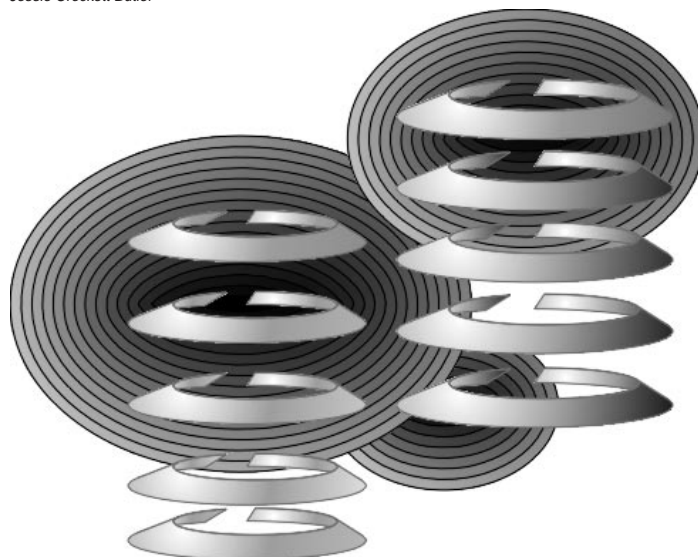


TR 9122

Product Analysis
GC Technical Report No. 9122

Simplified Design of a Temperature Programmable Multi-mode Injection GC Inlet System

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Introduction

A new Temperature Programmable Inlet (TPI) has been designed for capillary gas chromatography (GC). The Finnigan TPI is a universal inlet that may be used as a conventional split/splitless injector, large volume injector for trace analysis, cool on-column inlet for thermally labile compounds, and temperature program-mable inlet for high temperature analyses. The inlet is very simple to maintain with over a dozen application-specific liners. The only internal piece to the injector is the liner, *Figure 1*. All liners may be replaced from the top of the inlet by simply removing the septum nut, septum, and septum purge insert disc. The absence of sealing o-rings and internal graphite ferrules facilitates the replacement of the liner. The temperature of the inlet is electronically controlled through the microprocessor of the GC, opening a solenoid valve delivering a cooling gas of either CO₂ or air, *Figure 2*. The TPI utilizes a wound wire heating element to deliver uniform heating throughout the flash zone. The inlet may be operated isothermally or temperature programmed with up to four ramps and five levels. Several tests were performed to evaluate the performance of the injector in the following modes of operation: cool on-column, temperature programmed, large volume and conventional splitless.

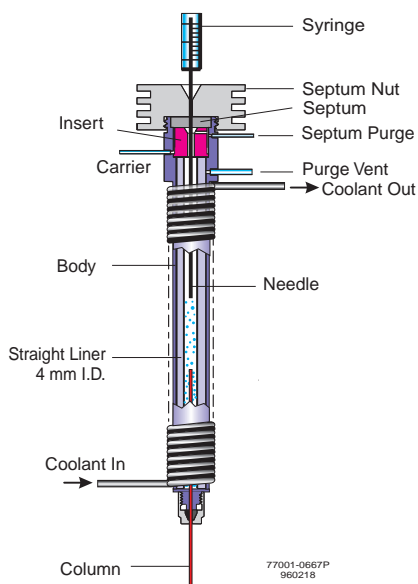


Figure 1. Temperature programmable capillary inlet

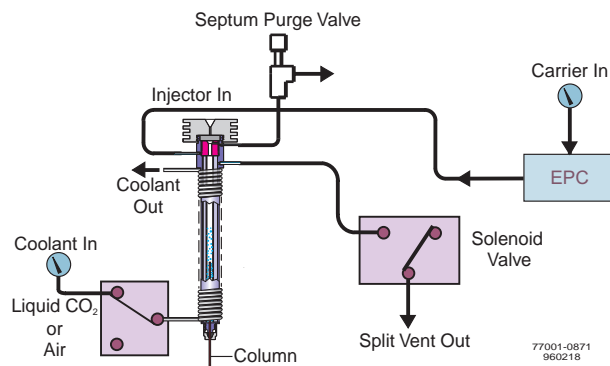


Figure 2. TPI inject mode

Cool On-Column Injection

In a cool on-column injection the sample is deposited inside the column using a unique liner, *Figure 3*. After the sample enters the column, the inlet is programmed to vaporize the compound for analysis without exposing it to the shock of a hot flash off-column injection. Thermally labile compounds usually break down in a standard hot flash injection before reaching the column. A 1 µL injection of the SW 846 Method 8270 check mix solution at 50 nanograms/µL was made by hot flash off-column injection and cool on-column injection. The TPI was run isothermally at 300°C for the flash injection and programmed for the on-column injection from 30°C to 300°C.

Experimental

Finnigan 9001 GC

TPI: Cool on-column: 30°C, 1 min; 10°C/min; 300°C, 2 min

TPI: Hot flash: 300°C

Liner: Uniliner

Guard Column: 1 meter phenylmethyl deactivated

Column: XTI-5, 0.53 mm x 30m, 1.0 µm film

Column flow EPC: 13.7 psi, 1 min; 1.05 psi/min; 40.75 psi, 2 min

FID: 320°C

Hydrogen: 18 mL/min

Air: 180 mL/min

Make up: 30 mL/min helium

AS 800 Autosampler

LabQuest Data System: 0-10 Volt

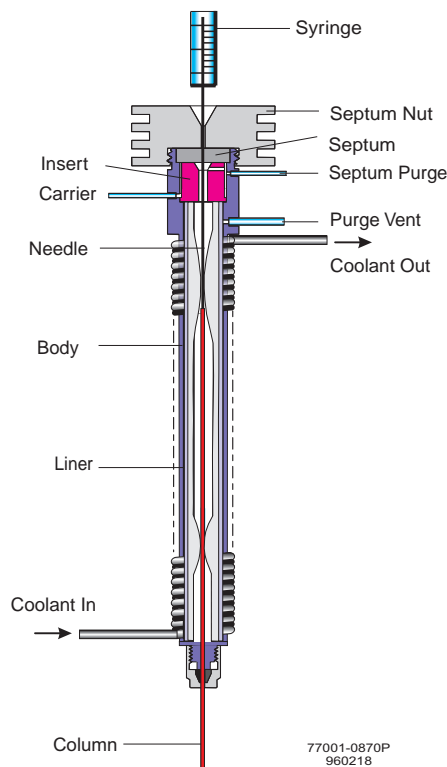


Figure 3. TPI on-column injection

Results

The cool on-column injection yielded increases in responses of 40%, 15%, 25% and 34% respectively for N-nitrosodi-n-propylamine, hexachlorocyclopentadiene, 2, 4-dinitrophenol, and 4-nitrophenol as illustrated in the bar graph in *Figure 4*. Some peak broadening was observed for N-nitrosodi-n-propylamine in the on-column injection, but the area counts were still higher than in the hot flash injection, *Figure 5*.

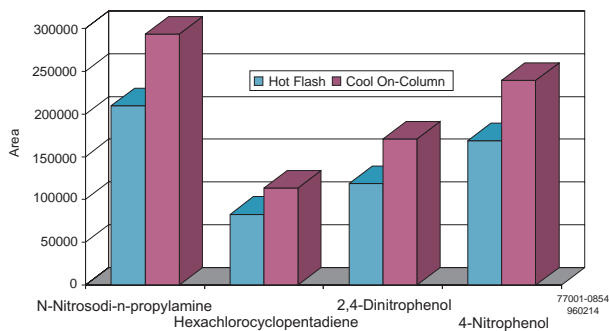


Figure 4. Enhanced response for semi-volatile performance check solution with cool on-column injection (FID)

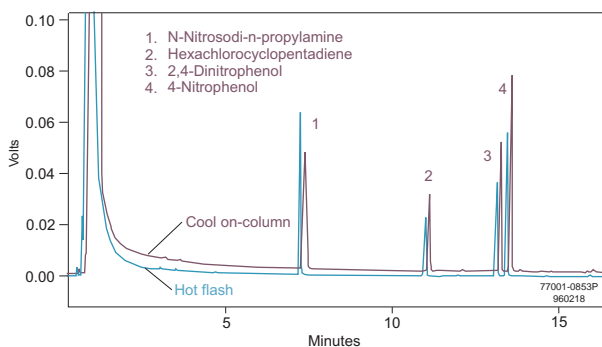


Figure 5. On-column and flash off-column megabore capillary analyses of performance check solution. (FID)

Temperature Programmed Injection of Waxes

The high temperature waxes C_9H_{20} through $C_{44}H_{90}$ were evaluated with the TPI in the split mode to check for discrimination. Two split injections were made, one at an isothermal temperature of 400°C and the other by a temperature programmed injection from 30°C to 400°C with the syringe directly contacting the glass wool in the liner.

Experimental

9001 Finnigan GC
 TPI: 30°C, 1 min; 3°C/sec; 400°C
 Liner: 4 mm straight with glass wool
 Column: SGE HT5, 0.25mm x 30m, 0.1 µm film
 Column flow EPC: 40 cm/sec constant flow
 Split: 100:1
 FID: 420°C
 Hydrogen: 18 mL/min
 Air: 180 mL/min
 Make up flow: 30 mL/min helium
 AS 800 Autosampler
 LabQuest Data System: 0-10 Volt

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Results

A temperature programmed injection gave better sample delivery of the hydrocarbon waxes than a conventional isothermal injection. High mass discrimination was seen in the hot flash injection but not observed in the temperature programmed injection. A chart of relative responses of the cold and hot split injections shows a more uniform response for the cold split temperature programmed injection, *Figure 6*.

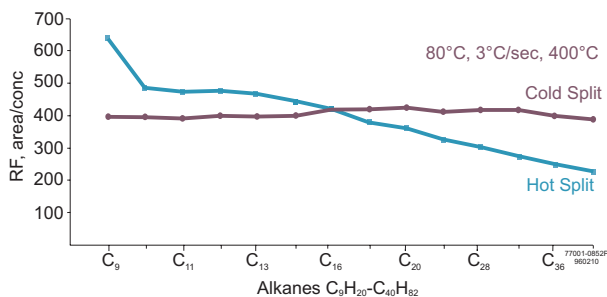


Figure 6. Uniform HC response on TPI in programmable mode

Large Volume Injections

A large volume injection of up to 500 µLs may be made with the TPI. The sample is injected into a 4 mm i.d. liner with glass wool. Holding the inlet temperature at the boiling point of the solvent but below that of the analytes, the compounds are trapped in the liner while the solvent is vented. After venting the bulk of the solvent, the purge vent valve is closed and the inlet is programmed to vaporize to the analytes into the column. Three injections of hydrocarbon solutions were made at 1 µL (100 ng/µL), 10 µL (10ng/µL) and 50 µL (2ng/µL) as shown in *Figure 7*. The 1 µL injection was made in the conventional splitless mode for comparison.

Experimental

9001 Finnigan GC
 TPI: 80°C, 2 min; 3°C/sec; 300°C, 15 min
 TPI splitless: 300°C
 Liner: 4 mm i.d. with glass wool
 Column: Rtx®-5, 0.53 mm x 15 m, 0.5 µm film
 Column Flow EPC: 80 cm/sec helium with 7.5 psi surge for 2 min
 FID: 320°C
 Hydrogen: 18 mL/min
 Air: 180 mL/min
 Make up: 30 mL/min helium
 AS 800 Autosampler
 LabQuest Data System: 0-10 Volt

Results

Consistent results were produced with varying injection volumes of 100 ng of the hydrocarbon solutions. Using the conventional splitless injection for comparison, the large volume injections gave uniform response for $C_{15}H_{32}$ through $C_{36}H_{74}$ with no loss of analyte.

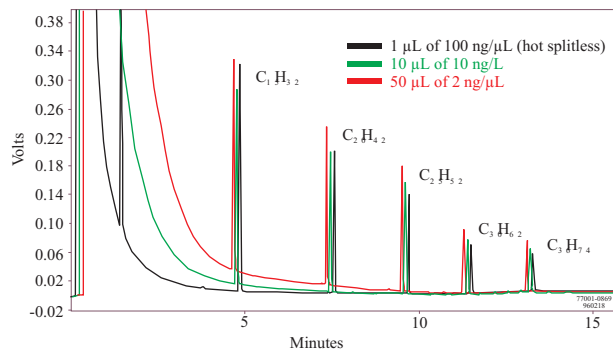


Figure 7. Large volume injection of hydrocarbons at 1, 10 and 50 μL

Conclusion

The Finnigan TPI is a simple universal injector for capillary chromatography. The chromatographer may now use the temperature of the inlet to optimize the sample delivery for lower detectability. In addition to performing the standard split/splitless isothermal injections, now both high boiling point compounds and thermally labile compounds may be successfully delivered into the column. The method detection limit may be lowered by two orders of magnitude by utilizing the large volume injection mode of the inlet. The TPI is a truly universal injector for the analysis of all volatile and semi-volatile compounds from the thermally labile nitrophenols to the high temperature waxes.

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