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SAMPLING AND VAPORIZING LIQUID SAMPLE

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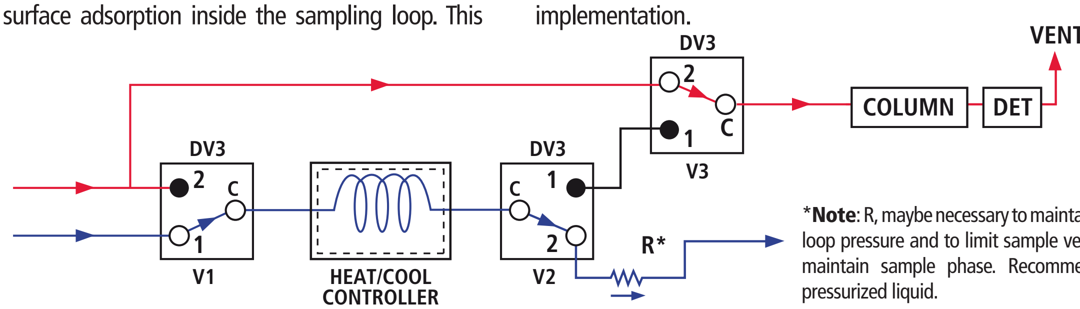
This method allows sampling fixed volumes of various liquid samples, and then vaporizing them before to be injected into separation column. This way, a gas phase of the sample is brought into the column. Vaporizing completely and quickly an isolated liquid sample volume avoids sample fractionation or distillation. This results in an accurate sample representation, since the flashing zone is well defined.This method has also the benefit of eliminating surface adsorption inside the sampling loop. This eliminates carry over or memory effect and improves response and stabilization time.

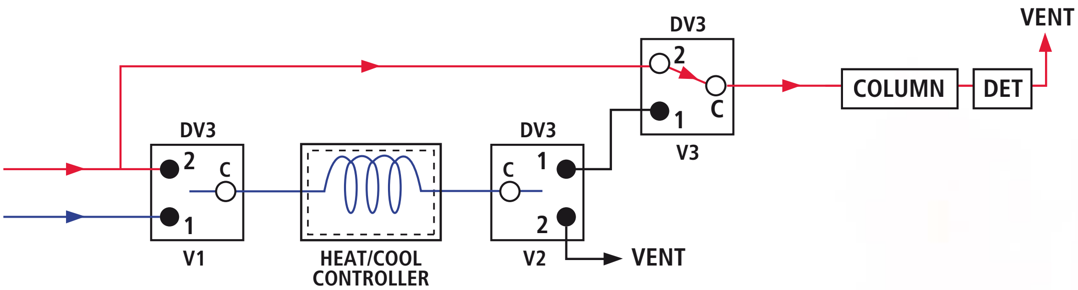
The method is simple, as explained in Figure 1 and following sequence description. This method was developed with the use of tight shut off DV series diaphragm valves. Both pneumatic and electric actuator configuration type could be used for its implementation.



*Note: R, maybe necessary to maintain sample loop pressure and to limit sample velocity and maintain sample phase. Recommended for pressurized liquid.

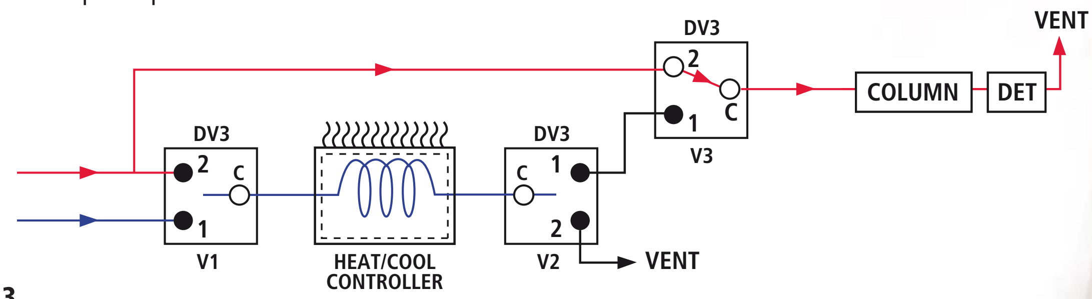
STEP 1

Figure 1.1 - Sampling at ambient or sub-ambient temperature.



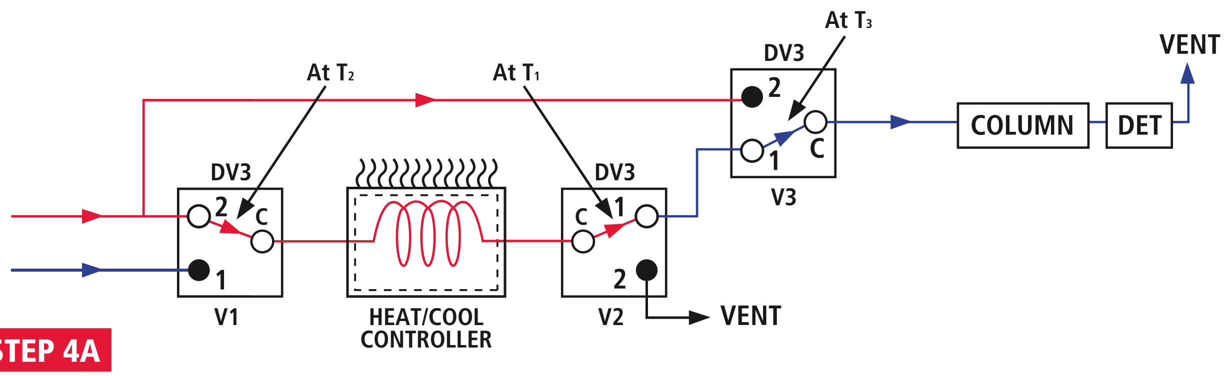
STEP 2

Figure 1.2 - Sample loop isolation.



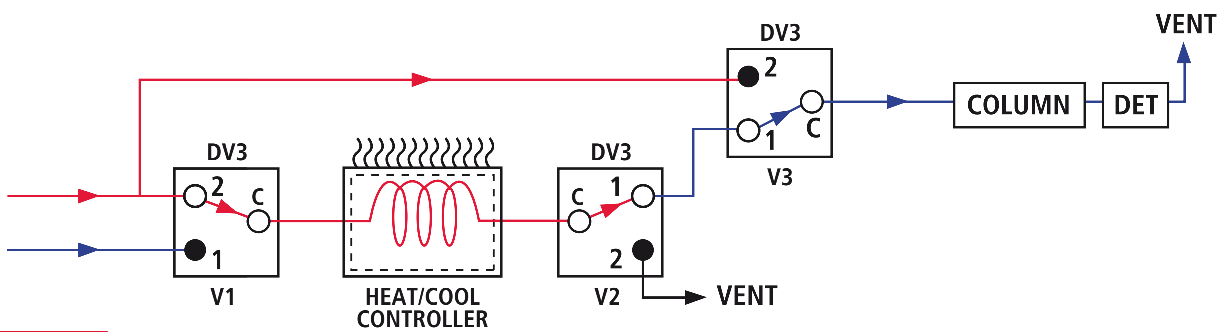
STEP 3

Figure 1.3
Sample loop is heated. Up to a preset point and optional a stabilization time maybe added before to go to the next step.



STEP 4A

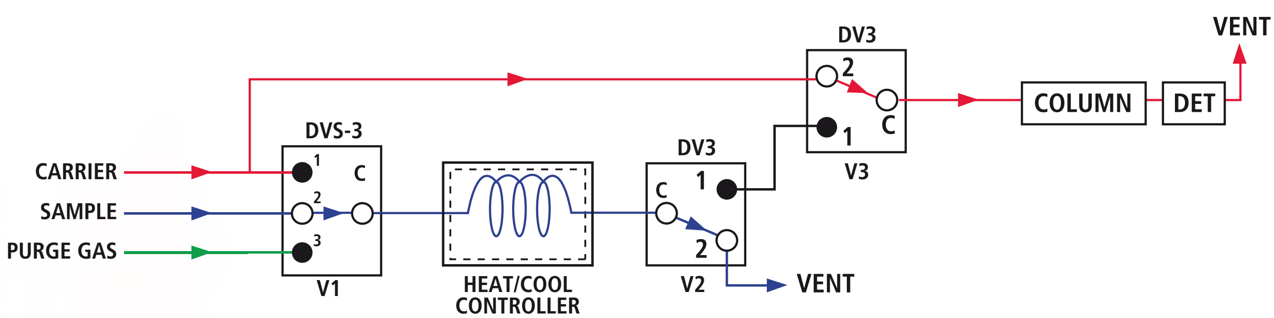
Figure 1.4



STEP 4B

Figure 1.5
Direct sample loop injection.

<u>Note 1:</u> Optionally, V1 could be replaced by a DVS-3. This allows purging the sampling system between cycles (see Figure 2).



STEP 5 **OPTIONAL**

Figure 2:
Configuration using a DVS-3 sample stream selection valve

This method could be used for reactive liquid sampling. It could be also used for cryogenic samples. In this case, a special version of DV valve having remote actuator with standoff mounting should be used for temperature below -30°C. This applies to V1 and V2. Proper insulation must be installed while V1 and V2 are mounted close to sample point vessel.

In the case of reactive sample, complete system purge must be done before allowing the sample to flow in it.

This method is also useful for various hydrocarbons sampling.

STEP DESCRIPTION

STEP 1
During this step the liquid sample flow through the sampling loop. Depend on the vaporizing pressure of the liquid it could be necessary to add a restrictor ''R", to make sure that the pressure stay at the require value in order to keep the sample in liquid phase. Another use oif the restrictor ''R" is too limit the pressure drop between the inlet and outlet of the sampling loop. The idea is too avoid fluid too high velocity is require to have a fast system reponse it is much better to use a sample by-pass flow before V1. Normally this could be done with a bypass value or rotometer. Generally in liquid sampling the sample loop is small normally lower than 500 µL. So, the sample loop volume is refresh several time per minutes at a relatively small sample flow trough it. But this flow, is generally too low to have an overall fast system response. This is generally true if sample point connector is far from the sampling loop. So, sample by-pass flow is a good idea to speed up the response time, without resulting in a too high fluid velocity through sample loop, or pressure expension that may lead to vaporisation into vent line, causing gas lock. Here we mean by speed of response the time that the fluid flowing into the sample volume correspond to the composition fluid being sample into the process.

In brief sampling sample vessel fo a distillation process remotely located will drive the decision to install a sample by-pass or not.

STEP 2 - figure 1.2
During this step the sample volume is completely isolated, i.e. inlet and outlet are shut off, and sample fluid flow is stopped.

STEP 3 - figure 1.3
Heat is applied to the sample loop until a preset temperature value is reach. At this point value is reach. At this point a stabilization time could be added. Normally at this point all sample phase should be gaseous.

Note : Step 2 and 3 may be combined in a single step.

STEP 4 - figure 1.4 and 1.5
This step is the dample volume injection one. This step could be done in an shot or sequentially. This depend of the sample fluid, and the sample size as show in figure 1.4, the injection could be done in 3 step. First at T1, V2, port 1 will be open to allow the pressurized gas sample to pressurize the line section between V2 Port 1 and V3 port 1. Then at T2, V1 port 2 is open to set the sample loop volume at the carrier pressure value. Then at T3, V3 port 1 is open and port 2 is close, this allow the sample volume injection. This 3 steps injection method could be use when sample volume is large and it pressure at or below carrier pressure. This avoid column and detector to be upset by suddend pressure and flow change up on injection. When the final pressure resulting from the liquid sample vaporazation is higher than the carrier pressure, it might be a good idea to open first V2 port 1 follow by the closing of V3 port 1. A short period of time after these, V1 port 2 could be open. This allow the depressurisation of the sample volume into column flow path, before to push this sample gas volume by the carrier. This until reduce over all system upset if any.

Here some trial and error experimantation will help to select the right injection sequence. The fact to be capable to control indepently each valve ports open the doors to system flexibility.

STEP 5 - figure 2, optimal purge of sampling system.
Here, V1 have been replace by a DVS series valve. The one show is a 3 inlet sample stream selecion valve, DVS-3 (Pneumatic Actuation) or EDVS-3 (Electronic Actuation). Here this design variation allow to switch the sampling volume and associate valve's internal parts and related tubing to a clean and innert purging gas. This could be done after the sample have been inject. It is useful to eliminate reactive sample out of the system and to keep it in standby. It may also be use to pre-conditioning the sampling system by evacuating atmospheric contaminant before to allow reactive compound to flow in it.

DESIGN VARIATIONS

1. Sample injection valve, i.e. V1 and V2, could be installed remotely from the GC system. This allows for a much faster response time of the sampling system.

2. When a liquid sample volume is flashed or vaporized, its volume increases anywhere from 600 to 900 times. In some case, this may overload the analytical column or detector.

   A convenient way to overcome this issue is to control the timing of V1 and V2. This way a repetitive gas volume representing a fraction of the total gas volume will be injected.

3. Different valve configurations could be used to answer specific needs or system requirements. For example, the DV series comes standard with a purge system, eliminating the risk of fugitive emission and inboard contamination. An optional sealing plate having extra purge connections could be provided, further improving the inertness of the system. This purge eliminates any atmospheric air out from the back side of the diaphragm. It also eliminates the possibility of having some sample vapor phase accumulating over the time. This purge eliminates the problem associated with permeation or diffusion through the diaphragm.

4. Adding some intelligence by monitoring purge gas vent, early warning could be issued in case of valve leak. This allows scheduling system maintenance and avoiding unplanned system shut down with the associated contamination problems. It also allows a safer working environment by evacuating away hazardous gases and preventing total system failure and leakage into working environment.

