BRITISH STANDARD

Railway applications — Railway rolling stock cables having special fire performance — Test methods

The European Standard EN 50305:2002 has the status of a British Standard

ICS 13.220.20; 29.060.20; 45.060.01

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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Railway applications - Railway rolling stock cables having special fire performance - Test methods

Applications ferroviaires - Câbles pour matériel roulant ferroviaire ayant des performances particulières de comportement au feu - Méthodes d'essais

Bahnanwendungen - Kabel und Leitungen für Schienenfahrzeuge mit verbessertem Verhalten im Brandfall - Prüfverfahren

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CENELEC

European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
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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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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.

Contents

Introduction **5**

1 Scope **6**

2 Normative references **6**

3 Definitions **7**

4 Applicability, sampling, test-piece preparation and test conditions **7**

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

5 Mechanical tests **8**

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

6 Electrical tests **12**

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

7 Ageing and thermal tests **15**

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

8 Tests in fluids, including water **23**

8.1 Mineral and fuel oil resistance 23
8.2 Acid and alkali resistance 23
8.3 Water absorption of sheath 23

9 Reaction to fire tests **24**

9.1 Flame propagation 24
9.2 Toxicity 24

10 Miscellaneous tests **27**

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 (normative) **Procedure for checking the efficacy of the method of spark testing** (with reference to 6.5) **30**

Annex C (informative) **Long term ageing test – Significance and use** **32**

Annex D (informative) **Illustration of an Arrhenius plot** **33**

Annex E (normative) **Analysis methods for toxicity** **34**

Figure 1 – Test arrangement for abrasion of insulation and sheath 9
Figure 2 – Pliability test rig 10
Figure 3 – Assembly for adhesion test 11
Figure 4 – Arrangement of electrodes for test sample 14
Figure 5 – Suggested method of attachment of insulated wire test specimen to mandrel 18
Figure 6 – Clamping device 20
Figure 7 – Flat topped cone 22
Figure 8 – Schematic diagram of apparatus for production, collection and analysis of gases 26
Figure B.1 – Removal of insulation segment 30
Figure B.2 – Overlap position for tape 31

Table 1 – Tolerances for temperature values 7
Table 2 – Parameters for impact test at low temperature 8
Table 3 – Recommended exposure times in days per cycle 17
Table 4 – Requirements for wrapping test 17
Table 5 – Determination method to be applied 25
Table 6 – CC_z values for various gases 27

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^2 up to 400 mm^2.

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^2.

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

The definitions given in EN 50264-1 and EN 50306-1, shall apply to this standard.

4 Applicability, sampling, test-piece preparation and test conditions

4.1 Applicable tests

Tests applicable to each type of cable are given in the particular cable standard.

4.2 Classification of tests

The classification of tests is given in the general requirements of the relevant cable standard.

4.3 Sampling

The size and number of samples for each particular test is given either in this EN or the relevant cable standard.

4.4 Test-piece preparation

The preparation of test pieces shall be as described in the particular test method or in the cable standard.

NOTE Attention is drawn to the fact that some insulation systems used for railway cables are composites (multilayer). In such cases special preparation techniques and requirements are given in the particular cable standard.

4.5 Test conditions

4.5.1 Ambient temperature

Tests shall be made at an ambient temperature within the range 5 °C to 35 °C, unless otherwise specified in the details for the particular test.

4.5.2 Tolerance on temperature values

The tolerances which shall apply to the temperature values are given in Table 1.

Table 1 – Tolerances for temperature values

Specified temperature (T) °C	Tolerance °C
$-40 \leq T \leq 0$	± 2
$0 < T \leq 50$	According to relevant clause
$50 < T \leq 150$	± 2
$T > 150$	± 3

4.5.3 Frequency and waveform of power frequency test voltages

Unless otherwise specified, the test voltage shall be a.c. 49 Hz to 61 Hz of approximately sine-wave form; the ratio peak value/r.m.s. value being equal to $\sqrt{2}$ with a tolerance of ± 7 %.

The values quoted are r.m.s. values.

4.5.4 Pre-conditioning

Unless otherwise stated the tests shall be carried out not less than 16 h after the extrusion or cross-linking, if any, of the insulating or sheathing compounds.

5 Mechanical tests

5.1 Impact test at low temperature

The impact test in accordance with 8.5 of EN 60811-1-4 shall be used except that the mass of hammer, intermediate test piece and height of drop shall be as given in Table 2.

Table 2 – Parameters for impact test at low temperature

Cable diameter (D) mm	Mass of hammer g	Mass of intermediate test piece g	Height of drop mm
D ≤ 15	1 000	200	100
15 < D ≤ 25	1 500	200	150
D > 25	2 000	200	200

The inside and outside of the sheath and the insulation of unsheathed cables shall then be examined with normal or corrected vision, without magnification. The insulation of sheathed cables shall be examined on the outside only.

5.2 Abrasion resistance

The test shall be carried out at a temperature of (20 ± 5) °C, using a machine similar to that shown in Figure 1.

The cutting edge shall be either a polished steel spring wire needle of (0,45 ± 0,01) mm diameter, and of material according to ISO 8458-2, held in a suitable support (Figure 1 b)), or a rectangular shaped steel blade (Figure 1 a)) mounted at 90° to the axis of the cable. The setting shall be arranged so as to wear the surface of the core or cable lengthwise over a distance of 10 mm to 20 mm, with a frequency of (55 ± 5) cycles per minute. The machine shall be fitted with a counter which shall stop automatically when the cutting edge touches the conductor or electrical screen.

For cables of diameter less than or equal to 6 mm the needle shall be used, and for cables with diameter greater than 6 mm the steel blade shall be used, unless otherwise specified in the particular cable standard.

The load on the cutting edge shall be defined in the cable standard.

The test specimen shall consist of a single 0,75 m sample of core or cable.

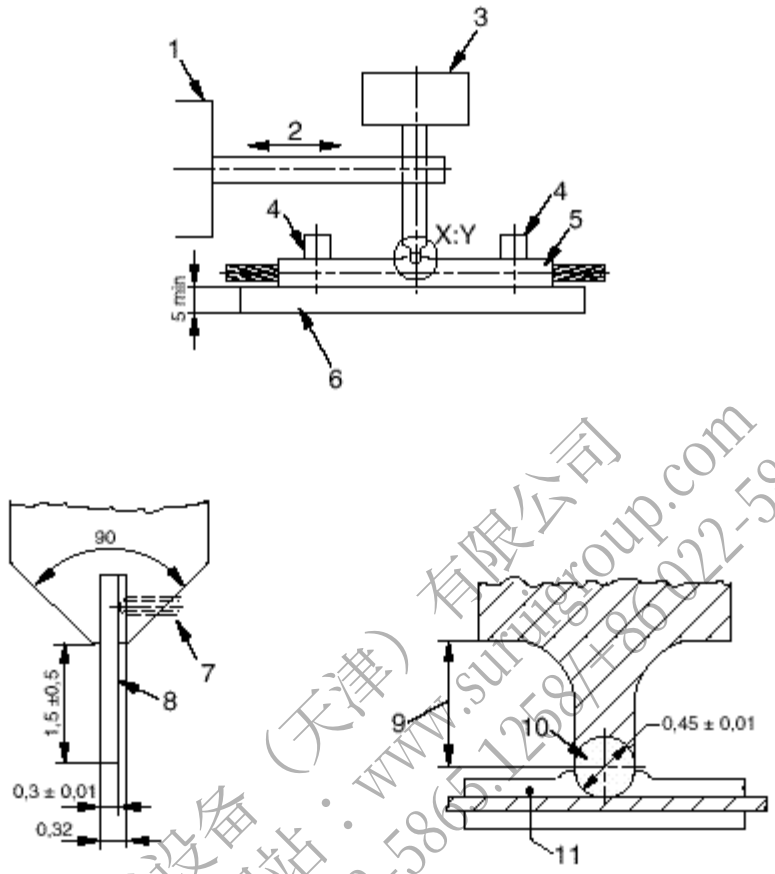
The test specimen shall be held securely on the plate by 2 cable clamps.

Each test specimen shall undergo four tests. After each single test it shall be moved approximately 100 mm and turned by a 90° angle, clockwise.

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.

Each test is finished when the cutting edge touches the conductor or electrical screen.

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



a) Detail X
(edges not rounded; free of burr)

b) Detail Y

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		
5	Test specimen	10	Cutting edge - needle
6	Supporting plate	11	Cable under test

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 °C, one at ambient temperature and one at 85 °C, in all cases for 3 h, after which time they shall be wound on to a mandrel, (3 ± 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.

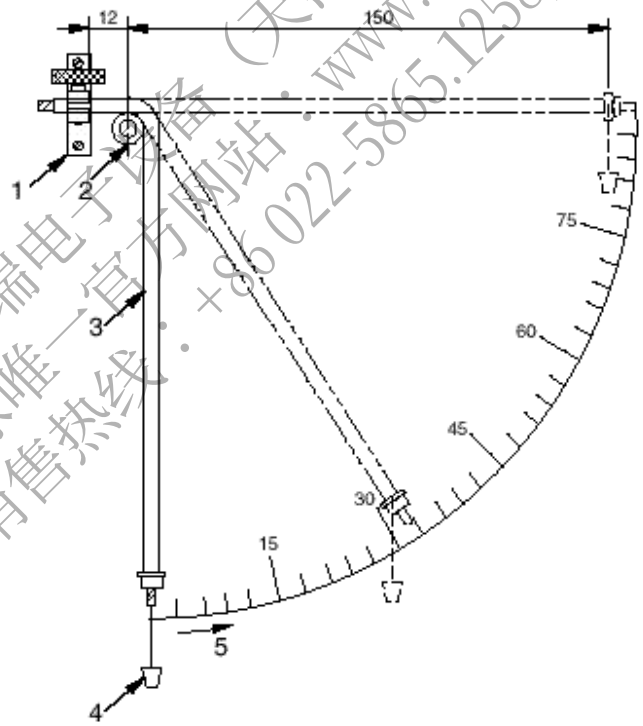
5.4 Pliability

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 $(90\ ^{0}_{-1})°$.

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.



Dimensions in millimetres

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

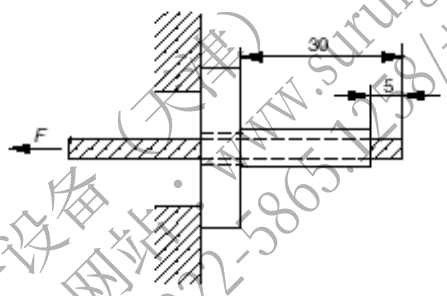
Stripping of 5 mm of insulation from each end of a 50 mm sample shall be easily carried out with normal stripping pliers.

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 ± 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_{20} = resistance at 20 °C, in ohm/kilometre;
R_t = 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 previously heated to 90 °C; a length of 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 then be 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 related to 1 km.

6.5 Spark test

6.5.1 General

This test shall be carried out as a routine test in the final stage of manufacture either on delivery lengths or on manufacturing lengths before cutting them into delivery lengths.

The test shall be carried out either on insulated cores or on the sheath of cable where there is a metallic screen or braid under the sheath.

6.5.2 Method

Test requirements : The cable shall withstand the test voltage specified in the particular cable standard without failure of the insulation or sheath as appropriate. The spark test equipment shall detect a puncture in the insulation or sheath having a diameter equal to or greater than half of the specified insulation or sheath thickness. The recovery time of the spark tester shall be not greater than one second.

Test voltage : The voltage applied by the spark tester may be power frequency a.c., d.c., high frequency or of other form.

The magnitude and the presence of the voltage shall be such that with the electrode system employed and at the speed employed for the passage of the cable through the spark tester the test requirements are effectively met.

The reference method to be used to establish the efficacy of the spark testing equipment is given in Annex B.

6.6 Surface resistance

The test specimen shall be prepared as follows.

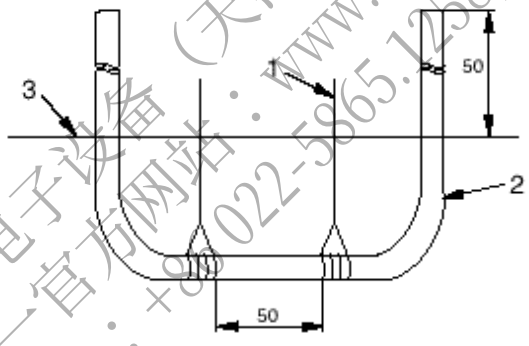
Each specimen shall have two annular electrodes fitted as shown in Figure 4. The two electrodes shall consist of copper wire with a diameter of about 1,8 mm and they shall be arranged in the centre part of the specimen with their internal windings about 50 mm from each other. When positioning the electrodes it is essential to avoid any serious indentation of the wire into the protective cover.

The specimens, fitted with their electrodes, shall be immersed in water held at (20 ± 5) °C for 4 h with the ends of the specimen protruding by about 50 mm above the level of the water.

Upon completion of this operation, the specimens shall be removed from the water and excess water removed from their surfaces.

The specimens prepared in this way shall be immediately subjected to 2,0 kV a.c. applied between the electrodes. The leakage current shall be measured, using an appropriate ammeter, between the two electrodes after 30 s voltage application.

Immediately after the determination of the leakage current, the voltage applied between the electrodes shall be increased at a constant rate of 100 V/s until breakdown occurs or the voltage limit given in the particular specification is exceeded.



Dimensions in millimetres

Key

1 Copper wire
2 Test specimen
3 Water level

Figure 4 – Arrangement of electrodes for test sample

6.7 D.C. stability

A test specimen of minimum length of 5 m of cable shall be immersed in distilled water containing 3 % NaCl. At least 300 mm of additional cable shall protrude at each end. The salt solution shall be maintained at an elevated temperature of (85 ± 2) °C for (240 ± 2) h, and the specified voltage applied between the conductor and the salt solution with the conductor at the negative potential. The test shall then be repeated, using a new test specimen, with the conductor at the positive potential.

The current flowing in the circuit shall be measured periodically throughout the test at intervals not greater than 24 h. Continuous measurement is preferred, if possible.

The current measurement data shall be plotted in the form of a current versus time curve and shall indicate an approach to a period of stability.

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 ± 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.

NOTE This method may also be used for certain sheathing materials when required by the particular cable specification.

A brief explanation of the significance and use of the test is given in Annex C.

7.2.2 Summary of test method

Three or four sets of test specimens of a given sample of insulated wire are exposed for selected periods of time at several fixed temperatures. After each exposure period the specimen is wrapped on a mandrel to simulate a flexing stress and then immersed in a water bath where it is subject to a voltage test. A given specimen is subjected to a continued series of exposures at its designated test temperature until failure occurs.

The life data at different temperatures are analysed on the basis of the Arrhenius equation which relates exposure time to failure to the reciprocal of the absolute temperature of exposure. The method is based on EN 60216-1.

7.2.3 Apparatus

The ageing shall be carried out in a circulating air oven, meeting the general requirements of 8.1.2 of EN 60811-1-2 and capable of operating at the required temperature. The vertical internal dimension of the oven shall be at least 500 mm.

A rack shall be provided for holding insulated wire specimens. A simple one can consist of 6 mm rods located horizontally approximately 25 mm below the top of the chamber. These can be mounted as a part of the chamber or as a removable rack carrying the specimens.

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 [a]	Duration of exposure in days for an estimated temperature value (°C) of:			
°C	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			

[a] Tolerances are given in Table 1.

Begin the test sequence with the highest temperatures, since exposure times will be relatively short. On the basis of these results, review the proposed lower exposure temperatures and revise if necessary.

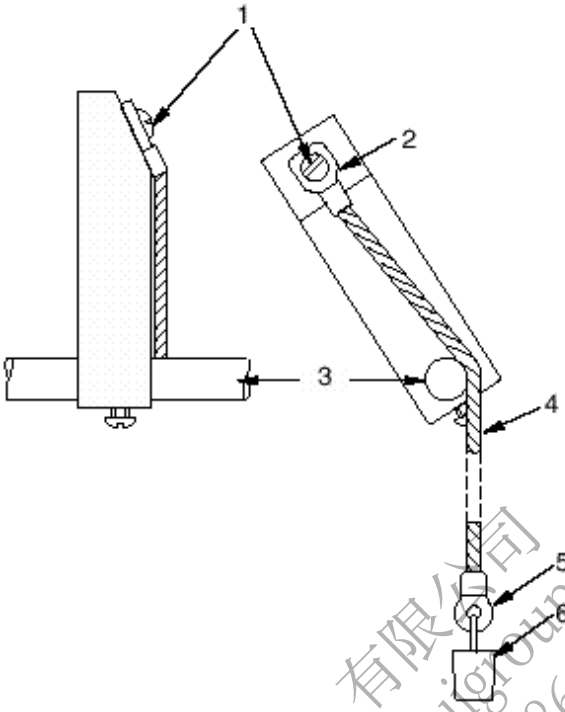
7.2.4.3 Wrapping procedure

After each period of exposure remove the group of specimens from the oven. Allow them to cool to room temperature.

A mandrel shall be provided, supported horizontally, and fitted at one end with a crank for mandrel wrapping of the specimens. Support the rod at least 600 mm above a horizontal work surface. The mandrel shall be provided with some convenient means for attaching one end of the insulated wire for wrapping. A suggested method of attachment is the crank arm shown in Figure 5. The diameter of the round mandrel and weights for attaching to the lower end of the specimens during the mandrel wrap shall be as in Table 4.

Table 4 – Requirements for wrapping test

Conductor cross section mm^2	Mandrel diameter mm	Weight kg
0,5	13	0,50
0,75	15	0,50
1,0	20	0,75
1,5	20	0,75
2,5	25	1,00



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

Attach one end of the specimen to the mandrel. Hang the prescribed added weight to its lower end. Rotate the mandrel so that the specimen is wrapped on it, first in one direction and then in the opposite direction. Do this twice. During the wrapping allow the insulated wire to twist freely and seek its own position on the mandrel with only the limitation that the core or cable must stay in contact with the mandrel and not wind upon itself. The speed of winding shall be uniform at a rate of one turn in 3 s to 5 s. It is preferable to have the winding motorised, but it may be done by hand. Then unwind the specimen at the same rate, remove the weights and detach the specimen from the mandrel.

7.2.4.4 Voltage test

Connect the two ends of a specimen together and immerse the specimen at room temperature in a bath containing 1 % of sodium chloride (NaCl) with 25 mm to 30 mm of each end of the insulated wire above the surface. Soak for 1 h.

Subject the specimens to the voltage test as specified in 6.2. at 1,5 kV a.c. unless otherwise specified in the product standard.

7.2.4.5 Continuation of exposure

A given specimen shall be subjected to a continued series of exposures at the designated test temperature, including wrapping sequence and voltage test, until electrical failure occurs.

7.3 Long term ageing for sheath

Samples of sheath from cable shall be aged as in EN 60216-1, except that after each exposure time three test pieces shall be tested for elongation at break in accordance with 9.2 of EN 60811-1-1 and the mean value recorded. The method of treatment of the results shall be as given in EN 60216-1 in which the exposed time to failure is based on a value of 50 % elongation at break.

7.4 Ozone resistance

7.4.1 Electrical test

The test sample shall be wound for at least 6 complete turns on to a cylindrical mandrel of diameter (10 ± 1,0) times the minimum diameter of the cable.

The assembly shall be subjected to ozone exposure as specified in the product specification. At the conclusion of the exposure it shall be examined for cracks and then subjected to the voltage test as specified in 6.2.

The examination and voltage test shall be carried out after cooling to ambient temperature and before 24 h have elapsed.

7.4.2 Non-electrical test

7.4.2.1 Test apparatus and testing devices

a) suitable ozone test chamber with uniform ozone concentration;

b) cutting device for preparation of test pieces;

c) clamping device according to Figure 6, or a similar device;

d) cylindrical mandrels consisting of wood or metal;

e) desiccator filled with silica gel or an equivalent material.

7.4.2.2 Selection and cutting of test pieces

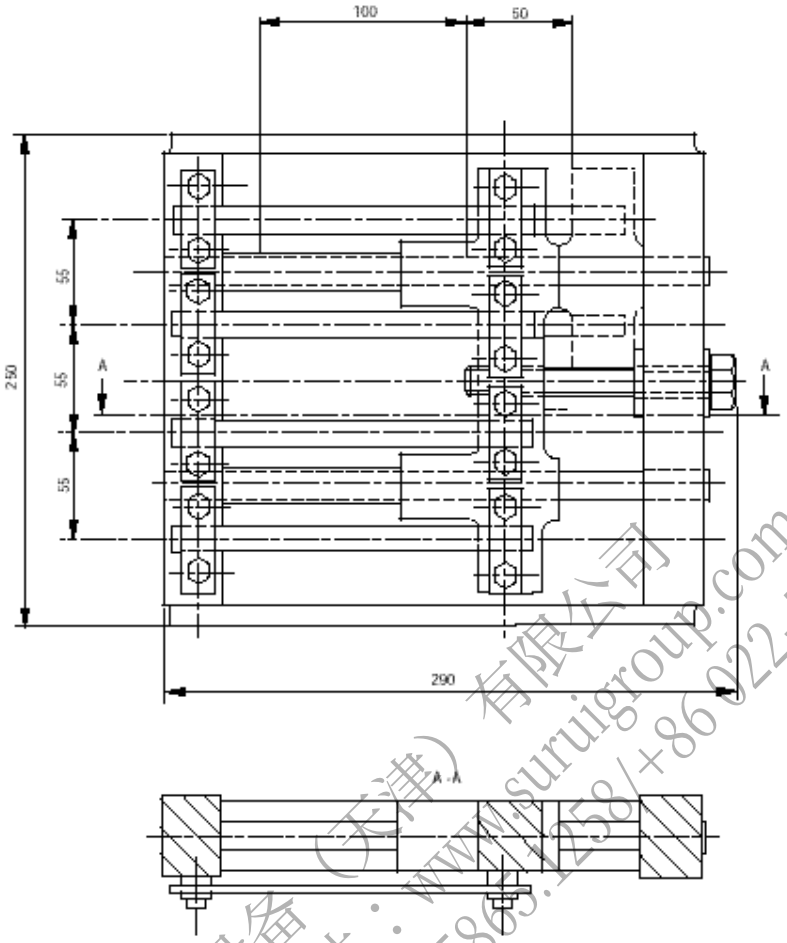
Three test pieces of at least 200 mm length consisting of the complete core or cable shall be used whenever possible under the proviso however that any coverings over the insulation or the sheath to be tested have been removed, care being taken not to damage the insulation or the sheath.

If it is impossible to use the complete core or cable, the insulation or sheath respectively shall be cut longitudinally and the required three test pieces of at least 5 mm but not more than 10 mm width shall be punched by using the cutting device according to 7.4.2.1 b). The test pieces shall be ground or cut, so as to obtain test pieces of uniform thickness, care being taken to avoid undue heating. After grinding or cutting the thickness of test pieces shall be not less than 0,8 mm and not more than 2,0 mm.

7.4.2.3 Conditioning and preparation of test pieces

The test pieces shall be wiped with a clean cloth to remove dirt or moisture and stored in the desiccator according to 7.4.2.1 e) for at least 16 h.

Test pieces consisting of the complete core or cable shall be wound around the mandrel according to 7.4.2.1 d). The diameter of the mandrel shall be (2 ± 0,1) D (D = outer diameter of test piece). Both ends of the pieces shall be fixed on the mandrel in order to keep the windings in position.



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 ± 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 the middle of the test chamber according to 7.4.2.1 a) so that each piece is at least 20 mm from any other piece and exposed to the ozone concentration required.

The ozone concentration shall be measured inside the test chamber and the other test conditions shall comply with the appropriate values given in the cable standard.

The air with the required ozone concentration shall have a flow rate from 0,2 up to 0,5 times the chamber content per minute. To avoid laminar flow along the samples the speed shall be ≥ 500 mm/s. This can be achieved by a built-in fan and can be checked by an anemometer.

7.4.2.5 Requirements

After the specified test duration the test pieces shall be removed from the test chamber, and while still elongated, shall show no cracks when examined with normal or corrected vision.

Any cracks near the fixing point on the mandrel and/or near the clamps when using test strips shall be disregarded.

7.5 Pressure test at high temperature

The test shall be in accordance with EN 60811-3-1, clause 8, except that the recommendation on minimum applicable thickness shall be disregarded and the samples shall be taken from the whole cable and shall be subjected to the voltage test in 6.2 after cooling to ambient temperature and before 24 h have elapsed. The test voltage shall be as specified in the cable specification.

NOTE Sufficient length of sample is required to carry out the voltage test.

7.6 Shrinkage test for insulation

The method of test shall be in accordance with EN 60811-1-3 except that the length of insulated core shall be 0,3 m.

The test specimen shall be placed in a oven pre-heated to the temperature specified. The test specimen shall remain in the oven for the specified time. The test specimen shall be then taken out and left to cool to ambient temperature.

The maximum shrinkage of the insulation at either end shall be measured.

7.7 Stress cracking test

7.7.1 General

The resistance to cracking under stress, at a test temperature related to the 20 000 h thermal endurance temperature of the cable, shall be demonstrated by the procedure in 7.7.2 to 7.7.4.

7.7.2 Preparation of test assemblies

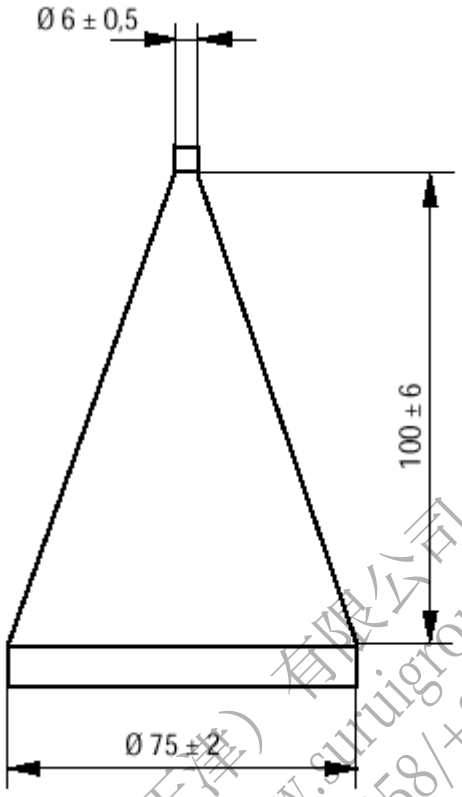
A test assembly shall be prepared in the form of a fully wound mandrel. The mandrel shall be in the form of a cone (see Figure 7), in which the cable sample forms a helix along the mandrel from a bending diameter of 6 mm to one of 75 mm, each turn of the helix touching the previous turn.

It is recommended that the mandrel is metal covered with a smooth inert coating.

No cable shall be wound to a diameter of less than 3 times the minimum specified diameter of the cable or less than 6 mm whichever is the greater.

The cone shall be in the form of a flat topped cone shaped mandrel with the following dimensions:

- diameter of base (75 ± 2) mm;
- diameter of flat top (6 ± 0,5) mm;
- vertical height (100 ± 6) mm.



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).

A parallel line is drawn through the point with co-ordinates of 20 000 h and 110 °C or 125 °C for maximum operating temperatures of 90 °C or 105 °C respectively.

For the stress cracking test the ageing temperature for the particular product is determined by

- finding the temperature corresponding to 168 h on the parallel line referred to above,
- subtracting 8 °C from that temperature.

However, in no instance should the test temperature be greater than the highest temperature of test used in determination of the Arrhenius plot.

NOTE In the example in Annex D the 168 h point is 182 °C, therefore the stress cracking test would normally be performed at 174 °C, for that particular product.

7.7.4 Test method

The test assembly shall be aged for 168 h in an oven as specified in EN 60811-1-2. The cone mandrel shall be arranged in the oven so that no pressure is exerted on the cable.

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 ± 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 ± 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 conditions as given in the particular cable standard, after which time it shall be examined for cracks and subjected to the voltage test as specified in 6.2.

The examination and voltage test for the assembly shall be carried out after cooling to ambient temperature and before 24 h have elapsed.

8.2 Acid and alkali resistance

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

The assembly shall be subjected to immersion, using the acid or alkali solution and the immersion conditions as given in the particular cable standard, after which time it shall be examined for cracks and subjected to the voltage test as specified in 6.2.

The examination and voltage test for the assembly shall be carried out after cooling to ambient temperature and before 24 h have elapsed.

8.3 Water absorption of sheath

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

The assembly shall be immersed in water using the immersion conditions as given in the particular cable standard, after which time it shall be subjected to a voltage test as specified in the particular cable standard.

The voltage test for the assembly shall be carried out after cooling to ambient temperature and before 24 h have elapsed.

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

The cables in the bundle shall be laid in the formation given and then subjected to a uni-directional twist to give a lay length of approximately 15 D (where D is the bundle diameter).

The maximum extent of the charred portion measured on the test sample shall not have reached a height above the bottom edge of the burner greater than that specified in the relevant cable specification.

9.2 Toxicity

All materials subject to this test shall be halogen-free, as defined in subclause 3.3 of EN 50306-1.

A preliminary qualitative analytical examination is required in order to determine the presence of sulfur and nitrogen.

Subsequent analysis techniques depend upon the outcome of this evaluation.

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 Evalution

Nitrogen

Add a few cm³ 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, $Fe(OH)_2$, is formed;
- boil the resulting product, then add a few cm³ of ferric chloride ($FeCl_3$) solution and acidify with a few drops of hydrochloric acid; the presence of nitrogen is indicated by the formation of a blue ferrocyanide (Prussian blue) compound.

Sulfur

Add a few drops of an approx. 1 % sodium nitroprusside solution to the solution. A deep purple colour indicates the presence of sulfur.

NOTE These qualitative examinations cannot detect the relevant elements when their content is below 0,1 %.

9.2.2 Quantitative analysis

9.2.2.1 General

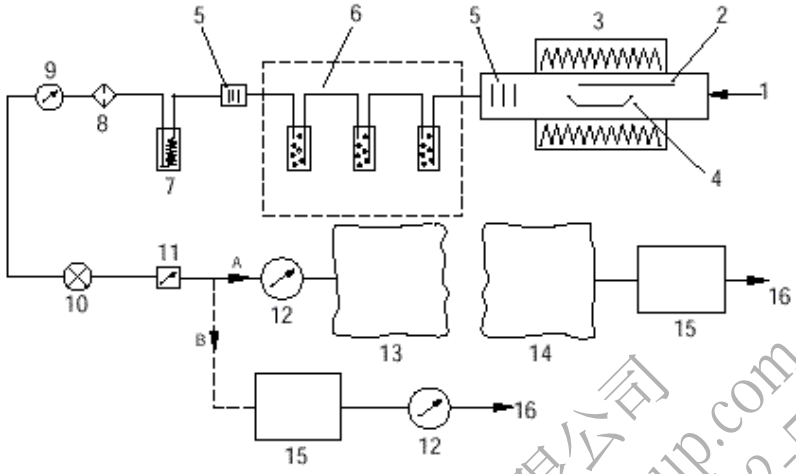
Depending upon the results from 9.2.1.2 further testing shall be in accordance with Table 5.
The apparatus used is described in EN 50267-1. In this procedure, a test specimen of material is introduced into a tube and a current of air is passed through the tube over the test specimen to support combustion. The effluent is then analysed.
The air supply can be pushed (see EN 50267-1) or pulled. In the latter case air gas bags should be used to collect gases at the end of the circuit (see Figure 8).
When air is pushed, continuous analysis is possible for carbon monoxide, carbon dioxide, hydrogen cyanide and sulfur dioxide according to the methods in E.1. Discontinuous analysis is also possible for these gases as well as for oxides of nitrogen, according to E.2.
Due to the effect of some elements on the absorbing solutions used for titration Table 5 gives annex references, which define appropriate analysis methods.

Table 5 – Determination method to be applied

Nature of detected element	Analysis				
	CO	CO_2	HCN	SO_2	NO_X
None (absence of both sulfur and nitrogen)	E.1.1 or E.2.1	E.1.1 or E.2.1	-	-	-
Sulfur	E.1.1 or E.2.1	E.1.1 or E.2.1	-	E.1.2 or E.2.2	-
Nitrogen	E.1.1 or E.2.1	E.1.1 or E.2.1	E.1.3.2 or E.2.4	-	E.2.3

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_2 – continuous mode only)
5	Silica wool	13	Gas bag 1
6	Bubbling line (only for HCN/NO_x and SO_2)	14	Gas bag 2
7	Cold trap	15	CO/CO_2 analyser
8	Dust filter	16	External outlet
A	Route for discontinuous analysis	B	Route for continuous analysis

Figure 8 – Schematic diagram of apparatus for production, collection and analysis of gases

9.2.2.3 Test specimen and pre-conditioning

The test specimen (approximately 1 g) shall be a piece of insulation or sheath (or other non-metallic component under investigation) taken from the relevant cable.

The test specimen shall be stored for at least 48 h in a testing room adjusted to (23 ± 2) °C and (50 ± 5) % relative humidity.

9.2.2.4 Combustion procedure

The test specimen, weighed to an accuracy of 0,001 g, is placed in the previously weighed combustion boat.

The furnace is established at its operating temperature of (800 ± 10) °C.

At the beginning of the test, marked by the start of a timer, the compression-air blower is operated and the combustion boat with the sample is immediately inserted into the central part of the combustion tube. The temperature is checked throughout the test by means of a temperature probe.

The air flow is adjusted by the needle valve so as to ensure a rate of (120 ± 5) $dm^3\ h^{-1}$, that is, approximately 33 $cm^3\ s^{-1}$ (a flow rate corresponding to a continuous bubbling in the wash bottles).

The pyrolysis or combustion is continued for 20 min. The gas flow arising from the combustion tube is purified by passing through a dust filter, then analysed according to the methods specific to the nature of the burnt material.

The combustion boat is then removed from the combustion tube, even if the combustion or pyrolysis is not completed. The amount of the evolved gas is then driven out by approximately 40 dm^3 of air. At the end of the test, the device is purged by about 80 dm^3 of air.

9.2.2.5 Analysis methods

The detailed analysis methods are given in

- E.1: Continuous analysis methods, and
- E.2: Discontinuous analysis methods.

9.2.3 Index calculation

The toxicity index (ITC) shall be calculated, based on the result of the relevant analysis and the titration of noxious gases produced by the pyrolysis procedure using the following formula:

$$ITC = \frac{100}{m} \bullet \sum \frac{M_z}{CC_z}$$

where

m = weight of the sample, g;

M_z = weight of gas z produced by the sample combustion, mg;

CC_z = critical concentration for a 30 min exposure for gas z, mg/m^3 given in Table 6.

Table 6 – CC_z values for various gases

Gases		CC_z
Carbon monoxide	CO	1 750
Carbon dioxide	CO_2	90 000
Sulphur dioxide	SO_2	260
Nitrogen oxides	NO_x	90
Hydrogen cyanide	HCN	55

10 Miscellaneous tests

10.1 Durability of marking

The durability of printed marking or of colour shall be checked by trying to remove the marking or colour by rubbing lightly 10 times with a piece of cotton wool or cloth soaked in water.

10.2 Blocking of cores

The test sample, which shall be of suitable length, shall be wound in two layers of six to eight turns each around a mandrel covered with PTFE or silicone oil and its diameter shall be between nine and ten times that of the core.

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

Ref	Test method	Standard
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 [a]
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

[a] 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.

a) The insulation shall be removed from the core for a length of about 5 times the nominal insulation thickness.

b) 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).

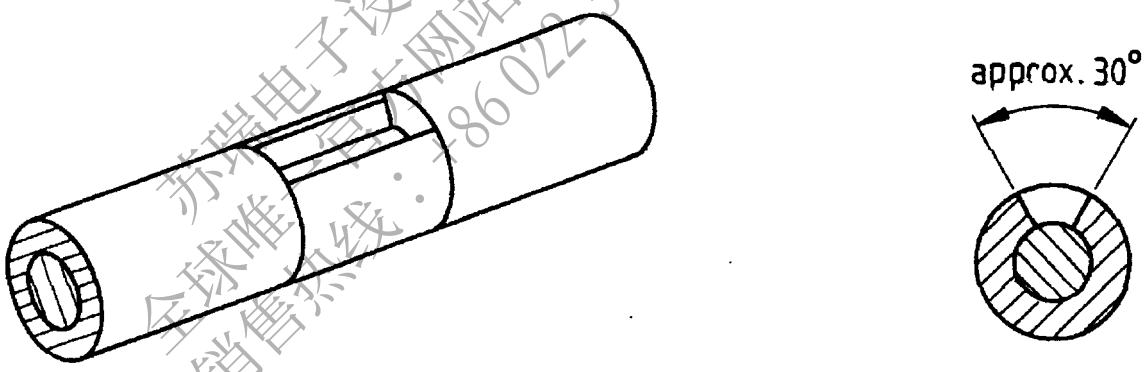


Figure B.1 – Removal of insulation segment

c) 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).

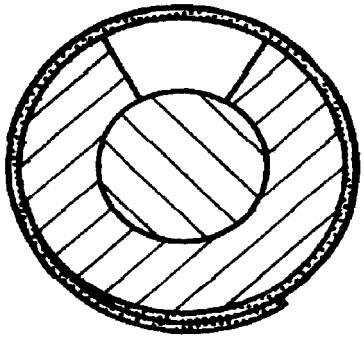


Figure B.2 – Overlap position for tape

The layer shall have a length of at least ten times the nominal insulation thickness.

d) In this layer, in the middle of the place where the insulation has been removed, a hole in the tape shall be punched with a hot needle. The diameter of this hole shall be equal to half of the allowed minimum insulation thickness.

The other test piece shall be prepared in the same way.

B.2.3 The prepared test-pieces should then be passed through the spark test equipment at the highest speed for which the equipment is intended, the voltage applied between the electrode and the conductor being that normally used.

A fault shall be registered as each test piece is passed through the equipment.

B.2.4 Method to check the recovery time

At least two faults shall be passed through the spark-test equipment at its actual operating speed v (in metre per second), the distance in metres between two faults being not greater than the value of v.

All the faults shall be registered by the equipment.

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 lower temperatures providing that the extrapolation is limited and is based on adequate data with sufficient linearity.

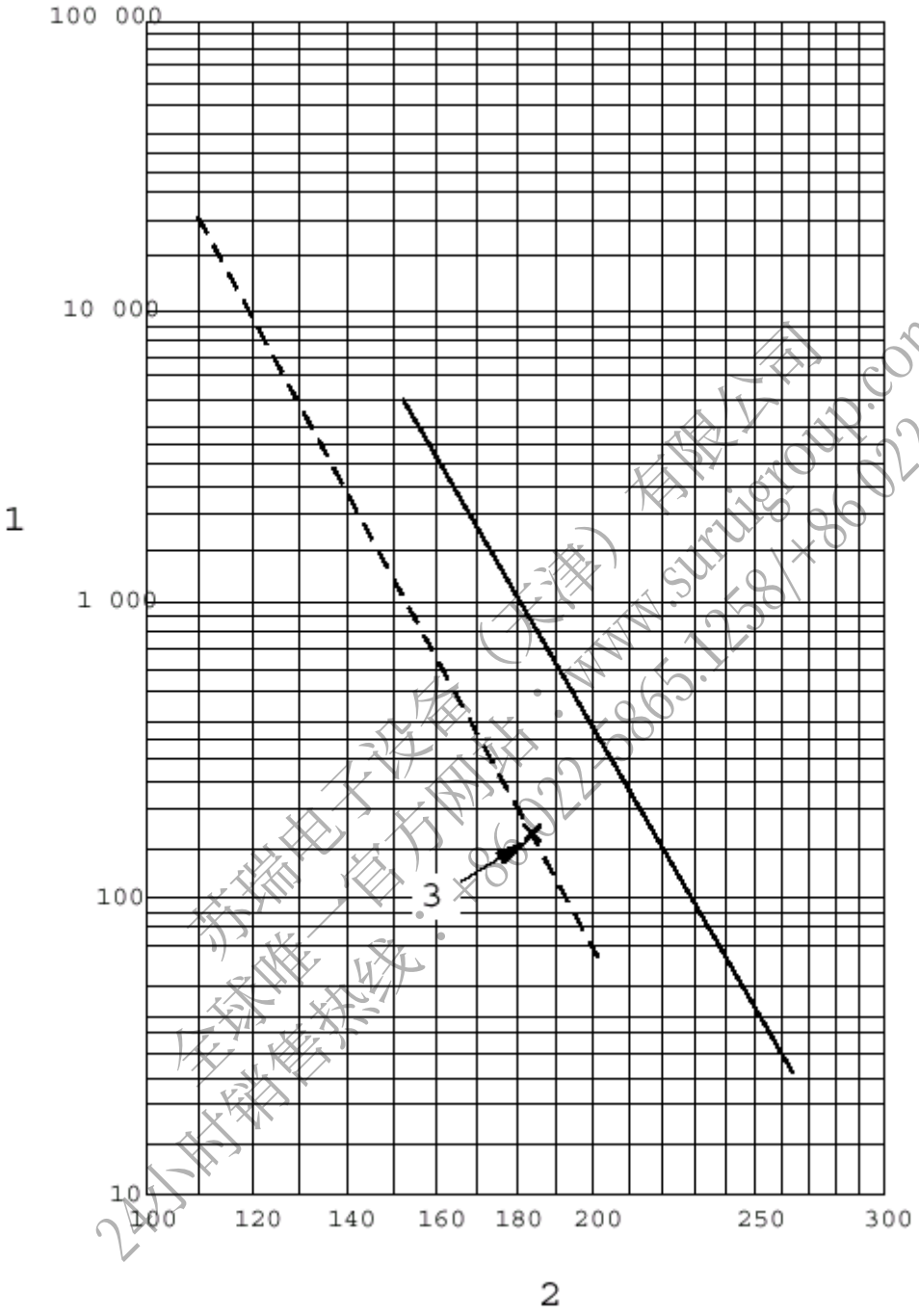
Embrittlement of the insulation and the loss of its electrical strength are the usual causes of failure due to thermal ageing on insulated wire in practical applications; hence the failure points for these accelerated conditions are determined by standard tests of embrittlement and electrical strength.

In comparing different systems, it is important that the dimensions and constructions of each are those to be used in the intended application.

It is important to know that changing the conditions of test will change the results. Decreasing the mandrel size, increasing the weight applied during the mandrel bend, bending at too high a rate, or increasing the proof voltage will decrease life. Too few temperature exposure cycles will result in erroneously long life values.

Annex D
(informative)

Illustration of an Arrhenius plot



Key

1 Exposure time, h
2 Temperature, °C
3 168 h point (used to calculate stress cracking test temperature – see 7.7.3)

_____ Line drawn from the long-term ageing test
------- Parallel line through 20 000 h and 110 °C

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_2. 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:

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

where

V_1 is in dm³
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 °C (298 K)

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

where

V_m^0 = molar volume at T_0 = 0 °C (273 K), that is 22,4 dm³ and
T/T_0 = 298/273

that is

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

Therefore

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

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

– Determination of the carbon dioxide (CO_2)

In a similar way, the apparatus calibrated in "CO_2 volume %" gives the % vol/vol ratio X_2 of the CO_2 contained in the gas volume V_1.

The amount of CO_2, evolved from the sample, is in milligrams:

$$CO_2 \text{ (mg)} = 1\,000 \cdot X_2 \cdot (44/24{,}45) \cdot V_1$$

where

M = the molar mass of CO_2 (44 g)
V1 is measured in dm³, and
24,45 dm³ = the molar volume of the CO_2 at T = 25 °C,

i.e. $CO_2 \text{ (mg)} = 1\,800 \cdot X_2 V_1$

– 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 test specimen indicates the amounts of CO and CO_2 contained in the ambient air, to be deducted from the determined values.

E.1.2 Materials containing sulfur - determination of sulfur dioxide

E.1.2.1 General

After a first test for the determination of CO and CO_2 a separate second combustion or pyrolysis shall be carried out for the determination of sulfur dioxide.

The sulfur, present in the composition of materials, under combustion or pyrolysis conditions, in the presence of oxygen, forms sulfur dioxide and is determined as such.

A discontinuous quantitative measurement of the sulfur dioxide may be carried out by means of detector tubes – see E.2.2.

E.1.2.2 Apparatus

- Wash bottles
- Laboratory graduated glassware
- Absorption spectrophotometer in the visible range
- Water bath
- Sulfur dioxide permeation tube
- Compressed dry nitrogen bottle
- U-shaped glass tube filled with glass balls 4 mm diameter
- Thermometer
- Ball flowmeter

E.1.2.3 Reagents

- Mercuric chloride ($HgCl_2$), analytical
- Sodium chloride, analytical
- Trapping solution: sodium tetrachloromercurate (0,1 M). For 1 dm³ of solution, dissolve 27,2 g of mercuric chloride with 11,7 g of sodium chloride
- Formaldehyde at 0,2 % : 6,6 cm³ of commercial solution of 30 % by mass diluted to 1 dm³ at the time of use
- Hydrochloric acid 12 N
- Para-rosaniline original solution at 1 %
- Decoloured solution of para-rosaniline at 0,04 % in hydrochloric acid : to 4 cm³ of original solution of para-rosaniline, add 6 cm³ of hydrochloric acid 12 N; dilute to 100 cm³ with demineralized water. The resultant solution shall be colourless or pale yellow
- Sulfamic acid solution at 0,6 % in mass

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 ± 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.

NOTE 1 Recommended operating conditions:
- water bath temperature : 25 °C ± 0,1 °C;
- DYNACAL or equivalent permeation tube, length: 2 cm with standard emission nitrogen flow : 0,6 dm³/min.

The permeation tube is immersed in the water bath and allowed to stabilise for 48 h , then removed and rapidly weighed within 0,1 mg. It is replaced under the same conditions and weighed after 168 h (1 week).
The recorded loss in mass corresponds to the mass of sulfur dioxide released by the tube.

NOTE 2 Using the recommended operating conditions, this mass release rate ranges between 0,5 to 0,6 × 10^{-6} dm³/min.

The calibration curve is obtained as follows.

The sulfur dioxide, evolved at the outlet of the stabilised tube under the above mentioned conditions, is absorbed in 10 cm³ of the trapping solution for specific durations calculated to obtain masses of sulfur dioxide ranging between 0 and 15 x 10^{-6} g.

Colorimetric calibration is then carried out as described below.

Calibration

To 10 cm³ of the solution containing the sulfur dioxide, add successively 2 cm³ of formaldehyde and 1 cm³ of decoloured para-rosaniline.

If the material is likely to give off nitrous oxides, add also 1 cm³ of sulfamic acid solution.

In the presence of sulfur dioxide, a violet colouration develops. Wait 20 min at the ambient temperature, then measure the optical density at 560 nm after zero adjusting with the reference solution.

This optical density value is plotted on a calibration curve to correlate with the mass, m, in grams, of sulfur dioxide present in 10 cm³ of solution.

The total mass of sulfur dioxide given off is:

$$SO_2\ (g) = V \cdot m / 10$$

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 determination of hydrogen cyanide below this value, down to the limit of 0,000 4 mg.

In the presence of a sodium picrate solution, the hydrogen cyanide ion gives a steady red colouring of isopurpurate.

Apparatus: Spectrophotometer (500 nm wavelength) with 10 mm thick cells.

Reagents : Bubbling reagent: a standard sodium hydroxide solution (NaOH).

Titration reagent: an aqueous solution containing 3 g × dm^{-3} of picric acid and 50 g × dm^{-3} of sodium carbonate.

Analysis

The hydrogen cyanide contained in the combustion gases is converted into sodium cyanide through bubbling in three bottles, each containing 50 cm^3 of a 0,1 N sodium hydroxide solution. The solutions and the washings are combined and made up to a known volume, V_1, for example 200 cm^3.

Add 30 cm^3 of titrating reagent to an aliquot portion, V_2, of the above solution, e.g. 20 cm^3. After the mixture has been heated in a water bath at 90 °C for 10 min, the colouring develops. The analysis is carried out by spectophotometry at 500 nm wavelength in 10 mm thick cells, by reference to a blank (reagent only). After cooling to room temperature, the hydrogen cyanide content, X (in mg), is obtained by comparison with a calibration curve, previously plotted, using titrated potassium cyanide solutions mixed, in the same conditions, with the titrating reagent and giving in respect of the optical density, hydrogen cyanide contents ranging from 1 mg × dm^{-3} to 10 mg × dm^{-3}.

If the hydrogen cyanide concentration is too high, the volume of aliquot portion shall be reduced.

The amount of the hydrogen cyanide released by the sample is calculated by means of the relationship:

$$HCN\ (mg) = (V_1/V_2)\ .\ X$$

E.2 Discontinuous analysis

The apparatus used is shown in Figure 8.

If the analysis is not carried out continuously (see E.1), a gas bag, (a saran lined polyvinyl chloride inflatable bulb or balloon of a 40 dm^3 capacity) is placed at the end of the circuit to recover the gas mixture, so that additional analytical determinations may be performed with infrared analysers, detector tubes, or gas chromatography, after the soluble gases have been absorbed through bubbling. After a 20 min test, this bag is replaced by another one, of the same capacity. The total volume of the samples drawn from the gas bags is 80 dm^3.

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_2. 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_2)

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_2 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 $\times$ 10^{-4} (vol/vol);
- 5 to 100 $\times$ 10^{-4} (vol/vol);
- 20 to 500 $\times$ 10^{-4} (vol/vol);
- 500 to 5 000 $\times$ 10^{-4} (vol/vol).

E.2.4 Analysis of the hydrogen cyanide

A spectrophotometric analysis is carried out on a aliquot portion according to E.1.3.2.

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