

Large Volume Injections with a Programmed Temperature Vaporizing Injector for Gas Chromatography

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Overview

The TRACE™ GC Programmed Temperature Vaporizing (PTV) injector is an extremely versatile injector for Gas Chromatography. The operating principle for PTV injection is simple. The sample is deposited into the inlet at a low temperature, then the inlet temperature is rapidly raised to vaporize the desired components and cause their transfer into the column. The PTV can operate effectively with Large Volume Injections allowing venting of solvent and backflushing of undesired compounds to vent. With the options of sub-ambient cooling and a heated solvent vapor exit valve, the PTV becomes one of the most versatile sample introduction devices for Gas Chromatography.

Introduction

Conventional vaporizing injectors are designed to maintain temperature stability with high mass injector bodies and heating blocks. They are normally operated at high temperature so that samples are immediately vaporized. Sample types that contain thermally labile compounds or a wide boiling range of compounds do not perform well in conventional Split/Splitless injectors. Because the PTV provides both variable and precise temperature control, discrimination of high boiling components or sample breakdown in the inlet can be greatly reduced.

In a Split/Splitless injector, the entire sample is vaporized and introduced onto the column at once. Large volumes of solvent cause column and detector overload, especially in the case of a mass spectrometer, limiting sample size to a few microliters. The PTV injector is designed to allow the inlet to perform a pre-separation of target analytes from solvent or other components of the sample. The PTV is constructed with a low mass injector body and insert allowing it to be heated with precise control at rates up to 14.5°C per second. The control precision of the PTV inlet allows techniques such as solvent evaporation from analytes of interest, pre-cutting of light analytes from a heavier matrix, and concentration of heavier impurities from gaseous samples.

The PTV can utilize multiple temperature ramps enabling several techniques to be applied to each sample (Figure 2). PTV provides sample injections with low discrimination to beyond C₅₀. The Cold On-Column (OCI) inlet can successfully inject analytes to the limits of column technology, for example: microcrystalline waxes up to C₁₀₀.

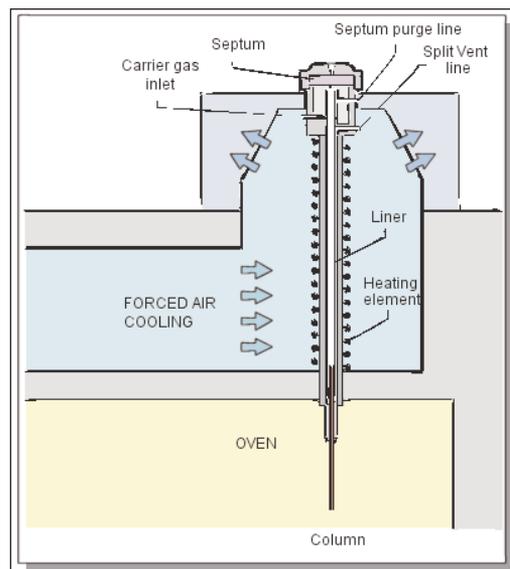


Figure 1: Detail of PTV Injector

Large Volume with PTV or OCI?

TRACE GCs are available with two inlets designed for large volume injection, Large Volume PTV (PTVLVI) and Large Volume On-Column Injection (LVOCI). Both are suitable for injecting large volume samples. The primary selection criteria between the two inlets are sample cleanliness, analyte boiling range, and mass spectrometer operation. LVOCI utilizes a partially coated pre-column to aid the separation of volatile analytes from solvent. Samples must be clean so the pre-column does not become contaminated with heavy residue. LVOCI can be used successfully with analytes more volatile than those that can be retained by PTVLVI. PTVLVI is a better choice for samples containing heavy components beyond the analytes of interest because the high boiling fraction can be retained in replaceable inlet liners or backflushed. Because PTVLVI does not employ a pre-column for solvent separation, it cannot retain analytes with volatility close to the solvent.

Key Words

- PTV Injector
- Large Volume Injections
- Shorter Sample Prep

Adding a selective packing to the liner of the PTV can help to retain volatile analytes, but in general, LVOCI better retains analytes whose volatility is near the solvent. In LVOCI injections, however, significant amounts of solvent can be eluted through the analytical column and into the mass spectrometer. PTVLVI, when equipped with the backflush option, prevents solvent from entering the analytical column, ensuring that the mass spectrometer vacuum system is protected, Figure 3.

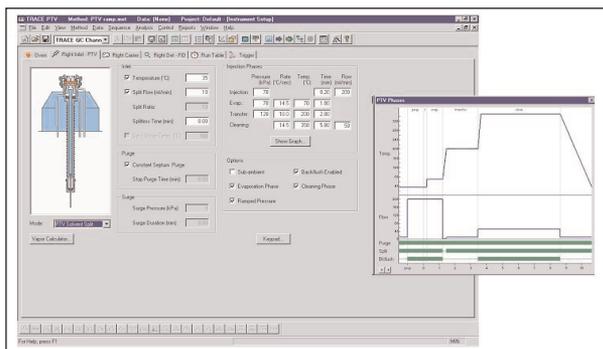


Figure 2: Control parameters for PTV Injector

Description

Samples are injected as a liquid into the PTV at a low temperature. The PTV temperature is then rapidly heated to vaporize solvent or low boiling components with the split vent open, sending these components to vent. With the addition of a heated solvent vapor exit valve, large volumes of solvent can be vented while retaining heavier analytes of interest for injection. The vapor exit valve must be heated to reproducibly vent large volumes of solvent vapors. The injection of large sample volumes can be used to lower method detection limits or to eliminate the need for concentration of extracts.

With the backflush option installed, solvent can be prevented from entering the column during this phase, making this technique ideal for use with Mass Spectrometers. The split vent is then closed and a second temperature ramp initiated, transferring target analytes to the analytical column. The split vent is reopened and backflush flow begun with a third temperature ramp to clean the inlet before cooldown for the next sample cycle.

There are many chromatographic methods for trace contamination where the extraction and concentration of solvent extracts requires more time than the chromatography. In these methods, the ability to inject large solvent volumes of less concentrated extracts can mean a significant increase in laboratory productivity.

The PTV is very useful in separating light analytes from a heavy matrix. Olive oil is primarily made up of triglycerides, which are very high boiling and difficult to chromatograph. The PTV with backflush can be used to prevent the triglycerides from entering the analytical column, shortening the analysis time (Figure 4).

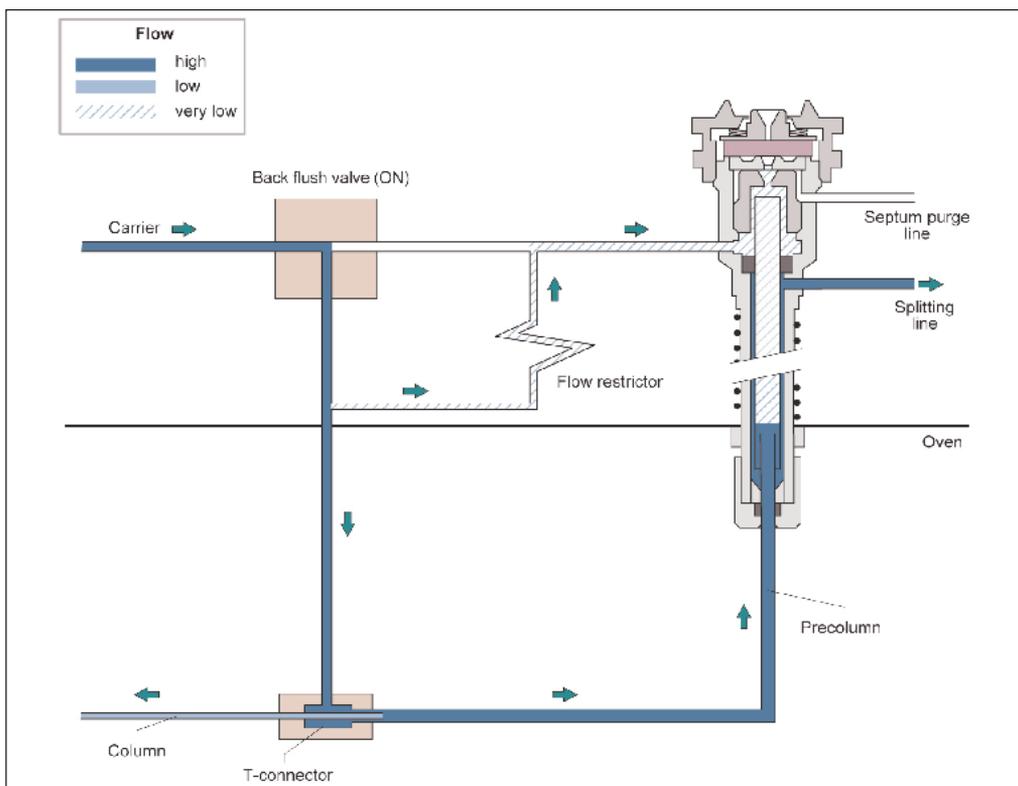


Figure 3: Detail of Backflush Valve Accessory for PTV Injector

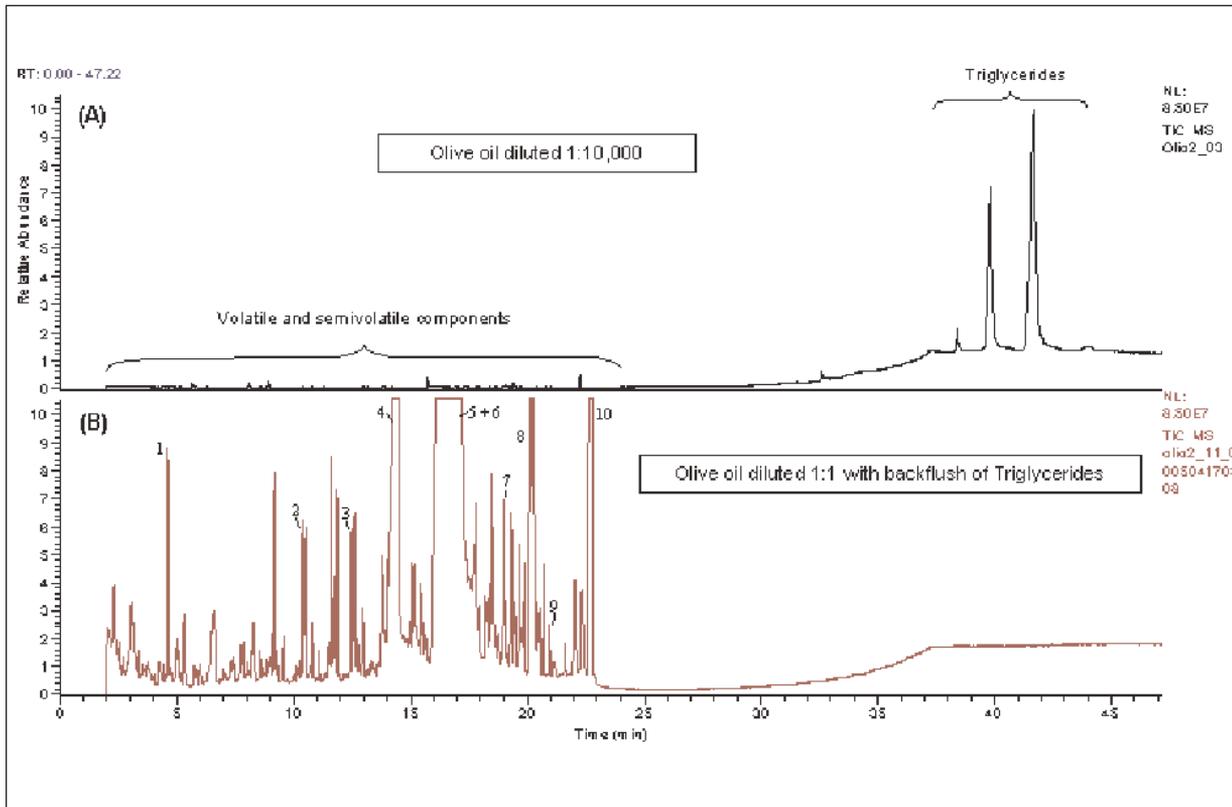


Figure 4: Example of backflush used to prevent heavy analytes from entering analytical column

Conclusion

A PTV injector can be very useful as a solution to a number of difficult sample types. The PTV can be used to concentrate portions of a sample, allowing larger volume injections than an analytical column normally accepts. The PTV can be used as a Large Volume Injector for samples that may contain heavy components or particulates. The PTV can function as a cryofocusing device for gaseous samples. The use of the PTVLVI allows higher productivity while maintaining method sensitivity and protecting Mass Spectrometers from the introduction of large volumes of solvent. The ability to inject large volumes reproducibly can reduce preparation time for solvent extraction methods. This is an important consideration for an analytical laboratory utilizing methods where sample preparation time is lengthy. Solvent extraction and evaporation is a labor intensive procedure which is not easily automated.

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