



## National foreword

This British Standard is the official English language version of EN 50305:2002.

The UK participation in its preparation was entrusted by Technical Committee GEL/20, Electric cables, to Subcommittee GEL/20/12, Railway cables, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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### Summary of pages

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**CENELEC**

European Committee for Electrotechnical Standardization  
Comité Européen de Normalisation Electrotechnique  
Europäisches Komitee für Elektrotechnische Normung

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### Foreword

This European Standard was prepared for the Technical Committee CENELEC TC 20, Electric cables by WG 12, Railway cables, on behalf of the Technical Committee CENELEC TC 9X, Electrical and electronic applications for railways.

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- latest date by which the EN has to be implemented  
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with the EN have to be withdrawn (dow) 2008-07-01

Annexes designated "normative" are part of the body of the standard.

Annexes designated "informative" are given for information only.

In this standard, annexes B and E are normative and annexes A, C and D are informative.

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Contents

<b>Introduction</b> .....	<b>5</b>
<b>1 Scope</b> .....	<b>6</b>
<b>2 Normative references</b> .....	<b>6</b>
<b>3 Definitions</b> .....	<b>7</b>
<b>4 Applicability, sampling, test-piece preparation and test conditions</b> .....	<b>7</b>
4.1 Applicable tests.....	7
4.2 Classification of tests .....	7
4.3 Sampling.....	7
4.4 Test-piece preparation .....	7
4.5 Test conditions.....	7
<b>5 Mechanical tests</b> .....	<b>8</b>
5.1 Impact test at low temperature .....	8
5.2 Abrasion resistance.....	8
5.3 Notch propagation.....	9
5.4 Pliability .....	10
5.5 Strippability and adhesion of insulation.....	11
5.6 Dynamic cut-through .....	11
<b>6 Electrical tests</b> .....	<b>12</b>
6.1 Electrical resistance of conductors .....	12
6.2 Voltage test on completed cable.....	12
6.3 Voltage test on sheath.....	12
6.4 Insulation resistance .....	13
6.5 Spark test .....	13
6.6 Surface resistance .....	14
6.7 D.C. stability .....	14
6.8 Dielectric strength .....	15
<b>7 Ageing and thermal tests</b> .....	<b>15</b>
7.1 Compatibility .....	15
7.2 Long term ageing for insulation .....	15
7.3 Long term ageing for sheath.....	19
7.4 Ozone resistance .....	19
7.5 Pressure test at high temperature .....	21
7.6 Shrinkage test for insulation .....	21
7.7 Stress cracking test.....	21
<b>8 Tests in fluids, including water</b> .....	<b>23</b>
8.1 Mineral and fuel oil resistance .....	23
8.2 Acid and alkali resistance.....	23
8.3 Water absorption of sheath .....	23
<b>9 Reaction to fire tests</b> .....	<b>24</b>
9.1 Flame propagation .....	24
9.2 Toxicity .....	24
<b>10 Miscellaneous tests</b> .....	<b>27</b>
10.1 Durability of marking .....	27
10.2 Blocking of cores.....	27

**Annex A** (informative) **List of other test methods applicable to rolling stock cables .....29**

**Annex B**

## Introduction

The railway industry is generally concerned with the movement of people as well as goods. It is therefore essential that a high level of safety is achieved, even when failures occur which may involve fire, howsoever caused, affecting railway rolling stock.

Hence it is necessary to provide cables for use in railway environments which minimise the hazard to people when a fire may damage the cable, irrespective of whether the fire is caused by an external source or from within the electrical system.

European Standards EN 50264 and EN 50306 specify cables which, in the event of fire will limit risk to people and improve the safety on railways in general. They cover cables based on halogen free materials, for use in railway rolling stock.

EN 50264 covers a range of sheathed and unsheathed cables, with standard wall thickness of insulation, rated at up to 3,6/6 kV with conductor sizes 1,0 mm<sup>2</sup> up to 400 mm<sup>2</sup>.

EN 50306 covers a range of sheathed and unsheathed cables with thin wall insulation, and restricted to a rating of 300 V to earth and a maximum conductor size of 2,5 mm<sup>2</sup>.

This standard EN 50305, gives particular test methods applicable to the cables at present covered by EN 50264 and EN 50306.

## 1 Scope

This standard specifies special test methods applicable to cables, and their constituent insulating and sheathing materials, for use in railway rolling stock. Such cables are specified in the various parts of EN 50264 and EN 50306.

Other test methods required for railway rolling stock cables and their insulating and sheathing materials are listed in Annex A.

## 2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

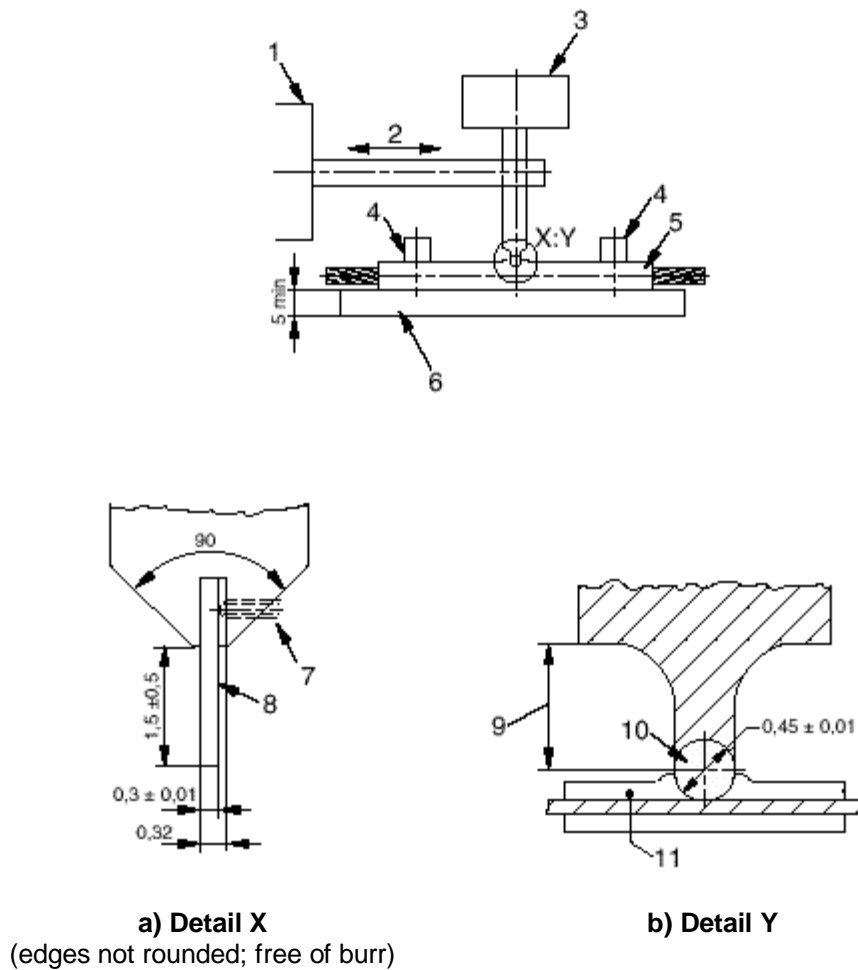
EN 50264-1	Railway applications - Railway rolling stock cables having special fire performance - Standard wall -- Part 1: General requirements
EN 50266-2-4:2001	Common test methods for cables under fire conditions - Test for vertical flame spread of vertically-mounted bunched wires or cables -- Part 2-4: Procedures - Category C
EN 50267-1	Common test methods for cables under fire conditions – Test on gases evolved during combustion of materials from cables -- Part 1: Apparatus
EN 50306-1	Railway applications - Railway rolling stock cables having special fire performance - Thin wall -- Part 1: General requirements
EN 60216-1	Electrical insulating materials - Properties of thermal endurance -- Part 1: Ageing procedures and evaluation of test results (IEC 60216-1)
EN 60811-1-1	Insulating and sheathing materials of electric cables - Common test methods -- Part 1-1: General application - Measurement of thickness and overall dimensions - Tests for determining the mechanical properties (IEC 60811-1-1)
EN 60811-1-2	Insulating and sheathing materials of electric cables - Common test methods -- Part 1-2: General application - Thermal ageing methods (IEC 60811-1-2)
EN 60811-1-3	Insulating and sheathing materials of electric cables - Common test methods -- Part 1-3: General application - Methods for determining the density - Water absorption tests - Shrinkage test (IEC 60811-1-3)
EN 60811-1-4	Insulating and sheathing materials of electric cables - Common test methods -- Part 1-4: General application - Test at low temperature (IEC 60811-1-4)
EN 60811-3-1	Insulating and sheathing materials of electric cables - Common test methods -- Part 3-1: Methods specific to PVC compounds - Pressure test at high temperature - Tests for resistance to cracking (IEC 60811-3-1)
ISO 6349	Gas analysis - Preparation of calibration gas mixtures - Permeation method
ISO 8458-2	Steel wire for mechanical springs -- Part 2: Cold-drawn carbon steel wire



### **3 Definitions**

#### **4.5.4 Pre-conditioning**

The measure of abrasion resistance shall be the average value of the number of cycles in the four tests.



Dimensions in millimetres

**Key**

- |   |                  |    |  |
|---|------------------|----|--|
| 1 | Stroke generator | 7  | Fastening screw                                  |
| 2 | Stroke           | 8  | Blade reamer                                     |
| 3 | Loading weight   | 9  | Shoulder of sufficient depth to clear insulation |
| 4 | Clamp            | 10 | Cutting edge - needle                            |
| 5 | Test specimen    | 11 | Cable under test                                 |
| 6 | Supporting plate |    |  |

**Figure 1 – Test arrangement for abrasion of insulation and sheath**

**5.3 Notch propagation**

Three samples of the cable shall be notched, to a depth of 0,05 mm of the insulation or sheathing, at four points equally spaced with respect to one another around the circumference and 25 mm apart along the length, and in a plane mutually perpendicular to the conductor.

NOTE In the case of 2 core cable, 3 core cable or those cables not substantially circular, the notches should be made at the highest points on the circumference of the cable.

One of the samples shall be conditioned at  $-15\text{ }^{\circ}\text{C}$ , one at ambient temperature and one at  $85\text{ }^{\circ}\text{C}$ , in all cases for 3 h, after which time they shall be wound on to a mandrel,  $(3 \pm 0,3)$  times the minimum specified diameter of the cable, whilst at the conditioning temperature. The notched sample shall be wrapped around the mandrel such that at least one notch is on the outside of the cable.

The sample shall be allowed to return to ambient temperature and then subjected to the voltage test given in 6.2. but at half the rated voltage  $U_0$ .

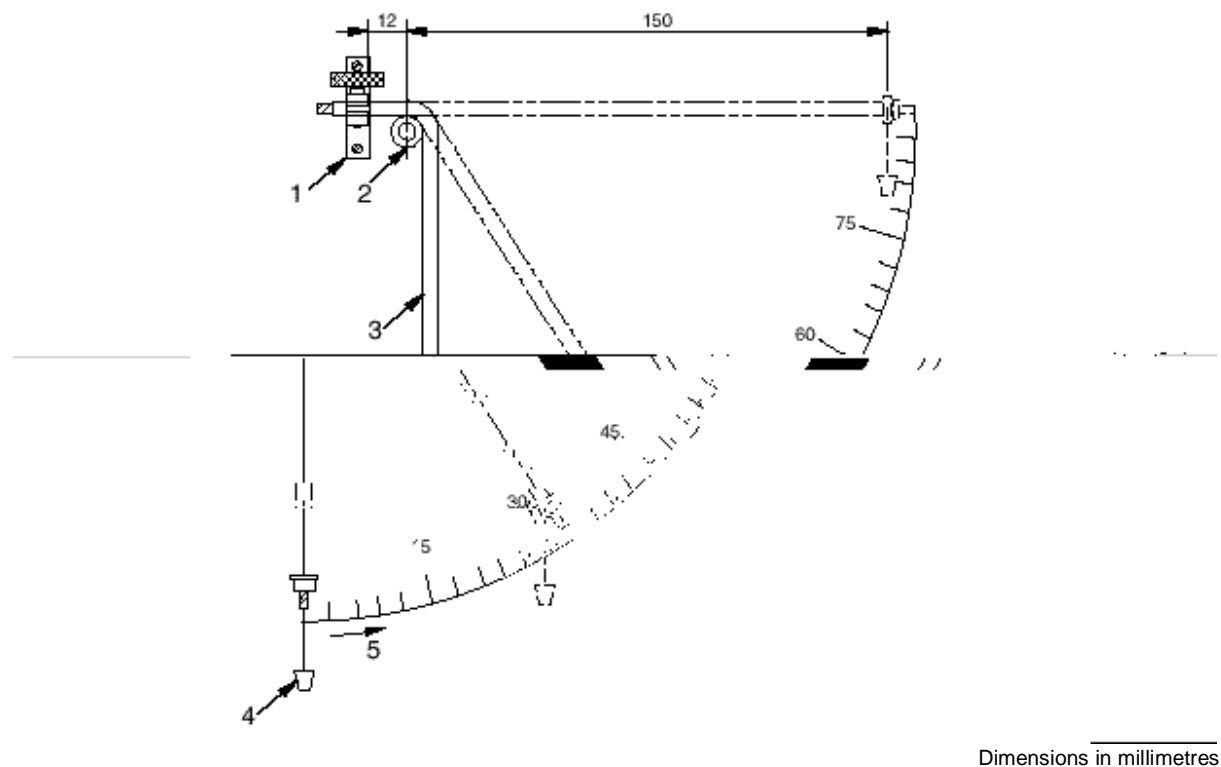
#### 5.4 Pliability

From a single coil of cable cut consecutive test specimen lengths, each of approximately 200 mm.

Suspend each specimen vertically for 24 h in an oven with a mass attached to its free end. The applied mass and oven temperature shall be as stated in the cable specification. Immediately after removal from the oven, store the specimens at the temperature, relative humidity and period of time specified in the cable specification.

Test each specimen using the test rig shown in Figure 2; the diameter of the mandrel in the test rig shall be as the minimum bend diameter unless specified in the cable specification. Gradually apply a mass to the cable, at the position shown in Figure 2, sufficient to bend the cable downwards through  $(90 \pm 1)^{\circ}$ .

Ensure that the specimen remains in this position for 5 min and record the mass. After this time, remove the mass and allow the specimen to recoil towards its original position. At a time 5 min after removal of the mass, record the recoil distance.



#### Key

- |   |                |
|---|----------------|
| 1 | Clamp          |
| 2 | Mandrel        |
| 3 | Test specimen  |
| 4 | Mass container |
| 5 | Recoil         |

Figure 2 – Pliability test rig

## 5.5 Strippability and adhesion of insulation

### 5.5.1 Strippability

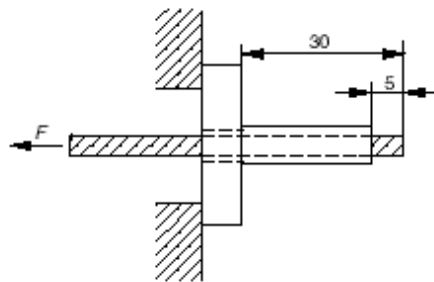
Stripping of 5 mm of insulation from each end of a 50 mm sample shall be easily carried out with normal stripping pliers.

### 5.5.2 Adhesion

Three test specimens, each of 50 mm length, shall be cut at regular intervals from a test sample of 3 m of core or cable.

On each specimen the insulation shall be cut 5 mm and 30 mm from one end. The insulation shall be stripped from the cuts to each end, so that insulation is left intact in-between the two cuts. The core shall then be passed through a calibrated hole the diameter of which is that of the core + 0,05 mm (see Figure 3).

Using a pulling speed of  $(100 \pm 10)$  mm/min a force shall be applied to the conductor until it slips inside the insulation. The force (F) required to produce the slippage shall be recorded.



Dimensions in millimetres

Figure 3 – Assembly for adhesion test

## 5.6 Dynamic cut-through

A tensile tester (or equivalent apparatus) shall be operated in a compression mode and shall be equipped with a means to record the force necessary to drive the needle cutting edge (see Figure 1 b)) through the insulation or sheath of a finished sample of cable. A low voltage detection circuit, designed to stop the tester when the edge cuts through the cable insulation or sheath and contacts the conductor or electrical screen, shall be attached.

Carry out the test at the temperature specified in the individual cable specification. The force on the cutting edge driving it through the insulation or sheath shall be increased at the constant rate as specified in the product standard until contact with the conductor or metallic screen occurs. Perform four tests on each test sample, and record the force measured at electrical contact. Move the sample forward a minimum of 25 mm and rotate 90° clockwise between each test.

NOTE In the case of 2 core cables, 3 core cable or those cables not substantially circular, the cutting edge should be applied to the highest points on the circumference of the cable.

The average of the four results shall not be less than the specified minimum.

## 6 Electrical tests

### 6.1 Electrical resistance of conductors

The electrical resistance of each conductor shall be measured on a sample of cable of at least 1 m in length, and the length of this sample shall be measured.

If necessary a correction to 20 °C and to a length of 1 km shall be obtained by the formula:

$$R_{20} = R_t \times \frac{254,5}{234,5+t} \times \frac{1\,000}{L}$$

where

- t = temperature of the sample at the moment of measurement, in degrees Celsius;
- R<sub>20</sub> = resistance at 20 °C, in ohm/kilometre;
- R<sub>t</sub> = resistance of L metres of cable at t °C in ohm;
- L = length of the sample of cable, in metres (length of the complete sample and not of the individual cores or wires)

The measured resistance shall not exceed the value in the particular standard.

### 6.2 Voltage test on completed cable

#### 6.2.1 Cable without metallic layer

If the cable has no metallic layer, a sample of the cable as delivered shall be immersed in water for a minimum period of 1 h. The length of the sample, the temperature of the water and the duration of application of voltage shall be as given in the cable specification.

A voltage shall be applied in turn between each conductor and all the others connected together and to the water.

#### 6.2.2 Cable with one or more metallic layers

- a) If the cable has a metallic layer, a sample of the cable shall be taken of the length defined in the cable specification.

A voltage shall be applied in turn between each conductor and all the others connected together and to the metallic layer.

The voltage and the duration of its application are given for each case in the cable specification.

- b) If the cable has more than one screened and sheathed unit a sample of the complete cable shall be taken of the length defined in the cable specification.

A voltage shall be applied in turn between each screen and all other screens and conductors connected together.

The voltage and the duration of its application are given for each case in the cable specification.

### 6.3 Voltage test on sheath

The test shall be made on sheathed cable where there is a metallic screen or braid under the sheath.

A length of complete cable shall be immersed in water, and an a.c. or a d.c. voltage applied between the metallic screen or braid and the water.

The sample length, test temperature, voltage level and duration of its application shall be as given in the particular cable standard.

## **6.4 Insulation resistance**

### **6.4.1 Test at ambient temperature**

The test shall be made on the core samples, 5 m long, previously submitted to the test described in 6.2.1.

The sample shall be immersed in water at ambient temperature; a length about 0,25 m at each end of the sample being kept above the water. The duration of immersion shall be a minimum of 1 h.

A d.c. voltage of between 80 V and 500 V shall be then applied between the conductor and the water.

The insulation resistance shall be measured one minute after application of the voltage and this value shall be corrected to 20 °C and related to 1 km.

### **6.4.2 Test at elevated temperature**

The test shall be made on the core samples, 5 m long.

The sample shall be immersed in water at the temperature specified in 6.4.1. The duration of immersion shall be a minimum of 1 h.

## **6.6 Surface resistance**

The test specimen shall be prepared as follows.



NOTE A period of stability is, for instance, less than 10 % increase in current leakage averaged over any 24 h period (this is subject to review in the light of practical experience).

On completion of the immersion, the cables shall be removed from the salt solution and subjected to the voltage test specified in 6.2 except that the test voltage shall be the rated voltage (U) of the cable, unless otherwise specified.

## **6.8 Dielectric strength**

5 m of cable sample shall be immersed in water for a period as given in the cable standard, maintained at a temperature of  $(20 \pm 5)$  °C with at least additional lengths of 150 mm protruding at each end. The voltage test specified in 6.2 shall be applied for 1 min between the conductor and the water. The test voltage shall be as specified in the cable specification. Immediately afterwards the voltage shall be increased by 500 V every 30 s until a disruptive discharge occurs.

The recorded value shall exceed the specified minimum.

## **7 Ageing and thermal tests**

### **7.1 Compatibility**

Samples of completed cables shall be aged in accordance with the time and temperatures given in the appropriate cable specification. The testing shall be in accordance with 8.1.4 of EN 60811-1-2 for single layer. For multi-layered insulating systems special techniques are given in the particular cable standard (see 4.4).

### **7.2 Long term ageing for insulation**

#### **7.2.1 General**

This test method provides a standard test and procedure for determining the 20 000 h life versus temperature curve for insulating materials.

Stabilising weights, each with a hook, shall be provided for holding insulated wire specimens straight in the oven during ageing. The appropriate weight size is about one-half of the wrapping test weight shown in Table 4. It is suggested that this weight also has a hook on the bottom so that the additional weight required for the mandrel wrap can be added without removing the stabilising weight.

## **7.2.4 Method**

### **7.2.4.1 Test specimens**

Each specimen shall be a 300 mm to 400 mm length of insulated wire whose insulation is free from visible imperfections. It is convenient to strip approximately 6 mm of insulation at each end and apply a lug from which the weights can be suspended. The lug shall be of a type that not only contacts the conductor but also clamps the insulation to prevent shrinkage with temperature exposure.

### **7.2.4.2 Temperature selection and exposure**

The selection of temperatures for test shall be achieved by adding 20 °C to the expected end-of-life temperature for the lowest temperature and two further temperatures at 10 °C to 20 °C successive steps. If the average life at the highest test temperature is found to be less than 100 h, too high a test temperature has been selected and the data should be discarded. The test should be repeated at a lower temperature.

Extrapolation to a temperature should not exceed 25 °C below the lowest ageing test temperature. If extrapolation beyond 25 °C is required, an additional series of tests shall be made at an even lower temperature. If, in addition, the average life found at the lowest test temperature is less than 5 000 h, tests shall be made at lower temperatures until at least 5 000 h average life data are achieved.

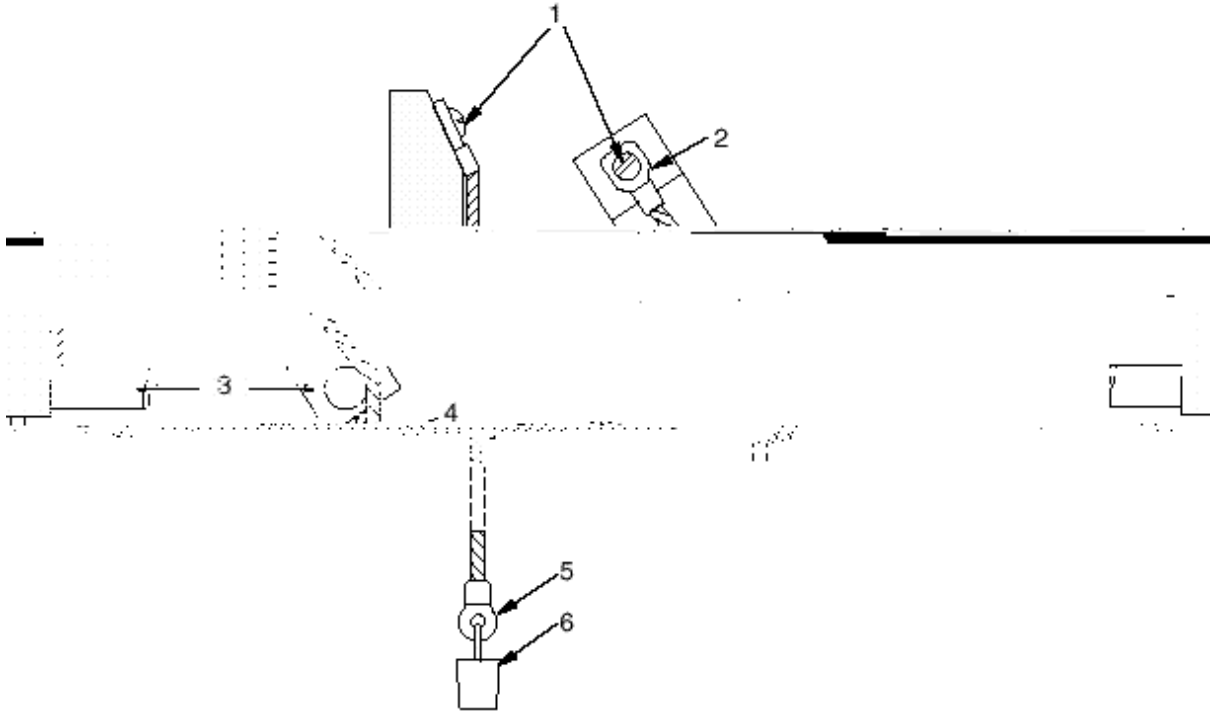
The average life of the specimen may be affected by the number of cycles; therefore to maintain a consistency in the procedure that will ensure a reliable degree of reproducibility, make an effort to expose each to an average of not less than eight cycles and not more than fifteen cycles. A first estimate of cycle time is given in Table 3. This table provides a selection of the days per cycle and the recommended ageing temperatures for cables having 20 000 h thermal endurance temperatures ranging from 105 °C to 180 °C. This range could be extended easily if necessary. During the course of the test, increase or decrease the length of the remaining cycles if necessary.

Make a quick estimate of the highest test temperature by running cycles of approximately one day in length at 80 °C to 100 °C above the normal rating temperature of the cable or at a point just below the melting point of the insulation if it is within this range.

**Table 3 – Recommended exposure times in days per cycle**

Ageing temperature <sup>a</sup> °C	Duration of exposure in days for an estimated temperature value (°C) of:			
	105	130	155	180
250				1
240				2
230				4
220			1	7
210			2	14
200		1	4	28
190		2	7	49
180	1	4	14	
170	2	7	28	
160	4	14	49	
150	7	28		
140	14	49		
130	28			
120	49			

<sup>a</sup> Tolerances are given in Table 1.

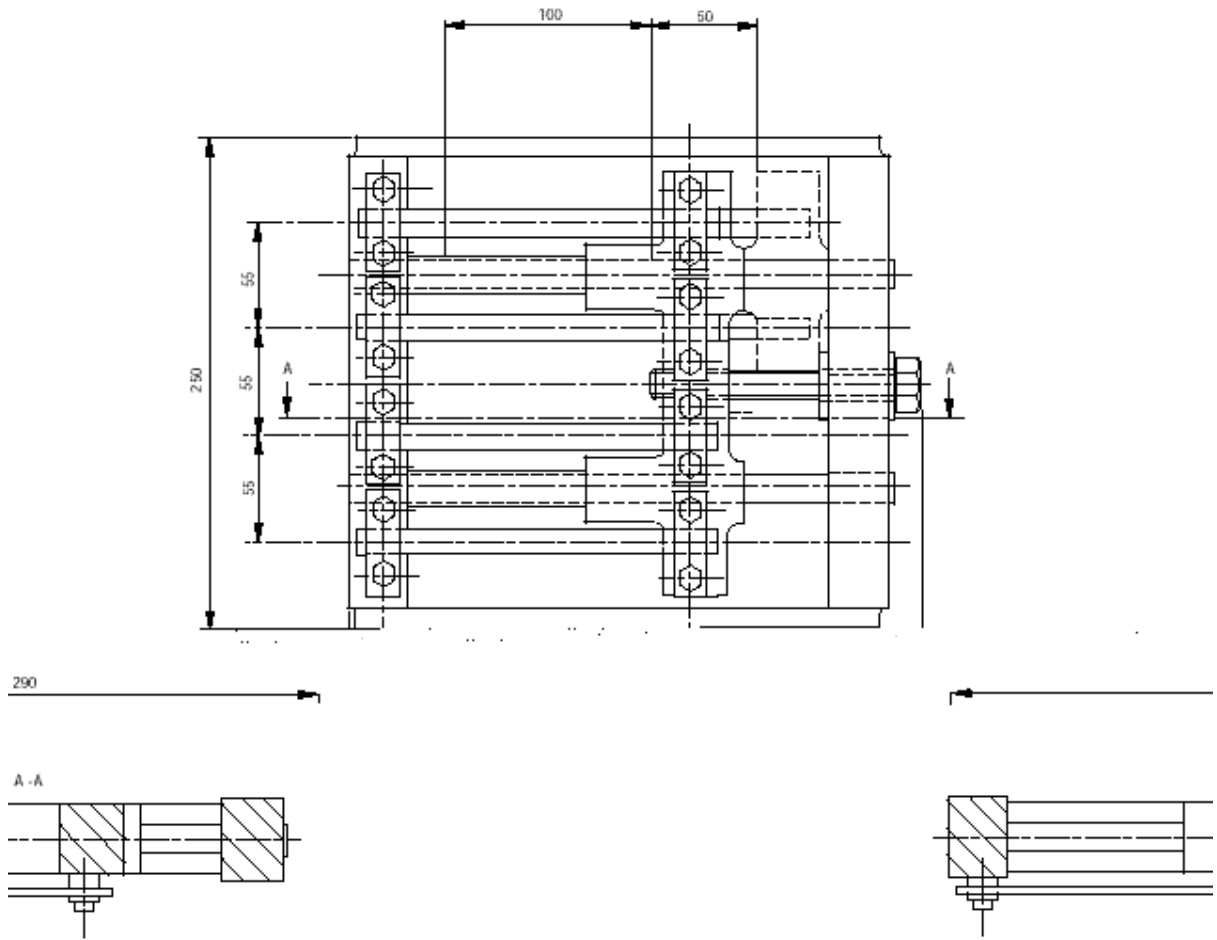


**Key**

- |   |                              |   |               |
|---|------------------------------|---|---------------|
| 1 | Set screw                    | 4 | Test specimen |
| 2 | Lug type solderless terminal | 5 | Eyelet        |
| 3 | Mandrel                      | 6 | Mass          |

**Figure 5 – Suggested method of attachment of insulated wire test specimen to mandrel**





Dimensions in millimetres

**Figure 6 – Clamping device**

Test pieces shall be clamped on both ends in the clamping device according to 7.4.2.1 a) in such a way as to obtain a free length between the clamps of 100 mm. Subsequently the test pieces shall be elongated by  $(33 \pm 2) \%$ .

NOTE To avoid possible ozone cracks near the clamps the test pieces may be covered locally by a suitable resistant lacquer.

**7.4.2.4 Test procedure**

The required number of test pieces prepared according to 7.4.2.3 shall be placed substantially in accordance with 7.4.2.1 a) and b).



Dimensions in millimetres

### **Figure 7 – Flat topped cone**

Suitable fixings may be added to the basic shape to allow the sample to be fixed conveniently at both ends.

#### **7.7.3 Determination of the 168 h thermal ageing test temperature**

To obtain the stress cracking ageing test temperature referred to in 7.7.1 the least squares best fit straight line regression curve of the Arrhenius plot obtained from the long term ageing test (7.2 for insulation or 7.3 for sheath) is drawn (refer to Annex D as an example).



The cable shall be aged at the selected temperature (see 7.7.3) and then allowed to cool to ambient temperature. The cable shall then be examined, without disturbance, for cracks.

The cable sample from the cone shall be unwound, straightened and again inspected for cracks. At intervals along the cable, so that a test occurs within each original complete turn on the cone mandrel, the cable shall be bent, for one complete turn, around a cylindrical metal mandrel of diameter  $(2 \pm 0,2)$  times the minimum specified diameter of the cable and further examined for cracks using four times magnification.

The cable sample shall then be subjected to the voltage test specified in 6.2, at 1,5 kV a.c. for 1 min. During this test the whole cable sample length previously in contact with the cone mandrel shall be completely immersed in the water.

## **8 Tests in fluids, including water**

### **8.1 Mineral and fuel oil resistance**

The test sample shall be wound on to a cylindrical mandrel of diameter  $(10 \pm 1,0)$  times the minimum diameter of the cable.

The assembly shall be subjected to oil immersion, using the type of oil or fuel and immersion

## 9 Reaction to fire tests

### 9.1 Flame propagation

#### 9.1.1 Cables with overall diameter greater than 6 mm and less than 12 mm

The test shall be carried out as for EN 50266-2-4 except that the nominal total volume of non-metallic material (NMV) shall be 0,5 l/m.

Cable mounting shall be in one (or more) layer(s) up to a maximum of 300 mm width on the 500 mm width ladder.

#### 9.1.2 Cables with overall diameter not greater than 6 mm

The test shall be carried out as for EN 50266-2-4 except that the ladder loading shall be bundles of cable of approximate diameter 20 mm spaced by half the bundle diameter, and:

- a) a minimum of two bundles shall be tested;
- b) the number of bundles shall be determined as that necessary to give a nominal total volume of non-metallic material (NMV) of 0,5 l/m subject to a) above;
- c) the bundle length shall be 2,5 m;
- d) the bundle formation shall be as given below;
- e) the number of bundles tested and NMV of each shall be recorded;
- f) the cable to be tested shall be selected such that the total volume of non-metallic material (NMV) in the bundles to be tested is  $> 0,4$  l/m and  $\leq 0,6$  l/m.

The number of cables in each bundle shall be as follows :

Cable diameter (d) mm	Number of cables in bundle
$d \leq 3,3$	37
$3,3 < d \leq 4,3$	19
$4,3 < d \leq 6,0$	12

## 9.2.1 Qualitative analysis for nitrogen and sulfur using molten sodium

### 9.2.1.1 Preparation of test solution

Place about 0,1 g of the finely divided sample with a pea-sized piece of metallic sodium in a test tube. Heat to red heat and then quench the tube in about 10 ml of water in a beaker, with all the necessary precautions taken to avoid projectiles and splinters. The test tube breaks and the soluble material dissolves. The resulting product is filtered in order to obtain a clear solution, which is divided into two equal portions.

### 9.2.1.2 Evaluation

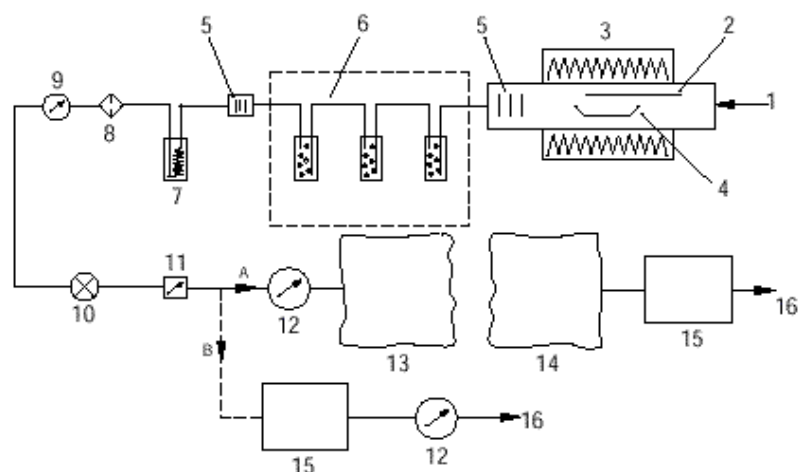
#### Nitrogen

Add a few cm<sup>3</sup> of a 10 % ferrous sulfate solution to an aliquot portion of the initial solution:

- in the presence of sulfur, black iron sulphide is formed;
- in the absence of sulfur, dark green ferrous hydroxide,  $\text{Fe}(\text{OH})_2$

### 9.2.2.2 Apparatus

A schematic diagram of the apparatus, including arrangements for continuous analysis (without gas bags) or discontinuous analysis (with gas bags), is shown in Figure 8.



#### Key

- |   |   |    |  |
|---|---|----|--|
| 1 | Dry air   | 9  | Needle valve   |
| 2 | Temperature probe   | 10 | Compression air blower                                 |
| 3 | Electric tube furnace   | 11 | Flow meter   |
| 4 | Combustion boat with sample                                       | 12 | Dry gas meter (SO <sub>2</sub> – continuous mode only) |
| 5 | Silica wool   | 13 | Gas bag 1  |
| 6 | Bubbling line (only for HCN/NO <sub>x</sub> and SO <sub>2</sub> ) | 14 | Gas bag 2  |
| 7 | Cold trap   | 15 | CO/CO <sub>2</sub> analyser                            |
| 8 | Dust filter   | 16 | External outlet  |
| A | Route for discontinuous analysis                                  | B  | Route for continuous analysis                          |



The assembly shall be supported horizontally in an oven preheated to the temperature specified, and shall remain in the oven for the specified time.

At the end of the specified time remove the assembly from the oven and leave it to cool for at least 1 h. The core shall be unwound from the mandrel and the turns shall easily separate from the mandrel and each other, without damage to their outer layer.

NOTE Discolouration of the core may be ignored.

**Annex A**  
(informative)

**List of other test methods applicable to rolling stock cables**

<b>Ref</b>	<b>Test method</b>	<b>Standard</b>
A.1	Measurement of dimensions	EN 60811-1-1
A.2	Tensile strength and elongation at break	EN 60811-1-1
A.3	Air oven ageing	EN 60811-1-2
A.4	Water absorption (gravimetric)	EN 60811-1-3
A.5	Tests at low temperature	EN 60811-1-4
A.6	Mineral oil test	EN 60811-2-1 <sup>a</sup>
A.7	Hot set test	EN 60811-2-1
A.8	Ozone resistance	EN 60811-2-1
A.9	Pressure test at high temperature	EN 60811-3-1
A.10	Flame propagation (single cable or core)	EN 50265-2-1
A.11	Flame propagation (bunched cables)	EN 50266-2-4
A.12	Acid and corrosive gas emission	EN 50267-2-1 and EN 50267-2-2
A.13	Smoke emission	EN 50268-2
A.14	Fluorine content	EN 60684-2
A.15	Elongation of conductors	EN 10002-1
A.16	Marking or cores by numbers	EN 50334
A.17	Electrical resistance of conductors	HD 383
A.18	Methods for the analysis of gases by the absorption of an undispersed infrared radiation beam, published by AFNOR	Booklet NF X 20-301 - January 1978
<sup>a</sup> This method is also used for acid and alkali resistance, and for fuel resistance, but using other specified fluids.		

## Annex B (normative)

### Procedure for checking the efficacy of the method of spark testing (with reference to 6.5)

#### B.1 Object

The object of this procedure is to standardise the method by which manufacturers may demonstrate that their spark testing method is effective in detecting faults in the insulation as specified in 6.5.1.

The manufacturer's instructions for production and control procedures shall provide that cables for which spark testing is required shall be effectively tested in practice.

#### B.2 Procedure

**B.2.1** Manufacturers should have available two test-lengths of cores which have been specially prepared. One of the cores should have the smallest insulation thickness for the relevant types of cable, the other core should have the largest insulation thickness for the relevant types of cable.

**B.2.2** The preparation of the punctures in the insulation shall be effected as follows.

- The insulation shall be removed from the core for a length of about 5 times the nominal insulation thickness.
- From the piece of insulation which has been removed, a segment of about 30° shall be removed; the remaining piece of the insulation shall then be replaced on the conductor (see Figure B.1).

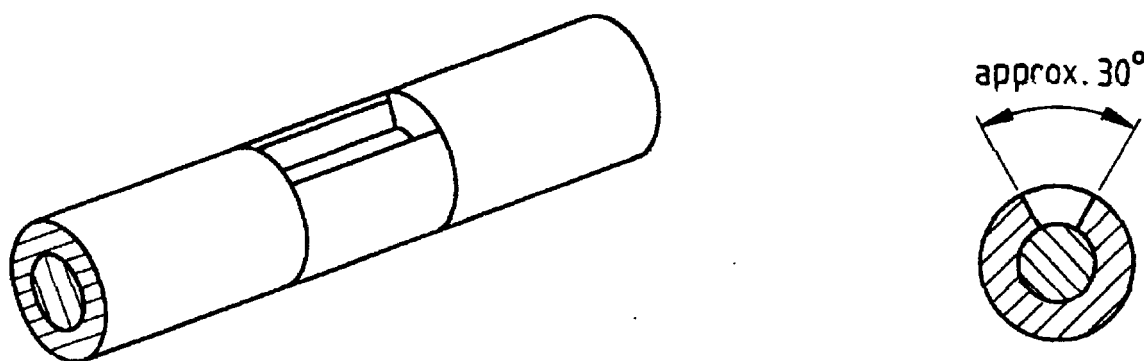
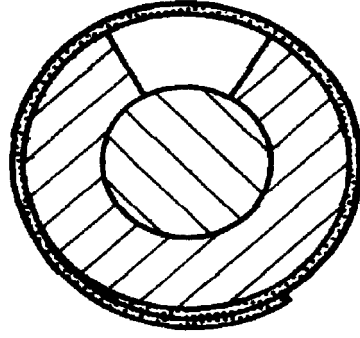


Figure B.1 – Removal of insulation segment

- Over the replaced piece of the insulation, one layer of adhering tape (e.g. Polyethylene terephthalate) shall be placed in a longitudinal direction, with an overlap. This overlap shall be situated on the opposite side of the core to the position where the insulation was removed (see Figure B.2).





**Annex C**  
(informative)

**Long term ageing test – Significance and use**

The chemical changes that degrade the physical and electrical properties of insulation on wire are accelerated when the insulated wire is exposed to elevated operating temperatures.

This test method can be used to determine the relative effects of different temperatures on the life of a given insulating system or to compare different insulating systems at a given temperature.

The times to failure in this test cannot be quantitatively related to the life of insulating materials in actual service, but do provide an indication of such life under the specific parameters of the test. The test results of these shorter time tests at higher temperatures can be extrapolated to longer times at



**Annex E**  
(normative)

**Analysis methods for toxicity**

**E.1 Continuous analysis methods**

**E.1.1 Oxides of carbon**

The necessary appliances are

- a carbon monoxide infrared analyser with several measuring ranges, for example, from 0 % to 10 %,
- a carbon dioxide infrared analyser with several ranges covering the concentrations from 0 % to 20 %.

NOTE The principle and the description of these appliances is dealt with in NF X 20-301 - January 1978 - Methods for the analysis of gases by the absorption of an undispersed infrared radiation beam.

These determinations cover only the oxides of carbon, CO and CO<sub>2</sub>. The gas mixture, after having passed through the cold trap to remove the water vapour excess, is then analysed by means of two series connected infrared analysers.

**- Determination of the carbon monoxide (CO)**

The integration curve gives the ratio  $X_1$  of CO contained in the gas of volume  $V_1$  recorded by the dry gas meter placed in the analysis circuit.

The amount of CO evolved from the test specimen is, in milligrams:

$$\text{CO (mg)} = 1\,000 \cdot X_1 V_1 \cdot (M/V_m^{25})$$

where

$V_1$  is in dm<sup>3</sup>

$X_1$  is the vol/vol ratio of CO in the gas sample

$M$  = molar mass of CO (28 g)

$V_m^{25}$  = molar volume of CO at the temperature  $T = 25\text{ °C}$  (298 K)

$$V_m^{25} = V_m^0 T/T_0$$

where

$V_m^0$  = molar volume at  $T_0 = 0\text{ °C}$  (273 K), that is 22,4 dm<sup>3</sup> and

$T/T_0 = 298/273$

that is

$$V_m^{25} = 22,4 \frac{298}{273} = 24,45 \text{ dm}^3$$

Therefore

$$\text{CO (mg)} = 1\,000 \cdot X_1 V_1 \cdot (28/24,45)$$

i.e.  $\text{CO (mg)} = 1\,145 \cdot X_1 V_1$

**– Determination of the carbon dioxide (CO<sub>2</sub>)**

#### **E.1.2.4 Solution of gases**

At the end of the combustion process, the volumes of solutions in which the sulfur dioxide was dissolved are combined. After filtering, the trapping solution in which the silica wool wadding plug was rinsed is added. The volume is adjusted to a volume V, with the trapping reagent.

The solution contains the total amount of sulfur dioxide which should be detected by colorimeter.

#### **E.1.2.5 Colorimetric titration analysis**

The use of a permeation bench for gas calibration is described in ISO 6349 (see Figure 5) which represents the arrangement to be made including

- a compressed dry nitrogen bottle used as carrier gas,
- a ball flowmeter,
- a water bath controlled to  $\pm 0,1$  °C,
- a U-shaped tube filled with glass balls on which the permeation tube stands,
- a thermometer placed in the emergent portion of the U-shaped tube in which the gas flow is effected,
- a gas outlet by flexible tube to which a glass tapered tube (used for bubbling in a beaker containing the trapping solution) may be adjusted.

### **E.1.3 Materials containing nitrogen**

#### **E.1.3.1 General**

The pyrolysis of the organic products containing nitrogen gives variable quantities of hydrogen cyanide and of the nitrogen oxides. The determination of the gases requires two combustions, one for oxides of carbon (see E.1.1) the second for hydrogen cyanide and nitrogen oxides.

A quantitative estimate of the nitrogen oxides may be carried out, by means of detectors, only in a discontinuous procedure, see E.2.3.

#### **E.1.3.2 Determination of the hydrogen cyanide**

Spectrophotometric titration: the picrate method.

NOTE This method is applicable if the amounts of hydrogen cyanide are above 0,3 mg. The dimedone method allows

### **E.2.1 Determination of oxides of carbon using infrared analysers.**

The necessary appliances are

- a carbon monoxide infrared analyser with several measuring ranges, for example, from 0 % to 10 %, and
- a carbon dioxide infrared analyser with several ranges covering the concentrations from 0 % to 20 %.

NOTE The principle and the description of these appliances is dealt with in NF X 20-301 - January 1978 - Methods for the analysis of gases by the absorption of an undispersed infrared radiation beam.

These determinations cover only the oxides of carbon, CO and CO<sub>2</sub>. The gas mixture, after having passed through the cold trap to remove the water vapour excess, is picked up in the gas bags, then analysed by means of two series connected infrared analysers (see E.1.1).

#### **– Determination of the carbon monoxide (CO)**

See E.1.1.

#### **– Determination of the carbon dioxide (CO<sub>2</sub>)**

See E.1.1.

#### **– Correction due to the amounts of carbon monoxide and dioxide in the atmosphere if the air used is not synthetic air**

A determination without a sample indicates the amounts of CO and CO<sub>2</sub> contained in the ambient air, to be deducted from the determined values with sample.

### **E.2.2 Determination of sulfur dioxide with a detector tube**

The gases are collected in a gas bag until the combustion of the test specimen is completed. No bubblers are used because sulfur dioxide is very soluble in water.

An appropriate detector tube is fitted on to the gas bag.

The following sensitivities are commonly used:

- 1 ppm to 200 ppm;
- 50 ppm to 8 000 ppm.

If the amount of sulfur in the composition of the material is high, a reduction of the sample from 1 g to 0,5 g may be necessary.

### **E.2.3 Determination of nitrogen oxides with a detector tube**

The gases are collected in a gas bag until the combustion of the test specimen is completed.

An appropriate detector tube is fitted on to the gas bag.

The following sensitivities are commonly used:

- 0,5 to 10 ×





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