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Near-Infrared Transmittance Handbook (NIRT)

Program Handbook

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Near-Infrared Transmittance Handbook

Foreword

The Near-Infrared Transmittance (NIRT) Handbook covers procedures for making official protein, wet gluten, oil, and starch determinations using the Foss-Infratec 1225, 1226, 1227, 1229, 1241, and NOVA NIRT (whole grain) analyzers. All official inspection personnel authorized or licensed to perform NIRT testing shall reference this handbook for procedures.

This handbook has been updated to incorporate FGIS's agency move to AMS, contact information, and the inclusion of information for the Foss NIRT model NOVA, as well as general revisions and formatting. In addition, definitions, tables, and processes were updated to the current standards. The specific updates are listed in chapter 6 of this handbook.

This handbook supersedes the NIRT Handbook, dated 12-18-06.

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CHAPTER 1 INTRODUCTION

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1.1 PURPOSE

This handbook establishes procedures for determining and certifying official protein and wet gluten content in wheat; official protein content in barley; official protein and oil content in soybeans; official protein, oil, and starch content in corn; monitoring the accuracy of official results; and maintaining near-infrared transmittance (NIRT) equipment accuracy.

1.2 SCOPE

Testing wheat, barley, soybean, and corn constituents as "official criteria" is authorizedunder Section 7(b) of the United States Grain Standards Act (USGSA), as amended.

Sections 800.125 and 800.135 of the USGSA permit a review inspection on either official grade/factors or official criteria. When requested, a review inspection for official grade or official factors and official criteria may be handled separately even though both sets of results are reported on the same certificate.

All official analysis as shown in Table 1-1 under the USGSA shall be performed in accordance with procedures prescribed in this handbook and the instrument manual. Testing must be performed only by authorized or licensed employees of the Federal Grain Inspection Service (FGIS) or delegated/designated agencies.

Grain	Wet Gluten	Protein	Oil	Starch
Wheat	✓	✓		
Barley		✓		
Soybean		✓	✓	
Corn		✓	✓	✓

TABLE 1.1 - TEST CONSTITUENTS BY GRAIN

1.3 **DEFINITIONS**

- a. **Application Model (AM)** Calibration software as developed in collaboration with Foss instruments. A collection of prediction models bundled together to predict multiple constituents at the same time (e.g., protein and oil).
- b. **ANN** Artificial Neural Network. The ANN is used to determine the calibration curve for some grains.
- c. **Baseline** Known protein, oil, and starch values determined by Technology and Science Division (TSD) for the Standard Reference Sample (SRS) sets.
- d. **Bias** The average difference between NIRT instrument results and the baseline values assigned by TSD to the Standard Reference Samples. To correct for the bias, an instrument adjustment of the calibration curve intercept constant is made. This is also sometimes referred to as "performing a bias" (e.g., bias correction).
- e. **Calibration Constants** The instrument requires a calibration containing the necessary calibration constants. The NIRT instrument uses these numerical values to calculate the constituent results.
- f. **Calibration Disk** A diskette or USB flash drive from which the NIRT calibration constants are loaded into the NIRT instrument. Models 1229 and older without IRIS use RMX format; all other models use DOS format.

- h. **Coarse Foreign Material** Foreign material found in soybean samples that consists of the following:
 - (1) Whole kernels of corn. Whole kernels of corn are kernels with one-fourth or less of the kernel removed.
 - (2) Cockleburs.
 - (3) Sticks meeting the following criteria:
 - (a) Approximately 1 inch or more in length.
 - (b) Approximately 1/2 inch or more with a thickness of 5/32 of an inch (width of the largest soybean slotted sieve).
 - (4) Pods (one-half pod or more). If pods contain soybeans, remove the soybeans and return to sample.
 - (5) Other coarse foreign material may include, but is not limited to, corn cobs, large feed pellets, pieces of dirt larger than soybeans, sweet corn, and edible beans that are generally larger than soybeans.
- i. **Collaborative Study** A study designed to compare constituent values determined by different laboratories.
- j. **Combustion Method** A chemical analysis used to determine the percent nitrogen in a sample using the AOAC International Method 992.23.
- k. **Constituent** Compounds for which an analysis is made in a product (i.e., protein and wet gluten in wheat; protein in barley; protein and oil in soybeans; protein, oil, and starch in corn).
- I. **Correlation** The interdependency of one variable on another (i.e., solvent oil extraction and NIRT oil).
- m. **Equipment Capability Testing (ECT)** The FGISOnline program in which standard reference sample daily bias data is inputted by instrument.
- n. **FO** Field Office is an office of the Service designated to perform or supervise official inspection services and Class X and Class Y weighing services.
- o. **Instrument Constants** Several constants (some of which are different for each instrument) which must be present in the instrument prior to using the instrument for official determinations.
 - The instrument constants that must be present in each unit include "O" and "P" constants (which characterize the instrument's optical components), and the slope and intercept constants that adjust each instrument for optimum agreement with the standard reference method. Only the intercept constant may be altered by the operator. Only the Technology and Science Division (TSD) can authorize change to the instrument's slope.
- p. **Moisture Basis** The use of Standard Moisture Basis (official basis) or Alternate Moisture Basis (i.e., client designated, As-Is, Dry Matter) to calculate the constituent value.
- q. **Monitor Samples** Samples selected by a specified process from the officially inspected samples analyzed during a specific date range. The official results on these samples are compared to the results obtained by a monitoring office to detect developing inaccuracies.
- r. **Near-Infrared Transmittance (NIRT) Determination** A spectrophotometric determination of a sample's constituents by measuring the amount of light transmitted through a sample at specific wavelengths in the near-infrared region of the spectrum.
- s. **OA** Official Agency is any State or local government agency, or any person, designated by the Administrator pursuant to subsection (f) of section 7 of the Act for the conduct of official inspection (other than appeal inspection), or subsection (c) of section 7A of the Act for the conduct of Class X or Class Y weighing (other than review of weighing).
- t. **Oil** Lipids (oils and fats) that are liquid at room temperature.
- u. **Pathlength** The distance between the two glass windows on opposite sides of the grain stream.

- v. **Prediction Model (PM)** A term used for the file containing the calibration constants, native calibration moisture basis, default or displayed moisture basis, universal slope, and universal intercept settings for each unique grain and constituent combination. Several PMs may be bundled together into an AM that the user selects prior to testing a sample.
- w. **PN** Part Number is an identifier of a particular part design or material used in a particular industry.
- x. **Polarimetry** A technique that measures the rotation of the plane of polarized light or the degree of polarized light passing through an optical sample to determine the starch content using the Corn Refiners Association Method A-20.
- y. **Protein** A naturally occurring complex combination of amino acids joined by peptide bonds that contain the elements carbon, hydrogen, nitrogen, oxygen, sulphur, and, to a lesser degree, other elements.
- z. **Solvent Extraction** A process that uses a non-polar solvent such as petroleum ether to extract the oil from a sample using the FGIS solvent oil extraction method.
- aa. **Specified Service Point (SSP)** A city, town, or other location specified by an agency for the performance of official inspection or Class X or Class Y weighing services and within which the agency or one or more of its inspectors or weighers is located.
- bb. **Standard Reference Method** A standardized chemical method used to determine a constituent value for a sample. FGIS standard reference methods are used to develop the calibration constants used with NIRT instruments for wheat, barley, soybeans, and corn.
- cc. **Standard Reference Samples (SRS)** A set of samples with established protein values for wheat, protein values for barley, protein and oil values for soybeans, and protein, oil, and starch values for corn which are used to maintain NIRT instrument accuracy.
- dd. **Standard Slope Settings** Average slope value used by all instruments within the official system. It is based on the individual slope values for instruments maintained by the TSD and covers grain from multiple crop years.
- ee. **Starch** Any of a group of polysaccharides composed of long-chain polymer of glucose in the form of amylose and amylopectin. It is the chief storage form of energy reserve (carbohydrates) in plants.
- ff. **Wet Gluten** Wet gluten is a protein *(or proteinaceous)* substance that remains after starch is washed out of dough. Gluten gives dough it's viscoelastic properties. The NIRT-wet gluten test is currently offered for Hard Red Winter and Hard Red Spring wheat only.

1.4 RESPONSIBILITIES

The general responsibilities for the wheat protein and wet gluten; barley protein; soybean protein and oil; and corn protein, oil, and starch testing programs are listed below.

- a. Responsibilities of the Technology and Science Division (TSD), NIRT Program.
 - (1) Maintain the FGIS National Standard NIRT instruments.
 - (2) Develop, evaluate, maintain, and implement the calibrations for official NIRT instruments. This includes developing the standard slope settings associated with an updated calibration and issuing the standard slope settings used by official NIRT instruments.
 - (3) Establish the official SRS protein contents for wheat and barley; protein and oil contents for soybeans; and protein, oil, and starch contents for corn.
 - (4) Develop and distribute SRS sets to FGIS field offices and official agencies.
 - (5) Prepare and distribute check samples (annual for corn) to monitor the accuracy of official testing locations providing corn protein, oil, and starch testing service.
 - (6) When necessary, review NIRT procedures at FGIS field offices and official agencies.
 - (7) Monitor the accuracy of official results at testing locations.
 - (8) Initiate, conduct, and report collaborative and special studies as needed.

- (9) Provide technical support and training to official inspection personnel in matters relating to NIRT determinations, maintenance, service, and repair.
- (10) Provide or coordinate maintenance, service, and repair of federally owned instruments.
- (11) Maintain a master list of all NIRT instruments, along with their approved calibration information, in the official system.
- (12) Upon request, forward a list of all instruments and their approved calibration information to the appropriate Field Office or Official Agency.
- b. Responsibilities of TSD, Board of Appeals and Review (BAR).
 - (1) Coordinate requests for Board appeal inspection services.
 - (2) As applicable, issue certificates and assess fees for Board appeal inspection services.
- c. Responsibilities of TSD, Biotechnology and Analytical Services (BASB).
 - (1) Maintain the FGIS standard reference laboratories.
 - (2) Establish the official constituent values for calibration samples.
- d. Responsibilities of FGIS Field Office Managers (FOM).
 - (1) Select an NIRT coordinator to serve as the primary contact within the respective circuit and to TSD.
 - (2) Perform original, reinspection, and appeal inspection services as applicable.
 - (3) Retrieve and distribute the wheat, barley, and soybean control charts and collaborate with TSD in monitoring specified service points within the circuit.
 - (4) Routinely review NIRT procedures in the respective area of responsibility.
 - (5) When necessary, inform TSD of problems detected in respective areas of responsibility and initiate appropriate corrective action.
 - (6) Provide technical support and training to official inspection personnel.
 - (7) Assist TSD in conducting collaborative and/or special studies.
 - (8) Assure that official agencies select and forward wheat, barley, and soybean monitoring samples to the appropriate field office and/or TSD.
 - (9) Ship file samples for Board appeal inspection services to the BAR. Write the words "Protein Board Appeal" in the "Remarks" section of the grain sample ticket and on the package.
 - (10) On a case-by-case basis and with the approval of the Director, Field Management Division, provide original testing service in areas where agencies are unable to arrange for adequate services (see Section 1.4.e(2)).
 - (11) Ensure the upkeep of calibrations according to Chapter 2 of this handbook.

e. Responsibilities of Agency Managers.

- (1) Coordinate and maintain a wheat protein and wet gluten; and/or barley protein; and/or soybean protein and oil; and/or corn protein, oil, and starch testing program in assigned geographic areas.
- (2) Perform original and reinspection NIRT inspection services and forward file samples for appeal to the FGIS field office or TSD. In some instances, the demand for the testing service may not warrant the purchase of NIRT equipment by an agency. If this occurs, the agency must survey industry representatives, determine the need for service, notify FGIS of their findings, and locate a mutually agreeable agency to provide the service. If an agency cannot arrange for adequate service, FGIS will determine on a case-by-case basis whether FGIS will provide original testing service.
- (3) Select and forward wheat, barley, and soybean monitoring samples to the appropriate FGIS field office and/or TSD.

- (4) Routinely review the NIRT operators' procedures at testing sites within the assigned geographic area.
- (5) Provide technical support and training to NIRT technicians.
- (6) Assist TSD/FGIS field offices in conducting collaborative and special studies.
- (7) Notify the monitoring FGIS field office concerning NIRT related problems and initiate follow-up action.
- (8) Maintain complete work records (log book).
- (9) Ensure office personnel enter complete and up-to-date daily instrument testing results into the Equipment Capability Testing (ECT) program.
- (10) Ensure the upkeep of calibrations according to Chapter 2 of this handbook.

1.5 DISCLAIMER CLAUSE

The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned.

CHAPTER 2 NIRT EQUIPMENT

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2.1 OFFICIAL EQUIPMENT

Approved equipment recognized for use in the NIRT program are as listed in table 2.1, by grain and constituent.

Note: All instrument models shown in the table are approved for official soybean and corn constituent determinations as shown in table 2.1. Only models NOVA, 1241, or other instruments equipped with IRIS operating software, are approved for official wheat and barley constituent determinations.

TABLE 2.1 – APPROVED INSTRUMENT MODELS

Grain	Constituent	Models 1225, 1226, 1227, and 1229	Models 1225, 1226, 1227, and 1229 <i>With IRIS Softwar</i> e	Model 1241	NOVA
Wheat	Protein	no	yes	yes	yes
(ANN ¹)	Wet gluten	no	yes	yes	yes
Barley (ANN¹)	Protein	no	yes	yes	yes
Soybeans	Protein	yes	yes	yes	yes
(ANN¹)	Oil	yes	yes	yes	yes
_	Protein	yes	yes	yes	yes
Corn (PLS²)	Oil	yes	yes	yes	yes
, ,	Starch	yes	yes	yes	yes

¹ Artificial Neural Network calibration

2.2 CALIBRATION SOFTWARE

- a. All approved models require that official calibrations be loaded into the instrument prior to use. Calibrations are loaded via disk or USB, and a separate disk or USB is required for <u>each</u> NIRT instrument. TSD has either developed application models or worked with Foss, to prepare official calibration disks or USB drives containing all application models needed.
- b. Information regarding the acceptable calibration software is summarized in Table 2-2. All calibrations are available from FOSS using the FOSS Part Number(s) shown in the table. Evaluation of the calibration for instrument accuracy is discussed in Chapter 3.

² Partial Least Squares calibration

TABLE 2.2 – APPROVED CALIBRATION SOFTWARE

Grain	Instrument Model(s)	Constituent	FOSS Part No.	Application Model
Wheat	1241 & older ¹	Protein & Wet Gluten	10014260 -or- "Small Grain F"	WH050101 ²
	NOVA	(HRW/HRS)	Per instrument SN	WBPR0028-0
Barley	1241 & older ¹	Protein	10014260 -or- "Small Grain F"	BA050101
	NOVA		Per instrument SN	BAPR0025-0
	1229 & older ³	Protein &	1229-60044633	00400450
Soybean	1241	Oil	1229-60044632	SO100152
Ooybean	NOVA	Protein	Per instrument SN	SOPR0016-0
	NOVA	Oil	T et instrument on	SOO10213-0
	1241 & older ¹	Protein, Oil, & Starch	FGIS NIRT Calibration Version: Sept. 1, 2001	CN081401
Corn		Protein		COPR0801-0
	NOVA	Oil	Per instrument SN	COO10801-0
		Starch		COST0801-0

¹ With IRIS software.

- c. FGIS FOMs and official agency managers shall verify the following:
 - (1) The calibration name is identical to that currently specified by TSD.
 - (2) The calibration disk is the current version approved by TSD.
 - (3) Slope values agree with TSD records.
 - (4) Slope values are not changed without the approval of TSD.
 - (5) NIRT instruments are configured by FGIS calibration to give readings corrected to the appropriate moisture basis listed in Table 2.3.

TABLE 2.3 – DISPLAYED MOISTURE BASIS

Constituent	Moisture Basis
Wheat protein	12.0 percent
Wheat wet gluten	14.0 percent
Barley protein	dry matter
Soybean protein and oil	13.0 percent
Corn protein, oil, and starch	dry matter

d. Operators shall use the standard slope settings, Standard Reference Sample (SRS) sets, and baseline values provided by TSD.

2.3 NIRT LOCATION AND ENVIRONMENT

² Protein only; is an all-wheat class model.

³ With IRIS software version 2.6 or higher.

Equipment location and environmental factors can affect the performance of NIRT equipment.

- a. <u>Location of Equipment</u>. NIRT instruments must be placed in a location conducive to a dust-free and stable environment. If the NIRT instrument is not located in its own room, all dust-emitting devices located in the same room must be operated with a functional dust collection system. The NIRT instruments must be protected from drafts, heating and cooling vents, and windows. Also, a vibration-free table is recommended to support the NIRT instrument.
- b. <u>Environmental Requirements</u>. The space and facilities required to perform official NIRT determinations must meet the specifications outlined below:
 - (1) Temperature affects the stability of NIRT instruments. Each testing site shall install a thermometer near the NIRT instrument(s). The temperature of the room where official testing occurs must be maintained between 60° and 80°F (16° and 27°C). Official testing shall be suspended if the room temperature is outside the acceptable range. Once the temperature is restored to the acceptable range, check instrument accuracy using the SRS set and, if necessary, bias adjust the instrument.
 - If the room temperature changes by ±5°F (2.5°C) or more from the temperature recorded during the daily instrument check, retest the SRS and, if necessary, bias the instrument.
 - (2) Relative Humidity (RH) must be kept between 20 and 75 percent. Each testing site shall install a hygrometer (calibrated to ± 3 percent RH) near the NIRT instrument(s). When the laboratory's RH is outside of the acceptable range, retest the SRS and, if necessary, bias adjust the instrument based on LEVEL-I tolerances. Once the laboratory's RH returns to the acceptable range, the SRS need to be retested only if a bias adjustment was made while the RH was outside the acceptable range. If necessary, bias adjust the instrument based on the LEVEL-I tolerances. SRS sets collected when the RH is outside of the acceptable range may not be used for the LEVEL-II and higher tolerances. All LEVEL tolerances are listed in section 3.2 for wheat, section 3.3 for barley, section 3.4 for soybeans, and section 3.5 for corn.
 - (3) The power for all NIRT instruments shall be supplied by a 120 ± 10 VAC/15-20-amp dedicated circuit. A maximum of two electronic instruments (i.e., NIRT, NMR or Hardness Tester) plus their associated printers and/or computers may be placed on one dedicated circuit. No other equipment shall be used on the circuit. The Tripp-lite line protector supplied with the Infratec 1241 should be used when a dedicated circuit is available.

If a dedicated circuit cannot be provided, a standby uninterruptable power supply (UPS) is an acceptable alternative. The UPS should be rated as shown.

Capacity: at least 300VA

Runtime: at least 5 minutes at full load

Switching time: less than 4 milliseconds

Note: FGIS strongly recommends using a UPS instead of the Tripp-lite. If an instrument is relocated and unknowingly connected to an undedicated circuit, the use of the UPS will still protect the instrument. Whereas, if the Tripp-lite is used and the instrument is moved from a dedicated to and undedicated circuit, the instrument will not be protected against any surges in the power.

Note: If other equipment (e.g., external computer) is used on the UPS, the capacity should be upgraded accordingly.

The UPS should incorporate surge suppression and filtering. The Tripp-lite protector supplied with the Infratec 1241 should not be used in conjunction with a UPS.

The Foss NOVA model contains an internal surge protector and should be used in conjunction with a UPS.

A power line conditioner is recommended for use if line voltage variation is a suspected problem. Before purchasing and installing a voltage regulation device, contact the instrument manufacturer

to determine which device is best suited for this purpose.

An NIRT instrument may be turned off if it will not be used for at least 8 hours. After turning the instrument on, it must be allowed to warm up at least 15 minutes before testing. Outliers in the "A" or "B" position of the outlier code may be indicated because of insufficient warm up.

(4) Smoke and Dust. Post "**NO SMOKING**" signs in the testing area. Follow good housekeeping practices to maintain a clean and dust-free environment.

Use a vacuum cleaner or brush for proper laboratory cleanup. Do not use compressed air for cleanup purposes.

2.4 SETUP

Official testing agencies and FGIS field offices must observe certain guidelines when establishing new laboratories, placing new equipment on-line, or relocating NIRT equipment.

a. <u>Laboratory Setup</u>.

- (1) Official agency managers must notify the appropriate FGIS field office manager concerning plans for a new laboratory and provide a diagram of the proposed design. The diagram should contain the proposed locations of NIRT equipment, location of major inspection equipment, and description of the power supply. Any additional information regarding the laboratory setup or equipment should also be included.
- (2) Upon request, TSD will assist official agencies in planning and preparing laboratories for official NIRT testing. The field office manager should forward a copy of all submitted information to TSD for review. Upon receipt, TSD will advise the official agency and field office manager.

b. Equipment Setup.

- (1) Official personnel shall notify TSD and the appropriate field office when new NIRT instruments are purchased. TSD will provide the necessary samples and instructions to check the accuracy of the instrument(s). Contact TSD as soon as possible because the checkout process may take several days to complete.
- (2) When an NIRT instrument is moved to a new location, the instrument must be allowed to reach temperature equilibrium with its environment before performing official tests. Generally, the instrument should sit for at least 2 hours before use after being moved. If the instrument might have been subjected to extreme temperatures during shipment, allow the unit to sit overnight in the new location before operating it.

2.5 EQUIPMENT MAINTENANCE

a. <u>General</u>.

- (1) Using a brush or cloth, dust out the sample hopper and path at the end of each day.
- (2) Replacement lamps for the instrument are expensive and, therefore, the lamp life should be extended as long as possible. Turning the lamp on and off frequently decreases its life. Turn the instrument off only if it will not be used for a period of 8 hours or more.

b. Repair of FGIS-owned NIRT Equipment.

- (1) Repair and service of FGIS-owned instruments are the responsibility of the respective Field Office.
- (2) TSD personnel are assigned to assist field office personnel in:
 - (a) Maintaining instruments,
 - (b) Performing diagnostic tests needed to verify acceptable performance, and
 - (c) Performing modular replacement when required.
- (3) Repair Procedures.

- (a) If an NIRT instrument malfunctions, the designated field office NIRT coordinator should contact TSD at (816) 702-3886 to report the problem.
- (b) The NIRT coordinator should be prepared to answer all questions regarding the symptoms of the failure (error codes, erroneous readings, malfunctioning display, etc.) and to perform diagnostic tests while maintaining telephone communications with TSD.
- (c) TSD will take one of the following actions:
 - 1 If the NIRT instrument is determined to be field-repairable, TSD will coordinate the shipment of replacement parts (boards, etc.) to the field office.
 - If it is not field-repairable, TSD will recommend return of the instrument to Foss. The field office will be responsible for shipping and repair costs. If possible, a replacement instrument will be furnished to the field office by TSD. A written summary of the malfunction should be sent to TSD.

c. Repair of Other NIRT Equipment.

Official Inspection Agency managers must ensure that only qualified technicians perform repairs on NIRT instruments used for official testing. Operators must notify the field office NIRT coordinator and TSD when instruments malfunction.

d. NIRT Lamp Replacement.

- (1) Replacement lamps for Infratec models 1241 and NOVA must be purchased directly from the instrument manufacturer. Refer to the instrument manual or the back of the instrument for the proper Foss part number.
- (2) Replacement lamps for older Infratec models 1225/1226/1229 are no longer supported by FOSS.
- (3) If the replacement lamp does not work upon receipt, or if it does work and the Infratec instrument displays an error code denoting no light is reaching the detector, contact the instrument manufacturer immediately.
- e. <u>Equipment Maintenance Log</u>. Record any information pertaining to instrument repairs (e.g., lamp replacement) and other relevant information concerning unusual instrument operation. Information entered in the log is used as a troubleshooting aid for repair personnel and provides the agency with a maintenance history of the instrument.

CHAPTER 3 DETERMINING INSTRUMENT ACCURACY

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3.1 STANDARD REFERENCE SAMPLES

a. General.

- (1) Standard Reference Samples (SRS) consist of bulk quantities of wheat, barley, soybeans, and corn with established protein, oil, and starch values (as applicable) determined by TSD.
- (2) SRS baseline values are corrected to a 12 percent moisture basis (mb) for wheat, dry matter basis for barley, 13 percent mb for soybeans, and a dry matter basis for corn before being released with the SRS sets.
- (3) SRS sets are used to test instrument accuracy and to determine any needed bias adjustments.
- (4) Contact your NIRT coordinator or TSD to request SRS sets. TSD will supply SRS that weigh between 675 and 725 grams for wheat or barley, and between 850 and 900 grams for soybeans and corn. Refer to Table 3.1 for information on SRS sizes and number of bags.

Grain	Amount per Bag (grams)	Number of Bags
Wheat	675 -725	6
Barley	675 -725	5
Soybean	850 – 900	5
Corn	850 – 900	4

TABLE 3.1 - SRS SAMPLE AMOUNTS

- b. <u>Number of Samples</u>. TSD will select a set of six (6) samples for wheat, five (5) samples for barley, five (5) samples for soybeans, and four (4) samples for corn. Perform a duplicate determination on each wheat, barley and corn SRS and a single determination on each soybean SRS.
- c. <u>Developing Baseline Values</u>. The constituent baseline values are determined by TSD on the standard NIRT instrument. TSD instruments are directly compared and adjusted to optimally agree with the standard reference method.
- d. Replacing Reference Samples. TSD will maintain bulk reference samples to replace the field supply as they become infested, contaminated, or depleted. Testing locations must replace the wheat SRS set after 6 months. The 6-month period starts on the day the SRS are first used. It should be within 2 weeks of receiving the SRS from TSD; if not, the SRS should be refrigerated until they are ready for use. Barley, soybean, and corn SRS sets will be replaced on a yearly basis unless they become infested, contaminated, or depleted. Request a replacement supply from TSD as far in advance as possible (30 days recommended).

Immediately notify your NIRT coordinator or TSD if any SRS becomes contaminated or infested and obtain replacement(s).

In case of emergencies, SRS can be shipped overnight during the week. Otherwise, the SRS will be shipped through United Parcel Service (UPS) and should arrive within 5 business days of placing an order for replacement samples.

- (1) <u>Collecting and Testing Replacement Samples</u>. TSD will collect, evaluate, and select SRS. TSD will evaluate and select slope sets when calibrations are updated. TSD may request selected testing locations to provide samples for this purpose.
- (2) Replacing SRS or Baseline Value(s). Use this procedure when SRS or baseline values are replaced or when the calibration is updated. Test the new SRS two times. Use the Level II tolerances to determine if a bias adjustment is needed. If baseline values are replaced or calibration is updated, contact TSD with your final intercept values for the updated and/or adjusted calibrations.
- (3) Storage of Standard Reference Samples.
 - (a) Store reference samples at room temperature in plastic containers with screw tops or securely fastened lids and place away from vents, heating devices, and direct sunlight.

- To prevent insect infestation, operators may place mothballs sealed in plastic bags (with small holes) with the reference samples. The mothballs must remain sealed in the plastic bags at all times.
- Operators must remove any mothballs before testing the SRS set.
- (b) SRS may be stored under refrigeration. However, the SRS must be allowed to reach room temperature (± 5°F) before testing.
- (c) Discard any SRS if it becomes infested or contaminated. Immediately inform your NIRT coordinator or TSD and obtain replacement sample(s).
- (d) Protect reference samples from manipulation by unauthorized persons to maintain sample integrity.
- e. <u>Using Standard Reference Samples</u>. Before testing market samples, the operator must first test, evaluate, and, if necessary, bias adjust the instrument using the appropriate SRS set and baseline values for wheat, barley, soybeans, or corn.

Review the following information before performing the required SRS testing as required under Section 3.2 through 3.5.

- (1) <u>Wheat Data Evaluation</u>. Evaluate the SRS values by comparing individual and average NIRT protein to the corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein testing service:
 - (a) <u>Duplicate Difference</u>. If the difference between the first and second analysis of an individual SRS exceeds ± 0.20 percent protein, then reanalyze the sample. Record the results from the two analyses closest to each other and discard the third result.

The duplicate difference is calculated as follows:

- Duplicate Difference = an individual sample NIRT value first analysis minus (-) individual sample NIRT value second analysis.
- (b) <u>Individual Difference</u>. An individual reference sample NIRT protein value differs from its baseline value by more than ± 0.40 percent protein.

The individual difference is calculated as follows:

Individual Difference = an individual sample NIRT protein value minus (-) sample baseline value.

- (c) <u>Average Differences</u>. The average difference between the SRS NIRT protein values and the baseline values exceeds the applicable tolerance level. See section 3.2 for more information.
- (2) <u>Barley Data Evaluation</u>. Evaluate the SRS values by comparing individual and average NIRT protein to the corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein testing service:
 - (a) <u>Duplicate Difference</u>. If the difference between the first and second analysis of an individual SRS exceeds ± 0.25 percent protein, then reanalyze the sample. Record the results from the two analyses closest to each other and discard the third result.

The duplicate difference is calculated as follows:

- Duplicate Difference = an individual sample NIRT value first analysis minus (-) individual sample NIRT value second analysis.
- (b) <u>Individual Difference</u>. An individual reference sample NIRT protein value differs from its baseline value by more than ± 0.40 percent protein.

The individual difference is calculated as follows:

- Individual Difference = an individual sample NIRT protein value minus (-) sample baseline value.
- (c) <u>Average Differences</u>. The average difference between the SRS NIRT protein values and the baseline values exceeds the applicable tolerance level. Refer to Section 3.3 for more information.
- (3) <u>Soybean Data Evaluation</u>. Evaluate the SRS values by comparing individual and average NIRT protein and oil to their corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein and oil testing services:
 - (a) <u>Individual Difference</u>. An individual reference sample NIRT protein value differs from its baseline value by more than ± 0.40 percent protein. An individual reference sample NIRT oil value differs from its baseline value by more than ± 0.30 percent oil.

The individual difference is calculated as follows:

Individual Difference = an individual sample NIRT value minus (-) sample baseline value.

- (b) <u>Average Differences</u>. The average difference between the SRS NIRT protein and/or oil values and the baseline values exceed the applicable tolerance level (see Section 3.4 for more information.)
- (4) <u>Corn Data Evaluation</u>. Evaluate the SRS values by comparing individual and average NIRT protein, oil, and starch to their corresponding baseline value(s). During data evaluation, if any of the following conditions occur, the operator must take the necessary corrective action before providing official protein, oil, and starch testing services:
 - (a) <u>Duplicate Difference</u>. If the difference between the first and second analysis of an individual SRS exceeds \pm 0.30 percent protein, \pm 0.40 percent oil, or \pm 0.90 percent starch, then reanalyze the sample for the constituent of interest. Record the results from the two analyses closest to each other for the constituent of interest and discard the third result.

The duplicate difference is calculated as follows:

- Duplicate Difference = an individual sample NIRT value first analysis minus (-) individual sample NIRT value second analysis.
- (b) <u>Individual Difference</u>. An individual reference sample NIRT protein value differs from its baseline value by more than ± 0.40 percent protein. An individual reference sample NIRT oil value differs from its baseline value by more than ± 0.50 percent oil. An individual reference sample NIRT starch value differs from its baseline value by more than ± 0.80 percent starch.

The individual difference is calculated as follows:

Individual Difference = an individual sample NIRT value minus (-) sample baseline value.

(c) <u>Average Differences</u>. The average difference between the SRS NIRT protein and/or oil and/or starch values and the baseline values exceed the applicable tolerance level (see section 3.5 for more information.)

3.2 SRS PROCEDURES - WHEAT BIAS ADJUSTMENTS

- a. General.
 - (1) The NIRT instrument may, at times, either read high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official wheat protein testing services are performed.
 - (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to TSD standard NIRT instruments. Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT

constant for the calibration.

(3) Prior to official wheat protein testing, perform a bias check and, if necessary, adjust the intercept constant for wheat.

The ANN wheat calibration package contains seven (7) separate named calibrations as noted in section 2.2, c. The calibrations are all identical (except "Wheat", which does not display unofficial moisture). For official protein measurements, it is acceptable to use any of the calibrations, which are interchangeable as long as the same intercept constant is entered in each.

Note: To maintain instrument standardization, it is required that, when a bias adjustment is made on a given wheat calibration, the new intercept must be entered in all other wheat calibrations. For simplicity, it is suggested that only the calibration "Wheat" be loaded on the instrument unless there are other reasons for using one of the named-class calibrations. For example, the operator might want a printout or results display showing the class name.

- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
- (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain SRS and bias records at each location at all times.
- b. <u>Tolerance Levels</u>. In addition to the individual sample difference limit of ± 0.40 percent protein, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available. All the tolerances are listed in appendix 3.1 at the end of chapter 3.

Presently, the four tolerance levels are set as follows:

(1) LEVEL-I (± 0.10 percent protein tolerance):

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 12 results).

(2) LEVEL-II (± 0.07 percent protein tolerance):

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 24 results) collected within a 2-week period and within a 5°F temperature range.

(3) LEVEL-III (± 0.05 percent protein tolerance):

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 36 results) collected within a 2-week period and within a 5°F temperature range.

(4) LEVEL-IV (± 0.03 percent protein tolerance):

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 60 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive or negative.

Record all bias adjustment data in the maintenance log and on the SRS log and/or worksheets (see attachments for examples). Include the date of the bias adjustment, the suspected source of error (if known), any action taken to correct the problem, direction provided by the NIRT coordinator or TSD, and the magnitude of the adjustment. All locations must ensure records

(SRS, bias, and maintenance logs) are complete, legible and in chronological order. The field office manager may request copies of SRS worksheet information if a problem is suspected (unusually high or low results, board appeal, or foreign complaint, etc.) for further review.

c. <u>Performing a Bias Check or Adjustment</u>. Previous SRS NIRT results are invalid after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than ± 5°F. Perform a bias check: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instruments accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than ± 5°F from the temperature recorded during the daily check.

Note for Infratec Models 1225, 1226 and 1227: After changing between the 18-millimeter and 30-millimeter sample cell, select the high and low protein samples from the wheat SRS. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

- **STEP 1**: Mix each SRS thoroughly before analyzing.
- **STEP 2**: Calculate the difference between the duplicate analyses for the same sample.

Does the duplicate difference for any sample differ by more than ± 0.20 percent protein?

- (1) If **NO**, proceed to STEP 3.
- (2) If **YES**, reanalyze the sample. Record and use the results from the two analyses closest to each other and discard the third result. Proceed to STEP 3.
- STEP 3: Calculate individual analysis differences between the NIRT and baseline. Does any SRS differ by more than ± 0.40 percent protein from its baseline value?
 - (1) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
 - (2) If **YES**, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.50 percent protein?

The range difference is found by identifying the most positive individual difference and identifying the most negative individual difference then calculating the difference between the two extreme values. See the following examples for more information:

Example 1. Where positive and negative differences are observed:

If the largest positive difference is + 0.34 and the largest negative difference is - 0.39, then the range difference is = (+ 0.34) - (- 0.39) = + 0.73 percent protein.

Example 2. Where all observed differences are positive:

If the largest positive difference is + 0.34 and the smallest positive difference is +0.09, then the range difference is = (+ 0.34) - (+ 0.09) = + 0.25 percent protein.

Example 3. Where all observed differences are negative:

If the smallest negative difference is -0.09 and the largest negative difference is -0.39, then the range difference is = (-0.09) - (-0.39) = +0.30 percent protein.

(a) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more + 0.40 percent protein different from its baseline value and notify your NIRT

- coordinator. Calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- (b) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- **STEP 4**: Review available data sets. Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid.
 - (1) Determine whether the average difference between the NIRT and baseline is \pm 0.10 percent protein.
 - (a) If the average difference between the NIRT and baseline is ± 0.10 percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.b.
 - (b) If the average difference between the NIRT and baseline is greater than ± 0.10 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.05 percent protein or less, proceed to analyze market samples. If using the wet gluten calibration, compute and enter the new wet gluten intercept according to:
 - Wet gluten intercept = Protein Intercept x = 3.029
 - If the difference is still greater than \pm 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
 - (2) If the previous data set is valid, calculate the average difference from the baseline for the two sets (24 individual analyses.)
 - (a) If the average difference is ± 0.07 percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.c.
 - (b) If the average difference is greater than \pm 0.07 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.05 percent protein or less, proceed to analyze market samples. If using the wet gluten calibration, compute and enter the new wet gluten intercept according to:
 - Wet gluten intercept = Protein Intercept x 3.029
 - If the difference is still greater than \pm 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
 - (2) If the previous two data sets are valid, calculate the average difference from the baseline for the three sets (36 individual analyses.)
 - (a) If the average difference is ± 0.05 percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.d.
 - (b) If the average difference is greater than ± 0.05 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.05 percent protein or less, proceed to analyze market samples. If using the wet gluten calibration, compute and enter the new wet gluten intercept according to:

- Wet gluten intercept = Protein Intercept x 3.029
- If the difference is still greater than \pm 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- (3) If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (60 individual analyses), otherwise proceed to analyze market samples.
 - (a) If the average difference is \pm 0.03 percent protein or less, proceed to analyze market samples.
 - (b) If the average difference is greater than \pm 0.03 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.05 percent protein or less, proceed to analyze market samples. If using the wet gluten calibration, compute and enter the new wet gluten intercept according to:
 - Wet gluten intercept = Protein Intercept x = 3.029
 - If the difference is still greater than \pm 0.05 percent protein, recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.

3.3 SRS PROCEDURES - BARLEY BIAS ADJUSTMENTS

a. General.

- (1) The NIRT instrument may, at times, either read high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official barley protein testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which,on average, are accurate with respect to TSD standard NIRT instruments. Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the calibration.
- (3) Prior to official barley protein testing, perform a bias check and, if necessary, adjust the intercept constant for barley.
 - The ANN barley calibration package contains three (3) separate named calibrations as noted in section 2.2,b,1. The barley calibrations are named "Barley", "Six-rowed Barley" and "Two-rowed Barley". The calibrations are all identical (except "Barley", which does not display unofficial moisture.) For official protein measurements, it is acceptable to use any of the calibrations, which are interchangeable as long as the same interceptconstant is entered in each.

Note: To maintain instrument standardization, it is required that, when a bias adjustment is made on a given barley calibration, the new intercept must be entered in all other barley calibrations. For simplicity, it is suggested that only the calibration "Barley" be loaded on the instrument, unless there are other reasons for using one of the named- class calibrations. For example, the operator might want a printout or results display showing the class name.

- (4) Determine instrument accuracy by comparing SRS results to their establishedbaseline values.
- (5) Only SRS results obtained using procedures identical to market sampleprocedures should be

used in computing bias adjustments.

- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, checkfor possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain bias records at each location at all times.
- b. <u>Tolerance Levels</u>. In addition to the individual sample difference limit of ± 0.40 percent protein, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicabletolerance level is based on the number of valid SRS data sets available. All the tolerances are listed in appendix 3.1 at the end of chapter 3.

Presently, the four tolerance levels are set as follows:

LEVEL-I (± 0.12 percent protein tolerance):

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set(typically an average of 10 results).

(1) LEVEL-II (± 0.09 percent protein tolerance):

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 20 results) collected within a 2-week periodand within a 5°F temperature range.

(2) LEVEL-III (± 0.06 percent protein tolerance):

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 30 results) collected within a 2-week period and within a 5°F temperature range.

(3) LEVEL-IV (± 0.04 percent protein tolerance):

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 50 results) that are all positive or negativeand were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive or negative.

Record all bias adjustment data in the maintenance log and on the SRS log and/or worksheets (see attachments for examples). Include the date of the bias adjustment, the suspected source of error (if known), and, any action taken to correct the problem, direction provided by the NIRT coordinator or TSD, and the magnitude of the adjustment. All locations must ensure records (SRS, bias, and maintenance logs) are complete, legible and in chronological order. The field office manager may request copies of SRS worksheet information if a problem is suspected (unusually high or low results, board appeal, or foreign complaint, etc.)for further review.

c. <u>Performing a Bias Check or Adjustment</u>. Previous SRS NIRT results are invalid after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than ± 5°F. Perform a bias check: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instruments accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH wasoutside the acceptable range; or (4) the temperature changes by more than ± 5°F from the temperature recorded during the daily check

Note for Infratec Models 1225, 1226 and 1227: After changing between the 18-millimeter and 30-millimeter sample cell, select the high and low protein samples from the barley SRS. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield results similar to the results made prior to changing the sample cell. If not, reinstallthe sample cell making sure the area is clear of any obstructions.

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and

- (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRSbaseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.
- **STEP 1**: Mix each SRS thoroughly before analyzing.
- **STEP 2**: Calculate the difference between the duplicate analyses for the same sample.

Does the duplicate difference for any sample differ by more than \pm 0.25 percent protein?

- (a) If **NO**, proceed to STEP 3.
- (b) If **YES**, reanalyze the sample. Record and use the results from the two analyses closest to each other and discard the third result. Proceed to STEP 3.
- STEP 3: Calculate individual analysis differences between the NIRT and baseline. Does anySRS differ by more than ± 0.40 percent protein from its baseline value?
 - (1) If NO, calculate the average difference between the NIRT values andbaseline. Proceed to STEP 4.
 - (2) If YES, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.60 percent protein?

Examples for calculating the range difference between the two extremevalues can be found on page 3-9.

- (a) If YES, reanalyze deviating sample(s). Drop from the average any sample that remains more ± 0.40 percent protein different from its baseline value and notify your NIRT coordinator. Calculate the averagedifference between the NIRT values and baseline. Proceed to STEP 4.
- (b) If NO, calculate the average difference between the NIRT values andbaseline. Proceed to STEP 4.
- **STEP 4**: Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).
 - (1) Determine whether the average difference between the NIRT and baselineis ± 0.12 percent protein.
 - (a) If the average difference between the NIRT and baseline is ± 0.12 percentprotein or less, and there are no more valid SRS data available, proceed analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.b.
 - (b) If the average difference between the NIRT and baseline is greater than \pm 0.12 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.06 percent protein or less, proceedto analyze market samples.
 - If the difference is still greater than \pm 0.06 percent protein, recheckyour calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.

- (2) If the previous data set is valid, calculate the average difference from thebaseline for the two sets (20 individual analyses.)
 - (a) If the average difference is ± 0.09 percent protein or less, and thereare no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.c.
 - (b) If the average difference is greater than ± 0.09 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.06 percent protein or less, proceedto analyze market samples.
 - If the difference is still greater than \pm 0.06 percent protein, recheckyour calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- (3) If the previous two data sets are valid, calculate the average difference from the baseline for the three sets (30 individual analyses.)
 - (a) If the average difference is ± 0.06 percent protein or less, and there are no more valid SRS data available, proceed to analyze market samples. If thebias is OK and more valid SRS data are available, proceed to STEP 4.d.
 - (b) If the average difference is greater than \pm 0.06 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.06 percent protein or less, proceedto analyze market samples.
 - If the difference is still greater than \pm 0.06 percent protein, recheckyour calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- (4) If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets(50 individual analyses), otherwise proceed to analyze market samples.
 - (a) If the average difference is ± 0.04 percent protein or less, proceed to analyze market samples.
 - (b) If the average difference is greater than ± 0.04 percent protein, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.06 percent protein or less, proceedto analyze market samples.
 - If the difference is still greater than \pm 0.06 percent protein, recheckyour calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.

3.4 SRS PROCEDURES - SOYBEANS BIAS ADJUSTMENTS

a. General.

- (1) The NIRT instrument may, at times, read either high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official soybean protein and oil testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to TSD standard NIRT instruments. Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the constituent of interest.
- (3) Prior to official soybean protein and oil testing, perform a bias check and, if necessary, adjust the intercept constant for each constituent.
- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
- (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain bias records at each location at all times.
- b. <u>Tolerance Levels</u>. In addition to the individual sample difference limit of ± 0.40 percent protein and ± 0.30 percent oil, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available. All the tolerances are listed in appendix 3.1 at the end of chapter 3.

Presently, the four tolerance levels are set as follows:

(1) LEVEL-I (\pm 0.17 percent protein and \pm 0.12 percent oil tolerances):

Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 5 results).

(2) LEVEL-II (\pm 0.12 percent protein and \pm 0.09 tolerances):

Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 10 results) collected within a 2-week period and within a 5°F temperature range.

(3) LEVEL-III (\pm 0.10 percent protein and \pm 0.07 percent oil tolerances):

Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 15 results) collected within a 2-week period and within a 5°F temperature range.

(4) LEVEL-IV (± 0.08 percent protein and ± 0.05 percent oil tolerances):

Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 25 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive or negative.

Record all bias adjustment data in the maintenance log and on the SRS log and/or worksheets (see attachments for examples). Include the date of the bias adjustment, the suspected source of error (if

known), and, any action taken to correct the problem, direction provided by the NIRT coordinator or TSD, and the magnitude of the adjustment. All locations must ensure records (SRS, bias, and maintenance logs) are complete, legible and in chronological order. The field office manager may request copies of SRS worksheet information if a problem is suspected (unusually high or low results, board appeal, or foreign complaint, etc.) for further review.

c. <u>Performing a Bias Check or Adjustment</u>. Previous SRS NIRT results are invalid after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than ± 5°F. Perform a bias check when: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instrument's accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than ± 5°F from the temperature recorded during the daily check.

Note for Infratec Models 1225, 1226 and 1227: After changing between the 18-millimeter and 30-millimeter sample cell, select the high and low protein SRS. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield protein results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

- **STEP 1**: Mix each SRS thoroughly before analyzing.
- STEP 2: Calculate individual sample differences between the NIRT and baseline. Do any SRS differ by more than \pm 0.40 percent protein or \pm 0.30 percent oil from its baseline value?
 - (1) If NO, calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.
 - (2) If YES, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.60 percent protein or 0.45 percent oil?

Examples for calculating the range difference between the two extreme values can be found on page 3-10.

- (a) If **YES**, reanalyze deviating sample(s). Drop from the average any sample that remains more ± 0.40 percent protein or ± 0.30 percent oil different from its baseline value and notify your NIRT coordinator. Only one sample can be dropped from the average. If more than one sample exceeds the tolerance, contact TSD. Calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.
- (b) If **NO**, calculate the average difference between the NIRT values and baseline. Proceed to STEP 3.
- STEP 3: Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).
 - (1) Determine whether the average difference between the NIRT and baseline is ± 0.17 percent protein or ± 0.12 percent oil.
 - a) If the average difference between the NIRT and baseline is ± 0.17 percent protein or less and ± 0.12 percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 3.b.
 - (b) If the average difference between the NIRT and baseline is greater than ± 0.17 percent protein or ± 0.12 percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the

SRS set.

- If the corrected difference is \pm 0.08 percent protein or less or \pm 0.05 percent oil or less, proceed to analyze market samples.
- If the difference is still greater than \pm 0.08 percent protein or \pm 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- (2) If the previous data set is valid, calculate the average difference from the baseline for the two sets (10 individual analyses.)
 - (a) If the average difference is \pm 0.12 percent protein or less and \pm 0.09 percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 3.c.
 - (b) If the average difference is greater than ± 0.12 percent protein or ± 0.09 percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.08 percent protein or less or \pm 0.05 percent oil or less, proceed to analyze market samples.
 - If the difference is still greater than \pm 0.08 percent protein or \pm 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- (3) If the previous two data sets are valid, calculate the average difference from the baseline for the three sets (15 individual analyses.)
 - (a) If the average difference is \pm 0.10 percent protein or less and \pm 0.07 percent oil or less, and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 3.d.
 - (b) If the average difference is greater than \pm 0.10 percent protein or \pm 0.07 percent oil, adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.08 percent protein or less or \pm 0.05 percent oil or less, proceed to analyze market samples.
 - If the difference is still greater than \pm 0.08 percent protein or \pm 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- (4) If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (25 individual analyses) otherwise proceed to analyze market samples.
 - (a) If the average difference is \pm 0.08 percent protein or less and \pm 0.05 percent oil or less, proceed to analyze market samples.
 - (b) If the average difference is greater than \pm 0.08 percent protein or greater than
 - ± 0.05 percent oil or less, adjust the NIRT intercept constant by an amount equal to the difference from the baseline.

Check the adjustment by reanalyzing the SRS set.

- If the corrected difference is \pm 0.08 percent protein or less or \pm 0.05 percent oil or less, proceed to analyze market samples.
- If the difference is still greater than \pm 0.08 percent protein or \pm 0.05 percent oil, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.

3.5 SRS PROCEDURES - CORN BIAS ADJUSTMENTS

a. General.

- (1) The NIRT instrument may, at times, read either high or low when compared to the SRS baseline values. Systematic deviation from the baseline values greater than the allowable tolerance limits must be corrected before official corn protein, oil, and starch testing services are performed.
- (2) All NIRT instruments are equipped with an "intercept" constant which is used to adjust the instrument's bias with respect to the baseline. If the intercept constant is set properly, the instrument should give results which, on average, are accurate with respect to TSD standard NIRT instruments. Adjust the bias of the NIRT calibration by subtracting the calculated average difference between the SRS results and the baseline value from the instrument's INTERCEPT constant for the constituent of interest.
- (3) Prior to official corn protein, oil, and starch testing, perform a bias check and, if necessary, adjust the intercept constant for each constituent.
- (4) Determine instrument accuracy by comparing SRS results to their established baseline values.
- (5) Only SRS results obtained using procedures identical to market sample procedures should be used in computing bias adjustments.
- (6) Any change in bias (especially if the bias has been stable for some time) may be an indication of procedural or equipment problems. If in doubt, check for possible problems before making an adjustment.
- (7) It is permissible to repeat the entire SRS set and discard prior SRS results if the results appear to be in error.
- (8) Maintain bias records at each location at all times.
- b. <u>Tolerance Levels</u>. In addition to the individual sample difference limit of \pm 0.40 percent protein, \pm 0.50 percent oil, and \pm 0.80 percent starch, four average difference tolerance levels were developed. These tolerance levels allow testing locations to bias to tighter tolerances. The applicable tolerance level is based on the number of valid SRS data sets available. All the tolerances are listed in appendix 3.1 at the end of chapter 3.

Presently, the four tolerance levels are set as follows:

- (1) LEVEL-I (\pm 0.15 percent protein, \pm 0.15 percent oil, and \pm 0.35 percent starch tolerances):
 - Apply a LEVEL-I tolerance when evaluating a single valid SRS data set (typically an average of 8 results.)
- (2) LEVEL-II (\pm 0.10 percent protein, \pm 0.10 percent oil, and \pm 0.25 percent starch tolerances):
 - Apply a LEVEL-II tolerance when evaluating the average of two valid SRS data sets (typically an average of 16 results) collected within a 2-week period and within a 5°F temperature range.
- (3) LEVEL-III (\pm 0.07 percent protein, \pm 0.07 percent oil, and \pm 0.20 percent starch tolerances):
 - Apply a LEVEL-III tolerance when evaluating the average of three consecutive SRS data sets (typically an average of 24 results) collected within a 2-week period and within a 5°F temperature range.
- (4) LEVEL-IV (\pm 0.05 percent protein, \pm 0.06 percent oil, and \pm 0.15 percent starch tolerances):
 - Apply a LEVEL-IV tolerance when evaluating the average of five valid SRS data sets (typically an average of 40 results) that are all positive or negative and were collected within a 2-week period and within a 5°F temperature range. An average difference between a single valid SRS data set and the baseline of zero is neither positive nor negative.

Record all bias adjustment data in the maintenance log and on the SRS log and/or worksheets (see attachments for examples). Include the date of the bias adjustment, the suspected source of error (if known), and, any action taken to correct the problem, direction provided by the NIRT coordinator or TSD, and the magnitude of the adjustment. All locations must ensure records (SRS, bias, and

maintenance logs) are complete, legible and in chronological order. The field office manager may request copies of SRS worksheet information if a problem is suspected (unusually high or low results, board appeal, or foreign complaint, etc.) for further review.

c. <u>Performing a Bias Check or Adjustment</u>. Previous SRS NIRT results are invalid after a change in the standard slope, bias adjustment, instrument repair or replacement, or when the recorded temperature has varied by more than ± 5°F. Perform a bias check when: (1) once a day when the instrument is turned on and warmed up, or after power is restored after a power outage; (2) the instrument's accuracy is questioned; (3) the RH is outside of the acceptable range or RH returns to the acceptable range and a bias adjustment was made while the RH was outside the acceptable range; or (4) the temperature changes by more than ± 5°F from the temperature recorded during the daily check.

Note for Infratec Models 1225, 1226 and 1227: After changing between the 18-millimeter and 30-millimeter sample cell, select the high and low oil SRS samples. Run these samples as a check to verify that the sample cell is installed correctly. The samples should yield oil results similar to the results made prior to changing the sample cell. If not, reinstall the sample cell making sure the area is clear of any obstructions.

The following procedure outlines the instrument check and evaluation of two kinds of data. These data concern (1) individual differences from the baseline and (2) the data related to the average differences from the baseline. Each is used to determine the relative accuracy of the equipment and the amount of correction (biasing) needed to allow the equipment to duplicate the values of the known SRS baseline values. The instrument check and data evaluation procedure start with "STEP 1" then proceed through a series of "YES" and "NO" responses to questions until being directed to analyze market samples.

- **STEP 1**: Mix each SRS thoroughly before analyzing.
- **STEP 2**: Calculate the difference between the duplicate analyses for the same sample.

Does the duplicate difference for any sample differ by more than \pm 0.30 percent protein, or \pm 0.40 percent oil, or \pm 0.90 percent starch?

- (1) If **NO**, proceed to STEP 3.
- (2) If **YES**, reanalyze the sample. Record and use the results from the two analyses closest to each other for the constituent of interest and discard the third result. Proceed to STEP 3.
- STEP 3: Calculate individual analysis differences between the NIRT and baseline for each constituent. Does any SRS differ by more than \pm 0.40 percent protein, or \pm 0.50 percent oil, or \pm 0.80 percent starch from its baseline value?
 - (1) If NO, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
 - (2) If YES, calculate the range of difference between the NIRT and baseline. Is the range greater than 0.50 percent protein, or 0.60 percent oil, or 1.50 percent starch?

Examples for calculating the range difference between two extreme values can be found on page 3-10.

- (a) If YES, reanalyze deviating sample(s). Drop from the average any sample that remains more ± 0.40 percent protein, or ± 0.50 percent oil, or ± 0.80 percent starch different from its baseline value and notify your NIRT coordinator. Calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- (b) If NO, calculate the average difference between the NIRT values and baseline. Proceed to STEP 4.
- **STEP 4**: Review available data sets. (Data sets older than 2 weeks or intervening bias adjustment, new standard slope, instrument repairs, etc., render SRS data invalid).
 - (1) Determine whether the average difference between the NIRT and baseline is \pm 0.15 percent protein, or \pm 0.15 percent oil, or \pm 0.35 percent starch.

- (a) If the average difference between the NIRT and baseline is ± 0.15 percent protein or less, or ± 0.15 percent oil or less, or ± 0.35 percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.b.
- (b) If the average difference between the NIRT and baseline is greater than \pm 0.15 percent protein, or \pm 0.15 percent oil, or \pm 0.35 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.07 percent protein or less, or \pm 0.07 percent oil or less, or \pm 0.20 percent starch or less proceed to analyze market samples.
 - If the difference is still greater than \pm 0.07 percent protein, or \pm 0.07 percent oil, or \pm 0.20 percent starch, recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.
- (2) If the previous data set is valid, calculate the average difference from the baseline for the two sets (16 individual analyses.)
 - (a) If the average difference is \pm 0.10 percent protein or less, or \pm 0.10 percent oil or less, or \pm 0.25 percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.c.
 - (b) If the average difference is greater than \pm 0.10 percent protein, or \pm 0.10 percent oil, or \pm 0.25 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.07 percent protein or less, or \pm 0.07 percent oil, or \pm 0.20 percent starch proceed to analyze market samples.
 - If the difference is still greater than \pm 0.07 percent protein, or \pm 0.07 percent oil, or \pm 0.20 percent starch recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- (3) If the previous two data sets are valid, calculate the average difference from the baseline for the three sets (24 individual analyses).
 - (a) If the average difference is \pm 0.07 percent protein or less, or \pm 0.07 percent oil or less, or \pm 0.20 percent starch or less and there are no more valid SRS data available, proceed to analyze market samples. If the bias is OK and more valid SRS data are available, proceed to STEP 4.d.
 - (b) If the average difference is greater than \pm 0.07 percent protein, or \pm 0.07 percent oil, or \pm 0.20 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.
 - If the corrected difference is \pm 0.07 percent protein or less, or \pm 0.07 percent oil or less, or \pm 0.20 percent starch or less proceed to analyze market samples.
 - If the difference is still greater than \pm 0.07 percent protein, or \pm 0.07 percent oil, or \pm 0.20 percent starch recheck your calculations and entered intercept constant, and if no errors are found, repeat the biasing procedure.
- (4) If the previous data sets are valid, check the last five runs to see if they are all positive or negative, excluding any zeroes. If they are all positive or negative, then calculate the average difference from the baseline for five sets (40 individual analyses) otherwise proceed to analyze market samples.
 - (a) If the average difference is \pm 0.05 percent protein or less, or \pm 0.06 percent oil or less, or \pm 0.15 percent starch or less proceed to analyze market samples.
 - (b) If the average difference is greater than \pm 0.05 percent protein, or \pm 0.06 percent oil, or \pm 0.15 percent starch adjust the NIRT intercept constant by an amount equal to the difference from the baseline. Check the adjustment by reanalyzing the SRS set.

- If the corrected difference is \pm 0.07 percent protein or less, or \pm 0.07 percent oil or less, or \pm 0.20 percent starch or less proceed to analyze market samples.
- If the difference is still greater than \pm 0.07 percent protein, or \pm 0.07 percent oil, or \pm 0.20 percent starch recheck your calculations and entered intercept constant(s), and if no errors are found, repeat the biasing procedure.

3.6 INSTRUMENT CHECKOUT

Certain checks and maintenance steps must be performed to verify that the NIRT instruments are functioning properly prior to providing official testing services.

- a. Instrument Checkout Schedule.
 - (1) Locations providing NIRT testing on a daily basis must complete these checks for each day.
 - (2) Locations providing infrequent NIRT testing and/or those that monitor NIRT activities of specified service points must complete these checks before any official NIRT results are provided.
 - (3) The instrument checkout procedures outlined below are general. If the instrument does not pass the checkout sequence, official testing shall be suspended until the problem is resolved or corrected. The first step toward resolution is to repeat the test in question and seek advice from your NIRT coordinator. If the problem cannot be resolved, seek advice from TSD.
- b. <u>Instrument Checkout Procedures</u>.
 - (1) Each instrument must have the sample cell pathlength standardized (unless you have an Infratec 1229, 1241, or NOVA with a variable sample cell), and use the appropriate standard slope settings for wheat and/or barley and/or soybeans and/or corn tested at the specified service point.
 - (2) Analyze the appropriate SRS following official procedures each day or prior to use (see 3.2.a, 3.3.a, 3.4.a or 3.5.a, as applicable).
 - (3) Dust out the sample hopper and path at the end of each day.
- c. <u>Other Tests</u>. The NIRT instrument performs extensive operational checks on itself whenever it is powered on and during operation. Record any error messages that appear for use in troubleshooting instrument problems.

It is the responsibility of the NIRT coordinator and technicians to alert the supervisor and suspend official testing if the NIRT performance is questionable. **Use the SRS to test instrument accuracy if instrument performance is questioned**. Official testing may resume when acceptable instrument performance is demonstrated.

APPENDIX 3.1 – SRS TOLERANCE LEVELS

Constituent	# of SRS	Daily	Level I (1 run)	Level II (2 runs)	Level III (3 runs)	Level IV (5 runs)
Wheat Protein	6	Dup ± 0.20 % Ind ± 0.40 % Range 0.50 % Avg →	(12 results) ± 0.10 %	(24 results) ± 0.07 %	(36 results) ± 0.05 %	(60 results) ± 0.03 %
Barley Protein	5	Dup ± 0.25 % Ind ± 0.40 % Range 0.60 % Avg →	(10 results) ± 0.12 %	(20 results) ± 0.09 %	(30 results) ± 0.06 %	(50 results) ± 0.04 %
Soybean Protein	5	Dup n/a Ind ± 0.40 % Range 0.60 % Avg →	(5 results) ± 0.17 %	(10 results) ± 0.12 %	(15 results) ± 0.10 %	(25 results) ± 0.08 %
Soybean Oil	5	Dup n/a Ind ± 0.30 % Range 0.45 % Avg →	(5 results) ± 0.12 %	(10 results) ± 0.09 %	(15 results) ± 0.07 %	(25 results) ± 0.05 %
Corn Protein	4	Dup ± 0.30 % Ind ± 0.40 % Range 0.50 % Avg →	(8 results) ± 0.15 %	(16 results) ± 0.10 %	(24 results) ± 0.07 %	(40 results) ± 0.05 %
Corn Oil	4	Dup ± 0.40 % Ind ± 0.50 % Range 0.60 % Avg →	(8 results) ± 0.15 %	(16 results) ± 0.10 %	(24 results) ± 0.07 %	(40 results) ± 0.06 %
Corn Starch	4	Dup ± 0.90 % Ind ± 0.80 % Range 1.50 % Avg →	(8 results) ± 0.35 %	(16 results) ± 0.25 %	(24 results) ± 0.20 %	(40 results) ± 0.15 %

If wheat is adjusted, wet gluten must be adjusted also. Wet gluten intercept = Protein Intercept x 3.029.

ATTACHMENT 1 - NIRT DAILY WHEAT SRS WORKSHEET

NIRT DAILY WHEAT SRS WORKSHEET

Location:		Serial #:		Date:				
Operator:		Temperatu	ıre:	R.Humidity:				
Instrument Con	stants:	"O"		"P"				
Protein Constar			000 Intercept					
Wet Gluten Cor				(= protein int. x 3.029)				
		PRO	OTEIN					
SRS	Value	Run 1	Difference	Repeat	Difference			
1								
1								
2								
2								
3								
3								
4								
4								
5								
5								
6								
6								
Average								
Range of Differ	ences (Maximur	n 0.50)						
Bias Calculatior	n: Protein Ave	erage						
	Minus Bas	eline						
	Today's P.	Bias						

Transfer range and bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust the intercept constant for a constituent if it is out of tolerance.

REPEATRULES:

- 1. Repeat any sample if an outlier is reported.
- 2. Repeat an individual SRS if the difference between the first and second analysis exceeds 0.20 percent. Record the results from the two analyses closest to each other and discard the third result.
- 3. Repeat any individual SRS that deviates by more than 0.40 from its target value and the range tolerances are exceeded.
- 4. If an individual SRS deviates from its target by more than 0.30 for five consecutive runs, contact TSD to have the SRS replaced.

ATTACHMENT 2 - NIRT DAILY SOYBEAN SRS WORKSHEET

NIRT DAILY SOYBEAN SRS WORKSHEET

Location:		Serial #:		Date:				
Operator:		Temperatu	re:	R.Humidity:_				
Instrument Const	tants:	"O"		"P"				
Protein Constant	s:	Slope	Intercept					
OIL Constants:		SlopeIntercept						
		PRO	TEIN					
SRS	Value	Run 1	Difference	Repeat	Difference			
1								
2								
3								
4								
5								
Average								
		C)IL					
SRS	Value	Run 1	Difference	Repeat	Difference			
1								
2								
3								
4								
5								
Average								
Range of Differer	nces (Maximur	n 0.60)		-				
Bias Calculation:	Protein Ave	erage	Oil Average					
	Minus Base	eline	Minus Base	line				
	Today's Bia	as	Today's Bias					
Transfer range and	bias results to th	ne SRS Bias Log,	calculate multi-run	averages, and ap	oply tolerances.			

Transfer range and bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust the intercept constant for a constituent if it is out of tolerance.

REPEAT RULES:

- 1. Repeat any sample if an outlier is reported.
- 2. Repeat the SRS set if the range tolerances are exceeded.
- 3. If the range tolerances are exceeded on two consecutive runs and an individual SRS deviates by 0.40 for protein and 0.30 for oil, that sample may be temporarily dropped from the average. Contact TSD for a replacement.

ATTACHMENT 3 – NIRT WEEKLY WHEAT SRS WORKSHEET

NIRT WEEKLY WHEAT SRS WORKSHEET

Location	n:		Serial #:					Date:			
Instrume	ent Consta	ants:		"O"_					P"		
Protein	Constants	:		Slop	e 1.00	0 Interce	pt				
Wet Glu	ten Const	ants:		Slop	e 1.00	0 Interce	rcept(= protein int. x 3.029)				.029)
					PROT	EIN					
Date:											
Tech:											
Temp:											
Humid	ity:										
SRS	Baseline	Result	Diff	Result	Diff	Result	Diff	Result	Diff	Result	Diff
1											
1											
2											
2											
3											
3											
4											
4											
5											
5											
6											
6											
Average											
Range of D)ifferences (M	ences (Max. 0.50)									
	Bias Calculation										
2nd P	revious Run										
Pi	revious Run										
One Run	(.10)										
Two Run	s (.07)										
Three Ru	ıns (.05)										
Five Run	s (all +/03)										

ATTACHMENT 4 - NIRT WEEKLY SOYBEAN SRS WORKSHEET

NIRT WEEKLY SOYBEAN SRS WORKSHEET

Location	1:			Seria	l #:			[Date:			
Instrume	ent Consta	ants:		"O"_					'P"			
Protein	Constants	:				ln						
				Soyl	bean Pro	otein & C)il					
Date:												
Tech:												
Temp:	itar											
Humid	ity.					<u> </u>					1	
CDC	Basslins	Dogult	Diff	Dogult	PROT		Diff	Dogult	Diff	Booult	Diff	
SRS 1	Baseline	Result	Diff	Result	Diff	Result	Diff	Result	Diff	Result	Diff	
2												
3												
4												
5												
Average												
Range of D	ifferences (M	ax. 0.60) _										
				В	ias Calc	ulation						
	revious Run											
	revious Run									т т		
One Run Two Run												
Three Ru												
	s (all +/08)											
				Clar		l n	taraant					
OIL Con	stants.			210		ln	tercept					
CDC	Basslins	Dogult	Diff	Dogult	OIL		Diff	Dogult	Diff	Booult	Diff	
SRS 1	Baseline	Result	Diff	Result	Diff	Result	Diff	Result	Diff	Result	Diff	
2												
3												
4												
5												
Average												
Range of D	ifferences (M	ax. 0.60)										
				В	ias Calc	ulation						
	revious Run											
	revious Run											
One Run												
Two Run												
Three Ru												
Five Run	s (all +/05)											

ATTACHMENT 5 - NIRT WHEAT SRS BIAS LOG

NIRT WHEAT SRS BIAS LOG

Tecator Model:	_Serial # :	Location:
Results obtained after a slope or be be averaged with results obtained	ias adjustment, SRS replacer prior to the change (also, the tive or all negative). Every efl	emperature range may be averaged. ment, or NIRT repair or replacement cannot 5-Run correction can only be applied if five fort should be made to maintain the bias

NIRT WET GLUTEN: For any protein bias adjustment, wet gluten must be updated according to:

Wet Gluten Intercept = Protein Intercept x 3.029

			Today's	Multi-Run Averages					
Date			Bias	2-Run	3-Run	5-Run	В*	Oper	
mm/dd/yy	Temp	RH	± 0.10	± 0.07	± 0.05	± 0.03	R*	Init	Comments
* (B) bias	adjustme	ent, or ((R) SRS re	placed. If the	ne NIRT is	repaired or	r repl	aced, s	tart a new log.

ATTACHMENT 6 - NIRT SOYBEAN SRS BIAS LOG

NIRT SOYBEAN SRS BIAS LOG

Tecator Model:	Serial # :	Location:	
NOTE O. I. II. II. II. II.			
NOTE: Only results less than two weeks	old and within a 5E	F temperature range may b	e averaged. Results obtained after
a slope or bias adjustment, SRS replace	ment, or NIRT repai	r or replacement cannot be a	averaged with results obtained prior

a slope or bias adjustment, SRS replacement, or NIRT repair or replacement cannot be averaged with results obtained prior to the change (also, the 5-Run correction can only be applied if five consecutive, valid runs are all positive or all negative). Every effort should be made to maintain the bias within the 0.08% Protein and 0.05% Oil tolerances

					Protein			Oil					
			Today's		Run Ave		S*	Today's		Run Ave		S* B*	Oper
Date			Bias	2-Run			В*	Bias	2-Run			B*	Init
mm/dd/yy	Temp	RH	± 0.17	± 0.12	± 0.10	± 0.08	R*	± 0.12	± 0.09	± 0.07	± 0.05	R	
*(S) slope			(D) I:	L				1 1641 - 1	UDT:			L	_, .

*(S) slope adjustment, (B) bias adjustment, or (R) SRS replaced. If the NIRT is repaired or replaced, start a new log.

ATTACHMENT 7 - NIRT DAILY CORN SRS WORKSHEET

NIRT DAILY CORN SRS WORKSHEET

Loca	ition:_			Serial #:							Date:				
Ope	rator:_					_Tem	peratui	re:			_R. Hu	umidity	r:		
		Const									"P"				
				<u>Protei</u>	<u>n</u>		<u>Oil</u>				<u>Starch</u>				
Slop	е	_				_									
Inter	cept	_				_				_					
S		ı	Proteir	n				Oil			Starch				
R S	Value	Run 1	Diff	Repeat	Diff	Value	Run 1	Diff	Repeat	Diff	Value	Run 1	Diff	Repeat	Diff
1															
1															
2															
2															
3															
3															
4															
4															
AVG															
						<u> </u>									<u> </u>
	ge of rence:	S _									_	_		_	
	mum			0.50		-			0.60		-			1.50	
								_			_				
Level	Tolerar	ices (ave	erage o	of 8 results	s):	Proteir	$1 = \pm 0.13$	5	Oil = ± 0.15 Starch = ± 0.35				= ± 0.35		

Transfer bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust intercept constant for a constituent if it is out of tolerance

ATTACHMENT 8 - NIRT WEEKLY CORN SRS WORKSHEET

NIRT WEEKLY CORN SRS WORKSHEET

tants:		Temp "O"_ Interd	perature	e:		R. Hu "P"					
		Interd									
		Interd									
		_									
				T							
	SRS Results										
Result	Diff	Result	Diff	Result	Diff	Result	Diff	Result	Diff		
	e Result	e Result Diff	e Result Diff Result								

intercept constant for a constituent if it is out of tolerance

ATTACHMENT 9 - NIRT CORN SRS BIAS LOG

NIRT CORN SRS BIAS LOG

NIRT Model:	Serial #:	Location:
		20041011

NOTE: Only results less than two weeks old and within a \pm 5°F temperature range may be averaged. Results obtained after a slope or bias adjustment, SRS replacement, while outside of the RH limit, or NIRT repair or replacement cannot be averaged with results obtained prior to the change (also, the 5-Run correction can only be applied if five consecutive, valid runs are all positive or all negative). Every effort should be made to maintain the bias within the \pm 0.07 percent protein, \pm 0.07 percent oil, and \pm 0.20 percent starch tolerances.

			Protein					Oil				Starch					
			Today's	Multi-Run Averages S*		S*	Today's	Multi-	Run Ave	rages	S*	Today's	Multi-	RunAve	rages	s	
			Bias	2-Run	3-Run	5-Run	B*	Bias		3-Run		B*	Bias			5-Run	
Date	Temp	RH	± 0.15		±0.07		R*	± 0.15		±0.07	± 0.06		± 0.35		±0.20		
																	Т
																	Г
																	L
																	\vdash
																	\vdash
																	Г
																	L
																	\vdash
																	\vdash
																	Т
																	╙
																	⊢
																	\vdash
																	Г
																	╙
																	\vdash
																	\vdash
																	\vdash
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																	\vdash
																	Г
																	\perp
																	\vdash
	1					l	I	I	l	ı	1	ı		l	I	l	1

*(S) slope adjustment, (B) bias adjustment, or (R) SRS replaced. If the NIRT is repaired or replaced, start a new log.

ATTACHMENT 10 - NIRT DAILY BARLEY SRS WORKSHEET

NIRT DAILY BARLEY SRS WORKSHEET

Location:		Serial #:		Date:		
Operator:						
Instrument Const	ants:	"O"		"P"		
Protein Constants				Intercept:		
		Prot	ein			
SRS	Value	Run 1	Diff	Repeat	Diff	
1						
1						
2						
2						
3						
3						
4						
4						
AVERAGE						
Range of Differen	ces (Maximun	n 0.60)				
Diag Calaulatian	Duntain A					
Bias Calculation:		•				
		eline				
	Today's Bia	ıs				
Transfer range and	bias results to th	e SRS Bias Log	calculate multi-ru	un averages and ap	ply tolerances	

Transfer range and bias results to the SRS Bias Log, calculate multi-run averages, and apply tolerances. Adjust the intercept constant for a constituent if it is out of tolerance.

REPEATRULES:

- 1. Repeat any sample if an outlier is reported.
- 2. Repeat an individual SRS if the difference between the first and second analysis exceeds 0.25 percent. Record the results
- from the two analyses closest to each other and discard the third result. Repeat any individual SRS that deviates by more than 0.40 from its target value and the range tolerances are exceeded.
- 4. If an individual SRS deviates from its target by more than 0.30 for five consecutive runs, contact TSD to have the SRS replaced.

ATTACHMENT 11 - NIRT WEEKLY BARLEY SRS WORKSHEET

NIRT WEEKLY BARLEY SRS WORKSHEET

_ocatio	า:										
nstrum	ent Consta	ants:		"O"				"P"			
Protein	Constants	:	Slope:					Interd	cept:		
					DDOT	EINI					
D (1 1		1	PROT	EIN T		1			
Date: Tech:											
Temp:											
Humid											
SRS Results											
SRS	Baseline	Result	Diff	Result	Diff	Result	Diff	Result	Diff	Result	Diff
1											
1											
2											
2											
3											
3											
4											
4											
5											
5											
Avg.											
Range of [Differences (M	ax. 0.60) _				_				_	
				В	ias Calc	ulation					
	revious Run revious Run										
One Run											
Two Run											
Three Ru											
	s (all +/04)										
					<u> </u>						

ATTACHMENT 12 - NIRT BARLEY SRS BIAS LOG

NIRT BARLEY SRS BIAS LOG

NIRT Model:	_Serial #:	Location:	

NOTE: Only results less than two weeks old and within a 5° F temperature range may be averaged. Results obtained after a bias adjustment, SRS replacement, or NIRT repair or replacement cannot be average with results obtained prior to the change (also, the 5-run correction can only be applied if five consecutive, valid runs are all positive or all negative). Every effort should be made to maintain the bias within the 0.06% Protein tolerance.

			Today's	Multi	-Run Aver	ages			
Date			Bias	2-Run	3-Run	5-Run	В*	Oper	
mm/dd/yy	Temp	RH	± 0.12	± 0.09	± 0.06	± 0.04	R*	Init	Comments

CHAPTER 4 SAMPLE PREPARATION AND ANALYSIS

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4.1 BASIS OF DETERMINATION

The following table summarizes the basis of determination and sample portion requirements for NIRT analysis of grain constituents:

TABLE 4.1 – BASIS OF DETERMINATION

		Size of Represe	ntative Portion		
	Basis of Determination	Models 1225, 1226, 1227, 1229	Models 1241 & NOVA		
Wheat Protein Wet gluten	After the removal of dockage	From approximately 500g to work portion size	From approximately 650g to work portion size		
Barley Protein	After the removal of dockage	(IRIS only)	to work portion size		
Soybeans Protein Oil	After the removal of FM	F			
Corn protein Oil Starch	After the removal of BCFM	From approximately 500g to work portion size	From approximately 825g to work portion size		

4.2 CLEANING SAMPLES

a. Wheat or Barley.

Remove dockage using an approved dockage tester. The dockage tester uses aspiration (air) and a combination of riddles and sieves to remove any readily separable foreign matter. Generally, the foreign matter removed consists of all matter lighter, larger, or smaller than grain. If excessive quantities of wild buckwheat, cob joints, flaxseed, chess, and/or similar types of seeds are found additional sieving procedures are required. Refer to Book II of the Grain Inspection Handbook, Chapter 13 – Wheat, or Chapter 2 – Barley, for detailed instructions.

b. Soybeans.

Clean samples by hand sieving over an 8/64-inch round-hole sieve to remove any fine foreign material (FM). Handpick the portion remaining on top of the sieve to remove any coarse FM. Refer to NIRT Handbook, Chapter 1, Section 1.3 for the definition of coarse FM. Use the portion remaining on top of the sieve after the removal of fine and coarse FM for the analysis.

c. Corn.

Remove broken corn and foreign material (BCFM) using an approved dockage tester or by hand sieving over a 12/64-inch round-hole sieve. Handpick any material other than corn (e.g., soybeans, pieces of corn cob) that remains in the sample. Use the clean portion after the removal of BCFM for the analysis.

d. <u>Mechanical/Hand Sieving Procedures</u>.

Use the following procedures to mechanically/hand sieve foreign material (FM in soybeans, BCFM in corn).

- (1) For mechanical sieving, refer to Book II of the Grain Inspection Handbook, Chapter 1 General Information, Section 1.13, for detailed instructions.
 - (a) Do not sieve more than 500 grams at a time.
 - (b) Set the sizer's counter to the appropriate number of strokes (20 for corn, and 5 for soybeans) and then turn it on.

- (2) For hand sieving:
 - (a) Do not sieve more than 500 grams at a time.
 - (b) Hold the sieve and bottom pan level and, using a steady motion, move the sieve from right to left approximately 10 inches. Return from left to right to complete one sieving operation. Repeat this operation 20 times for corn, and 5 times for soybeans.

4.3 NIRT ANALYSIS

Operators must read the user's manual and familiarize themselves with the instructions before operating the NIRT instrument.

a. Sample Cells.

- (1) For Infratec Models 1225, 1226, 1227 and 1229: Use the 30-millimeter cell for soybeans and corn determinations.
- (2) For Infratec Model 1229 with IRIS: Use the 18-millimeter cell for wheat or barley determinations.
- (3) For Infratec Model 1241: The Model 1241 and NOVA are programmed through the calibration files to automatically adjust the sample cell to 18-millimeters for wheat and barley, or to 30-millimeters for corn and soybeans.
- (4) For Infratec NOVA: Variable cell automatically controlled from 6 33 mm.
- b. <u>Sample Temperature</u>. The sample's temperature must be between 60 °F and 80 °F. If necessary, use a liquid-in-glass or digital thermometer specified to 2 °F (± 1 °C) accuracy or better to measure the sample temperature. If the sample is not in the acceptable temperature range, allow it to cool or warm to within the limits before testing.
- c. <u>Calibration</u>. Ensure that the displayed calibration ID is a TSD approved calibration for the type of grain being tested.
- d. <u>Standard Reference Samples</u>. Check that the SRS for that grain type have been tested that day.
- e. Pour the entire sample into the NIRT hopper, enter the sample identification number, and press the "RUN" button.
- f. The instrument provides a display (and printout, if a printer is attached) of the percentage of the constituent being measured. For wet gluten, see below.
- g. Retain the sample for further analysis if needed.

h. Wet Gluten.

- (1) If the calibration model is installed on the official NIRT instrument, the wet gluten percentage is shown directly on the instrument display. The official wet gluten calibration is packaged with the Foss global wheat protein calibration and distributed by AMS, Technology and Science Division (TSD), to those official testing locations which have purchased the Foss calibration diskette "Small Grain F" (Foss part number 10014260).
- (2) If the NIRT does not display wet gluten results directly, you may calculate the result manually by solving the equation using the NIRT (shown to 0.01 percent) protein result:
 - Wet gluten (14.0 percent mb) = Official protein (12.0 percent mb) x 3.029 7.83
 - For example: a sample with an NIRT wheat protein of 12.79 percent would have a wet gluten content of 30.9 percent. Official protein (12.79 percent) x 3.029 7.83 = 30.91, rounded to 30.9 percent.
- (3) You may use Appendix 1, Conversion Table, at the end of this chapter.

4.4 OUTLIER INDICATIONS

An outlier indication is a warning that the result may be in error. Outlier indications should rarely occur. If frequent outlier indications are observed, contact TSD.

- a. <u>Types of Outliers</u>. There are four possible outliers (A, B, C, and D). The values for each can range in degree from 0 (no outlier) to 5 (very extreme). The greater the value, the less reliable the predicted result. Review the user's manual for more information.
 - (1) "A" Outlier (Residual). The sample's scan doesn't fit the calibration.
 - (2) <u>"B" Outlier (Leverage)</u>. The sample's scan doesn't fit the calibration and, if added, will have a strong influence.
 - (3) <u>"C" Outlier (Standard Deviation)</u>. The standard deviation (variability) between subsamples is beyond the specified limit.
 - (4) <u>"D" Outlier (Outside Range Limits)</u>. One or more of the subsamples have been predicted above or below the constituent value limits for the calibration.
- b. <u>Causes of Outliers</u>. Some conditions will cause intermittent outlier indications (not repeated on reruns) and others will cause consistent outlier indications. The most likely causes for outliers are:

For example:

- (1) Wrong calibration selected. Testing a wheat sample with the soybean calibration;
- (2) Wrong sample cell (Infratec Models 1225, 1226 and 1227). Testing a wheat sample using the soybean/corn (30 mm) cell or vice versa;
- (3) Extremely dirty samples;
- (4) Poorly mixed samples;
- (5) Sample packing or settling problems;
- (6) Electrical interference;
- (7) Measurement of samples not represented in the calibration set; or
- (8) Improper instrument warm up.
- c. What to do when an outlier occurs? When any outlier indication occurs, the operator must perform the following:
 - (1) Verify that the correct calibration and sample cell (Infratec Models 1225, 1226 and 1227) were used (18 mm for wheat and barley, or 30 mm for soybeans and corn).
 - (2) Make sure the sample is cleaned per official procedures.
 - (3) Make sure the room and sample temperature and the room relative humidity are within the specified ranges.
 - (4) Mix the sample thoroughly before retesting.
 - (5) Rerun the sample through the Infratec.
 - (6) If the second run does not show an outlier indication, certify the results of the second run.
 - (7) If the second run shows an outlier indication, certify on the basis of the average of the two runs.
 - (8) Never certify a result with a level "5" outlier in the "A" or "B" positions.

Note: The NIRT determines both protein and oil in soybeans, and protein, oil, and starch in corn on each run. Use the second run result for the constituent in question only. Use the first run result for the other constituent.

4.5 TESTING INDIVIDUAL AND SUBLOT SAMPLES

- a. <u>Individual Sample</u>. Official sample-lot or submitted samples of wheat, barley, corn, or soybeans are tested and certificated for protein, wet gluten, oil, or starch as applicable, in conjunction with official grade determinations or as a separate testing service.
- b. <u>Shiplot and Lash Barges</u>. Testing for protein in wheat is based on sublot results in accordance with the cusum loading plan. Testing for other constituents in wheat, barley; soybeans; and corn can be determined on an average of individual sublot results or on the basis of a composite sample representing the entire lot. Refer to the Grain Inspection Handbook, Book III, for details concerning the cusum loading plan.
- c. <u>Unit Trains</u>. Testing for protein in wheat is determined on individual or sublot basis in accordance with the cu-sum loading plan. Testing for other constituents in wheat, barley, soybeans, and corn can be determined on individual, sublot or composite sample basis. Applicants can request wheat wet gluten, corn protein, oil, and/or starch; and soybean protein and/or oil on a sublot basis while requesting inspection for grade on an individual carrier basis. When articulated railcars are used, each car is tested as a sublot.

Note: The maximum size sublot for protein testing is five railcars for unit trains consisting of less than 200,000 bushels, or less than 50 railcars. For unit trains consisting of 200,000 bushels or more, or 50 railcars or more, the maximum sublot size is ten railcars.

4.6 CERTIFYING OFFICIAL RESULTS

- a. <u>Moisture Basis</u>. NIRT instruments are programmed to determine official criteria results (i.e., protein, wet gluten, oil, starch) on a moisture basis that is commonly used for trading purposes. The instruments will automatically report wheat protein results on a 12.0 percent moisture basis; wheat wet gluten results on a 14.0 percent moisture basis; barley protein results on a dry matter basis; soybean protein and oil results on a 13.0 percent moisture basis; and corn protein, oil, and starch results on a dry matter basis.
- b. <u>Alternate Moisture Basis</u>. An applicant can request an alternate moisture basis be used in lieu of the standard moisture basis for certifying a result. If an applicant requests an alternate moisture basis for a wheat protein result, show both results (12.0 percent standard basis and the alternate specified moisture basis) on the work record and certificate. Wet gluten may not be reported on an alternate moisture basis.
- c. <u>General Procedures</u>. All official criteria results (e.g., protein, wet gluten, oil, starch) shall be recorded on the work record and in the "Results" section of the official inspection certificate to the nearest tenth percent on the applicable moisture basis (wheat protein 12.0 percent, wheat wet gluten 14.0 percent, barley dry matter basis, soybeans 13.0 percent, corn dry matter basis). Upon request, an applicant can request an alternate moisture basis be used in lieu of the standard moisture basis for certifying wheat protein, barley protein, soybean protein and oil, or corn protein, oil, and starch.

If an applicant requests an alternate moisture basis for a wheat protein result, show both results (12.0 percent basis and the specified moisture basis) on the work record and certificate.

Show the official results on the inspection certificate using an approved statement as shown in the next section. Upon request of the applicant for service, official criteria results may be stated on a certificate separate from the grade certificate or on letterhead stationery in lieu of an official certificate.

4.7 STANDARD REPORTING AND CERTIFICATE STATEMENTS: PROTEIN, WET GLUTEN, OIL, AND STARCH

Below are standard templates for certificate statements for reporting results to the standard moisture basis or an alternate percent moisture basis. Please see each subsection for examples of reporting constituents by grain.

Standard Moisture Basis:
Protein/Oil/Starch%, (standard moisture basis) % moisture basis.
Alternate Moisture Basis:
<u>Protein/Oil/Starch</u> content%, (requested) % moisture basis, which equates to <u>Protein/Oil/Starch</u> %, (standard moisture basis) % moisture basis. <u>Protein/Oil/Starch</u> content is reported on an alternate moisture basis in addition to the standard % moisture basis at the applicant's request.

Note: Dry matter basis may also be written as 0.0 percent moisture basis.

<u>Whea</u>	<u>t</u> .
(1)	General. 12.0 Percent Moisture Basis Only.
	Protein results determined only on the 12.0 percent basis are certified with the following statement:
	"Protein%, 12.0% moisture basis."
(2)	12.0 Percent and Specified Moisture Basis.
	Protein results determined on the 12.0 percent basis and an alternate moisture basis are certified with the following statements:
	"Protein content%, (requested) moisture basis, which converts to protein, 12.0% moisture basis. Protein content reported on an alternative moisture basis in addition to the U.S. standard 12.0 percent moisture basis at applicant's request."
	As an example, where a protein result is 13.5 percent (12.0 percent moisture basis) is converted to 15.3 percent (dry matter basis, 0.0% moisture basis), the following statement would be shown on the certificate:
	"Protein 15.3%, dry matter basis, which converts to protein 13.5%, 12.0% moisture basis. Protein content reported on an alternative moisture basis in addition to the U.S. standard 12.0 percent moisture basis at applicant's request."
(3)	<u>Protein/Wet Gluten Only.</u> When certifying protein alone (without official grade and factors), protein and wet gluten alone (without official grade and factors), or wet gluten alone (without official grade and factors), show only the class of wheat on the grade line of the certificate.
	Examples:
	"Hard Red Winter wheat"
	"Hard Red Spring wheat"
	"Soft Red Winter wheat"
	"Durum wheat"
	"Hard White wheat"
	"Soft White wheat"
(4)	Shiplot and Combined Lots. Upon request of the applicant for service, show the range of protein results using the following statement:
	"Sublot protein results range from (lowest) % to (highest)%."
(5)	Wet Gluten. Show the official results (to the nearest 0.1 percent) on the inspection certificate using the following statement:
	"Wet gluten%, 14.0% moisture basis."
	Note: Wet gluten must be certified to the standard moisture basis and cannot be certified to any alternate moisture basis.
<u>Barle</u>	ر. Certify protein in the "Results" section of the official inspection certificate to the nearest tenth

b. <u>Barley</u>. Certify protein in the "Results" section of the official inspection certificate to the nearest tenth percent on a dry matter basis. Applicants have the option of requesting the results on a specified moisture basis or an "as is" moisture basis in addition to or instead of the dry matter basis. Upon request, official criteria results may be stated on a certificate separate from grade or on letterhead stationery in lieu of an official certificate.

a.

Use the following statements to report results: (1) Individual results (protein). "Protein%, dry matter basis." For protein results reported on a *specified moisture basis*, substitute the specified "moisture basis" requested for the words "dry matter basis." For protein, oil, or starch results reported on an as is moisture basis, substitute "as is" moisture basis for the words "dry matter basis." Protein Only. When certifying protein alone (without official grade and factors), show only the sub-(2) class of barley on the grade line of the certificate. **Examples:** "Six-rowed barley" "Two-rowed barley" "Barley" Corn. Certify protein, starch, and oil in the "Results" section of the official inspection certificate to the C. nearest tenth percent on a dry matter basis. Applicants have the option of requesting the results on a specified moisture basis or an "as is" moisture basis in addition to or instead of the dry matter basis. Upon request, official criteria results may be stated on a certificate separate from grade or on letterhead stationery in lieu of an official certificate. Use the following statements to report results: (1) Individual results (protein, oil, starch). "Protein, oil, or starch (as applicable)___ _____%, dry matter basis." (2) A combination of results (protein, oil, starch). %, Oil _%, and starch__ basis." For protein, oil, or starch results reported on a specified moisture basis, substitute the specified "moisture basis" requested for the words "dry matter basis." For protein, oil, or starch results reported on an as is moisture basis, substitute "as is" moisture basis for the words "dry matter basis." Protein and/or Oil and/or Starch Only. When certifying protein and/or oil and/ or starch alone (without (3) official grade and factors), show only the class of corn on the grade line of the certificate. **Examples:** "Yellow Corn" "White Corn" "Mixed Corn"

- (4) <u>Composite Samples</u>. For results determined on the basis of a composite sample include the following statement in addition to the appropriate statement used to certify protein, oil, and starch. "Results based on composite sample analysis."
- d. <u>Soybeans</u>. Certify protein and oil in the "Results" section of the official inspection certificate to the nearest tenth percent on a 13.0% moisture basis. Applicants have the option of requesting the results on a specified moisture basis or an "as is" moisture basis in addition to or instead of the 13.0% moisture basis. Upon request, official criteria results may be stated on a certificate separate from grade or on letterhead stationery in lieu of an official certificate.

(1) <u>Protein</u>. Certificate all official soybean protein results (domestic and export lots) using the following statement:

"Protein %, 13.0% moisture basis."

(2) <u>Oil</u>. Certificate all official soybean oil results (domestic and export lots) using the following statement:

"Oil %, 13.0% moisture basis."

(3) <u>Protein and Oil</u>. Certificate all official soybean protein and oil results (domestic and export lots) using the following statement:

"Protein % and Oil %, 13.0% moisture basis."

(4) <u>Protein and/or Oil Only</u>. When certifying protein and/or oil alone (without official grade and factors), show only the class of soybeans on the grade line of the certificate.

Examples:

"Yellow Soybeans"

"Mixed Soybeans"

(5) Composite Samples. For results determined on the basis of a composite sample include the following statement in addition to the appropriate statement used to certify protein, and oil.

"Results based on composite sample analysis."

4.8 CONVERTING RESULTS TO AN ALTERNATE MOISTURE BASIS

a. Conversion Formulas.

Examples of alternate moisture basis specifications and formulas for correcting the NIRT results are:

(1) Converting a wheat protein, barley protein, soybean protein/oil, or corn protein/oil/starch result from the standard moisture basis to an "as is" moisture basis.

$$A = P \times (100 - M)$$

Where:

A = Percent protein/oil/starch on an "as is" moisture basis.

P = NIRT protein/oil/starch result on the standard moisture basis (based on NIRT result rounded to the nearest tenth percent.)

M = Official moisture result for the sample/lot (as applicable.)

C = 87 for soybeans, 88 for wheat, or 100 for corn and barley.

(2) Converting a barley protein or corn protein, oil, or starch result from the dry matter basis to another specified moisture basis.

$$A = P \times (100 - M)$$

Where:

A = Percent protein/oil/starch on a specified moisture basis.

P = NIRT protein/oil/starch result on a dry matter basis (based on NIRT result rounded to the nearest tenth percent.)

M = Moisture basis specified by applicant.

(3) Converting a soybean protein or oil result from the 13.0 percent moisture basis to another specified moisture basis.

$$A = P \times (100 - M)$$

87

Where:

A = Percent protein/oil on a specified moisture basis,

P = NIRT protein/oil result on the 13.0 percent moisture basis (based on NIRT result rounded to the nearest tenth percent.)

M = Moisture basis specified by applicant.

(4) Converting a soybean protein result to an oil-free and moisture-free basis.

$$B = P \times 100$$
(100 - (O + 13))

Where:

- B = Percent protein on an oil-free and moisture-free basis. P =NIRT protein result on a 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent.)
- O = Percent oil on a 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent.)
- (5) Converting a soybean protein result to an oil-free and specified moisture basis.

$$C = \frac{P \times (100 - M)}{100 - (13 + O)}$$

Where:

C = Percent protein on an oil-free and specified moisture basis.

P = NIRT protein result on a 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent.)

M = Moisture basis specified by the applicant.

- O = NIRT oil result on 13 percent moisture basis (based on NIRT result rounded to the nearest tenth percent.)
- (6) Converting a wheat protein result from the 12.0 percent moisture basis to another specified moisture basis.

$$A = P \times (100 - M)$$

Where:

A = Percent protein on a specified moisture basis.

P = NIRT protein result on the 12.0 percent moisture basis (based on NIRT result rounded to the nearest tenth percent.)

M = Moisture basis specified by applicant.

b. <u>Conversion Guidelines for Submitted Samples and Single Lot Inspections</u>.

For submitted samples and single lots, use the official moisture result for the lot if the applicant requests an "as is" moisture basis.

- c. <u>Conversion Guidelines for Sublot Testing</u>.
 - (1) When sublot testing is performed on unit trains, lash barges, or ships inspected as single lots, and the applicant does not specify limits for protein, oil, or starch content on an alternate moisture basis, record individual sublot results and calculate CuSum values on the basis of the standard moisture basis. Upon completion of loading, convert the final average protein/oil/starch result to the specified moisture basis. For an "as is" moisture basis, use the official average moisture result in the conversion formula.
 - (2) If a load order specifies limits on the protein, oil, or starch content on a specified moisture basis, convert the individual sublot protein/oil/starch results to the desired moisture basis. For an "as is" moisture basis, use the official sublot moisture result in the conversion formula.

Using two separate columns on the loading log, record the individual sublot results (from the standard moisture basis and specified moisture basis) and apply the CuSum loading plan information (e.g., breakpoints, starting values) to the specified moisture basis results column.

Upon completion of the lot, average the protein/oil/starch results in the standard moisture basis column and convert this value to the applicable protein/oil/starch value on the specified moisture basis. For an "as is" moisture basis, use the official average moisture result in the conversion formula.

Enter the converted protein/oil/starch value as the final "average" under the specified moisture basis column (protein/oil/starch results) on the log. Do not average results from the specified moisture column to obtain a "final" specified moisture basis result.

APPENDIX 4.1 – PERCENT PROTEIN TO PERCENT WET GLUTEN CONVERSION TABLE

PE	RCENTAGE PR	OTEIN (12.0 %	6 mb) TO	PERCE	NTAGE WET GL	UTEN (14.0 %	mb)
Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten
8.01	16.4	8.51	17.9	9.01	19.5	9.51	21.0
8.02	16.5	8.52	18.0	9.02	19.5	9.52	21.0
8.03	16.5	8.53	18.0	9.03	19.5	9.53	21.0
8.04	16.5	8.54	18.0	9.04	19.6	9.54	21.1
8.05	16.6	8.55	18.1	9.05	19.6	9.55	21.1
8.06	16.6	8.56	18.1	9.06	19.6	9056	21.1
8.07	16.6	8.57	18.1	9.07	19.6	9.57	21.2
8.08	16.6	8.58	18.2	9.08	19.7	9.58	21.2
8.09	16.7	8.59	18.2	9.09	19.7	9.59	21.2
8.10	16.7	8.60	18.2	9.10	19.7	9.60	21.2
8.11	16.7	8.61	18.2	9.11	19.8	9.61	21.3
8.12	16.8	8.62	18.3	9.12	19.8	9.62	21.3
8.13	16.8	8.63	18.3	9.13	19.8	9.63	21.3
8.14	16.8	8.64	18.3	9.14	19.9	9.64	21.4
8.15	16.9	8.65	18.4	9.15	19.9	9.65	21.4
8.16	16.9	8.66	18.4	9.16	19.9	9.66	21.4
8.17	16.9	8.67	18.4	9.17	19.9	9.67	21.5
8.18	16.9	8.68	18.5	9.18	20.0	9.68	21.5
8.19	17.0	8.69	18.5	9.19	20.0	9.69	21.5
8.20	17.0	8.70	18.5	9.20	20.0	9.70	21.6
8.21	17.0	8.71	18.6	9.21	20.1	9.71	21.6
8.22	17.1	8.72	18.6	9.22	20.1	9.72	21.6
8.23	17.1	8.73	18.6	9.23	20.1	9.73	21.6
8.24	17.1	8.74	18.6	9.24	20.2	9.74	21.7
8.25	17.2	8.75	18.7	9.25	20.2	9.75	21.7
8.26	17.2	8.76	18.7	9.26	20.2	9.76	21.7
8.27	17.2	8.77	18.7	9.27	20.2	9.77	21.8
8.28	17.3	8.78	18.8	9.28	20.3	9.78	21.8
8.29	17.3	8.79	18.8	9.29	20.3	9.79	21.8
8.30	17.3	8.80	18.8	9.30	20.3	9.80	21.9
8.31	17.3	8.81	18.9	9.31	20.4	9.81	21.9
8.32	17.4	8.82	18.9	9.32	20.4	9.82	21.9
8.33	17.4	8.83	18.9	9.33	20.4	9.83	21.9
8.34	17.4	8.84	18.9	9.34	20.5	9.84	22.0
8.35	17.5	8.85	19.0	9.35	20.5	9.85	22.0
8.36	17.5	8.86	19.0	9.36	20.5	9.86	22.0
8.37	17.5	8.87	19.0	9.37	20.6	9.87	22.1
8.38	17.6	8.88	19.1	9.38	20.6	9.88	22.1
8.39	17.6	8.89	19.1	9.39	20.6	9.89	22.1
8.40	17.6	8.90	19.1	9.40	20.6	9.90	22.2
8.41	17.6	8.91	19.2	9.41	20.7	9.91	22.2
8.42	17.7	8.92	19.2	9.42	20.7	9.92	22.2
8.43	17.7	8.93	19.2	9.43	20.7	9.93	22.2
8.44	17.7	8.94	19.2	9.44	20.8	9.94	22.3
8.45	17.8	8.95	19.3	9.45	20.8	9.95	22.3
8.46	17.8	8.96	19.3	9.46	20.8	9.96	22.3
8.47	17.8	8.97	19.3	9.47	20.9	9.97	22.4
8.48	17.9	8.98	19.4	9.48	20.9	9.98	22.4
8.49	17.9	8.99	19.4	9.49	20.9	9.99	22.4
8.50	17.9	9.00	19.4	9.50	20.9	10.00	22.5

PERCENT	AGE PROTEIN	(12.0 % mb)	ТО	Р	ERCENTAGE V	VET GLUTEN	(14.0 % mb)
Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten
10.01	22.5	10.51	24.0	11.01	25.5	11.51	27.0
10.02	22.5	10.52	24.0	11.02	25.5	11.52	27.1
10.03	22.6	10.53	24.1	11.03	25.6	11.53	27.1
10.04	22.6	10.54	24.1	11.04	25.6	11.54	27.1
10.05	22.6	10.55	24.1	11.05	25.6	11.55	27.2
10.06	22.6	10.56	24.2	11.06	25.7	11.56	27.2
10.07	22.7	10.57	24.2	11.07	25.7	11.57	27.2
10.08	22.7	10.58	24.2	11.08	25.7	11.58	27.2
10.09	22.7	10.59	24.2	11.09	25.8	11.59	27.3
10.10	22.8	10.60	24.3	11.10	25.8	11.60	27.3
10.11	22.8	10.61	24.3	11.11	25.8	11.61	27.3
10.12	22.8	10.62	24.3	11.12	25.9	11.62	27.4
10.13	22.9	10.63	24.4	11.13	25.9	11.63	27.4
10.14	22.9	10.64	24.4	11.14	25.9	11.64	27.4
10.15	22.9	10.65	24.4	11.15	25.9	11.65	27.5
10.16	22.9	10.66	24.5	11.16	26.0	11.66	27.5
10.17	23.0	10.67	24.5	11.7	26.0	11.67	27.5
10.18	23.0	10.68	24.5	11.18	26.0	11.68	27.5
10.19	23.0	10.69	24.6	11.19	26.1	11.69	27.6
10.20	23.1	10.70	24.6	11.20	26.1	11.70	27.6
10.21	23.1	10.71	24.6	11.21	26.1	11.71	27.6
10.22	23.1	10.72	24.6	11.22	26.2	11.72	27.7
10.23	23.2	10.73	24.7	11.23	26.2	11.73	27.7
10.24	23.2	10.74	24.7	11.24	26.2	11.74	27.7
10.25	23.2	10.75	24.7	11.25	26.2	11.75	27.8
10.26	23.2	10.76	24.8	11.26	26.3	11.76	27.8
10.27	23.3	10.77	24.8	11.27	26.3	11.77	27.8
10.28	23.3	10.78	24.8	11.28	26.3	11.78	27.9
10.29	23.3	10.79	24.9	11.29	26.4	11.79	27.9
10.30	23.4	10.80	24.9	11.30	26.4	11.80	27.9
10.31	23.4	10.81	24.9	11.31	26.4	11.81	27.9
10.32	23.4	10.82	24.9	11.32	26.5	11.82	28.0
10.33	23.5	10.83	25.0	11.33	26.5	11.83	28.0
10.34	23.5	10.84	25.0	11.34	26.5	11.84	28.0
10.35	23.5	10.85	25.0	11.35	26.5	11.85	28.1
10.36	23.6	10.86	25.1	11.36	26.6	11.86	28.1
10.37	23.6	10.87	25.1	11.37	26.6	11.87	28.1
10.37	23.6	10.88	25.1	11.38	26.6	11.88	28.2
10.39	23.6	10.89	25.2	11.39	26.7	11.89	28.2
10.39	23.7	10.90	25.2	11.40	26.7	11.90	28.2
10.41	23.7	10.91	25.2	11.41	26.7	11.91	28.2
10.41	23.7	10.92	25.2	11.42	26.8	11.92	28.3
10.42	23.8	10.93	25.3	11.42	26.8	11.93	28.3
10.43	23.8	10.94	25.3	11.44	26.8	11.94	28.3
10.44	23.8	10.95	25.3	11.45	26.9	11.95	28.4
10.45	23.9	10.96	25.4	11.46	26.9	11.96	28.4
10.46	23.9	10.96	25.4	11.46	26.9	11.97	28.4
		10.97	25.4	11.47	26.9	11.98	28.5
10.48	23.9					11.98	
10.49	23.9	10.99	25.5 25.5	11.49	27.0		28.5
10.50	24.0	11.00	25.5	11.50	27.0	12.00	28.5

PERCENTA	GE PROTEIN	(12.0 % mb)	ТО	Р	ERCENTAGE V	WET GLUTEN	I (14.0 % mb)
Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten
12.01	28.5	12.51	30.1	13.01	31.6	13.51	33.1
12.02	28.6	12.52	30.1	13.02	31.6	13.52	33.1
12.03	28.6	12.53	30.1	13.03	31.6	13.53	33.2
12.04	28.6	12.54	30.2	13.04	31.7	13.54	33.2
12.05	28.7	12.55	30.2	13.05	31.7	13.55	33.2
12.06	28.7	12.56	30.2	13.06	31.7	13.56	33.2
12.07	28.7	12.57	30.2	13.07	31.8	13.57	33.3
12.08	28.8	12.58	30.3	13.08	31.8	13.58	33.3
12.09	28.8	12.59	30.3	13.09	31.8	13.59	33.3
12.10	28.8	12.60	30.3	13.10	31.8	13.60	33.4
12.11	28.9	12.61	30.4	13.11	31.9	13.61	33.4
12.12	28.9	12.62	30.4	13.12	31.9	13.62	33.4
12.13	28.9	12.63	30.4	13.13	31.9	13.63	33.5
12.14	28.9	12.64	30.5	13.14	32.0	13.64	33.5
12.15	29.0	12.65	30.5	13.15	32.0	13.65	33.5
12.16	29.0	12.66	30.5	13.16	32.0	13.66	33.5
12.17	29.0	12.67	30.5	13.17	32.1	13.67	33.6
12.18	29.1	12.68	30.6	13.18	32.1	13.68	33.6
12.19	29.1	12.69	30.6	13.19	32.1	13.69	33.6
12.20	29.1	12.70	30.6	13.20	32.2	13.70	33.7
12.21	29.2	12.71	30.7	13.21	32.2	13.71	33.7
12.22	29.2	12.72	30.7	13.22	32.2	13.72	33.7
12.23	29.2	12.73	30.7	13.23	32.2	13.73	33.8
12.24	29.2	12.74	30.8	13.24	32.3	13.74	33.8
12.25	29.3	12.75	30.8	13.25	32.3	13.75	33.8
12.26	29.3	12.76	30.8	13.26	32.3	13.76	33.8
12.27	29.3	12.77	30.9	13.27	32.4	13.77	33.9
12.28	29.4	12.78	30.9	13.28	32.4	13.78	33.9
12.29	29.4	12.79	30.9	13.29	32.4	13.79	33.9
12.30	29.4	12.80	30.9	13.30	32.5	13.80	34.0
12.31	29.5	12.81	31.0	13.31	32.5	13.81	34.0
12.32	29.5	12.82	31.0	13.32	32.5	13.82	34.0
12.33	29.5	12.83	31.0	13.33	32.5	13.83	34.1
12.34	29.5	12.84	31.1	13.34	32.6	13.84	34.1
12.35	29.6	12.85	31.1	13.35	32.6	13.85	34.1
12.36	29.6	12.86	31.1	13.36	32.6	13.86	34.2
12.37	29.6	12.87	31.2	13.37	32.7	13.87	34.2
12.38	29.7	12.88	31.2	13.38	32.7	13.88	34.2
12.39	29.7	12.89	31.2	13.39	32.7	13.89	34.2
12.40	29.7	12.90	31.2	13.40	32.8	13.90	34.3
12.41	29.8	12.91	31.3	13.41	32.8	13.91	34.3
12.42	29.8	12.92	31.3	13.42	32.8	13.92	34.3
12.43	29.8	12.93	31.3	13.43	32.8	13.93	34.4
12.44	29.9	12.94	31.4	13.44	32.9	13.94	34.4
12.45	29.9	12.95	31.4	13.45	32.9	13.95	34.4
12.46	29.9	12.96	31.4	13.46	32.9	13.96	34.5
12.47	29.9	12.97	31.5	13.47	33.0	13.97	34.5
12.48	30.0	12.98	31.5	13.48	33.0	13.98	34.5
12.49	30.0	12.99	31.5	13.49	33.0	13.99	34.5
12.50	30.0	13.00	31.5	13.50	33.1	14.00	34.6

PERCENT	TAGE PROTEIN	(12.0 % mb)	ТО	Pi	ERCENTAGE W	ET GLUTEN	(14.0 % mb)
Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten	Protein	Wet Gluten
14.01	34.6	14.51	36.1	15.01	37.6	15.51	39.1
14.02	34.6	14.52	36.2	15.02	37.7	15.52	39.2
14.03	34.7	14.53	36.2	15.03	37.7	15.53	39.2
14.04	34.7	14.54	36.2	15.04	37.7	15.54	39.2
14.05	34.7	14.55	36.2	15.05	37.8	15.55	39.3
14.06	34.8	14.56	36.3	15.06	37.8	15.56	39.3
14.07	34.8	14.57	36.3	15.07	37.8	15.57	39.3
14.08	34.8	14.58	36.3	15.08	37.8	15.58	39.4
14.09	34.8	14.59	36.4	15.09	37.9	15.59	39.4
14.10	34.9	14.60	36.4	15.10	37.9	15.60	39.4
14.11	34.9	14.61	36.4	15.11	37.9	15.61	39.5
14.12	34.9	14.62	36.5	15.12	38.0	15.62	39.5
14.13	35.0	14.63	36.5	15.13	38.0	15.63	39.5
14.14	35.0	14.64	36.5	15.14	38.0	15.64	39.5
14.15	35.0	14.65	36.5	15.15	38.1	15.65	39.6
14.16	35.1	14.66	36.6	15.16	38.1	15.66	39.6
14.17	35.1	14.67	36.6	15.17	38.1	15.67	39.6
14.18	35.1	14.68	36.6	15.18	38.2	15.68	39.7
14.19	35.2	14.68	36.7	15.19	38.2	15.69	39.7
14.20	35.2	14.70	36.7	15.20	38.2	15.70	39.7
14.21	35.2	14.71	36.7	15.21	38.2	15.71	39.8
14.22	35.2	14.72	36.8	15.22	38.3	15.72	39.8
14.23	35.3	14.73	36.8	15.23	38.3	15.73	39.8
14.24	35.3	14.74	36.8	15.24	38.3	15.74	39.8
14.25	35.3	14.75	36.8	15.25	38.4	15.75	39.9
14.26	35.4	14.76	36.9	15.26	38.4	15.76	39.9
14.27	35.4	14.77	36.9	15.27	38.4	15.77	39.9
14.28	35.4	14.78	36.9	15.28	38.5	15.78	40.0
14.29	35.5	14.79	37.0	15.29	38.5	15.79	40.0
14.30	35.5	14.80	37.0	15.30	38.5	15.80	40.0
14.31	35.5	14.81	37.0	15.31	38.5	15.81	40.1
14.32	35.5	14.82	37.1	15.32	38.6	15.82	40.1
14.33	35.6	14.83	37.1	15.33	38.6	15.83	40.1
14.34	35.6	14.84	37.1	15.34	38.6	15.84	40.1
14.35	35.6	14.85	37.2	15.35	38.7	15.85	40.2
14.36	35.7	14.86	37.2	15.36	38.7	15.86	40.2
14.37	35.7	14.87	37.2	15.37	38.7	15.87	40.2
14.38	35.7	14.88	37.2	15.38	38.8	15.88	40.3
14.39	35.8	14.89	37.3	15.39	38.8	15.89	40.3
14.40	35.8	14.90	37.3	15.40	38.8	15.90	40.3
14.41	35.8	14.91	37.3	15.41	38.8	15.91	40.4
14.42	35.8	14.92	37.4	15.42	38.9	15.92	40.4
14.43	35.9	14.93	37.4	15.43	38.9	15.93	40.4
14.44	35.9	14.94	37.4	15.44	38.9	15.94	40.5
14.45	35.9	14.95	37.5	15.45	39.0	15.95	40.5
14.46	36.0	14.96	37.5	15.46	39.0	15.96	40.5
14.47	36.0	14.97	37.5	15.47	39.0	15.97	40.5
		14.98	37.5	15.47	39.1	15.98	40.5
14.48	36.0						
14.49	36.1	14.99	37.6	15.49	39.1	15.99	40.6
14.50	36.1	15.00	37.6	15.50	39.1	16.00	40.6

16.01 40.7 16.51 42.2 17.01 43.7 17.51 4 16.02 40.7 16.52 42.2 17.02 43.7 17.52 4 16.03 40.7 16.53 42.2 17.03 43.8 17.53 4 16.04 40.8 16.54 42.3 17.04 43.8 17.54 4 16.05 40.8 16.55 42.3 17.05 43.8 17.56 4 16.06 40.8 16.56 42.3 17.06 43.8 17.56 4 16.07 40.8 16.56 42.3 17.06 43.8 17.56 4 16.07 40.8 16.57 42.4 17.07 43.9 17.57 4 16.08 40.9 16.58 42.4 17.09 43.9 17.59 4 16.10 40.9 16.60 42.5 17.10 44.0 17.60 4 16.11 41.0 16.61	
16.02 40.7 16.52 42.2 17.02 43.7 17.52 4 16.03 40.7 16.53 42.2 17.03 43.8 17.53 4 16.04 40.8 16.54 42.3 17.04 43.8 17.54 4 16.05 40.8 16.55 42.3 17.05 43.8 17.55 4 16.06 40.8 16.56 42.3 17.06 43.8 17.56 4 16.07 40.8 16.57 42.4 17.07 43.9 17.57 4 16.08 40.9 16.58 42.4 17.08 43.9 17.58 4 16.09 40.9 16.59 42.4 17.09 43.9 17.59 4 16.10 40.9 16.60 42.5 17.10 44.0 17.60 4 16.11 41.0 16.61 42.5 17.11 44.0 17.61 4 16.13 41.0 16.63	Gluten
16.03 40.7 16.53 42.2 17.03 43.8 17.53 4 16.04 40.8 16.54 42.3 17.04 43.8 17.54 4 16.05 40.8 16.55 42.3 17.05 43.8 17.55 4 16.06 40.8 16.56 42.3 17.06 43.8 17.56 4 16.07 40.8 16.57 42.4 17.07 43.9 17.57 4 16.08 40.9 16.58 42.4 17.08 43.9 17.58 4 16.09 40.9 16.59 42.4 17.09 43.9 17.59 4 16.10 40.9 16.69 42.4 17.09 43.9 17.59 4 16.10 40.9 16.69 42.4 17.09 43.9 17.59 4 16.10 40.9 16.60 42.5 17.10 44.0 17.60 4 16.11 41.0 16.61	5.2
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16.33 41.6 16.83 43.1 17.33 44.7 17.83 4	6.2
16.34 41.7 16.84 43.2 17.34 44.7 17.84 4	6.2
16.35 41.7 16.85 43.2 17.35 44.7 17.85 4	6.2
16.36 41.7 16.86 43.2 17.36 44.8 17.86 4	6.3
	6.3
16.38 41.8 16.88 43.3 17.38 44.8 17.88 4	6.3
16.39 41.8 16.89 43.3 17.39 44.8 17.89 4	6.4
16.40 41.8 16.90 43.4 17.40 44.9 17.90 4	6.4
16.41 41.9 16.91 43.4 17.41 44.9 17.91 4	6.4
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CHAPTER 5 NIRT MONITORING PROGRAM

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5.1 GENERAL INFORMATION

- a. The NIRT monitoring program is designed to monitor the accuracy of official wheat, barley, soybean, and corn testing services. Wheat wet gluten content is determined directly from the NIRT protein, and no additional monitoring is required. TSD will review calibration performance using the existing HRW and HRS protein monitoring sample submissions.
- b. Accuracy is evaluated by comparing field office and specified service point results with TSD results. Field offices with NIRT instruments may conduct supplemental file sample monitoring of specified service points within the circuit in addition to TSD monitoring to evaluate the performance of instruments and technicians.
- c. Monitoring information is used by specified service points to evaluate the performance of their testing program. Field offices use the monitoring information to evaluate their own accuracy as well as the accuracy of the testing service program within their circuit. TSD uses the monitoring information to evaluate the accuracy of individual field locations as well as the entire program.
- d. TSD monitoring will identify service points generating questionable results.
 - Field offices and specified service points must initiate corrective action and follow up whenever wheat, barley, soybean, or corn testing problems are detected.
 - Corrective action and follow-up include investigating, identifying, correcting, and documenting the cause of accuracy problems.
- e. Several methods are utilized to monitor particular elements in the testing process. These methods include review of SRS worksheets, check samples, monitoring file samples (quality control charts), intermarket sample exchanges, and special studies. Procedures regarding these monitoring methods are discussed in the following sections.

5.2 REVIEW OF SRS DAILY BIAS RESULTS

TSD, field offices, and specified service points record the results of daily and weekly standard reference sample checks on SRS worksheets (examples are shown in Chapter 3).

Any instrument in the NIRT Program must have acceptable SRS checks daily before the start of sample analysis (as discussed in Chapter 3). ECT parameters of the SRS bias checks should be immediately reviewed by the responsible person (however named: operator, FO, OA, SSP) for acceptability. This is typically done by looking in the "Calibration" box to see if the "In Tolerance, No Bias Adjustment" box is checked.

- a. <u>Troubleshooting</u>. TSD may request SRS information if a problem is suspected (unusual high or low results, board appeal, foreign compliant, etc.) NIRT coordinators and specified service points must review these worksheets for accuracy and completeness.
- b. <u>Evaluation of Results</u>. NIRT coordinators must evaluate the SRS worksheets to determine if: (1) bias adjustments were completed, when necessary, (2) instrument accuracy was maintained, or (3) bias adjustments were required frequently (which may indicate the need for operator training, instrument repair, or SRS replacement).

5.3 MONITORING FILE SAMPLES

Wheat, barley, and soybean testing accuracy are evaluated through the file sample monitoring system. Official inspection points select and forward samples to TSD. Accuracy is determined by comparing original results with the average of the TSD master instruments' results. If a field office has a local, supplemental monitoring program, official agencies shall also forward samples to the field office location.

- a. When selecting weekly monitoring samples follow the following guidelines.
 - (1) Select monitor samples representing the range of constituents observed during the week. Include a low and a high protein sample each week with three intermediate samples. When applicable, include a high oil and low oil sample.
 - (2) Avoid selecting all samples tested from the same day. When less than five samples of a wheat class or barley subclass are tested during a week, select all samples tested for that class or subclass. Do not make up monitoring samples to fulfill the minimum number of samples. Do not select mixed wheat samples.

- (3) Each dockage-free wheat or barley sample submitted for monitoring must be at least 600 grams unless special arrangements have been made with TSD. Each soybean sample should be at least 800 grams.
- b. Select five samples of wheat per week. Do not submit five samples for each wheat class tested; submit five samples total. The samples should all be the same class. Locations routinely certifying NIRT protein on more than one wheat class should, where practical, rotate weekly submissions among those classes.
- c. Select five samples per barley subclass tested, per week.
- d. Select five samples of soybeans tested, per week.
- e. Field offices and/or TSD may request additional samples for monitoring and/or special study purposes.
- f. <u>Shipping Instructions</u>. Seal the samples in individual 6-mil plastic bags. Mark the sample number and wheat class or barley subclass on each bag using an indelible marker. Place the samples and sample information (monitor submission form) in a suitable package and indicate "NIRT Monitoring Sample" on the package. This will assist in separating protein monitoring samples from other samples received.
- g. <u>Monitoring Results</u>. TSD will email monitoring results to the FOs and OSPs upon completion and review of submitted monitoring sample data. The FOs and OSPs will review the information, and immediately initiate follow-up action when accuracy deficiencies are indicated by monitoring results. If any monitoring results exceed the tolerance limits TSD will work with the respective FO or OSP for follow up testing or troubleshooting.
- h. <u>Evaluating Monitoring Results</u>. Field offices and specified service points will evaluate completed quality control charts to determine if any action limit (tolerance limits, absolute limits, and/or run limit) violations occurred.
 - (1) Action limit violations occurring on the average difference chart generally indicate a bias-related problem. Action limit violations occurring on the range difference chart generally indicate inconsistency due to fluctuating laboratory conditions, failure to follow procedures consistently, instrument problems, or improper instrument slope or bias adjustment. Violations on the range difference chart are more serious than those on the average difference chart because if the results are inconsistent, the average differences are not meaningful.
 - (2) Monitoring field offices and agencies must initiate corrective action when quality control chart rule violations occur. Field office managers must document any action taken to resolve the differences. This documentation includes action taken to identify the cause and extent of the problem and steps to resolve the problem and/or reasons why no further action is necessary. Documentation may be placed directly on the control chart indicating action limit violations.

5.4 WHEAT/BARLEY/SOYBEAN MONITORING CONTROL REPORTS AND CHARTS

- a. <u>General</u>. A NIRT Monitoring Control Report and Chart are visual displays of monitoring data. The report and chart effectively display extreme variations, shifts, and trends. Also, the report and chart illustrate the difference between results while statistically defining expected variability using control limits. These limits are established based on the normal expected variation of results.
- b. <u>Control Charts</u>. The protein/oil monitoring program utilizes Average Difference and Range Difference quality control charts. The average difference chart illustrates the difference between a specified service point's average for five weekly monitoring samples and the TSD's average for the same samples. The range difference chart plots the range of individual sample differences for the corresponding weekly monitoring sample set. Refer to table 5.1 for more information.
 - (1) <u>Average Difference Chart</u>. The average difference chart includes a zero or Center Line (CL), upper and lower Tolerance Limits (TL), and upper and lower Absolute Limit lines (AL).

The center line is the control chart reference point. Points plotted above the center line indicate a positive difference when compared to the monitoring result. Points plotted below the center line indicate a negative difference when compared to the monitoring result.

(a) For wheat, the absolute limit lines are set at \pm 0.20 percent protein from the center line. The tolerance limit lines are set at \pm 0.15 percent protein from the center line. The run limit is set at \pm 0.10 percent protein from the center line.

- (b) For barley, the absolute limit lines are set at \pm 0.25 percent protein from the center line. The tolerance limit lines are set at \pm 0.20 percent protein from the center line. The run limit is set at \pm 0.10 percent protein from the center line.
- (c) For soybean protein, the absolute limit lines are set at ± 0.25 percent protein from the center line. The tolerance limit lines are set at ± 0.20 percent protein from the center line. The run limit is set at ± 0.15 percent protein from the center line.
- (d) For soybean oil, the absolute limit lines are set at \pm 0.20 percent oil from the center line. The tolerance limit lines are set at \pm 0.15 percent oil from the center line. The run limit is set at \pm 0.10 percent oil from the center line.

These tolerances are determined statistically based on the systems actual performance and may be revised from time to time. A violation of any of the established tolerances means that there is less than one chance in one hundred that the observed error level occurred due to random chance. Therefore, it is very likely that a correctable problem exists. Average and Range Difference results are shown in chart 5.1.

- (2) Range Difference Chart. The range difference chart indicates how much difference is observed within a set of monitoring samples.
 - (a) For wheat, the absolute limit line is set at 0.60 percent protein. The tolerance limit line is set at 0.40 percent protein.
 - (b) For barley, the absolute limit line is set at 0.70 percent protein. The tolerance limit line is set at 0.50 percent protein.
 - (c) For soybeans, the absolute limit line is set at 0.80 percent protein. The tolerance limit line is set at 0.40 percent protein.
 - (d) For soybeans, the absolute limit line is set at 0.60 percent oil. The tolerance limit line is set at 0.45 percent oil.

TABLE 5.1 – NIRT MONITORING ACTION LIMITS

	Av	erage Difference Ch	Range Difference Chart		
Violation If:	Average Difference exceeds limit on any run Average Difference exceeds limit on 2 consecutive runs		(a) 4 of 5 consecutive runs are all either + or - and (b) all 4 runs exceed limit	Range of Differences exceeds limit	Range of Differences exceeds limit on 2 consecutive runs
Constituent	Absolute Limit	Tolerance Limit	Run Limit	Absolute Limit	Tolerance Limit
Wheat Protein	0.20	0.15	0.10	0.60	0.40
Barley Protein	0.25	0.20	0.10	0.70	0.50
Soybean Protein	0.25	0.20	0.15	0.80	0.60
Soybean Oil	0.20	0.15	0.10	0.60	0.45

c. <u>Plotting Control Charts</u>. Control charts are generated using the data listed in the Monitoring Control Report as seen in Figure 5.1.

FIGURE 5.1 - EXAMPLE NIRT MONITOR CONTROL REPORT



Official Agency Name Location Name SSP Number

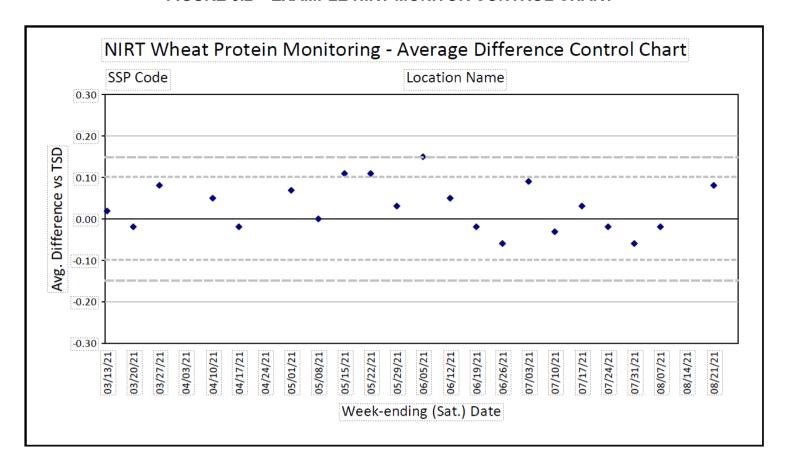
Report Notes:
1. Reports are issued once per week
2. A report is issued only if TSD has completed a monitor set since last report issue date
3. Table and charts are based on last six months data

Six digit	WE Date	Count	Class	SP Average	TSD Average	SP minus TSD	Range
XXXXXX	8/27/2021	5	000	11.17			
XXXXXX	8/21/2021	5	000	11.02	10.94	0.08	0.09
XXXXXX	8/7/2021	5	000	10.51	10.53	-0.02	0.28
XXXXXX	7/31/2021	5	000	10.84	10.90	-0.06	0.29
XXXXXX	7/24/2021	5	000	10.72	10.74	-0.02	0.30

- (1) <u>Sequential Plotting</u>. Control charts depict how a process or system is varying over time. In order for the results to be meaningful, data is plotted in the sequence in which the original results were obtained. That is, when plotting the Average/Range chart, samples originally tested during week 3 are plotted after samples originally tested during week 2, but before samples originally tested during week 4 (see Figures 5.1 and 5.2).
- (2) <u>Average Difference</u>. The difference between the original average result and the monitoring average results is obtained by subtracting the monitoring office result from the original protein result.
- (3) Range Difference. The difference between the original result and the monitoring result is determined for each individual sample monitored within a sample set. The range difference value that is plotted is the difference between the smallest and the largest difference observed in one set. If these two differences are of the opposite sign, add the magnitudes of the two numbers. The range difference is calculated and plotted on the range difference chart.
- (4) <u>Plotted Results</u>. The difference between the monitoring average result and the original result obtained from step 2 is plotted on the average difference chart. The positive differences (original results higher in protein then the monitoring results) are plotted above the center line. Negative differences (original results lower in protein then the monitoring results) are plotted below the center line. Results having no difference (original result is identical to monitoring result) are plotted on the center line.
- d. <u>Reviewing Control Charts—Action Limits</u>. Protein testing problems are indicated on the control charts either by a large difference between the average protein results or a consistent pattern of smaller differences on a series of average results. Three action limits are used for rapid identification of protein testing problems through interpretation of the control chart. Refer to figure 5.2 for more information.
 - (1) <u>Absolute Limit (AL)</u>. This action limit is intended to identify excessive differences between results and indicates a potential protein testing problem. An absolute limit violation occurs if any plotted value is equal to or greater than the absolute limit lines.
 - (2) <u>Tolerance Limit (TL)</u>. This action limit controls the number of consecutive data sets with a large difference between original and monitoring results but not so large as to exceed the absolute limit. An average difference limit violation occurs if two consecutive data sets are either equal to or above the upper tolerance limit line or both equal to or below the lower tolerance limit line. A range difference tolerance limit violation occurs if two consecutive data sets are equal to or greater than the tolerance limit line.
 - (3) Run Limit. This action limit controls the number of consecutive comparisons which are all above or all below the average difference chart center line (CL).

A violation occurs if four out of five consecutive results are either all above or all below the CL, and the average difference from the center line for these four results exceed the applicable limit from table 5.1. Run limits do not apply to the range difference chart.

FIGURE 5.2 - EXAMPLE NIRT MONITOR CONTROL CHART



5.5 COLLABORATIVE CHECK SAMPLES AND SPECIAL STUDIES

a. <u>Collaborative Check Samples</u>. Collaborative check samples may be initiated by TSD for cross-checking other data. TSD will select and send enough of each sample selected so that specified service points can retain a portion for rechecking purposes. Upon receipt, participants must complete the analysis and report the data within 5 working days.

Use the forms provided with the samples to report analysis results. Retain a copy of the completed form and return the original immediately to TSD. Results from all locations included in the collaborative study will be compiled by TSD and reported participating field offices and specified service points.

b. <u>Special Studies</u>. TSD may initiate and conduct special studies. These studies are designed for a specific purpose (i.e., resolving differences either within or between markets, evaluating calibration performance, or updating the calibrations).

When special studies are initiated by TSD, it is required that all participants (as designated by TSD) respond with utmost priority, as these are normally of an urgent nature and an expedient resolution of the problem is essential.

5.6 INTERMARKET SAMPLE EXCHANGE

An intermarket sample exchange helps isolate protein and/or oil differences between inspection points. Protein and oil testing laboratories will determine protein and oil results on separate portions obtained from the same sample. Protein and oil results arethen compared to determine if any significant differences exist.

There are no restrictions as to which offices may exchange samples. Specified service points are encouraged to exchange samples with other specified service points and field offices for the purpose of resolving intermarket inspection differences. A copy of the results of the exchange must be provided to the field office and/or TSD for review if they were not participants in the exchange.

CHAPTER 6 REVISION HISTORY

CHANGE NO. 8:	MARCH 25, 2022	6-1
CHANGE NO. 7:	MARCH 24, 2008	6-1
CHANGE NO. 6:	DECEMBER 18, 2006	6-1
CHANGE NO. 5:	JULY 25, 2005	6-1

CHANGE NO. 8: MARCH 25, 2022

The NIRT Handbook was revised and reformatted for to incorporate FGIS's agency move to AMS, contact information, and the inclusion of information for the Foss NIRT model NOVA, as well as general revisions and formatting.

Chapter 1 was updated with new definitions, tables, and responsibilities. Specific changes were made to sections 1.2, 1.3, and 1.4.

Chapter 3 was updated to standardize the SRS weights for the Foss 1241 and NOVA. Specific changes were made to section 3.1.a.

Chapter 4 was updated to include addition of a table, updating certificate language and notes for further clarification. Specific changes were made to sections 4.1, 4.6, and 4.7.a(5).

Chapter 5 was updated to revise old processes to align with current processes, remove soybean and corn check samples. Specific changes were to sections 5.2, 5.3.f-g, 5.4.c-d (figures updated to new format and removed old), and 5.5.b.

CHANGE NO. 7: MARCH 24, 2008

The NIRT Handbook Chapter 3 was revised and reformatted to remove the testing requirements for analyzing two sets of Standard Reference Samples (SRS) if an instrument has been out of service more than two weeks and that instruments be kept within Level III bias tolerance at all times for approved calibrations. Additional revisions include minor editorial changes to certification statements for Soybean Protein and Oil.

Specifically, changes were made to Chapter 3 section 3.1, d (2), 3.2, a (2), 3.3, a. (2), 3.4, a. (2), 3.5, a (2); Chapter 4 section 4.7, d, (1), (2), & (3), and Chapter 5 section 5.4, a (3).

CHANGE NO. 6: DECEMBER 18, 2006

This updated the handbook to include instructions for testing wet gluten; and the wheat worksheets in Chapter 3 to include wet gluten constants. Additionally, the handbook nowprovides appropriate certificate statements to use for protein, wet gluten, oil, and starch.

This handbook superseded:

- NIRT Handbook, dated 7-25-05.
- Grain Inspection Handbook, Book IV, Section 3.8, Certificate Statements: Proteinand Oil, dated 1/1/91
- Program Notice FGIS-PN-01-16, Protein Instrument Approval, dated 11-14-01
- Program Notice FGIS-PN-05-03, NIRT Wheat Protein Calibration, dated 03-24-05
- Program Notice FGIS-PN-05-06, NIRT Barley Protein Program, dated 06-20-05
- Program Notice FGIS-PN-06-03, NIRT Wet Gluten Program, dated 4-24-06
- Program Notice FGIS-PN-06-09, NIRT Monitoring Program Wheat and Soybeans, dated 07-25-06

CHANGE NO. 5: JULY 25, 2005

The NIRT Handbook has been revised to incorporate changes made to the official NIRTtesting program since May 1, 2005. The revisions include procedures for analyzing barley protein.