- TMP – thermostats of the oven, methanizer, transfer line, columns gases.
- INL – inlets parameters.
- AUX – parameters of auxiliary (external) devices, such as autosamplers.
- VALVE – parameters of rotary valves and solenoid valves.

SIG key menu:



In this option detectors signals and corresponding start channels are displayed.

DET key menu (as an example, FID-1):



FID-1 – select and press ENTER key to edit parameters of this detector

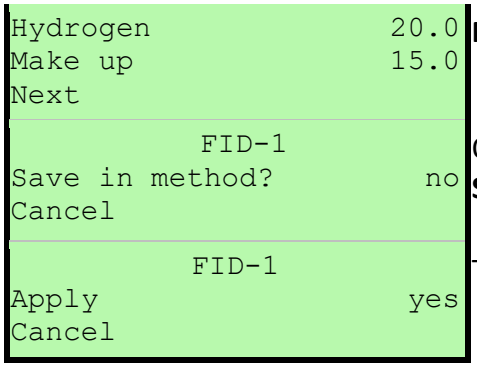
Manual ignition – provides forced turning on wires for 5 seconds to ignite detector.

Current operating state for the detector is displayed here

Figures 200.0/198.5 – set/measured parameters.

Edit – enter this option to change parameters. Editing could be carried out either with saving method or without it.

Change required parameters in this option

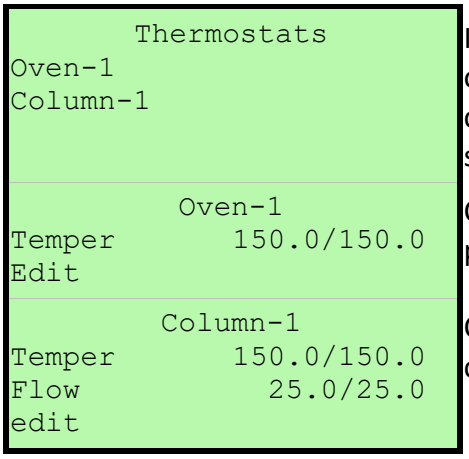


Next – go ahead to save changes

Once any change has been performed, the question appears: **Save in method?** If so, press SELECT, then ENTER.

To apply changed parameter, select **Yes**.

TMP key menu



In this menu user can view and edit temperature parameters of the oven, auxiliary thermostats, methanizer, as well as columns operating parameters (gas flow and pressure rates, split ratio etc.)

Current state of the oven is displayed in this option. To change parameters, select **Edit** command.

Current state of the column-1 is shown in this option. To change parameters, select **Edit** command.

Editing could be carried out either with saving method or without it.

Operation with other quick access keys is performed in the same manner.

4 PREPARATION FOR OPERATION

4.1 Packed columns preparation procedures

4.1.1 General information

Packed columns usually made from glass or metal, of 0.5 … 6 m length (glass columns – up to 3m) of 2- or 3-mm internal diameter (i.d.). Columns of other length and internal diameter are available by order. Packed columns with length above 3m usually consist of sections up to 3mm joint with each other.

Packed columns made in Chromatec may be filled with any commercially available sorbent, by order. Column's test is carried out according to test methods requirements. At delivery, each column is accompanied with passport and check out chromatogram.

Column's material

Packed columns of two types are mainly used: glass and metal. Metal columns are more practical in operation. Glass columns are more fragile, but more inert to analytes. Usually test procedures recommend preferable columns material.

Metal columns, depending on analyzed sample composition and sensitivity requirements, may be of: stainless steel 12X18H10T (Russian marking), SS316 (AISI), deactivated stainless steel SS 316, at (activity tested), nickel. Stainless steel silanized columns or fused silica packed columns (Silcosteel®) are also available by order.

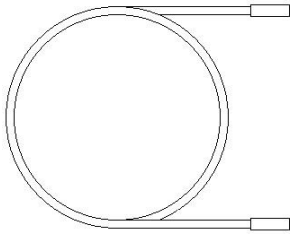
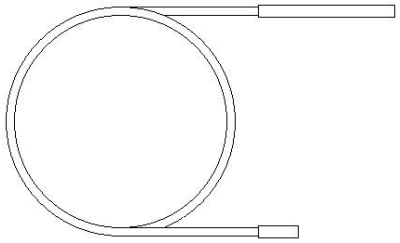
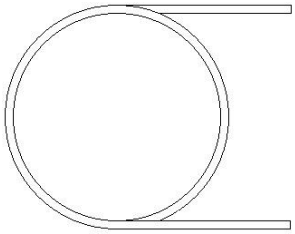
Columns types

Metal columns types are classified depending on installation features, outer diameter of the ends (ends o.d.).

Distance between ends 163mm is for all packed columns intended for installation into GC oven.

Types of the columns are described below (Table 4.1).

Table 4.1 – Description of packed columns types

Type	Description	Overview
1	Symmetrical ends, o.d. 5mm. Standard fitting for packed column in the oven. Graphite or rubber ferrules. Nuts and ferrules are not included.	
2	Ends o.d. 5mm. Elongated front end for entering into inlet (on-column injection). When attaching to inlet, fitting 8.652.376-06 is required (by order). Graphite or rubber ferrules. Nuts and ferrules are not included.	
3*	Symmetrical ends, o.d. 3mm. (for 2 and 3mm i.d. columns) or 1/8" for columns made from corresponding tubing. This is recommended to eliminate influence of diffusion of air compound when traces are to be analyzed. Nuts and stainless-steel ferrules are supplied with the column on default: Nut 8.930.285, Ferrule 8.658.038-01 (3mm) or Ferrule 8.658.038-03 (1/8").	
7	Internal diameter 2mm, reeling diameter 70mm. Intended for using in auxiliary column thermostat (see section 3.3.3). Metal sealing. Nuts and ferrules are not included.	

* – The GC suitable for columns of 3 type has proper fittings in the oven, which are not compatible to common columns (type 1).

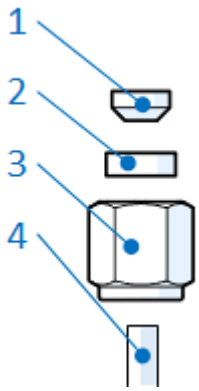
4.1.2 Preparation of column sealing elements

The seals of the packing columns (type 1) differ in material: graphite and rubber.

Ferrule 8.220.380 (rubber) is used in conjunction with sleeve 8.223.013 and nut 8.930.172.

It is recommended to use rubber seals at a temperature not exceeding 240 °C.

The operation of rubber seals at temperatures above 200 °C requires frequent replacement. The tightening force of the nut is 8.930.172 "by hand".



1 – Ferrule 8.220.380 (rubber), 2 – sleeve 8.223.013, 3 – nut 8.930.172, 4 – packed column

Figure 4.1 – Rubber seal

Before using the ferrule 8.220.380, it is recommended to bake at a temperature of (280-290) °C for 20 minutes. At this temperature, the surface of the rubber seal "ages", as a result, rubber seals "do not stick" to metal surfaces at high temperature.

It is not recommended to lube the surfaces of rubber seals with graphite. In this case, along with reducing the "sticking" at high carrier gas pressure, it is possible to squeeze the columns out of the rubber seals and depressurize the gas line.

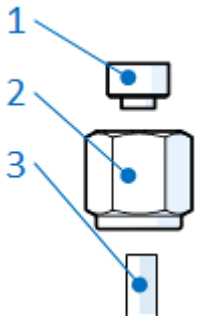
The condition of the rubber seals is recommended to be checked periodically. If there are visible traces of dirt, deformation or cracks, the sealing ferrule must be replaced.

Ferrule 6.453.038 (with graphite) is used with nut 8.930.172.

Graphitic seals are more reliable and especially preferable when working at high temperatures (above 200 °C).

The tightening force of the nut is 8.930.172 "by hand", followed by turning the wrench by (30-60) degrees.

When sealing the glass column with graphitic ferrules, care should be taken to avoid its breakage.



1 – Ferrule 6.453.038 (with graphite), 2 – nut 8.930.172, 3 – packed column

Figure 4.2 – Graphitic seals

Before using graphite ferrules 6.453.038, it is recommended to bake in a thermostat at a temperature of (250-300) °C for 30 minutes to remove organic components absorbed by graphite.

4.1.3 Preparation units for column installation

Connecting adapters to detectors, inlets (if necessary) for packed columns' connecting is described in corresponding parts of part 2 User Manual.

TCD detector and the fittings of the lines from gas valves do not require special adapters to connect the packed columns.

4.1.4 Packed column installation

Before column installation prepare required sealing elements, connect adapters to units according to directions of previous information and sections of part 2 UM.

Distance between packed columns ends is (163±1) mm. Glass packed columns installation should be avoided if distance differs from above mentioned. At installation glass packed column ends should be placed in inlet and detector simultaneously, column should be located parallel to columns oven door.

At glass packed column sealing by graphitic ferrules safety rules should be observed for its failure avoidance.

- Put appropriate sealing elements on the column ends.
- Install column into fittings up to the stop. Pay attention to proper installation relative to gas flow (column beginning is usually marked by label).
- Tighten nuts thoroughly "by hand".
- If graphitic seals used, perform additional turning by wrench by (30-60) degrees (at first use angle shall be a little bit bigger, up to 90-120 degrees).
- For metal ferrules, act additional turning up to (30-60) degrees.

Montage procedures for columns with elongated front end into inlet, is described in section 1.3.4 of part 2 UM.

4.2 Capillary columns preparation procedures

4.2.1 Column location in the oven

Capillary column is located in the oven with the help of a bracket 6.133.078 (Figure 4.3), which is fit to oven top wall. For location up to three columns, the bracket 6.133.089 is recommended.

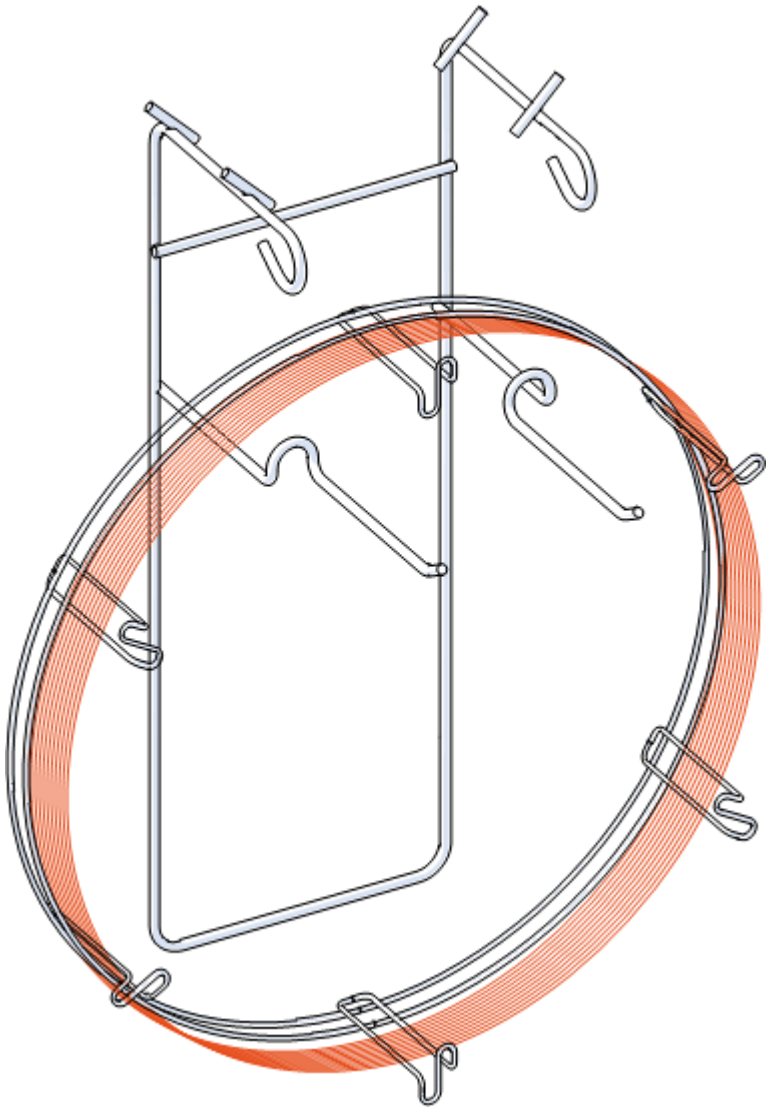


Figure 4.3 – Capillary column location on the bracket 6.133.087

Column shall be disposed in the center part of the oven, do not allow touching column loops to oven walls. When connecting column to detector and inlet, dispose column so that loops of the column were smooth (especially for wide-bore columns).

4.2.2 Preparation for columns install

4.2.3 lation

Required accessories:

- Capillary column;
- Nuts 8.930.339;
- Ferrules (graphite #072626 SGE or similar, or vespel/graphite, like #072663)
- Wrenches 7mm and 8mm size;
- Adjustable spanner wrench;
- column measuring tool 6.236.249 (see Figure 4.4);
- Column cutter;
- lint-free tissue;
- Isopropyl (or ethyl) alcohol.

Graphite ferrules are recommended to bake at (250–300) °C for 30 min before usage in order to remove volatile organic compounds.

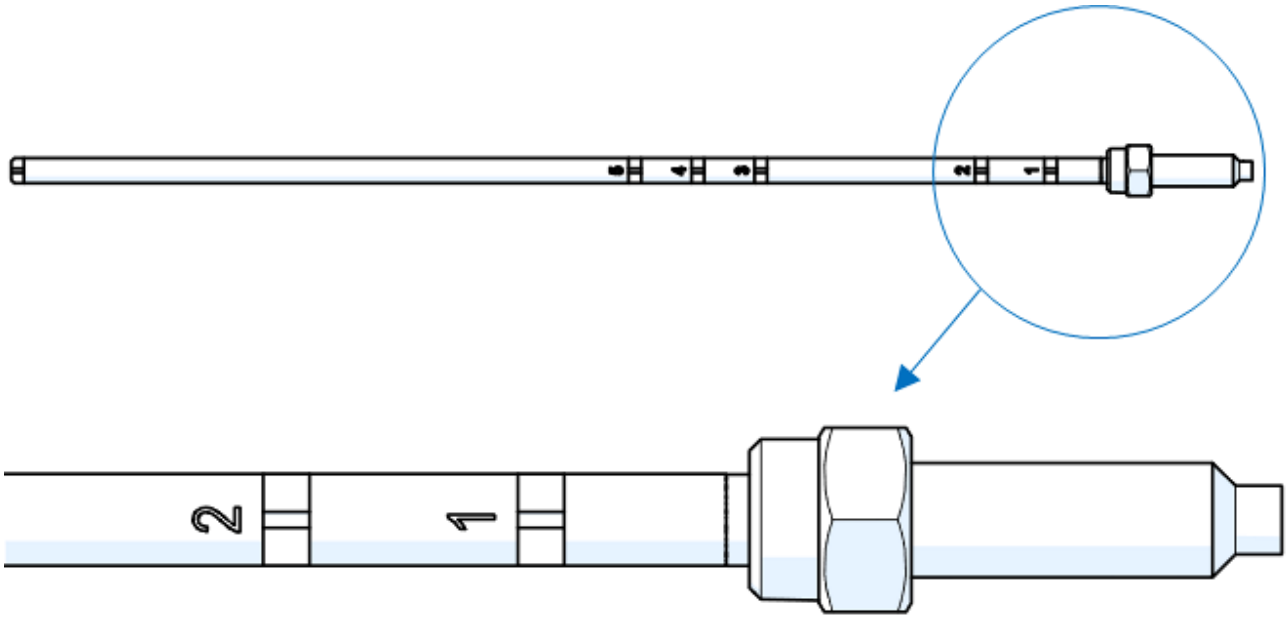


Figure 4.4 – Column measuring tool 6.236.249

Distance for:

1. SSL or PSSI8.1 (liner 113mm),
2. SSL or PSSI8.1 (liner 99mm),
3. ECD,
4. NPD,
5. FID

End section for MSD

4.2.4 Capillary column installation

Capillary column installation procedure for particular units (detectors, inlets, valves) is described in corresponding sections of part 2 UM.

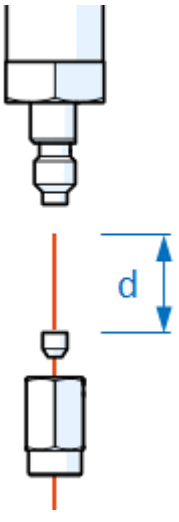
Common recommendations on capillary column installation are described below.

Before column installation prepare required accessories, connect adapters to units according to directions of previous information and sections of part 2 UM.

First connect column to the inlet.

The procedure of column installation:

- Put on the nut 8.930.339 and the ferrule (e.g., #072626 SGE, with its cone into nut) on the end of column to be installed into inlet or detector.
- Cut column end at (5–20 mm) to avoid graphite ingress into column. The cut should be flat without chips and scratches.
- Wipe cut end of the column with lint-free tissue wetted in alcohol in order to remove dust and other contaminants;
- Locate nut and ferrule at required distance (d) from the column end. For convenience, use column measuring tool 6.236.249 from shipping kit. Install column end into measuring tool and pre-swage the nut in the tool fingertight. Align column end at required tool mark. Then holding column position, screw nut by wrenches at about 60 – 90 degrees so that column is fixed tightly by ferrule. Use wrenches of 7 and 8 size for swaging in tool.
- Then release nut.

Tool marking	Unit	d (distance)		
1	Inlet	32 mm	with liner 113 mm	
2	Inlet	43 mm	with liner 99 mm	
	On column	124-0.5 mm		
3	ECD	78 mm		
4	NPD	88 mm		
5	FID	98 mm		
	FPD	using the stop, see sect. 3.5.4-part 2 UM		
	µ-TCD	0.8 mm	vespel/graphite ferrule only	
	PID	20-25 mm		

- Insert the column into adapter of inlet or detector, tighten nut by hand.
- Then tighten the nut by wrench at 45-60 degrees so that column cannot be withdrawn from the fitting (column is tightened by ferrule holder).

Once the column is connected to inlet it should be blown by carrier gas from oxygen at ambient temperature at least for 10-15 min.

If the column is new, it should be conditioned as described in section 4.3.

Connect the column to detector in the same manner.

4.3 Column conditioning

4.3.1 Column preparation for conditioning

Conditioning shall be applied as for new columns as well as columns being working. Purpose of column conditioning is removal volatile contaminants from the column. Contaminants may cause excessive column bleeding, unstable baseline especially at temperature increasing. High boiling contaminants, if got into column with sample, may cause deterioration of compounds separation, tailing peaks and even irreversible sorption of some compounds.

Before conditioning make sure that carrier gas is sufficient by purity grade. For many columns low oxygen content is very important parameter. Unwanted impurities affection at high temperature may damage column.

Column preparation for conditioning is carried out as follows:

- Column is disconnected from detector. Detectors fitting in the oven shall be plugged;

When operating with FPD, do not disconnect column from the detector.

- for NPD detector, set Hydrogen flow as 0 ml/min in GC operating mode and switch off flame control in Configuration;
- if column is not installed yet, it shall be connected to inlet (or gas line fitting from sampling valve). Procedure describing column installation has been given above.

Hardly contaminated column is recommended to expose long-term conditioning under high temperature, and the column re-connected with its back end to inlet, front end is stayed free.

When using TCD detector, filaments shall be turned off before conditioning.

Setting parameters, such as oven temperature and gas flows the columns at conditioning, is carried out in the same way as setting operating GC mode.

The chromatograph control program provides for the possibility of setting automatic conditioning when the device is turned on (see section 3.2.1 of the Chromatec Analytic user manual).

4.3.2 Packed columns conditioning

Before starting conditioning, make sure that stable carrier gas flow is passing through column.

Procedures performed at packed column conditioning and parameters:

- Set required carrier gas flow through the column (usually at 30 ml/min);
- Keep carrier gas flowing through the column at ambient temperature in oven for (10–15) min in order to eliminate the rest of oxygen;
- Carry out conditioning with temperature programming at (1–5) °C/min;
- Do not exceed maximum allowable temperature for the column. Set it at (20–30) °C below maximum value;
- Time of conditioning depends on column sorbent. It may vary from several hours to several days;
- If column is not going to be used immediately, it shall be taken out from the oven. For storage, ends of the column shall be sealed with plugs to prevent ingress moisture and other contaminants into the column.

4.3.3 Capillary columns conditioning

Before starting conditioning be sure that stable carrier gas flow is passing through column.

Procedures performed at capillary column conditioning and parameters:

- Set required carrier gas flow through the column (usually at (1-2) ml/min); for wide-bore columns 0.53 mm i.d. – up to (6–8) ml/min);
- Keep carrier gas flowing through the column at ambient temperature in oven for (10–15) min in order to eliminate the rest of oxygen;
- Carry out conditioning with temperature programming at (5–10) °C/min. At reaching maximum isotherm, keep column at about 1 hour in this condition;
- Do not exceed maximum allowable temperature for the column. Set it at (20–30) °C below maximum value;
- Time of conditioning lasts about 4 hours;
- If column is not going to be used immediately, it shall be taken out from the oven. For storage, ends of the column shall be sealed to prevent ingress moisture and other contaminants into the column.

Other conditioning mode may be recommended for some columns. Follow columns manufacturer instructions.

4.3.4 Other ways of column regeneration

If column conditioning doesn't lead to desired results, other procedures may be applied to gain necessary performance:

For packed column, re-pack input section of the column.

For capillary column, cut off front end of the column (20 – 30 cm).

Capillary columns with bonded phase are allowed to rinse with suitable solvent or solvent mix.

If nothing helps, it's recommended to change the column.

4.4 Liquid and gas samples preparation

4.4.1 Samples preparation

Sample preparation in chromatographic analysis is multistage procedure. It includes all operations which sample is undergone by, starting from sampling and finishing by injection a substance being under analysis in chromatograph.

Sample preparation procedure before analysis is carried out in the following cases:

- Analyzing substances quantity in the sample is small;
- Sample contains components which are unwanted for chromatographic system ingress;
- Analyzing substances by their physicochemical properties are not suitable for gas chromatography.

Sample preparation means such operations connected with use of the following physicochemical properties conducting as: adsorption, absorption, extraction, distillation, chemisorption, etc. In case when initial substances estimation is difficult from them compounds are obtained, that suitable for analysis.

Detailed operations connected with sample preparation are given in procedures for analysis execution (MVI, GOST, RD etc.).

Chromatograph should pass presales tests and modes testing at the factory-manufacturer in accordance with testing procedures.

Manufacturer guarantees branch fittings accurate characteristics of chromatographic analysis results for samples, specified in appropriate testing procedures sections.

Sampling and sample injection are the most important steps in sample preparation. Some general recommendations concerning sampling and samples injection from which accurate results obtaining to greater extent depends on should be used.

4.4.2 Liquid samples preparation

liquid samples should be kept at indoor temperature before being injected into the GC. Exceptions are volatile liquids samples, with boiling point (15-45) °C. Such liquids are sampled in cold state with the help of a cooled micro syringe. Sampling temperature should provide stable liquid aggregative state of obtaining sample, without degassing.

High viscosity liquids samples (for example, crude oil) are allowed being diluted by less viscous solvents, not affecting analysis results.

Additionally at operation with micro syringes for liquid samples injection it's recommended to study subsection 4.5.

4.4.3 General recommendations for gas samples preparation

For injection, temperature of gas sampling container should be 10 °C higher than temperature at loading. Samples should be kept at this temperature for at least 3 hours.

If there are dead volumes in sampling gas lines, the following blowing procedure is recommended: increase pressure in dead volume up to (3-4) kg/cm^2 (≈3-4 Bar) then drop pressure down to atmosphere level. This procedure is recommended to eliminate the rest of oxygen and nitrogen, when these compounds analyzed at 0,01 % vol. or below.

However, it should be considered that abrupt pressure drop may cause concentration distortion in flow can be observed for components with boiling point over 60°C and also for components tend to sorption on gas channels internal surfaces. For such samples blowing by moderate stable flow rate for (2-5) minutes is recommended.

If there is any flow rate control device for analyzed gas (for example, Gas flow indicator 5.183.004), valve-sampler is recommended to rotate from "Sampling" position into "Analysis" position without flow break. Valve-sampler loop purging time is 1-2 min at analyzing gas sample flow rate (30-40) ml/min. Loop purging modes within analyses consequence should be reproduced by blowing time at least 10%, by gas sample analyzable gas flow rate at least 20%.

It should be considered that at analyzable gas flow break at sampling valve failures, concentration figures for analyzed components tend to be lower than real.

When using blocking liquid at the output of loop for sample purging, be aware that blocking liquid doesn't exclude errors in oxygen and nitrogen concentration determination (especially at low level). Blocking liquid is in contact with indoor air at balanced distribution of dissolved oxygen and nitrogen in liquid. At loop blowing balance in sampling line is upset to concentration decrease over liquid. At analyzing gas sampling line blowing stoppage diffusion of dissolved oxygen and nitrogen in liquid in sampling line is possible.

At long operation interruption liquid may be drawn into sampling line. Output tube should be removed from blocking liquid.

It's recommended to use the following blocking liquids: 22% sodium chloride as per GOST 13830 in distilled water as per GOST 6709 or mix of glycerin equal volumes in accordance with GOST 6259 and distilled water.

For analyzed gas flow regulation, it's recommended to use adjustable restrictor without dead volumes. Gages in sampling line should be installed with blowing measuring cell.

4.5 Operation with syringes

Syringes for GC applications can be classified in design:

- "Plunger in needle" type (0.5 to 5 µL volume);
- Syringes for liquid samples with metal plunger fitted in glass barrel (5 to 500 µL volume);
- Gastight syringes have plunger with PTFE tip, intended for gaseous or liquid samples injection (usually volume from 10 to 100 µL).

Depending on syringe type, recommendations on syringe care, recommended replaceable parts and lifetime are different. Common recommendations for syringes intended for liquid sample injections are described below. Detailed instructions for particular syringes are specified in corresponding user manuals.

4.5.1 Common recommendations for syringes usage

Liquid samples are injected into GC inlet by means of syringe.

When using syringe, follow recommendations specified in accompanied user manual as well as recommendations described below.

Minimize shifting dry plunger in the syringe. If moving plunger not wetted with liquid, it may be jammed in syringe barrel. So if there appear any force at plunger shifting, it's recommended to wet it with solvent (not applicable to gastight syringes).

Excessive forces should be avoided at syringe plunger movement as it can result in plunger bending.

Syringe plunger is carefully fitted to syringe barrel by diameter at manufacturing. So plungers are not replaceable (except "plunger in needle" type and gastight syringes).

For sampling, immerse needle into liquid and prime syringe (aspirate and dispense syringe several times by cycling plunger). This allows displace air or previous liquid from the syringe. When priming, make 15 – 20 cycles, at that 3 – 4 initial aspirations shall be wasted.

When sampling viscous liquids, make aspiration slowly, this allows prevent cavitation, gaseous bubbles appearance.

If plunger is completely come out from syringe cylinder accidentally, carefully wipe it by lint-free issue before reinstallation into cylinder. Do not touch plunger as fingers' grease often prevents plunger reliable functioning.

At measuring too low sample volumes comparatively to total syringe capacity, be aware that inaccuracy of measurement goes up dramatically. For precise sampling, using less than 10% of syringes scale is not recommended.

4.5.2 Manual injection

The following is a simple algorithm for injection a sample with a syringe, used in the vast majority of cases.

In some cases, other algorithms for injecting a sample with a syringe are recommended, for example, a sandwich with a preheated needle or a sandwich with a solvent (not described in this manual). Such input methods can reduce peak blurring or discrimination of sample components, for example, when introducing a volatile solvent into a hot evaporator.

To measure the required sample volume, fill the syringe with a sample volume slightly exceeding the required one. If a syringe with a volume of 5 or 10 ml is used when injecting liquid, the last air bubble should be removed by turning the syringe over and slightly pushing the plunger. Then gently lower the plunger to the required level. When adjusting the plunger position, hold the syringe at eye level with the divisions to yourself, so that the scale is clearly visible. Remove remnants of the sample from the needle tip with lint-free issue.

When making an injection, puncture needle through the septum gingerly in order to prevent needle from bending. For new septum, previous puncturing septum by needle 6.054.005 is recommended.

When injecting a sample, it is recommended to always hold the syringe the same way (for example, with divisions to yourself), monitor the vertical position of the syringe body when injecting.

The injection should be carried out at the same speed from repeat to repeat, avoid unnecessary delays when injecting the sample. Correct and proven injection technique allows you to get good convergence of results between analyses.

4.5.3 Syringe cleaning

Syringe cleaning agents will usually depend on the contaminating material. Different organic components could be used for this purpose, commonly used solvents are ethanol or methylene chloride.

Do not immerse the entire syringe in solvent as this may damage the adhesive used to bond parts of the syringe. Clean syringe externally by wiping with a tissue wetted in solvent.

To clean plunger, take it out from syringe barrel and wipe carefully with lint free tissue wetted in solvent. Then insert plunger into barrel and aspirate pure water or other solvent several times. Before storage dry syringe and plunger in air.

4.6 GC configuration setting

The GC is supplied in composition according to its gas diagram, which is specified in attachment B of part 3 UM. Initial configuration setting according to gas diagram is carried out by manufacturer. At configuration setting parameters for detectors, inlets, rotary valves, solenoid valves are set. Setting is performed by chromatograph keyboard or with the help of Chromatec Analytic software on PC.

Parameters of configuration may need to be changed in some cases, such as:

- Replacement or conditioning chromatographic columns;
- Installation or replacement of devices (detectors, inlets, valves, methanizer);
- Replacement of detectors being in operation;
- Simultaneous and independent operation of two chromatograph channels and in a combination with it use of two automatic peripheral devices;
- DFPC replacement;
- Chromatograph gas diagram overcommutation.

Configuration settings are described in details:

- in section 3.2.1 of the Chromatec Analytic User Manual when performing settings from a personal computer (recommended).
- in section 3.7.6 of this User Manual.

5 CHROMATOGRAPH DAILY WORK

5.1 Chromatograph operation

Chromatograph operating modes are controlled in form of dialogue by means of personal computer (PC) or by means of the control panel.

- At work with PC, with the help of "Chromatec Analytic" software it is necessary to use user's guide 214.00045-51И.
- At operation with chromatograph using the control panel it is necessary to learn its description (section 3.7, current User Manual).

GC mode shall be created by user in accordance with recommended parameters specified analysis test method.

At method parameters take into account:

- specific features of particular gas diagram of the GC (gas diagram is given in attachment B of the part 2 current UM;
- configuration settings should comply with gas diagram and analysis procedure (for example, detectors location, flow rate controllers' parameters setting, etc.).

Procedure of GC mode creation is described in section 3.7.5 of current UM.

In menu of CONTR key, operational and diagnostic parameters, external and built-in auxiliary devices parameters could be observed.

5.2 Operability check

Operability check is carried out in order to estimate readiness of the GC to analyses performance and also to reveal if any kind of technical maintenance or repair is required.

Recommended daily practice in operability check is following:

- The GC shall be turned on, at this there should be **no strange noise** (from any operating mechanism, gas leaks etc.);
- Before setting GC mode, it’s recommended to check (by display on keyboard or PC) **thermostats temperatures** (figures should be the same as ambient temperature) and **gas flow figures** (these should be equal to nil, admissible figure – 0,3 ml/min);
- Set GC mode and control how parameters keep set (thermostats temperatures, flow and pressure rates of gas controllers).
- During operation, calibration or unknown samples analysis, check detectors baseline constancy (detectors bleed, noise amplitude, baseline drift, baseline shift at oven temperature raise), efficiency on peaks separation, and repeatability in analyses results.

If necessary, at detectors and inlets contamination, sensitivity decreasing etc., as well as at long work break, it’s recommended to estimate as many parameters as possible. For this following section 6.2 "Test run" in Performance verification procedures 214.2.840.043Д is recommended.

Depending on GC configuration, operational mode and sample character, normal parameters may significantly vary. Pay attention to stability of parameters mentioned above in daily practice.

If necessary, consult manufacturer or its regional representative.

5.3 Operation sequence

Information about GC location and installation on workplace, gas lines connection, and electrical connections is described in User Manual, part "Installation instructions".

Chromatograph operation procedures are specified in section 5.1. At using with Chromatec Analytic software, before start in operation all preliminary settings shall be performed (software installation, connection to GC, working project creation etc.). Below links refer to "Chromatec Analytic" version 3.0 User Manual.

Before start in operation, procedures recommended in section 4 should be carried out.

Below typical order on user procedures in daily work with GC and connected devices is described. Nevertheless, suggested operation sequence may be different for particular situation depending on GC configuration, analyses method, hardware and software parameters.

5.3.1 Operation start

- Power on computer (PC), then turn on GC and connected devices (if available). For GC turning on, power on/off switcher on rear panel, then push on/off button on right wall. At turning on the GC passes through initialization state and starts hardware.
- Supply carrier gas into the GC. For this purpose, open on/off valve on carrier gas cylinder (anticlockwise). Then by rotating pressure regulator handle clockwise set up carrier gas pressure rate within range 0,4 – 0,64 MPa (or up to 1,00 MPa if higher pressure required to set at column inlet).
- If flame detectors are available, turn on hydrogen generator and compressor or other hydrogen and air sources.
- Set working mode to the GC. Setting mode procedure is described in section 6.3.1 of "Chromatec Analytic" UM. If GC mode had been created before, it can be transferred to the GC. After setting mode, the GC goes to "Not ready" state, gas controllers keep flows and thermostats start heating.
- If flame detectors are available, automatic ignition of detectors (FID, NPD, FPD) starts in 20-25 minutes after GC mode setting, state "Ignition" takes place.

Turning to "Ignition" state takes place from the state "Not ready" if the following conditions fulfilled:

- There is at least one active flame detector (FID, NPD, FPD).
- Signal of the detector is below than set threshold within 10 sec.
- If NPD detector is available, actual temperature of detectors reached set value.
- If NPD detector is not available, actual temperature of detectors reached 150 °C (inclusive).
- At the moment of setting operating mode, the lowest value of detectors temperature must be higher than 80 °C (inclusive).

If at the moment of setting operating mode the lowest temperature of detectors value is below 80 °C (inclusive), 10 minutes delay takes place from the time when lowest temperature reaches 80 °C.

- Once GC comes to "Ready" state (all parameters keep normal), it's useful to wait for some time to allow baseline stabilization, when working at oven temperature programming, initial blank analysis (without sample injection) is recommended to carry out.
- Then go to metering.

5.3.2 Metering

Metering, depending on analysis method and operation step may purpose:

- Calibration;
- Unknown sample analysis.

These procedures are described at length in sections 2 and 3 of "Chromatec Analytic" UM.

Operation with the GC during these procedures are similar:

- Before injection, sample preparation shall be carried out.
- Once the GC comes to state "Ready", inject sample into the GC by means of microsyringe, sampling valve or other sampling device, at the same time press Start button on status board. The GC start "Analysis" state.
- At termination "Analysis" state, the GC goes to "Post conditioning" (if set) or to "Not ready" state.
- Perform chromatogram processing, if necessary, print report.

In some configurations, the following steps are also performed:

- When using "PrepRun", programming of gas flow rates and pressures, after "Not ready" state the GC goes to "Waiting" state (Indicators NOT READY and READY blink in turn). To go to "Ready" state, press Start button.
- After coming to "Ready" state, the GC is ready to start next run.
- If gas save mode is set, and analysis was not started timely, the GC goes to "Gas save" state. To come back to "Ready" state, re-set GC mode.

5.3.3 Operation termination

- At termination of GC operation, before turning off, temperature zones of the GC should be cooled down. For this purpose, "Cooling" mode shall be transferred to the GC (see section 6.8 of "Chromatec Analytic" UM).
- After cooling, the GC can be turned off. At daily work rear located On/Off switcher can be left on "On" position.

"Cooling" state also starts at on/off button press (Figure 3.1). In this case the GC will be turned off automatically, once oven temperature reaches 40°C.

- Close gas cylinder on/off valve (clockwise);
- Release pressure regulator spring (counterclockwise);
- Turn off all connected devices.

6 MAINTENANCE

Technical maintenance is performed in order to provide compliance of GC performance with its technical specification during operation.

At technical maintenance it is necessary to observe safety measures according to section 2.1.

Maintenance procedures for the GC and units are described in corresponding sections of current User Manual.

With no necessity, GC unites should not be disassembled and regulated.

Except for special provisions, repair and maintenance should be carried out with disconnected chromatograph electric supply and closed gas lines.

6.1 Types of maintenance

Two types of maintenance are foreseen for GC:

- Current maintenance. It's carried out by customer.
- Periodic maintenance. It's carried out by service personnel with qualification confirmed by certificate signed by manufacturer officials.

The requirement for periodic maintenance by a qualified service engineer applies only to work carried out during the warranty period.

Maintenance work carried out after the warranty period is over, fully transferred to the responsibility of the consumer, unless otherwise specified in the contract for maintenance.

Periodicity of technical maintenance both types is suggested on the basis of 20-30 analysis during working shift of eight hours period.

Maintenance periodicity can vary depending on:

- chromatograph work intensity;
- working temperatures of inlets, detectors and the oven;
- nature of samples;
- Ambient factors (pollution, temperature, presence of dust).

6.2 Current maintenance

Table 6.1 – Current maintenance procedures

Procedures	**Periodicity**
General evaluations: acoustic noise level evaluation; sealing state visual check; stability of keeping set gas flow, oven temperature	Daily
Inlet septum state evaluation	Daily
Inlet O-ring and glass liner state evaluation	Weekly
Replacing the septum	As it wears out (see section 1.6 of part 2 of this manual)
Replacing O-ring and glass liner	As wear or contamination occurs (see section 1.6 of part 2 of this manual)

General evaluations often are subjective and their adequacy mostly depends on operator's experience.

Although together with evaluated chromatograph technical parameters these evaluations can be considered in later actions for defects elimination.

Units, possible noises sources, have constructive safety margin.

Table 6.2 – Acoustic noises sources

Unit	**Evaluation parameters**
Electric motor of oven ventilator	Vibration increase
Oven flaps gear	At oven cooling the vent hole should be opened
	At oven heating the vent hole should be closed
	Mismatch of heating and cooling speed to GC technical specifications
Electric actuator of rotary valves	Valve switching should concur with set values
	Absence or mismatch of peaks on chromatogram in case if valve switching has not occurred

According to evaluation results periodic technical maintenance, repair or replacement should be conducted.

6.3 Periodic maintenance

Periodic maintenance parameters are listed below (Table 6.3):

Table 6.3 – Periodic maintenance parameters

Procedures	**Periodicity**	**Related section of UM**
Regeneration or replacement of adsorbent in gas traps	See section 3.2.7	3.2.7
Gas lines connections check for leaks	Once in half-year	3.4.4
Inlet maintenance (except replacement of septum)	After removing the O-ring and glass liner and regenerating the filters	section 1.6 part 2 of current UM
Gas flow check*	Yearly	3.1.3
Gas pressures check*	Yearly	3.1.4
Detector's maintenance	Once in half-year	section 3 part 2 of current UM

* If retention times are confirmed as stable, separation efficiency for compounds meets to test method requirements; this procedure is allowed to skip.

Replacement of chromatographic columns is not a maintenance operation.

The replacement of the chromatographic columns is made by the consumer when following the instructions of the operating manual.

6.4 Consumables for the GC

Consumables service life of the GC depends significantly on operating conditions (carrier gas purity, temperature, sample character etc.). The following table shows general consumables, signs of wear, factors having an influence on lifetime, and mean lifetime.

Table 6.4 – GC consumables

Description	**Sign of wear**	**Lifetime factors**	**Mean lifetime. Advice.**
GC traps (traps)	Excessive signal, drift, noise of detector, negative peaks on chromatogram	Purity of refined gas	6 – 12 months. Regeneration of sorbent, if stipulated by design; or replacement.
Sealing elements of the GC	Various signs of wear depending on material, impossibility to eliminate leak	Correct swaging (excessive force unacceptable), number of cycles of mounting / dismounting	Rubber: up to 1 year. Metal: 3 years and more; not more than 5 cycles of mounting / dismounting Replacement only
Inlet			
Septum	Inlet leak	Inlet temperature, needle diameter and tip style	50 – 300 injections. Replacement only.
Liner	Ghost peaks, broadening peaks or lost sensitivity due to sorption	Sample purity (non-volatile and heavy compounds undesirable)	5 – 500 injections. Rinsing or replacement.
Liner glass wool (untreated, silanized or quartz)	Ghost peaks, broadening peaks or lost sensitivity due to sorption	Sample purity (non-volatile and heavy compounds undesirable)	5 – 500 injections. Replacement only.
O-ring	Sample distortion, cracks	High inlet temperature	5 – 500 injections. Replacement only.
Detectors			
FID jet	Broken jet neck, unstable or impossible flame ignition	Neatness at maintenance. Heavy components capable to carborize nozzle of the jet	6 – 12 months. Cleaning; if broken – replacement only.

Ignition wire of flame detectors FID, NPD, FPD	Lack or poor glowing, unstable auto-ignition due to this reason.	Aggressive combustion products (acid vapors etc.)	6 – 12 months. Replacement only.
TCD sensor (TCD filaments)	TCD signal unbalance, excessive difference of resistance between filaments.	Carrier gas purity. Oxidants in sample.	Up to 1 year. Replacement only.
NPD sault source	Insufficient NPD signal at high rate of hydrogen flow.	High hydrogen flow, high detector temperature	3 – 6 months. Replacement only.
PID lamp	Low sensitivity, lamp cleaning doesn't help.	High operating temperature, close to maximum for lamp. Long operation at turned on condition.	According to lamp manufacturer instructions. Typically, up to 500 hours for Kr-lamp, up to 200 hours for Ar-lamp. Replacement only.
Methanizer			
Methanizer catalyst	Poor conversion or no peaks of CO and CO2 using FID with methanizer	Hydrocarbons and acid gases in gas flow through the catalyst	3 – 12 months. Follow catalyst regeneration procedure. If it doesn't help, replace catalyst.
Chromatographic columns			
Capillary or packed columns	Deterioration of separation of critical pars of peaks, general efficiency decreasing, retention time shift to lower value	Heavy compounds in sample, carrier gas insufficient purity, operating at oven temperature close to maximum for column.	From 3 months to 3 years. Follow conditioning or other regeneration ways (see section 4.3.4). If it doesn't help, replace the column.
Sealing elements for columns	Various signs of wear depending on material (rubber – cracks and loss of elasticity, graphite ferrule – subsiding in holder, metal – cracks and asymmetry at swaging) impossibility to eliminate leak	Correct swaging (excessive force unacceptable), number of cycles of mounting / dismounting. High temperature is unacceptable for rubber ferrules.	Rubber: 1-3 months. Graphite: up to 1 year Metal: 3 years and more. Graphite and metal sealing have limited cycles in mounting / dismounting Replacement only.

Accessories for sample preparation and injection of samples into GC

Manual syringes	Needle clogging. Damaged or bended needle (or plunger). Plunger stuck. Leak through the plunger (for gastight syringes). Unsuitable repeatability.	Neatness in work. Regular cleaning after operation. Sample character (viscosity, saults and solid particles presence etc.)	3 – 6 months. Cleaning plunger and syringe barrel. Cleaning needle and preparation of needle tip.
Syringes for autosampler	All signs specified for manual syringe.	All factors specified for manual syringe. Correct installation of the syringe in the autosampler, correct installation of the autosampler on the GC.	3 – 12 months. Cleaning plunger and syringe barrel. Cleaning needle and preparation of needle tip.
Vials	-		Disposable. Cleaning or replacement.
Septa for vials, Aluminum caps for vials	-		Disposable. Replacement only.

Recommended part numbers of consumables are determined by configuration of the GC and specific application.

Consumables for MSD, PDD, HID are not considered in this section.

Supplementary materials and chemicals

Lint-free fabric, cotton applicators for cleaning parts.

Ethyl alcohol or other cleaning agent (methyl acetate, acetone) is required in volume up to 100 ml per year.

Strong mineral acids (sulfuric acid, hydrochloric acid) or chloroazotic acid may be applied successfully for liners cleaning.

7 TROUBLESHOOTING

The GC is a complex analytical apparatus with microprocessor computer facilities, thus its maintenance should be carried out by manufacturer or its regional representative, except for:

- Fuses replacement;
- Elimination of leaks in gas connections by replacement of seals;
- Washing of detectors and inlets;
- replacement of consumables in inlet (liner, O-ring, septum);
- DFPC replacement;
- Replacement or regeneration of sorbent in gas traps;
- Replacement or conditioning chromatographic columns.

Parameters, effecting chromatographic analysis quality, are controlled mainly automatically, appropriate messages display on PC monitor and GC display once inadmissible deviations appear (Check prm LED indicator lights in this case).

Units built in GC and accompanied devices require periodic maintenance. Maintenance procedures for the GC and units are described in corresponding sections of current User Manual.

Below (Table 7.1) common malfunctions, causes and recommendations for their elimination are described.

Table 7.1 – Troubleshooting

Malfunction type	**Possible cause**	**Recommendation**
No peaks on chromatogram	Needle of sampling syringe has been jammed (the same for autosamplers)	Needle should be repaired (cleaned) or syringe should be replaced
	Gas leak in inlet, in columns connection joints, through inlet septum	Replace seals. Replace septum.
	Column is broken up	Change the column or joint split ends
	Column's oven temperature is too low	Increase oven temperature
	Inlet temperature is too low, sample is not vaporized	Increase inlet temperature
	Leakage in the section "DFPC – inlet – column – detector"	Tighten connections, replace sealing elements
	There is no flame in flame detectors due to hydrogen or air leakages	Eliminate leakages
	For ECD detector, signal went of scale	Blow out gas lines from oxygen, use high purity nitrogen only (oxygen shall not exceed 1ppm)
Reduction (below than usual) peaks heights at normal retention time	Sample leakage in syringe or gas leakage in the section "column-detector".	Replace syringe, eliminate leakage
	Detector low sensitivity	Wash detector. Check gases purity and diagnostic parameters of the detector
Reduction (below than usual) peaks heights at increasing retention time	Carrier gas flow reduction in column due to gas leakage at the section "DFPC – inlet – column"	Eliminate leakage
	Inlet liner is broken	Replace liner
Unstable drift of baseline	Detector, inlet are polluted	Wash detector, inlet
	Gas lines leakages	Reveal and eliminate gas leakages
	Carrier gas and auxiliary gases pollution	Gas-purifiers sorbent should be replaced or regenerated; gas cylinders should be replaced.
	Low gas pressure in gas cylinder (below 0.5MPa)	Replace gas cylinder

Abnormally big noise amplitude of the baseline	Chromatograph is not properly grounded	Provide proper apparatus grounding (additional as well)
	Causes similar to baseline unstable drift	
Inadmissible gas flow deviation in one channel	Gas leakage at the area "flow converter-valve" DFC (DPC). Failure of DFC (DPC)	Replace gas flow controller
Inadmissible voltage deviation on DFPC valve	Output pressure is out of range	Set required gas pressure value
	Failure of DFC	Replace DFC
	High column pneumoresistance	Replace sorbent in column, replace the column
No signal of detector	Failure of electrometric amplifier of appropriate channel. Micro controllers' failure	Contact with manufacturer
	Chromatograph is in "Test" mode	Set "Operation" mode
	Auxiliary gas lines leakages	Eliminate leakages
	Columns or plugs are not installed on detector fitting	Install columns or plugs
	Failure of ignition wire	Replace ignition wire
	No voltage on ignition wire	Contact with manufacturer
There is no heating of the column thermostat (the set value is 0). Control Panel displays an error – "Heating is prohibited. Check the carrier gas."	One or more DFPC does not work out the specified consumption.	Ensure that the carrier gas is supplied to the chromatograph.

8 APPENDIX A (INFORMATIONAL)

Referenced documents.

Document	Name
GOST 12.1.004-91	Fire safety. General requirements
GOST 12.1.005-88	General sanitary-hygienic requirements to air of working area
GOST 12.1.007-76	Occupational safety standards system. Noxious substances. Classification and general safety requirements
GOST 15150-69	Machines, instruments and other industrial products. Modifications for different climatic regions. Categories, operating, storage and transportation conditions as to environment climatic aspects influence
GOST 17433-80	Industrial purity. Compressed air. Pollution classes
NRB-99	Radiation standard rates
OSPORB -99	Basic Sanitary Regulations for Radiation Safety Provision
PBZ-576-03	Rules of lay-out and safe operation of vessels working under pressure
POT RM-016-2001	Inter-industrial rules of protection of labor (safety rules) during operation of electrical equipment
PR 50.2.006–94	Measuring instruments verification. Arrangement and procedures
SanPiN 2.6.1.1015-01	Hygienic Requirements to Radioisotope Instruments Design and Operation
214.00045-51I	"Chromatec Analytic" software. User manual
214.0.045.064I	Method for gas flow rate measurement using burette and stop-watch.
214.2.840.043DS	Hardware and software Bundle for medical researches on the basis of chromatograph "Chromatec – Crystal 5000". Operating documents sheet
214.2.840.043-01D	Hardware and software Bundle for medical researches on the basis of chromatograph "Chromatec – Crystal 5000". Performance verification procedure
214.2.840.043LB	Hardware and software Bundle for medical researches on the basis of chromatograph "Chromatec – Crystal 5000". Logbook
214.2.840.043UM	Hardware and software Bundle for medical researches on the basis of chromatograph "Chromatec – Crystal 5000". User manual
214.2.840.073DS	Chromatograph "Chromatec – Crystal 9000". Operating documents sheet
214.2.840.073V	Chromatograph "Chromatec – Crystal 9000". Performance verification procedure
214.2.840.073LB	Chromatograph "Chromatec – Crystal 9000". Logbook

Document	Name
214.2.840.073UM	Chromatograph "Chromatec – Crystal 9000". User manual
214.4.078.000UM	Gas fitting set. User manual
214.5.884.092UM	Trap 20.0. User manual
	Rules of operation of electrical equipment
	Rules of technical operation of consuming electrical equipment