

SAMPLING TECHNIQUES FOR OBTAINING REPRESENTATIVE SAMPLES FROM PROCESS STREAMS

Richard O. Speckman
VP & General Manager
Bristol Equipment Company
210 Beaver Street
Yorkville, IL 60560
Phone: 630-553-7161
Fax: 630-553-5981
DickS@Bristolequipment.com

Scott E. Raether
Executive VP & COO
Bristol Equipment Company
210 Beaver Street
Yorkville, IL 60560
Phone: 630-553-7161
Fax: 630-553-5981
ScottR@Bristolequipment.com

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ABSTRACT

The analysis of any process stream to determine its constituents or quality depends not only upon the laboratory instrumentation used but also on the quality and representivity of the process stream and sample itself.

Obtaining representative samples ⁽¹⁾ for accurate analysis is dependant on process conditions (pressure, temperature, flow rate, etc.), ambient conditions, media to be sampled (viscosity, pH, grain size, etc.) and environmental or agency requirements (3A Standards, ASME, OSHA, etc.) Various devices have been designed to meet the needs of the unique customer while ensuring a representative sample is delivered as needed while keeping the operator safe from any process or environmental hazards. . Caution must be taken to ensure that the devices used in collecting and transporting samples do not effect the composition of the sample taken.

It is the intent of this discussion to explore acceptable devices and methods for extracting quality samples from the various process categories.

INTRODUCTION

Representative sampling can be obtained from process streams by direct and/or indirect methods. Direct methods include dip type sampling or opening spigot valves. These and other unsafe direct collection procedures are considered unacceptable and do not warrant discussion.

Obtaining representative samples begins by:

- Outlining sample objectives
- Examination of product and site considerations
- Established methods and procedure
- Profile of the sampling system

OUTLINING SAMPLE OBJECTIVES

This is a commitment to provide a comprehensive guideline incorporating safe, modern methods and procedures for collection, conditioning, testing and analysis. No matter how sophisticated laboratory analysis equipment and procedures may be, final analysis is only as good as the quality of the sample collected.

EXAMINATION OF PRODUCT AND SITE CONSIDERATIONS

This is primary to define the sampling characteristics and restrictions. Food and Drug Administration (FDA), Pharmaceutical, Chemical and Nuclear all require similar yet unique and different approaches to equipment selection. Liquid, slurry and/or powders will further define the “approach” to representative sampling.

Examination of product and site considerations should include:

- Basic description of material to be sampled
- pH value
- Operating/design temperature
- Viscosity
- Slurry analysis
- Moisture content
- Dust considerations
- Gases or vapors present
- Unusual hazard concerns
- Explosion hazard rated areas
- Process line pressure and temperature
- Process line size, material and orientation
- Dew Point &/or Bubble Point

ESTABLISHED METHODS AND PROCEDURES

Procedures of modern sampling are most often influenced by environmental, agency, or process requirements. Health and safety requirements reinforce the concept of treating each sample site

individually; to obtain complete details required for the selection, installation, operation and maintenance of comprehensive sampling equipment. Carefully defined methods and procedures will minimize operator and/or environmental exposure to potentially hazardous conditions at the sample site while providing a quality process sample.

Of equal importance is the need to establish on-going control procedures with periodic review to determine if product or site conditions have changed; and if collection methods and procedures are still appropriate, accurate and useful. Testing and analysis procedures should be reviewed to insure data is appropriate, accurate and useful. Control procedures should involve consideration to changes in technology that could affect the objective as outlined in the original sampling program.

Assuming the sampling objectives have been established and proper identification has been made regarding the characteristics of the product and the sample site; appropriate methods and procedures should logically follow. Methods should involve selection and operation of the sampling instrument, the nature of operational control and the peripheral accessories required to meet sampling objectives.

Methods and procedures will likewise include a determination about the type of sample (batch or composite), as well as the time period over which the sample is to be collected. Procedures should clearly define how the sample is taken and subsequently handled to minimize operator exposure to potentially hazardous environmental conditions.

A PROFILE OF THE SAMPLING SYSTEM

A profile of the sampling system involves the analysis of appropriate component parts within the sampling system necessary to co-exist with the already established methods and procedures.

A sampling system, whether designed for liquid, slurry or dry powder; would include the appropriately designed sampling instrument, operational control and sample collection accessories necessary to secure and maintain the integrity of the collected sample. Proper selection and integration is essential to meet all of the program objectives.

IDENTIFICATION OF PRODUCT CHARACTERISTICS

The first step in evaluating the sampling system is to examine the aspects at the sample site and the material to be sampled. As shown in Figure 1, one approach that might serve as a useful introduction into this area is a simplified conceptual triangle ⁽²⁾ showing three basic states of matter; gas, liquids and solids at each of the extreme points.

Reviewing the conceptual model, it might initially suggest that these three states of matter are separate entities. This, of course, is rarely the case. It does, however, serve as a point of perspective and can be used to demonstrate how the states can interact within the same chemical process "stream" further complicating methods and procedures required to handle this material.

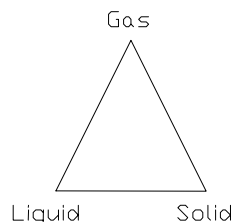


FIGURE 1- THREE STATES OF MATTER

For instance, it is quite common to find that most chemical process streams contain entrained gases that can be greatly affected by changes in operating pressures and temperatures. Thus, when sampling process streams that contain “outgases” which can expand upon decompression; provisions must be made to vent, contain or otherwise handle the pressurized material. If the gases are critical to the composition of the sample, appropriate sampling procedures and equipment must be used so that the sample is representative of the process stream.

The term “solids”, as defined here, refers to various materials normally ranging from small micron or mesh sizes to larger granular products that might be found in liquid process streams, slurries or by itself as a bulk solid. The concept of the above model, when used in conjunction with data obtained about the product to be sampled; and the sample site characteristics, can serve as a useful tool in considering alternate approaches to meeting those potential “hazards” at the sample point.

PRODUCT AND SAMPLE SITE CONSIDERATIONS

There is normally a considerable amount of information available within the chemical complex that can be used to profile characteristics of the product under consideration. In sorting through this material, it would be helpful to pay particular attention to those areas previously outlined in the “Examination of product and site considerations” section above.

BASIC DESCRIPTION OF MATERIAL TO BE SAMPLED.

A concise description of the material to be sampled serves as a basic point of identification to all individuals concerned in the project. The description often supplies hints about characteristics that may cause health and/or safety issues at the sample point.

pH VALUE.

This is an essential measurement when working with solutions to determine the nature (acidity – alkalinity) and extent of corrosion associated with the process. This information, when combined with a further description of the acid or caustic material, will give insight to material compatibility, isolation or other protective measures to be considered.

OPERATING/DESIGN TEMPERATURE.

Operating and design temperatures are critical for the selection of seal materials in direct contact with (or in close proximity to) the process material(s). Operating temperature further serves as an indicator about the type of, and amount of, provision required for safety, heat and/or freeze protection.

VISCOSITY.

There are two primary concerns associated with material viscosity levels. First, where the product has a viscosity less than water, potential sealing (or leakage) problems may be encountered between the sampling instrument and appropriate process line interface. Second, material with higher viscosity levels pose potential problems of plugging, air binding, non-flow ability, carry-over and cleaning. In turn, this can involve a series of health and safety concerns associated with the operation and maintenance of the sampling equipment.

SLURRY ANALYSIS.

Liquids and solids in “slurry” can exert various characteristics also affecting health and safety. This suggests the need to examine the constituents; paying particular attention to texture, stickiness, abrasiveness, particle size and entrained hazards (e.g. radioactive resin beads). Other considerations might include percent solids by weight (or volume) and potential product precipitation directly affecting the useful life of the dynamic seals.

MOISTURE CONTENT (DRY POWDER).

The amount of internal or surface moisture of solid “dry” material varies to some extent depending on the type of material and where in the process sampling is accomplished. Although other operating conditions may co-exist, the drier the material sampled, the easier it will “flow” allowing for less chance of plugging and/or rendering the sampling instrument unusable.

DUST CONSIDERATIONS (DRY POWDER)

One of the primary concerns when working with “dry” materials is associated with airborne contaminants from the product and related environment. This is especially the case when working with bagging machines or similar equipment causing turbulent conditions.

Although not considered hazardous by nature of toxicity, airborne contaminants can still cause concern from a health and safety standpoint. Suggested methods for handling this material might include enclosed sampling systems, ventilation hoods, suction units or alternate sample locations and handling techniques.

GASES OR VAPORS PRESENT.

It is not unusual (in sampling chemical process streams) to encounter the presence of liquids, vapors or gases that could be harmful if exposed to the operator or the environment. Toxic,

radioactive and other process materials may require further isolation or containment solutions both during sample collection and afterward in handling the collected sample.

UNUSUAL HAZARD ⁽³⁾ CONCERNS.

Each sample site should be considered on its own merits, since even minor differences can influence the behavior of the process and therefore affect the health and safety aspects of the sampling environment. Product characteristics should be looked at both separately and as they would interact with other variables within the process.

EXPLOSION HAZARD RATED AREAS.

Explosive hazard conditions are influenced by the product, as well as conditions in and around the sampling area. Acceptable techniques and “rated” commercial equipment allow reasonably safe operating conditions when used according to established methods and procedures.

PROCESS LINE PRESSURE AND TEMPERATURE.

Examination of line pressure and temperature is essential to the analysis and must be determined for both normal operating conditions, as well as maximum allowable design considerations. Operating pressures and temperatures are useful indicators of the expectant life of the instrument product contact seal materials and influence the capacity (rating) of the sampling instrument itself.

In addition to the requirements to seal and operate at given line pressures and temperatures, provisions should be considered to minimize possible product degradation during sample collection. Pressure reducing techniques, enclosure isolation, thermal stabilization, and security provisions should also be given consideration to ensure a representative sample and to minimize hazardous influences at the sample point.

PROCESS LINE SIZE, MATERIAL AND ORIENTATION.

Line size (vessel, chute, etc.), orientation and material of construction collectively influence the method of installation and establish the design configuration of the sampling instrument. Standards, codes, plant practices and other factors related to health and safety not only affect the selection of the instrument; but also, how and where it should be installed.

DEW POINT &/OR BUBBLE POINT

The dew point and/or bubble point of the fluid to be sampled must be considered to ensure that the sample contains all the constituents. Pressure or temperature change will cause the entrained gases in the fluid to separate which will lead to incorrect analysis of the fluid in the process lines due to the change in the fluids make-up.

ISOLOK® AND POSITIVE DISPLACEMENT SAMPLING

Whether for liquid, slurry or bulk solids applications; the most crucial component within the sampling system is the sampler instrument. Its design and materials of construction must be compatible with the attributes (corrosion, abrasion, particle size) of the process fluid to ensure a representative sample is delivered as needed. The sampling instrument must also keep the operator safe from any process or environmental hazards.

Sampling devices (instruments) using the positive displacement, or indirect method of sample collection, are most suited. These devices are designed to isolate line conditions (pressure and temperature) during the normal collection cycle; never does an open line exist between the potentially hazardous process and the sample collection container.

COMPOSITE SAMPLING

In many instances, the most representative liquid, slurry or powder sample is produced by automatically withdrawing a series of smaller, precisely measured amounts as a flowing material passes the sample point. The number of individual samples taken is adjusted according to time or by product flow; to generate the desired composite sample volume that most fully represents the process material composition. Samples might be collected in a common container or in individual containers as appropriate.

BATCH OR ANALYSIS SAMPLING

The positive displacement sampler is also applicable when an immediate sample is required, or when on-line analysis techniques are utilized. A single sampler cycle can be initiated, or a rapid cycling rate used to withdraw the precise material volume (batch) necessary.

When on-line analysis is used, the sampler is connected directly to the measurement instrument and the accurate material sample is conducted safely and directly to the analyzer.

POSITIVE DISPLACEMENT DESIGN FEATURES

As shown in Figure 2, the positive displacement type instrument device has many features over other direct or indirect types of devices. These include:

- A fixed amount of sample is drawn with each stroke of the plunger.
- Process media washes the annular sample spool each time it extends into the process line. Sample flows smoothly without slots or passages to clog.
- Process line port is always closed, helping to avoid leaks or product loss. Samples are transferred directly to a closed container, avoiding exposure to personnel or plant environment.

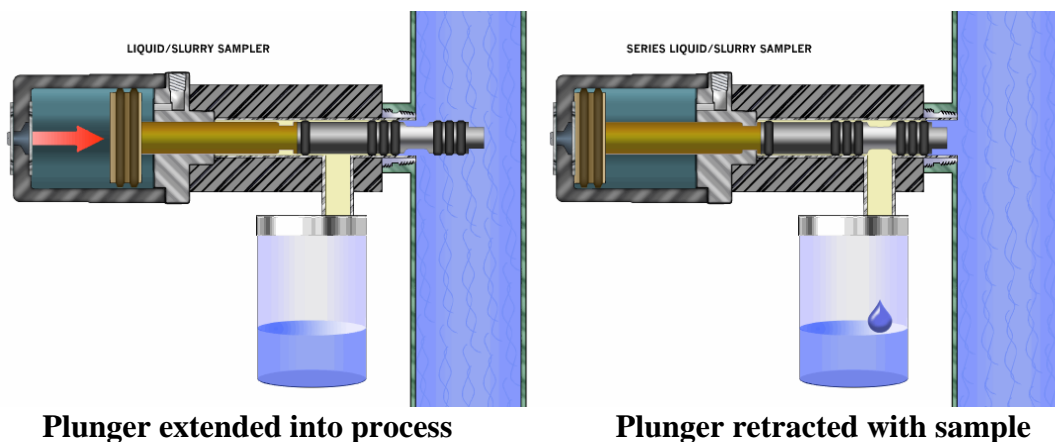


FIGURE 2 COMPOSITE SAMPLING DEVICE

CONCLUSION

The purpose of this discussion was to explore acceptable devices and methods for extracting quality samples from the various process categories. In retrospect, this involved the need to first define (and develop) a comprehensive approach to the development of the sampling program. Once completed, the design to effectively handle difficult chemical materials under a variety of potentially hazardous operating conditions would naturally evolve.

Because of the wide variety of potential hazards at the sample site, health and safety professionals need to work in close approximation with process engineers, operating and other management personnel to identify and isolate potential hazards during the sample collection process.

REFERENCES

1. Perry, Robert H., Green, Don W. Perry's Chemical Engineer's Handbook, Sixth Edition, McGraw-Hill Book Company, New York, 1984, Pages 19-109
2. Hawley, Gessner G., Hawley's Condensed Chemical Dictionary, Twelfth Edition/ revised by Richard J. Lewis, Sr., Van Nostrand Reinhold Company, New York, 1993, Pages 552, 703, 1074
3. Hawley, Gessner G., Hawley's Condensed Chemical Dictionary, Twelfth Edition/ revised by Richard J. Lewis, Sr., Van Nostrand Reinhold Company, New York, 1993, Pages 585

NOMENCLATURE

Powders are a subset of a larger classification of materials generally defined as bulk solids. Other material classifications within bulk solids includes, but is not limited to granules, flakes, fibrous materials, pellets, chunks, and grains.