The 3 Challenges in Plastics Testing

COMPLIANCE | VARIABILITY | EFFICIENCY

By Sammi Sadler

CONTENTS

- TEST RESULTS
  - Factors that Influence Results & Solutions
  - Troubleshooting
- INCREASING LABORATORY EFFICIENCY & THROUGHPUT
  - Factors that Influence Test Time
- TESTING STANDARDS

TEST RESULTS

Why are my results inconsistent?

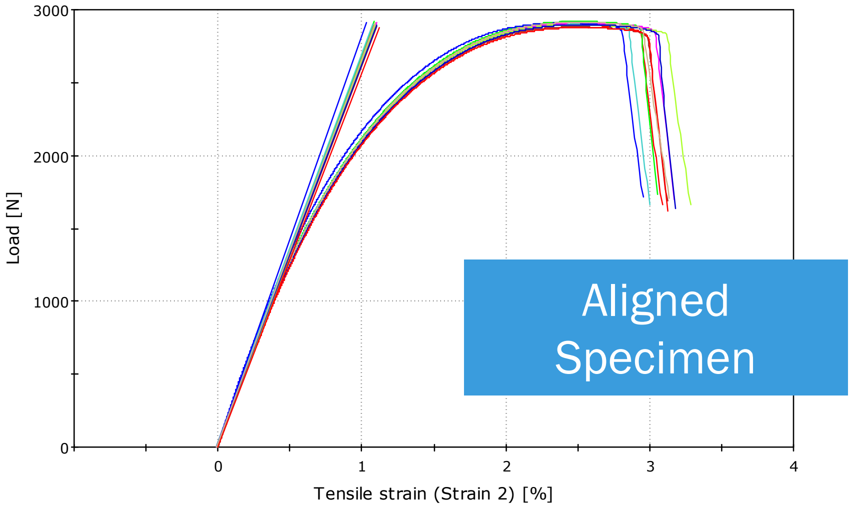
FACTORS THAT INFLUENCE RESULTS

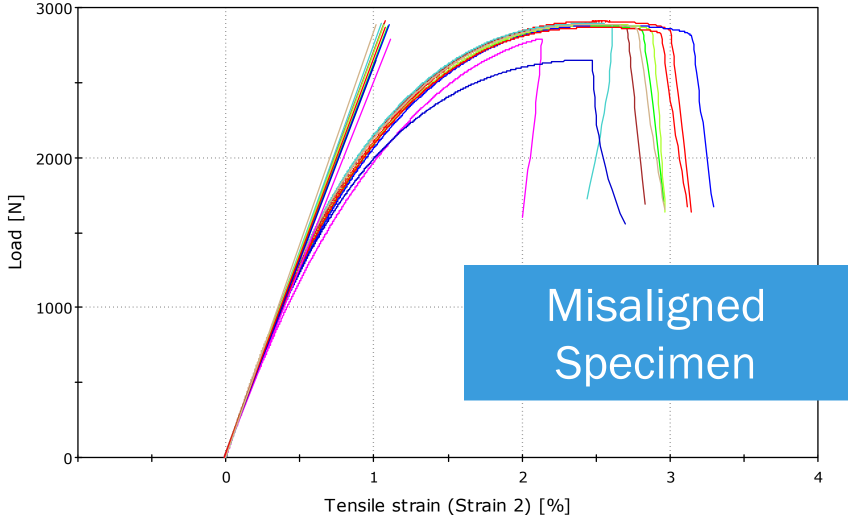
So, you're performing the tests to the standard and you're still seeing variability?
There are many factors that can contribute to variability in results…

Specimen Alignment	Thermal Effects	Grip Type	Extensometer Type
Grip Faces	Extensometer Attachment	Weight of Extensometer	Speed of Test
Method Setup	Technique	Specimen Measuring Anvils	Procedural Errors
Accuracy of Equipment	Data Rate	Algorithm	Rigid v. Flexible Load String

● = most common

SPECIMEN ALIGNMENT





Proper Alignment leads to

- Increased strain
- Better repeatability
- Higher yield stress
- Increased tensile strength

Affects proven on: PP (30% glass), ABS, various TPOs

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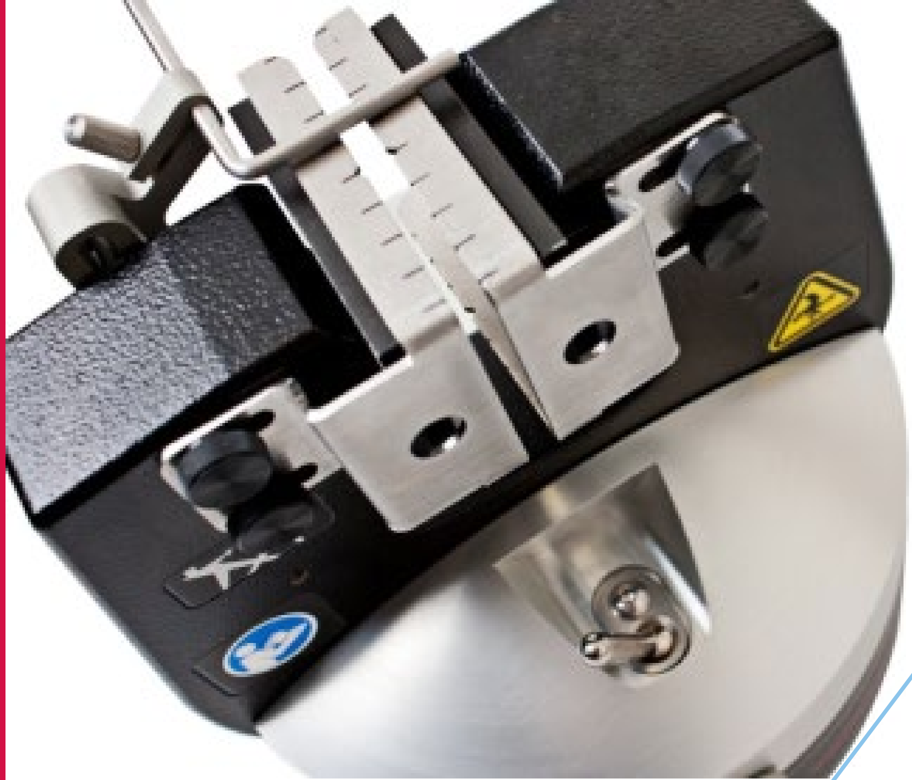
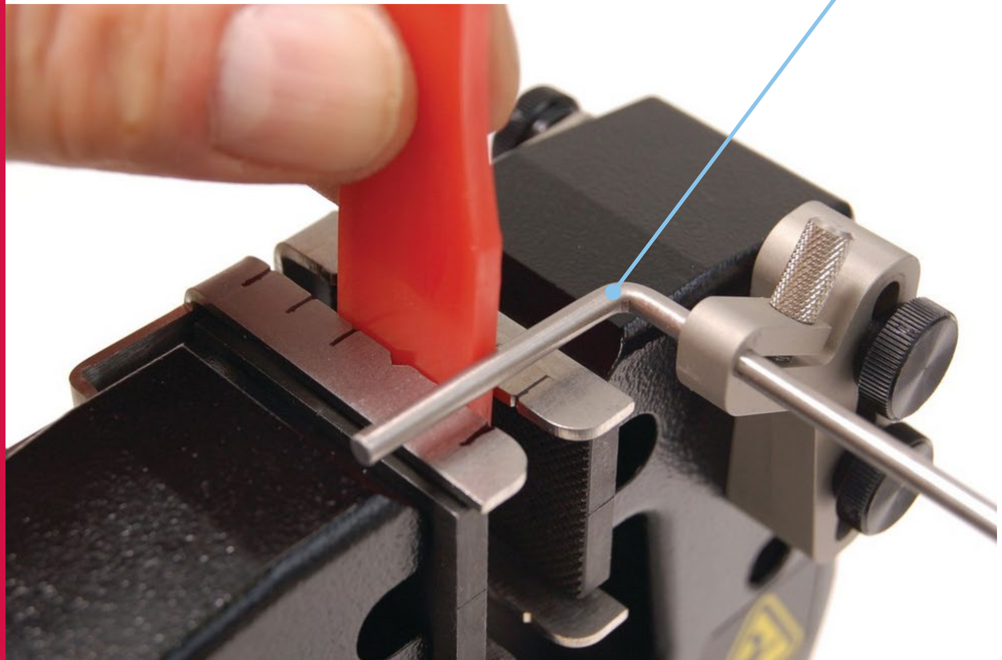
CORRECTING SPECIMEN ALIGNMENT





Specimen Centering Aids

- Ensure specimen is vertical and centered in grips

  Available for most side-acting and wedge grips

Jaw faces sized for your specimen

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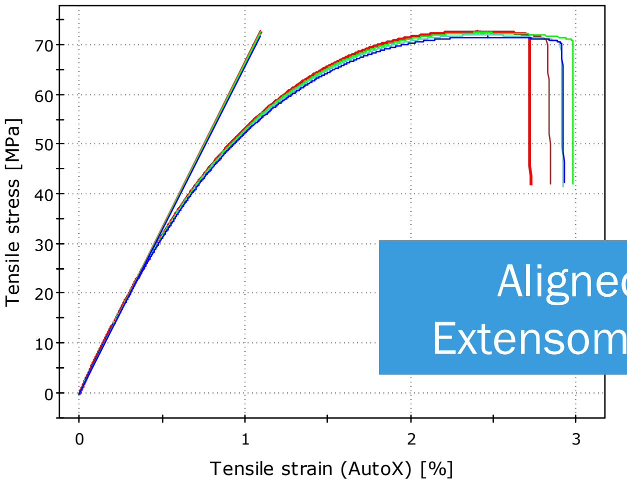
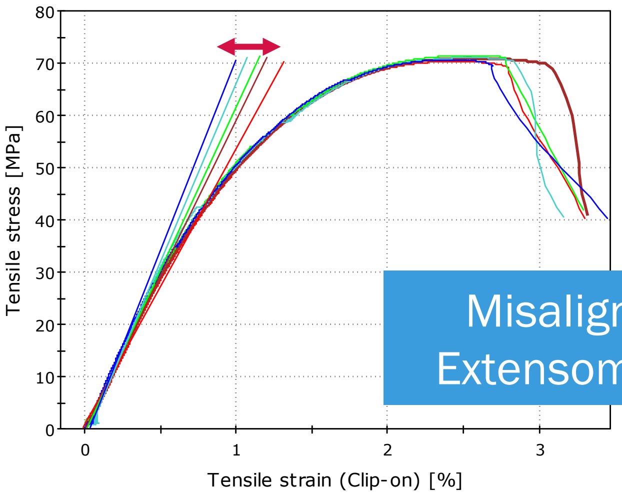
EXTENSOMETER PLACEMENT/ATTACHMENT



When extensometers are not attached properly, it can negatively affect the results.

misaligned, high, low

Manual devices are more susceptible to misuse but work well when used properly. Automatic extensometers eliminates this variable.



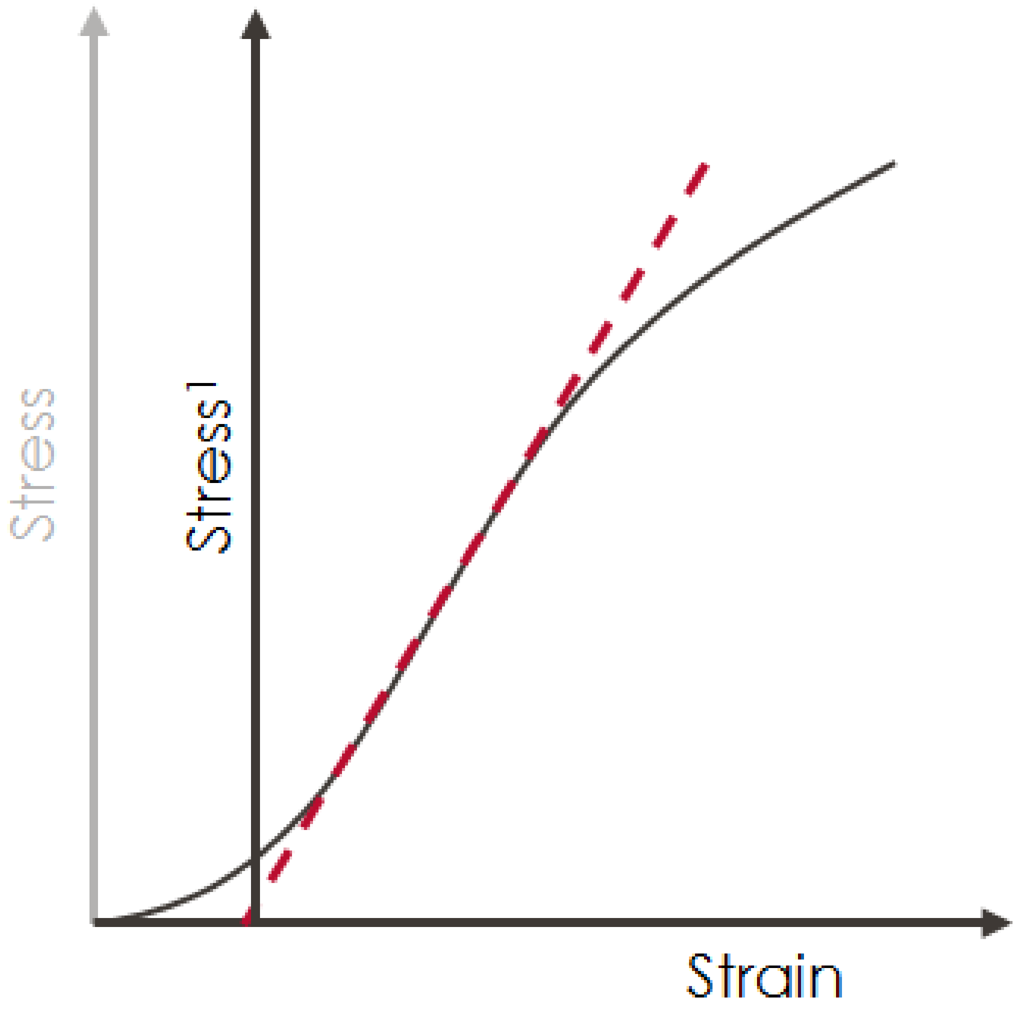
Automatic vs. Manual

- Modulus
  - Better repeatability
  - Higher mean
- Strain
  - Better repeatability

Affects proven on: PP (neat), PEEK, ABS, Data based off PP (30% glass)

Toe Compensation

- Required by both ASTM & ISO
- Recommendation: Use pre-load & auto-balance extension (NOT LOAD)



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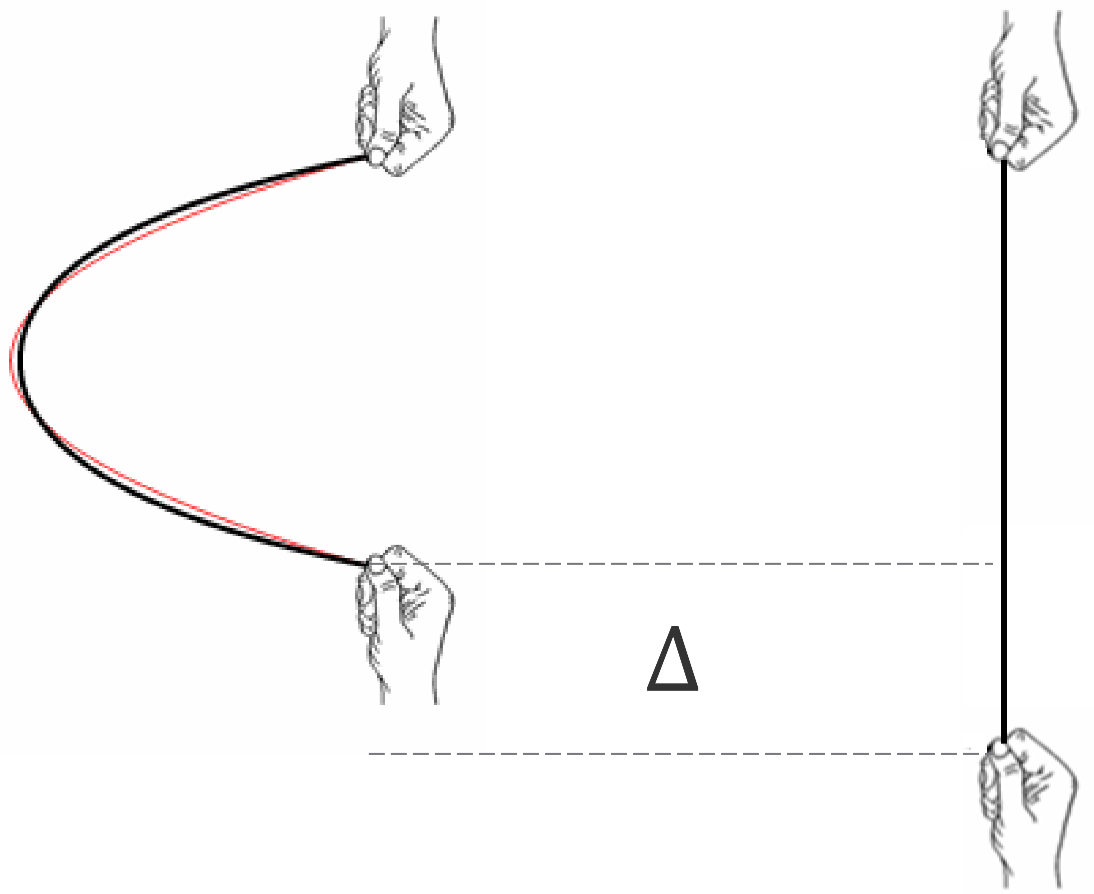
Toe Compensation – Why Balance Extension & Not Load?

- After a specimen is installed into grips there is some slack present
- Pre-load to remove the slack
- Distance traveled is not from specimen straining!
- Small load is real load!



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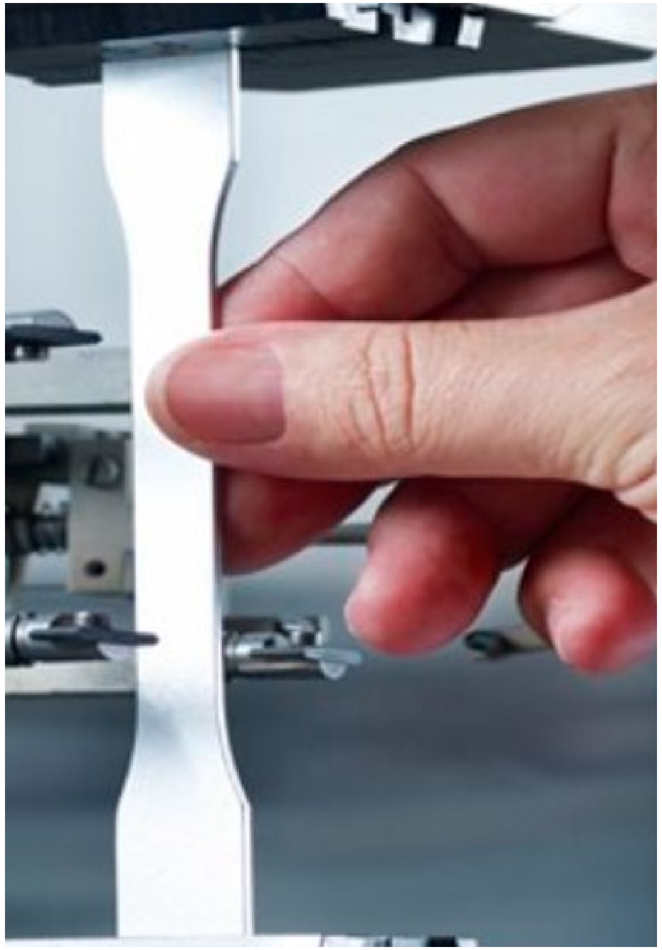
Thermal Effects

- Heat of user's hands effect material properties
  - Premature breaks
- Varies from person to person



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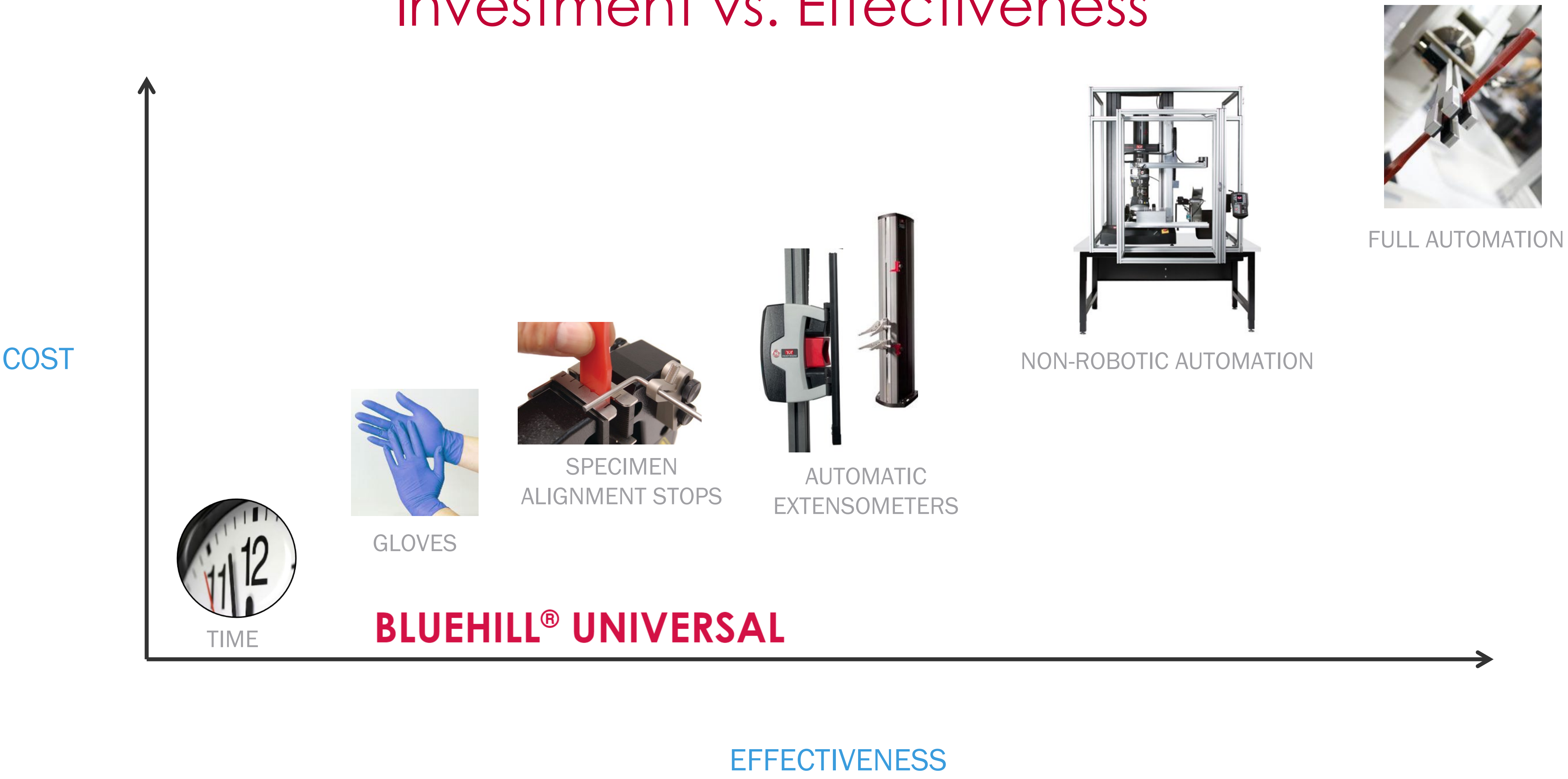
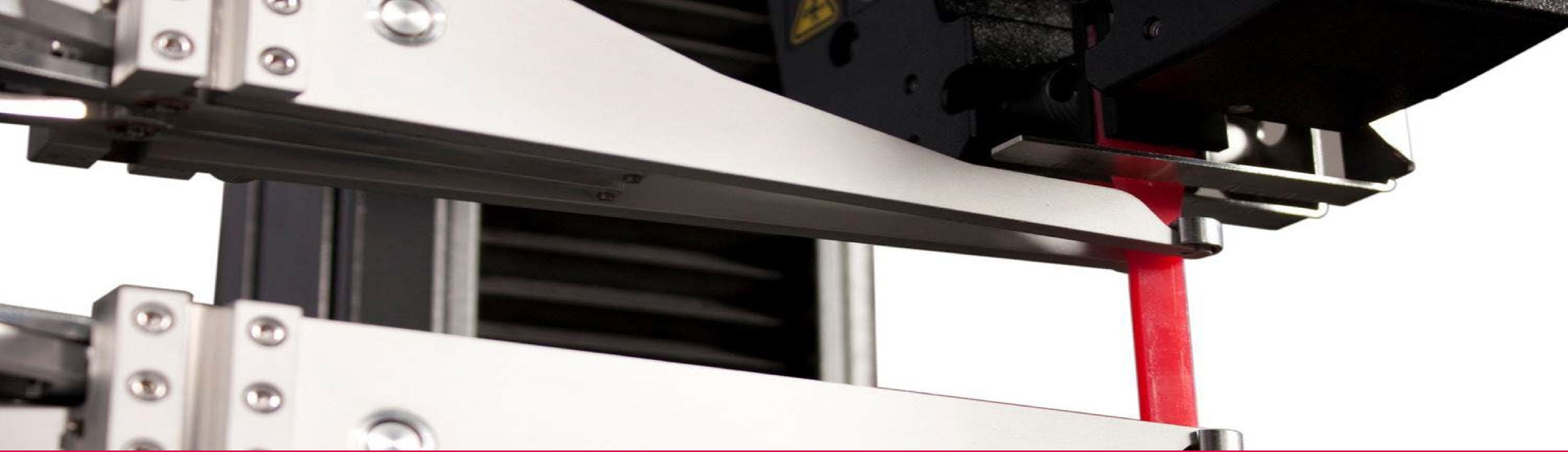
Repeatability
Investment vs. Effectiveness





INCREASING LABORATORY EFFICIENCY & THROUGHPUT

How much time can you gain?

TYPES OF SETUPS

TYPICAL

- Manual wedge grips
- Micrometer + calculator
- Clip-on extensometer

IDEAL

- Pneumatic grips with alignment aids
- Integrated micrometer
- AutoX750 extensometer

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THE CYCLE TIME FORMULA

- Fixed Times
- Variable with different setups

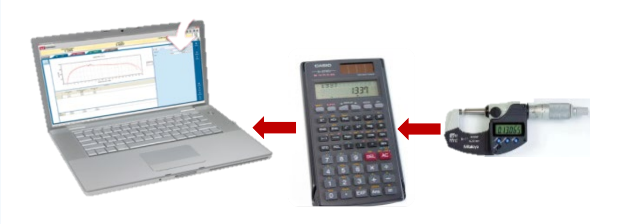
DIMENSIONAL MEASUREMENT	Integrated vs. Manual
SPECIMEN INSERTION	Time to Install Correctly & Grip Closing
EXTENSOMETER ATTACHMENT	Automatic vs. Manual
TEST TIME	
EXTENSOMETER REMOVAL	Automatic vs. Manual
+ SPECIMEN REMOVAL	Grip Opening

= CYCLE TIME

THE DIFFERENCES

DIMENSIONAL MEASUREMENT

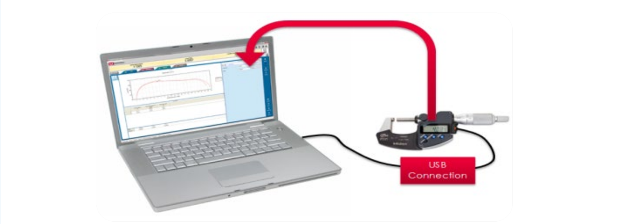
INCREASED USER INTERACTION



- 6 measurements/specimen
- ~42 keystrokes/specimen

10 TESTS = 420 KEY STROKES

MINIMIZED USER INTERACTION

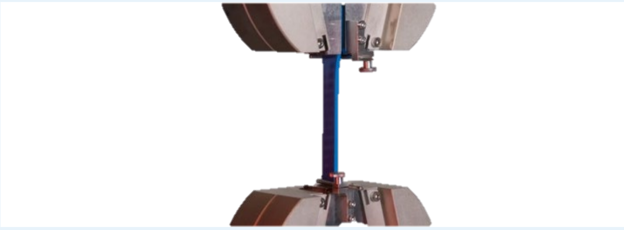


- 6 measurements/specimen
- ~7 keystrokes/specimen

10 TESTS = 70 KEY STROKES

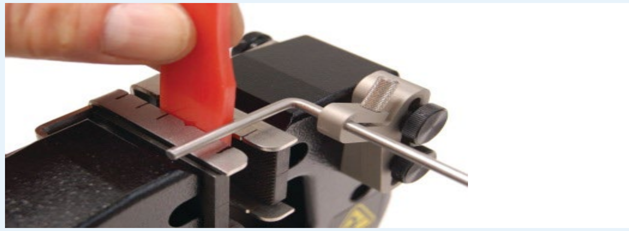
SPECIMEN INSERTION/ REMOVAL

INCREASED USER INTERACTION



- Time spent aligning by eye
- Grip closing time is longer

MINIMIZED USER INTERACTION



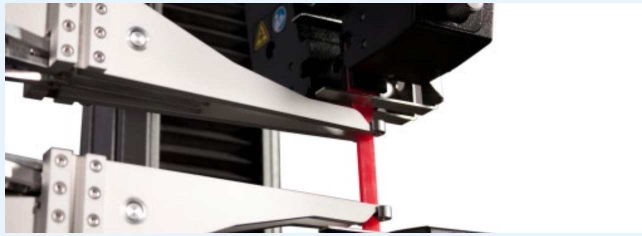
- Specimen inserted quickly and easily with alignment devices
- Grips close quickly

EXTENSOMETER ATTACHMENT/ REMOVAL

INCREASED USER INTERACTION

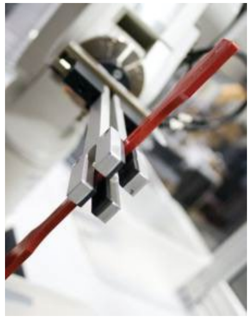
- Time spent aligning by eye
- Time to pause test to remove

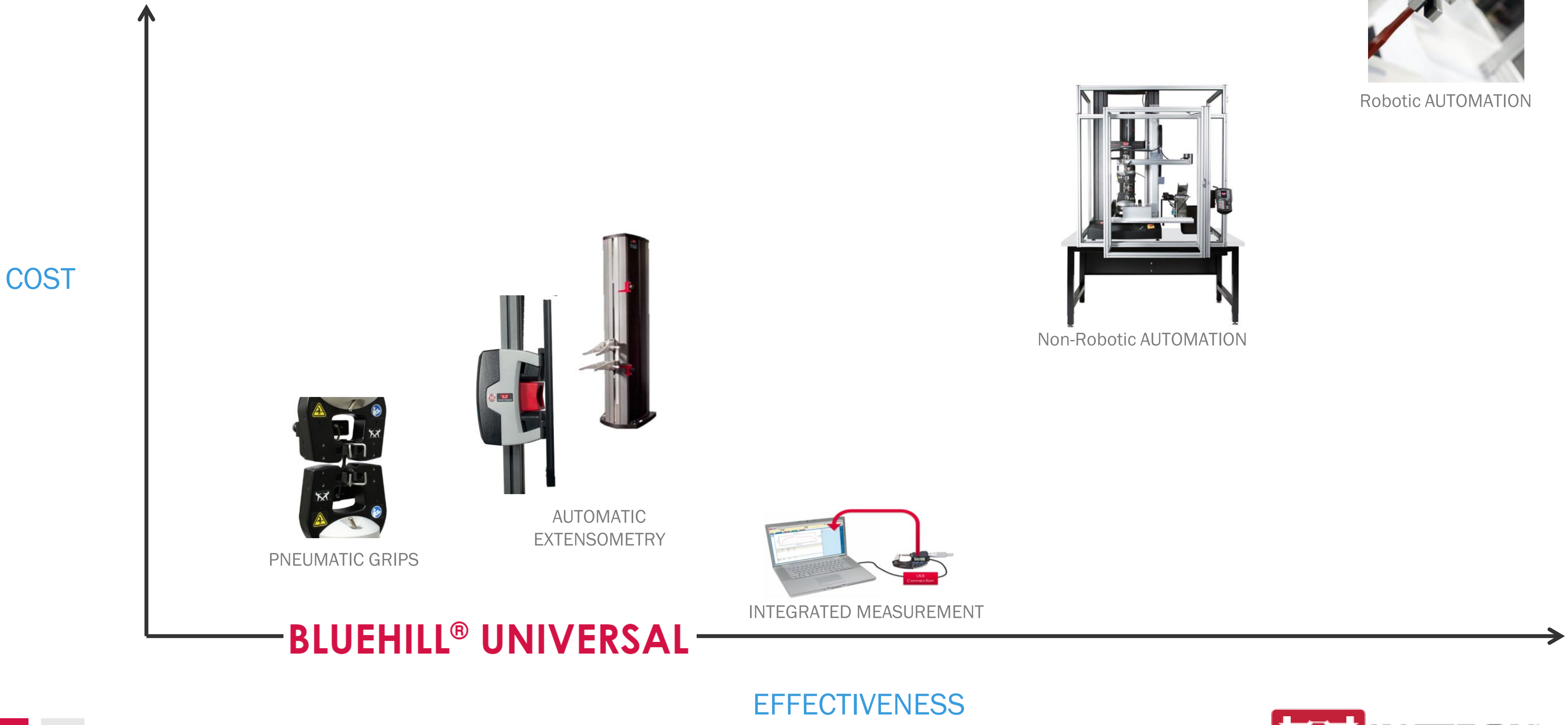
MINIMIZED USER INTERACTION



- Attaches aligned every test
- Automatically removes without pausing

Time Savings
Investment vs. Effectiveness





TESTING STANDARDS

ASTM D790

What's Changed?

- *Latest Revision in 2017*
- Most equivalent to ISO 178 but not technically equivalent

ASTM D790

	2000, 2002, 2003, 2007 2007(e1), 2010, 2015, 2015e1	2015e2	2017
TYPE I VERIFICATION		ASTM E2309 Class D	ASTM E2309 Class B
TYPE II VERIF			ss B-2
COMPLIANCE CORRECTION		Mention without detail	Added Note 10 for more detail Added Appendix X1 to outline a procedure

NOTE 10—Machine compliance correction may be applied to correct for lost motion and deflection in the load frame, drive mechanism, load sensor and other elements in order to give a more precise measurement of the deflection of the test specimen. Many manufacturer's machines and/or software packages perform this machine compliance correction. Appendix X1 also provides a means of determining the compliance correction.

THE IMPACT

Changes

	RESULTS	METHOD	EFFICIENCY	SERVICE
TYPE I VERIFICATION				●
TYPE II VERIFICATION				●
COMPLIANCE	●	●		

ASTM D882

What's Changed?

- Latest Revision in 2018
- Most equivalent to ISO 527-3 but not technically equivalent

ASTM D882

	1997 (1st Edition) / 2012	2018
SPECIMEN MEASUREMENT	"Several points along its length"	"Three points along its length" Center and within 13mm of each end of gage length
UNIFORM THICKNESS	Vague wording	Thickness is uniform to within 5 or 10% of the average thickness
BREAKING FACTOR CALCULATION	Divide max force by minimum width	Divide max force by average width
TENSILE STRENGTH CALCULATION	Divide max force by minimum area	Divide max force by average area
TENSILE YIELD STRENGTH	Divide the force at the yield point by minimum area	Divide the force at the yield point by average area

1997 (1st Edition) → 2012 → 2018

HOW WILL THESE IMPACT YOU?

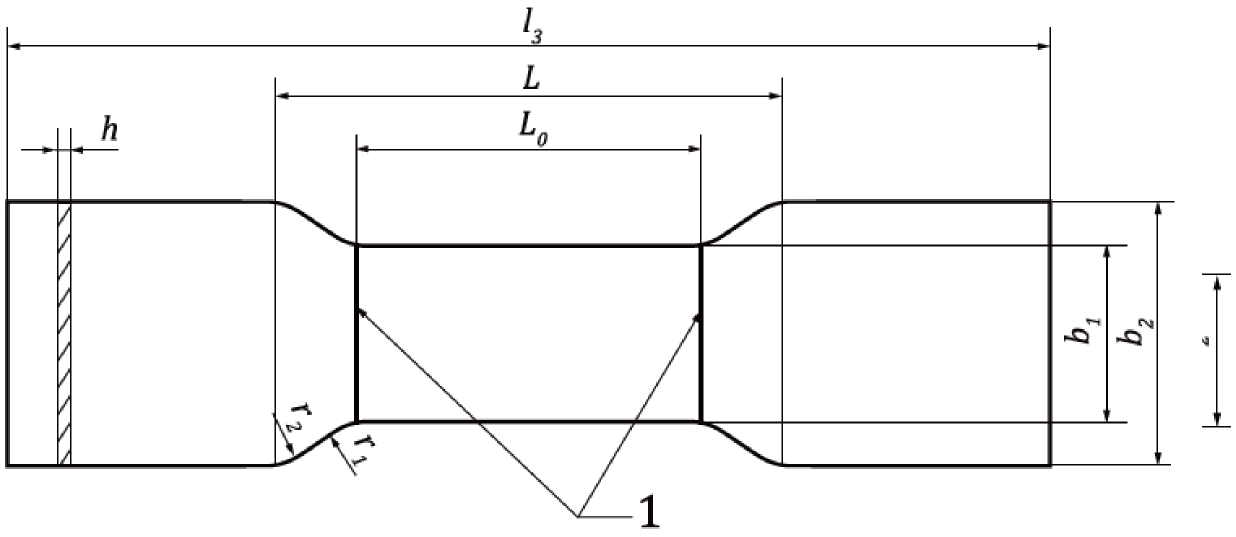
Changes	RESULTS	METHOD	EFFICIENCY	PRODUCT
SPECIMEN MEASUREMENT	●	●		
UNIFORM THICKNESS	●	●		
BREAKING FACTOR	●	●		
TENSILE STRENGTH	●	●		
TENSILE YIELD STRENGTH	●	●		

ISO 527-3

What's Changed?

- Latest Revision in 2018
- Most equivalent to ASTM D882 but not technically equivalent

ISO 527-3



Key

1	gauge marks
b_1	width of narrow papallel-sidedportion: 25,4 mm ± 0,1 mm
b_2	width at ends: 38 mm
h	thickness: ≤1 mm
L_0	gauge length: 50 mm ± 0,5 mm
L	initial distance between grips: 98 mm
l_3	overall length: ≥152 mm
r_1	small radius: 22 mm
r_2	large radius: 25,4 mm

Figure 4 — Specimen type 4

HOW WILL THESE IMPACT YOU?

Changes	RESULTS	METHOD	EFFICIENCY	PRODUCT
TYPE 2		●		●
TYPE 1B				●
TYPE 4	●	●		●

ASTM D638

Just an update!

- *Latest Revision in 2022*
- Most equivalent to ISO 527-1,2 but not technically equivalent
- Adding an annex for testing additive manufactured specimens
- This will create an amendment to the standard when it gets added

ASTM D638/ISO 527 Yield Point

ASTM

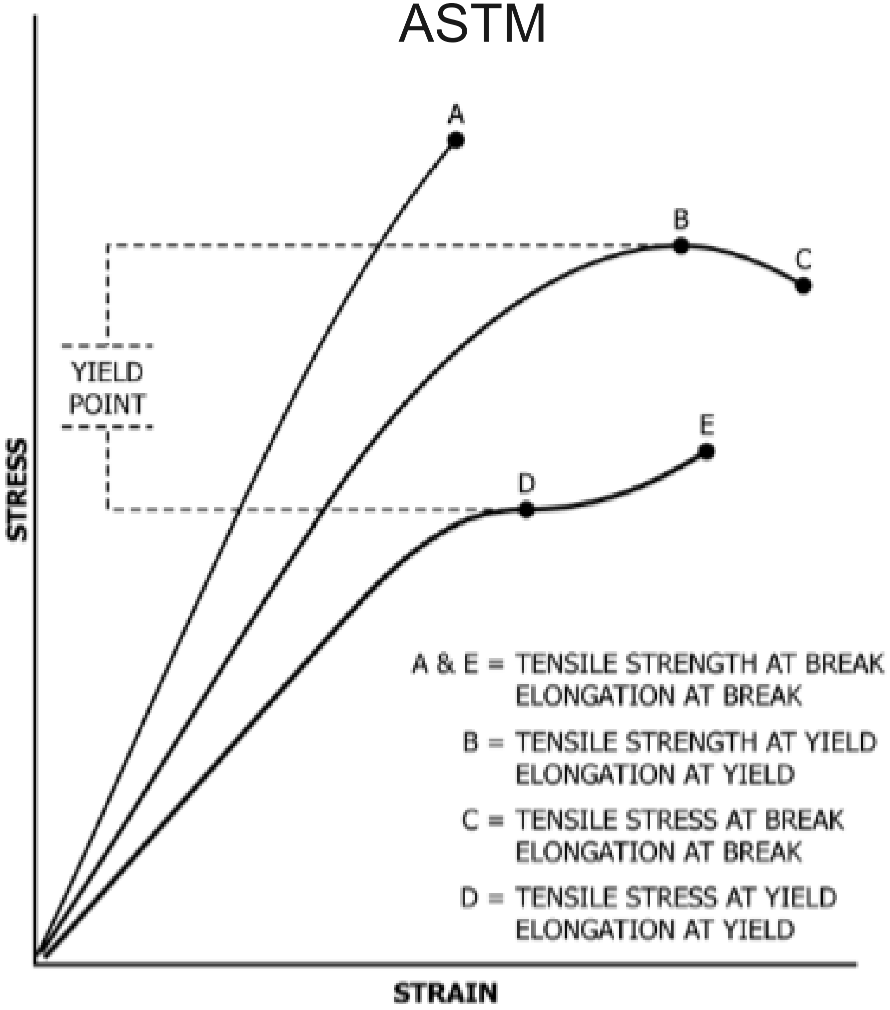
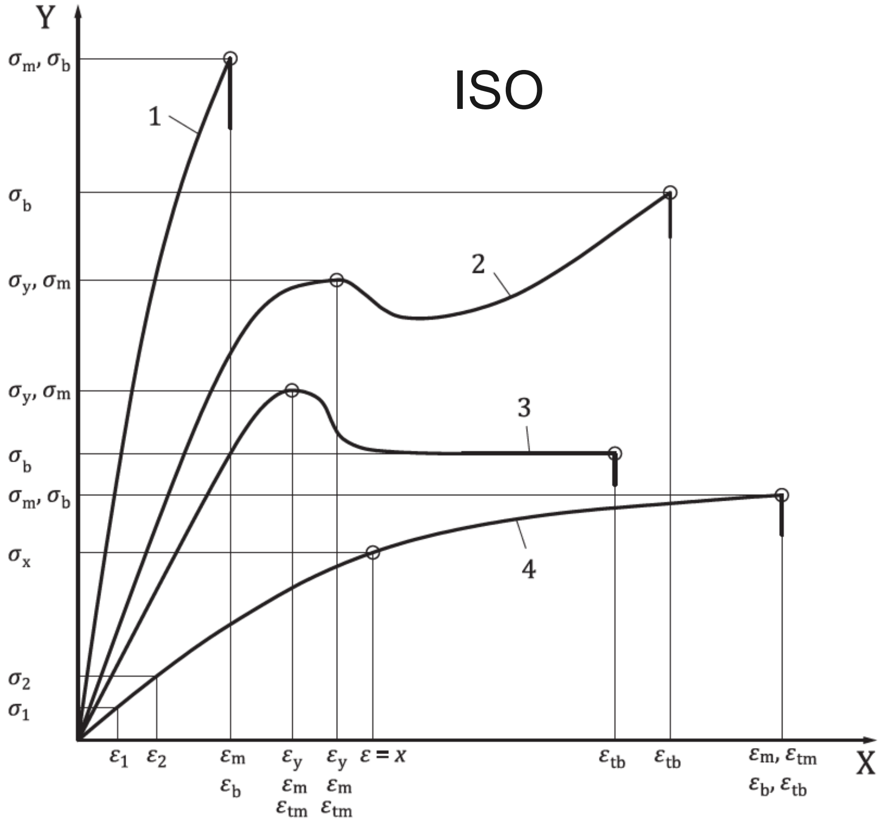


FIG. A2.3 Tensile Designations

ISO



Key

X strain and/or nominal strain

Y stress

1 Curve (1) represents a brittle material, breaking without yielding at low strains. Curve (4) represents a soft rubberlike material breaking at larger strains (>50 %).

2, 3 Curves (2) and (3) represent materials that have a yield point with (2) or without (3) stress increase after yielding. Curves (2) and (3) are curves “stress vs. strain” up to the yield point and “stress vs. nominal strain” beyond the yield point.

4 Curve (4) may be either stress vs. strain or stress vs. nominal strain depending on equipment used.

ASTM D638/ISO 527 Nominal Strain

No Yield

- Width
- Thickness
- Modulus of Elasticity
- Secant Modulus
- Tensile Strength
- **Percent Elongation**
- **Percent Elongation at Yield**
- **Percent Elongation at Break**

Yield

- Width
- Thickness
- Modulus of Elasticity
- Secant Modulus
- Tensile Strength
- **Nominal Strain**
- **Nominal Strain at Yield**
- **Nominal Strain at Break**

Most Recent Standard Revisions

ASTM Standards:

- ASTM D638 (2022)
- ASTM D695 (2015)
- ASTM D790 (2017)
- ASTM D882 (2018)
- ASTM D1708 (2018)
- ASTM D3574 (2017)
- ASTM D6272 (2017)

ISO Standards:

- ISO 178 (2019)
- ISO 527 – 2 (2012)
- ISO 527 – 3 (2018)
- ISO 604 (2002)

The 3 Challenges in Plastics Testing – Melt Flow

COMPLIANCE | VARIABILITY | EFFICIENCY

By Stephanie Williams

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ITW

CONTENTS

- Melt Flow Testing
  - What it is
  - How it is done
- TESTING STANDARDS
  - Changes in Key Standards
- TEST RESULTS
  - Factors that Influence Results & Solutions
  - Troubleshooting
- INCREASING LABORATORY EFFICIENCY & THROUGHPUT
  - Factors that Influence Test Time

Melt Flow Testing

What is Melt Flow testing?

Test that determines the flow rate of a polymer material in its molten state under specific load/temperature conditions

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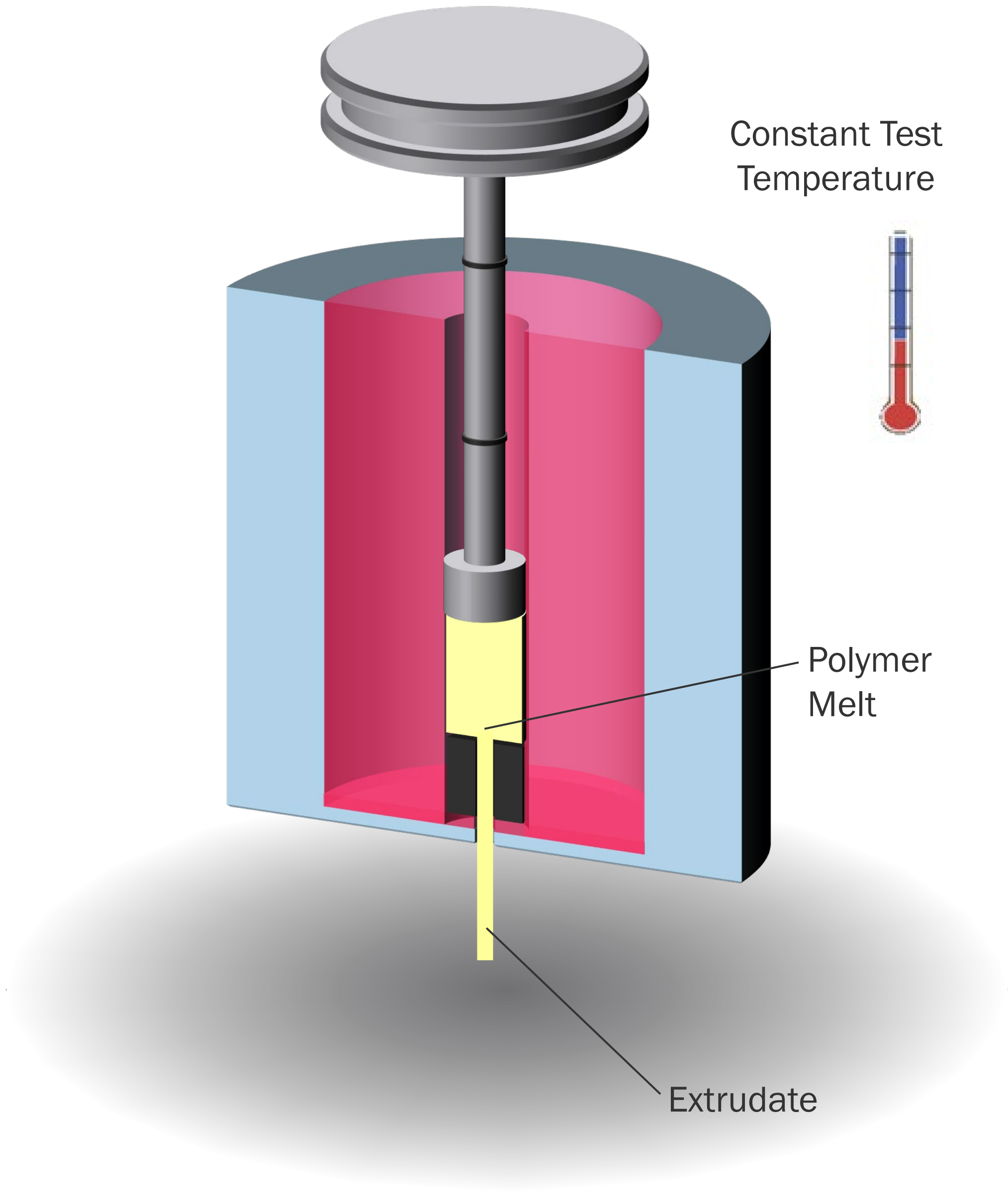
Melt Flow Testing



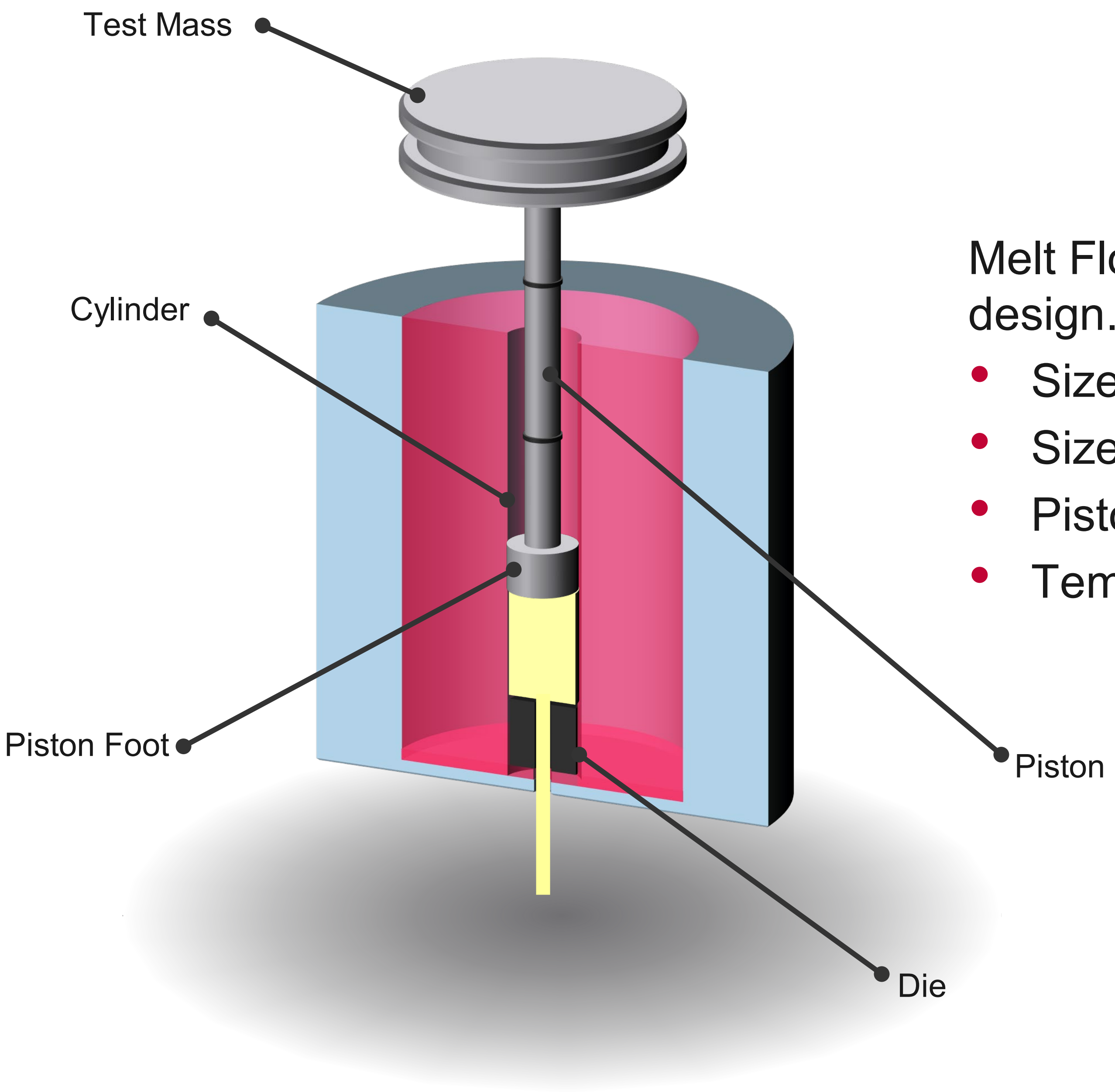
Basic test Procedure:

- ✓ Preheat Test Barrel, Piston & Die
- ✓ Insert the Polymer Sample
- ✓ Apply the Test weight
- ✓ Measure the amount of sample extruded
- ✓ Calculate the MFI Value = grams/10 min

Input Parameters:

- ✓ Geometry: Specified die/nozzle
- ✓ Material
- ✓ Temperature
- ✓ Weight: Specified mass

Melt Flow Testing



Melt Flow Testers are standards driven in their design. The standards define the:

- Size of the cylinder bore
- Size of the die and the orfice in the die
- Piston & landing foot of the piston
- Temperature Control System:

TESTING STANDARDS

Melt Flow Test Standards

General standards for all materials:

- ASTM D1238, Method A, B, C and D
- ISO 1133-1,-2, Procedure A, B

For specific materials:

- ASTM D3364 (for PVC)
- .. plus all individual material standards (e.g. ISO 1872-1 for PE, ISO 2580-1 for ABS, …) specifying key parameters but referring to the general ones for machine construction and method settings

ASTM D1238

What's Changed?

- *Latest Revision in 2023*
- Covers same subject matter as ISO 1133 but differs in technical content.
- Allows for use of load cell to apply force to specimen.

ISO 1133-1, -2

What's Changed?

- *Latest Revision in 2022*
- Reference for most local standards on MF tests worldwide
- Similar to ASTM D1238 but differs in technical content.

ASTM D1238 (2023) vs ISO 1133-1,2 (2022)

ASTM D1238 (2023)		ISO 1133-1,2 (2022)
Test Procedure allows application of test weight by dead-weight stack OR a force/load	**Test Procedure**	Test Procedure allows application of test weight by dead-weight stack OR a force/load
Defines specific Procedure D with details and prescriptions	**Procedure for multi-weight tests**	Just mentioned
Mentions die plugging and piston holder, defines specific Procedure C with half die	**Procedure for high-flow materials**	Mentions die plugging and piston holder, half die is allowed
Start 46 mm above die, measure length 1 inch or 1/4 inch depending on expected MFR value	**Typical measure start point and length** ★	Start 50 mm above die, measure length 30 mm
Maximum absolute deviation, defined for all materials and as a function of different temperature ranges	**Temp. accuracy & consequently verification, calibration** ★	Max abs. dev. plus (strict) maximum difference from min to max actual temperature along the barrel, required only for sensitive materials but applied flat to whole working range

★ = Significant impact

TEST RESULTS

Why are my results inconsistent?

Key Factors that influence test results

Temperature calibration ★	Preparation of sample (moisture) ★	Sample compacting ★	Method settings
Temperature stability	Choice of procedure ★	Encoder calibration	Extrudate cutting precision
Melt density value ★	Manual operations within test ★	Maintenance of die & piston ★	Cleaning procedures ★

★ = most common sources of issues

Checklist: Reducing Sources of Error

- ✓ Are you using the correct test procedure recommended for your sample?
- ✓ Does the equipment meet the standard requirements? Is it calibrated?
- ✓ Is the preheat time 7± 0.5 min?
- ✓ Is the piston cold? Is there > 5 min interval between 2 test runs?

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Checklist: Reducing Sources of Error

✓ Are you using the right amount of sample based on your expected MFI value per standard?

✓ Do your test results vary with operator?
- Non uniform compaction
- Imprecise extrudate cuts (Method A)

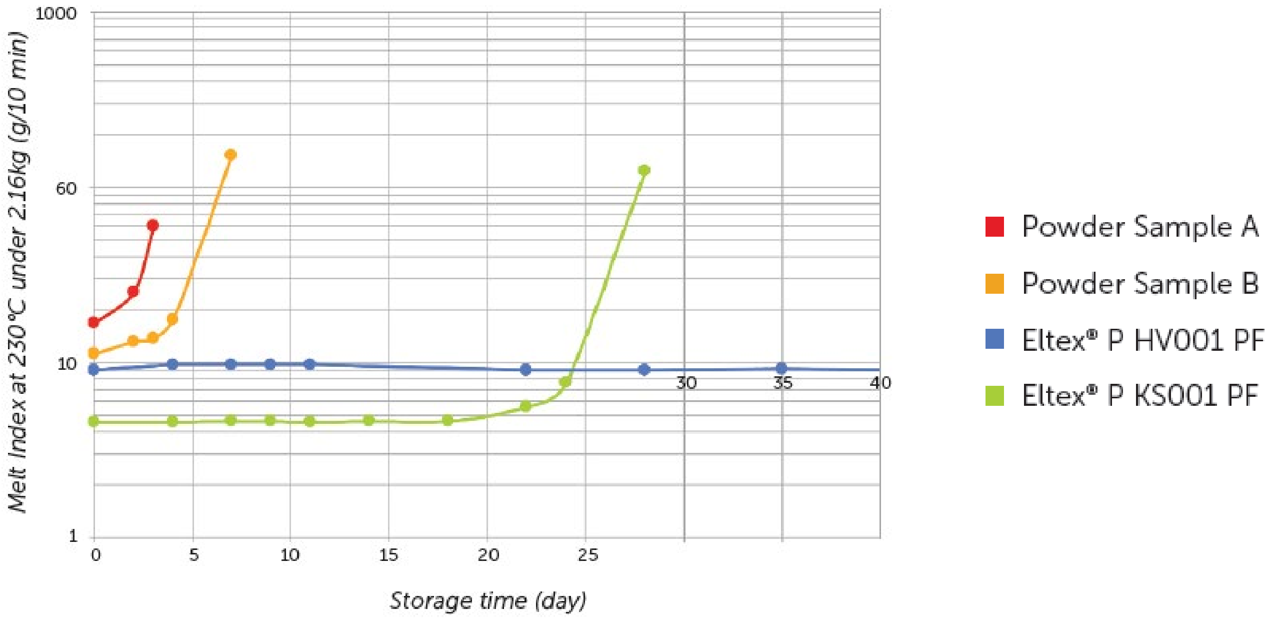
✓ Are you using Density or Melt Density values for your calculating MFR?

✓ Is your equipment (barrel, piston & die) cleaned thoroughly after every test run?

Additional Factors affecting Melt Index Values

- ✓ Polymer material Degradation due to UV/storage times

- ✓ Test sample
- ✓ Contaminati



Reference: INEOS PolyOlefins - Literature

INCREASING LABORATORY EFFICIENCY & THROUGHPUT

How much time can you gain?

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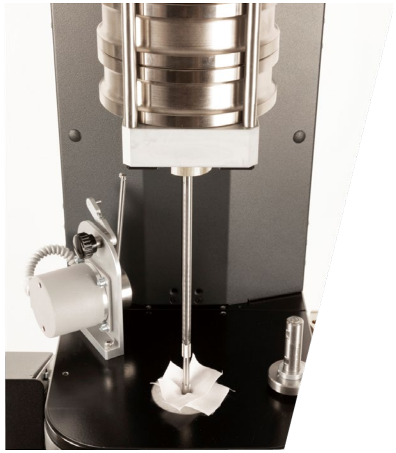
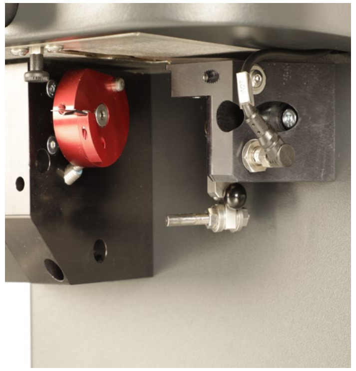
The difference is measurable®

TYPES OF SET UP

Manual

Automatic

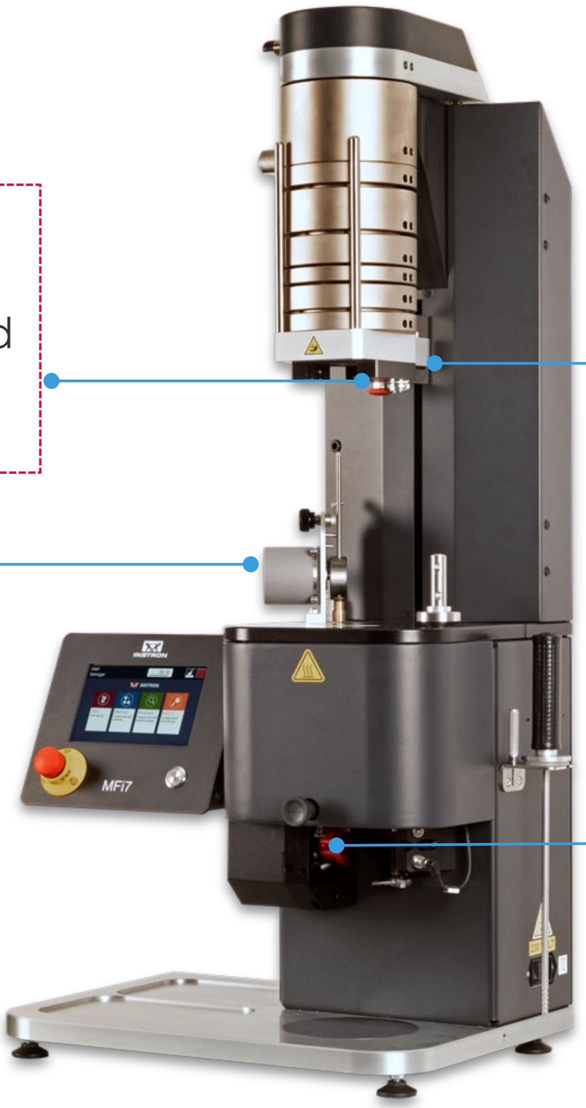




Improved User Experience & Test Efficiency

Load Cell
for automatic compaction and purging of material.

Optical Digital Encoder
for accurate reproducible results



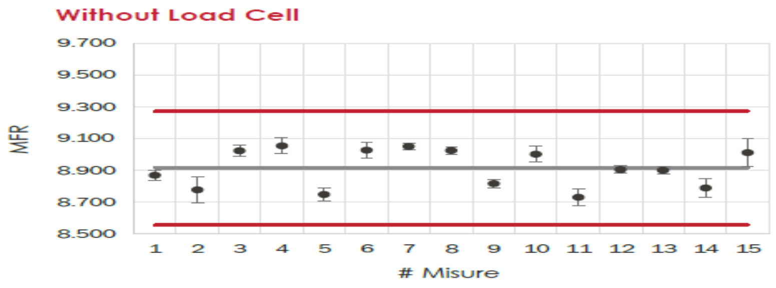
Weight Lifter
does the lifting job, no additional power or compressed air is required for Mass Selector

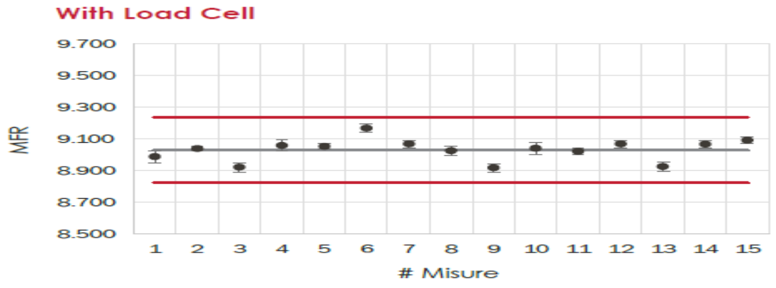
Motorized extrudate cutter,
T / P - based

Improved User Experience & Test Efficiency

Compacting

- Controlled Compacting
  - Better reproducibility and less scattering of results
  - No physical effort required by operator (reduces risk of injury)





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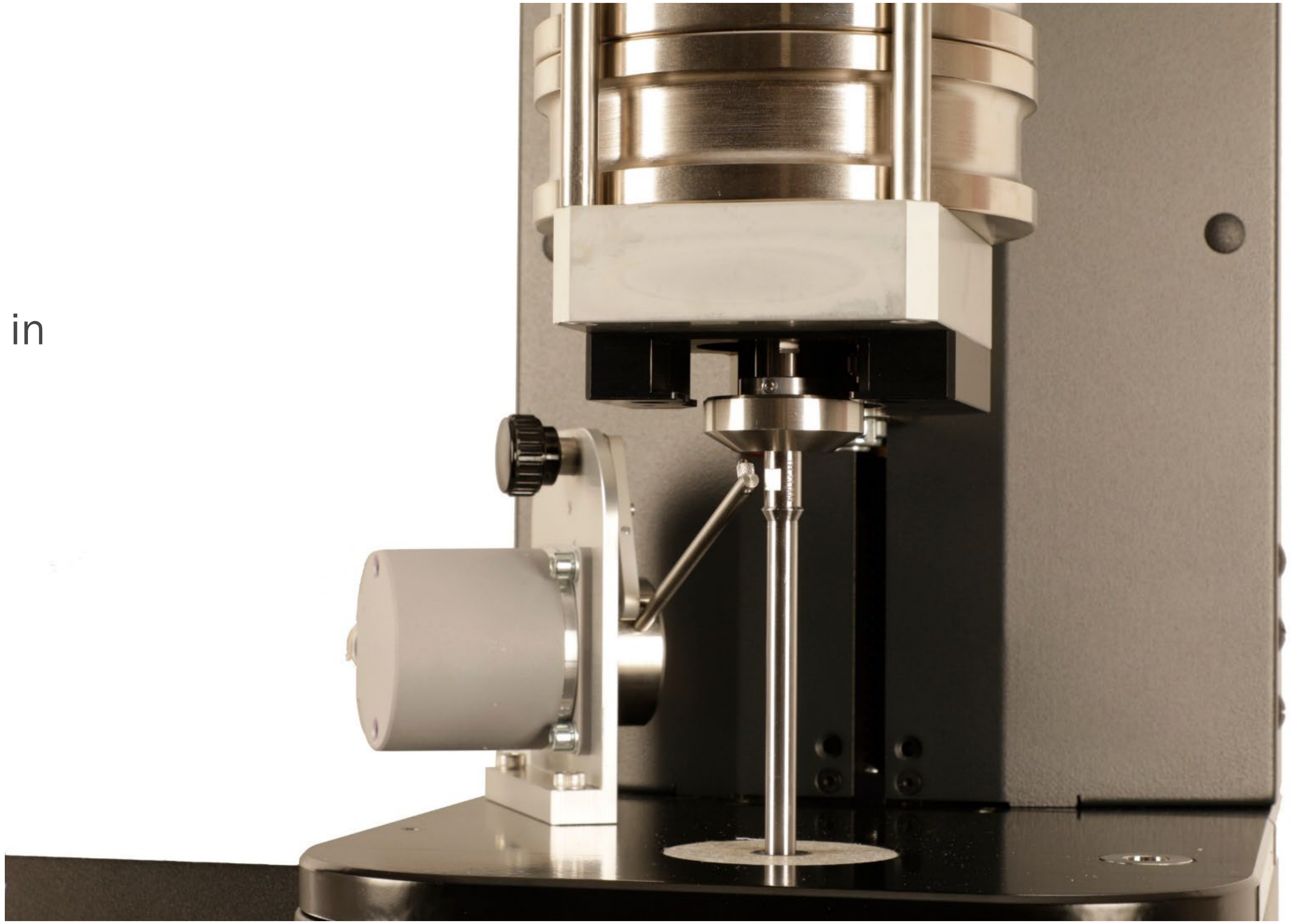
Improved User Experience & Test Efficiency

Cutting

Improved User Experience & Test Efficiency

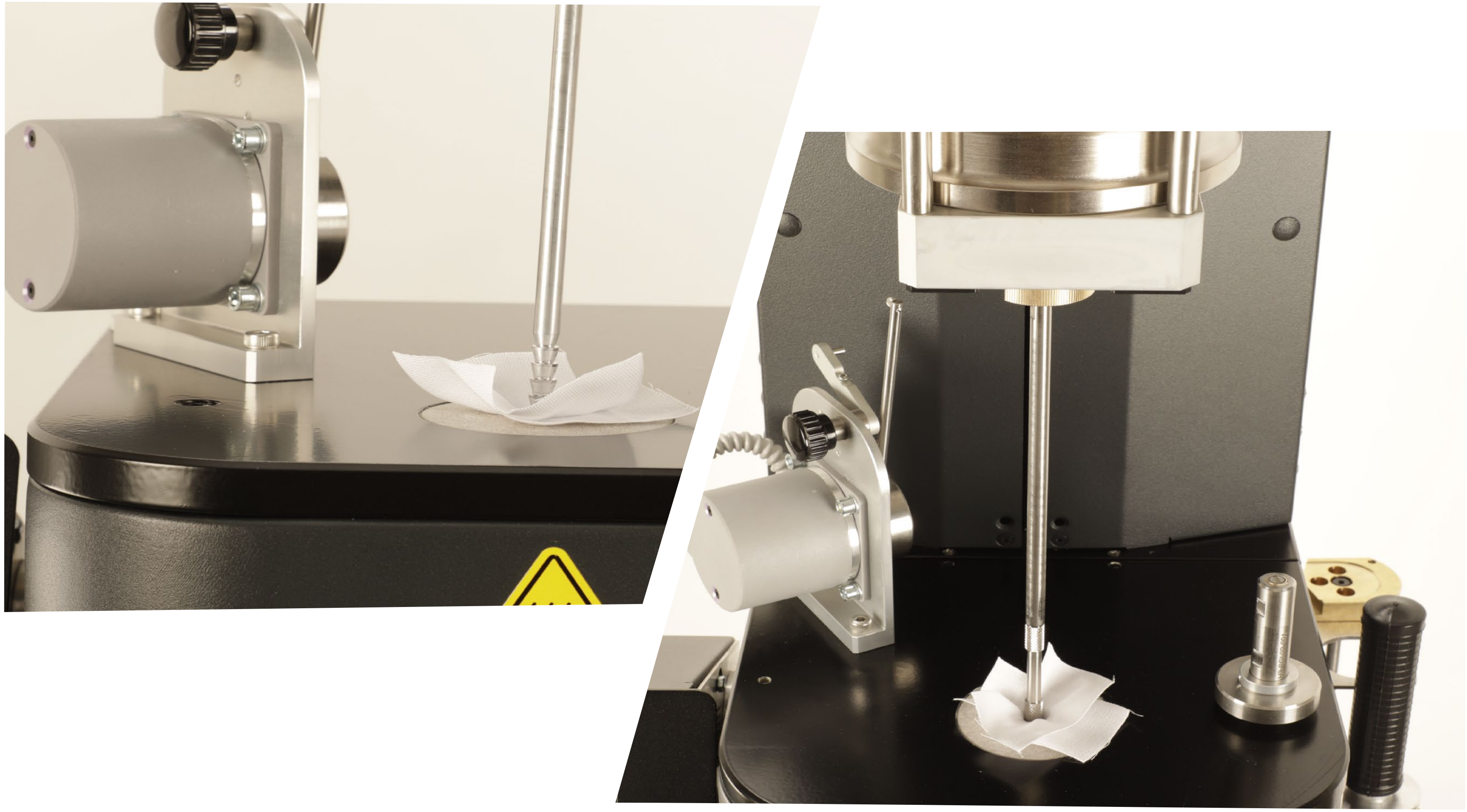
PURGING

Automated removal of excess material in the barrel immediately after the test.



Improved User Experience & Test Efficiency

CLEANING

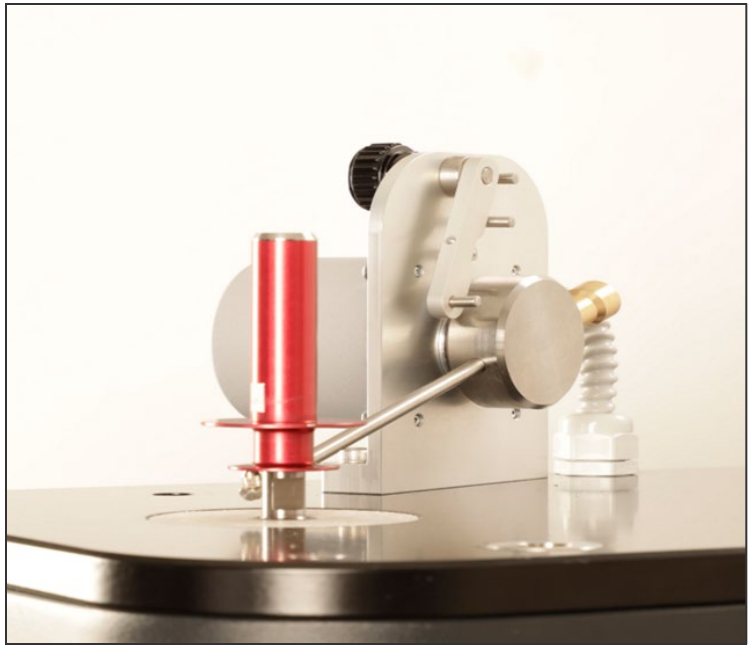


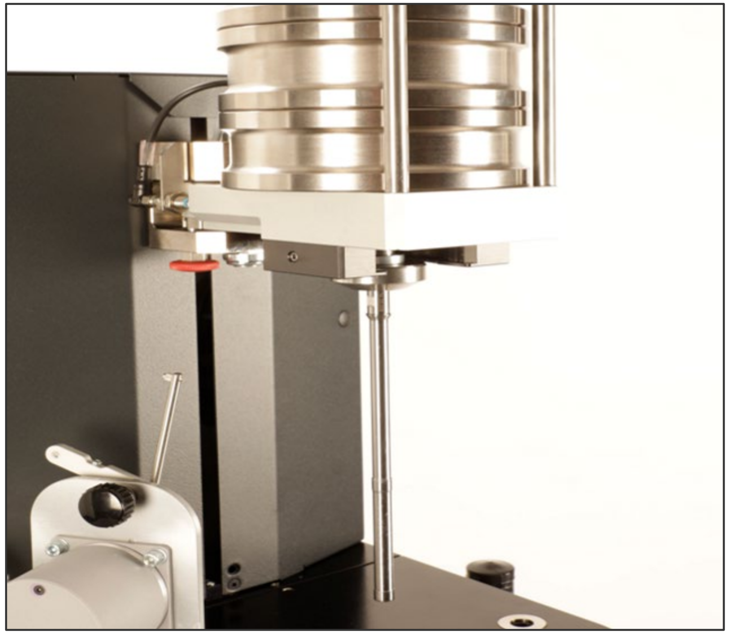
Improved User Experience & Test Efficiency

Motorized Mass Lifter

Manual Mass Selector

Improved User Experience & Test Efficiency





High Flow Rate Materials

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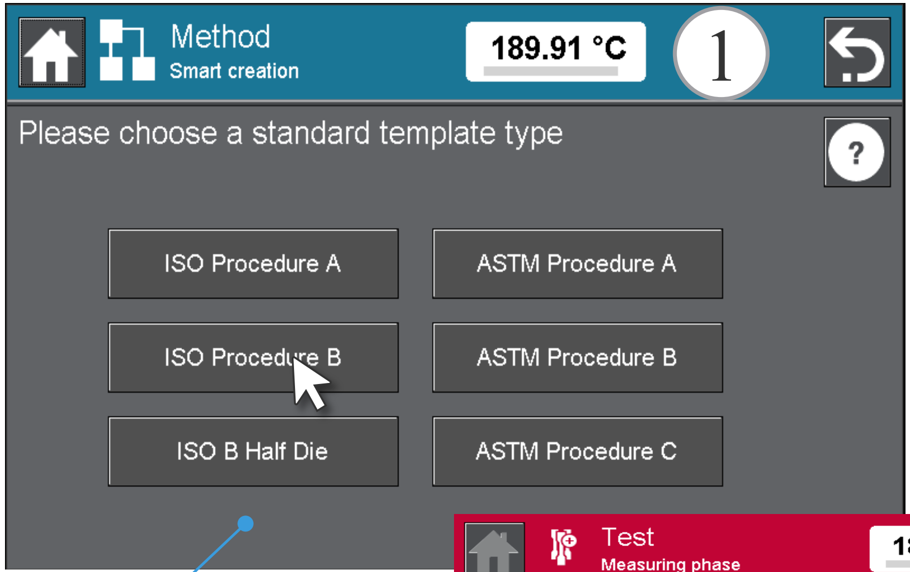
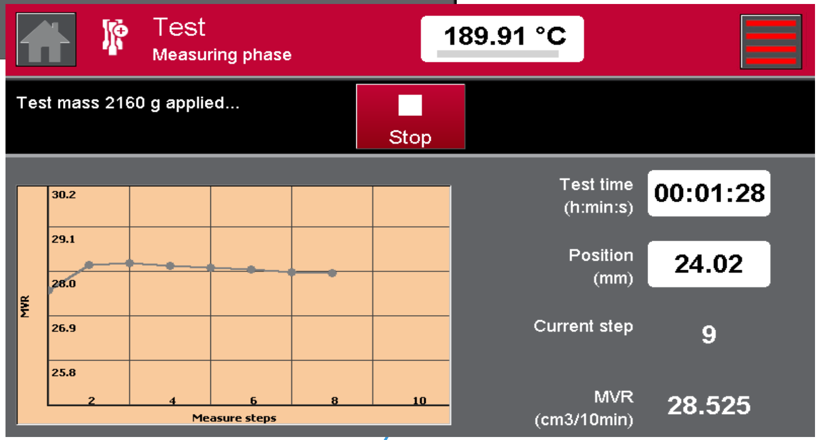
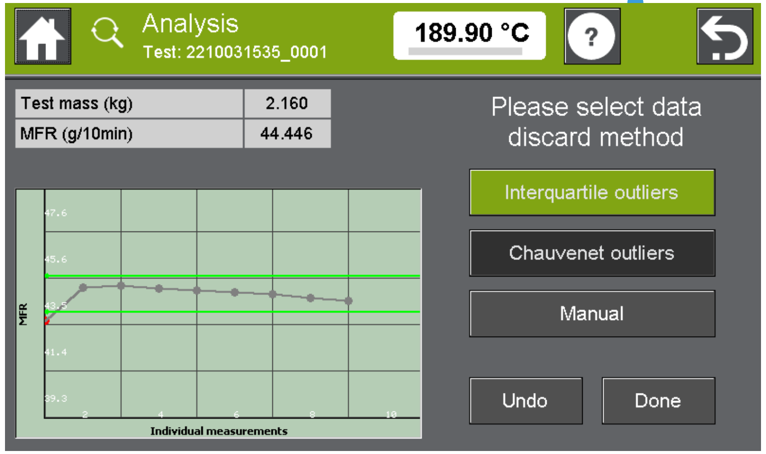
Improved User Experience and Test Efficieny

Software







Method Set-up
pull down pre-set standard formats (customizable)

Database Storage / Visual graph

Discard steps with air-voids/bubbles

MULTIPLE LICENSE SOLUTION

If you need to manage multiple MFi instruments in a laboratory from a central node

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Most Recent Standard Revisions

ASTM Standards:

ASTM D1238 (2023)

ASTM D3364 (2019)

ASTM D4000 (2023)

ASTM D5947 (2018)

ISO Standards:

ISO 19062 (2019)

ISO 19065 (2019)\

ISO 19066 (2020)

ISO 21301 (2019)

ISO 21302 (2019)

ISO 21305 (2019)

ISO 24026 (2020)

ISO 29988 (2018)

ISO 24022 (2020)

Thanks for Joining Us

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Applications Engineer @ Instron

Stephanie Williams

Senior Product Specialist @ Instron

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Thank You for Listening