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Fundamentals of Purge and Trap

Application Note

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Introduction

Purge and Trap has been used for decades to extract volatile organic compounds (VOCs) from a solid or liquid matrix for introduction into a Gas Chromatograph (GC) for separation and identification. The VOCs are concentrated onto an absorbent trap, followed by thermal desorption into a GC. Sample matrices can range from soil, plastics, foods, flavor, fragrance, emulsions and water.

From its early body fluids' applications in the 1960's, Purge and Trap has evolved to the standardized protocol for analyzing environmental samples (i.e., soil, water). Several regulatory agencies including the United States Environmental Protection Agency (USEPA), the United States Department of Energy (USDOE), and the United States Department of Defense (USDOD) has instituted methodologies based on Purge and Trap.

The Tekmar 3000 purge and trap concentrator has been designed to perform these functions. The Purge and Trap concentrator is interfaced to a GC through its injection port or directly to the column based on the configuration desired. The purge gas is supplied directly on the concentrator and is flow regulated before purging the sample. The desorption gas is supplied and controlled by the GC or a separate regulator. These gas flows are routed through the concentrator by solenoids and a six port switching valve.

The Purge and Trap primarily performs three functions: Purge, Desorb, and Trap Bake. A detail description of the operation of the system is outlined below.

Operation

Purge Mode

The VOCs are removed from the sample matrix during this process. The purge gas (helium or nitrogen 99.999% purity) passes through the bottom of a fritted sparge vessel before it makes contact with the sample. The frit disperses the gas into finely divided bubbles allowing a large surface area of the sample to be contacted. This process allows the inert gas stream to strip the analytes from the sample matrix and concentrate them on an adsorbent trap.

The purge efficiency of an analyte is based on vapor pressure, solubility, temperature of sample and purge volume. The higher the vapor pressure of the analyte, the quicker it vaporizes and leaves the sample. Highly polar organic compounds are quite soluble in water and are held together by very strong dipole-dipole interaction and hydrogen bonds. These compounds will have poor purge efficiencies. Increasing the temperature of the sample puts enough thermal energy into the molecule to break the dipole-dipole interaction thus increasing the purge efficiency. The total amount of VOCs removed from the sample is directly proportional to the purge volume. The purge volume is a product of the purge flow rate and the purge time. The recommended setting for the system is flow rate of 40 ml/min for a period of 11 minutes for a purge volume of 440 ml.

Desorb Mode

The purged analytes are now trapped onto a solid sorbent. The Desorption mode requires the heating and backflushing of the trap with desorption gas to release and transfer the analytes of interest to the GC. The GC carrier gas is used as the desorb gas and involves switching the six port valve to place the trap in line with the GC column.

The trapping material (adsorbents) efficiency is greatly affected by temperature and desorption time. The two most widely used adsorbent traps are the three stage Tenax/silica gel/ charcoal and the Vocarb 3000. Desorption

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profiles of these traps are based on the adsorption of the analytes onto/into the trap. The further the analytes move into the bed, the broader the desorption profile is going to be. This can be overcome by using desorb preheat, which involves the heating of the trap without flow to release the analytes. This will allow the analytes to desorb as a narrow band during desorption.

While purge efficiency is based only on the amount of analyte recovered from a purged sample, the percent recovery takes into account both the desorption and transfer efficiencies. The percent recovery is calculated as follows:

$$\% \text{ Recovery} = A/B * 100$$

where A = the area counts of analyte by Purge and Trap and B = the area counts of analyte by direct injection.

A moisture control system (MCS) is place between the trap and the GC to remove water from the desorb stream. This is important for detectors that are sensitive to water.

Trap Bake Mode

Trap bake mode is designed to clean up the trapping material prior to next run. Baking a trap is analogous to baking a GC column. This process requires the switching of the six-port valve allowing the trap to be flushed with purge gas. The time and temperature required for baking is related to the trapping material and the compounds analyzed. The purge gas in this mode goes through the purge vessel or can be bypassed (bake gas bypass) depending on the configuration desired.

Conclusion

Purge and Trap is a widely used technique for VOCs for a multitude of sample types. The Purge and Trap technique can be fully utilized by understanding the Purge, Desorb and Bake functions.