Applying near infrared spectroscopy to the needs of US grain inspection

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Introduction

Near infrared (NIR) spectroscopy has a long history of use in determining grain constituents such as moisture, protein and oil. Grain related applications represent one of the most widespread uses of NIR spectroscopy. This includes the use of NIR spectroscopy in official grain inspection. The purpose of this paper is to discuss general objectives and procedures of the Official Inspection System and show how the use of NIR spectroscopy technology helps provide official inspection results that meet customer expectations. The paper also discusses newly developed programs to standardize the results of unofficial commercial grain inspection.

Offical inspection system

The Federal Grain Inspection Service (FGIS) of the Grain Inspection, Packers and Stockyards Administration manages the Official Inspection System and helps facilitate grain marketing by (i) developing and maintaining US grain standards and (ii) developing inspection and weighing procedures. In addition to FGIS, the Official Inspection System consists of designated agencies and delegated states whose licensed personnel officially inspect and weigh grain and serve as an independent third party in grain transactions. The Official Inspection System does not market grain, mandate quality, arbitrate disputes or set prices.

FGIS official inspection activities are authorized by the US Grain Standards Act, as amended. The Act requires that most export grain be inspected and weighed, prohibits deceptive practices and criminal acts with respect to the inspection and weighing of grain, provides penalties for violations and authorizes the charging of fees for official inspection. Official inspection is defined as determination and certification of the kind, class, quality or condition of grain under standards provided in the Grain Standards Act. The Grain Standards Act states that official certificates setting out the results of official inspection shall be received by all courts of the United States as *prima facie* evidence of the truth of the facts stated therein. Applicants for official inspection have the option of requesting, a reinspection that is conducted by the agency performing the original inspection, an appeal inspection that is conducted by the FGIS field office overseeing the agency that provided the original service and a board appeal inspection conducted by the Board of Appeals and Review. Results of the board appeal inspection are accepted as final and there are no further appeals.

Under provisions of the Grain Standards Act, official inspection and weighing is mandatory for most grain exported from the United States. FGIS and delegated states provide on-site inspection and weighing services at export grain elevators. In contrast, official inspections in domestic commerce are performed upon request and FGIS typically acts in a supervisory capacity for authorized state and private agencies to ensure that official inspection procedures are observed.

Table 1. Official grading factors and official criteria used in wheat inspection.

Offical grading factors	Official criteria
Test weight	Moisture content (required)
Defects	Protein
Damaged kernels	Falling number
Foreign material	TCK (smut)
Shrunken and broken kernels	Aflatoxin
Total defects	Dockage (required)
Wheat of other classes	

Inspection results are classified as either official factors (grade determining factors) or official criteria. Official factors and criteria for wheat are shown in Table 1. All official factors must be measured in order to assign an official grade. Those official criteria having broad market application, such as moisture content and dockage, are also required as part of all official inspections. Official criteria that apply to a narrower market segment or that may not be important to all users within a market segment, including NIR protein and oil determinations, are optional and not required even at export. If, however, measurement of official criteria is requested, an official inspection result must be provided. Wheat protein and soybean protein and oil are two of the most requested optional official criteria.

NIR spectroscopy in official inspection

Wheat protein and soybean protein and oil determinations are currently the only uses of NIR in the Official Inspection System. These programs have proven to be valuable in the marketing of wheat and soybeans. In 1994, there were 16,500 official inspections for soybean protein and oil. An average of nearly 500,000 official wheat protein determinations per year have been requested over the last ten years. The Official Inspection System certified results for over 680,000 wheat protein determinations in 1994. Official wheat protein determinations for six classes of wheat, Hard Red Winter wheat, Hard Red Spring wheat, Soft White wheat, Durum wheat, Soft Red Winter wheat and Hard White wheat are included in this total. Because wheat protein levels are affected by environmental conditions such as drought or frost, protein premiums and the demand for official inspection can vary significantly from year-to-year.

Customers of the Official Inspection System expect and demand a high level of accuracy and consistency of official protein and oil determinations because grain is typically bought and sold several times before finally being utilized. Unofficial commercial users often depend upon official inspection results to standardize their NIR equipment. Their customers in turn demand results that agree with official inspection results. The demand for accuracy and consistency is particularly high for wheat protein determinations because protein content affects dough characteristics and the suitability of wheat flour for use in making bread, pastries or pasta. If the protein content is too low, the product quality cannot be improved by simply adding more flour. Wheat must be purchased at a protein content suitable for the desired end use. In extreme instances, the protein premium for providing 15 percent protein wheat has approximately equaled the base price offered for wheat with a protein content of 12 percent.

To provide practical and acceptable inspection results, FGIS has developed detailed procedures and guidelines for the wheat protein and soybean protein and oil programs. Official protein and oil determinations require:

1. Use of official equipment. Currently the Infratec Models 1225 and 1226 are used for wheat protein and soybean protein and oil determinations.
2. Use of FGIS developed calibrations. FGIS develops official calibrations using a national grain sample set representing multiple crop years and diverse growing conditions and geographic regions. FGIS maintains reference methods and laboratories for use in calibration development and monitoring. A combustion nitrogen method is used to determine wheat and soybean reference protein contents and solvent oil extraction is used to determine reference oil content.
3. Adherence to approved operating procedures. FGIS publishes and distributes grain inspection handbooks that contain detailed operating procedures and program guidelines.
4. That inspections be performed by licensed personnel. Inspection personnel receive training and must pass qualification testing.
5. That individual instruments be standardized. Instrument standardization is used to assist in calibration transfer and assure consistency of results between official inspection laboratories.

While these activities all contribute to the accuracy and consistency of protein and oil determinations, standardization of individual instruments is the key to elevating system performance to the highest level.

FGIS uses grain samples to standardize individual official instruments to master instruments and to the reference method. Standard slope samples are analyzed on all instruments to compensate for differences in optics and other components. Reference values for slope samples are determined using the combustion nitrogen method. Slope adjusted instruments will agree closely with the reference method over the full protein (or oil) range. After slope adjustment of wheat protein instruments, for example, the difference in slope between instrument and reference results is typically well within the tolerance limits of 0.98 and 1.02. Slope adjustments are made when calibrations are updated or instruments are repaired. Instrument slope adjustments are generally reviewed annually.

A set of national standard reference samples (SRS) is used to ensure that official instruments are closely aligned with the master instruments and with each other. Protein baseline values are assigned to SRS samples by running them on master instruments that have been slope adjusted to agree with the reference method. SRS samples must be run each day for each class of wheat that will be analyzed before official instruments can be used to make protein determinations. Tolerances for daily wheat protein SRS checks are ±0.10 for the average of 10 analyses (duplicate analyses on a set of five SRS samples). The tolerance decreases to 0.07 for the average of 20 analyses and to 0.05 for the average of 30 analyses. Adherence to these relatively tight tolerances is reflected in correspondingly tight system performance.

FGIS monitors system performance and uses standard statistical quality control methods to verify that standardization procedures achieve the desired result. The performance of official instruments is regularly monitored and compared to that of the master instruments. Official inspection laboratories submit five samples per week per class of wheat to be analyzed on the master instruments. Figure 1 shows typical system performance for Hard Red Winter wheat for the time period April–July 1995. For the 3266 samples tested, the field instruments as a group were biased within 0.01 percent protein of the master instruments. The standard deviation of the difference between protein predictions of field and master instruments on individual samples was 0.11 percent protein. Standard quality control rules and limits are used to identify problem instruments. For example, an immediate check is initiated when the bias for a set of five samples from an individual laboratory exceeds 0.20 percent protein.

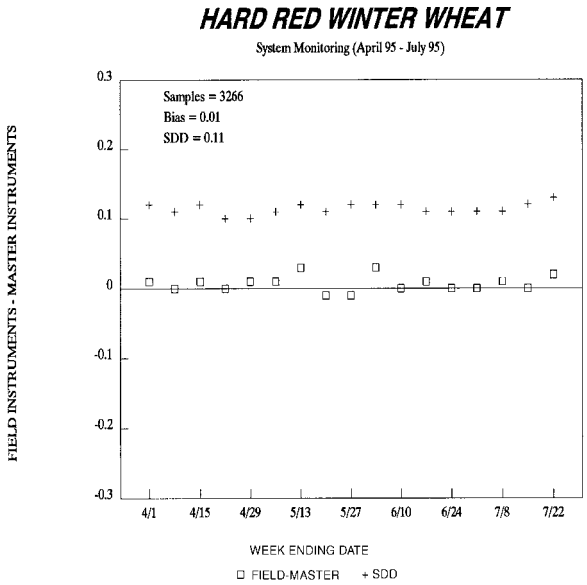

Figure 1. Performance comparison between official instruments (field) and FGIS master instruments on Hard Red Winter wheat.

Calibration accuracy is monitored by submitting selected samples for reference analysis and comparing the results to protein contents predicted using the master instruments. This aspect of system monitoring is important because the end user is primarily concerned with the accuracy of protein and oil results. Typical results for Hard Red Winter wheat are shown in Figure 2. Fewer samples are analyzed in monitoring calibration accuracy because of the time and cost associated with reference analyses. The results indicate a small overall bias between instrument and reference values of 0.13 percent protein and a standard deviation of differences of 0.22 for samples analyzed during the four-month period from April 1995 to July 1995. In addition to an on-going check of calibration accuracy, an annual review is conducted to discuss calibration concerns and establish priorities for calibration updates. FGIS standardization, monitoring and quality control programs represent a commitment of resources that may not be available to commercial users wishing to achieve similar levels of accuracy and consistency.

Standardizing commercial inspection

Because of the economic benefits of producing unofficial commercial inspection results that agree with those provided by official inspection, there has been on-going interest in seeing FGIS standardization efforts extended beyond official inspection. Provisions of the Grain Quality Incentives Act of 1990 authorized FGIS to work in conjunction with the National Institute of Standards and Technology (NIST) and National Conference on Weights and Measures (NCWM) to:

1. Identify inspection instruments requiring standardization;
2. Establish performance criteria for commercial grain inspection instruments;
3. Develop a national program to approve grain inspection instruments for commercial inspection; and

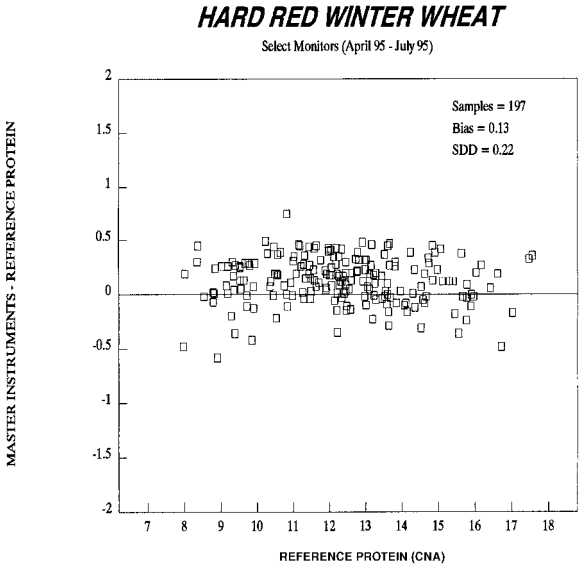

Figure 2. Typical calibration monitoring data comparing protein results for FGIS master instruments to combustion nitrogen reference values on Hard Red Winter wheat.

4. Develop standard reference materials or other means necessary for calibration or testing of approved instruments.

Representatives from FGIS, NIST and NCWM identified standardization of commercial grain moisture meters and near infrared wheat protein analyzers as program priorities. These areas were selected on the basis of economic significance, availability of design and performance criteria and the opportunity to coordinate efforts with existing inspection and regulatory programs.

The grain moisture meter program has progressed at a faster pace than the wheat protein program. Design criteria and performance specifications, developed by a technical committee, have been adopted by the NCWM. FGIS has been authorized as a National Type Evaluation Program (NTEP) Laboratory for evaluating grain moisture meters. Type evaluation testing has been completed for five instrument models, two of which are near infrared transmittance instruments. FGIS is continuing to conduct NTEP evaluations and has begun collecting calibration data on NTEP meters for the national sample set used to calibrate the official grain moisture meter. Funding has been secured to support the first five years of the calibration program.

A similar NTEP program is scheduled to begin later this year for wheat protein analyzers. One difference from the moisture meter program is that the FGIS NTEP laboratory will work with manufacturers to help standardize instruments and calibrations to official samples and reference methods before conducting type evaluation testing. Details of an on-going calibration monitoring program are still being discussed by the technical committee.

Implementation of these state regulated programs has implications for both manufacturers of NIR spectroscopic equipment and for the Official Inspection System. As currently envisioned, calibrations on NTEP instruments will be reviewed and updated on an annual basis and at a specified time. Scheduled calibration updates, intended to promote system uniformity, may present an interesting dilemma when trying to address customer concerns. Program participants

may no longer be able to act independently when making calibration or instrument standardization changes.

Summary

Official inspection and certification of wheat protein and soybean protein and oil contents clearly facilitate the market of wheat and soybeans, as evidenced by the number of requests for official determinations and by high customer expectations of the accuracy and consistency of results. Official NIR spectroscopy inspection results provide a much needed reference standard for the competitive free market system in the United States. Cooperative efforts to standardize commercial inspection will have an impact on the Official Inspection System, state regulatory authorities and NIR instrument manufacturers. Implementation of NTEP grain moisture meter and wheat protein analyzer programs should increase the demands for nationwide accuracy and consistency of NIR moisture and protein determinations. To meet NTEP program demands, manufacturers are already providing audit trails to log changes to metrological adjustments such as slope, bias or calibration constants. Additional effort is being directed toward minimizing sample temperature effects. These, and future efforts, will help ensure the continued importance of NIR spectroscopy technology in grain marketing.