

# *Elastocon*<sup>®</sup>

Testing with precision

## Rubber testing



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# Rubber testing

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# Standardisation

## What is a standard and standardisation?

To create a standard is to set up rules, create order and make selections, in order to rationalize as well as to define various requirements, e.g. concerning matters of safety etc. A standard tells you how to do something how to produce a particular object, use a terminology, and to employ a method of testing or some other procedure in a specific manner.

An International standard has first been set up in accordance to a particular set of rules and it is then approved and implemented. A standard can be regarded as an agreement between the authorities, manufacturers, consumers and other interested parties in all participating nations and it is an effective and powerful tool precisely because these parties have worked out the standard together.

## Why are standards needed?

Without standards life would be much more complicated. Let us take paper sizes as an example. When we use A4 and A5 paper sizes, the paper fits most typewriters, most envelopes, most folders, files, drawers and shelves. Using A4 and A5 sized paper makes it simple and cheap to write and file letters.

## When did standardisation begin?

Standards have always existed in all periods of history, even though they come in other forms than the ones we know today. Examples of old standards are coins, measurements, weights, road construction and building standards. There are, however, not many standards which have remained unchanged until the present day. When techniques and fashions change, standards change as well. There is however one particular standard which is exactly the same today as when it was first created and came into general use during the Middle Ages. This is, of course, the standard of how to design musical notes.



## What use are the standards?

Without standards many products and services would become more costly. If we can reach an agreement about something and that is what standards are infact all about, like limiting the available range of varieties, companies will only need to manufacture a limited number of varieties of their products, e.g. nuts and bolts. They can then produce longer runs, which will in turn help to reduce the price of the article.

Without standards many products would be of an inferior quality. In standards, agreement is made about the important quality requirements and specifications which a product must comply with. Using the same standard, the products can also be tested to ensure that they meet these requirements. Standards also determine the significance and meaning of various words and terms. We are then able to express ourselves more clearly and hence avoid misunderstandings.

## How is a standard created?

A great deal of work is done before a standard is completed and accepted. A technical committee has to produce a proposal. The committee consists of representatives for the manufacturers, customers, government authorities and testing and research institutions. When a proposal has been drafted, it is sent for consideration to all those who might be affected by the new standard.

When replies and criticism of the draft have been reviewed and changes have been made, it is submitted to the technical committee of the concerned National Standardization Institute for final approval. The time taken to produce the final version of a standard can vary from a couple of months to several years.

## Who pays for all this?

The costs of creating a new standard are generally paid for with subsidies from the industry in general and from governments. Companies and the various authorities normally pay the costs for their participation in the technical committees. When a standard has been approved, it is printed and published. All the interested parties then purchase copies of the standards.

## How is international standardisation carried out?

Internationally, the co-operation in standardisation is carried out within the ISO, the International Organisation for Standardisation, which has 87 member countries. The counterpart within electrical standardisation is the IEC, International Electro technical Commission. In addition to the ISO and IEC there are the European organisations for standardisation, CEN (European Committee for Standardisation) and CENELEC (European Committee Electro technical for Standardisation). The importance of these organisations has greatly increased in recent years since the European Union uses EN standards to harmonize the requirements on products within EU countries. In the USA the ASTM (American Society for Testing and Materials) is a dominating organisation in the testing area.

There are approximately 10 000 standards published by ISO and IEC. Many of these have also been approved as national standards in many countries.

The work within the ISO is delegated to technical committees (TC) of which TC45 deals with rubber and rubber products and TC 61 deals with plastic and plastic products.

Through the international co-operation in the field of standardisation, the standards of all the various countries tend, especially with regard to methods of testing, to be similar to each other. This is the whole aim of international standardisation.



Meeting of TC 45/SC2/WG1, physical testing.

Photo: Göran Spetz

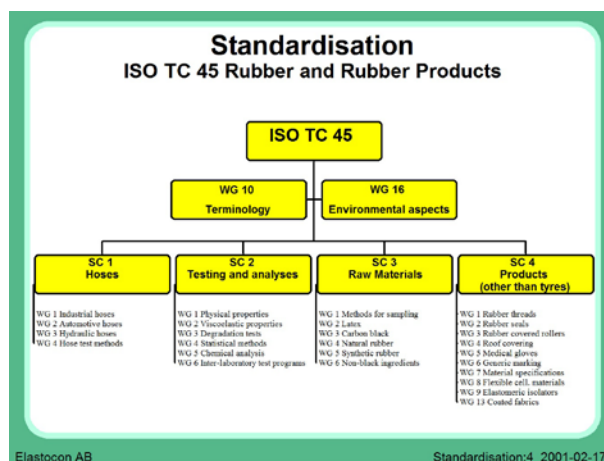
## Quality and environmental standards

In recent years within the ISO a series of standards for quality systems in companies has been produced, known as the ISO 9 000 series. ISO 9 000 standards have been produced in order to standardize the quality work in companies and they specify the minimum requirements for organisations, procedures, documentation systems and the calibrating of testing instruments. More and more companies are now being certified according to ISO 9 000. ISO 14 000 is also a similar standard, which applies to environmental work in companies.



Plenary meeting of ISO TC 45.

Photo: Göran Spetz





# Preparation of test pieces

## Preparation of test pieces

The testing of rubber is either starting from uncured rubber or from cured rubber products. Testing can also apply to the uncured or cured state. The preparation of the test pieces is an extremely important part of the testing itself and it must not be the case that the test results reflect the effects of the preparation rather than the properties of the materials being tested.

Temperature conditioning of the test material is also an important part of the preparation.

## Testing cured properties ISO 4661

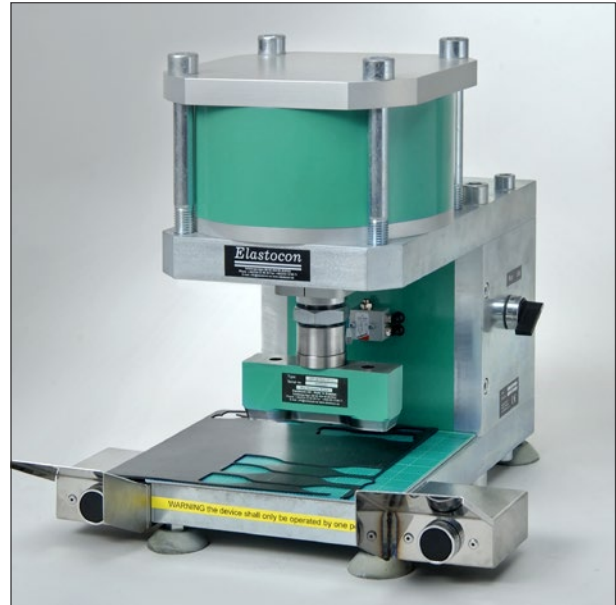
In order to test cured properties, specimen test sheet and other test pieces need to be produced. Nearly all rubber testing is carried out on  $2 \text{ mm} \pm 0,2 \text{ mm}$  thick test sheets, which are vulcanised in a mould, often with four cavities, each  $150 \times 150 \text{ mm}$ .

Cylindrical test pieces are used for compression set, relaxation, abrasion and compression testing and are also produced in a mould. There are two common sizes, the so called small test piece with a diameter of 13 mm and a height of 6,3 mm, and a large test piece 29 mm in diameter and 12,5 mm high.

Test pieces are punched out of the test sheets in order to perform tests for tensile strength, tear strength and tension set etc. It is important that sharp punching cutters or dies are used against a soft surface when punching out the test specimens. Punching cutters with poor cutting edges or nicks may cause rupture notches in the test piece and thus produce incorrect results.

## Testing uncured properties

When testing uncured rubber, it is often sufficient to cut off a piece of rubber of the appropriate size using a pair of scissors. In certain tests, as for example, the determination of cure curves, the test must have a specific volume in order to be able to provide reproducible test results. In these cases, a volume press is used to provide test pieces with a constant volume.



Cutting press with cutting dies.

Photo: Elastocon



Specimen volume press. Photo: MonTech

## Testing materials in products

In certain cases, one is interested in testing the material properties of a rubber product. Sheets 2 mm thick or cylindrical pieces are cut from the product for testing. A slicing machine or a cutting machine is very useful for this purpose and consists of a band knife or a rotating blade, which can cut off thin strips from a product.

The cylindrical pieces are cut from thicker parts of a product with a rotating knife.

## Conditioning ISO 23529

Prior to testing, the test material must often be conditioned. After test sheets are cured, there is a specified waiting time of at least 16 hours before testing, in order to allow the material to stabilise. Test pieces must also be given time to assume the testing temperature, which may take a few hours. Certain materials such as cellular materials must also be conditioned to achieve the correct balance of moisture, which may take a considerably long time. The standardised climatic conditions in a laboratory are  $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  and  $50\text{ }\%$  RF  $\pm 5\text{ }\%$  RF.

## Conditioning time for rubber

Time in minutes to obtain evenness of temperature.

Thickness mm	Temp $^{\circ}\text{C}$	Time min
25	-50	135
	$\pm 0$	95
	100	140
10	-50	45
	$\pm 0$	30
	100	45
5	-50	20
	$\pm 0$	15
	100	20



Rotating knives for cutting out compression set test pieces. Photo: Elastocon



Slicing machine for preparing 2 mm test sheets. Photo: Bizerba

# Testing of uncured rubber

## Batch control

Batch control is the name given to the tests, which entails that each batch of mixed rubber is checked before it is used in any further processing. The testing can include the determination of viscosity, cure curve, density and hardness.

## Viscosity – Plasticity

In the processing of rubber, it is important that the material has a suitable viscosity for the purpose. If the viscosity is too low the material will become difficult to process, e.g. due to increased stickiness, cold flow and low strength. If the viscosity is too high, processing requires great amounts of power and energy. The mechanical energy used in the processing is transformed into heat, which increases the risk of scorching. Measuring the viscosity of rubber polymers and compounds is therefore one of the more usual controls when producing rubber.

The term "plasticity" is often used simultaneously with "viscosity". High plasticity is the same as low viscosity. Rubber polymers and rubber compounds have very complicated flow properties or rheological properties as they are also called. For normal liquids of low viscosity (Newtonian fluids) such as water, one single material constant – viscosity – is sufficient to be able to characterise their flow properties.

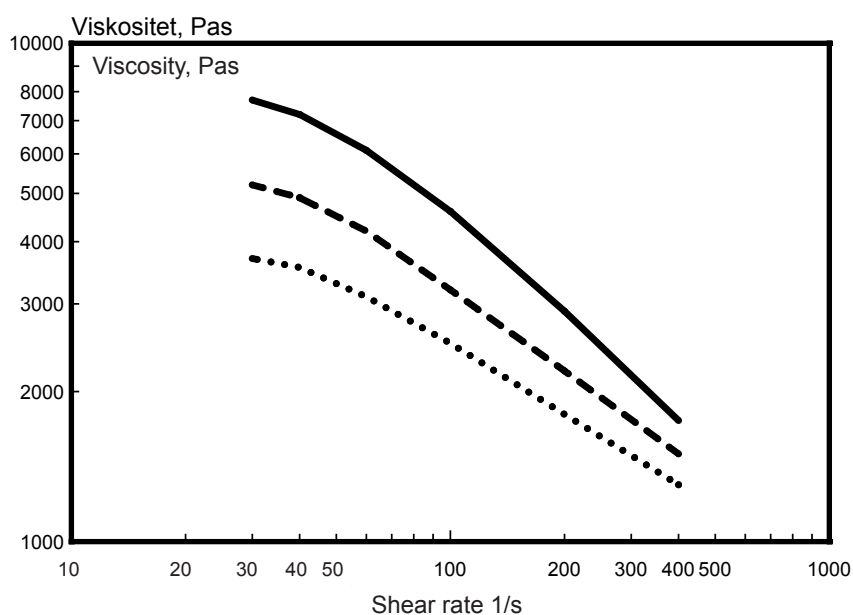


Laboratory for batch control.

Photo: Alf Gustavsson

For rubber, however, viscosity varies with the shear rate. In addition, viscous deformation is combined with the effects of elastic deformation.

Finally, rubber is more or less thixotropic, which is why the flow properties depend on the degree of processing that has been used in previous processes. All this entails different measuring methods for the determination of the rheological properties can rank materials differently depending on the test conditions used. This also means that laboratory results could differ greatly from what they are in practice, e.g. if the shear rate differs in both cases.



Viscosity as a function of the shear rate for three rubber compounds.



## Rotation viscometers ISO 289

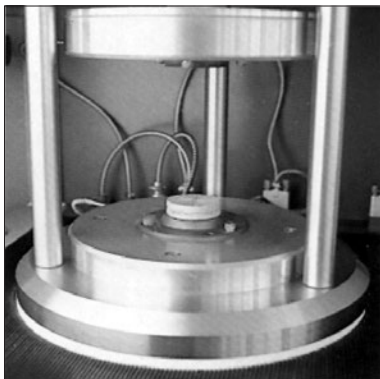
The most common instrument used in determining the viscosity of rubber polymers and compounds is the Mooney viscometer. The Mooney viscometer can be described as a rotation viscometer, in which the rubber is sheared between an inner rotor and an outer die. The die has a diameter of 50 mm and a height of 10 mm. The normal rotor has a diameter of 38 mm. For the measurement of high viscous materials, there is a smaller rotor with a diameter of 30 mm. The temperature of the die can be adjusted and normally 100 °C is used. The rotor is rotated with a speed of 2 rpm. This corresponds to an average shear rate of 1 s<sup>-1</sup>. The torque required for driving the rotor, which is a measurement of the viscosity, is measured and presented in Mooney units.

8,3 ± 0,02 Nm corresponds to 100 Mooney units. Placing two circular pieces of the testing material in the die, one under and one above the rotor does the testing. The die is closed using a force of 11 500 N and the material fills the die. Preheating is normally carried out for 1 minute after which the rotor is started. The viscosity decreases at first because of the heating and degradation of thixotropic properties. Readings are then normally taken after 4 minutes.

The result is normally given as follows:

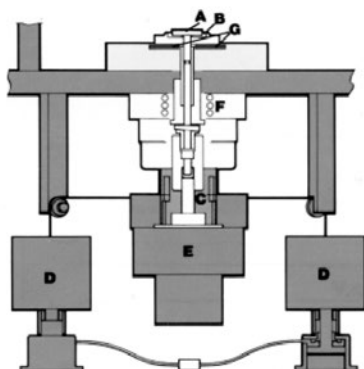
**55 ML (1+4) 100 °C.** M here stands for Mooney, L large rotor and 1 + 4 the preheating time and reading time.

Values under 40 can be designated as low, values between 40 and 80 as normal and values over 80 as high.



Mooney viscometer.

Photo: Flexys



Principle drawing of a Mooney viscometer.

A: Rotor  
B: Die  
C: Force sensor  
D: Calibration weights  
E: Motor and gearbox  
F: Bearing  
G: Heating element

Illustration: Flexys

## Extrusion plastometers

In an extrusion plastometer and a capillary rheometer, rubber is forced through an extruder nozzle, capillary, at a known pressure or with a constant speed for a specified time and the volume that is extruded is measured.

The method is somewhat similar to extrusion and injection moulding in the rubber manufacturing industry and is often used as a test of extrudability. A capillary rheometer is different from an extrusion plastometer because in a capillary rheometer the pressure is measured in the cylinder just before the die, the die often is longer and that it is easy to change the pressure on the rubber.

Extrusion plastometers have been used for a long time in the rubber industry, but to a limited extent and they have not been standardised. Capillary rheometers have been used quite a lot as research instruments this is due to their greater ability to carry out testing over a large range of shear rates. One of the greatest advantages of extrusion plastometers is also the high shear rate that can be obtained, over 100 s<sup>-1</sup>.

High shear rates can lead to over heating of the material, but this is not necessarily so if the capillary tube is short. The time taken for the rubber to pass through the opening is then very short. On the other hand, it is too short to break down the rubber's thixotropic properties.

The Mooney viscometer provides better degradation of the structure, for example.

A simple extrusion plastometer functions so that rubber is pressed out through the extruder nozzle by a plunger under a constant load. The flow rate and the apparent viscosity in Poise are obtained as a result.

A more advanced extrusion plastometer or capillary rheometer, as it should be known, functions so that rubber is pressed at a constant speed through an extruder nozzle and the pressure at the entrance to the nozzle is measured using a pressure sensor. This allows the real shear viscosity to be calculated. Some capillary rheometers can automatically test the viscosity at a series of shear rates in the same test. The result is presented as a graph of the viscosity as a function of the shear rate.

Some instruments measure the dimensions of the extruded string using a laser device. In other instruments, measuring in both short and long capillary cylinders can be taken and thus the elongation viscosity can be calculated. Both these measurements provide information about the material's elasticity, which is the property, which is most often the cause of problems in processing.

# Testing of uncured rubber

## Compression plastometers

The principle used in a compression plastometer is very simple – the testing piece is pressed together between two parallel plates using a constant load and the compressed thickness is measured. This simplicity explains the previous widespread use of the method within the rubber industry.

Apart from the simplicity, the method has few advantages. It does however have a whole series of disadvantages:

- a) The shear rate is low, between  $0.1$  to  $1 \text{ s}^{-1}$ .
- b) The rubber is not sufficiently deformed in order to break down the thixotropic structure this applies especially to materials filled with carbon black
- c) The shear rate is not uniform in the test piece and in addition it also changes during the testing.

The disadvantage of the low shear rate may also be regarded as an advantage, since in recent years it has been found that the difference in viscosity between various materials is greatest at low shear rates.

## Scorch – Cure

During the curing of rubber, crosslinks are formed via chemical reactions between the molecules in the compound, which is then transformed from a plastic formable compound to an elastic material. With the increase in the number of crosslinks, the tendency of the material to flow and creep decreases. The hardness and tensile strength of the material increases, swelling in liquids decreases and the elasticity increases etc.

The curing process can thus be followed by studying how one of these properties changes by testing a number of test pieces cured at different curing times. Today, however, there are methods with which you can continuously follow the curing process using both considerably faster and simpler means. Using these methods, one or several of the following properties can be measured:

1. Scorching, that is to say the material's tendency to start to cure. Short scorching time lead to the material starting to cure when being processed, which ruins it and makes it impossible to form. In extrusion, scorching can be seen as "burnt pieces", which give the extruded string an uneven, pimple-like surface.  $t_s(1)$  is a measure on the scorch time.
2. The curing rate, that is to say how fast the material is completely cured after vulcanisation has started. This is an important factor for productivity in the production curing process. The values  $t_c(50)$  och  $t_c(90)$  are measures on the curing rate.
3. Viscosity before the vulcanisation reaction starts.  $M_L$  is a measure of the viscosity.
4. Stiffness, the maximum stiffness after the curing process is finished, is also a measurement of the crosslink density.  $M_H$  is a measure of the maximal stiffness.
5. Reversion, that is to say the decrease in stiffness in cases of overcure. This can be attributed to the degradation of the rubber material at the high curing temperature. The value  $R_p$  is the reversion of one unit, and is a measure of the reversion.



Parallell plate plastometer.

Photo: Elastocon



Capillary rheometer.

Photo: CEAST

## Measurement of the curing process in instruments with oscillating movements

ISO 6502

### Curemeter with rotor, ISO 3417

The construction and design of this instrument is similar to the Mooney viscometer with certain differences in the dimensions of the dies and the design of the rotor. The rotor is formed so that it is thicker in the centre and tapers off towards the extremities so as to obtain the same shear stress throughout the whole of the test material. The rotor oscillates sinusoidally at 1,7 Hz and normally with an amplitude of  $\pm 1^\circ$ . A torque sensor attached to the torque arm of the rotor registers the torque required to oscillate the rotor.

When carrying out batch control, a temperature of around 190 °C is often chosen, in order to obtain a short testing time. When development testing is being done however, a lower temperature is chosen, 150 to 170 °C, in order to obtain a better resolution. From the curves obtained, the previously mentioned properties, scorch time, cure rate, viscosity, elasticity and reversion can be determined.

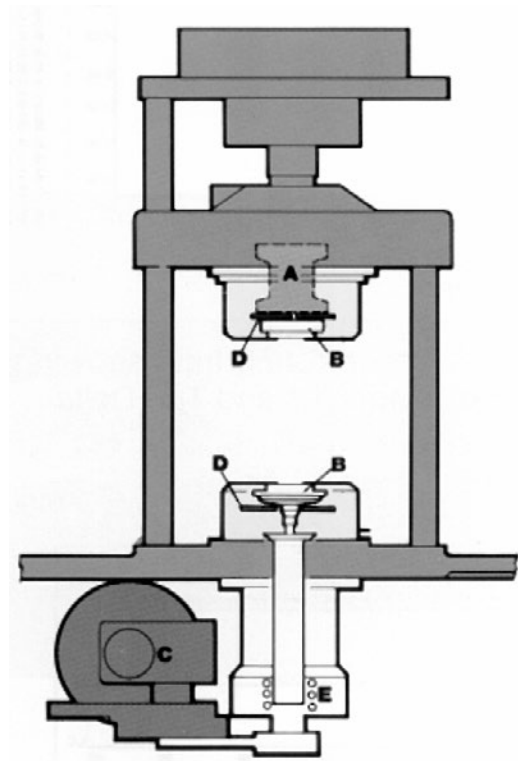
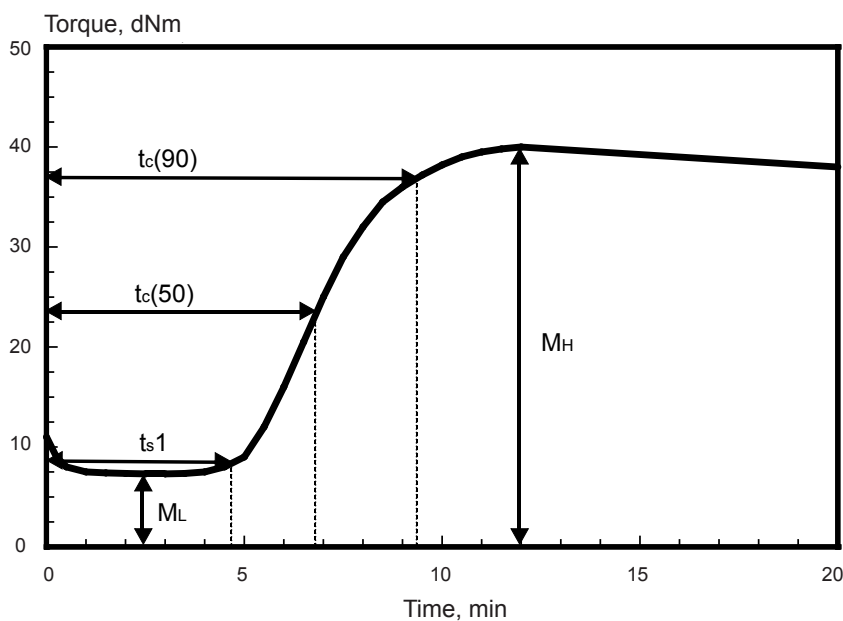


Diagram of a curemeter.

A: Torque sensor  
B: Die  
C: Motor and gearbox  
D: Heating element  
E: Bearing

Illustration: Flexys



Parallel plate plastometer.

Photo: Elastocon

# Testing of uncured rubber

## Rotorless Curemeter

In the rotorless curemeter, the lower half of the die rotates and the torque is measured by a torque sensor. The amplitude is normally lower than that of the rotor type, namely  $\pm 0,2^\circ$ . In addition to the torque, which is a measurement of the rigidity, the damping and the crosslink rate can be registered.

A rotorless curemeter has the advantage that it allows changes of the test specimens to be made quickly, since you do not need to peel off the cured sample from any rotor.

On the other hand, however, it must be loaded with test pieces with an exact volume, in order to avoid too much flash to get between the die halves. This will influence the result of the measurement. In order to obtain the exact volume, a special volume punch is used.

Curemeters without rotors have in recent years more and more replaced curemeters with rotors, since they among other things provide a higher testing capacity.

In later years a more advanced type of curemeters have been introduced, where the angle, temperature and frequency can be changed during the test. This type of instrument is called Dynamic Mechanical Rheological Testers (DMRT) or Rubber Process Analyzer (RPA).



*Curemeters for measuring curing properties. Rheometer and curemeter are names of instruments for determination of flow and curing properties of rubber compounds.*

*Photo: MonTech*

# Mechanical testing

## Important factors

### Climates ISO 23529

Polymer materials are sensitive to temperatures close to normal ambient temperatures in a completely different way than what metals are. Careful control of the temperature is therefore necessary when carrying out tests.

Many polymer materials, e.g. cellular material, are sensitive to humidity and particularly the relative humidity of air. It is therefore necessary to control this factor.

It is not sufficient, however, to perform tests in a well-defined climate. The material must be in a state of equilibrium with the surrounding climate. This is obtained by conditioning, which means that the test material is stored in the test climate conditions for a sufficient period of time so that it acquires the same conditions as the surroundings. The normal standard climate for conditioning and testing of plastic and rubber is  $23 \pm 2^\circ\text{C}$  with a relative humidity (RH) of  $50 \pm 5\%$ . Other climatic conditions are applied in special contexts. When the requirements of climate control are stricter the tolerances are reduced to  $\pm 1^\circ\text{C}$  and  $\pm 2\%$  RH.

## History

Polymer materials adapt slowly to changes, sometimes so slowly that “the memory” of the previous state remains in the material. In particular, this applies to the memory of the manufacturing process from raw material to finished product. This must be borne in mind when interpreting test results.

## Deformation rate

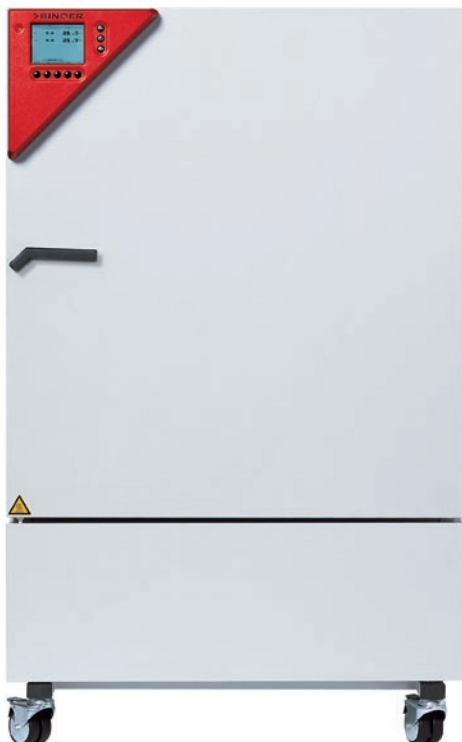
The resistance of a polymer material to an imposed deformation often greatly depends on the deformation rate. If the deformation is very slow, the material can flow like a liquid, almost without resistance. If the deformation is very fast, many materials react elastically, that is to say the reactive forces are proportional to the size of the deformation.

## Test preparation

The greatest risks in preparation of test pieces are that defects may occur due to the handling. In that case the test results will mirror the preparation rather than the object tested. Experience, through knowledge of the materials and good engineering common sense are the elements that can prevent this.

## Material data or product data

The crucial question when you choose the type of procedure for preparation and testing is if you want the results to reflect data from the material or data about the product itself. In the first case, the aim is to delete the memory of the material's previous processing history, to obtain a condition equal to the standard climate and select a deformation rate, type of test piece and other prerequisites according to the material standards. In the latter case, the conditions under which the product is to be used must be known and then simulated to the fullest possible extent.



*Climate chamber for conditioning of test pieces.*

*Photo: Binder*



# Mechanical testing

## Static or dynamic testing

Nearly all tests nowadays are static tests, that is to say you pull, press or shear using a constant speed in one direction, often until the test sample breaks or yields in some way or another. Most products are subjected to dynamic stress in their everyday use. There could be regular or random variations in the deformation rate and direction, often linked to other changes in the environment in which they are used. In principle, a product should always be tested dynamically if it is to be subjected to dynamic stress in its normal use.

## Testing methods

The prerequisite in order to be able to determine the properties of rubber material is to have appropriate testing methods. Comprehensive international cooperation in standardisation has provided us today with a number of generally accepted methods; we will deal with the most important of these.

## Density ISO 2781

The most common method of determining the density of a rubber material is to weigh a test piece in air and water. Weighing in air provides the actual weight and weighing in water provides the volume. The density is then calculated by dividing the weight by the volume.



Shore handheld tester. Photo: Bareiss



Thickness gauge.

Photo: Elastocon

## Dimensions ISO 23529

When measuring the dimensions of rubber test samples and products, most normal measuring tools can be used, e.g. measuring tapes and rulers, vernier callipers, thickness gauges and profile projectors. Since rubber is a soft material, it is important not to deform the rubber when measuring it.

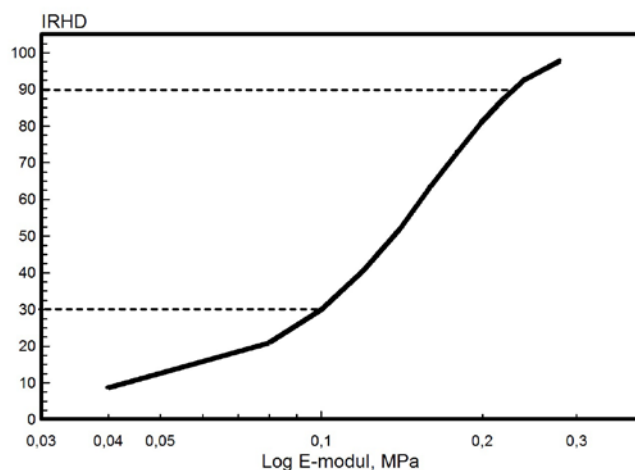
In order to accurately measure the thickness, a thickness gauge having a specified measuring load of 22 kPa should be used. The diameter of the measuring foot can be from 2 mm up to 10 mm and the load can be adjusted to reach the specified pressure.

Another good way of measuring the cross section of rubber profiles is to use a profile projector, which allows remote, measuring without having to touch the profile itself.

## Hardness

The rubber's hardness or stiffness (modulus) is determined by measuring how far a blunt measuring probe can be pressed into the rubber.

Originally there were several methods for hardness measuring, today however there are mainly two methods used, and a third for measuring hardness on big rubber covered rolls.



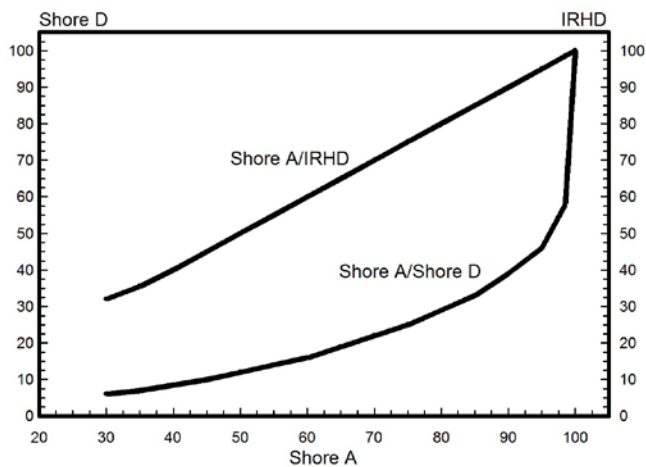
Hardness as a function of the E-modulus.

## Shore ISO 48-4

The oldest method for measuring hardness is the Shore method from 1915, where a truncated cone is pressed into the rubber with a spring force and the reading is carried out after 3 seconds. Earlier the reading time was expressed “within 1 second”, but changed in ISO 7619 to 3 s for better accuracy. The Shore method has a number of scales (A, B, C, D, AO, DO, O, OO and AM). Among these the most common are specified in the ISO standard. Shore A is used for normal rubber hardness, Shore D for hard rubber and Shore AO for soft rubber. Shore AM is a Shore A micro method for thin test pieces.

All measuring scales have the end positions 0 and 100, where 0 is infinitely soft and 100 infinitely hard. The difference between the scales consists of different probes and spring forces. See the table. The Shore meters can be used either as handheld meters or as stand mounted. Both analogue and digital meters are available.

The measuring accuracy is noticeably improved when the meter is stand mounted, as the meter is parallel with the testpiece and the correct load is used. If a timer is used, the precision is further improved.



Comparison between IRHD, Shore A and Shore D.

## IRHD ISO 48-2

The IRHD method was developed within ISO in order to improve the accuracy in measuring hardness. This method is called “International Rubber Hardness Degrees” or IRHD. The scale for IRHD equipment corresponds to Shore A. What differentiates the methods is that the IRHD equipment is always mounted on a stand, the loading is done with a deadweight the measuring probe is ballshaped and the reading is taken after 30 seconds.



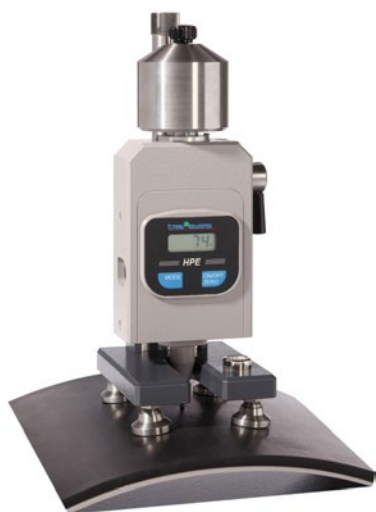
Digital automatic hardness meter with exchangeable measuring probes for Shore A, D, AM IRHD-N and IRHD-M.  
Photo: Bareiss

	Shore A	Shore D	Shore AO	Shore AM	IRHD-N	IRHD-M
<b>Measuring probe, type</b>	truncated cone	cone	ball	cone	ball	ball
<b>Measuring probe, size</b>	diam 1,25 mm	diam 1,25 mm	diam 5 mm	diam 0,79 mm	diam 2,5 mm	diam 0,395 mm
<b>Load, type</b>	spring	spring	spring	spring	weight	weight
<b>Load, probe</b>	822 g	4 550 g	8,05 N	0,764 N	5,4 N	145 mN
<b>Load, measuring foot</b>	1 000 g	5 000 g	1 000 g	250 g	8,3 N	235 mN
<b>Measuring time, rubber</b>	3 s	3 s	3 s	3 s	30 s	30 s
<b>Measuring time, TPE and plastic</b>	15 s	15 s	15 s	15 s	30 s	30 s
<b>Thickness of testpiece, mm</b>	>6 mm	>6 mm	>6 mm	>1.5 mm	>8 mm	>1.5 mm

# Mechanical testing

## Pusey & Jones ISO 48-8

Pusey & Jones hardness meters are mainly used to measure the hardness of larger rubber covered rolls, since the device can be used on curved surfaces. The device consists of a tripod stand, which is placed on the surface that is to be measured. A 3,175 mm ball is pressed with a constant weight of 1 000 g into the rubber surface and the result is given in 1/100 mm.



*Pusey & Jones hardness meter. Photo: Bareiss*

## Tensile test ISO 37

Tensile testing is normally used to determine the following properties of rubber materials:

- Stress at a particular elongation, e.g. 100 % or 300 %, expressed in MPa, is sometimes called the “rubber modulus”.
- Tensile strength in MPa, which is the strength at break.
- Elongation at break in %.

The testing is normally conducted on dumbbell test pieces, which are punched out of 2 mm test sheets. The test is done in a tensile testing machine using a speed of 500 mm/min. To be able to determine the elongation and the stress at different elongation, an extensometer is also needed. The extensometer can be mechanical with balanced clamps that measure the elongation or optical e.g. a laser that measures two reflecting benchmarks on the test piece.



*Computer controlled universal tester. Photo: Tinius Olsen*

## Compression tests ISO 7743

Compression tests to measure the rubber's stiffness, spring constant or modulus, can be done in modern tensile testers, which can be used in both tension and compression. The normal method is to deform the rubber by 25 % and measure the force. It is common to do a mechanical condition by compressing the rubber three times and carrying out the measurement on the fourth compression. The result can be displayed as a load/deformation curve, from which you can read off the force at e.g. 10 and 20 % deformation.

## Tear test ISO 34

In a tear test the material's tear resistance is determined. Three methods are common, namely:

### Trouser test piece (method A)

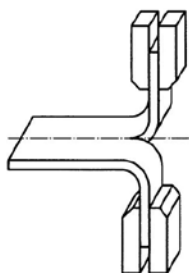
A trouser test piece consists of a rectangular strip 15 x 75 mm with a notch 25 mm in from one short side. The strip is elongated in a tensile tester with 100 mm/min, so that the notch gets bigger. The results are produced as a tear diagram with a number of tops and the median top is given as the result.

### Angle test piece (method B)

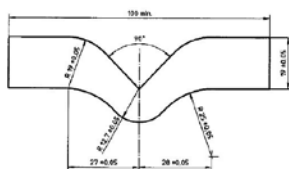
A 90° angle test piece is elongated using a speed of 500 mm/min to break. The test can be performed with a 1 mm nick or without a nick. The test without the nick measures a combination of the force of tear initiation and propagation.

### Crescent test piece (method C)

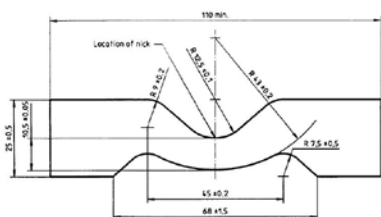
A crescent shaped test piece with a 1 mm deep nick is elongated using a speed of 500 mm/min to break. The test can be considered to be a tensile test with an indication of fracture. The result is given in kN/m (N/mm) test piece thickness.



Tear test with trouser test piece.



Tear test piece, angle type.



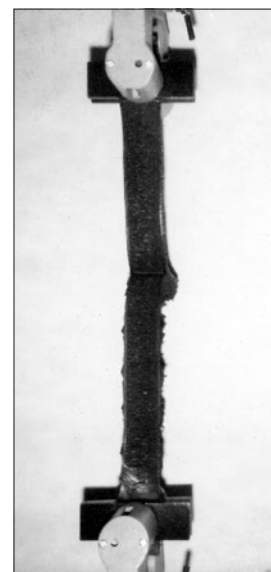
Tear test piece, crescent type.

## Adhesion tests

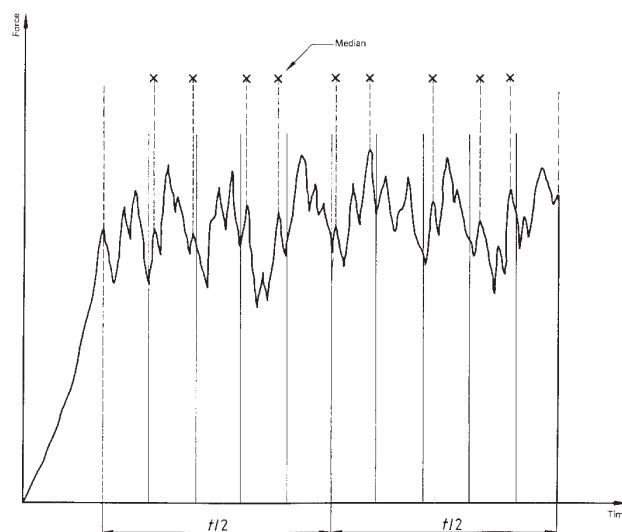
### ISO 36, ISO 813, ISO 814, ISO 1827

Many rubber products have different reinforcement materials inserted, primarily metal and textile. The function of the products often depends on a good adhesion between the rubber and reinforcement materials. There are several methods for testing adhesion, tensioning, shearing or peeling. In certain cases, special test pieces are produced, in other cases test pieces are taken from the products, such as tyres, conveyor belts etc.

The most interesting aspect of adhesion testing is to see where the break occurs. If the break is in the rubber this is considered to be good, and poor if the break is at the surface of the reinforcement material since in that case the bonding system is the weak link. In peeling tests, a curve is obtained similar to the one with trouser tear tests and it is evaluated in the same way, but the result is given in N/mm test width.



Adhesion testing of car tyres.  
Photo: Göran Spetz

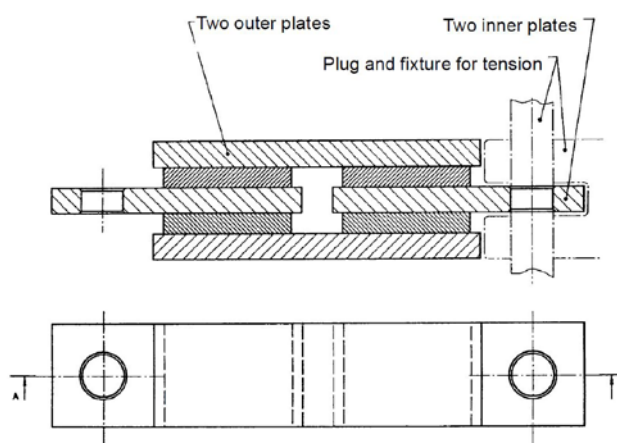


Evaluation of tear test and adhesion test ISO 6133.

# Mechanical testing

## Shear Modulus ISO 1827

Shear modulus is often an important property when calculating rubber products. The most common way to determine the value is to do measurements on a so called quadruple shear test piece. The quadruple shear test piece consists of four rubber pieces 4 mm thick, 20 mm broad and 25 mm long, glued together or cured between two plates as in the figure. After mechanical conditioning the test piece is elongated to 30 % deformation with a speed of 5 mm/minute. The shear modulus is calculated at a deformation of 25 %.



Tear test piece, crescent type.

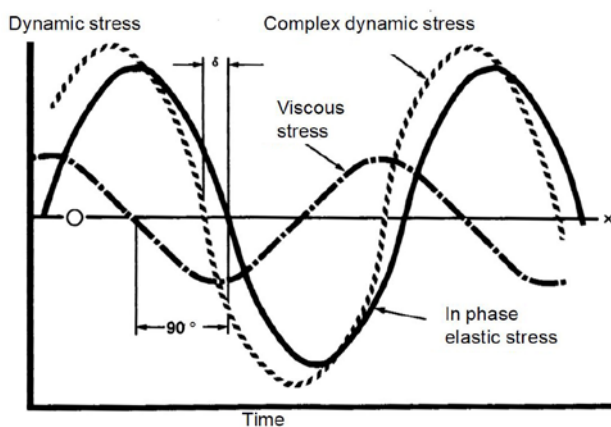
## Dynamic testing ISO 4664

Many rubber products are used under dynamic conditions and for that reason it is interesting to be able to measure dynamic properties. The properties often measured are modulus and damping as a function of deformation or frequency. Material testing is mostly done in small instruments under compression, tension, bending or shearing and is called Dynamic Mechanical Analysis, DMA. The instrument contains a device for vibrating the test-pieces from 0 to some twenty or thirty Hz and one load-cell for measuring the force. The test piece is enclosed in a temperature chamber where it can be both cooled and heated between about -70 and +200 °C. Thereby a temperature sweep can be done, where the modulus is measured as function of the temperature (see further Thermal tests).

The dynamic properties of products are tested in servo hydraulic universal testing equipment, available in sizes with loads up to several 100 kN. The machines have a hydraulically driven piston, which can vibrate the product under test up to about 100 Hz and a loadcell measuring the force.

As rubber is a viscoelastic material modulus and damping vary with frequency, deformation and temperature.

By dynamic testing the so-called complex dynamic modulus and the loss angle are determined. The damping is expressed as tangens for the loss angle ( $\delta$ ). Damping is a measure of the energy loss and of heat generation in the test piece.



Tear test piece, crescent type.



## Rebound resilience ISO 4662

Rebound resilience is a basic property of rubber and it is also one of the oldest and simplest tests. The testing is normally conducted as a rebound test, in which a weight is bounced against the rubber, either in the form of a free falling ball or a pendulum. By comparing the height of the rebound with the original height you get a measurement of the energy that has been absorbed by the damping effect of the rubber. The results are normally given as the rebound height in percentages of the fall height.

The two most common devices are the Lüpke and Schob pendulums.



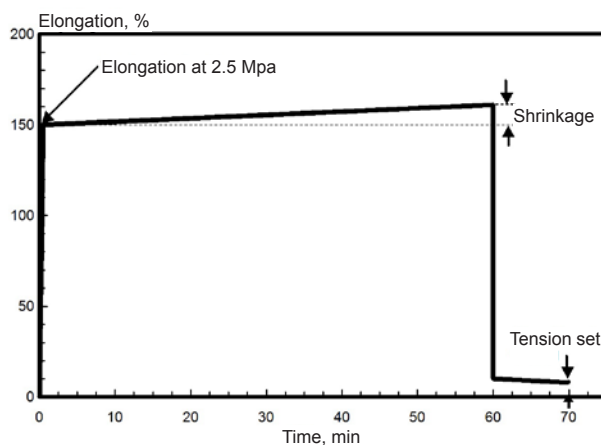
Schob pendulum.

Photo: Bareiss

## Tension set at constant load ISO 2285

If a rubber test piece is stretched for a time and then unloaded, it does not completely retract to its initial length. The difference is called tension set.

In this test, rubber test specimens are loaded with weights using a load of 2,5 MPa at room temperature. After an hour the samples are unloaded and the tension set is determined after a 10 minutes recovery period. Often the elongation for 2,5 MPa load is determined after 30 s and the creep after being loaded for 1 hour.



Tension set.

# Thermal testing

## Thermal testing

Temperature influences the properties of polymer materials much more than is the case with metals and ceramics. This is not only seen as changes in the actual properties when the temperature changes but also that critical temperatures are reached relatively early.

## Glass transition temperature

One of the critical temperatures is the glass transition temperature ( $T_g$ ). This is the temperature where an amorphous polymer is transformed from a hard and rigid state (glass) to a rubber-like state. The glass transition is found in pure form only in completely amorphous polymers. The transition occurs within a temperature interval of 20-30 °C, where  $T_g$  normally marks the middle of the interval.

For obvious reasons, the  $T_g$  defines the upper temperature limit for amorphous thermoplastics.

In polymers with partially crystalline structures, the glass transition strength decreases with increasing degree of crystallinity, since glass transition only occurs in the amorphous parts. This explains why certain polymers, e.g. polyethylene and polypropylene, whose  $T_g$  is far below room temperature, have such high stiffness. For partially crystalline plastics, the  $T_g$  is not therefore the upper temperature limit.

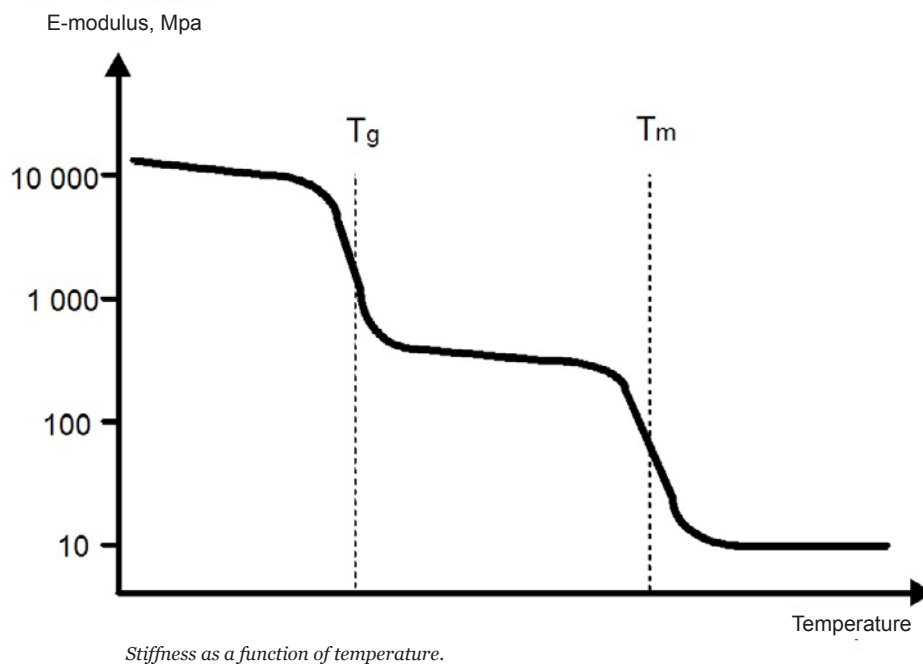
For normal thermoset plastics, the molecules are closely crosslinked, which are why mobility is limited and the effect of glass transition relatively modest.

A borderline case of crosslinked polymers is elastomers (rubber). In these materials, the degree of crosslinking is relatively low, which is why the glass transition occurs to an almost normal degree. The softness and elasticity that are obtained above the  $T_g$  is used in these materials. Rubber is thus a slightly crosslinked, melted polymer.

Plasticisers are the most effective way of lowering the  $T_g$ .

The  $T_g$  is not a fixed material constant but is partly affected by the heating and cooling rate. Slow temperature changes lower the  $T_g$ .

The brittle temperature and the extensibility partly depend on the  $T_g$  and are normally used as practical measurements of this. There are standardised test methods to determine, among other things, the brittle, stiffness temperature and elastic retraction, TR.



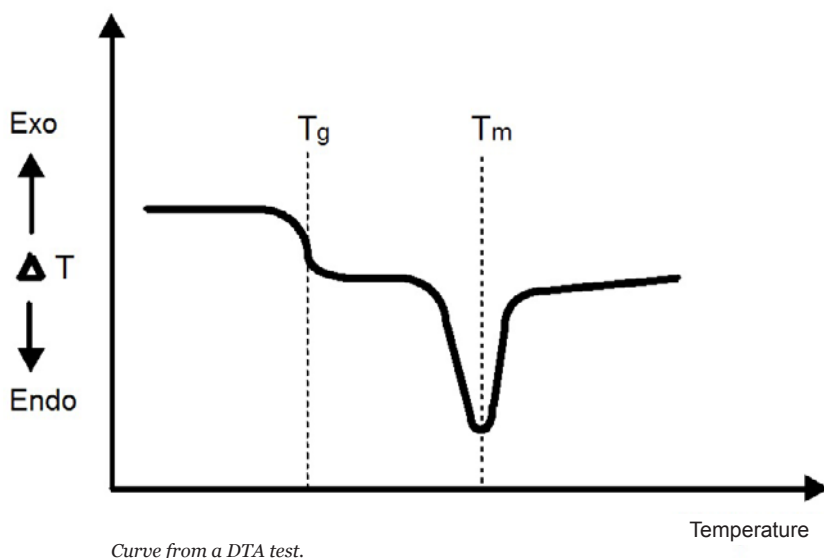
## Crystal melt temperature

The other critical temperature is the melt temperature ( $T_m$ ) that is the temperature at which the remaining crystalline parts of a partially crystalline polymer melt. The melting process in a polymer, in contrast to metals and low molecular substances (common salt, sugar etc.), covers a relatively wide temperature range (about ten degrees).

Since no plastic material is 100 % crystalline, each partially crystalline polymer will undergo both glass transition and melting. The  $T_g$  is then always lower than the  $T_m$ , which gives the upper limit of use for partially crystalline plastics.

Due to the crosslinking in rubber and thermoset plastics no melting of these occurs. When heated, if the temperature is sufficiently high, chemical degradation of the material takes place. The form of the melting interval depends on the molecular structure and molecule weights and the distribution of the sizes of the crystallites. Normally the smallest crystallites melt first and the larger last. Plasticisers have little effect on the  $T_m$  while fillers or reinforcements do not influence the  $T_m$  at all.

Diagram from Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC) as well as Dynamic Mechanical Analysis (DMA) can be used in the classification of melting intervals and phase transformations.



## Low temperature tests

There are several methods to determine the properties of a rubber material in low temperatures.

### TR tests ISO 2921

The TR test is a common method used and TR stands for "Temperature Retraction".

The testing is done so that the test pieces are elongated, normally by 50 %, and are then cooled after this down to  $-70^{\circ}\text{C}$  for 10 min. Most rubber materials then become completely rigid. The clamping of the test pieces is released and after this the samples are heated up by  $1^{\circ}\text{C}$  per minute. The temperatures at which the samples have regained 10, 30, 50 and 70 % of their original lengths are registered and are named  $\text{TR}_{10}$ ,  $\text{TR}_{30}$ ,  $\text{TR}_{50}$  and  $\text{TR}_{70}$ .



Automatic TR tester.

Photo: Elastocon

# Thermal testing

## Brittleness temperature ISO 812

Testing of the brittleness temperature is a method where a hammer is used to pound the test pieces after these have been cooled down in a cooling bath. The brittleness point is the temperature where the samples just not brake when struck with the hammer.

## Gehman test ISO 1432

When Gehman testing it is common to determine the relative torsional stiffness at 23 °C and lower temperatures. If the dimensions of the test piece are known, the torsional modulus can be calculated too.

When testing a test piece with the square section 2 x 3 mm is mounted in series with a calibrated wire made out of spring steel. The torsional stiffness is measured by turning both the rubber and the wire 180°. That turn is distributed between the rubber test piece and the wire in proportions to their torsional stiffness, and the turning angel of the test piece is determined.

First the torsional stiffness is determined at 23 °C and after that the mounted test pieces are placed in a cooling bath, with a temperature where the rubber is at least 100 times stiffer than it is in room temperature. The temperature in the bath is then heated up by 1 °C per minute, and the temperature where the rubber is 100, 10, 5 and 2 times stiffer than in room temperature is determined. These temperatures are called  $RM_{2}$ ,  $RM_{5}$ ,  $RM_{10}$  and  $RM_{100}$ .



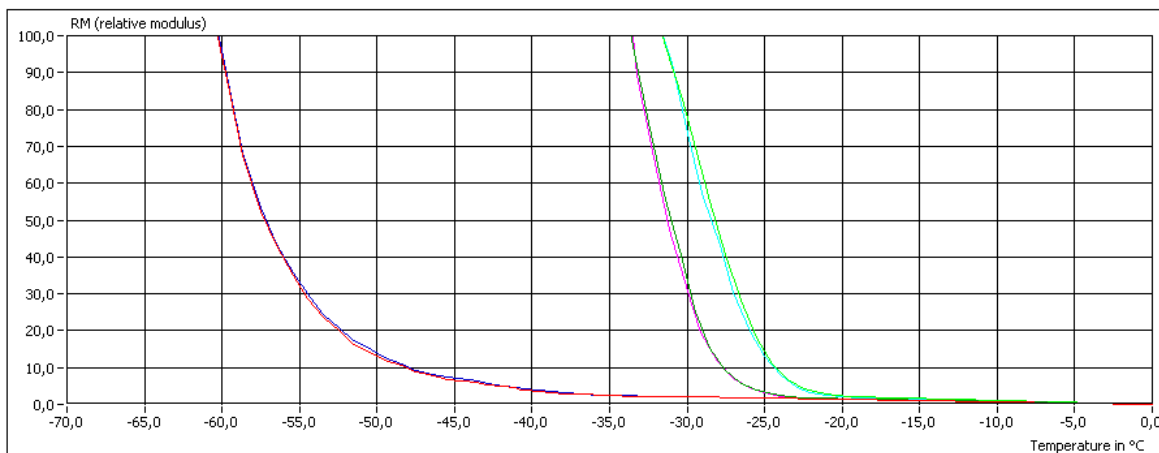
Brittleness temperature tester.

Photo: Elastocon



Automatic Gehman tester cobined with TR tester.

Photo: Elastocon



Freezing for Windows

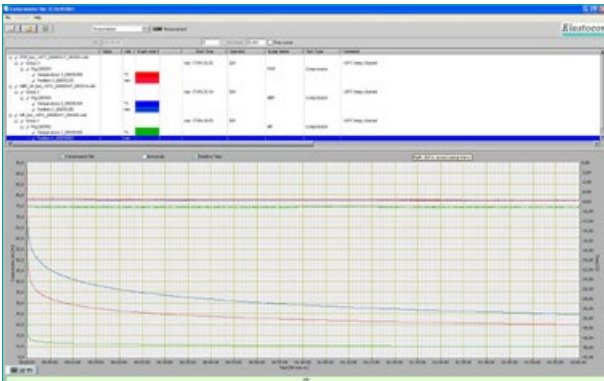
Stiffness curves from Gehman test.

## Compression set at low temperature ISO 815-2

In order to determine the rubber's elasticity at low test temperature, a compression set test can be carried out at low temperatures. Both measuring and recovering are carried out at the low test temperature.

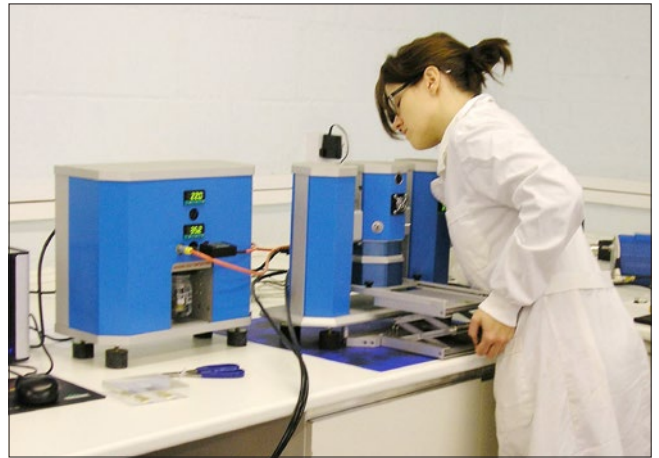


Semiautomatic test of Low Temperature Compression Set, LTCS.  
Photo: Elastocon



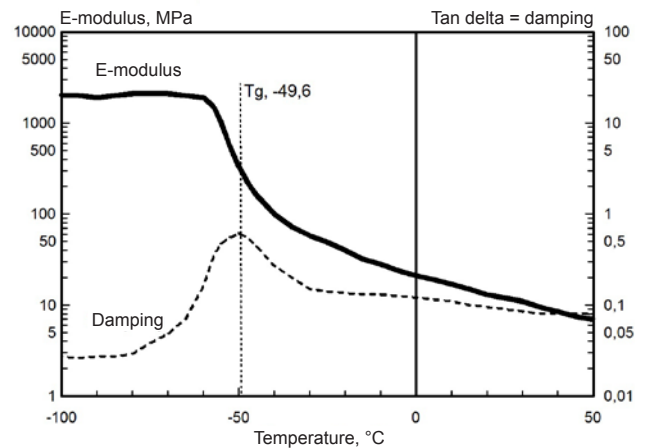
## Dynamic mechanical analysis, DMA ISO 4664

If you wish to determine the modulus as a function of the temperature, modern DMA equipment can be used for this. In DMA tests, the viscous and elastic modulus are measured together with damping for a particular temperature interval. The testing is done dynamically either in bending, tension, compression or shear. This test can replace most other cold tests the only disadvantage is the high price for the equipment.



Instrument for DMA analysis.

Photo: Triton



DMA curve for EPDM rubber.



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# Durability testing

## Durability testing

The durability of rubber materials is affected by a number of environmental factors in combination with the mechanical stresses that are caused by the use of the product itself.

## Influencing factors

It was well known from an early stage in the development of rubber materials that factors like heat, sunlight, oxygen in air and humidity in general accelerate the degradation of rubber. Mechanical loads, erosion, impurities, microorganisms and other special influences occur depending on the application of the rubber.

## Accelerated ageing

Normally there is no time to wait for a test under real conditions. It could in actual fact take decades to get the natural results. Accelerated ageing is therefore used. This means the factors that cause natural ageing are reinforced. This could take place both outdoors – in a desert or tropical rain forest – and indoors in ovens, climate chambers or weather simulators. Unfortunately this is often done with no proper critical analysis. The ageing process is accelerated far too much. The material is literally grilled. The accelerating ageing process then becomes completely different from the natural process. The result is incorrect predictions of the actual durability.

## The philosophy of ageing processes

The functional environment must first of all be carefully analysed, so that the most important degradation factors in each application are identified. Using the available knowledge, it is then determined how far the acceleration can be taken. The available knowledge and facts are seldom sufficient in order to determine the maximum permissible acceleration or to translate the results into an exact number of years under real conditions. The acceleration has therefore to be carried out in moderation and using rules of thumb.

If durability testing is to be carried out seriously, long testing times – a year is not uncommon – must be expected. It is surely always better to wait a long time for a more correct result than to get an incorrect one quickly.

## What to remember about the ageing process

In all ageing processes it is especially important to keep a constant temperature and in certain cases a constant relative humidity in air. This is because the speed of a chemical reaction is roughly doubled for a temperature increase of 10 °C – and ageing is in most cases a chemical reaction. Normally, the highest deviation of  $\pm 1$  °C and  $\pm 5$  % RH is allowed. In all ageing, and especially for long testing times (up to a year and more can occur), it is extremely important to be sure that the temperature has been maintained within the permitted tolerance during the whole testing procedure.

Another important factor is the flow of air. During the ageing process, the oxygen in the air is used up and degradation products are formed. In order to make the testing reproducible, the oxygen concentration must be kept at a constant level, and the degradation products ventilated off. In order to meet these requirements, the air must be changed between 3 and 10 times per hour.

The device must therefore be equipped with an air supply and flow meters. The air speed must also be low otherwise the oxidation rate can increase and plasticisers and antioxidants be ventilated off.

## Ageing

When rubber material ages with time, this normally manifests it in increased stiffness and reduced extensibility. Easily oxidised materials, as for example natural rubber, become softer for longer ageing times.

When a rubber material ages, among other things, the following reactions take place:

- a) Oxidative degradation, caused by oxygen, which creates breaks in the polymer chain.
- b) Thermal degradation, caused by thermal movements in the polymer chains, which cause breaks in the polymer chain.
- c) Additional cross-linking caused by the remains of curing agents. In curing systems with high sulphur content, polysulphide and disulphide links can break up and form new crosslinks of the di- and mono-sulphide type.

The changes in a rubber material when ageing can be examined by testing for several properties. The most common way to test the effect of ageing on a rubber material is to do a tensile test and measure the change in hardness. The total ageing effects are most apparent in the decrease in elongation at break. The additional crosslinking is most apparent in the increase in hardness and increase in tensile strength.

For testing tension set, the oxidative reaction is normally dominant, caused by the thin cross section of the test piece, which allows oxygen to penetrate.



Cell ageing oven.

Photo: Elastocon

For compression set testing, thermal degradation can be dominant, since the test pieces are relatively thick and protected on two sides by the compression plates, which leave only a small surface area exposed to the air.

For compression set testing, additional crosslinking is a main reaction during the first hours and days, while degradation reactions dominate in longterm tests. A 24 hours compression set test is therefore often used as a control test of the degree of vulcanisation of the rubber.

In relaxation testing, additional crosslinking does not influence the result, since the newly formed crosslinks do not contribute to any change in the counter forces measured.

This means that a relaxation test in elongation reveals both oxidative and thermal degradation, while a relaxation test for compression shows mainly thermal degradation. If a relaxation test is carried out in an inert atmosphere, such as nitrogen, it is possible to separate the thermal degradation from the oxidative. Stress relaxation is normally divided into two types of relaxation, physical and chemical relaxation. Physical relaxation takes place mainly during the first minutes and hours of a relaxation test and are caused by the relocation of filler particles, in which these together with the chains of polymers find new states of rest. Chemical stress relaxation consists mainly of breaks in the polymer chain and is caused by oxidative and thermal degradation.

Ageing effects increase at increasing temperature and as a rule, a temperature rise of 10 °C decreases the life cycle by half.

The service life of rubber materials is affected not only by chemical reactions but also by mechanical influences, such as abrasion and dynamic fatigue.

# Durability testing

## Heat ageing ISO188

Heat ageing is carried out in cell ovens or special ageing cabinets with constant temperature, low air speed and an air change of 3–10 times per hour. Normally you measure the change in hardness, tensile strength and elongation at break and tear resistance, by storing test pieces at a higher temperature. The results are given as a percentage of the change. The test time is often one or several weeks. 1 000 hours (6 weeks) samples are common in the material specifications for the car industry.

## Tension set at constant elongation

### ISO 2285

If a rubber sample is kept elongated for a period of time and is then unloaded, it will not retract completely to its initial length. This is called tension set.

For constant elongation testing, the test pieces are elongated, normally to 100 % and are kept elongated, often at increased temperature, from 24 hours to several weeks. When the samples have been taken out of the oven they are left to cool in the elongated state after which they are unloaded and allowed to recover for 30 minutes before measuring.

## Compression set ISO 815-1

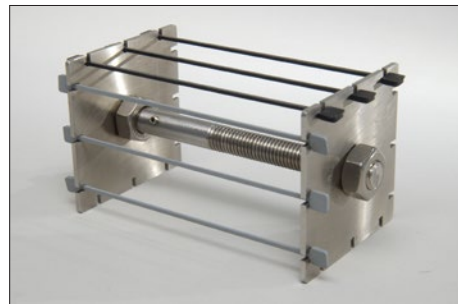
Compression set is the permanent set that occurs when rubber has been compressed and then unloaded and allowed to recover.

The testing is normally done on standardised cylindrical test pieces. The test pieces are normally compressed by 25 % in a rig with smooth polished surfaces. The compression set rig, with test samples, is stored in an ageing cabinet at increased temperature, for periods from 24 hours up to several weeks. The rig is taken from the cabinet and the samples are unloaded and are allowed to recover for 30 minutes before testing. Sometimes an alternative method is used where the samples are allowed to cool in the compressed state. This normally gives higher compression set values.



*Rig for compression set testing.*

*Photo: Elastocon*



*Rig for tension set.*

*Photo: Elastocon*

## Relaxation ISO 3384-1, ISO 6914

Relaxation is the decrease in counter pressure, which can be measured in a rubber test piece exposed to constant deformation. The deformation method can either be compression or tension.

Relaxation measuring can be done in two different ways. Either the measurements of the original force and the force after different times can be taken at the test temperature, or all measurements are taken at room temperature, while the samples are stored at increased temperature in between these measurements. In certain cases, relaxation measuring is also done in liquids e.g. oil. This is common for e.g. O-rings.

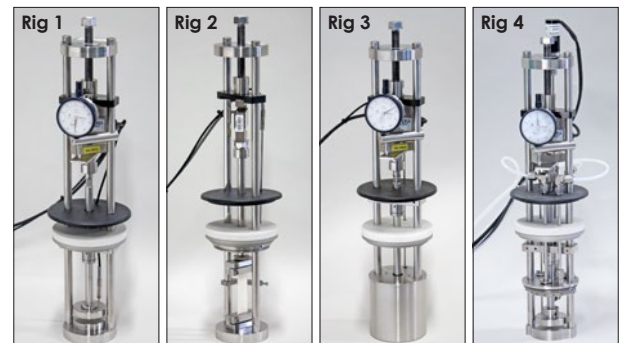


Relaxation testing equipment.

Photo: Elastocon

## Cycling relaxation test ISO 3384-2

Some years ago the automotive industry started to specify stress relaxation tests with temperature cycling. The temperature is cycled between a high temperature for ageing, e.g. 125 °C, and a lower temperature, e.g. -40 °C, to check if there is still a sealing pressure. Rubber shrinks much more than steel, so a seal in a motor is functioning when the motor is running, but if the car is standing in a low temperature the seal may shrink so there is no sealing pressure left.



Relaxation rigs arranged for testing in compression, tension, liquid and ALE-test.

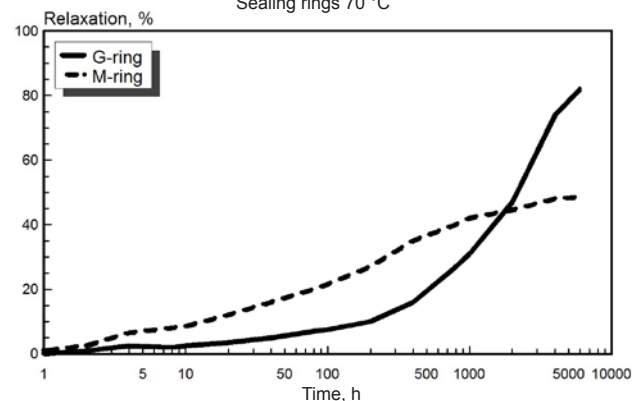
Images: Elastocon

## Estimation of service life ISO 11346

Often some form of the expected service life is required as the result of an ageing test. Many different attempts to predict the service life of a material have been made, often simply by extrapolating the short time test over a longer period. This procedure can lead to completely incorrect results, as shown in the diagram. Two different materials for sealing rings for waste water pipes are shown here, both of which have been tested for relaxation for one year.

If the testing had been interrupted after one week or one month and a linear extrapolation carried out, the result would have been completely incorrect, since one of the curves bends sharply after a time. The fact that a material's properties deteriorates at a faster rate after long periods is very common and is due to the fact that the antioxidants and stabilisers added have been consumed and the degradation process speeds up.

Relaxation test  
Sealing rings 70 °C



Comparative relaxation test for two materials for sealing rings.

Example of a cycling test

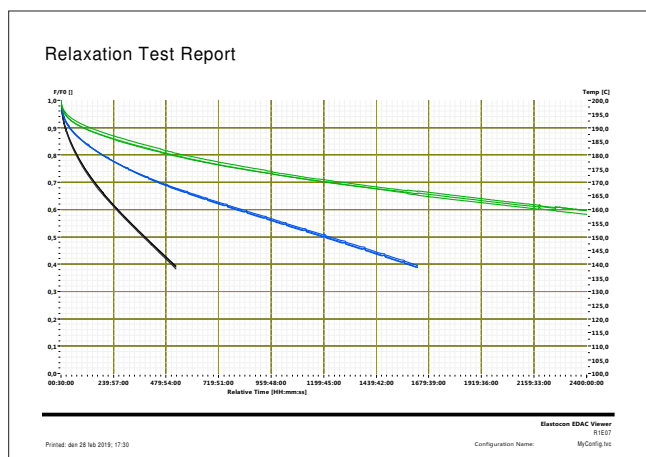


# Durability testing

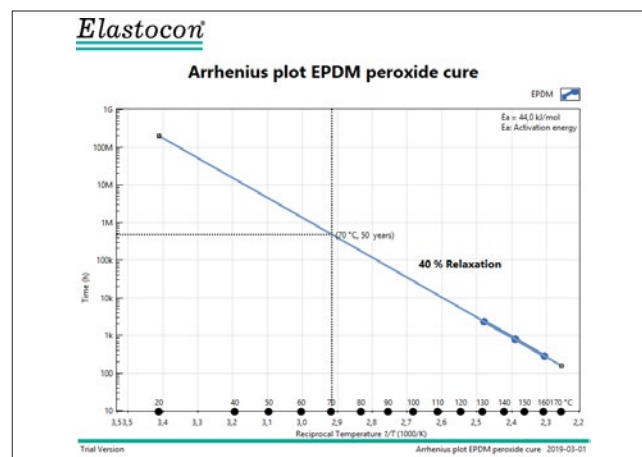
A more serious approach to estimate the life time for a material is to use an Arrhenius diagram. In order to be able to draw an Arrhenius diagram, you first need to determine the life time for a material for at least three temperatures. The properties used in order to determine the life time are often elongation at break, tensile strength, compression set and relaxation. The properties chosen and the level, at which the function of the material ceases, depend on what the material is to be used for. Often the time used is that which the particular property deteriorates to 50 % of the initial level. To be able to determine the time when the criteria is reached, you perform the testing using logarithmic time intervals, e.g. 1, 2, 4, 8, 16 and 32 etc. days. The testing times can become long, especially at the lowest temperature, up to a year is not uncommon.

When the life time from at least three temperatures have been obtained, these are entered on the Arrhenius diagram, with the Y-axis as Ln time and X-axis as temperature, expressed in  $1/T$ , where  $T$  is the temperature in degrees Kelvin. Then a straight line is drawn through the time/temperature points and which can then be extrapolated for the life length at lower temperatures.

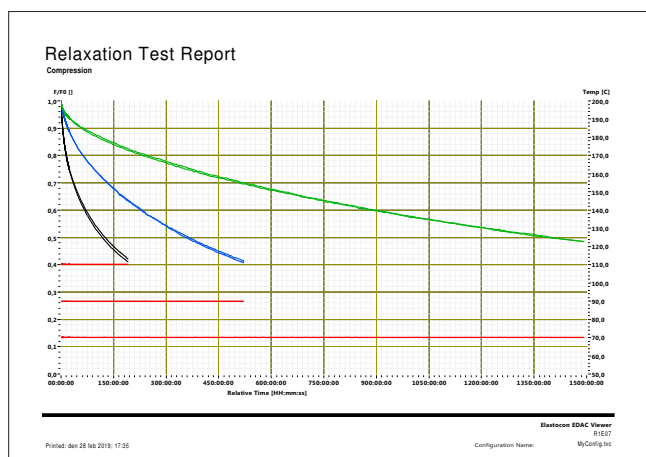
What may sometimes occur is that a straight line cannot be drawn between the points and which then shows that the ageing reaction has not been the same at all temperatures. Often it is the highest temperature that differs and which means that a lower temperature may need to be used. It should also be noted that the further you extrapolate from the lowest tested temperature, the larger the uncertainty becomes.



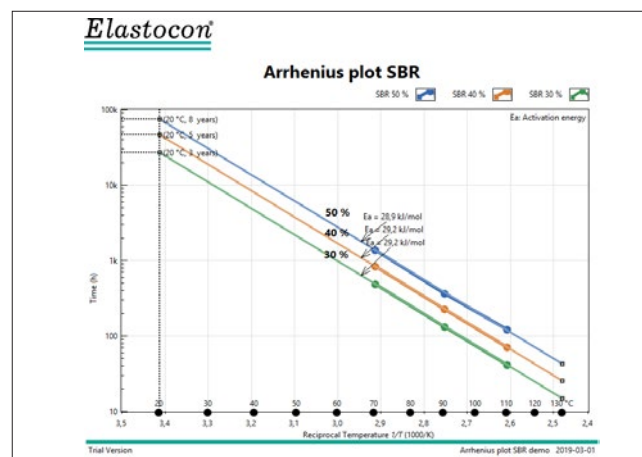
EPDM relaxation curves at three temperatures.



Arrhenius plot at 40 % relaxation.



SBR relaxation curves at three temperatures.



Arrhenius plot at 30 %, 40 % and 50 % relaxation.



## Resistance to liquids ISO 1817

When testing for resistance to liquids, the effects that various liquids have on rubber materials are determined.

The most common liquids are:

- Oils
- Fuels
- Water
- Chemicals

Oils, fuels and water penetrate the rubber and make it swell, without destroying the material. Fuels and certain oils can also dissolve plasticizers in the rubber, so that it decreases in weight when the fuel is dried off. Oxidising chemicals can break down the chains of molecules in the rubber.

The testing is carried out by immersing rubber samples with a volume of 1–3 cm<sup>3</sup> into the liquid. The testing is normally carried out at an increased temperature for 72 hours. In certain cases, the testing is carried out for longer periods, until a balance is reached, which can take several weeks.

The properties that are tested are normally changes in weight, volume, and hardness. Sometimes also the change in tensile strength is tested. The result is given as a percentage change.

## Weather ageing

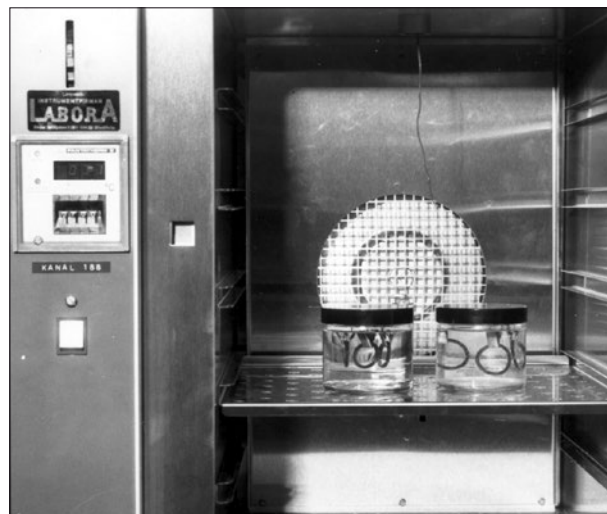
The factors, which influence rubber outdoors, in addition to oxygen and heat, are light, ozone and rain.

Sunlight, especially the short UV rays, causes degradation of the chains of polymers. This is not a problem if the rubber material is black because the light will then be absorbed and cannot penetrate into the material. In light coloured materials, titanium dioxide, a white pigment can be added. If you have sufficient titanium dioxide in the rubber the light will be reflected and cannot penetrate into the material. There is also special UV protection that can be added.

In cases where rubber products are manufactured in light colours, sunlight can cause changes in colour.

Ozone, which is an especially reactive form of oxygen, attacks rubber materials with unsaturated carbon chains, that is to say material with double bonds. The attack increases the more the material is elongated. The materials that are most sensitive to reaction with ozone are natural, styrene butadiene and nitrile rubbers. Butyl and chloroprene rubbers have better resistance, while EPDM, silicone and fluorocarbon rubbers are completely resistant.

Ozone is formed in UV light and by electrical discharging. Ozone is found in the atmosphere in small amounts, normally not above 10 parts per hundred million parts air (pphm). Normally the concentration lies between 0 and 5 pphm.



Swelling test of O-rings in fuel.

Photo: Göran Spetz



Weather simulating device.

Photo: Q-Lab

# Durability testing

Rain is normally not a problem for most types of rubber, except for some, which are sensitive to hydrolysis, which means that damp environments increase the degradation. The rubber type that is most sensitive to hydrolysis is polyurethane rubber, especially the ester types. Even silicone rubber is affected by hydrolysis, but only at high temperature in steam. Rain, in combination with sunlight, can however cause chalking on light rubber materials. Chalking arises when the rain washes away the outer layer of oxidised and degraded rubber polymers so that the light filler granules emerge. Weather testing can be conducted indoors or outdoors.

## Weather simulation ISO 4665

Weather simulation is testing that is carried out indoors and is conducted in special weather ageing cabinets. In a weather cabinet, sun, temperature and rain are simulated. The normal testing cycle has a light intensity of  $1\,000\text{ W/m}^2$ ,  $55^\circ\text{C}$  black panel temperature and a rain cycle of 18 minutes of rain and 102 minutes dry. In certain simpler devices only light is used.

The properties most often examined are colour changes and changes in tensile strength and elongation at break. The testing is moderately accelerated and a 6 weeks test corresponds to approximately 2–3 years outdoors.

## Outdoor ageing

Outdoor ageing of rubber is often carried out with elongated test pieces in order to form an opinion of the effect of ozone, in addition to changes in the appearance and tensile strength. The testing is carried on for at least a year and is often done as a comparison between different materials.



Ozone cabinet.

Photo: SATRA

## Ozone tests ISO 1431

Testing a rubber material's ozone resistance is done in special ozone chambers, normally at an ozone concentration of 50 pphm (parts per hundred million) and at a temperature of  $+40^\circ\text{C}$ . Since rubber is more easily attacked by ozone when it is elongated, the testing is carried out in a rig with varying degrees of elongation, from 5 to 80 %.

The samples are checked at certain time intervals, 2, 4, 8, 24, 48, 72 and 96 hours, and the time taken to form the first crack in each elongation is noted.

## Abrasion testing ISO 4649

Abrasive resistance is one of the rubber's most important properties, but it is at the same time difficult to test in the laboratory.

In laboratory tests, results must be obtained in a short time and therefore the testing is accelerated, but this means that the abrasion mechanism is not the same as in practice and the results do not correlate to what actually occurs in reality.



Abrasion tester.

Photo: Bareiss

There are normally four different abrasion mechanisms for rubber materials:

Type	Abrasion mechanism	Abrasion patterns	Example
<b>Pyrolysis wear</b>	Frictional heat breaks down the rubber in liquid and gaseous substances.	Sticky surface.	Braking tracks on asphalt.
<b>Abrasive wear</b>	Sharp particles tear parts of the rubber surface.	Scores in the longitudinal direction.	Sandpaper wear.
<b>Wear by chip</b>	The friction exceeds the rubber's shear formation strength, whereby the material is removed as small chips.	Wave shape patterns, with wave patterns in the direction of glide.	Eraser wear.
<b>Fatigue wear</b>	The forces in the surface are too small to break at the first cycle, but repeated cycles lead to the material being removed as very small particles.	Smooth, polished surface.	Fatigue wear, polishing wear.

The most common method of testing abrasion for a rubber material is to let a loaded sample slip against a roller covered in emery cloth. After a wear distance of 40 m, the weight loss is determined and calculated over to a volume loss. Eventual variations in the emery cloth are corrected by using a reference piece of rubber and correct the results. The disadvantage with this method is that the wear intensity is higher than what is permitted in practice.

## Dynamic fatigue

ISO 132, ISO 4666, ISO 6943

Dynamic fatigue testing can be carried out in several ways. Products are often tested in ways similar to their actual usage in servo hydraulic machines or eccentric driven test rigs. The time taken until break is determined.

Dynamic fatigue test on materials is often conducted as bending or elongation fatigue in a so-called De Mattia machine. Test pieces are bent or stretched using a frequency of 5 Hz, until a break occurs.

An interesting fact to note is that a rubber which is tested for elongation, lasts much longer if it is preloaded, than if it has zero tension during part of the elongation cycle.



Fatigue testing in a De Mattia machine.

Photo: MonTech



# Other testing

## Discoloration ISO 3865

Discoloration from rubber is caused by migration of substances from the rubber material to adjacent materials. Especially sensitive in this respect are light painted surfaces, plastic mats, etc. The substances that migrate are primarily antioxidants and plasticizers. The most discolouring substances are the effective amine antioxidants. If discoloration could cause problems, the non-discolouring phenol antioxidants must be chosen instead. These are unfortunately both more expensive and have poorer protection effects.

Discoloration shows itself as a faint light brown to black surface on the material that has come in contact with the rubber. The discoloration often shows more clearly if the surface, which has come in contact with the rubber is subjected to sunlight. Discoloration is primarily a problem of appearance.

Discoloration is usually divided into the following types:

- Contact staining, discoloration in the area of contact between the rubber and an underlying surface.
- Migration staining, discoloration that migrates even outside the contact surface.
- Penetration staining, discoloration that penetrates through a material that is in contact with the rubber.
- Extraction staining, discoloration that arises when water, flows over a rubber material and afterwards flows over e.g. a light painted surface. The water in this case dissolves discolouring substances and then discolours the painted surface.

Do not confuse discoloration with smearing. Smearing arises when a black rubber material is rubbed against a surface and some of the rubber is worn off. Smearing can be washed off, discoloration cannot, since the discolouring substance has penetrated into the discoloured surface.



Equipment for determining carbon black dispersion. Photo: MonTech

## Dispersion ISO 11345

It is important that the filler and especially the carbon black are fully dispersed in the rubber compound otherwise it will deteriorate the mechanical properties such as tensile strength and abrasion resistance. The more fine-grained a filler is, the better reinforcing properties it has, but it is more difficult to disperse.

The commonest way to determine the dispersion of carbon black is to cut off a cured rubber piece using a sharp knife. Then the cross-sectional surface is studied in a microscope at 30 times magnification, with diagonal lighting. Poor dispersion can then be seen as unevenness and compared with a reference scale in order to grade the dispersion.

There are also special devices produced to determine the dispersion, based on the TV technique, with built in reference scales for easier estimation.

## Permeability ISO 6179, ISO 2782

Liquids and gases diffuse through rubber. The speed of diffusion depends on the rubber type, the composition of the formula and the diffusion medium. Diffusion is usually divided into three phases:

1. The medium dissolves into the rubber from one side.
2. Diffusion (migration) through the rubber.
3. Evaporation from the opposite surface.

Different rubber materials have different degrees of gas permeability. Butyl rubber has the lowest permeability of the rubber materials.

There are several different methods to determine permeability or diffusion.



In order to determine the diffusion of liquids, the rubber can be placed as a wall in a receptacle with the liquid you want to test. The receptacle is weighed at regular intervals in order to determine the weight loss due to diffusion. The result is usually given in  $\text{g/m}^2 \times \text{h}$ .

In order to determine permeability to gas, the rubber material can be placed as a partition wall between two chambers. One of the chambers is pressurized with the gas which is to be investigated, i.e. air, at a pressure of 0,3–1,5 Mpa. The other chamber is kept at atmospheric pressure. The permeability is measured and expressed in  $\text{m}^2/\text{Pa} \cdot \text{sec}$ .

## Fire testing SIS 162222

All rubber materials are more or less combustible. The rubber materials which contain halogens such as chlorine, fluorine and bromine can be difficult to ignite and they are self-extinguishing. In order to improve the fire resistance of a rubber material, substances which contain chlorine can be added, like for an example chlorparaffin. Antimony trioxide and aluminium hydrate with large amounts of crystal water are also used. Substances which contain chlorine are no longer recommended from an environmental point of view, which is why aluminium hydrate is now the most popularly used additive. Aluminium hydrate works in the way that a lot of energy is required in order for the crystal water to be converted into vapour thus making the rubber difficult to ignite.

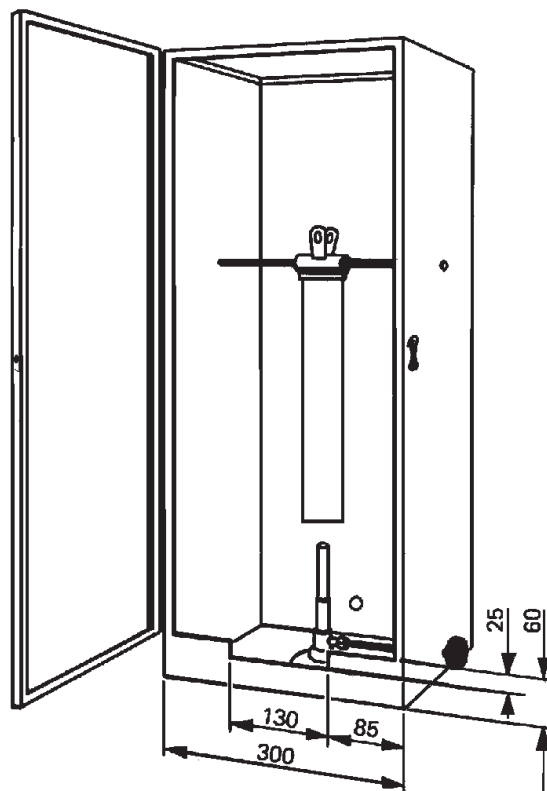
Rubber's fire resistance can be tested using vertical or horizontal methods. The most common method of testing rubber is to ignite a test strip at one end using a Bunsen burner and then observe the time of burning of the strip after the flame has been removed.

## Electrical testing

ISO 1853, ISO 1278, ISO 2951

Rubber polymers are in themselves electrical insulators and rubber is also used in many instances as insulation for electrical cables etc. When carbon black is compounded in the rubber, the electrical conductivity is increased and the rubber becomes a semi-conductor. With special types of carbon black, the rubber can also be made more conductive.

In order to determine the electrical conductivity or resistance, several different methods can be used. In principle, a voltage is applied across a rubber test piece and the electrical current, which passes through the rubber, is measured. This may appear uncomplicated, but it is difficult to achieve in practice since often very small currents have to be detected in addition the resistance of rubber changes with temperature, time of electric exposure and physical stress.



Vertical fire test.



Equipment for measuring volume resistivity of conductive and semiconductive rubber materials.

Photo: Elastocon



# Other testing

## Chemical analysis

Today the content of a rubber material or compound can be well defined by chemical analysis.

In general two types of analysis are used: Qualitative analysis is where the nature of the ingredients is determined. Quantitative analysis is where the amount of each ingredient is determined.

## Analytical instruments

A number of instruments and methods are available today for chemical analysis, and the ones described below are the most commonly used methods for analysis of rubber materials.

## IR-spectroscopy

With IR-analysis an infrared light source is used to pass infrared radiation through the test sample. A spectrum is obtained showing the energy absorption at the different wavelengths in the infrared spectrum. Each substance exhibits its own characteristic absorption spectrum, which is then compared with reference spectra in order to identify the material.

When analysing elastomers or compounds, which have no transparency, the polymer is first pyrolyzed in nitrogen where the polymer is degraded into its fundamental molecules. This pyrolysate which normally is in liquid form, can then be analysed.

In the last few years a new more sensitive type of IR spectrometers is used, referred to as a FTIR-spectrometer. FT stands for Fourier Transformation and it constitutes a way to increasing the resolution by utilizing computerized Fourier analysis. Also black rubber can be analysed by measuring reflected radiation without the need of pyrolysis when using a FTIR spectrophotometer.



FTIR instrument.

Photo: PerkinElmer

## Chromatography

In gas chromatograph, GC, gaseous break down substances are collected and sorted in molecule weight fractions when a gaseous sample is passing a thin capillary in an oven. The type of substance is determined by the so-called retention time, which is the time it takes for the substance to pass the capillary. Even in this method reference spectra are used to identify the substances. A solid material as rubber has to be first subjected to pyrolyzed.

Today a combination of a gas chromatograph and a mass spectrometer, MS, is increasingly used. A mass spectrometer ionises and separates the fractioned samples according to mass-to-charge ratios. The compounds are then identified on the basis of their fragmentation pattern upon ionisation. A mass spectrometer is often used as a detector for a gas chromatograph since it provides structural information about the analysed compound.

## Thin layer chromatography

When using this method plates coated with a thin layer of silica gel is used. The sample is dissolved in a solvent and applied to the plate with the silica gel. Identification is based on the fact that different substances have different rates of diffusion through the silica gel.

## Extraction

Extraction is used to extract different substances from a material, i.e. plasticizers from a rubber compound. For such a purpose a Soxhlet extractor is commonly used. It consists of a glass flask, a cooler and between them an extraction cup.

The sample is placed in the extraction cup and the solvent in the flask. The solvent is brought to boiling, and the volatiles are condensed in the cooler to drip on the sample. When the extraction cup is filled with solvent a siphon will empty it and new solvent will drip on the sample. Normally after 16 hours the extraction is terminated, as most substances should have been extracted after that time. The amount of extracted constituents is determined by weight calculations.

## Thermogravimetric analysis, TGA

A TGA-instrument consists of a very sensitive balance inside an oven, where the temperature can be regulated from room temperature to about 1 000 °C. The atmosphere in the oven can be changed to be i.e. air, nitrogen or oxygen.

## Analysing methods

### Type of elastomer ISO 4650, ISO 7270

Identification of type of rubber can be done in an IR-spectrophotometer or with a gas chromatography. The most commonly used is Pyrolysis- IR, where the rubber is subjected to pyrolysis and identification of the decomposition products is made with IR-spectroscopy.

### Type of antioxidant ISO 4645

Analysis of protective agents such as antioxidants is normally done with thin layer chromatography.

### Ash content ISO 247

The ash residue of a polymer or an elastomeric material is determined by heating a sample in a crucible, at first on a Bunsen to drive off the volatile substances, and later on in an oven at 550 °C. The ash residue is calculated as percentage of the sample weight. The ash residue in a polymer is a measure of the inorganic impurities, while ash of a rubber material is a measure of inorganic constituents such as zinc oxide, whiting, silica, clay etc.

### Carbon black content ISO 1408

When analysing the carbon black content an earlier extracted sample is used. To drive out the polymer from the sample it is pyrolyzed in nitrogen at 850 °C. The sample is then placed in a muffle furnace and the carbon black is burned off at 850 °C. The amount of carbon black is calculated from the weight loss in the different steps of the procedure.



TGA instrument.

Photo: PerkinElmer

### Analysis of free sulphur ISO 7269

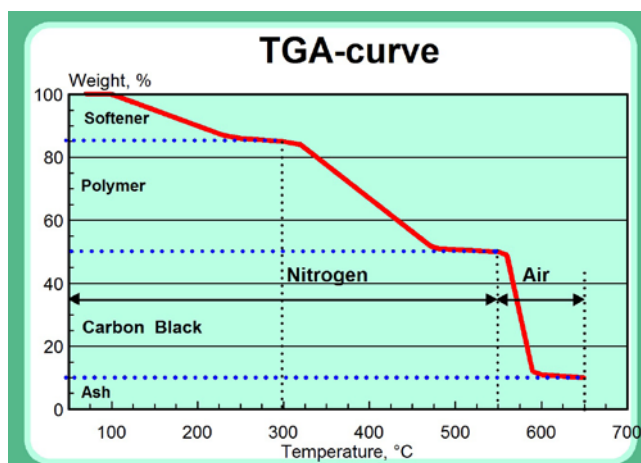
The amount of free sulphur is of interest as it is a measure of the curing degree.

Several methods are available. For example in one method the free sulphur reacts with a solution of sodium sulphite and the thereby formed sodium thiosulphate is determined by titration.

### Analysis of material composition by TGA ISO 9924

Thermogravimetical analysis is used to determine the amount of plasticizer, polymer, carbon black and ash residue in a rubber material.

A sample is weighed and heated in nitrogen, from 70 to 300 °C and in doing so the volatile substances (more or less corresponding to the plasticizers) evaporate. The heating then continues, still in the nitrogen atmosphere, to 550 °C. During that period the remaining organic constituents mostly the polymer, will be removed. Finally the atmosphere in the oven is changed to air and the temperature increases to 650 °C, during which phase the carbon black will burn. The remaining part is the ash residue consisting of inorganic constituents.



TGA-analysis.

# Measurement techniques

## Measurement techniques

Originally various parts of the body were used as measurement units, e.g. the yard, which was the same as the length of the lower arm = 0,594 m, 1 foot = 0,305 m and an imperial inch = 25,4 mm.

## Measurement systems

The modern measurement system is based on the work that was started in 1790 by a commission in France, whose task it was to develop a standardised measurement system. Their starting point was the circumference of the earth. One ten millionth part of the distance between the equator and the North Pole was said to be a metre. A standard metre rod was produced in platinum. In the year 1875, 40 prototype metres were made of platinum-iridium, in the form of a beam with a profiled cross-section, one for each country which had signed the Metre Convention.

The system with metre prototypes is today not sufficiently accurate, since the rods age. Since 1960, a metre is defined on the basis of the wavelength of light. It was also decided that the mass 1-kg was one thousandth of the weight of one cubic metre of water at +4° C. Also kilo prototypes were produced.

The metre system was developed further into the MKS system of units (Meter, Kilogramme, Second) and to the MKSA system, in which the unit Ampere was added for electrical current. These measurement systems were developed further into today's SI system of units.

The SI units are made up of seven basic units, two supplementary units and certain derived units.

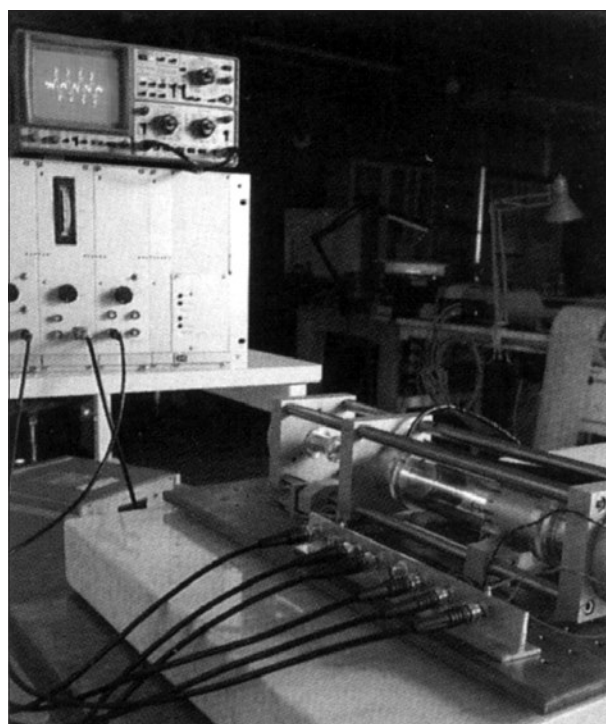
### Basic units

length	meter, m
plane angle	radian, rad
mass	kilogram, kg
solid angle	steradian, sr
time	second, s
electrical current	ampere, A
temperature	kelvin, K
luminous intensity	candela, cd
amount of substance	mole, mol

### Supplementary units

plane angle	radian, rad
solid angle	steradian, sr

Over and above these units, 17 derived units have been added which all have their own designations.



The new prototype metre.

Photo: SP

Multiple units are made from increasing or decreasing powers of 10 from the standard SI units, by adding a prefix before the unit.

#### Prefix

$10^{18}$	E	exa*	$10^{-1}$	d	deci
$10^{15}$	P	peta	$10^{-2}$	c	ti
$10^{12}$	T	tera	$10^{-3}$	m	milli
$10^9$	G	giga	$10^{-6}$	$\mu$	mikro
$10^6$	M	mega	$10^{-9}$	n	nano
$10^3$	k	kilo	$10^{-12}$	p	piko
$10^2$	h	hekto*	$10^{-15}$	f	femto
10	da	deka*	$10^{-18}$	a	atto

Prefixes with \* should be avoided.



The Swedish national archive kilo, nr 40.

Photo: SP

## The measurement of various quantities

### Giving measurements

In the following example: the length = 15 meter = 15 m, length is a quantity that is to say a property which can be measured. The figure 15 is called the measured value, metre is called the unit of measurement (unit) and m is called the unit designation.

A quantity value (a size) is the product of the measured value and the unit, e.g. 15 m. Each unit has both a name, and a designation. The name of the unit of length is the metre and it is designated m. All designations are written with a small letter, unless the designation derives from the name of a person such as for example joule, watt and pascal.

The designations, such as for example m, kg, J, W, Pa, V and N are international. They are written in the same way in all languages. Therefore we should always use the proper designations and not idiomatic ones.

### Writing a measured value

Note that there should be a space between the measured value and the unit: 435 mm.

Write number in groups of three with a space between.

Never use full stop to indicate a thousand, e.g. 1 023 450.

Never use full stop as a decimal point, but use the comma.

Remember that the number in a measurement result, which is not followed by information concerning its accuracy, should reflect the precision of the result. For example, 1 000 m or 1,000 km indicates that the length is known with the accuracy of one metre.

# Measurement techniques

## Length

Measuring tapes and steel rulers are used in the measurement of longer lengths, where the accuracy is approximately 1 mm. Callipers are used for lengths of up to approximately 0,5 m and the accuracy can vary from 0,02 to 0,1 mm. Higher accuracy can be achieved with modern digital callipers. Dial gauges and micrometers are used when the accuracy is required to be 0,001 to 0,01 mm.

The thickness of rubber sheets and compression set buttons, etc. are measured using a dial gauge with a resolution of at least 0,01 mm, mounted in a stand and with a measuring pressure of 22 kPa on the plane measuring foot. The low measuring pressure is required in order not to deform the rubber during the measurements.

## Volume

The volume of a solid body is easily measured by immersing it into water, e.g. in a measuring cylinder and measuring the rise of the surface of the water. The subsequent volume of a liquid is simply measured with graded measuring containers of various kinds.

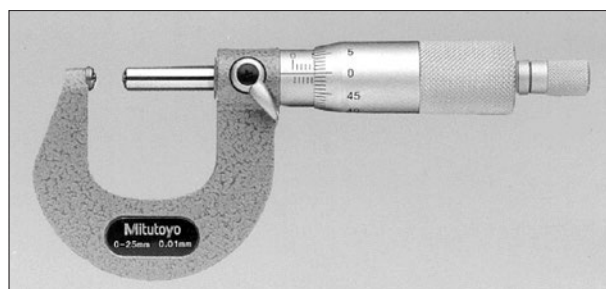
## Mass

The mass or weight of a body is measured using a balance. Several different types of balances are used. The balancing scale is an old construction, where two scale pans are balance against each other. The sample is placed in one pan and weights with known mass are added into the other pan until the two pans have reached balance.



Digital calliper.

Photo: Mitutoyo



Top, an analogue micrometer with vernier scale.  
Below, digital micrometer.

Photo: Mitutoyo



Digital dial indicators.

Photo: Mitutoyo

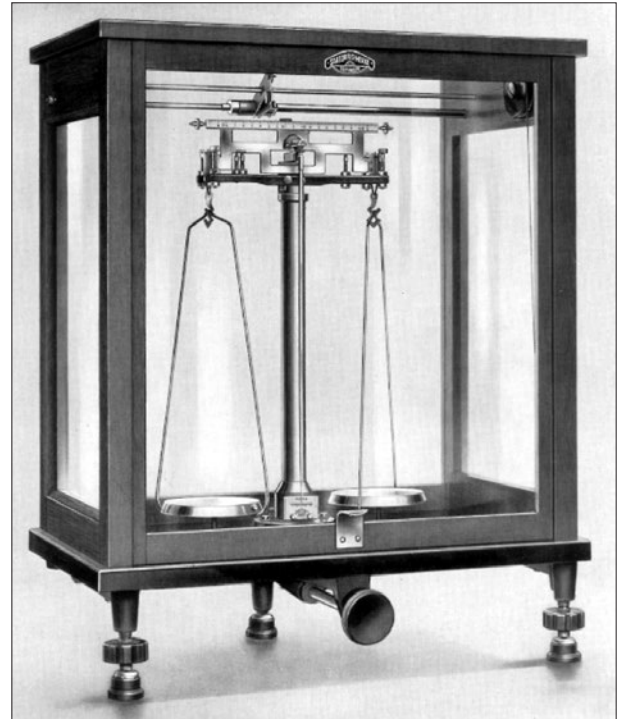


## Balance

Today electronic balances are common. The principle of these balances is to measure the deformation of a metal spring by means of a strain gauge. The deformation is proportional to the mass of the load on the balance. The strain gauges, often several (four of them often used) are combined to form a load cell, which is connected to an electronic amplifier. Through the use of electronics more complicated measuring systems became available, such as automatic tare correction, accumulated weighing, etc.

## Force

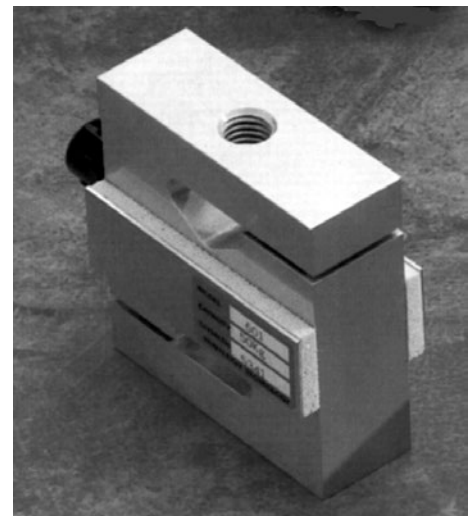
The measuring of force is used in several contexts in the rubber testing, e.g. when carrying out tensile or compression tests. The tensile tester is equipped with a load cell designed in the same way as in an electronic scale. The only difference from these are that the load cell and amplifier are calibrated in N instead of in grammes.



*An old type of balance.*



*Electronic balances.*



*Load cell for measurement of mass or force.*

# Measurement techniques

## Pressure

Pressure e.g. the air pressure of pneumatic systems or of liquids in hydraulic systems can be measured in different ways. The conventional way of measuring pressure has been using a pipe in the shape of a spring or a membrane pressure gauge. The pipe spring pressure gauge or manometer consists of a bent metal pipe connected to an indicator needle. When there is an overpressure in the pipe of the spring, the pipe strains to straighten out and the indicator moves. In the membrane pressure gauge, there is a folded membrane connected to the indicator.

**Electronic pressure sensors** can be designed in different ways, but the principle is that the deformation of a material is measured and then converted into a pressure. A common type is built up like a load cell with a **strain gauge**, in another type the sensor consists of a **piezo-electric crystal**, which produces a change when it is deformed.

Low pressures can also be measured with **liquid pressure gauges**, which consists of a U-pipe half-filled with liquid. The liquid can be alcohol, water or mercury, each providing different measuring ranges.



Digital pressure gauge. Photo: HBM    Analogue pressure gauge. Photo: Wika

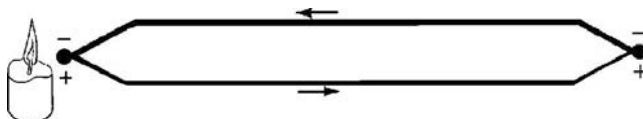
## Temperature

Temperature is a very important quantity in the rubber testing, since it is used in order to control many processes. The temperature can be measured in different ways.

The most common method to measure temperature has been traditionally with a mercury or alcohol filled liquid thermometer. The principle of this thermometer is that the liquid has a greater expansion resulting from changes in temperature than has the glass.

Nowadays electronic thermometers are the most popular. They can be based on several different types of sensing elements, e.g. semi-conductors, resistance thermometers, thermocouples, etc.

The most accurate sensor type is a resistance sensor with a platinum thread wound round a frame and the resistance of which is 100 ohm at 0 °C. The resistance then increases with the temperature and reaches 138 ohm at 100 °C. The sensor is connected to an amplifier which measures the resistance and gives the result converted to the corresponding temperature.



Temperature measurement with thermocouples is based on the principle that an electrical voltage differential occurs between two wires of different metals, which are soldered together. This happens when the points of junctions are subjected to different temperatures. The voltage differential generated is proportional to the difference in temperature between the two junctions of the wires.

The wires are connected to an amplifying measuring instrument, which measures the voltage differential (mV) and converts it into a temperature. In order for the instrument to be able to give the temperature with any accuracy, the temperature at the connection point to the instrument must also be known. In order to measure the temperature at the connection a semi conductor or PT 100 sensor is used.



Digital temperature instrument for resistance sensors, PT 100.

Photo: Testo

## Speed

Classification of linear speed is carried out simplest by the measurement of distance and time.

## Time

Time is a quantity, which we today can measure very accurately with relatively inexpensive instruments. In industry, there is often a need to measure elapsed time, which can be done with a stopwatch with an accuracy of 1/10 sec. There are some clocks which are set by a radio transmitter in Germany and which have an accuracy of 1 s. These clocks correct themselves once per hour via the German radio transmitter which is powered by an atomic clock.

## Calibration – traceability

In order to make sure that the measuring instruments being used show the correct measurements, regular calibration needs to be carried out on the instrument.

Calibration means that the instrument being used is compared to a standard gauge. The standard gauge is an instrument which should have a higher accuracy and which has been calibrated with a known accuracy.

If the instrument gives the wrong readings, it can sometimes be adjusted so that the values it shows are more accurate. After the adjustment repeated calibration is carried out.

To *calibrate* an instrument is to determine the deviation compared to a reference instrument (the standard gauge). To *adjust* an instrument is to rectify it so that it shows values more accurately.



Force calibration of the Swedish national laboratory for force testing.  
Photo: SP


# Measurement techniques

## Traceability

In the ISO 9000 quality system, it is required that instruments which can have an influence on the quality of manufacture are calibrated and that the documentation for this can be traced in accordance with the requirements of standard. By traceability is meant that the calibration is carried out against a reference instrument, and that this is documented and traceable to national and international standards.

**Elastocon**

Datum/Date  
2010-11-12

**KALIBRERINGSBEVIS**  
Statistiskt av ackrediterat kalibreringslaboratorium  
**CALIBRATION CERTIFICATE**  
issued by an Accredited Calibration Laboratory  
ISO/IEC 17025

Martin Spetz/ms k101653

Bevis/Certificate  
1678-k101653

Page 1 (2)

Customer

**Commission:**

Calibration of temperature indicators and control of airflow meters.

**Equipment:**

Elastocon Cell Oven

**Type:**

EB 07

**Serial no:**

201091

**Internal no:**

-

**Indicator:**

Shinko

**Sensor:**

Pentronic

**Type:**

JCS

**Type:**

Pt 100

**Serial no:**

10203158/10A042470/10203170

**Serial no:**

10380009/-10/-11

**Int. no:**

-

**Runtime:**

0 h

**Arrival:**

**Date:** - **State:** Normal

**Calibration:**

**Date:** 101112 **Location:** Elastocon, workshop

**Temp.:** 22,0 °C **Other:** -

**Performance**

Calibration of indicator and sensor by comparison with a reference sensor and reference instrument. The sensors were mounted in a block calibrator.  
Method described in KMT001.  
Verification of airflow meters by comparison with a reference airflow meter. Not included in the accredited calibration.

**Traceability:**

The reference instruments used are calibrated according to certificate MTv F917462-K04 and are there by traceable to the Swedish national laboratory for temperature, SP in Borås.  
Temperature scale ITS-90.  
The reference instrument for air flow are calibrated according to calibration certificate k070363.

**Instruments:**

Indicator Unisystem U24102, Serial no. 09714, inv.no E64  
Sensor Pt 100, inv.no. E222  
Block Calibrator EK 02, serial no. 9902, inv.nr. E130  
Air flow meter, Platon, 0,1-1,2 l/min, serial no. F121360/6, inv.no. E214


**Uncertainty of measurements:**

The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor  $k=2$ , which for a normal distribution corresponds to a coverage probability of approximately 95 %. The standard uncertainty of measurement has been determined in accordance with EAs publication EA - 4 / 02.

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**Elastocon**

Datum/Date  
2010-11-12

**KALIBRERINGSBEVIS**  
Statistiskt av ackrediterat kalibreringslaboratorium  
**CALIBRATION CERTIFICATE**  
issued by an Accredited Calibration Laboratory  
ISO/IEC 17025

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Bevis/Certificate  
1678-k101653

Page 2 (2)

**Result:**

Reference		Object			
Inv. no.	Temperature (°C)	Label	Indication (°C)	Correction (°C)	Uncertainty (°C)
Cell 1					
E64+E222	49.9		49.8	0.1	±0.2
	100.0		99.9	0.1	
	150.0		150.0	0.0	
	199.8		199.8	0.0	
Cell 2					
E64+E222	49.9		49.9	0.0	±0.2
	100.0		100.0	0.0	
	150.0		150.0	0.0	
	199.8		199.9	-0.1	
Cell 3					
E64+E222	49.9		49.8	0.1	±0.2
	100.0		100.0	0.0	
	150.0		150.0	0.0	
	199.8		199.9	-0.1	

Indicated temperature + Correction term = Actual temperature

**Control of airflow meters (not included in the accredited calibration):**

Set value (cm <sup>3</sup> /min)	Actual air flow (cm <sup>3</sup> /min)		
	Cell 1	Cell 2	Cell 3
200	190	180	190
500	450	440	440
800	770	770	770

The uncertainty of the measurements is estimated to be ±25 cm<sup>3</sup>/min.

Borås 2010-11-12  
ELASTOCON AB

Jonas Nilsson  
Responsible for the Laboratory

Martin Spetz  
Responsible for the Calibration

The results relate only to the objects specified in this document.  
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Example of a certificate of calibration.

42 Elastocon – Rubber testing

Testing accuracy means the accuracy that you can expect from a certain method of testing. ISO and ASTM have started to state this in their testing standards. Values of reproducibility and repeatability are given in the standards.

R = Reproducibility = between different laboratories  
r = Repeatability = within an individual laboratory

R is given in the actual units of measurement and endorses that two labs, with a 95 % probability, do not differ more from each other than the value of R.

r means the same thing and refers to tests that are made repeatedly within the same lab, but at different times. If (R) or (r) is given within brackets this then means that the deviation is given as a percentage of a total measurement.

The values for the accuracy are obtained by organising an Interlaboratory Test Program, (ITP). In such a program, at least 10 laboratories should participate in order to allow statistically correct procedure. One of the laboratories manufactures and distributes test material to the other laboratories. Each laboratory then conducts the testing according to the applicable standard. The results are collected and are processed statistically where among other things the results from laboratories with measuring results deviating too greatly (outliers) are removed.

In the table below, repeatability and reproducibility are shown for the most common testing methods for rubber. The results were obtained using ISO standards and from interlab tests conducted in Sweden. The differences between the various laboratories are often much greater than what is expected, which is why there is a great room for improvement in order to refine methods and the equipment for rubber testing.

	<b>r</b>	<b>(r)</b>	<b>R</b>	<b>(R)</b>	
Hardness, IRHD	1.7	2.9	3.5	6.0	1)
Hardness, IRHD micro	3.7	6.2	6.4	10.7	1)
Hardness, Shore A	–	–	7.2	–	2)
Tensile strength, MPa	–	–	–	13.4	2)
Elongation at break %	–	–	–	17.6	2)
Tear resistance, N/mm	–	–	–	30.3	2)
Compression set, %	3.6	15.0	8.6	36	1)
Tension set					
constant elongation, %	–	–	17.3	44	2)
constant load, %	–	–	–	44	2)
Cold test, TR <sub>30</sub> , °C	–	–	7.5	–	2)
Oil resistance weight change, fuel, %	–	–	9.8	12.2	2)
volume change, oil 1, %	–	–	11.3	47.8	2)
volume change, oil 3, %	–	–	8.9	9.3	2)
Heat ageing change in hardness, IRHD	–	–	10.4	–	2)
change in tensile strength, %	–	–	15.0	–	2)
change in elongation at break, %	–	–	16.4	–	2)

1) ISO ITP

2) Swedish ITP





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