

A NOVEL APPROACH TO LARGE VOLUME LIQUID INJECTIONS IN GAS CHROMATOGRAPHY

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INTRODUCTION

Up to 100 μ L of liquid sample can be injected into a standard gas chromatograph without having to install a new injector or overloading the existing one. SGE has developed a new technique in which a wide bore capillary column can be used in a similar way to a Programmed Temperature Vaporizing Injector. There are two stages to SGE's Large Volume Injection System:

1. The sample is injected into a 0.53mm ID capillary column that is under high pressure and the solvent is separated from the compounds of interest by using the solvent effect on a large scale.
2. The compounds are transferred to an analytical column and analyzed using a standard method.

HOW DOES IT WORK?

STAGE 1 - Solvent Separation

The injection is carried out into a conventional Splitless injection system, except the pressure in the injection port is typically 60psi or higher. The sample then enters a short 0.53mm ID capillary column, under the same pressure, where it recondenses. At such high pressures, the volume that the sample can expand to is restricted and the recondensation of the solvent onto the precolumn occurs instantly. The volume that can be injected will depend on the MW of the solvent but for most solvents 100 μ L is not uncommon.

Figure 1 shows the amount of sample that can be injected before loss of solute occurs.

The high carrier gas pressure combined with the large surface area of the 0.53mm ID precolumn causes the Solvent Effect to be very efficient. The Solvent Effect is used to selectively remove all the solvent while leaving the solute molecules in the stationary phase of the precolumn. Once the solvent is separated from the solute, it is removed from the precolumn and burnt by a Flame Ionization Detector. The detector is solely used to monitor the solvent as it exits the precolumn. This allows the user to ensure that all the solvent is disposed of before the system transfers the solute to the analytical column.

STAGE 2 - Compound Transfer & Analysis

To retrieve the compounds of interest, the oven is heated and a compressed air cold trap collects the solute as it elutes from the 0.53mm ID column. The system then switches into analytical mode, where the solute molecules are directed into a narrow bore analytical column. An overview of the system setup is shown in **Figure 2**.

An example is shown in **Figure 3** where 80 μ L of solvent was injected and separated from C24 and C26.

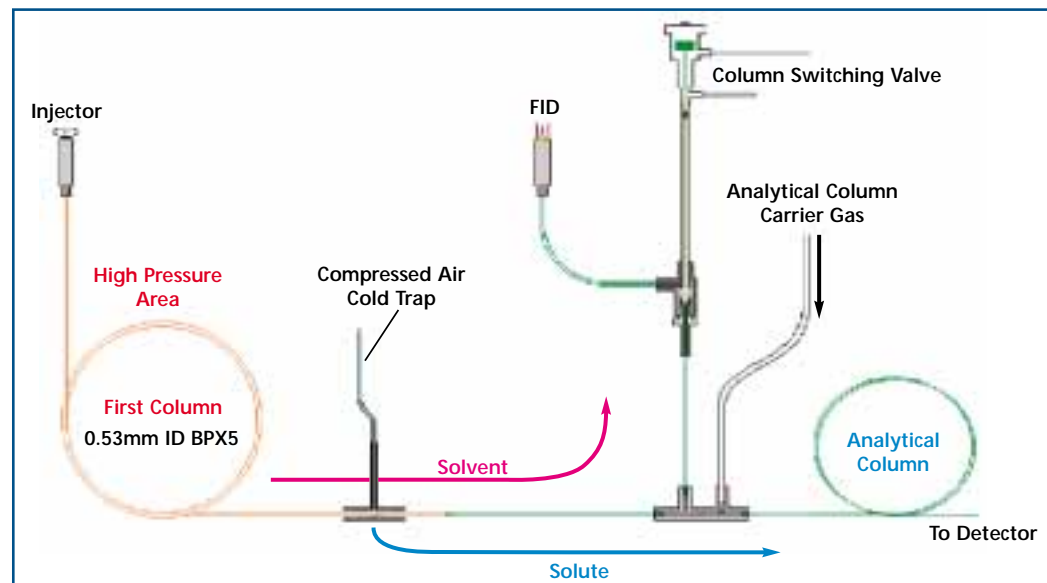


Figure 2. The large volume injection system.

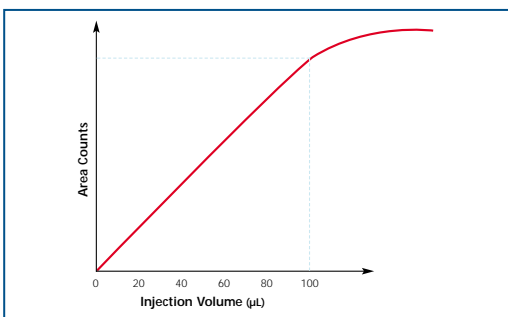


Figure 1. Injection volume linearity, C24 in decane.

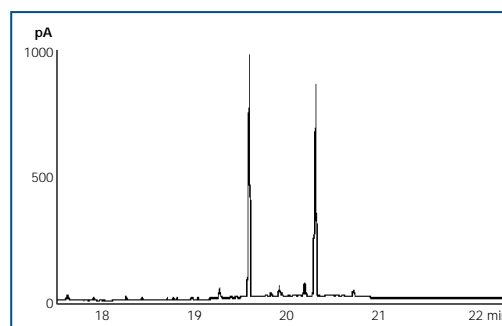


Figure 3. 80 μ L of 1ng/ μ L C24 and C26 in iso-octane.

CONCLUSION

It is possible to make large volume injections without using a Programmed Temperature Vaporizing Injector. This injection system makes it possible to dispose of large volumes of solvent with minimal modification to the instrument and uses the existing splitless injection system. Trace analysis of pesticide residues and dioxins are routinely being carried out using this technique.

