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Helping our world work better

ASTM International Interlaboratory Study Program (ILS)

www.astm.org

What is an ILS?

An interlaboratory study (ILS) is a multi-lab study done for the specific purpose of producing data that will be used to develop a Precision & Bias statement and Research Report in order to demonstrate the expected variability of a test method.

We'll discuss…

Who we are and what we do …

- In 2004, the **Board of Directors** approved the creation of a unit that would help to strengthen the perceived quality of ASTM Test Methods by:
  - Facilitating the **production of data**, with the goal of developing Precision & Bias statements and Research Reports to demonstrate the **variability** of our test methods
  - Providing **administrative and financial support** to all ASTM committees
  - Helping to ensure the **confidentiality** of participating labs

ASTM is not a lab.
We aren't able to test or receive samples.

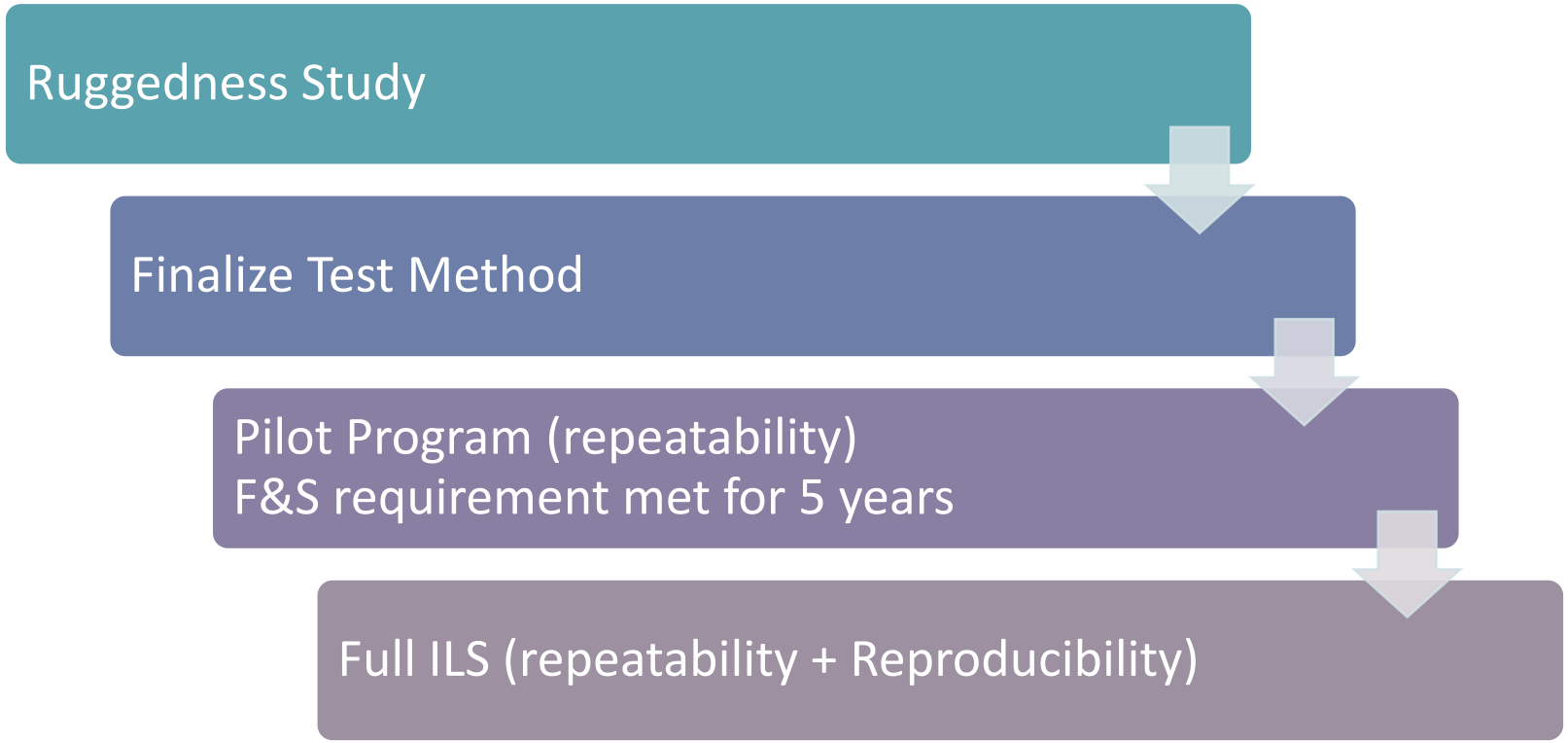
Form & Style Manual (Blue Book)

A21. Precision and Bias **(Mandatory)**

A21.2.1 A statement on precision allows potential users of the test method to assess in general terms its usefulness in proposed applications.

A21.2.3 **Every test method** shall contain: (1) a statement regarding the **precision of test results** obtained in the same laboratory under specifically defined conditions of within-laboratory variability (**repeatability** conditions); and (2) a statement regarding the precision of test results obtained in different laboratories (**reproducibility** conditions).

ILS Phases New Test Method



Test Result

- A test result should be uniquely defined by the Test Method- review your standard for specifics.
  - Single test determination
  - The average of two or more determinations
  - Subject to multiple if/then statements

- One **test result** = one reportable replicate for ILS purposes.

- A **test result** is the actual number you would report to a client.

- We will **need multiple test results (replicates),** on each material, from every operator, in order to calculate precision.

Test Result *Example*

- Test Method X requires the **average of 5** individual measurements to be reported as a single test result (replicate). Your ILS calls for 3 replicates.
  - A total of **15 individual measurements** must be taken to produce the 3 replicate test results (each the average of 5 measurements) required from each participating laboratory in this hypothetical.
- Laboratory Data Report Form

Hardness					
Individual Measurement 1	3	Individual Measurement 1	4	Individual Measurement 1	4
Individual Measurement 2	4	Individual Measurement 2	6	Individual Measurement 2	7
Individual Measurement 3	3	Individual Measurement 3	5	Individual Measurement 3	5
Individual Measurement 4	7	Individual Measurement 4	3	Individual Measurement 4	4
Individual Measurement 5	5	Individual Measurement 5	6	Individual Measurement 5	5
Average:	**4.4**	**Average:**	**4.8**	**Average:**	**5**

3 Replicate Test Results

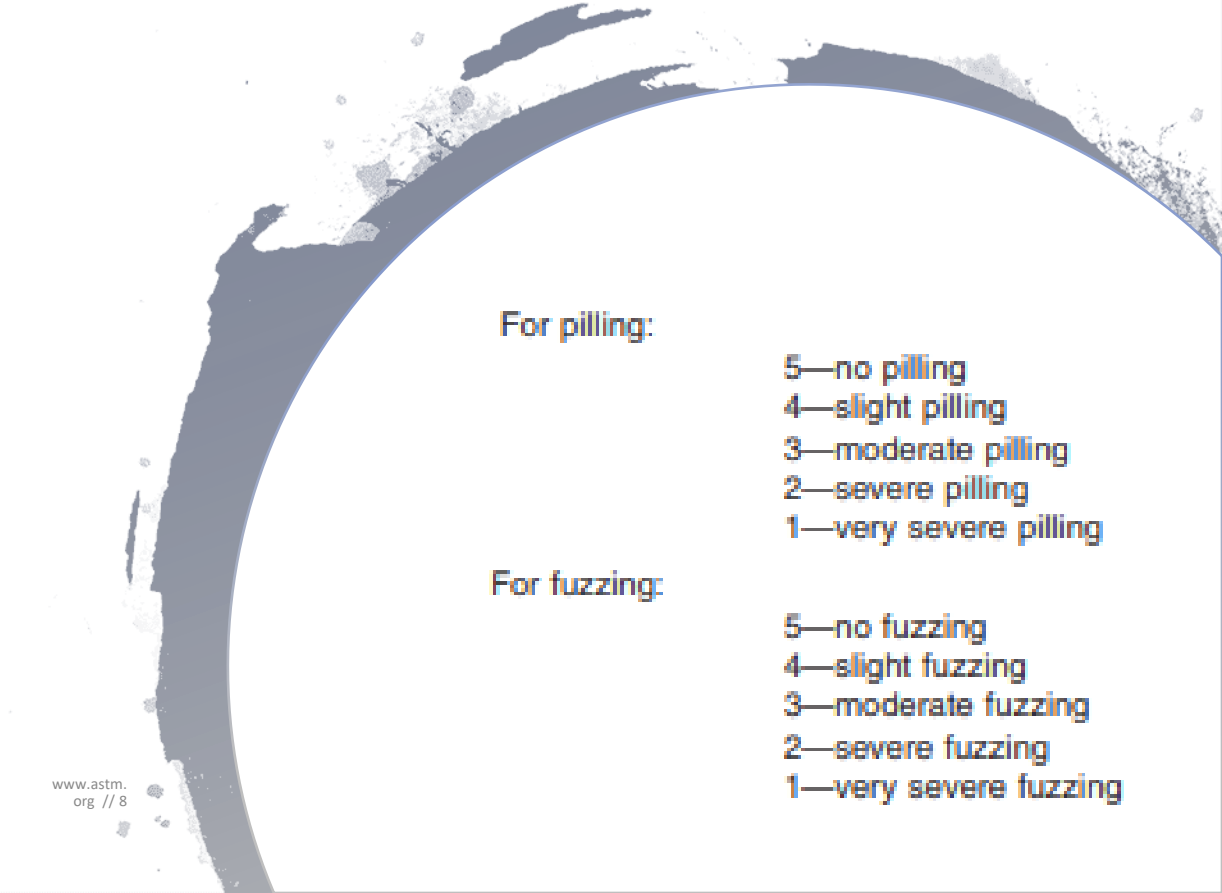
Nonquantitative Test Results

- An ILS is **not** required
- Examples: pass fail tests, rating scale, color change …

From ***Form and Style***:
A21. Precision and Bias (Mandatory)

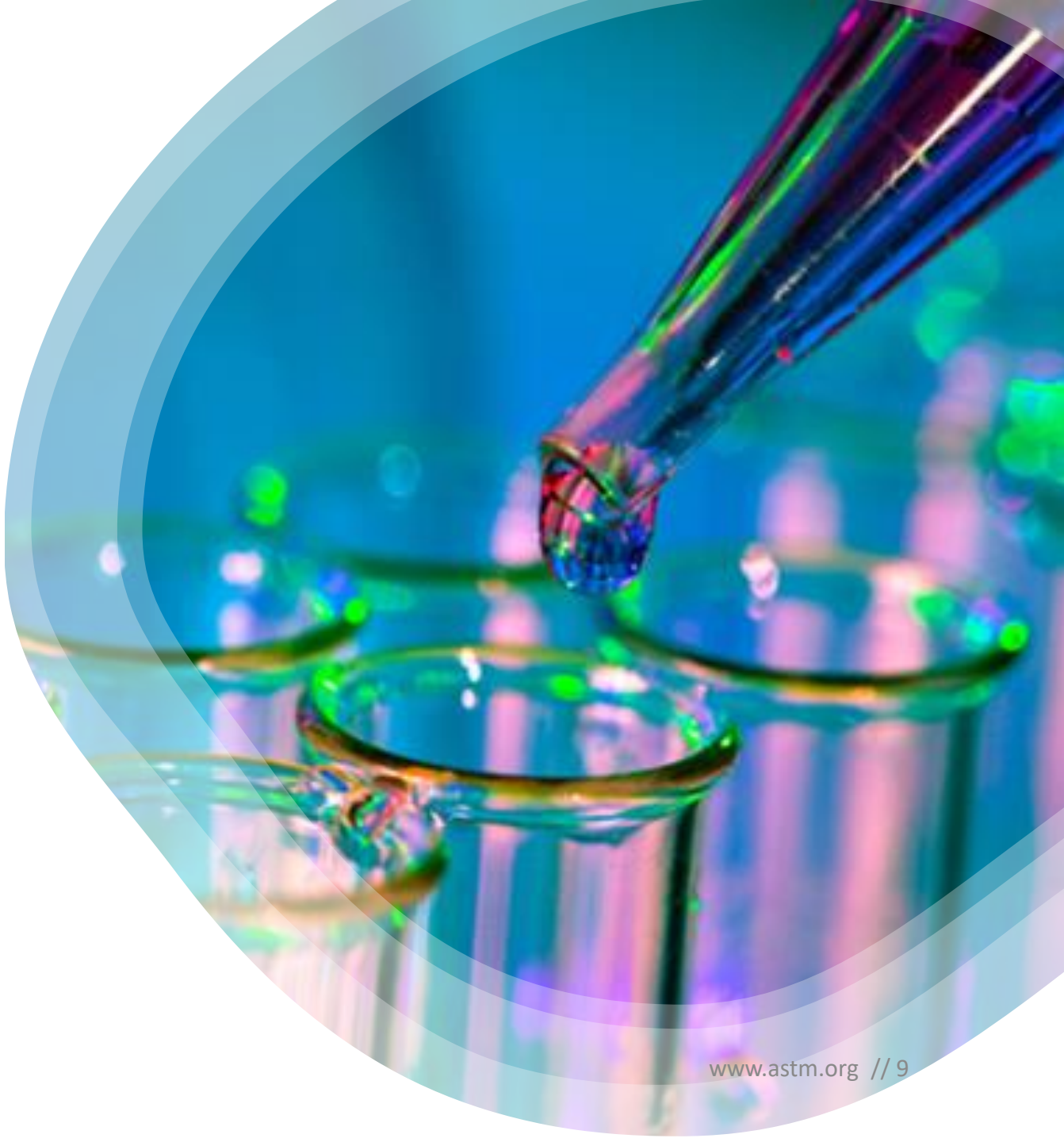
A21.5.4 When a test method specifies that a test result is a ***nonnumerical*** *report of success or failure or other categorization or classification based on criteria specified in the procedure, use a statement on precision and bias such as the following:*

Precision and Bias—No information is presented about either the precision or bias of Test Method X0000 for measuring (insert here the name of the property) since the test result is nonquantitative.



Administrative Support

- Scheduling of conference calls and WebEx meetings
- Review of Experimental Design
- Assistance identifying volunteer laboratories
- Identification of sample vendors
- Coordination of sample distribution
- Data collection
- Statistical processing
- Generation of reports
- Assistance with the adjudication of negative votes



Strengths

- Input encouraged from active Committee volunteers, as well as non-members, broadening the range and diversity of the study participants, allowing the study to most accurately demonstrate expected “real-world” precision
- Scientific neutrality of ASTM in reviewing test data (random lab numbers are assigned to all of the participants)
- Provides a value-added Quality Assurance Program to participating laboratories

Potential Lab Benefit of Participation

- Statistical program to monitor strengths and weaknesses of lab testing when compared to peers
- Assess testing performance and adherence to written procedures by lab technicians
- Recognition in the final Research Report

Benefits to the Committees

- Meet the requirements of the Form and Style Manual
- Obtain valuable feedback on methods, leading to the correction of errors and omissions, as well as highlighting the need for technical updates
- Resource for increased membership

"I think there was a typo in the lab instructions."

Blind Sample Matrix

- To generate, the ILS Staff must have:
  - Laboratory Names
  - Material Names
  - Number of Replicates
- Sample Labeling Matrix below was sent to the distributor by the ILS Staff
- Utilized by Committees D02, D16, D19 & D28

Sample Name/ Lab Name:	University of Calgary	Marathon Oil	Alberta Research Council	Phillips 66 OK	LyondellBasell	ExxonMobil Research	Agilent Technologies	Triton Analytics Corp	Envantage Inc.
Kerosene	1, 7	2, 12	5, 6	7, 9	4, 6	4, 7	2, 5	8, 11	7, 10
High Sulfur Diesel	3, 6	3, 10	9, 10	5, 10	2, 11	5, 10	9, 12	1, 4	2, 6
#2 Low Sulfur Diesel	5, 12	5, 11	4, 11	3, 11	8, 9	11, 12	1, 10	6, 10	4, 11
Aviation Turbine Jet A	2, 4	4, 6	2, 8	1, 6	3, 10	2, 8	3, 4	2, 9	5, 9
Ultra Low Sulfur Diesel	8, 11	7, 9	3, 7	2, 4	5, 7	1, 6	6, 11	3, 12	1, 3
Light Cycle Oil	9, 10	1, 8	1, 12	8, 12	1, 12	3, 9	7, 8	5, 7	8, 12

Data Report Form- Instructions

ASTM INTERNATIONAL
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ILS# 1446

ASTM F3203 - Test Method for Determination of Gel Content of Crosslinked Polyethylene (PEX) Pipes and Tubing

Basic Study Information:

Please have one Laboratory Technician conduct all ILS testing.
Please follow ASTM standard provided to you for this study.
Please complete the testing in the shortest possible period of time.

Data Entry:

Do not enter commas (1,234 --> 1234)
Do not report units

Submitting Data:

Please submit completed data report forms to ILS@astm.org

Laboratory understands and agrees that the data generated as a result of the services and provided to ASTM will be used in ASTM's business, to assist in developing a research report, consensus standard or adjunct thereto. Laboratory agrees to keep such data and results confidential and not to disclose or share the data/results with anyone else, without ASTM's written consent.

Click here for a copy of the ASTM International's Intellectual Property Policy.

Data Report Form

ASTM INTERNATIONAL
Helping our world work better

ILS#1446

F3203, Standard Test Method for Determination of Gel Content of Crosslinked Polyethylene (PEX) Pipes and Tubing

Laboratory Name:

Laboratory Technician:

Date:

Please submit completed data report forms to ILS@astm.org

Measured % Gel Content			
Material X	Material X-1	Material X-2	Material X-3
Replicate 1			
Replicate 2			
Replicate 3			

Measured % Gel Content			
Material Y	Material Y-1	Material Y-2	Material Y-3
Replicate 1			
Replicate 2			
Replicate 3			

Measured % Gel Content			
Material Z	Material Z-1	Material Z-2	Material Z-3
Replicate 1			
Replicate 2			
Replicate 3			

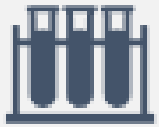
Comments:

Laboratory understands and agrees that the data generated as a result of the services and provided to ASTM will be used in ASTM's business, to assist in developing a research report, consensus standard or adjunct thereto. Laboratory agrees to keep such data and results confidential and not to disclose or share the data/results with anyone else, without ASTM's written consent.

Administering Programs

- Conference Calls and WebEx Meetings with the participants to discuss specific study instructions
- Coordination of acquisition and distribution of study material
  - Physical
  - Electronic
- Collection of data report forms & analysis of data
- Assist with the adjudication of negative votes

Precision



To calculate precision, we need usable data from at **least 6 laboratories**

(The closer to 30, the better)

Each lab should report **2-10 replicate** test results per material

The precision statement in an ASTM test method is not meant to qualify it as good or bad

Example:
% Moisture in mulch vs. aspirin

The published precision is there to help a user of the standard understand what can be expected based on the real world results of others

Repeatability (r) ranges

- With 95% confidence, the same operator, in the same laboratory, using the same equipment, under the same conditions, should obtain results when testing the same material that agree within this range.

  - **Example**: published repeatability range = 2.4 ppm

    Test Result 1: 79.1 ppm

    Test Result 2: 81.6 ppm

Results differ by 2.5 ppm, therefore: **Suspect**

*Internal laboratory investigation may be advisable

Reproducibility (R) ranges

- With 95% confidence, two operators, in different laboratories, using different equipment, under conditions meeting those specified in the standard, should obtain results when testing **the same material** that agree within this range.

  - **Example**: published reproducibility range = 3.2 ppb

    Test Result from Lab 1: 50.8 ppb

    Test Result from Lab 2: 47.9 ppb

Results differ by 2.9 ppb, therefore: **As Expected**

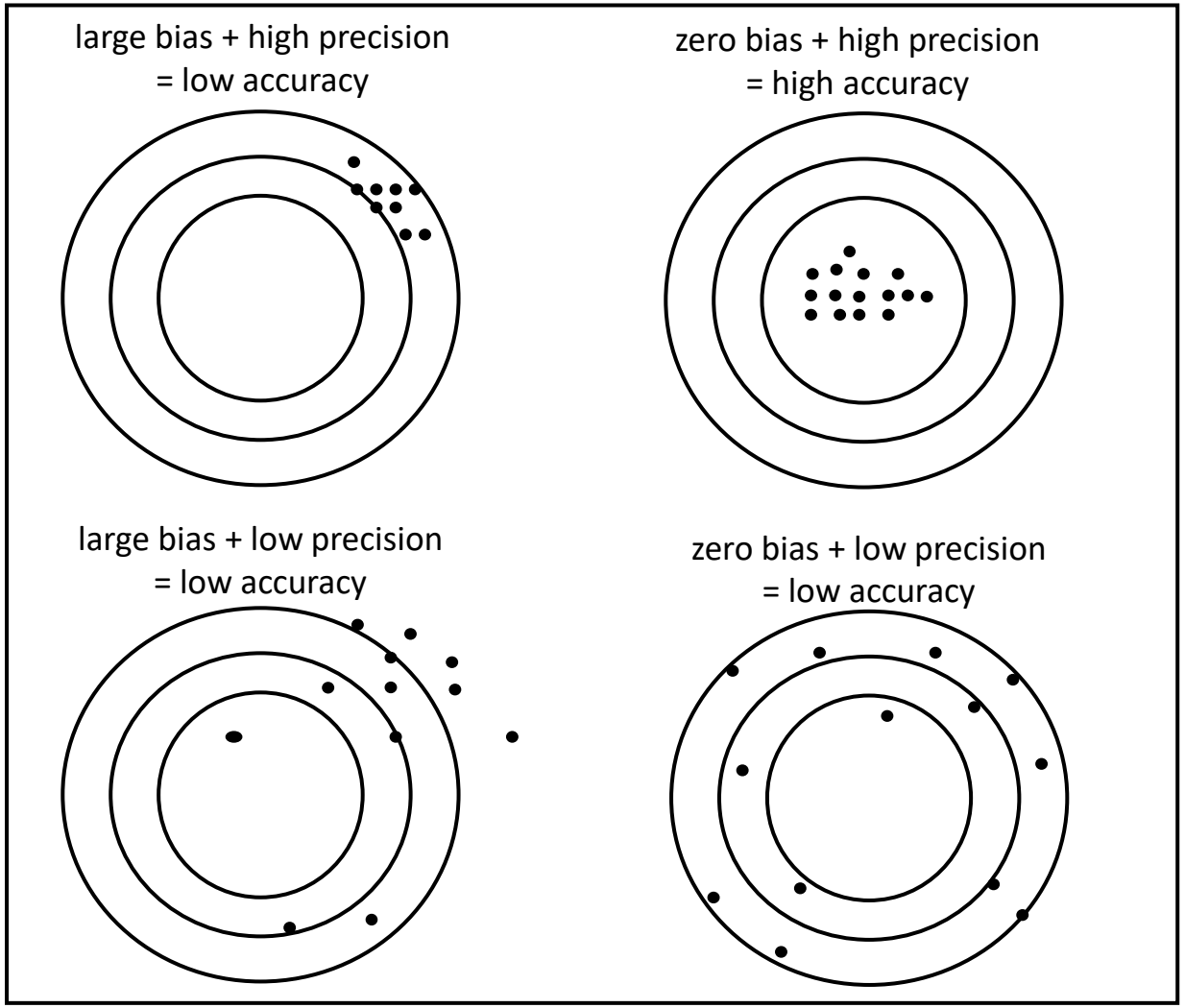
The Statistics (in a nutshell) ASTM E691

- E691 is useful for estimating the precision of different materials, at varying levels, with a repeatability and reproducibility range being calculated for each.
    - Usually 3-7 different materials span the range stated in the Scope of the standard
- Within laboratory precision is evaluated against a **k-statistic**.
    - Variability among replicates in any one lab
- Between laboratory precision is evaluated against an **h-statistic**.
    - Lab averages compared between all participants

Bias

- To calculate bias, we may be able to include a reference “standard” among the sample specimens distributed to the participating laboratories.
    - Bias may be determined as the average discrepancy between the “known” value and the reported values.

Precision and Bias



Remember …

- Your standard may allow you to correct for bias.
- You cannot correct for imprecision.
- An ILS may be used to demonstrate improvement as standards are modified.
    - For example: Compare results from Method A with those from Method B
- Whatever materials we are testing, they should be as homogeneous as possible (i.e. from the same batch and lot).

Establishing New Programs

- Concept registered as a **Work Item**
- Program registered through **MyASTM** as an ILS Program
- Initial conference call, with the technical contact from the committee, to establish the **basic study parameters**
- Experimental design (with input from the committee's **statistical support** person, if available)
- Identification of study materials, suppliers, a distributor, and volunteer laboratories

Committee Week Reports

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ILS# 0018 Committee Week Status Report

D3942, Test Method for Determination of the Unit Cell Dimension of a Faujasite-Type Zeolite

Subcommittee: D32.05
Technical Contact: Thomas Szymanski
Staff Manager: Kelly Paul
Work Item Number: WK29961
Registered Date: April 26, 2006
Statistical Support: THOMAS SZYMANSKI
Tests:

1. Relative Crystallinity %

Materials:

1. D32-08-002
2. Standard - Supplied by: Acme
3. D32-08-004 - Supplied by: Acme
4. D32-08-003 - Supplied by: Acme

Labs:

Lab Name	Contact Name		Data Submitted
Albemarle Bayport	Mark	James	✓
Chevron	James	Oliver	✓
Haldor Topsøe A/S	Shawn	Beegle	✓
INEOS	Joe	Little	✓
Lummus Technology Inc-TL-I	Diane	Williams	✓
Saint Gobain Norpro	Jacki	Cullen	✓
UOP	Terri	Ross	✓

From: ils@astm.org
Sent:
To: Technical Contact
Cc: Staff Manager, Sub Chair
Subject: ILS# 0458 - Committee Week Status Report

Dear Larry,

Please see the attached Committee Week Status Report for ILS# 0458, on ASTM - D0664- Test Method for Acid Number of Petroleum Products by Potentiometric Titration, for your upcoming Committee Week Meeting. This summary is to assist you in providing an update to your committee. Please review and let us know if any changes need to be made.

For your reference, attached are the following files:

P-and-B-Statement-For-ILS-0458.doc

Regards,

The ILS Staff
ILS@astm.org

W.R.Grace & Co	Jen	Smith	✓

Distributor:

Acme

Status: Precision statement on main ballot.

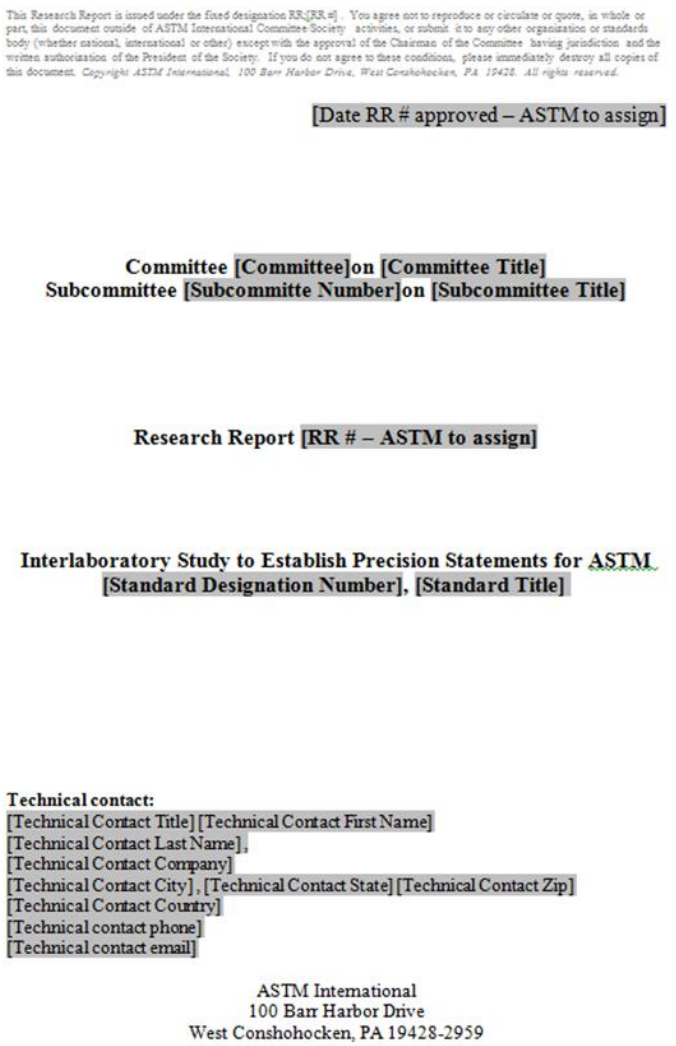
Please email any missing information regarding this Interlaboratory Study to ILS@astm.org. ASTM will need all of the study information to complete the research report.

Research Reports

- The ASTM Form and Style Manual (Section A29.1) states,
  - *"Where numerical data have been generated to establish the precision and bias of a test method, a research report is required."*
- ASTM Word Research Report Template
- The draft research report should be made available to committee members while the related precision statement is on ballot.
- Research report numbers are assigned after ballot approved.

Parts of a Research Report

- List of participating laboratories
- Description of samples with their suppliers
- Laboratory instructions
- General description of equipment/ apparatus used
- Raw data (lab name's hidden)
- Statistical summary
- Precision and bias statement



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[Date RR # approved – ASTM to assign]

Committee [Committee]on [Committee Title]
Subcommittee [Subcommitte Number]on [Subcommittee Title]

Research Report [RR # – ASTM to assign]

Interlaboratory Study to Establish Precision Statements for ASTM [Standard Designation Number], [Standard Title]

Technical contact:
[Technical Contact Title] [Technical Contact First Name] [Technical Contact Last Name] ,
[Technical Contact Company]
[Technical Contact City] , [Technical Contact State] [Technical Contact Zip]
[Technical Contact Country]
[Technical contact phone]
[Technical contact email]

ASTM International
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959

Research Report Numbers

- A research report number will be assigned by ASTM when all of the following have been completed:
  - The research report is submitted to ILS
  - It has been reviewed for completeness
  - The ballot item to include the corresponding precision and bias statement is approved for publication

Select: Register a New ILS Study www.astm.org/ILS

Interlaboratory Study Program

The Interlaboratory Study Program is provided at no cost as a benefit for all ASTM members to help you meet the precision statement requirements in ASTM test methods.

Register for a New ILS Program | Active ILS Program list | FAQs | Contact

The ASTM ILS Team Is Ready to Assist

Developing a Precision and Bias statement using an Interlaboratory Study can be a complicated process, but our experienced team is here to help. Contact the ILS Team for assistance with:

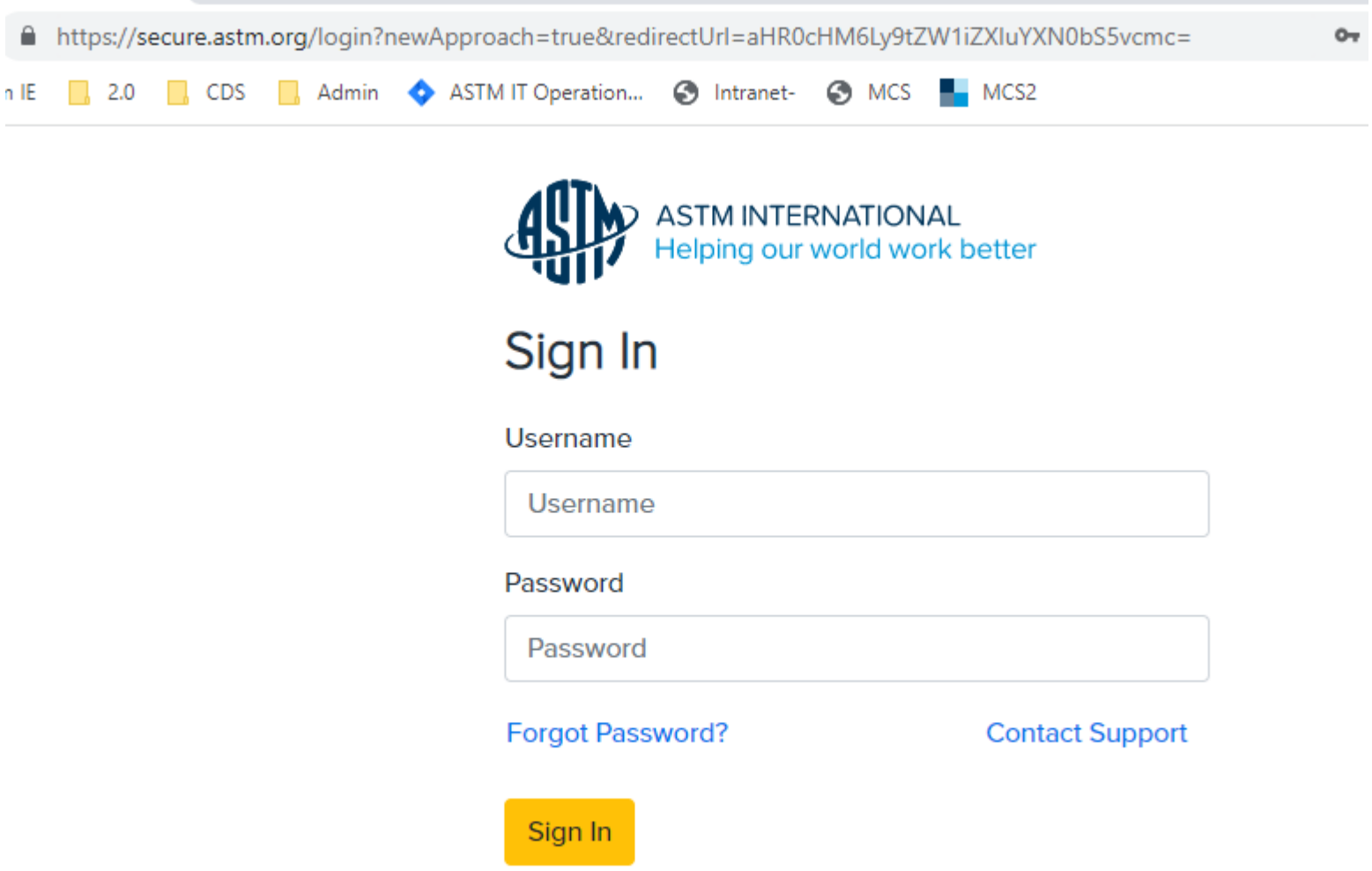
- Designing an Interlaboratory Study
- Identifying potential samples
- Soliciting volunteer laboratories
- Finding an available supplier
- Ordering and purchasing samples
- Contracting with a distributor
- Reviewing laboratory instructions
- Shipping expenses
- Collecting data
- Analyzing data
- Producing a draft precision statement
- Compiling information for the Research Report
- Recognition of participating labs

How It Works – The Interlaboratory Study (ILS) Process

Download the Infographic: Interlaboratory Studies Program

1. **Register Your Work Item**
2. **Register for Your ILS Program** using your MyASTM area of the website.
3. **Conference Call** ILS reps will schedule a call with the Technical Contact (TC). The TC should be prepared to discuss things like test specimens, lab supplies, potential suppliers, a distributor, a potential list of labs, and what data should be collected.
4. **Data Report Forms** We'll collect data using an excel data report form. The data report form will be sent to the TC for review before sending to the lab.
5. **Sample Distribution** Once the study materials are secured, it may be necessary to send them to a distributor for final prep and packaging.The distributor will then ship the samples to the participating labs, and ASTM will send the data report forms and instructions.
6. **Data Submission** ASTM will track data submitted by the lab participants.
7. **Statistical Summary** Once all data is received, we will compute the repeatability and reproducibility using ASTM Statistical Software. Initial statistics and a draft precision and bias statement will be sent to the TC for review.
8. **Research Report (RR)** ASTM will a draft research report for the TC to review.
9. **Precision and Bias Statement** The precision and bias statement (along with any other revisions to the standard) should be placed on the next ballot.
10. **Approval** After the ballot is approved and the research report is complete, ASTM will assign a Research Report number. Copies of the report will be sent to the TC and participating labs.

MyASTM Login



Register a New Study

MyASTM

ILS Home

ASTM.org

Register or Edit a Study

Need Help?

The ILS Staff can help you finish your registration.

email: ILS@astm.org

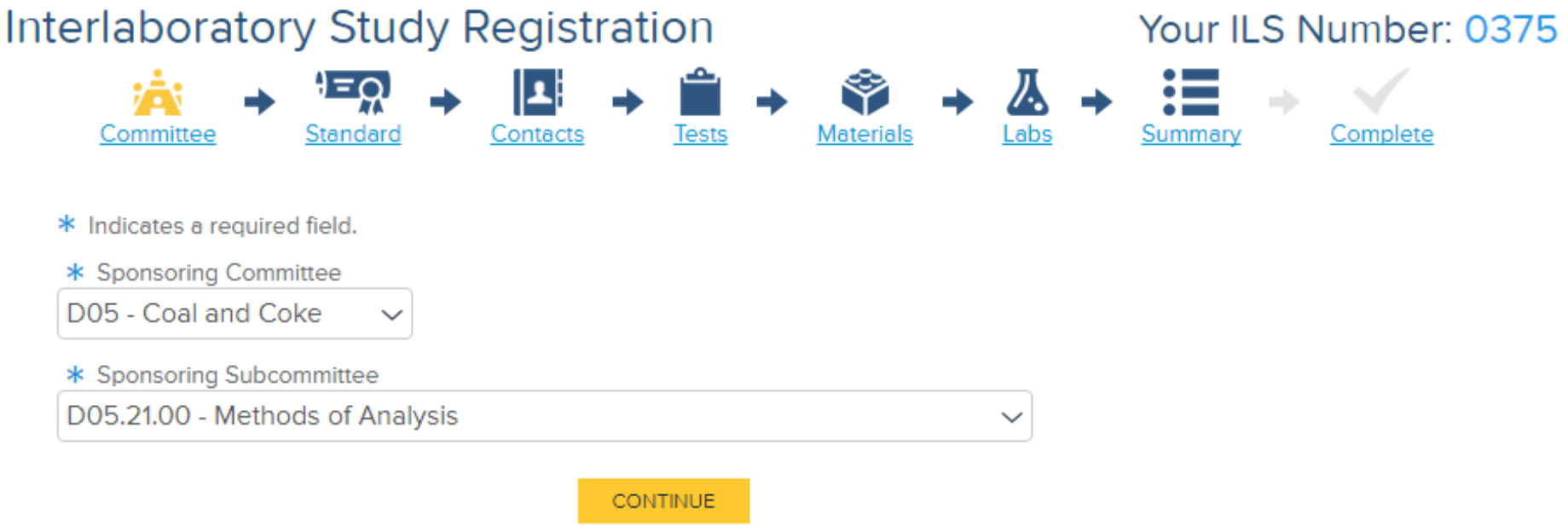
tel: +1.610.832.9746

Interlaboratory Study Registration

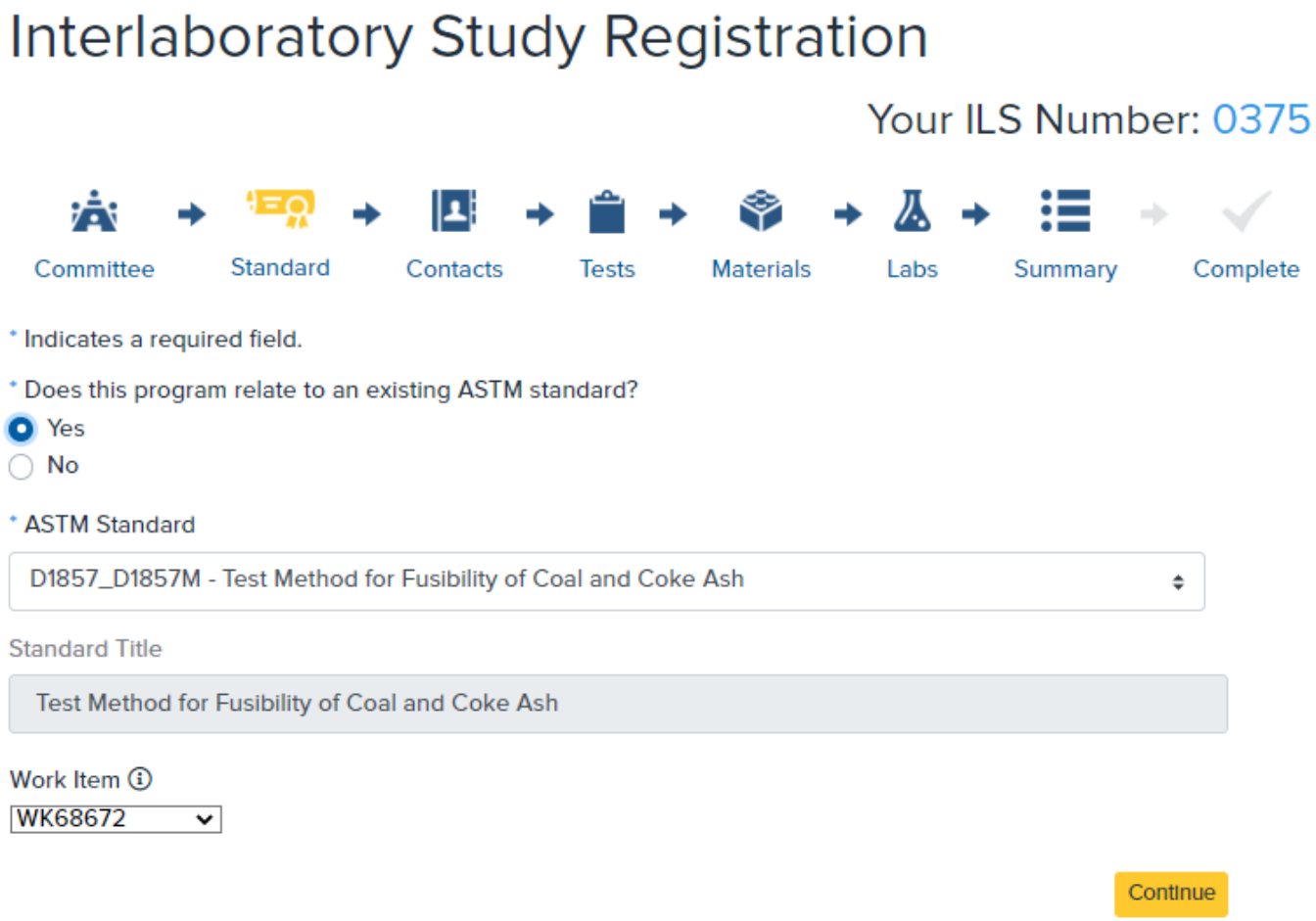
REGISTER A NEW STUDY

Technical contact understands and agrees that the data generated as a result of this study, regardless of any further support or services provided by or to ASTM, will be used in ASTM's business, to assist in developing a research report, consensus standard or adjunct thereto. Technical contact agrees to keep such data and results confidential and not to disclose or share the data/results with anyone else, without ASTM's written consent. For a copy of the ASTM International's Intellectual Property Policy click here.

Committee



Standard



Contacts

Tests

Interlaboratory Study Registration

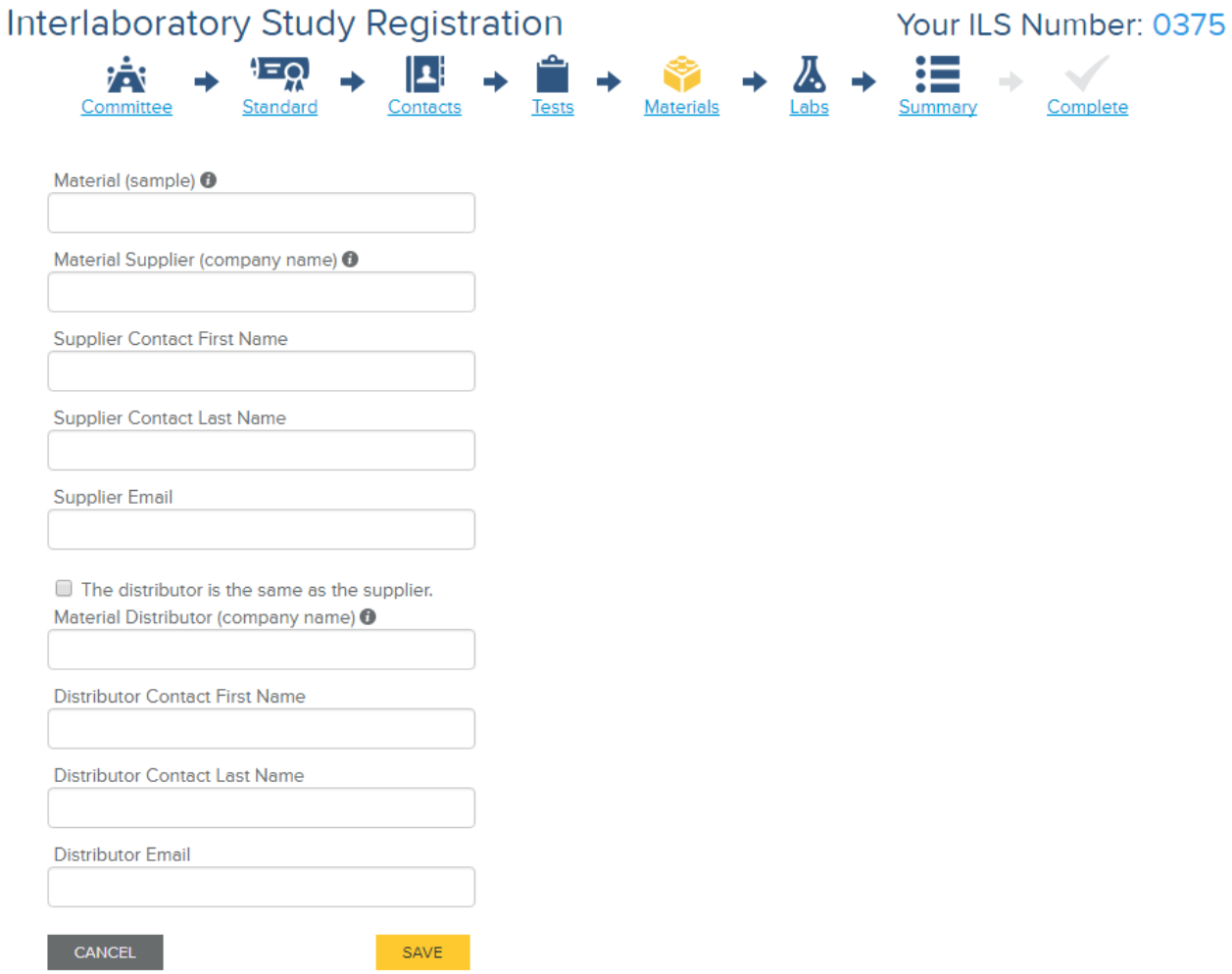
Your ILS Number: 0375

Committee → Standard → Contacts → Tests → Materials → Labs → Summary → Complete

Tests can be re-ordered by clicking and dragging the numbers, or by using the up/down arrow buttons.

	Tests ⓘ	Units of Measure ⓘ	
1.)	Day 1 Air- EqM- Pre-Wet		Remove
2.)	Day 1 Air- EqM- Not Pre-Wet		Remove
3.)	Day 2 Air- EqM- Pre-Wet		Remove
4.)	Day 2 Air- EqM- Not Pre-Wet		Remove
5.)	Day 1 Nitrogen- EqM- Pre-Wet		Remove
6.)	Day 1 Nitrogen- EqM- Not Pre-Wet		Remove
7.)	Day 2 Nitrogen- EqM- Pre-Wet		Remove

Materials, Supplier(s) & Distributor(s)



Materials

Materials can be re-ordered by clicking and dragging the numbers, or by using the up/down arrow buttons.

	Testing Material (sample) ⓘ	Material Supplier ⓘ	Distributor ⓘ	
1.)	CO-Bituminous	Colorado, USA	Standard Laboratories, Inc.	Edit Remove
2.)	PA-Bituminous	Pennsylvania, USA	Standard Laboratories, Inc.	Edit Remove
3.)	VA-Bituminous	Virginia, USA	Standard Laboratories, Inc.	Edit Remove
4.)	MT-Subbituminous B	Montana, USA	Standard Laboratories, Inc.	Edit Remove
5.)	WY-Subbituminous I	Wyoming, USA	Standard Laboratories, Inc.	Edit Remove
6.)	WY-Subbituminous	Wyoming, USA	Standard Laboratories, Inc.	Edit Remove
7.)	MS-Lignite	Mississippi, USA	Standard Laboratories, Inc.	Edit Remove
8.)	ND-Lignite	North Dakota, USA	Standard Laboratories, Inc.	Edit Remove
9.)	TX-Lignite II	Texas, USA	Standard Laboratories, Inc.	Edit Remove

Add Material

Continue

Laboratories

Download Lab Contact Information Form (Excel)

ADD LABS

Please fill in the Lab Contact Information form and email it back to lls@astm.org

Laboratory ⓘ	
DONG Energy	Edit Remove
SGS Newcastle	Edit Remove
ALS	Coal Division GV	Edit Remove
ALS	Coal Division GD	Edit Remove
BHP Billiton Mitsubishi Alliance	Edit Remove
Standard Laboratories, Inc. CY	Edit Remove
Standard Laboratories, Inc. RW	Edit Remove
Mineral Labs, Inc.	Edit Remove
SGS North America Inc.	Edit Remove
SGS Henderson KY US	Edit Remove
Standard Laboratories, Inc. AS	Edit Remove
SGS China Tianjin Energy Lab	Edit Remove
SGS - Tianjin China	Edit Remove
WY Analytical Laboratories, Inc	Edit Remove
Incolab Services Colombia S.A.S.	Edit Remove
BVIT Newcastle	Edit Remove

Add Another Lab

Continue

Laboratory Information Form

ASTM INTERNATIONAL
Helping our world work better

ILS # 0375

Lab Info Form

Click here to email this completed lab info form to: ils@astm.org

Laboratory Name	Contact First Name	Contact Last Name	Email	Phone #	Address 1	Address 2	City	State (Full name)	Zip Code	Country (Full name)

Registration Summary – Submit to ASTM

Please review the following summary of your ILS program before submitting to ASTM.

PRINT FRIENDLY

SUBMIT REGISTRATION TO ASTM

Select to Edit

Committee

Committee	D05 - Coal and Coke
Sub Committee	D05.21.00 - Methods of Analysis

Standard

Related Standard	D1857_D1857M - Test Method for Fusibility of Coal and Coke Ash
ILS Title	Test Method for Fusibility of Coal and Coke Ash
Work Item	WK68672

Contacts

Technical Contact		
First Name	Last Name	Email
Melissa	Marcinowski	mmarcinowski@astm.org

Statistical Support
ASTM to provide statistical support

Tests

Tests	Units of Measure
Day 1 Air- EqM- Pre-Wet	
Day 1 Air- EqM- Not Pre-Wet	
Day 2 Air- EqM- Pre-Wet	
Day 2 Air- EqM- Not Pre-Wet	
Day 1 Nitrogen- EqM- Pre-Wet	
Day 1 Nitrogen- EqM- Not Pre-Wet	
Day 2 Nitrogen- EqM- Pre-Wet	
Day 2 Nitrogen- EqM- Not Pre-Wet	

Materials

Testing Material (sample)	Material Supplier	Distributor
CO-Bituminous	Colorado, USA	Standard Laboratories, Inc.
PA-Bituminous	Pennsylvania, USA	Standard Laboratories, Inc.
VA-Bituminous	Virginia, USA	Standard Laboratories, Inc.
MT-Subbituminous B	Montana, USA	Standard Laboratories, Inc.
WY-Subbituminous I	Wyoming, USA	Standard Laboratories, Inc.
WY-Subbituminous	Wyoming, USA	Standard Laboratories, Inc.
MS-Lignite	Mississippi, USA	Standard Laboratories, Inc.
ND-Lignite	North Dakota, USA	Standard Laboratories, Inc.
TX-Lignite II	Texas, USA	Standard Laboratories, Inc.

Labs

Laboratory
DONG Energy
SGS Newcastle
ALS I Coal Division GV
ALS I Coal Division GD
BHP Billiton Mitsubishi Alliance
Standard Laboratories, Inc. CY
Standard Laboratories, Inc. RW
Standard Laboratories, Inc. ES
Mineral Labs, Inc.
SGS North America Inc.
SGS Henderson KY US
Standard Laboratories, Inc. AS
SGS China Tianjin Energy Lab
SGS - Tianjin China
WY Analytical Laboratories, Inc
Incolab Services Colombia S.A.S.
BVIT Newcastle

PRINT FRIENDLY

Technical contact understands and agrees that the data generated as a result of this study, regardless of any further support or services provided by or to ASTM, will be used in ASTM's business, to assist in developing a research report, consensus standard or adjunct thereto. Technical contact agrees to keep such data and results confidential and not to disclose or share the data/results with anyone else, without ASTM's written consent. For a copy of the ASTM International's Intellectual Property Policy click here.

SUBMIT REGISTRATION TO ASTM

Key Takeaways

- In each participating laboratory, **one lab technician** should conduct **all ILS testing**
- Labs should follow the ASTM standard provided to them for the ILS, completing the testing in the **shortest** possible period of time
- Full ILS - we need good usable data from a **minimum of 6** labs
- Do not send samples to **ASTM Headquarters**

The ILS Program is a **FREE** member benefit, available to all committees, for members working on ASTM Test Methods.

Questions

Additional Classroom for Member Trainings

- New Member Orientation & Training
- Balloting & Handling Negatives Votes
- WebEx Training
- Roster Maintenance
- Process of Developing & Revising a Standard
- Task Group Chair & Technical Contact Responsibilities
- Subcommittee Chair's Duties and Responsibilities
- Interlaboratory Studies Program
- Planning Symposia & Workshops
- Collaboration Area Training

Contact Information

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Caitlin Farrell,
Project Manager
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Interlaboratory Study Program (ILS)

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