



HIGH TEMPERATURE HPLC USING STATIONARY PHASES BASED ON DIVINYLBENZENE (DVB)

INTRODUCTION

Instrumentation for performing temperature programmed HPLC utilizing temperatures up to 200°C is now possible with the Polaratherm™ Total Temperature Controller, but traditional silica columns cannot withstand the elevated temperatures or extremes in pH that are often required to optimize a separation. Stationary phases based on rigid polymeric spheres of divinylbenzene (DVB) can be used from pH 0-14. Because of their superior pH stability, these stationary phases should also have enhanced temperature stability. Three DVB column packings were evaluated for high temperature HPLC use.

EXPERIMENTAL

Three different stationary phases were evaluated. They are summarized in Table 1. Columns were flushed with 1,000 column volumes of 50:50 Acetonitrile:water starting at 100°C, increasing the temperature by 20°C increments until evidence of stationary phase degradation was observed. HPLC column evaluation conditions are summarized in Table 2. The columns were cooled to 50°C and evaluated with a three component test mix consisting of uracil, phenol, and benzene between column flushings at high temperature.

Table 1.
Jordi Columns Evaluated

Column	Packing	Particle Size (micron)	Pore Size (Angstroms)	Dimensions (mm)
DVB	DVB divinylbenzene	5	300	100 x 4.6
C ₁₈	DVB bonded with C ₁₈	5	500	100 x 4.6
Hydroxylated (OH)	Hydroxylated DVB	5	500	100 x 4.6

Table 2 HPLC conditions for Jordi Column Evaluation	
Columns:	See Table 1
Mobile Phase:	50:50 Acetonitrile:Water, isocratic
Flow:	1.0
Detection:	UV @ 254 nm
Injection:	5.0 uL
Temperature:	50°C

RESULTS

Figures 1-3 show the effect of temperature on theoretical plates. After being exposed to 1,000 column volumes of mobile phase at 160°C, all three columns showed a loss of theoretical plates. This was accompanied by longer retention times, and higher back pressure. These are all indicators of stationary phase degradation. Further work indicated that the columns could be used without evidence of degradation of the column packing at temperatures up to 150°C.

CONCLUSIONS

The three Jordi FLP columns based on DVB appeared to be stable to 140°C, but showed a loss of theoretical plates at 160°C (see Figures 1-3). Additional experiments indicated that the columns could be used up to 150°C.

ACKNOWLEDGEMENT

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Figures 1-3. The effect of temperature on efficiency for the three Jordi columns evaluated.

Figure 1.

Effect of Temperature on Efficiency of Jordi DVB Column

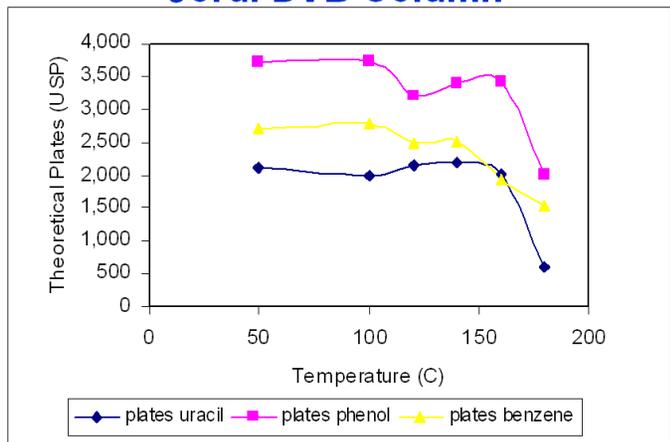


Figure 2.

Effect of Temperature on Efficiency of Jordi DVB C₁₈ Column

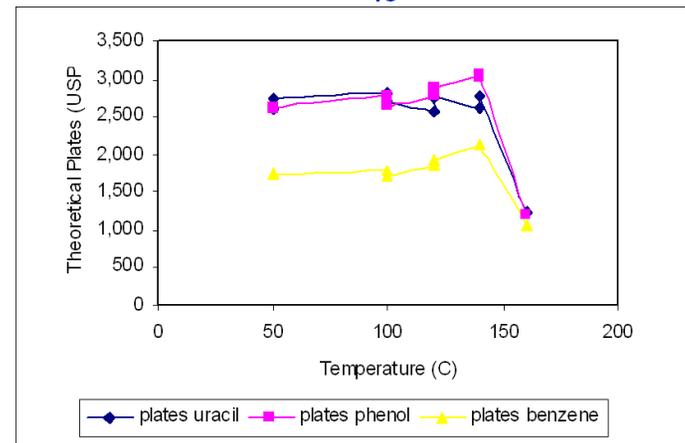


Figure 3.

Effect of Temperature on Efficiency of Jordi DVB OH Column

