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Plastics piping and ducting systems — Determination of the long-term hydrostatic strength of thermoplastics materials in pipe form by extrapolation

Systèmes de canalisations et de gaines en matières plastiques — Détermination de la résistance hydrostatique à long terme des matières thermoplastiques sous forme de tubes par extrapolation

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9080 was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 5, *General properties of pipes, fittings and valves of plastic materials and their accessories — Test methods and basic specifications*.

This second edition cancels and replaces the first edition (ISO 9080:2003), which has been technically revised. The following changes have been made:

— all references to lifetime have been removed, as this standard only deals with the mathematics for extrapolation and the calculation of long-term strength;

— a more accurate description of the number and distribution of the observations and of the use of the extrapolation has been included;

— the observations in the example of Annex C have been modified in order to comply with the specifications of this standard and, consequently, the results of the regression calculations have been updated;

— a second set of observations has been added in Annex D in order to provide an evaluation according to the 3-parameter model (see Annex C), and according to the 4-parameter model (see Annex D);

— a second software package has been evaluated and included in Annex E.

Introduction

0.1 General

This Standard Extrapolation Method (SEM) is meant to be used to evaluate the long-term hydrostatic strength of a material in pipe form. Product standards have specific requirements for the physical and mechanical properties of the material used for the intended application. It is emphasized that the Standard Extrapolation Method (SEM) described in this document is not intended to be used to disqualify existing procedures for arriving at design stresses or allowable pressures for pipelines made of plastics materials, or to disqualify pipelines made of materials proven by such procedures, for which experience over many years has been shown to be satisfactory.

Software packages have been developed for the SEM analysis as described in Annex A and Annex B. Windows-based programmes are commercially available (see Annex E). Use of these software packages is recommended.

0.2 Principles

The suitability of a plastics material for a pressure pipe is determined by its long-term performance under hydrostatic stress when tested in pipe form, taking into account the envisaged service conditions (e.g. temperature). For design purposes, it is conventional to express this by means of the hydrostatic (hoop) stress which a plastics pipe made of the material under consideration is expected to be able to withstand for 50 years at an ambient temperature of 20 °C using water as the internal test medium. The outside test environment can be water or air. This method is not intended to imply service life. In certain cases, it is necessary to determine the value of the hydrostatic strength at either shorter design times or higher temperatures, or on occasion both. The method given in this International Standard is designed to meet the need for both types of estimate. The result obtained will indicate the lower prediction limit (LPL), which is the lower confidence limit of the prediction of the value of the stress that can cause failure in the stated time at a stated temperature.

This International Standard provides a definitive procedure incorporating an extrapolation method using test data at different temperatures analysed by multiple linear regression analysis. The results permit the determination of material-specific design values in accordance with the procedures described in the relevant product standards.

This multiple linear regression analysis is based on the rate processes most accurately described by $\log_{10}$(stress) versus $\log_{10}$(time) models.

In order to assess the predictive value of the model used, it has been considered necessary to make use of the estimated 97,5 % lower prediction limit (LPL). The 97,5 % lower prediction limit is equivalent to the lower 97,5 % confidence limit for the predicted value. This convention is used in the mathematical calculations to be consistent with the literature. This aspect necessitates the use of statistical techniques.

The method can provide a systematic basis for the interpolation and extrapolation of stress rupture characteristics at operating conditions different from the conventional 50 years at 20 °C (see 5.1.5).

Thermoplastic materials in pipe form such as mineral filled thermoplastic polymer, fibre reinforced thermoplastics, plasticized thermoplastics, blends and alloys may have further considerations with regards to prediction of long term strength which have to be taken into account in the corresponding product standards.

It is essential that the medium used for pressurizing the pipe does not have an adverse effect on the pipe. In general, water is considered to be such a medium.

Long consideration was given to deciding which variable should be taken as the independent variable to calculate the long-term hydrostatic strength. The choice was between time and stress.

The basic question the method has to answer can be formulated in two ways, as indicated below:

a) What is the maximum stress (or pressure) that a given material in pipe form can withstand at a given temperature for a defined time?

b) What is the predicted time to failure for a material in pipe form at a given stress and temperature?

Both questions are relevant.

If the test data for the pipe under study does not show any scatter and if the pipe material can be described perfectly by the chosen empirical model, the regression with either time independence or stress independence will be identical. This is never the case because the circumstances of testing are never ideal nor will the material be 100 % homogeneous. The observations will therefore always show scatter. The regressions calculated using the two optional independent variables will not be identical and the difference will increase with increasing scatter.

The variable that is assumed to be most affected by the largest variability (scatter) is the time variable and it has to be considered as a dependent variable (random variable) in order to allow a correct statistical treatment of the data set in accordance with this method. However, for practical reasons, the industry prefers to present stress as a function of time as an independent variable.

0.3 Use of the methods

The purpose of this extrapolation method is to estimate the following:

a) The lower prediction limit[1)] (at 97,5 % probability level) of the stress which a pipe made of the material under consideration is able to withstand for 50 years at an ambient temperature of 20 °C using water or air as the test environment. In accordance with ISO 12162, the categorised value of this lower prediction limit is defined as MRS and is used for classification of the material.

b) The value of the lower prediction limit (at 97,5 % probability level) of the stress, either at different design times or at different temperatures, or on occasion both. In accordance with ISO 12162, the categorised value of this lower prediction limit is defined as CRS$_{\theta,t}$ and is used for design purposes.

There are several extrapolation models in existence, which have different numbers of terms. This SEM will use only models with two, three or four parameters.

Adding more terms could improve the fit but would also increase the uncertainty of the predictions.

The SEM describes a procedure for estimating the lower prediction limit (at 97,5 % probability level) whether a knee (which demonstrates the transition between data type A and type B) is found or not (see Annex B).

The materials are tested in pipe form for the method to be applicable.

The final result of the SEM for a specific material is the lower prediction limit (at 97,5 % probability level) of the hydrostatic strength, expressed in terms of the hoop stress, at a given time and a given temperature.

For multilayer pipes, the determination of the long-term hydrostatic pressure strength of the products is carried out in accordance with ISO 17456.

For composite and reinforced thermoplastics pipes, guidance on the use of this method is given in the product standards.

Guidance for the long-term strength of a specific material with reference lines is given in the appropriate product standard.

1) In various ISO documents, the lower prediction limit (LPL) is defined incorrectly as the lower confidence limit (LCL), where LCL is the 97,5 % lower confidence limit for the mean hydrostatic strength.

Plastics piping and ducting systems — Determination of the long-term hydrostatic strength of thermoplastics materials in pipe form by extrapolation

1 Scope

This International Standard specifies a method for predicting the long-term hydrostatic strength of thermoplastics materials by statistical extrapolation. The method is applicable to all types of thermoplastics pipe at applicable temperatures. It was developed on the basis of test data from pipe systems.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1167-1, *Thermoplastics pipes, fittings and assemblies for the conveyance of fluids — Determination of the resistance to internal pressure — Part 1: General method*

ISO 1167-2, *Thermoplastics pipes, fittings and assemblies for the conveyance of fluids — Determination of the resistance to internal pressure — Part 2: Preparation of pipe test pieces*

ISO 2507-1:1995, *Thermoplastics pipes and fittings — Vicat softening temperature — Part 1: General test method*

ISO 3126, *Plastics piping systems — Plastics piping components — Measurement and determination of dimensions*

ISO 11357-3, *Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization*

ISO 12162, *Thermoplastics materials for pipes and fittings for pressure applications — Classification, designation and design coefficient*

ISO 17456, *Plastics piping systems — Multilayer pipes — Determination of long-term strength*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
internal pressure
p
force per unit area exerted by the medium in the pipe, in bars

3.2
stress
σ
force per unit area in the wall of the pipe in the hoop (circumferential) direction due to internal pressure, in megapascals

NOTE It is derived from the internal pressure using the following simplified equation:

$$\sigma = \frac{p(d_{\text{em}} - e_{\text{y,min}})}{20 e_{\text{y,min}}}$$

where

p is the internal pressure, in bars;

d_{em} is the mean outside diameter of the pipe, in millimetres;

$e_{y,min}$ is the minimum measured wall thickness of the pipe, in millimetres.

3.3
test temperature
T_t
temperature at which stress rupture data have been determined, in degrees Celsius

3.4
maximum test temperature
$T_{t,max}$
maximum temperature at which stress rupture data have been determined, in degrees Celsius

3.5
service temperature
T_s
temperature at which the pipe will be used, in degrees Celsius

3.6
time to failure
t
time to occurrence of a leak in the pipe, in hours

3.7
maximum test time
t_{max}
time obtained by averaging the logarithms of the five longest times to failure, in hours

3.8
extrapolation time
t_e
time limit for which extrapolation is allowed, in hours

3.9
long-term hydrostatic strength
σ_{LTHS}
quantity with the dimensions of stress, which represents the predicted mean strength at a temperature T and time t, in megapascals

3.10
lower confidence limit of the predicted hydrostatic strength
σ_{LPL}
quantity with the dimensions of stress, which represents the 97,5 % lower confidence limit of the predicted hydrostatic strength at a temperature T and time t, in megapascals

NOTE It is given by

$$\sigma_{LPL} = \sigma(T, t, 0{,}975)$$

3.11
knee, data type A, data type B
point of intersection of two branches at the same temperature; data points used to calculate the first branch are designated as type A, data points used to calculate the second branch are designated as type B

3.12
branch
line of constant slope in the $\log_{10}$ (stress) versus $\log_{10}$ (time) plot representing the same failure mode

3.13
extrapolation time factor
k_e
factor for calculation of the extrapolation time

4 Acquisition of test data

4.1 Test conditions

The pipe stress rupture data shall be determined in accordance with ISO 1167-1 and ISO 1167-2. The determination of the resistance to internal pressure shall be carried out using straight pipes.

The mean outside diameter and minimum wall thickness of each pipe test piece shall be determined in accordance with ISO 3126.

For all calculations, the pipes tested shall be of the same nominal dimension and made from the same batch of material and come from the same production run.

For existing materials evaluated according to ISO/TR 9080:1992 or ISO 9080:2003, the initial data set may be complemented by additional data produced from other batches to meet the requirements of 4.2. In such case, the additional data should be spread regularly at each temperature and documented in the test report.

4.2 Distribution of internal pressure levels and time ranges

4.2.1 For each temperature selected, a minimum of 30 observations shall be obtained, spread over the testing time. Internal pressure levels shall be selected such that at least four observations will occur above 7 000 h and at least one above 9 000 h (see also 5.1.5). In the event of prediction based on the second branch, a minimum number of 20 observations is required for the second branches, with a minimum of 5 observations per temperature.

4.2.2 For all temperatures, times to failure up to 10 h shall be neglected.

4.2.3 At temperatures $\leq$ 40 °C, times to failure up to 1 000 h may be neglected, provided that the number of remaining observations conforms to 4.2.1. In that case, at the selected temperature(s), all points below the selected time shall be discarded.

4.2.4 Test pieces which have not failed above 1 000 h may be used as observations in the multiple linear regression computations and for the determination of the presence of a knee. Otherwise, they should be disregarded, provided that the number of remaining observations conforms to 4.2.1.

5 Procedure

5.1 Data gathering and analysis

5.1.1 General

The method is based on multiple linear regression and calculation details given in Annex A. It requires testing at two or more temperatures and times of 9 000 h or longer and is applicable whether or not indications are found for the presence of a knee.

5.1.2 Required test data

Obtain test data in accordance with Clause 4 and the following conditions, using two or more temperatures T_1, T_2, …, T_n:

a) Each pair of adjacent temperatures shall be separated by at least 10 °C and at most 50 °C.

b) One of the test temperatures shall be 20 °C or 23 °C.

c) The highest test temperature $T_{t,max}$ shall not exceed the Vicat softening temperature, VST_{B50}, determined in accordance with ISO 2507-1:1995 minus 15 °C for glassy amorphous polymers, or the melting temperature determined in accordance with ISO 11357-3 minus 15 °C for semi-crystalline polymers.

d) The number of observations and the distribution of internal pressure levels at each temperature shall conform to 4.2.

e) To obtain an optimum estimate of σ_{LPL}, the range of test temperatures shall be selected such that it includes the service temperature or range of service temperatures.

Failures resulting from contamination may be disregarded, provided that the number of remaining observations conforms to 4.2.1.

All valid data points shall be used in the calculations.

For most materials, the test environment and test temperatures are specified in the relevant product standards.

5.1.3 Detection of a knee and validation of data and model

Use the procedure given in Annex B to detect the presence of any knee.

After detecting a knee at any particular temperature, split the data set into two groups, one belonging to the first branch (data type A), the other belonging to the second branch (data type B).

Fit the multiple linear regression as described in Annex A independently, using all first-branch (type A) data points for all temperatures and all second-branch (type B) data points for all temperatures.

When studying the data for the occurrence of a knee, attention should be paid to the occurrence of a degradative failure. Such data (usually characterized by a nearly stress-independent line and visually recognizable) should not be considered for the calculation, but should only be used for determination of the extrapolation time (see 5.1.5).

If the automatic knee detection does obviously not correspond with the visual examination of the diagram, then the data points type A and type B in the region of the predicted knee can be manually reclassified to better align the knee point position with the data. In this case all data points at higher stresses than the stress level of the reclassified transition from type A to type B data points shall be declared type A and all data points at lower stresses shall be declared type B. The extrapolation shall be performed again without automatic knee detection. It is recommended in this case that more data points beyond the time of the predicted knee are obtained.

The reasons for following the manual procedure and details of the changes made for the analysis shall be justified and included in the test report, see Clause 7.

5.1.4 Visual verification

Plot the observed data points, the σ_{LTHS} linear regression lines and the σ_{LPL} curves as a graph on a $\log_{10}\sigma$ / $\log_{10}$ (time) scale.

5.1.5 Extrapolation time and extrapolation time factor

Determine the extrapolation time t_e using the following information and procedures.

The time limits t_e for which extrapolation is allowed, are bound to be temperature-dependent values. The extrapolation time factor k_e as a function of ΔT is based on the following equation:

$$\Delta T = T_t - T$$

where

T_t is the test temperature to which the extrapolation time factor k_e is applied, $T_t \leq T_{t,max}$, in degrees Celsius;

$T_{t,max}$ is the maximum test temperature, in degrees Celsius;

T is the temperature for which the extrapolation time is calculated, $T_s \leq T$, in degrees Celsius;

T_s is the service temperature, in degrees Celsius.

Calculate the extrapolation time t_e, using the following equation:

$$t_e = k_e\, t_{max}$$

Obtain the maximum test time t_{max}, by averaging the logarithms of the five longest times to failure, which are not necessarily at the same stress level, but at the same temperature. Test pieces that have not yet failed may be considered as data points for this purpose. All those points shall belong to the population with which all calculations are performed.

If the data at the maximum test temperature are not used for determination of the regression model, these data are only used for the determination of the maximum test time t_{max} and consequently for the extrapolation time t_e. Such choices of calculation shall be justified and reported. Extrapolation is not permitted above the temperature range of the regression model.

The data obtained may be used for predicting the strength down to 20 °C below the lowest test temperature, provided that there is no change of state of the material, e.g. glass transition.

NOTE It is recommended that test data be generated at the lowest predicted temperature to demonstrate performance.

Examples of the application of the extrapolation time factor are presented in Figures 1 to 3. Figure 2 represents the case that a knee has been detected only at the highest temperature. Figure 3 refers specifically to the case that a knee has been detected at higher temperatures. Values of the extrapolation time factor k_e are assigned in 5.2 and 5.3.

NOTE In cases like Figure 2, t_{max} is positioned at the time of the knee point.

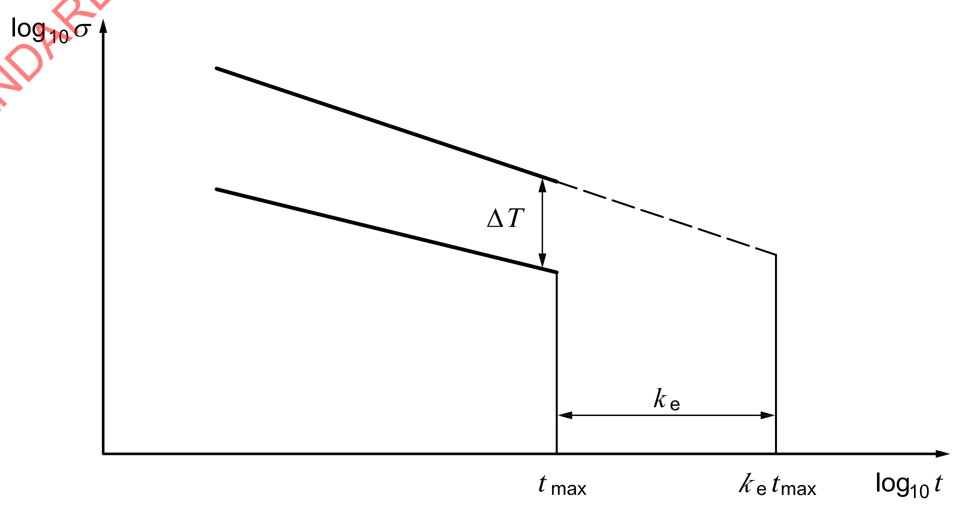


Figure 1 — Extrapolation time in the case of extrapolation without a knee at the highest test temperature

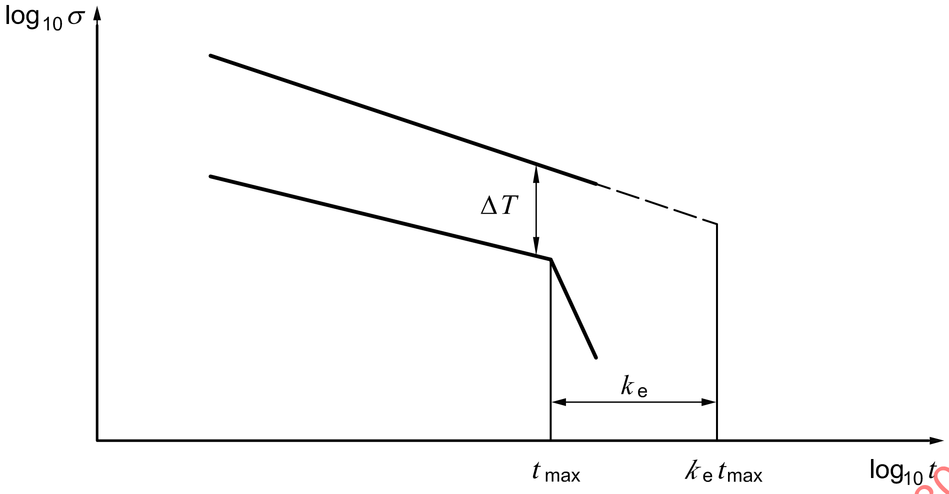


Figure 2 — Extrapolation time in the case of extrapolation with a knee only at the highest test temperature

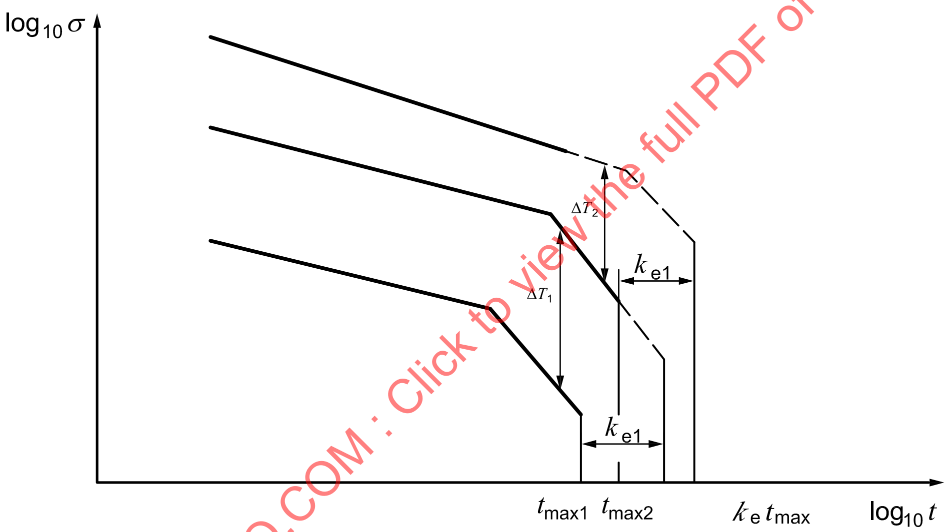


Figure 3 — Extrapolation time in the case of extrapolation with knees at different test temperatures

5.2 Extrapolation time factors for polyolefins (semi-crystalline polymers)

For extrapolation of stress rupture data of polyolefins, the extrapolation time is based on an experimentally determined time to failure at the relevant maximum test temperature and an Arrhenius equation for the temperature dependence using the apparent activation energy calculated from the third (degraded) branch of the curve for stabilized polyolefins (which is 110 kJ/mol, i.e. a conservative value for the activation energy from the third branch). This yields the extrapolation time factors k_e given in Table 1.

Table 1 — Relationship between ΔT (= $T_t - T$) and k_e for polyolefins

ΔT °C	k_e
≥ 10 but < 15	2,5
≥ 15 but < 20	4
≥ 20 but < 25	6
≥ 25 but < 30	12
≥ 30 but < 35	18
≥ 35 but < 40	30
≥ 40 but < 50	50
≥ 50	100

5.3 Extrapolation time factors for glassy, amorphous vinyl chloride based polymers

For extrapolation of stress rupture data for vinyl chloride based polymers, the extrapolation time is based on an experimentally determined time to failure at the maximum test temperature, which is 15 °C below the Vicat softening temperature, and an Arrhenius equation for the temperature dependence, which employs the estimated activation energy calculated from the assumed third (degraded) branch of the curve for vinyl chloride based polymers (which is 178 kJ/mol). This yields the extrapolation time factors k_e given in Table 2.

Table 2 — Relationship between ΔT (= $T_t - T$) and k_e for vinyl chloride based polymers

ΔT °C	k_e
≥ 5 but < 10	2,5
≥ 10 but < 15	5
≥ 15 but < 20	10
≥ 20 but < 25	25
≥ 25 but < 30	50
≥ 30	100

For modified PVC materials, the extrapolation time factors given in Table 2 shall be used if the continuous phase of the modified PVC material is a vinyl chloride based polymer.

5.4 Extrapolation time factors for polymers other than those covered in 5.2 and 5.3

For the polymers not mentioned in this International Standard, the extrapolation time factors given in Table 1 shall be used. If experimental evidence can be given that, for a specific polymer, other extrapolation time factors are justified, these may be used instead of the factors given in Table 1.

6 Example of calculation, software validation

For an example of a semi-crystalline polymer, the calculation of the regression curves and an example of knee detection, in accordance with the procedures described in Clause 5, is given in Annex C.

The calculation of the regression curves for an example of a vinyl chloride based polymer, in accordance with the procedures described in Clause 5, is given in Annex D.

The data sets given in Clause C.1 and D.1 are considered as the software validation data sets. If a programme other than that mentioned in Annex E is used, the calculations performed with the software validation data sets given in Annex C and D shall yield the same results, accurate to the last three decimals as presented in Annex C and D.

7 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) complete identification of the material and samples, e.g. manufacturer, material type, grade name, batch number, source and previous significant history, if any;

c) the dimensions of the pipes used for testing;

d) the outside test environment and the pressure medium inside the pipes;

e) a table of the observations, including, for each observation, the test temperature (in degrees Celsius), the pressure level (in bars), the stress (in megapascals), the time to failure (in hours), a physical observation of the type of failure (ductile, brittle or unknown), the data type (A or B), the date of the test and any other observations which could be relevant;

f) the number of neglected data points under 1 000 h and the corresponding temperature, time to failure and type of failure;

g) if applicable, the reclassified data points and justification for reclassification;

h) the model used to estimate σ_{LTHS} and σ_{LPL};

i) the estimated parameters c_i and their standard deviations s_i, for each branch separately;

j) a graph presenting observed failure points, σ_{LTHS} linear regression line(s) and σ_{LPL} curve(s);

k) details of the software package used for the calculations;

l) any factors which could have affected the results, such as any incidents or any operating details not specified in this International Standard.

Annex A
(normative)

Methods of analysis

A.1 General model

The general 4-parameter model used in this International Standard is the following:

$$\log_{10}t = c_1 + c_2/T + c_3\log_{10}\sigma + c_4(\log_{10}\sigma)/T + e \tag{A.1}$$

where

t is the time to failure, in hours;

T is the temperature, in kelvins (°C + 273,15);

σ is the hoop stress, in megapascals;

c_1 to c_4 are the parameters used in the model;

e is an error variable, having a Laplace-Gaussian distribution, with zero mean and constant variance (the errors are assumed to be independent).

NOTE For calculation purposes in Annex A, the temperature T is expressed in kelvins.

The 4-parameter model shall be reduced to a 3-parameter model if the probability level of c_3 is greater than 0,05. In that case, $c_3 = 0$, i.e.:

$$\log_{10}t = c_1 + c_2/T + c_4(\log_{10}\sigma)/T + e \tag{A.2}$$

A 2-parameter model shall be chosen if all the data points relate to the same temperature:

$$\log_{10}t = c_1 + c_3\log_{10}\sigma + e \tag{A.3}$$

The calculations for the 4-parameter model are given below. The corresponding calculations for other models can be obtained by removing the appropriate terms. Due to possible ill-conditioning of the matrices to be inverted, it is required to perform the computations in double precision arithmetic (14 significant digits). The inversion of the matrices is performed by the classical Gauss-Jordan approach described in Reference [1] (see the Bibliography).

The following matrix notations are used:

$$\boldsymbol{X} = \begin{bmatrix} 1 & 1/T_1 & \log_{10}\sigma_1 & (\log_{10}\sigma_1)/T_1 \\ \vdots & \vdots & \vdots & \vdots \\ 1 & 1/T_N & \log_{10}\sigma_N & (\log_{10}\sigma_N)/T_N \end{bmatrix}; \; y = \begin{bmatrix} \log_{10}t_1 \\ \vdots \\ \log_{10}t_N \end{bmatrix}; \; e = \begin{bmatrix} e_1 \\ \vdots \\ e_N \end{bmatrix}$$

where N is the total number of observations.

With $c = (c_1, c_2, c_3, c_4)^T$, ^{T} being the transposition operator, model (A.1) can be written:

$$y = \boldsymbol{X}c + e$$

The least-squares estimates of the parameters are given by:

$$\hat{c} = (\boldsymbol{X}^T\boldsymbol{X})–1\boldsymbol{X}^T y$$

and the residual variance estimate is given by:

$$s^2 = (y - \boldsymbol{X}\hat{c})^T(y - \boldsymbol{X}\hat{c})/(N - q)$$

where q is the number of parameters in the model.

The predicted stress value corresponding to a given time to failure t and temperature T is given by:

$$\log_{10}\sigma = (\log_{10}t - c_1 - c_2/T)/(c_3 + c_4/T)$$

To calculate σ_{LPL}, corresponding to a given time to failure t and temperature T, the inversion operation in the following relationship is carried out:

$$\log_{10}t = c_1 + c_2/T + c_3\log_{10}\sigma + c_4(\log_{10}\sigma)/T - t_{St}s[1 + x(\boldsymbol{X}^T\boldsymbol{X})–1x^T]^{1/2}$$

where

t_{St} is Student's t-value corresponding to a probability level of 0,975 and a number of degrees of freedom of $N - 4$;

x represents the vector $[1, 1/T, \log_{10}\sigma, (\log_{10}\sigma)/T]$

The result is:

$$\log_{10}\sigma_{LPL} = \frac{-\beta - \sqrt{\beta^2 - 4\alpha\gamma}}{2\alpha}$$

where

$$\alpha = (c_3 + c_4/T)^2 - t_{St}^2 s^2(K_{33} + 2K_{43}/T + K_{44}/T^2)$$

$$\beta = 2(c_1 + c_2/T - \log_{10}t)(c_3 + c_4/T) - 2t_{St}^2 s^2[K_{31} + (K_{41} + K_{32})/T + K_{42}/T^2]$$

$$\gamma = (c_1 + c_2/T - \log_{10}t)^2 - t_{St}^2 s^2(K_{11} + 2K_{21}/T + K_{22}/T^2 + 1)$$

K_{ij} is the element $(\boldsymbol{X}^T\boldsymbol{X})^{-1}$ corresponding to the parameters c and c_j in the model (A.1).

The value of σ_{LPL} can then be calculated from the following equation:

$$\sigma_{LPL} = 10^{\log_{10}\sigma_{LPL}}$$

A.2 Reduced models

In the case of a 3-parameter model (i.e. $c_3 = 0$), the third column of $\boldsymbol{X}$ is deleted and K_{ij} will now represent the element of $(\boldsymbol{X}^T\boldsymbol{X})^{-1}$ corresponding to the parameters c_i and c_j calculated using this new $\boldsymbol{X}$ matrix:

$$\log_{10}\sigma = (\log_{10}t - c_1 - c_2/T)\frac{T}{c_4}$$

and

$$\alpha = (c_4/T)^2 - t_{St}^2 s^2 K_{44}/T^2$$

$$\beta = 2(c_1 + c_2/T - \log_{10}t)c_4/T - 2t_{St}^2 s^2(K_{41}/T + K_{42}/T^2)$$

$$\gamma = (c_1 + c_2/T - \log_{10}t)^2 - t_{St}^2 s^2(K_{11} + 2K_{21}/T + K_{22}/T^2 + 1)$$

t_{St} is now based on $N - 3$ degrees of freedom.

In the case of a 2-parameter model (i.e. $c_2 = 0$, $c_4 = 0$), the second and last columns of $\boldsymbol{X}$ are deleted and K_{ij} will now represent the element of $(\boldsymbol{X}^T\boldsymbol{X})$–1 corresponding to the parameters c_i and c_j calculated using this new $\boldsymbol{X}$ matrix:

$$\log_{10}\sigma = (\log_{10}t - c_1)/c_3$$

and

$$\alpha = c_3^2 - t_{St}^2 s^2 K_{33}$$

$$\beta = 2(c_1 - \log_{10}t)c_3 - 2t_{St}^2 s^2 K_{31}$$

$$\gamma = (c_1 - \log_{10}t)^2 - t_{St}^2 s^2(K_{11} + 1)$$

t_{St} is now based on $N - 2$ degrees of freedom.

A.3 Calculation of σ_{LTHS} and σ_{LPL} when a knee has to be taken into account

As outlined in Annex B, it is assumed that two failure mechanisms may be operating, each in its own range of temperatures and times to failure. The two data sets corresponding to each data type must be fitted independently to the model. In order to do this, the available test data have to be subdivided into two groups, one of the mechanisms being assumed to be operating in each group.

In each group, σ_{LTHS} and σ_{LPL} can now be calculated using the general procedure, provided sufficient data are available and its distribution over the temperature range is suitable (see 4.2 and 5.1.2).

Apply the automatic knee detection method separately for each temperature as described in Annex B. By this method the data are divided into two groups. These two data sets are then fitted independently to the model, in accordance with the general procedure described in this Annex.

A.4 Test of fit

To test the fit of the model to the data, the following statistics method is used:

$$F = \frac{(SS_{\mathrm{H}} - SS_{\mathrm{e}})/(\nu_{\mathrm{H}} - \nu_{\mathrm{e}})}{SS_{\mathrm{e}}/\nu_{\mathrm{e}}}$$

where

F is the Fisher statistic;

SS_{e} is the sum of the squares of the differences between each individual observation and its corresponding mean, i.e. the mean of the repetitions at the experimental conditions at which the observations were made (the model being used does not affect this calculation);

SS_{H} is the sum of the squares of the differences between each individual observation and the corresponding prediction, i.e. the prediction made by the model for the experimental conditions at which the observations were made;

ν_{e} is the number of degrees of freedom for SS_{e}, i.e. the number of observations minus the number of different experimental conditions;

ν_{H} is the number of degrees of freedom for SS_{H}, i.e. the number of observations minus the number of parameters in the model being used.

Under the hypothesis that the model being fitted to the data are correct, the above statistics operation will deliver an F-distribution with $\nu_{\mathrm{H}} - \nu_{\mathrm{e}}$ degrees of freedom for the numerator and ν_{e} degrees of freedom for the denominator.

Using the F-distribution, available as tables and computer programmes, the probability that F would exceed the value calculated using the above formula is obtained. This probability is then compared with a significance limit of 0,05. If this limit is exceeded, the hypothesis that the model is correct is accepted. If the limit is not exceeded, the hypothesis is rejected.

NOTE 1 This test can only be considered as an indication of the fit of the model to the observations.

NOTE 2 An example of a test-of-fit calculation for the 20 °C observations of Table C.1 is given below, assuming a 2-parameter model:

The value of $F(13;15)$ is 0,478. The probability (Pr) that the F-distribution with the given numbers of degrees of freedom exceeds this value is:

$\Pr[F(13;15) > 0{,}478] = 0{,}906$

With the significance limit set at 0,05, the model is accepted, as the probability exceeds the limit.

Annex B
(normative)

Automatic knee detection

B.1 Principle

This procedure detects the presence of any knee by means of calculations performed at each individual temperature.

It is assumed that, for a given temperature and data type, a linear relationship exists between $\log_{10}$ of the hydrostatic stress to which the pipe sample is subjected and $\log_{10}$ of the time to failure of the pipe test pieces. Furthermore, it is assumed that measurements of the time to failure are subject to a random error.

The idea developed in this method is that the data type depends on the value of the hydrostatic stress. Data points are of type B at stresses below the knee value, and of type A at stresses above it.

B.2 Procedure

The model expressing these assumptions and taking into account the data type is written as follows:

$$\log_{10} t = \begin{cases} c_1 + c_{31}(\log_{10}\sigma - \log_{10}\sigma_k) + e_i, \text{ if type A } (\sigma > \sigma_k) \\ c_1 + c_{32}(\log_{10}\sigma - \log_{10}\sigma_k) + e_i, \text{ if type B } (\sigma < \sigma_k) \end{cases}$$

where σ_k is the stress at which the knee occurs, c_1 is the log(time) at that stress, c_{31} and c_{32} are the slopes of the two branches and e_i is the error variable.

NOTE The errors are assumed to be independent and normally distributed with constant variance.

A practical way of fitting this model to the measurements is to scan σ_k over the experimental range of stress values and to calculate the residual sum of squares of the corresponding linear fit and choose the value of σ_k corresponding to the minimum. Statistical software will give the degrees of freedom from this linear fit as N 3, when they should in fact be $N - 4$, since σ_k has been estimated. The correct value of s_k^2 is obtained by dividing the residual sum of squares by $N - 4$.

An F-test is then carried out to compare s_k^2, the residual variance corresponding to the model with a knee, to the residual variance obtained using a model without a knee (s^2). The Fisher statistic F is calculated as:

$$F_{2,N-4} = \{(N\text{-}2)\, s^2 - (N\text{-}4)\, s_k^2\} / 2\, s_k^2$$

This statistic has, under the hypothesis of no knee, an F-distribution with 2 degrees of freedom for the numerator and $N - 4$ degrees of freedom for the denominator, N being the number of measurements.

The hypothesis that no knee was present is accepted at a probability level of 5 % if the probability associated with the calculated value of F is greater than 0,05. Otherwise it is rejected, and the presence of a knee is accepted.

Annex C
(informative)

Application of SEM to stress rupture data of a semi-crystalline polymer

C.1 List of observations

Stress rupture data at 20 °C, 40 °C and 60 °C for a semi-crystalline polymer are included in Tables C.1, C.2 and C.3.

Table C.1 — Stress rupture data at 20 °C

Temperature °C	Stress MPa	Time h	Temperature °C	Stress MPa	Time h
20	15,0	10	20	13,5	411
20	15,0	14	20	13,5	412
20	14,5	32	20	13,5	3 368
20	14,5	24	20	13,5	865
20	14,3	46	20	13,5	946
20	14,1	111	20	13,4	1 220
20	14,0	201	20	13,3	1 112
20	14,0	260	20	13,3	2 108
20	14,0	201	20	13,2	4 524
20	13,9	250	20	13,0	5 137
20	13,7	392	20	13,0	7 651
20	13,7	440	20	12,8	7 760
20	13,7	512	20	12,8	8 240
20	13,7	464	20	12,7	10 837
20	13,7	536			
20	13,6	680			

Table C.2 — Stress rupture data at 40 °C

Temperature °C	Stress MPa	Time h	Temperature °C	Stress MPa	Time h
40	11,1	10	40	10,0	2 076
40	11,2	11	40	10,0	1 698
40	11,5	20	40	9,5	1 238
40	11,5	32	40	9,5	1 790
40	11,5	35	40	9,5	2 165
40	11,5	83	40	9,5	7 823
40	11,2	240	40	9,0	4 128
40	11,2	282	40	9,0	4 448
40	11,0	1 912	40	8,5	7 357
40	11,0	1 856	40	8,5	5 448
40	11,0	1 688	40	8,0	7 233
40	11,0	1 114	40	8,0	5 959
40	10,8	54	40	8,0	12 081
40	10,5	5 686	40	7,5	16 920
40	10,5	921	40	7,5	12 888
40	10,5	1 145	40	7,5	10 578
40	10,5	2 445	40	6,5	12 912
40	10,0	5 448	40	6,0	11 606
40	10,0	3 488			
40	10,0	1 488			

Table C.3 — Stress rupture data at 60 °C

Temperature °C	**Stress** MPa	**Time** h	**Temperature** °C	**Stress** MPa	**Time** h
60	9,6	10	60	7,5	351
60	9,5	13	60	7,0	734
60	9,5	32	60	7,0	901
60	9,5	34	60	7,0	1 071
60	9,5	114	60	7,0	1 513
60	9,5	195	60	6,5	1 042
60	9,2	151	60	6,5	538
60	9,0	242	60	6,0	4 090
60	9,0	476	60	6,0	839
60	9,0	205	60	6,0	800
60	9,0	153	60	5,5	339
60	9,0	288	60	5,5	2 146
60	8,9	191	60	5,5	2 048
60	8,5	331	60	5,5	2 856
60	8,5	296	60	5,0	1 997
60	8,5	249	60	5,0	1 647
60	8,5	321	60	5,0	1 527
60	8,5	344	60	5,0	2 305
60	8,5	423	60	5,0	2 866
60	8,5	686	60	4,0	6 345
60	8,5	513	60	3,5	15 911
60	8,5	585	60	3,4	7 841
60	8,5	719	60	3,4	8 232
60	7,5	423	60	2,9	15 090
60	7,5	590			
60	7,5	439			
60	7,5	519			

C.2 Example of automatic knee detection

This example uses the set of observations given in Table C.2 for 40 °C.

Assume there is no knee and fit a single straight line through all the data points. The residual variance obtained is equal to 0,409 15, with 36 degrees of freedom.

Then assume the presence of a knee and determine its position by scanning 50 stress values regularly spaced over the experimental range of the logarithm of the stress. The value corresponding to the lowest residual sum of squares of the fit (constrained model involving two straight half-lines) occurs at a stress of 10,57 MPa (log stress 1,024), when the estimated time to failure is 1985 h (log time 3,298-). In this case, the residual sum of squares is 7,941 90. The residual variance is thus $s_K^2 = 7{,}941\ 90/34 = 0{,}233\ 6$, with 34 degrees of freedom.

The Fisher statistic used to test for the presence of the knee is equal to 14,528. The probability that the Fisher statistic with 2 and 34 degrees of freedom is greater than 14,528 is 0,000 027 5. As 0,000 027 5 $<$ 0,05, it is decided that a knee is present.

The resulting classification of the data types is given in Table C.4.

Table C.4 — Classification of data types

Temperature °C	Stress MPa	Time h	Data type	Temperature °C	Stress MPa	Time h	Data type
40	11,1	10	Type A	40	10,5	2 445	Type B
40	11,2	11	Type A	40	10,0	5 448	Type B
40	11,5	20	Type A	40	10,0	3 488	Type B
40	11,5	32	Type A	40	10,0	2 076	Type B
40	11,5	35	Type A	40	9,5	1 790	Type B
40	10,8	54	Type A	40	9,5	2 165	Type B
40	11,5	83	Type A	40	9,5	7 823	Type B
40	11,2	240	Type A	40	9,0	4 128	Type B
40	11,2	282	Type A	40	9,0	4 448	Type B
40	11,0	1 688	Type A	40	8,5	7 357	Type B
40	11,0	1 114	Type A	40	8,5	5 448	Type B
40	11,0	1 912	Type A	40	8,0	7 233	Type B
40	11,0	1 856	Type A	40	8,0	5 959	Type B
40	10,5	921	Type B	40	8,0	12 081	Type B
40	10,0	1 488	Type B	40	7,5	16 920	Type B
40	10,0	1 698	Type B	40	7,5	12 888	Type B
40	9,5	1 238	Type B	40	7,5	10 578	Type B
40	10,5	1 145	Type B	40	6,5	12 912	Type B
40	10,5	5 686	Type B	40	6,0	11 606	Type B

NOTE This example of automatic knee detection illustrates the methodology used. The calculation programme presents these results in a different way.

C.3 Regression calculations carried out on stress rupture data

C.3.1 Estimation (see Figure C.1)

C.3.1.1 Model used

As the probability level of the parameter c_3 applying the 4-parameter model is greater than 0,05, the model is reduced to a 3-parameter model.

$$\log_{10} t = c_1 + c_2/T + c_4(\log_{10}\sigma)/T + e$$

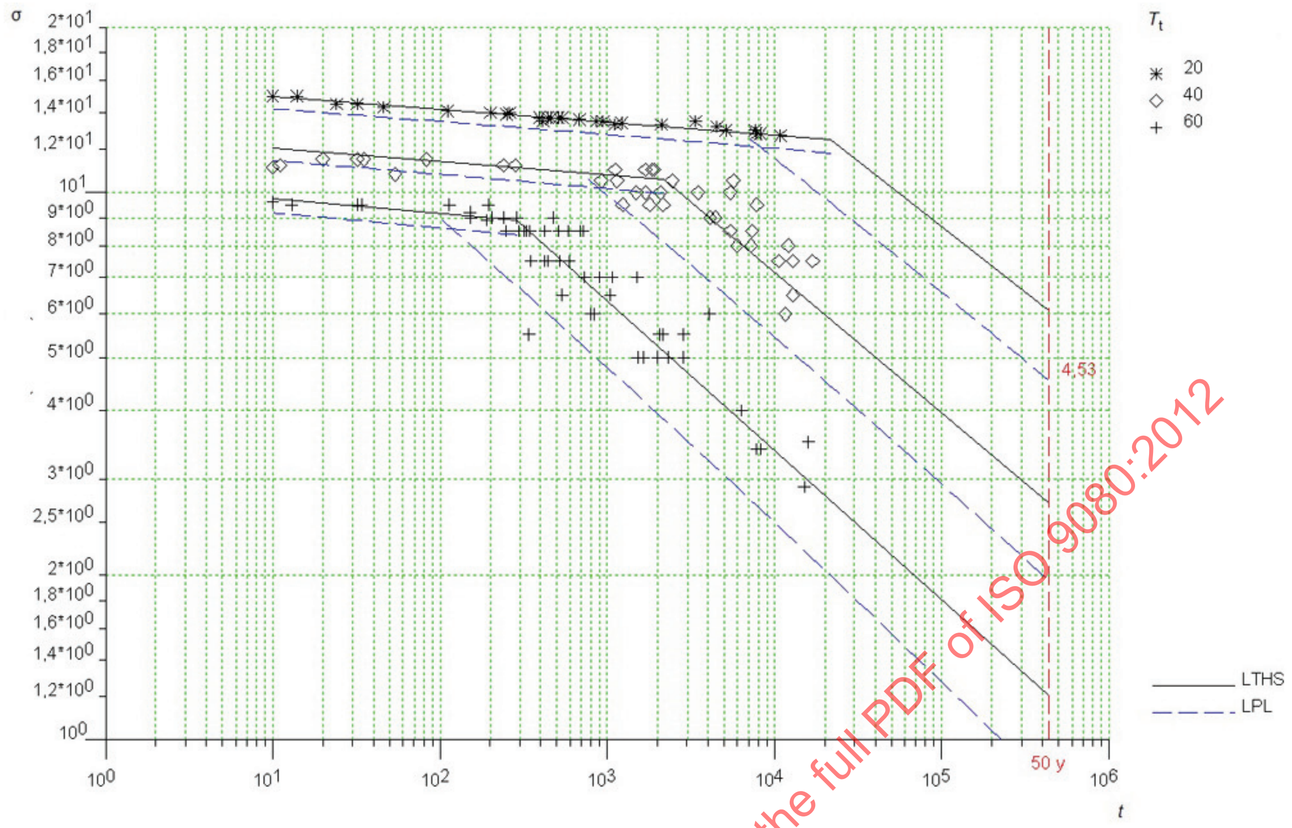
C.3.1.2 Data type A

Residual variance 0,205 909

Number of points 49

Number of parameters 3

Number of degrees of freedom 46



Key

t time to failure (hours)

σ stress (megapascals)

T_t test temperature (°C)

Figure C.1 — Graphical presentation of the results of the SEM analysis for a semi-crystalline polymer as described in this annex

Details of the parameters and statistics for data type A are given in Table C.5 (further details on the statistics are given in Reference [2]).

Table C.5 — Estimated parameters for data type A

Parameter	Value	Standard error	t-value	Pr$(>\|t\|)$
c_1	–57,751	5,297	–10,902	0,000
c_2	32 021,416	2 950,432	10,853	0,000
c_4	–12 596,346	1 255,311	–10,034	0,000

Test of fit of model: Pr[F(19;27) > 3,020] = 0,004

C.3.1.3 Data type B

Residual variance 0,048 195

Number of points 70

Number of parameters 3

Number of degrees of freedom 67

Details of the parameters and statistics for data type B are given in Table C.6.

Table C.6 — Estimated parameters for data type B

Parameter	Value	Standard error	t-*value*	$\Pr(>\lvert t \rvert)$
c_1	–15,794	1,008	–15,673	0,000
c_2	7 239,786	365,424	19,812	0,000
c_4	–1 219,841	76,695	–15,905	0,000

Test of fit of model: Pr[F(20;47) > 0,741] = 0,764

C.3.2 Prediction

C.3.2.1 General

Predicted values of strength are given in Tables C.7, C.8, C.9 and C.10.

The extrapolation time is given in Tables C.11 and C.12.

C.3.2.2 Data type A

Table C.7 — Predicted values of strength for data type A

Temperature °C	Time h					
	1	10	100	1 000	10 000	100 000
	σ_{LTHS} MPa					
20	15,780	14,956	14,176	13,436	12,735	B
40	12,776	12,065	11,394	10,760	B	B
60	10,344	9,733	9,158	B	B	B
	σ_{LPL} confidence level (one-sided) = 0,975 MPa					
20	14,964	14,221	13,488	12,762	12,046	B
40	12,095	11,441	10,796	10,161	B	B
60	9,752	9,178	8,616	B	B	B

Table C.8 — Predicted values of strength for data type A

Temperature °C	Time years			
	0,5	1	10	50
	σ_{LTHS} MPa			
20	12,982	12,774	B	B
40	B	B	B	B
60	B	B	B	B
	σ_{LPL} confidence level (one sided) = 0,975 MPa			
20	12,302	12,087	B	B
40	B	B	B	B
60	B	B	B	B

C.3.2.3 Data type B

Table C.9 — Predicted values of strength for data type B

Temperature °C	Time h					
	1	10	100	1 000	10 000	100 000
	σ_{LTHSL} MPa					
20	A	A	A	A	A	8,668
40	A	A	A	A	7,140	3,954
60	A	A	A	6,343	3,382	1,803
	σ_{LPL} confidence level (one-sided) = 0,975 MPa					
20	A	A	A	A	A	6,569
40	A	A	A	A	5,444	2,934
60	A	A	A	4,787	2,495	1,275

Table C.10 — Predicted values of strength for data type B

Temperature °C	Time years			
	0,5	1	10	50
	σ_{LTHS} MPa			
20	A	A	8,948	6.078
40	8,825	7,387	4,090	2,706
60	4,237	3,506	1,870	1,205
	σ_{LPL} confidence level (one sided) = 0,975 MPa			
20	A	A	6,789	4,533
40	6,762	5,638	3,041	1,955
60	3,160	2,592	1,326	0,822

C.3.2.4 Extrapolation time

Table C.11 — Extrapolation time for T_t = 40 °C, t_{max} = 13 160,5 h

T °C	ΔT °C	k_e	t_e h	t_e years
20	20	6	78 963	9,01

Table C.12 — Extrapolation time for T_t = 60 °C, t_{max} = 9 966,4 h

T °C	ΔT °C	k_e	t_e h	t_e years
20	40	50	498 321	56,89
40	20	6	59 799	6,83

C.3.3 Knee position

The positions of the knees are given in Table C.13.

Table C.13 — Knee positions

Temperature °C	Stress MPa	Time h
20	12,51	21 745
40	10,55	2 183
60	8,90	289

Annex D
(informative)

Application of SEM to stress rupture data of a vinyl chloride based polymer

D.1 List of observations

Stress rupture data at 20 °C, 65 °C, 82 °C and 95 °C for a vinyl chloride based polymer are included in Tables D.1, D.2, D.3 and D.4.

Table D.1 — Stress rupture data at 20 °C

Temperature °C	Stress MPa	Time h	Temperature °C	Stress MPa	Time h
20	42,08	191	20	35,54	2 059
20	42,08	250	20	35,54	3 949
20	40,96	124	20	35,32	21 025
20	40,96	484	20	35,32	3 306
20	39,62	123	20	35,08	10 512
20	39,62	1 409	20	35,08	25 919
20	38,52	2 486	20	34,86	16 864
20	38,52	2 761	20	34,86	6 383
20	37,48	938	20	34,55	15 078
20	37,48	4 402	20	34,55	24 671
20	37,00	5 294	20	33,99	27 177
20	37,00	953	20	33,99	30 229
20	36,33	907	20	33,49	8 947
20	36,33	8 035	20	33,49	36 166
20	35,80	7 710			
20	35,80	12 042			

Table D.2 — Stress rupture data at 65 °C

Temperature °C	Stress MPa	Time h	Temperature °C	Stress MPa	Time h
65	20,48	370	65	17,84	3 876
65	20,48	429	65	17,10	2 377
65	19,93	457	65	17,10	3 383
65	19,93	677	65	16,68	4 417
65	19,58	577	65	16,68	3 893
65	19,58	503	65	16,48	6 543
65	19,14	745	65	16,00	6 373
65	19,14	865	65	16,00	7 862
65	18,93	1 010	65	15,78	9 592
65	18,93	1 542	65	15,78	8 533
65	18,66	1 854	65	15,61	9 095
65	18,36	2 850	65	15,61	10 429

