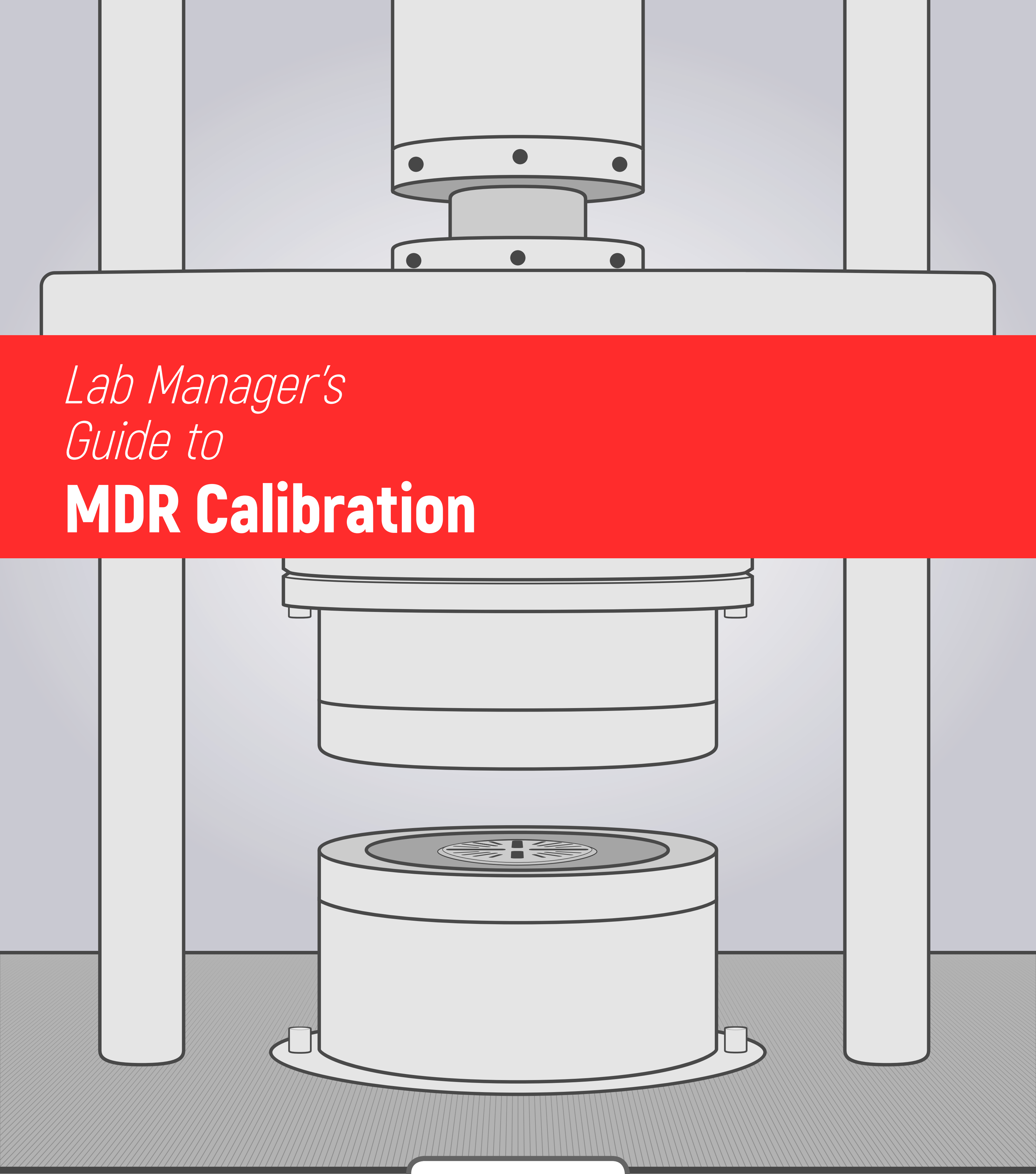
Lab Manager's
Guide to

MDR Calibration



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What does ISO/IEC 17025 accreditation mean?

Why calibrate?

All process and measuring equipment experience degradation over time. This can lead to a drift away from product quality specifications. Calibration is a process of comparing testing instruments (*MDRs*) to traceable standards. This improves product quality by keeping products in tolerance.

What is ISO/IEC 17025 Accreditation?

ISO 17025 provides the general requirements for the competence of testing and calibration laboratories. This standard is used by calibration laboratories in developing their management system for quality, administrative, and technical operations.

Typically, calibration standards are 3 to 10 times the accuracy of the instrument under test, while providing traceability of measurements to International System of Units (**SI**). To ensure consistency and reliability for ISO 17025 laboratories, part of the requirements of accreditation is verifying that the technicians are well-trained, knowledgeable, and properly follow standard procedures. Accredited technicians' training includes instrument principles of operation, observations by a senior metrology technician in performing calibrations, and proficiency testing to complete training in the following disciplines and ASTMs:

- *Force*
- *Frequency*
- *Time*
- *Temperature*
- *Torque*
- *Angle*
- *Physical/Dimensional*
- *ASTM D5289-17 (Vulcanization Using Rotorless Cure Meters)*

What can you expect in the on-site calibration process?

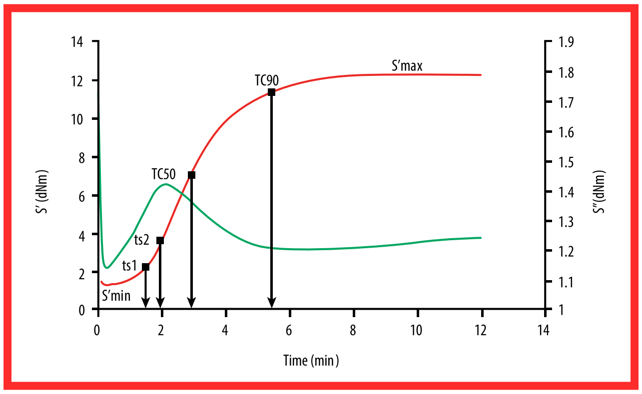
When receiving on-site ISO/IEC 17025 calibration services, it is important to know what to expect. Here is a breakdown of the sequence of events in a typical calibration process performed by an accredited service provider:

Opening Meeting	This is a meeting between the lead Calibration Field Engineer **(CFE)** and company Point of Contact **(POC)** to discuss the statement of work **(SOW)** as well as any business rules to be followed in the service visit.
Take As-Found Data	The **CFE** takes and records **As-Found** calibration data on the unit under test **(UUT)** to start the calibration process. This is a baseline with respect to the performance of the instrument being calibrated.
Calibrate / Adjust	The **CFE** follows 17025 compliant procedures to compare **UUT** measurements to certified calibration standards. Adjustments are made to the bring **UUT** into the tolerances if contracted for in the **SOW**.
Take As-Left Data	Take and record the "**As-Left Data**" and compare to the "**As-Found Data**" to determine if the **UUT** is in tolerance.
Closing Meeting	This is a meeting between the **CFE** and company **POC** to review and discuss the service report; a summary of the work performed, out-of-tolerance units **(OOTs)**, observations made, parts used, etc., during the service visit.
Enter Data	Raw data in the field worksheets is entered and preserved into the Calibration System by the **CFE**. This includes the **"As-Found Data"** and the **"As-Left Data"**, **OOTs**, changes in status, etc.
Check Uncertainties	Calculate the Uncertainties of Measurement, then check, record, and enter them into the calibration system.
Quality Review	Regarding the calibration performed, this is a review of the information and data collected that will used to complete the accredited "Calibration Certificate" prior to posting to catch any errors in the certificate requirements, notes, comments, procedures used, standards used, etc.
Post Certificates	This is the final step in the process that makes the certificates available to the customer.
End Process	Process complete.

Moving Die Rheometer *(sealed die)*
Theory of Operation

The Moving Die Rheometer (*MDR*) is a bench top press with a pneumatic cylinder, upper and lower heated platens, and upper and lower dies (*mould*) that create the die cavity. PT100 sensors in the dies independently measure the upper and lower die temperatures.

To create the angular oscillation of the lower die, MDRs use a motor and a gearbox with an eccentric that turns at 100 RPM. A link arm connects the eccentric to a torque arm, defining the chord of the arc driving the oscillatory motion of the central shaft ±0.5 degrees. Torque measurement is accomplished by a torsion sensor, with strain gages in a full bridge configuration fixed to the upper die. With excitation applied to the bridge, it outputs a low-level signal - which is amplified in a signal conditioner. When plotted against time, the signal is a sine wave with a frequency of 1.667 Hz. A simple timing circuit then allows the capture of the peak voltage at 90 degrees in the positive half of the cycle. A plot of this value against time is the S' cure curve.



Cure test performed on MDR

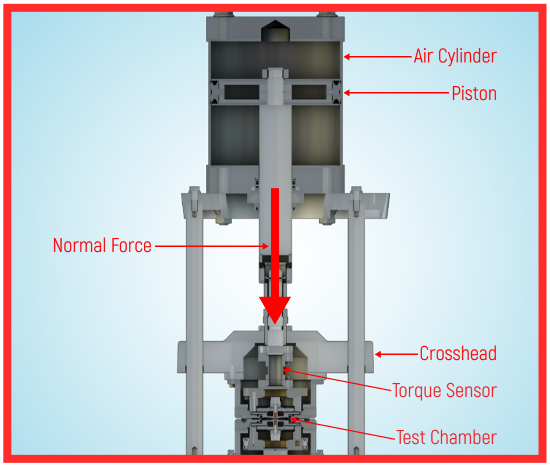
Force & Frequency / Time

Force - Closing Force

Closing force in an MDR comes from air pressure in the pneumatic cylinder, which applies a normal force through the piston rod to the cross head. The cross head applies force to the upper platen, closing to the lower platen similar to a molding press. Most of the force is applied to the sealing ring on the seal plates defining the perimeter of the die cavity. The upper and lower conical dies, seals, and sealing ring on the seal plate form the cavity.

The upper die-heater assembly is in-line with the torque transducer and attached to the bottom of the cross head, making it independent from the seal plate. With the sample placed in the die cavity, and with platens closed, torque is transmitted by the oscillatory motion of the lower die, through the sample, to the upper die and is measured by the torque sensor. Part of the normal force is now applied to the surface area of the conical die face that can be measured as die cavity pressure.

Closing force calibration is performed using a certified force gage and indicator to verify that the instrument's closing force is within tolerance.

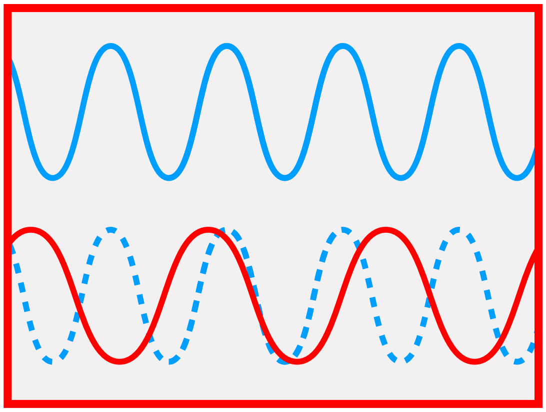


Cross-section of MDR 2000 air cylinder and platens. Normal force is applied to crosshead to close platens and seal test chamber.

For further reference see: ASTM D5289-17 section 6.2.3

Frequency/Time - Oscillation Frequency - Plotting Against Time

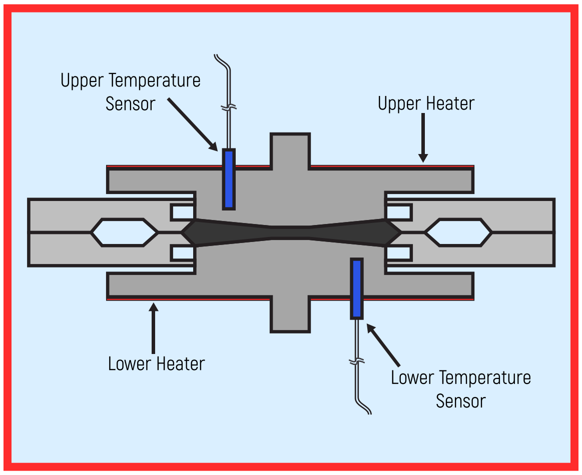
Oscillation frequency and time calibrations are performed using time standards. The oscillation frequency of an MDR is 1.667 Hz and is dependent on motors with gearboxes spinning an eccentric at 100 RPM. Speed affects the frequency and requires calibration. Calibration methods are dependent on eccentric or dynamic motor configurations.



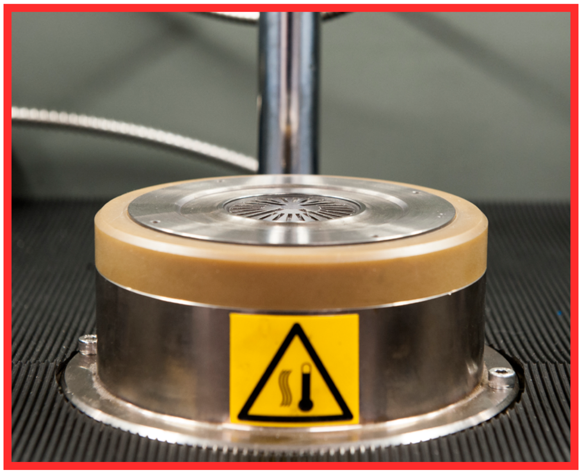
Visualization showing correct oscillation frequency (blue), overlaid on slower, incorrect frequency (red)

For further reference see: ASTM D5289-17 section 6.3.2

Temperature



Cross section of MDR's test chamber showing seal plates, dies, heaters, and temperature sensors.



Lower Die of MDR 2000

Temperature - Die Cavity Temperature

Temperature also plays a key role in the rates of vulcanization reactions, namely scorch and cure times. Therefore, the controlling system must be capable of maintaining die temperature within ±0.3°C (*±0.5°F*) of the set point, according ASTM specification.

All manufactured rubber goods have an ideal manufacturing temperature range. Most production processes such as molding, extruding, and calendaring include heat exposure to improve processability and to vulcanize. The MDR test chamber simulates the temperature in these processes.

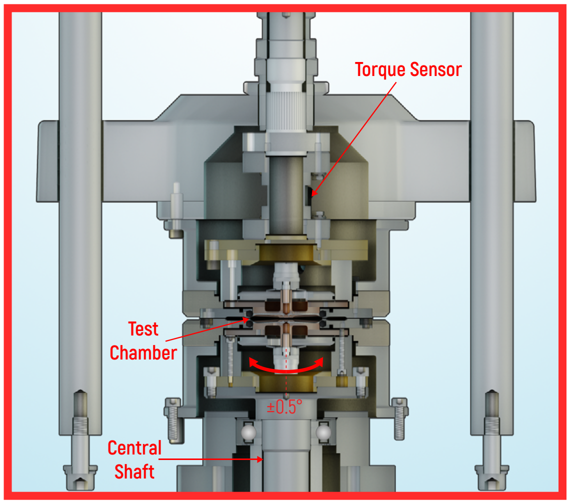
To calibrate, a temperature standard consisting of a certified sensor and indicator, is used by inserting the sensor into the test chamber, allowed to stabilize, to verify temperature is in tolerance. If the temperature is "Out of Tolerance", the temperature controllers are adjusted to bring them back in.

For further reference see: ASTM D5289-17 section 6.6

Torque

Torque - Torque Sensor

The torque sensor is essentially a precision spring with strain gages that produces an oscillatory electrical signal (sine wave) when strain, imparted by the oscillating lower die is transmitted through the sample. The amplitude of this signal in the positive half of the sine wave is captured and represents the torque signal. As the compound is heated within the MDR die cavity, the amplitude of torque signal will initially decrease, then increases significantly as the compound stiffens due to vulcanization. The peak value when plotted against time represents the cure curve.

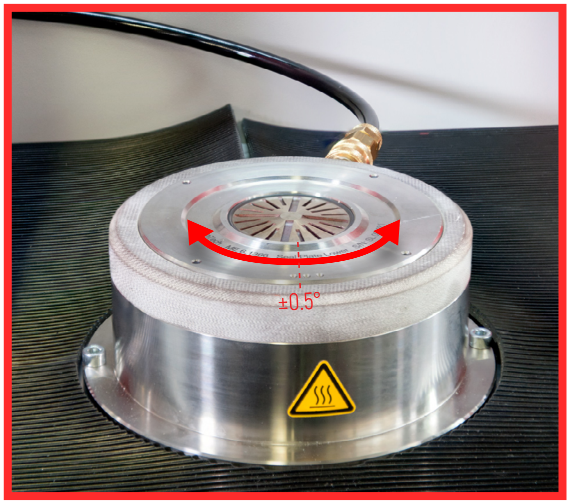


Cross-section showing die-heater sub-assembly oscillation.

The calibration process is similar to measuring a rubber sample through vulcanization except a certified torque standard is inserted between the conical dies. The elastic torque value of the precision torsion spring is compared to the instrument sensor to ensure it is within tolerance.

Control Standard Compound

A Standard Control Compound with known reference values is used as a final test. This test records data points for historical value, and ensures the results are within tolerance to meet the ASTM.



Lower die of MDR oscillates at ±0.5° to apply torsional force to the sample to measure torque.

For further reference see: ASTM D5289-17 sections 6.4.1 - 6.5.1.2

Angle & Physical / Dimensional

Angle - Angle of Oscillation

Calibration of angle of oscillation is typically derived using a certified torque standard.

For further reference see: ASTM D5289-17 section 6.3.1

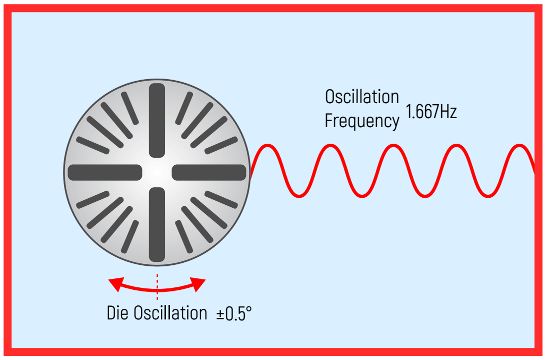


Illustration to visualize oscillation frequency and oscillation angle in an MDR.

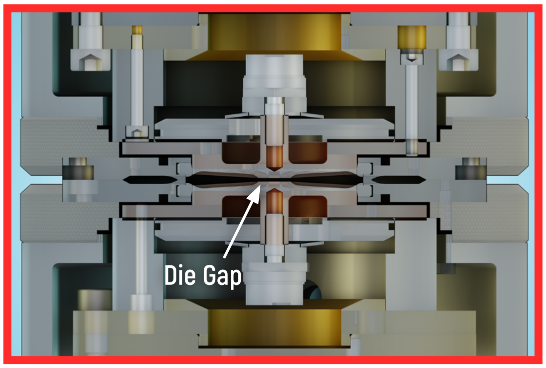
Physical / Dimensional - Die Diameter Calibration

Die diameter is measured using a certified Physical/ Dimensional measurement standard. This measurement is associated with the area of the die face.

Physical / Dimensional - Die Gap Calibration

Die gap calibration is performed using a certified rubber standard. Die gap is the measurement of height of the center of the cured standard rubber sample made with a certified control compound.

Note: Die gap is dependent on the modulus of compound being tested.



Cross section of MDR's test chamber to show die gap

Standard Control Compound

A Standard Control Compound with known reference values is used in setting the die gap. This test records data points for historical value, and ensures the results are within tolerance to meet the ASTM.

For further reference see: ASTM D5289-17 sections 6.2.1 - 6.2.2

ASTM D5289-17 Accredited Calibration

Calibration to the ASTM incorporates calibrating measurements in the above metrology disciplines.

About Us

With a 40 page scope of capability, **MonTech USA** has been providing 17025 accredited calibrations dedicated to the rubber and polymer industry for over 45 years.

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