ASDevices

µInProve™

IMPROVING THE PAST...
REDIFINING THE FUTURE

AN-05
PURGED LIP SEALING VALVE TECHNOLOGY AND APPLICATIONS

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REV. 01

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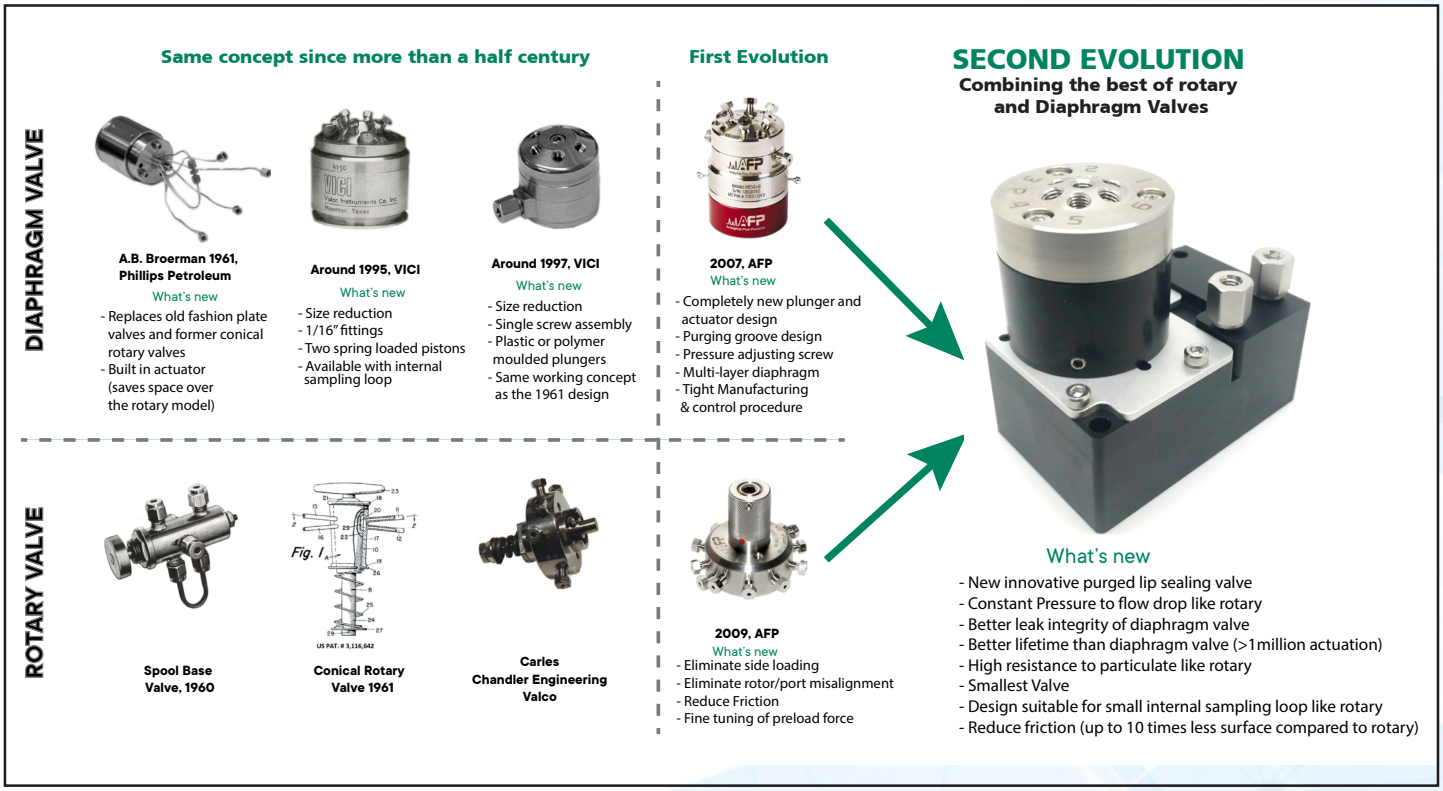
60 years of Evolution

During the past 60 years, chromatographic valves have been improved but the base concepts remained the same: conical rotary and diaphragm. A few innovations were introduced by Yves Gamache and his team at Contrôle Analytique and AFP as they were confronted to performance limitations. The valve performances at the time were the main limitations in his quest to offer better gas chromatographs. In order to achieve the performance he needed, he introduced and patented many design improvements which allowed to get the most out of the rotary and diaphragm concepts. However, some problems remained unsolved due to fundamental limitations. Based on decades of expertise in designing chromatographic methods and valves, a revolutionary concept is born, the Purged Lip Sealing Valve (PLSV). The PLSV combines the best of diaphragm and conical rotary valves without any of their drawbacks.

The below figure summarises the evolutions.



Conical Rotary Valve

People like conical rotary valves because

- ✓ No dead volume
- ✓ Constant pressure/flow drop between positions
- ✓ Can operate at high pressure
- ✓ Good resistance to particulates
- ✓ Design can accommodate very small internal sampling loops
- ✓ Low cost

People don't like conical rotary valves because

- ✗ Poor life time
- ✗ Bulky (valve plus actuator)
- ✗ Require more complex machining prcessus

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Diaphragm Valve

People like diaphragm valves because

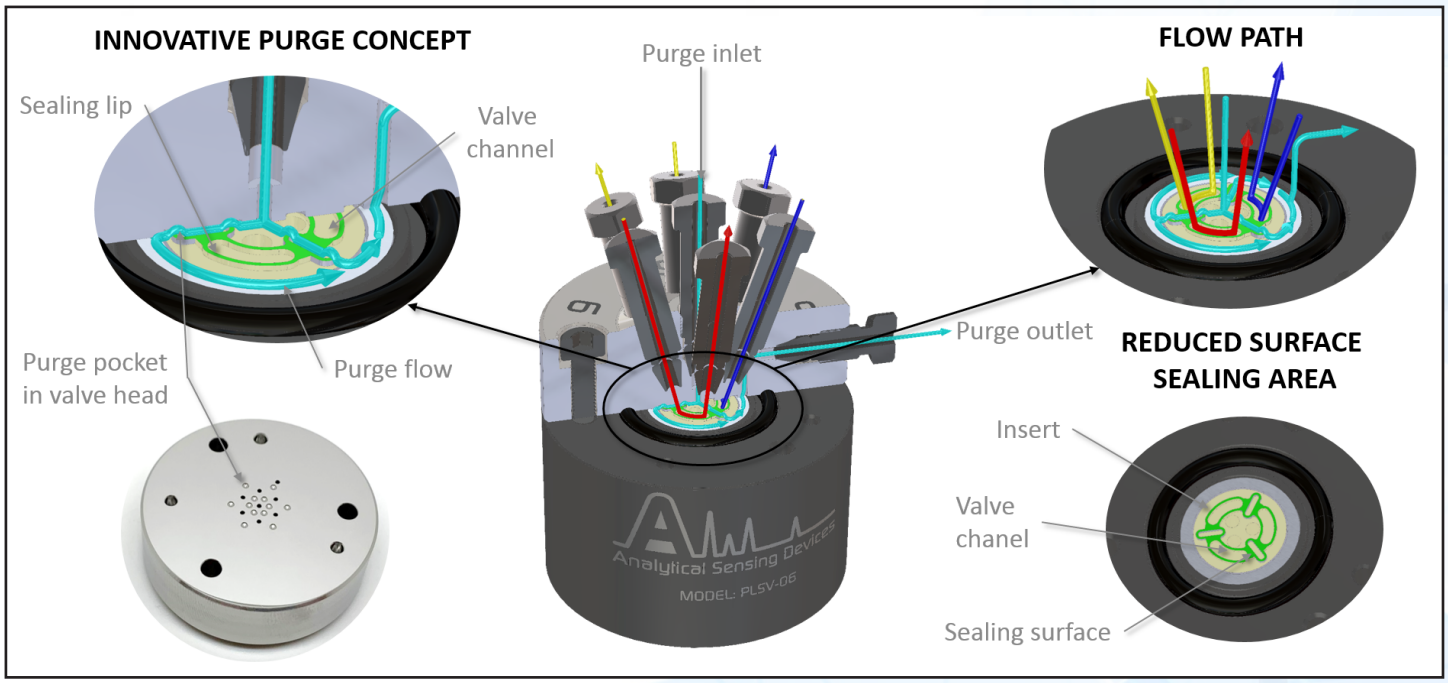
- ✓ Very good life time
- ✓ Very good leak integrity
- ✓ Compact with integrated actuator

People don't like diaphragm valves because

- ✗ Dead volume
- ✗ Poor pressure/flow drop characteristic
- ✗ Expensive
- ✗ Performance impacted by particulates

µInProve™ Purged Lip Sealing Valve*

The Purged Lip Sealing Valve (PLSV) concept is the result of many years of experience, frustration and customer feedback in a quest to improve the diaphragm and conical rotary valve concept. The outcome is the uInProve PLSV valve which has shown, following intensive testing, a superior performance compared to any other valve on the market. It combines the best of conical rotary and diaphragm valves.



µInProve™ Valve

Purged Lip Sealing Valve unique features

- ✓ No dead volume
- ✓ Constant pressure/flow drop
- ✓ Operate at high pressure
- ✓ Good resistance to particulates
- ✓ Design can accommodate very small internal sampling loop
- ✓ Very good life time
- ✓ Very good leak integrity
- ✓ Very compact

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* patent pending

This section is a the summary of different text books that teach how to do various gas chromatographic configurations to allow the measurement of multiple components in several types of backgrounds. Most of the following configurations and text were adapted from the book "The analysis of gases by chromatography" by C.J. Cowper and A.J. Derose, Pergamon Press. Another source of useful information was found in "Fundamentals of Gas Analysis by Gas chromatography" by Brian Thompson of Varian Associates Inc. As far as we know, both publications are out of print.

All of the following configurations could be done with diaphragm or standard rotary valves. However, the best performances are achieved with our PLSV technology. This is the reasons why all valve drawings show the internal purging grooves that characterize the µInProve PLSV and their inserts.

COLUMN SELECTION

A four-port valve is used as shown in Figure 1. The valve operation allows the injected sample to go onto either column. The two detectors could be the sensor and reference cells of a thermal conductivity detector.

When used this matter, the system may not offer any advantages over the use of another gas sampling valve. With a gas sampling valve in each carrier gas line, samples can be injected onto either column without the baseline disturbances which may attend the use of switching valves.

If the effluent from a single column is to be switched to different detectors during an analysis, then the configuration of Figure 1 can be modified by fitting the column before the valve. The effluents from the two columns can then be switched between the two detectors.

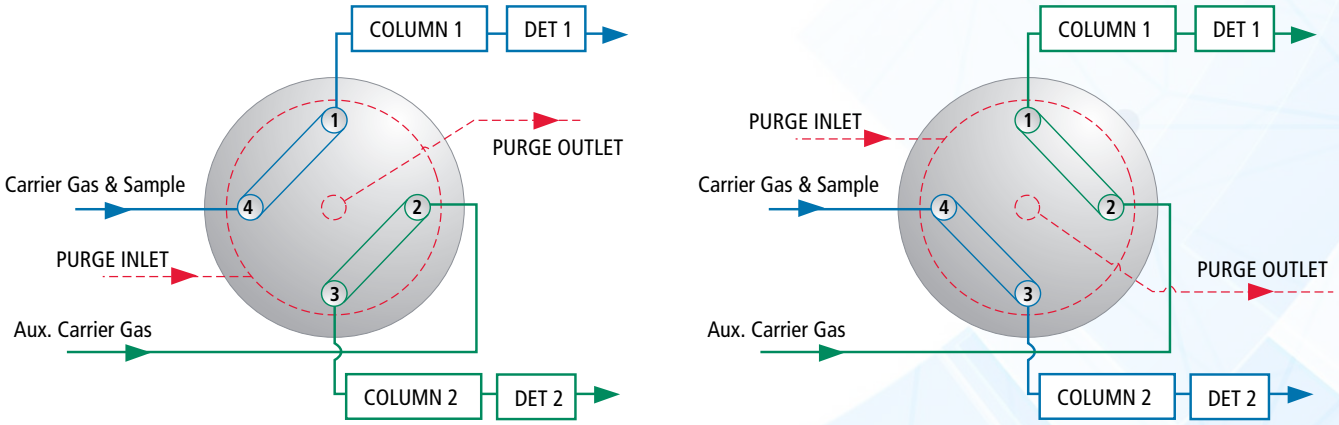

Figure 1: Column selection

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BACKFLUSHING

If the sample is complex, optimizing the separation of a group of components may mean that heavier components have very long retention time. Conditions may not favor their measurement, and they can cause an excessive delay between analyses. The gas flow in the column can be reversed after elution of last component of interest, and the heavier components eluted backwards: this should take approximately as long as they have spent travelling forward in the column.

The backflushed components can be vented, or fed to the detector so that a total figure for heavy components may be measured. While components are being backflushed, they are to some extent regrouped: those components which travel faster in forward flow also travel faster in backflush, and so catch up with the slower-moving ones. This is a, somewhat, simplified view and depends on the pressure drop and the ratio of retention time of the heavier components.

Peak broadening cannot be reversed, but the backflushed components often appear as a single, perhaps rather unsymmetrical peak. The limit of detection of this group is obviously much lower than that of the individual components which comprise it.

Backflush column to detector

A four-port valve is used as shown in Figure 2. If the detector is an FID, the pressure surge, when flow is reversed, may extinguish the flame. A restrictor should be placed between the valve and the detector.

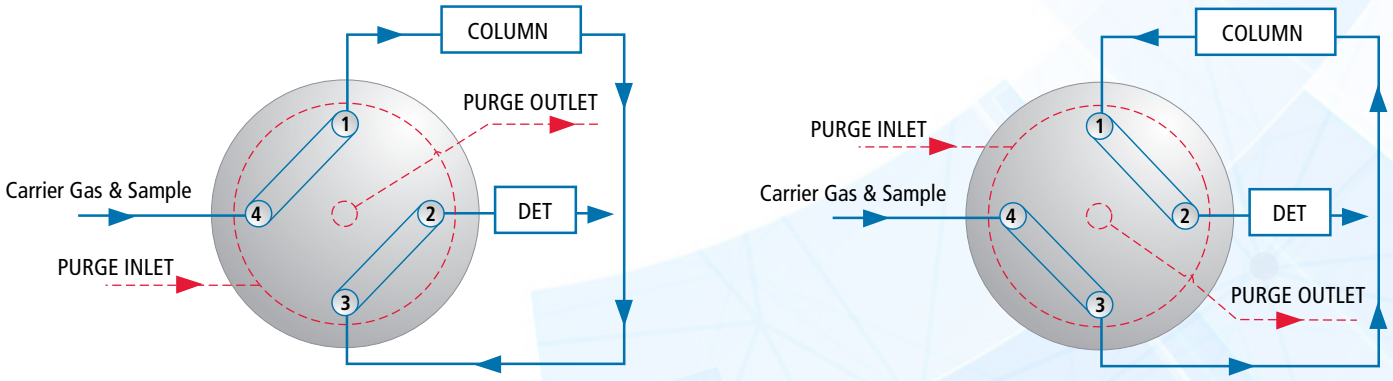

Figure 2: Backflush column to detector

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BACKFLUSHING (CONT'D)

Backflush pre-column

With the above configuration, the total time taken is approximately twice than the forward flow. An alternative approach uses two columns, the first of which (pre-column) only needs to be long enough to separate the group of desired components from the group to be backflushed. The first group is passed to the second (analytical) column in which they are separated. Simultaneously, the second group is backflushed from the first column. A six-port valve is used as shown in Figure. 3.

Restrictors R1 and R2 are set to have similar pneumatic resistances, to the analytical column and the pre-column respectively. This should ensure that the flow through the analytical column is undisturbed by the operation of the valve.

The backflushed group of components can be measured, if desired, by placing another detector on the vent line. Alternatively, the vent line can be teed in to the existing detector after the analytical column. In this case, the column length and switching time must be chosen so as to avoid interference between the backflushed group and the normally eluting components.

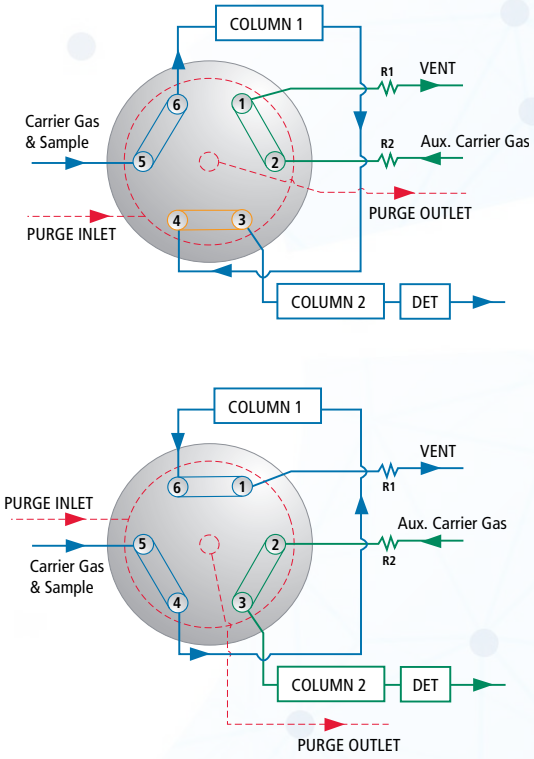

Figure 3: Backflush pre-column to vent

Backflush and sequence reverse

A six-port valve is used with a pre-column and an analytical column. A single carrier gas source is used, and the plumbing arranged so that switching both reverses the flow through the pre-column and positions it after the analytical column. Figure 4 shows the configuration.

The pre-column would normally be chosen to be considerably shorter than the analytical column. This means that it has little effect on the normally eluted components, even though they pass through it twice. The backflushed group of components, although it has spent only a short time travelling in either direction will emerge as a sharp peak. It can be arranged for the backflushed group to be the first to elute: this allows much lower detection limits than with a more conventional broad backflushed peak.

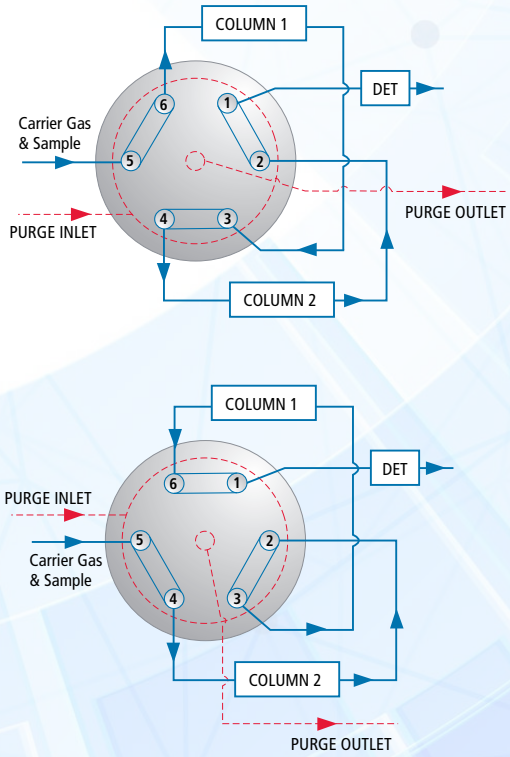

Figure 4: Backflush and sequence reverse

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COLUMN ISOLATION

Series operation of columns allows measurement of groups of components separated on columns whose characteristics are different. The drawback of the technique is that all the components eluted from the first column pass through the second. This can mean excessive retention time, which prolongs the interval between analyses, or deactivation of the second column (e.g. H2O, CO2 on molecular sieve).

A switching valve can be used to isolate and by-pass the second column after the appropriate components have entered it. Elution proceeds from the first column directly to the detector, then subsequently the second column is reconnected and the components which have been stored in it are passed to the detector. Alternatively, it may be possible to continue elution through the columns in series, until all components from the second column have been measured, then by-pass the second column before the heavier components have reached it.

Figure 5 shows the use of conventional six-port valves. The restrictor is adjusted to have the same resistance as the column, so that the flow is independent of the valve position. A disadvantage of the configuration shown in Figure 5 arises from the fact that both ends of the column are connected via the valve when it is isolated. During isolation, the pressure of the carrier gas in the column will adopt a value intermediate between the inlet and outlet pressures under flow conditions. This is achieved two ways: the first way is by continuing forward flow through the column, and the second is by reverse flow from the high-pressure to the low-pressure end of the column via the valve. There is a danger that a component which has just entered column 2 before it is isolated, may be transferred after isolation to the low pressure end of the column. This is avoided by the configuration shown in Figure 6.

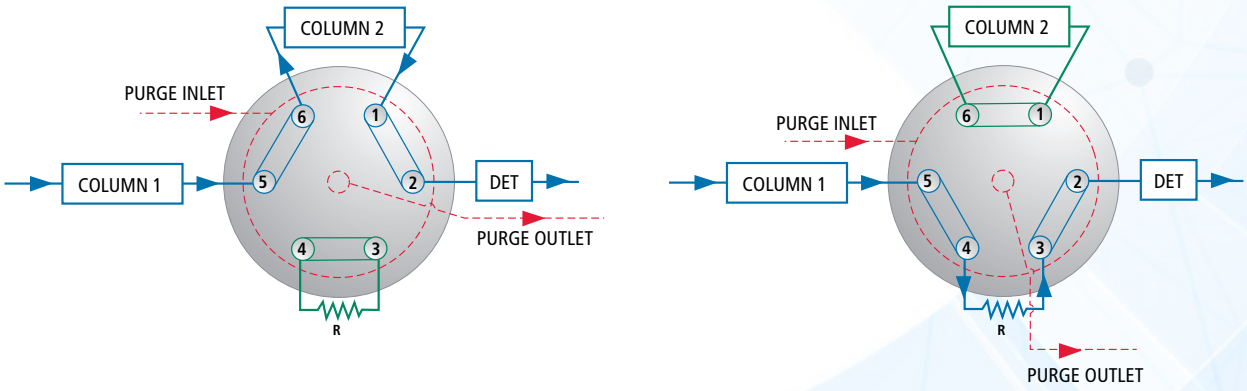

Figure 5: Column isolation - method 1

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COLUMN ISOLATION (CONT'D)

In this case the use of a rotor with only two slots means that equalization of pressure in the isolated column is only achieved by forward flow of carrier.

When components are isolated in a column in this way, the chromatographic process stops, but the component bands are still subject to diffusion in the mobile phase, which causes some broadening. The degree of broadening depends upon the time and upon the partition coefficient of the component: significant band broadening only occurs in the mobile phase and hence affects lighter components to a greater extent. In general, within the time-scale of most analyses, the degree of broadening will be perceptible but not troublesome. Furthermore, this type of operation will almost invariably be controlled by an automatic timer, and so the peak shape and resolution will be consistent.

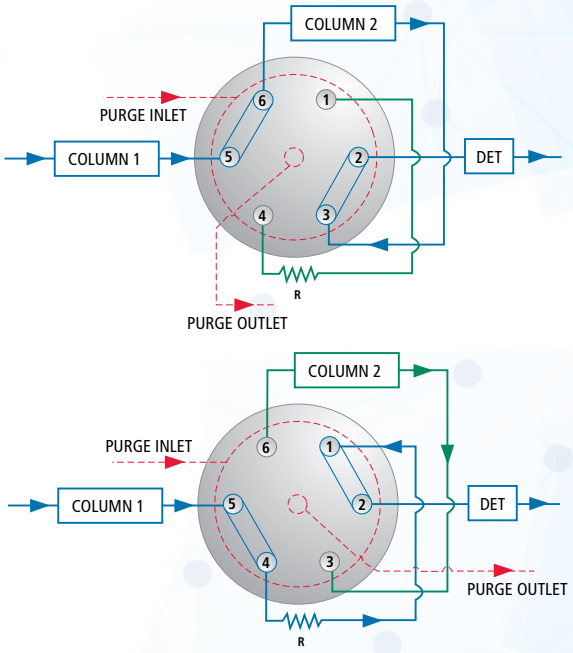

Figure 6: Column isolation - method 2

FOREFLUSHING

Column isolation allows maximum flexibility when multiple columns are being needed, as components can be stored until the most appropriate time in the analytical cycle. There is the danger, however, that if the isolating valve is not completely leak-tight, stored components may leak out or small amount of air may leak in. The µInProve PLSV eliminate these problems. Foreflushing has been described by Willis (1978) as a way of avoiding this. Figure 7 shows the use of a six-port valve for this purpose.

After sample injection, and when the lighter components have been passed on to column 2, the valve is rotated, and the heavier components eluted directly from the column 1 to the detector. The lighter components, having been separated on column 2, now pass through column 1 for a second time and are then detected. Provided that they have been sufficiently well separated on column 2, the small extra amount of peak broadening caused by this second passage through column 1 is insignificant. Column lengths must be selected so that there aren't any overlaps of components.

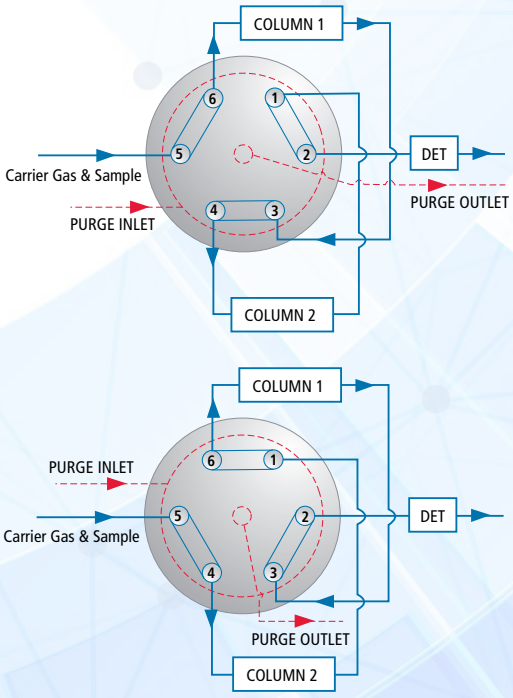

Figure 7: Foreflushing

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MULTIPLE FUNCTIONS

When an analysis requires more than one of the above operations, or a combination of any of these with sample injection, a series of valves can be used. Alternatively, where two functions can occur at the same time, they can be dealt with by a single multi-port valve. Some typical examples follow.

Gas sampling and backflush to detector

An eight-port valve with 45° rotation is used as shown in Figure 8. The left hand position shows the sample being loaded into the loop. The right hand position shows the sample being injected and flowing through the column in what would be considered to be the forward direction. Components separated in this way continue on the further columns or to the detector. When the valve is returned to the left hand position, the column is backflushed and the sample loop returned to the load position.

The timing of the backflush operation is dictated by the requirements of the analysis. The time at which the sample loop is returned to the load position is not critical, and so the fact that these operations necessarily occur simultaneously is not disadvantage.

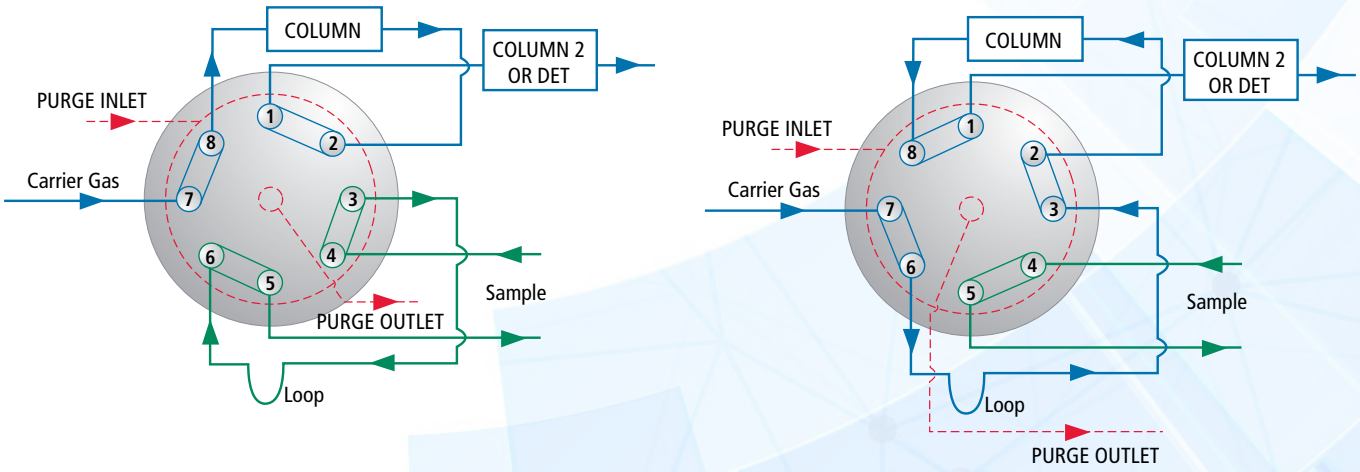

Figure 8: Gas sampling and backflush to detector

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MULTIPLE FUNCTIONS (CONT'D)

Gas sampling and backflush to vent

A ten-port valve is used with an auxiliary supply of carrier gas as shown in Figure 9. Restrictor 1 is adjusted to have the same resistance as with column 1, and restrictor 2 to the same resistance as any downstream columns.

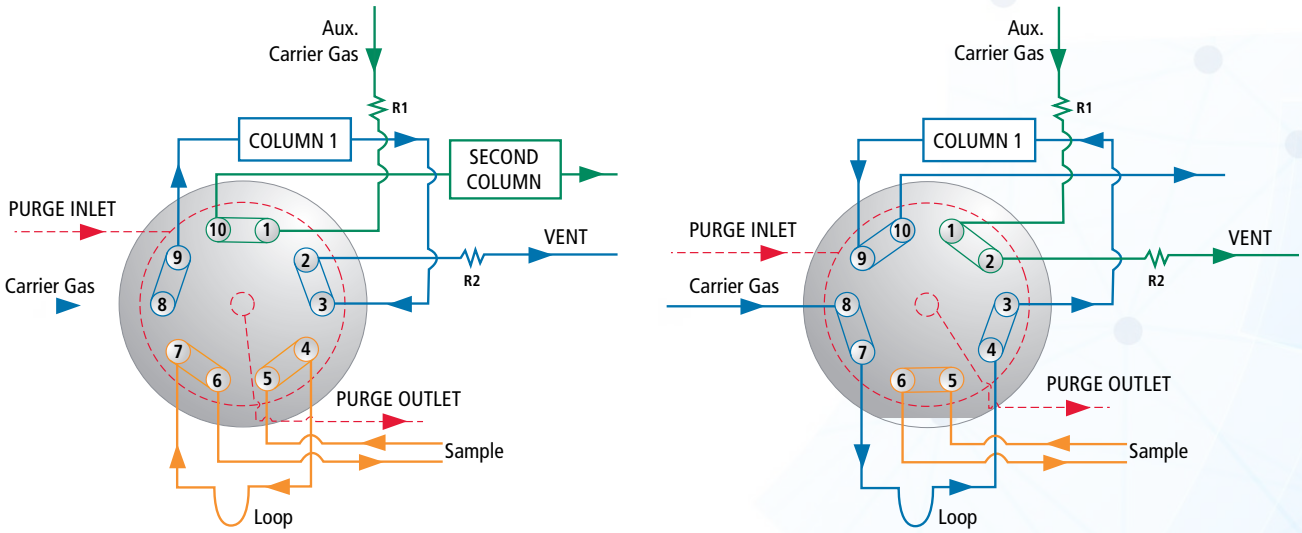

Figure 9: Gas sampling and backflush to vent

Gas sampling and sequence reverse

A ten-port valve combines these functions as show in Figure 10.

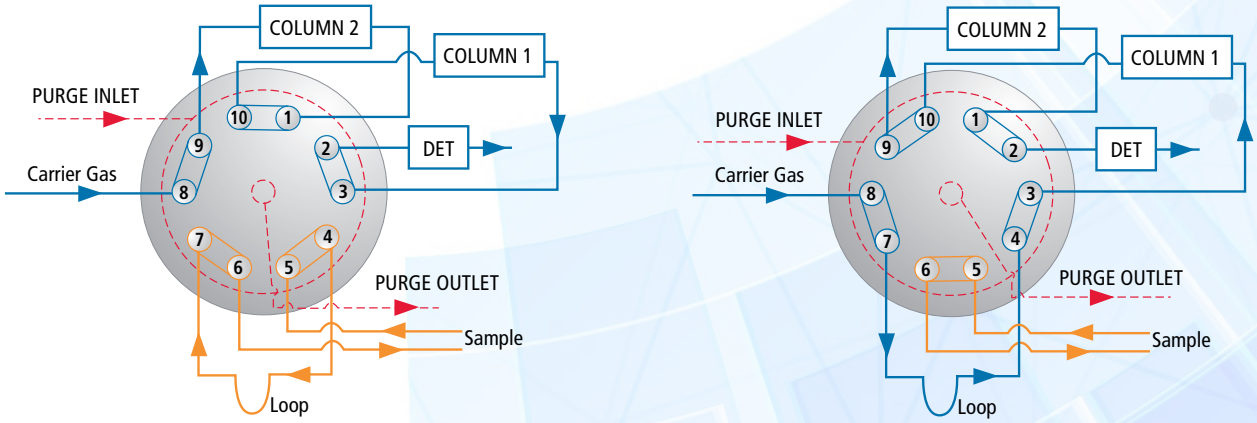

Figure 10: Gas sampling and sequence reverse

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GROUP ISOLATION / ENHANCE SELECTIVITY

This configuration uses two different types of columns and two different sizes of sampling loops. Normally SL1 is smaller than SL2. This allows to transfer a poorly resolved peaks from column 1 to SL2, and then re-injects SL2 into column2 that has appropriate separation for the peaks of interest.

This works better than standard heartcut since SL2 is quickly injected into the column 2, reducing peak broadening and improving separation. It may also reduce the load on column 2 by first pressurizing SL2 before injecting it.

This is simply a matter of timing. Optionally an auxiliary detector could be added on V2 vent line for parallel measurement of impurities resolved by column 1. The addition of V4 allows fast tuning of the system, this could be useful for LAB GC where the configuration is frequently changed.

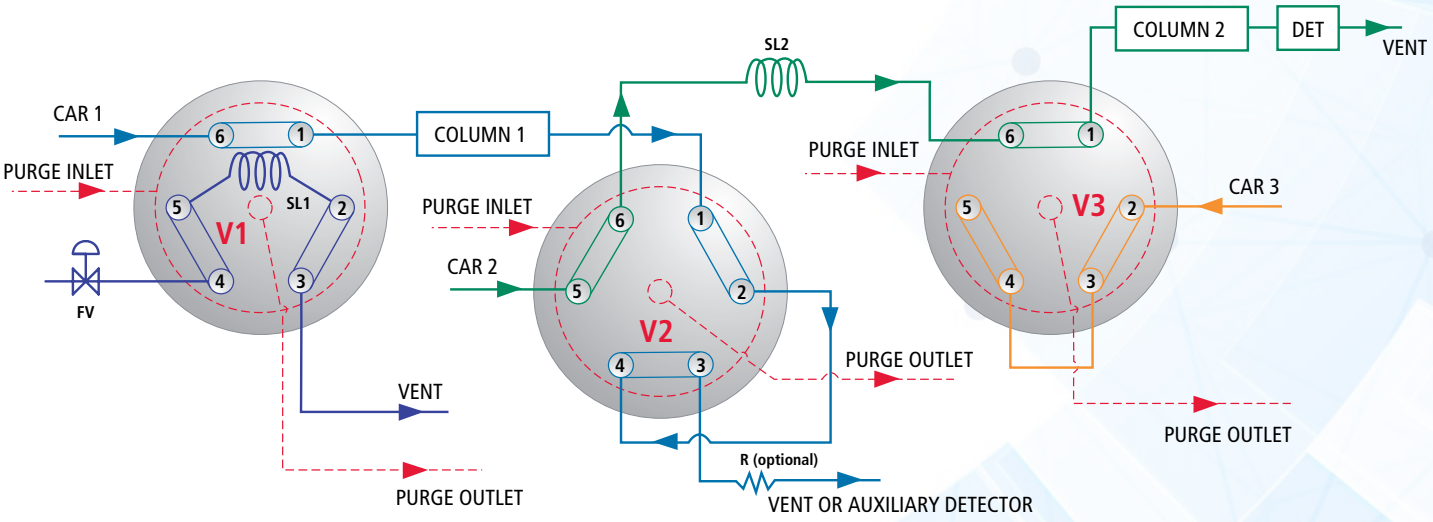

Figure 11:
- Filling SL1
- Injecting SL2 on column 2
- V3 port #5 plugged

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GROUP ISOLATION / ENHANCE SELECTIVITY (CONT'D)

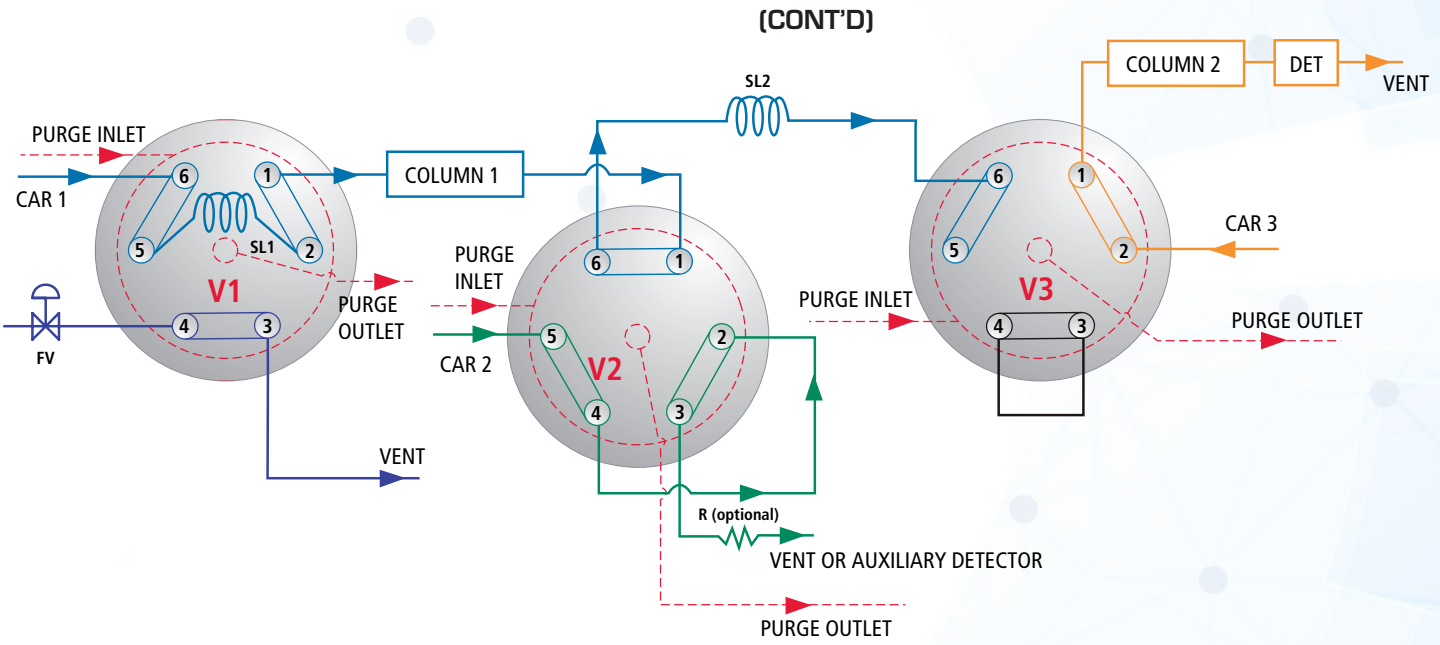

Figure 12:
- Filling SL2 with the selected peak or group of column 1
- Injecting SL1 on column 1

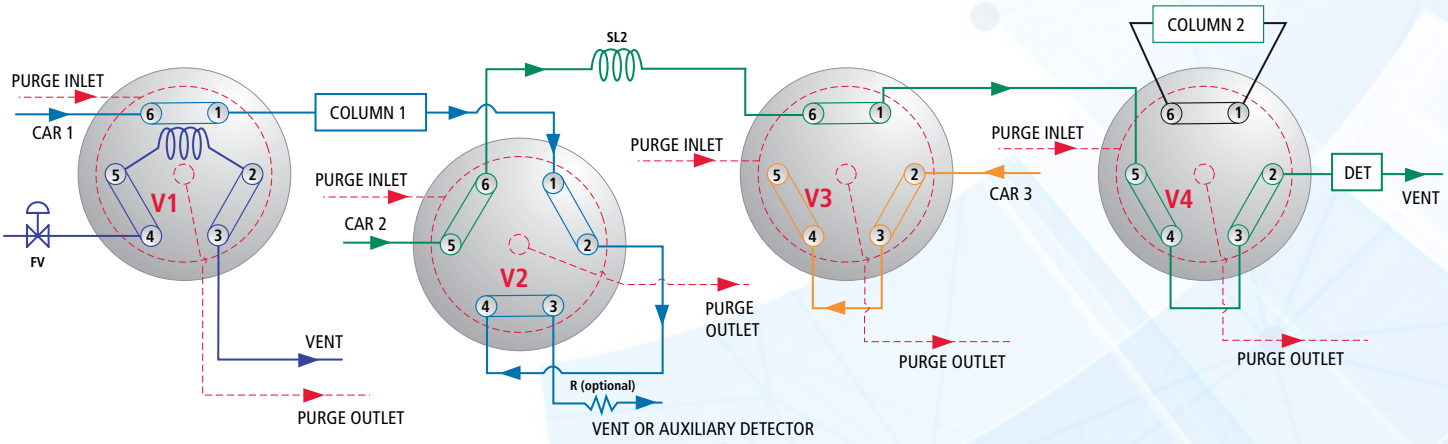

Figure 13: **Configuration variation**
Here V4 is added to allow column 2 by-pass.
This is a useful tool to allow proper timing FV3, in order to synchronize V3 operation.

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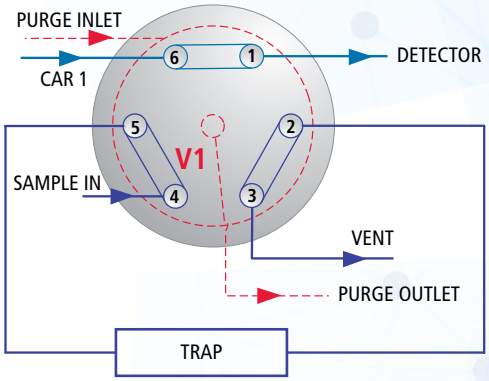
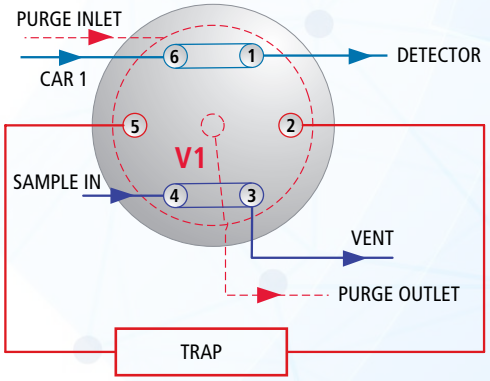
PURGE AND TRAP AND SAMPLE CONCENTRATION

Purge and trap and sample concentrator systems are all designed around the same 2 steps sequence principle. During a first step, a 6 ports valve is used to accumulate the sample. Following a period of time, the valve is actuated in order to release the accumulated sample by heating the trapping material. As the trap has a thermal mass, heating is not instantaneous. The result is a slow and gradual release of the accumulated sample. The sample is consequently not released in a sharp and focused pulse. It is well known in chromatography that injecting the sample rapidly is important in order to obtain good chromatography and good results.

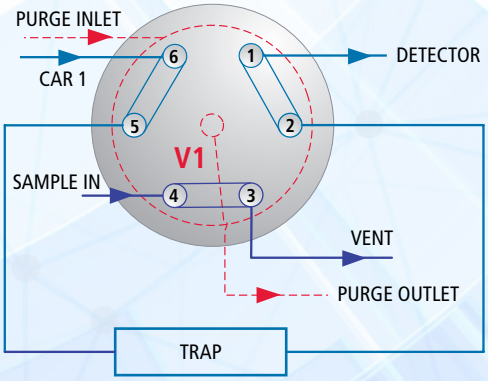


SAMPLE TRAPPING / CONCENTRATION

Our unique valve design allows to do the process in 3 steps. An additional trap isolation step is introduced. To do this, the valve is positioned in an intermediate position. In that position, both carrier and sample flow continue to circulate normally while the trap is being isolated. During this isolation phase, the trap is heated until it reaches the optimum release temperature. Once the temperature is reached, the valve position is changed in order to release the sample in a short and focus pulse. The sequence is then repeated for another analysis.



SAMPLE ISOLATION



SAMPLE RELEASE

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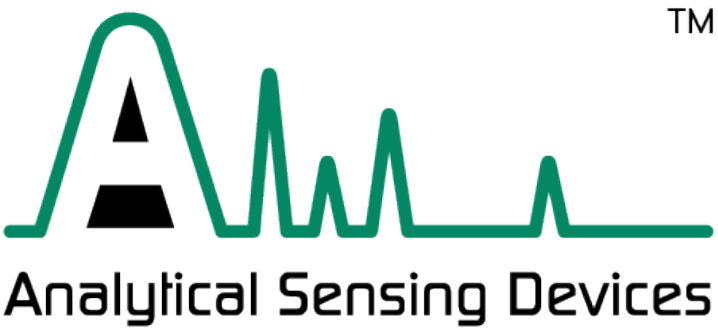

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