LAB MANAGER'S GUIDE TO

ODR CALIBRATION

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Rubber Testing Solutions

What does ISO/IEC 17025 accreditation mean?

What is ISO/IEC 17025 Accreditation?

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

Why calibrate?

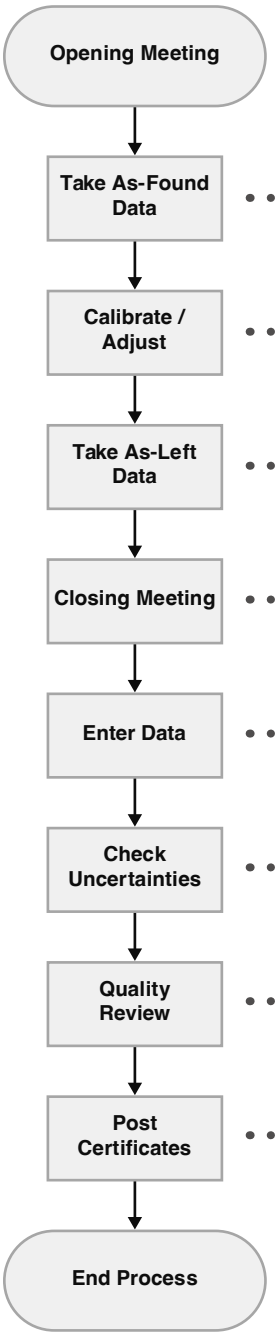
All process and measuring equipment experience degradation with time. Calibration is a process of comparing testing instruments, typically 3 to 10 times the accuracy of the instrument under test, while providing traceability of measurements. This improves product quality by keeping your products in tolerance.

To ensure consistency and reliability for ISO/IEC 17025 laboratories, part of the requirements of accreditation is verifying that the technicians are well-trained, knowledgeable, and properly follow procedures. Accredited technicians training includes principles of operation, observations by a metrologist in performing calibrations, and proficiency testing to complete training in the following disciplines and ASTMs:

- *Torque*
- *Oscillation Frequency*
- *Temperature*
- *Time*
- *Force*
- *Physical/Dimensional*
- *ASTM D2084 - Vulcanization Using an ODR*

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.

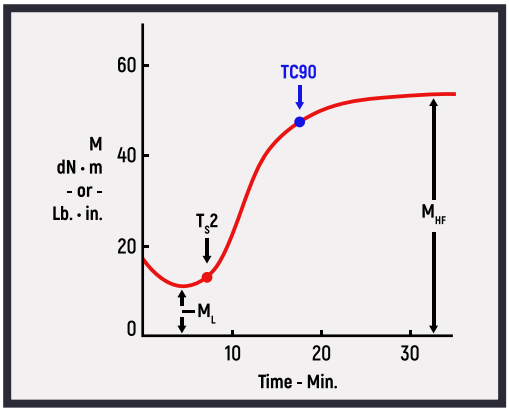
ODR (R-100) Theory of Operation

The ODR is a bench top press with cylinder, upper and lower heated platens, and upper and lower dies (moulds) that create the die cavity. The lower die allows for the insertion of the rotor into a collet in the central shaft located below the die. PT100 sensors in the platens indirectly control die temperatures also measured with PT100 sensors.

To create the angular oscillation of the rotor, the ODRs use a motor and a gearbox with and eccentric turning at 100 RPM to convert rotational motion to linear motion. A link arm connects the eccentric to a torque arm defining the chord of the arc driving the oscillatory motion of central shaft ±1, 3, or 5 degrees.

The central shaft assembly consist of a central shaft with a collet at one end and a draw bar connecting the collet to a clamp cylinder at the other end. A threaded rod, passes up through the clamp cylinder, the draw bar, and through the collet allowing adjustment of the rotor height. The torque arm with torque sensor is fixed to the central shaft. When testing the clamp cylinder draws down the collet fixing the rotor to the central shaft.

Torque measurement is accomplished with strain gages in full bridge configuration fixed to the torque arm. With excitation applied to the bridge, the output, a low-level signal is amplified in a signal conditioner and when plotted against time, shows a sine wave with a frequency of 1.667 Hz. A simple timing circuit then allows the capture the peak voltage at 90 degrees in the positive half of the cycle. A plot of this value against time is a plot of S', the cure curve.



Cure curve from ODR Test.

Why perform ODR calibrations to an ASTM?

The data an Oscillating Disk Rheometer (ODR) gathers enables rubber processors to understand the cure profile of a compound. If an ODR is out of calibration, the data gathered is subject to inaccuracy. To ensure that testing data is as accurate as possible, it is important to have regularly scheduled ISO/IEC 17025 calibrations performed on testing instruments.

An ASTM calibration for an ODR includes calibrating the instrument's: *Torque, Oscillation Frequency, Oscillation Amplitude, Temperature, Pressure/Closing Force, and Rotor Disk Diameter.*

Why is it important to receive a 17025 calibration to an ASTM?

The purpose of rubber testing is to accurately characterize the properties of a compound – and to have confidence in the validity of those measurements. If an ODR test is performed and any one point of measurement isn't within tolerance, it can negatively impact results (i.e., accuracy, repeatability, reproducibility). ODRs must be functioning properly and its measurements must be consistent with established standards to ensure the accuracy of test results. In practice, calibration is making a comparison to known certified standards.

Do all calibration service providers calibrate to an ASTM?

Quality/laboratory managers are responsible for the calibrations required, unless contracted for from an outside service. Not all calibration providers perform complete calibrations to an ASTM. When purchasing ODR calibration, review the calibration provider's technical scope of capability to ensure that parts of the ASTM are not excluded.

When should calibrations be performed?

Calibrations should be performed on a routine basis with an interval supported by data. Typically, the more an instrument is used, the frequency of calibration increases (i.e., 24/7). Calibration is also necessary if data is not in tolerance or suspect/questionable.

Torque Calibration

Torque

Torque is the primary measurement for understanding the cure profile of your compounds. It (S') is the opposing force to the oscillation of the rotor when testing a rubber compound. As the compound is heated within the ODR, the torque will initially decrease, then as the compound cures, it increases significantly. This torque measurement, when plotted against time, is the cure curve.

Overview of Torque Calibration

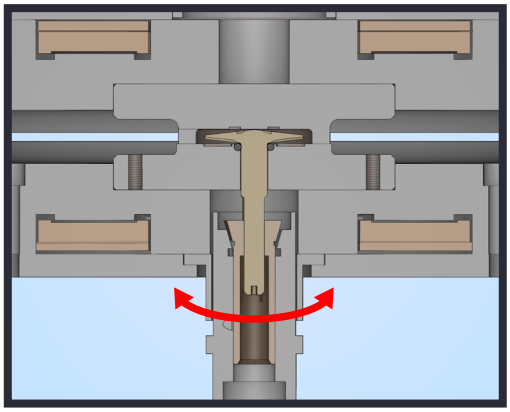
Torque transducers must be able to measure the amount of force imparted by the sample during oscillation with an accuracy that meets ASTM specifications.

"Running Zero"

This is the first step in the torque calibration, verifying that an empty test chamber torque reading is zero. The torque reading is taken and, if needed, adjustments are made to bring the reading to zero.

Calibration Standard Measurement

For the second step, torque measurements must be verified using a calibration standard. The standard is installed in place of the rotor and the platens are closed. After stabilization, the torque reading recorded in the test is compared with the standard's charted value to determine the corresponding degree of deflection.



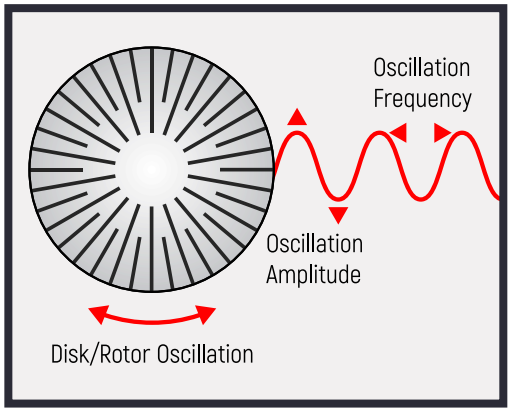
Cross-section of ODR test chamber. A motor/eccentric oscillates the central shaft and rotor to apply torsional force to the sample.

Rubber Control Sample

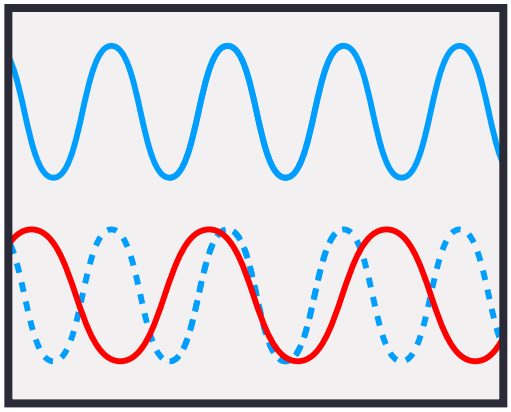
The final test must be performed using a control sample with known reference values and standard of deviation. This test records data points to ensure the results are within tolerance to meet the ASTM.

For further reference see: ASTM D2084-17 section 6.4

Oscillation Calibration



Rotor Oscillation in an ODR: *Frequency and Amplitude of Oscillation shown as a sine wave.*



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

Oscillation Frequency Calibration

Oscillation frequency calibration is performed using a timing standard. Oscillation frequency, part of the shear equation in ODR testing, is normally 1.667 Hz ±1% - though other frequencies may be used if required. Going above or below ±1% of the specified frequency will drastically affect the accuracy of the data produced by the ODR.

Oscillation Amplitude Calibration

Oscillation amplitude (oscillation angle) is driven by the eccentric in the instrument. Calibration of the oscillation angle is actually tied to torque calibration. The calibration standard used for torque calibration has a given rating for different angles of oscillation. If the torque value matches up with the rating at the instrument's set angle of oscillation, it can be deduced that the instrument is oscillating at the correct angle.

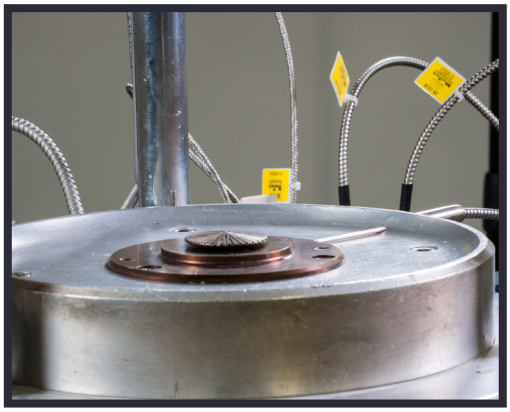
For further reference see: ASTM D2084-17 section 6.2.3

Temperature Calibration

Temperature

Temperature also plays a key role in an ODR's calibration. All manufactured rubber goods must undergo heat exposure during their production processes (molding, extruding, calendaring) and within the die cavity of the ODR. The heat in the die chamber vulcanizes the rubber sample enabling the ODR to gather torque data.

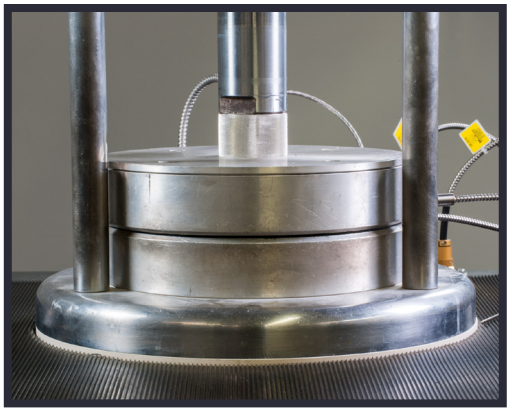
The temperature controlling system must be able to maintain a die temperature within ±0.5°C, or ±1°F of the specified test temperature.



Lower platen of an ODR

Temperature Calibration

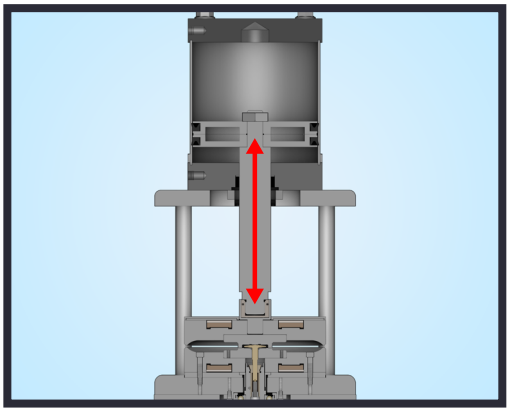
With the rotor and seal in place, the platens are closed and the temperature stabilizes to the temperature specified by the ASTM. A temperature standard is inserted into the hole in the upper platen used for measurement of the upper die. After stabilization, the temperature is verified. This is repeated for the lower die.



Platens closed for measurement of temperature.

For further reference see: ASTM D2084-17 section 6.3

Closing Force/Rotor Diameter Calibration



Cross-section of ODR platens and pneumatic cylinder.

Closing Force Calibration

The closing force of the platens comes from pressurized air in its pneumatic cylinder. This creates a sealed chamber, securing the sample and enabling the test to begin. If the positive pressure being exerted on the sample from the closing force is not high enough, or if the sample is too small, it can result in the sample not being securely held between the upper and lower dies – which can cause slippage, and affect torque measurements.

Closing force calibration is performed using a force standard to verify that the instrument's closing force between its platens is within tolerance, as specified by ASTM D2084-17.

For further reference see: ASTM D2084-17 section 6.2.4

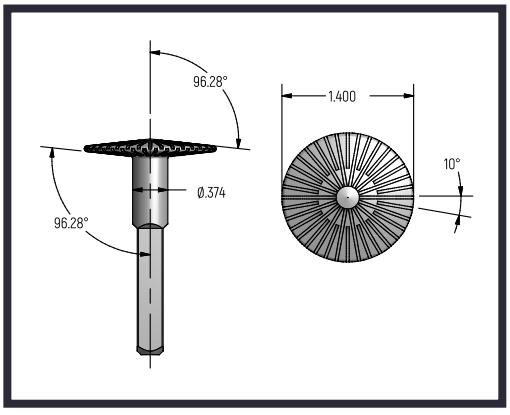


Diagram showing top and side views of an ODR rotor.

Rotor Diameter Calibration

The diameter of the rotor disk in the ODR must have the right dimensions to ensure accurate torque readings during testing. If a rotor is excessively worn, it creates extra space in the test chamber. This extra space can cause the sample to slip as the rotor oscillates - causing the torque reading to lower.

Using a dimensional standard, the diameter of the rotor disk is measured to verify it is within tolerance specified by the ASTM. The average value from multiple measurements using the dimensional standard is the value recorded as the rotor's diameter.

For further reference see: ASTM D2084-17 section 6.2.3

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