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# Establishing a quality management system in a university-based analytical laboratory

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# Establishing a quality management system in a university-based analytical laboratory

by

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Signatures have been redacted for privacy

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## ABSTRACT

Analytical laboratory organization is a complex task. Equally as complex is the establishment of a quality management system (QMS) that functions consistently and efficiency. The objectives of this research are to develop the quality manual for the Iowa State University Grain Quality Laboratory (ISU-GQL) to apply for ISO/IEC 17025 accreditation and to provide a structure for university-based analytical laboratories to establish quality management systems. Important considerations for implementing a QMS include writing and implementing laboratory procedures, standardizing job descriptions, creating a quality manual, and generating working control charts. Typical word processing and spreadsheet software along with custom program routines were utilized to simplify the ISU-GQL quality management system for technicians and managers, because of the more continuous turnover and generally less structured practice of the university researcher. Special attention was paid to the initial and continuing training of technicians and support staff. The conclusions reached from this research include that a quality management system must originate and be endorsed by management to succeed. Considerable time and energy must be expended by dedicated personnel to establish and maintain an effective quality management system. And that a quality management system, which is well organized and composed initially, can experience great success.

# CHAPTER 1. LITERATURE REVIEW AND INTRODUCTION

Analytical laboratory organization is a complex task. Equally as complex is the establishment of a quality management system that functions consistently and efficiently. The process for applying the ISO/IEC 17025 standard (ISO/IEC, 1999) to analytical laboratories is well defined, as shown by Tholen (2002). Development and implementation resources are readily available for inexperienced laboratory staff, especially in the pharmaceutical field. Unfortunately, these resources are difficult to use in areas other than pharmaceuticals (Dux, 1990). Many courses and materials are so specific to the pharmaceutical industry that extrapolation is not easily made. Quality management systems in general have much wider application.

The essence of a quality management system (QMS) is that an organization documents what it is doing, then proves it is doing what is claimed. In the process of creating documentation and proof, operations are analyzed in detail for efficient contribution to customer satisfaction. A QMS does not define what quality is, nor does it set a product standard that all organizations must meet. In a laboratory setting, the emphasis is rather on discipline and reproducibility. (Tholen, 2002) Therefore, a quality management system must be developed individually for each laboratory.

The background for quality management systems is the philosophy of the International Organization for Standardization (ISO) and International Electrotechnical Commission (IEC) focusing on customers, leadership, personnel involvement, continual improvement, factual approach to decision-making, and mutually beneficial supplier relationships (Hurburgh, 2003). The ISO standard

provides a framework under which to evaluate operations. ISO is not the only QMS approach available, but generally industry specific QMS programs are designed around the ISO standard. ISO/IEC 17025 accreditation is a natural fit for a laboratory seeking to establish a quality management system. Many of the same processes must be done to succeed with both.

ISO/IEC 17025 (http://www.iso.org) was developed to update ISO/IEC Guide 25, create a laboratory application for ISO 9001/9002, and provide a basis for international acceptance. It is essentially a laboratory version of the ISO/IEC 9000 series. ISO/IEC 17025 is not an award for proficiency but instead is a statement that the lab has in place a quality system sufficient to assure accurate and reliable tests within customer requirements, if followed. (Tholen, 2002)

The ISO/IEC Guide 25 focused on laboratory technical capability. Example questions answered using this guide were: Are the technicians using the methods correctly? Are reports being generated that contain the correct interpretation of results? Competence rather than quality management was the main goal of ISO/IEC Guide 25. ISO 9001/9002 (1994 version) focus was still not on specific product quality; but it worked to help organizations establish quality systems and the required documentation. By verifying that quality policies were stated and followed, that there was a commitment from upper management to monitor and update the quality system, and that customer requirements were met, ISO 9001/9002 helped to initiate successful quality systems. (Tholen, 2002)

Laboratory Quality Management Systems (LQMS) are based on the total quality management (TQM) concept. There were five major contributors (Deming, Juran,

Feigenbaum, Crosby, and Ishikawa) to the TQM concept comes from combining their thoughts and ideas, quality management was created. (Kruger, 2001)

Deming's fourteen-point program is listed below (Deming, 1982). It contains many points specific to manufacturing. However, there are many underlying concepts that have been applied to any setting.

1. Create constancy of purpose for improvement of product and service.
2. Adopt a philosophy that mistakes/defects are unacceptable.
3. Cease dependence on mass inspection of finished products.
4. End the practice of awarding business on price tag alone.
5. Constantly and forever improve the system of production and service.
6. Institute modern methods of training on the job.
7. Institute modern methods of supervision (without pressure and distrust).
8. Drive out fear as a management tool.
9. Break down barriers between staff areas.
10. Eliminate numerical goals for the workforce.
11. Eliminate work standards and numerical quotas.
12. Remove barriers that hinder the production worker (i.e. incorrect supervision, poor quality raw materials, faulty equipment)
13. Institute a vigorous program of education and training for all employees
14. Create a structure in top management that will push every day on the above 13 points.

Three of Deming's major ideas were used to focus the work on the Iowa State University Grain Quality Laboratory's (ISU-GQL) QMS - continuous improvement,

training and elimination of fear (numbers 5, 6/13 and 8). These were chosen because they were the most applicable to a laboratory setting and fit best with the goals of the project.

Quality in any organization is an ever changing and improving process. This project is not the end of what needs to be done in the ISU-GQL. It is a good start that can be built upon in the future as more work is done, expectations of the clients are better known and processes are refined.

Deming emphasizes modern training methods. The idea that a new worker learns from an experienced worker, who learned from a more experienced worker, is ineffective. Such a system promotes inconsistency and fault. Training is a structured program that identifies to new workers what is expected of them and gives them the information they need to do their job effectively. Deming saw training as a long-term investment in the company. A university-based analytical laboratory is unique in that it experiences high personnel turnover. Turnover does not negate the value of long-term investment in training. This investment may or may not be fully realized in employees who only work for three years, but it can be realized in the improvement of training processes and trainers.

By eliminating fear, employees feel free to express their ideas and ask questions. This provides an invaluable resource on which to base review and improvement in the processes and employees themselves. Without their input, deficiencies may not be identified or fixed.

Juran, Crosby and Ishikawa felt strongly that quality must be lead by example. (Kruger, 2001) Employees will not see the importance of a quality management

system if all levels of management do not embrace and practice continuous improvement. And in addition, Feigenbaum felt that in TQM the responsibility for quality falls on all employees of a company. Quality improvement can not be assigned to a specific person or department, and be expected to succeed. Although the quality manager can be a very good resource for a laboratory, operators must practice good quality measures. A quality manager can be very useful as the group expert and as the person responsible for the upkeep of records and training.

The benefits of a well planned, implemented and maintained laboratory quality management system range from increased profit to a better working environment. Hurburgh stated in 2003 that organizations pursuing QMS can experience company growth, methods for problem recognition and resolution, time and effort savings, improvement in record maintenance, and overall laboratory consistency. Quality management systems in most cases are a means to increase profit from existing operations, rather than marketing tools for gaining new business.

## CHAPTER 2. CHALLENGES

The high personnel turnover is one of the areas that make a university-based research and service laboratory different from commercial analytical laboratories. Many university-based laboratories employ student workers that naturally move on to other employment. Students are permitted to work no more than twenty hours per week; they are enrolled in scheduled academic courses and outside class work; and they rarely work more than three years due to the usual four-year course plan.

The student worker is a valuable asset to a university-based analytical laboratory. They are primed for learning due to their college activities along with being able to learn at a rapid rate. Because of the large emphasis on computer skills at the university level, most student workers have at least a basic knowledge of computer software and hardware. This attribute allows more time to be spent in training on topics other than computer basics. Very often the skills and problem solving abilities that student workers learn and apply in a university-based analytical laboratory can be carried over to their academic field of study. At the Iowa State University Grain Quality Laboratory (ISU-GQL) such skills are seen as a significant benefit due to its commitment to the university's mission of further education. The ability to offer academic credit as well as pay for specialized, more creative work can be a strong incentive for employment.

There are also drawbacks with having a workforce that consists entirely of undergraduate students. Their short term of employment causes the training to hours worked ratio to be larger than that of a typical analytical laboratory. For the ISU-GQL this ratio is one hour trained for every 32 hours worked on average, based

on five hours trained per semester and working an average of ten hours per week. A typical laboratory has one hour trained for every 83 hours worked. The large discrepancy between university-based analytical laboratories and typical laboratories also accentuates the importance of continued data capture, especially from longer-term student workers. Frequently, these workers have gained a large amount of hands-on knowledge that can only be learned from experience.

Rapid turnover in a workforce also makes the use of custom programmed laboratory information management systems (LIMS) a problem. Custom LIMS can be very powerful, but significant training is usually required to use such a system. In settings where training time must be optimized, the use of a LIMS may not be efficient. For the ISU-GQL, off-the-shelf systems and task specific software were used. Such software, with which the students were familiar, allowed them to spend time learning the background of the processes they were to conduct. The drawback is that off-the-shelf software requires that the system conform to its constraints and configurations.

Employee responsibility is another concern with a rapid turnover workforce. Because of the length of time most students are employed, it is essential that responsibility be granted sooner. Allowing personnel to have more responsibility removes management pressure to micromanage. This requires upfront personnel evaluation at the beginning of employment to verify that responsibility is not being erroneously assigned. In most cases individuals can be identified easily for key tasks and trained accordingly. Retaining personnel that were placed in unsuitable positions causes animosity, friction and ultimately analysis delays or errors.

Earlier responsibility assignment requires frequent productivity assessments to allow for the maximization of output. Productivity assessments will be different for each laboratory. A major criterion is often analysis of samples per employee per a unit time. Comparison can sometimes tell if an employee needs more training or is not utilizing time wisely. As Deming observed, prudence should be used with this kind of analysis to identify the real reason for low productivity (Kruger, 2001). It may have nothing to do with the employee's abilities or work ethic, but rather with special customer requirements, unique situations, research projects, or equipment breakdown.

Rate of analyses per month is not viable for the ISU-GQL because the laboratory operates on a crop year basis with thousands of samples coming in the fall. These are analyzed as fast as possible and the late spring/early summer is spent with calibration, planning for the next crop year and maintenance. For laboratories who have a relatively consistent sample flow, monthly analysis rate can be helpful. The next sections describe the development of the quality management system for the ISU-GQL, beginning with the quality manual.

Another challenge the university-based analytical laboratory faces is the incorporation of research activities. Research in this laboratory setting is inherently unstructured, transient and innovative. It does not lend itself to future forecasting or easy definition. Typical laboratory practices can be defined and documented because they are run everyday and conform to accepted methods.

Indirect, secondary analysis methods utilized in the ISU-GQL, such as near-infrared spectroscopy (NIR), pose yet another type of challenge to a university-

based analytical laboratory. NIR relies on calibrations, which provide more possibilities for error and their reliability is based on the quality of samples used in the calculations. The flow-through method of some NIR machines makes it difficult to use absolute reference standards. Therefore, a check sample with the same structural properties (i.e. size, shape and flow characteristics) as normal samples must be used. To meet this problem, NIR technology developers and their partners are working to formulate reference standards that mimic the structural properties of common analysis materials such as corn and soybeans.

## CHAPTER 3. OBJECTIVES AND ORGANIZATION

A quality management system (QMS) and/or ISO/IEC 17025 accreditation can be beneficial to any analytical laboratory interested in improving results, efficiency, employee performance, and customer relations. Therefore, the objectives of this research were to develop a laboratory quality management system for the ISU-GQL, to apply for ISO/IEC 17025 accreditation and to provide a structure for university-based analytical laboratories to establish quality management systems.

ISU-GQL upper management's specific goals for pursuing ISO/IEC 17025 accreditation were 1) to improve accuracy, 2) reduce cost, 3) increase throughput, and 4) provide visible credibility. Only one of these objectives, credibility, directly used the quality management system in marketing of services.

Important considerations in this work included writing and implementing laboratory procedures, standardizing job descriptions, creating a quality manual, and generating working control charts. Typical word processing and spreadsheet software along with custom program routines were utilized to simplify the ISU-GQL quality management system. Custom software and operations are not well suited to the more continuous personnel turnover and historically less structured practice of the university research setting. Special attention was paid to the initial and continuing training of technicians and support staff.

This thesis was organized to touch on the central parts of a quality management system. Documentation examples were included to provide direction for other university-based analytical laboratories.

## CHAPTER 4. QUALITY MANUAL

A quality manual contains all the processes that an organization uses to execute its quality management system, technical competence, and validated results. The Iowa State University Grain Quality Laboratory (ISU-GQL) Quality Manual was created using the SHOQ Quality Assurance Manuals Inc. software ISO 17025 Quality Manual Template v.3 (2000) obtained from the Association of Analytical Communities (AOAC International). Although the template did require an up-front investment, it was well worth the time saved in development. Templates can be very useful for laboratories without parent companies or who are the first of a series to develop a quality manual compliant with the ISO 17025 standard. There are many templates available as well as companies that will develop a quality manual for a fee. Examples of these companies include Quality Systems Innovations, Incorporated (Effort, PA http://www.qsinnovations.com); EHS Services, Incorporated (1-877-670-7841 http://www.ehsservices.com); and To The Point Consulting (Oudenaarde, Belgium http://www.17025.homestead.com). (web search 6-10-04, www.google.com) The SHOQ template was very effective at providing a structured base. It was already cross-referenced and numerically aligned to the ISO 17025:1999 and 9001:2000 standards, which will allow the manual to be used in an audit situation.

Additions, substitutions and modifications were made to allow the Quality Manual to be unique to the ISU-GQL. A major benefit of a unique quality manual is acceptance of laboratory staff to the manual and its use, taking away the stigma of a cookie-cutter model being applied to every laboratory. A unique quality manual also

allows for maximization by streamlining it to include only items appropriate and rewriting sections for easy understanding by personnel who are not quality management system experts. The laboratory logo was added for aesthetic purposes and to make the quality manual more identifiable.

The quality manual is a key place to identify the organizational structure and typical processes of a laboratory. A full laboratory organizational chart for the ISU-GQL can be seen in Figure 1. Management was defined in the ISU-GQL as the professor in charge/general manager, laboratory and service manager, and the quality manager.

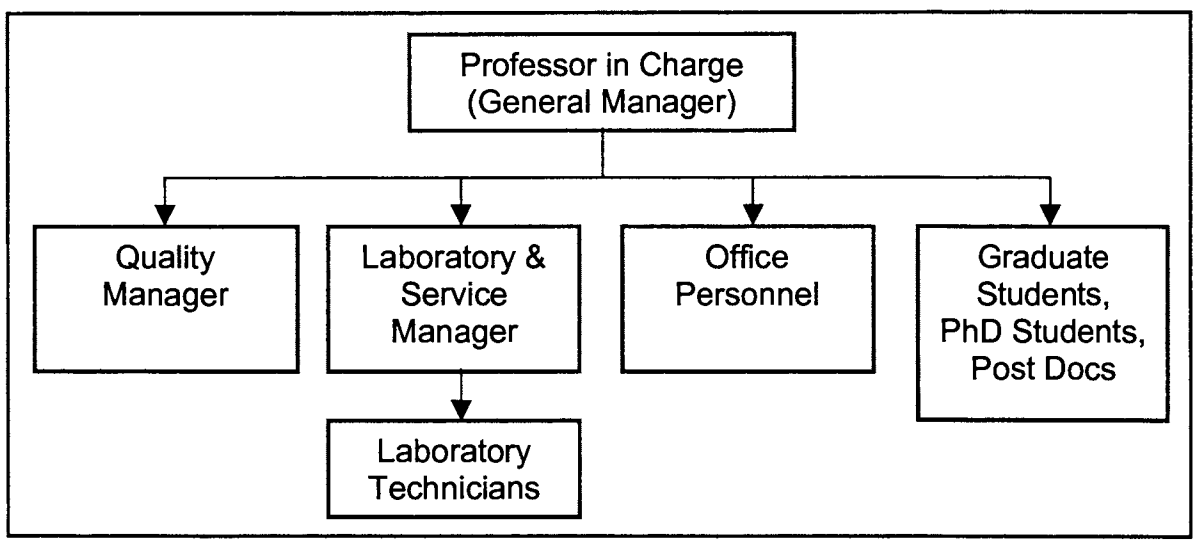


**Figure 1.** Iowa State University Grain Quality Laboratory Organizational Chart

Typical processes in a laboratory can also appear in the quality manual. Figure 2 is an example of one major process in the ISU-GQL, the route of service samples through the laboratory. This particular flowchart was helpful to standardize the treatment of service samples.

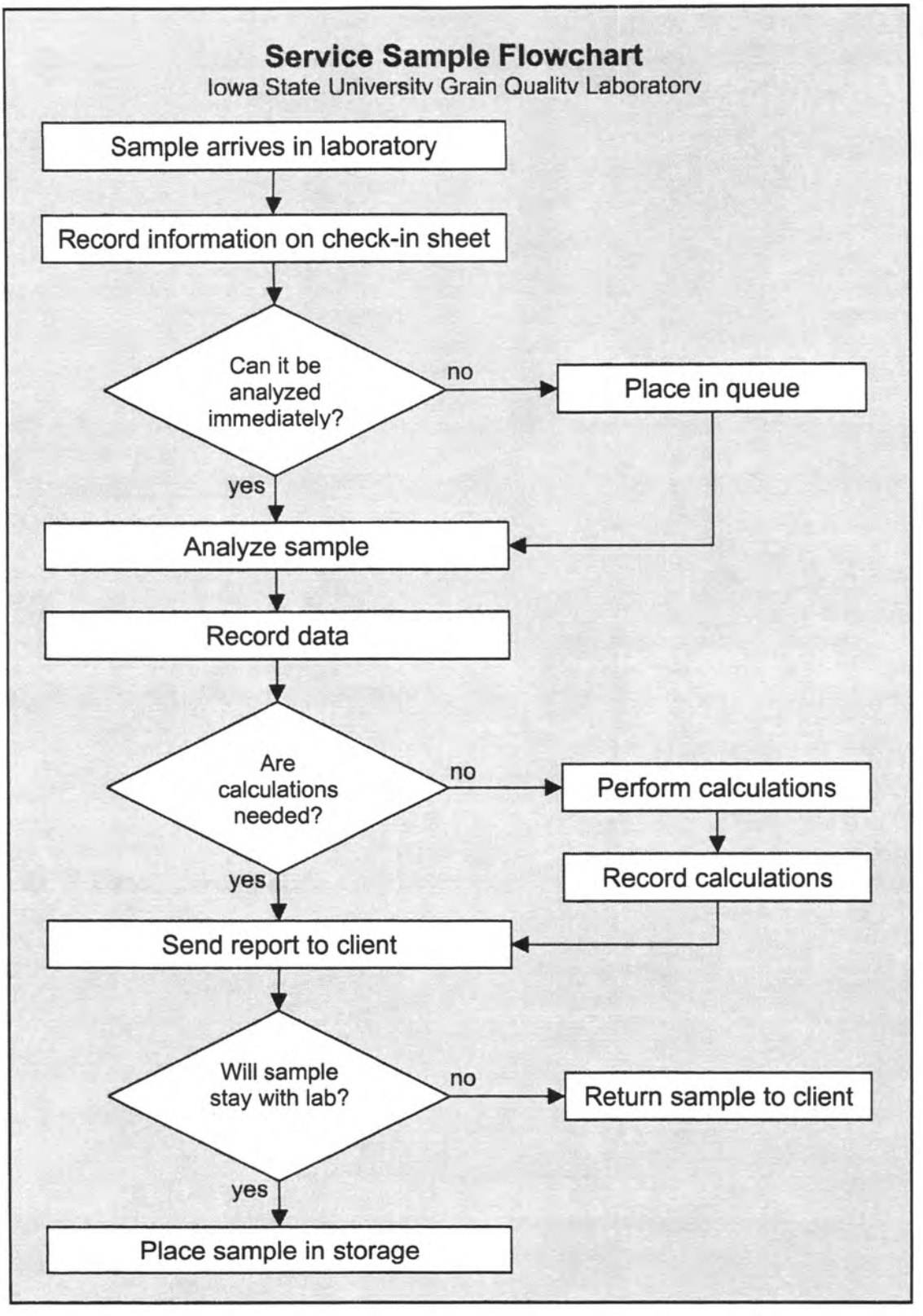


**Figure 2.** Flowchart example for route of service samples

The quality manual is a controlled document. The definition of a controlled documents is that only copies that are officially issued to an individual or area are a true record of what the organization will be judged against. This prevents the quality manual from being maliciously altered or from multiple versions being referenced by personnel.

A website can be a very effective way of controlling documents. It allows one person to keep one record up to date while multiple people can view it in many locations. Software knowledge is a draw-back of a website based quality manual. Locking records and documents in read-only format and including a copyright and uncontrolled watermark addressed document safety. For the ISU-GQL, records contain information pertaining to analysis of samples and documents include the quality manual, laboratory procedures and job descriptions. The ISU-GQL maintains the official quality manual on the internal server in read-only Microsoft Office software with user password entry. Appendix B contains an uncontrolled copy of the ISU-GQL's quality manual as indicated by the words UNCONTROLLED printed on all pages.

## CHAPTER 5. LABORATORY PROCEDURES

Each laboratory should use a procedure format that works well for them. The original ISU-GQL procedure format was created using input from upper management along with requirements of the ISO 17025 standard. An example of a laboratory procedure used in the ISU-GQL can be found in Appendix C.

A laboratory procedure is meant to be concise and easy to read, but also give enough information to be understood by anyone with general laboratory knowledge. Procedure formats contained the basic parts as defined in Table 1. Beneficial additions could include the company or laboratory logo, departmental differentiation,

**Table 1.** Laboratory procedure component definition

| *Component* | *Purpose* |
|---|---|
| Header | document tracking, identify when and who made changes |
| Procedure Name | unique to procedure, identifiable by laboratory personnel |
| Background | places each procedure in the context of the specific laboratory |
| Objective | what should be accomplished when the procedure is carried out correctly |
| Procedure Detail | specific steps, numbered, divided as needed for different sub-processes (ex. starting machine, running sample, shutting down machine) |
| Evaluation and Monitoring | when the procedure should be reviewed, definition of non-conformance to procedure |
| Corrective Actions | Definition of actions to be taken if evaluation and monitoring shows the procedure is not effective |

pictures of equipment or typical results. Laboratory Procedures, for ease of use, should be explicit enough to carry out the work but as short as possible. The goal was laboratory procedures that were no more than two pages. The unique format utilized for the ISU-GQL contained the seven major components, as shown in Table 1. For the purposes of the ISU-GQL quality system, standard operating procedures (SOPs) and laboratory procedures were deemed one and the same. SOPs are most commonly utilized in industry to define the processes used in the production environment, while laboratory procedures refer to in-house analytical laboratories.

Technicians wrote the majority of the laboratory procedures. This was beneficial for many reasons. It gave laboratory technicians ownership of the quality management system and enabled them to know that they were a part of the process. It also improved the accuracy of the laboratory procedures. Not all procedures followed the exact layout and steps as those defined by the manufacturer. It was important to capture the true process as performed. By having the laboratory technicians write the procedures, deviations from the manufacturer's suggestions were identified and training could be altered if needed.

The first round of writing laboratory procedures captured what was happening in the laboratory. Later reviews and revisions will update the correctness and efficiency of procedures. An example was the Grainspec instrument. While the technical manual provided by Foss Electric was very thorough, it contained 43 pages. The ISU-GQL laboratory procedure written for this instrument was one page. The technical manuals that accompany machines are important for specific problems and troubleshooting, but are harder to use for day-to-day operations.

Finally, having the laboratory technicians write laboratory procedures allowed them to put into words what they have been taught first hand through oral training. This decreased the chance that data will be lost when technicians leave the laboratory. It also allowed the Laboratory and Service Manager to assign laboratory procedure writing to those technicians that have been working with that particular process the longest.

Information that could not be filled in by laboratory technicians was supplied in by management or by a combined effort of management and the laboratory technicians. Management also completed laboratory procedures for which technicians had little or no experience. Examples of these procedures include administrative processes (those processes done only by management) and very technical processes that would be given to laboratory technicians later. The approval chain for laboratory procedures worked up from the bottom of the organizational chart. The process started with the laboratory technicians writing the laboratory procedures, the quality manager and laboratory and service manager then reviewed them and gave feedback and final approval was given by the professor in charge/general manager.

Laboratory procedures accentuate the problem of controlled copies. Until the laboratory is operated entirely electronically, printed copies of documents will be necessary. All laboratory staff must understand that the printed copies are uncontrolled and if in doubt, they should refer back to the controlled copy stored on the internal server. During development of the ISU-GQL's quality system, these documents (laboratory procedures and the quality manual) were located on a shared server, allowing easy access for key personnel. In the final stages of development,

all documents were moved to a password-protected section of the ISU-GQL's server. Access to make changes is only granted to the quality manager while all other personnel may view documents in read-only format.

A header tracking system is also a vital part of document control. An example of a header used for the ISU-GQL laboratory procedures is found in Figure 3. It contains four sections, which are location and name of the file, page number and number of total pages per procedure, version tracking (changes made when and by whom), and ISO/IEC 17025 reference section identification.

\\Grainbin\users\Shared\Laboratory Quality System\Lab Procedures\Lab Tech Training.doc 1/2
Original Version: 2-18-2004; By: AmH Last Revision: 6-11-2004; By: AmH Approved: 6-30-2004; By: CRH
Next Scheduled Revision: XX-XX-XX ISO 17025 Reference Section(s): X.XX

**Figure 3.** Example of a document tracking header

Revision plays a considerable role in effectively using laboratory procedures in a laboratory quality management system. Although the short length of this project did not allow for the review and revision of all laboratory procedures, it is important to continue this revision process on a scheduled basis. Due to the mass of samples that are analyzed in the fall semester (beginning of the crop year), a late spring, early summer revision schedule would be appropriate. The first step in revision is management review sessions. Management can provide insight into maximizing productivity and technical accuracy. These sessions should also review productivity and outputs from the previous season. Later laboratory technicians should be able to give feedback as to the practicality of the proposed new or updated procedure.

# CHAPTER 6. TRAINING

The Iowa State University Grain Quality Laboratory (ISU-GQL) training program has progressed over the last two years. Before the start of this project, no formal training was given. All training was done hands-on by more experienced personnel, and no records were kept. Safety was the initial stimulus for a training program. Starting with the guidelines provided by the Iowa State University's Department of Environmental Heath and its Safety Chemical Hygiene Plan (ISU-EH&S, 1991), a program was designed to address important laboratory safety considerations. Areas of focus included content of laboratory safety standard, material safety data sheet location and usage, standard operating procedure location and usage for gas tanks, emergency procedures, personal protective equipment location and usage, Chemical Hygiene Plan location and content, and general procedures for cleaning and timesheets. It was conducted in the laboratory as a discussion/demonstration session.

As the training program progressed additional sessions about laboratory operations were added to the safety content. The current training program includes all the safety areas as well as laboratory protocol, semester project overviews, instrumentation discussion, laboratory procedure review, instrument demonstration, and individual evaluation. The forms, an example of materials provided for a typical training session, and Microsoft PowerPoint presentations are included in Appendix B.

Training is done every academic semester (fall, spring, summer). All personnel must go through full training the first semester of employment and every fall,

thereafter. The other semesters they are allowed to attend a refresher session. If personnel are found by the quality control charts to be lacking in their competency, the Laboratory and Service Manger can require that they attend the next full session to continue employment. This system enables personnel to be adequately trained without extra or redundant time.

To emphasize the importance of training, personnel are compensated for training time. Although this system works well for the ISU-GQL, other analytical laboratories may find other or additional training incentives. Examples of the training incentives in the corporate world could include vacation time, stock options, certificates, recognition events, etc.

The training program is now divided into three sessions.

1) Safety and laboratory operations. This session takes up to one hour depending on the number of questions asked and the length of discussion. The number of laboratory technicians in each of the safety and laboratory operations sessions varies from three to ten. A Microsoft PowerPoint presentation highlights the most important information and gives a guide as the session progresses. An example of a presentation can be found in Appendix B. Since the laboratory technicians are undergraduate students with classroom experience, the PowerPoint presentation is beneficial to give the session a sense of structure and routine.

2) The second session addresses laboratory standard procedures. This session takes two to four hours. Presentation time and depth are variable depending on the number of technicians in the session (5 maximum) and the experience of each participant. Small sessions are important to allow the trainer enough time with each

person to evaluate their abilities. This session includes a conference room and a hands-on laboratory component. A Microsoft PowerPoint presentation is used in the conference room section, as a guide. An example of this presentation is also provided in Appendix B. The main focus of the conference room part of this session is discussion. This time helps the trainer to identify specific areas that need to be addressed or expanded in the hands-on part of training. After sufficient discussion, which is decided subjectively by the trainer, the group then moves into the laboratory for demonstration on all major instrument groups.

The laboratory technicians are also shown where they can access laboratory procedures and safety information. Currently, this repository of information is found in one central location in the laboratory space. A current uncontrolled copy of each laboratory procedure is also located next to the machine or in the area where it is used. Work is underway to make the documents available on-line for quick query and reference. Due to the large number of instruments utilized in the laboratory and the frequency of their use, instruments are selected for the hands-on section based on current work demands. The laboratory and service manager gives specific instruction to individuals who will use more advanced instrumentation. The final step of laboratory procedures training is for the trainer to sign-off each instrument on the technician's lab procedure training record grid, a brief example of the grid can be seen in Figure 4. Appendix B contains the complete training grid. This system requires the trainer to observe the technician reading, observing and performing the procedure before approval is granted.

| Employee Name: | | | | Date: | | |
|---|---|---|---|---|---|---|
| *Procedure / Task* | *Lab Procedure Code / Revision #* | *Employee Read Procedure* * | *Employee Observed Procedure* ** | *Employee Performed Procedure* ** | *Trainer Comments* ** | *Training Approval* ** |
| Foss Infratec 1229 | | | | | | |
| Foss Infratec 1241 | | | | | | |
| Bruins Omega Analyzer G flow | | | | | | |
| * Employee initials column entries<br>** Trainer initials or completes comments for column | | | | | | |

**Figure 4.** Example of a laboratory procedure training record grid

3) The third training session is a refresher session. It includes updates made to laboratory procedures, a semester project overview and announcements that are pertinent. This session is meant to be short, less than 30 minutes. It emphasizes the importance of continued training and gives formal time for interaction and knowledge sharing. The only limit on group size is the number of continuing technicians. The refresher session is required by all laboratory technicians who are returning and wish to continue their employment. The past training records are reviewed by the laboratory and service manager and the quality manager to determine those returning.

Training sessions are scheduled in the third week of fall and spring classes and the second week of summer classes. This method allows the Laboratory and Service Manger to finish hiring new personnel and confirm those that are returning,

while still providing training quickly enough to be beneficial. Two or three date and time combinations for each session are planned to accommodate the variety of course schedules of the technicians. This is a major difference from other analytical laboratories in that the ISU-GQL personnel are undergraduates, who work only up to 20 hours per week, scheduled around their class schedules. In a typical analytical laboratory, sessions could be planned based on other factors such as time of day, date of hire, scheduling of laboratory work, etc.

Historically, formal training was done solely by the Quality Manager. This was primarily due to the way the training program was developed - being led by the Quality Manager from initiation. In the future, the Laboratory and Service Manager will perform the formal training in collaboration with the Quality Manager. The role of the Laboratory and Service Manager in training helps to emphasize the importance of training for the laboratory's quality management system, while also making sure information provided is accurate.

The Laboratory and Service Manager, along with other laboratory technicians, frequently provide informal training. Daily, guidance is given concerning the laboratory's analysis flow, review of control chart results, instrument trouble-shooting, new instrument use, and general laboratory operations. This kind of training can sometimes teach more than a lecture or demonstration, although it is not easily documented. It also gives more experienced technicians the opportunity to pass their knowledge along. This activity is a vital type of data capturing activity that may not produce a document, but is still carrying on knowledge to future employees.

Training records are kept in the Chemical Hygiene Plan book found in the laboratory. They include signature sheets for each session attended, copies of handouts and presentations used, and reference to who provided the training. Individual technician training records are kept in the human resource files. The lab procedure training record, confidentiality agreement, and personal information and ISU-GQL identification can be found here. Training records are vital for the identification of technicians who are due for further training as well as for keeping a history of which technicians are trained to perform specific processes. An example of all records used can be found in Appendix B.

Control chart results can also indicate if further training is needed. Figure 5 shows an example of a control chart that may indicate the need for further training. Research is done to discover the root cause of divergence. It may have resulted from a keying error or from technical skill deficiencies. There are many factors that contribute to the variation in data: sample, environment, machine, or technician. If the first three are found reasonable, the operator may be at fault. Therefore, when alarms are present in the control charts, or patterns are detected, further technician training may be needed. Usually, only a refresher on the proper sample preparation and instrument procedure is needed. In extreme cases, technicians need to be thoroughly retrained.

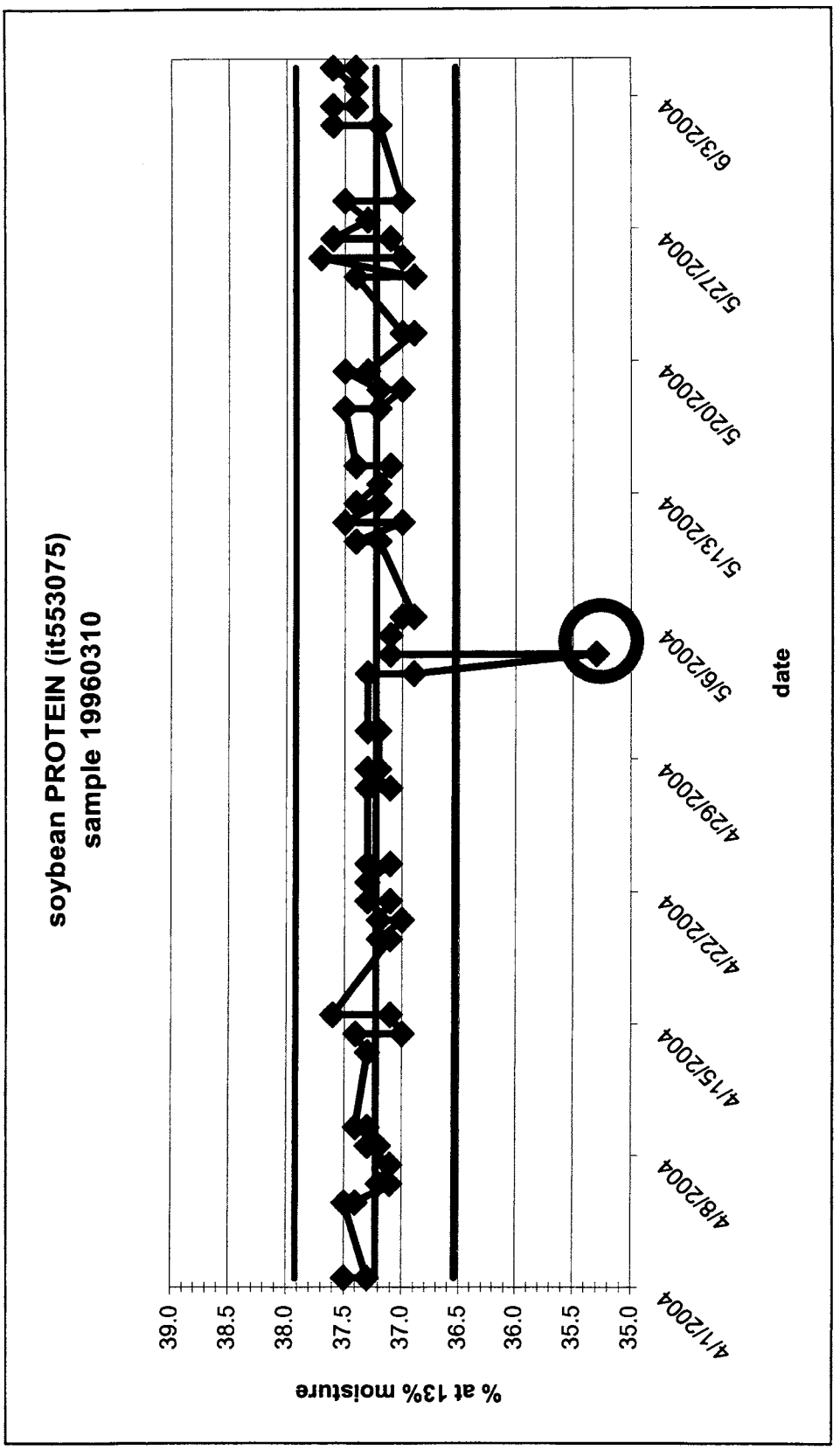


**Figure 5.** Sample quality control chart indicating the possible need for further training

# CHAPTER 7. QUALITY CONTROL

A quality control program was developed to support ISO 17025 section 4.2 and enable ongoing evaluation of internal and external results. Quality control is an essential part of the ISO 17025 standard and the laboratory quality management system philosophy. There is no requirement that quality be high, although this would be favorable for laboratories who want to keep and gain clients, only that quality be maintained at a level that is efficient for the laboratory. (Tholen, 2002) The Iowa State University Grain Quality Laboratory (ISU-GQL) has collected data for many years, which makes current analysis useful. The goal of this project was to develop a quality control process that could be utilized in the future, not to fix problems that were found. Quality control measures utilized in the laboratory included:

- near-infrared spectroscopy (NIR) daily check samples
- reference checks
- working accuracy of instrumental analysis
- precision of instrumental analysis

## Near-Infrared Spectroscopy Daily Check Samples

Daily analysis of check samples run on near-infrared spectroscopy (NIR) machines evaluates the stability of instrumental analysis. Using the same sample over a long period of time enables the ISU-GQL to identify departures from normal analysis before service or research work is run each day. The General Manager and/or Laboratory and Service Manager select check samples. The main attribute that makes a service sample a good check sample is the volume the laboratory has

in long-term storage. There must be enough sample to run for an adequate length of time while taking samples to send for chemistry and replacing the sub-sample stored in the laboratory. Periodically, the sub-sample that is run everyday must be replaced. This need occurs due to operator error, which causes sample to be lost or contaminated by other samples, time expiration (usually one year), visible sample deterioration, container deterioration or other causes.

The large sample is stored in an industrial freezer that is held at 2-4 °C. The bulk sample is mixed for 5 minutes making sure to reach all the way to the bottom of the barrel; this guarantees the next sub-sample taken is representative. The container is filled and labeled as a check sample with the sample number. The new check sample is returned to the lab at least the night before it is to be run for the temperature to equilibrate. The check sample log records times when samples are replaced.

The operator records daily check sample data on check sample data sheets. The data is later keyed into a Microsoft Excel Spreadsheet for analysis. Check samples are always run as the first activity of the shift. This routine represents a significant expense for the laboratory; in that approximately one hour per day of technician time is taken ($50/week, approximately $2000/year assuming 40 weeks/year operation).

The quality control charts for the ISU-GQL were constructed using standard quality control chart format and daily check sample data. The standard format can be seen in Figure 6. The centerline was set at the overall sample average ($X_{bar}$) which included yearly fluctuation. The Upper Control Limit (UCL) and Lower Control

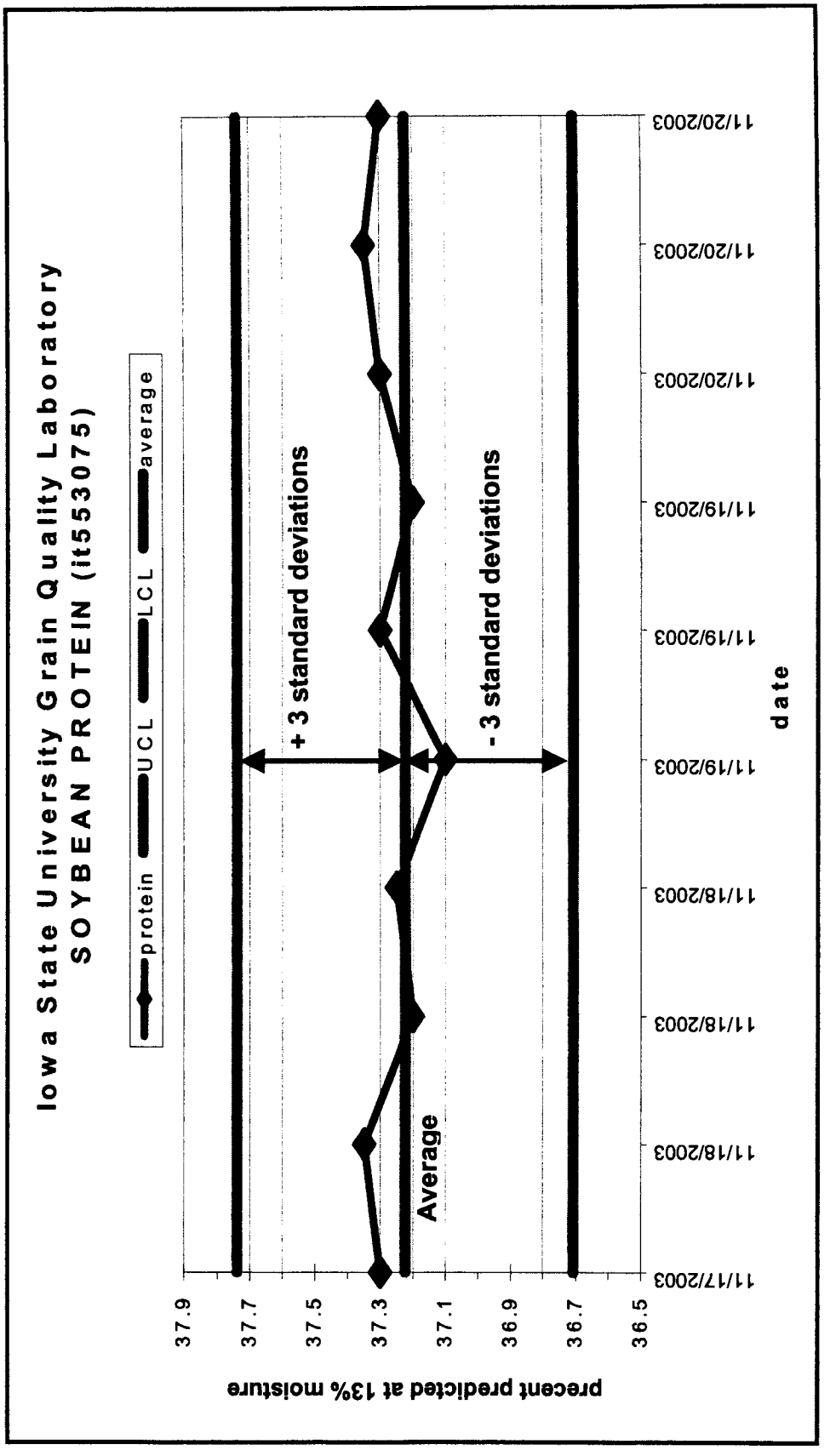


**Figure 6.** Standard quality control format used by the Iowa State University Grain Quality Laboratory

Limit (LCL) were set at the centerline plus or minus three standard deviations. An action point is one data point outside the UCL or LCL. The assumption is that if a data point is beyond three standard deviations, the probability of that event occurring randomly is very small. Other lines can be set at plus or minus 2 standard deviations and plus or minus 1 standard deviations and can be earlier indicators of process change. A benefit of using extra lines is that there is more control over the slightest change in the process. More control limit lines were not utilized in the ISU-GQL due to the apparent natural, yearly fluctuation in check sample results.

An example of the yearly fluctuation can be seen in Figure 7. The chart was constructed using check sample data collected over the last five years. For this specific chart, the soybean check sample 19960310 was used on the Infratec model 1229 serial number 553075 and the predicted protein value was charted.

Pressure (mb), relative humidity (%), temperature (°C), date, and time were all analyzed for a correlation that could be used to predict the fluctuation. A low in the check sample data can always be seen in the winter months. If the correlation analysis would have succeeded, the quality control charts could be constructed using a centerline that was predicted using correlating factors. The yearly fluctuation would then be taken into account and any alarms that were set off by the control charting rules would have represented a process change. Unfortunately, no correlations involving laboratory climate factors explained the fluctuations. The comparison between relative humidity and soybean protein predictions from Infratec serial number 553075 can be seen in Figure 8. The plot shows that there is no direct correlation due to the even coverage of points throughout. Also, the

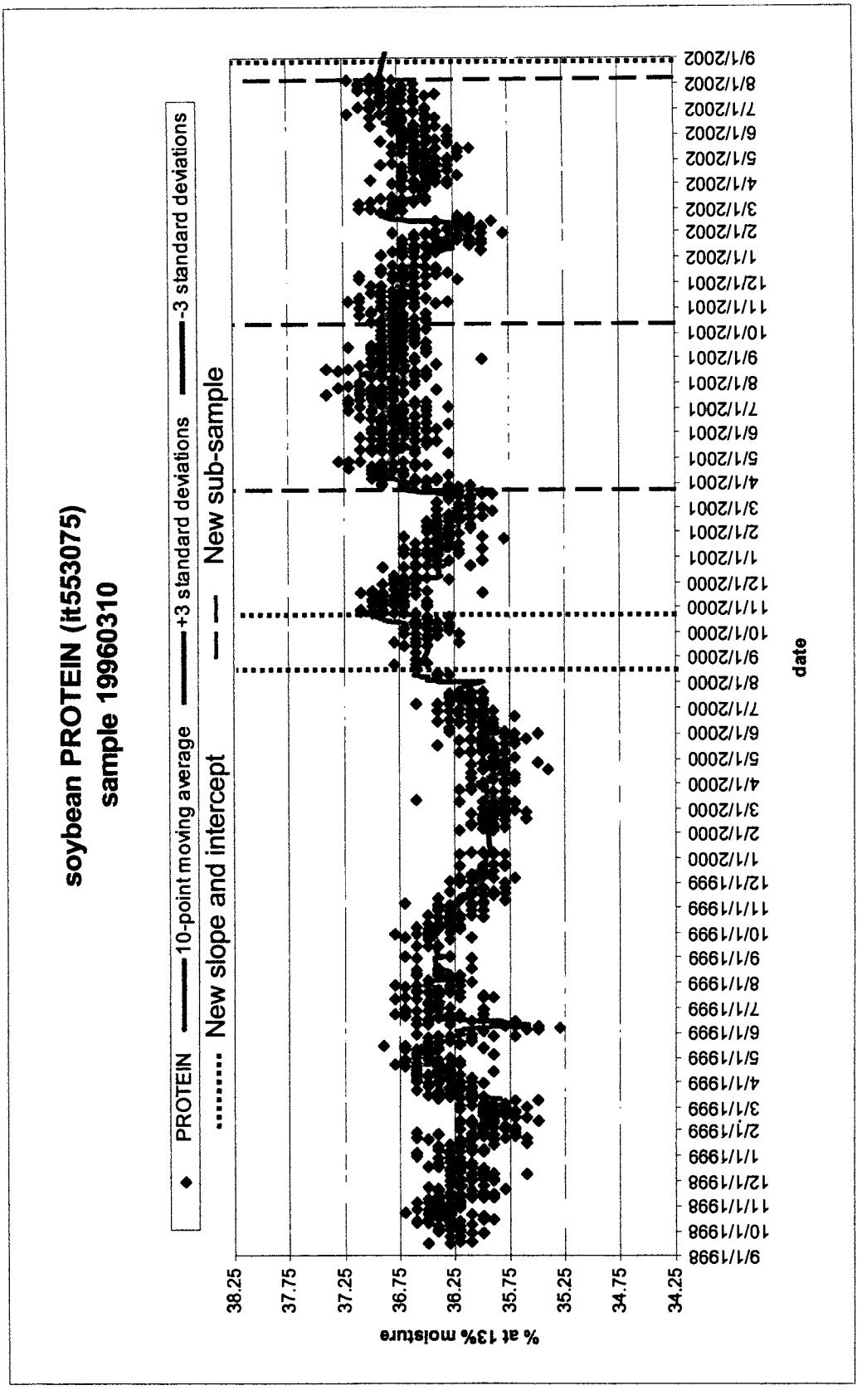


**Figure 7.** Problems created by yearly check sample prediction fluctuation

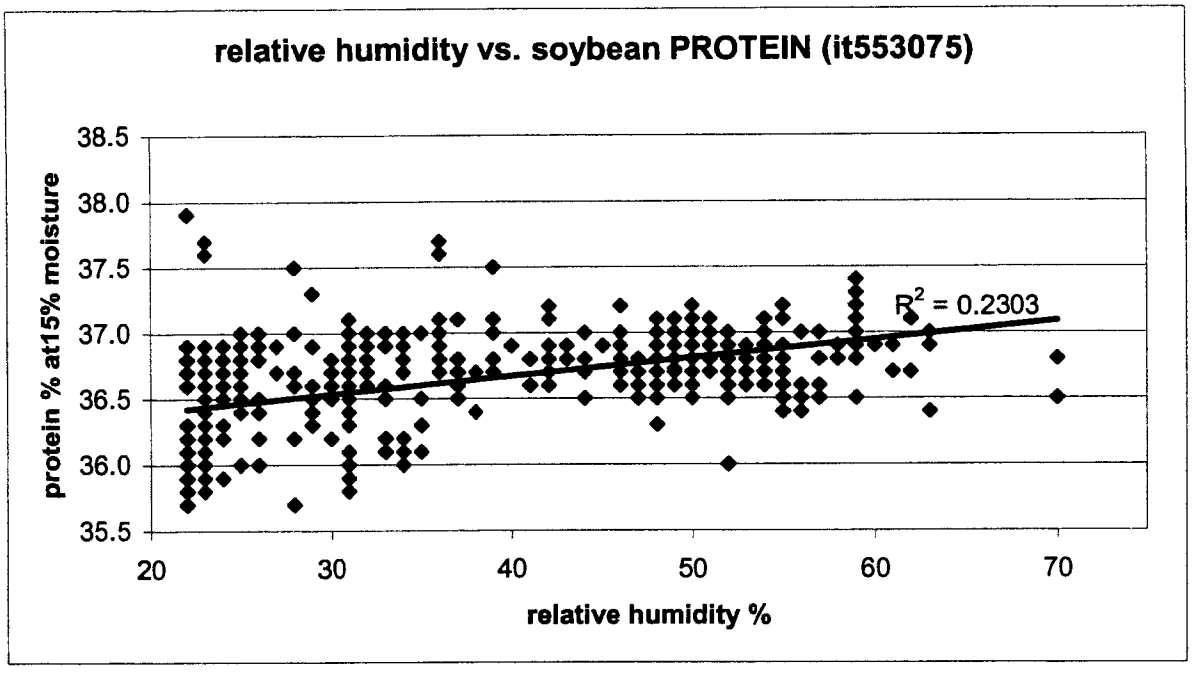


**Figure 8.** Correlation between environmental relative humidity and soybean protein predictions for Infratec serial number 553075

coefficient of determination ($R^2$) value is only 0.2303, which is well below an acceptable number for confidence in a relationship between relative humidity and soybean protein on the Infratec 553075. The other correlation factors displayed similar charts with no $R^2$ value above 0.4006.

## Reference Checks

Proximate, amino acid and fatty acid reference analysis are currently outsourced to external laboratories that can do these tests more efficiently than the ISU-GQL. These tests use commonly accepted direct primary methods, rather than indirect secondary methods. Reference checks are performed to verify the stability of check samples, verify the stability and precision of reference tests, and provide data for calibration purposes. The stability of check samples must be verified due to the

confidence placed in the sample when used for control chart analysis. If the sample composition is changing, the laboratory and the NIR instrument readings must be able to identify the change. Because external laboratory results are used for calibration, customer reports and sample status verification, they must be verified. When data is used for customer reports, usually one sample is sent for analysis and reported. The ISU-GQL must have assurance in the daily analysis of samples by its outsourced laboratory. When check or standardization samples are resubmitted over time or blind duplicated on one date, they can be used to evaluate the stability and precision of reference tests, respectively.

In the effort to determine if the fluctuations in NIR check sample data were from deterioration of the sample or from laboratory problems, external laboratory chemistry checks were performed. This effort is also an example of control chart development for external laboratory data. Although this example was for a check sample, it also shows how similar checks are performed to document stability of externally supplied data allowing for correction of bad sets of data.

Two samples of each, corn and soybeans, were taken from the sub-sample located in the laboratory. They are submitted to the outsourced laboratory company as per the contract held with them. The samples were blind-coded so they could not be identified by the outsourced laboratory as a check sample or as a duplicate. This coding was vital to the integrity of the process to assure that results were representative. In some cases the four samples were sent with others at the same time. The raw data can be seen in tables 2 and 3. Whether they were sent separately or with other samples, the data received was not satisfactory. Chemistry

**Table 2.** Soybean check sample #19960310 chemistry data over time

| Date | Moisture (%) | Protein (%) | | | Oil (%) | | |
|---|---|---|---|---|---|---|---|
| | Forced Draft Oven [a] | Combustion-Feed [a] | 13% moisture [b] | dry basis [b] | Crude Fat[a] | 13% moisture[b] | dry basis[b] |
| 1996 crop year [c] | 10.66 | 37.80 [d] | 36.81 | 42.31 | 18.27 [d] | 17.79 | 20.45 |
| 6/10/2003 | 3.07 | 41.06 [d] | 36.85 | 42.36 | 19.36 [d] | 17.38 | 19.97 |
| 6/10/2003 | 3.47 | 39.98 [d] | 36.03 | 41.42 | 19.00 [d] | 17.12 | 19.68 |
| 8/19/2003 | 3.46 | 40.29 [d] | 36.31 | 41.73 | 18.85 [d] | 16.99 | 19.53 |
| 8/19/2003 | 3.46 | 40.10 [d] | 36.14 | 41.54 | 18.69 [d] | 16.84 | 19.36 |
| 12/2/2003 | 5.99 | 39.62 [d] | 36.67 | 42.14 | 19.45 [d] | 18.00 | 20.69 |
| 12/2/2003 | 6.53 | 38.30 [d] | 35.65 | 40.98 | 18.14 [d] | 16.88 | 19.41 |
| Average | 5.23 | 39.59 | 36.35 | 41.78 | 18.82 | 17.29 | 19.87 |
| Standard Deviation | 2.76 | 1.15 | 0.45 | 0.51 | 0.50 | 0.46 | 0.52 |

[a] taken from the external laboratory chemistry report

[b] calculated

[c] represents the original analysis reported by the external laboratory in the year of harvest

[d] mean values with the same superscript within each column are not significantly different ($P > 0.05$)

**Table 3.** Corn check sample #20020020 chemistry data over time

| Date | Moisture (%) | Protein (%) | | | Oil (%) | | |
|---|---|---|---|---|---|---|---|
| | Forced Draft Oven [a] | Combustion-Feed [a] | 15% moisture [b] | dry basis [b] | Crude Fat [a] | 15% moisture [b] | dry basis [b] |
| 2003 crop year [c] | 8.06 | 8.59 [d] | 7.94 | 9.34 | 6.71 [d] | 6.20 | 7.30 |
| 6/9/2003 | 11.15 | 8.51 [d] | 8.14 | 9.58 | 6.77 [d] | 6.48 | 7.62 |
| 6/9/2003 | 10.99 | 8.23 [d] | 7.86 | 9.25 | 7.00 [d] | 6.68 | 7.86 |
| 8/19/2003 | 11.50 | 8.04 [d] | 7.72 | 9.08 | 6.76 [d] | 6.49 | 7.64 |
| 8/19/2003 | 11.51 | 8.30 [d] | 7.97 | 9.38 | 6.62 [d] | 6.36 | 7.48 |
| 11/26/2003 | 9.99 | 8.04 [d] | 7.59 | 8.93 | 6.63 [d] | 6.26 | 7.37 |
| 11/26/2003 | 10.22 | 8.02 [d] | 7.59 | 8.93 | 6.64 [d] | 6.29 | 7.40 |
| Average | 10.49 | 8.25 | 7.83 | 9.21 | 6.73 | 6.39 | 7.52 |
| Standard Deviation | 1.22 | 0.23 | 0.21 | 0.24 | 0.13 | 0.17 | 0.19 |

[a] taken from the external laboratory chemistry report

[b] calculated

[c] represents the original analysis reported by the external laboratory in the year of harvest

[d] mean values with the same superscript within each column are not significantly different ($P > 0.05$)

analysis was reported on an as-is basis by forced draft oven (moisture percentage) combustion feed (protein percentage) and crude fat (oil percentage). The calibrations used in predicting machines at ISU-GQL rely on a calculation for protein and oil based on moisture content. Therefore, these constituents were calculated on a 13% and 15% moisture basis for soybeans and corn respectively. Issues with the data have been discussed with the outsourced laboratory company and efforts are being made to improve results for this purpose and others throughout the laboratory.

While the theory behind this method was valid, it proved to emphasize chemistry problems more than providing evidence to the deterioration of check samples. Some indication was given by the data that the check samples, if changing at all, are doing so at such a nominal rate as to not be a concern. Figures 9 and 10 show that when the duplicates were averaged, they stayed consistent with the original chemistry done.

Internal laboratory duplicates are performed when samples that are scanned using predicting machines (Infratec, Grainspec, GAC, etc.) are also analyzed within ISU-GQL (as opposed to outsourced results) with basic analytical methods. At this point the tests covered by this process are oven moisture, pycnometer density and test weight. Students are running internal laboratory duplicates. Although they are trained, it is not the same as having professional technicians performing advanced chemical analysis. For this reason, reference checks are not the only quality control measure utilized.

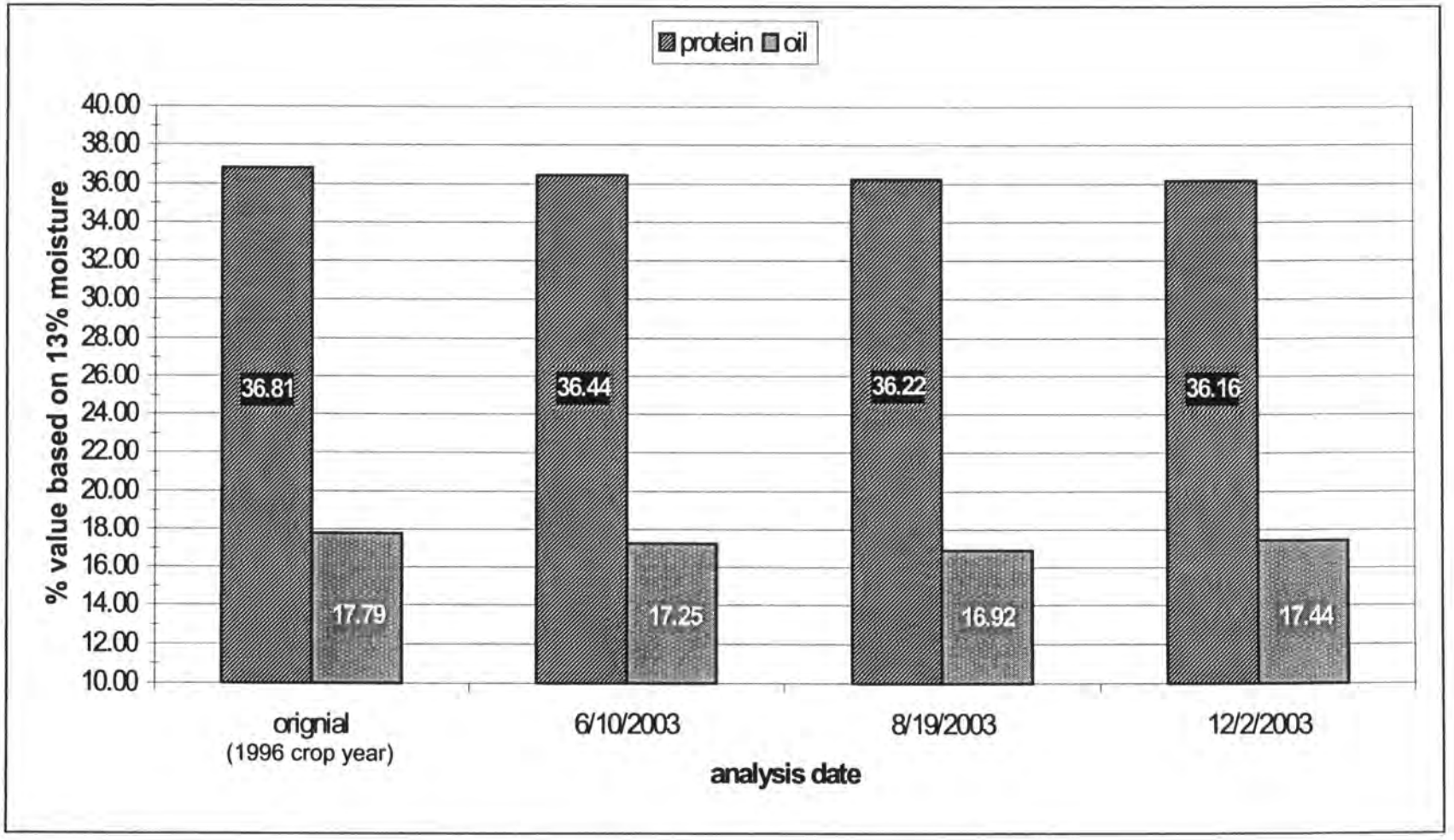


**Figure 9.** Soybean check sample chemistry over time (average of duplicate by date)

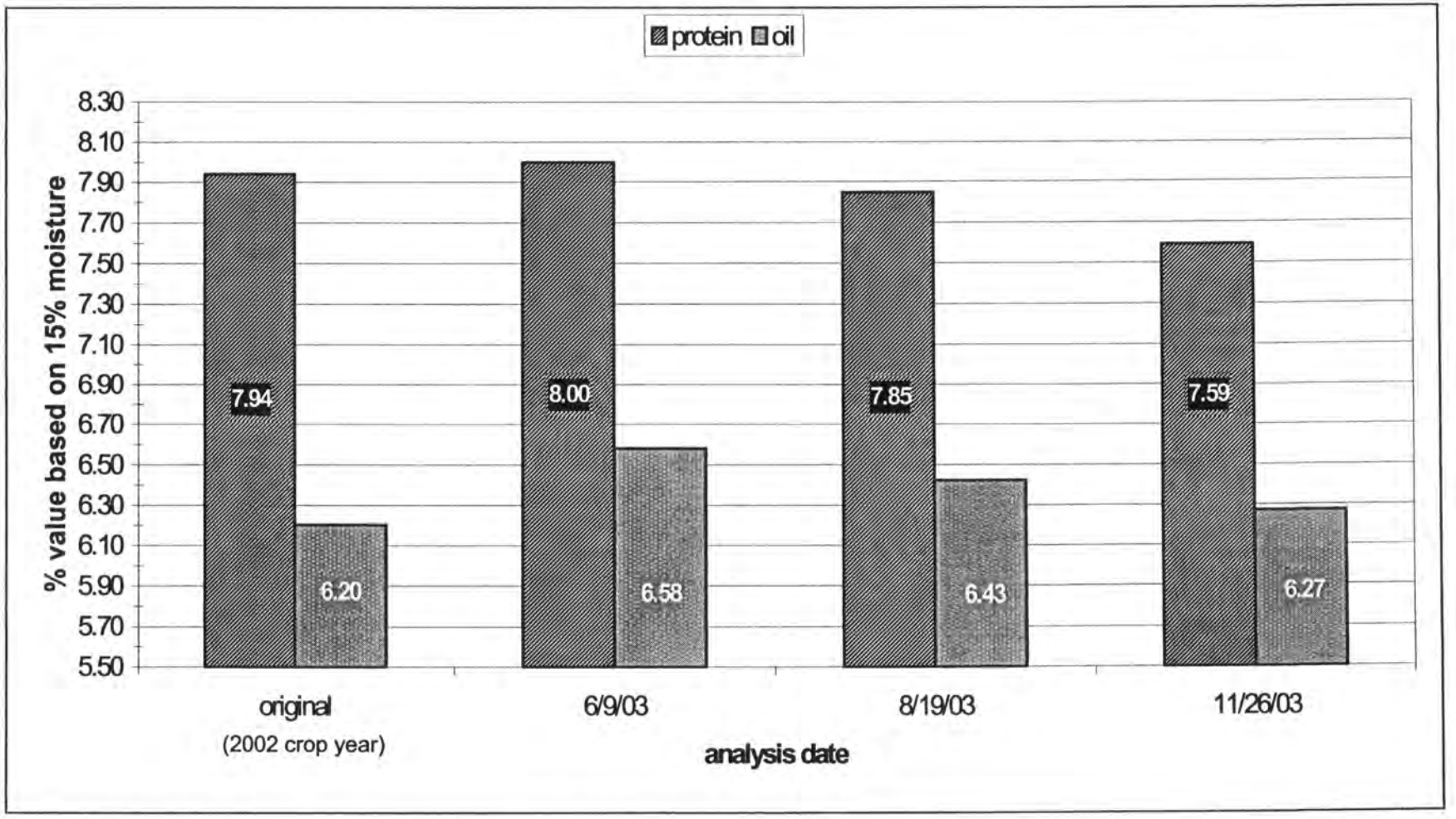


**Figure 10.** Corn check sample chemistry over time (average of duplicate by date)

## Working Accuracy of Instrumental Analysis

Running standardization samples and comparing original NIR analyses to reference chemistry are two ways the ISU-GQL analyzes working accuracy of instrumental analysis. Conventional accuracy is determined when two methods are compared at the same time. Comparably, working accuracy is the ability of an NIR instrument to predict the true constituent content of a sample during routine sample analyses.

Standardization samples are run at least once a year for corn and soybeans. Standard samples are chosen that have a long-term average of 3 or more repeat reference chemistry values. Samples are run three times each and bias and slope/bias settings are calculated. Based on analysis of the data obtained, adjustments are made to the calibrations used in NIR instruments so performance across copies of the same instrument is identical.

When the chemistry results are compared back to the original NIR reading, it provides a basis for analyzing the accuracy of the calibration model developed and used. Select samples are sent to external laboratories for chemical analysis as close to the first NIR reading as possible. The ISU-GQL assumes that the external chemistry results are an accurate depiction of the sample at the time of first analysis. Samples are sent for outsourced analysis based on the request of the client or the uniqueness of the sample. Samples that represent extremes of the compositional bell-curve are always in high demand. Distinctive samples are not always readily available and are essential to increase the accuracy and reliability of calibration models.

## Precision of Instrumental Analysis

The precision of instrumental analysis is analyzed for the same reason the precision of external laboratory chemistry are checked. During the beginning of a new crop year (September to December), the laboratory runs thousands of service samples. It is financially and logistically impossible to run every sample in duplicate, which emphasizes the importance of having calibrations and methods that enable the ISU-GQL to run service samples only once. By verifying the precision of instrumental analysis with duplicates, this goal is accomplished.

Ten percent of near-infrared spectroscopy (NIR) instrument readings are duplicated. The duplicates are compiled and a summary is produced at the end of a crop year. Figure 11 shows the difference between duplicates in soybean protein for the Infratec serial number 553075 for the 2002 crop year. The chart demonstrates that as protein levels increased, the ability of the instrument to reproduce the result diminished. Possible explanations for this phenomenon include the prediction of protein values extrapolated beyond the calibration set; fewer samples in the higher protein range available for inclusion in calibration sets; and more specialty (inherently variable) germplasm in higher protein samples. The majority of duplicates gathered around zero giving the ISU-GQL confidence in their machinery.

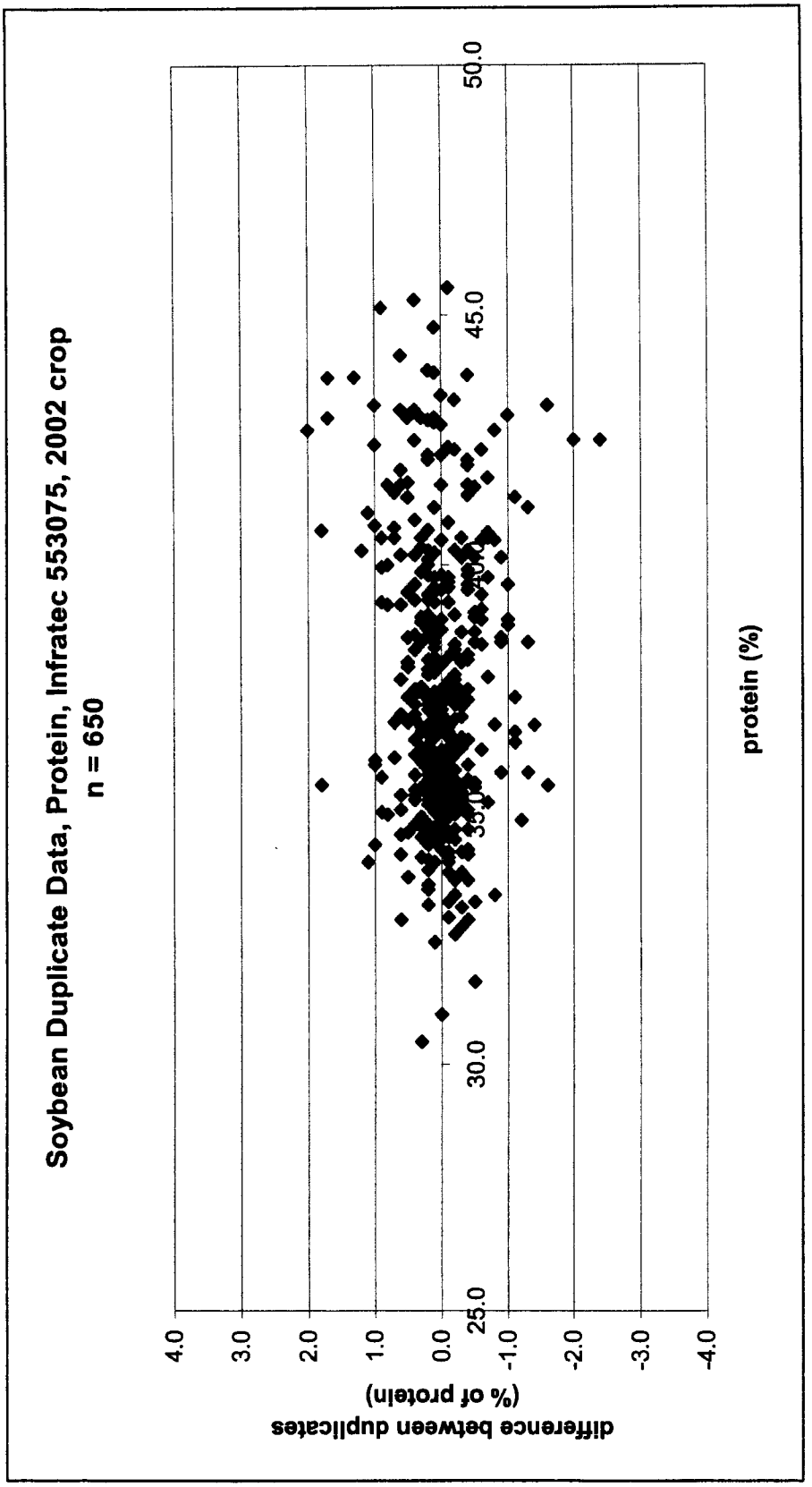


**Figure 11.** Example of instrument precision check data

## CHAPTER 8. CONFORMANCE WITH ISO 17025

Table 4 is a matrix comparing the requirements of the ISO 17025 standard and what has been completed by this project. The matrix was completed using the ISO 17025 standard and interpreted to be more succinct and easily understood. (ISO/IEC, 1999) Some sections of the standard were not described in detail because these sections did not require direct action by the laboratory.

ISO 17025 sections one through three do not contain any requirements that must be met by a laboratory. They, instead, provide background and information that make it easier to read and understand the standard. These sections include a table of contents and a detained definition of terms.

Section four contains the specific information pertaining to management requirements. Organization of the laboratory was a major focus for this project and all requirements set by section 4.1 were completed. One example was the training system (section 4.1.5a) which was developed and implemented. The quality system and manual for the ISU-GQL was established fulfilling section 4.2. Document control, section 4.3, was addressed by the use of a document header on all laboratory procedures. The sub-sections of section four identified as not completed were not addressed by this project and are included in the future direction recommendations.

Section five contains the specific information pertaining to technical requirements. Personnel requirements from section 5.2 were completed with the training program in place. Job descriptions are another example of the completion of section 5.2.4. Except for controlled access to the laboratory, the accommodation

**Table 4.** Conformance by the Iowa State University Grain Quality Laboratory to the ISO/IEC 17025 standard at completion of project[a]

| Section Number | Section Title | Guidance Requirements | Completed |
|---|---|---|---|
| 1 | Scope | none | |
| 2 | Normative references | none | |
| 3 | Terms and definitions | none | |
| 4 | Management requirements | | |
| 4.1 | Organization | | |
| 4.1.5 a | | personnel with authority and resources to work and identify departures from quality system (training) | yes |
| 4.1.5 b | | remove internal or external pressures | yes |
| 4.1.5 c | | protection of confidential information and electronic storage | yes |
| 4.1.5 d | | avoid questionable practices | yes |
| 4.1.5 e | | define organizational structure | yes |
| 4.1.5 f | | | yes |
| 4.1.5 g | | provide adequate supervision | yes |
| 4.1.5 h | | technical management with resources | yes |
| 4.1.5 i | | appoint a quality manager | yes |
| 4.1.5 j | | appoint deputies for key managerial personnel if practical | yes |
| 4.2 | Quality System | | |
| 4.2.1 | | establish, implement and maintain a quality system with appropriate documentation | yes |
| 4.2.2 | | quality manual in place | yes |
| | | with a quality policy statement | no |
| 4.3 | Document control | | |
| 4.3.1 | General | procedures for document control | yes |
| 4.3.2 | Document approval and use | 4.3.2.1 current revision status list | no |
| | | 4.3.2.2 ensuring current edition is the only one available | |
| | | 4.3.2.3 uniquely identified quality system documents | yes |
| 4.3.3 | Document changes | how made and controlled | yes |
| 4.4 | Review of requests, tenders and contracts | | no |
| 4.4.1 | | procedures for review of requests, tenders and contracts | no |
| 4.4.2 | | | no |
| 4.5 | Subcontracting of tests and calibrations | | |

[a] only sections included that require action by lab (ISO/IEC, 1999)

**Table 4.** (continued)

| Section Number | Section Title | Guidance Requirements | Completed |
|---|---|---|---|
| 4.5.1 | | choose subcontractors who comply with ISO 17025 for the work in question | no |
| 4.5.2 | | advise clients of subcontractors in writing | no |
| 4.5.4 | | register of subcontractors | yes |
| 4.6 | Purchasing services and supplies | | |
| 4.6.1 | | policy for service an supply purchase | no |
| 4.6.2 | | records of purchased services and supplies compliance | no |
| 4.6.3 | | purchase order documentation | no |
| 4.6.4 | | evaluation of suppliers with records | no |
| 4.7 | Service to the client | cooperation and communication with clients | yes |
| 4.8 | Complaints | policy for recording and resolving complaints | no |
| 4.9 | Control of nonconforming testing and/or calibration work | | |
| 4.9.1 | | procedure for identification of nonconformance | partially |
| 4.10 | Corrective action | | |
| 4.10.1 | General | corrective action procedures with authorized personnel | some |
| 4.10.2 | Cause analysis | root cause analysis | no |
| 4.10.3 | Selection and implementation of corrective actions | documentation of corrective action investigations | no |
| 4.10.5 | Additional audits | procedure for and when needed in accordance with 4.13 as soon as possible | no |
| 4.11 | Preventive action | development of action plans with implementation and monitoring procedures | no |
| 4.12 | Control of records | | |
| 4.12.1 | General | | |
| 4.12.1.1 | | | no |
| 4.12.1.2 | | stored and kept in organized and legible fashion | no |
| 4.12.1.4 | | record backup | yes |
| 4.12.2 | Technical records | | |
| 4.12.2.1 | | maintain all raw data with operator | yes |
| 4.12.2.3 | | measures to avoid loss or change of original data | no |
| 4.13 | Internal audits | | |

**Table 4.** (continued)

| Section Number | Section Title | Guidance Requirements | Completed |
|---|---|---|---|
| 4.13.1 | | plan for internal audits | no |
| 4.13.2 | | timely corrective actions and notification of clients | no |
| 4.13.3 | | documentation of audit findings and corrective actions | no |
| 4.13.4 | | follow-up audit activities documented | no |
| 4.14 | Management reviews | | |
| 4.14.1 | | procedure and schedule for management reviews | no |
| 4.14.2 | | record of findings and actions to be taken with timescale | no |
| 5 | Technical requirements | | |
| 5.1 | General | | |
| 5.2 | Personnel | | |
| 5.2.1 | | ensure competence of personnel | yes |
| 5.2.2 | | policy for identifying training needs | yes |
| 5.2.4 | | maintain job descriptions | yes |
| 5.2.5 | | personnel records | yes |
| 5.3 | Accommodation and environmental conditions | | |
| 5.3.1 | | lab conditions are appropriate for testing being performed | yes |
| 5.3.2 | | monitor and record environmental conditions | yes |
| 5.3.3 | | separation between incompatible activities | yes |
| 5.3.4 | | controlled lab access | no |
| 5.3.5 | | good housekeeping practices | yes |
| 5.4 | Test and calibration methods and method validation | | |
| 5.4.1 | General | select appropriate methods and have procedures for them | yes |
| 5.4.2 | Selection of methods | inform clients of methods used | no |
| 5.4.3 | Laboratory-developed methods | must be a planned activity and documented | no |
| 5.4.4 | Non-standard methods | agreement with client for use | no |
| 5.4.5 | Validation of methods | | |
| 5.4.5.2 | | validate non-standard methods | no |
| 5.4.5.3 | | range and accuracy of validated methods | no |
| 5.4.6 | Estimation of uncertainty of measurement | | no |
| 5.4.7 | Control of data | documentation of custom software | no |
| 5.5 | Equipment | | |
| 5.5.1 | | furnished will all required equipment | yes |

**Table 4.** (continued)

| Section Number | Section Title | Guidance Requirements | Completed |
|---|---|---|---|
| 5.5.2 | | equipment and software shall be capable of the accuracy needed | yes |
| 5.5.3 | | operated by authorized personnel with procedures provided | yes |
| 5.5.4 | | equipment uniquely identified | yes |
| 5.5.5 | | records maintained | yes |
| 5.5.6 | | | no |
| 5.5.7 | | suspect equipment taken out of use | yes |
| 5.5.8 | | equipment calibration records | yes |
| 5.5.9 | | equipment that leaves lab should be checked upon return | yes |
| 5.5.11 | | procedures for updating all correction factors | no |
| 5.5.12 | | safeguards in place so invalidation of equipment does not occur | no |
| 5.6 | Measurement traceability | | |
| 5.6.1 | General | procedure for calibration of equipment | yes |
| 5.6.2 | Specific requirements | | |
| 5.6.2.1 | Calibration | must be traceable to SI (international system of units) | no |
| 5.6.2.2 | Testing | same requirements as 5.6.2.1 for testing equipment | |
| 5.6.3 | Reference standards and reference materials | | |
| 5.6.3.1 | Reference standards | procedure for calibration of reference standards | no |
| 5.6.3.2 | Reference materials | traceable to SI units if possible | no |
| 5.6.3.3 | Intermediate checks | procedures and schedules | no |
| 5.6.3.4 | Transport and storage | procedures | no |
| 5.7 | Sampling | | |
| 5.7.1 | | sampling procedures | no |
| 5.7.2 | | deviations for clients recorded | no |
| 5.7.3 | | records of sampling | no |
| 5.8 | Handling of test and calibration items | | |
| 5.8.1 | | retention, disposal | no |
| 5.8.2 | | system for identifying samples | no |
| 5.8.3 | | abnormalities recorded upon receipt | yes |
| 5.8.4 | | procedures for storage | no |
| | | facilities for storage | yes |
| 5.9 | Assuring the quality of test and calibration results | procedures for quality control monitoring | yes |
| 5.10 | Reporting the results | | |
| 5.10.1 | General | results reported clearly in test report/calibration certificate | yes |

**Table 4.** (continued)

| Section Number | Section Title | Guidance Requirements | Completed |
|---|---|---|---|
| 5.10.2 | Test reports and calibration certificates | | no |
| 5.10.3 | Test reports | additional requirements | no |
| 5.10.4 | Calibration certificates | additional requirements | yes |
| 5.10.5 | Opinions and interpretations | document when used | no |
| 5.10.6 | Testing and calibration results obtained from subcontractors | clearly identify to clients | no |
| 5.10.7 | Electronic transmission of results | control see 5.4.7 | no |
| 5.10.8 | Format of reports and certificates | minimize misunderstanding or misuse | yes |
| 5.10.9 | Amendments to test reports and calibration certificates | only made by additional report that notes it is an amendment | no |

and environmental conditions of section 5.3 were completed. Instrument log maintenance was defined for sections 5.5.5 and 5.5.8. As with section four, the sub-sections of section five identified as not completed were not addressed by this project and are included in the future direction recommendations. Specifically, a system for estimation of the uncertainty of measurements, which addresses section 5.4.6 and the definition of reference materials for section 5.6 require future attention.

# CHAPTER 9. FUTURE DIRECTION

Although much progress has been made in developing a laboratory quality management system for the Iowa State University Grain Quality Laboratory (ISU-GQL), there is still more work to be done. Procedures for document and record management need to be developed. They should include a specific plan for the storage and retrieval of analytical data produced in the laboratory along with administrative documents. Benefits of clear document and record management procedures include a decrease in retrieval time, an increased in data release efficiency, and a decrease in animosity between those who must deal with different parts of data management and analysis.

Continual revision and addition to laboratory procedures is always a must. Previously written procedures need to be scrutinized and changes made if necessary. Also new procedures should be added for recently added processes and those that were not completed in this study.

A management review schedule should be developed and observed. It is vital that management does not loose sight of what the laboratory quality management system can provide the lab and what must be done to accomplish this end. The schedule should include at least one major review per year. The most reasonable time is in the late spring or early summer after fall samples and calibrations samples have been completed, but before preparations for the next crop year begin. This will give management the opportunity to analyze what was done in the previous crop year to decide if things should change or if they worked well. Other shorter

management review sessions can be scheduled as need to address specific topics of concern brought my members of management of laboratory technicians.

Work should be done to analyze the specific needs and expectations of all the ISU-GQL clients. Due to the addition of research interests of a university-based analytical laboratory, clients will include, not only the typical service clients, but graduate students, other departments and the Iowa Grain Quality Initiative. A discussion should be included in the management review session to ensure that the laboratory is meeting the needs and expectations of its clients. If the clients are not satisfied, the laboratory does not have much hope of survival.

More attention needs to be paid to the outsourced chemistry companies. Data in the past has been questionable and changes need to be made in the future. If the outsourced chemistry does not improve, consideration should be made to the possibility of choosing a new chemistry company. Pros and cons should be weighed. Cost is not the only factor that needs to be considered. If another chemistry company can provide more reliable results, it should be considered. The old premise applies here that no data is better than bad data. At least with no data, assumptions and calibrations are not made that could be in error.

Finally, more work needs to be done with the ISU-GQL's quality control charts. Time limitations of this project did not permit the exhaustive study of all the possible causes for the yearly fluctuation. It is possible that multiple correlations with climate information could explain the data fluctuations. A programmer, could be helpful for the implementation of real-time online control charts. In the interest of laboratory technician use, the continued utilization of Microsoft Excel software is valuable.

# APPENDIX A. QUALITY MANUAL

**Iowa State University Grain Quality Laboratory**
**1563 Food Sciences Building**
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Grain Quality
LABORATORY

# Quality Manual

**Quality Manager:** Audrey M. Hansen

**General Manager:** Dr. Charles R. Hurburgh, Jr.

**Laboratory Manager:** Glen Rippke

**Date of Issue:** 06/12/2004

**Controlled Copy #:** UNCONTROLLED

## Quality Manual

This Quality Manual meets the requirements of ISO 17025 and ISO 9001. This Quality Manual is confidential and assigned as outlined below.

Issued to: Audrey Marie Hansen thesis appendix A

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## Table of Contents





## Introduction – the READ ME page!

### Purpose

This Quality Manual contains all the requirements that our laboratory uses to demonstrate our quality system, technical competence, and valid results.

Section 4 specifies how we demonstrate sound management and maintain client satisfaction.

Section 5 specifies how we demonstrate technical competence in our laboratory.

In addition, this Quality Manual outlines how we meet:

- ISO 17025
- ISO 9001

All personnel are to take an active role in establishing, implementing, and maintaining our quality management program. We do not separate quality from our daily business. Quality cannot be something that we do just to pass audits. Quality is involved in every facet of the decision-making process in the management of our laboratory and the science that we practice.

### Distribution List

The Quality Manager maintains a distribution list for this Quality Manual.

## 1. Scope

This Quality Manual facilitates:

- recognition of technical competence for standardized methods, non-routine methods, and laboratory-developed methods we perform
- inspection and product certification capabilities and/or services we provide
- total quality for our administrative and technical systems
- audits by clients, regulatory authorities and accreditation bodies
- meeting the requirements of ISO 17025 and ISO 9001
- client satisfaction



## 2. Normative References

### Reference List

ISO/IEC Guide 2 - General terms and their definitions concerning standardization and related activities.

VIM: 1993 - International vocabulary of basic and general terms in metrology, issued by BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML.

ISO 9001:2000 – Quality Management Systems – Requirements.

ISO 17025:1999 – General Requirements for the Competence of Testing and Calibration Laboratories.

### Cross-references (helpful during audits!)

This manual is numerically aligned with the international standard ISO 17025. It is expected that this will prove useful during accreditation audits and expedite the process.

Furthermore, each section cross-references the ISO 9001 standard to assist the laboratory during the ISO 9001 registration process (if applicable).

For ease of use, each section starts with a brief summation of what the section addresses and a listing of the quality terminology and key words.

## 3. Terms and Definitions

For the purposes of this manual, the following relevant definitions apply: ISO/IEC Guide 2; ISO/IEC Guide 30; ISO Council Committee on Conformity Assessment (CASCO); ISO 8402; ISO 10015; ISO 5725-1; ISO 17025; the Food Laboratory Accreditation Working Group (FLAWG); AOAC; American Chemical Society (ACS); and International Vocabulary of Basic and General Terms in Metrology (VIM).

**Accreditation** – formal recognition of a laboratory by an independent science-based organization that the laboratory is competent to perform specific tests (CASCO).

**Accuracy** – the closeness of agreement between a test result and the accepted reference value (ISO 5725-1, ISO Guide 30).

**Calibration** - a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system or values represented by a material measure or a reference material, and the corresponding values realized by the standard (VIM).

Notes
1. The result of a calibration permits either the assignment of values or measurands to the indications or the determination of corrections with respect to indications.
2. A calibration may also determine other metrological properties such as the effect of influence quantities.
3. The result of a calibration may be recorded in a document sometimes called a calibration certificate or a calibration report.

**Certification** - procedure by which a third party gives written assurance that a product, process, or service conforms to specified requirements (ISO 8402).

**Certified Reference Material** – a reference material, one or more of, whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body (ISO Guide 30).

**Client** – an entity (customer, agency, company, person, etc.) who receives a test result done according to specified requirements (FLAWG).

**Competence** – ability consisting of theoretical knowledge, practical skills, and attitudes (ISO 10015).

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Section 3 - Terms and Definitions


**Corrective Action** – action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence (ISO 8402).

**Holding Time** – elapsed time between sample collection and either sample preparation or analyses, as appropriate.

**Inspection** - evaluation for conformity by measuring, observing, testing, or gauging the relevant characteristics (ISO/IEC Guide 2). Activity such as measuring, examining, testing or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformity is achieved for each characteristic. (ISO 8402)

**Limit of Detection** – the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte (ACS). The mean value of the matrix blank readings plus 3 standard deviations of the mean, expressed in analyte concentration. For methods with less than 100% recovery the limit of detection should be corrected for recovery (AOAC).

**Limit of Quantification** – lowest concentration of analyte that can be determined with an acceptable level of accuracy and precision. Determined by actual analysis of at least 6 fortified test samples per matrix. It is not determined by extrapolation (AOAC).

**Linearity** – is determined by the analysis of samples with analyte concentrations spanning the claimed range of the method. The results are used to calculate a regression line against analyte calculation using the least squares method. It is convenient if a method is linear over a particular range but it is not an absolute requirement. Where linearity is unattainable for a particular procedure, a suitable algorithm for calculations should be determined (AOAC).

**Measurement Uncertainty** - parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand (International Vocabulary of Basic and General Terms in Metrology).

**Precision** – the closeness of agreement between test results obtained under stipulated conditions (ISO 5725-1, ISO Guide 30).

**Preventive Action** – action taken to eliminate the causes of a potential nonconformity, defect, or other undesirable situation in order to prevent occurrence (ISO 8402).

**Proficiency Testing** – determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons (ISO/IEC Guide 2).

**Quality Assurance** – all those planned and systematic actions necessary to provide adequate confidence that a product or service will satisfy given requirements for quality (ISO 8402).

**Quality Control** – the operational techniques and activities that are used to fulfill requirements for quality (ISO 8402).

**Quality Manual** – a document stating the quality policy, quality system, and quality practices of an organization (ISO 8402).

**Quality System** – the organizational structure, responsibilities, procedures, processes, and resources for implementing quality management (ISO 8402).

**Range** – the difference between the largest and smallest observed value of a quantitative characteristic. For quantitative analysis the working range for a method is determined by examining samples with different analyte concentrations and determining the concentration range for which acceptable accuracy and precision can be achieved. The working range is generally more extensive than the linear range. The working range is determined by the analysis of a number of samples of varying analyte concentrations and calculating the regression from the results, usually using the method of least squares. The relationship of analyte response to concentration does not have to be perfectly linear for a method to be effective (AOAC).

**Reference Material** – a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (ISO Guide 30).

**Reference Standard** – a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived (VIM). Generally, this refers to national traceable standards such as those from the National Institute of Standards and Technology (NIST).

**Repeatability (r)** – precision under the *same conditions* (same method, same test item, same operator, same apparatus, same laboratory, short interval of time) (ISO 5725-1).

**Reproducibility (R)** – precision using the *same method* on identical items obtained by operators in different laboratories using different equipment (ISO 5725-1).

**Ruggedness** – the ruggedness of a method is tested by deliberately introducing small changes to the method and examining the consequences. A large number of factors may need to be considered, but because most of these will have a negligible effect, it will normally be possible to vary several at once (AOAC).



**Selectivity** – the extent that a specific analyte can be determined from a complex mixture without interference from the other components in the mixture. A method that is perfectly selective for an analyte or group of analytes is said to be specific. The applicability of the method should be studied using various samples, ranging from pure standards to mixtures with complex matrices. In each case the recovery of the analyte(s) of interest should be determined and the influences of suspected interference duly stated. Any restrictions in the applicability of the technique should be documented in the method (AOAC).

**Sensitivity** – the difference in analyte concentration corresponding to the smallest difference in the response of the method that can be detected. It is represented by the slope of the calibration curve and can be determined by a least squares procedure, or experimentally, using samples containing various concentrations of the analyte (AOAC).

**Skill** – ability to apply knowledge effectively and readily in performance (ISO 10015).

**Specific** – see selectivity.

**Standard Operating Procedure** – a document that specifies or describes how an activity is to be performed. It may include methods to be used and sequence of operations (FLAWG).

**Test** - technical operation that consists of the determination of one or more characteristics of a given product, process, or service according to a specified procedure (ISO Guide 2: 1991).

**Traceability** – the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons (VIM).

**Training** – a process to provide and control competence to meet requirements (ISO 10015).

**Validation** - confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled (ISO 8402).

**Verification** - confirmation by examination and provision of objective evidence that specified requirements have been fulfilled (ISO 8402).

## 4.1 Organization

### Cross-references

ISO 17025:1999 Section 4.1
ISO 9001:2000 Section 5.1, 5.3, 5.4.1, 5.5.1, 5.5.2, 6.1, 6.2.1, 6.3

### 4.1.1 Legal Identification / Registration

Iowa State University Grain Quality Laboratory
1563 Food Sciences Building
Telephone: (515) 294-5387
Fax: (515) 294-6383

### 4.1.2 Laboratory Requirements

The work areas of Iowa State University Grain Quality Laboratory have been organized to satisfy the needs of the client and regulatory authorities and to operate to the international standards ISO 17025 and ISO 9001. Iowa State University Grain Quality Laboratory is composed of the following work areas:

Professor in Charge (General Manager's) Office (1541 Food Sciences Building)
Laboratory and Service Manager's Office (1547 Food Sciences Building)
Administrative Personnel's Office (1563 Food Sciences Building)
Proximate Analysis Laboratory (1546 Food Sciences Building)
Physical Properties Laboratory (1544 Food Sciences Building)
Near-Infrared Instrumentation Laboratory (1515 Food Sciences Building)
Dry Grinding Laboratory (1513 Food Sciences Building)

### 4.1.3 Scope of Management System

The management system covers activities in the laboratory's permanent facility. The fields of activities include:

[List the field of activities and give a brief description]

The laboratory's scope of tests is listed in the <Click here and type 'RECORD LOCATION'>.



### 4.1.4 Potential Conflicts of Interest

Not applicable - the laboratory is not part of a larger organization.

### 4.1.5 Organization

#### A) Management and Technical Personnel

**Policy:**
The laboratory managerial and technical personnel have the necessary authority and resources needed to meet the mandates assigned to their areas.

**Details:**
Responsibilities are detailed in 4.1.4 (F).

Departures from the organizational and management policies in this manual can only be approved by the professor in charge (general manager).

Departures from quality system procedures can only be approved by the professor in charge (general manager).

Departures from test methods or technical standard operating procedures (SOPs) can only be approved by the laboratory and service manager.

See also section 5.2.

#### B) Undue Pressure

**Policy:**
Management and personnel are to be free from any undue internal and external commercial, financial and other pressures that may adversely affect the quality of their work. The integrity of test results is the responsibility of all personnel. Management ensures that employees are never instructed or forced to alter or falsify data.

**Details:**
The following list provides some guidelines on how employees avoid conflict of interest situations. Employees shall not:
- falsify records, prepare fraudulent reports, or make false claims

- seek or use privileged or confidential company information, or data from any client, for any purpose beyond the scope of employment
- conduct non-laboratory business on laboratory time, or use company facilities or equipment to conduct outside interests in business, unless prior approval has been obtained
- solicit business on their own behalf (rather than the laboratory) from a client
- be employed by, or affiliated with, organizations whose products or services compete with laboratory products or services
- have employment that negatively affects or interferes with their performance of laboratory duties
- compete with the laboratory in the purchase, sale, or leasing of property or goods
- allow association, family, or friends to influence business decisions to their benefit - decisions must be made on a strictly business basis, always in the best interest of the laboratory
- make any decision that provides gains or benefits to the employee and/or others
- have personal financial dealings with an individual or company that does business with the laboratory which might influence decisions made on the laboratory's behalf

Firm adherence to this code of values forms the foundation of our credibility. Personnel involved in dishonest activities are subject to a range of disciplinary action including dismissal.

### C) Client Confidentiality

**Policy:**
It is the policy of our laboratory to protect the confidential information and proprietary rights of our client including the electronic storage and transmission of results.

**Details and Procedures:**
All employees sign an Employee Confidentiality Agreement. The signed agreement is retained in each employee's Human Resources file.

Test results are only released to the client. Release to someone other than the client requires the express permission of the client, except when the situation contravenes State or Federal Legislation and the results must be provided to the appropriate agency. The release of test results to anyone other than the client requires the permission of management. Laboratory reports are reviewed for accuracy prior to release.



### D) Operational Integrity

**Policy:**
The laboratory will avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment, or operational integrity.

**Details and Procedures:**
To ensure confidence in laboratory operations a formal quality assurance program is implemented. Technical competence is ensured through check sample programs. Impartiality is assessed through audits and approvals. Judgment is ensured through the hiring of qualified personnel and by continuously refining, upgrading and improving his or her skills. Operational integrity is reviewed by management on a regular basis at management review meetings to ensure continued suitability and effectiveness of laboratory policies and procedures. Any problems are acted on immediately through corrective action procedures.

### E) Organizational Structure

**Policy:**
The organization and management structure of the laboratory, and the relationships between management, technical operations, support services, and the quality system is defined through the aid of an organizational chart.

**Details:**
Senior management keeps the most current organizational chart on file. An organizational chart is available with this manual as a reference record and is considered the official record on the date it is marked.

### F) Responsibility and Authority

The following positions are described in their individual job descriptions:

- **Professor in Charge (General Manager)**
- **Laboratory and Service Manager**
- **Quality Manager**
- **Laboratory Technicians**
- **Administrative Personnel**

### G) Laboratory Supervision

**Policy:**
Adequate supervision is provided in each area of the laboratory for all testing and calibration personnel, including trainees, by persons familiar with the methods and procedures.

**Details:**
Adequate supervision is ensured through designated supervisors as well as through documentation such as this Quality Manual, test methods and SOPs. A thorough orientation and training program is adhered to for all new employees. Ongoing training for regular personnel is required.

### H) Technical Management

**Policy:**
A technical manager (the laboratory and service manager) is assigned to the laboratory. They have overall responsibility for the technical operations and the provision of resources needed to ensure the required quality of laboratory operations.

**Details:**
While the technical manager may at times delegate duties to other personnel, the technical manager is accountable for any nonconforming activities.

### I) Quality Manager

**Policy:**
The Quality Manager is appointed by the highest level of management. The Quality Manager, who, irrespective of other duties and responsibilities, has defined responsibility and authority for ensuring that the quality system is implemented and followed. The Quality Manager has direct access to the highest level of management where decisions are taken on laboratory policy or resources.

**Details:**
This statement notifies all laboratory personnel that Audrey M. Hansen is the Quality Manager as authorized below by the Professor in Charge (General Manager). Any change in this position requires the reissue of this section to all holders of controlled copies of the Quality Manual. The following signature also serves as approval for this Quality Manual and affirms senior management's commitment to the policies and procedures set forth in this manual.



Dr. Charles R. Hurburgh, Jr.
Professor in Charge (General Manager)

### J) Managerial Substitutions

**Policy:**
Deputies for key personnel are appointed to fulfill the key personnel's duties in their absence.

**Details:**
In the absence of the Quality Manager, the Laboratory and Service Manager will assume his/her responsibilities.

In the absence of the Technical/Laboratory Manager, the Professor in Charge (General Manager)will assume his/her responsibilities.

Management is responsible for ensuring that current and/or increased workload requirements are met. This includes making adjustments as a result of employee absence. Only fully trained employees are utilized to fulfill the duties of personnel who are absent. If sufficient human resources are not available, management will identify the best possible solution to meet operational requirements.

## Revision History

Revision 1

## 4.2 Quality System

### Cross-references

ISO 17025:1999 Section 4.2

ISO 9001:2000 Section 4.1, 4.2.1, 4.2.2, 5.1, 5.4.1, 5.4.2, 5.5.1, 6.2.1, 7.1

### 4.2.1 Policies and Procedures

**Policy:**
The Quality System is established, implemented, and maintained by management. It is applicable to all the fields of testing and activities in which the laboratory is involved and undertakes. All policies, systems, programs, procedures and instructions are documented to the extent necessary to enable the laboratory to assure the quality of results generated. These documents are communicated to, understood by, available to, and implemented by the appropriate personnel.

**Details:**
The purpose of our Quality System is to ensure that all services and products satisfy the client's requirements and have been designed, manufactured, and delivered under controlled conditions.

The effectiveness of the Quality System is assessed in several ways:
- by a program of planned internal audits, covering all aspects of the operation of the quality system
- by regular management reviews of the suitability and effectiveness of the quality system
- by analysis of potential and actual problems as shown by client complaints and supplier and subcontractor assessments
- by other methods approved from time to time by the Professor in Charge (General Manager)

This Quality Manual and associated documents (including procedures) and records serves as the quality plan for the laboratory. Other documents and records include:
- standard operating procedures
- quality control plans in test methods
- organizational charts
- proposals
- project management schemes



### 4.2.2 Quality Policy Statement

**Policy:**
The policies and objectives for laboratory operations are documented in this Quality Manual. The overall objectives are set out in the Quality Policy Statement. The Quality Policy Statement is issued under the authority of the Professor in Charge (General Manager) on the effective date.

**Quality Policy Statement:**
To ensure accurate and timely analytical, research, calibration, and technical services and to continuously meet or exceed the stated or implied expectations of our clients through day-to-day interactions.

Effective Date: 06/17/2003

a) *Management commitment to good professional practice and quality of services provided to the client*: Tests and calibrations are always carried out in accordance with stated standardized methods and clients' requirements. Requests to perform tests that may jeopardize an objective result or have a low validity are rejected.

b) *Standards of service include*:
- Client Satisfaction
- Accurate
- Timely

Excellence in the workplace is promoted by providing all employees with the knowledge, training, and tools necessary to allow for the completion of accurate and timely work.

c) *Purpose*: to manage our business by meeting the needs of our clients.

d) *Personnel*: familiarize themselves with quality documentation and implement the policies and procedures in their work.

e) *Management is committed to complying with ISO 17025 and ISO 9001 international standards*: the objective of this Quality Manual is to document the compliant policies and associated procedures that are integrated into our daily activities.

Additional objectives include:

- to establish the level of the laboratory's performance
- to make test method changes to improve performance
- to participate in proficiency testing or quality evaluation programs with peer laboratories

- to ensure that all personnel are trained to a level of familiarity with the quality system appropriate to the individual's degree of responsibility
- to improve and validate laboratory methodologies by participation in method validation collaborative tests
- to establish and report on quality savings

### 4.2.3 Quality Manual

**Policy:**
This Quality Manual outlines the structure of the documentation used in the quality system. This Quality Manual makes reference to supporting procedures including technical procedures and is maintained up to date.

**Details:**
This quality system is structured in three tiers of documentation. The tiers are as follows
I. Quality Manual
II. Standard Operating Procedures and Test Methods
III. Records

For most clients, this Quality Manual and the associated documents form a general Quality Plan. If necessary, specific Quality Plans will be prepared on a 'per-client' basis. These Quality Plans will modify the general requirements stated in the Manual and associated documents.

All of the above documents are controlled documents.

The following records and directive documents are referenced in the Quality Manual, but maintained separately:
- organizational chart (section 4.1.5.E)
- copies of the Quality Policy Statement posted in the laboratory (section 4.2.2)
- identification of resources and management review (section 4.14.1)
- job descriptions (section 5.2.4)
- statistical techniques (section 5.9)
- test reports (section 4.12.2)
- identification of the laboratory's approved signatures (section 5.10.2)
- laboratory's scope of tests (section 4.1.3)
- equipment inventory and records (sections 5.5.4 and 5.5.5)
- calibration status indicators (section 5.5.8)
- reference standards inventory (section 5.6.3)
- verification records (section 5.9)
- quality control plan / criteria for workmanship (section 5.4.1)



- corrective action records (section 4.10)
- preventive action records (section 4.11)
- client complaint records (section 4.8.1)
- audit schedule and records (section 4.13.3)
- procurement and subcontracting records (sections 4.6 and 4.5.4)
- training records (section 5.2.5)
- master list of documentation (section 4.3.2)
- confidentiality agreements (section 4.1.5 C)
- contract review (section 4.4.2)
- validation of test methods (section 5.4.5)
- facility floor plan (section 5.3.1)

### 4.2.4 Technical Management and the Quality Manager

The roles and responsibilities for technical management and the Quality Manager are outlined in section 4.1.4 (F) of this manual.

Technical management ensures that section 5 of this manual is implemented and maintained. The Quality Manager ensures that section 4 of this manual is implemented and maintained.

### Revision History

Revision 1

## 4.3 Document Control

### Cross-references

ISO 17025:1999 Section 4.3

ISO 9001:2000 Section 4.2.1, 4.2.3

### 4.3.1 Policies and Procedures

**Policy:**
The SOP# QSP 4-3-1 is used to control all quality system documents (internally generated and from external sources). These include documents of external origin, such as regulations, standards, other normative documents, test and/or calibration methods, as well as drawings, specifications, instructions, and manuals.

**Details:**
Document means any information or instructions including policy statements, procedures, specifications, calibration tables, charts, text books, posters, notices, memoranda, software, drawings, and plans. These may be in various media, whether hard copy or electronic and they may be digital, analog, photographic or written.

The documents to be controlled include:
- Quality Manual
- Standard Operating Procedures and test methods
- Forms
- Standards

The control of data related to testing and calibration is covered in section 5.4.7. The control of records is covered in section 4.12.

### 4.3.2 Document Approval and Issue

#### 4.3.2.1 Review / Approval / Master List

**Policy and Details:**
All documents issued to personnel in the laboratory as part of the quality system are reviewed and approved for use by authorized personnel prior to issue (i.e., reviewed by personnel knowledgeable in the documented activity and then approved by management). A master list identifying the current revision status and distribution of documents in the



quality system is readily available in order to preclude the use of invalid and/or obsolete documents (see SOP# QSP 4-3-1). A revision history of documents is also maintained. Documents are formally reviewed on a biennial basis to ensure their continuing suitability.

#### 4.3.2.2 Availability and Obsolete Documents

**Policy and Details:**
The master list shows the current status of all controlled documents. The master list document is organized with the following information:
- Document #
- Title
- Revision #
- Date of issue
- Date of last review
- Locations

Controlled documents are approved before issue.

The SOP# QSP 4-3-1 for document control ensures that:
- authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed
- documents are periodically reviewed and where necessary revised to ensure continuing suitability and compliance with applicable requirements
- invalid or obsolete documents are promptly removed from all points of issue or use to assure against unintended use
- obsolete documents retained for either legal or knowledge preservation purposes are suitably marked (i.e., stamped "OBSOLETE" and dated)

#### 4.3.2.3 Identification

**Policy and Details:**
All quality system documentation is identified by:

- date of issue and/or revision number
- page numbering
- total number of pages (e.g., page 5 of 5)
- issuing authority (i.e., approval signature)

## 4.3.3 Document Changes

### 4.3.3.1 Review / Approval

**Policy:**
Changes to documents are reviewed and approved by the same function (i.e., personnel or position) that performed the original review.

**Details:**
Developments in policies and procedures require documents to be changed from time to time. Changes to documents receive the same level of review and approval as the originals.

The Quality Manual is reviewed annually by the Quality Manager. Records are kept of this review.

Test methods and SOPs are reviewed on a biennial basis. Procedures for this are outlined in SOP# QSP 4-3-1.

Obsolete documents are withdrawn, but are retained for archive purposes and clearly labeled as obsolete.

### 4.3.3.2 Identification of Changes

**Policy:**
The nature of document changes is identified in the document.

**Details:**
As outlined in SOP# QSP 4-3-1.

In general, the nature of changes is identified in the document with a vertical bar in the left-hand margin. Revision history is recorded at the end of the document.

### 4.3.3.3 Amendments by Hand

**Policy and Details:**
Hand-written amendments to documents are not permitted

### 4.3.3.4 Computerized Documents



**Policy and Details:**
The SOP# QSP 4-3-1 details how changes in documents maintained in computerized systems are made and controlled.

## Revision History

Revision 1

## 4.4 Review of Requests, Tenders, and Contracts

### Cross-references

ISO 17025:1999 Section 4.4

ISO 9001:2000 Section 5.2, 7.2.1, 7.2.2, 7.2.3

### 4.4.1 Policies and Procedures

**Policy:**
The SOP# QSP 4-4-1 is used to review requests, tenders, or contracts. This procedure ensures that:

a) the client requirements including the methods to be used are adequately defined, documented and understood (see section 5.4.2)
b) the laboratory has the capability and resources to meet the requirements
c) the appropriate test and/or calibration method is selected and capable of meeting the client's requirements (see section 5.4.2)

Any differences between the request or tender and the contract are resolved before any work commences. Each contract must be acceptable by both the laboratory and the client.

**Details:**
The request, tender and contract review is conducted in a practical and efficient manner, and the effect of financial, legal, and time schedule aspects are taken into account.

The review of capability establishes that the laboratory possesses the necessary physical, personnel, and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the tests and/or calibrations in question. The review may also encompass results of earlier participation in inter-laboratory comparisons or proficiency testing and/or the running of trial test or calibration programs using samples or items of known value in order to determine uncertainties of measurement, limits of detection, and confidence limits.

The contract review ensures that each client's requirements are adequately defined and documented before the service or product is ordered or dispatched. This should ensure that any order, once accepted, can be completed without delay, and that the client's requirements including delivery date, technical specification, and cost can be met.



If the contract review highlights any ambiguities or uncertainties then the client will be contacted and the problem resolved before the order is accepted.

The SOP# QSP 4-4-1 also describes the activities that take place should there be a subsequent amendment to a client's order.
Typical types of contracts include:

- approved service quotations
- confidentiality agreements
- non-disclosure agreements
- sample submission requests
- memorandum of agreement
- memorandum of understanding
- research proposals and contracts
- verbal orders (oral agreements)
- activity plans

### 4.4.2 Records of Review

**Policy:**
Records of request, tender and contract review, including significant changes, are maintained. Records of pertinent discussions with a client relating to the client's requirements or the work during the period of execution of the contract are also maintained.

**Details:**
For review of routine and other simple tasks, the date and the identification (e.g., initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate. For repetitive routine tasks, the review need be made only at the initial enquiry stage or on grant of the contract for on-going routine work performed under a general agreement with the client, provided that the client's requirements remain unchanged. For new, complex or advanced testing and/or calibration tasks, a more comprehensive record is maintained.

### 4.4.3 Review of Subcontracted Work

**Policy:**
Request, tender, and contract review also includes work that is subcontracted by the laboratory.

**Details:**
Subcontractor laboratories are reviewed as described in section 4.5.

### 4.4.4 Notification of Client

**Policy and Details:**
Clients are informed of deviations from the contract. This is typically communicated to the client prior to the performing the deviation.

### 4.4.5 Contract Amendment

**Policy and Details:**
If a contract needs to be amended after the work has commenced, the same contract review process is repeated and any amendments are communicated to all affected personnel.

### Revision History

Revision 1



## 4.5 Subcontracting of Tests and Calibrations

### Cross-references

ISO 17025:1999 Section 4.5

ISO 9001:2000 Section 7.4.1, 7.4.3, 8.2.4

### 4.5.1 Subcontractor Competence

**Policy:**
Work that must be subcontracted due to:
- unforeseen circumstances
- workload
- large contracts
- contracts requiring some extra technical expertise

is subcontracted to a technically competent laboratory.

**Details:**
The subcontracted laboratory demonstrates technical competence by possession or receipt of one or more of the following:
- recognized technical accreditation
- registration under the ISO 9001 standard
- satisfactory performance of appropriate quality control check samples (certified reference material, in-house reference material or replicate analysis
- audit of the subcontractor's quality system by our auditors

It is the responsibility of the Quality Manager to assess and approve the competence level of subcontractor laboratories.

### 4.5.2 Client Approval

**Policy:**
Clients are advised of work (or any portion thereof) that is being subcontracted to another laboratory and their approval is obtained (preferably in writing).

**Details:**
Clients are advised of subcontracted work through fee schedules or any type of contract listed in section 4.4.1.

### 4.5.3 Assurance of Subcontractor Competence

**Policy:**
The laboratory is responsible to the client for the subcontractor's work. Technical competence of subcontractor laboratories is demonstrated through various records.

> **Note** – there may be circumstances where the client specifies which subcontractor is to be used. In such cases we may not be able to demonstrate the competence of the subcontractor and therefore are not responsible for the results.

**Details:**
Records of subcontractor competence include, but are not limited to, the following:
- accreditation certificates or documentation
- registration certificates
- check sample results
- audit results
- approval by the Quality Manager

### 4.5.4 Subcontractor Register

**Policy:**
A register of all subcontractors performing tests and calibrations is maintained.

**Details:**
The approved register of subcontractors and all assessment records are maintained by the Quality Manager.

### Revision History

Revision 1



## 4.6 Purchasing Services and Supplies

### Cross-references

ISO 17025:1999 Section 4.6

ISO 9001:2000 Section 7.4, 8.2.4

### 4.6.1 Policies and Procedures

**Policy:**
The SOP# QSP 4-6-1 is used to select and purchase services and supplies. The SOP# QSP 4-6-1 is used for procurement, reception, and storage of supplies.

**Details:**
Consumable materials are stored according to the appropriate test method, SOP, or work instruction.

### 4.6.2 Specifications

**Policy:**
Only services and supplies of the required quality are used. These quality requirements are detailed in laboratory SOPs under the "*Materials Required*" section and will identify the appropriate minimum specifications when necessary.

**Details:**
Packing slips are checked against package content labels and matched with the Purchase Order if accepted. Once accepted, the packing slip is dated and initialed as evidence of compliance. Certificates of analysis (COA) are maintained on file after the COA is checked to ensure the received item meets minimum specifications.

Chemicals are purchased with manufacturer's certificates where possible. Uncertified chemicals are purchased from ISO 9000 registered companies. Whatever the source, the laboratory verifies the quality of the standards by comparing the new batch of standards to the old. Due regard is paid to the manufacturer's recommendations on storage and shelf life.

Reagents are generally purchased from manufacturers who have a quality system based on ISO 9000. The grade of any reagent used (including water) is stated in the method together with guidance on any particular precautions to be observed in its preparation or use.

Where no independent assurance of the quality of procured goods or services is available or the supplier's evidence is insufficient the laboratory ensures that purchased goods and services comply with specified requirements. Where possible and practical the laboratory ensures that goods are inspected, calibrated, or are otherwise in compliance with any standard specification relevant to the calibrations or tests concerned.

### 4.6.3 Purchasing Documents

**Policy:**
Purchasing requests are recorded on the Purchase Order form and contain data describing the product ordered. The Purchase Order is reviewed and approved for technical content prior to release.

**Details:**
The description may include type, class, grade, precise identification, specifications, drawings, inspection instructions, other technical data including approval of test results, quality required and quality system standard under which they were produced.

The completion of the Purchase Order is the responsibility of the laboratory and service manager. They review the Purchase Order for accuracy and approve the technical content prior to release with their signature and the date.

### 4.6.4 Approved Suppliers

**Policy:**
Suppliers of critical services are evaluated and approved before use. An approved supplier list is maintained.

**Details:**
Audits or tender evaluation is conducted to qualify suppliers of critical services prior to use. The criteria for evaluation may include, but is not limited to the following:

- references
- accreditation
- formal recognition

The records are maintained by purchasing personnel.

### Revision History

Revision 1



## 4.7 Service to the Client

### Cross-references

ISO 17025:1999 Section 4.7

ISO 9001:2000 Section 7.2.1, 7.2.3, 7.4.3, 7.5.1

### 4.7.1 Policies and Procedures

**Policy:**
Client requests are clarified for the clients or their representatives. Furthermore the client or their representative will be afforded the right to monitor the performance of the laboratory in relation to the work performed.

**Details and Procedures:**
Service to the client includes:

- Affording the client or the client's representative reasonable access to relevant areas of the laboratory for the witnessing of work performed for the client; it is understood that such access should not conflict with rules of confidentiality of work for other clients or with safety.
- Preparing, packaging, and dispatching of test and calibration items needed by the client for verification purposes.
- Maintaining of open contacts. The client values advice and guidance in technical matters, and opinions and interpretations based on results. Contact with the client, especially in large assignments, should be maintained throughout the work. The laboratory should inform the client of any delays or major deviations in the performance of the tests and calibrations.
- Obtaining feedback from the client. Positive and negative feedback can be obtained passively through ongoing communications with the client or actively through client satisfaction surveys. The feedback is used to improve the quality system, testing and calibration activities, and client service.

### Revision History

Revision 1

## 4.8 Complaints

### Cross-references

ISO 17025:1999 Section 4.8

ISO 9001:2000 Section 7.2.3

### 4.8.1 Policies and Procedures

**Policy:**
The SOP# QSP 4-8-1 is used for resolving complaints received from clients or other parties. Records are maintained of all complaints and follow-up.

**Details:**
Records of complaints include the following information:

- details of the complaint
- investigation
- corrective action
- follow-up verification

See also section 4.10.

All personnel are responsible for recording and responding to complaints.

### Revision History

Revision 1



## 4.9 Control of Nonconforming Testing and Calibration Work

### Cross-references

ISO 17025:1999 Section 4.9

ISO 9001:2000 Section 5.5.1, 7.4.3, 7.5.1, 8.2.4, 8.3

### 4.9.1 Procedures to Control Nonconforming Work

**Policy:**
The SOP# QSP 4-9-1 is used to control any aspect of testing and/or calibration work, or the results of this work, when they do not conform with the test methods or the agreed requirements of the client.

**Details:**
The procedure ensures that:

- Responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports [and calibration certificates] as necessary) are defined and taken into consideration when nonconforming work is identified
- an evaluation of the significance of the nonconforming work is made
- remedial actions are taken immediately, together with any decision about the acceptability of the nonconforming work
- where necessary, the client is notified and the work is recalled
- the responsibility for authorizing the resumption of work is defined

Identification of nonconforming work or problems with the quality system or with testing [and/or calibration] activities can occur at various locations within the quality system and technical operations such as:

- client complaints
- quality control
- instrument calibration
- checking of consumable materials
- staff observations or supervision
- test report [and calibration certificate] checking
- management reviews
- internal or external audits

## 4.9.2 Root Cause Analysis

**Policy:**
Where evaluation indicates that nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in 4.10 are followed to identify the root cause(s) of the problem and to eliminate this (these) cause(s).

**Details:**
The SOP# QSP 4-10-1 outlines recording the root cause analysis for investigating nonconforming work.

Situations warranting corrective action investigation include:
- failure to comply with test method including all applicable procedures necessary to ensure the integrity and representative nature of the sample
- presentation of uncertain knowledge as to compliance with test methods including all applicable procedures necessary to ensure the integrity and representative nature of the sample
- failure or suspected failure in method performance as demonstrated by results provided by quality control samples
- lack of relevant evidence provided by quality audit, proficiency testing, or client feedback
- lack of relevant evidence provided by data validation
- neglect to check the inherent property of the sample that compromises the testing

## Revision History

Revision 1



## 4.10 Corrective Action

## Cross-references

ISO 17025:1999 Section 4.10

ISO 9001:2000 Section 5.5.1, 8.1, 8.2.2, 8.2.3, 8.4, 8.5.2, 8.5.3

## 4.10.1 General

**Policy:**
The SOP# QSP 4-10-1 is utilized for implementing corrective action when nonconforming work or departures from policies and procedures in the quality system or technical operations have been identified. The procedure requires that appropriate authority be designated for the implementation of corrective actions. The procedure includes cause analysis, selection and implementation of corrective action, and monitoring of actions.

**Details:**
Problems with the quality system or technical operations of the laboratory may be identified through a variety of activities, such as control of nonconforming work, internal or external audits, management reviews, feed-back from clients, or staff observations.

Corrective action investigations are documented and required changes to operational procedures are implemented. The corrective action request (CAR), investigation and resolution are recorded on a CAR form.

## 4.10.2 Cause Analysis

**Policy:**
Corrective action always begins with an investigation to determine root cause(s) of the problem (see SOP# QSP 4-10-1).

**Details:**
Potential causes of the problem could include client requirements, the samples, sample specifications, methods and procedures, personnel skills and training, consumable materials, or equipment and its calibration.

### 4.10.3 Selection and Implementation of Corrective Actions

**Policy and Details:**
After determining the cause(s) of the problem, potential corrective actions are identified. The most likely action(s) (this includes practical and/or reasonable) are selected and implemented to eliminate the problem and to prevent recurrence. It should be noted that any corrective actions taken to eliminate the cause(s) of nonconformances or other departures are to an appropriate degree to address the magnitude of the problem and commensurate with the risks encountered (Note – in plain language, this means determine whether the benefit outweighs the cost). Controls are applied to prevent recurrence. The laboratory documents and implements the required changes resulting from corrective action investigations.

### 4.10.4 Monitoring of Corrective Action

**Policy:**
After implementing the corrective action(s), the laboratory monitors the results to ensure that the actions taken have been effective in overcoming the problems originally identified.

**Details:**
Monitoring is assigned to an appropriate individual such as the originator of the CAR or the originator's manager. Changes resulting from corrective action are documented.

### 4.10.5 Additional Audits

**Policy:**
Where the identification of nonconformances or departures casts doubts on compliance of policies, procedures, regulations, international quality standards, the appropriate areas of activity are promptly audited in accordance with section 4.13.

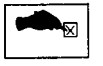
**Details:**
Special audits follow the implementation of corrective actions to confirm their effectiveness. A special audit is only necessary when a serious issue or risk to the business is identified. Special audits are carried out by trained and qualified personnel who are independent of the activity to be audited. See section 4.13 for more details.

### Revision History
Revision 1



## 4.11 Preventive Action

### Cross-references



ISO 17025:1999 Section 4.11

ISO 9001:2000 Section 8.4, 8.5.2, 8.5.3

### 4.11.1 Preventive Action Identification

**Policy:**
Opportunities for needed improvement and potential sources of nonconformances, either technical or with the quality system shall be identified. If action is required, action plans are developed, implemented and monitored, to reduce the likelihood of occurrence of such nonconformances and to take advantage of the improvement opportunities.

**Details:**
Records of preventive action are maintained in the Preventive Action Request (PAR) file and include the following information:

- details of potential nonconformances
- investigation
- preventive action
- follow-up verification

### 4.11.2 Preventive Action Plans

**Policy:**
The preventive action procedure includes the initiation of such actions and application of controls to ensure that they are effective.

**Details:**
Preventive action may result from the review of operational procedures and analysis of data. Analysis of data includes trend analysis, analysis of proficiency testing results, and risk analysis. The SOP# QSP 4-11-1 is utilized to implement opportunities for needed improvement and prevent potential sources of nonconformances.

### Revision History
Revision 1

# 4.12 Control of Records

## Cross-references

ISO 17025:1999 Section 4.12

ISO 9001:2000 Section 4.2.4, 6.3, 6.4, 7.1, 7.5.1, 7.5.2, 7.5.3, 8.1, 8.2.2, 8.2.3, 8.2.4

## 4.12.1 General

### 4.12.1.1 Procedures

**Policy:**
The SOP# QSP 4-12-1 is used to identify, collect, index, access, file, store, maintain, protect, backup, and dispose quality and technical records. Quality records include reports from internal audits and management reviews as well as corrective and preventive action records.

**Details:**
Records are available to demonstrate conformance to requirements and effective operation of the Quality System. Quality records from suppliers are also controlled.

All records, including test reports, are safely stored and held secure in locked areas, and in confidence to the client. Records are maintained in the designated archival area for at least three (3) years.

The master list of records is organized with the following information:
- Record No. / Form No.
- Record Name
- Filing Method (loose forms filed monthly, quarterly, semi-annual, annual or electronic)
- Active Files (files referred to within the work area) / Retention Period / Location
- Inactive Files (files referred to but not often and kept in storage) / Retention Period / Location
- Persons / Positions Responsible / Users

The dating format for records is MM/DD/YYYY.



### 4.12.1.2 Record Integrity

**Policy:**
All records are to be legible and shall be retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss.

**Details:**
The retention times for records are generally set at three (3) years. Exceptions to this are calibration sample records, electronic files, and chemistry, which are maintained indefinitely. Records may be in the form of any type of media, such as hard copy or electronic media.

### 4.12.1.3 Record Security

**Policy:**
All records are held secure and in confidence.

**Details:**
Access to records is secured through locked rooms and filing cabinets.

### 4.12.1.4 Record Backup

**Policy:**
The SOP# QSP 4-12-1 is followed to protect and backup data/records held on computers at all times and to prevent unauthorized access to or amendment of data/records on computers.

**Details:**
Data is password protected. Backups ensure integrity and availability of data / information in the event of a system / power failure.

## 4.12.2 Technical Records

### 4.12.2.1 Record Information

**Policy:**
Original observations, calculations, derived data and sufficient information to establish an audit trail, calibration records, personnel records and a copy of each test report [or calibration certificate] issued are retained for at least three (3) years.

The records for each test or calibration shall contain sufficient information to facilitate, if possible, identification of factors affecting the test uncertainty and to enable the test or calibration to be repeated under conditions as close as possible to the original. The records include the identity of personnel responsible for sampling, performing of each test and/or calibration and checking of results.

**Details:**
Technical records are accumulations of data (see 5.4.7) and information that result from carrying out tests and/or calibrations and which indicate whether specified quality or process parameters are achieved. They may include forms, contracts, work sheets, work books, note books, instrument printouts, magnetic media, check sheets, work notes, control graphs, test reports, calibration certificates, client's notes, papers and feedback, and test reports [and calibration certificates] to clients.

The records for each test contain sufficient information to permit its repetition. Records include:

- date of sampling
- sample receipt
- sample handling, storage, and disposal
- identification of personnel
- analyst proficiency
- equipment identification and performance
- calibration records
- media performance, where appropriate
- test organism batch # or lot #, where appropriate
- results
- reports (mailed, faxed)
- review

*Note* – the above records may be stored in separate locations. They are cross-referenced for easy retrieval.

#### 4.12.2.2 Recording

**Policy:**
Observations, data, and calculations are clearly and permanently recorded and identifiable to the specific job at the time they are made.

**Details:**
Handwritten records must be legible and made with indelible ink immediately after an observation, after data is collected and/or after calculations are made.



#### 4.12.2.3 Corrections to Records

**Policy:**
Changes to test data are made so as not to obscure or delete the previous data entry.

**Details:**
Mistakes are crossed out and the correct value entered alongside. Mistakes are not erased, made illegible, or deleted. All alterations to records are signed or initialed by the person making the correction. In the case of computer-collected data, similar measures are taken to avoid loss or change of original data.

## Revision History

Revision 1

## 4.13 Internal Audits

### Cross-references

ISO 17025:1999 Section 4.13

ISO 9001:2000 Section 8.1, 8.2.2, 8.2.3

### 4.13.1 Internal Audit Program

**Policy:**
The internal audit program involves periodic audits conducted according to a predetermined schedule for each year. This program is defined on an annual basis and conducted as outlined in this section with further details found in SOP# QSP 4-13-1. All elements of this Quality Manual will be audited each year and all relevant laboratory records are available to personnel conducting the audit. These audits are performed to verify operations continue to comply with the requirements of this Quality Manual and are effective.

**Details:**
The Quality Manual, test procedures, and laboratory results are verified for compliance. It is the responsibility of the Quality Manager to plan and organize audits as required by the schedule and requested by management. Audits are carried out by trained and qualified personnel who are wherever resources permit independent of the activity to be audited. Personnel are not to audit their own activities except when it can be demonstrated that an effective audit will be carried out (see also 4.10.5). Audits are performed through the aid of a checklist prepared in advance to minimize the possibility of overlooking any details during the audit.

Generally, the types of audits include:
- quality management system
- processes and procedures
- products, services, and reports

### 4.13.2 Corrective Action

**Policy:**
When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of test or calibration results, timely corrective action is taken and clients are notified if investigations show that laboratory results may have been affected.



**Details:**
Nonconformances that can be resolved easily are to be corrected immediately, ideally during the audit. Records are made on the audit checklist. Nonconformances that require a more involved resolution are recorded on a CAR and resolved as described in section 4.10.

Corrective actions and client modifications must be kept on record for each audit deviation that casts doubt as described in this section.

### 4.13.3 Records and Management

**Policy:**
Records are made of the activity being audited, the audit findings, and corrective actions that arise. Management ensures that corrective actions are discharged within an appropriate and agreed timeline.

**Details:**
A report is prepared by the auditors and distributed to those audited and/or the area manager/supervisor within an appropriate and agreed timeline. The audit report may include the following sections, as appropriate:
- audit objective and scope
- area or section audited
- personnel involved – auditors and auditees
- date of audit
- reference documents
- observations including nonconformances and commendations
- opening and closing meetings
- recommendations
- audit report distribution

The appropriate manager is responsible for ensuring that corrective actions are sufficiently recorded. Follow-up is performed by the auditor and recorded when corrective action is complete and deemed effective. The audit records are kept in the laboratory.

### 4.13.4 Follow-up Audits

**Policy:**
Follow-up audits are performed to verify and record the implementation and effectiveness of the corrective action taken.

**Details:**
The follow-up audit is performed at a mutually acceptable time between the area implementing corrective action and the auditor. This time is determined when the CAR is issued.

### Revision History

Revision 1



## 4.14 Management Reviews

### Cross-references

ISO 17025:1999 Section 4.14

ISO 9001:2000 Section 5.1, 5.4.2, 5.6, 6.2.1, 7.1, 8.5.1

### 4.14.1 Review of Quality System and Testing

**Policy:**
Management periodically (at least annually) and in accordance with a predetermined schedule and SOP# QSP 4-14-1, conduct a review of the laboratory's quality system and testing and/or calibration activities to ensure their continuing suitability and effectiveness and to introduce any necessary changes or improvements.

**Details:**
The review takes account of:
- suitability of policies and procedures
- reports from managerial and supervisory personnel
- the outcome of recent internal audits
- corrective and preventive actions
- assessments by external bodies
- results of inter-laboratory comparisons or proficiency tests
- changes in the volume and type of work undertaken
- feedback from clients, including complaints and client satisfaction surveys
- other relevant factors, such as quality control activities, resources and personnel training

A minimum period for conducting a management review is once a year. Results of the review feed into the laboratory planning system and include goals, objectives and action plans for the coming year.

A management review can be supplemented by consideration of related subjects at regular management meetings.

## 4.14.2 Findings, Actions, and Records

**Policy and Details:**
Findings from management reviews and the actions that arise are recorded in the minutes of the meeting. Management will ensure that the actions are discharged within an appropriate and agreed timeline.

### Revision History

Revision 1



## 5.1 General

### Cross-references

ISO 17025:1999 Section 5.1
ISO 9001:1994 Section N/A
ISO 9001:2000 Section N/A

### 5.1.1 Correctness and Reliability

**Policy and Details:**
Correctness and reliability of the tests and/or calibrations performed have many contributing factors including:

- human factors (see section 5.2)
- accommodation and environmental conditions (see section 5.3)
- test and calibration methods and method validation (see section 5.4)
- equipment (see section 5.5)
- measurement traceability (see section 5.6)
- sampling (see section 5.7)
- handling of test and calibration items (see section 5.8)

### 5.1.2 Measurement Uncertainty

**Policy:**
When developing test and calibration methods and procedures, total measurement uncertainty must be accounted for in the training and qualification of personnel, and in the selection and calibration of equipment.

**Details:**
The extent to which the factors contribute to total measurement uncertainty differs between types of tests and between types of calibrations.

See section 5.4.6 for more details.

### Revision History

Revision 1

## 5.2 Personnel

### Cross-references

ISO 17025:1999 Section 5.2

ISO 9001:2000 Section 5.5.1, 6.2.1, 6.2.2, 7.5.1

### 5.2.1 Competence and Qualification

**Policy:**
Management ensures the competency of all specific equipment operators, those performing tests and/or calibrations, those evaluating results and sign test reports and calibration certificates. Appropriate supervision is provided for employees undergoing training. Personnel performing specific tasks are qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

In addition, personnel responsible for the opinions and interpretations included in test reports also have:

- relevant knowledge of the technology used for the manufacturing of the items, materials, products tested, or the way they are used or intended to be used and of the defects or degradation that may occur during or in service
- knowledge of the general requirements expressed in the legislation and standards
- an understanding of the significance of deviations found with regard to the normal use of the items, materials, or products concerned

**Details:**
Management defines the minimum levels of qualification and experience necessary for all posts within the laboratory, and can be found within each job description.

Continued competence is monitored and where this is not achieved, the need to retrain personnel is considered. Where a method or technique is not in regular use, verification of personnel performance before they undertake tests, may be necessary.

### 5.2.2 Training Policies and Procedures

**Policy:**
Management will formulate the goals with respect to the education and the skills of the laboratory personnel. The training program is relevant to the present and anticipated tasks



of the laboratory. SOP# QSP 5-2-1 is utilized to identify training needs and providing the necessary training for personnel.

**Details:**
The skills and knowledge are defined in the job description for each job function as described in section 5.2.4. Management compares the job description to the skills and knowledge of the new incumbent to determine the training needs. All new employees must undergo the same base training, regardless of their incoming skills or knowledge.

Training in the laboratory must include all methods or parts of methods and techniques that personnel are asked to perform. Minimally, the analyst must demonstrate competency by observation by management and verification using replicate and/or check samples. For technicians who perform only parts of the method, confirmation of competency may be verified by observation only. Re-verification of all personnel must be performed annually on all methods or techniques pertinent to their job description.

In some cases it may be appropriate to define competence related to a particular technique or instrument rather then methods. If so, it will be necessary to define for each method, the necessary technique-based competence required together with any additional requirements.

### 5.2.3 Employees

**Policy:**
Competent permanent or contractual employees are employed in the laboratory. The technical manager ensures that contractual, additional technical employees, and key support personnel are supervised and work in accordance to the policies and procedures of this Quality Manual.

**Details:**
Testing must be either performed or supervised by an experienced person qualified to degree level. Personnel have relevant practical work experience (at least 2 years) before being allowed to perform accredited work.

### 5.2.4 Job Descriptions

**Policy:**
Current job descriptions for managerial, technical and key support personnel involved in tests and/or calibrations are maintained centrally in the administration area of the laboratory.

**Details:**
Minimum contents of job descriptions include:
- Background
- Purpose
- Responsibilities
- Qualifications
- Relationships
- Compensation
- Duration Reporting and Evaluation

A date and signature on the Lab Operations training paperwork demonstrates that each incumbent has read it and is in agreement. They are maintained current.

## 5.2.5 Authorized Personnel

**Policy:**
Management authorizes specific personnel to perform particular types of sampling, test and/or calibration, to issue test reports and calibration certificates, to give opinions and interpretations and to operate particular types of equipment. Records of the relevant competence, educational and professional qualifications, training, skills and experience of all technical personnel and contracted personnel are maintained by employee in the Personnel files. This information is readily available and includes the date on which authorization and/or competence was confirmed and the criteria on which the authorization is based and the confirming authority.

**Details:**
The purpose of these records is to provide evidence that personnel have been adequately trained and their competence to perform particular tests has been assessed. In some cases it may be pertinent to state any particular limitations to competence. The records include:
- academic and professional qualifications
- external and internal courses attended
- relevant on-the-job training and retraining as necessary (i.e., demonstration of competence)
- skills and experience (i.e., resume)
- relevant authorizations

Records are held centrally in the administration area.

## Revision History
Revision 1



# 5.3 Accommodation and Environmental Conditions

## Cross-references

ISO 17025:1999 Section 5.3

ISO 9001:2000 Section 6.3, 6.4, 7.1, 7.5.1, 7.5.2, 7.6, 8.2.3

## 5.3.1 Facility

**Policy:**
Laboratory facilities are appropriate to attain correct performance of tests and/or calibrations. This may include, but not limited to, energy sources, lighting, heating, ventilation and any other environmental conditions.

Appropriate care is taken to ensure that the environment does not invalidate the results or adversely affect the required quality of any measurement. Particular care is taken when sampling, tests and/or calibrations are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of tests and calibrations are documented.

**Details:**
This section deals with the test areas in the laboratory and premises for support such as sample receipt and storage. Central laboratory supplies and services, such as water purification systems, air supply, vacuum source, and sample storage, are appropriate to facilitate proper performance of tests.

## 5.3.2 Monitoring

**Policy:**
Critical environmental conditions are monitored, controlled and recorded as required by the relevant specifications, methods, and procedures or where they may influence the quality of the results. Due attention is paid, for example, to dust, air quality, humidity, temperature, and sound and vibration levels, as appropriate to the technical activities concerned. Tests and calibrations are stopped when the environmental conditions jeopardize the results of the tests and/or calibrations.

**Details:**
Laboratories are ventilated to reduce the levels of contamination, lower humidity, and control temperature. Laboratories' test areas are air-conditioned. The relative humidity in test areas is 45-50 and the temperature is 20-25 °C.

Bench tops and floors are made of impervious, smooth easily cleaned materials. There is at least two linear meters workspace per analyst while working. Walls and ceilings are made of materials that are smooth and easily cleaned.

[If appropriate, the SOP#[PROCEDURE#] is used].

### 5.3.3 Separation of Incompatible Activities

**Policy:**
Effective separation between neighboring areas is made when the activities are incompatible. Measures are taken to prevent cross-contamination.

**Details:**
Reference materials and certified reference materials must be kept separated from samples (log-in and storage). Sample log-in and storage must be segregated, ideally in a separate area from the testing laboratory, and include proper sanitation to exclude the possibility of cross-contamination. Segregation of activities is achieved through time and space allocations.

An example of space segregation would be for a trace analysis. Physical separation of the trace analysis from high-level analysis is achieved through the use of separate rooms.

An example of time segregation would be the coordination of activities at different times. It may be appropriate to perform work on "cleaner" samples first before starting "dirtier" type samples.

### 5.3.4 Controlled Access

**Policy:**
Access to and use of areas affecting quality of the tests and/or calibrations is defined and controlled.

**Details:**
Access to the laboratory is restricted to authorized personnel. The authorized personnel are made aware of the following items:
- the intended use of the area
- the restrictions imposed on working within such areas
- the reasons for imposing the restrictions



### 5.3.5 Good Housekeeping

**Policy:**
Measures are taken to ensure good housekeeping in the laboratory. Special procedures are prepared when necessary.

**Details:**
Controlled use of cleaning and pest control materials is exercised. The laboratory complies with the local health and safety requirements. [If appropriate, the SOP#[PROCEDURE#] is used].

### Revision History

Revision 1

## 5.4 Tests and Calibration Methods and Method Validation

### Cross-references

ISO 17025:1999 Section 5.4

ISO 9001:2000 Section 4.2.1, 4.2.3, 6.3, 6.4, 7.1, 7.2.1, 7.3, 7.4.3, 7.5.1, 7.5.2, 7.6, 8.1, 8.2.3, 8.2.4

### 5.4.1 General

**Policy:**
Methods and procedures used for all tests and/or calibrations are appropriate as per:
- sampling, handling, transport, storage, and preparation of items to be tested and/or calibrated
- an estimation of the measurement of uncertainty as well as statistical techniques for analysis of test and/or calibration data where appropriate

Instructions on the use and operation of all relevant equipment and on the handling and preparation of items for testing and/or calibration are available. All instructions, standards, manuals and reference data relevant to the work of the laboratory are maintained current and readily available to personnel. Deviation from test and calibration methods must be documented, technically justified, authorized, and accepted by the client.

**Details:**
There are SOPs for sampling, sample handling, transport, storage, preparation of test items, QA/QC procedures (media QC, incubation times and temperatures, equipment calibration and maintenance, process control QC), and standards for approving / rejecting results. These may be combined with or separate from the method. The content of a test method includes:
- scope
- description of test items
- holding times
- quantities to be tested
- materials and equipment required
- physical environmental conditions required (incubation times and temperatures, pH requirements)
- description of procedures
- sample identification
- method of recording observations and results
- safety measures



- documentation
- method for data analysis and presentation
- sensitivity of method
- quality control plan

International, national, or regional standards or other recognized specifications that contain sufficient and concise information on how to perform the tests and/or calibrations are not necessarily supplemented or rewritten as an internal procedure when they are written in a way that can be used as published by laboratory staff. Consideration may need to be given to providing additional documentation for optional steps in the method.

### 5.4.2 Selection of Methods

**Policy:**
Test and/or calibration methods, including methods for sampling, meet the needs of the client and are appropriate for the tests and/or calibrations it undertakes. Preference is given to reference methods published as international, national, or regional standards. The laboratory ensures that the latest edition of a standard is used unless it is not appropriate or possible to do so. When necessary, the standard is supplemented with additional details to ensure consistent application.

**Details:**
Methods that have been published either in international, national, or regional standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer are selected when the client does not specify the method to be used. These methods may be adopted from the {AOAC, FDA BAM, USDA FSIS & AMS, APHA SMEDP, ISO, ICMSF, National Food Processors, American Association of Cereal Chemists, Environmental Protection Agency, OIE, ASTM, etc.}

The ability of the laboratory to achieve satisfactory performance against documented performance characteristics is verified before samples are analyzed.

Laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated. The client is informed as to the method chosen. The laboratory confirms that it can properly operate standardized methods before introducing the tests or calibrations. If the standardized method changes, the confirmation is repeated.

The client is informed when the method proposed by the client is considered to be inappropriate or out of date.

## 5.4.3 Laboratory-Developed Methods

**Policy:**
Introduction of test and calibration methods developed internally is a planned activity and is assigned to qualified personnel equipped with adequate resources. Plans are updated as development proceeds and ensures effective communication amongst all personnel involved.

**Details:**
Methods developed in-house are validated and authorized before use. Where available, Certified Reference Materials (CRMs) are used to determine any systemic bias, or where possible results are compared with other techniques, preferably based on different principles of analysis. Determination of uncertainty must be part of this validation process and is essential for ongoing quality control.

## 5.4.4 Non-Standard Methods

**Policy:**
Utilization of non-standard methods is subject to agreement with the client and includes a clear specification of the client's requirements and the purpose of the test and/or calibration. The developed method is validated appropriately before use.

**Details:**
Discussion and agreement for the use of non-standard methods is recorded as part of contract review procedures (see section 4.4).

All non-standard and new tests are validated for their intended purpose. Qualitative test methods must be validated to demonstrate estimated sensitivity and specificity, relative accuracy to official methods (if appropriate), positive and negative deviation, limit of detection, matrix effect, repeatability, and reproducibility.

Quantitative test methods are validated to demonstrate specificity, sensitivity, relative accuracy, positive and negative deviation, repeatability, reproducibility, and limit of determination.

For new methods where procedures are developing rapidly, especially for emergency situations, it may be necessary to circumvent normal validation procedures. Minimally, this must be a demonstrated recovery in replicate.

New test and/or calibration methods are documented prior to providing test and/or calibration results to clients and contain at least the following information:



- appropriate identification
- scope
- description of the type of item to be tested or calibrated
- parameters or quantities to be determined
- apparatus and equipment, including technical performance requirements
- reference standards and reference materials required
- environmental conditions required and any stabilization period needed
- description of the procedure, including
  - affixing identification marks, handling, transporting, storing and preparing of items
  - ensuring checks are made before the work is started
  - checking that the equipment is working properly and, where required, calibrating and adjusting the equipment before each use
  - listing method of recording the observations and results
  - indicating any safety measures to be observed
- criteria and/or requirements for approval/rejection (quality control plan)
- data to be recorded and method of analysis and presentation
- uncertainty or procedure for estimating uncertainty

## 5.4.5 Validation of Methods

### 5.4.5.1 Performance Characteristics

**Policy:**
Validation of a method establishes, by systematic laboratory studies, that the performance characteristics of the method meet the specifications related to the intended use of the test results.

**Details:**
The performance characteristics of a validation plan includes, as applicable:
- selectivity and specificity
- range
- linearity
- sensitivity
- limit of detection
- limit of quantitation
- ruggedness
- accuracy
- precision
- reporting limit
- repeatability
- reproducibility

- recovery
- confirmation techniques
- criteria for the number of samples tested to validate method as per defined scope of method
- action levels where defined by regulation
- quality control incorporating statistics as applicable
- interpretation of population results as applicable

Performance characteristics that are selected take into account the intended use of the method, whether for screening, confirmatory analysis, or quantitation.

The design, verification of the method and documentation procedures for validation are planned and conducted by qualified personnel, equipped with adequate resources.

This section lists a few acceptable validation procedures. The choice of the procedure depends on the extent of the deviation from the published method.

Validation of methodology is a value judgment in which the performance parameters of the method are compared with the requirements for the test data. A prerequisite for a valid method is that data produced by the method must attain a state of statistical control. Such a state is obtained when the mean value of a large number of individual values tends to approach a limiting value called the limiting mean.

Methods may be validated by one or more alternative procedures. Some of these procedures are described below. Apparent differences can be analyzed statistically to confirm their significance. In all cases, the reasons for choosing one or more alternatives must be documented.

- analysis of standard reference materials (SRM) that are identical or almost identical to the test samples
- in the absence of suitable SRMs, analysis of reference materials that are similar in all respect to the test samples; the use and validity of this reference material must be documented
- using an alternative method to measure the same parameter provides a very high level of confidence if results are confirmed
- recovery studies by the addition of a known concentration of the parameter of interest to some of the replicates being measured

The parameters to be determined include:

- the scope of the method and any known interference
- detection limit
- the range of concentration where the method is valid
- precision and bias
- intra-laboratory variations



- inter-laboratory variations

Judgment is required to determine if some or all of the above is required. Requirements will depend largely on the extent of deviation from the original method.

Developments in methodology and techniques require methods to be changed from time to time. The difference in performance between revised and obsolete methods is established so that it is possible to compare old and new data.

Where a change in method involves only minor adjustments, such as sample size, or different reagents, the amended method is validated and the changes brought to the attention of the accreditation body at the next accreditation audit. Where the proposed change involves technology or methodology, the laboratory seeks the approval of the accreditation body.

Records are kept on all validation activities. The records include any of the performance characteristics chosen, reference procedures or guidance documents followed to validate the method or custom validation procedure, and a final confirmation (memo to file) that the method validation results are acceptable for continued use of the method. An example statement would be "This memo serves as record that the validation of the XYZ Test Method has been approved for use by [name and title of approver]".

### 5.4.5.2 Fit for Use

**Policy:**
The laboratory validates non-standardized methods, laboratory-designed/developed methods, standardized methods used outside their intended range, and amplifications of standard methods to confirm that the methods are fit for the intended use. The validation is as extensive as is necessary to meet the needs in the given application or field of application (may include procedures for sampling, handling, and transportation). The laboratory records the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

**Details and Procedure:**
Validation records are kept as in section 5.4.5.1. Included in these records is the validation procedure. The procedure used for the validation is likely to vary between different methods. Therefore, the procedures included in the laboratory records are not as detailed as a typical SOP, but are sufficient enough to re-create how the method was validated.

The techniques used for the determination of the performance of a method, are one of, or a combination of, the following:

- calibration using reference standards or reference materials
- comparison of results achieved with other methods
- inter-laboratory comparisons
- systematic assessment of the factors influencing the result
- assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.

When changes are made in the validated non-standard method, the influence of such changes carried out is documented and if appropriate a new validation is performed.

#### 5.4.5.3 Client's Needs

**Policy:**
The range and accuracy of the values obtainable from validated methods (e.g., the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object) as assessed for the intended use is relevant to the client's needs.

**Details:**
Validation includes the specification of the requirements, determination of the characteristics of the methods, the comparison of the requirements with the values of the characteristics of the method, and a statement on the validity.

As method development proceeds, regular review is required to verify that the needs of the client are still being fulfilled. Changing requirements requiring modifications to the development plan are approved and authorized.

Validation is always a balance between costs, risks, and technical possibilities.

### 5.4.6 Uncertainty of Measurement

#### 5.4.6.1 Calibration

**Policy:**
Physical, chemical, and biological standards are calibrated or characterized by qualified subcontractors.

**Details and Procedures:**
Repeatability and reproducibility data are components of measurement uncertainty and are determined as a first step towards producing estimates of this parameter. The



uncertainty of measurement is available on the certificate of analysis or calibration certificate from a subcontractor.
**Note** - in-house calibrations include procedures for uncertainty of measurement estimates where this is common practice.

#### 5.4.6.2 Testing

**Policy:**
The SOP# QSP 5-4-1 is utilized to estimate uncertainties of measurement in testing, except when the test methods preclude such rigorous calculations. In certain cases it is not possible to undertake metrologically and statistically valid estimations of uncertainty of measurement. In these cases the laboratory attempts to identify all the components of uncertainty and make the best possible estimation, and ensure that the form of reporting does not give an exaggerated impression of accuracy. Reasonable estimation is based on knowledge of the performance of the method and on the measurement scope and makes use of previous experience and validation data.

**Details:**
The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:
- requirement of the test method
- requirement by the client
- if there are narrow limits on which decisions on conformance to a specification are based

In cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied the estimation uncertainty of measurement by following the reporting instructions (see section 5.10).

#### 5.4.6.3 Uncertainty Components

**Policy:**
When estimating the uncertainty of measurement, all uncertainty components that are of importance in the given situation are taken into account using accepted methods of analysis.

**Details:**
Sources contributing to the uncertainty include, but are not necessarily limited to, the reference standards and reference materials used, methods and equipment used, the environmental conditions, the item being tested or calibrated and the operator.

The predicted long-term behavior of the tested and/or calibrated item is normally not taken into account when estimating the measurement uncertainty.

For further information, see ISO 5725 and the Guide to Expression of Uncertainty in Measurement.

## 5.4.7 Control of Data

### 5.4.7.1 Calculations and Data Transfers

**Policy:**
Calculations and data transfers are subject to appropriate checks in a systematic manner.

**Details:**
Test data are validated through the following arrangements by the Laboratory and Service Manager:

- checks to determine accuracy of calculations, conversions, and data transfers
- checks for transcription errors, omissions, and mistakes
- checks to determine consistency with normal or expected values

For those analyses where manual data reduction is required, it is performed according to the instructions provided in the test method or SOP.

### 5.4.7.2 Computers and Automated Equipment

**Policy:**
When computers or automated equipment are used for the acquisition, processing, manipulation, recording, reporting, storage or retrieval of test or calibration data, the laboratory ensures that:

- computer software developed by the user is documented in sufficient detail and suitably validated or otherwise checked as being adequate for use
- procedures are established and implemented for protecting the integrity of data; such procedures include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission, and data processing (see section 4.12.1.4)
- computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of test and calibration data
- data is securely maintained by preventing unauthorized access to, and unauthorized amendment of, computer records



**Details and Procedures:**
Data generated using computer software programs that are interfaced directly to instruments incorporates all dilutions and calculations, thereby eliminating the need for manual data reduction.

Commercially developed software in general use within its designed application range may be considered sufficiently validated. Laboratory software configuration / modifications are validated as outlined in SOP# QSP 5-5-1.

Electronic records, electronic signatures, and handwritten signatures executed to electronic records must be equivalent to proper records and handwritten signatures to paper and are validated by procedures in 21 CFR. Part II (Docket No. 92NO251) RIN0910-AA29; Federal Register: March 20, 1997, Volume 62, Number 54), Rules and Regulations, pages 13429-13466. For further details see:

http://www.fda.gov/cder/esig/index.htm

## Revision History

Revision 1

## 5.5 Equipment

### Cross-references

ISO 17025:1999 Section 5.5

ISO 9001:2000 Section 4.2.1, 4.2.3, 5.1, 7.1, 7.4, 7.5.1, 7.5.2, 7.5.3, 7.6, 8.1, 8.2.3, 8.2.4

### 5.5.1 Required Equipment

**Policy:**
The laboratory is furnished with all items for sampling, measurement and test equipment required for the correct performance of the tests and/or calibrations (including sampling, preparation of test and/or calibration items, processing and analysis of test and/or calibration data). When equipment is used outside the laboratory's permanent control, it ensures that the requirements of this Quality Manual are met.

**Details:**
Equipment is used in an environment appropriate to its proper performance. All equipment required by a test is described in each method, including the equipment's tolerances.

### 5.5.2 Required Accuracy

**Policy:**
Equipment and software used for testing, calibration and sampling are capable of achieving the accuracy required and comply with specifications relevant to the tests and/or calibrations concerned. Calibration programs are established for key quantities or values of the instruments where these properties have a significant affect on the results. When received, equipment, including that used for sampling, is checked to establish that it meets the laboratory's specification requirements, complies with the relevant standard specifications, and is checked and/or calibrated in accordance with section 5.6 before use.

**Details:**
The procedures for checking newly received equipment are as determined by manufacturers' specification and/or those determined by the laboratory during procurement.



### 5.5.3 Authorized Personnel

**Policy:**
Equipment is operated by authorized personnel. Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) is readily available for use by the appropriate laboratory personnel.

**Details:**
Access to laboratory equipment is controlled to ensure that only authorized personnel use equipment.

### 5.5.4 Unique Identification

**Policy:**
Each item of equipment used for testing and calibration is uniquely identified as appropriate.

**Details:**
Measuring and testing equipment is uniquely identified through a unique serial number. Measuring and testing equipment includes any instrument that could affect the quality of test results. Components that can be interchanged between various instruments are tracked in equipment logbooks, but are not assigned individual serial numbers.

### 5.5.5 Inventory and Maintenance Records

**Policy:**
Records are maintained of each item of equipment significant to the tests and/or calibrations performed. The records include the following:

- identity of the item of equipment (and its software)
- manufacturer's name, type identification, and serial number and/or other unique identification
- checks that equipment complies with the specification (see section 5.5.2)
- current location, where appropriate
- the manufacturer's instructions, if available, or reference to their location
- dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and due date of next calibration
- maintenance carried out to date and the maintenance plan (includes calibration)
- damage, malfunction, modification or repair to the equipment

**Details:**
The above information related to service and maintenance is kept in individual equipment binders. Other information kept in these binders may include:

- date received and date placed in service
- condition when received (e.g., new, used, refurbished)
- dates and results of calibration and/or verification and date of next calibration and/or verification
- performance history, where appropriate (e.g., response time, drift, noise level)

## 5.5.6 Equipment Procedures

**Policy:**
The SOP# QSP 5-5-1 is utilized as an established plan for safe handling, transport, storage, use and maintenance (including calibration) of measuring equipment, and appropriate use of correction factors to ensure proper functioning and in order to prevent contamination or deterioration.

Note – additional procedures may be necessary when measuring equipment is used outside the permanent laboratory for tests, calibrations, or sampling (currently not applicable at our laboratory).

**Details and Procedures:**
The procedures for each piece of measuring equipment are located in the appropriate equipment binder. These procedures detail any information for safe handling, transport, storage, use, and maintenance of measuring equipment.

## 5.5.7 Out of Service Equipment

**Policy:**
Equipment that has either been subjected to overloading or mishandling, or gives suspect results, or has been shown to be defective or outside specified limits, is taken out of service, clearly marked, and appropriately stored until it has been repaired and shown by calibration or test to perform correctly.

**Details:**
Routine testing work is completely discontinued on equipment that even shows minor nonconformances. Not only do we do this for ethical reasons in support of our client, but minor nonconformances are often indicative of major breakdowns in expensive equipment. These breakdowns need to be avoided wherever possible. Out of service equipment is clearly marked as outlined in section 5.5.8.



The laboratory examines the effect of the defect or departure from specified limits on previous test and/or calibrations and institutes the "Control of Nonconforming Work" procedure as outlined in section 4.9.

## 5.5.8 Calibration Status

**Policy:**
Equipment requiring calibration is labeled to indicate the calibration status and/or operational status and the date when re-calibration is due when appropriate.

**Details:**
All calibration information is recorded in the equipment binders found by each instrument and include the person who performed the calibration, the date it was performed, the date it is due for re-calibration, and the equipment's identification number.

Measuring equipment that has failed calibration or is deemed out of service is deemed to be out of service according to section 5.5.7:

A piece of equipment that is not calibrated or checked is identified in its equipment binder to not be used for sample analysis.

## 5.5.9 Return to Service

**Policy:**
When equipment goes outside the direct control of the laboratory for a period, the laboratory ensures that the function and calibration status of the equipment are checked and validated and shown to be satisfactory before the equipment is returned to service.

**Details and Procedures:**
The procedures used to check and ensure that the function and calibration status of the equipment are satisfactory before the equipment is returned to service are outlined in the manufacturer's equipment manual. Any additional quality control checks are outlined in the "Quality Control Plan" section of the appropriate test method.

## 5.5.10 Periodic Checks

**Policy:**

When intermediate checks are needed to maintain confidence in the calibration status of equipment, these checks are carried out periodically according to defined procedure.

**Details and Procedures:**
As stated in section 5.5.6, the procedures for each piece of measuring equipment are located in the appropriate equipment binder. SOP# QSP 5-5-1 outlines a general maintenance plan for equipment and includes various checks. Internal quality control checks are specified in individual test methods that are located in the appropriate laboratory areas thereby providing procedures for intermediate checks.

## 5.5.11 Correction Factors

**Policy**
Calibrations that give rise to a set of correction factors are updated along with all copies of this data (e.g., in computer software).

**Details and Procedures:**
The updating of correction factors including all copies is assured by following the appropriate test method or SOP. It is the responsibility of the Laboratory and Service Manager to ensure that all copies are updated.

## 5.5.12 Safeguards against Adjustments

**Policy:**
Test and calibration equipment, including hardware and software, are safeguarded from adjustments that invalidate test and/or calibration results/status.

**Details:**
Safeguards against adjustment for laboratory equipment include:
- detailed SOPs and manufacturer's manuals on the operation of the equipment
- policies permitting only fully trained and competent personnel to operate equipment
- access to the laboratory is restricted to authorized personnel

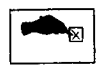
Safeguards against adjustment for software includes:
- password protection for important files and packages
- access to the laboratory is restricted to authorized personnel

## Revision History
Revision 1



# 5.6 Measurement Traceability

## Cross-references



ISO 17025:1999 Section 5.6

ISO 9001:2000 Section 7.1, 7.6

## 5.6.1 General

**Policy:**
Test and/or calibration equipment for subsidiary measurements (e.g., for environmental conditions) having a significant effect on the accuracy or validity of the result of the test, calibration, or sampling are calibrated before being put into service. All measurement and test equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before being put into service. As mentioned in section 5.5, the SOP# QSP 5-5-1 outlines an established program for the maintenance of equipment and includes calibration.

**Details:**
The program includes a system for selecting, using, calibrating, checking, controlling, and maintaining:
- measurement standards
- reference standards used as measurement standards
- measuring and test equipment used to perform tests and calibrations

Procedures are documented where appropriate. All measurements that play a defining role in testing accuracy are based directly or indirectly on reference standards, reference materials, certified reference materials, or other standards or materials having appropriate traceability.

[Records are maintained for each standard. These records include, as applicable:
- supplier, grade, batch#
- dates of preparation or verification
- measurement of weights, volumes, time intervals, temperatures, and pressures and related calculations
- relevant processes (e.g., pH adjustment, sterilization)
- verification results
- identification of personnel involved

Records are maintained for each lot of test organisms. These records include, as applicable:
- source, including age, species, and lot#
- date of arrival

- arrival condition
- culture and/or holding conditions
- feeding history
- health history including mortality, disease and treatment
- acclimation history

Disposal of test organisms is carried out humanely and conforms to applicable legal requirements.

Reagents prepared in the laboratory are labelled to identify substance, strength, solvent (where not water), any special precautions or hazards, restrictions of use, and date of preparation and/or expiry. The person responsible for the preparation of the reagent is identified either from the label or from records.]

## 5.6.2 Specific Requirements

### 5.6.2.1 Calibration

**Policy:**
The program for calibration equipment is designed and operated to ensure that calibration measurements are traceable to the Système International (SI) units of measurement.

**Details:**
Traceability of measurement is assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories show that there is a link to a primary standard or to a natural constant realizing the SI unit by an unbroken chain of calibrations. The calibration certificates contain the measurement results including the measurement uncertainty and/or a statement of compliance with an identified metrological specification (see also section 5.10.4.2).

Calibration laboratories accredited to ISO 17025 are considered competent to provide the appropriate calibration services.

Traceability to SI units of measurement may be achieved by reference to an appropriate primary standard or by reference to a natural constant the value of which in terms of the relevant SI unit is known.

The term "identified metrological specification" means that it must be clear from the calibration certificate against which specification the measurements have been compared with, by including the specification or by giving an unambiguous reference to the specification.



When the terms "international standard" or "national standard" are used in connection with traceability, it is assumed that these standards fulfil the properties of primary standards for the realization of SI units.

Maintain certificates of all reference standards, measuring equipment, or certified reference material used in ensuring traceability. Where traceability to national standards of measurement is not applicable, the laboratory provides satisfactory evidence of correlation of results, for example by participation in a suitable program of inter-laboratory comparisons or proficiency testing.

Reference standards, such as thermometers and weights are traceable to a national or international standard (e.g., NIST).

### 5.6.2.2 Testing

5.6.2.2.1

**Policy:**
The requirements given in section 5.6.2.1 apply to measuring and test equipment with measuring functions used, unless it has been established that the associated calibration uncertainty contributes little to the total uncertainty of the test result. When this situation arises, the laboratory ensures that equipment used can provide the accuracy of measurement needed.

**Details:**
The extent to which the requirements in section 5.6.2.1 are followed depends on the relative contribution of calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements are strictly followed. If, however, calibration is not one of the major contributors to the total uncertainty, other ways for providing confidence may be used, as given in section 5.6.2.2.2.

5.6.2.2.2

**Policy:**
Where traceability to SI units of measurement is not possible and/or not relevant, other means for providing confidence in the results are applied such as:
- the use of suitable reference materials certified to give a reliable characterization of the material
- mutual-consent standards or methods which are clearly specified and agreed upon by all parties concerned

- participation in a suitable program of inter-laboratory comparisons or proficiency testing

**Details:**
Reliable characterization involves an estimate of recovery.

The laboratory participates in proficiency testing and/or check sample programs. The list of programs is maintained by the Quality Manager.

## 5.6.3 Reference Standards and Reference Materials

### 5.6.3.1 Reference Standards

**Policy:**
The SOP# QSP 5-6-1 outlines the program for the calibration of reference standards. Reference standards are obtained or calibrated by a body that can provide traceability as described in section 5.6.2.1. Such reference standards of measurement held by the laboratory are used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

**Details:**
Reference standards are obtained from the National Institute of Standards and Technology (NIST) [National Research Council for certain measurements are available in Canada], if applicable.

### 5.6.3.2 Reference Materials

**Policy:**
Where possible, reference materials are traceable to SI units of measurement, or to certified reference materials. Internal reference materials are checked as far as is technically and economically practicable.

**Details:**
Reference materials, including calibration standards, used in chemical measurement are prepared so that the point of measurement is similar or equivalent to that of the samples. The matrix, prior to the addition of the analyte does not have a detectable concentration of the analyte. Reagents used in the preparation of reference materials, including calibration standards are of certified purity.

[Certified reference cultures are traceable to a national or internationally recognized type culture collection. Reference cultures from laboratory sources must be identified to standard reference sources. These reference cultures must be handled to maintain their biochemical



reaction and physiological characteristic integrity. All Reference Cultures and Certified Reference Cultures are not transferred more than five times from a type culture collection. Alternatively, re-identify the culture for key biochemical and physiological characteristics using national or internationally recognized reference sources. Another alternative is to grow the type culture, then freeze it (or freeze-dry it), and use periodically. Thus, extending the length of time required before repurchase or re-identification. These may also be commercially available and purchased for use. Companies selling Certified Reference Cultures must comply with the requirements of ISO 17025 for a calibration laboratory.]

### 5.6.3.3 Intermediate Checks

**Policy:**
Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials are carried out according to defined procedures and schedules.

**Details and Procedures:**
The control check standards used to verify the accuracy of all the other standards are prepared independently from all the other standards used to establish the original calibration. These control check standards are preferably prepared from a separate lot # or source. It is the responsibility of the <Click here and type appropriate TITLE> to establish and maintain the individual schedule for each SOP and/or test method.

### 5.6.3.4 Transport and Storage

**Policy:**
The SOP# QSP 5-6-1 outlines safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

**Details:**
Additional procedures may be necessary when reference standards and reference materials are used outside the permanent laboratory for tests, calibrations, or sampling.

Proper conditions are established for housing, handling, and care of {reference standards/reference materials/test organisms}. [Test organisms are acclimatized to the test environment for an adequate period before a test is initiated]. All information needed to properly identify references appears on their housing or containers.

## Revision History
Revision 1

## 5.7 Sampling

### Cross-references

ISO 17025:1999 Section 5.7

ISO 9001:2000 Section 4.2.4

### 5.7.1 Sampling Plan and Procedures

**Policy:**
The SOP# QSP 5-7-1 outlines the sampling plan and procedures for sampling for any laboratory sampling of substances, matrices, materials or products for subsequent testing or calibration. The sampling plan and procedures are available at the location where sampling is performed. Sampling plans are based on appropriate statistical methods whenever reasonable. The sampling process addresses the factors to be controlled to ensure validity of the test and calibration results.

**Details:**
Sampling is a defined procedure whereby a part of a substance, matrix, material or product is taken to provide for testing or calibration as a representative sample of the whole. Sampling can also be required by the appropriate specification for which the substance, matrix, material or product is to be tested or calibrated. In certain cases (e.g., forensic analysis), the sample may not be representative, but determined by availability.

The sampling plan describes the allocation, withdrawal and preparation of a sample or samples from a substance, matrix, material or product to yield the required information. All samples are collected and placed in sealed containers.

### 5.7.2 Deviations, Additions or Exclusions

**Policy:**
Where the client requires deviations, additions or exclusions from the sampling procedure, these are recorded in detail with the appropriate sampling data and included in all documents containing test and/or calibration results, and communicated to the appropriate personnel.

**Details:**
The physical appearance and temperature of all test items is observed and recorded upon receipt. Any deviations from specifications or observations are discussed with the client as to the suitability of the sample. Cross-contamination is the most critical issue from broken, leaking samples for both qualitative and quantitative tests.



### 5.7.3 Records

**Policy:**
The SOP# QSP 5-7-1 outlines the procedures for recording relevant data and operations relating to sampling that forms part of the testing or calibration that is undertaken. These records include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and any diagrams or other equivalent means to identify the sampling location as necessary, and, if appropriate, the statistics upon which the sampling procedures are based.

**Details:**
Adequate sample identification upon receipt in the laboratory includes:
- unique and unambiguous sample identification, usually a number or alphanumeric identification, retained throughout the testing life of the test item
- name of person(s) the report will be sent to
- sample source and date if available
- identification number or description from (client) if any
- product description
- tests desired and/or methods requested
- date of receipt
- delivery carrier
- sample condition, including temperature

### Revision History

Revision 1

## 5.8 Handling of Test and Calibration Items

### Cross-references

ISO 17025:1999 Section 5.8

ISO 9001:2000 Section 6.3, 6.4, 7.1, 7.4.3, 7.5, 8.2.4

### 5.8.1 Procedures

**Policy:**
The SOP# QSP 5-8-1 outlines the procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of test and/or calibration items, including all provisions necessary to protect the integrity of the test or calibration item, and the interests of the laboratory and the client.

**Details:**
Samples, reagents, and standards are stored so as to ensure their integrity by preventing against deterioration, contamination, and loss of identity. It is recognized that this is a general statement, but details are elaborated upon in SOP# QSP 5-8-1.

### 5.8.2 Identification of Test and/or Calibration Items

**Policy:**
Test and/or calibration items are systematically identified as they arrive at the laboratory. The identification is retained throughout the life of the item in the laboratory. The system is designed and operated so as to ensure that items cannot be confused physically, or when referred to in records or other documents. The system accommodates a sub-division of groups of items and the transfer of items within and from the laboratory when appropriate.

**Details:**
Sample labelling indicates the unique identification and conforms to applicable legal requirements. Where conformity of possession of a test sample must be maintained for forensic or other purposes, the laboratory establishes and documents a system for appropriate chain-of-custody (forensic samples may be used in a court of law for evidentiary purposes).



### 5.8.3 Receipt

**Policy:**
Upon receipt of the test or calibration item, any abnormalities or departures from normal or specified conditions, as described in the relevant test or calibration method, are recorded. When there is any doubt as to the suitability of an item for test or calibration, or when an item does not conform to the description provided, or the test or calibration required is not specified in sufficient detail, the laboratory consults the client for further instructions before proceeding and keeps a record of the discussion.

**Details:**
Conform to applicable regulations or contractual arrangements. The condition of sample may include or relate to damage, quantity, preparation, packaging, or temperature. Preparation may include addition of chemical preservative, removal of moisture, isolation of portion of sample to be tested, homogenization, or subsampling.

Arrangements are in place to ensure that elapsed time between sampling and testing does not exceed test method specifications (holding time).

### 5.8.4 Protection

**Policy:**
The SOP# QSP 5-8-1 outlines the procedures and appropriate facilities for avoiding deterioration, loss or damage to the test or calibration item during storage, handling and preparation and testing; instructions provided with the item are followed. When items have to be stored or conditioned under specified environmental conditions, these conditions are maintained, monitored, and recorded. Where a test item is to be held secure (e.g., for reasons of record, safety or value, or to enable complementary test and/or calibrations to be performed later), the laboratory has arrangements for storage and security that protect the condition and integrity of the secured item concerned.

**Details:**
Where test items are to be returned into service after testing (e.g., for non-destructive testing or human beings subject to medical tests), special care is required to ensure that they are not damaged or injured during the handling, testing or storing/waiting processes.

A sampling procedure and information on storage and transport of samples, including all information that may influence the test or calibration result, is provided to those responsible for taking and transporting the samples.

The laboratory establishes whether the sample has received all necessary preparation or whether the client requires preparation to be undertaken or arranged by the laboratory. Proper requirements for packaging, environmental conditions, and separation from incompatible materials are observed. Where samples have to be stored or conditioned under specific conditions, these conditions are maintained, monitored, and recorded, where necessary.

Where a sample, or portion of a sample, is to be held secure (e.g., for reasons of record, safety, or value, or to enable check tests to be performed later), the laboratory has storage and security arrangements that protect the condition and integrity of the sample.

## Revision History

Revision 1



## 5.9 Assuring the Quality of Test and Calibration Results

### Cross-references

ISO 17025:1999 Section 5.9

ISO 9001:2000 Section 6.3, 6.4, 7.1, 7.4.3, 7.5.1, 7.5.2, 7.5.3, 7.5.5, 8.1, 8.2.3, 8.2.4, 8.4

## 5.9 Assuring the Quality of Test and Calibration Results

**Policy:**
Quality control procedures are utilized to monitor the validity of test and/or calibration results. These procedures are for each test method utilized in the laboratory. The resulting data are recorded so that trends are detectable (and where practicable, statistical techniques are applied to the reviewing of the results. This monitoring is planned and reviewed and may include, but not limited to, the following:

- regular use of certified reference materials and/or internal quality control using secondary reference materials
- participation in inter-laboratory comparisons or proficiency testing programs
- replicate tests or calibrations using the same or different methods
- re-testing or re-calibration of retained items
- correlation of results for different characteristics of an item

**Details:**
The methods utilized from the above list will be appropriate for the type and volume of the work undertaken. Records are maintained of assurance activities and any actions taken.

As a guide, for routine analyses the level of internal quality control is typically 5% of the sample throughput. For more complex procedures, 20% is not unusual and on occasions even 50% may be required. For analyses performed infrequently, a full system validation is performed on each occasion. This may typically involve the use of a reference material containing a certified or known concentration of analyte, followed by replicate analyses of the sample and spiked sample. For analyses undertaken more frequently, systematic quality control procedures incorporating the use of control charts and check samples are implemented. These procedures are documented in the "Quality Control Plan" of each test method.

Internal quality control schemes using statistics include:

- design of experimental/factorial analysis
- variation/regression analysis

- safety evaluation/risk analysis
- tests of significance
- quality control charts
- statistical sampling inspection

Proficiency testing helps to highlight not only repeatability and reproducibility performance between laboratories, but also systematic errors such as bias. It is important to monitor proficiency testing results as a means of checking quality assurance and take action as necessary.

The Quality Manager maintains a list of all the current proficiency testing programs the laboratory participates in, monitors the results, and notifies the appropriate personnel of both problematic and successful results.

Technical personnel use certified reference materials and reference materials to evaluate test performance on a daily basis and include daily process control checks. These data are used to evaluate the validity of the test results.

Replicate tests may be used if suitable reference material is available. These materials and proficiency test materials are available for improving repeatability.

Re-testing of test items is performed occasionally at the discretion of the supervisor or when test results seem anomalous.

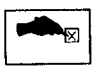
## Revision History

Revision 1



# 5.10 Reporting of Results

## Cross-references



ISO 17025:1999 Section 5.10

ISO 9001:2000 Section 7.1, 7.4.3, 7.5.1, 7.5.4, 8.2.4

## 5.10.1 General

**Policy:**
The results of each test, calibration, or series of tests or calibrations are reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test or calibration methods.

The results are reported, normally in a test report [or a calibration certificate] and include all the information requested by the client and necessary for the interpretation of the test [or calibration] results and all information required by the method used. This information may include what is outlined in section 5.10.2, 5.10.3 and 5.10.4.

In the case of tests or calibrations performed for internal clients, and in the case of a written agreement with the client, the results may be reported in a simplified way. The information listed in section 5.10.2 to 5.10.4, and not reported, is kept readily available.

**Details:**
Test reports [and/or calibration] reports are issued as either hard copy or by electronic data transfer.

## 5.10.2 Test reports and calibration certificates

**Policy:**
Test reports and/or calibration certificates include the following information, as appropriate:
- a title (e.g., "Test Report" [or Calibration Certificate])
- name and address of laboratory, and location where tests and/or calibrations were carried out if different from the address of the laboratory
- unique identification of the test report [or calibration certificate] (such as a serial number), and on each page an identification in order to ensure that the page is recognized as a part of the test report [or calibration certificate], and a clear identification of the end of the test report [or calibration certificate]
- name and address of the client

- identification of the method used
- description, condition, and unambiguous identification of the item(s) tested [or calibrated]
- date of receipt of test [or calibration] items (where this is critical to the validity and application of the results) and date(s) of performance of the test [or calibration]
- reference to sampling procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results
- test [or calibration] results with, where appropriate, units of measurement
- the name(s), function(s) and signature(s) or equivalent of person(s) authorizing the test report [or calibration certificate]
- where relevant, a statement to the effect that the results relate only to the items tested [or calibrated]

**Details:**
Signing authority for test reports is the responsibility of the <Click here and type the appropriate TITLE(S)>. Records for individuals with signing authority for test reports are approved by the <Click here and type appropriate TITLE> and maintained by the Quality Manager.

Hard copies of test reports [or calibration certificates] include the page number and total number of pages.

A statement is included specifying that the test report [or calibration certificate] is not to be reproduced except in full, without written approval of the laboratory. Data reported to the client contains the appropriate significant digits for each test method. Low level data are identified as being below specified limits.

## 5.10.3 Test Reports

### 5.10.3.1

**Policy and Details:**
In addition to the requirements listed in section 5.10.2, test reports include the following, where necessary for the interpretation of results:

- deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions
- where relevant, a statement of compliance/non-compliance with requirements and/or specifications
- where applicable, a statement on the estimated uncertainty of measurement of the test result; information on uncertainty is needed in test reports when its is relevant to the validity or application of the test results, when a client's instruction so requires, or when uncertainty affects compliance to a specification limit



- where appropriate and needed opinions and interpretations (see section 5.10.5)
- additional information required by specific methods, clients, or groups of clients

### 5.10.3.2

**Policy and Details:**
In addition to the requirements listed in sections 5.10.2 and 5.10.3.1, test reports containing the results of sampling include the following, where necessary for the interpretation of test results:

- date of sampling
- unambiguous identification of substance, matrix, material or product sampled (including name of manufacturer, model or type of designation and serial numbers as appropriate)
- location of sampling, including any diagrams, sketches or photographs
- reference to sampling plan and procedures used
- details of any environmental condition during sampling that may affect the interpretation of the test results
- any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned

## 5.10.4 Calibration Certificates

### 5.10.4.1

**Policy:**
The testing laboratory [generally] does not issue calibration certificates. However, the laboratory often receives calibration services from a calibration laboratory and needs to be familiar with the information on a calibration certificate.

**Details:**
In addition to the requirements listed in 5.10.2, the calibration certificate could include the following, where necessary for the interpretation of calibration results:

- the conditions (e.g., environmental) under which the calibrations were made that have an influence on the measurement results
- the uncertainty of measurement and/or a statement of compliance with an identified metrological specification or clauses thereof
- evidence that the measurements are traceable (see 5.6.2.1.1)

**5.10.4.2**

**Policy:**
This section is not applicable to a testing laboratory.

[For the calibration laboratory - The calibration certificate relates only to metrological quantities and the results of functional tests and specifically states which clauses of the specification are met or not met. When a statement of compliance with a specification is made omitting the measurement results and associated uncertainties, the laboratory records those results and maintains them for possible future reference. When statements of compliance are made, the uncertainty of measurement is accounted for.

**Note** - When an instrument for calibration has been adjusted or repaired, the calibration results before adjustment or repair, if available, are reported.]

**5.10.4.3**

**Policy:**
This section is not applicable to a testing laboratory.

[For the calibration laboratory - A calibration certificate (or calibration label) does not contain any recommendation on the re-calibration interval except where this has been agreed with the client. This requirement may be superseded by legal regulations.]

## 5.10.5 Opinions and Interpretations

**Policy:**
When opinions and interpretations are included in the test report, the basis upon which the opinions and interpretations have been made is documented. Opinions and interpretations are clearly marked as such in the test report.

**Note** - opinions and interpretations should not be mixed-up with inspections and product certifications as intended in ISO/IEC 17020 and ISO/IEC Guide 65.

**Details:**
Opinions and interpretations included in a test report may comprise, but not be limited to the following:

- opinion on conformity of the results with requirements
- fulfilment of contractual requirements
- recommendations on how to use the results
- guidance to be used for improvements



In many cases it is appropriate to communicate the opinions and interpretations by direct dialogue with the client. This dialogue is written down.

## 5.10.6 Testing and Calibration Results Obtained from Subcontractors

**Policy and Details:**
Test reports containing the results of tests performed by subcontractors are clearly identified for the subcontracted results. The subcontractor reports the results either in writing or electronically to our laboratory.

## 5.10.7 Electronic Transmission of Results

**Policy:**
In the case of transmission of test [or calibration] results by telephone, telex, facsimile or other electronic or electromagnetic means, the requirements of the policies and procedures of this Quality Manual continue to apply (see also 5.4.7).

**Details:**
Reports that are "published" electronically contain the statement that signatures are on file.

## 5.10.8 Format of Reports

**Policy:**
The format of reports is designed to accommodate each type of test [or calibration] carried out and to minimize the possibility of misunderstanding or misuse.

**Details:**
The layout of the test report [or calibration certificate] is such that the presentation of the test [or calibration] data facilitates ease of assimilation by the reader.

The headings are standardized as far as possible.

## 5.10.9 Amendments to Reports

**Policy:**
Material amendments to a test report [or calibration certificate] after issue are made only in the form of a further document, or data transfer, which includes the statement "Supplement to Test Report [or Calibration Certificate], serial number...[or as otherwise identified]", or an equivalent form of wording. Such amendments meet all the requirements in this Quality Manual.

**Details:**
When it is necessary to issue a complete new test report [or calibration certificate], it is uniquely identified and contains a reference to the original that it replaces.

## Revision History

Revision 1

# APPENDIX B. TRAINING DOCUMENTATION

# Grain Quality Lab Training

**Safety & Lab Operations Seminar**

Spring 2004
provided by: Audrey Hansen & Glen Rippke

## Topics

- Safety
- Lab protocol
- Semester project overview
- Questions
- Quiz
- Paperwork

## Introduction

- Charlie's comments on the activities of the lab techs, our quality management system, etc.

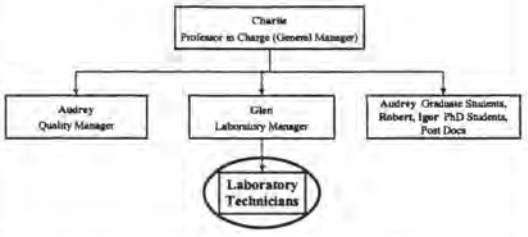
## Safety

- Terms Defined
- Site-Specific Training
  - Emergency procedures
  - Personal Protective Equipment
  - Chemical Hygiene Plan
  - Material Safety Data Sheets
  - Laboratory Procedures
  - Lab Safety Standard
- Further Resources

goggles
gloves
dust masks

## Lab Protocol

- Job description (review handout)




## Lab Protocol, cont.

- Hourly Time Clock (Tracy Time System)
- Food & Drink Policy
- Operator ID
- Saturday Work

## Project Overview

25,000 samples in 3 months

=

8,333 samples in 1 month

=

416 samples in 1 day

## Project Overview, cont.

- Soybean Calibration
- Corn Calibration
- Temperature Scans - on selected instruments
- Single Seed Analyzer
- Rosemary Study
- Soybean Variety Identification
- Various service projects as they come in

## Your Questions

## Quiz

- When can you have food or drinks in the lab?
  - Never
- What do you do if you forgot to clock in/out?
  - Talk to Glen, he will amend your record
- Where is the meeting place for a fire evacuation?
  - The new parking area by the green house
- Where can you get further training for things like fire extinguisher use?
  - ISU Environmental Health & Safety

## Quiz

- What does MSDS stand for?
  - Material Safety Data Sheet
- What are some examples of PPE in our lab?
  - Ear muffs, gloves, dust masks, safety goggles, coats
- Where is the meeting place when a tornado alarm sounds?
  - Down the hall, in basement
- What do you do if you forgot to clock in/out?
  - Talk to Glen, he will amend your record

## PAPERWORK

- Chemical Hygiene Signatures
- Lab Operations Signatures
- Personnel files
- Assign Operator IDs
- Confidentiality Agreement

# Grain Quality Lab Training

**Lab Procedures Seminar**

Spring 2004
provided by: Audrey Hansen & Glen Rippke

## Topics

- Instruments
- Lab Procedures
- Questions
- Demonstration
- Practice
- Evaluation & Paperwork

## Instruments

- General Categories
  - Near Infra-Red (NIR)
  - Wet Chemistry
  - Official Instrumentation
  - Physical Properties
  - Experimental / Prototype
- Purpose of each
  - (see handout)

## Lab Procedures

- Portable Zeltex with sample loading
- GrainSpec
- Foss Infratec 1229
- Foss Infratec 1241

## Lab Procedures

- Briuns OmegAnalyzerG flow
- Oven Moisture
- Dickey John GAC
- Seed Counter
- Test Weight

## Your Questions

Grain Quality LABORATORY

# Lab Procedure Training Record

Employee Name:_____ Start:_____

| *Procedure/Task* | *SOP Code* | *Revision #* | *Employee Read Procedure** | *Employee Observed Procedure*** | *Employee Performed Procedure*** | *Trainer Comments ** | *Training Approval*** |
|---|---|---|---|---|---|---|---|
| Foss Infratec 1229 | | | | | | | |
| Foss Infratec 1241 | | | | | | | |
| Bruins Omega Analyzer G flow | | | | | | | |
| Oven Moisture | | | | | | | |
| Dickey-John GAC | | | | | | | |
| Seed Counter | | | | | | | |
| Test Weight | | | | | | | |
| Zeltex 50 with sample loading | | | | | | | |
| GrainSpec | | | | | | | |
| | | | | | | | |

* Employee initials and dates column entries

** Trainer initials and dates or completes comments for column

\\129.186.30.108\Users\audrey\THESIS\Employee Confidentiality Agreement.doc 1/1
Original Version: 06-18-2003; By: AmH Last Revision: 7/14/04; By: AmH Next Scheduled Revision: XX-XX-XX
**ISO 17025 Reference Section(s): X.XX**

# Iowa State University Grain Quality Laboratory
# Employee Confidentiality Agreement

As an employee or affiliate of the Iowa State University Grain Quality Laboratory, I agree not to disclose to any person, any confidential information or documentation that comes to my knowledge or into my possession through my employment or affiliation, except with the prior written permission of the Professor in Charge (General Manager) of the Iowa State University Grain Quality Laboratory or as required by law. I shall treat all information related to clients in a confidential manner.

Specifically I will:

- Access data solely in order to perform my job responsibilities.
- Not seek personal benefit or permit others to benefit personally from any data that has come to me through my work assignments.
- Not make or permit unauthorized use of any information in the Iowa State University Grain Quality Laboratory's information system or records.
- Not enter, change, delete or add data to any information system or files outside the scope of my job responsibilities.
- Not include or cause to be included in any record or report, a false, inaccurate or misleading entry.
- Not alter or delete or cause to be altered or deleted from any record, report or information system, a true and correct entry.
- Not release Iowa State University Grain Quality Laboratory data other than what is required in completion of my job responsibilities.
- Not exhibit or divulge the contents of any record, file or information system to any person except as it is related to the completion of their job responsibilities.

Employee (print):_____

Employee's Signature:_____

Date:_____

Laboratory and Service Manager:_____Glen Rippke_____

Laboratory and Service Manager's Signature:_____

Date:_____

Grain Quality LABORATORY

# Employee Detail Sheet

## Local Contact Information

Name: _____ Initials: _____

Job Title _____

Paycheck ID: _____

GQL ID: _____

Address: _____
City: _____
State: _____
Zip: _____

Phone: _____

E-mail: _____

## Training Specifics

Please see training record for Lab Procedure training details

*Sessions Attended:*

| **Annual Safety** | **Annual Quality** | **Safety & Quality Refresher** |
|---|---|---|
| date: | date: | date: |

*Notes:*

# CHEMICAL HYGIENE TRAINING

Iowa State University

**DEPARTMENT:** Agriculture and Biosystems Engineering

**BUILDING:** Food Sciences

**ROOM NUMBERS:** 1513, 1515, 1544, 1546

**LABORATORY:** Grain Quality Research

**PROFESSOR IN CHARGE:** Dr. Charles Hurburgh

**SUPERVISOR:** Glen Rippke

The Laboratory Standard requires that the employee's supervisor provide training, which covers the specific topics described in the "Information and Training" section of the Chemical Hygiene Plan. This training must be provided at the time of the employee's initial assignment and on a refresher basis thereafter. The Site-Specific Training sheet is attached and has been provided to all employees. Specific employee training is documented below:

| **Description of Training** | |
|---|---|
| Lab Safety Standard content | **Dates sessions provided** |
| MSDS – location, usage | 2/23/04, 2/27/04 |
| SOP – location, usage | |
| Emergency procedures | **Provided by** |
| Personal Protective Equipment – location, usage | |
| Chemical Hygiene Plan – location, content | Audrey Hansen Graduate Research Assistant |
| General Procedures – cleaning, timesheets | |
| Safety Hints from A-Z | Glen Rippke Lab & Service Manager |

OSHA's Laboratory Standard (29CFR1910.1450) requires that each laboratory employee be made aware of the location and content of the laboratory's Chemical Hygiene Plan. By your signature below, you acknowledge that you have read and understood the contents of this plan and know its location within the laboratory.

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Address, Phone: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Address, Phone: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Address, Phone: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Address, Phone: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Address, Phone: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Address, Phone: _____

*** END RECORD ***** END RECORD ***** END RECORD ***** END RECORD ***** END RECORD ***

# LAB OPERATIONS TRAINING

Iowa State University

**DEPARTMENT:** Agriculture and Biosystems Engineering

**BUILDING:** Food Sciences

**ROOM NUMBERS:** 1513, 1515, 1544, 1546

**LABORATORY:** Grain Quality Research

**PROFESSOR IN CHARGE:** Dr. Charles Hurburgh

**SUPERVISOR:** Glen Rippke

The ISO/IEC 17025:1999 requires that the employee's supervisor provide training, which covers the specific topics described in the "Management Requirements – Organization" section (4.1) of the International Standard. This training must be provided at the time of the employee's initial assignment and on a refresher basis thereafter. The materials utilized to provide reference as to methods and procedures used is attached and has been provided to all employees. Specific employee training is documented below:

| **Description of Training** | **Dates training sessions provided** |
|---|---|
| Lab Protocol | |
| Job Description review | |
| Project overview for semester | **Provided by** |
| Instrumentation categories | |
| Instrumentation purpose and use | Audrey Hansen Graduate Research Assistant |
| Chemical Hygiene Plan Requirements (for specifics see Chemical Hygiene Plan Training) | |
| | Glen Rippke Lab & Service Manager |

By your signature below, you acknowledge that you have read and understood the contents of the materials provided and know the location within the laboratory of all resources needed to carry out your duties.

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Employee Name: _____ Classification: _____

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Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

Employee Name: _____ Classification: _____

Signature: _____ Date: _____

*** END RECORD ***** END RECORD ***** END RECORD ***** END RECORD ***** END RECORD ***

Grain Quality LABORATORY

# Terms Defined

**Chemical Hygiene Plan (CHP)** – documentation issued by EHS to ensure that Iowa State University meets the governmental requirements for safety

**Department of Environmental Health and Safety (EHS)** – a service unit of Iowa State University, devoted to ensuring the occupational health and safety of all ISU personnel, students and visitors. They are located in 118 Agronomy Lab Ames, IA 50011-3200 Phone: (515) 294-5359 Fax: (515) 294-9357

**Environmental Protection Agency (EPA)** – a federal government agency; mission is to protect human health and to safeguard the natural environment (air, water, and land); website located at http://www.epa.gov/

**Iowa Occupational Safety and Health Act (IOSHA)** – applies OSHA standards as adopted by Iowa Workforce Development, Division of Labor; investigates safety and health complaints in construction and general industry and performs general scheduled inspections

**Lab Procedures** – generalized version of SOPs; describes the task and its function; provides a quick reference when doing your job

**Lab Safety Standard** – required by EHS to be done departmentally; Grain Quality Laboratory's guidelines to ensure safety in the lab

**Material Safety Data Sheet (MSDS)** – required by OSHA and EPA; designed to provide both workers and emergency personnel with the proper procedures for handling or working with a particular substance; includes information such as physical data (melting point, boiling point, flash point etc.), toxicity, health effects, first aid, reactivity, storage, disposal, protective equipment, and spill/leak procedures

**Occupational Safety and Health Administration (OSHA)** – a federal government agency in the U.S. Department of Labor; primary goals are to save lives, prevent injuries and protect the health of America's workers; website located at http://www.osha.gov/

**Personal Protective Equipment (PPE)** – includes, but is not limited to, the following: safety goggles, aprons, gloves, protective clothing, ear muffs or plugs, respirators for both dusts and toxic materials and other similar items; the department is responsible for providing the necessary PPE. All PPE items shall meet or exceed the IOSHA standards and requirements

# Site-Specific Training for the Grain Quality Laboratory

**1513, 1515, 1544, and 1546 Food Science Building**

1. In case of emergency call 911. The closest readily available phone during office hours is in 1546 Food Science.
2. All injuries must be reported to the lab supervisor or the professor in charge.
3. Fire and tornado emergency plans are posted on the laboratory doors.
4. Food and drink for human consumption will not be allowed or consumed in the laboratory.
5. All backpacks and personal items must be placed in the cabinet provided in room 1546.
6. Smoking is not allowed in the building or the laboratory.
7. A First Aid Kit is located on the shelf in each laboratory room.
8. A fire extinguisher is located next to the exterior door of each laboratory room.
9. An eyewash station is located attached to the sink in 1513, 1544, and 1546 Food Science and freestanding in 1515 Food Science.
10. There are two-safety showers located on the outside of both labs next to the door.
11. The chemical hygiene plan (contains training and lab safety inspection documentation, as well as the chemical inventory), MSDS catalogs, SOP manual (also contains prior approval documentation for very hazardous chemicals), and chemical inventory sheets are present in 1546 Food Science.
12. Go to the lab and learn the locations of each of the items mentioned in Items 6-10. Read the Standard Operating Procedures (SOPs) for the lab and the prior approval sheets for hazardous chemicals.
13. When bringing chemicals into the lab, check the chemical inventory to see if they are on the list. If they are not, check to see whether there is an MSDS for that chemical. If not, one must be located immediately and added to the MSDS catalog. This must be done before any work with the chemical is done in the lab. Please contact Glen Rippke or Audrey Hansen for assistance.
14. **All** personnel who are working in the lab will be responsible for the cleanliness of the lab. The lab supervisor will evaluate the lab for cleanliness as needed and at the end of the semester.

## Resources

*Environment Health and Safety - ISU*

Gas Cylinder Guide:

http://www.ehs.iastate.edu/publications/manuals/gascylinder.pdf

Smoking Guidelines:

http://www.ehs.iastate.edu/publications/policies/smoking.pdf

Fire Safety Guide:

http://www.ehs.iastate.edu/publications/manuals/fireguide.pdf

Worker Right to Know:

http://www.ehs.iastate.edu/publications/manuals/wrtk.pdf

Hearing Conservation:

http://www.ehs.iastate.edu/publications/manuals/hearing.pdf

Respiratory Protection:

http://www.ehs.iastate.edu/publications/manuals/respirator.pdf

*Oxford University*

How to interpret MSDS sheets:

http://ptcl.chem.ox.ac.uk/MSDS/interpretingmsds.html

# Iowa State University Grain Quality Laboratory
## Job Description

**Title:** Laboratory Technician (Lab Tech)

**Background:**
The Grain Quality Laboratory's major objective is to reliably and effectively analyze samples obtained from clientele for research and production purposes. The Laboratory Technician is vital in achieving this objective through their hard work and attention to detail.

**Purpose:**
Provide labor for testing and analysis completed in the lab.

**Responsibilities:**
- work a reasonable number of hours per week
- perform duties effectively and in a timely manner
- maintain cleanliness in the lab environment
- attend regular training sessions (at least once per semester and as announced)

**Qualifications:**
- Iowa State University student – currently enrolled
- some experience with computers and Microsoft Excel software
- willingness to work time blocks of at least 2 hours
- ability to work well with others
- some capability to lift heavy boxes
- reliability to accomplish responsibilities listed above

**Relationships:**
Will interact with certain personnel including the lab manager, quality control technician, service-contract representatives, delivery staff, etc.

**Compensation:** beginning at $ 7.00 / hour

**Duration:** determined by the individual's interests

**Reporting and Evaluation:**
All laboratory work and testing will be coordinated by the Lab Manager (as of last update Glen Rippke). Reporting will be done directly to the Lab Manager or other individual who has been left in charge. Evaluation will be conducted through the Operator ID records and as needed to ensure effective output and working environment.

# APPENDIX C. LABORATORY PROCEDURE EXAMPLE

## Grain Quality Laboratory
## Iowa State University

## PROCEDURE: Writing Laboratory Procedures

**Background:**
There are many tasks that must be done in the Grain Quality Laboratory. Each of these tasks needs to be understood by the operator so that it may be done correctly. Lab Procedures are a way to standardize the tasks done in the lab, teach new employees, and also provide a reference for all those working in the laboratory.

**Objective:**
To write effective lab procedures that can be easily understood and executed.

**Procedure Detail:**
1. Familiarize yourself with the task.
2. Gather background information (from your knowledge or the lab manager).
3. Outline the major steps.
4. Fill in the specifics that are important to the completion of the task (although this is subjective, the procedures need to be kept within 2 pages if possible, so some very specific steps that are generally know by most operators may not need to be included).
5. Complete the Lab Procedure Template found on the 3.5 floppy disk given to you.
    a. Fill in all the information that you know (other items such as corrective actions can be filled in later by someone else as they are defined)
    b. Make sure to fill in the information in the header. This includes the filename and path and the original version date and author's initials.
6. Have another operator complete the task using the newly created lab procedure to verify that it is clear and concise.

**Evaluation and Monitoring:**
Completed lab procedures will be reviewed by at least the Laboratory Quality Representative after creation and then biannually in January and July. The biannual review will consist of the following.
1. Observation of personnel doing each process making notes of inconsistencies between what is actually happening and what the lab procedure says should happen.
2. Discussion with lab personnel and the lab manager to determine if the inconsistencies are valid or if the personnel need further training.
3. If the inconsistencies are valid, changes should be made to the lab procedure.

**Corrective Actions:**
Upon creation, if a completed lab procedure is found to be wrong, the error(s) will be reported back to the individual who wrote it to be corrected as soon as possible.

During the biannual review, error(s) will be corrected by the Laboratory Quality Representative or other appointed personnel. Additional training of personnel will be assigned as necessary.

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