Improved Food and Feed Safety through Systematic Planning and the Theory of Sampling (TOS): An Introduction to "GOODSamples"

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The Food Safety Modernization Act (FSMA), signed into law by President Barack Obama on 4 January 2011, provides the US Food and Drug Administration (FDA) with a framework to better protect public health by strengthening the food safety system. Its primary purpose is to ensure the safety of the US food supply by shifting focus to prevention of food and animal feed contamination through enhanced partnerships and integration among federal, state, local, tribal and territorial partners. FSMA is the most sweeping reform of US food safety laws in 70 years. FSMA addresses Preventive Controls, Inspection and Compliance, Response, Imported Food Safety, and Enhanced Partnerships. Title II—Improving Capacity to Detect and Respond to Food Safety Problems—addresses Laboratory Accreditation for Analyses of Feeds in Section 202. Section 202(a)(6) states that Model Standards will require appropriate sampling.

The US FDA awarded a five-year cooperative agreement to the Association of Public Health Laboratories (APHL), Association of Food and Drug Officials (AFDO) and the Association of American Feed Control Officials (AAFCO) to support the implementation of The Food Safety Modernization Act (FSMA). One of the Specific Aims in the cooperative agreement is "Harmonized Policies and Procedures for Equivalency of Data". A task under this Aim is to establish a working group to develop harmonised policies and procedures for sample collection, shipment, analysis, storage and retention of food and feed materials. The *Sampling and Sample Handling Working Group* effort is led by AAFCO due to its long history of recognition of sampling and sample preparation as critical aspects of the regulatory process.

Currently, procedures for sample collection are as varied as the number of agencies that collect samples. This wide variety of sample collection techniques does not lend itself to data equivalency among the various agencies, a prerequisite for inter-agency data sharing because of uncontrolled sampling bias (which cannot be corrected) and other sampling errors (see further below). The goal of the working group is to develop a common sampling strategy for sampling food and feed. With this common sampling strategy, data can be evaluated with respect to "fit for purpose" or, more aptly, "fit for decision" criteria for any agency, project or situation. This will allow for harmonised data collection, defensibility of analytical results and, ultimately, the ability of agencies to share data with confidence. The main audience for this document is regulatory programmes and their associated laboratories, including management, inspectors, quality assurance officers and laboratory personnel.

The guidance document currently under construction has been titled *Guidance on Obtaining Defensible Samples* or *GOODSamples*.

GOODSamples, the philosophy

The common perception is that all that is needed are more standard operating procedures (SOP), but as there are already thousands available; it is doubtful that "just a few more" will meet the goals of FSMA. Unfortunately, there are an infinite number of sampling scenarios. SOPs are stagnate and not responsive to new analytes, new regulations, new sampling techniques and tools, unanticipated field conditions, new field analytical techniques etc. But nowhere has the characteristic "representative" been given a full, operative definition by the US FDA or food/feed regulatory bodies. Therefore, the philosophy of *GOODSamples* is not to develop more Standard Operating Procedures for sample collection, but to provide a practical and complete framework for field inspectors, project managers, chemists etc. to work together to develop and implement sampling protocols to meet the objectives of FSMA. This can only be met by insisting on documented representative sampling procedures through the entire field-to-aliquot pathway (see Figure 1).

The FDA developed many sampling protocols based on attribute sampling strategies *ca* WWII that predated the Theory of Sampling (TOS). The scientific basis for these protocols has changed little over time. FSMA has now brought an opportunity to change and update the science behind sampling food and feed.

GOODSamples, the document

It should be evident that data equivalency is dependent upon the collection of representative samples for specific objectives; it may not be so readily evident that representative sampling is a function of the whole organisation and a management system that is committed and promotes communication.

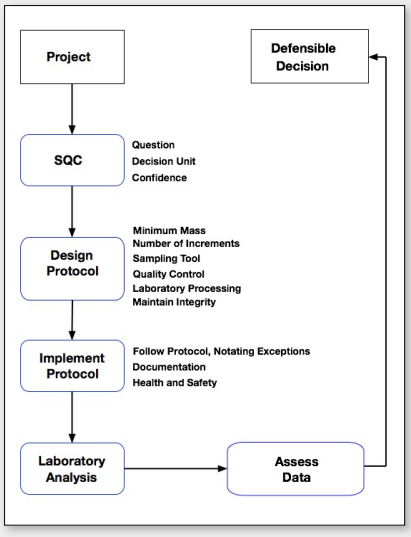

Figure 1. GOODSamples pathway to a defensible decision

GOODSamples address the entire process of sampling from development of objectives to final assessment. Communication between all disciplines involved in sample collection is stressed throughout the document. The specific chapter titles and the rationale for its inclusion are as follows:

Definitions: A common vocabulary is essential! Each segment of the intended audience currently has different terms for the same concept.

Management Considerations: Supportive and knowledgeable management is critical to a successful sampling programme. Communication among management, quality, sampling and laboratory staff is needed to develop competent sampling protocols. This chapter provides a rationale for the importance of management in the overall effort.

Sampling Quality Criteria (SQC): SQC provides the framework for planning and managing practical sampling and analytical operations consistent with the food/feed programme needs. It is a series of statements that clarify technical and quality criteria to support defensible decisions. This chapter introduces the key elements of SQC.

Theory of Sampling (TOS): The most important part of sample collection is a basic understanding of the TOS and what makes a representative sample. Understanding TOS is key for management, quality assurance staff, inspectors, laboratory analysts and data users. Only a brief introduction to the topic is presented here; the standard *DS 3077—Horizontal*[1] gives additional background information and references and Gy[2] and Pitard[3] provide greater detail on the TOS.

Quality Control: This chapter describes the three of the four general types of quality control checks used in the sampling process. These include checks for contamination from various sources, sampling repeatability (precision) and laboratory consistency.

Sampling Tools: This chapter is divided into two sections: *The Theory of Sampling Tools* and *Sampling Tools.* The first section addresses the theory of the selection of equiprobable particles, sample correctness and the dimensions of decision units. The second section addresses considerations in choosing the correct tool for a specific sampling effort.

Maintaining Integrity This chapter is divided into sections on *Evidentiary Integrity* and *Analyte Integrity*. Evidentiary integrity is maintained by thorough documentation, including chain of custody. *Analyte Integrity* is maintained by proper preservation, proper choice of containers, observance of holding times and proper handling, packaging and shipping.

Health and Safety: SAFETY FIRST! No sample is as important as your safety.

Sampling Protocol Design: A sampling protocol is a detailed procedure for obtaining a representative primary sample of appropriate mass and number of increments from a specific decision unit to meet the SQC. The protocol includes the appropriate quality control and directions for maintaining evidentiary and analyte integrity, tool usage, sample processing etc.

Examples of Sampling Protocols: Examples of protocols for a specific SQC are provided.

Laboratory Sampling, Handling and Preparation: This section provides guidance on how laboratories should handle and process samples received for analysis, keeping in mind two primary responsibilities: ensuring that the target analyte(s) are not compromised during sample preparation and storage; and obtaining representative analytical samples and test portions from the laboratory sample.

Process Assessment: Assessment of the entire process is critical to determine whether it meets the objectives set forth (SCQ) and is suitable to make decisions at the specified confidence.

GOODSamples, the approach

The approach promulgated in *GOODSamples* is that all sampling protocols must begin with development of appropriate objectives. Too often, data is generated without objectives first being defined. A lack of objectives, or poorly defined objectives, unavoidably leads to undesirable outcomes. These include inconsistency in interpretation of results; questions are not answered directly; insufficient confidence; and/or inefficient allocation of resources. As Bernard Baruch has stated, "A problem well stated is a problem half solved."

Sample Quality Criteria (SQC) provides the framework to determine project objectives and is the basis of design for a sampling protocol to answer a specific question with a known confidence (see Figure 2). Once the SQC is established, the sampling protocol can be developed based on TOS incorporating necessary quality control.

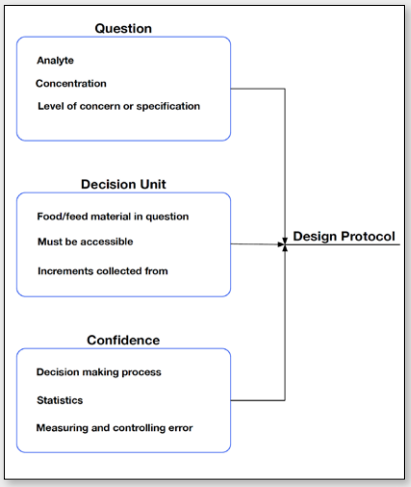

Figure 2. Sample quality criteria (SQC) inputs.

Care must be taken to ensure the analyte integrity is maintained through the entire process including transportation to the laboratory. Laboratory sub-sampling and processing protocols need to be considered and included as part of the protocol. The *AAFCO Guidelines for Preparing Laboratory Samples*[4] provides guidance to feed laboratories with basis in TOS and was used as a basis for International Standard 6498:2012 *Animal feeding stuffs – Guidelines for Sample Preparation*.[5] Both will serve as normative references for *GOODSamples*. Petersen *et al.*[6] also provide a TOS-approach to laboratory processes for dry granular feed materials.

There must be established a unified responsibility (institutionally, through normative good practice documents). Three primary elements of SQC in *GOODSamples* are:

What is the question sampling and analysis is intended to answer?

Identification of the analyte(s) and concentration level(s) of concern is the first consideration in SQC. It is critical that this is known in advance so planning ensures that appropriate sample containers are used, sampling tools and techniques can maintain the integrity of the analyte(s) are utilised, analytes are preserved appropriately and health and safety is addressed.

Determination of the expected analyte concentration of concern is also important in the development of the sampling protocol. If the concentration is unknown and a reasonable estimate is not available,

a specification limit may be used as the concentration estimate since this is the concentration where the error must be closely controlled.

In situations where there are multiple analytes of concern, this information is required for all analytes.

What is the decision unit (population, lot) the sample is intended to represent?

For some scenarios this is an obvious and easy question to answer, but in reality, identification of the decision unit is typically not considered. "Just take some samples" is a typical approach. This aspect of sampling can be the most difficult to understand initially, but it is the most fundamental aspect of sampling. The decision unit determines what needs to be accessible; where increments are collected from, where inferences are made to, which tools will select the right shape and mass of increments. It is critical that the entire decision unit be available to the sampler; this is termed the fundamental sampling principle (FSP) in TOS.

What is the desired confidence in the final decision?

Selecting the level of confidence can be difficult for those without some level of statistical understanding, especially if a specific number on the level of confidence (e.g. 95%, 99%) is desired. Confidence is actually a function of consequences. The more serious the consequences of the ultimate decision, the greater the level of confidence needed. Confidence does not have to be statistical, but it does have to be agreed on by all the parties involved.

Knowing how the data is going to be applied is critical to ensure that the appropriate data is collected. An often-overlooked aspect in the planning stage is to specify how the data will be applied in making the decision. This may include the number of samples, types of sampling, allowable sampling error, quality control, sample processing, analytical methods and a host of other important design aspects. All too often, the intended decision cannot be made because the data are inadequate for the type of decisions required by the SQC.

Quality control is an important, yet often overlooked, element in the confidence realm. Quality control demonstrates that the system is in control and allows an empirical estimation of the effective, total sampling and analysis error. One type of quality control is a control for the detection of contamination. The contamination may be from the environment, tools or containers. This is important for sampling of trace, volatile or biological analytes. Replication (in the form of a "replication experiment", DS 3077 82013) is another approach that can be used to determine the total measurement uncertainty (MU) [sampling + analysis] associated with the analytical results. Esbensen and Wagner[7] outlined the complementary, interacting competences between $TOS_{sampling}$ and $MU_{analysis}$.

Once the SQC process is complete, the design of the sampling protocol can begin. The sampling protocol is impossible to develop without a competent understanding of TOS. To the knowledge of the authors, TOS has never been comprehensively included in food and feed SOPs in the United States.

Once the sampling protocol has been designed, implementation can begin. Unfortunately, field situations are seldom what were anticipated during the development of the sampling protocol and sometimes adjustments must be made. If the sampler is following a protocol blindly, unrepresentative samples may be the result. The ultimate data user is typically unaware of the field conditions and makes decisions based on results from samples that may not be adequate for the objectives of the project. Therefore, it is of paramount importance that the people collecting the primary samples have sufficient training in all aspects of SQC and TOS so they can adapt to unanticipated conditions in the field without compromising the integrity of the primary samples and the resulting decision. Training is an important part of FSMA and critical to the implementation of *GOODSamples*.

Often the data is used without any determination or assessment as to whether it meets the objectives set forth and is suitable to make decisions. Assessment of data includes evaluation of the appropriateness of the SQC, critical review of the quality control data (not just pass/fail), error propagation calculations, verification of data assumptions if statistical calculations are performed etc. In other words, did everything go as planned? If not, what impact does that have on the confidence in the final decision? (see Figure 3).

Summary

Sampling is more than a collection of Standard Operating Procedures that are selected for ease of use or availability of equipment. Simply filling containers will not

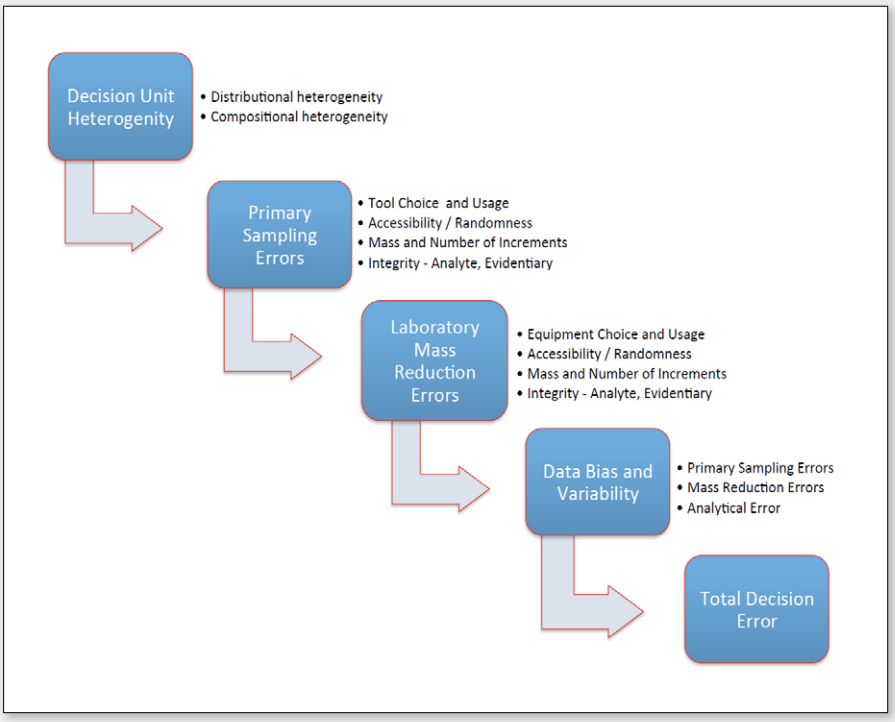

Figure 3. Contributions to the Total Decision Error.

provide useful data for defensible decision-making. Sampling is about meeting project objectives. A systematic approach to meeting project objectives is the most critical, but largely overlooked, part of the process. *GOODSamples* provides the necessary and sufficient framework to allow for defensible decisions.

The guidance provided in GOODSamples is not unique to food and feed sampling but should be applicable to any sampling effort. The process from determining objectives to a developing a final sampling protocol must be based on science. While there are many proposals for practical approaches to sampling, mostly of a highly specific, "home-grown" variety, from which no general conclusions could possibly be drawn, TOS reigns as the most comprehensive approach for the types of materials encountered in the food and feed industries. In the recently codified form, *DS 3077*[1] (2013) will be a normative reference for *GOODSamples*. While the focus of *GOODSamples* is food and feed regulatory programmes and their associated laboratories, the document will also be suited for producers, distributors and manufacturers of food and feed. Other industries such a fertiliser, pharmaceutical and supplement producers can also benefit from a systematic approach such as outlined in *GOODSamples*.

Many readers of the *TOS Forum* have experience with the sampling of food, feed, fertiliser, pharmaceuticals, supplements and other related commodities. The present authors seek relevant references to cite in the new guidance document. We wish to include as much international "flavour" as possible, since most of the work in TOS does not take place in the United States. Please feel welcome to contact the corresponding author with any comments, questions, issues, concerns and references you have.

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