Overview of ASTM F04.15.14 Task Group on Corrosion Testing

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ASTM F04.15.14 Corrosion Testing

- F746-04(2014) Standard Test Method for Pitting or Crevice Corrosion of Metallic Surgical Implant Materials
- F897-02(2013) Standard Test Method for Measuring Fretting Corrosion of Osteosynthesis Plates and Screws
- F1089-10 Standard Test Method for Corrosion of Surgical Instruments
- F1801-97(2014) Standard Practice for Corrosion Fatigue Testing of Metallic Implant Materials
- F1875-98(2014) Standard Practice for Fretting Corrosion Testing of Modular Implant Interfaces: Hip Femoral Head-Bore and Cone Taper Interface
- F2129-15 Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices
- F3044-14 Test Method for Standard Test Method for Evaluating the Potential for Galvanic Corrosion for Medical Implants
- WK52215 * Ion Release Evaluation of Medical Implants

ASTM F04.15.14 Corrosion Testing

- This talk is going to focus on work that has been done by the Corrosion Testing Task Group to plan and implement inter-laboratory testing programs to create ASTM Precision and Bias Statements

- Currently, the Task Group is planning an inter-laboratory testing protocol to generate a Precision and Bias statement for F3044-14 Test Method for Standard Test Method for Evaluating the Potential for Galvanic Corrosion for Medical Implants

- Previously, the Task Group planned and completed an inter-laboratory testing program to generate a Precision and Bias statement for F2129 Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices

ASTM F04.15.14

- **Precision and Bias Statement for F2129**

✓ 12 Laboratories and 4 materials

✓ Each Laboratory tested 8 samples per material

✓ Repeatability and Reproducibility statement for 4 variables

1. Rest Potential, Er
2. Breakdown Potential, Eb
3. Repassivation Potential, Ep
4. Breakdown Potential minus Rest Potential, Eb - Er

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11.5 A copy of the cyclic polarization curve should be provided in the report.

11.6 A generic description of the appearance of any corrosion observed on the specimen should be described. Photographic documentation may be appropriate.

12. Precision and Bias

12.1 An interlaboratory study was conducted in accordance with Practice E691 in twelve laboratories with four different materials. Each laboratory tested eight samples per material. The details of this study are provided in an ASTM Research Report.[5] The results are summarized in Tables 1-4, which provide the repeatability and reproducibility statistics for each output parameter from the test. The terms repeatability limit and reproducibility limit are used as specified in Practices E177 and E691. As defined in Practice E691, repeatability is concerned with the variability between independent test results within one laboratory under tightly controlled conditions. Reproducibility is concerned with the variability between independent test results in different laboratories. No measurement bias is possible with this test method since there is no accepted reference material. No precision statement is possible for the repassivation potential, E_p, for 316LVM stainless steel or 455 stainless steel since there was insufficient data to generate the statistics. Neither of these materials exhibited repassivation in the majority of the experiments.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F04-1011.

TABLE 1 Precision of Rest Potential E_r (mV)

Material	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	–7	33	64	93	178
455 SS	–30	38	67	105	187
Nitinol A	–519	35	49	98	137
Nitinol B	–482	21	49	60	138

TABLE 2 Precision of Breakdown Potential E_b (mV)

Material	Grand Mean	Repeatability StandardDeviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	679	161	190	451	531
455 SS	269	36	40	100	113
Nitinol A	160	82	108	230	302
Nitinol B	180	54	94	152	263

TABLE 3 Precision of Repassivation Potential E_p (mV)

Material	Grand Mean	Repeatability StandardDeviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	...	...	...	...	...
455 SS	...	...	...	...	...
Nitinol A	–171	57	108	160	302
Nitinol B	–126	38	58	107	162

13. Keywords

TABLE 4 Precision of Breakdown Potential minus Rest Potential: $E_b - E_r$ (mV)

Material	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	674	154	176	432	494
455 SS	298	47	69	132	192
Nitinol A	679	83	110	232	309
Nitinol B	662	57	92	159	257

ASTM F 3044 Inter-laboratory Testing Program*

- **Background of ASTM F 3044**
  - ✓ ASTM F 3044 was formally approved in 2014
  - ✓ Test method for evaluating galvanic corrosion in medical implants. Prompted by concern with possible galvanic corrosion of overlapping cardiovascular stents. However, standard has many other applications, including dentistry (e.g., gold alloy crown touching amalgam restorative).
  - ✓ Per ASTM, test methods require a Precision and Bias Statement
  - ✓ Currently, no Precision and Bias statement – requires inter-laboratory testing to establish

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ASTM F 3044 Inter-laboratory Testing Program*

- **ASTM Inter-laboratory Study (ILS) Program – Provides support for ILS studies**
  - ✓ Review of experimental design
  - ✓ Assistance identifying volunteer laboratories
  - ✓ Identification of sample vendors
  - ✓ Coordination of sample distribution
  - ✓ Data Collection
  - ✓ Statistical Processing
  - ✓ Generation of Reports

Questions: ILS@astm.org
Philip Godorov, ILS Director
Caitlin Farell, ILS Project Manager
https://www.astm.org/ILS/

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ASTM F 3044 Inter-laboratory Testing Program*

- **Requirements for an Inter-laboratory Study**

✓ Multiple participating laboratories (n=6 minimum required per ASTM E691, within 5 years of the standard being published)

1. To establish reproducibility (R) from laboratory to laboratory
2. One operator; one piece of equipment per laboratory; and all tests should be run within as short of a time frame as possible

✓ Multiple replicate samples – to establish repeatability (r) within a single laboratory

✓ Testing conditions – what materials to test, how many material combinations, etc.

✓ Will provide a provide a Precision statement (R and r) for ASTM F3044. We would need a certified standard material to create a Bias statement

- **F 3044 ILS chaired by Dr. Shari Rosenbloom, WL Gore**

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ASTM F 3044 Inter-laboratory Testing Program*

- **Participating Laboratories –** (demonstrates diverse membership in Corrosion Testing Task Group)
  - ✓ WL Gore
  - ✓ Corrosion Testing Laboratories, Inc.
  - ✓ FDA
  - ✓ Memry
  - ✓ Heraeus Precious Metals GmbH
  - ✓ American Dental Association
  - ✓ Univeristy of Mississippi, Dept of Biomedical Materials Science
  - ✓ PneumRx, Inc.
  - ✓ Cortronik GmbH
  - ✓ EndoLab Mechanical Engineering GmbH
  - ✓ ?

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ASTM F 3044 Inter-laboratory Testing Program*

- **Replicate tests – how many replicates per test condition should be run?**
  - ✓ Depends on the variability in the data
  - ✓ Also, it depends on the test itself:
    1. How burdensome is it to run the standard?
    2. How expensive is it to run the standard?
  - ✓ Need to get enough data to be able to develop the Precision and Bias statement
  - ✓ ASTM does not have a hard-and-fast rule on the number of replicates. They have had as few as n=2 (example, an ASTM standard that required burning down a house) and as many as 100 (example, testing hospital gloves).
  - ✓ In the experience of a corrosion testing laboratory that has run multiple galvanic corrosion tests, n=3 has been adequate for reproducibility. Some of their clients ask for running n=5 replicates. Total number still open for discussion depending on the final number of material combinations...

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ASTM F 3044 Inter-laboratory Testing Program*

- **Material Combinations**
  - ✓ Considerations:
    1. Testing the Test Method, so not trying to evaluate the material. Therefore, we want to choose materials so that they exhibit consistent behavior.
    2. Use different surface area ratios?
    3. Different materials?
    4. Don't' have to use medical grade materials. However, it would probably be a good idea.
  - ✓ Suggestions:
    1. 316 stainless steel
    2. Cobalt Chromium Alloy
    3. Platinum/Iridium Alloy
  - ✓ Questions:
    1. Reuse the cathode (due to expense)?
    2. How many different combinations of materials?
    3. Which combinations of materials
  - ✓ Ft. Wayne Metals has offered to donate the materials

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ASTM F 3044 Inter-laboratory Testing Program*

- **Some Details of the Inter-laboratory Testing Program**

✓ Data/Laboratories will be blinded:

1. Each laboratory will know their own data.
2. Each laboratory will receive all other results as blinded
3. ASTM ILS Study Program will do data collection and blind the data

✓ Next Steps:

1. Register the study through the ASTM ILS Study Program
2. Continue meeting with ILS volunteers throughout the year through conference call or WebEx

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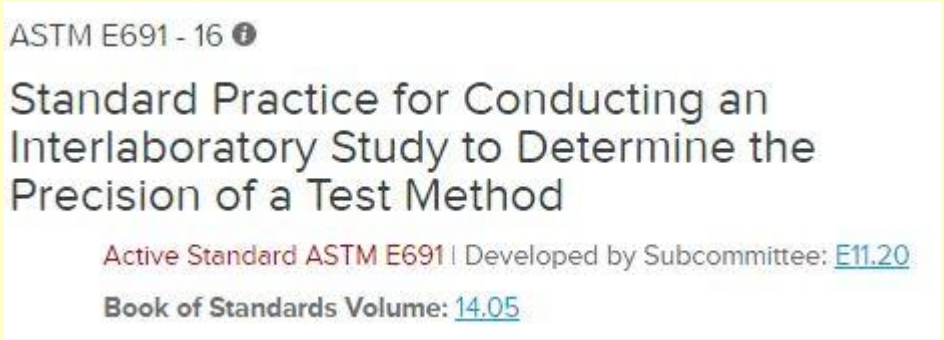
ASTM F 2129 Inter-laboratory Study*

- **This part of talk presents some of the inter-laboratory test results for ASTM F 2129**



- Purpose of analysis according to ASTM E691

1. To determine whether the data is consistent enough to form the basis for a test method Precision Statement.
2. To act on any data considered to be inconsistent.
3. To obtain precision statistics on which the precision statement can be based.

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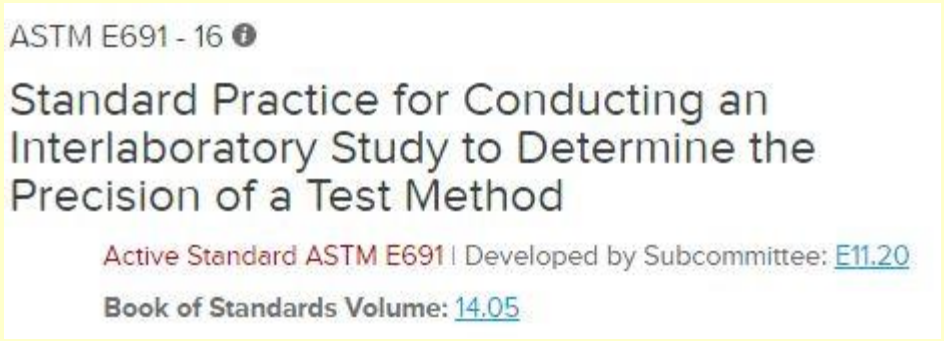
ASTM F 2129 Inter-laboratory Study*


ASTM E691 - 16

Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

Active Standard ASTM E691 | Developed by Subcommittee: E11.20

Book of Standards Volume: 14.05

- Consistency Statistics from ASTM E691:
  - ✓ **h** measures repeatability between laboratories.
  - ✓ **k** measures reproducibility within laboratories.
  - ✓ Critical values listed in E691 in Table 5 for 12 laboratories and 8 samples/material:
    1. **h**crit = 2.38
    2. **k**crit = 1.64
  - ✓ If all **h** and **k** values are less than **h**crit and **k**crit then data is consistent enough to form basis for precision statement.

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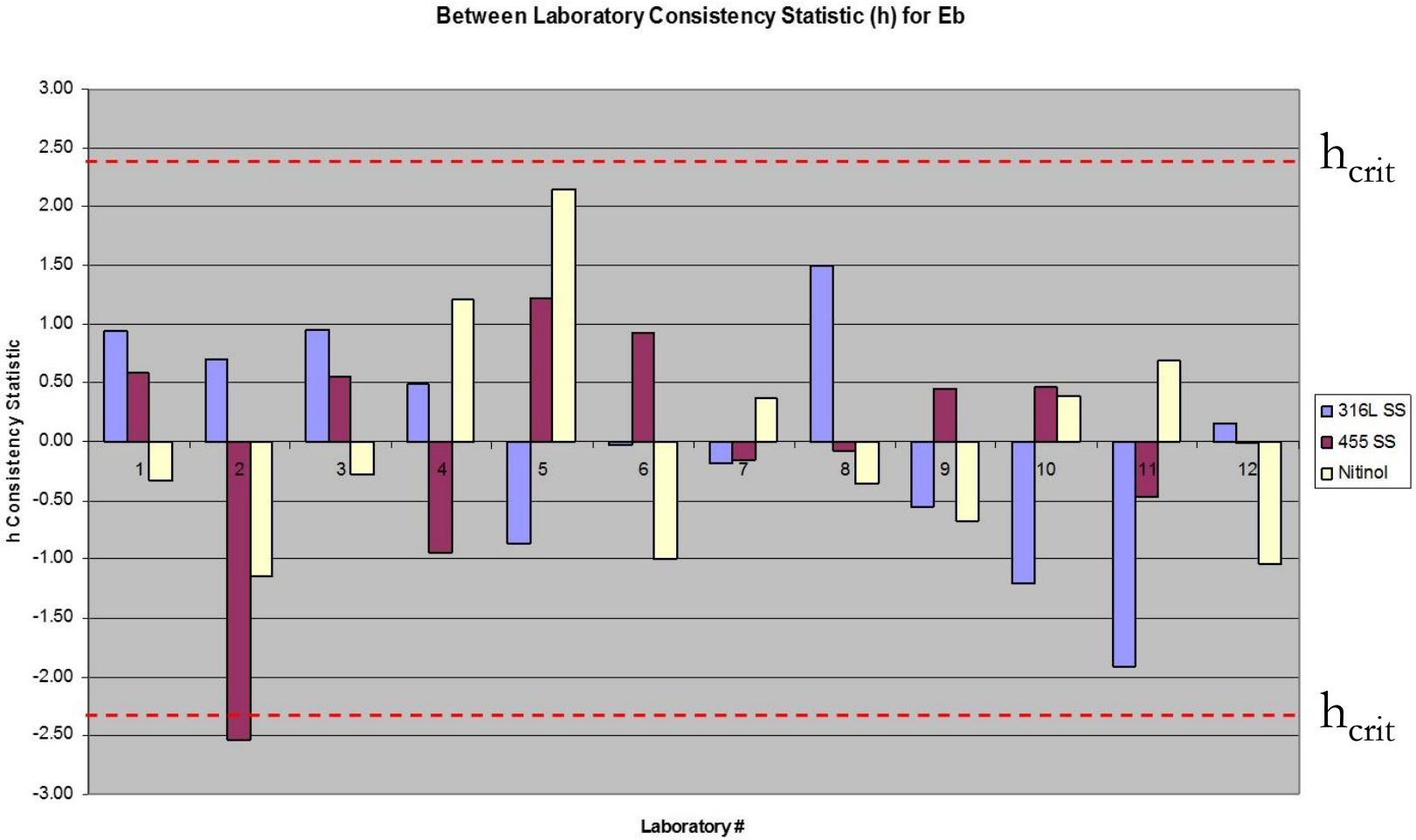
ASTM F 2129 Inter-laboratory Study*

Breakdown Potential (Eb) values all within **h**crit values except 1



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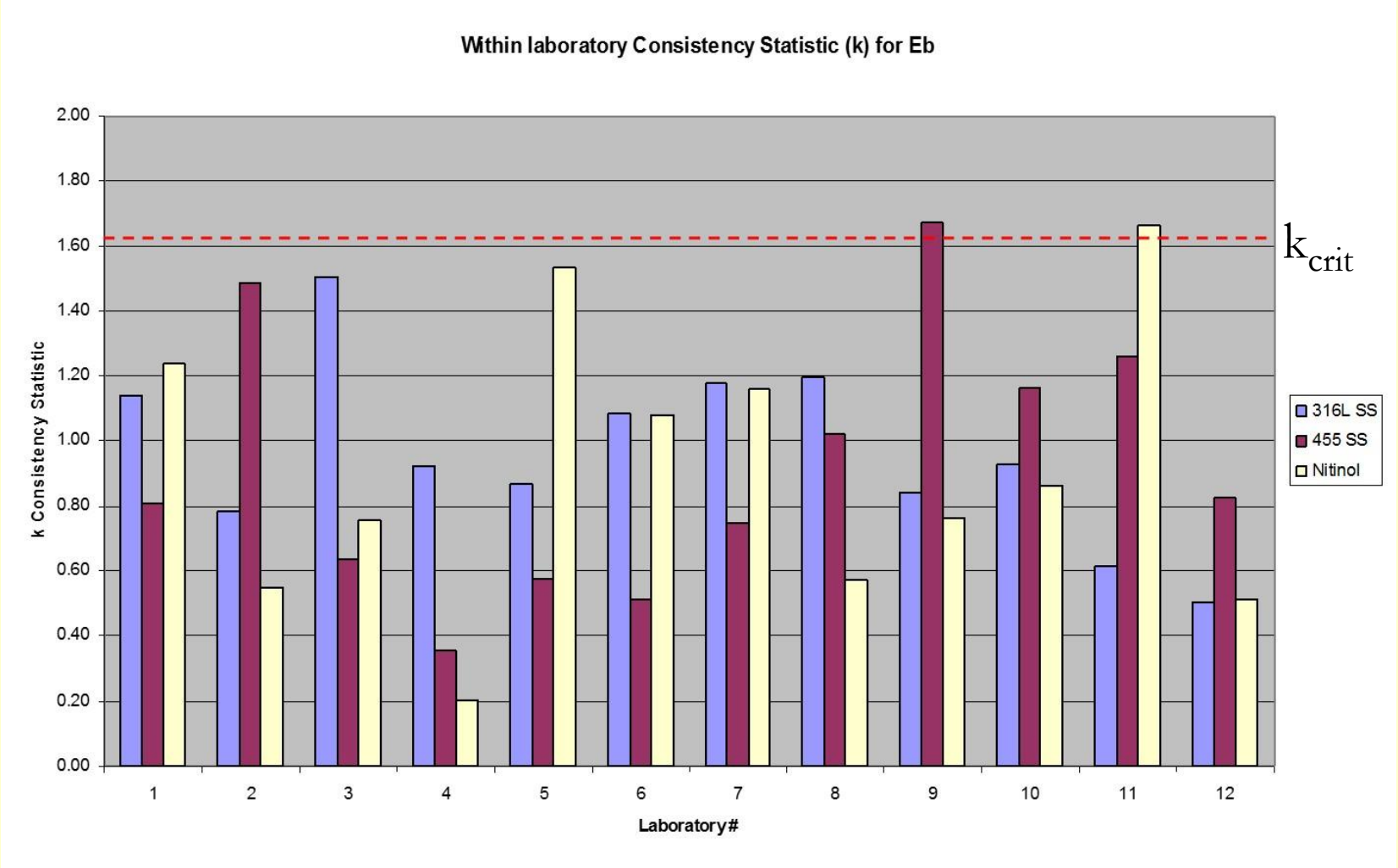
ASTM F 2129 Inter-laboratory Study*

Breakdown Potential (Eb) values all within **k**crit values except 2



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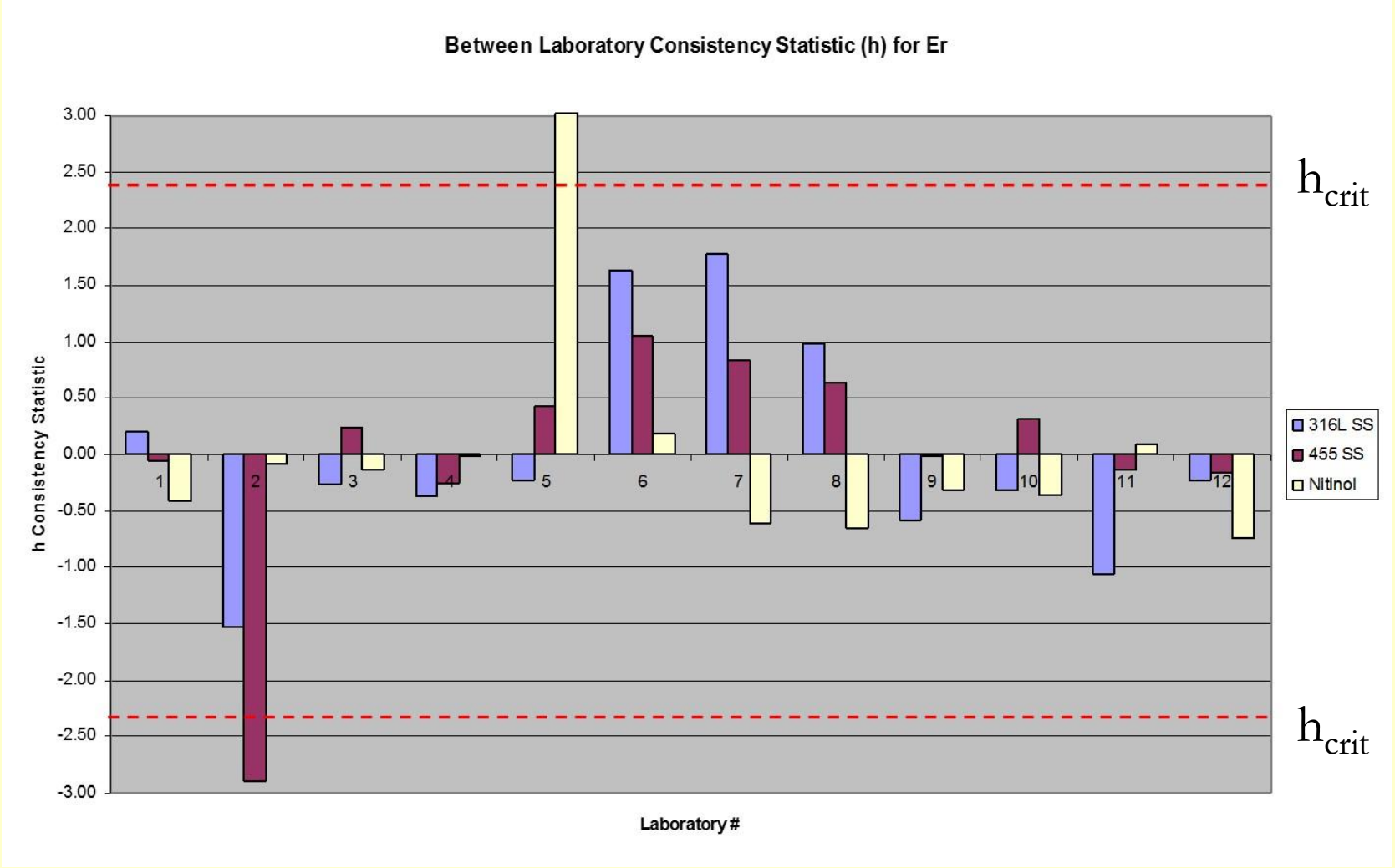
ASTM F 2129 Inter-laboratory Study*

Rest Potential (Er) values all within **h**crit values except 2



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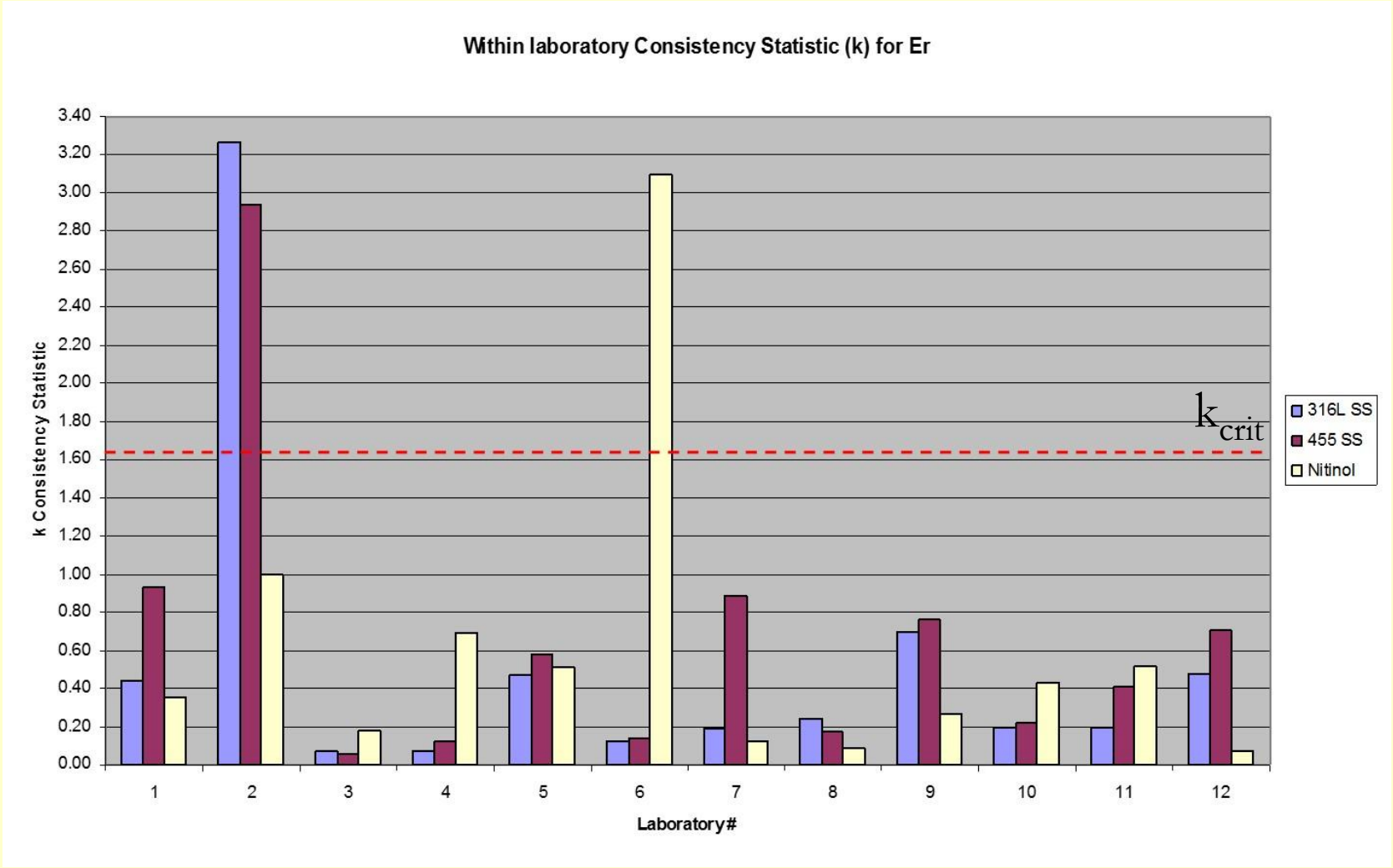
ASTM F 2129 Inter-laboratory Study*

Rest Potential (Er) values all within **k**crit values except 3



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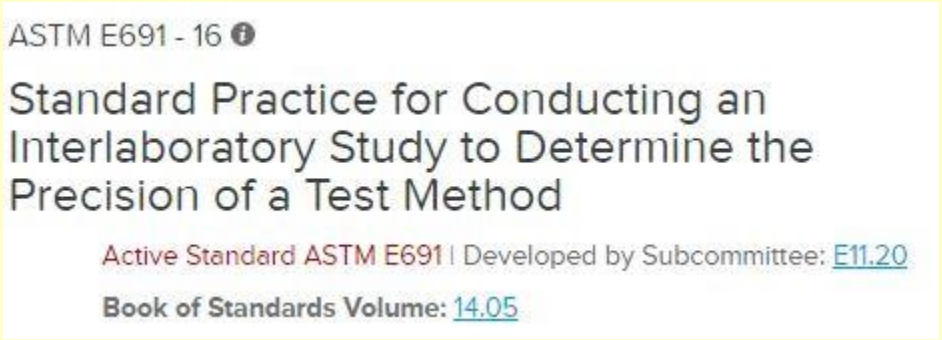
ASTM F 2129 Inter-laboratory Study*


ASTM E691 - 16

Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

Active Standard ASTM E691 | Developed by Subcommittee: E11.20

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- Consistency Statistics from ASTM E691:
  - ✓ There are **h** and **k** values in excess of **h**crit = 2.38 and **k**crit = 1.64 for Eb and Er.
  - ✓ The cause of these values needs to be investigated, and do one of the following:
    1. Find causes and resolve them, or
    2. Exclude data (allowed to exclude up to 20% of data according to ASTM E691).

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ASTM F 2129 Inter-laboratory Study*

- **Interpretation of Breakdown Potential, Eb**
  - ✓ Significant differences existed between laboratories as to how to interpret Eb and the 2 orders of magnitude criteria in current specified in F 2129 for scan reversal.
  - ✓ The following 2 slides show where the ILS Program Chair, Dr. Cliff Warner identified clear differences in interpretation and implementation of the standard. It is important to note that all interpretations were allowable within the standard. Therefore, it may be necessary to change the language in the standard.

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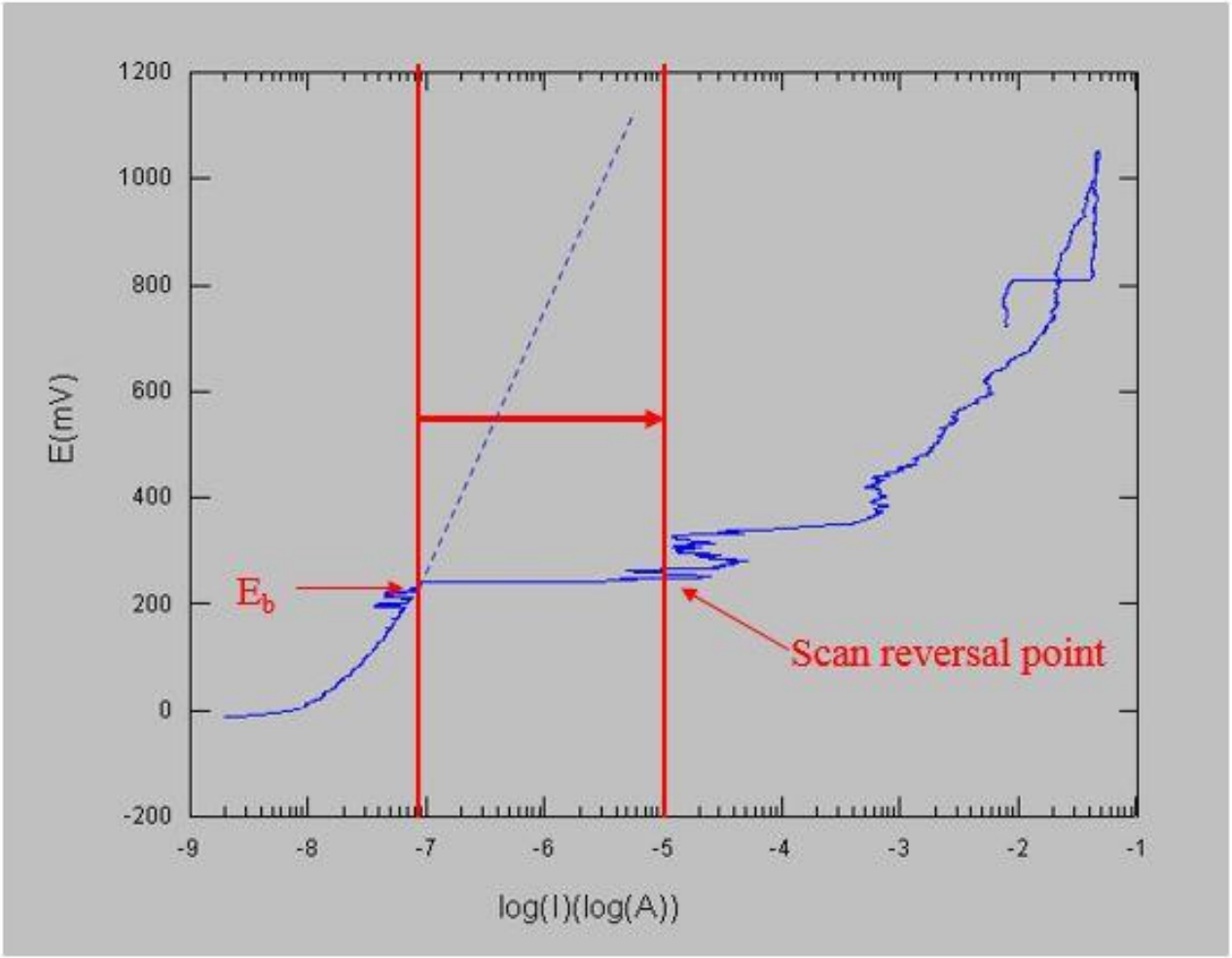
ASTM F 2129 Inter-laboratory Study*

Eb and Scan Reversal:



In this case, the laboratory interpreted the scan as having two breakdown events: one at about 220mV and a second at about 350mV.

The consensus at the meeting was that **Eb is about 220mV** because the current never dropped back to the initial current level (10 to 7 Amps) or where the current would have been in the passive region (dashed blue line).

Most laboratories would have reversed the scan at the point indicated on the plot. However, due to the wording of F2129-06, this interpretation and way of running the test is completely valid!

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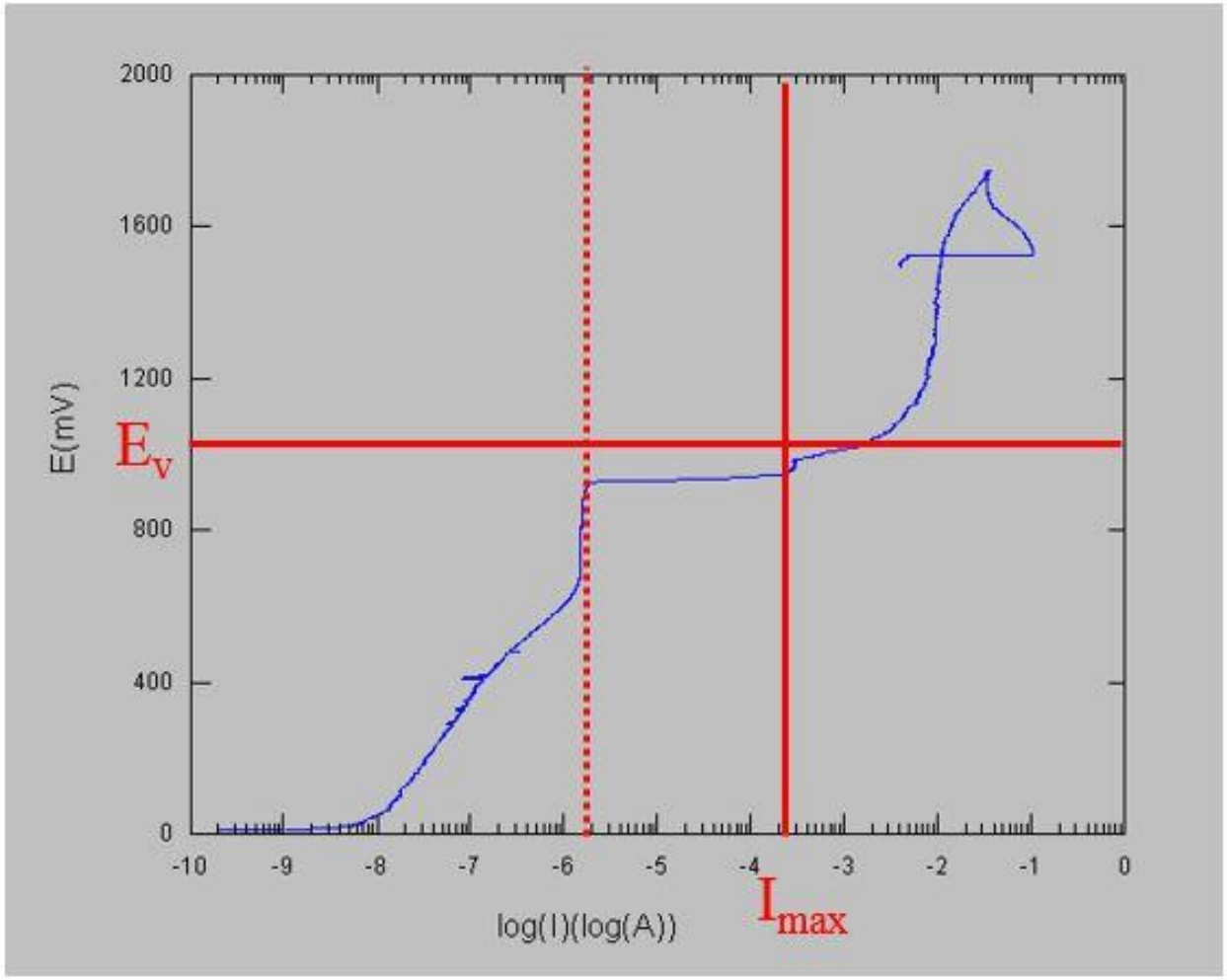
ASTM F 2129 Inter-laboratory Study*

Eb and Scan Reversal:



Note that Imax is at a higher current density than in the previous case. This shows that if Eb varies significantly within the sample set, one will need to manually reverse the scan to meet the criteria of 2 orders of magnitude increase in current density, which is specified in F2129.

Going forward, F04.15.14 may want to consider changing how the scan reversal is specified to make implementation of the scan reversal more consistent.

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Thank You!

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