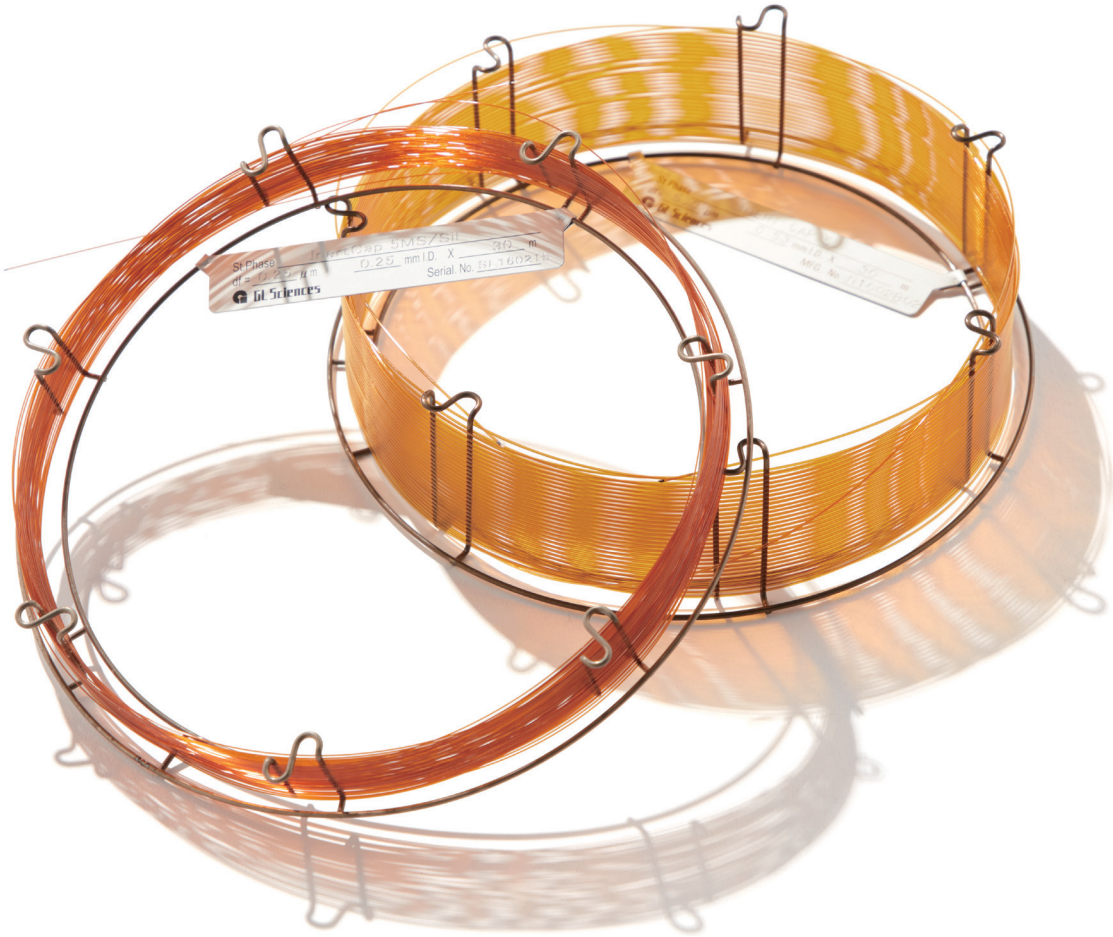
GC

Gas Chromatography

Troubleshooting Guide



GL Sciences

GC Troubleshooting Guide

Abnormal peaks

Tailing peak (Analyte)

Tailing peak (Solvent)

Leading peak

Split peak

Fluctuation in retention time

Retention times decrease

Peaks not detected

All peaks are too small

Poor repeatability of peak area and response

Ghost peaks: a peak appears where you do not expect a peak

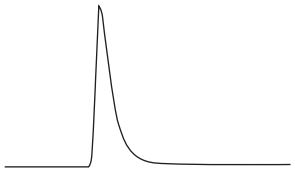
Decrease in column performance: peak broadening / peak tailing

"Batman" peaks or "low-slope" leading peaks

A cluster of peaks elute before the main component

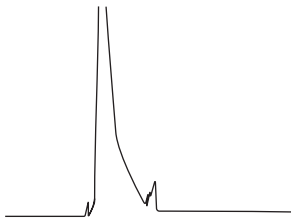
Abnormal peaks

Tailing peak (Analyte)



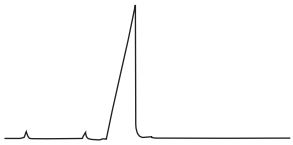
Main cause	Solution
Column installation position in the injection port and/or the detector is incorrect.	Check the insertion length of the column and ensure it is in the correct position as specified in the instruction manual for each instrument. Make sure that the column is positioned just below the flame tip (FID) or near the point of detection.
Contamination of the liner by matrix, septa or graphite. Liner temperature too low.	Replace the liner or replace wool. Be carefull as wool can break and cause activity;Increase liner temperature.
Contamination or activity in the column.	Cut-off about 50 cm from the injection port side of the column. If still not OK, cut another 50 cm. If not OK, connect detector side to injector and recondition the column for several hours at its maximum operation temperature. If not OK, replace column.
Split ratio is too low.	Increase the split ratio.
Dead volume present.	Check column position in inlet and outlet. If coupling is used check that column ends are correctly cut using the wafer cutting device supplied with the column.
A very polar, basic or acidic component.	When the component has high polarity, it may show adsorption. If possible, Increase initial oven temperature and use a higher programming rate. Try using a thicker film and/or derivatize the component.
The tailing peak elutes just before a big solvent peak.	Reduce sample size by smaller injection volume or increase split ratio. Choose a thicker film or a stationary phase where the solvent peak elutes later or earlier then the tailing peak.
Overloaded peak on a PLOT column.	Reduce sample size by smaller injection volume or increase split ratio. Use a PLOT column with thicker layer or larger diameter.

Tailing peak (Solvent)



Main cause	Solution
Injection volume too big.	Reduce injection volume. Increase split ratio.
Liner diameter to small.	Use a larger diameter liner.
O-ring in liner is not sealing.	Replace O-ring.
Column inlet position too low in injection port.	Check required length for your instrument and adjust. To secure position especially when a new ferrule is used, slide the column inlet first through a used septum, then put the nut and ferrule on the column, cut 1 cm from the inlet using the ceramic wafer and use the septum to secure the correct insertion depth.
Injector temperature to low.	Increase injection port temperature.

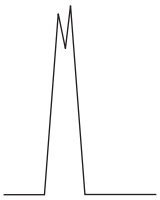
Leading peak



Main cause	Solution
The amount of analyte injected exceeds the column loading capacity (liquid phase). Retention time increases.	Reduce the injection volume and/or increase the split ratio so that it does not exceed the loading capacity of the column. Dilute the sample. Use a column with a thicker film or a wider diameter to increase the sample loading capacity.
The polarity of the analyte is not compatible with the stationary phase.	Use a stationary phase with better solvability for this component. (For polar compounds, use a polar stationary phase, like InertCap PureWax. For non polar compounds, use a non-polar stationary phase (like InertCap1 or 5).

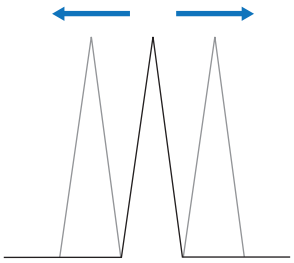
Abnormal peaks

Split peak



Main cause	Solution
Injection conditions are incorrect.	When making a manually injection, the injection speed is unsuitable and/or the syringe is faulty. Replace the syringe or use an autosampler.
The column is not correctly connected to the injection port.	Check the insertion length of the column as recommended in the instruction manual for your GC.
The injection solvent (the sample) is a mixed solvent.	This occurs when using splitless injection and on-column injection. Change to a single solvent.
Mismatch polarity of stationary phase and solvent of sample. With splitless injection, multiple injection bands may be formed due to droplet formation.	Change either the stationary phase that is compatible with the solvent or use a sufficiently long retention gap to make sure all solvent (droplets) are evaporated in the retention gap.

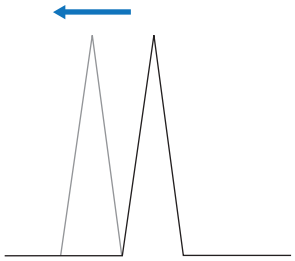
Fluctuation in retention time



Main cause	Solution
Leak of carrier gas.	Check for leaks (use digital leak detection device), around the injection port and column connections. When the inlet septum leaks, replace it. Check how many injections(xx) are required before a serious leak of septum develops. Then replace septa after xx injections before the leak developes.
Different initial oven temperature.	Check initial oven temperature.
Mixing of carrier gases via leaking gas selection valve.	Replace the gas selection valve.
Restriction built-up in detector flame tip (FID).	Replace or clean flame tip.
Restriction built-up in the tubing, malfunction of the flow control device. Clogging of the split line filter.	When the split vent line becomes blocked, wash or replace it. When the flow control device malfunctions it should be replaced. Replace split line filter. Make this routine maintenance.
The carrier gas supply is too low.	Verify the supply pressure to the GC is correct.
Alumina or Molsieve PLOT column not sufficiently conditioned. Retention depends on residue adsorbed water and CO_2.	Condition the column for several hours at maximum temperature to remove all water and CO_2.

Abnormal peaks

Retention times decrease



Main cause	Solution
Column looses stationary phase (bleed is high) because air/water is entering the system.	Check for leaks at column connections and carrier gas tubing connections. Check gas filtration systems, replace filters.
Column operated at too high temperature.	Decrease final programmed oven temperature; Use flow programming. Use a more temperature stable phase.
Water is adsorbed on a Alumina or Molsieve PLOT column.	Remove water from the sample. Replace moisture filter for carrier gas. Remove water after each analysis by heating the column to Tmax for 10 minutes. Use a precolumn that retains the water and can be backflushed.
Column becomes shorter after maintenance.	Adjust integration windows. Adjust the flow to get the same retention time. Replace the section that was cut-off with a similar section from a second column or use deactivated fused silica by using a suitable coupling.

Peaks not detected

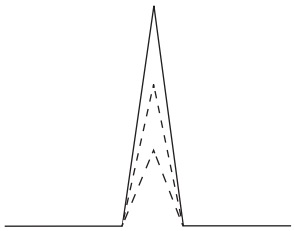
Main cause	Solution
Blocked syringe needle.	Replace the needle or the syringe.
The column is not correctly connected to the injection port and/or detector.	Check the insertion length of the column as recommended in the instruction manual for your GC.
Column flow blocked (by septum particles, graphite or pieces of wool).	Replace liner. Cut a 2 cm section from inlet, connect column at injector, set flow and check that the column has flow by dipping the column end in a vial with some acetone. Connect detector side and inject methane or a solvent to verify linear gas velocity.
Carrier gas leak and/or inadequate supply.	Check that there is a pressure in the injection port; Check for (big) leaks around the injection port and column connections. Also check that septum cap is sufficiently tightened. When there is a leak from the injection port septum, replace the septum. Check the flow rate is correct at the detector outlet and the column outlet.
Faulty detector.	Check the condition of the detector. Check for broken wire filaments with MS and TCD and check that the FID flame is lit. Check the cable connection between the detector and data acquisition system.
Detector temperature too low.	Detector temperature must be set at least 20 degrees higher then final temperature of the oven temperature program.

Abnormal peaks

All peaks are too small

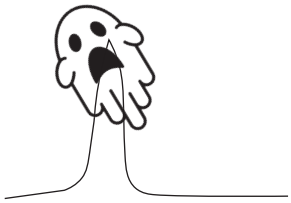
Main cause	Solution
Partial blockage of the syringe.	Wash and/or replace the syringe.
Wash and/or replace the syringe.	Check column position. Column inlet must be 4-8 mm above the bottom of the liner.
There is a leak at the injection port.	Check for leaks around the injection port. If the injection port septum is leaking, replace the septum.
The split ratio is too large.	Reduce the split ratio.
Leaking column coupling.	Replace column coupling.
The injection time is too short(When using splitless injection).	Check the splitless injection parameters and optimize the injection time. Typically injection times are 60-80 seconds.
There is an abnormality in the sample.	Check the sample concentration and stability etc. make sure the sample preparation and storage conditions are suitable.
The injection port temperature is too low (especially when analyzing high boiling point samples).	Check the injection port temperature and optimize it.
The detector is unsuitable for the component being analyzed.	Check whether the detector is compatible for detecting the analyte at required levels.
The detector sensitivity is to low (not optimized / contamination).	FID: Check the flows for H_2, air and make-up gas. Verify and adjust detector temperature. Clean detector according to manufacturers procedures.
Setting parameter error in the detector and/or integrator.	Check that the sensitivity setting of the detector is correct. Check that the attenuation setting is correct for the integration system used.

Poor repeatability of peak area and response



Main cause	Solution
Carrier gas leak and/or defective supply.	Check for leaks around the injection port. If the injection port septum is leaking, replace the septum. Check that the supply pressure is correct.
Discrimination, injection conditions are incorrect.	Select a liner and injection volume suitable for the injection method. If injecting manually, pay attention to discrimination. Consider to use a liner with some wool for best results. Check column position in the liner.
Faulty syringe, incorrect autosampler parameter setting.	The amount of sample picked up is not correct. Wash and/or replace the syringe. If the sample viscosity is high; reduce the syringe suction speed.
Contamination and/or deterioration of the liner.	Replace the liner. Depending on the type of sample and injection technique, select an appropriate liner with or without wool and type of wool material.
Split ratio is unstable.	If the split ratio is too small the split will not stabilize; adjust the split ratio to be suitable for the column and the GC. If short 0.53 mm columns are used, add a few meters of 0.25 mm ID deactivated fused silica as restriction tubing.
Faulty detector.	Check the detector is working correctly.

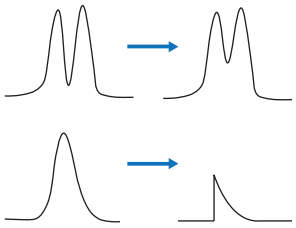
Ghost peaks: a peak appears where you do not expect a peak



Main cause	Solution
Not all peaks have eluted from previous run.	Wait until all peaks from previous run have eluted. To speed up, you may increase the oven temperature and/or flow.
Contamination of carrier gas.	Install or replace filtration system.
Trapped bleed products from septum. Siloxanes, phthalates from septa and septa particles deposited in the liner.	Replace liner; Use high temperature septa with a center guide; Use septum purge; Reduce injection port temperature. Consider a Merlin Seal septum.
The ghost peak is formed by a reaction in the liner.	Use liner without wool; Reduce liner temperature. Use PTV or on-column injection.
O-ring can produce siloxanes and triphenyl phosphine oxide (m/z = 277).	Replace O-ring; condition the liner for 16 hours at 300℃.
Contamination of sample by: sealing septum, micropipette tips, gloves or syringe.	If the syringe is contaminated, wash or replace it. If continually analyzing from the same vial, it is possible that sample will become contaminated by material that is stuck on the outside of the needle while moving through the seal. Work with larger volume in the vial which will dilute the contamination. When shaking vial, make sure that the liquid does not contact the seal of the vial. Micropipette tips can release eurucylamide.
Contamination of the injection port by sample matrix.	Replace the liner. Inject smaller amounts or dilute the sample. Consider using splitted injection.
Built up of matrix residues in the split line that diffuse back in the injection port during maintenance.	Disconnect the split line and rinse it with some solvents similar to what is used for the samples or replace the splitline.
There is a backflash: Injection volume is too large, so part of the sample is pushed back in the carrier gas line.	The injection volume, liner size, septum purge flow rate and injection port temperature may cause sample overflow from the liner and result in carryover (ghost peaks are teh same peaks as you analyze, but now are causing a bias). Select more suitable injection parameters. Check system cleanliness by running a blanc (only temperature programming, no injection).
Reaction product formed in activated part of the column in the (hot) detection port liner. Compound can react with the stationary phase creating a ghost peak. For example O_2 on a porous polymer PLOT column.	Without flow, hydrogen and air will be entering the column end creating high activity. Make sure that your column is always under positive flow. Only stop the flow when the detector is cooled down. To reduce or check impact, decrease the detector temperature. At lower temerature the ghost peak area should decrease.

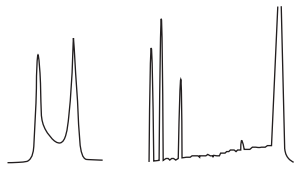
Abnormal peaks

Decrease in column performance: peak broadening / peak tailing



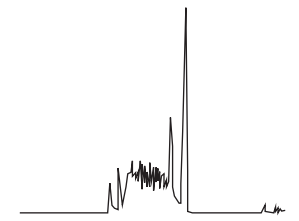
Main cause	Solution
Carrier gas purity is not sufficient.(Oxygen and moisture present in the carrier gas).	The column (liquid phase) will deteriorate rapidly with the presence of water and oxygen and with increased temperature. Install a moisture removal filter and an oxygen removal filter in the carrier gas line. Position the filtration device as close as possible to the GC. It filters are already installed, replace the filters.
There is a leak in the system where air can enter the carrier gas.	Do leak test with digital leak detection device. Check all connections including the septum.
Contamination of column inlet.	Cut-off about 50 cm from the injection port side of the column. If still not OK, cut another 50 cm. If not OK, connect detector side to injector and recondition the column for several hours at its maximum operation temperature. If not OK, replace the column. If the decrease in performance happens too fast, consider to do more sample cleanup or use guard- or precolumns.
A sample which causes chemical damage to the column has been injected.	Avoid injecting samples which contain inorganic bases (KOH, NaOH etc.), inorganic acids (HCl, HNO3, and HF etc.), perfluoro acetic acid (CF3COOH, C2F5COOH etc.) and salt. These classes of compounds cause chemical damage to the column and the liner.
Insufficient sample cleanup.	If the sample contains a matrix which may adversely affect the column's liquid phase, more or better sample pretreatment should be done before injection.
The column has been subjected to temperatures above the recommended maximum operation temperature.	Pay close attention to the final temperature of the column oven program and do not exceed the maximum temperature of the column.
The column has been subjected to higher temperatures without carrier gas flow.	Check flow settings. Make sure there is sufficient carrier gas available.
Peakbroadening due to depolymerization of the stationary phase at the inlet side.	Cut-off about 50 cm from the injection port side of the column. If still not OK, cut another 50 cm. If not OK, connect detector side to injector and recondition the column for several hours at its maximum operation temperature. If not OK, replace the column. If the decrease in performance happens too fast, consider to do more sample cleanup or use guard- or precolumns.

"Batman" peaks or "low-slope" leading peaks



Main cause	Solution
Component decomposes, changes configuration (cis to trans or trans to cis) or reacts while moving through the column. New product formed has a different retention time.	Reduce elution temperature by using higher flow rate or flow programming. Use slower temperature programming. Use shorter columns, larger ID. Thinnest possible film. Choose phases with lowest retention or a combination of these actions.

A cluster of peaks elute before the main component



Main cause	Solution
Formation of multiple injection bands during splitless injection caused by mismatch of polarity of solvent and stationary phase. As a result droplets are formed which move into the column and form multiple injection bands.	Choose splitted injection. Choose matching solvent with phase(polar solvent, polar phase, non-polar solvent, non-polar phase). Use correct polarity retention gap. Add a co-solvent to reduce droplet formation (toluene). Use a longer retention gap and make sure that the droplets do not reach the analytical column. Start at a higher oventemperature.

GC Troubleshooting Guide

Abnormal baseline

Column bleeding or backgrounds is very high

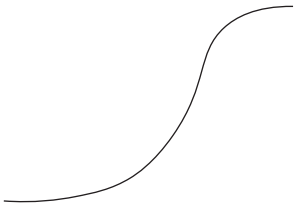
Spikes

Noise

Instability / oscillation of baseline / many ghost peaks at same distance

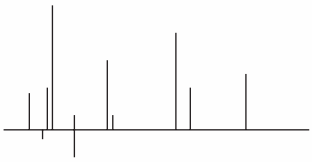
Abnormal baseline

Column bleeding or backgrounds is very high



Main cause	Solution
Column conditioning is too short.	Perform column conditioning using the procedure detailed in the instruction manual supplied with the capillary column, or condition the column for several hours 20 degrees higher then used for your application. Do not exceed the maximum temperature.
Carrier gas contains traces of air (water and/or oxygen) causing stationary phase to degrade.	Check gas purity, filtration and check all fittings and septum for leaks.
Normal ageing of the column by running applications.	Injecting smaller amounts will slow down bleed formation and will increase life time.
Sample contains small amounts of high boiling material which is not eluting during the application.	Best way is to reverse the column and connect detectorside to Inlet. Connect inlet site to detector and condition the column at its maximum temperature to clean it. Keep the column at this temerature until the baseline is horizontal. Now reinstall the column again in original configuration. This procedure can be repeated periodically, but depends how much high boiling material is injected.
Contamination in the detector.	Clean the detector. Consult your instrument manual.
High bleed by using thick-film columns.	Bleed is a direct function of the film thickness. If retention of components allows, you can choose for a thinner film; Also the flow can be increased for late eluters to make components elute at lower temperatures and the final temperature of the oven program can be reduced. Roughly a 4 times higher flow results in approximate 30 degrees lower elution temperature. As for most GC liquid phases, the column bleed is not depending on the flow, 30 degrees lower elution temperature results in about 4 times lower bleed.
Liner is contaminated.	Replace liner.
Splitline is contaminated.	Clean or replace splitline. Don't forget to replace the splitline filter.
Septum is bleeding too much.	Choose better septum. Check for leaks. Use septum purge. Consider using a Merlin seal.

Spikes



Main cause	Solution
Column connection (detector side) is incorrect.	A spike may occur when the column is inserted too far into the detection port liner. Check the insertion length of the column as recommended in the GC instruction manual.
Contamination of the detector by silicium oxide.	Clean the detector. For FID, replace flame tip.
Faulty signal cable.	Check the cable is securely connected and the insulation is intact. Replace if necessary.
Abnormality in the power source cable and/or power supply.	Ensure the power supply is stable.
Particle elution from a PLOT column.	Use a particle trap. Connect the PLOT column with a 1-2 m length of silicone coated capillary. The particles with be cought by the silicone coating.

Abnormal baseline

Noise

Main cause	Solution
High background caused by contamination in the detector.	Clean the detector. Set detector temperature 20℃ higher then final oventemepature used for the application.
High backgrounds caused by contamination on the column.	Cut-off about 50 cm from the injection port side of the column. If still not OK, cut another 50 cm. If not OK, connect detector side to injector and recondition the column for several hours at its maximum operation temperature. If not OK, replace the column. If the decrease in performance happens too fast, consider to do more sample cleanup or use guard- or pre-columns.
Contamination of the injection port.	Due to contamination in the liner by residual material and/or sample on the injection port septum. Replace the liner. Replace the injection port septum.
Column connection (detector side) is incorrect.	Check for leaks. Check the insertion length of the column as recommended in the GC instruction manual.
High bleed caused by too high oventemperature for the column used or by presence of water / oxygen in the carrier gas.	Reduce final oven temperature. Use a column with a thinner film; Use a column with higher temperature stability. Check filtration system, replace filters.
A leak of the carrier gas (MS, FID, TCD, PDD and ECD).	Check for a leak and fix it.
Detector gases are not clean enough.	Check gas filtration for all gases used for the detector. Use charcoal filters.
Detector cable not correctly connected.	Check connection. replace cable.
Faulty detector.	Faulty filament, electron multiplier, amplifier or baseplate etc. Repair or replace the faulty component.
Electronic noise picked up from other instrument (or vacuum cleaner).	Check if noise is happening in certain times of the day. If so, check impact systematically by turning off instruments close to the GC.

Instability / oscillation of baseline / many ghost peaks at same distance

Main cause	Solution
Column conditioning is insufficient.	Perform the column conditioning procedure.
Contamination is present in the whole column.	Perform the column conditioning procedure. Cut-off about 50 cm from the injection port side of the column. If still not OK, cut another 50 cm. If not OK, connect detector side to injector and recondition the column for several hours at its maximum operation temperature. If not OK, replace the column. If the decrease in performance happens too fast, consider to do more sample cleanup or use guard- or pre-columns.
Contamination of the injection port.	Contamination in the liner, the carrier gas line or the split line with residual material and/or sample on the injection port septum. Wash or exchange the liner. Clean the gas lines. Replace the injection port septum.
Condensation of stationary phase bleed products on one side of the column during oven cooling down. Typical phenomena with high bleed phases.	Use negative cooling down program for the oven, -10 or -20℃/min until a temperature where the column bleed is low, then cooling down can be done faster.
The detector temperature is unstable.	This may occur when using a TCD. Check the temperature stability.
The flow rate regulation of the detector gases are unstable.	Check the flow rates.
The temperature environment around the instrument is not suitable.	Check the temperature environment and make sure it is within the recommended specification for the instrument.

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