

GC TROUBLESHOOTING

USER MANUAL

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Table of context

1 Introduction 4
2 GC diagnostyc by chromatogram image 5
2.1 No peaks on the chromatogram 5
2.2 Peaks heights reduction 6
2.3 Bursts 8
2.4 Negative peaks, negative bursts 8
2.5 False peaks 9
2.6 Downward drift 10
2.7 Monotonous upward drift 11
2.8 Rapid upward drift 11
2.9 "Plateau" 12
2.10 Shift of baseline 13
2.11 "Tailing" peaks 13
2.12 "Fronting" peaks 14
2.13 Width increasing 15
2.14 Irregular shape of the top 16
2.15 Unwanted humps 16
2.16 The peaks have shifted to the right 17
2.17 The peaks have shifted to the left 18
2.18 Split peaks 19
2.19 Poor resolution 20
2.20 High noise, waves, high background 21

1 INTRODUCTION

The chromatograph has a high level of self-diagnosis. Many errors that occur during operation are identified and indicated to the operator.

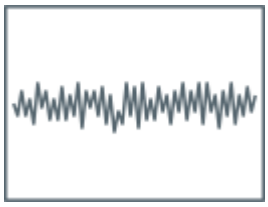
However, when performing measurements, the user may encounter analytical errors that are not detected by the instrument. Typical examples are loss of sensitivity, deterioration in separation of components, smearing of peaks, poor convergence of results.

When evaluating the analysis requirements, it is necessary to consider not a gas chromatograph separately, but the entire gas chromatographic complex, as an integral system, including a chromatograph with all devices installed in it, gases and filters, devices for sampling, transportation and storage of samples, sample injection devices, software.

Analysis history is important in evaluating emerging problems and correcting them. Sometimes the problem cannot be identified from the latest chromatograms, but it is immediately diagnosed if you look at chromatograms, for example, a month ago.

2 GC DIAGNOSTYC BY CHROMATOGRAM IMAGE

2.1 No peaks on the chromatogram



The syringe is blocked with septa particles

Clean the needle or use another syringe. The needle can be cleaned by hydraulic shock:

- Remove the plunger.
- Pour the solvent used for sample preparation into the syringe channel from above.
- Insert the plunger up to the stop. The plunger should be moved quite rapidly but carefully, because there is a risk of its bending.

Capillary column is broken

If the column is broken in the beginning or in the end, then cut off its short part. If the break is in the middle part, you can use a glass or quartz connector. In case it is not available, replace the column.

Losses at working with sample

Check the storage, preparation and transportation of the sample.

No flame in FID, NPD or FPD

The problem may occur when there is no flame, but chromatograph recognizes the presence of flame. Cases of false flame are described in the lessons devoted to flame detectors.

The column is selected incorrectly

Perhaps you just did not wait for the peaks to come out. Increase the temperature in the column oven (in case its upper limit will not be exceeded).

Too low temperature of the column oven and insufficient analysis time

This problem is related with the previous one. You did not wait for the peaks to come out. Change the temperature mode.

Dirty detector or dirty gas

It is typical for ECD. The chromatogram in this case is a straight line on the level of 10000 mV. Make sure that the carrier gas is pure and perform the detector maintenance.



Leakage in the inlet

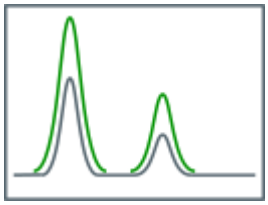
It is unlikely reason. The peaks will be absent if their heights were at the noise level during operation with a properly working inlet.

No carrier gas flow through the column

If carrier gas flow is not set, chromatograph will not reach the "Ready" stage. However, if there are two columns and the flow is set and goes only through one of them, the chromatograph will not recognize this situation as a failure.

When using capillary column in on-column mode it may become blocked with a phase or a piece of septa. In this case chromatograph reaches the "Ready" stage as the pressure is normal but there is a poor flow or not flow through the column. To solve this problem, cut off the beginning of the column for the length of syringe immersion.

2.2 Peaks heights reduction



Detector sensitivity deterioration

Estimate peaks heights against the reference standard comparing chromatograms which was recorded today and earlier with the same standard. Perform the detector maintenance.

Poor tightness of sample injection port

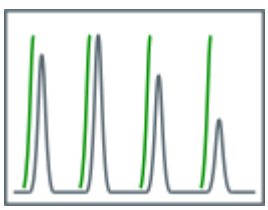
When working with packed column it occurs along with retention time increasing. Fix the poor tightness (replace the inlet septa). The seals of column and tubes on the sample injection line can be the source of poor tightness.

The mode of sample splitting in the inlet has changed

It is unlikely reason. It will happen only if operator works with many methods with different modes and they were mixed up.

Fractionation

When the samples containing heavy components are injected into the inlet, it is advisable to use on-column injection; in the split mode fractionation of components occurs.



Irreversible sorption of the sample in the liner and/or column

For example, it is typical for analysis of sulfur containing compounds on FPD – the pieces of septa which have entered the liner sorb the sample. Clean the liner, replace the filling regularly. Replace the septa.

Saturation of filters on the air line when working with FPD

Perform the regeneration of filters.

Leakage in the syringe

It is typical for gas-tight syringes at deterioration and reducing of sealing features of plunger. Replace the syringe.

Syringe needle contamination

It leads to the sampled volume reduction or boiling of the sample in the syringe. Clean the needle or use another syringe. The needle can be cleaned by hydraulic shock:

- Remove the plunger.
- Pour the solvent used for sample preparation into the syringe channel from above.
- Insert the plunger up to the stop. The plunger should be moved quite rapidly but carefully, because there is a risk of its bending.

Strong contamination of the column with changing of its characteristics

Cut off 0,5-1 meter of column. If the column contents fixed and bonded phases it can be washed with the solvent.

Changing of sample concentration

It can be caused by vaporization or degradation of sample or dependence of sample on temperature or pH.

Passage of the sample

Large carrier gas flow and less volume of solvent can help in this situation. Reduce the volume of sample.

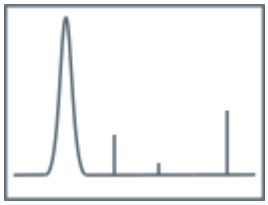
Destruction of the sample on the liner

Use only deactivated liners or liners with deactivated filling.

Losses of the sample

Check the storage, preparation and transportation of the sample.

2.3 Bursts

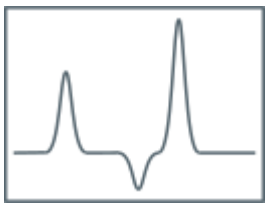


If the bursts are only upwards and dust cannot be blown by ordinary flow, the metal tubes should be tapped.

Poor contact of amplifier connector and FID, NPD, ECD electrode

If there are dips, check the quality of contact.

2.4 Negative peaks, negative bursts



The analyzed component presents in the carrier gas and its concentration in the sample is lower than in carrier gas

Use the gas of higher purity. Use filters removing this component from the carrier gas.

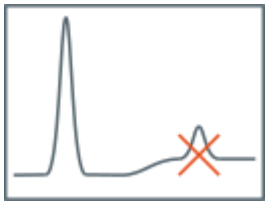
Negative bursts are the result of valves switching

It is necessary to reduce the "dead" zones in the flow diagram, but it is not always possible due to the design features. Use integration events to reduce the negative impact of negative peaks and bursts when integrating a chromatogram.

When working with FPD, hydrocarbon peaks can be negative

It is most often useless to battle this problem. Use integration events to reduce the negative impact of negative peaks and bursts when integrating a chromatogram.

2.5 False peaks



Incorrect storage of components of the sample

It can cause the reaction between the sample components and container structural elements. Use only inert containers.

Insufficient quality of solvent

If possible, use the solvent of more purity.

The sample decomposes

Check the temperature program. If the analytes are temperature-sensitive, reduce the inlet temperature. Replace the liner.

Poor quality derivatization

Check the derivatization procedure.

Poor quality of carrier gas

Contaminants entering with the carrier gas (or entering the carrier gas flow in the chromatograph: from the inlet liner, liner sealing ring) can focus on the initial section of the column in the lower isotherm. The mentioned components elute as false peaks at temperature programming. Naturally for such a case, the longer the GC time between analyses is (in the first isotherm), the higher the amplitude of false peaks will be.

To eliminate these contaminants, it is necessary to regenerate or replace the carrier gas filters, replace or clean the inlet liner.

Dirty syringe

Use another syringe or wash it better.

Dirt from the inlet septa

Use high-temperature low bleeding septa, e.g. HTsepta #041883 SGE.

Semi-volatile impurities

If peak width of your sample is more than the peak width of the sample with similar retention, perform column conditioning.

Sample contamination before injection into chromatograph

Check the storage, sampling and injection conditions.

Dirt from the sampler

If you use the sampler, disconnect it and make sure that chromatograph is not a source of false peaks. Then check the sampler.

Cases of sample ingress from the vent channel

When working with pressure programming (including programming of temperature and constant flow rate through the capillary column) or in the splitless mode, the flow in the sample vent line slows down or stops for a while. It can lead to the ingress of sample components from the vent line to the inlet. Then the components can focus in the initial part of the column and come out as false peaks.

The retention times of these components will be close or identical to the corresponding times of target components.

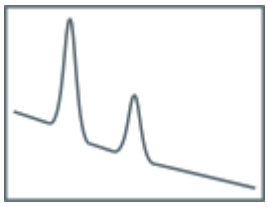
Possible solutions:

- Regenerate the sample vent filter.
- When working in a splitless mode use valve before the sample vent filter and electronic septum purge controller.
- Optimize the analysis mode.

Column contamination

False peaks due to the column contamination are the least possible reason among all abovementioned. That's why column conditioning should be the final step in false peaks elimination.

2.6 Downward drift



The gas flow varies along with the temperature gradient

Check the content of gas in the cylinder. The actual pressure should exceed the required one at maximum temperature. Otherwise, replace the gas cylinder.

Create a method with constant flow through the column.

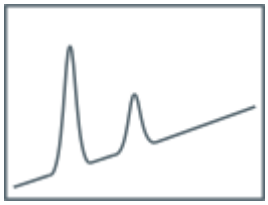
Poor quality of gas (at constant input pressure)

Check the quality of gas.

Gas lines have not purged from the residual air

Purge the lines.

2.7 Monotonous upward drift



Dirty detector

Perform detector maintenance.

Dirt in the column

Run a blank analysis with plugged detector fitting at ambient temperature of the column oven. If the baseline became better the problem was identified correctly. Do the following (perform each subsequent action in case the previous one did not help):

- Perform column conditioning.
- Cut off two turns from the column input.
- Wash the column with solvent (only chemically bound phases).
- Use pre-column (flow diagram of chromatograph should be modified).
- Replace the column.

The new column is not conditioned

Perform column conditioning.

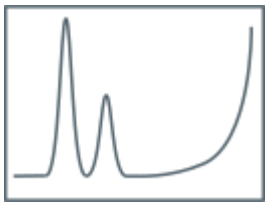
Dirt from septa or liner of the inlet

Run a blank analysis at room temperature of the inlet. If zero line became better, replace/wash the liner and install more high-temperature septa.

Poor quality of carrier gas

Use the gas which meets the requirements given in the operating documents for chromatograph.

2.8 Rapid upward drift



Decomposition of liquid phase of the column

Check the analyzed matrix for compatibility with the column.

Dirty detector

Perform detector maintenance.

Oxygen ingress

Column conditioning can help. If it did not help, replace the column.

To prevent this problem, perform leak tests and replace inlet septa regularly. Use pure gases and oxygen traps.

Dirt in the column, chemical damage of phase

Run a blank analysis with plugged detector fitting at ambient temperature of the column oven. If the baseline became better the problem was identified correctly. Do the following (perform each subsequent action in case the previous one did not help):

- Perform conditioning of column disconnected from detector (when working with FPD do not disconnect it from the detector).
- When working with capillary column, cut off two turns from the column input.
- Wash the column with solvent (only chemically bound phases).
- Use pre-column (flow diagram of chromatograph should be modified).
- Replace the column.

The new column is not conditioned

Perform column conditioning.

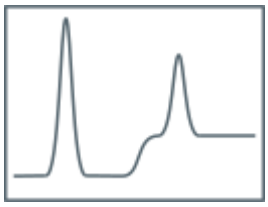
Column overheating

Check whether the temperature in analysis or conditioning do not exceed the maximum allowable one according to the column passport.

Poor quality of carrier gas

Use the gas which meets the requirements given in the operating documents for chromatograph.

2.9 "Plateau"



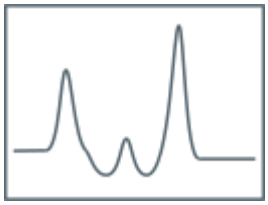
Too high speeds of heating in the temperature program of column

Reduce the speeds of heating.

Variation of carrier gas flow during analysis

It is the result of valves switching in the pneumatic circuit. MSD, TCD, ECD are sensitive to carrier gas flow reversion. This problem can be solved only by working parameters and flow diagram design adjustment. This effect often cannot be removed completely.

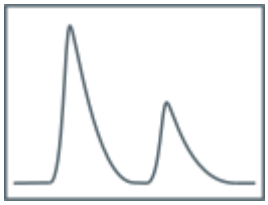
2.10 Shift of baseline



Column is not conditioned

Perform column conditioning.

2.11 "Tailing" peaks



The volume of vapors exceeds the capacity of inlet

Reduce the volume of sample.

Column overloading

Increase sample splitting in the injection port, reduce the volume of sample, use dilution.

The sample evaporates too slowly and irregularly and also condensates

Increase the inlet temperature.

Incorrect installation of column, uneven cut, leak in the system

Read the User manual. Cut and reinstall the column again. Check the leak tightness. The least held peaks tail most often.

Either liner or septa is contaminated

It occurs for chemically-active components. Replace the septa. Wash or replace the liner.

Non-conformance of sample and solvent

Change the solvent. Use pre-column.

Bad injection technique

It is often caused by irregular motion of syringe plunger at sample injection or its sampling into the syringe. Use autosampler.

Cold areas

Peaks with bigger retention time tail more. Condensation occurs somewhere in the sample line. Check the heat-up of sample line.

"Dead" volumes in the system

Peaks with bigger retention time tail more. Check the collections in the sample line to eliminate non-purged volumes.

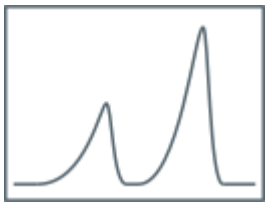
The features of analyzed component

Diffused rare front of the peak is typical for polar substances eluted on non-polar column with thin film. When working with packed columns the reason is not high enough quality of solid support. In this situation not every peak will have a tail.

Broadening in the external injection devices

Broadening due to incorrect operation of injection devices (headspace sampler, thermal desorber). For example, when working with one-stage thermal desorber with combined tubes for light components, desorption from the different layers of tube is performed with the different speed, that's why the components have diffused front.

2.12 "Fronting" peaks



Column overloading

Increase sample splitting in the injection port, reduce the volume of sample, use dilution.

Bad injection technique

It is often caused by irregular motion of syringe plunger at sample injection or its sampling into the syringe. Use autosampler.

Non-conformance of sample and solvent

Change the solvent. Use pre-column.

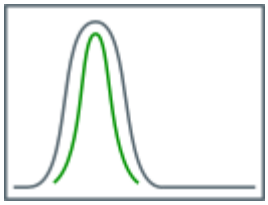
Superposition of peaks of solvent and sample components

Use the solvent with greater difference in polarity or boiling temperature.

The column is installed incorrectly

Read the User manual. Reinstall the column.

2.13 Width increasing



Large volume of sample

Excessive increasing of volume of injected sample leads to widening of chromatographic zones and distortion of peaks shape. It is normally typical for the initial section of chromatogram.

The concentration of the component increased

Width increases along with increasing of concentration, please note this during creating a method.

Strong contamination of column with changing of its characteristics

Cut off 0,5-1 meter of column. If the column contents fixed and bonded phases it can be washed with the solvent.

Liner contamination

Clean the liner or replace it.

Negative influence of the solvent

Reduce the column temperature. Change the solvent for the better conforming of polarity/sample/phase.

No focusing

Use pre-column.

The following reasons mean that the operator changed the mode of analysis himself, for example loaded another method without checking its parameters:

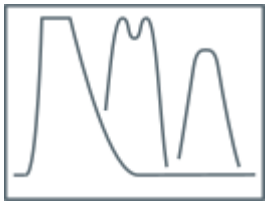
The flow rate of carrier gas has changed

This reason can be supposed in case of shifting of all peaks in one direction by approximately equal distance. Check the mode of analysis.

The mode of inlet has changed

Check the pressure, division, and inlet temperature.

2.14 Irregular shape of the top



Unretained component

It occurs for the substances that almost do not interact with the phase and are released immediately after the dead time on the short and wide capillary columns, as well as on all packed columns. There is often no way to solve this. Sometimes reducing temperature in the column can help (at initial temperature programming).

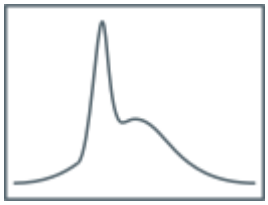
Off-scale detector readings

If the top is absolutely flat, it means that the signal of detector exceeds its working range. Increase the splitting in the injection port, reduce the volume of sample.

Detector overloading

If the top is rounded, not sharp or M-shape. One of the typical examples is analysis of sulfur-containing compounds on FPD. Increase the splitting of the sample in the injection port, reduce the volume of the sample.

2.15 Unwanted humps

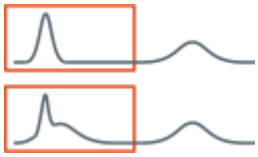


Moisture in the column

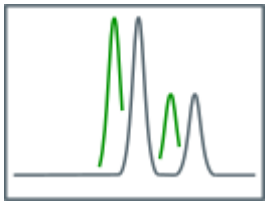
Run 1-2 blank analysis before starting work.

Superposition of components from the previous analysis

If there are heavy components in the sample and you have not wait enough for them to be released, they appear in the next analyses in the form of very wide peaks, so called “humps”. Always wait enough for releasing of all components of the sample before starting the next analysis. Increase the temperature of the last step of column programming. Use pre-column.



2.16 The peaks have shifted to the right



Poor tightness of injection port

Fix the poor tightness (replace the inlet septa). The seals of column and tubes on the sample injection line can be the source of poor tightness.

Low carrier gas pressure

This reason can be supposed if not all peaks have shifted to the right, but only ones close to the end of analysis; gas viscosity and column restriction increases at temperature programming, carrier gas flow rate through the packed column reduces. Increase carrier gas pressure on the cylinder.

Replacing the column or changing its dimension

After these procedures the method should be modified. Columns with the same parameters are still not absolutely identical.

Changing of the component's concentration

The tops of the peaks shift to the right at reducing of the concentration. It is especially obvious at significant variations of concentration and is usually accompanied with changing the peak shape. The shape of the peaks of high components concentration can differ from Gaussian.

The following reasons mean that the operator changed the mode of analysis himself, for example loaded another method without checking its parameters:

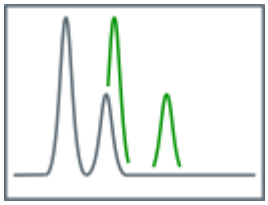
The flow rate of carrier gas has changed

This reason can be supposed in case of shifting of all peaks in one direction by approximately equal distance. Check the mode of analysis.

The column temperature has changed

This reason can be supposed in case of peaks shifting on the equal distance close to the end of chromatogram. Check the mode of analysis.

2.17 The peaks have shifted to the left



Reducing of column efficiency

Gradual changing of features of stationary phase due to its volatility (natural and inevitable process). In this case shifting of peaks occurs slowly and gradually during months and even years. It cannot be eliminated.

Carrier gas quality (especially presence of oxygen) can reduce the lifetime of the column. As was described above, the speed of deterioration of separation features and changing of times depends on the degree of impact (concentration of oxygen and heating to the high temperatures accelerate the processes of phase oxidation).

The columns which operate at the temperature close to the maximum have shorter lifetime and their phase is blown away more quickly at high temperature.

Possible solutions:

- Column replacement.
- Injection of preliminary purified samples during analysis and careful execution of recommendations on column usage are advised to extend the lifetime of the column.
- Check the quality of carrier gas. Replace or regenerate the filter removing oxygen.
- Do not heat the column to the temperatures close to the maximum unless necessary.
- If there are several columns in the column oven, do not forget to remove low-temperature columns during conditioning of high-temperature ones.

Many columns in gas-solid chromatography are rather water-sensitive. These columns change their sorption characteristics significantly at ingress of even small amount of moisture or other substances which fill the pores. Packed columns with molecular sieves and all capillary PLOT columns are among such columns.

The retention times of components for this moist column shift downwards. The retention times increase again after column conditioning.

PLOT columns generally have bigger instability of retention times in comparison with WCOT columns (with liquid stationary phase).

Possible solutions:

- Adjust a working mode of chromatograph so that the column is conditioned for a time after each analysis, and then the next analysis begins immediately. Reach the time consistency of all stages during the analysis cycle.
- Use the function "Preprun of the column oven".
- Use pre-column which will remove moisture and heavy components.
- If possible, replace PLOT column by WCOT column with liquid stationary phase.

Replacing the column or changing its dimension

After these procedures the method should be modified. Columns with the same parameters are still not absolutely identical.

Changing of the component's concentration

The tops of the peaks shift to the left at increasing of the concentration. This also can influence the neighboring peaks. Note the possibility of injection of the sample with such concentration when you create a method. The concentration can be reduced by increasing splitting in the injection port, reducing the volume of sample or using sample dilution.

The following reasons mean that the operator changed the mode of analysis himself, for example loaded another method without checking its parameters:

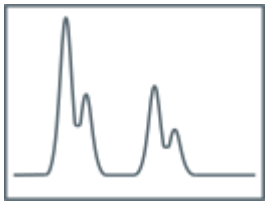
The flow rate of carrier gas has changed

This reason can be supposed in case of shifting of all peaks in one direction by approximately equal distance. Check the mode of analysis.

The column temperature has changed

This reason can be supposed in case of shifting of all peaks closer to the end of chromatogram by approximately equal distance. Check the mode of analysis.

2.18 Split peaks



The sample decomposes

Check the temperature program. If the analytes are temperature-sensitive, reduce the inlet temperature. Replace the liner.

Non-conformance of solvent and column

Change the solvent or use pre-column.

Detector overloading

Reduce the volume of the sample or increase the splitting of the sample in the injection port.

Sample degradation

Only some peaks split. Peaks widening may occur at reducing of the temperature. Reduce the inlet temperature. Use derivatization for obtaining thermostable derivatives. Use on-column injection (special inlet is required).

Mixtures of solvents with big difference in boiling temperature and polarity

Use only one solvent.

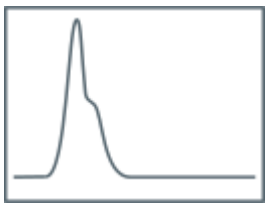
Incorrect installation of column, uneven cut, leak in the system

Read the User manual. Cut and reinstall the column again. Check the leak tightness.

Bad injection technique

It is often caused by irregular motion of syringe plunger at sample injection or its sampling into the syringe. Use autosampler.

2.19 Poor resolution



Reducing of column efficiency

It occurs due to phase aging (natural and inevitable process). In this case shifting of peaks occurs slowly and gradually. It cannot be eliminated.

One more reason is phase destruction as a result of improper operation of the column. For example, column overheating could take place during conditioning. Do not exceed the maximum permissible temperature of the column. If there are several columns in the column oven, do not forget to remove low-temperature columns during conditioning of high-temperature ones.

Strong contamination of column with changing of its characteristics

Cut off 0,5-1 meter of column. If the column contents fixed and bonded phases it can be washed with the solvent.

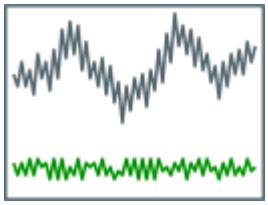
Co-elution with other components

Reduce the column temperature, check whether the "tail" of the peak appears.

Replacing the column or changing its dimension

After these procedures the method should be modified. Columns with the same parameters are still not absolutely identical.

2.20 High noise, waves, high background



Unstabilized detector

For some types of detectors (e.g. ECD) background stabilization can take several hours.

Dirty detector

Perform detector maintenance.

Dirt in the column

Disconnect the column from the detector and install the plug on the input fitting of the detector. Run a blank analysis. If the baseline became better the problem was identified correctly. Do the following (perform each subsequent action in case the previous one did not help):

- Perform column conditioning.
- If you use capillary column, cut off two turns from the column input.
- Wash the column with solvent (only chemically bound phases).
- Use pre-column (flow diagram of chromatograph should be modified).
- Replace the column.

The new column is not conditioned

Perform column conditioning.

Dirt from septa or liner of the inlet

Run a blank analysis at room temperature of the inlet. If zero line became better, replace/wash the liner and install more high-temperature septa.

Sample destruction

Background signal growth after large peak. Check the cleanliness of the liner. Use a new deactivated liner of replace the filling.

Defective pressure regulator in the air compressor (wave)

The repairing of compressor is required. An additional pressure regulator can be installed in the air line before chromatograph.

Poor quality of carrier gas

Use the gas which meets the requirements given in the operating documents for chromatograph. Perform the regeneration of filters.

Major leak through the septa straight after injection

Background changing occurs after the large peak of solvent. Replace the septa. If possible, use a narrower syringe needle.

The column is installed incorrectly

Read the User manual. Reinstall the column.

The gases in the detector are set incorrectly

Read the User manual. Set the correct gases mode.

Leakage when using MSD, TCD or ECD

Most often locations are places of column connection. Find the leak and fix it.

Deterioration of filament and PID lamp

Replace the part.

Deterioration of septa

Replace the septa. Use the appropriate consumables for high-temperature analyses.