Test Methods for Elastomers

1. Definitions

1.1. Repeatability \( r \):
Test results obtained on identical material by one operator in one laboratory within a short interval of time on a 95% confidence level.

1.2. Reproducibility \( R \):
Difference obtained between two single test results – using identical material by operators in different laboratories using the standardized test method – not being exceeded with 95% probability.

1.3. Test counts and repetitions
Repeat tests on one sample three times. Destructive tests should be repeated three times, better 5 or 7 times, using the median as test result not the mean value of individual test.

2. Common Methods

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Norm</th>
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<tbody>
<tr>
<td>Abrasion</td>
<td>DIN ISO 4649</td>
</tr>
<tr>
<td>Accelerated Aging and Heat Testing</td>
<td>ISO 188</td>
</tr>
<tr>
<td>Compression Set</td>
<td>DIN ISO 815</td>
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<tr>
<td>Curemetry</td>
<td>DIN 53 529</td>
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<tr>
<td>Density</td>
<td>DIN EN ISO 1183</td>
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<tr>
<td>Elongation at break</td>
<td>DIN 53 504</td>
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<td>Flex cracking and crack growth (De Mattia)</td>
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<td>Hardness</td>
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<td>Rebound Resilience</td>
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<tr>
<td>Tear Strength</td>
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<tr>
<td>Tensile Strength</td>
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<td>Tension Set</td>
<td>DIN 53 504</td>
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</tbody>
</table>

Out of the above mentioned methods let us go into detail just for the most important ones used to characterize compounds for waste water aeration, Density, Elongation at break, Flex cracking, Hardness, Rebound, Tear and Tensile Strength, and last not least Tension set. Curemetry is used only to test raw compounds before vulcanization, so we do not cover it here, for details see DIN 53529 or ISO 3417.
3. **De Mattia Test DIN ISO 132**

There are various methods to test rubber parts under dynamic load, none of which really can replace testing parts in the field under realistic conditions. We focus on a popular method to determine the influence of continuous flexing per ISO 132.

### 3.1. Determination of Flex Cracking

Flexing judges the resistance of an elastomer against the build-up of cracks during a given dynamical deformation. The test specimen (150 x 25 x 6.3 mm; 6 x 1 x ¼ inch) is grooved and deformed by an Exzenter at 300 cycles per minute. The specimen is inspected at regular intervals until the first cracks occur. Thereafter inspection takes place at given intervals and the cracks are classified according to number, size and depth of cracks on a 1 – 6 scale and plotted in a diagram vs. the cycle counter.

### 3.2. Determination of Crack Growth

This test is performed the same way as described above, with the exception that a precise slit is cut into the middle of the groove. Cycle number and growth of slit are recorded.

4. **Density DIN EN ISO 1183-1**

Determination of density is fast and easy and is used widely to check the consistency of batches of compounds. Changes in density during the lifetime of a diffuser indicate a physical and/or chemical influence on the compound. To calculate density you simply measure the weight of a specimen (any shape) in air and submerged into a liquid of known density (usually water with 0.1% surfactants). The probe is hold by a fine wire (maximum diameter 0.125 mm). No air bubbles should adhere to the probe after submergence.

Density is determined according to

\[ \rho = \rho_L \cdot \frac{m_1}{m_1 - m_2} \]  

[ g/cm³ ]

With:

- \( m_1 \): Mass of specimen weighed in air
- \( m_2 \): Mass of specimen weighed in a liquid (mass of specimen minus mass of displaced liquid)
- \( \rho_L \): Density of test liquid (water about 1)

5. **Hardness DIN ISO 7619-1 (Shore A)**

Hardness is the resistance of a material to localized plastic deformation. Hardness often is a good indication of tensile and wear properties of a material. Hardness of a rubber compound depends much on the modulus of elasticity and is expressed on a 0 – 100 scale (soft to hard).

Test specimens must have smooth and parallel surfaces, and a minimum thickness of 6.3 mm, as results depend on material thickness. Stacking specimens is OK as longs as each layer exceeds 2 mm. Tests on non-standardized samples or curved surfaces (O-Rings) will cause greater deviations.

Precision little depends on level of hardness; repeatability is 2 Shore A and reproducibility 3 Shore.

Tensile strength (at break) is defined as the ratio of the force at break to the initial cross-section of the specimen. Elastomers usually exhibit the maximum force in the moment of breaking apart; consequently “tensile strength at break” is identical with “tensile strength at yield” at room temperature and above. (In contrast to many metal compounds)

Elongation at break is defined as the ratio of change of length (at the moment of breaking) to the initial length of the specimen.

\[ \varepsilon_R = \frac{(L_R - L_0)}{L_0} \times 100\% \]

Next to hardness test stress-strain properties are the prevalent methods to characterize elastomers, although most rubber parts are rarely employed under extreme elongation. It is appropriate to display the entire stress-strain-diagram in order to better understand the behavior of a given compound under stress. The numbers alone are much less useful.

See the following table for the precision of the test methods:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Repeatability</th>
<th>Reproducibility</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile stress at bread</td>
<td>6%</td>
<td>8 - 16%</td>
</tr>
<tr>
<td>Elongation at break</td>
<td>10 – 12%</td>
<td>19 – 24%</td>
</tr>
</tbody>
</table>

7. *Tear Strength DIN ISO 34*

ISO 34-1:2004 specifies three test methods for the determination of the tear strength of vulcanized rubber, namely

1. Method A, using a trouser test piece;
2. Method B, using an angle test piece, with or without a nick of specified depth;

Method A, using the trouser test piece, is preferred because it is not sensitive to the length of the cut, unlike the other two test pieces in which the nick has to be very closely controlled.

Variance of test results is extremely high: Trouser specimen exhibits a repeatability of 45% and a reproducibility of 72%. Angle Specimen got 28% and 60% respectively, not much of an improvement.
8. Tension Set DIN ISO 2285

Extent to which vulcanized rubber is permanently deformed after being stretched a specified amount for a given time. It is expressed as a percentage of the original length or distance between gage marks. Results are significantly time-dependent. Tension set may be determined as “deformation after constant elongation” or “deformation under constant tensile load”. The first method requires less elaborate equipment and consequently is preferred by most manufacturers. Nevertheless diffusers are operated under constant tensile load, even increasing over the years of service.

Specimens look similar to the above mentioned tensile probes and are preferably 2 mm thick and 6 mm wide with 25 to 50 mm between distance marks. After measuring the original length, the specimen is slowly elongated to its test length; typical elongations are 25%, 50%, or 100%. After 10 to 20 minutes the elongated distance between markers is determined. Specimens are kept under constant temperature (room temperature or elevated temperature) for 24 hours. (72 and 168 hours are optional). Thereafter specimens are relaxed slowly and stored for 30 minutes on a non-sticking wooden surface; finally the relaxation length is determined and tension set is calculated:

\[
E = 100 \cdot \frac{(L_2 - L_0)}{(L_1 - L_0)} \quad \text{[%]}
\]

- \(L_0\): original length
- \(L_1\): length after elongation
- \(L_2\): length after relaxation

9. Material Analysis

9.1. Thermo-Gravimetric Analysis

Thermo-gravimetric analysis (TGA) uses heat to force reactions and physical changes in materials. TGA provides quantitative measurement of mass change in materials associated with transition and thermal degradation. TGA records change in mass from dehydration, decomposition, and oxidation of a sample with time and temperature. Characteristic thermo-gravimetric curves are given for specific materials and chemical compounds due to unique sequence from physicochemical reactions occurring over specific temperature ranges and heating rates. These unique characteristics are related to the molecular structure of the sample.

The analyzer usually consists of a high-precision balance with a pan loaded with the sample. The sample is placed in a small electrically heated oven with a thermocouple to accurately measure the temperature. The atmosphere may be purged with an inert gas to prevent oxidation or other undesired reactions. A computer is used to control the instrument. A derivative weight loss curve can be used to tell the point at which weight loss is most apparent.

Analysis is carried out by raising the temperature gradually and plotting weight against temperature. After the data is obtained, curve smoothing and other operations may be done such as to find the exact points of inflection.
Sampling material is heated under nitrogen gas until about 500 °C to determine low volatile ingredients like oil, waxes and the like (40 – 420 °C). Polymers degrade between 420 and 500 °C. Above 500 °C synthetic air is added to oxidize carbon black (500 – 650 °C). Calcium Carbonate is detected above 650 °C degrading to Calcium Oxide (Limestone to Quicklime reaction). The remaining residue is an indication for other inert fillers (china clay¹, Titanium oxide etc.)

**9.2. Extraction, IR-Spectroscopy, and more**

Most rubber compounds may be modified within broad limits in order to alter certain properties or to ease processing of compound. Plasticizers, waxes, anti-ageing and UV-protection additives as well as curing agents are supplemented to the main ingredients of a rubber compound. One way to analyze these substances is solvent extraction and sequential chemical and/or physical analysis of the extract. IR-spectroscopy is a fast and easy method to compare compounds with back-up samples from the past in order to determine any changes of constituents. Gas and liquid chromatography, mass spectrometry as well as IR and UV spectrometers may be employed to analyze new and used rubber parts for changes during operation. Interpretation of all methods mentioned above requires thorough comprehension of the application, experience, and intuition.

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¹ The word Kaolin is derived from the Chinese city 高岭 Gaoling (gāo lĭng = high mountain range)

[1] DIK-Homepage, Anwendungen + Methoden, [www.dikautschuk.de/angebot/bsp/_info.htm](http://www.dikautschuk.de/angebot/bsp/_info.htm)