ASTM International Interlaboratory Study Program (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...
• 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.
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

- Ruggedness Study
- Finalize Test Method
- Pilot Program (repeatability)
  F&S requirement met for 5 years
- Full ILS (repeatability + Reproducibility)
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

<table>
<thead>
<tr>
<th></th>
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</tr>
</thead>
<tbody>
<tr>
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<td>5</td>
<td>6</td>
<td>4</td>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>

**Average:** 4.4 4.8 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

<table>
<thead>
<tr>
<th>Sample Name/ Lab Name</th>
<th>University of Calgary</th>
<th>Marathon Oil</th>
<th>Alberta Research Council</th>
<th>Phillips 66 OK</th>
<th>LyondellBasell</th>
<th>ExxonMobil Research</th>
<th>Agilent Technologies</th>
<th>Triton Analytics Corp</th>
<th>Envantage Inc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kerosene</td>
<td>1, 7</td>
<td>2, 12</td>
<td>5, 6</td>
<td>7, 9</td>
<td>4, 6</td>
<td>2, 11</td>
<td>5, 10</td>
<td>2, 5</td>
<td>8, 11</td>
</tr>
<tr>
<td>High Sulfur Diesel</td>
<td>3, 6</td>
<td>3, 10</td>
<td>9, 10</td>
<td>5, 10</td>
<td>2, 11</td>
<td>5, 10</td>
<td>9, 12</td>
<td>1, 4</td>
<td>2, 6</td>
</tr>
<tr>
<td>#2 Low Sulfur Diesel</td>
<td>5, 12</td>
<td>5, 11</td>
<td>4, 11</td>
<td>3, 11</td>
<td>8, 9</td>
<td>11, 12</td>
<td>1, 10</td>
<td>6, 10</td>
<td>4, 11</td>
</tr>
<tr>
<td>Aviation Turbine Jet A</td>
<td>2, 4</td>
<td>4, 6</td>
<td>2, 8</td>
<td>1, 6</td>
<td>3, 10</td>
<td>2, 8</td>
<td>3, 4</td>
<td>2, 9</td>
<td>5, 9</td>
</tr>
<tr>
<td>Ultra Low Sulfur Diesel</td>
<td>8, 11</td>
<td>7, 9</td>
<td>3, 7</td>
<td>2, 4</td>
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<td>1, 6</td>
<td>6, 11</td>
<td>3, 12</td>
<td>1, 3</td>
</tr>
<tr>
<td>Light Cycle Oil</td>
<td>9, 10</td>
<td>1, 8</td>
<td>1, 12</td>
<td>8, 12</td>
<td>1, 12</td>
<td>3, 9</td>
<td>7, 8</td>
<td>5, 7</td>
<td>8, 12</td>
</tr>
</tbody>
</table>
Data Report Form- Instructions

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

<table>
<thead>
<tr>
<th>Measured % gel Content</th>
<th>Material X-1</th>
<th>Material X-2</th>
<th>Material X-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Replicate 1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicate 2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicate 3</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Measured % gel Content</th>
<th>Material Y-1</th>
<th>Material Y-2</th>
<th>Material Y-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Replicate 1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicate 2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicate 3</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Measured % gel Content</th>
<th>Material Z-1</th>
<th>Material Z-2</th>
<th>Material Z-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Replicate 1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicate 2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicate 3</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments:

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

Laboratory understands and agrees that the data generated as a result of the services 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

large bias + high precision = low accuracy
zero bias + high precision = high accuracy
large bias + low precision = low accuracy
zero bias + low precision = low accuracy
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
ILS# 0018 Committee Week Status Report

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

June 9, 2017

Subcommittee: D32.05
Technical Contact: Thomas Szymanski
Staff Manager: Kelly Paul
Work Item Number: WK29951
Registered Date: April 26, 2006
Statistical Support: THOMAS SYMANSKI
Tests:
1. Relative Crystallinity %

Materials:
1. D32-88-002
2. Standard - Supplied by: Acme
3. D32-88-664 - Supplied by: Acme
4. D32-88-003 - Supplied by: Acme

Tests:

<table>
<thead>
<tr>
<th>Lab Name</th>
<th>Contact Name</th>
<th>Data Submitted</th>
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</thead>
<tbody>
<tr>
<td>Abemarle Bayport</td>
<td>Mark</td>
<td></td>
</tr>
<tr>
<td>Chvron</td>
<td>James</td>
<td></td>
</tr>
<tr>
<td>Hakutzu Topco A/S</td>
<td>Shaun</td>
<td></td>
</tr>
<tr>
<td>INEOS</td>
<td>Joe</td>
<td></td>
</tr>
<tr>
<td>Lumus Technology Inc. TL-1</td>
<td>Diane</td>
<td></td>
</tr>
<tr>
<td>Sino-Guine Norpro</td>
<td>Jacki</td>
<td></td>
</tr>
<tr>
<td>UOP</td>
<td>Terri</td>
<td></td>
</tr>
</tbody>
</table>

W.R. Grace & Co    Jen       Smith    ✔

Distributors:
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.
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
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

Interlaboratory Study Program

ASTM’s Board of Directors adopted plans, in the fall of 2004, to launch the Interlaboratory Study Program (ILS) as part of their continuing pursuit of excellence in standards development. Responding to the need for standards in the marketplace to be of known and documented quality, a commitment was made to fund the development of the ILS Program. This commitment means that ASTM has been able to assist technical committees for which the prospect of implementing an interlaboratory study was either administratively daunting or financially impossible. The ultimate goal is to enhance the quality of ASTM standard test methods by aiding the Technical Committees as they develop Precision Statements backed by high quality laboratory data.

In order to support the committee’s efforts to produce precision statements for their test methods, so as to incorporate at least a repeatability statement, the ILS Program is available to assist with the following areas:

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

Once a Work Item has been requested (Instructions here), new programs should be registered through the Interlaboratory Study (ILS) link in the My Tools section of the member’s MyASTM page, available after logging into ASTM’s website.

Active ILS Programs &
ILS Process
Precision and Repeatability
Research Reports
FAQ

Interlaboratory Study (ILS) Process
1. Work Item - register a work item (if you have not done so already).
2. Registration - register the ILS through the MyASTM area of the website.
3. Conference Call - The Technical Contact (TC) will be contacted by ASTM to set up a conference call to discuss the ILS. During this call the TC should be prepared to discuss the following information: test specimen, lab supplies, potential suppliers, a distributor, a potential list of labs, and what data should be collected as part of the study.
4. Samples - if assistance is needed with finding or ordering materials to be tested, ASTM can provide support.
5. Sample Funding - ASTM will fund funds available to assist with purchasing samples, sample preparation and shipping. After a proposal of costs is developed, ASTM will work with the committee to determine how much assistance can be provided.
6. Lab Solicitation - if there are not enough labs to do the testing, ASTM can send out a solicitation to the committee looking for volunteers.
7. Lab Instructions - the study instructions should be developed and finalized before samples are distributed. Please send them to ASTM for review.
8. Data Report Form - ASTM will collect the study data using an online data report form or an excel data report form depending on the amount of complexity of the data. The data report form will be sent to the technical contact for review before the labs receive it.
9. Sample Distribution - once the study materials have been purchased, it may be necessary to send them to a distributor for final packaging and shipping (may be the same as the supplier). The distributor will then ship the samples. ASTM will send this data report form and the study instructions to the labs.
10. Data Submission - ASTM will track data submitted by the lab participants.
11. Statistical Summary - once all of the data has been received from the labs, ASTM will then complete the final report and statistical summary using ASTM StatCalc Software. A draft precision and bias statement will be written and sent to the TC for review.
MyASTM Login

Sign In

Username

Password

Forgot Password? Contact Support

Sign In
Register a New Study

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

Interlaboratory Study Registration

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

Your ILS Number: 0375

* Indicates a required field.

- Sponsoring Committee
  - D05 - Coal and Coke

- Sponsoring Subcommittee
  - D05.21.00 - Methods of Analysis

CONTINUE
Interlaboratory Study Registration

Your ILS Number: 0375

* Indicates a required field.

* Does this program relate to an existing ASTM standard?
  - Yes
  - No

* ASTM Standard

D1857_D1857M - Test Method for Fusibility of Coal and Coke Ash

Standard Title

Test Method for Fusibility of Coal and Coke Ash

Work Item

WK68672

Continue
Contacts

Interlaboratory Study Registration

Your ILS Number: 0375

Technical Contact

<table>
<thead>
<tr>
<th>First Name</th>
<th>Last Name</th>
<th>Email</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melissa</td>
<td>Marcinowski</td>
<td><a href="mailto:mmarcinowski@astm.org">mmarcinowski@astm.org</a></td>
</tr>
</tbody>
</table>

Statistical Support

Please indicate the person who will provide statistical support for this ILS, or indicate below that assistance is required.

- [ ] ASTM to assist with statistical support.
- [ ] I will provide statistical support for this ILS.
- [ ] A committee member will provide statistical support.

Select One

[ ] Statistical support will be provided by the following person:

Continue
## Interlaboratory Study Registration

Your ILS Number: 0375

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

<table>
<thead>
<tr>
<th>Tests</th>
<th>Units of Measure</th>
<th>Remove</th>
</tr>
</thead>
<tbody>
<tr>
<td>1)</td>
<td>Day 1 Air - EqM - Pre-Wet</td>
<td></td>
</tr>
<tr>
<td>2)</td>
<td>Day 1 Air - EqM - Not Pre-Wet</td>
<td></td>
</tr>
<tr>
<td>3)</td>
<td>Day 2 Air - EqM - Pre-Wet</td>
<td></td>
</tr>
<tr>
<td>4)</td>
<td>Day 2 Air - EqM - Not Pre-Wet</td>
<td></td>
</tr>
<tr>
<td>5)</td>
<td>Day 1 Nitrogen - EqM - Pre-Wet</td>
<td></td>
</tr>
<tr>
<td>6)</td>
<td>Day 1 Nitrogen - EqM - Not Pre-Wet</td>
<td></td>
</tr>
<tr>
<td>7)</td>
<td>Day 2 Nitrogen - EqM - Pre-Wet</td>
<td></td>
</tr>
</tbody>
</table>
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.

<table>
<thead>
<tr>
<th>Testing Material (sample)</th>
<th>Material Supplier</th>
<th>Distributor</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) CO-Bituminous</td>
<td>Colorado, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>2) PA-Bituminous</td>
<td>Pennsylvania, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>3) VA-Bituminous</td>
<td>Virginia, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>4) MT-Subbituminous B</td>
<td>Montana, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>5) WY-Subbituminous I</td>
<td>Wyoming, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>6) WY-Subbituminous</td>
<td>Wyoming, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>7) MS-Lignite</td>
<td>Mississippi, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>8) ND-Lignite</td>
<td>North Dakota, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
<tr>
<td>9) TX-Lignite II</td>
<td>Texas, USA</td>
<td>Standard Laboratories, Inc.</td>
</tr>
</tbody>
</table>
# Laboratories

Download Lab Contact Information Form (Excel)

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

<table>
<thead>
<tr>
<th>Laboratory</th>
<th>Edit</th>
<th>Remove</th>
</tr>
</thead>
<tbody>
<tr>
<td>DONG Energy</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SGS Newcastle</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ALS Coal Division GV</td>
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<tr>
<td>ALS Coal Division GD</td>
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<tr>
<td>BHP Billiton Mitsubishi Alliance</td>
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<tr>
<td>Standard Laboratories, Inc. CY</td>
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<tr>
<td>Standard Laboratories, Inc. RW</td>
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<tr>
<td>Mineral Labs, Inc.</td>
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<tr>
<td>SGS North America Inc.</td>
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<td>SGS Henderson KY US</td>
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<tr>
<td>Standard Laboratories, Inc. AS</td>
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</tr>
<tr>
<td>SGS China Tianjin Energy Lab</td>
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<tr>
<td>SGS - Tianjin China</td>
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<tr>
<td>WY Analytical Laboratories, Inc</td>
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<tr>
<td>Incolab Services Colombia S.A.S.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>BVIT Newcastle</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Laboratory Information Form

ILS # 0375

Lab Info Form

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

<table>
<thead>
<tr>
<th>Laboratory Name</th>
<th>Contact First Name</th>
<th>Contact Last Name</th>
<th>Email</th>
<th>Phone #</th>
<th>Address 1</th>
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<th>City</th>
<th>State (Full name)</th>
<th>Zip Code</th>
<th>Country (Full name)</th>
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</thead>
</table>
Registration Summary – Submit 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
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