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GC Troubleshooting

How to use this Troubleshooting Help system

This Help system is designed to help you diagnose and resolve problems that may occur with your Gas Chromatograph system.

Because there are a number of possible variations in the configuration of your system, the problem descriptions have been grouped partly by the general type of problem, and partly by the specific hardware components.

The main general problem type groups are **Baseline related problems, Peak related problems,** and **Results related problems.** All the suggested possible causes and required remedies within these general problems areas are applicable whatever your system configuration. However, you will also find that there may be some additional possible causes that are applicable only to one particular system configuration.

For example, the **Baseline related problems** area lists a number of generic possible causes. However, if your system utilises a Nitrogen Phosphorous Detector (NPD), you will find an additional possible cause listed under the NPD Troubleshooting section - Unstable Baseline (NPD).

To make sure that you don't overlook these additional problem descriptions, there is a **See also** list in each of the general problem areas, which lists the problems in the area that relate to specific system components.

The suggested method of use, therefore, is to start with the general problem list for the type of problem you are experiencing. If this doesn't resolve the problem, then try the **See also** list for any additional problem descriptions that apply to your system configuration. Finally, try the troubleshooting sections that refer specifically to the components in your system.

You can also use the **Index** and **Search** tabs on the Navigation panel to search for keywords that describe your problem.

If you encounter a problem that is not described here, or have a problem that is not resolved by the remedy suggested, contact your local service representative who will be able to provide additional advice.

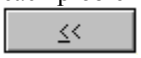
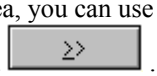
Click here to go to the top level troubleshooting list.

GC Troubleshooting - Top Level

This Help file is designed to help you diagnose and resolve problems that may occur from time-to-time with your GC system. Click here for advice on how to use this help system.

Choose from the following list the area that matches your category of problem. The first three problem areas describe general problem types that are independent of the particular system configuration being used. The remainder of the list takes you to troubleshooting topics for specific system components.

To move through the problem areas, and also to move through the separate problem descriptions within each problem area, you can use the Browse buttons

 and  .

Baseline related problems, such as rising or falling levels of Baseline, drifting, or Baseline noise.

Peak related problems, such as Ghost Peaks, Tailing, or No Peaks at all.

Results related problems, such as varying Retention Times, Poor Sensitivity, or Poor Reproducibility.

Flame Ionization Detector (FID) Troubleshooting

Electron Capture Detector (ECD) Troubleshooting

Nitrogen Phosphorous Detector (NPD) Troubleshooting

Flame Photometric Detector (FPD) Troubleshooting

Photoionization Detector (PID) Troubleshooting

Thermal Conductivity Detector (TCD) Troubleshooting

Mass Spectrometer (MS) Troubleshooting

PTV Injector troubleshooting

Gas Sampling Valve troubleshooting

Within each of these main problem-type areas, specific problem descriptions are listed. When you select the problem description that best matches your problem, you will see a list of the possible causes of the problem, with suggestions as to how each cause can be remedied.

If you encounter a problem that is not described here, or have a problem that is not resolved by the remedy suggested, contact your local service representative who will be able to provide additional advice.

Generic problem descriptions

Baseline related problems

Select the problem description that best matches your problem:

Baseline drifting

Baseline falling

Baseline falling away slowly after a high initial value

Baseline rising

Baseline rising under temperature program control

Baseline - high standing current

Baseline irregular shape: dip after solvent peak

Baseline irregular shape: S-shaped

Baseline irregular shape: square waves

Baseline high frequency noise

Baseline spiking

See also: Baseline related problems with specific system components

Back to top level troubleshooting list.

Peak related problems

Select the problem description that best matches your problem:

Peaks broadening

Peaks clipping at bottom

Peaks clipping at top

Double Peaks

Peak Fronting (excessive slope on left side)

Ghost Peaks

Broad Ghost Peaks

Irregular, Chair-shaped Peaks

Negative Peaks

No Peaks after solvent peak

No Peaks at all

Sample Peak Tailing (excessive slope on right side)

Solvent Peak Tailing (excessive slope on right side)

Unresolved Peaks

Discrete high-intensity contaminant peaks

See also: Peak related problems with specific system components

Back to top level troubleshooting list.

Results related problems

Select the problem description that best matches your problem:

Low reproducibility of peaks area

Poor sensitivity with increased retention time

Poor sensitivity with normal retention time

Retention times decreasing

Retention times increasing

Low reproducibility of retention times

Retention times are inconsistent

See also: Results related problems with specific system components

Back to top level troubleshooting list.

System-component related problems

Flame Ionization Detector (FID) Troubleshooting

To ensure optimum performance of the FID, you must keep it clean and free of dust and deposits. Symptoms such as reduced sensitivity and increased noise indicate that detector needs cleaning.

To properly maintain the FID, you should perform the following cleaning or replacement sequences:

- cleaning the jet
- replacing the jet
- cleaning the collecting electrode
- replacing the collecting electrode
- replacing the ignition assembly

These maintenance activities are described in Chapter 9 of the Maintenance and troubleshooting Manual.

Select the problem description that best matches your problem:

FID Sensitivity problems

FID Flame Ignition problems

FID Contamination problems

Back to top level troubleshooting list.

Electron Capture Detector (ECD) Troubleshooting

Select the problem description that best matches your problem:

ECD Contamination problems

ECD Sensitivity problems

High base frequency

Negative dips after peaks

Baseline drift with changing pulse voltage

Back to top level troubleshooting list.

Nitrogen Phosphorous Detector (NPD) Troubleshooting

To ensure optimum performance of the NPD, you must keep it clean and free of dust and deposits. Symptoms such as reduced sensitivity and increased noise indicate that detector cleaning or thermionic source replacement could be necessary.

To properly maintain the Nitrogen-Phosphorus Detector, you should perform the following sequences:

- cleaning the jet.
- replacing the jet.
- cleaning the collecting electrode.
- replacing the collecting electrode.
- replacing the thermionic source.

These maintenance activities are described in Chapter 11 of the Maintenance and Troubleshooting Manual.

Select the problem description that best matches your problem:

NPD Sensitivity problems

No NPD detector response

NPD detector response lower than expected

High background level (NPD)

NPD shows FID-like response for solvent and other carbon based compounds

Solvent quenching effect (NPD)

Unstable baseline (NPD)

Low carbon rejection (NPD)

Back to top level troubleshooting list.

Flame Photometric Detector (FPD) Troubleshooting

To ensure optimum performance of the FPD, you must keep it clean and free of dust and deposits. Symptoms such as reduced sensitivity and increased noise indicate that detector needs cleaning.

To properly maintain the FPD, you should perform the following cleaning or replacement sequences:

- cleaning the jet
- cleaning the interferential filter
- cleaning the mirror metal plug

- cleaning the flame-side heat shield
- replacing the jet
- replacing the interferential filter
- replacing the heat shields

These maintenance activities are described in Chapter 12 of the Maintenance and Troubleshooting Manual.

Select the problem description that best matches your problem:

FPD Sensitivity problems

High standing current and noise (FPD)

No standing current (FPD)

Unstable and excessively noisy baseline (FPD)

FPD temperature does not reach the set point

Low sensitivity (FPD)

Low sensitivity and water droplets generate between the heat shields (FPD)

Back to top level troubleshooting list.

Photoionization Detector (PID) Troubleshooting

The Photoionization Detector requires relatively little maintenance. To properly maintaining the detector, you should perform the following sequences:

- UV lamp replacement
- Cleaning the UV lamp window

These maintenance activities are described in Chapter 13 of the Maintenance and Troubleshooting Manual.

The UV Lamp must be replaced when exhausted, faulty or when a different class of compounds is to be analyzed. Click here for the available UV lamps and their application field.

Note: Keep always a spare UV lamp in your laboratory because the lamp could collapse suddenly, without warning signs.

CAUTION: Make sure to have appropriate filters installed on the carrier and make-up gas lines, and replace them regularly.

Select the problem description that best matches your problem:

PID Sensitivity problems

PID Contamination problems

UV lamp does not light immediately (PID)

UV lamp does not light at all (PID)

No standing current (PID)

Tailing of solvent peak (PID)

Tailing of sample peaks (PID)

Low sensitivity (PID)

Back to top level troubleshooting list.

Thermal Conductivity Detector (TCD) Troubleshooting

The TCD detector does not usually need current maintenance. Nevertheless, if you follow a few simple sequences, you will avoid troubles and prolong the detector's lifetime. Pay special attention to avoid contamination or damage of the filaments.

Follow these simple rules:

- Avoid activating/deactivating the bridge when not necessary. This operation would considerably reduce the filaments' lifetime.
- Avoid injecting samples that contain halogenated or acid compounds at high concentrations.
- Ensure that oxygen (air) could not enter into the filaments cells. Oxidation would irreversibly damage the filaments. Install traps for moisture and oxygen on the gas lines to reduce the hazard.

WARNING! Set Filament power to **Off** before disconnecting the column from the detector. When the column is disconnected, air will enter into the cell and the filament, if powered, will burn.

For the same reason set Filament power to **On** only if the column has been connected. It is a good practice to let the reference and make-up gases flow through the cells for 10-15 minutes before powering the filaments.

Select the problem description that best matches your problem:

TCD Sensitivity problems

Negative Peaks (TCD)

Baseline Fluctuation (TCD)

Baseline Drift (TCD)

Low Sensitivity (TCD)

TCD does not work

Back to top level troubleshooting list.

Mass Spectrometer (MS) Troubleshooting

This section covers only chromatography-related problems that may be observed when using a GC/MS system, where the cause may be with the MS rather than the GC. Refer to your MS troubleshooting documentation and help for specific help with MS problems.

Select the problem description that best matches your problem:

High Baseline (MS)

Peak Tailing (MS)

Low Sensitivity (MS)

Poor Reproducibility of Results (MS)

Back to top level troubleshooting list.

PTV Injector troubleshooting

The PTV injector will normally be serviced by ThermoFinnigan authorized technical personnel. In order to operate at peak performances, the injector requires periodic maintenance from the user. This maintenance includes:

- the replacement of the septum

- the cleaning or replacement of the liner.

These maintenance activities are described in Chapter 6 of the Maintenance and Troubleshooting Manual.

In addition, you will find information on how to adjust the quartz wool packing inside the liner used in the Large Volume version of the PTV injector (LV PTV).

Select the problem description that best matches your problem:

PTV Sensitivity problems

PTV Discrimination problems

Discrimination of heavy compounds in splitless mode (PTV)

Discrimination of volatile compounds in splitless mode (PTV)

Discrimination of volatile compounds in solvent split mode (PTV)

Discrimination in split mode (PTV)

Excessive broadening of solvent peak (PTV) (also with volatile compounds obscured and lack of sensitivity)

Sample degradation (PTV)

PTV Large Volume - Poor reproducibility of results

PTV Large Volume - Discrimination of volatile compounds

PTV Large Volume - Sample degradation

Back to top level troubleshooting list.

Gas Sampling Valve (GSV) troubleshooting

The Gas Sampling Valve is available in two versions: Manual and Automatic.

Both types will normally be serviced by ThermoFinnigan authorized technical personnel.

The Manual Gas Sampling Valve does not require periodic maintenance.

To operate effectively, the Automatic Gas Sampling Valve requires periodic maintenance from the user. This maintenance includes:

- the replacement of the diaphragm;
- the cleaning or replacement of the removable frit of the filter.

For either version, you could be required to replace the valve loop, if troubleshooting indicates that it needs to be cleaned or replaced.

These maintenance activities are described in Chapter 7 of the Maintenance and Troubleshooting Manual.

Incorrect operation of the gas sampling valves lowers the reproducibility of the injected volumes of sample causing unreproducible results.

Select the problem description that best matches your problem:

Analyses results are not reproducible (GSV)

No signal at all (GSV)

GSV does not work

Back to top level troubleshooting list.

Leak Checking

Checking for Leaks

There are various automatic and manual operations you can use to keep under control the tightness of your chromatographic system. The sequences of operations you can use to find and correct possible leaks in the system are organized according to the instrument configuration. Specifically, this means the type of column installed, and whether the system is equipped with the Digital Pressure Flow Control (DPFC) and Digital Gas Flow Control systems.

Click here for information on leak-checking DPFC-equipped systems.

Click here for information on leak-checking non-DPFC systems.

Select the relevant leak-checking procedure from the list below:

Performing an Automatic Leak Check

Performing an Automatic Column Evaluation

Checking for leaks - Capillary Columns, DPFC, DGFC

Checking for leaks - Capillary Columns, DPFC, Non-DGFC

Checking for leaks - Capillary Columns, Non-DPFC, Non-DGFC

Checking for leaks - Packed Columns, DPFC, DGFC

Checking for leaks - Packed Columns, DPFC, Non-DGFC

Checking for leaks - Packed Columns, Non-DPFC, Non-DGFC

Leak testing the lines from the Gas Source to the GC

Identifying and Removing Leaks

Back to top level troubleshooting list.

Leak-checking DPFC-equipped systems

With DPFC (Digital Pressure Flow Control), the circular sequence **Automatic Leak Check - Manual Check for Leaks - Column Evaluation - Automatic Leak Check** is the key for minimizing troubles related to leaks.

Automatic Leak Check

When you perform an Automatic Leak Check, the GC measures the column flow with a true mass flow sensor and compares it to the calculated flow from the original column constant to see if the numbers match. The instrument assumes a gas leak exists if there is a change and notifies you that the system is not leak tight. When the Automatic Leak Check highlights leaks, you should perform a manual leak check, to determine the location of the leak.

You should execute the Automatic Leak Check:

- once a day
- every time you have disconnected the gas lines for any reason (e.g. to clean or replace a component of the system or to install a new column).

Note that only a previous Column Evaluation, performed in a condition of true tightness, can ensure the validity of the subsequent Automatic Leak Check responses.

Column Evaluation

Once the leak has been removed and the tightness of the system is reasonably sure, you should perform the Column Evaluation automatic control and compare the response with the *K Factor values* reported in the **K Factor Quick Reference card** supplied with the Trace GC manuals. If the value obtained does not agree with the one reported on the card, the leaks have not been repaired.

Performing the Column Evaluation is the necessary condition for the success of any subsequent Automatic Leak Check. Click here for detailed instructions on Performing an Automatic Column Evaluation.

Leak-checking

Select the relevant procedure for leak-checking:

Performing an Automatic Leak Check

Performing an Automatic Column Evaluation

Checking for leaks - Capillary Columns, DPFC, DGFC

Checking for leaks - Capillary Columns, DPFC, Non-DGFC

Checking for leaks - Packed Columns, DPFC, DGFC

Checking for leaks - Packed Columns, DPFC, Non-DGFC

Leak testing the lines from the Gas Source to the GC

Identifying and Removing Leaks

Back to Leak-checking.

Back to top level troubleshooting list.

Leak-checking non-DPFC systems

To keep under control the tightness of a Non-DPFC equipped gas chromatograph you should check the system for leaks regularly, at least once a week.

In addition, you should also check for leaks every time you disconnect the gas lines for any reason (e.g. to clean or replace a component of the system or to install a new column).

Select the relevant procedure for leak-checking:

Checking for leaks - Capillary Columns, Non-DPFC, Non-DGFC

Checking for leaks - Packed Columns, Non-DPFC, Non-DGFC

Leak testing the lines from the Gas Source to the GC

Identifying and Removing Leaks

Back to Leak-checking.

Back to top level troubleshooting list.

Performing an Automatic Leak Check

(DPFC systems only)

1. Press LEAK CHECK to access the Leak Check control table.
2. Select the carrier gas channel you want to check and press ENTER. The channel selected is automatically pressurized with carrier gas and sealed to perform the leak check. The following message will be displayed:

   Please wait
   Leak check in progress
3. If the system is free of leaks, the following message is displayed:

Leak check: passed

4. If leaks are found, an error message will be displayed. In this case, eliminate leaks and repeat the leak check procedure.

Back to Leak-checking.

Back to top level troubleshooting list.

Performing an Automatic Column Evaluation

(DPFC systems only)

Note: Before you begin this procedure, make sure the GC is in stand-by condition at the initial temperature.

1. Press COLUMN EVAL to access the relevant control table.
2. Scroll to *Right column* or *Left column* (as required) and press ENTER. The applicable control table is displayed (parameters not editable). The GC automatically performs the column evaluation.
3. After a few minutes the following message will be displayed:

   L COL EVALUATION
   COMPLETED SUCCESSFULLY
   K = X.XX

4. Make a note of the displayed K Factor value.

Back to Leak-checking.

Back to top level troubleshooting list.

Leak testing the lines from the Gas Source to the GC

Single GC system

1. Switch off the gas chromatograph power supply.
2. Turn on all gas supply lines to the GC, setting the input pressure to 420 kPa (60 psi). Allow the gas lines to pressurize for several seconds.
3. Turn off the input gas source. The pressure value should not change more than 5% in 10 minutes. If the value drops down immediately, one or more leaks are present. In this case, carry on with step 4.
4. Check the connections with a handheld electronic leak detector to find the leaks.

Multiple GC systems

If more than one system is attached to the same gas source, you will need to isolate each GC system.

1. Install a cut-off valve and pressure gauge with an 1/8 inch input tee in the gas line leading to the GC that you are leak testing, as shown in the figure. Be sure the pressure gauge is connected in line between the on/off valve and the GC.
2. Turn on the gas supply and allow the system to pressurize.
3. Turn off the input gas source. The pressure value should not change more than 5% in 10 minutes. If the value drops down immediately, one or more leaks are present in the plumbing from the cut-off valve to the GC. In this case, carry on with step 4.
4. Check the connections of the line you are testing with a handheld electronic leak detector to find the leaks.

Back to Leak-checking.

Back to top level troubleshooting list.

Identifying and Removing Leaks

If the Automatic Leak Check (or any significant symptom) has notified a possible leak in the system, you should:

- Check the whole system for leaks, from the carrier gas inlet to the detector base body.
- Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum caps).

A possible source of leaks may be the gas bottles/gas chromatograph connections. Check these lines before the others, if specific symptoms indicate that the leak is outside the gas chromatograph.

Back to Leak-checking.

Back to top level troubleshooting list.

Baseline related problems with specific system components

Note: Use the **Back** button to return to this list after checking one of the possible causes below.

Baseline drift with changing pulse voltage (ECD)

Unstable baseline (NPD)

High standing current and noise (FPD)

No standing current (FPD)

Unstable and excessively noisy baseline (FPD)

No standing current (PID)

Baseline Fluctuation (TCD)

Baseline Drift (TCD)

High Baseline (MS)

Back to main Baseline problem list.

Baseline drifting

Possible Cause	Remedy
Accumulation of stationary phase.	Remove the end section of the column.
Carrier gas cylinder pressure too low to allow control.	Replace the carrier gas cylinder, or increase the pressure.
Drifting carrier gas or combustion gas flows.	Check the gas controllers.

Accumulation of impurities in the column.	Check impurity levels in the gas source. Use correct gas purity.

Back to main Baseline problem list.

Baseline falling

Possible Cause	Remedy
Carrier gas leak in the system.	Perform a leak test and check the tightness of the connections on the carrier gas line.
Column is baking out.	Allow enough time for the column to stabilize.

Back to main Baseline problem list.

Baseline falling away slowly after a high initial value

Possible Cause	Remedy
Purge valve left closed during acquisition.	Alter the GC program. See your GC user manual for details.
Inadequate purge flow rate.	Increase the purge flow rate.
Purge valve left closed for too long.	Shorten the purge time.
Solvent tail peak.	Increase the solvent delay or shorten the purge time.
Pre-filters are dirty. (When using a quadropole MS detector)	Contact your service representative.

Back to main Baseline problem list.

Baseline rising

Possible Cause	Remedy
Accumulation of impurities in the column.	Check impurity levels in the gas source. Use correct gas purity.
Contaminated detector.	Check the detector and clean it.
There is bleeding from the GC column.	Condition or change the column.
Air is leaking into the system. See Note 1.	Trace and repair the leak.

Note 1 With an air leak, the Baseline can also fall after an initial rise, as the GC ferrule expands with oven temperature and seals off the air.

Back to main Baseline problem list.

Baseline rising under temperature program control

Possible Cause	Remedy
Column contaminated.	Recondition the column.

Back to main Baseline problem list.

Baseline - high standing current

Possible Cause	Remedy
Carrier gas flow rate too high.	Reduce the carrier gas flow.
Column contaminated.	Recondition the column.

Contaminated gases.	Replace gas cylinders or gas filters.
Excessive column stationary phase bleeding.	Check the oven temperature, ensuring that it doesn't exceed the column upper limit. Recondition the column. Replace the column.
Loose connections.	Ensure that all interconnections and screw connections are tight.

Back to main Baseline problem list.

Baseline irregular shape: dip after solvent peak

Possible Cause	Remedy
Detector contaminated.	Bake out or clean the detector.

Back to main Baseline problem list.

Baseline irregular shape: S-shaped

Possible Cause	Remedy
Excessive column bleed during column temperature programming.	Reduce the upper column temperature. Bake out the column. Install a high temperature column.
Oxygen contamination is decomposing the stationary phase.	Install oxygen filters in the carrier gas line. Check the pneumatic and inlet systems for leaks. Use correct gas purity with low oxygen content.

Back to main Baseline problem list.

Baseline irregular shape: square waves

Possible Cause	Remedy
Large AC power fluctuations; heavy equipment on the same line.	Use a dedicated clean AC line of sufficient amperage.

Back to main Baseline problem list.

Baseline high frequency noise

Possible Cause	Remedy
Contaminated detector.	Isolate the detector from the electronics. If noise disappears, clean the collector.
Combustion gas flow too low or too high.	Check the detector gas flows.
Column contaminated.	Condition the column.
Contaminated detector gas supply.	Check the gas purity and install appropriate filters.
Defective electrometer.	Replace electrometer.
Detector temperature higher than column maximum temperature.	Reduce the detector temperature to the column temperature upper limit.
External electrical interference.	Attach an AC line monitor and check the AC supply for interference.
Loose column fittings.	Tighten fittings accordingly.

Loose detector electrical connections.	Make sure the leads are properly connected.

Back to main Baseline problem list.

Baseline spiking

Possible Cause	Remedy
Defective electrometer or amplifier.	Replace the electrometer or amplifier.
Column too close to flame. (When using an FID)	Lower the column to the correct position (2-3 mm below the tip of the jet).
Dirty jet or detector.	Isolate the detector from the electronics. If the spiking disappears, clean the jet and the collector.
FID temperature too low. (When using an FID)	Increase the FID temperature to at least 150°C.

Back to main Baseline problem list.

Peak related problems with specific system components

Note: Use the **Back** button to return to this list after checking one of the possible causes below.

Negative dips after peaks (ECD)
Tailing of solvent peak (PID)
Tailing of sample peaks (PID)
Negative Peaks (TCD)
Peak Tailing (MS)
Excessive broadening of solvent peak (PTV)

Back to main Peaks related problem list.

Peaks broadening

Possible Cause	Remedy
Column flow too high.	Reduce the flow to slightly above optimum.
Column flow too low.	Increase the flow to slightly above optimum.
Split flow too low in split injection.	Increase the flow to 40-50 ml/min.
Column performances degraded.	Test the column at the optimum flow rate.
Dirty injector.	Clean or replace the liner.
Stationary phase accumulated in the outlet.	Remove the last two coils from the column.
Detector base body temperature too low.	Increase the temperature to 5°C below the column maximum.
The sample is overloading the column.	Reduce the amount and/or concentration of the sample.

Back to main Peaks related problem list.

Peaks clipping at bottom

Possible Cause	Remedy
Detector or integrator zero set too low.	Set the zero correctly.

Back to main Peaks related problem list.

Peaks clipping at top

Possible Cause	Remedy
Data system zoomed in too close.	Zoom out to view the entire chromatogram.
Detector or integrator attenuation set too low.	Set the attenuation higher.
Detector range too sensitive.	Set a less-sensitive detector range.
Incorrect input to recording unit.	Correct and check the recording unit.

Back to main Peaks related problem list.

Double Peaks

Possible Cause	Remedy
Injection speed too low.	Inject more rapidly in a smooth motion.
Wrong autosampler injection speed or mode.	Use a higher speed.

Back to main Peaks related problem list.

Peak Fronting (excessive slope on left side)

Possible Cause	Remedy
Column or detector overloaded.	Decrease the injected amount and/or analyte concentrations. Increase the split ratio.
Column temperature too low.	Increase the temperature.
Stationary phase too	Use a thicker-film

thin.	column.
Poor injection technique.	Repeat, with better injection technique.

Back to main Peaks related problem list.

Ghost Peaks

Possible Cause	Remedy
Contaminated carrier gas.	Replace the cylinder or replace the filter.
Contamination from laboratory glassware.	Ensure the glassware is clean and contamination-free.
Decomposition of injected sample.	Decrease the injection port temperature. Use the on-column injection technique.
Dirty injection solution.	Carry out adequate clean-up of sample prior to injection.

Back to main Peaks related problem list.

Broad Ghost Peaks

Possible Cause	Remedy
Contaminated inlet or pneumatics.	Remove the column and bake out the inlet. Use a high-quality septum. Replace the split vent filter. Install an in-line filter between the pneumatics and the inlet.
Incomplete elution of previous sample.	Increase the final oven program temperature or total run time. Increase the column

flow rate.

Back to main Peaks related problem list.

Irregular, Chair-shaped Peaks

Possible Cause	Remedy
Solvent flooding of column.	Increase the initial oven temperature. Reduce the injection volume (OC). Install a retention gap (OC).

Back to main Peaks related problem list.

Negative Peaks

Possible Cause	Remedy
All Peaks negative	
Integrator wires reversed.	Correct the connections.
Some Peaks negative	
This symptom can be normal.	None required.

Back to main Peaks related problem list.

No Peaks after solvent peak

Possible Cause	Remedy
Carrier gas flow too high.	Reduce the carrier gas flow rate.
Combustion gas flow incorrect.	Check the combustion gas flow.
Detector contaminated.	Bake out or clean the detector.
FID flame	Check the detector

extinguished by solvent peak.	temperature.
Too much sample injected.	Inject less sample.
Incorrect column position in S/SL injector (too high).	Check the column position.

Back to main Peaks related problem list.

No Peaks at all

Possible Cause	Remedy
Clogged syringe needle.	Replace or repair the syringe.
Column broken or disconnected.	Check the column and connections.
Defective electrometer or amplifier.	Replace the electrometer or amplifier.
Defective recording device.	Replace the recording device.
FID flame is out.	Light the flame.
Poor or missing electrical connection.	Check the cable connections.
Incorrect column position in S/SL injector (too high).	Check the column position.

Back to main Peaks related problem list.

Sample Peak Tailing (excessive slope on right side)

Possible Cause	Remedy
Column degradation causing activity.	Inject a test mixture and evaluate the column.
Column/oven temperature too low.	Increase the column/oven temperature. Do not exceed the recommended maximum temperature for the stationary phase.
Dirty liner.	Clean or replace the liner.
Glass wool or inlet liner causing activity.	Replace with fresh silanized wool and a clean inlet liner.
Inlet temperature too low.	Increase the inlet temperature.
Poor or obstructed column connections.	Remake the column inlet connection.
Wrong stationary phase.	Replace the column according to the column manufacturer's literature.

Back to main Peaks related problem list.

Solvent Peak Tailing (excessive slope on right side)

Possible Cause	Remedy
Incorrect column position in inlet.	Reinstall the column.

Initial oven temperature too high (On Column).	Reduce the initial oven temperature.
Septum purge flow too low and/or split/splitless vent flow too low.	Check and adjust the septum purge and vent flows.
Too large injection size.	Reduce the injection size.

Back to main Peaks related problem list.

Unresolved Peaks

Possible Cause	Remedy
Carrier gas flow rate too high.	Reduce the carrier gas flow rate.
Column deteriorated.	Replace the column.
Column temperature too high.	Lower the column oven temperature.
Column too short.	Use a longer column.
Incorrect column choice.	Install a suitable column.
Injection technique is not adequate.	Choose a correct injection technique.

Back to main Peaks related problem list.

Discrete high-intensity contaminant peaks

Possible Cause	Remedy
Bleed from the GC column.	Condition or change the column.
Bleed from the septum.	Replace the septum.

Sample vial septa are contaminating the sample.	Discard sample. Store samples upright, in a refrigerator. Use teflon faced septa, with the teflon facing downwards (i.e. towards the sample).

Back to main Peaks related problem list.

Results related problems with specific system components

Note: Use the **Back** button to return to this list after checking one of the possible causes below.

FID Sensitivity problems
ECD Sensitivity problems
NPD Sensitivity problems
FPD Sensitivity problems
Low sensitivity (FPD)
Low sensitivity and water droplets generate between the heat shields (FPD)
PID Sensitivity problems
Low sensitivity (PID)
TCD Sensitivity problems
Low Sensitivity (TCD)
Low Sensitivity (MS)
Poor Reproducibility of Results (MS)
PTV Sensitivity problems
PTV Large Volume - Poor reproducibility of results
Analyses results are not reproducible (GSV)

Back to main Results related problem list.

Low reproducibility of peaks area

Possible Cause	Remedy
Concentration not compatible with the dynamic range of the detection system.	Ensure that the sample concentration is suitable for the detection system.
Inappropriate injection technique.	Try a different injection technique.

Injection parameters inappropriate.	Check the injection temperature and flow rates.
Non reproducible sample injection technique.	Evaluate the sample preparation sequences. Compare the results with a series of standard injections.
Leaking syringe or septum.	Check and replace the syringe and/or septum at regular intervals.
Leaks at the injection.	Check the column connections. Run a leak check.
Poor injection technique.	Carefully meter the injected amount. Use a clean, good-quality syringe.
Poor split flow or ratio control.	Monitor the flow. Replace the in-line filter.

Back to main Results related problem list.

Poor sensitivity with increased retention time

Possible Cause	Remedy
Carrier gas flow rate too low.	Increase the carrier gas flow rate. Locate and remove possible obstructions in the carrier gas line. Check the injector/column ferrules.

Back to main Results related problem list.

Poor sensitivity with normal retention time

Possible Cause	Remedy

Oven or injector parameters are not optimised.	Adjust the oven or injector parameters.
Leaks in the GC carrier gas line.	Run a leak test and correct leaks.
Syringe leaks during injection.	Replace syringe or piston seals, if applicable.
Split injection temperature too low.	Increase the temperature of the injector.
Column is in poor condition, or wrong column type used.	Condition or change the column.

Back to main Results related problem list.

Retention times decreasing

Possible Cause	Remedy
Stationary phase deteriorated by oxygen and/or water.	Use a carrier gas free of oxygen and water.
Stationary phase loss due to column bleeding.	Reduce the column temperature.

Back to main Results related problem list.

Retention times increasing

Possible Cause	Remedy
Increasing carrier leakage.	Check the septum and column connections.
Carrier gas supply running out.	Replace the bottle.

Back to main Results related problem list.

Low reproducibility of retention times

Possible Cause	Remedy
Drifting or unstable pneumatic controller.	Monitor the column pressure or flow. Check and replace the controller if necessary.
Poor injection technique.	Start the run at consistent time after injection.
Sample size is too large.	Reduce the injected amount and/or volume.
Unstable column temperature.	Check the main oven door and cooling flap. Monitor the column temperature.

Back to main Results related problem list.

Retention times are inconsistent

Possible Cause	Remedy
GC column is in poor condition.	Condition or change the column.
Insufficient equilibration time set on GC.	Increase equilibration time.
Poor injection.	Repeat with better injection technique.
Oven temperature programmed to rise too quickly.	Reduce oven temperature ramp rate.
Air is leaking into the system at the injector seal or the carrier gas manifold.	Trace and repair the leak.

Back to main Results related problem list.

FID Sensitivity problems

The sensitivity of the Flame Ionization Detector depends on the carrier and detector gases flow rates, and on the detector temperature.

The gas flow rates must be set correctly for proper FID operation.

Click here for the gases normally used with FID.

The carrier gas flow range depends on the type of gas used and on the type and diameter of the capillary or packed column installed.

Note: Make-up gas is not required when a packed column is used.

Click here for the recommended range of detector gas flow rates tolerated by the FID.

To gain optimum analytical performance from the FID, the hydrogen flow rate should be experimentally optimized, keeping the carrier and air flows constant, to obtain the maximum signal intensity for the components of interest.

The FID sensitivity will be reduced as hydrogen flow rate is above or below the optimal value. The flow rate of the air is less critical than the hydrogen one. An excessive amount of air will destabilize the flame, causing noise and eventual flameout.

Note: Generally the air flow rate must be set to about 10 times the hydrogen flow rate to keep the flame lit.

A low flow rate of air would reduce the detector sensitivity.

Click here for typical FID operating conditions.

For high sensitivity applications, it is essential that you exclude all traces of organic contamination from the chromatographic system and/or from the gas lines of the detector. Such contamination would cause ghost peaks in the chromatogram or, more commonly, an unstable baseline.

WARNING! It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details concerning hydrogen, please refer to **Using Hydrogen** on page xi of the Maintenance and Troubleshooting Manual.

Back to FID troubleshooting problem list.

FID gases

Carrier gas		Detector Gas	Make-up Gas
Capillary Columns	**Packed Columns**		
Helium	Helium	Hydrogen +	Nitrogen
Nitrogen	Nitrogen	Air	(recommended)
Hydrogen	Argon		Helium

FID detector gas flow rates

Gas	Flow rate
Hydrogen	30 - 50 ml/min

Air	300 - 600 ml/min
Make-up gas	10 - 60 ml/min

FID operating conditions

Gas	Capillary Columns	Packed Columns
Carrier	2 ml/min	40 ml/min
Hydrogen	35 ml/min	40 ml/min
Air	350 ml/min	500 ml/min
Make-up gas (Nitrogen)	30 ml/min	Not used

FID Flame Ignition problems

You can ignite the flame as soon as the detector temperature has reached 150°C.

After the flame appears to have ignited, check for water vapor condensed on a mirror or on the polished end of a wrench directly over the FID exit. You should observe steam condensing on the cold surface. If not, the flame is not ignited.

WARNING! Do not lean over the FID to see the flame, it is invisible.

The table below shows the possible causes of flame ignition problems and the relevant remedies.

Possible Cause	Remedy
Incorrect fuel gas flows.	Make sure that all gas flows are correct.
Defective igniter.	Check the igniter element. Refer to the **Replace the Ignition Assembly** Operating Sequence in Chapter 9 of the Maintenance and Troubleshooting Manual for instructions.
Broken or cracked flame jet.	Replace jet. Refer to the **Replace the Jet** Operating Sequence in Chapter 9 of the Maintenance and Troubleshooting Manual for instructions.
Blocked jet tip.	Check for a blocked jet by measuring the hydrogen flow with a

	flowmeter. Refer to the **Measure the Gas Flows with a Bubble Meter** Operating Sequence in Chapter 8 of the Maintenance and Troubleshooting Manual for instructions. If required, remove and clean the jet following the **Clean the Jet** Operating Sequence in Chapter 9 of the manual.
Faulty electronics.	Contact your service representative.

Back to FID troubleshooting problem list.

FID Contamination problems

All organic compounds that pass through the FID are burnt in the flame. Some compounds can produce detector contamination, resulting in a high noise baseline. If this symptom is found, clean the collecting electrode. Refer to the **Clean the Collecting Electrode** Operating Sequence in Chapter 9 of the Maintenance and Troubleshooting Manual for instructions.

Back to FID troubleshooting problem list.

ECD Contamination problems

The ECD, if properly used, has a good resistance against contamination thanks to its unique internal geometry. However, some critical operating conditions may give rise, in the long run, to contamination of the collecting electrode. This contamination is highlighted by an excessive increase of the base frequency and a baseline drift when the reference current or the pulse amplitude is changed. The collecting electrode, in these cases, can be easily removed and cleaned without disturbing the radioactive source. Refer to the **Clean the Collecting Electrode** Operating Sequence in Chapter 10 of the Maintenance and Troubleshooting Manual for instructions.

If contamination of the whole cell is suspected, heat the ECD at the maximum operating temperature with carrier and make-up gases flowing through the detector (thermal cleaning). The decontamination process can be followed by monitoring the base frequency. Initially the frequency value tends to increase to very high values, and then it decreases to acceptable ones. If irreversible contamination of the cell is suspected, please contact your local ThermoFinnigan Technical Service office for maintenance or replacement of the source, whichever is necessary. Strictly follow the existing regulations for the transport of radioactive material.

Detector contamination could be indicated by the following effects in the chromatogram:

- reduced signal to noise ratio
- high-noise baseline

- baseline drift with changing pulse voltage
- negative dips after peaks

Back to ECD troubleshooting problem list.

Warning: Radioactivity

WARNING! The Electron Capture Detector (ECD) contains a 63Ni beta-emitting radioactive source of 370 MBq (10 mCi).

The 63Ni radioisotope, electrically deposited as metal on a nickel foil, is inserted in a cylindrical holder made of 6 mm thickness stainless steel. The holder is fixed to the detector body, also made of stainless steel, in order to be totally inaccessible from the outside.

The radioisotope is not released by its support at temperatures lower than 450°C. This temperature can never be reached by the detector, whose maximum operating temperature is 400°C. A safety device prevents overheating.

In normal operating conditions, no dispersion of solid or gaseous radioactive material is involved. This eliminates any risk of direct or secondary radiation. For no reason should the detector be opened or handled by the user. Any maintenance or service operations involving even partial disassembling of the detector must be performed **ONLY** by CE Instruments factories (Milano and Austin) or by a laboratory specifically licensed to handle radioactive material.

ECD Sensitivity problems

The effect of a temperature change on the ECD response generally depends on the compounds analyzed.

The ECD behavior can be affected by a change of the total flow of the gases through the cell. The resulting response is also related to the nature and purity of the gases.

Click here for the gases normally used on the ECD.

When using helium or hydrogen as a carrier gas with capillary or wide bore columns, the detector should be fed with nitrogen or argon/methane through the make-up gas line.

The carrier gas flow rate must be set according to the type of the gas used and to the diameter and the stationary phase of the capillary (or packed) column installed.

Note: When packed columns are used, the make-up gas (as carrier gas type) is used only if strictly required.

WARNING! It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details concerning hydrogen, please refer to **Using Hydrogen** on page xi of the Maintenance and Troubleshooting Manual.

Back to ECD troubleshooting problem list.

ECD gases

Carrier Gas		Make-up Gas
Capillary Columns	**Packed Columns**	
Helium	Nitrogen	Nitrogen (recommended)
Nitrogen	Argon-Methane (5-10%)	Argon-Methane (5-10%)

Hydrogen

High base frequency (ECD)

Possible Cause	Remedy
Impure gas supply.	Use high purity grade gases and filters to trap moisture and oxygen.
No (or insufficient) flow of make-up gas.	Increase make-up gas flow rate.
Excess column bleeding.	Condition the column.
Leaks on carrier and/or make-up gas lines.	Run a leak test.
Contaminated collecting electrode.	Clean the collecting electrode, as described in the **Clean the Collecting Electrode** Operating Sequence in Chapter 10 of the Maintenance and Troubleshooting Manual, or replace it.
Chemically contaminated radioactive source.	Refer to ECD Contamination.
Pulse width not correctly set.	Set Pulse width to 1µs for Nitrogen and 0.1 µs for Ar/CH4.

Back to ECD troubleshooting problem list.

Negative dips after peaks (ECD)

Possible Cause	Remedy
Contaminated collecting electrode.	Clean the collecting electrode, as described in the **Clean the**

	Collecting Electrode Operating Sequence in Chapter 10 of the Maintenance and Troubleshooting Manual, or replace it.
Contaminated radioactive source.	Refer to ECD Contamination.

Back to ECD troubleshooting problem list.

Baseline drift with changing pulse voltage (ECD)

Possible Cause	Remedy
Contaminated collecting electrode.	Clean the collecting electrode, as described in the **Clean the Collecting Electrode** Operating Sequence in Chapter 10 of the Maintenance and Troubleshooting Manual. If the problem persists, replace the collecting electrode.

Back to ECD troubleshooting problem list.

NPD Sensitivity problems

The main cause of changes in sensitivity is related to the Thermionic source. Its gradual depletion results in a drop of sensitivity, that can be compensated by increasing the source current. Take into account that increasing current results in a shorter source life.

Loss of sensitivity can also be related to source contamination, due to high boiling sample compounds not completely eliminated.

Shift of detector temperature can reduce sensitivity.

Click here for the gases normally used for NPD. Consistent flows are necessary to maintain a constant and stable sensitivity.

Nitrogen is preferred over helium as make-up gas due to its lower thermal conductivity. Using nitrogen, the source requires a lower heating current.

Note: When a packed column is used, make-up gas generally is not necessary.

WARNING! It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details

concerning hydrogen, please refer to **Using Hydrogen** on page xi of the Maintenance and Troubleshooting Manual.

Click here for typical flow rates for detector gases.

Back to NPD troubleshooting problem list.

NPD gases

Carrier gas		Detector Gas	Make-up Gas
Capillary Columns	**Packed Columns**		
Helium	Helium	Hydrogen +	Nitrogen
Nitrogen	Nitrogen	Air	Helium
Hydrogen (only with DPFC)			

NPD operating conditions

Gas	Flow rate
Hydrogen	2 - 4 ml/min
Air	40 - 80 ml/min
Make-up gas	10 - 20 ml/min

No NPD detector response

Possible Cause	Remedy
Source heating current too low.	Increase the heating current.
No hydrogen flow.	Turn the hydrogen flow on and set it to a correct value.
No air flow.	Turn the air flow on and set it to a correct value.
Source turned off.	Turn the source on.
Source faulty.	Replace the source.

Back to NPD troubleshooting problem list.

NPD detector response lower than expected

Possible Cause	Remedy

Lower source temperature.	Check the base body temperature and source heating current.
Air contamination in hydrogen line.	Turn the source off. Increase the hydrogen pressure for 10-20 minutes to purge the hydrogen line. Check the line tightness.

Back to NPD troubleshooting problem list.

High background level (NPD)

Possible Cause	Remedy
Heating current too high.	Set the correct operating parameter.
Hydrogen flow too high.	Set the correct operating parameter.
Air and/or Make-up gas flow too low.	Set the correct operating parameter.
Excessive column bleed.	Condition the column.

Back to NPD troubleshooting problem list.

NPD shows FID-like response for solvent and other carbon based compounds

Possible Cause	Remedy
Hydrogen flow too high.	Reduce hydrogen flow to a lower value.

Back to NPD troubleshooting problem list.

Solvent quenching effect (NPD)

The "Solvent quenching effect" is observed when there is a large negative baseline upset at solvent elution with no recovery to the original baseline.

Possible Cause	Remedy
Heating current too low.	Slightly increase the source heating current.

Back to NPD troubleshooting problem list.

Unstable baseline (NPD)

Possible Cause	Remedy
Background current level too high.	Reduce the source heating current value.
Hydrogen flow fluctuation.	Check pressure regulators on hydrogen line.

Back to NPD troubleshooting problem list.

Low carbon rejection (NPD)

Possible Cause	Remedy
Hydrogen flow too high.	Reduce hydrogen flow to the correct operative conditions.

Back to NPD troubleshooting problem list.

FPD Sensitivity problems

The FPD sensitivity is affected by the temperature. The FPD response decreases with an increase in the detector temperature. In addition, the detector response can be severely reduced if a non-sulphur or non-phosphorus compound is partially or fully eluted with the analyte of interest (quenching).

Keep clean the interferential filter and the heat shields as described in the maintenance sequences, to avoid reducing sensitivity. Replace the heat shields if the deposits due to contamination cannot be eliminated.

A right choice of the flow rates of hydrogen and air (fuel gases) is of primary importance in determining FPD sensitivity. Flow rates will affect also selectivity and, to a great extent, peak shapes.

Optimum carrier gas flow rate depends on the type of gas used and on the diameter of the column installed.

Click here for the gases normally used with FPD.

Click here for the recommended values of flow rate for FPD.

The optimum air flow rate should be found experimentally, setting a proper value for the hydrogen and performing some analyses on a standard mixture at different air flow rates.

WARNING! It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details concerning hydrogen, please refer to **Using Hydrogen** on page xi of the Maintenance and Troubleshooting Manual.

Back to FPD troubleshooting problem list.

FPD gases

Carrier gas		Fuel Gas	Make-up Gas
Capillary Columns	**Packed Columns**		
Helium	Helium	Hydrogen +	Generally
Nitrogen	Nitrogen	Air	not required
Hydrogen	Argon		

FPD gas flow rates

Gas	Capillary Columns	Packed Columns
Carrier	1-3 ml/min	30-50 ml/min
Hydrogen	85-100 ml/min	100-120 ml/min
Air	100-120 ml/min	110-135 ml/min

The optimum air flow rate should be found experimentally, setting a proper value for the hydrogen and performing some analyses on a standard mixture at different air flow rates.

High standing current and noise (FPD)

Possible Cause	Remedy
Detector conditioning not completed.	Increase detector conditioning time.
Air flow rate set too high.	Reduce air flow rate.
Detector not light-tight.	Tighten the chimney cap and the knurled nut that fixes the photomultiplier tube.
lnterferential filter	Check the

missing.	interferential filter has been correctly installed.

Back to FPD troubleshooting problem list.

No standing current (FPD)

Possible Cause	Remedy
Signal or power cables incorrectly connected.	Check connections.
Faulty photomultiplier tube.	Contact your service representative.

Back to FPD troubleshooting problem list.

Unstable and excessively noisy baseline (FPD)

Possible Cause	Remedy
Faulty photomultiplier tube.	Contact your service representative.

Back to FPD troubleshooting problem list.

FPD temperature does not reach the set point

Possible Cause	Remedy
Power cable not properly connected.	Check cable connection.
Faulty heater.	Contact your service representative.

Back to FPD troubleshooting problem list.

Low sensitivity (FPD)

Possible Cause	Remedy

Low photomultiplier excitation voltage.	Set **High Voltage Mode** to **Yes** in the FPD control table.
Inadequate hydrogen flow rate.	Set the hydrogen flow rate to a proper value. Refer to Recommended gas flow rates for FPD.
Inadequate air flow rate.	Set the air flow rate to a proper value. Refer to Recommended gas flow rates for FPD and relevant note.
Dirty interferential filter.	Clean the interferential filter. Refer to **Clean the Interferential Filter** Operating Sequence in Chapter 12 of the Maintenance and Troubleshooting Manual.
The mirror of the combustion chamber is dirty.	Clean the mirror. Refer to **Clean the Mirror Metal Plug** Operating Sequence in Chapter 12 of the Maintenance and Troubleshooting Manual.
The flame-side heat shield is dirty.	Clean the flame-side heat shield. Refer to **Clean the Flame-side Heat Shield** Operating Sequence in Chapter 12 of the Maintenance and Troubleshooting Manual.
Reduced transparency of the heat shields.	Replace the heat shields. Refer to **Replace the Heat Shields** Operating Sequence in Chapter 12 of the Maintenance and Troubleshooting Manual.

Back to FPD troubleshooting problem list.

Low sensitivity and water droplets generate between the heat shields (FPD)

Possible Cause	Remedy
Leak at the flame-side heat shield.	Replace the heat shields. Refer to **Replace the Heat Shields** Operating Sequence in Chapter 12 of the Maintenance and Troubleshooting Manual. If the symptom does not disappear, contact your service representative.

Back to FPD troubleshooting problem list.

UV lamps for PID

Lamp Type	Application
8.4 eV	Determination of amines and polycyclic aromatic compounds.
9.6 eV	Determination of low-boiling aromatic compounds (BTEX analyses).
10.6 eV	General applications. Filled with krypton, emits at 10.0 and 10.6 eV.
11.8 eV	Determination of aldehydes and ketones.

PID Sensitivity problems

The most common cause of loss of sensitivity is condensation of the effluent on the lamp window, resulting in a progressive increase of absorption of the UV. For long life, the window must be cleaned periodically according to the operating sequence described in the **Clean the Lamp Window** Operating Sequence in Chapter 13 of the Maintenance and Troubleshooting Manual.

A Lamp failure message will be displayed when the lamp does not work. One of the possible causes is the breakdown of the lamp. In this case, replace the lamp following the instructions given in the in the **Replace the UV Lamp** Operation Sequence in Chapter 13 of the Maintenance and Troubleshooting Manual.

See also: Low sensitivity (PID)

Back to PID troubleshooting problem list.

PID Contamination problems

An abnormally high baseline may indicate contamination of the detector. In this case, turning off the lamp will not make the symptom disappear. This means that the detector cell has been contaminated.

Try to solve the problem by baking the cell for two hours at 400°C. If the symptom does not disappear, contact your service representative.

If you suspect contaminants are carried into the detector cell by the gas lines, try reducing the flow rate in each line, one at a time, to identify the contamination source.

To avoid detector contamination install gas filters between the gas cylinders and the detector and replace them periodically.

Note: To face more effectively possible chromatographic problems, you should carry out an analysis using the test mixture and evaluate the results.

Back to PID troubleshooting problem list.

UV lamp does not light immediately (PID)

Possible Cause	Remedy
The lamp has not been used for a long time.	Set **High current mode?** to **Y** until the lamp lights, then reset to **N**.

Back to PID troubleshooting problem list.

UV lamp does not light at all (PID)

Message: Lamp failure

Possible Cause	Remedy
Lamp not installed.	Install a suitable UV lamp. Refer to the **Replace the UV Lamp** Operating

	Sequence in Chapter 13 of the Maintenance and Troubleshooting Manual.
Lamp not correctly installed in the lamp holder.	Reinstall the UV lamp. Refer to the **Replace the UV Lamp** Operating Sequence in Chapter 13 of the Maintenance and Troubleshooting Manual.
Lamp holder not correctly fixed to the lamp housing.	Tighten the two knurled screws that fix the lamp holder to the housing.
Lamp power cable disconnected.	Check the connection.
Polarization cable disconnected.	Check the connection.
Safety interlock circuit is faulty.	Contact your service representative.
UV lamp is exhausted.	Replace UV lamp. Refer to the **Replace the UV Lamp** Operating Sequence in Chapter 13 of the Maintenance and Troubleshooting Manual.

Back to PID troubleshooting problem list.

No standing current (PID)

Possible Cause	Remedy
Power and signal cables not properly connected.	Check the connections of the cables.

Back to PID troubleshooting problem list.

Tailing of solvent peak (PID)

Possible Cause	Remedy
Insufficient make-up gas flow rate.	Increase the flow rate.

Back to PID troubleshooting problem list.

Tailing of sample peaks (PID)

Possible Cause	Remedy
Adsorption effects caused by degradation products inside the cell.	Remove deposits by baking the cell for two hours at 400°C. If the symptom does not disappear, contact your service representative.
Contamination of the cell.	Remove contaminants by baking the cell for two hours at 400°C. If the symptom does not disappear, contact your service representative.
Inadequate gas flow rate settings.	Set gas flow rates to proper values.
Leaks on the gas lines.	Perform a leak test.

Back to PID troubleshooting problem list.

Low sensitivity (PID)

Possible Cause	Remedy
Lamp window fogged by UVabsorbing deposits.	Clean the lamp window. Refer to the **Clean the Lamp Window** Operating Sequence in Chapter 13 of the Maintenance and Troubleshooting

	Manual.
Flow rate of the make-up gas is set too high.	Reduce the flow rate of the make-up gas down to10 ml/min or less.
UV lamp is not adequate to the compounds to be analyzed.	Replace with an appropriate one.

Back to PID troubleshooting problem list.

TCD Sensitivity problems

Sensitivity is related to the detector temperature (increasing temperature reduces sensitivity) and to the flow rate of the carrier, reference and make-up gas. A gain in sensitivity can be obtained by increasing the gap between the temperature of the block and that of the filaments, or increasing the filaments voltage.

Sensitivity is strictly related to the state of the filaments and to their operating conditions. A significant reduction of the detector sensitivity may be caused by the contamination of the filaments due to degradation of high molecular weight compounds inside the cell or to contaminated gases.

Low temperatures of the detector block may cause high boiling compounds to condensate on the filaments, reducing sensitivity.

See also Low Sensitivity (TCD).

Back to TCD troubleshooting problem list.

Negative Peaks (TCD)

Negative peaks are normally generated by the sample components that have a thermal conductivity higher than carrier gas. For instance, using nitrogen or argon as carrier gas, negative peaks are obtained for helium, hydrogen or methane.

To revert the polarity of the detector, refer to the Trace™ GC Operating Manual for instructions.

Back to TCD troubleshooting problem list.

Baseline Fluctuation (TCD)

Possible Cause	Remedy
Unstable regulation of the flow rate of the gases.	Check the controllers of the carrier, reference and make-up gases work well.
Leaks on the gas lines.	Leak test the connections of the carrier, reference and

	make-up gas.
Inlet pressure of gases set too low.	Set the pressure of the carrier, reference and make-up gas to a proper value. Refer to the Trace GC Operating Manual for instructions.
Trans temp set too high.	Set *Trans temp* to a proper value (about 10 to 20°C below the *Block temp* value).
Faulty temperature regulation.	Contact your service representative.

Back to TCD troubleshooting problem list.

Baseline Drift (TCD)

A small baseline drift normally occurs during a temperature program and does not indicate a problem. This effect is due to the decrease of the carrier gas flow rate as temperature increases.

A baseline that suddenly goes out of scale and a rapid growth of the signal could indicate that filaments are likely to be burnt and have to be replaced.

Possible Cause	Remedy
Unstable regulation of the flow rate of the gases.	Check the controllers of the carrier, reference and make-up gases work well.
Leaks on the gas lines.	Leak test the connections of the carrier, reference and make-up gas.
Column conditioning not correctly performed.	Recondition the column according to the manufacturer's instructions.
Septum bleeding.	Check the operating temperature of the septum is adequate. Replace the septum if necessary.

Faulty temperature control.	Contact your service representative.

Back to TCD troubleshooting problem list.

Low Sensitivity (TCD)

Possible Cause	Remedy
Leaks on the gas lines.	Leak test the connections of the carrier, reference and make-up gas.
Leak due to septum wearing.	Replace the septum.
Operating conditions of the detector not properly set.	Optimize working parameters of the detector according to the actual operating mode (Constant Voltage, Constant Temperature). Refer to the Trace GC Operating Manual for instructions.
Thermal conductivity of the carrier gas is too close to that of the compound to be analyzed.	Use a different carrier gas. Refer to the Trace GC Operating Manual for instructions.
Contaminated filaments.	Remove contamination by baking the filaments for one hour at a temperature higher than the boiling point of the most high-boiling compound. Perform baking twice if necessary. If the symptom does not disappear, contact your service representative.

Oxidized filaments.	Contact your service representative.

Back to TCD troubleshooting problem list.

TCD does not work

Message: Filament power Off

Possible Cause	Remedy
Lack of carrier, make-up or reference gas or pressure too low.	Check the feed of the carrier, make-up and reference gas.
Filament burnt.	Contact your service representative.

Back to TCD troubleshooting problem list.

High Baseline (MS)

Possible Cause	Remedy
Contaminated MS source.	Clean the Source.
Air is leaking into the GC/MS interface.	Trace and repair the leak.

Back to MS troubleshooting problem list.

Peak Tailing (MS)

Possible Cause	Remedy
MS Source or GC Interface is not hot enough.	Increase the Source or GC Interface temperature.

Back to MS troubleshooting problem list.

Low Sensitivity (MS)

Possible Cause	Remedy
MS ion source is dirty.	Clean the source.
MS ion source temperature is not optimized.	Re-tune the MS or adjust the source temperature.
MS is not tuned properly.	Tune the MS.
Incorrect column alignment in the MS source.	Re-align the column in the MS source.
Detector voltage set too low (Trace MS)	Increase the Detector Voltage in the Trace MS Tune window.

Back to MS troubleshooting problem list.

Poor Reproducibility of Results (MS)

Possible Cause	Remedy
MS ion source is dirty or badly assembled.	Disassemble, clean and reassemble the ion source.
Old or damaged MS source filament.	Replace the source filament.
Poor MS calibration.	Re-calibrate the MS.
Poor MS tuning.	Re-tune the MS.
Loose connections in the MS ion source.	Check the source connections.

Back to MS troubleshooting problem list.

PTV Sensitivity problems

Low sensitivity is commonly caused by incorrect operating conditions. It might also be due to a dirty liner or to a liner wrong sized with regards to the volume of the sample injected.

Lack of sensitivity for selected compounds can also be related to discrimination phenomena induced by inadequate injection conditions. See PTV Discrimination problems and the related topics.

Back to PTV troubleshooting problem list.

PTV Discrimination problems

Possible discrimination of the heavy or volatile fraction of the sample may be caused by operating conditions set incorrectly with regard to the chosen injection mode.

Discrimination could also be caused by:

- degradation of the thermally labile compounds due to the cathalytic effect of bad deactivated quartz wool inside the liner;
- by-products originated in previous injections that have been not completely eliminated from the liner during the cleaning phase or the cleaning operating sequence.

PTV Injector Troubleshooting provides a list of possible symptoms relating to discrimination when using a PTV Injector. Select the one that best matches your problem, and then investigate the probable causes and the suggested remedies.

Back to PTV troubleshooting problem list.

Discrimination of heavy compounds in splitless mode (PTV)

Possible Cause	Remedy
Transfer temperature too low.	Set *Transfer temp* and cleaning (without backflush) temperature to a higher value.
Transfer time too low.	Set transfer time or cleaning time to a value close to the whole GC run time.
Splitless time too short.	Set *Splitless time* to a higher value.
Initial temperature too high with regard to the solvent boiling point.	Set *Inject temp* to a value closer to the solvent boiling point.
Flooding of the liner	Set *Inject temp* to a

due to low injection temperature.	higher value. If the symptom does not disappear, insert quartz wool in the liner. Refer to **Adjust a Quartz Wool Packing** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Flooding of the liner due to injected volume too large.	Reduce the amount of sample to be injected. Replace the liner with one of adequate diameter. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Flooding of the liner due to insufficient size.	Replace the liner with one of larger diameter. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Dirty liner.	Clean or replace the liner. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Liner not suitable for the actual sample.	Replace the liner with an adequate one. A lower diameter liner would improve the sample transfer. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance

	and Troubleshooting Manual.
Quartz wool causing excessive retention of high molecular compounds.	Replace the packed liner with a new one filled will quartz wool. Replace the packed liner with an empty one. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.

Back to PTV troubleshooting problem list.

Discrimination of volatile compounds in splitless mode (PTV)

Possible Cause	Remedy
Initial temperature too high.	Set *Inject temp* to a lower value.
Liner diameter too small.	Replace the liner with one of larger diameter. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Volatiles are eliminated through the purge line.	Set *Const purge flow?* to **N**, then set *Stop purge for* to a value corresponding to *Splitless time.*

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Discrimination of volatile compounds in solvent split mode (PTV)

Possible Cause	Remedy
Initial temperature too high.	Set *Inject temp* to a lower value.
Injection and evaporation time are set as to exceed the time required for the solvent elimination.	Set *Inject time* and *Evaporation time* to adequate values.
Split flow set too high.	Set *Split flow* to a lower value.
Lack of an adequate quartz wool packing inside the liner.	Insert quartz wool in the liner.

Back to PTV troubleshooting problem list.

Discrimination in split mode (PTV)

Possible Cause	Remedy
Initial temperature too low.	Set *Inject temp* to a higher value.
Injected volume too large.	Reduce the amount of sample to be injected. Replace the liner with the 2 mm ID version. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Lack of an adequate quartz wool packing inside the liner.	Replace the packed liner with a new one filled will quartz wool. Fix the quartz wool packing inside the

	liner. Refer to **Adjust a Quartz Wool Packing** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.

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Excessive broadening of solvent peak (PTV)

Note: Volatile compounds may also be obscured and a lack of sensitivity observed.

Possible Cause	Remedy
Injection pressure set too high.	Set *Inject pressure* to a lower value.

Back to PTV troubleshooting problem list.

Sample degradation (PTV)

Possible Cause	Remedy
Dirty liner.	Clean or replace the liner. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Transfer temperature set too high.	Set *Transfer temp* to a value adequate to the nature of the sample compounds.
Liner diameter too large.	Replace the liner with one of smaller diameter to improve transfer efficiency. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting

	Manual.
Cathalytic and thermal degradation of sensitive compounds caused by the quartz wool inside the liner.	Use a new packed liner. If the symptom does not disappear, use an empty liner. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.

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PTV Large Volume - Poor reproducibility of results

Note: Excessive broadening of the solvent peak and broadening and irregular shape of sample peaks may also be observed.

Possible Cause	Remedy
Injection speed set too high.	Reduce injection speed.
Injected volume too large.	Reduce the amount of sample to be injected.
Initial temperature set too low.	Set *Inject temp* to a higher value.
Injection pressure set too high.	Set *Inject pressure* to a lower value.
Lack of an adequate quartz wool packing inside the liner.	Fix the quartz wool packing inside the liner. Refer to **Adjust a Quartz Wool Packing** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual. Replace the liner with another filled with quartz wool. Refer to

	Clean or Replace the Liner Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.

Back to PTV troubleshooting problem list.

PTV Large Volume - Discrimination of volatile compounds

Note: Solvent peak may be significantly reduced.

Possible Cause	Remedy
Injection speed set too low.	Set *Inject speed* to a higher value.
Initial temperature set too high.	Set *Inject temp* to a lower value.
Split flow set too high.	Lower the split flow.
Lack of an adequate quartz wool packing inside the liner.	Fix the quartz wool packing inside the liner. Refer to **Adjust a Quartz Wool Packing** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual. Replace the liner with another filled with quartz wool. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.

Back to PTV troubleshooting problem list.

PTV Large Volume - Sample degradation

Possible Cause	Remedy
Cathalytic degradation of sensitive compounds caused by the quartz wool.	Use an empty deactivated liner reducing the volume injected. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.
Cathalytic degradation of sensitive compounds caused by by-products accumulated on the quartz wool.	Replace the packed liner with a new one. Refer to **Clean or Replace the Liner** Operating Sequence in Chapter 6 of the Maintenance and Troubleshooting Manual.

Back to PTV troubleshooting problem list.

Analyses results are not reproducible (GSV)

Possible Cause	Remedy
Loading time is too short (loop not completely filled by sample).	Increase the loading time.
Injecting time is too short (sample not completely transferred to the column).	Increase the injection time.
Leaks on the valve or gas line connections.	Check all valve and gas line connections for leaks.
The rotor of the valve is worn and causes gas leaks.	Replace the valve body.

The diaphragm of the valve is worn and causes gas leaks.	Replace the diaphragm. Refer to **Replace the Diaphragm on an Automatic Valve** in Chapter 7 of the Maintenance and Troubleshooting Manual.
The frit of the filter is dirty.	Clean or replace the frit. Refer to **Clean or Replace the Filter on an Automatic Valve** in Chapter 7 of the Maintenance and Troubleshooting Manual.

Back to GSV troubleshooting problem list.

No signal at all (GSV)

Possible Cause	Remedy
The frit of the filter is obstructed.	Replace the frit. Refer to **Clean or Replace the Filter on an Automatic Valve** in Chapter 7 of the Maintenance and Troubleshooting Manual.

Back to GSV troubleshooting problem list.

GSV does not work

Possible Cause	Remedy
Servo air pressure absent or too low.	Check the servo air source.
Faulty electrovalve on the servo air line.	Contact your service representative.
The diaphragm is	Replace the

	damaged.	diaphragm. Refer to **Replace the Diaphragm on an Automatic Valve** in Chapter 7 of the Maintenance and Troubleshooting Manual.

Back to GSV troubleshooting problem list.

Checking for leaks - Capillary Columns, DPFC, DGFC

1. With the capillary column installed, cool down the injector and the detector base body to room temperature.
2. *Only for FID, NPD, FPD:* remove the detector. Do not remove ECD detector.
3. *Only for FPD:* remove the jet. Use typewriter correction fluid to mark the position of the column entering the detector base body. Lower the column end down to the top of the detector base body, then tighten the M4 fixing nut to restore system tightness.
4. Finger tighten the relevant blind cap (fitted with a silicon seal) on the detector base body, to seal the column flow path.
5. *Only for ECD:* remove the chimney and seal the line with the relevant blind 6MB nut.
6. Turn off any H2, air and make-up gas flows. Do not close the detector's gases supplies.
7. Turn off the split and septum purge vents (if any).
8. Set the inlet pressure to 100-200 kPa.
9. Set the flow of the make-up gas to the maximum acceptable value to speed the pressurization of the line.
10. Wait until the system is equilibrated. Air, Make-up and H2values need not necessarily be equal.
11. Turn off the inlet pressure and the make-up gas. The shown values should not change more than 5% in 10 minutes. If the values drop down immediately, one or more leaks are present. In this case, carry on with step 12.
12. Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum cap) with a handheld electronic leak detector to find possible leaks.
13. If no leak is detectable in this way, contact your service representative.

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Checking for leaks - Capillary Columns, DPFC, Non-DGFC

1. With the capillary column installed, cool down the injector and the detector base body to room temperature.
2. *Only for FID, NPD, FPD:* remove the detector. Do not remove ECD detector.

3. *Only for FPD:* remove the jet. Use typewriter correction fluid to mark the position of the column entering the detector base body. Lower the column end down to the top of the detector base body, then tighten the M4 fixing nut to restore system tightness.
4. Finger tighten the relevant blind cap (fitted with a silicon seal) on the detector base body, to seal the column flow path.
5. *Only for ECD:* remove the chimney and seal the line with the blind 6MB nut.
6. Turn off the H2 and air gas flows.
7. Turn off the split and septum purge vents (if any).
8. Set the inlet pressure to 100-200 kPa.
9. Open the make-up gas line to speed the pressurization of the detector side of the system. Use an external pressure gauge (e.g. the gauge installed on the bottle) to monitor pressure.
10. Wait until the system is equilibrated.
11. Turn off the inlet pressure and the make-up gas. The shown values should not change more than 5% in 10 minutes. If the values drop down immediately, one or more leaks are present. In this case, carry on with step 12.
12. Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum cap) with a handheld electronic leak detector to find possible leaks.
13. If no leak is detectable in this way, contact your service representative.

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Thermo ELECTRON CORPORATION

Checking for leaks - Capillary Columns, Non-DPFC, Non-DGFC

1. With the capillary column installed, cool down the injector and the detector base body to room temperature.
2. *Only for FID, NPD, FPD:* remove the detector. Do not remove ECD detector.
3. *Only for FPD:* remove the jet. Use typewriter correction fluid to mark the position of the column entering the detector base body. Lower the column end down to the top of the detector base body, then tighten the M4 fixing nut to restore system tightness.
4. Finger tighten the relevant blind cap (fitted with a silicon seal) on the detector base body, to seal the column flow path.
5. *Only for ECD:* remove the chimney and seal the line with the blind 6MB nut.
6. Turn off the H2 and air gas flows.
7. Turn off the split and septum purge vents (if any).
8. Open the ON/OFF knob that controls the carrier gas, then adjust the inlet pressure to 100-200 kPa using a screwdriver. You can monitor the pressure value on the gauge located close to the regulator.
9. Open the make-up gas line to speed the pressurization of the detector side of the system. Use an external pressure gauge (e.g. the gauge installed on the bottle) to monitor pressure.
10. Wait until the system is equilibrated.
11. Turn off the inlet pressure and the make-up gas. The shown values should not change more than 5% in 10 minutes. If the values drop down immediately, one or more leaks are present. In this case, carry on with step 12.
12. Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum cap) with a handheld electronic leak detector to find possible leaks.

13. If no leak is detectable in this way, contact your service representative.

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Checking for leaks - Packed Columns, DPFC, DGFC

1. With the packed column installed, cool down the injector and the detector base body to room temperature.
2. *Only for FID, NPD, FPD:* remove the detector. Do not remove ECD detector.
3. *Only for FPD:* remove the jet.
4. Finger tighten the relevant blind cap (fitted with a silicon seal) on the detector base body, to seal the column flow path.
5. *Only for ECD:* remove the chimney and seal the line with the blind 6MB nut.
6. Turn off any H2, air and make-up gas flows. Do not close the detectors gases supplies.
7. Turn off the split and septum purge vents (if any).
8. Set the inlet pressure to 100-200 kPa.
9. Wait until the system is equilibrated (Air, Make-up and H2 values need not necessarily be equal).
10. Turn off the inlet pressure. Pressure value should not change more than 5% in 10 minutes. If the value drops down immediately, one or more leaks are present. In this case, carry on with step 11.
11. Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum cap) with a handheld electronic leak detector to find possible leaks.
12. If no leak is detectable in this way, contact your service representative.

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Checking for leaks - Packed Columns, DPFC, Non-DGFC

1. With the capillary column installed, cool down the injector and the detector base body to room temperature.
2. *Only for FID, NPD, FPD:* remove the detector. Do not remove ECD detector.
3. *Only for FPD:* remove the jet.
4. Finger tighten the relevant blind cap (fitted with a silicon seal) on the detector base body, to seal the column flow path.
5. *Only for ECD:* remove the chimney and seal the line with the blind 6MB nut.
6. Turn off any H2, air and make-up gas flows.
7. Turn off the split and septum purge vents (if any).
8. Set the inlet pressure to 100-200 kPa.
9. Wait until the system is equilibrated.
10. Turn off the inlet pressure. The shown value should not change more than 5% in 10 minutes. If the value drops down immediately, one or more leaks are present. In this case, carry on with step 11.

11. Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum cap) with a handheld electronic leak detector to find possible leaks.
12. If no leak is detectable in this way, contact your service representative.

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Checking for leaks - Packed Columns, Non-DPFC, Non-DGFC

1. With the capillary column installed, cool down the injector and the detector base body to room temperature.
2. *Only for FID, NPD, FPD:* remove the detector. Do not remove ECD detector.
3. *Only for FPD:* remove the jet.
4. Finger tighten the relevant blind cap (fitted with a silicon seal) on the detector base body, to seal the column flow path.
5. *Only for ECD:* remove the chimney and seal the line with the blind 6MB nut.
6. Turn off any H2, air and make-up gas flows.
7. Turn off the split and septum purge vents (if any).
8. Open the ON/OFF knob that controls the carrier gas, then adjust the inlet pressure to 100-200 kPa using a screwdriver. You can monitor the pressure value on the gauge located close to the regulator.
9. Wait until the system is equilibrated.
10. Turn off the inlet pressure. The shown value should not change more than 5% in 10 minutes. If the value drops down immediately, one or more leaks are present. In this case, carry on with step 11.
11. Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum cap) with a handheld electronic leak detector to find possible leaks.
12. If no leak is detectable in this way, contact your service representative.

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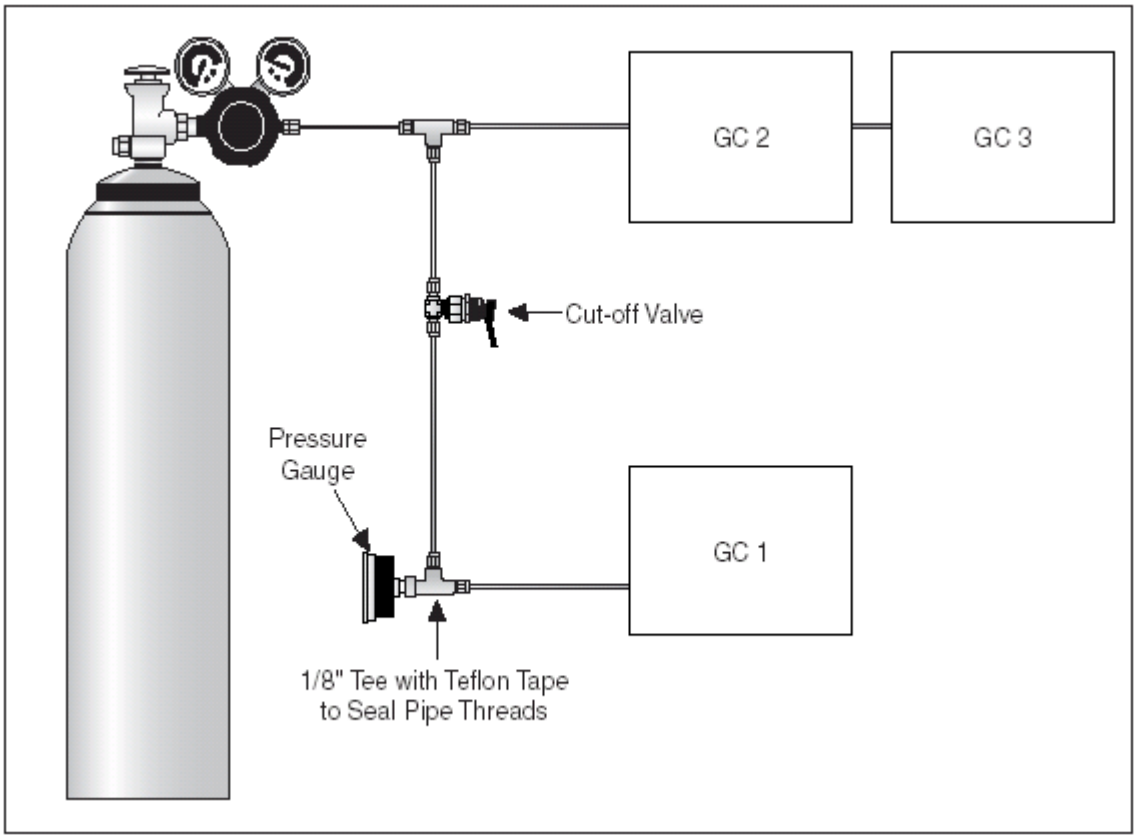
Multi GC system



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