



AB-02 APPLICATION BRIEF-02

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SAMPLE CONCENTRATION WITH THE USE OF DV SERIES DIAPHRAGM VALVES

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Using a sample concentrator is really useful when trying to determine very low level of impurities or to simply improve the sensibility of a system and/or to improve peak shape. The trapping or sorbent material could be of a single type or a mix of different sorbing material, each one targeting a single or a group of compounds.

The concept is simple and provides an inexpensive way to improve overall system sensitivity, without having to modify the detector technology. It may be used in front of a GC system or to be the sample injection by itself.

It has been used by many with great results. Thank to high level of performance of DV series tight shut off diaphragm valves, sample concentrators or isolation systems are easily achieved.

The system relies on proper timing sequence. This is easily achieved with the use of EDV series valves. This one has a built-in microcontroller, allowing to daisy-chain several valve blocks and downloading into each one the appropriate timing sequence. Then, the sequence could be triggered by a digital contact or through serial port. Please see the EDV products literature for more information about the features and benefits of this series.

Different types of trapping material could be used: Tenax™, molecular sieve, various catalysts, column packing materials. The choice depends of which type of impurity is to be trapped and injected into the system. Sample matrix may also guide the selection of trapping material.

A tube of appropriate dimensions for the application is filled with the selected material. This becomes the trap. The system should be capable to heat and cool down the trap quickly. The trap could also be cooled down at sub-ambient with the help of Peltier thermoelectric cooler. This enhances the trapping of volatile compounds.

The design of the heat/cool sub-system is application dependent.

In Figures 1, there is an example of such system. In this configuration, the trap effluent is directly injected into the GC separation column. The basic idea behind the concept is to allow the sample to flow at a predetermined fixed flow and pressure into the sample trap, for a predetermined amount of time. The trap should be maintained at constant temperature during this step. Then, the sample trap is isolated, heated, pressurized and injected. After the sample has been desorbed from the trap and injected into separation column, the trap could be purged with carrier gas, while column head pressure is maintained by another source of carrier.

The complete sequence is shown in Figures 1.

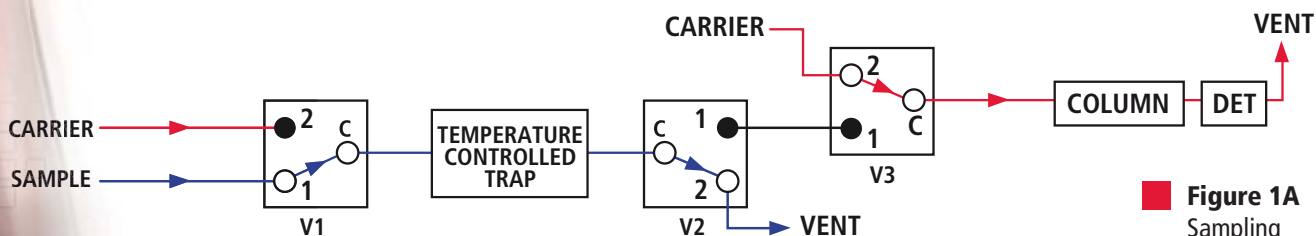


Figure 1A
Sampling

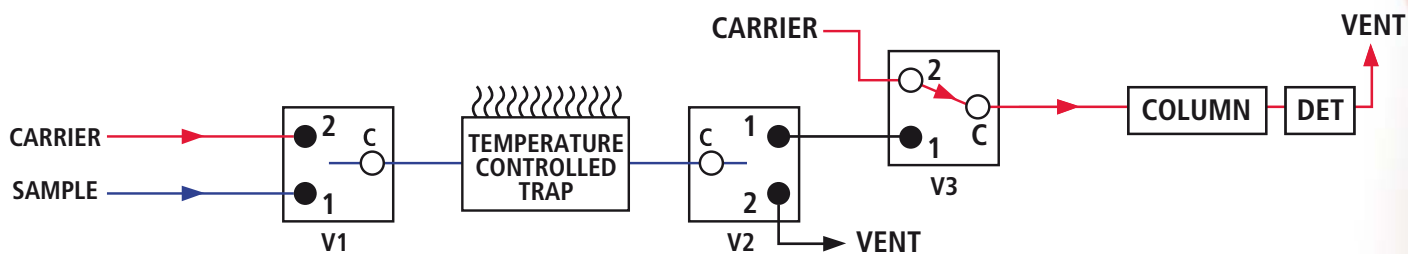


Figure 1B
Sample trap isolated and heated

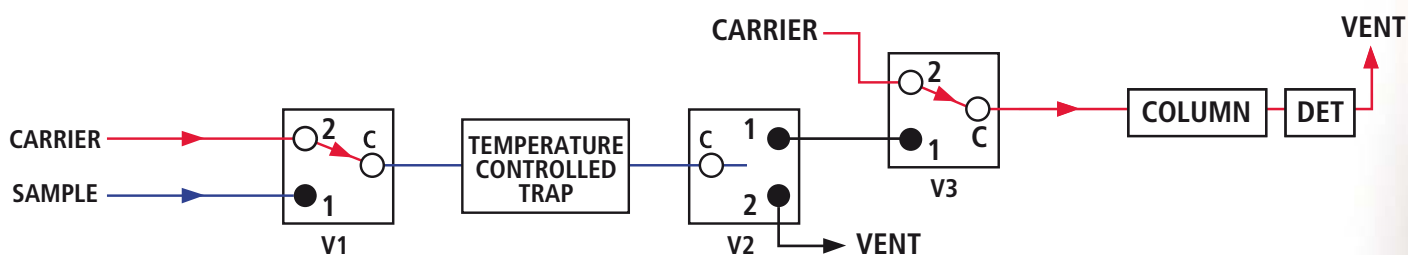


Figure 1C
Sample trap pressure is raised to carrier pressure

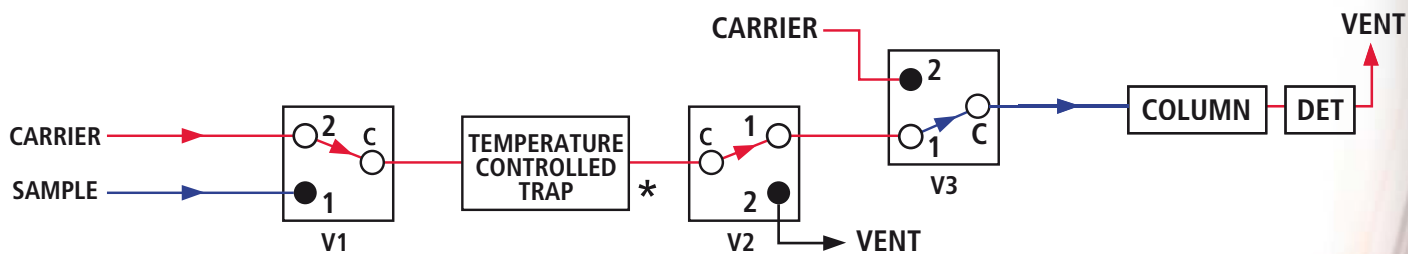


Figure 1D
Sample is injected into separation column; trap is still at high temperature
*tubing length long enough to cool down the gas

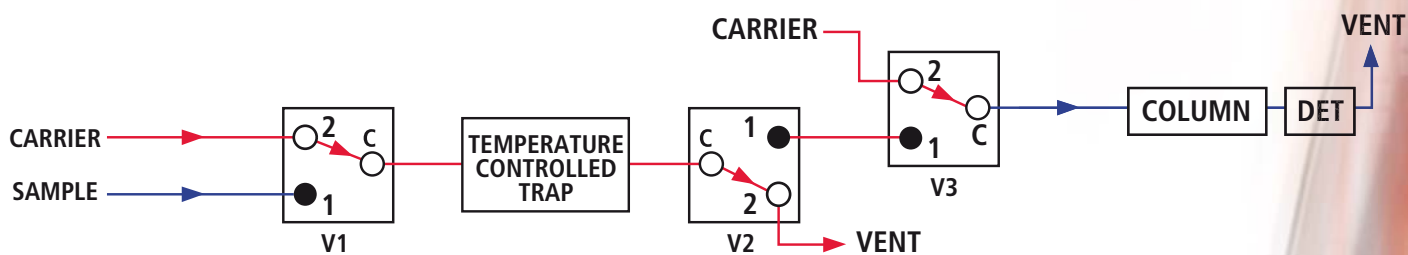


Figure 1E
Trap is purged while hot, and then cooled down, while having clean carrier gas flowing in it. At the same time, impurities are separated into columns.

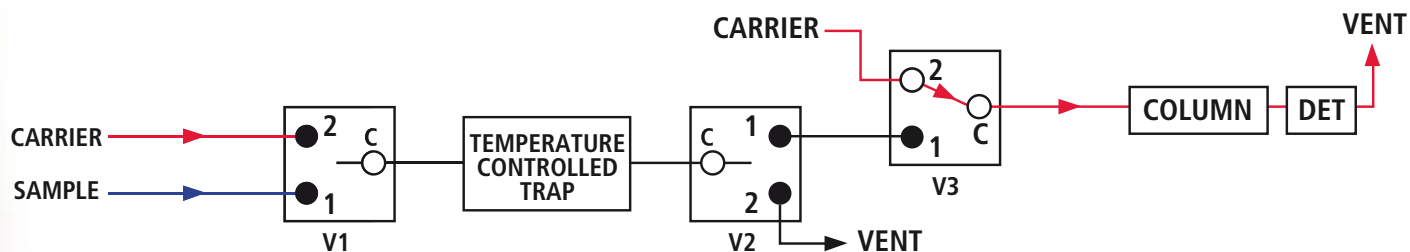


Figure 1F - Trap is isolated and ready for another cycle, i.e. go back to step 1.

The time that the sample should be allowed to flow into the trap is determined by the amount of impurities to be trapped, the targeted sensitivity, and of course trapping material characteristic.

Calculation could be made to determine the required quantity of material, based on manufacturer data. Experimentation could also be done to tune the system.

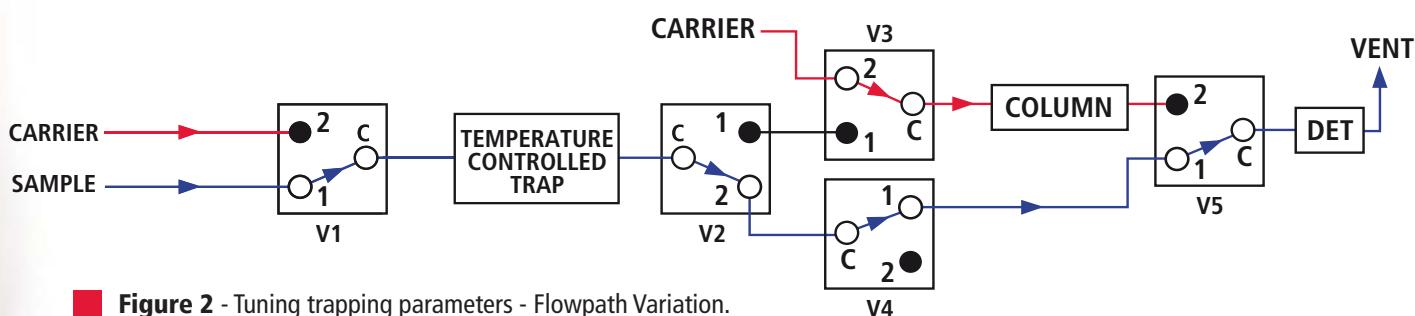


Figure 2 - Tuning trapping parameters - Flowpath Variation.

Figure 2 shows an interesting and practical flowpath variation.

It allows redirecting the effluent of the trap directly into the detector. This is useful to check trap performance and to tune timing sequence.

For example, if a calibration gas, typically carrier background having some level of targeted impurities in it, is used as a sample, while the trap is directly connected to the detector and GC column by-pass, it allows monitoring trap performance.

Indeed, monitoring the outlet of the trap with various sampling time, and at various temperature, is a practical method to determine proper timing. The idea is to make sure that nothing is coming out of the trap, while in sampling mode. If not, trap overloading will occur. This is very useful when trapping material is made of molecular sieves.

Many design variations could be done to fit any particular requirement. Liquid sample concentrators have been also done with a diaphragm valve. Impurities in drinking water is one example.

Figure 3 show another design variation. Here the valve V1 of figure 2 have been replace by a DVS series valve. In this case a DVS-3. The third port of V1 is connect to a source of regenerating gas. The purpose of this gas is to oxidize or reduce the trapping material when this one is base on a catalist. When reconditioning or regenerating the trap, the effluent is vented through V4 port 2. This design variation may be use to remove a specific reactive compound from a sample matrix. The output of such sub-system

may be connected to another concentrator sub-system. This way unwanted compound could be eliminate and specific one to be concentrate. This having the benefits to increase system sensitivity and separation.

Any number of DV valve blocks could be added in a system without adverse effects on the system.

Indeed, DV valves have no dead volume effect and there is no sample or carrier contamination. The pressure drop of a DV is the same than a standard 1/16" OD union having an internal diameter of .040». DV valves could be optimized for liquid or gas phase applications.

TIPS AND HINTS

- 1 Trap must have particles filter to avoid trapping material to flow into valves.
- 2 Sufficient length of tubing must be used to allow the gas to cool down before entering into the valve.
- 3 Depending of impurities to be concentrated, valves may require purged sealing plates. This eliminates problems related to permeation and diffusion through the diaphragm. Valves could also be heated to avoid impurities adsorption.
- 4 Proper valve sequence timing should be done to avoid reaching equilibrium level into the trap. It's better to have more trapping material or sorbents and shorter sampling interval.