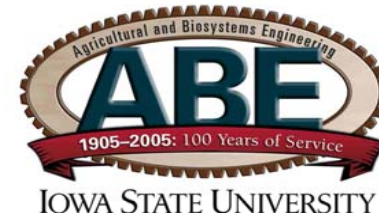


Ongoing Quality Control Program for NIRS-based Service and Research Laboratory



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World Grains Summit: Foods and Beverages
September 17-20, 2006 • San Francisco, California U.S.A.



ISU Grain Quality Laboratory

- ISU-GQL performs Near Infrared (NIRS) instrument calibration.
- ISU- GQL provides services of measuring the chemical composition and biological properties of agricultural products using NIRS instruments.
(e.g. corn and soybean, N ~ 20,000 samples/year)
- ISU-GQL has many brands of NIRS instruments:

Infratec, Bruins Omega, Perten DA7200, Zeiss Corona, NIRS6500.

Why a Quality Control Program?

- ISU-GQL wants to meet quality standard based on ISO 17025.
- Quality Control is specifically stated in ISO 17025 section 5.9.
- Other sections of ISO 17025 that may support Quality Control:
 - Section 4.12 - documentation
 - Section 5.2 - training
 - Section 5.3 - laboratory facilities
 - Section 5.5 - equipment

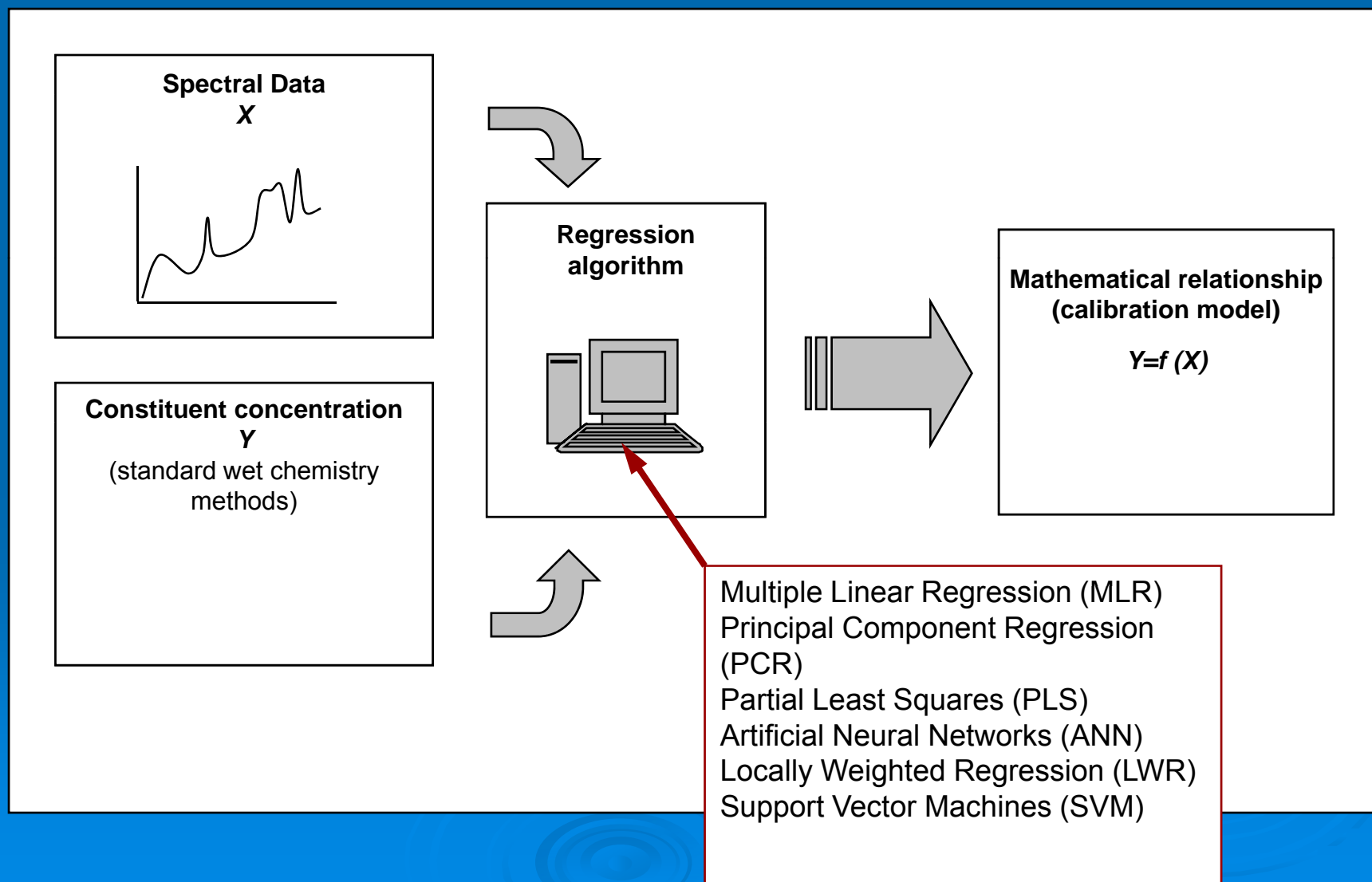
Project Objective

To develop an auditable quality control program for NIRS analyzers, supporting equipment and laboratory room conditions.

NIRS Quality Control Program



NIRS Calibration



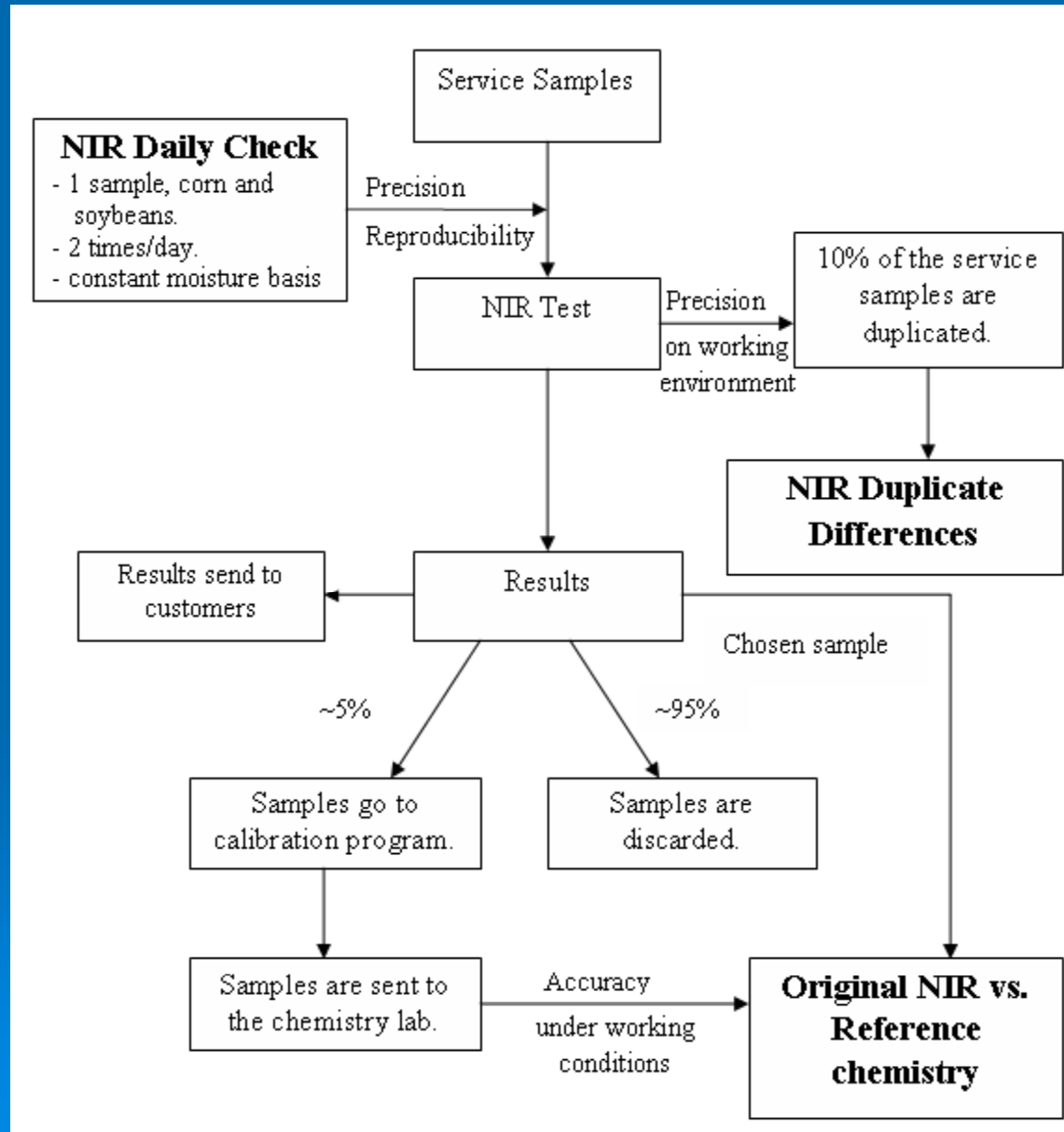
AACC Method 39-00 Validation

- Standard Error of Prediction (SEP) on new samples:

$$SEP = \sqrt{\frac{\sum (D - \bar{D})^2}{(n-1)}}$$

- Standard Error of Precision $\leq 0.33 * SEP$
- Transfer Std Error $< 0.50 SEP$

ISU-GQL NIRS Quality Control



Control Charts

- NIRS Daily Check (95% confidence level):
 - Method 1: Shewhart control chart
UCL/LCL = average \pm 2 standard deviation
 - Method 2: AACCC Method 39-00
UCL/LCL = 10-point moving average \pm 2 (0.33 SEP)

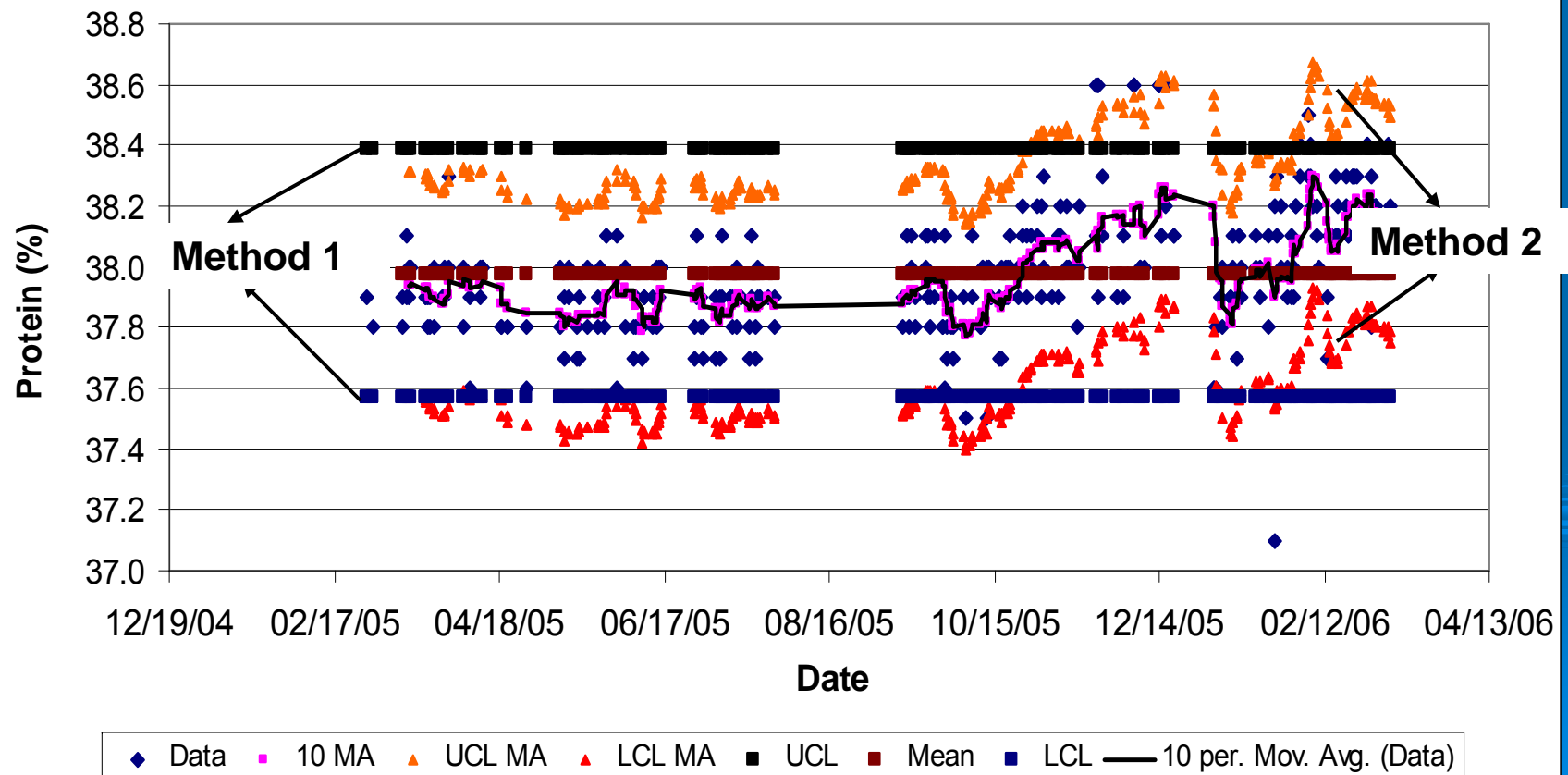


Control Charts

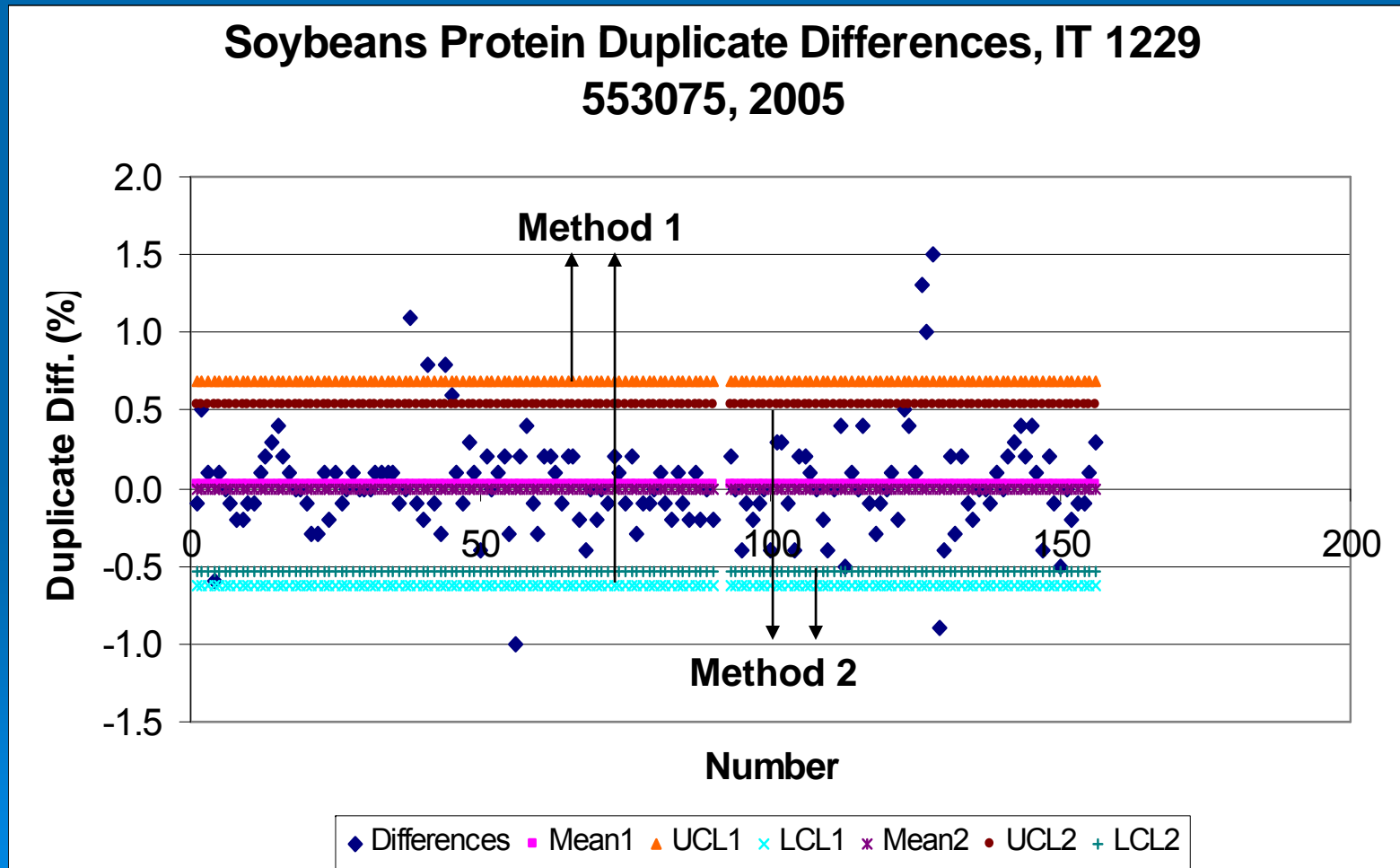
- NIR Duplicate Differences (95% confidence level):
 - Method 1: Shewhart control chart
UCL/LCL = average of differences \pm 2 standard deviation of differences.
 - Method 2: AACCC Method 39-00
UCL/LCL = \pm 2 ($\sqrt{2}$ * 0.33 SEP).
- Original NIR values vs. References (95% confidence level)
 - $Y = X$ as center line
 - $Y = x \pm 2 \text{ SEP}$ as UCL and LCL

NIR Daily Check Control Chart

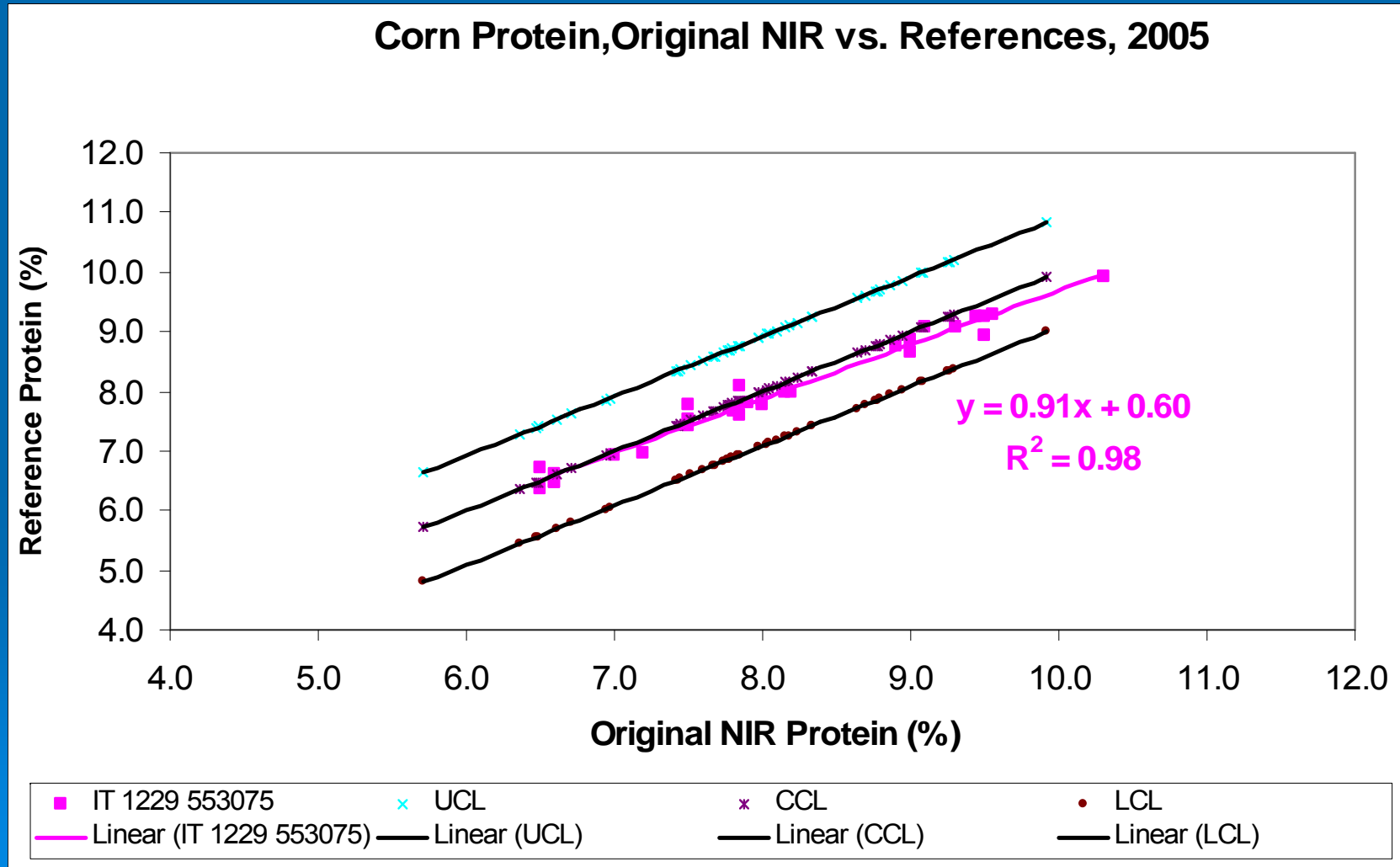
Soybeans Protein Daily Check, IT 1241 12410350, Sample 20010461



NIR Duplicates Differences Control Chart



Original NIR values vs. References Control Chart



Online Control

Microsoft Excel - soybeans service template

| AN13 | A | B | C | AD | AE | AF | AG | AH | AI | AJ | AK | AL | AM | AN |
|------|----------------|-------------------------------|---|----|------------------------|----|-------|----|-------|----|-------------|--------|----|----|
| 6 | Source: | Roland | | | 0.04 | | -0.01 | | 0.01 | | 0.01 | | | |
| 7 | Date: | 12/2/2005 | | | 0.9 | | 0.6 | | 0.5 | | 0.1 | | | |
| 8 | Instrument: | 553792 | | | -0.3 | | -0.6 | | -0.3 | | -0.1 | | | |
| 9 | Feeder | Flow | | | 0.19 | | 0.23 | | 0.15 | | 0.05 | | | |
| 10 | Calibration ID | SB200301 | | | | | | | | | | | | |
| 11 | Filename: | C:\Documents and Settings\... | | | | | | | | | | | | |
| 12 | Moist. Basis: | 13 | | | | | | | | | | | | |
| 13 | Date Ended: | | | | Differences> | | | | | | | | | |
| 14 | ID Number: | 972 | | | | | | | | | | | | |
| 15 | | ISU ID | | | 96 | 4 | 96 | 2 | 96 | 1 | 96 | 22 | | |
| 16 | | | | | 4.17% | | 2.08% | | 1.04% | | Sats | 22.92% | | |

Microsoft Excel - soybeans service template

| AN6 | A | B | C | D | E | F | G | H | I | J | K | L | M |
|-----|----------------|-------------------------------|---------------------|--------------------|------------|---------------|--------------|-----------------|----------------|------------|--------------|-------------|-------------|
| 6 | Source: | Roland | | | | | | 7.89 | 35.66 | 19.45 | 4.70 | #DIV/0! | 55 |
| 7 | Date: | 12/2/2005 | | | | | | 9.8 | 39.0 | 22.0 | 5.0 | 0.0 | 56 |
| 8 | Instrument: | 553792 | | | | | | 6.2 | 31.9 | 17.2 | 4.4 | 0.0 | 57 |
| 9 | Feeder | Flow | | | | | | 0.59 | 1.20 | 0.66 | 0.11 | #DIV/0! | 0 |
| 10 | Calibration ID | SB200301 | | | | | | | | | | | |
| 11 | Filename: | C:\Documents and Settings\... | | | | | | | | | | | |
| 12 | Moist. Basis: | 13 | | | | | | | | | | | |
| 13 | Date Ended: | | | | | | | | | | | | |
| 14 | ID Number: | 972 | | | | | | | | | | | |
| 15 | | ISU ID | | | | | | | | | | | |
| 16 | | | | | | | | | | | | | |
| 17 | Cal | Date | Universal ID | Command | ID1 | Plot # | ID | Moisture | Protein | Oil | Fiber | Sats | Lino |
| 417 | 12/6/2005 | 200502007996 | | | | | 200502007996 | 8.9 | 34.5 | 19.2 | 4.9 | | 53 |
| 418 | 12/6/2005 | 200502008002 | | | | | 200502008002 | 8.4 | 35.5 | 19.8 | 4.7 | | 54 |
| 419 | 12/6/2005 | 200502007995 | | | | | 200502007995 | 8.2 | 34.9 | 19.0 | 4.8 | | 53 |
| 420 | 12/6/2005 | 200502007998 | | | | | 200502007998 | 8.6 | 33.0 | 19.9 | 4.9 | | 52 |
| 421 | 12/6/2005 | 200502007999 | | RUN AGAIN ! | | | 200502007999 | 8.6 | 36.4 | 19.2 | 4.7 | | 56 |
| 422 | 12/6/2005 | 200502007993 | | | | | 200502007993 | 7.9 | 34.7 | 20.1 | 4.7 | | 54 |
| 423 | 12/6/2005 | 200502007992 | | | | | 200502007992 | 7.9 | 33.7 | 19.6 | 4.9 | | 53 |
| 424 | 12/6/2005 | 200502007990 | | | | | 200502007990 | 8.0 | 35.1 | 18.9 | 4.8 | | 54 |
| 425 | 12/6/2005 | 200502007989 | | | | | 200502007989 | 8.4 | 36.1 | 19.1 | 4.7 | | 56 |

Tolerance Setting for Method 2

| Item | SEP | Type | Cum. Ave | 95% CI | 0.33* SEP | 95% CI | SED | 95% CI |
|--|------|------|-------------|-------------|-------------|-------------|-------------|-------------|
| Base calibration | 0.57 | SECV | | | | | | |
| Base Calibration Temp + Stabilization | 0.55 | SECV | | | | | | |
| Original Calibration 2004 | 0.47 | SECV | | | | | | |
| Base Calibration Temp + Stabilization (Val 2004) | 0.32 | SECV | | | | | | |
| Validation 553075 (Crop 2004) | 0.38 | SEP | | | | | | |
| Validation 1241350 (Crop 2004) | 0.48 | SEP | 0.46 | 0.92 | 0.15 | 0.30 | 0.21 | 0.43 |

Supporting Equipment Quality Control Program

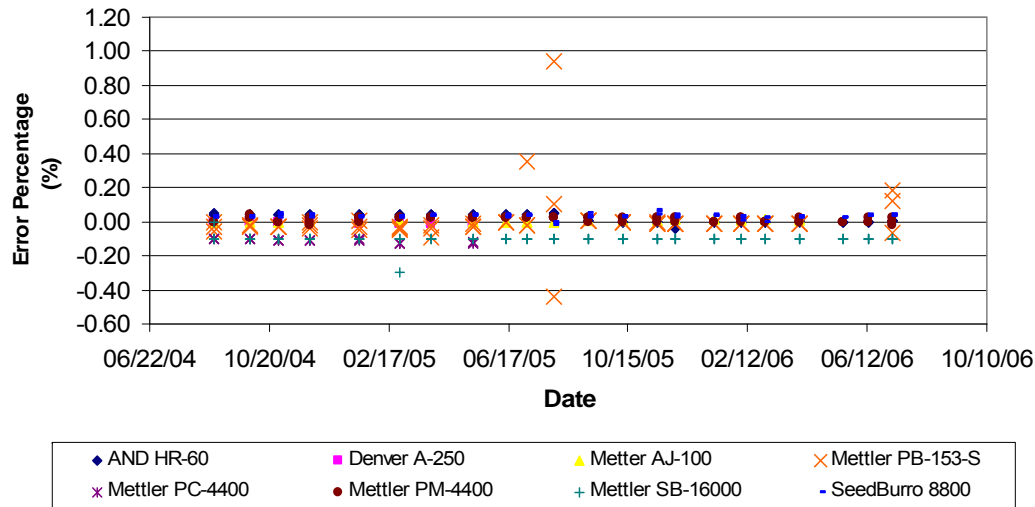


Equipment Quality Control

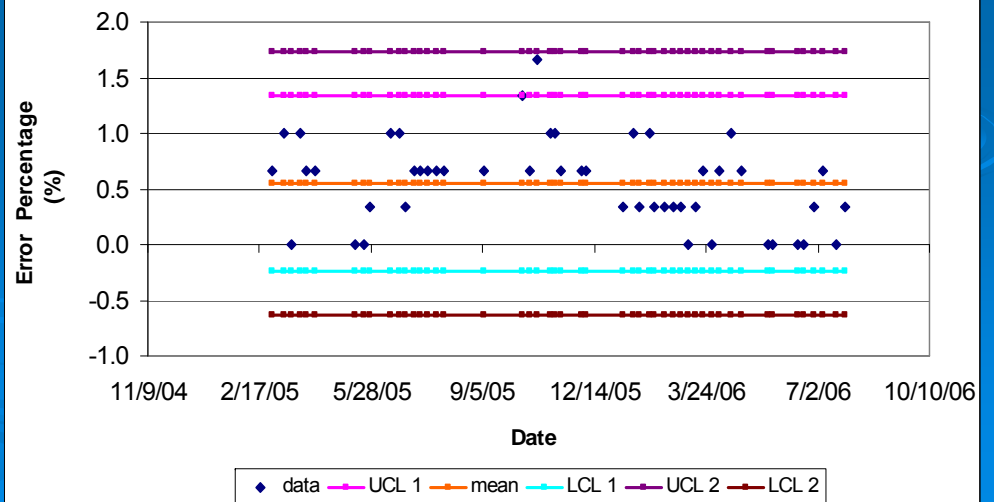
| Item | Quality Control | Documentation | Comments |
|--|--|------------------------------|---|
| Balances (7) | Chart of Date vs. Error Percentage | Lab notebooks Spreadsheet | Monthly single point check Yearly full scale check Monthly printed chart Quarterly updated report |
| Thermometers (2) Infrared Mercury | 95% and 99% CL of Schewhart control chart Percentage differences of IR from Mercury Boiling Water, room and refrigerator temperatures | Lab notebooks Spreadsheet | Monthly check Monthly printed control chart Quarterly updated report |
| Divider (2) Rotary Boerner | 95% and 99% CL of Schewhart control chart Percentage differences between known and measurement 1000 gram of corn seeds | Spreadsheet Lab notebooks | Monthly check Monthly printed control chart Quarterly updated report |
| Seed counter (1) | 95% and 99% CL of Schewhart control chart Percentage differences between known and measurement Corn and soybeans | Lab notebooks Spreadsheet | Single point weekly check (300 seeds) Yearly full scale check Monthly printed control chart Quarterly updated report |
| Grain Test Weight (kettle and GAC) | Chart Date vs. Test Weight Quart/Pint Chart Date vs. Test Weight GAC 2000/2100 TW corrected= TW original+ (0.25*(current moisture-15%)) | Lab notebooks Spreadsheet | Weekly check Quarterly printed chart Quarterly updated report |
| Water Test Weight | 95% and 99% CL of Schewhart control chart (Percentage differences of known and measurement) Quart= 1099.98 gram water Pint= 550.68 gram water | Lab notebooks Spreadsheet | Yearly check Yearly printed chart Yearly updated report |

Equipment Charts

Balances Q/C

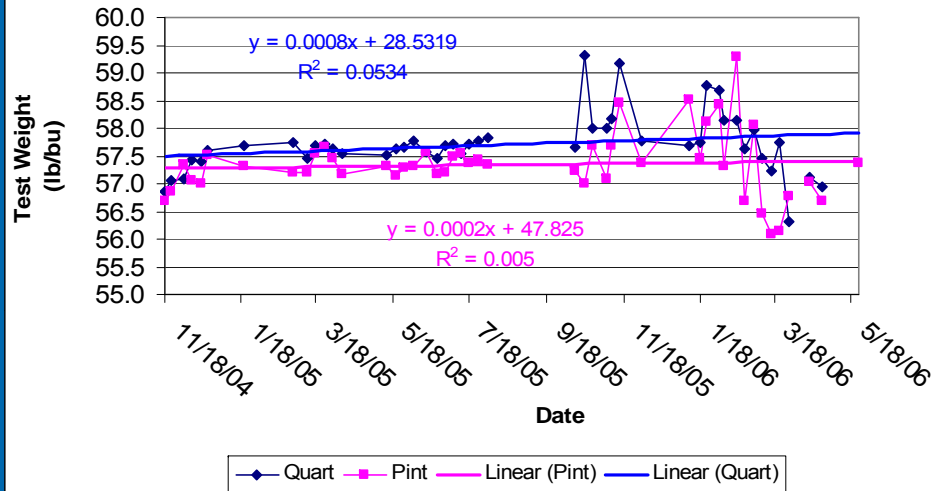


Seed Counter Q/C, Soybeans (300 seeds)

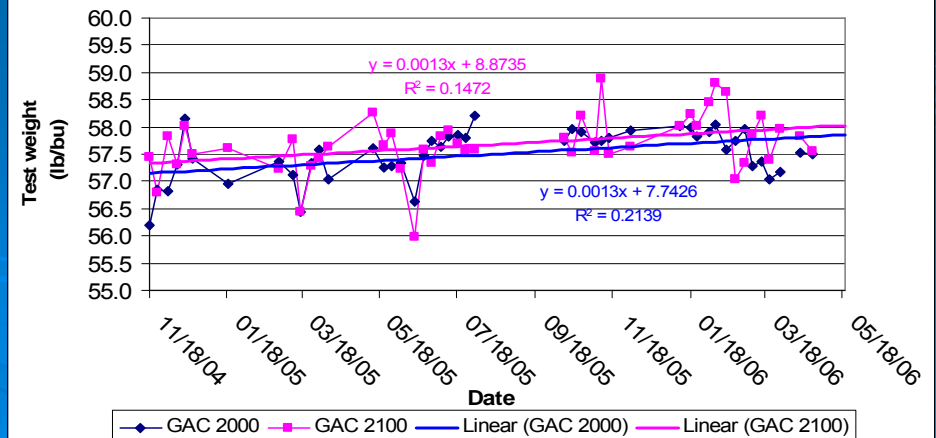


Equipment Charts

Quart/Pint Test Weight Q/C - Corn, Sample 20040049



GAC 2000/2100 Test Weight Q/C - Corn, Sample 20040049



Laboratory Climate

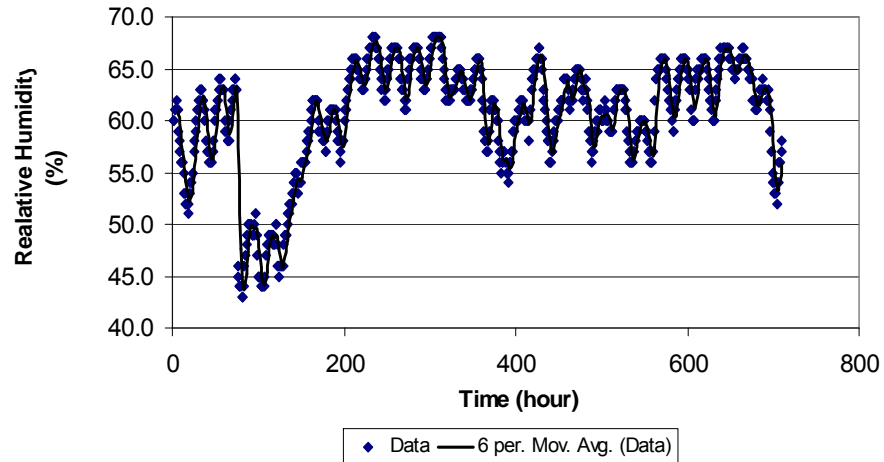


Climate

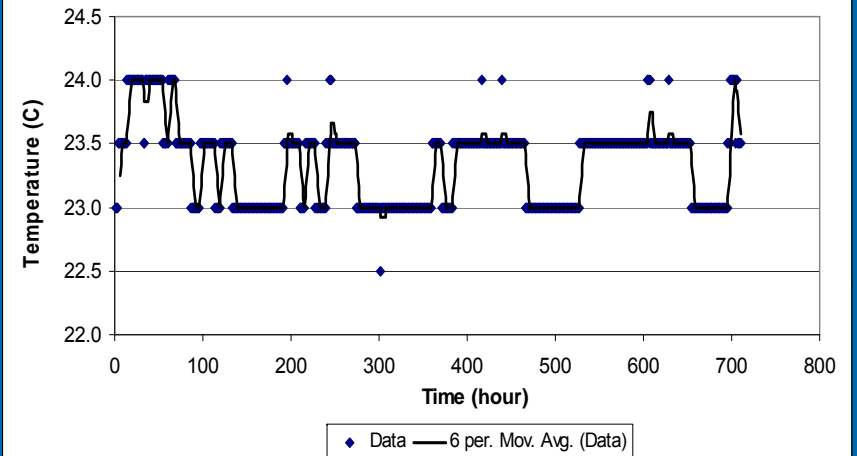
- Climate:
 - Temperature
 - Humidity
 - Pressure
- Climate software capture data hourly.
- Data are download monthly.
- Documentation: spreadsheet.
- Chart:
 - Chart of time vs. temperature.
 - Chart of time vs. humidity.
 - Chart of time vs. pressure.

Monthly Climate

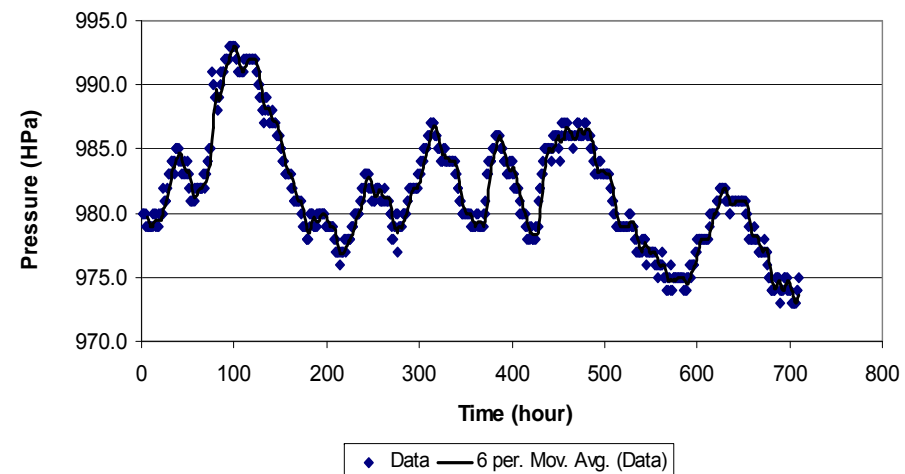
Humidity, July 2006



Temperature, July 2006



Pressure, July 2006



Laboratory Procedures

- Header:
 - File path
 - Original version, by
 - Last revision, by
 - Next scheduled revision
 - ISO 17025 Reference Section
- Procedure title
- Background
- Objectives
- Operation and data collection
- Quality control and data handling
- The use of data
- Control chart guidelines
- Evaluation and monitoring
- Corrective Actions

\\Grainbin\qcdata\Lab Procedures\QC Procedures\Online Procedure\Duplicate Summary.doc 1/3
Original Version: 05-01-05 By: MS Last Revision: 05-01-06; By: MS Next Scheduled Revision: 05-01-07
ISO 17025 Reference Section(s): 4.9, 4.11, 4.15, 5.9

Grain Quality Laboratory Iowa State University

PROCEDURE: **NIR Instrument Duplicates**

Background:

The NIR instruments especially the instruments that are used for the services need to be checked for their precision and reproducibility. The ten percent of measurements using NIR instruments are duplicated to measure the reproducibility over time

Objective:

To measure the reproducibility over time of the NIR instruments used for the services.

OPERATION AND DATA COLLECTION

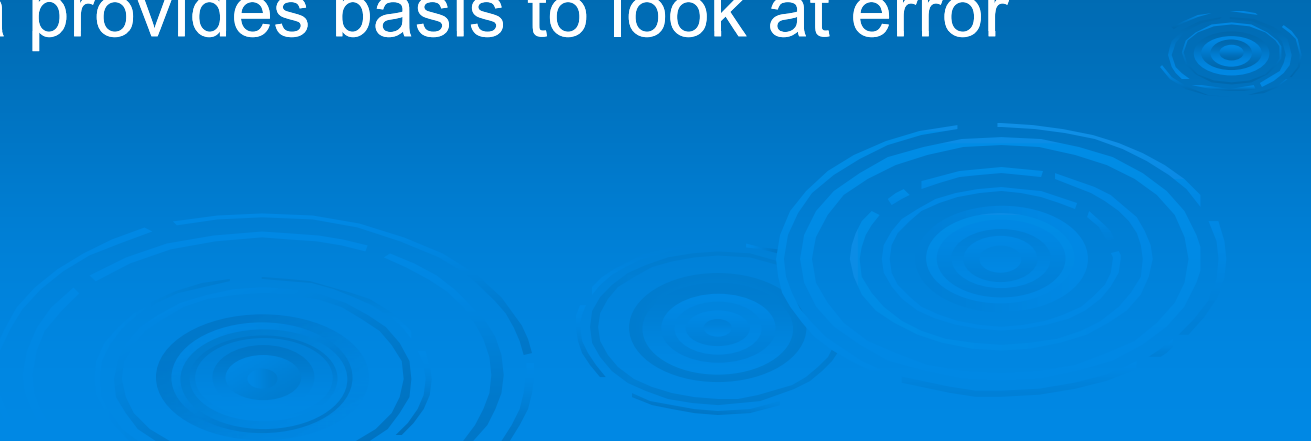
1. Open the standard service template found in
\\Grainbin\Service\200X\Corn\CNDATAFORMDX.xls for corn -or-
\\Grainbin\Service\200X\Soybeans\sbDATAFORMDX.xls for soybeans.
"X" is the last digit of the year.
2. "Save As" the spreadsheet in the correct folder for corn or soybeans using the following naming convention: \R cn (or sb) project descriptor set number.
3. Complete the date and source of the test for every line of data.
4. Run samples, collect duplicate data at least every tenth sample. Confirm the number of duplicates to be taken with the supervisor. Duplicate data records update automatically on the Dup Collection tab.
5. Save the spreadsheet usually after every sample.

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:

1. Open the duplicate summary from the previous year at \\Grainbin\QCdata\Duplicate Summary\XXX, copy them to the new file, save as **Duplicate Corn** or **Duplicate Soybeans**, put them to the new folder, name the folder using the respective year.
2. Delete the data except the first data to keep the formula.
3. Collect the duplicate data from service file at :
\\Grainbin\Service\XXXX\Corn
\\Grainbin\Service\XXXX\Strip Plots\Corn
\\Grainbin\Service\XXXX\Strip Plots\Soybeans
\\Grainbin\Service\XXXX\Soybeans

Conclusion

- Control charts using fixed tolerances that derived from SEP as described in AACCC Method 39-00 are more appropriate than Shewhart control charts: Fixed tolerances come from the expected performance of NIRS instruments.
 - Equipment quality control can improve consistency of data generated by lab.
 - Climate data provides basis to look at error patterns.
- 

Conclusion

- ISU-GQL satisfies some requirements stated in ISO 17025 : quality control, documentation, laboratory facilities, and equipment.
- Lab data generated by ISU-GQL is prepared for third party audit.

QUESTIONS ?

