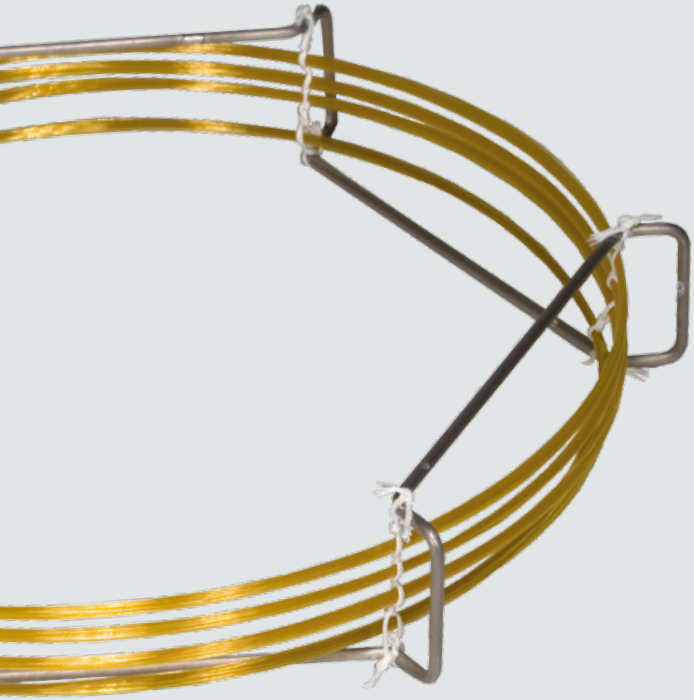

Manufactured by MACHEREY-NAGEL

OPTIMA®
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Since 1911

User Manual

GC

Fused silica capillary column

MACHEREY-NAGEL

MACHEREY-NAGEL

Fused Silica Kapillarsäulen für die GC · Einbau- und Gebrauchsanleitung

Fused silica capillary columns for GC · Installation and operating instructions

Please read these instructions BEFORE installing – and certainly before heating – your column!

The present capillary column from MACHEREY-NAGEL has been manufactured in our own facilities and will be the heart of your gas chromatographic analytical system. It has been produced following state-of-the-art technologies and has been individually tested; the enclosed certificate documents its quality standard.

In order to obtain good results from your column as long as possible, you should carefully read and follow these instructions. We have grouped them into 4 chapters:

I Column installation

II Start-up and testing column and chromatograph

III General information for handling

IV Technical support and customer services

Important note: *Do not remove the cords from the capillary column! The cords attach the column to the cage and prevent unnecessary contact between the column and metal parts of the cage. The cords are temperature stable.*

Caution: *Without carrier gas flow at high temperatures any stationary phase in a capillary column will be destroyed in no time!*

I. Column installation

The following items are required for proper column installation:

- Ferrules (with hole appropriate for the respective column outer diameter)

Column ID [mm]	Column OD [mm] (= bore of ferrule)
0.05–0.20	0.40
0.25	0.40
0.32	0.50
0.53	0.80

For GC instruments of Agilent special ferrules are available – please see our website ***www.mn-net.com*** or our catalog Chromatography.

- Diamond file (e.g., REF 708300)
- Magnifying lens (e.g., REF 706296)
- Vial with volatile solvent (e.g., pentane, cyclohexane, acetone)
- Leak detector or leak check liquid
- Flow meter

Now you are ready for installation:

Caution: *Avoid sharp bends. The column should be installed in an unstrained configuration. A column hanger should only contact the cage. For coupling of columns use only zero dead volume fittings or devices to avoid peak broadening, tailing or even memory effects.*

a) Open the plastic packing (cut – not tear) and remove the column.

b) Check the column for mechanical transport damage; there must be no free ends visible (ends not melted)! If this should be the case, return the column to your distributor for replacement.

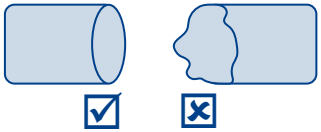
c) The melted column ends have to be removed. For this purpose, score the polyimide layer about 5 cm from the melted end using a diamond file and break the end off straight. Make sure that no column fragments get into the column. For this purpose it is advisable to take off the removed end in a downward movement.

d) Unwind both column ends from the cage for about one turn.

e) Suspend the column in the middle of the oven.

f) Place a column nut and a ferrule over each column end, then cut about 5 cm from each column end using a diamond file (in order to remove any ferrule particles from the column).

g) Examine the column ends with a magnifying lens (REF 706296). If the column ends are not smooth or even show strong burrs, cut off another piece from the column.



h) Consult the manual of your GC instrumentation because it is very important to install the column entrance at the correct insertion distance in the injector. Insert one column end into the injector and attach it according to the instructions of the instrument manufacturer, i.e., fix it only slightly more than finger-tight.

i) Adjust the flow of the carrier gas through the injector according to the instrument manual, the analytical instructions or the average values of the following table:

ID [mm]	Length [m]	Pressure [bar]	Approximate dead time [min]
0.05	10	2.5 (H2)	1.5
	20	4.5 (H2)	3
0.20	12	0.8 (He)	1.0
	25	1.5 (He)	1.8
	50	3.0 (H2)	3.0
0.25	10	0.3 (He)	1.0
	15	0.4 (He)	1.2
	25	0.7 (He)	2.5
	30	0.8 (He)	2.8
	50	1.5 (He)	5
	60	1.8 (He)	5.5
0.32	10	0.3 (He)	0.8
	15	0.4 (He)	0.9
	25	0.5 (He)	1.4
	30	0.6 (He)	1.6
	50	1.0 (He)	3.5
	60	1.2 (He)	4.0
0.53	10	0.2 (He)	0.6
	15	0.2 (He)	0.7
	25	0.3 (He)	1.0
	30	0.4 (He)	1.1
	50	1.0 (H2)	1.5

j) Place the free end of the capillary column into a vial with a volatile solvent. After not more than 30 s gas bubbles should indicate a steady flow through the column. If no bubbles appear, repeat steps f) – i). If you still don't see a gas flow check the following:

- the supply of carrier gas
- the pressure of the carrier gas in the supply pipes (pressure regulator, valves)
- if the capillary column is broken

k) Insert the second column end into the detector and attach it according to the instructions of the instrument manufacturer, i.e., fix the nut slightly. Exception: If using highly sensitive detectors (MS, ECD) you should first condition the column (see below) and then connect it to the detector.

Hint: *For subsequent column installations you may mark the installation depth of the column in the injector and detector in the gas chromatograph using a moisture-proof felt tip pen (e.g., on the door or the edge of the oven).*

l) Check the column connections in the oven for leaks (e.g., with leak check liquid; this is especially important when using hydrogen as carrier gas)!

m) Close the oven door, start the gas chromatograph:

- Heat the injector and the detector (maximum to the long-term temperature limit of the capillary column) and ignite, if required (e.g., FID).
- Heat the oven (choose a temperature between 60 and 100 °C).
- Start the integrator or the data processing system.
- Adjust the split ratio (typical 1:30).
- Inject methane or a volatile solvent to check the overall performance of the gas-chromatographic system.

n) Before first usage and as required, condition the column, (see chap. II.1).

o) Possibly reproduce the column test from certificate (see chap. II.2) – now the column is ready for use.

II. Start-up and testing column and chromatograph

1. Conditioning

Each column has been conditioned prior to its quality control. Conditioning is used to elute volatile components of sample molecules and stationary phase and to remove active sites of the inner column surface.

Conditioning should always be performed after a longer storage of the column and performed until the baseline is stable (usually approx. 4 h).

The two numerical temperature values mentioned in the certificate indicate the (lower) isothermal long-term temperature (= conditioning temperature) and the (higher) short-term temperature (= maximum in temperature gradients during 5–15 min).

During conditioning never exceed the long-term temperature, and make sure that the gas supply is never interrupted.

2. Column test

Each column from MACHEREY-NAGEL is tested in a gas chromatograph, and the individual chromatogram with conditions (the test certificate) is supplied with the column. It should be retained for future reference.

If possible you should reproduce the included test chromatogram before using the column for your analytical tasks. The respective test mixture can be separately purchased (see ***www.mn-net.com***).

This will indicate whether

- column installation is correct
- samples are properly injected
- the overall chromatographic system including evaluation works accurately.

By reproducing the column test you document the performance of the column, and thus you can at any time compare the state of the column with the initial quality.

Using the column test, you can identify a number of typical failures; this is the reason why reproduction of this test should always be the first step in troubleshooting.

If the test result is satisfactory, the planned application can be operated.

3. Checking column installation

What should you do when your own test does not show the same result as the test certificate?

a) Check the correct installation of the column and test again.

b) Compare the faulty chromatogram with the examples in our troubleshooting and try to correct the problem.

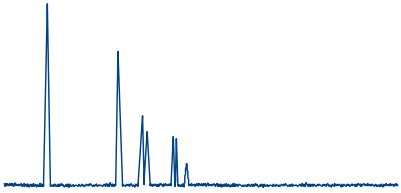
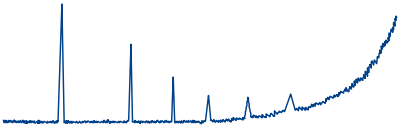
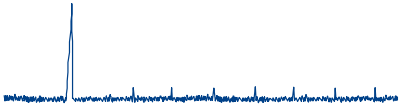
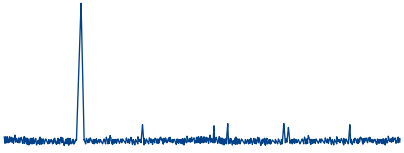
c) Contact your nearest MACHEREY-NAGEL office or your local distributor and ask for help.

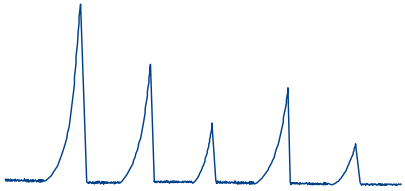
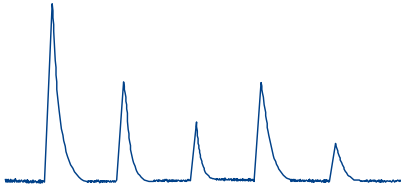
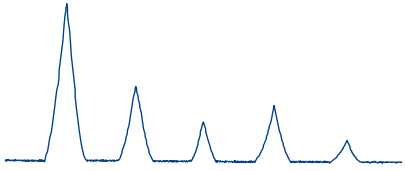
4. Troubleshooting

If you cannot reproduce the test chromatogram of your capillary column, this may be due to one of the following reasons (see page 11 and 12).

For example, a dead volume in the injector produces tailing. This is especially strong for early eluting components in a temperature program. Dead volume in the detector also results in tailing, however, in this case all components are affected.

Fused silica capillary column for GC

Observation	*Possible cause*	*Suggested remedy*
Shortcoming A  **Missing or overlapping peaks**	Sample too diluted	Increase injection volume, concentrate sample
	Column connection leaks, column not properly installed	Check for leaks, change ferrules, reinstall column
	Injector temperature too low	Check temperature program, increase injector temperature
	Sample decomposes in the injector	Check temperature program, reduce injector temperature, change liner, check capillary ends
	Column adsorbs substances or decomposes them	Check capillary ends, check intact deactivation using a suitable test mixture; for poor results shorten both column ends by about 10 cm; or replace column
		If the column test does not show any defects: a) use a column with thicker film; b) use alternative phase
Shortcoming B  **Increasing baseline, at high temperatures bleeding, strong noise**	Septum particles in the column entrance	Cut off one turn from the column entrance, change injection septa
	Heating program too fast, final temperature too high	Decrease temperature gradient and final temperature
	Column bleeding due to incomplete column conditioning	Disconnect detector and condition column (also see instructions for your GC instrumentation and detector)
	Soiled detector	Clean detector according to manufacturers instructions
Shortcoming C  **Regular interfering peaks**	Bleeding of silicone septa	Use new septum or septum grade with lower bleeding (high temperature septum)
Shortcoming D  **Irregular interfering peaks / spikes / ghost peaks**	Impurities from sample vials and / or sample preparation	Check sample preparation and autosampler vials, use septa with lower bleeding
	Dirty syringe	Use clean syringe
	Sample is decomposed	Check temperature program, oven temperature (thermometer); when chromatographing thermolabile compounds, reduce injector temperature, change liner
	Column adsorbs substances or decomposes them	Check capillary ends, check intact deactivation using a suitable test mixture; for poor results shorten both column ends by about 10 cm; or replace column
		If the column test does not show any defects: a) use a column with thicker film; b) use alternative phase
	Bleeding of butyl septa	Use silicone septa for injector and sample vials

Observation	*Possible cause*	*Suggested remedy*
Shortcoming E	Column overload	Reduce injection volume
	Sample is decomposed	Check temperature program, oven temperature (thermometer); when chromatographing thermolabile compounds, reduce injector temperature, change liner
	Column adsorbs substances or decomposes them	Check capillary ends, check intact deactivation using a suitable test mixture; for poor results shorten both column ends by about 10 cm; or replace column
Fronting, heavy peak broadening of the peak front		If the column test does not show any defects: a) use a column with thicker film; b) use alternative phase
Shortcoming F	Sample with high boiling point	Derivatize polar, basic or high-boiling samples
	System leaks	Check for proper column installation and leaks, change ferrules
	Sample is decomposed	Check temperature program, oven temperature (thermometer); when chromatographing thermolabile compounds, reduce injector temperature, change liner
	Column adsorbs substances or decomposes them	Check capillary ends, check intact deactivation using a suitable test mixture; for poor results shorten both column ends by about 10 cm; or replace column
Tailing, heavy peak broadening of the peak back		If the column test does not show any defects: a) use a column with thicker film; b) use alternative phase
	Compounds which always show strong tailing	No remedy possible
Shortcoming G	Split flow too low	Increase split flow
	Column overload	Inject less or increase split flow
Broad peaks		

III. General information for handling

1. Column regeneration

If contamination is the cause of a loss in column performance (compare shortcoming D) you may try one of the following remedies:

a) Remove the contaminated end from the column, i.e. in general shorten the injector column end by about 50 cm (at least one column turn).

b) Remove column end in the transfer line of the detector, because here phase deterioration may be faster than in the rest of the column due to the permanent high temperatures.

c) You may also try to ”flood“ the column in the GC using 20 µL acetone followed by a normal heating program.

d) The column can be rinsed *(only chemically bonded phases)*. For this purpose disconnect the capillary column from the gas chromatograph and connect the detector end (mark it!) to the rinsing device. For rinsing use 10 mL pentane, acetone or toluene for nonpolar – medium polar phases or dichloromethane for polar phases. After rinsing dry the stationary phase for at least 2 h in a stream of carrier gas at ambient temperature before you install the column in the GC and heat it in a temperature program (1 °C/min) up to the maximum isothermal temperature. Hold this temperature for about 1 h.

2. Column storage

If you want the capillary column to stay in the GC over night we recommend to reduce the split flow and adjust the oven temperature to 80–100 °C.

If you have to turn off the carrier gas or if it is necessary to change the column, the oven should be cooled to ambient temperature before disconnecting the column. In order to prevent oxygen damaging of the stationary phase the column should be stored with sealed column ends *(especially important for Wax and FFAP columns)*. Fused silica columns can be easily sealed by sticking the ends (sideways) into a septum. For long-term storage we recommend flame sealing and storage in the original shipping container.

3. Maintenance of value

The column at hand consists of a tube of fused silica manufactured with high precision and coated with a protective polyimide layer which results in a high column flexibility.

Caution: *It is very important to avoid damage to the polyimide layer, since unprotected fused silica becomes brittle and thus easily breaks.*

The column is coiled onto a cage which largely protects it from mechanical damage. Avoiding mechanical or extreme thermal strain (e.g., opening the oven door at high temperatures!).

In addition, you can protect your column from chemical stress by performing a suitable sample preparation, by controlling the pH value of your sample (neither acid nor basic) and by using a precolumn. If matrix problems occur, a change in sample preparation can considerably extend the lifetime of the column.

We recommend that you use CHROMAFIL® syringe filters for sample clarification, CHROMABOND® SPE columns for selective sample preparation and / or deactivated capillary columns (precolumns) to protect the separation column from contamination. If necessary 0.5–1 m (always at least one column turn) of the guard column can be removed without need to shorten the analytical column.

IV. Technical support and customer services

If you have any questions concerning the use of capillary columns please contact us:

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We will be glad to help you with:

- Technical support by phone
- Technical documentation as comprehensive collections of applications for our products
- Local technical support (e.g., by your local distributor)
- Our extensive internet collection of applications for GC, HPLC, TLC, SPE
- Visit our chromatography pages: ***www.mn-net.com***

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This warranty does not cover defects in or damage to the products which are due to improper installation or maintenance, misuse, neglect or any use other than ordinary commercial application.

Any discharge from liability will be void if a defect results from a heavily negligent or intentional breach of contract or if the buyer will be bodily injured as a result from a negligent or intentional breach.

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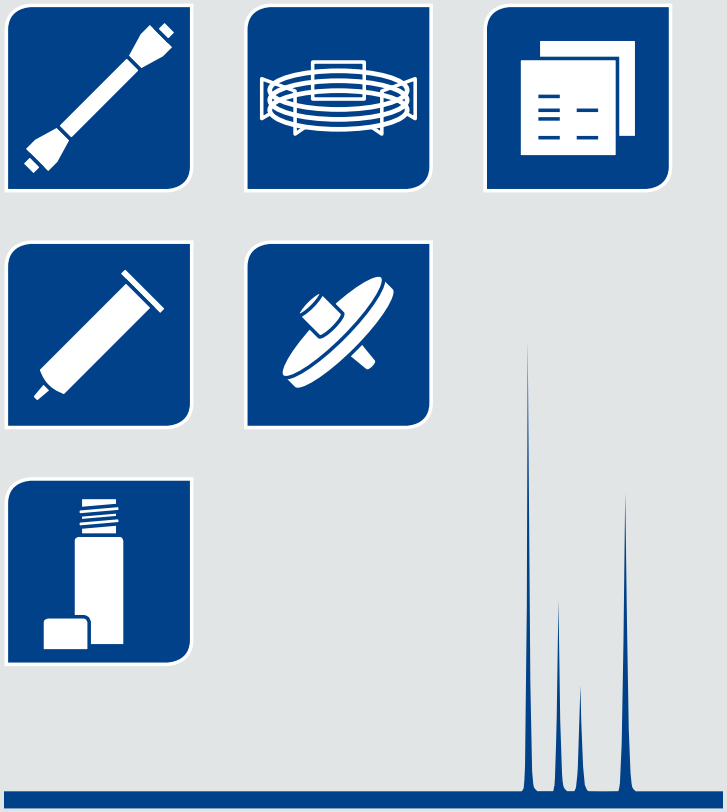
If not superseded by these terms the terms and conditions for the sale of goods (export version) are additionally valid.



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