Product Bulletin 102

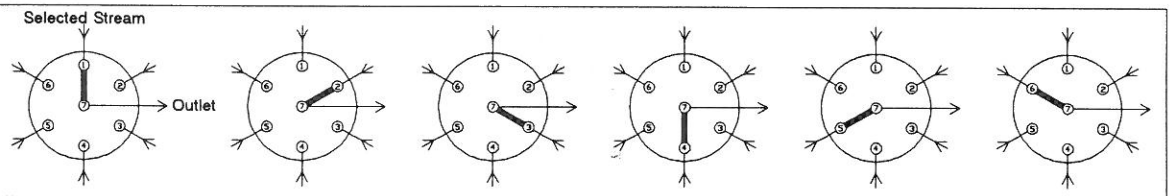
Valco Instruments Co. Inc.

LC Applications for In-Line Rotary Valves

These applications are valid for all types of in-line rotary valves. Generally, substituting a single 8 or 10 port valve for a multiple 6 port system reduces extra column volumn and minimizes the maintenance, service, and risk of leaks. A single valve also costs less than a multiple valve system.

MULTIPOSITION VALVES

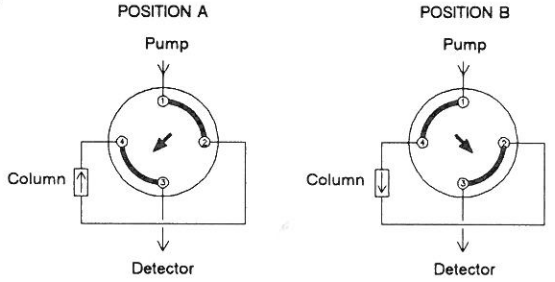
6 POSITION
Also available with
4, 8, 10, 12
and 16 positions



A multiposition valve allows selection of any one of four, six, eight, or ten dead-ended streams, depending on the valve in use. One stream at a time flows to a sample valve, pressure sensor, detector, etc. This configuration may also be used for directing one stream to a number of outlets in applications such as fraction collection. For information about other types of multiposition configurations, refer to pages 26-29 in our catalog.

4 PORT SWITCHING VALVES

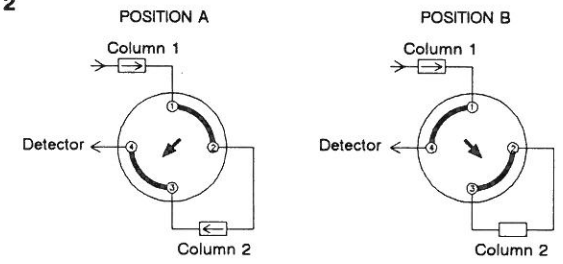
1



Backflush to Detector

Operation of the valve after the last resolved peak of interest causes heavy, slowly eluting compounds to be eluted rapidly from the column to the detector by flow direction reversal. The late eluting compounds may be determined as a group.

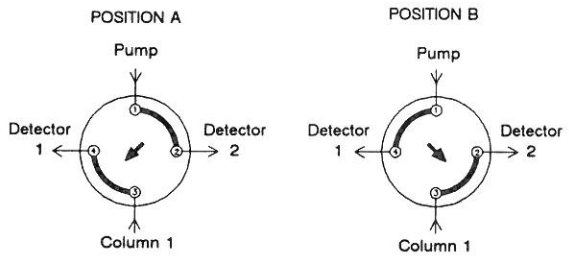
2



Column Isolation

A 4 port valve permits isolation of a column to prevent compounds emerging from an upstream column from being irreversibly retained.

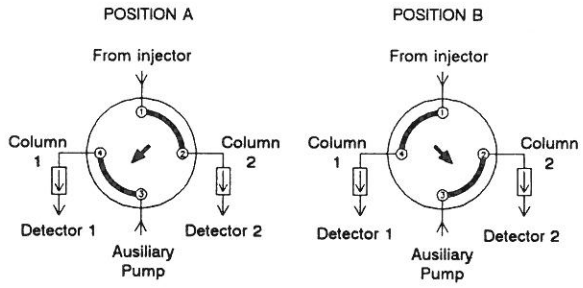
3



Detector Selection From Two Columns or One Column and Auxiliary Pump

This unique configuration allows analysis of different parts of one analysis with two different detectors, without multiple injections.

4



Column Selection with Maintained Flow - Two Detectors

Sample may be introduced to the selected column while flow is maintained to the unselected column.

6 PORT INJECTORS AND SWITCHING VALVES

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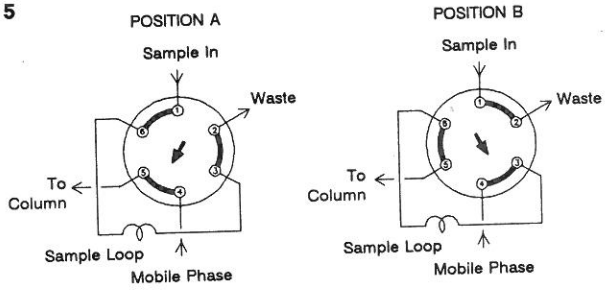
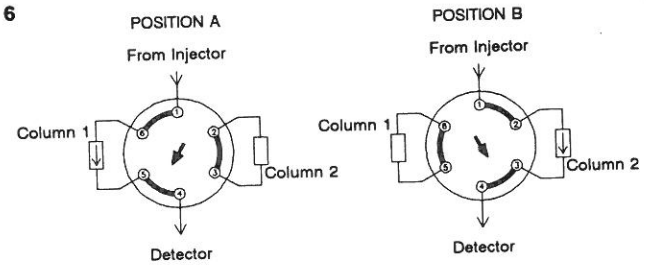


Sample Injector

With the valve in Position A, sample flows through the external loop while the mobile phase flows directly through to the chromatographic column. When the valve is switched to Position B the mobile phase flow is diverted through the loop, displacing the sample contained in the sample loop and valve body and injecting it into the column.

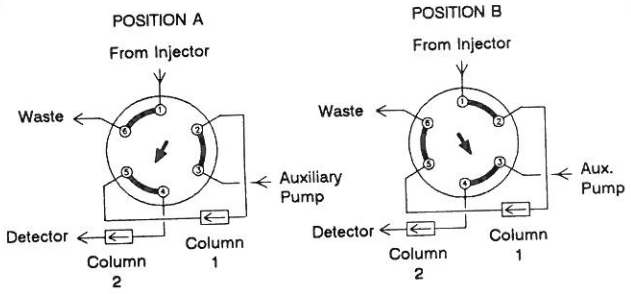
6



Two Column Selection

When two different columns are required at frequent intervals at similar oven temperatures, a 6 port valve can provide rapid selection of the one to be used. The column not in use is protected by a blanket of carrier gas and may be rapidly brought to equilibrium when required. (If flow must be maintained to the non-selected column, an 8 or 10 port valve is required.)

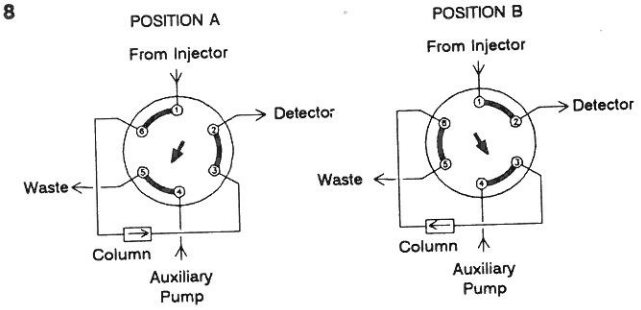
7



Heart Cutting or End Cutting to a Second Column

Certain applications require analysis only of a single heart or end cut, while venting the early eluting components. In Position B, the sample is injected into Column 1 and the front end elutes to vent. Position A directs the heart cut or end cut into Column 2 and the detector. The valve is returned to Position B (if heart cutting) to elute the end cut from Column 1 to vent.

8



Backflush to Vent

Use of this arrangement permits "dumping" of late eluting components which need not be quantitated and which may interfere with a detector's operation. (Example: water with an electron capture detector.) If a large change in pressure drop is anticipated when the column is switched in and out of the carrier flowpath, the related 8 port application should be used.

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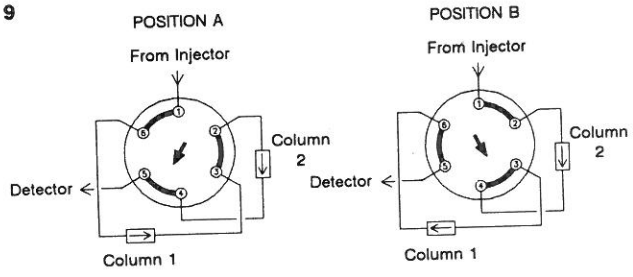

Two Column Switching with Backflush of First Column

This configuration, often done with a 10 port valve, allows backflushing of late eluting components into a detector prior to the emergence of the first peak. The valve is switched after all components of interest are in the second column. Since the components in the second column must make a second pass through the first column, there is time for the backflush peak to emerge early.

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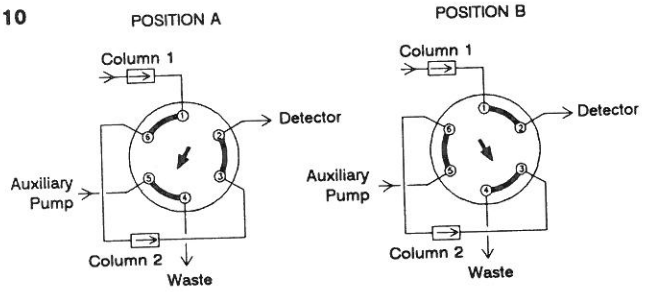

Column Addition

This arrangement shows how a valve can be used to provide flexibility in column selection. In this example, a 30 cm. column is usually required, but if additional length is needed, a simple rotation of the valve adds an additional 30 cm. without removing any connections.

8 PORT INJECTORS AND SWITCHING VALVES

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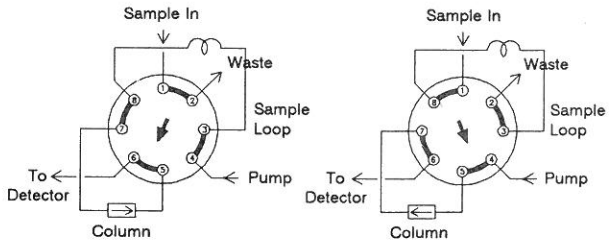

Loop Sampling with Backflush to Detector

One valve functions as sampling and backflush valve, simplifying operation and reducing cost. When components of interest are detected, the strongly retained components are backflushed and removed from the column without temperature programming.

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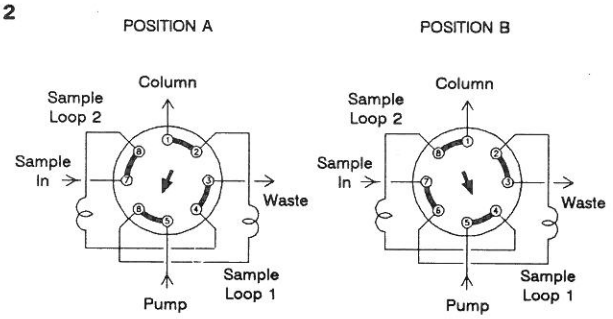

Dual Loop Sampling

In this mode a sample is injected each time the valve turns. The loops may be different sizes for simultaneous analyses of dilute and concentrated samples.

10 PORT INJECTORS AND SWITCHING VALVES

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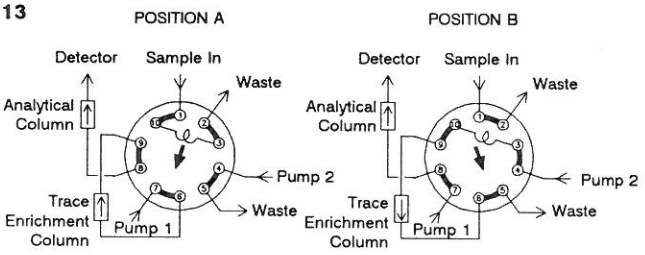
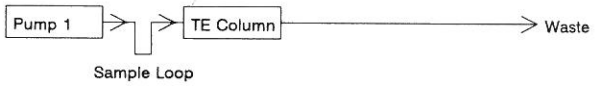
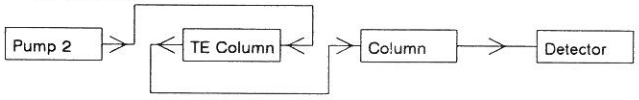


A. Fill sample loop with waste water.
B. Inject sample into trace enrichment column to waste.

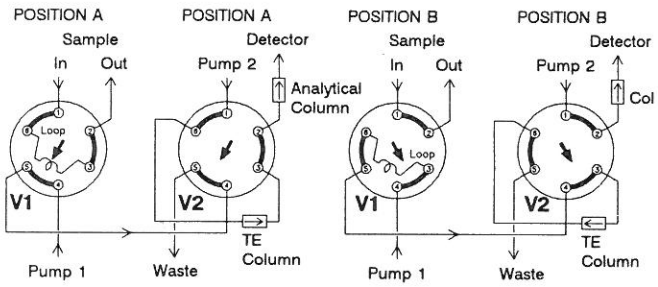


A. Backflush trace enrichment column to analytical column to detector.



Sample Introduction with Trace Enrichment Followed by Backflush of Pre-column

Sample cleanup and separation can be done with two 6 port valves as shown below. However, such trace enrichment can also be accomplished using one 10 port valve as shown at left. In both cases, a desired material is isolated by sorption onto the head of a short LC column. The desired material is backflushed into a second column where separation occurs. In the diagram at left, the sample loop is filled in Position A. The valve is moved to Position B, and Pump 2 displaces the sample from the sample loop into the short trace enrichment column. The valve is returned to Position A and Pump 1 uses a mobile phase to backflush the trace enriched material from the short column to the analytical column where the trace enriched material is separated. During the separation, the sample loop can be filled for another trace enrichment.

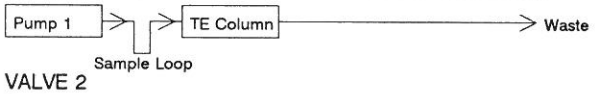


VALVE 1

A. Fill sample loop with waste water.

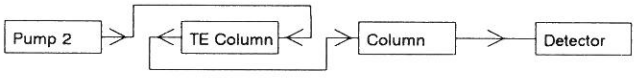
VALVES 1 and 2

B. Inject sample into trace enrichment column to waste.



VALVE 2

A. Backflush trace enrichment column to analytical column to detector.



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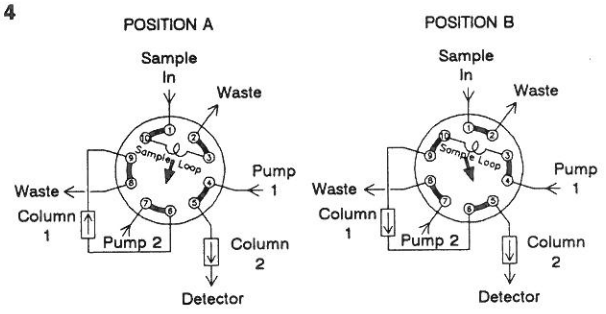
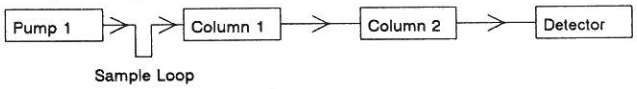


Front Cut to Column 2 and Detector

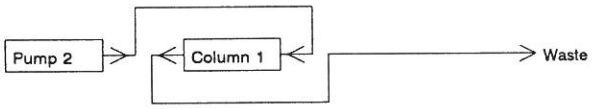
The 10 port valve can be used to inject a sample into the LC column and then to direct the front, heart, or end cuts to a second column, to waste, or to a detector.

Although this procedure has been reported with two 6 port valves, the total operation can be done with a single 10 port valve as shown. If the heart or end cuts are retained strongly by Column 1, it may be necessary to backflush them from Column 1 to waste.

A. Fill sample loop.
B. Inject sample into column 1 to column 2 to detector.



A. Column 1 end cut backflushed to waste.



Column 1 front cut into column 2 to detector.

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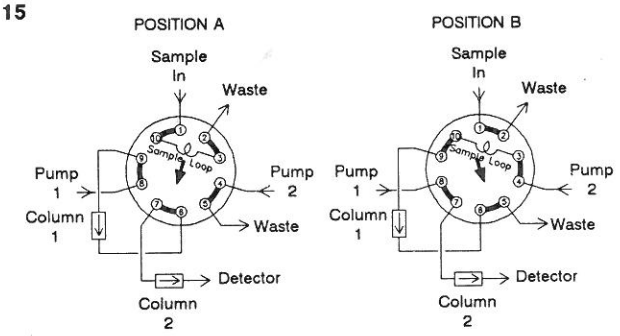
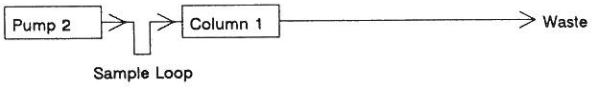


Heart Cut to Column 2 and Detector

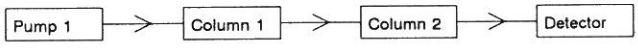
The 10 port valve can be used to inject a sample into the LC column and then to direct the front, heart, or end cuts to a second column, to waste, or to a detector.

Sample injection and heartcutting can be done with one 10 port valve as shown. Both early and late eluting fractions are discarded.

A. Fill sample loop.
B. Inject sample into column and front cut to waste.

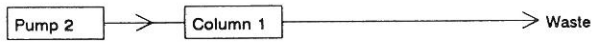


A. Heart cut to column 2.



B. Heart cut from column 2 to detector.

End cut to waste.



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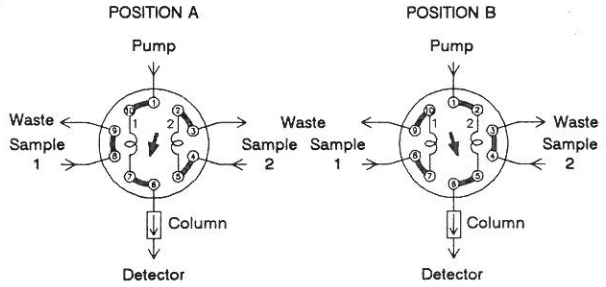
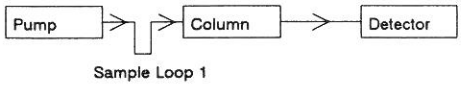
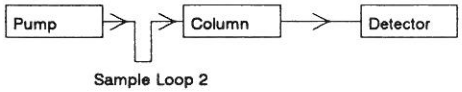


A. Fill sample loop 2 with sample 2.
Inject sample loop 1 into column.



A. Fill sample loop 1 with sample 1.
Inject sample loop 2 into column.



Random Access of Two Different Loops to One LC System

Using a 10 port valve as an injector with a single column permits injection from either of two loops, which may be identical or of different sizes. A single mobile phase, column, and detector are employed. This technique replaces a 4 port selector valve and 6 port sample injector. Since the samples always alternate, this flowpath is usually employed in automatic, unattended operation. Or, one loading port can be connected to an autosampler and the other used as a manual loading port, or two syringe loading ports may be installed.

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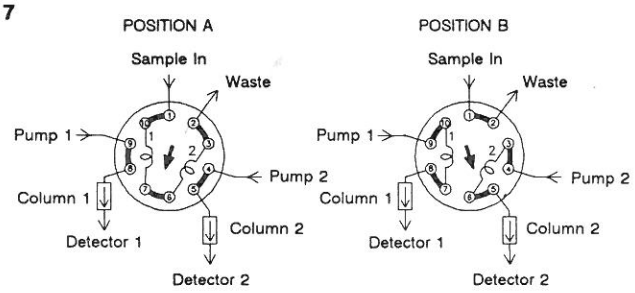
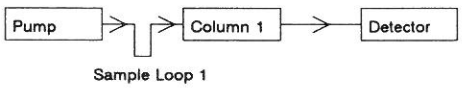
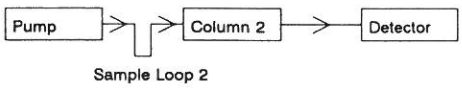


A. Fill sample loop 1 and sample loop 2 with same sample.
B. Inject sample loop 1 into column 1 to detector 1.



Inject sample loop 2 into column 2 to detector 2.



Simultaneous Injection of the Same Sample Into Separate Columns

A single loading of two loops introduces samples into two separate flow systems which may have different mobile phases, columns, and detectors. This technique loads both sample loops in series (Position A) followed by injection at the same time (Position B) of both loops into two separate columns. A single auto-sampler used with this flowpath can automate two analytical procedures for the same sample. In an important non-chromatographic application, the roles of carrier and sample are reversed, permitting two different quantities of two different materials to be dispensed together, as in automatic dilution.

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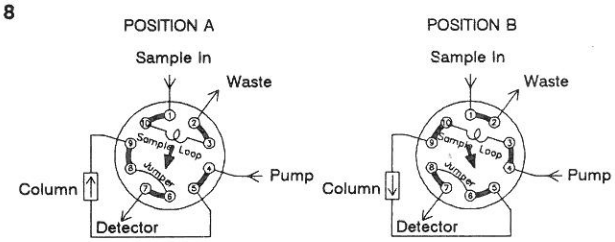
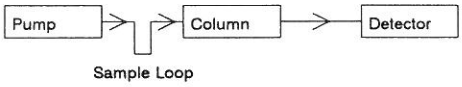


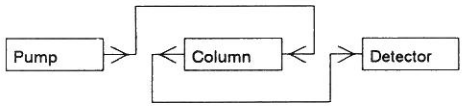
Loop Sampling with Backflush to Detector

Sample injection into an LC column, followed by backflushing of the column, is one of the simplest examples of column switching. After all the early eluting components of interest have been detected, the remaining components are backflushed to the detector and measured as a composite peak.

A. Fill sample loop.
B. Inject sample into column.



A. Backflush column to detector.



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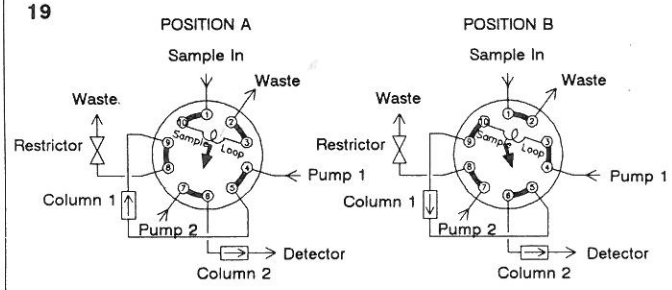
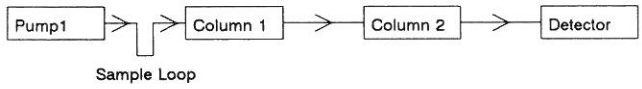


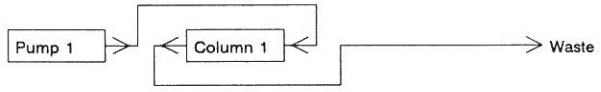
Loop Sampling with Backflush of Pre-column to Vent

Often, the components of interest in a sample will be those which are early eluting. This method allows strongly retained components not pertinent to the analysis to be backflushed to vent. After the sample loop has been loaded in Position A, the valve is switched to Position B to inject the sample into Column 1. As soon as all components of interest have entered Column 2, the valve is switched back to Position A. Column 1 is backflushed to vent during the analysis, thus reducing the total analysis time.

A. Fill sample loop.
B. Inject sample into column 1 to column 2 to detector using Pump 1.



A. Pump 2 to column 2 to detector.



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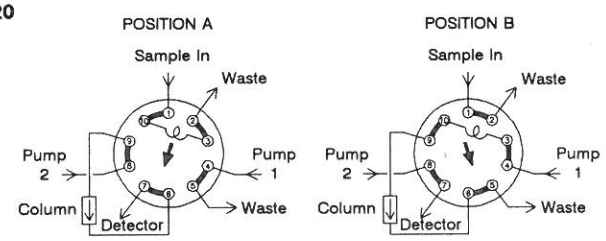
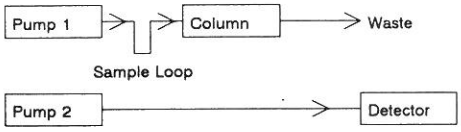


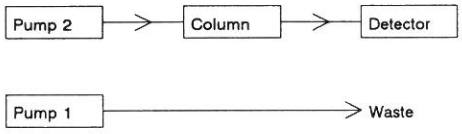
Loop Sampling with Foreflush to Vent, Same Carrier to Detector in Both Modes

Certain applications require analysis of only the strongly retained minor components in a low boiling matrix. The venting of a solvent peak to protect a detector is a common example. The foreflush technique vents the early major component while the loop is in series (Position B) with the column. When the valve is switched to Position A, Mobile Phase 2 drives the remaining components through the column for analysis. Note that the column is foreflushed to vent in Position B by Mobile Phase 1 and foreflushed to detector (Position A) by Mobile Phase 2.

A. Fill sample loop.
B. Inject sample into column 1 to waste using Pump 1. Pump 2 maintains flow to the detector.



A. Pump 2 to column to detector. Pump 2 to vent.



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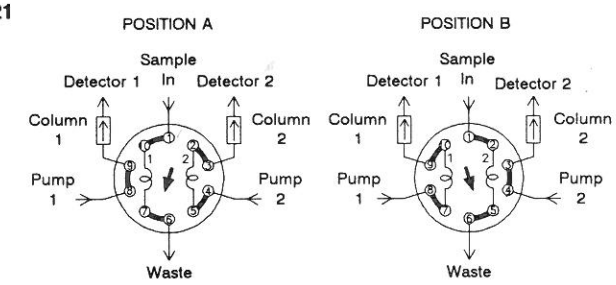
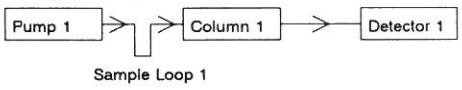


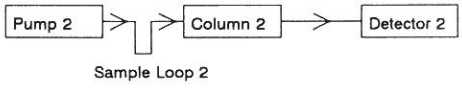
Random Access Injection Into Two Separate LC Systems

This configuration utilizes one 10 port valve to serve two different columns and detectors. Each column can be randomly accessed by introducing the sample in the appropriate valve position. Injection may be made repeatedly into the same column since the unused loop only contains mobile phase.

LC System 1
A. Fill sample loop 1.
B. Inject sample loop 1 into column 1.



LC System 2
B. Fill sample loop 2.
A. Inject sample loop 2 into column 2.



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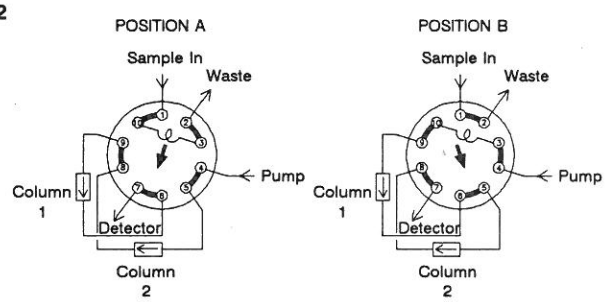
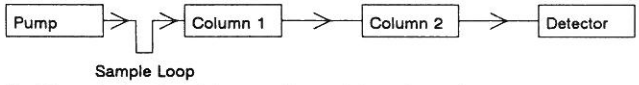
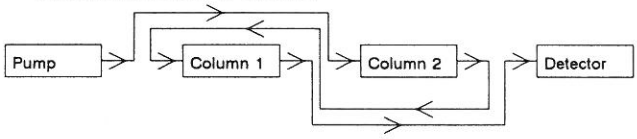


A. Fill sample loop.
B. Inject sample into column 1 and column 2.
Front cut to detector.



A. Recycle heart cut from column 2 to column1.
Column 1 end cut to detector



Recycle

Recycling can be used to give increased resolution between components in an LC fraction. The simplest recycling technique consists of connecting the eluent from the detector back to the pump inlet and recycling the eluent through the LC system. However, since this leads to remixing of separated material during each recycle, most recycling is done using a valve to feed the eluent from one column to another. The 10 port injection/ recycling system shown has much lower extra column volume than valve systems previously reported.

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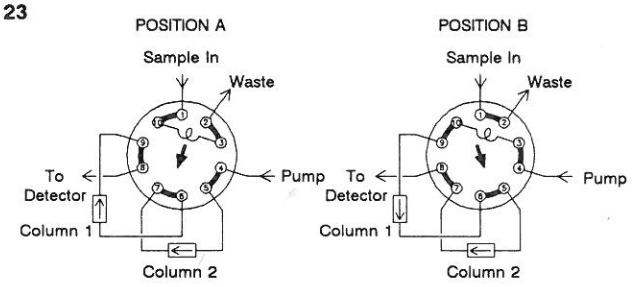
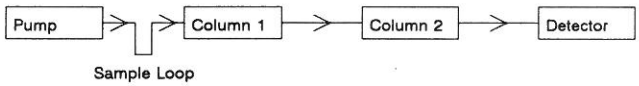
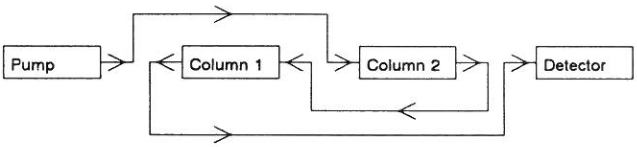


A. Fill sample loop.
B. Inject sample into column 1 to column 2 to detector.



A. Column 1/column 2 sequence reversal with column 1 end cut backflushed to detector



Loop Sampling with Two Column Sequence Reversal and Backflush of Column 1 to Detector

This flowpath is ideal for many natural gas analyses. The valve is plumbed so that a short pre-column (Column 1) is used ahead of a typical long analytical column (Column 2). The sample loop is loaded in Position A. Injection is made into Column 1 when the valve is switched to Position B. The sample is swept efficiently into the column by the pressure/flow "burst" effect caused by reversing the pre-column and moving it upstream of the analytical column at the moment of injection. The light hydrocarbons quickly elute into Column 2 leaving the "heavies" behind in Column 1. The valve is switched at this time, which reverses the flow through Column 1 and reverses the column sequence. The "heavy" fraction is backflushed directly to the detector as a single composite peak which is narrow and easier to quantify than the composite peak obtained by backflushing a long column.

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