ASTM INTERNATIONAL
Helping our world work better

DDGS Proficiency Test Program

Fuel Ethanol Laboratory Conference
October 4, 2022
Chris McCullough, General Manager, Program Development, Laboratory Services

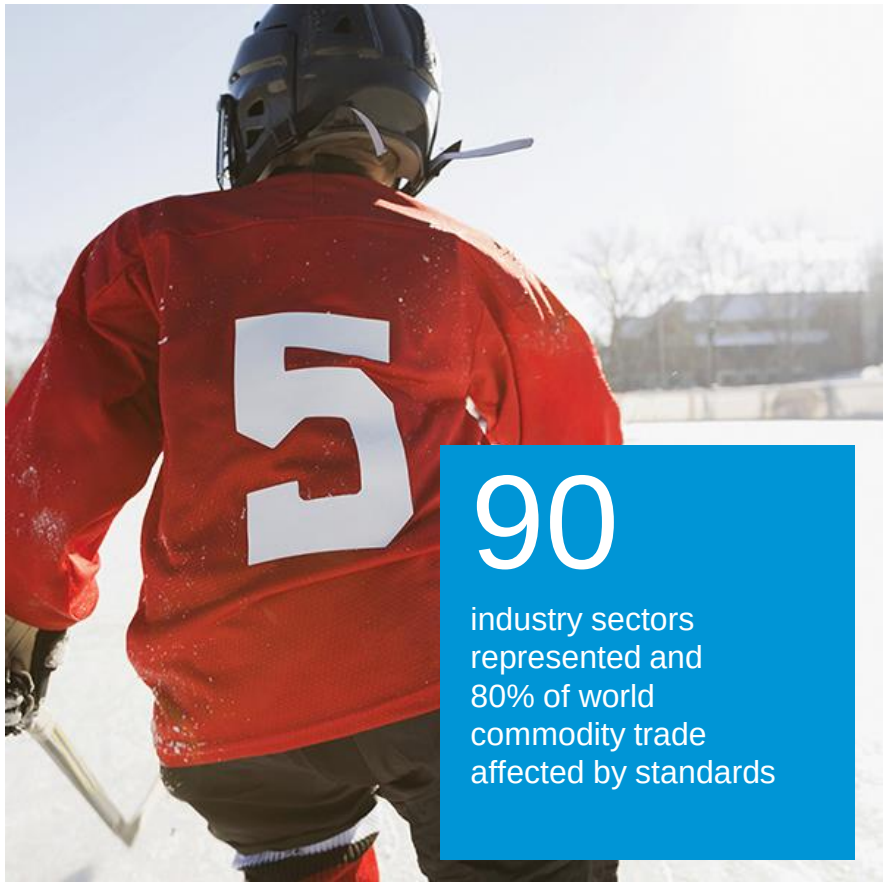
www.astm.org

Agenda

- Introduction to ASTM - Who we are / What we do
- DDGS Proficiency Test Program Details
- Overview of Summary Report (DDGS2203)
- Additional Programs and Services

Helping Our World Work Better

Touching Every Part of Everyday Life



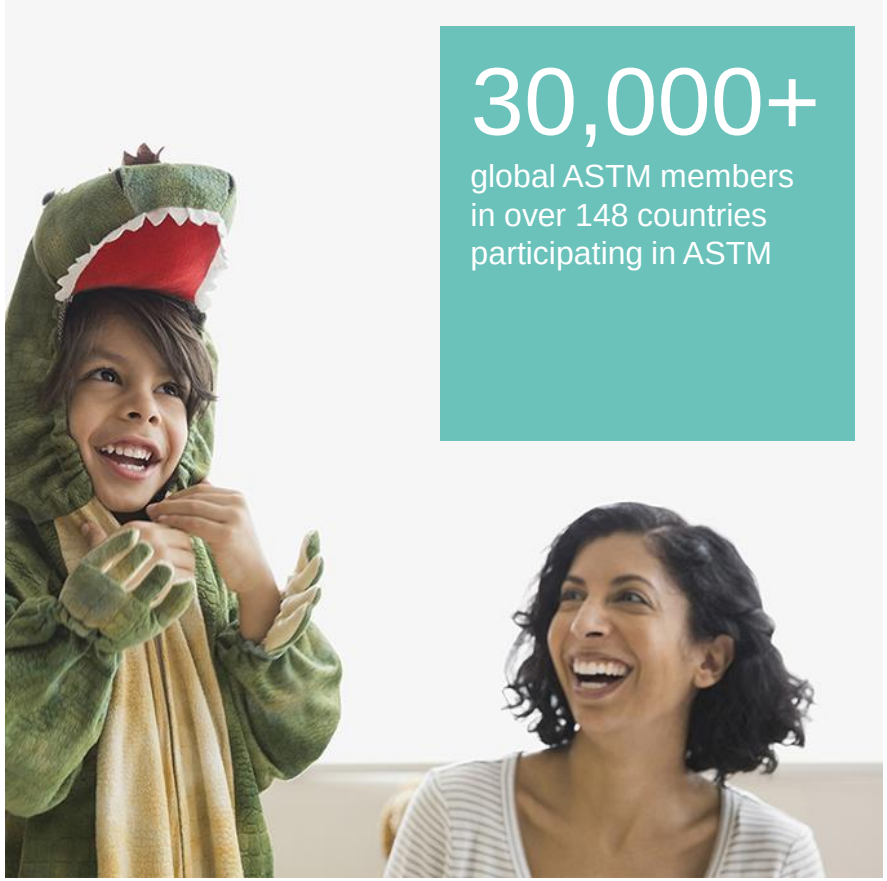
90

industry sectors represented and 80% of world commodity trade affected by standards

145

main committees plus 2,017 subcommittees

Over a Century of Openness



30,000+

global ASTM members in over 148 countries participating in ASTM

1,500

people trained annually across 145 ASTM courses

Independent and Objective

Testing and Certification

- Proficiency Testing Services: confirm and improve competence; determine method accuracy; instill the ability to judge, assess and compare test methods
- Resulting data creates statistical summary reports
- Certification and Declaration Programs provide independent evaluation of compliance to standards or regulations
- Available worldwide, they provide a level playing field, answer regulatory needs and reinforce consumer confidence
- ASTM also offers Personnel Certification Programs

ASTM DDGS Proficiency Test Program

Program Details

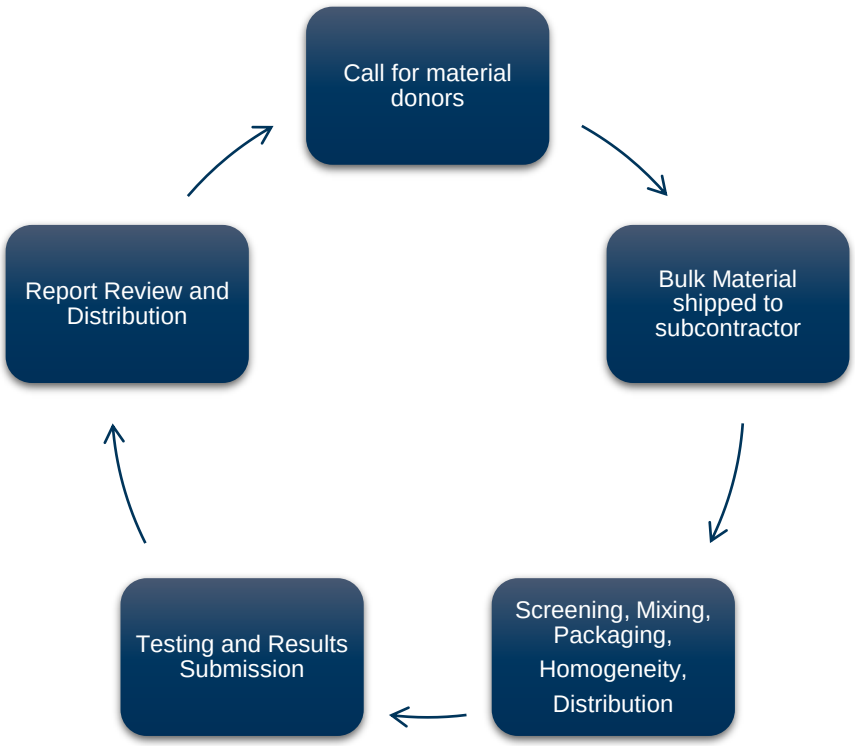
- 2 Cycles per year (March, August) $750/yr
- 3 x 300 g samples per cycle
- Upon completion of testing, each laboratory submits their own data online, electronically to ASTM for use in generating electronic statistical summary reports that contain:
  - Coded laboratory test results
  - Statistical analyses of test data
  - Charts plotting test results versus laboratory code
- Test instructions and data report forms are distributed electronically to each participant on the date samples are distributed. Labs have approximately 8 weeks to submit test data with the final statistical summary reports being electronically distributed in approximately 25 business days.
- https://www.astm.org/STATQA/index.html

ASTM DDGS ILCP

Test List

- Moisture [NFTA 2.2.2.5]
- Crude Protein [AOAC 990.03 and AOAC 2001.11]
- Crude Fat [AOAC 945.16]
- Crude Fiber [AOAC 978.10]
- Amylase-treated Neutral Detergent Fiber (NDF) [AOAC 2002.04 and ISO 16472]
- Acid Detergent Fiber (ADF) [AOAC 973.18-1977 and ISO 13906:2008]
- Ash [AOAC 942.05 and ISO 5984]
- Sulfur [AOAC 923.01 and ISO 27084]
- Loose Bulk Density
- Particle Size (ANSI/ASABE S319.4 FEB2008 (R2017)
- Color – Hunter L*, a*, and b*
- pH
- Water Activity
- Mycotoxins (Multiple AOAC,AOCS Test Methods)

Cycle Overview



- Call for Material Donors
- Bulk Material Shipped to Subcontractor
  Cost of shipment paid for by ASTM International
- Screening Tests (Protein and Moisture)
- Mixing and distribution using Boerner Divider
- Packaging in sealed double bagged
- Homogeneity Testing – to validate between sample homogeneity (Protein – later, will add Particle Size)
- Sample Distribution – domestic shipping included in registration fee. Options for international participants.

DDGS 2208 Participants

COMPANY	CITY	COUNTRY
Alcoholes Del Uruguay S.A	Paysandu	Uruguay
CHS- Rochelle	Rochelle, IL	United States
Eurofins Food Testing Rotterdam	Barendrecht	Netherlands
Foundation Analytical Laboratory	Cherokee, IA	United States
FS Agrisolutions Industria de Biocombustiveis Ltda	Sorriso, MT	Brazil
FS Agrisolutions Industria de Biocombustiveis Ltda	Lucas do Rio Verde, MT	Brazil
Green Plains	Superior, IA	United States
Green Plains	Atkinson, NE	United States
Green Plains	Madison, IL	United States
Green Plains	Central City, NE	United States
Green Plains	Fairmont, MN	United States
Green Plains	Mount Vernon, IN	United States

COMPANY	CITY	COUNTRY
Green Plains	Rives, TN	United States
Green Plains	Fergus Falls, MN	United States
Green Plains	Shenandoah, IA	United States
Green Plains	Wood River, NE	United States
Green Plains	York, NE	United States
ICM Inc.	Saint Joseph, MO	United States
Mid Iowa Grain Inspection, Inc.	Elwood, IL	United States
Poet Research Inc.	Sioux Falls, SD	United States
Romer Labs	Union	United States
Trilogy Analytical Laboratory	Washington	United States
Ward Laboratories, Inc.	Kearney	United States

Coverpage

ASTM INTERNATIONAL
Helping our world work better

Committee DGTC
Proficiency Test Program

Distillers Dried Grain with Solubles
Sample ID: DDGS2203
March 2022

Report Issue Date: July 25, 2022

Table of Contents

ASTM INTERNATIONAL
Helping our world work better

Table of Contents

Report Guidance

ASTM INTERNATIONAL
Helping our world work better

How to Use This Laboratory Report

Revision 5 Issued August 18, 2021

1. Overview

The Laboratory Report is customized for your laboratory. Results are displayed only for those test methods for which your laboratory submitted results. See the Program Report for results on other test methods for which your laboratory did not submit results.

1.1. ***Request to Appeal Against the Statistical Evaluation.*** **Any participant who wishes to appeal against the evaluation of their performance in a proficiency testing program may do so. To request an appeal, send an email to ptp@astm.org providing details such as program, cycle, method, data field and description and reason for the appeal. Requests will be evaluated and the PTP Center will provide a response.**

1.2. **The PTP can request reporting results in units of measure that are different than that specified in the test method, can require reporting one or more of the result**

Listing of Participant Laboratories

Alphabetical Listing of Laboratories
Submitting Results for DDGS2203

Lab Name (as given in Registration)	City	State/Province	Country
CHS- Rochelle	Rochelle	IL	United States
Eurofins Food Testing- Rotterdam	Barendrecht		Netherlands
FS Agrisolutions Industria de Biocombustiveis Ltda	Sorriso	MT	Brazil
FS Agrisolutions Industria de Biocombustiveis Ltda- Lab #2	Lucas do Rio Verde	MT	Brazil
Foundation Analytical Laboratory	Cherokee	IA	United States
Gpre Superior	Superior	IA	United States
Green Plains - Madison	Madison	IL	United States
Green Plains Fairmont Inc.	Fairmont	MN	United States
Green Plains Mt Vernon Llc	MOUNT VERNON	IN	United States
Green Plains Obion	Rives	TN	United States
Green Plains Otter Tail, Llc	Fergus Falls	MN	United States
Green Plains Shenandoah Llc	Shenandoah	IA	United States
Green Plains Wood River Llc	Wood River	NE	United States
Green Plains York LLC	YORK	NE	United States
Romer Labs	Union	MO	United States
Trilogy Analytical Laboratory	Washington	MO	United States
Ward Laboratories, Inc.	Kearney	NE	United States

Results Summary

Results Summary for All Methods

Distillers Dried Grain with Solubles - DDGS2203

Numeric Methods			Method Performance Statistics									
Measured Property With Conditions	Designation	Units	Conforming Results	Results Used	Average	StdDev	ASTM R	These Data R	Precision Performance	TPI	Anderson Darling	Normal?
Sample A	Moisture, Dry Matter	%	13	10	11.655	0.851		2.357			0.72	Normal
Sample A	pH		4		NSP							
Sample A, (As Is)	Ash	mass %	6	6	4.388	0.101		0.280			0.40	Normal
Sample A, (As Is)	Crude Fat	mass %	7	7	6.661	0.926		2.566			0.33	Normal
Sample A, (As Is)	Crude Fiber	mass %	4		NSP							
Sample A, (As Is)	Crude Protein	mass %	7	7	22.891	2.091		5.793			0.71	Normal
Sample A, (As Is)	Sulfur	mg/kg	4		NSP							
Sample A, ADF (As Is)	Acid Detergent Fiber	%	2		NSP							
Sample A, Aflatoxin B1	Aflatoxins, Mycotoxins	µg/kg (ppb)	6	6	1.608	1.331		3.686			0.46	Normal
Sample A, Aflatoxin B2	Aflatoxins, Mycotoxins	µg/kg (ppb)	3		NSP							
Sample A, Aflatoxin G1	Aflatoxins, Mycotoxins	µg/kg (ppb)	3		NSP							
Sample A, Aflatoxin G2	Aflatoxins, Mycotoxins	µg/kg (ppb)	2		NSP							
Sample A, Color - L*	Color		6	6	63.623	0.915		2.536			0.56	Normal
Sample A, Color - a*	Color		3		NSP							
Sample A, Color - b*	Color		3		NSP							
Sample A, Deoxynivalenol	Deoxynivalenol, Mycotoxins	µg/kg (ppb)	8	8	749.095	839.487		2325.378			0.89	Marginally Normal
Sample A, Fumonisin B1	Fumonsins, Mycotoxins	µg/kg (ppb)	5		NSP							
Sample A, Fumonisin B2	Fumonsins, Mycotoxins	µg/kg (ppb)	5		NSP							

Statistics Summary

Sample A, (As Is), Crude Protein

Data Report
Sample A, (As Is) by Crude Protein (mass %)
Distillers Dried Grain with Solubles - DDGS2203

Summary of Results

Conforming Results	7	
Results Used	7	
Average	22.891	
StdDev	2.091	
ASTM R		There is no ASTM Reproducibility for this method.
These Data R	5.793	
TPI		
ADrs Statistic	0.71	Normal

Legend

NDS	No Data Submitted
NCR	Non Conforming Results
1	Test result outside ± 3 sigma range for These Data
2	Test result outside ± 3 sigma range for ASTM Reproducibility
3	Z-Score outside range of -2 to 2
#	Z-Score > 90
$	Z-Score not calculated.
$$	Z-Score not calculated - submitted /Not Included in Statistics was non-conforming (see NCR or NIS).
$$$	Z-Scores non-existent - all lab results submitted were the same.
$$$$	ZScore is not displayed as StdDev is greater than 20% of Mean
NSP1	No Statistics Performed, <6 results submitted.
NSP2	No Statistics Performed, mixed data set w/ >18% non-numeric results.
NSP3	No Statistics Performed, as recommended by program reviewer.
NSP4	No Statistics Performed, mixed data set w/ >18% ND results.
R	Rejected by GESD

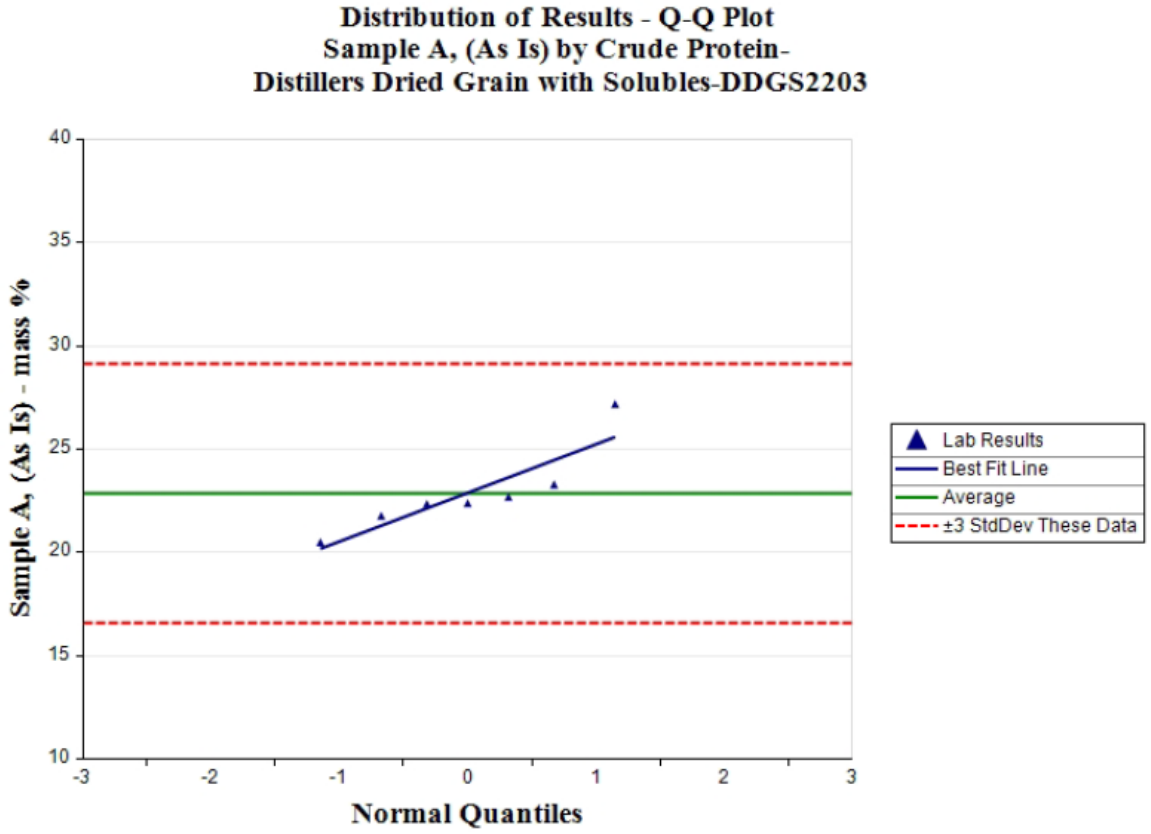
Results Summary

Results Table and Z-Scores
Sample A, (As Is) by Crude Protein (mass %)
Distillers Dried Grain with Solubles - DDGS2203

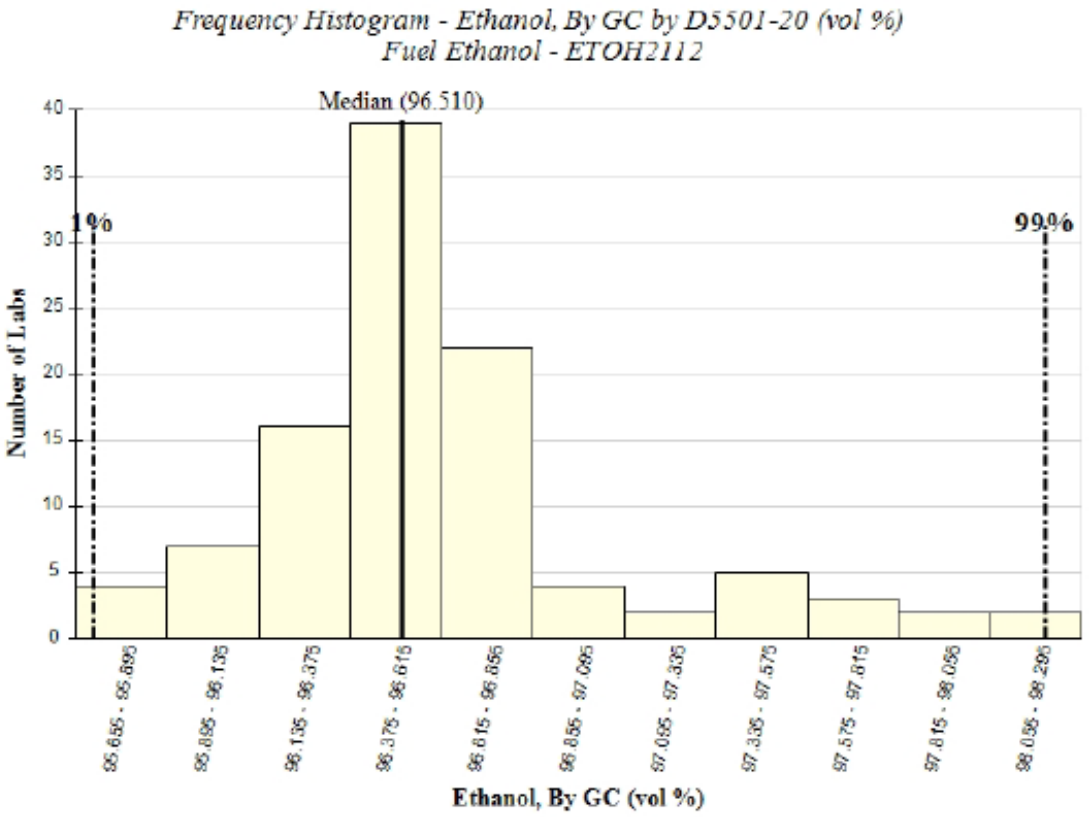
Lab Code	DDGS2203 Result	Dev	Z 2203	Notes	Z Count	Avg Z	StdDev Z-Score
0003	20.50	-2.391	-1.14		1	-1.14	
0004	22.36	-0.531	-0.25		1	-0.25	
0009	21.78	-1.111	-0.53		1	-0.53	
0015	22.70	-0.191	-0.09		1	-0.09	
0017	22.40	-0.491	-0.23		1	-0.23	
0018	27.20	4.309	2.06	3	1	2.06	
0020	23.30	0.409	0.20		1	0.20	

Distribution Graphs

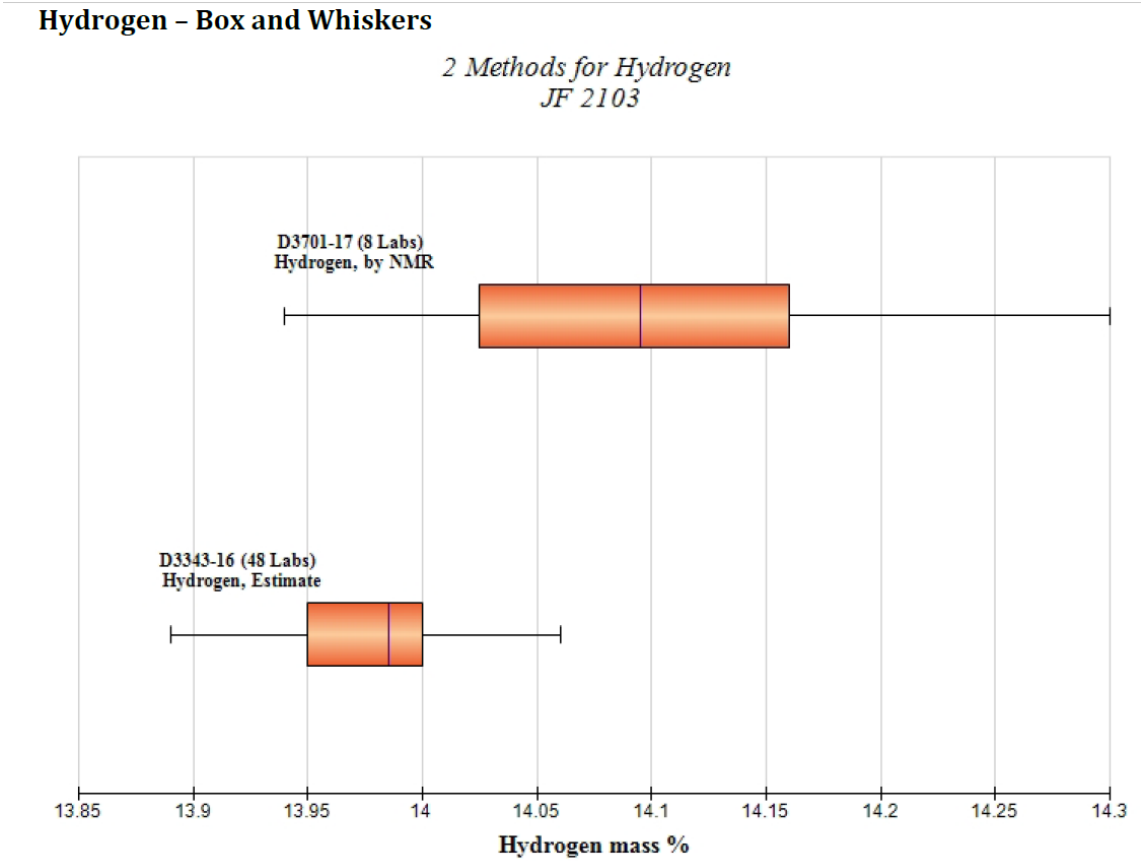
Distribution of Results



Results Histogram



Box and Whiskers to show bias and precision comparison



Future Consideration

Expansion of participant base

Standards Development

Addition of new tests

Laboratory Certification Program

Addition of New Programs

Splitting of data sets

ASTM Fuel Ethanol PTP

Program Details

- 3 Cycles per year (Apr, Aug, Dec)
- Annual Fee for all three cycles: $969
- 1 Liter Sample
- 113 Participants Laboratories (18 International)

https://www.astm.org/ptpetoh2022.html

Test Parameters

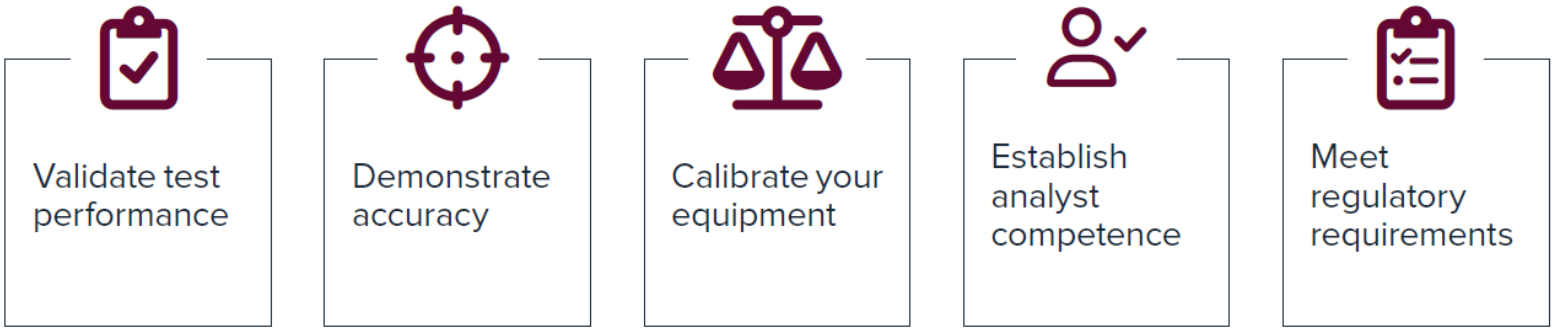
D1613, D7795	Acidity
D1688 (per D4806)	Copper Content
D5501	Ethanol/Methanol
D7319	Inorganic Chloride
D6423	pHe
D381	Solvent Washed Gum
D4052	Specific Gravity
D7319, D7328	Sulfate Content
D5453, D7039	Sulfur
D7923, E203, E1064	Water

Ethanol Reference Materials

Property	Test Methods
Acidity	D1613, D7795
Ethanol and Methanol	D5501
Inorganic Chloride and Sulfate	D7319, D7328
pHe	D6423
Specific Gravity at 60°F	D4052
Sulfur	D5453, D7039
Water	E203, E1064

Reduce risk, improve processes, and capture the accurate and precise data you need with ASTM International Reference Materials

- Wide variety of products, properties and levels
- Accurate reference values with low uncertainties
- Validated for homogeneity and stability
- Clear and concise Certificates of Analysis
- Quality packaging to safeguard materials during shipment and storage



ASTM Training Courses and E-Learning

Training for Your Industry

Choose a training program or course that's right for you. Whether it's our award-winning in-person continuing education courses or our industry-leading online training programs, you'll find the focused, practical and expert training you need.

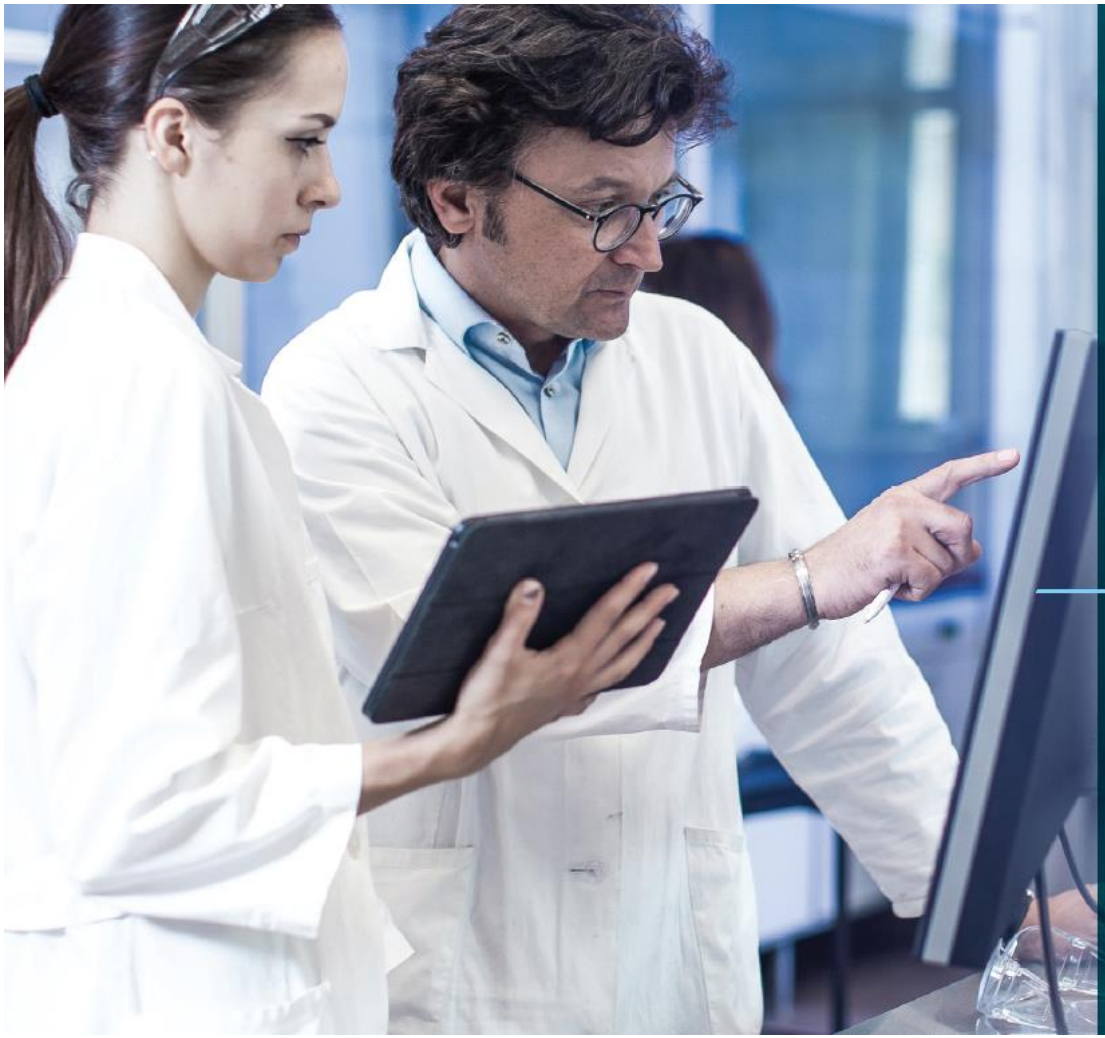
Instructor Led Training Courses

- Fuels Technology Hand-On Training
- Diesel Fuel: Specifications and Test Methods
- Gasoline: Specifications, Testing, and Technology
- Statistics in ASTM Standard Test Method Development Application, and QA
- And many more…

E-Learning Modules

- Various ASTM Ethanol Test Methods
- ASTM D4057 Sampling
- And many more…

ASTM Insight SQC



What sets ASTM Insight SQC apart?

- easy-to-use data integration tools
- straightforward interface witih easy setup for staff at all levels
- extensive dashboard for spotting issues and identification of trends
- configurable auto scheduling of QC runs and auto creation of rechecks
- configurable control strategies
- comprehensive view of individual or multiple labs with customized management reports

---

- automated chart construction with automatic statistical calculations
- flagging of warning violations and out of control violations
- automated assessment process
- assessment records to provide evidence of how control limits were established
- management reports to help with audits
- sortable resolution tools for use by all analysts

https://www.astm.org/ptpddgs2022.html

ASTM INTERNATIONAL
Helping our world work better

Thank you

www.astm.org

Chris McCullough
General Manager, Laboratory Services
cmccullough@astm.org

Amy Meacock
Director, Proficiency Testing Programs
ameacock@astm.org

John Gallagher
Director, Sales
jgallagher@astm.org