9. Test Procedures

There are standard ASTM, ISO, and DIN procedures for conducting most of the tests on rubber materials. It is important to follow these procedures carefully in conducting tests if uniform results are to be obtained. For instance, in pulling specimens to find tensile strength, elongation and modulus values, ASTM method D412 requires a uniform rate of pull of 20 inches (500 mm) per minute. In a test, tensile strength can be found to decrease 5% when the pulling speed is reduced to 2 inches per minute, and a decrease of 30% when the speed is reduced to 0.2 inches per minute.

Different international norms for elastomers exist. In the following table is a comparison of the DIN, ISO, and ASTM norms. These norms are comparative, but can be slightly different.

Test Specimens
ASTM test methods include description of standard specimens for each test. Often two or more specimens are permitted, but results from the different specimens will seldom agree. The way that properties vary with the size of the specimen is not consistent. For instance, as the cross section increases, nitrile O-rings produce lower values of tensile strength, elongation, and compression set. Likewise, ethylene propylene O-rings produce a similar pattern for tensile and elongation values but not for compression set. In Fluorocarbon compounds, only the elongation changes.

In fluid immersion tests, O-rings with a smaller cross section can be found to swell more than larger O-rings, while in explosive decompression tests the smaller cross sections will have better resistance to high pressure gas.

<table>
<thead>
<tr>
<th>DIN Standard</th>
<th>ISO Standard</th>
<th>ASTM Standard</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>DIN 53519 T2</td>
<td>ISO 48 M</td>
<td>ASTM D 1415</td>
<td>Hardness, IRHD</td>
</tr>
<tr>
<td>DIN 53479</td>
<td>ISO 2781</td>
<td>ASTM D 1817</td>
<td>Specific Weight</td>
</tr>
<tr>
<td>DIN 53505</td>
<td>ISO 868</td>
<td>ASTM D 2240</td>
<td>Hardness, Shore A</td>
</tr>
<tr>
<td>DIN 53517</td>
<td>ISO 815</td>
<td>ASTM D 395 B</td>
<td>Compression Set</td>
</tr>
<tr>
<td>DIN 53504</td>
<td>ISO 37</td>
<td>ASTM D 412</td>
<td>Tensile Strength</td>
</tr>
<tr>
<td>DIN 53504</td>
<td>ISO 37</td>
<td>ASTM D 412</td>
<td>Elongation at Break</td>
</tr>
<tr>
<td>DIN 53518</td>
<td>ISO 2285</td>
<td></td>
<td>Tension Set</td>
</tr>
<tr>
<td>DIN 53521</td>
<td>ISO 1817</td>
<td>ASTM D 471</td>
<td>Immersion Test</td>
</tr>
<tr>
<td>DIN 53508</td>
<td>ISO 188</td>
<td>ASTM D 573</td>
<td>Aging in Air</td>
</tr>
<tr>
<td>DIN 53509</td>
<td>ISO 2921</td>
<td>ASTM D 1329</td>
<td>Low Temperature Behaviour, TR10-Test</td>
</tr>
<tr>
<td>DIN 53515</td>
<td>ISO 1431</td>
<td>ASTM D 1149</td>
<td>Ozone Resistance</td>
</tr>
<tr>
<td>DIN 53515</td>
<td>ISO 34-1</td>
<td>ASTM D 624</td>
<td>Tear Resistance</td>
</tr>
</tbody>
</table>
9. Test Procedures

How to properly deal with Hardness and Compression Set

Hardness Testing is the easiest test to carry out on O-rings, but a proper interpretation of the hardness may be difficult. The hardness commonly mentioned in the various data sheets refers to the standard measuring method by DIN or ASTM. This means that the test has been carried out on a standard slab of .08 inch (2 mm) or a button of .5 inch (12 mm) thickness. O-ring hardness measurements differ from measurements on slabs. In addition, the value per each individual O-ring cross section will vary as well: a small cross section of e.g. .08 inch (2 mm) will give higher values than a crosssection of .275 inch (7 mm) for the same compound.

To make it even more complex, a distinction must be made between the two measuring standards: Shore A and IRHD. IRHD is more and more being used for O-rings. Measuring results of both methods may differ. It is peculiar that the difference depends on the kind of rubber. HNBR will show more deviations than FKM.

What conclusion should consequently be made? With a view to the application, hardness is a parameter of relatively minor importance. The service life of an O-ring will not drastically be changed by a small difference in hardness. Please also note that data sheets always state ±5 points on IRHD or Shore A values. It is recommended that when testing hardness, testing methods should always be the same (same equipment, same specimen). In these circumstances a comparison is useful.

Hardness Shore A readings taken on actual O-rings are notorious ly variable because O-rings do not have flat surface and operators will vary in the accuracy with which they apply the indenter to the crown of the O-ring, the point that gives the best reliable reading. Therefore it is better to order compression set buttons from the same batch as the O-ring for the hardness test.

As to compression set it should be observed that one should carefully watch the information in the data sheet. In most cases the compression set stated has been measured on a slab or a button. This gives totally different values than measurements on actual O-rings. O-rings will show different values depending on the thickness of the sample. Small crosssections will give a higher value than large crosssections. The NBR and EPDM compression set values are commonly stated at 100°C (212°F); for EPDM PC at 150°C (300°F) and for VMQ and FKM at 200°C (390°F).

The lower the values, generally the better the sealing performance. See also the calculations of service life in which this subject is discussed extensively.

Compression set of the standard ERIKS O-rings is measured on an O-ring with a crosssection of .139 inch (3.53 mm). Consequently all qualities can be compared.

Changes in Environment
Changes in a fluid medium can occur due to the effect of heat and contaminants during service so that a rubber that is virtually unaffected by new fluid may deteriorate in the same fluid after it has been used for a certain time. For this reason it is sometimes better to run tests in used fluids.

Aging
Deterioration with time or aging relates to the nature of the bonds in the rubber molecules. Three principle types of chemical reactions are associated with aging.

• Cracking. The molecular bonds are cut, dividing the molecular chain into smaller segments. Ozone, ultraviolet light, and radiation cause degradation of this type.

• Cross linking. An oxidation process whereby additional intermolecular bonds are formed. This process may be a regenerative one. Heat and oxygen are principle causes of this type of attack.

• Modification of side groups. A change in the molecular complex due to chemical reaction. Moisture, for example, could promote this activity.

All mechanisms by which rubber deteriorates with time are attributable to environmental conditions. Selection and application of O-rings to provide acceptable service life is the main subject of this handbook.
9. Test Procedures

Service life of an O-ring
Calculations of Service Life

For calculating the service life of O-rings ERIKS makes the assumption that the sealing life of an O-ring is zero, once the compression set has reached 100%. This indicates that the rubber has practically no elasticity and that the sealing force has become minimal so that leakage can easily develop. The best method to determine these values is to carry out “long-term” tests at a certain temperature under a certain pressure in a certain media. This is generally done in air, being the most receptive to aging. Field tests have shown that within the same polymername service life can vary widely e.g. 1000 to 6000 hours. Several compounds have been field tested by ERIKS for thousands of hours to determine the time at which the leakage occurs under specified conditions.

As mentioned before, the service life of O-rings depend on both the formulation quality and the product quality. The formulation quality indicates the maximum properties of the compound. This quality is generally tested on each batch. The product quality strongly depends on the production process control.

Life Tests

The extensive tests which preceded the determination of the O-ring service life have enabled ERIKS to give an indication of service life by means of short term tests i.e. life time tests. A service life test is a laboratory procedure used to determine the amount and duration of the resistance of an article to a specific set of destructive forces or conditions. Today, it is possible to perform these tests under the same conditions as those prevailing in many actual situations. In this way a picture of the service life of the seal can be achieved.

Please observe that service life varies depending on the cross section and the temperature. The latest, most state-of-the-art computer programs can now predict these service life graphs by each individual cross section.

This chapter gives an example of the results of life time tests according ISO 815 for different NBR compounds: A, B, and C, 70 shore O-rings with cross section .139 inch (3.53mm). This shows that different NBR-70 compounds give different life times.

Life tests for O-rings NBR 70° shore
.139 inch (3.53 mm) / acc. ISO 815 / compression set 100% / hot air

<table>
<thead>
<tr>
<th>NBR compound</th>
<th>Compound A</th>
<th>Compound B</th>
<th>Compound C</th>
</tr>
</thead>
<tbody>
<tr>
<td>At 140°F (60°C)</td>
<td>65,000 hr (6.5 yr.)</td>
<td>48,000 hr (4.8 yr.)</td>
<td>40,000 hr (4 yr.)</td>
</tr>
<tr>
<td>At 175°F (80°C)</td>
<td>17,000 hr (1.7 yr.)</td>
<td>10,000 hr (1 yr.)</td>
<td>9,500 hr (11 m.)</td>
</tr>
<tr>
<td>At 212°F (100°C)</td>
<td>3,000 hr (4 m.)</td>
<td>1,900 hr (2.3 m.)</td>
<td>1,800 hr (9 wk)</td>
</tr>
<tr>
<td>At 257°F (125°C)</td>
<td>740 hr (4 wk.)</td>
<td>500 hr (18 d.)</td>
<td>450 hr (16 d.)</td>
</tr>
</tbody>
</table>

* Consult ERIKS for other lifetime tests.
9. Test Procedures

Life tests on 70 shore O-rings with a cross section of .139 inch (3.53 mm) in a sulphur cured EPDM and a peroxide cured EPDM give the following values. These values show the large difference in results between the two curing systems.

<table>
<thead>
<tr>
<th>EPDM compound</th>
<th>Sulphur cured</th>
<th>Peroxide cured</th>
</tr>
</thead>
<tbody>
<tr>
<td>At 140°F (60°C)</td>
<td>100,000 hr (10 yr.)</td>
<td>1,000,000 hr (100 yr.)</td>
</tr>
<tr>
<td>At 175°F (80°C)</td>
<td>52,000 hr (5.2 yr.)</td>
<td>250,000 hr (25 yr.)</td>
</tr>
<tr>
<td>At 212°F (100°C)</td>
<td>8,500 hr (10 m.)</td>
<td>34,000 hr (3.4 yr.)</td>
</tr>
</tbody>
</table>

Factors for small c.s. .070 inch (1.78 mm)

<table>
<thead>
<tr>
<th></th>
<th>For large c.s. .275 inch (6.99 mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>At 175°F (80°C)</td>
<td>: x 0.75</td>
</tr>
<tr>
<td>At 212°F (100°C)</td>
<td>: x 0.65</td>
</tr>
<tr>
<td>At 257°F (125°C)</td>
<td>: x 0.65</td>
</tr>
</tbody>
</table>

Generally, in air, the service life for NBR 70 O-rings at 100°C (212°F) in the various compounds range from 2000 to 3000 hours. EPDM O-rings at 100°C (212°F) sulphur cured or peroxide cured range from 8,500 to 34,000 hours, depending on the compound. Other Eriks O-ring compound life test results are available. Please contact an ERIKS representative for more information.

These test results give the number of expected hours, during which the compression set reaches (100%) when tested in air. When testing in oils, the service life is considerably higher (not for EPDM). Warm air can be a very aggressive environment for rubber.

Since the interpretation of this data requires some explanation, please contact an ERIKS representative for more information.
9. Test Procedures

Extra service for O-rings
In addition to test procedures specified by ERIKS, there are also different possibilities for specific quality assurance systems:

• compression set testing
• hardness control following Shore A or IRHD
• surface control to Sortenmerkmal S (surface defect control)
• specific measurement to special tolerances
• special surface control
• tear strength test
• tensile strength test
• ozone testing
• lifetime testing
• chemical resistance tests
• infrared spectroscopy
• TGA-analysis
• FDA migration test
• TOC analysis
• FEA calculations
• Clean Room

These tests are carried out in our MTC (Material Technology Center) in Manchester, UK.
9. Test Procedures

Testing of hardness
The most used method for hardness testing of rubber parts is the Shore A. On O-rings or moulded parts the Microhardness in IRHD is more specific. The drawing shows the two methods.

IRHD: the norm is measuring on a 2mm sheet during 30 seconds. The shore A is measured on a 6mm sheet during 3 seconds. The first method is very sensitive for surface imperfections, the second is difficult for measuring at small cross-sections.

Compression set:
In designing an O-ring seal, it is important to determine the O-ring compound early, as the compound selected may have an influence on the gland design. The application determines the rubber compound, the primary factor being the fluid to be sealed. But the elastomer must also resist extrusion when exposed to the maximum anticipated pressure and be capable of maintaining good physical properties through the full temperature range expected.

This chapter discusses the next criteria that must be considered like compression set, hardness, tensile strength, chemical compatibility, thermal effects, pressure, and extrusion. Data and procedures enabling the designer to meet particular requirements or obtain specific performance from the seal will be found in this chapter.

Compression Set and Squeeze
Compression set is the percentage of deflection that the elastomer fails to recover after a fixed period of time under a specific squeeze and temperature. Compression set is a very important sealing factor, because it is a measure of the expected loss of resiliency or “memory” of a compound. Compression set is generally determined in air and measured as a percentage of original deflection. Although it is desirable to have a low compression set value, this is not so critical as it might appear because of actual service variables. For instance, an O-ring may continue to seal after taking a 100% compression set, provided the temperature and system pressure remain steady and no motion or force causes a break in the line of seal contact. Also, swelling caused by contact with the service fluid, may compensate for compression set. The condition most to be feared is the combination of high compression set and shrinkage. This will lead to seal failure unless exceptionally high squeeze is employed. Compression set is calculated as follows:

\[ C = \frac{t_0 - t_1}{t_0 - t_s} \times 100 \% \]
9. Test Procedures

**Tensile strength and elongation:**
In the tensile strength the tension limits of the rubber are determined. Tensile strength and elongation at break are the outcome of the test. The test is foll DIN 53504 or ISO37.

Elastomers are determined as follows:
- 0-5 MPa: low
- 5-10 MPa: middle
- 10-15 MPa: high
- 15-20 MPa: very high

The DIN 53504 or ISO 37 is made as follows:
- S2 rod
- Speed: 200mm/min

**Chemical resistance:**
Chemical resistance tests which determine:
- Volume increase
- Hardness change
- Change of network structure in the rubber
are possible on all compounds.
Tests are carried out foll DIN ISO 1817 and specific VDA test-methods.

**Aging properties/lifetime tests:**
Due to tests on hardness, volumeswell, tensile strength and elongation at different temperatures it is possible to define the heat resistance of seals in different compounds. Tests are carried out foll ISO 188.

**Ozon resistance:**
Due to climate changes ozon at higher concentreat in can influence highly the lifetime of a seal or rubber part. Mainly NR and NBR at 3 to 5% elongation are very ozon-sensitive. NBR with ozon protection gives much better results. Tests at different ozon concentrations, different elongations and different time (e.g. 48 hours) give comparative results. Ozon in these conditions gives scratches in the rubber.
Tests are carried out foll ISO 1431.
9. Test Procedures

**TGA (thermogrific analysis):**
In this method a compound is burnt till only the ashes remain. At different temperature stages different ingredients burn and the weight change is put in a diagram. This method gives a good idea of the composition of the compound.

**Infrared spectroscopy (FT/R):**
The resonance vibrations of the molecular connections are registered by this method. In this way functional groups are defined and this allows to control the chemical composition of the elastomer.

**Basler inspection machine: 100% inspection of O-rings:**
Via Basler inspection machines the tolerances and the surface imperfections are controlled by a camera system.

**Cross section measurement:**
Via a laser controlled system the coresection is controlled automatically.

**Surface Defects by magnification:**
A 2x or 4x magnification allows us to control the surface defects of small quantities of seals.

**Finite Element Analysis (FEA):**
Our Finite Element Analysis capability allows us to provide an accurate mathematical prediction of seal performance. This technology allows us to model seal performance on the computer and ensure right first time solutions to sealing applications. The type of analysis we undertake can be as simple as that of calculating the maximum force required to insert a seal or as complex as predicted a seals performance after many years of service. We undertake customer specific projects that can combine our material testing and dynamic testing capabilities, in conjunction with FEA prediction, to provide data that provides a high level of confidence the seal will retain its integrity over the designed life of the application.
9. Test Procedures

Clean Room:
A clear vision on clean products
Due to a growing awareness in the field of purity, industry focuses more and more on effectively controlling manufacturing processes. Goal is to prevent technical components from being the cause of pollution in processes and systems. Pollution can originate from airborne solid particles, which can be visible or invisible. These particles are called aerosols. To avoid this type of pollution is an important factor in the pharmaceutical-, semiconductor and food industry.

ISO class 7
The size of a human hair is 100 µm on average. Nowadays it is possible to filter up to 0.5 µm with very good results and relatively little effort. This filtering takes place in the cleanroom. This sealed space is filled with air in which only a limited number of particles, with a size of 0.5 µm or more, float about. The current norm for the industry is ISO 14644-1. Within this norm Eriks uses ISO class 7 (also known as class 10,000) for her cleanroom. The cleaning and packing takes place in an, even tighter, class 100 environment which is accepted by 90% of the industry.

Cleanroom cleaning and packing
ERIKS is fully equipped to take over your cleaning and packing activities in one’s own cleanroom. Out-gassing of rubber products by means of vacuum ovens is also an added ERIKS-value to the market.