PROCESS STREAM SAMPLING
SAFER DESIGNS FOR MANUAL SAMPLING OF LIQUID PROCESS STREAMS

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(Much of the information about to be given was developed from a paper of the same title written for presentation through the American Institute of Chemical Engineers. Requests for copies of the original paper should be handled through that organization.)

Taking process samples is one of the most common daily operations used in the chemical industry. It can be an activity which may pose a significant hazard to personal safety, if done improperly. Yet, it often received little attention in the design and operation of chemical processing plants. With increasing emphasis on industrial safety, coupled with a heightened awareness of the hazards of handling chemicals, process sampling can no longer be a haphazard or poorly planned operation.

There is a growing need to develop a reasonable approach to safer sampling which can cover a diversity of sampling situations. I will limit my comments to liquid line sampling, however the approach can be applied to other types of sample systems.

Safe sampling involves a careful balance of design and procedure. The design is important, for it can help reduce the potential risks involved. Design implies a systematic definition of the process requirements, and an understanding of the hazards and conditions under which the sample must be obtained. Only with this information in hand, can the proper equipment and procedure be developed to reduce the risk of exposure.

Whether designing for sampling in a new process, or an existing plant, the method is the same. First, the sampling needs of the process must be anticipated and understood. The reasons for taking samples can be grouped into four areas:

- to establish a material balance
- to evaluate process operations
- to trouble-shoot equipment
- to aid in product quality control

Often, by examining the need for a particular sample, we can establish the type of information the sample should provide.

The diverse conditions under which samples may be taken often compound the problems of safe handling of hazardous materials (Refer to Table 1). The interaction of these problems may greatly affect the complexity of the sampling equipment, and the procedures used.

The second step is to interpret each sample point as a distinctive piece of equipment - a piece of hardware which must provide two functions: to obtain the desired process sample; and to protect the individual during the procedure.

This is helped by the use of a specification or data sheet as shown in Figure 1, for each sample location. Each sample point in the process is assigned a unique equipment number, and has its own corresponding design document. On this sheet are summarized the operating conditions for each sample. The definition of these process conditions should be done with the same deliberation that would be used for a pump or a heat exchanger. The specification sheet is used to organize the thinking. It asks specific questions to assist in defining the needs of the sample equipment. The items on the specification sheet are by no means all inclusive, they are simply to act as a focus for evaluating some of the pertinent facts for each sample location. Let's take a closer look at some of its major areas.

This first section is used to help define the hazards asociated with a particular sample material. In it is noted whether the material is lethal, toxic at high temperature or pressure, or is flammable. Space is provided for key items of data. The limits as to the degree of hazard are completely arbitrary. They may be changed according to the interpretation of the engineer, or to reflect the practices of a particular production unit or company.

The second sector focuses on some of the specific conditions involved in taking the sample, such as why is it in, how much and how often is it to be taken. Sample size is very important - it should reasonably match the requirements of the end use. There is no need to take eight ounces of material if only five microliters are required for an analytical procedure. Oversize samples simply increase the potential for exposure, and may create handling problems when finally disposing of the excess. Likewise, the size and material of the sample container should be checked to be compatible with the procedure and process materials. Some materials may also warrent comments regarding environmental toxicity and related handling precautions.

The third major section summarizes some of the other special handling conditions which may be necessary to get a good, safe sample. Foaming, viscosity, chemical reaction, heat treating and ventilation are just some of the items which could have a significant impact on the overall design of the sampler system. At times, characteristics of the materials or the analytical procedures may impose further special handling considerations. All of these factors may require a specialized design that is unique to that particular process sample.

Table 1. Materials and conditions affecting process sampling.

Consider both type of material and physical conditions. Plan procedures to meet needs.

Solid Liquid Gas	Type of Container
Sensitizer Toxic Corrosive	Isolate
Flammable	Ventilate Inert Atmosphere
Hot/Cold Pressure/Vacuum	Isolate & Readjust to ambient conditions by special handling
Reactive Chemical	Quench/Inhibit
Contamination	Materials/Procedure

PLANT		FILE/JOB NO.
LOCATION	BLDG. NO.	CHARGE NO.
MANUFACTURER	NO. UNITS	B/M NO.
		P.O. NO.

No.	Section	Item				
1	Hazards	Lethal	Yes	No		
2		Hot - Greater than 60°C (140°F)	Yes	No		°C
3		High Pressure - Greater than 30 psig	Yes	No		psig
4		Flesh Burning	Yes	No		
5		Toxic Liquid	Yes	No		
6		Toxic Vapor Fumes Dust	Yes	No		
7		Flammable <140°F Flash Point	Yes	No		°F
8						
9						
10	Conditions	Sample Type	Solid	Liquid	Gas	Other
11		Sample Size				
12		Sample Container	Type	Size	Material	
13		Sample Frequency	/hr	/day	/wk	/mo
14		Boiling Point	°C			
15		Freeze Point	°C			
16		Environmental Toxicity				
17						
18						
19	Special Considerations	Other Sample Considerations:				
20		A. Foaming				
21		B. Flashing				
22		C. Sublime				
23		D. Viscous				
24		E. Reactive Chemical				
25		F. Quench Sample				
26		G. Heat/Cool Sample				
27		H. Ventilation (Positive or Negative)				
28		I. Inert Atmosphere				
29		J. Corrosivity				
30		K. Thermal Shock (Container)				
31		L. Pressure Limit (Container)				
32		M. Other				
33						
34						
35	Materials	Item or Part	Material Information			
36		Process Piping				
37		Sample Valve(s)				
38		Sample Line				
39		Vent Line(s)				
40		Sample Enclosure				
41		Sample Container				
42		Sample Support Plate				
43		Overflow				
44						
45	Installation	Sample Enclosure:				
46						
47		Piping Installation:				
48						
49						
50						

SPEC. BY								EQUIP. NO.
CHECKED:	SERVICE							
APP'D:								SAMPLER SPECIFICATIONS
DATE:	REVISION DATE	A		B		C		SPEC. NO.
VENDOR TO COMPLETE ALL INFORMATION MARKED					SHEET	OF		

Figure 1. Process sampling specification sheet.

The last two sections deal with the details relating to the actual installation of the sample system. Most important are the materials of construction to be used, for both the process piping, and the various components of the sample system. Choosing the proper materials is usually influenced by the corrosivity of the process materials, but construction materials may also effect aspects of sample quality such as color or metals contamination.

Once the requirements or problems related to getting a particular sample have been identified, the job is only half done. There is a large selection of sampling equipment which can be used, no single design may effectively handle all situations. We need to understand the strengths and limitations of each system to choose the one that best fits the needs of utility and safety.

Despite efforts to establish and insist on the use of proper sampling procedures, there is no guarantee that the person in the plant will always wear the prescribed clothing and equipment, or always follow the procedures. Instead of isolating the man, it may be possible to reduce the risk of exposure by isolating the sample in an enclosure. This enclosure has to be simple to use, permit easy visual operation, and offer increased protection of the individual. If it does not fit these guidelines of being simple to use, it probably won't be used at all.

Figure 2 shows a plastic sample enclosure made up in one of our shops. It is a stock design and a few extra units are kept in inventory. It has a Plexiglas® window which provides good visibility of the sample inside the box. The window is easily removed for cleaning or replacement, and usually the plants keep extra precut windows on hand. Inside the box, the perforated floor will support a sample container, and allow any overflow or spills to drain into the bottom. This unit can be piped directly to a spill containment system, or the funnel could be outfitted with a collection bottle instead. In actual operation, the sample piping would extend down through a hole in the top of the box, with the control valves mounted outside. The valves should be positioned so that the person would stand in front of the window, away from the opening while taking the sample. Any splash or spray is deflected away by the sheild along the front edge of the opening. This design is very easy to modify with the addition of a door, exhaust ventilation, rubber gloves, adsorbent materials for spill, or any other features required by the process conditions.

A substantially less exotic design is the simple plywood box shown in Figure 3. The front and rear sliding plastic panels serve as doors. Units such as this are relatively inexpensive, but very effective when used properly.

The enclosure shown in Figure 4 was made from a steel Freon® can. The plastic window is easily removable for cleaning or replacement. This enclosure also has a swing and bottle holder to place the sample bottle

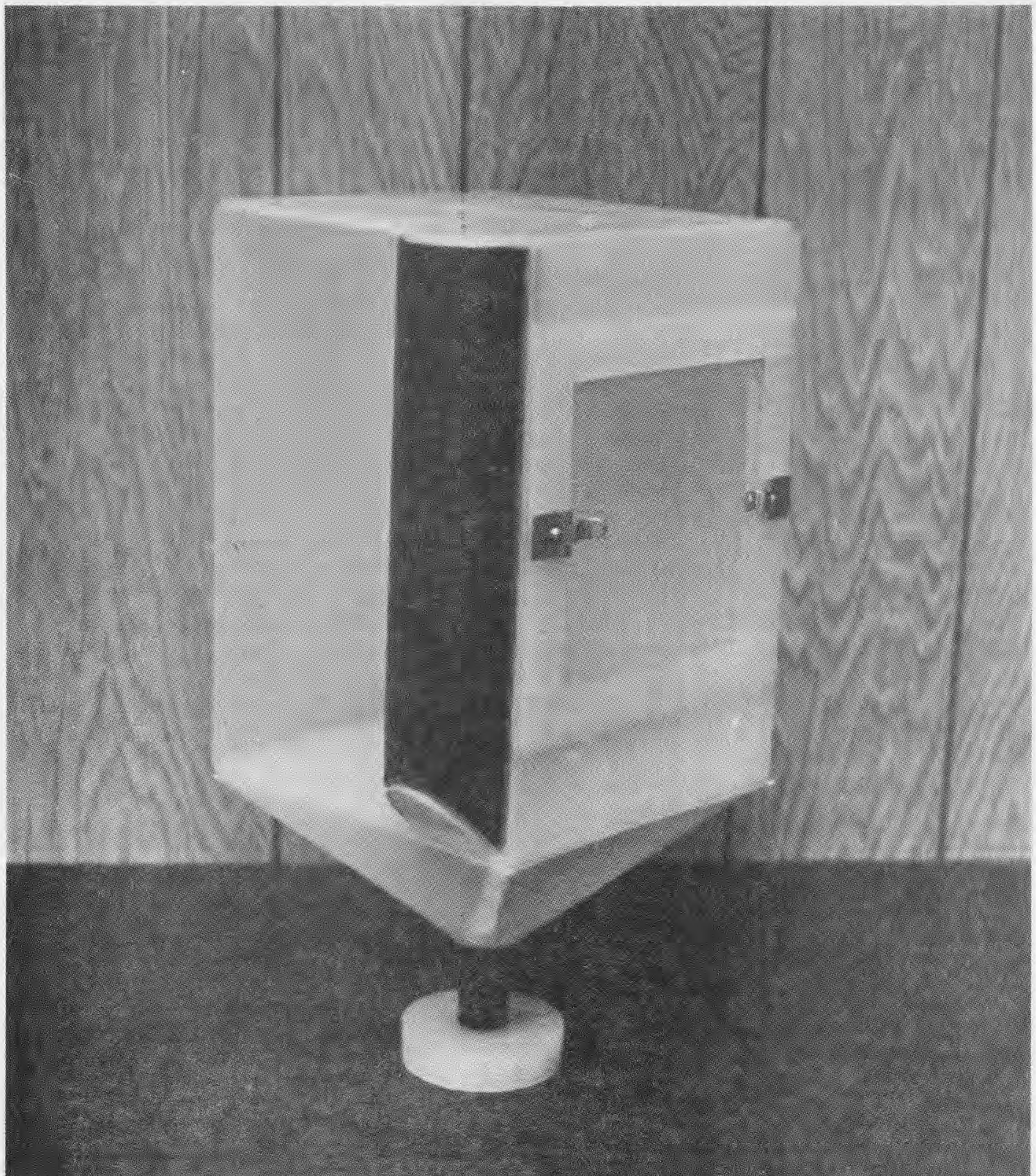

Figure 2. Plastic Sample Box Enclosure.

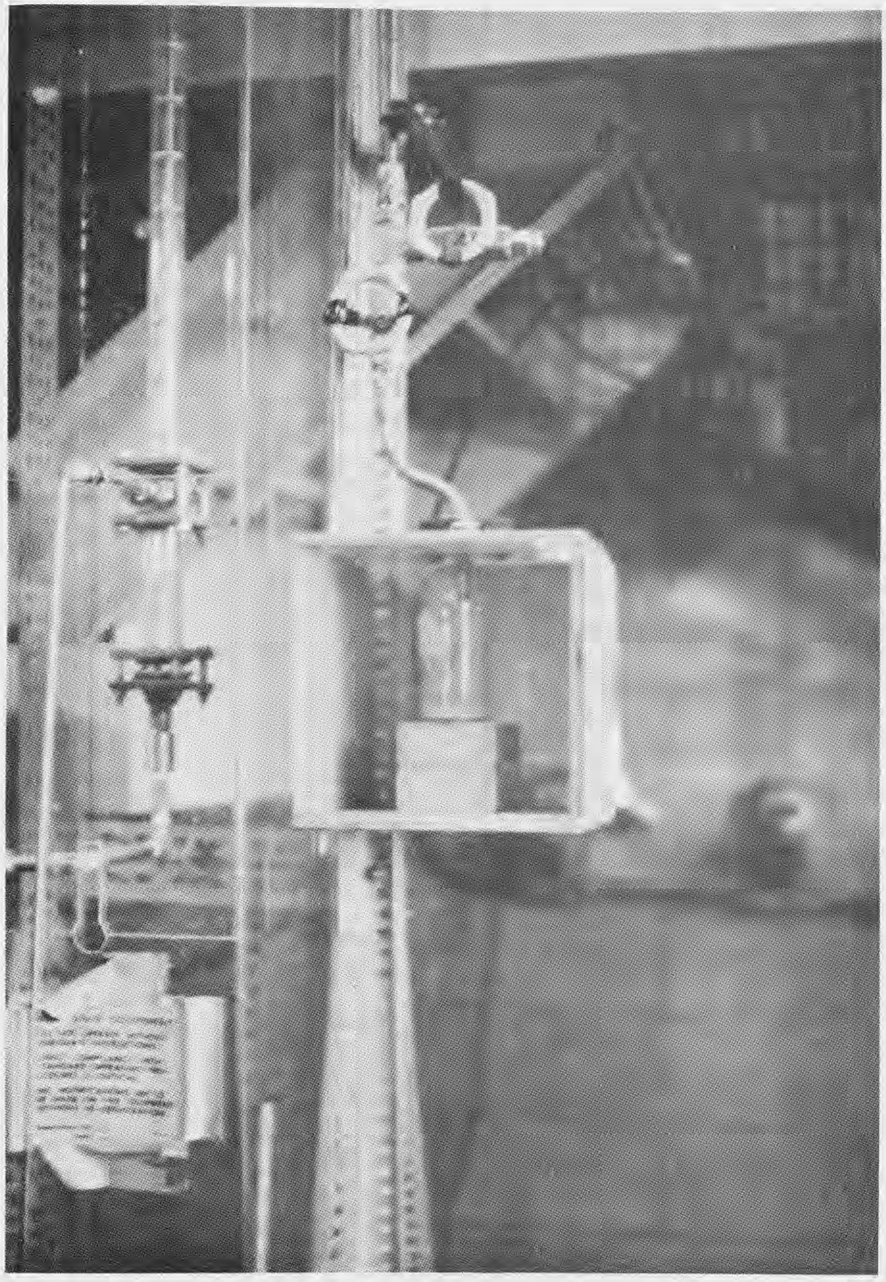

Figure 3. Plywood Sample Box Enclosure.

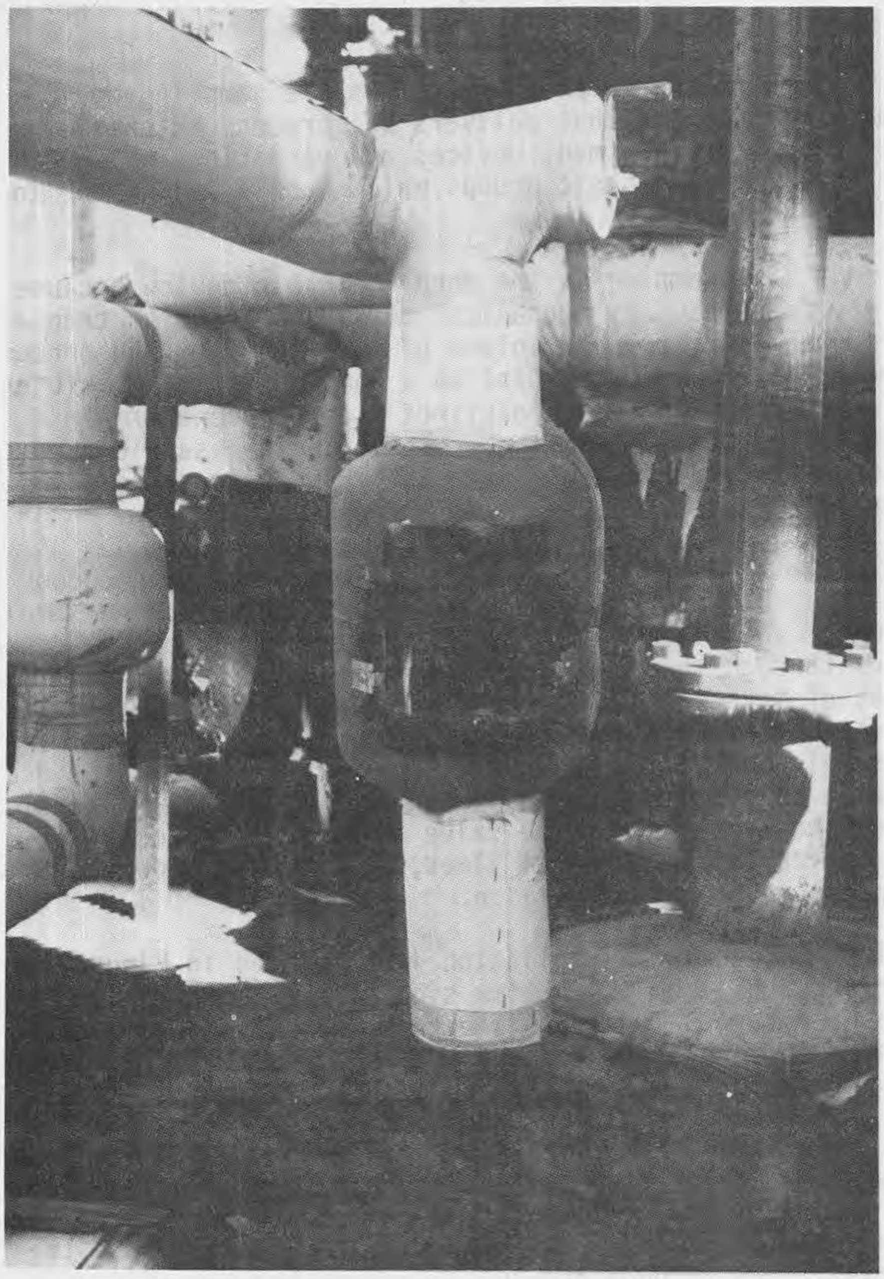

Figure 4. Freon Can Sample Box Enclosure.

in the proper position inside. Metal units such as this may be preferred when high temperatures or chemical solvents may damage plastic materials. The plastic units are effective in corrosive environments. The common key in all of these is simply reduction of risk exposure by containment.

The second part of the sampling system is the sampling device. This is the actual working piece that delivers the process material. Like the sample enclosures, there are many devices and variations which can be used. These fall into two basic groups which I call Direct and Indirect line samplers.

The Indirect line sampler is the more desirable device because of its inherent safety features. By mechanical means, these units trap and isolate a predetermined, precise volume of material from the process line, and then delivers this material to a sample bottle, within an appropriate enclosure. These two distinct steps prevent sample containers at full process conditions. Isolating the sample from the process and restricting the volume improves chances to anticipate and control potential problems.

The simplest of the Indirect samplers is the sampling plugcock shown in Figure 5. The plugcock valve has been modified by adding sample and vent ports to the valve body. In the normal open position the process material flows through the opening in the plug. As the valve is closed, a small amount of material is trapped within the cavity of the plug. When the valve is turned to the full closed position, the plug cavity is open to the sample connection, allowing the trapped material to drain out. This valve delivers a precise, predetermined volume of sample, depending upon the line size of the valve used. The valve may be installed in horizontal or vertical lines, in the main flow, or in a by-pass, in pressure or vacuum service.

An actual sampling plug installation is presented in Figure 6. The sample connection runs from the bottom side of the valve through the top of the sample enclosure. The vent line from the top side of the valve runs down behind the enclosure and connects into the lower part of the funnel, below the support plate. Any spilled or vented materials will be collected in a bottle to be placed under the funnel.

A somewhat more complicated system uses a pair of two position, three-ported valves. This arrangement allows more flexibility in obtaining larger sample sizes. During normal flow conditions, the process material flows through the two valves as shown in Figure 7A. The sample volume is the amount contained within and between the valve plugs. This volume is determined by the length of the piping between the valves. The sample is taken by turning both valves simultaneously. In the 90^{o} position (Refer to Figure 7B), the sample is trapped between the plugs, and is isolated from both process and sample connections. Turning the valves through the full 180^{o} opens the sample ports, and lets the trapped sample drain out.

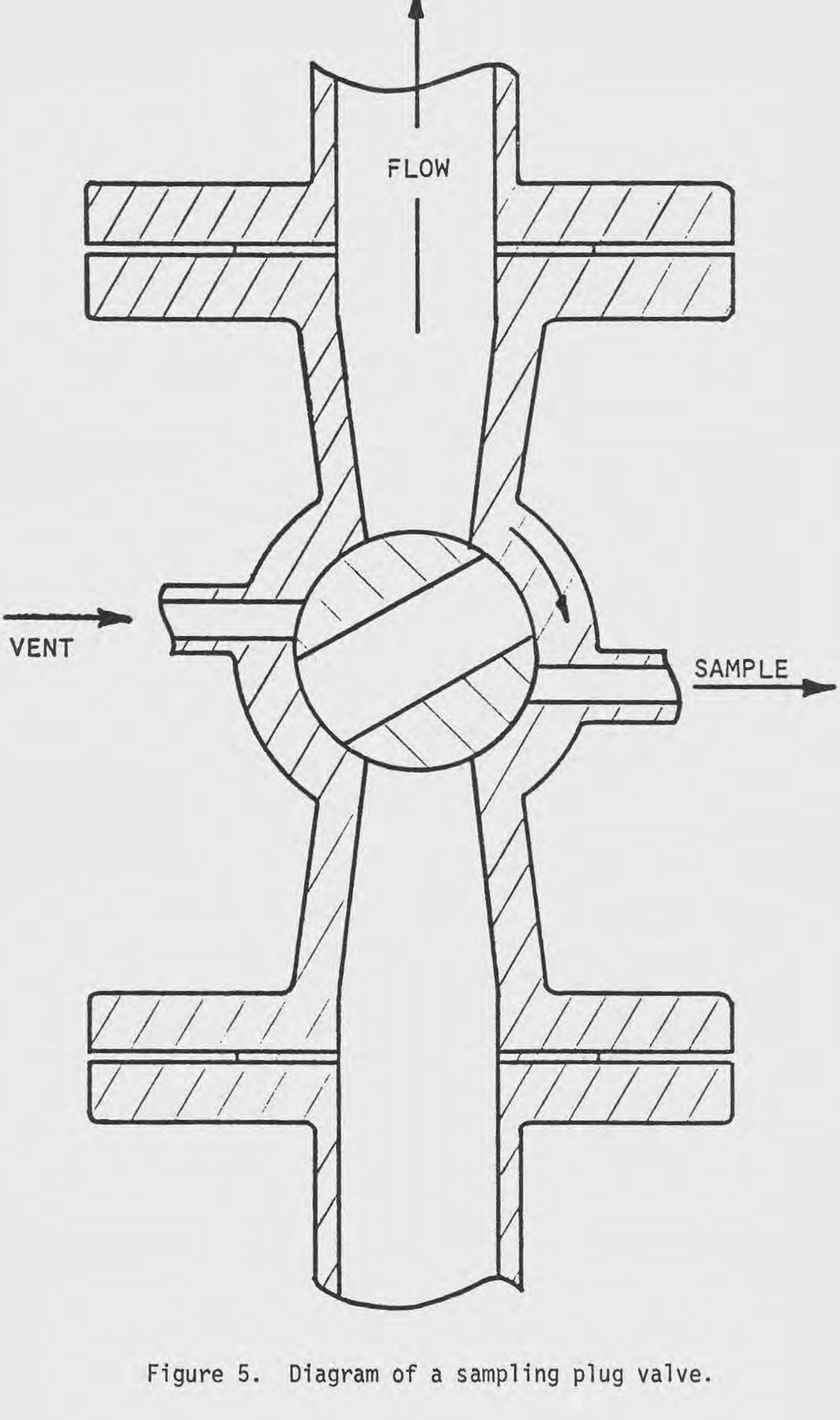

Figure 5. Diagram of a sampling plug valve.

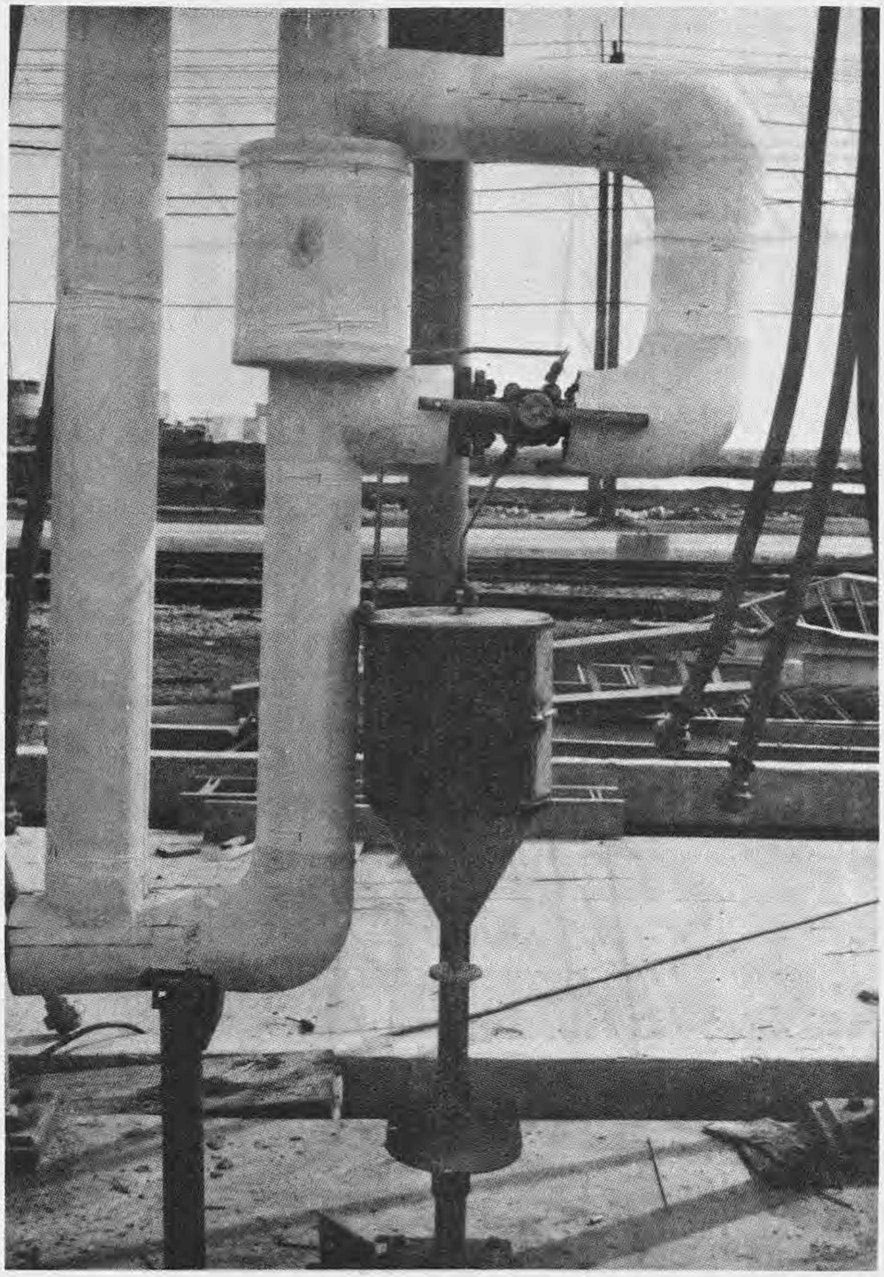

Figure 6. Sampling plug valve, used with a stainless steel sample box enclosure.

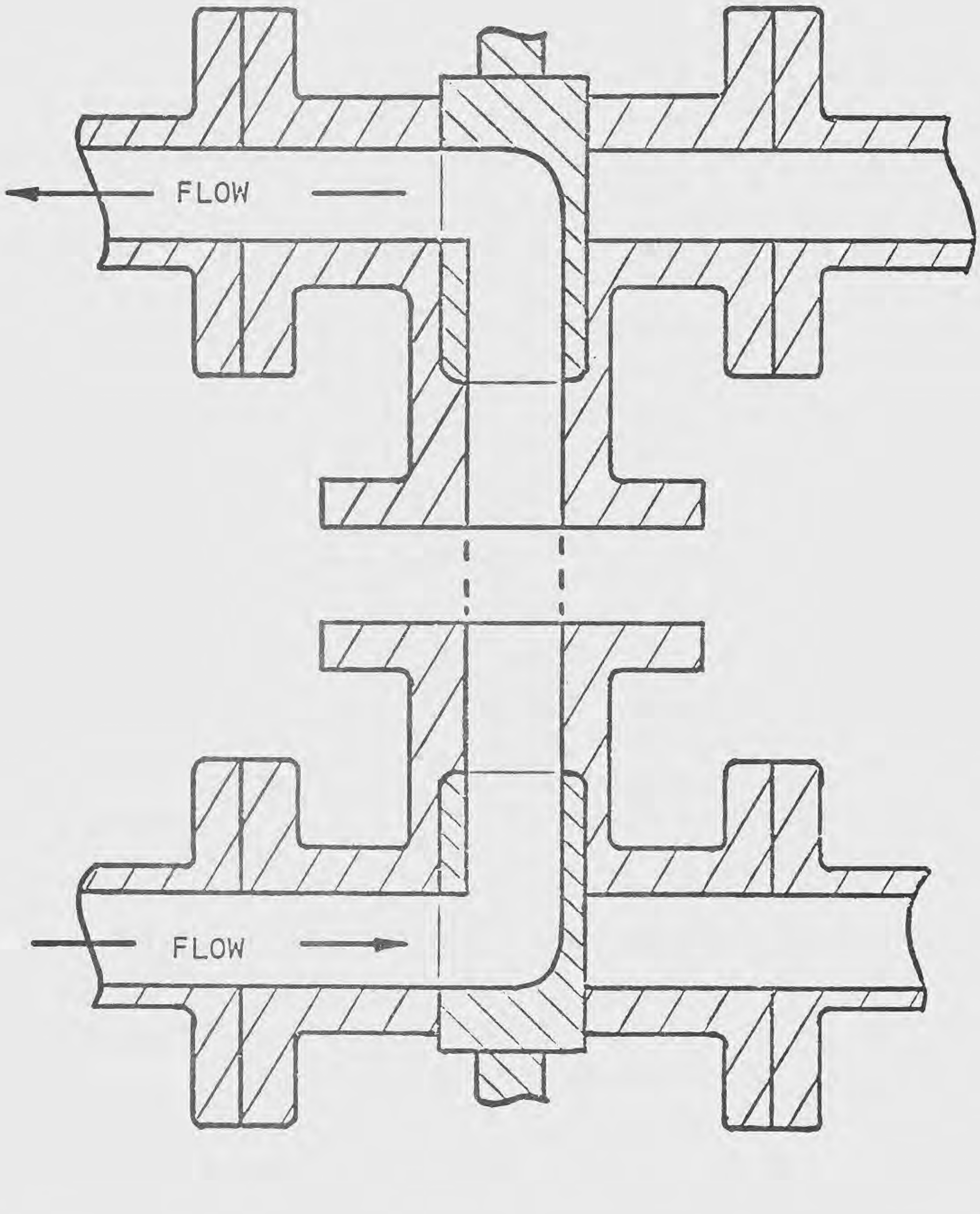

Figure 7a. Diagram of a double three-part valve arrangement normal flow position.

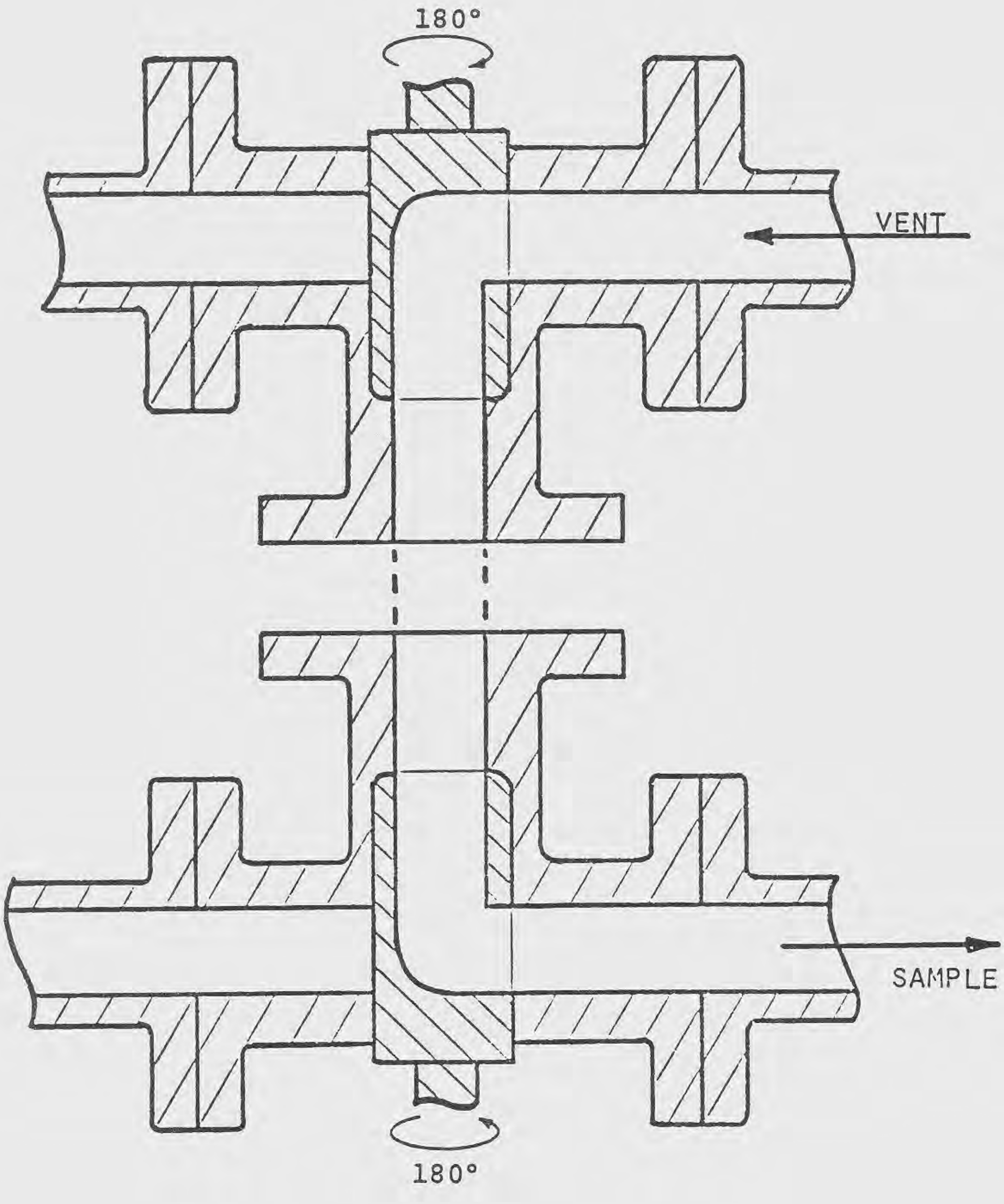

Figure 7b. Diagram of a double three-part valve arrangement - sampling position.

There are two important precautions to be exercised when using this system. The valves MUST NOT be transflow-type plugs, but must offer complete shut-off in the intermediate position. Second, the two valves must operate simultaneously. If both valves do not turn at the same time, full time pressure and flow could be discharged from either the sample or vent ports. There are various gear set and mechanical linkages which can be used to ensure positive, simultaneous movement. In this double valve system, under construction, the interlocking gear mechanism has been removed, and placed off to the side (Refer to Figure 8). When completed, the two valves will be operated by a single handle, and will deliver one quart of sample per cycle of the valves.

There are a number of companies which do manufacture liquid samplers like the ones shown in Figure 9. Typical operation of these units uses a air or hydraulic actuator to insert a hollow piston into a process pipe. As the piston retracts through the barrel of the sampler, a small, precise volume is trapped within the cavity of the piston. The piston stroke first seals the cavity from the process, and then opens it to the sample connection, to which a bottle may be attached. The sampling cavity within the piston usually has a capacity of only a few milliliters, so larger samples may require repeated cycling, either manually, or with automatic timers.

Another method uses a sample chamber attached directly to a process line as illustrated in Figure 10. Sample chambers can be designed to match process conditions and materials of construction, and may be sized for any desired volume. This particular arrangement uses a remotely operated control valve to admit a reaction mixture into a sample chamber. The chamber was preloaded with a quench solution to stop the reaction mixture from continuing. This system takes a good representative sample, but is somewhat difficult to use frequently. Furthermore, it may expose a person to potential chemical exposure during the step of breaking the union connection to remove the chamber from the system.

In the area of Direct line sampling, the piston ram type valve is finding increased use (Refer to Figure 11). This valve has a retracting piston which ensures that the process connection has no dead zone cavity, and that the valve is rodded out with each use. This makes the valve useful with process materials which could freeze or plug in the process connection. The disadvantage of this valve is that it is Direct line sampling - that is it has the potential to expose a person to full line pressure and flow. There is little positive control over the amount of sample discharged, and the valve may be difficult to close quickly. However, these potential drawbacks can be reduced significantly when this valve is used in combination with other sampling features, such as a sample chamber or sample enclosure.

Still another method of sample taking involves the use of a hypodermic type syringe to withdraw material through a septum attached to the process line (Refer to Figure 12). This method is restricted to

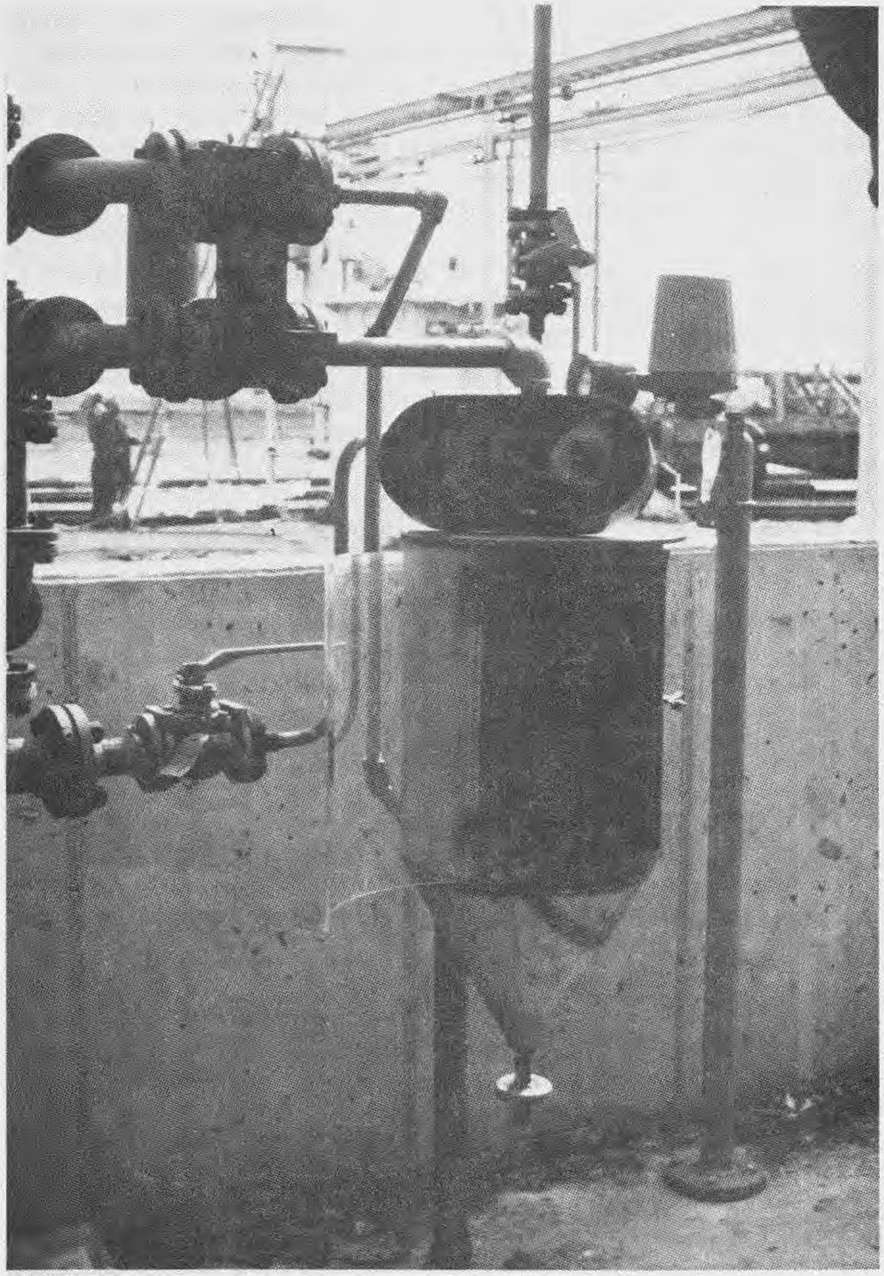

Figure 8. Double three-port valve installation with interlocking gear set removed.

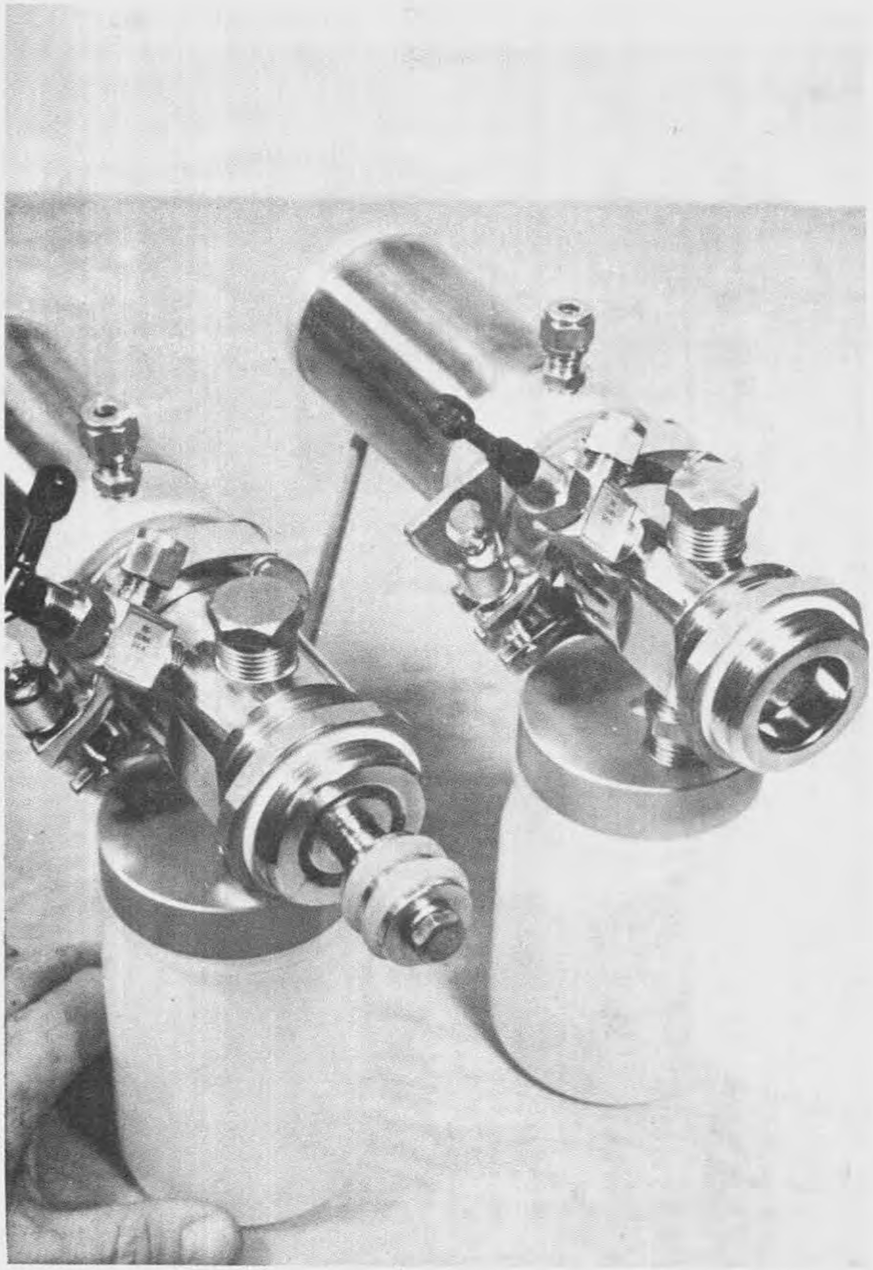

Figure 9. Isolok$^{(R)}$ liquid sampler.

Courtesy of Bristol Engineering Company

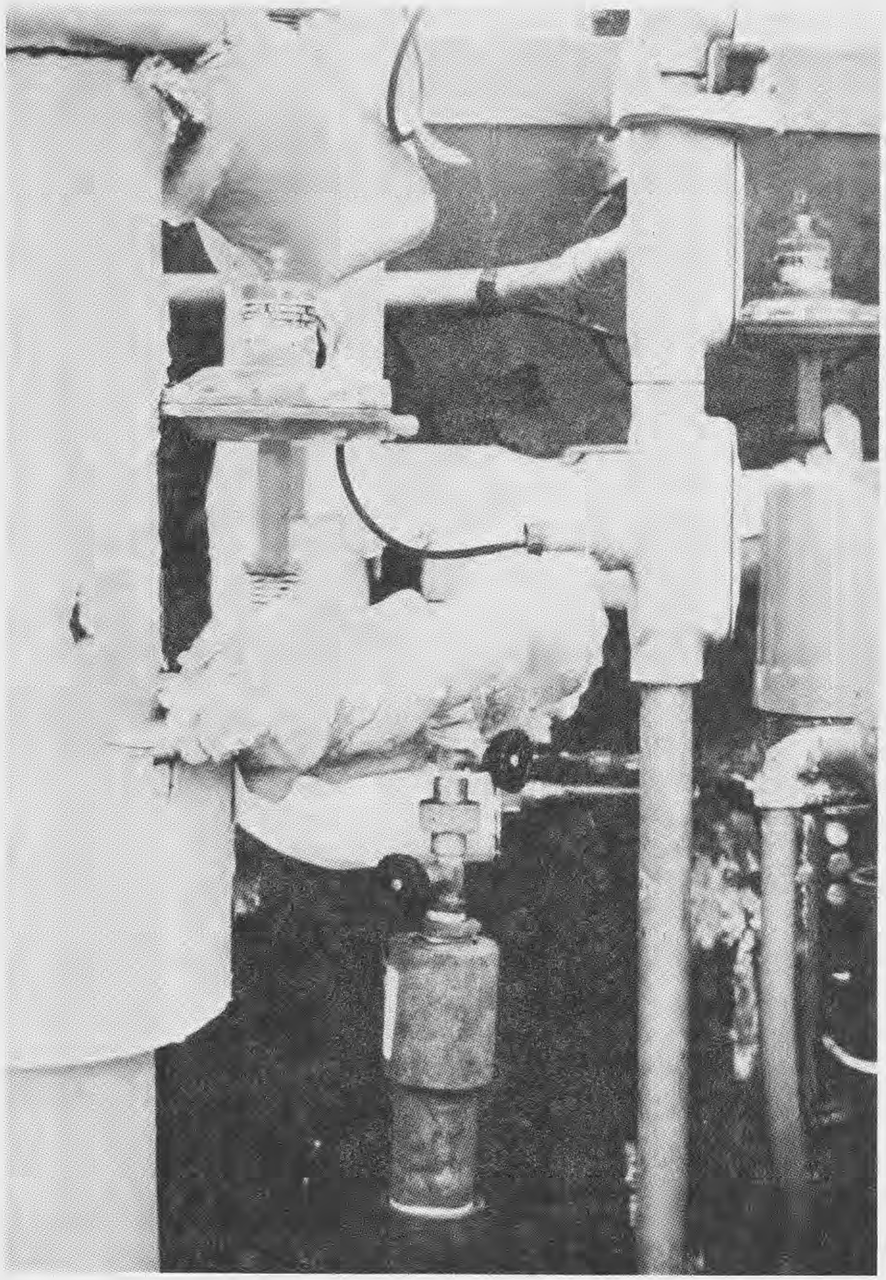

Figure 10. Sample chamber filled by remotely operated valve.

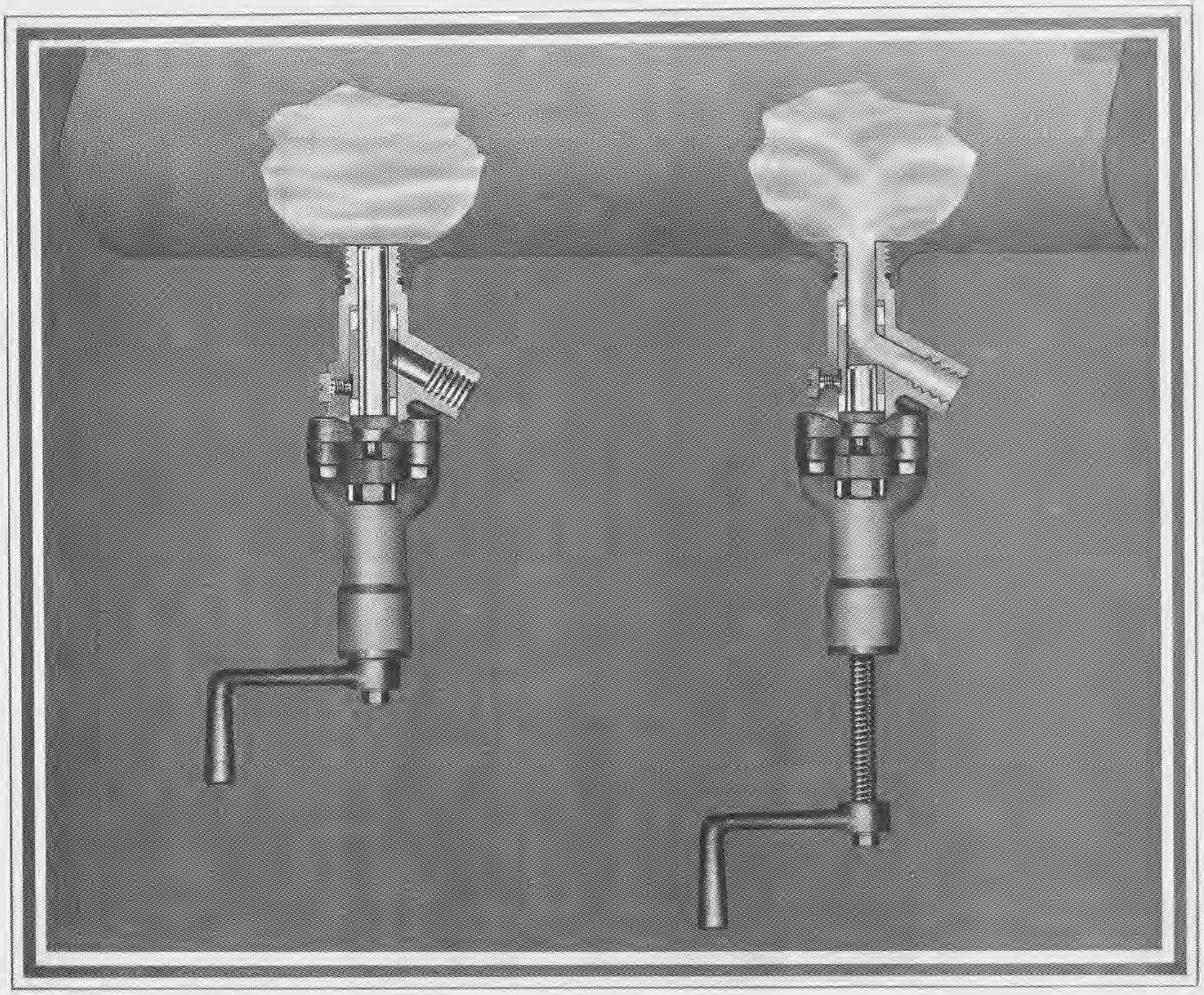

Figure 11. Strahman piston type sampling valve.

Courtesy of Strahman Valves, Inc.

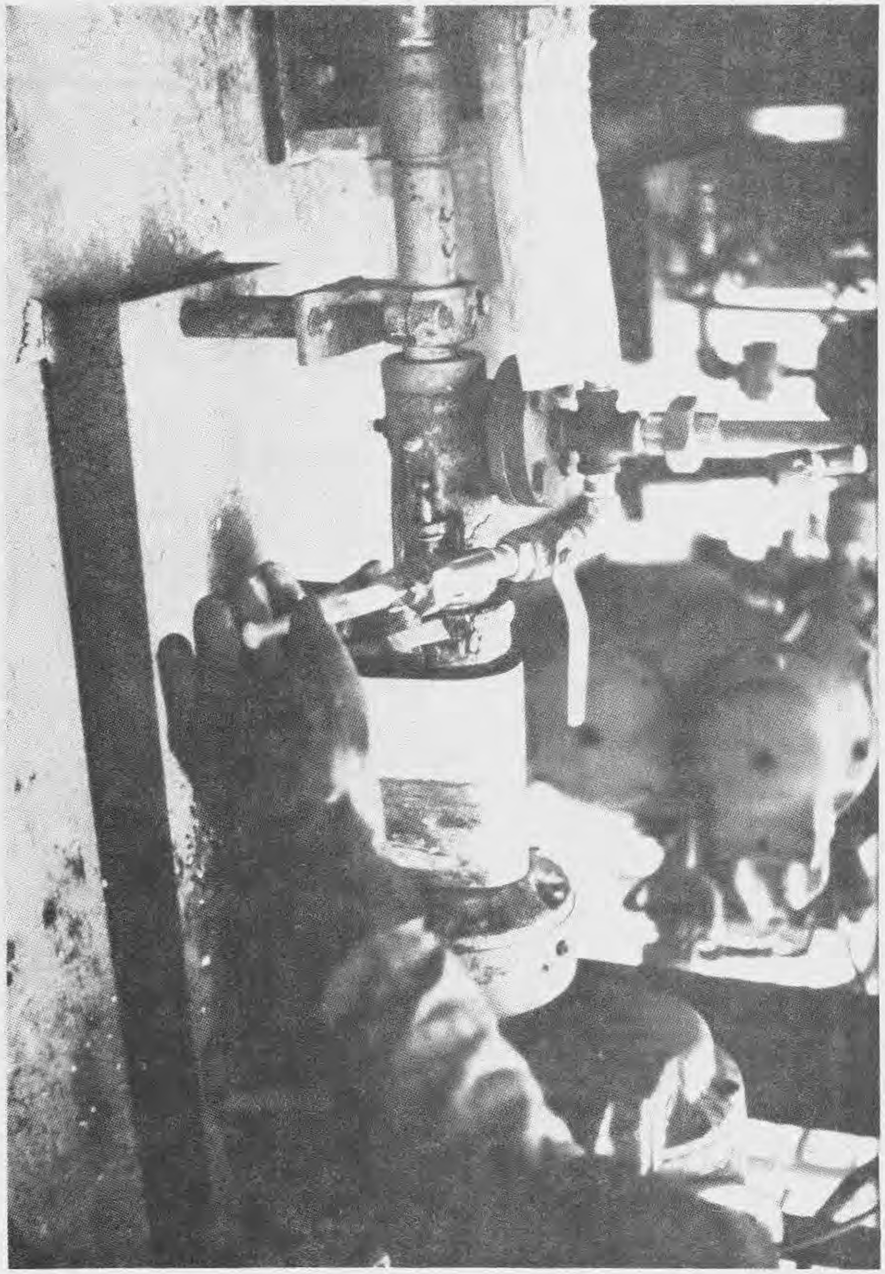

Figure 12. Septum and syringe method sampling installation.

conditions where the required sample is very small, the process material is nonviscous, nonfreezing, or does not contain solids which may clog the needle. Also, it should only be used with chemicals which will not attack or degrade the septum material. In this installation, the septum is at the end of the pipe stub, and can be isolated by means of the shut-off valve. However, this arrangement creates a substantial amount of dead volume. To manually bleed the dead volume out through the red handled bleed valve defeats the purpose of using the syringe to reduce personal exposure to hazardous chemicals.

An improvement on this method uses a three port, or a cross pattern valve shown in Figure 13. This valve is installed in a by-pass loop of the process line. The process material flows through the side ports, and the septum is installed on the bottom port. When the valve is opened, the needle may be inserted through the septum to extract a sample from the flowing stream. When the valve is closed, the septum is isolated from direct exposure to process conditions, and can be replaced with minimum interference and risk. The amount of dead volume within the valve is negligible, and the valve can be fitted with a needle guide to reduce damage to the syringes.

In conclusion, process sampling is a necessary facet of daily operation in the chemical industry. To be done safely, it can not be left to chance. The risks involved with sampling can be reduced by good design, and well planned and followed procedures. The sampling need must be anticipated and carefully considered; the sampling equipment should be uniquely identified, and selected or designed. The final design will be that sample system which best meets the requirements of process conditions, sample quality, safety, and costs.

* * *

DISCUSSION:

MR. JERRY SCHROY: What is the potential for leakage due to needle punctures or permeating simple diffusion through the organic material through the membrane. Is there any possibility of the process pressure enhancing leakage through a needle puncture?

MR. BRUCE LOVELACE: On this particular application we were using the septum in ethyl benzene process stream, the rubber that we were using was a silicone rubber and we would periodically change the septum whether it developed a leak or not. By using this particular valve, we only had the septum exposed process conditions at the time that we needed to get a process sample, the rest of the time it was isolated so that it wouldn't develop a leak. Care should be exercised in selecting the septum material both with regard to mechanical durability and chemical permeation.

MR. SCHROY: Have you ever reversed the situation, where you put the needle in the process and the septum in the sample bottle?

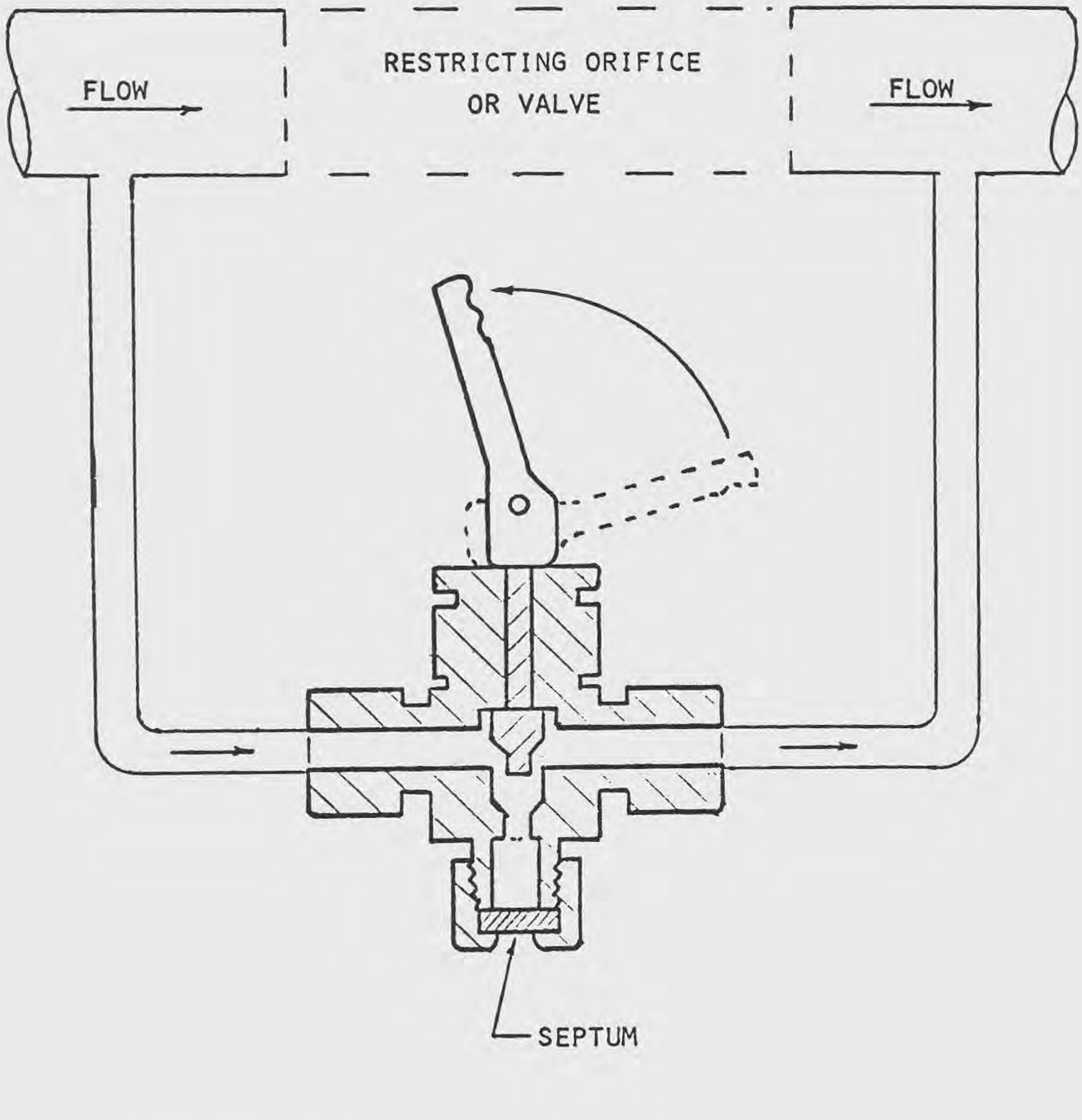

Figure 13. Diagram of syringe septum mounted on an isolating valve.

MR. LOVELACE: No we haven't. That is something we would like to consider.

MR. SCHROY: Your paper that was based on the AIChE paper, what meeting was it given at?

MR. LOVELACE: The paper has been written for some time but will be presented at the 86th National Meeting in Houston, Texas, this coming April (1979). It will be part of the Safety and Loss Prevention Symposium.

MR. SCHROY: You have two valves which turn simultaneously in one valve position, the process flows through the valve and in the second position, the sample is taken. Do you find that it is necessary to have a dead zone when the valve postion is changed from position 1 to position 2 to avoid process conditions being exerted on the sampling system to avoid cross-flow or is that not necessary in your system?

MR. LOVELACE: Yes, a dead zone is necessary. We have preferred to use a valve that has a dead zone in the center simply because in these particular applications we feel very strongly about making sure that there is no possibility that process conditions could be discharged through the sample ports. There have been applications that ran into problems where people have used the wrong valve and have found there is discharge during the actual procedure of turning the valves. We have also had installations where instead of a hard mechanical linkage to operate the two valves, the valves were being operated by two separate air driven actuators. The problem that we encountered is that both actuators did not respond to the air signal at exactly the same time and again one valve turned faster than the other one and allowed some leakage through one of the ports where they were not expecting it, this is why we are going back to a hard mechanical linkage.

MR. SCHROY: In dealing with a needle and septum sampler, have you ever reversed the position to put the needle on the process side and the septum in the sample bottle?

MR. LOVELACE: No, we have not used this technique, in my experience, however I see no major problems in doing this. (This technique is highlighted in Chemical Engineering, March 12, 1979, Plant Notebook Section.)

MR. DONATO R. TELESCA: The last speaker this session will be Arthur Spiegelman, who has his B.S. in Chemical Engineering from Cooper University. He is a registered Professional Engineer in New York and New Jersey, and has 25 years experience in the insurance industry. He was Vice President of Engineering of the American Insurance Association until he retired in 1979. At present he is a consulting engineer in all phases of loss control. Mr. Spiegelman's talk will be on the role of the insurance industry in motivating loss prevention measures.

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Control Technology in the Plastics and Resins Industry

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Held at the
Atlanta Hilton Hotel
Atlanta, Georgia

February 27-28, 1979

U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES
Public Health Service
Centers for Disease Control
National Institute for Occupational Safety and Health
Division of Physical Sciences and Engineering
Cincinnati, Ohio 45226

January 1981

For sale by the Superintendent of Documents, U.S. Government
Printing Office, Washington, D.C. 20402

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DHHS (NIOSH) Publication No. 81-107