

CHIRALDEX™ GC Column Care & Use

Maximum Allowable Operating Temperatures (MAOTs)

Columns that have been used at high temperatures (>180 °C) (except PM) for extended periods of time lose their efficiency when used at low temperatures, even though their performance remains constant at high temperatures. Consequently, if one is interested in reading enantiomers at temperatures >180 °C, we recommended that a column be dedicated for that purpose.

Phase	MAOT °C Isothermal	MAOT °C Programmed
TA	180	180
PN	200	220
BP	200	220
DP	200	220
DM	200	220
PM	200	220
DA	200	220
PH	200	220

Optimal Temperatures

Enantiomeric selectivities can be dramatically enhanced with small temperature reductions at temperatures below 150 °C. Above 150 °C, this temperature effect is less pronounced. Increasing carrier gas linear velocities (50 cm/sec.) is often more useful than increasing column temperatures to affect retention and resolution. To this end, hydrogen and helium are the best carrier gas choices.

The most selective temperature range for the PH and DA series of CHIRALDEX columns is -5 to 200 °C. In no event should these columns be heated above 220 °C. For the TA, PN, and BP series, the range is from -5 to 170 °C and never higher than 190 °C.

Temperature Ramp Rates

CHIRALDEX stationary phases are sensitive to thermal shock. Never heat or cool the column at more than 15 °C/min.

Sample Preparation

The capacity of chiral capillary columns is very low, so samples should be sufficiently pure to protect the stationary phase. **For the TA series, it is especially important to have the sample free of moisture.** Methylene chloride extracts of aqueous samples contain >100 ppm water, sufficient to cause the hydrolysis of the TA. Evaporation to near dryness in the presence of dimethoxypropane will adequately dry the sample. An additional step for routine analysis that can protect the TA columns would be to use a retention gap (methylphenyl) as a guard column. In addition to picking up residual moisture, the retention gap will protect the TA column from the high temperature of the injector. Typically 5 meters is used. If the TA column has been hydrolyzed, it may be regenerated using the regeneration procedures. This procedure can be obtained by contacting Supelco Technical Service at 814-359-3041 or techservice@sial.com. Solvents routinely used to dissolve or dilute samples are: ethyl ether, hexane, methylene chloride, methanol, and ethanol.

Derivatization Effects

Enantiomeric separation data for a variety of acyl derivatized analytes indicates there is an enantioselective dependency on the type of acylation reagent used. The acyl derivative can play an important role in the enantioselective mechanism. In many cases, changing the acyl derivative provides resolution. The optimal acyl derivative on one CHIRALDEX column may not be the optimal derivative on another column. For compounds that require derivatization, it is prudent to prepare a variety of derivatives.

Injection Technique

CHIRALDEX stationary phases are not bonded and can only be run in a split mode unless a retention gap is used. Make sure the splitter flow is on and the split ratio is $\geq 30:1$ before injection. Splitless or on-column injections will permanently damage CHIRALDEX capillary columns (without the use of a retention gap).

Detection

All capillary columns have extremely low capacities. To obtain highest column efficiency and chiral selectivity, set the FID detector at the highest usable sensitivity and inject the lowest amount of sample (e.g. 0.2-4.0 μ L of 1 mg/mL solution with a split ratio of 100:1).

Storage

CHIRALDEX columns must be protected from moisture during storage. With carrier gas on, heat the column in a GC to 150 °C for 30-60 minutes. This will remove residual moisture from the column. Flame seal one end, pull a vacuum on the other end (5-10 min.) and flame seal. By flame sealing the column ends under vacuum, column life will be extended, particularly in the TA series.

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