CHROM G10 FE

GAS CHROMATOGRAPH

OPERATING MANUAL

1. Introduction

CHROM G10 FE is an analytical, two channel gas chromatograph equipped with a flame ionisation detector (FID) and an electron capture detector (ECD). The instrument is controlled by an in-built microcomputer. Operating conditions are entered by means of a keyboard situated in the left side of an upper slanting panel. The entered and measured values are displayed on an eight lines graphic display above the keyboard. The temperature of the injector and the detector can be set in broad range, the column oven temperature can be programmed in six isothermal segments and five segments of linearly increasing temperature. The carrier gases pressure and flow are controlled electronically and can be programmed in two isobaric segments and one segment of linearly rising pressure. The flow rates of gases entering the FID (hydrogen, air, make-up gas) are also controlled electronically and can be set by means of a keyboard. Make up gas for ECD is controlled manually.

The column oven (overall volume 10 l) can house one or two standard columns of any manufacturer. Its strong side fan ensures good homogenity of the hot air stream and uniform temperature of the air inside the oven. Two additional small fans are used to pump cold air into the oven during cooling or working in lower temperature range. There are three flaps which position - and thus an amount of cold and hot air in the oven - is controlled by a processor unit. It allows to keep perfect homogenity of inner temperature in periodes of increasing or decreasing temperature as well.

The injectors block, the electromagnetic valves closing septums washing capillaries, the detector heads, the splitter electromagnetic valves and high-precision needle splitter valves are installed inside the oven cover. Electronics of the flame ionisation detector is mounted in the oven cover too inside a shielded aluminium box and connected firmly to the detector head - an arrangement that reduces noise and increases detector sensitivity.

This manual will teach you how to operate the gas chromatograph and perform analyses on your own. Chapter 1 describes the instrument and explains the functions of individual control elements, Chapter 2 deals with gas chromatographic analyses, Chapter 3 explains the effect of individual parameters on the measurement conditions, and Chapter 4 deals with defects caused by incorrect operation and how to remove them.

ATTENTION

The gas chromatograph you have received is accessorized with ECD detector, which contents beta radiator – a foil with isotopic Ni64 covering. Servicing the instrument usual way assures no contamination danger of your working environment and no harm to the ECD detector. However it is necessary to attend to the following rules:

1. **maximum working temperature of the detector is 350 °C. This value is also the maximum working temperature of the entire gas chromatograph. You should not increase this temperature by eventual adjusting of the instrument. By increasing the temperature the volatile Ni carbonile could occur and your working environment could be contaminated by leaked radiation.**
2. **do not in any case disassemble the ECD detector as by the current assembly the beta radiation is screened out so you cannot come in contact with it.**
3. **do not warm up the detector for the working temperature, if there is now carrier gas flowing through it. Otherwise the Ni foil can be damaged by creation of oxides.**
4. **before heating up the thermostat room, the detector needs to be heated up on the temperature at least 10 °C higher. By following this procedure you avoid the contamination of the radioactive foil by substances coming out of the column. Such a contamination could decrease the sensitivity of the detector. Please note that the minimum working temperature of the detector is 300 °C.**

2. Fundamental Technical Specifications of Gas Chromatograph CHROM G10 FE

Weight	35 kg
Dimensions (h x w x d)	415 mm x 420 mm x 540 mm
Oven dimensions	120 mm width x 240 mm height , volume 10 l
Conditions of use	10 °C – 40 °C
Power	230 V, 1800 W
Oven temperature programme	5 ramps, 6 isothermal, max. increase 100 °C/min 1 °C temperature setting
Oven temperature	Ambient + 4°C – 450 °C
Oven cooling	From 400 °C to 50°C in 5 min From 300°C to 50°C in 3,5 min
Injectors simultaneously working, common temp. control	2 x split-splitless for capillary columns and on column for packed columns
Split-splitless injector	Programmed splitter closing, programmed delay
Detectors, 2 simultaneously working, common temp. control	a) FID (+ NPD possibility) d) ECD, Ni 64, AC (+ DC) mode
FID detector	Max. temperature 450°C Detection limit 2 pg C/s Linearity 10,000,000
ECD detector	Max. temperature 350°C Detection limit 20 fg/s (lindane) Linearity 10,000
Carrier gas, 2 independent	El. pressure control, 2 ramp, 3 isobaric El. flow control 5,0 – 50,0 ml/min Max. Pressure 2,5 bar Max. pressure increase 1 bar/min Precision 0,005 bar Max. total flow 150 ml/min
Make up gas (FID, ECD)	El. flow control 14 – 32 ml/min (nitrogen or helium setting)
Air (FID)	El. flow control 50 - 156 ml/min
Hydrogen (FID)	El. flow control 22 – 61 ml/min
Display	graphic, 240x60, illuminated, 7 lines
Keyboard	8 buttons

3.1 Gas Chromatograph Control Elements

The user communicates with the instrument *via* an alphanumeric display and a keyboard situated in the upper part of the front panel of the unit.

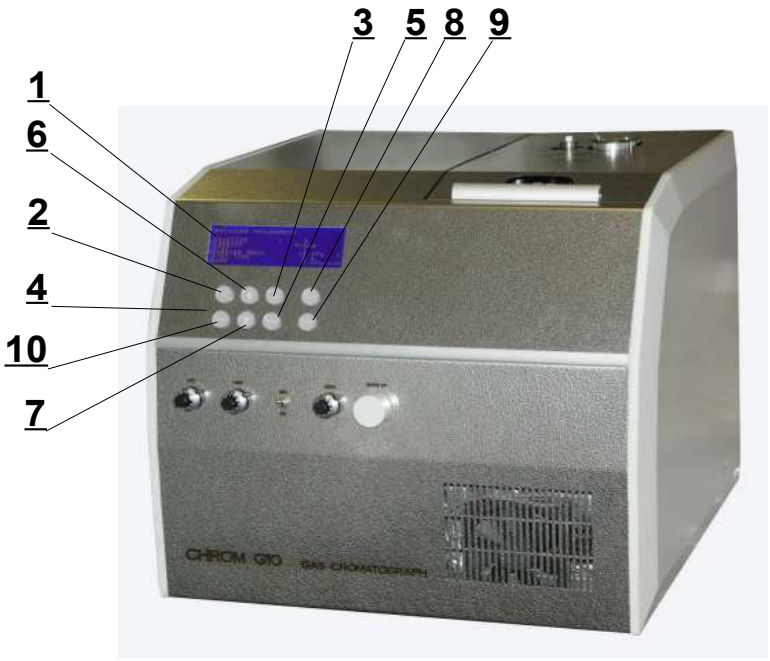
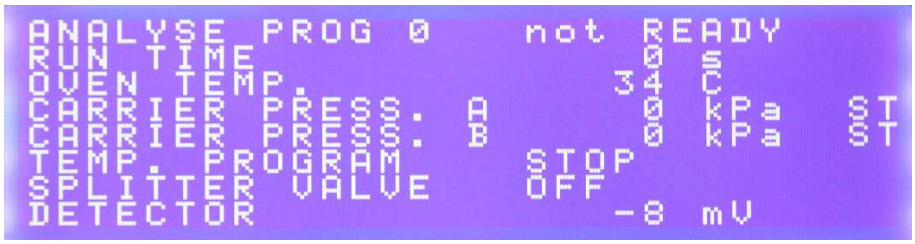


Fig. 1 : The front panel of gas chromatograph

1 Graphic display
2 Button used to decrease the entered numeric value
3 Button used to increase the entered numeric value
4 Keyboard
5 ENTER key used to confirm displayed value
6 Button used to list through the operating parameters table upwards
7 Button used to list through the operating parameters table downwards
8 START button used to start the time program.
9 STOP button used to stop the time program
10 SHIFT button for switching among screens

The instrument is switched on by means of the power switch MAINS in the right side of the rear panel; the message

is displayed and the gas chromatograph then goes directly to analyses or over to the mode where individual operating conditions can be entered. Left side arrow indicates which part of the nemu will be selected using ENTER button. Using START ANALYSE choice, the display indicates the instrument status:



First line of the display indicates program number (from a memory) and at the moment status (not READY, READY, RUN, STOP, COOLING).

Second line indicates time when program is running, third oven temperature, forth and fifth carrier gas pressures, sixth program status, seventh splitter status and the last one FID signal level in mV. Program is started by a START button.

Before the above described procedure is possible, right data have to be entered into the instrument and optionally saved in the memory going from first display to the line PROGRAMME TEMPERATURE and pressing ENTER, next display is shown:

On the second line injector and on third one detector temperature can be set in each case confirmed by ENTER. Heading plates are common for A and B channels and only one value is set for both. Number of a step (1 - 5) is then selected on next row and ginning step oven temperature, step time and a ramp parameters are defined. For example if you like to program a standard analysis with one temperature increase, following data are entered

STEP 1
OVEN TEMP. 50
STEP TIME 300
RAMP 0

then

STEP 2
OVEN TEMP. 50
STEP TIME 600
RAMP 10

and

STEP 3
OVEN TEMP. 150
STEP TIME 500
RAMP 0

It is not necessary to enter temperature decreasing data, cooling is done automatically.

Using SHIFT button one comes again to the first display and can choose pressure and flow programming on third row. Following display appears:

Injector A or B can be selected. Carrier gas (second line) pressure or flow rate can be programmed. Up to 5 pressure steps number can be programmed on fourth line and corresponding pressure and time on the fifth and sixth lines. A ramp between first and second step can be set on the last line.

If “flow” selection on third row is used next display appears:

Flow rate relatives to atmospheric pressure can be set either for A or B channel in the range 5 ml/min. - 50 ml/min.

Using repeatedly SHIFT button and coming back to the first screen, a FID detector properties can be programmed on following screen:

Activating second line instruction, detector signal is going near to 0 V value. Third line allows to shift signal zero to defined level (offset function) in mV. Sensitivity of the detector can be set on 1, 10, 100, 1000 level. Highest sensitivity is on level 1. Last line informs about actual value of detector signal.

Using again repeatedly SHIFT button and coming back to the first screen, auxiliary gases and splitter properties can be programmed on following screen:

Gases programming is relative to FID detector. ECD detector make up flow rate is set manually. Splitter opening time (fifth row) is common for both channels as well as splitter delay time value. Splitter opening delay is used in some highly sensitive analyses and means that splitter is not opened when START button is activated, but later.

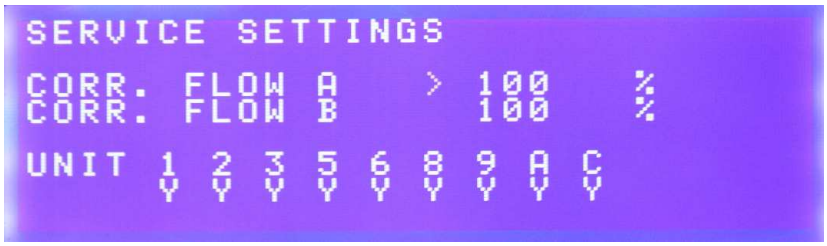
Using again repeatedly SHIFT button and coming back to the first screen, a memory programming is to be selected on seventh row.

This screen is simple and allows to enter all programmed parameters to inner computer memory. Up to 10 different programs can be stored and restored in memories 0 - 9.

The first screen has one next (ninth) row which is obviously not seen:

Using a selection SERVICE SETTINGS, following screen appears:

Using proper password, one can go to the last screen in the selection:



Service settings allows to calibrate carrier gas flow rates depending on the type of gas used. This process is reserved for servicemen only. Last two lines are used to check a function of electronic boards.

3.2 ECD control elements

ECD control elements are focused on the left bottom part of the front panel - see Fig. 2. There is (from left to the right): DC mode current control potenciometer, pulse mode current potenciometer, pulese mode/DC mode switch, zero potenciometer and make-up needle valve

Fig. 2 : The ECD control elements

3.3 Splitter flow rate control

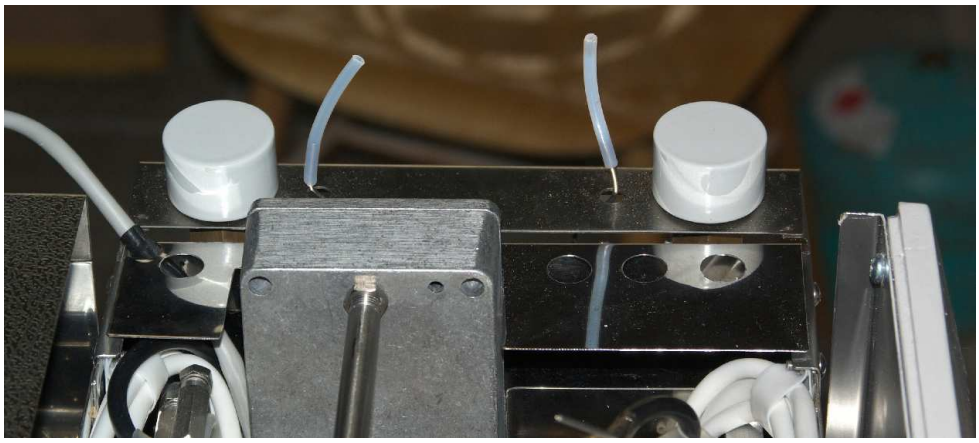


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Fig. 3 : The splitter valves

Splitter valves are situated in the most rear part of oven cover. On the left side is a needle valve for channel A and on the right side is a needle valve for channel B. There are short metal capillaries with PTFE end parts on each valve to allow flow rate measurements. Please turn needle valves gently in order not to destroy precise needles inside.

3.4 Gas Distribution and Control

In connection with chromatographic process and the flame ionisation detector operation the chromatograph uses three gases: nitrogen (alternatively helium) as the carrier gas, hydrogen, and air necessary for FID flame burning. In connection with chromatographic process and the electron capture detector operation the chromatograph uses nitrogen

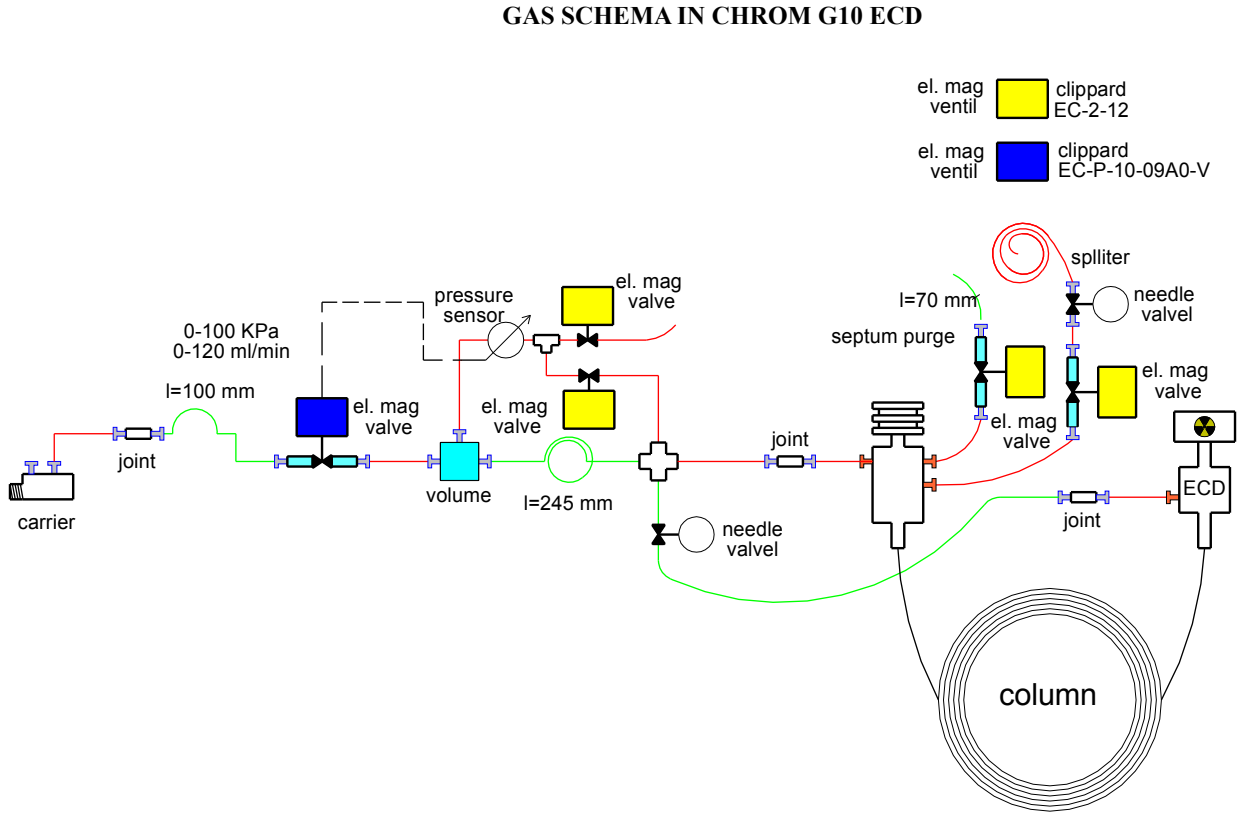

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Fig. 4 : The gas distribution in ECD channel

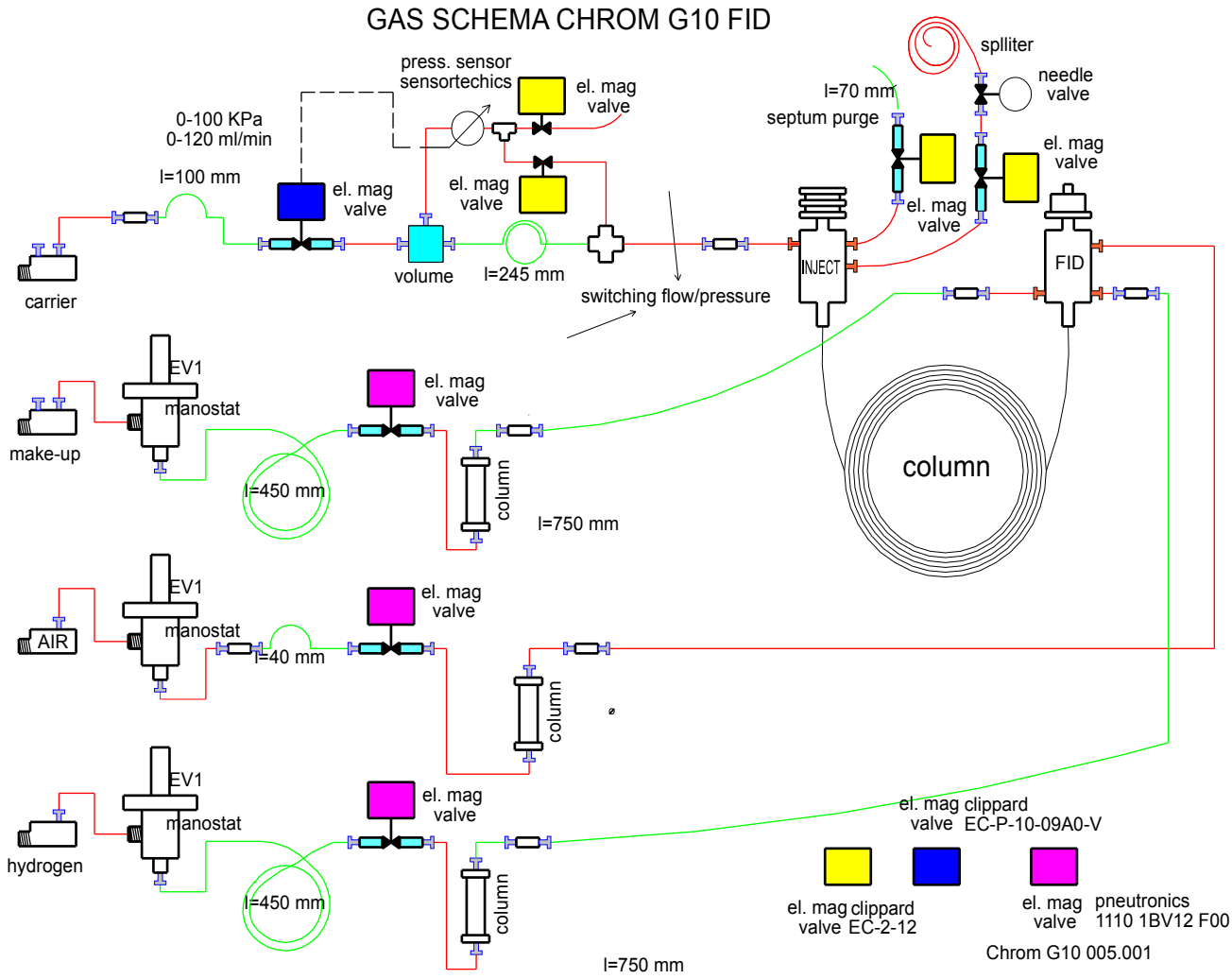

Fig. 5 : The gas distribution in FID channel

(alternatively argon) as the carrier gas. The gases proceed *via* capillaries from individual pressure cylinders to the appropriate connectors situated in the rear chromatograph panel. The inlet capillaries are attached to the connectors by cap nuts provided with rubber O-rings. The gas distribution is shown schematically in Figure .

A part of ECD carrier gas passes to an electronic feedback controller with proportional electromagnetic valve and to the detector as the so-called make-up gas to ensure optimum operating conditions of the detector and to keep highest purity of the make-up gas. FID make-up gas is entered from an independent inlet through the same type regulator as are used for other auxiliary gases.

The carrier gases go to an proportional electromagnetic feedback control valve with pressure sensor securing electronically constant pressure across the column and then to the injection block; a minor fraction is branched off *via* capillary to provide for permanent septum purge.

The carrier gases proportional electromagnetic feedback control unit is designed such way, that pressure sensor controls not an absolute pressure, but a differential pressure on a calibrated capillary. This way is used in so called “flow mode” which is securing electronically constant flow across the column. The permanent septum purge is closed by an electromagnetic valve and is open for short time only during start procedure.

From each injector one part of the carrier gas proceeds to the column, another part is led to an electromagnetic valve and the splitter needle valve where the so-called splitter ratio is set to divide the injected volume to the part entering the capillary column and the part vented outside the instrument. The above arrangement is only important for analyses with a capillary column where the injected sample must be further divided into a part entering the column and a second part leaving the system. If a packed column is mounted, the splitter outlet capillary is closed and the flow is not split.

Make-up gas, air and hydrogen for FID proceed from their respective connectors through electronic controllers to the detector where a hydrogen flame burns between two electrodes; ionisation of the flame indicates the arrival of an elution zone.

Figure 3 shows a detailed view inside chromatograph cover (without upper sheet). Carrier gas proceeds from the electromagnetic valve through capillary (33) to the injector block, where it passes to the liner where it its divided into a stream led to the column and a stream vented *via* the splitter. The latter is led by capillary (36) from the bottom of the injection block to electromagnetic valve (37) and thereafter by capillary (38) to needle valve (39) used to set the flowrate of the carrier gas stream vented by means of outlet capillary (40). The ratio of flowrates of carrier gas entering the detector *via* the column and that vented *via* the splitter defines the proportion in which the injected sample is divided into a part entering the column and the vented part.

3.5 Rear Panel

The power switch is in the right side close to the power connector. Inlet connectors for individual gases are situated in the upper right part of the unit; the copper inlet capillaries for carrier gases, FID make-up, hydrogen, and air are connected by means of nuts and O-rings.

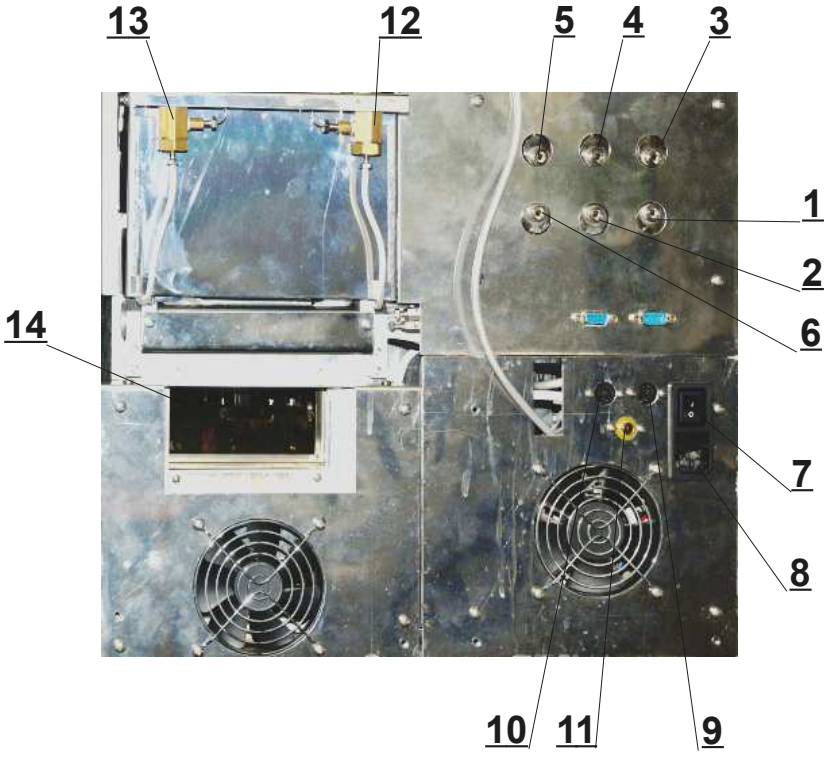

Fig. 6 : The rear panel

1 - inlet carrier channel A, FID
2 - inlet carrier channel B, ECD
3 - inlet make up FID
4 - inlet air FID
5 - inlet hydrogen FID
6 - not used
7 - main switch
8 - fuse 10 A
9 - datastation Clarity, channel A (FID)
10 - datastation Clarity, channel B (ECD)
11 - datastation Clarity START cable
12 - splitter needle valve channel A - FID
13 - splitter needle valve channel B ECD
14 - exhaust flap

A five-pin connectors for the integrator are under gases inputs. There is also the start connector and autosampler connector.

3.6 Oven Cover

Figure 7 shows the gas chromatograph from above. The cooling devices for the injector block with the needle guide for injecting a sample through a septum onto the column is in the front part of the cover; the rear part of the oven cover houses the FID detector and the ECD

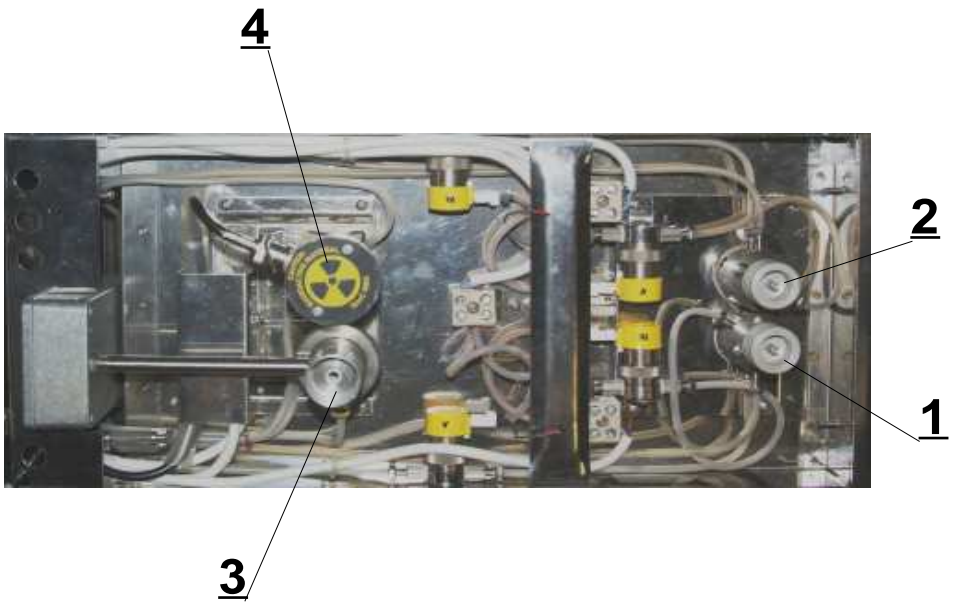

Fig. 7 : Upper view

1 - Injector channel A, FID
2 - Injector channel B, ECD
3 - FID
4 - ECD

detector both with the inlet capillaries of hydrogen, air and the make-up gases. The metal lid of the cover ensuring constant temperature inside is fastened by two hand bolts on instrument back and two supporting members on the inner cover front side. It is recommended to remove and to install the lid when cover is partially opened. Both bolts are released and the lid is turneda bit back. There is the possibility to remove it then.

3.7 Oven

Figure 8 depicts a cross-section of the thermostat. Its side part includes the electrically driven large fan ensuring intensive air circulation. Air is heated by heating coils with computer-controlled power depending to the required, preset oven temperature. Air from the fan passes the heating coils and is directed to the oven space. In addition to the heating coils the temperature inside the thermostat is controlled by two cooling flaps (equipped with small fans each) and one big flap for hot air exhaust. Cooling flaps - when open - allow cool air from the surroundings to enter the thermostat. The flaps are fully opened only when the oven is cooled to the initial temperature at the end of an analysis. In instances when the preset oven temperature is below 120°C they are opened partially and the stage of opening is controlled by a processor unit.

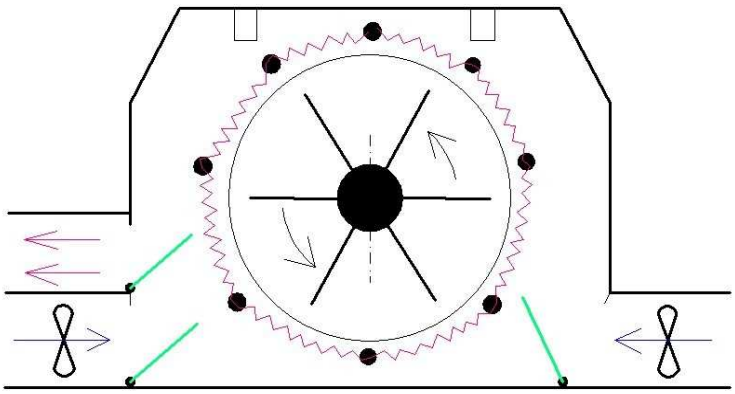

Fig. 8: The gas chromatograph CHROM G10 oven crossection

The column (capillary or packed) is attached to the hinged thermostat cover. A packed column is attached to the injector block and the detector block by a stainless steel clamp, a plastic cone seal and a cap nut. A capillary column is attached by means of a metal holder with a clamp and screw used to fix a wire frame supporting the coils of the capillary column. Once the column is so fixed the inlet and outlet is attached to the thermostat head by cap nuts and stainless steel inserts and - the column is run through the holes in the insert and fixed by a small cap nut, metal washer and plastic seal as described below.

3.8 Injector Block

A cross-sectional view of the injector block with attached capillary column is in Fig. 9. The block consists of two metallic parts held together by three screws. An air cooler that also

presses the needle guide to septum is screwed onto the upper part. The needle is led through the septum to the injection chamber formed when the upper and bottom part of the injector block are screwed together. The capillary feeding carrier gas from the control valve enters the upper part of the injector block; two capillaries and from the upper part of the block lead a small stream of carrier gas across the septum to flush it. All inlet and outlet capillaries are attached to the injector block by through-bolts and cone metallic seals.

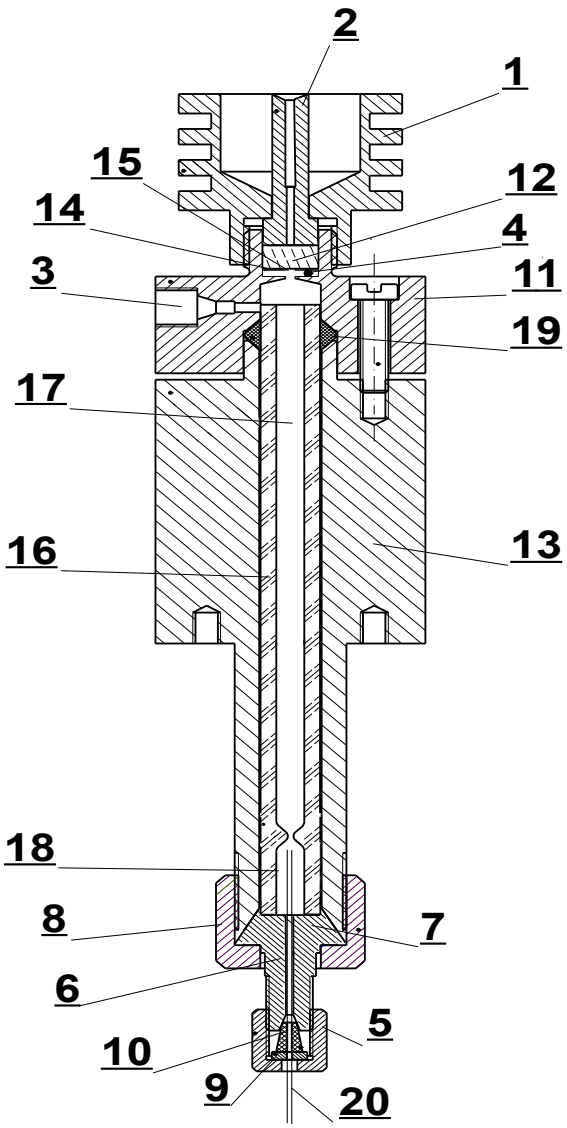

Fig. 9: Cross – section of the injector block with attached capillary column

1 – Cooling device
2 – Needle guide
3 – Carrier gas inlet.
4 – Outlet capillary.
5 – Detector cap nut
6 – Drilled hole for capillary column.
7 – Capillary column brass insert
8 –Insert cap nut.
9 –Metal washer.

10 –Plastic seal.
11 –Upper part of injection chamber.
12 – Septum.
13 –Botton part of injection chamber.
14 –Presed in metal cap.
15 –Drilled hole for needle.
16 – Liner.
17 –Upper part of liner.
18 –Bottom part of liner.
19 – Teflon ring.
20 –Capillary column.

The capillary feeding carrier gas from the control valve enters the upper part of the injector block; capillary from the upper part of the block lead a small stream of carrier gas across the septum to flush it.

To prevent possible contamination of the column by products of septum degradation the space beneath the septum is separated from the injection chamber by the pressed-in metal cup with drilled hole through which the needle passes.

The injected sample is evaporated in liner - a glass rod constricted in the lower part. Glass wool is put in the upper part beneath the needle end to ensure that the sample is wiped off the needle tip. Sample vapour leaves the upper part of the liner and enters the bottom part with the inlet of the capillary column; the stream is split here to a part entering the column and a part vented out of the splitter.

A circular plastic seal or silicon rubber ring is slid onto the upper part of the liner to separate its upper and bottom parts and ensure appropriate carrier gas stream. In this arrangement carrier gas passes from the upper part of the liner *via* the constriction to the bottom part where it is split into a stream entering the column and that vented through outlet capillary (36), connected to the electromagnetic splitter valve.

Capillary column passes through a hole drilled in the stainless seel insert and is attached to it by means of cap nut, metal washer and plastic cone seal. The metal washer is attached to the block by cap nut. To prevent potentially harmful contamination of the column by products of septum degradation when the septum is overheated above the maximum allowed temperature, hole is drilled transversally through the brass washer with metal cap fitted on its upper part to separate the part of the liner with the column end from the space in contact with the plastic seal.

To ensure adequate sample evaporation the bottom part of the liner is inserted in a heated block whose temperature can be set separately from the thermostat temperature. It is necessary to take in the account that temperature inside the liner is in any case lower than displayed temperature of the heated injector block. It can have influence on the analyses results when boiling points of detected substances are to near to the temperature of the injector.

3.9 Flame Ionisation Detector

Similarly to the injector block a thermostated metal block houses the detector. The column is connected to the detector inlet in its bottom part, but - again as in the injector block - the connection differs between capillary and packed columns.

A packed column is attached by a cap nut and a cone plastic seal with a stainless steel insert in-between; the column is inserted end-to-end and its opening abuts the flame ionisation detector nozzle.

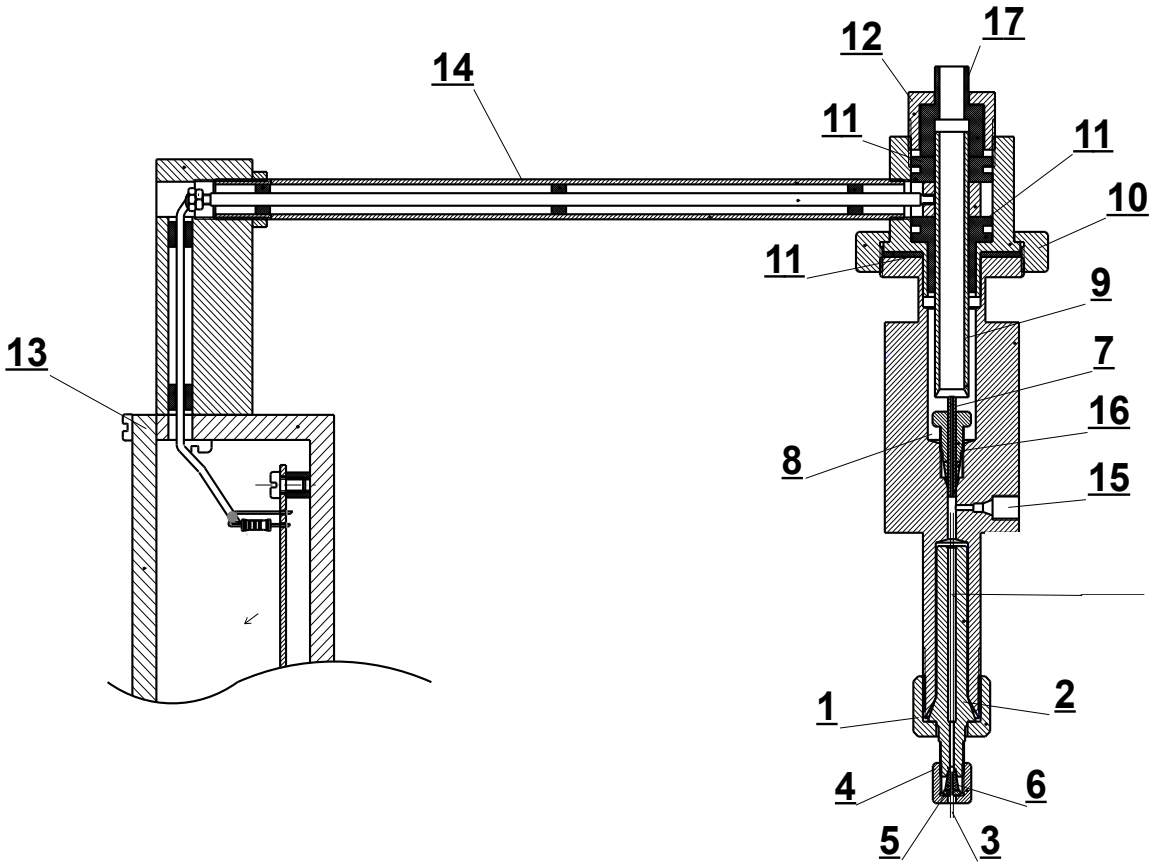

Fig. 10 : Cross section of the FID. Attachement of a capillary column

1 – Cap nut
2 – Capillary column insert.
3 –Capillary column.
4 – Cap nut catching capillary column to insert.
5 – Washer.
6 –Cone seal.
7 – FID nozzle
8 – Carrier gas inlet.
9 – Metal tube
10 –Cap nut.
11 – PTFE insulation.
12 – Metal cap with thread.
13 – Aluminium casing with electronics of FID detector.
14 –Metal tube connecting body of FID with electronics
15 – Hydrogen inlet
16 – Nozzle screw
17 –Gas outlet.

A capillary column must be connected to the detector by means of the connecting insert, consisting of a stainless steel cylinder with recess for a seal, pressed by means of a screw cup to the bottom detector inlet.

Capillary column is attached to it - as in case of the connecting insert of the injector block - by means of a small cap nut and cone seal. The capillary column is first inserted in the insert with the larger screw cup in place and attached by the small screw cup in a manner ensuring that its end is at the level of the upper part of hole in the centre of the insert.

Carrier gas passes from the column to the space beneath the nozzle where it is mixed with hydrogen entering *via* the capillary from the corresponding control element; the mixture then enters the FID nozzle. The part housing the nozzle is threaded and screwed onto the detector body, and can be removed by means of a special wrench.

Air and carrier gas added as make-up to ensure optimum conditions of the detector operation are led to the nozzle by the appropriate capillaries.

The sample concentration the flame ionisation detector (Fig. 8) is monitored such way, that the electric current passing through the flame burning between the electrodes is measured. The nozzle represents one electrode; the other electrode is a metal tube situated above the flame. Thus, the detector body consists of two insulated parts - the bottom part attached to the thermostat cover, and the upper part connected to the detector electronics units situated in an aluminium casing.

The upper detector part can be removed by unscrewing the large screw cup; the two parts are insulated from each other by a PTFE seal. The tube comprising the working electrode is inserted in a PTFE insert to insulate it from those parts of the detector electrically connected to the other electrode; a PTFE cap with a vent-pipe outlet that vents the mixture of gases leaving the detector is fitted onto the working electrode and attached to the detector by a threaded metal part.

The reason underlying the above arrangement is the necessity to prevent condensation of water inside the detector, since - when wet - the Teflon insert holding the electrode short-circuits the electrodes and disables the detector temporarily (see Chapter 4 - Removal of Minor Defects - for additional details).

To reduce detector noise the FID electronics is situated in an aluminium casing (82) connected to the upper part of the detector by tube (82) protecting the connecting cable.

3.10 Flectron Capture Detector

Please see the picture Fig. 9 for the cross-section of the ECD detector. Detector (similarly to the injector) is placed in a heated metal block, whose temperature is settable by 1 °C. The foil covered with isotopic Ni 64 (beta radiator) and rolled in the respective detection unit.

The foil constitutes a cylinder, which in the same time represents one of the electrodes. The second electrode is a wire placed as the axis of this cylinder. ECD detector itself is stored in a tube, through which is flowing the carrier gas coming from the column. A make-up gas is added to the carrier gas flow using a needle valve in order to guarantee optimal flow rate in the detector.

This so-called make up of the carrier gas needs to be done in the case of the capillary columns with usual flow-rate of 1 – 5 ml/min. In the case of packed column the carrier gas make up is usually not proceeded.

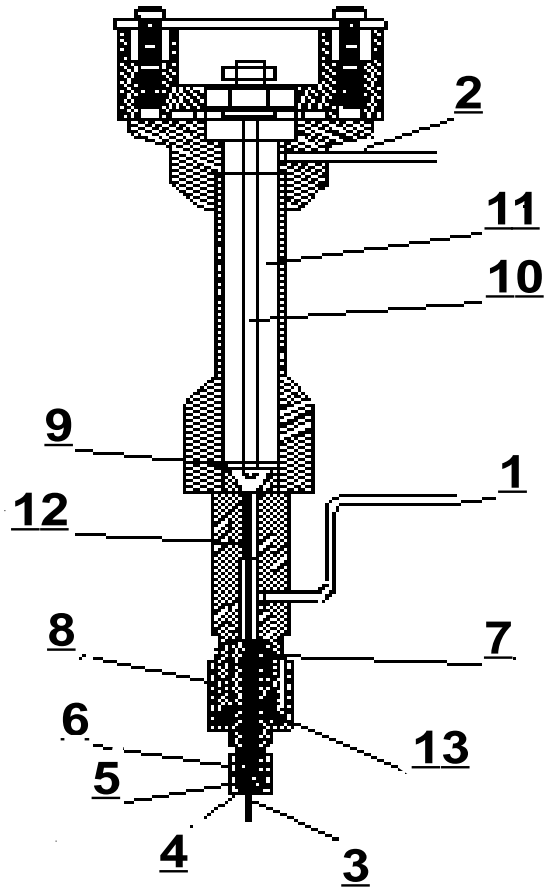

Fig. 11: ECD detector cross section

1 – make up gas
2 – carrier gas output
3 – column
4 – metal insert
5 – ferrule
6 – nut fixing capillary column
7 – connecting insert
8 – nut fixing capillary column insert to the detector
9 - Ni 64 plate
10 – collecting electrode
11 – detection space
12 – carrier gas channel
13 – sealing surface

The carrier gas is drained away from the detector by means of a capillary. The space where the detection unit is placed shapes a room connected with the input of the column by a narrow channel. Geometric alignment disallows the flashing of a the beta radiation and protects the detection unit during the column mounting.

The connection of columns to the detector is identical with the connection of injector. After insertion into the input of the ECD detector, the packed column is fixed by means of nut, metal pad and conical gasket. The capillary column is connected by means of brass pad, which is fixed to the input of the detector by means of conical gasket lathed into this pad and a nut. Capillary column is inserted into the mouth bored in the centre of the brass pad in such a way, that the capillary passes through the pad at the full length and is fixed to it by a conical gasket, metal pad and small nut.

3.11 Mounting Chromatographic Columns

Chromatographic column, where components of the analysed mixture are separated, is the core element of a gas chromatograph. Two types of columns - packed and capillary - are used in the Chrom G10 gas chromatograph.

A packed column is a coiled tube. Components are separated on a layer of appropriate material packed in the column. Glass wool is placed at both column ends to prevent the sorbent from escaping and reduce the undesirable dead volumes. The column is mounted in the chromatograph by means of a ring seal and a cap nut.

In mounting a capillary column a glass insert - liner - constricted in the middle and a metal insert are placed in the bottom part of the injection block. The capillary column protrudes up to the constriction in the glass liner rod. Both the upper part of the liner insert and the upper part of the capillary column are sealed by PTFE O-rings. Similarly as in the packed column a metal insert - reduction - is mounted by means of a cap nut. The capillary column is attached to the reduction by a smaller cap nut, a metal washer and a cone seal. To prevent contamination an adjusting piece is inserted to separate the lower part of the liner from the space where the capillary column seal is situated and prevents the products of its thermal degradation, released when the maximum allowed temperature is exceeded, from entering the column.

The capillary column is attached in the same manner to the metal reduction piece inserted in the detector instead of a packed column (see Figure 9).

To fix the flexible capillary column it is mounted by means of a metal sheet holder screwed onto the thermostat cover.

4.0 Analyses Performed with the CHROM G10 FE Gas Chromatograph

4.1 Gas Chromatograph Installation

1) Unpack the instrument and attach the inlet capillaries to the corresponding pressure vessels by screwing them onto the manometer assembly outlet.

Note: Successful gas chromatographic analyses with a flame ionisation detector require gases of the highest purity - at least medicinal (better synthetic) air, nitrogen 4.8 and hydrogen 4.0. Any admixtures of organic compounds are undesirable since they increase the residual current and reduce detector sensitivity.

2) Run the ends of the inlet capillaries through the black rubber O-rings and attach the capillaries by means of the cap nuts to the connectors situated at the rear panel of the instrument. Tighten the cap nuts by means of a spanner.

3) Connect the integrator board cable to the five-pin connector at the rear panel of the chromatograph.

4) Set 3 bar (40 PSI) on the pressure cylinder regulators. Open the regulator, leave the outlet valve open, check by means of a detergent solution tightness of all connections of all gases at the respective pressure cylinders and tighten the nuts as required. Make sure that the needle valves at the front panel are closed, open the outlet valves one by one and check by a detergent solution tightness of all connections to the gas chromatograph.

5) Unscrew the cooler of the injector block and remove it together with the needle guide.

6) Use a new septum and fix it with addition of a stainless steel wire (in chromatograph accessories). String on the wire the cooler block following with the septum (if the septum has not a predrilled hole, take care to pierce the wire in the centrum of the septum).

7) Put all parts into the injector. Be sure that metal insert on the top of the injector is turned with the larger central hole (hole is shaped like the conus) up. Screw tightely the cooler to its place.

8) The instrument is now ready for installation of a chromatographic column. The following procedure differs depending on whether a capillary or a packed column is to be installed.

4.1.1 Installing a Packed Column

1) On each column end place the cup nut, the brass insert and the cone seal in that order.

2) Unscrew the injector block cooler and remove it together with the septum. Unscrew the three screws in the upper part of the injector block and remove the upper part.

3) Push the column into the injector block and (all the way) into the detector. If the column length is correct, it will protrude from the lower part of the injector block by 2 to 3 mm.

4) Place the Teflon O-ring onto the column end protruding from the bottom part of the injection block, and fix the column by tightening the cup nut on the detector - do not use a wrench yet.

5) Carefully place the upper part of the injector block on top of the column end protruding from the bottom part and fix it by the three screws. Carefully tighten the screws one by one to prevent damage to the column end.

6) Insert the needle guide into the air cooler and the septum beneath it. Screw the cooler onto the upper part of the injector block and tighten by hand. Finally, tighten the cup nut at the inlet to the injector block by a wrench.

7) Switch the gas chromatograph on, set the carrier gas pressure to 100 kPa and check tightness of all connections by a detergent solution: the capillary that leads carrier gas from the control valve, the capillaries at the septum flush and the capillary leading carrier gas to the splitter (just to make sure - nothing should happen here). Check for leaking carrier gas between the two parts of the injector block where the cooler is screwed on and, finally, at the two cup nuts. Be careful not to contaminate the system - use only very dilute detergent solution and wipe the checked points with distilled water. The check may be performed only when the chromatograph has cooled down.

8) Make sure that the carrier gas needle valve is closed and measure by means of bubble flowmeter the flowrate of the gas leaving the FID detector to verify that carrier gas flows through the column. The actual flowrate depends on the sorbent packed in the column, the column length and the method of packing used.

If all connections are tight and carrier gas passes through the column, the gas chromatograph is ready for measurements with a packed column.

4.1.2 Installing a Capillary Column

A capillary column is connected to the injector block and the detector by brass connectors; a glass constricted tube, the so-called liner, is inserted in the injector block ensuring adequate sample evaporation and its separation into a part entering the column and the other, vented part.

Prior to installing a column it is advisable to examine Figures 6 and 8 that depict the cross-section views of the injector block and the detector entry point. Install a capillary column as follows:

1) Insert a stainless steel inserts into injector and detector (short to the injector, long to the detector. In case of injector put in the insert a matallic cap (67). Fix both inserts with nuts.

2) Unscrew the air cooler from the injector block together with the needle guide and the septum.

3) Remove the fixing screw, the cover handle and the cover lid of the oven part.Unscrew the upper part of the injector block held by three screws and push it to one side.

4) Insert a glass liner into the injector block together with a PTFE sealing ring.

N o t i c e : The length of liner must be 80 mm. If you install new, check it.

Carefully fit the upper part of the injection block onto the bottom part, fix by the three screws and carefully tighten them to ensure appropriate fit and tightness of the two parts. Screw back the air cooler with the needle guide and the septum. Use steel wire to fix the septum in proper position. Assemble the cover lid.

5) Open the oven and fix the capillary column by the wire frame in the column holder by means of the clamp in the upper part of the holder. Fix both ends of the column and check whether the clamp is well screwed down and the wire frame securely fixed to the holder; check whether the column cannot be damaged by the insert in the bottom part of the thermostat when the oven cover is closed, preferably by means of a steel rule, and only then close the oven cover carefully.

6) Put the small cap nut, the metal plate, and the graphite cone seal in that order onto the column and fix it to the injector insert (length of free capillary part is about 40 mm). Correct position of the capillary inside the liner is important: if the end of the capillary reaches

the liner constriction, the injection is not split at all and, if it is too far below it, the actual split ratio may differ from that originally set up.

7) Put the small cap nut, the drilled metal plate and the cone seal in that order on the column end earmarked for the detector, insert the column (free part of the column is 60 mm) in the brass insert and tighten the small cap nut.

8) Open the carrier gas pressure cylinder, switch on the chromatograph and set the pressure across the column to 100 kPa (it is equal 1 bar or 14 PSI).

9) Use a detergent solution to check tightness of all connections - the entry point of carrier gas to the injector block, the outlets of capillaries for septum flushing, and the outlet of the capillary column.

10) Measure the carrier gas flow rate through the column by bubble flowmeter attached to the detector output (all other gases are closed).

11) The capillary column is now installed.

4.1.3 FID Flame Ignition

Provided a stream of carrier gas passes through a column installed in the gas chromatograph already connected to the Claritydatastation, the flame in the detector may be ignited and the FID activated. First it is however necessary to heat the detector block to prevent condensation of water vapour and a short-circuit. Proceed as follow

1) Set up the detector temperature (displayed to the left) to lie at least 20 °C above the highest column temperature and at any rate higher than 180 °C. Confirm the setting by hitting ENTER.

3) Wait at least 15 minutes to allow the detector to reach the preset temperature. Thereafter - provided the relief vent of the detector is warm – set the hydrogen flow from the keyboard to 35 ml/min.

4) Set the air flow rate from the keyboard to about 120 ml/min. This is the optimum value but the detector sensitivity remains essentially constant over a broad range of air flow rates.

5) Wait not less than 10 minutes to stabilize the flow of gases.

6) Set the piezoelectric lighter onto the FID outlet and press the lighter button to ignite the hydrogen flame. Check by formation of water droplets on a mirror or a beaker positioned close to the FID outlet whether the flame is burning.

Note: The flame ignites if the mixture inside the detector lies within the flammability range. A failure to ignite the flame is probably due to the fact that hydrogen concentration lies outside this range - change the air flow

7) Using capillary column set the make up gas flow rate from the keyboard to the value about 15 ml/min.

4.1.4. Preparation of ECD detector for the measuring

We can start with preparing the measuring if: (i) column is installed already on the channel B, (ii) carrier gas is flowing through and (iii) the Clarity data unit is prepared for the measuring as described.. Before the measuring always assure yourself if the right “make up” is set up and if the carrier gas flows through the column.

It is necessary to purge the system before starting the analyses, to remove the substances with a chlorine content. Their transport into the detection cell causes decrease of the sensitivity of the detector.

It is necessary to heat up the detector to temperature of minimum 300 °C first and to start to heat up the column and the injector only after the detector reaches this minimal temperature. If done the other way round, the substances flowing thorough the column would condensate on the detection unit and the detector would lose its sensitivity.

During the preparing steps and purging process, it is absolutely necessary to monitor the flow of the basic line and in case it increases over observable value, decrease the temperature of the thermostat and an injector, so you avoid the contamination. For activating the detector please follow these steps:

1) Switch the chromatograph to pressure setting mode. Using the proper buttons set up the value of the overpressure of the carrier gas on the channel B. Confirm the value by pressing ENTER and wait when the display stops showing a < mark on the right side of the numeric value. The appearance of this mark means that pressure is not yet stable. The display should stop showing this mark within few seconds. If that does not happen, it points to a leakage somewhere on the system and it is necessary to control all the connections.

2) Use the needle valve on the front panel of the instrument to set up the make up to such a value, that the flow-rate on the output of the detector stays between 20 – 30 ml/min

3) On the computer monitor click on the Clarity icon. Enter your name and confirm. In the next window click on the menu “Method“ and choose „Acquisition“. In the „Method Setup window“ is a smaller „Range window“ . Set up the integration scale on 10000 mV.. By clicking on the menu „Monitor“ and choosing „detector signal“ you switch the Clarity into the mode „collection of chromatographic data“ (if in an offer “detector signal” is selected).

4) Set up 600 parts on the potentiometer and wait when the signal becomes stable. Wait for about 60 seconds and then switch the working mode of the ECD to the position IMP.

5) Set up the I-IMP potentiometer to zero and set up the detector signal by potentiometer ZERO to 0 mV. which can be seen on a computer monitor in a window Data Acquisition.

Set up 500 parts on the I-IMP potentiometer. The current will increase to about 2000 mV or more.

6) Enter the 300 °C as detector temperature, confirm by pressing ENTER. Wait till the detector is heated up. Signal is going through a maximum and decreases to initial value. As soon as the signal is stable, set up the temperature of the thermostat to the value, by which the purging of the system should be done. During the purging the baseline decreases. Purging can take even some days and it is proceeded so long until the flow of the basic line is stable. After this stabilization the detector is prepared for the measuring.

7) Change between DC and IMP mode. Polarizing the Ni64 detection foil by DC voltage the measuring is not usually proceeded. This mode is used just for the activation of the detector. After these steps are done, the detector is activated, therefore switch the working mode into the IMP position.

8) Using the potentiometer ZERO set up the detector output signal approximately on value 0 mV. If it is impossible repeat a purging procedure and try again to switch between DC and IMP mode. Check if in each mode signal react on change of current (left side potenciometers). If you can keep in IMP mode current nearly 500 parts on the potenciometer and zero can be set to by a ZERO potenciometer, the ECD detector is prepared for measurement.

4.1.5 Setup of operating conditions

4.1.5.1 Setting up operating conditions from the gas chromatograph keyboard

Once a column has been installed and the detector is heated to the operating temperature, the remaining operating conditions can be set; follow the procedure described below.

1) Set the required injector temperature. Confirm the set value by hitting ENTER.

2) Enter the oven temperature programme the same manner as described. Note that temperature of the oven automatically reaches the preset temperature 1 as the value is confirmed by ENTER. Be sure that sufficient carrier gas flow is secured for the column and it is flushed enough.

3) If the capillary column is used, define the time of splitter opening from the keyboard (usually about 120 s). The time of splitter valve opening is the last parameter set.

4) Set the splitter flow. Splitter needle valve is located on the rear side of the oven cover (see Fig.). Start the programme (opening elmg. splitter valve) and turning needle valve axis you can set proper ratio between column and splitter flow. I this ratio (usually 1:10) the detetector response is going lower. The main reason for splitter use is to prevent large quantity of solvent to come into the small hole of capillary column.

4.1.5.2 Storing the operating conditions in the chromatograph memory

If the currently set operating conditions will be subsequently used for chromatographic analyses, it is advisable to store them in the instrument memory for future use according described procedure..

4.1.6 Column and Gas Chromatograph Conditioning

Since the FID reacts to the presence in carrier gas of any organic compound, the system must be conditioned to raise the detector sensitivity. The objective is to reduce the residual current due to organic compounds released from the column and from the heated parts of the instrument. During the conditioning stage the temperatures of the injector block and the thermostat are maintained at the maximum working limits at which the column will be subsequently operated, and the detector at a temperature higher by 20 °C than that of the thermostat. Leave the instrument running under the above conditions and with the flame burning until the residual current stabilises.

4.2 Analyses with the CHROM G10 FE Gas Chromatograph

4.2.1 Sample injecting

1) For the injecting of the liquid sample (usually 0,5 – 2 ul) use proper microsyringe having injecting needle diameter about 0,5 mm and needle length 50 - 70 mm (for example 1 ul Hamilton 7001 type). For gas injections (volume about 20 – 200 ul) use gastight syringe with the same needle parameters. Using larger needle diameter the septum is damaged soon.

2) Own injection process is important for successful analysis (reproducibility). Prepare the sample for injection. Rinse the syringe sometimes with the sample (if use the syringe with piston in glass part take care not have bubbles in the syringe). Check the temperature of injector cooler to prevent the accident before injecting. Press the START button and wait about one second. Insert the needle into the needle guide, press it firmly but fast into the injector, press the piston and – without waiting – put the needle away of the injector.

4.2.2 Chromatogram registration

Once the gas chromatograph is made ready for measurement by the procedure described in Chapter 2.1 and the integration programme and integrator are connected, a chromatogram can be measured under the preset operating conditions. Proceed as follows:

1) Be sure the display in ANALYSIS mode shows the message READY and an integrator remote cable is connected to JACK type connector on the back of the chromatograph. Note: Remote connector is common for both channels.

2) Fill up the syringe with the analysed sample, adjust the volume, wipe the needle end and inject the sample: insert the needle in the guide and pierce the septum. Simultaneously with sample injection press the START button situated at the front instrument panel, or start the integration programme.

3) Once the chromatogram has been registered, stop the temperature programme by pressing the STOP button. The programme will terminate, both cooling flaps and exhaust flap will open and the displayed message Run will be replaced by Cooling, indicating that rapid cooling of the thermostat to the initial temperature is in progress.

The analysis is thus complete. The operator must now wait for the end of the cooling stage until the thermostat temperature stabilises at the initial value and the message READY is displayed.

4.2.3 End of work and instrument shutdown

Attention!! When terminating work with a gas chromatograph with a capillary column installed, do not forget to cool it down prior to switching the instrument off to prevent clogging. The following procedure is then mandatory:

1) Close the hydrogen and air flow to extinguish the flame (in case FID is used), and close the valves of the corresponding pressure cylinders. Leave the carrier gas pressure cylinder open.

2) Set the injector block temperature to 0 °C. Wait about 10 minutes and only then set the column temperature to 0 °C. Allow the oven to cool down and then set the detector temperature to 0 °C.

Note: The above procedure prevents contamination of the chromatographic system by compounds that might be released from its heated parts and condense on the cool parts.

3) Switch off the integration programme and the computer.

4) Wait until all parts of the chromatograph cool down below a temperature that would endanger the column and only then switch the instrument off and close the carrier gas control elements.

5. Effect of Operating Conditions on the Resulting Chromatogram

In appropriate setup of operating conditions might result in incorrect results of chromatographic analyses; accordingly, this Chapter deals with the effects of resettable parameters on the analytical result - the registered chromatogram.

5.1 Injector Block Temperature

The injected sample evaporates in the injector block. Thorough evaporation of the injected sample and all its components ranks among conditions prerequisite for the success of a chromatographic analysis. Accordingly, the injector temperature must lie above the boiling point of the solvent and to the maximum extent possible prevent condensation of components present in the analysed sample.

If the injector temperature is too low, peaks of less volatile components will be reduced or might disappear altogether. Remedying this defect is easy: increase the injector temperature up to the maximum allowed temperature of 450 °C. When increasing the injector block temperature always keep in mind the following:

a) The analysed sample might change owing to chemical reactions proceeding at elevated temperatures, e.g., at the surface of stainless-steel columns, and the results of analysis need not correspond to the composition of the injected sample.

b) Each column exhibits maximum temperature resistance specified in the manufacturer's technical specifications; the maximum temperature must not be exceeded, not even at the column inlet, otherwise the packing will decompose and result in increased baseline level and after some time also detector contamination. Chromatographic characteristics of a column subject to such temperature load will change.

c) The septum, the glass wool inserted in a packed column or the liner, if chemically modified, and also the plastic seals all exhibit limited temperature resistance. When the maximum working temperature is exceeded, the baseline level is again raised owing to thermal degradation, and leakage at the septum and individual seals might occur.

For these three reasons in deciding on the injector temperature a judicious compromise must be resorted to, taking into account the analysed mixture and the chromatograph at hand. One must also keep in mind that the preset temperature is maintained at the point of the heated block where the temperature sensor PT100 is situated, and may be different at some other places of the injector block. It in fact reaches the maximum value at the point where the injection chamber penetrates the heated metal block, and at other points depends on conditions of heat flow.

5.2 Detector temperature

The detector is heated similarly to the injector since it is important to prevent condensation of compounds leaving the column, a circumstance that may happen if the detector temperature is lower that that of the column.

The hydrogen flame burning inside the detector produces water vapour and, accordingly, all detector parts must be heated above 100 °C to prevent condensation, since a liquid water film on the Teflon insert would short-circuit the electrodes and put the detector temporarily out of commission.

Thus, the detector temperature should be at least 180 °C and is it advisable to wait some 15 minutes before attempting to ignite the flame.

5.3 Oven temperature, temperature programme

As already mentioned, the maximum thermostat temperature is limited by the thermal resistance of the column; always examine the manufacturer's data to learn the range of working temperatures and never exceed the recommended maximum to prevent column destruction.

Upon decomposition of the packing the chromatographic characteristics of the column change and the column becomes useless!!!

Decomposition of the packing contaminates the detector and reduces its sensitivity; the remedy might sometimes prove difficult.

Although the column may be heated to a higher operating temperature, it is questionable whether the seals will withstand it. The Teflon and PEEK seals lose appropriate mechanical properties and must be replaced by graphite seals.

Chromatographic separation of compounds is based on the fact that the rate with which individual components of the separated mixture pass through the column and, accordingly, their retention times depend on the column temperature; since individual component differ in that respect, one must identify a suitable temperature programme ensuring that the peaks of all components are separated adequately.

The rate of elution generally increases with increasing column temperature. At sufficiently low temperatures all components may be completely retained and remain in the stationary phase; on the other hand, above a certain temperature there is no retention and the retention times of all components are equal to the dead time. Since separation at any given temperature may prove to be inadequate and/or the analysis can last too long, chromatographic analyses are often performed with a variable, pre-programmed thermostat temperature; the problem then is to find a suitable temperature programme.

In searching for the optimum column temperature one should keep in mind that temperature affects the carrier gas viscosity as well and, accordingly, also its flowrate.

Contrary to liquids the viscosity of gases increases with increasing temperature and, in an ideal gas (both helium and nitrogen behave almost like the ideal gas), is directly proportional to $T^{1/2}$ (where T is temperature in K). This should be taken into account together with the fact that with variable operating temperature the split ratio and thus also the sensitivity can change. Temperature may also affect column efficiency and the carrier gas flowrate may cease to be optimal.

5.4 Carrier gas pressure

The flowrate of carrier gas is directly proportional to its pressure drop and decides on the time of analysis - the time can be reduced by increasing the carrier gas pressure. The carrier gas flowrate is an important parameter decisive for chromatographic separation since it affects the column efficiency characterised by the plate number N:

$$N = 5.545/t_R/w_{1/2})^2 .$$

Another parameter often used to characterise column efficiency is the height equivalent of a theoretical plate; theory of chromatographic separation states that the latter depends on the mobile phase flowrate according to the so-called van Deempter isotherm exhibiting two asymptotes and a minimum, where the optimum separation efficiency is reached; the position of the minimum defines the optimum carrier gas pressure across a given column; separation efficiency deteriorates when the pressure is increased.

5.5 Overall flowrate of carrier gas through the detector

All gas chromatographs of series CHROM G10 are equipped with an electronic valve to set up the overall flow rate of carrier gas (make up gas) through the detector. The dependence of peak area on the overall flow rate of carrier gas through the FID detector demonstrates that the flow rate should be some 25 ml/min, where the detector sensitivity is the highest. Since for capillary columns the carrier gas flow rate lies usually between 0.25 and 5 ml/min, it is advisable to supply additional carrier gas to the detector (with packed columns this is usually not the case).

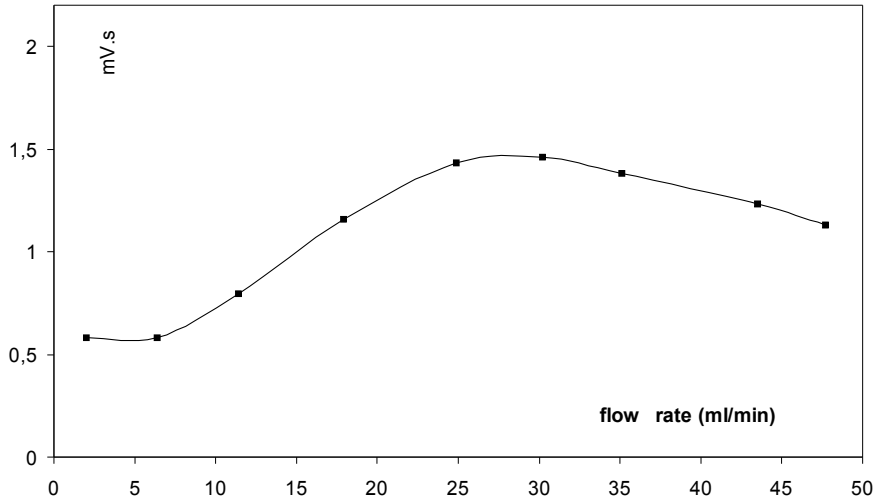

Fig. 12 :Dependence of peak area on carrier gas flow rate.

5.6 Flowrate of hydrogen through the detector

Figure 10 shows detector sensitivity as a function of hydrogen flowrate. The flowrate should lie somewhere between 25 and 40 ml/min where the curve exhibits a broad maximum.

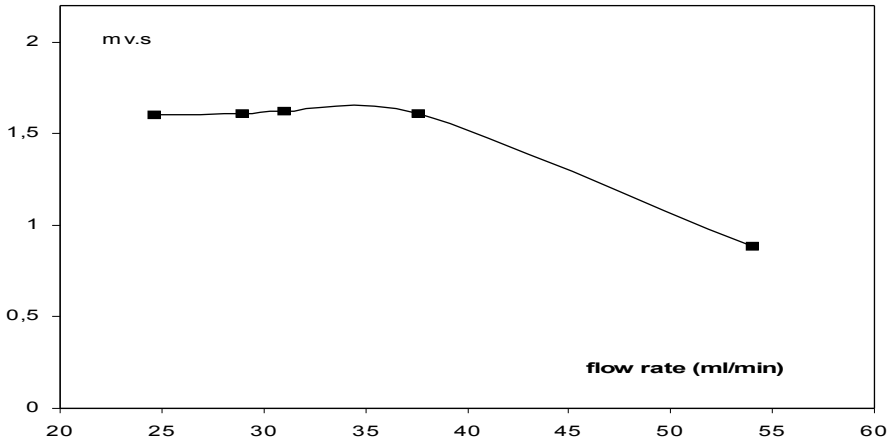

Fig. 13 : Detector sensitivity as a function of hydrogen flow rate.

5.7 Flow rate of air

The dependence of sensitivity on air flowrate is only moderate and shows a broad plateau between 110 and 190 ml/min. Ignition of the hydrogen flame however requires a presence of an explosive mixture in the detector; it is therefore important to use a correct ratio of the hydrogen and air flowrates. Without make-up carrier gas the hydrogen flowrate should be 35 ml/min and that of air 120 ml/min; at any rate the appropriate ratio for flame ignition must be determined by trial and error since it depends on the flowrate of carrier gas through the detector and therefore also on the chromatographic column employed.

6. Removal of Minor Defects

This Chapter offers advice to all beginners who might encounter difficulties when operating a gas chromatograph - a relatively complex instrument. The reasons underlying the defects described below can be identified according to the description of the defective behaviour, always followed by a hint how to remove it without calling a serviceman.

1) The detector does not react to injection, the recorder or integrator register a flat baseline

The defect might be caused by several reasons. What is the output voltage? Is it within the preset integrator range (e.g. for example 1250 mV)? Is it possible to bring it into the above range by autozero? If yes, possible reasons include the following:

a) Flame not burning in the detector. The hydrogen/air mixture exhibits a lower and upper critical composition; outside these limits the flame either cannot be ignited at all or a miniexplosion takes place but the flame is immediately extinguished.

When the flame burns water droplets condense on a cold glass object held close to the detector outlet. Ignition and extinguishment of the flame affect the baseline level.

Remedy:

Check whether the outlet valves of the hydrogen and air pressure cylinders are open!

Check the hydrogen and air pressure on cylinders! Are they within the prescribed limits? They might have changed and caused the defect. Keep in mind that the system reacts to the setting of a values with a certain time lag - always wait about 10 minutes before try to ignite the flame again. If you subsequently succeed in igniting the flame, reset the original value - the flame mostly persists.

If the flame is burning, as indicated by water droplets formed on a glass object held close to the detector outlet, then the underlying reason can be any of the following:

b) No carrier gas passes the column and there is thus no elution.

Open the carrier gas make-up flow and check whether the outlet valve on the pressure cylinder is open. Check flow on the detector output. Another possible but rather improbable reason is a clogged inlet capillary or a leakage in the system.Close pressure cylinder and check the pressure indicated by the manometer then drops rapidly. The leak is usually at the connection of the inlet capillary or at the connecting point of the two parts of the injector block. Check all connections by a dilute detergent solution. Is the column OK? Another possible reason is the following:

c) Broken or clogged column. Close all auxiliary gases wait about 10 min. and measure the flowrate at the detector outlet. If the measured flow rate is zero, the column is either broken or clogged.

Remedy:

Remove and check the column output due an immersion into destilled water. If it appears to be OK and bubbles are going out, a capillary column is not clogged. Since clogging usually occurs at one end, examine the ends by a magnifying glass - the defect is often immediately apparent. If a column is clogged at either end, cut off a small segment, otherwise the column must be discarded and replaced.

2) At the range 1000 mV set the detector output corresponds to the maximum value possible

There are two possible reasons:

d) Short-circuited FID detector:

The culprit is almost always water condensed on the PTFE part separating the measuring electrode from the detector body. The defect persists also with extinguished hydrogen flame.

Remedy:

Extinguish the flame by closing hydrogen and air inflows. Remove the metal detector cap and swab water beneath, if any.

Remove the cap nut holding the detector in place and remove the upper detector part by lifting the electronics block. Examine and if need be dry the electrodes. Dry the detector, preferably by a stream of hot air. Fit the upper part onto the bottom part - the electronics block must snap in. Fix the upper part by the cap nut.

In fitting the electrode block back in place take care of the connecting cables. A Teflon ring seal must be beneath the metal block fitted onto the bottom detector part.

Set the detector temperature to 300 °C and wait about 30 to 60 minutes. If the condensed water has been completely removed, proper detector function will resume.

e) Too high level of impurities in ECD

Remedy:

Check all gases in use if quality is good enough for ECD. Let the sysstem to be purged overnight. Switch for a while to DC mode.

e) Compounds eluted from the column raise the signal level. The column has been either not conditioned properly or the column packing decomposes. In this instance the baseline level drops down upon changing the attenuation from 1 to 1000 as well as upon extinguishing the flame.

Remedy:

Lower the temperature. Decrease sensitivity and condition the column if new; replace it if the operating temperature has been exceeded and then condition the system. If the defect persist, bridge over both the injector block and the detector by short stainless-steel capillaries and condition the whole system at the maximum temperature allowed in view of the seals employed: 300 °C the injector block, 300 °C the thermostat, 350 °C the detector. The conditioning lasts 24 hours. The residual current must decrease gradually during the conditioning operation; if it remains high, the upper part of the detector and the nozzle must be removed and, if need be, the contaminating layer responsible for the high residual current cleaned.

3) Signal is almost zero, chromatogram peaks are minute or non-existent

There is a leak in the system, usually at the cap nuts fixing the column to the thermostat cover (these can get loose when exposed to increased temperature), but in fact anywhere from the carrier gas inlet to the injector block.

Remedy:

Prior to looking for a leak, check whether the outlet from the splitter is set correctly. Set the time of valve opening to 180 s and measure the flowrate at the splitter capillary outlet. It must be zero for a packed column and usually between 10 and 100 ml/min for a capillary column.

If the splitter is set correctly, extinguish the flame, allow the injector block, the detector and the thermostat to cool down and check for a leak:

1) At the carrier gas inlet.
2) At the outlet of the septum flush outlet.
3) At the connection between the upper and bottom part of the injector block.

4) At the splitter outlet capillary.
5) At the inlet and outlet capillary of the splitter electromagnetic valve.
6) At the inlet and outlet capillary of the splitter needle valve.
7) At the cap nuts at the inlet to the injector block.
8) At the cap nut at the detector inlet.

Tighten the leaking cap nut or replace a defective seal.

4) Capillary column efficiency is poor, peaks are large and broad, the solvent peak exhibits major tailing and prevents the analysed compounds to be evaluated properly:

Injection is not split. There are two possible reasons:

a) You have failed either to set the time of valve opening or to press ENTER.

Remedy:

Select item **Splitter time** in the menu of operating parameters. The valve opening time should be at least 30 s. The default value is 0 s.

b) The capillary column is incorrectly installed and its end protrudes to and clogs the constricted part of the liner.

Remedy:

Allow the thermostat, the injector block and the detector to cool down.

Disassemble the injector block and remove the liner. Swing back the thermostat cover, unscrew the larger cup nut, remove the column along with the connecting piece and the protective cap and put the liner on the column. The end of the capillary should be about 1 mm below the constriction. If necessary, move the column and fix by tightening the small cup nut, check the position of the column end, and reconnect.

c) Wrong injection method is used – in any case press first START and then inject the sample

5) High noise and high baseline level in analyses with a capillary column

The capillary column is improperly connected to the detector, its end protrudes from the brass connecting piece and reaches the nozzle.

Remedy: Extinguish the flame, allow the thermostat, the injector block and the detector to cool down; remove the detector by unscrewing the brass connecting piece, check and if necessary adjust the position of the column end.

6) The baseline drifts during an analysis with a temperature programme.

The reason is improperly conditioned column that releases organic compounds when heated. Another possible reasons involve decomposition of a seal, or organic compounds present in carrier gas whose flowrate depends on temperature.

Remedy: Condition the system properly. Replace the defective seal - use a graphite seal instead of PEEK seal when working close to 270 °C. Use high-purity nitrogen in all chromatographic analyses.

7. Servicing organisation

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