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Rubber — Estimation of uncertainty for test methods — Non-functional parameters

Caoutchouc — Estimation de l'incertitude des méthodes d'essai — Paramètres non fonctionnels

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Foreword

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ISO/TR 12134 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Introduction

It is now a requirement that laboratories accredited to ISO/IEC 17025[2] take into account the measurement or calibration uncertainties associated with any work they have performed when assessing conformity of the material or product to a given specification. As there is an increasing requirement for traceability of measurement, more and more technical staff find themselves faced with the task of carrying out an uncertainty evaluation on their reported measurement results.

Currently, the primary source document for guidance on measurement uncertainty is ISO/IEC Guide 98-3[1], to which the interested reader is referred for details.

Eurolab Technical Report No. 1/2007[5] is a very useful guide to alternative approaches to uncertainty evaluation, whilst ISO/TS 21748[3] gives guidance on the use of repeatability/reproducibility data for uncertainty estimation.

Rubber — Estimation of uncertainty for test methods — Non-functional parameters

1 Scope

This Technical Report provides guidance to scientists, engineers and technicians, working in the field of rubber materials and products, to supplement ISO/IEC Guide 98-3 and to provide additional guidance in situations where functional relationships between input quantities (such as temperature, strain rate and time) and derived output quantities (such as tensile strength and compression set) are unknown and where no other guidance is available.

This Technical Report provides a summary of the classical approach that is taken in the preparation of uncertainty budgets and provides in Clause 4 a list of selected test methods and, for each, an indication of the factors that will make a contribution to the uncertainty budget. Clause 5 discusses how “non-functional” factors can be taken into account in the classical approach.

2 Summary of preparing an uncertainty budget

The analysis of most measurement uncertainties can be reduced to a step-by-step procedure. This procedure comprises the following steps:

a) define the functional relationship between the input measurements and the measurand (the quantity being measured, e.g. tensile strength);

b) compile a list of all the factors that are expected to contribute to the uncertainty in the measurand;

c) for each of the uncertainty sources, estimate the magnitude of the uncertainty;

d) from the relationship defined in step a), estimate the effect that each functional quantity has on the measurement result, using direct mathematical techniques;

e) for the non-functional quantities, estimate their effect through other sources, such as secondary experimentation or expert opinion;

f) combine the uncertainties in all the input quantities to obtain the uncertainty in the output quantity;

g) express the expanded uncertainty as an interval about the measurement result within which it is anticipated, with a stated level of confidence, that the measurand will lie.

3 The elements of the uncertainty budget

Taking each of the seven steps in turn:

a) The functional relationship between the measurand and its input variables is given in the International Standard for the test method being examined. For example, the functional relationship for tensile strength, σ, is given by:

$$\sigma = \frac{F}{wt}$$

where

F is the force at break;

w is the test piece width;

t the test piece thickness.

b) See Clause 4 for listings of factors that can be expected to have some influence on the result of the test.

c) Estimating the magnitude of the uncertainty, $u(x)$, is often the most difficult part in preparing the uncertainty budget. Two main types are identified in ISO/IEC Guide 98-3.

Type A uncertainties relate to random effects. Typically, the type A evaluation will be applied to the material property data that has been determined by the test method, e.g. the tensile strength, compression set or volume swell. Such data will generally be normally distributed about their mean (or sufficiently close to a normal distribution for the deviation to be insignificant) so that an estimate of the standard uncertainty can be deduced by means of the usual statistical procedures. The standard uncertainty is given by the standard error of the mean.

The second, type B, relates to systematic effects and is applied to the analysis of such parameters as the calibration of an instrument or the drift between calibrations. Such sources of uncertainty should be evaluated on the basis of the information available, such as a calibration certificate, the manufacturer's specifications or professional judgement and past experience. Part of that experience is in deciding what kind of distribution the uncertainty will take. Often this is not a normal distribution, and rectangular or sometimes triangular distributions are often encountered. Reference should be made to ISO/IEC Guide 98-3 for further details, but in all cases the standard uncertainty is given by the standard deviation of the distribution that has been chosen.

d) Once the standard uncertainty for each of the functional factors has been estimated, the sensitivity coefficient for each must be found. This is the first derivative of the measurand with respect to the parameter being considered. Thus, for tensile strength, the sensitivity coefficient, c, for the force is simply:

$$c_F = \frac{\partial \sigma}{\partial F} = \frac{1}{wt}$$

while for the width it is:

$$c_w = \frac{\partial \sigma}{\partial w} = \frac{-F}{w^2 t}$$

An alternative to formal differentiation is to add a small increment to the factor (for example, add δw to w), calculate the new value for the tensile strength (call this $\sigma+$), then subtract this same increment, δw, from w and recalculate tensile strength (call this $\sigma-$). Then determine the sensitivity coefficient from the expression:

$$c_w = \frac{(\sigma+)-(\sigma-)}{2\delta w}$$

Having found the sensitivity coefficient, the contribution that the factor makes to the uncertainty of the measurand is simply the product of its standard uncertainty and its sensitivity coefficient.

e) Those factors that have an influence on the measurand but which do not contribute to the functional relationship given in step a) above similarly need to be quantified. This aspect of deriving the uncertainty budget is not explicitly covered by ISO/IEC Guide 98-3 or other documents, and yet typically there are many more of these “non-functional” parameters in rubber testing than there are functional ones. Suggestions as to how these may be taken into account are given in Clause 5 of this Technical Report.

f) The combined standard uncertainty of all the individual factor uncertainties quantified in steps d) and e) above are combined by means of a root mean square process. Taking just the three functional parameters for tensile strength by way of illustration:

$$u(\sigma) = \sqrt{u(F)^2 c_F + u(w)^2 c_w + u(t)^2 c_t}$$

g) This combined standard uncertainty is the equivalent of one standard deviation of a normal distribution function. It is conventional, however, for us to work at a confidence level of 95 %, and so this combined standard uncertainty needs to be multiplied by a coverage factor, k, in order to increase the probability that the true value of the property we are considering lies within the expanded combined uncertainty of our measured value — more specifically, that this will happen 95 % of the time. It is conventional to take the coverage factor as being 2. Thus:

$$U(\sigma) = 2u(\sigma)$$

The end result of this process is to enable the test result we have obtained to be quoted with an associated level of uncertainty at a given level of confidence. Thus an uncertainty statement would be of the form: The tensile strength of compound X was ($Z \pm U$) MPa, with an estimated uncertainty of 95 %.

4 Selected test methods and sources of uncertainty

4.1 General

In all cases, the uncertainty arising from material variability will be evaluated using the type A method of calculation. In addition to the non-functional parameters listed below, almost every test method is subject to uncertainties due to the temperature and humidity of test. It is generally assumed that, if the temperature is within the relevant tolerance given in ISO 23529[4], the associated uncertainty is negligible. Humidity is only considered significant for electrical tests.

Similarly, for many test methods the quality of the cutting out of test pieces could influence the result. It is generally assumed that, for standard test pieces, if the cutting process and the quality of the cutter conform to ISO 23529, the associated uncertainty can be neglected.

4.2 Density

Functional parameters		
Accuracy of balance (at least two weighings)		
Non-functional parameters		
Immersion-liquid density at test temperature (this can be calculated)	Test temperature	Absorption of liquid by test material
Effect of suspension thread (this can be calculated)	Air bubbles adhering to material surface (this will be an inseparable part of the between-test-piece variability)	

4.3 Tensile strength (or modulus)

Functional parameters		
Force measurement	Test piece width	Test piece thickness
Non-functional parameters		
Test speed	Alignment of test piece (this will be an inseparable part of the between-test-piece variability)	

4.4 Elongation at break

Functional parameters		
Initial gauge length	Extensometer accuracy	
Non-functional parameters		
Test speed	Alignment of test piece (this will be an inseparable part of the between-test-piece variability)	

4.5 Tear strength

Functional parameters		
Force measurement	Test piece thickness	
Non-functional parameters		
Test speed	Depth of nick (some methods)	Accuracy of angle (angle tear)
Alignment of test piece (this will be an inseparable part of the between-test-piece variability)		

4.6 Compression set

Functional parameters		
Initial thickness of test piece	Final thickness of test piece	Spacer thickness
Non-functional parameters		
Ageing temperature	Test duration	Recovery time
Slippage of, or adhesion between, test piece and metal compression plate (these will be an inseparable part of the between-test-piece variability)		

4.7 Hardness

Non-functional parameters		
Indentor dimensions (including angles for Shore durometers)	Dial accuracy	Parallax errors in reading dial
Load or spring forces	For durometers, the force applied to the foot	Time of force application
Friction in meter		

Hardness is unusual in that approximate relations between several of the parameters and the hardness are readily available and these can be used to estimate the uncertainty using either the tolerances given in the standard or the measured dimensions for the apparatus in question, as considered appropriate. Such estimates for hardness have been given by Brown and Soekarnein[6].

4.8 Heat ageing

Where properties such as tensile strength are measured before and after the ageing process, the factors associated with that parameter apply to both the “before ageing” and the “after ageing” data as well as the factors associated with the ageing process itself given below:

Non-functional parameters		
Ageing temperature	Duration of ageing	Effect of air flow/ air changes
Interval between ageing and testing		

4.9 Effect of liquids (volume swell)

All the effects relating to density (with up to eight weighings to consider) will apply to this test, plus the following:

Non-functional parameters		
Immersion temperature	Test duration	Volatility of test liquid/time interval between weighings
For volume swell, interaction between the test liquid and the liquid (usually water) in which the test piece is weighed		

Where other properties like tensile strength are measured, the factors associated with that parameter apply to both the “before immersion” and the “after immersion” data as well as the factors associated with the immersion process itself given above.

4.10 Abrasion resistance

Functional parameters		
Weighing errors		
Non-functional parameters		
Abrasive grit size	Grit size distribution	Grit shape factors
Applied load	Relative speed between test piece and grit surface	
Removal of fine abraded crumb from test piece surface (this is likely to be an inseparable part of the material variablility)		

Many of these factors will be intractable even to rigorous analysis.

4.11 Gehman low-temperature stiffness

Functional parameters		
Test piece width	Test piece thickness	Free length of test piece
Torsional constant of wire	Twist angle being measured	
Non-functional parameters		
Accuracy of measured angle	Length of time at test temperature	Speed and time twist is applied
Nature of immersion liquid	Slope of modulus/temperature curve	

4.12 Impact brittleness

Non-functional parameters		
Test piece width	Test piece thickness	Free length of test piece
Radius of fulcrum causing bending	Speed of hammer	Operator judgement on crack/no-crack condition
Length of time at test temperature	Nature of immersion liquid	

4.13 Temperature of retraction test

Non-functional parameters		
Initial extension of test piece	Measurement of length of test piece	Friction in retraction apparatus
Length of time at test temperature	Nature of immersion liquid	

4.14 Ozone resistance

Non-functional parameters		
Ozone concentration	Test humidity	Test duration
Strain applied to test piece	Magnifying lens used	Operator judgement

4.15 Stress relaxation

Functional parameters		
Force measurement		
Non-functional parameters		
Mechanical pre-conditioning of test piece	Thermal pre-conditioning of test piece	Applied compression
Jig “break point” consistency	Ageing temperature	Test duration
Test environment (e.g. liquid)	Measurement temperature	
Cooling/heating effects (when measurement and test temperatures are not the same)		
Regression analysis (when average rates of relaxation are required)		

5 Methods of deriving uncertainty estimates for non-functional parameters

5.1 Empirical evaluation

For those factors that do not have a known functional relationship to the measurand, a programme of experimental work can be undertaken to vary systematically the factor of interest (say the test speed for a tensile-strength determination) over a wide enough range that a quantitative estimate of the effect can be established. This can then be evaluated as an uncertainty contribution over the (normally much smaller) range that the parameter is expected to vary under test conditions.

Thus, test speed might be varied between 100 mm/min and 500 mm/min in 100 mm/min increments and the resulting tensile strength plotted as a function of speed, from which the slope can be derived and hence the change in tensile strength that can be expected over the probable speed uncertainty under test conditions of ±5 mm/min (or whatever the likely speed variation is with the specific item of test equipment being used).

For temperature, the normal allowable variation is ±1 °C or ±2 °C but, to assess the temperature effect, ±10 °C or even ±20 °C might have to be applied to get a sufficiently measurable effect.

In principle, this can be applied to each factor for each test. However, it is to be expected that different rubbers will have different sensitivities, so a rigorous evaluation would have to take this into account as well. Such an extensive test programme is impractical, so the best that can be achieved is to establish the “generic” effects, based on a limited number of polymer types being tested.

It is rare for such investigations to be published, but any that have been can clearly be used.

5.2 Calculation

In a few cases, the effect of a parameter can be calculated, even although it does not appear in the function given in the test method for obtaining the result. Such cases have been indicated in the tables.

5.3 Expert opinion

Instead of formal experimental work, which is costly and time consuming, it might be that many aspects of the uncertainty budget can be established by expert opinion agreeing what the likely contribution of a particular factor to a particular test method should be. This is particularly likely to be the case for parameters that are expected to have a negligible effect if controlled within limits specified in the test method standard. Indeed, the object of such limits is to minimize uncertainty.

5.4 Interlaboratory testing

As an alternative approach to the problem, interlaboratory testing can be a source of extra information that, in principle, could be applied to the task of data gathering so that quantitative estimates of uncertainty can be derived. ISO/TS 21748[3] indicates one such methodology.

It is worth noting that, if interlaboratory testing can be used, then this would replace the whole of the involved and often complex process of assessing uncertainties using the conventional “functional relationship” method.

Since interlaboratory testing is not set up to provide uncertainty estimates as such, there is still some debate as to whether this is even applicable in principle or, if it is, how to establish the uncertainty in the test result from the repeatability and reproducibility data obtained from the trial. The repeatability factor is almost certainly too small as it does not take account of the finite errors associated with the instrumentation being used. On the other hand, the reproducibility is certainly too large because it takes account of the interlaboratory differences (e.g. bias) which, for any one laboratory's uncertainty estimation, is not relevant.

Bibliography

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