

ASTM INTERNATIONAL
Helping our world work better



ASTM Proficiency Testing and Standards for Distillers Dried Grains

Distillers Grains Symposium
St. Louis

Chris McCullough

ASTM International
General Manager, Program Development, Laboratory Services

November 13, 2024
www.astm.org



ASTM Proficiency Testing Programs

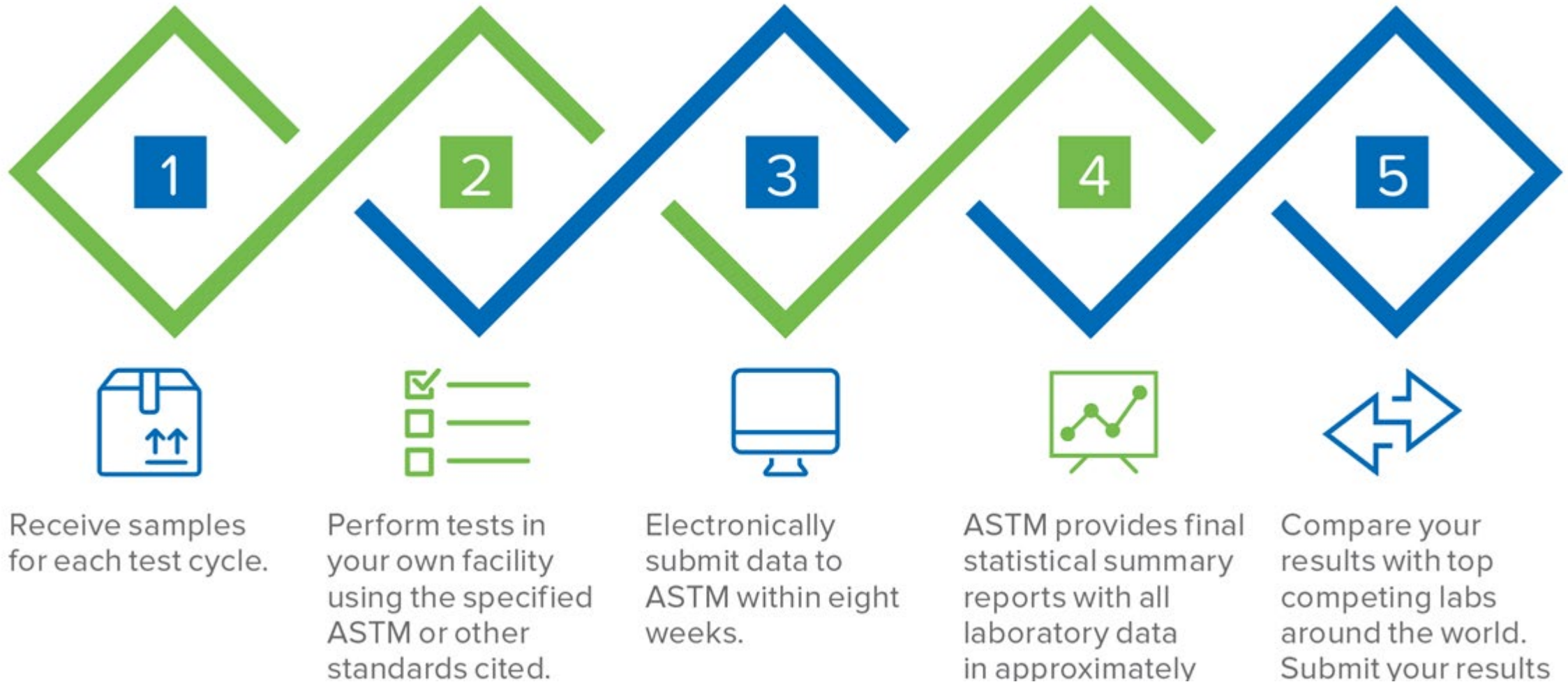
- Statistical quality control tool - assess your lab's performance in conducting test methods
- Open to all labs regardless of location – 52% of participating labs for ASTM PTP outside U.S.
- Thousands of participants
- No need to be an ASTM member
- Programs run all year – ample time for accreditation needs
- ISO/IEC 17043 Accredited (www.astm.org/ptp)

ISO/IEC 17025 Accreditation

- Many bodies require participation in at least one PTP to achieve initial accreditation
- Ongoing participation to maintain accreditation – have a PT plan!
- Check with your accreditation body for full requirements and questions (A2LA, etc.)



How it works



Data Entry and Submission



- Base Number
- Color
- Density
- Evaporation Loss
- Flash Point
- Viscosity, Apparent, HTHS
- Nitrogen
- Pour Point
- Saponification Number
- Shear Stability
- Sulfur
- Viscosity Kinematic
- Viscosity, Low Temperature
- Viscosity, Low Temperature Scanning Brookfield
- Viscosity, Tapered Bearing
- Viscosity, Tapered Plug
- Volatility, Calculated
- Water
- General Comments

Acid Number, A, 125 mL, Buffer EP

Report buffer end point to 3 significant figures mg KOH/g

mg KOH/g

Acid Number, A, 60 mL, inflection EP

Report inflection end point to 3 significant figures mg KOH/g

mg KOH/g

Acid Number, A, 60 mL, Buffer EP

Report buffer end point to 3 significant figures mg KOH/g

mg KOH/g

D974-14e2

(Report values <0.02 mg KOH/g as "<0.02." Report values \geq 0.02 and <1.0 to the nearest 0.01 mg KOH/g. Report values \geq 1.0 to the nearest 0.10 mg KOH/g.)

Acid Number, by Color Indicator

mg KOH/g

Dashboard - Laboratory



Home Participants ▾ Users Samples ▾ Reports ▾ Test Results ▾

Lab Reports

Company: Lab: Program: Program Cycle:

Show entries

Test	Method	Report	Lab Status
Water and Sediment	All	Numeric	
Water and Sediment	D1796	Numeric	Accepted
Water and Sediment	D6304	Numeric	Accepted
Water and Sediment	D2709	Category	Accepted
Water and Sediment	D2709	Numeric	Rejected
Acid Number	All	Numeric	
Acid Number	D974	Numeric	Rejected
Acid Number	D664	Numeric	Accepted

Reports for Download - Program



Results Table

Results Table and Z-Scores
 HC Type, Aromatics, Oxy-Free by D1319-19 (vol %)
 No 2 Diesel Fuel - DF21910

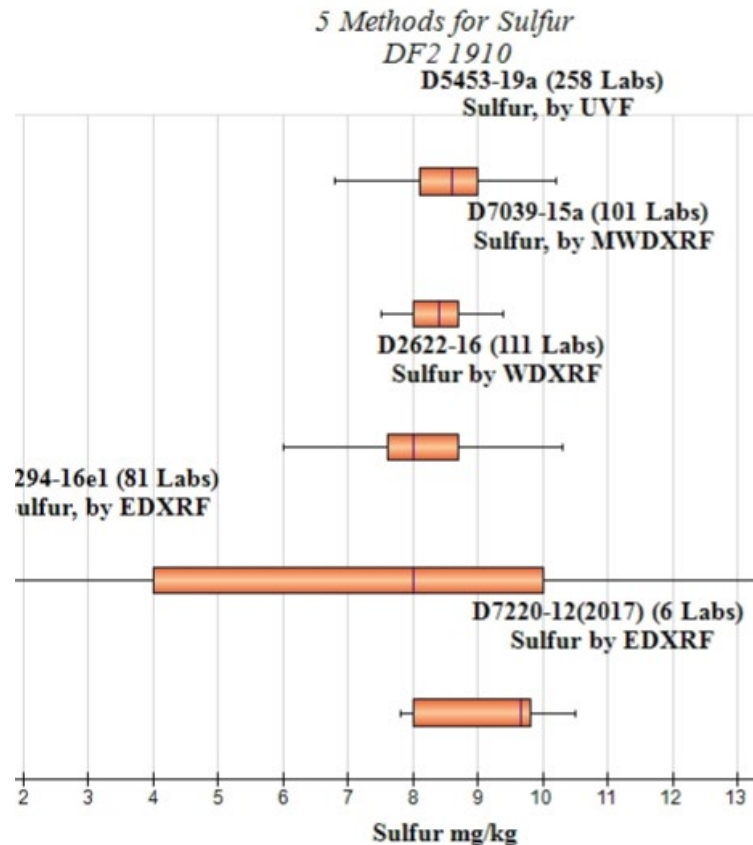
sult	Dev	Z 1910	Notes	1906	1902	1810	1806	1802	1710	Z Co
-0.09	-0.06			-0.25	-0.74	0.13	-0.87	-0.31	0.01	
-5.29	-3.25	1,2,3		-1.82	NDS	NDS	NDS	NDS	NDS	
1.71	1.05			0.49	0.93	-0.78	-0.19	2.72	NDS	
0.01	0.00			-0.06	0.72	1.32	0.12	1.49	0.17	
0.21	0.13			2.43	2.18	1.84	1.29	1.29	-0.83	
-0.79	-0.49			-0.25	NDS	-0.39	NDS	NDS	NDS	
2.01	1.23			NDS	-0.95	-0.50	0.91	1.21	0.09	
0.51	0.31			0.58	-0.95	-0.33	0.53	0.49	NDS	
-2.99	-1.84			NDS	NDS	-0.84	-0.46	-0.51	-0.50	
0.51	0.31			NDS	0.09	0.36	-0.19	0.29	0.76	
4.01	2.46	2,3		1.69	-0.54	0.98	-0.08	0.29	0.17	
-0.99	-0.61			0.03	-0.12	-0.39	0.81	0.49	0.43	
-1.49	-0.92			0.03	NDS	-0.61	-0.02	-0.35	-0.91	
-0.49	-0.30			0.03	-0.95	-0.61	0.09	0.41	0.93	
0.01	0.00			0.12	-0.95	-0.21	0.09	-0.79	0.09	
3.31	2.03	2,3		2.71	1.35	-1.07	0.05	-0.15	0.59	
-0.09	-0.06			0.58	0.30	1.38	0.91	0.81	0.76	
-1.69	-1.04			NDS	NDS	0.18	-0.67	-0.43	-0.66	
-0.09	-0.06			1.14	-0.95	1.61	NDS	NDS	NDS	
1.71	1.05			1.42	0.93	0.98	-2.96	0.33	NDS	
1.71	1.05			NDS	NDS	NDS	-0.02	-0.11	1.26	
-0.69	-0.43			0.68	NDS	-1.01	-0.25	-0.39	1.85	
-0.59	-0.36			0.21	NDS	-0.21	NDS	0.25	-0.33	
1.61	0.99			0.95	1.14	-3.01	1.18	1.81	1.51	
2.81	1.72			0.77	0.51	0.36	0.29	0.61	0.68	
0.01	0.00			0.77	1.35	-0.04	-1.97	0.53	-0.24	
-3.89	-2.39	2,3		0.31	4.49R	1.67	-5.74R	NDS	NDS	
-0.99	-0.61			-3.02	-0.95	-1.47	0.60	NDS	-0.24	
-0.59	-0.36			-0.15	-0.12	0.13	-0.60	-0.51	-0.08	
0.11	0.07			0.77	NDS	0.30	0.67	0.01	0.34	
0.01	0.00			NDS	NDS	NDS	NDS	NDS	0.17	
-0.99	-0.61			0.31	\$\$	-0.33	0.05	-0.19	0.09	

4, 2020

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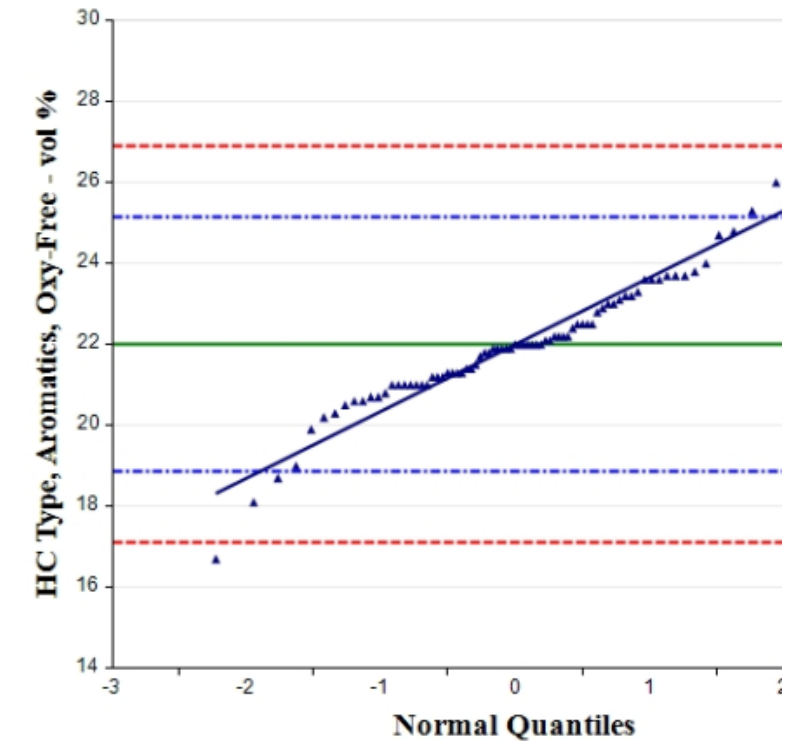
Box and Whisker Graphs

Box and Whiskers



q-q Plot

Distribution of Results - Q-Q
 HC Type, Aromatics, Oxy-Free by D1319-19 (vol %)
 No 2 Diesel Fuel-DF21910





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DDGS Proficiency Testing Program

Dried Distillers Grains with Solubles



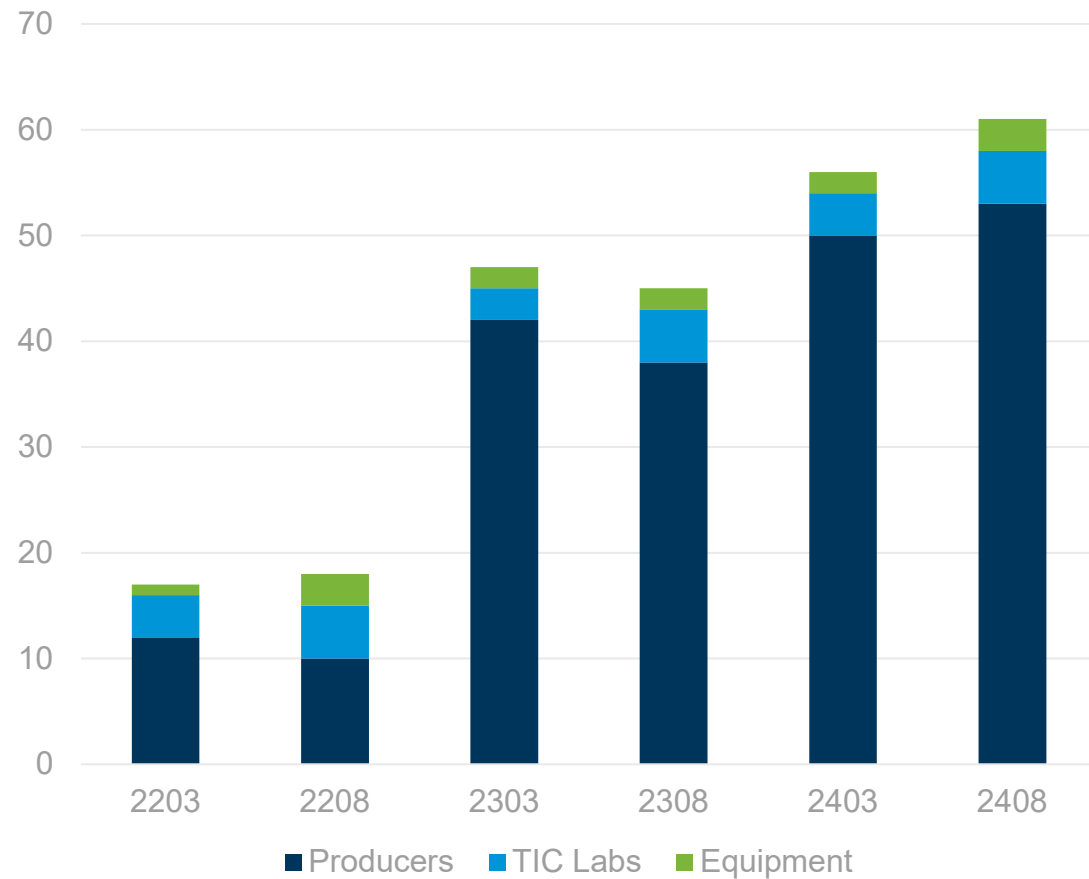
- Program Details:**
- Materials: One 300g sample of Dried Distillers Grains each cycle
 - Cycle Months: March, August, November (3 cycles/yr)
 - Annual Subscription Fee: \$870

Tests	Test Methods	Tests	Test Methods
Acid Detergent Fiber (ADF)	AOAC 973.18, ISO 13906	Loose Bulk Density	
Amylase-treated Neutral Detergent Fiber	AOAC 2002.04, ISO 16472	Moisture	NFTA Method 2.1.4 (September 2006)
Ash	AOAC 942.05, ISO 5984	Particle Size	
Color (L*, a*, and b*)		pH	
Crude Protein	AOAC 990.03 and AOAC 2001.11	Sulfur	
Crude Fat	AOAC 945.16	Mycotoxins, Aflatoxins (B1, B2, G1, G2, Total), Deoxynivalenol, Fumonisin (B1, B3, B3, Total), Other Mycotoxins (Ochratoxin, T-2, HT-2), Zeralenone	AOAC 2005.08, AOCS Aa11-05, AOAC 991.31, AOCS Ab 7-91, AOAC 986.18, AOCS Aj 5a-97, AOCS Aj 2-86, ISO 17372, Rapid Test Kits
Crude Fiber	AOAC 978.10		

Program Trends



Program Growth

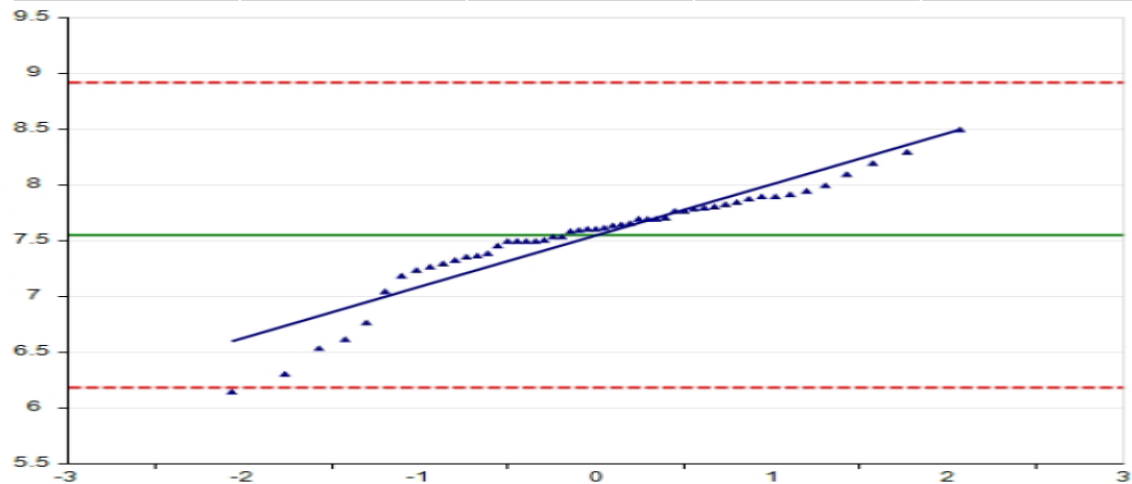


Summary of Results



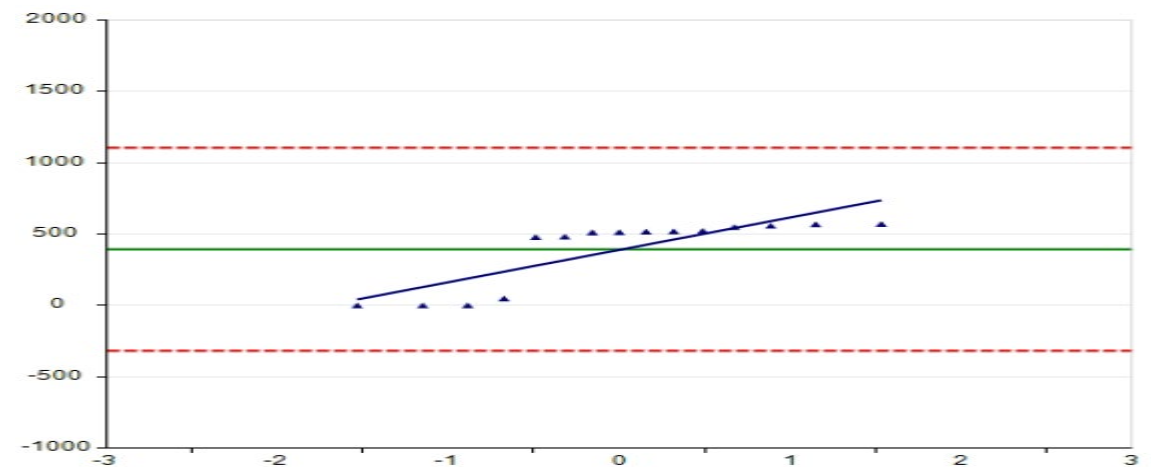
Moisture, %

	2303	2308	2403	2408
N	44	39	52	52
Avg	13.163	11.718	8.058	7.549
SD	0.940	0.435	0.437	0.456
RSD	7.14 %	3.71 %	5.42 %	6.04 %
AD	0.84	0.65	1.29	1.61



Loose Bulk Density, kg/m³

	2303	2308	2403	2408
N	24	22	18	15
Avg	454.85	443.45	503.29	389.95
SD	95.59	63.75	82.85	237.29
RSD	21.0 %	14.4 %	16.5 %	60.9 %
AD	2.64	2.58	2.74	2.52



Summary of Results



Ash, mass %

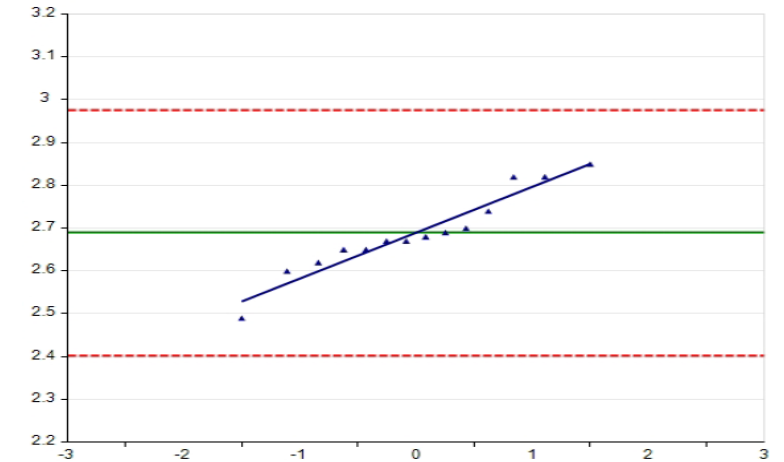
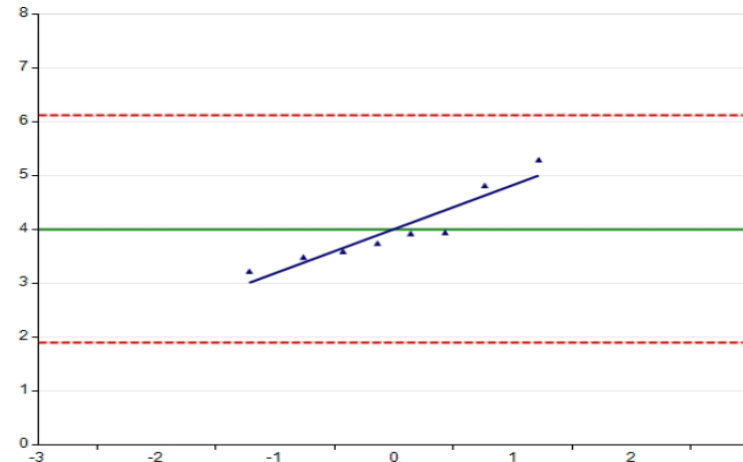
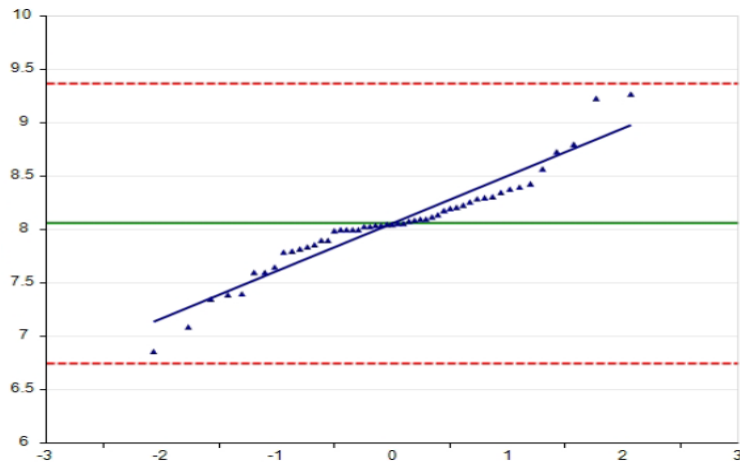
	2303	2308	2403
N	44	39	52
Avg	13.163	11.718	8.058
SD	0.940	0.435	0.437
RSD	7.14 %	3.71 %	5.42 %
AD	0.84	0.65	1.29

Ash (Near IR), mass %

	2408
N	8
Avg	4.009
SD	0.701
RSD	17.5 %
AD	0.55

Ash (Furnace), mass %

	2408
N	15
Avg	2.689
SD	0.096
RSD	3.57 %
AD	0.43

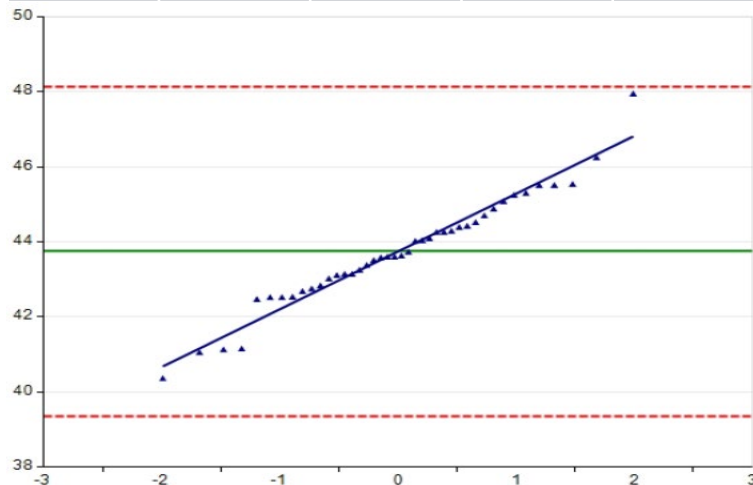


Summary of Results



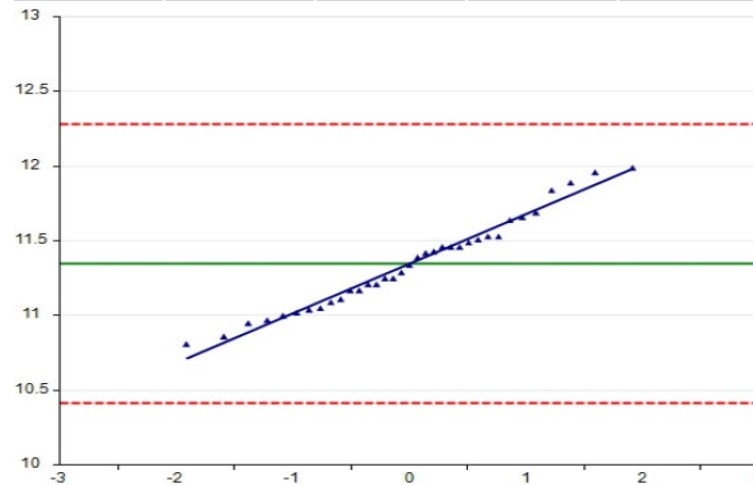
Color – L*

	2303	2308	2403	2408
N	34	33	40	42
Avg	49.40	55.18	43.42	43.75
SD	0.99	1.28	1.16	1.47
RSD	2.0 %	2.3 %	2.6 %	3.4 %
AD	1.02	0.59	0.37	0.34



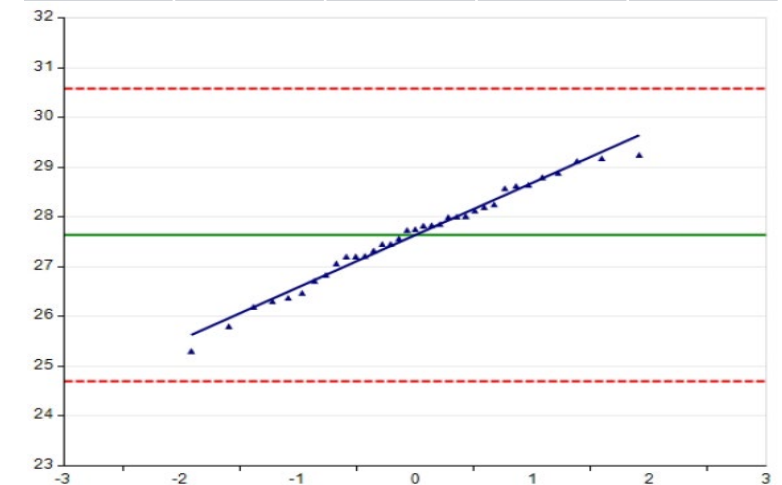
Color a*

	2303	2308	2403	2408
N	27	24	33	35
Avg	14.58	13.00	11.21	11.35
SD	0.31	0.74	0.41	0.31
RSD	2.2 %	5.4 %	3.7 %	2.7 %
AD	0.29	1.27	0.93	0.29



Color b*

	2303	2308	2403	2408
N	27	24	33	35
Avg	38.55	39.02	27.24	27.64
SD	1.47	1.04	0.88	0.98
RSD	3.8 %	2.7 %	3.2 %	3.4 %
AD	0.63	0.53	0.38	0.22



Summary of Results



Crude Protein (As Is), mass %

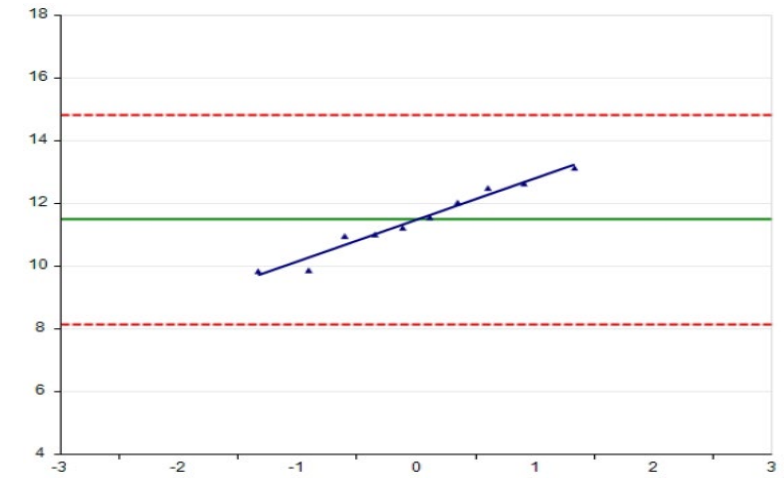
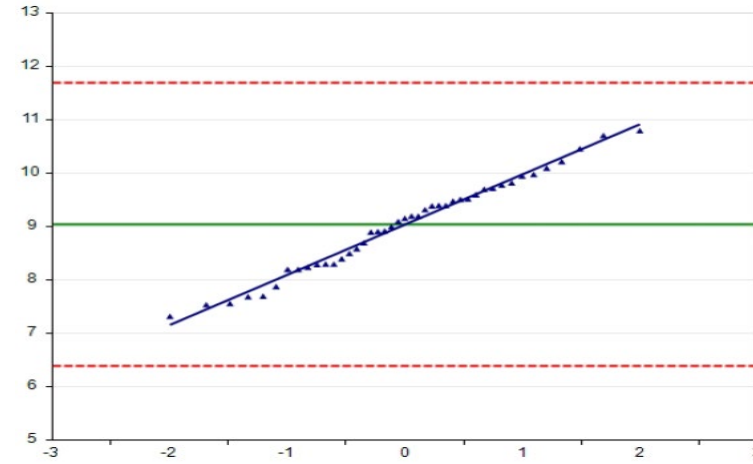
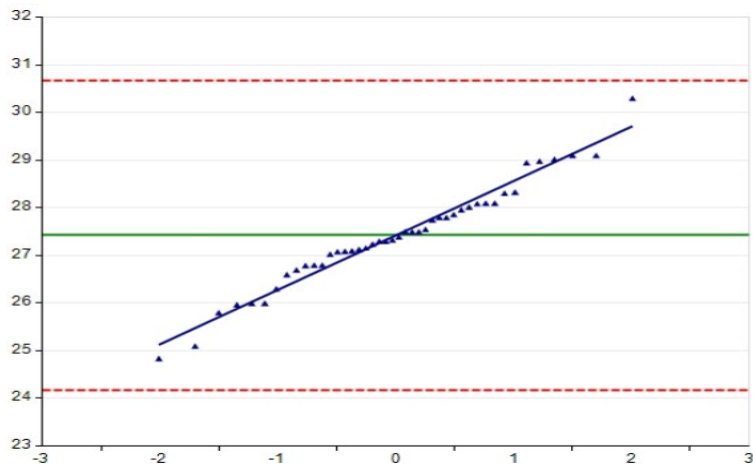
	2303	2308	2403	2408
N	35	34	45	45
Avg	29.30	30.32	27.28	27.42
SD	1.01	1.25	1.21	1.09
RSD	3.5 %	4.1 %	4.4 %	4.0 %
AD	0.33	0.47	0.43	0.38

Crude Fat (As Is), mass %

	2303	2308	2403	2408
N	35	34	43	43
Avg	6.64	6.20	8.72	9.04
SD	1.01	0.88	0.86	0.88
RSD	15 %	14 %	9.8 %	9.7 %
AD	1.18	0.35	0.45	0.27

Crude Fiber (As Is), mass %

	2303	2308	2403	2408
N	4	7	10	10
Avg	NSP	8.06	10.67	11.48
SD		1.20	1.62	1.12
RSD		15 %	15 %	9.8 %
AD		0.47	0.43	0.25

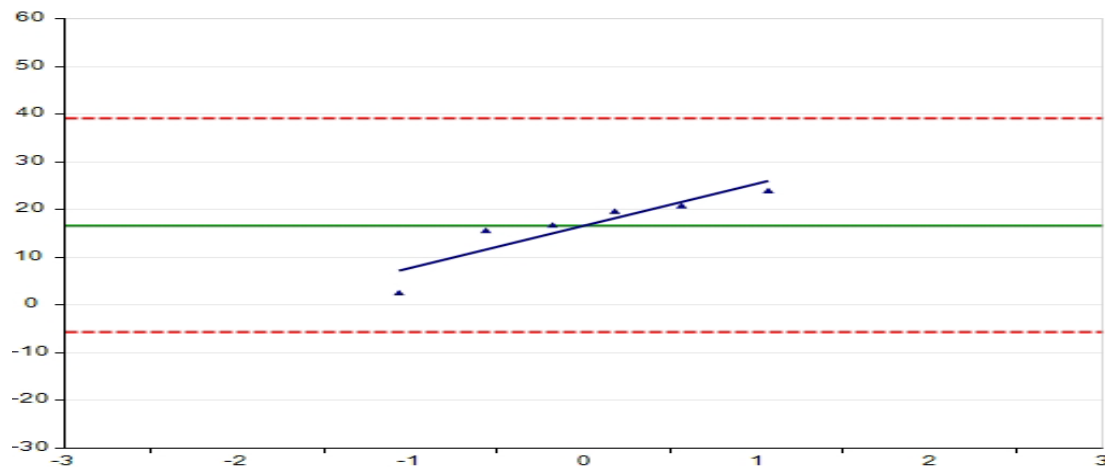


Summary of Results



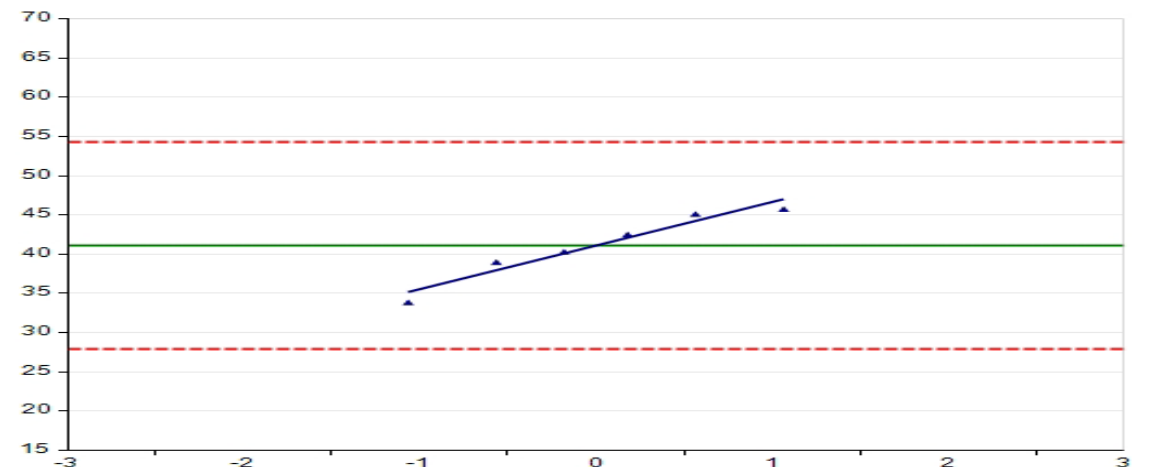
Acid Detergent Fiber (As Is), %

	2303	2308	2403	2408
N	2	4	4	6
Avg	NSP	NSP	NSP	12.628
SD				7.466
RSD				44.9 %
AD				0.55



Amylase-treated Neutral Detergent Fiber (As Is), %

	2303	2308	2403	2408
N	2	4	4	6
Avg	NSP	NSP	NSP	41.107
SD				4.409
RSD				10.7 %
AD				0.27

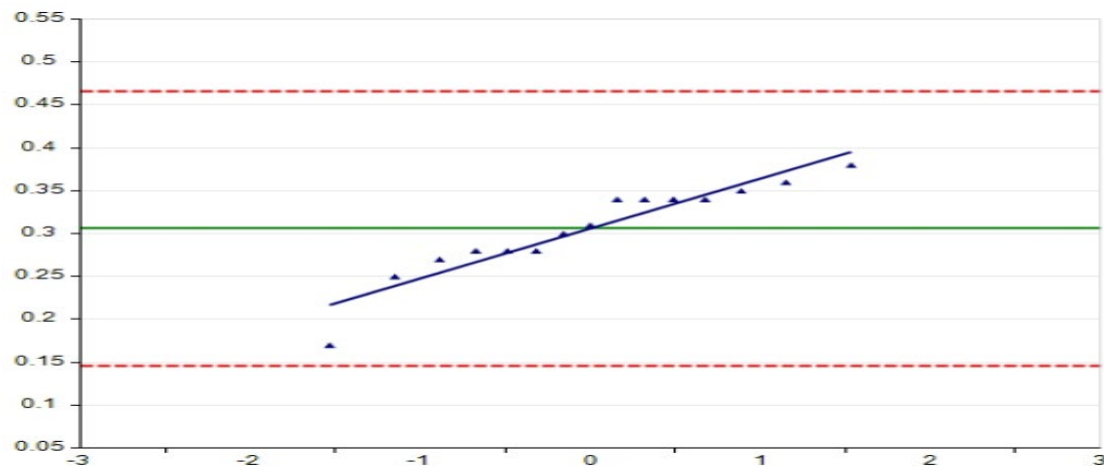


Summary of Results



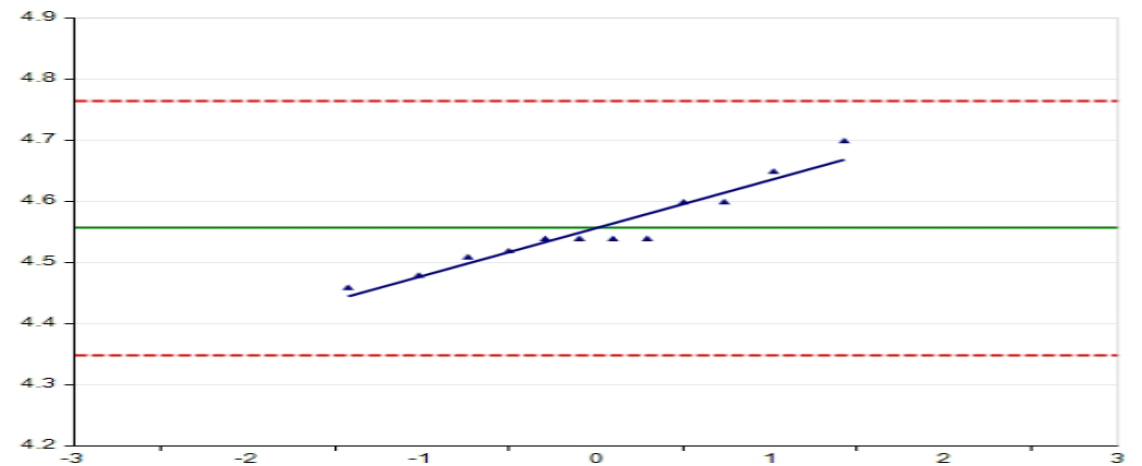
Sulfur, mg/kg (ppm) ► wt %

	2303	2308	2403	2408
N	6	11	9	17
Avg	NSP	0.653	947.7	0.306
SD		0.049	1467.0	0.053
RSD		7.50 %	154.8 %	17.32 %
AD		0.51	1.62	0.47



pH

	2303	2308	2403	2408
N	5	8	7	12
Avg	NSP	4.629	4.576	4.557
SD		0.121	0.096	0.069
RSD		2.61 %	2.10 %	1.51 %
AD		0.94	0.21	0.42

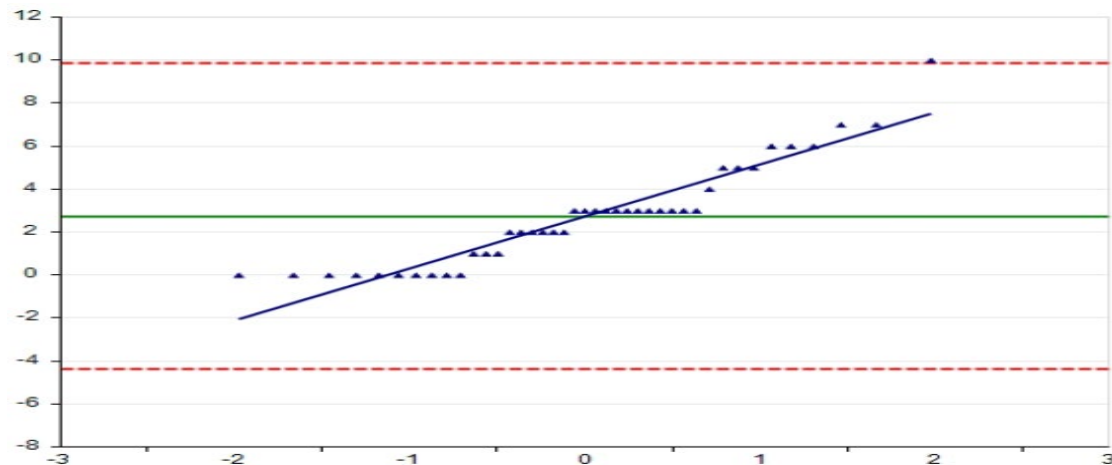


Summary of Results



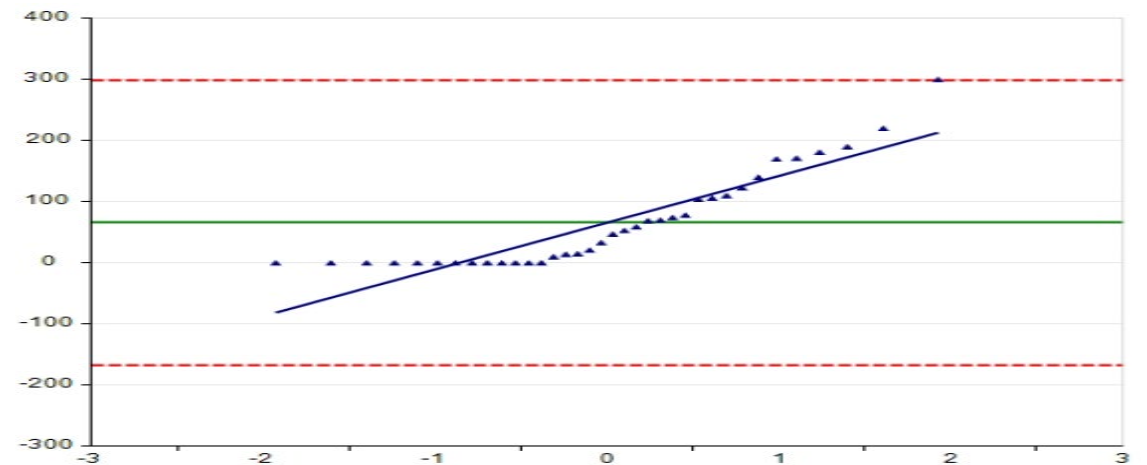
Total Aflatoxins, $\mu\text{g}/\text{kg}$ (ppb)

	2303	2308	2403	2408
N	36	34	42	43
Avg	1.596	2.350	3.480	2.7
SD	0.952	1.400	2.418	2.4
RSD	59.6 %	59.6 %	69.5 %	89.9 %
AD	0.34	0.44	0.91	1.33



Zeralenone, $\mu\text{g}/\text{kg}$ (ppb)

	2303	2308	2403	2408
N	36	31	42	39
Avg	172.1	105.5	34.31	65.5
SD	143.3	68.0	46.70	77.8
RSD	83.3 %	64.5 %	136 %	119 %
AD	2.27	0.29	3.74	2.20

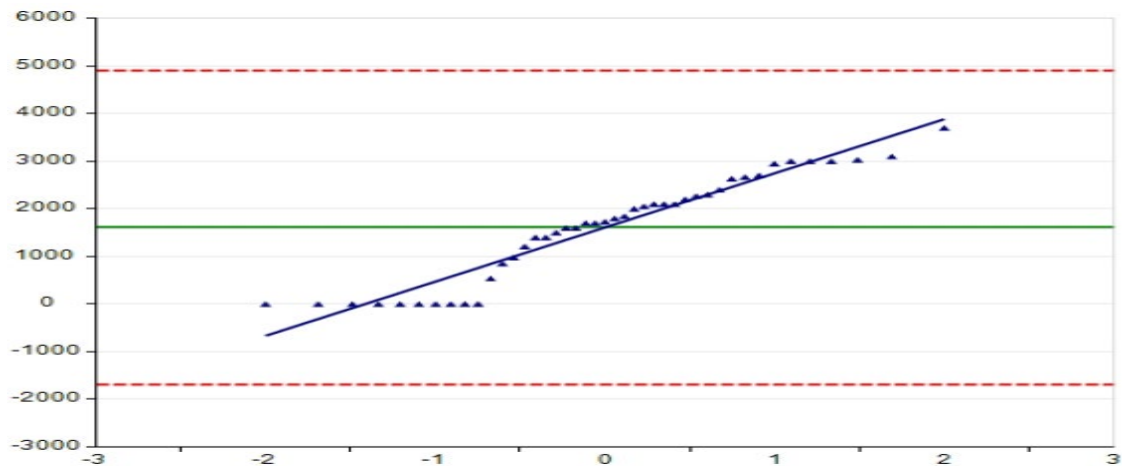


Summary of Results



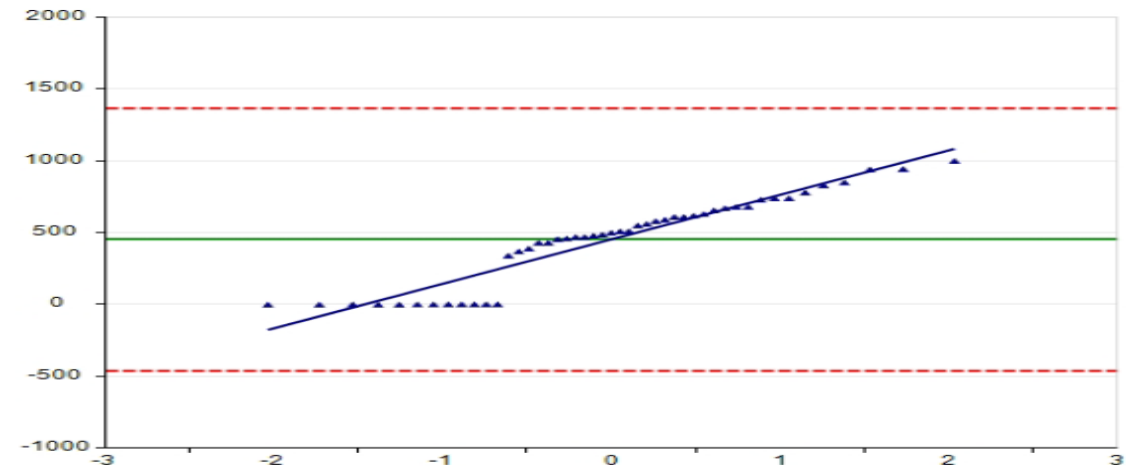
Total Fumonisin, $\mu\text{g}/\text{kg}$ (ppb)

	2303	2308	2403	2408
N	33	31	43	43
Avg	248.7	4347.5	909.8	1608.8
SD	781.9	1660.9	1203.8	1100.2
RSD	314 %	38.2 %	132 %	68.4 %
AD	9.00	0.48	4.82	1.20



Deoxynivalenol, $\mu\text{g}/\text{kg}$ (ppb)

	2303	2308	2403	2408
N	38	37	44	47
Avg	93.06	1229.2	264.0	453.1
SD	312.51	457.4	348.4	304.7
RSD	336 %	37.2 %	132 %	67.2 %
AD	10.78	0.62	5.35	1.91



Key Sources of Errors

0.623 ► 0.263

–Transcription Errors

1 + 1 = 3?

–Calculation Errors

mg/kg vs µg/kg

–Units of Measurement

Moisture 3 h @ 105 °C vs
 1 h @ 130 °C

–Inconsistent Procedures

–Insufficient Test Specimen Prep or Volumes
for Test Kits

For Consideration:

Utilization of “Reference” Laboratories for establishment of ARVs and Tolerance Limits



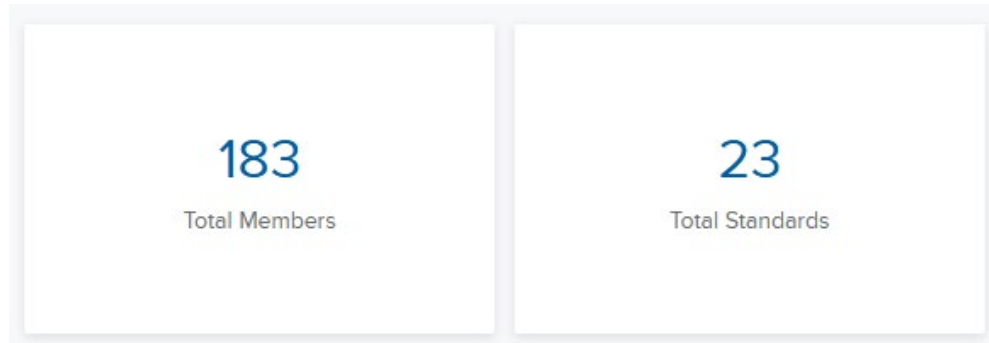
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ASTM Committee E48 on Bioenergy and Industrial Chemicals from Biomass Subcommittee E48.D0 on Distillers Co-Products

Committee E48 on Bioenergy and Industrial Chemicals from Biomass



Scope - The promotion of knowledge, stimulation of research, and the development of standards and methods, and development of recommended practices and guides related to the analysis of distillers grains and co-products produced from the biorefinery and distillery industries.



- [E48.05 Biomass Conversion](#)
- [E48.80 Sustainability of Bioenergy and Industrial Chemicals from Biomass](#)
- [E48.90 Executive](#)
- [E48.91 Terminology](#)
- [E48.A0 Product Specifications](#)
- [E48.D0 Distillers Co-Products](#)



ASTM International Technical Committee E48 on Bioenergy and Industrial Chemicals from Biomass

Scope

The promotion of knowledge and the development of standards for bioenergy and industrial chemicals from biomass. The focus of the committee shall be on bioenergy and industrial chemicals from biomass, from characterization through manufacturing. The work of this Committee will be coordinated with other ASTM Committees and other organizations having mutual interests.

Technical Subcommittees

- E48.05 Biomass Conversion
- E48.48 US TAG to ISO TC 248 on Sustainability Criteria for Bioenergy
- E48.80 Sustainability of Bioenergy and Industrial Chemicals from Biomass
- E48.90 Executive
- E48.91 Terminology

Key Documents

- E870 Standard Test Methods for Analysis of Wood Fuels
- E871 Standard Test Method for Moisture Analysis of Particulate Wood Fuels
- E873 Standard Test Method for Bulk Density of Densified Particulate Biomass Fuels
- E1756 Standard Test Method for Determination of Total Solids in Biomass
- E1821 Standard Test Method for Determination of Carbohydrates in Biomass by Gas Chromatography
- E3050 Standard Specification for Denatured Ethanol for Use as Cooking and Appliance Fuel



Quick Facts

Established 1985
Number of Members 160+
Number of Standards 15
Global Participation
15 Countries represented
The standards are available in
Volume 05.06 in the *Annual Book of ASTM Standards*
Meetings E48 meets twice each year, in June and December

Staff Manager

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<https://www.astm.org/get-involved/technical-committees/committee-e48>

Benefits of Participation - Laboratories



Visibility Amongst Customers and Employee Candidates – Through active involvement in the development of test methods, you will be visible to potential talent as well as customers establishing your position as a leading Subject Matter Expert. Though this visibility, potential employee candidates might have appreciation and even admiration for organizations permitting their staff for spending time and effort for such activities.

Opportunity to Develop Practical Test Methods – Being the one driving the initiative, you have control over how practical the standardized test method is, rather than being subjected to the application of a test method that is not as practical in terms of the equipment, standards and reagents, procedural and QA/QC requirements. It should be desirable to control one's own destiny and if ASTM Test Methods are available for reference, state regulators are more likely to mandate the use of such standards rather than going through the trouble of developing their own. There is already much precedent for regulators making reference to ASTM Standards.

Eliminate the Need for Re-Training Staff and Equipment Purchase – If you utilize your own laboratory's test method as the basis for the ASTM Test Method, you will not need to train staff or make costly purchases for new equipment and consumables. Being subject to utilizing a test method that your laboratory did not develop, this might not be the case.

Expert Input via Peer Review – While you might be quite proud of your own laboratory's test method, there might be potential for making it even better (e.g. more robust, improvement in precision, decreasing turn around time, etc.). Through the development process, your test method will be reviewed by industry experts and improvement suggestions will be made along the way – this is an opportunity for your laboratory to leverage this input for instigating your own laboratory's continuous improvement efforts.

Validation Testing and Development of Interlaboratory Precision – Part of the process of developing an ASTM Test Method is the performance of an Interlaboratory Study (ILS). This is a very robust validation and entails the testing of several different samples in duplicate by several different laboratories. Obtaining an equivalent number of degrees of freedom from within a single laboratory is a monumental effort and is likely to be more open for scrutiny of individual laboratory bias. The concern for individual laboratory bias is removed as the ILS will involve participation from multiple laboratories.

Precision Statements – Once the ILS is complete, ASTM Staff will develop a precision statement to state the repeatability and between laboratory reproducibility for the test method. This is useful for a laboratory to evaluate their own performance (vs the industry) as well as in evaluation and defense of results.

December 11, 2024 at the Anaheim Marriott

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Thank you

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