

# TECHNICAL SPECIFICATION

# IEC TS 61244-3

Second edition  
2005-11

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## Long-term radiation ageing in polymers –

### Part 3: Procedures for in-service monitoring of low-voltage cable materials



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### Part 3: Procedures for in-service monitoring of low-voltage cable materials

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International Electrotechnical Commission, 3, rue de Varembé, PO Box 131, CH-1211 Geneva 20, Switzerland  
Telephone: +41 22 919 02 11 Telefax: +41 22 919 03 00 E-mail: [inmail@iec.ch](mailto:inmail@iec.ch) Web: [www.iec.ch](http://www.iec.ch)



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## INTERNATIONAL ELECTROTECHNICAL COMMISSION

**LONG-TERM RADIATION AGEING IN POLYMERS –****Part 3: Procedures for in-service monitoring  
of low-voltage cable materials**

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Technical specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC 61244-3, which is a technical specification, was prepared by subcommittee 15E: Methods of test, of IEC technical committee 15: Insulating materials, which has now been merged with IEC technical committee 98: Electrical insulation systems into IEC technical committee 112: Evaluation and qualification of electrical insulating materials and systems (provisional title).

This second edition cancels and replaces the first edition, published in 1998, and constitutes a technical revision. The main technical changes with regard to the previous edition are as follows:

- a) as there have been technical advances in established test methods and newer methods have become available, several additions have been made to the techniques available in Clause 5;
- b) some of the techniques listed in the previous edition were found to be either unsuitable for use as cable monitoring methods in plants, or less sensitive to radiation ageing than other methods; these techniques have now been removed;
- c) a list of abbreviations and their meanings has been added.

The text of this technical specification is based on the following documents:

Enquiry draft	Report on voting
15E/252/DTS	15E/258/RVC

Full information on the voting for the approval of this technical specification can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

IEC 61244 consists of the following parts, under the general title *Long-term radiation ageing in polymers*:

- Part 1 Techniques for monitoring diffusion-limited oxidation  
Part 2: Procedures for predicting ageing at low dose rates  
Part 3: Procedures for in-service monitoring of low-voltage cable materials

The committee has decided that the contents of this publication will remain unchanged until the maintenance result date indicated on the IEC web site under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- transformed into an International standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

## INTRODUCTION

Polymers are widely used as electric insulating materials (e.g. in cables for control, instrumentation and power) in environments in which they are exposed to radiation. In such applications, these materials may well be required to survive the full working life of the plant, which may be more than 40 years, and possibly accident conditions up to and at the end of their working life. Although considerable data are available on the behaviour of polymeric insulating materials under irradiation, there is still some uncertainty on the effects of long-term, low-dose rate irradiation such as would be experienced by cables. There is, therefore, a requirement for techniques for monitoring the state of degradation of cable materials *in situ* throughout the lifetime of the plant. Suitable cable monitoring techniques would also be important to surveillance programmes in support of plant life extension and licence renewal. Although this technical specification is primarily aimed at cable condition monitoring in radiation environments, it can also be applied to other polymeric components. Many of the techniques are equally applicable to thermal-only ageing of polymeric components.



## LONG-TERM RADIATION AGEING IN POLYMERS –

### Part 3: Procedures for in-service monitoring of low-voltage cable materials

#### 1 Scope

This part of IEC 61244, which is a technical specification, summarizes the main cable monitoring techniques which are currently being assessed worldwide. These techniques are primarily aimed at monitoring degradation of low-voltage cables. Most of the methods are at the development stage and require more in-plant evaluation before they could be recommended as standard techniques. The advantages and disadvantages of each method, and its current state of development, are outlined in the following clauses.

#### 2 Normative references

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

IEC 60544-5:2003, *Electrical insulating materials – Determination of the effect of ionizing radiation – Part 5: Procedures for assessment of ageing in service*

#### 3 Abbreviations

BR	Butyl rubber
CM	Condition monitoring
CSPE	Chlorosulphonated polyethylene
CP	Chloroprene
DLO	Diffusion limited oxidation
DSC	Differential scanning calorimetry
EPR	Ethylene propylene rubber
EVA	Ethylene vinyl acetate
IR	Infrared
OIT	Oxidation induction time
OITP	Oxidation induction temperature
NIR	Near infra-red reflectance
NMR	Nuclear magnetic resonance
PE	Polyethylene
PVC	Polyvinyl chloride
PEEK	Polyetheretherketone
SBR	Styrene butadiene rubber
TGA	Thermo-gravimetric analysis
XLPE	Cross-linked polyethylene
XLPO	Cross-linked polyolefin

## 4 Requirements of a monitoring technique

There is a range of requirements which the ideal cable monitoring technique would need to satisfy. In practice, no one technique can currently satisfy all of the requirements and a range of techniques is likely to be needed. In each case, baseline data (i.e. data on unaged material of the same formulation and manufacturer) are needed to make full use of the techniques.

The ideal monitoring technique would have the following attributes:

- non-intrusive, causing minimal cable disturbance;
- capable of use during normal operation;
- not require disconnection of equipment;
- related to an identifiable degradation criterion;
- applicable to a wide range of cable materials and configuration;
- applicable at accessible locations;
- capable of measuring degradation at hot-spots;
- reproducible and capable of compensating for environmental conditions (temperature, humidity);
- less expensive to implement than periodic cable replacement;
- readily available reference data.

## 5 Techniques available

### 5.1 General

There is a wide range of possible techniques which are being considered for cable monitoring. A few are already in use in-plant, whilst others are only at the laboratory evaluation stage. Those methods for which there is most experience have been published in IEC 60544-5. Such methods consist of:

- indenter;
- oxidation induction time and oxidation induction temperature;
- thermo-gravimetric analysis;
- density measurements;
- equipment deposit.

The monitoring methods which have been evaluated can be grouped together under generic types, as follows:

#### a) Local tests without sampling

- indenter;
- sonic velocity;
- near infrared reflectance;
- torque testing;

#### b) Local tests with micro-sampling

- modulus profiling;
- NMR relaxation;
- infrared spectroscopy;
- oxidation induction time (OIT) and temperature (OITP);

- thermogravimetric analysis (TGA);
- density measurements;
- gel fraction and solvent uptake.

Each of these types of test is described in more detail in the following subclauses.

## 5.2 Local tests without sampling

### 5.2.1 General

The term "local" refers to techniques which give information on the state of the cable at the measuring point only and are thus likely to miss localized degraded areas. These methods can only be applied in man-accessible areas and are generally limited to tests of the cable jacket material except at terminations where the insulation is exposed. Where the techniques have been cross-correlated with changes in elongation at break, which is a consistent indicator of degradation, these methods have a predictive capability. This type of test will provide immediate data in-plant on the state of the cable. Where the cable jacket is more likely to degrade than the insulation (which is often true), the methods provide early warning of cable failure. Local bend tests by manipulation of the cable by hand can give qualitative information when carried out by experienced personnel.

### 5.2.2 Indenter

The indenter is an instrument that determines a parameter related to the compressive modulus of a polymer. By driving an instrumented probe of known shape into the surface of the polymer, the load exerted is measured. Details of the method are given in IEC 60544-5.

### 5.2.3 Sonic velocity

This technique is under development and at present (2005), has only been tested on PVC based cables [1-3]<sup>1</sup>. Sonic velocity testing is based on the fact that the velocity of sound in a solid medium is dependent on both the density and the elastic modulus and is given by:

$$C^2 = \frac{E}{\rho}$$

where

$C$  is the sonic velocity,

$E$  is the elastic modulus,

$\rho$  is the polymer density.

Since both modulus and density can change during ageing of cable materials, changes in sonic velocity would be expected to occur on ageing.

The tester uses piezoelectric transducers to transmit and receive a series of pulses as shown schematically in Figure 1. The signal transit times can be plotted as a function of transducer separation distance (up to a few centimetres) to obtain the slope which represents velocity. Sonic velocity measurements have been made at 20 kHz on a series of PVC jacketed cables and on strips of jacket material cut from the cables. Comparison between the data obtained on the test strips and the complete cables has shown that the technique is dependent on the cable geometry and adjacent shielding and insulation components. The magnitude of the sonic velocity at this frequency also varies considerably with different formulations of PVC, therefore baseline data would be required for each type of cable used in a plant if the technique was to be of practical use [1]. Other work, using 1MHz pulses [2][3], found the sonic velocity to be strongly dependent on the degradation of PVC jacket materials but independent of cable geometry and PVC formulation (Figure 2).

<sup>1</sup> Figures in square brackets refer to the bibliography.

### *Limitations*

The sonic velocity tester measures properties of the cable jacket over a small volume between the transducer probes. The measurements obtained can be strongly dependent on the cable construction and the specific formulation of the jacket material. Therefore, extensive baseline data may be required. The technique is still under development and has so far only been tested on PVC jacketed cables. At present, a prototype portable tester has been developed but it has not been used for field use. Its high sensitivity to ageing degradation indicates that it may well be worth further development.

#### **5.2.4 Near infrared reflectance**

Changes in the infrared spectrum of polymers are known to occur with ageing, primarily in functional groups such as carbonyl (C=O), hydroxyl (O-H) and carboxyl (COOH). These functional groups are normally observed in the infrared spectrum in transmission, but for use in-plant a portable unit based on near-IR reflectance (NIR) using a fibre optic probe would be more suitable. NIR is also being used as a method for identifying cable materials and compounds [4].

NIR reflectance measurements have been carried out on PVC jacketed cables in the wavelength range 1 300 nm to 2 100 nm ( $7\,692\text{ cm}^{-1}$  to  $4\,762\text{ cm}^{-1}$ ) [1] using a fibre optic probe pressed against the cable jacket. Baseline shifts arising from small changes in optical path length are often seen in the absorbance spectra. These small baseline shifts can be eliminated by using the first derivative of the absorbance spectra (Figure 3), since the first derivative is the slope of the absorbance spectrum at each wavelength. Various regions of the IR spectrum were found to correlate well with changes in the elongation at break of the PVC material tested; for example, changes in the spectrum at 1 640 nm to 1 650 nm ( $6\,098\text{ cm}^{-1}$  to  $6\,060\text{ cm}^{-1}$ ) are shown in Figure 4 for thermal ageing of PVC. This technique using fibre optic probes for reflectance measurements is at an early stage of development.

### *Limitations*

The NIR reflectance spectrum obtained is only from a thin surface layer of the cable material being tested. This surface may not be representative of the state of degradation of the bulk of the material. As in the other techniques in this category, data are limited to the immediate area of the probe. Care must also be taken in interpreting the data as some stabilizing additives in polymers have peaks in the same range of wavelengths. The technique is not sensitive to the cable geometry or construction but calibration curves would be required for each cable material. NIR is not applicable to carbon-filled materials.

#### **5.2.5 Torque tester**

The degradation of cable jacket materials can also be determined using a torque-strain response method [5]. A pair of chucks are used to grip the outside of the cable and a small angle torque, in the range  $5^\circ$  to  $10^\circ$ , is applied to one of the chucks at up to 2 Hz. A schematic diagram of the apparatus for such measurements is shown in Figure 5a; Figures 5b and 5c show prototype benchtop and portable versions of the torque tester. Preliminary data on the behaviour of PVC cables have been used to optimize the test conditions for the torque method.

The effect of torsion frequency on the torque values obtained is small in the frequency range 0 Hz to 2 Hz; for PVC the optimum frequency is 0,8 Hz. In both as-received and aged cables the torque values measured increase linearly with the applied torque angle up to  $10^\circ$ . At higher torque angles, components other than the jacket material will significantly contribute to the values obtained. For a non-destructive technique, a maximum torque angle of  $10^\circ$  is recommended. The effect of the length of cable between the chucks has also been investigated. At shorter cable lengths, the measured response will be strongly dependent on the insulation, conductors and any shielding components, whereas as the cable length increases, the sensitivity of the torque-strain response will decrease. The optimum cable length between chucks is 50 mm for PVC cables.

There is a strong correlation between the torque values measured using the prototype tester and elongation at break, both for thermally aged material and for cables subjected to sequential radiation and thermal chemical ageing (Figures 6 and 7). A linear relationship between elongation and torque is found over a wide range of elongation values. Deviations from this linear relationship are only observed when heterogeneous oxidation has occurred in the accelerated tests (Figure 8). The torsion test is a measure of the bulk properties of the jacket material whereas elongation at break is determined by crack initiation in the more highly oxidised surface layer in this material. In most cable applications in nuclear plants, homogeneous oxidation is likely to occur.

A portable version of the torque tester which could be clamped onto sections of cable in-plant has been developed (Figure 5c). This enables data to be taken non-destructively on accessible lengths of cable *in situ*.

### Limitations

As in all local tests, measurements are limited to man-accessible areas. Since torque values will be significantly affected by differences in cable construction and geometry, baseline data for a wide range of cable types would be needed in practice. If there are environmental conditions in-plant which give rise to heterogeneous oxidation, the technique would tend to underestimate the degree of degradation of the jacket material. The technique is invasive as it requires significant manipulation of the cable, but is essentially non-destructive.

## 5.3 Local tests with micro-sampling

### 5.3.1 General

These methods are also limited to man-accessible areas and mainly provide information on the jacket materials. Insulation materials can only be accessed at terminations. Where the methods are cross-correlated with baseline data on changes in elongation at break they have a predictive capability. All of these techniques are laboratory based with a varying degree of complexity of equipment required.

Although some experience of use of these methods in-plant has been built up, there is considerable resistance from utilities in some countries over the application of sampling techniques. Each of these methods is briefly described in the following subclauses. For details of these methods, the cited references should be consulted.

### 5.3.2 Modulus profiling

Modulus profiling is a technique that has been developed over the past 15 years that allows quantitative mapping of inverse tensile compliance measurements (closely related to tensile modulus) to be made with a resolution of  $\sim 50 \mu\text{m}$  [6][7]. The resolution implies that measurements can be made on small pieces of cable jacket or insulation material. The technique is based on extensive but relatively simple modifications of a commercial thermo-mechanical analyser. It involves sequential two-step loading of a stainless steel, paraboloidally-shaped tip into the polished cross-section of the sample of interest with quantitative monitoring of the indentation using a linearly variable differential transducer. The amount of indentation coupled with the probe geometry and the loading history leads to "modulus" values with typical scatter usually around  $\pm 5\%$ .

Since the technique is sensitive to changes in modulus, it has been found to be applicable to the same types of materials as the indenter. Figures 9 and 10 show typical correlations found between tensile elongation results and modulus measurements from accelerated thermal ageing studies. Figure 9 gives the correlations found for five ageing temperatures for thin (0,4 mm thick) individual chlorosulphonated polyethylene (CSPE) jackets, whereas Figure 10 shows such correlations at three accelerated ageing temperatures for a chloroprene (CP) jacket. Figure 11 shows results for a CSPE jacket aged at the four different combined radiation plus temperature environments indicated on the figure. For all three examples it is clear that an excellent correlation exists that appears to be independent of the ageing condition.

This observation is extremely encouraging since this suggests that similar correlations might be expected under the lower temperatures and dose rates occurring in actual plant environments. In addition it is clear that 50 % absolute elongation (a “failure” criterion that is often used) is reached when the modulus value reaches ~25 MPa to 35 MPa. Similar results taken on one CP jacket and seven CSPE jackets aged in thermal-only and combined environments indicate that 50 % absolute elongation is typically reached when the modulus attains a value of around 35 MPa  $\pm$  15 MPa [8][9].

Therefore a “universal” failure value of ~35 MPa for CP and CSPE jacket materials might be appropriate when monitoring modulus results, a tremendous potential advantage when monitoring materials that have not been or cannot be studied under accelerated ageing conditions.

#### *Limitation*

Sampling can only be made in man-accessible areas and is therefore limited mainly to the jacket materials or insulation materials at accessible conductor terminations. Considerable baseline data may be needed to find the correlation of the technique to elongation.

#### **5.3.3 NMR relaxation**

This method is based on increasing the sensitivity of nuclear magnetic resonance (NMR) relaxation measurements by swelling the sample in a suitable solvent [8][10-12]. Measurements are easily done on many commercial NMR machines, are extremely reproducible and typically require less than 15 min to 20 min for sample preparation, data accumulation and data analysis.

Screening studies of thermally aged materials indicate that the NMR approach is applicable to most important cable materials. This applicability includes XLPO insulations [12] that are generally difficult to evaluate with many available CM techniques, because their material properties are dominated by the presence of a high modulus crystalline phase. Since NMR relaxation times are sensitive to the crosslink density in the amorphous phase of a material, the confounding effects of the crystallites on mechanical properties are eliminated.

Figures 12 and 13 show the correlation of NMR results (proton spin-spin relaxation time  $T_2$ ) with elongation results for a thermally aged CP jacket and a thermally aged XLPO insulation, respectively. Typically, 10 mg samples are utilized for the measurements. However, since results have been demonstrated on samples as small as 0,1 mg [11], the NMR approach can be considered as essentially non-destructive. In addition, the ability to monitor such small samples might prove useful for assaying the condition of samples that would otherwise be difficult to study such as composite thin insulations.

#### *Limitations*

Even though extremely small samples are needed for this technique, sampling can only be made in man-accessible areas and is therefore limited mainly to the jacket materials or insulation materials at accessible conductor terminations. Considerable baseline data are required on the specific polymers used in cables in order to determine how much the NMR relaxation time must change before concern over degradation exists.

#### **5.3.4 Infrared spectroscopy (IR)**

Changes in the infrared spectrum of polymers are known to occur with ageing, primarily in functional groups such as carbonyl (C=O), hydroxyl (O-H) and carboxyl (COOH). An alternative to reflectance measurements in-plant is to take samples from the cable material and measure the IR spectrum in the laboratory. Such samples would usually be in the form of scrapings or slivers of material cut from the surface, but sufficiently small to not affect the operation of the cable. Changes in the carbonyl peak at about 1 720 cm<sup>-1</sup> are most frequently used for monitoring changes in the state of degradation of the material [13-15].



Figure 14 shows the change in carbonyl absorbance for radiation aged XLPE and Figure 15 shows the change for thermal ageing of XLPE. This functional group is sensitive to the early stages of oxidative degradation but may not be sufficiently sensitive in the later stages when mechanical properties are significantly degraded.

#### *Limitations*

IR spectra taken from samples are limited to the surface layers of the cable jacket unless exposed insulation is accessible. The depth of material sampled is greater than for the in-plant IR reflectance measurements, but may still not be representative of the bulk material. The technique is limited to those parts of the cables that are man-accessible for sampling and is not very sensitive to the later stages of degradation. The technique is not suitable for carbon-filled polymers.

#### **5.3.5 Oxidation induction time (OIT) and temperature (OITP)**

Oxidation induction tests utilize micro-samples of material which can be taken from the component (e.g. cable jacket material) without affecting functionality. These tests utilize thermal analysis using commercial differential scanning calorimetry (DSC) equipment to determine either an oxidation induction time at constant temperature, or an oxidation induction temperature (OITP) at a constant temperature ramp rate. The two methods are complementary, in that OITP is often useful where OIT is difficult to determine. The OIT decreases with increasing degradation of the material. Details of the method are given in IEC 60544-5.

#### **5.3.6 Thermogravimetric analysis (TGA)**

Like oxidation induction tests, thermogravimetric analysis utilizes micro-samples of material which can be taken from the component without affecting functionality. These tests use commercial standard laboratory thermal analysis equipment to monitor the mass loss in the sample as the sample temperature is ramped up at a constant rate. The absolute values obtained in the TGA tests are dependent on the oxygen content in the sample chamber, with higher onset values being observed with lower oxygen content. The TGA temperature tends to decrease with increasing radiation degradation. Details of the method are given in IEC 60544-5.

#### **5.3.7 Density**

When polymeric materials age in air, oxidation normally dominates the degradation. The oxidation involves a mixture of scission and crosslinking processes, leading to the generation of oxidation products along the polymer chain and the generation of gaseous degradation products. Easily measurable density increases result from these chemical changes. Details of the method are given in IEC 60544-5.

#### **5.3.8 Gel fraction and solvent uptake measurements**

These relatively simple measurements [8][16-18] require small samples of material to be removed from cables. The sample is initially weighed then boiled in a good solvent after which it is weighed when swollen by the solvent. The sample is then dried in a vacuum oven to drive off the solvent and finally weighed when dry. The solvent uptake is defined as the swollen weight over the final dried weight and the gel fraction is given by the final weight divided by the initial weight. The gel fraction goes up when crosslinking processes dominate scission and down when the opposite occurs. Higher crosslinking leads to less swelling, whereas swelling goes up when scission dominates crosslinking. Since changes in modulus and hardness have similar sensitivities to the mix between crosslinking and scission, swelling and gel measurements are found to be good condition monitoring techniques for materials and environments similar to those found appropriate to the indenter and modulus profiling. The measurements have been found to be useful for PVC and many rubbers including butyl, CP, and CSPE [8][18]. Figure 16 for example shows solvent uptake ratio versus elongation results

for a CP jacket at three different ageing temperatures [8]. These techniques also show promise for XLPO materials as seen for an XLPE material [18] aged at 130 °C (Figure 17) and an XLPO material aged at 125 °C (Figure 18). When solvent uptake shows sensitivity to ageing, the difficulties associated with weighing swollen samples accurately leads to a sample size requirement of ~50 mg [8]. On the other hand, when gel is an appropriate technique, the ease of obtaining very accurate values for the initial and final dry weights means that samples weighing 1 mg or less can be used.

#### *Limitations*

Sampling can only be made in man-accessible areas and is therefore limited mainly to the jacket materials or insulation materials at accessible conductor terminations. Considerable baseline data are required on the specific polymers used in cables in order to determine how much the gel and solvent uptake must change before concern over degradation exists.

## **6 Summary**

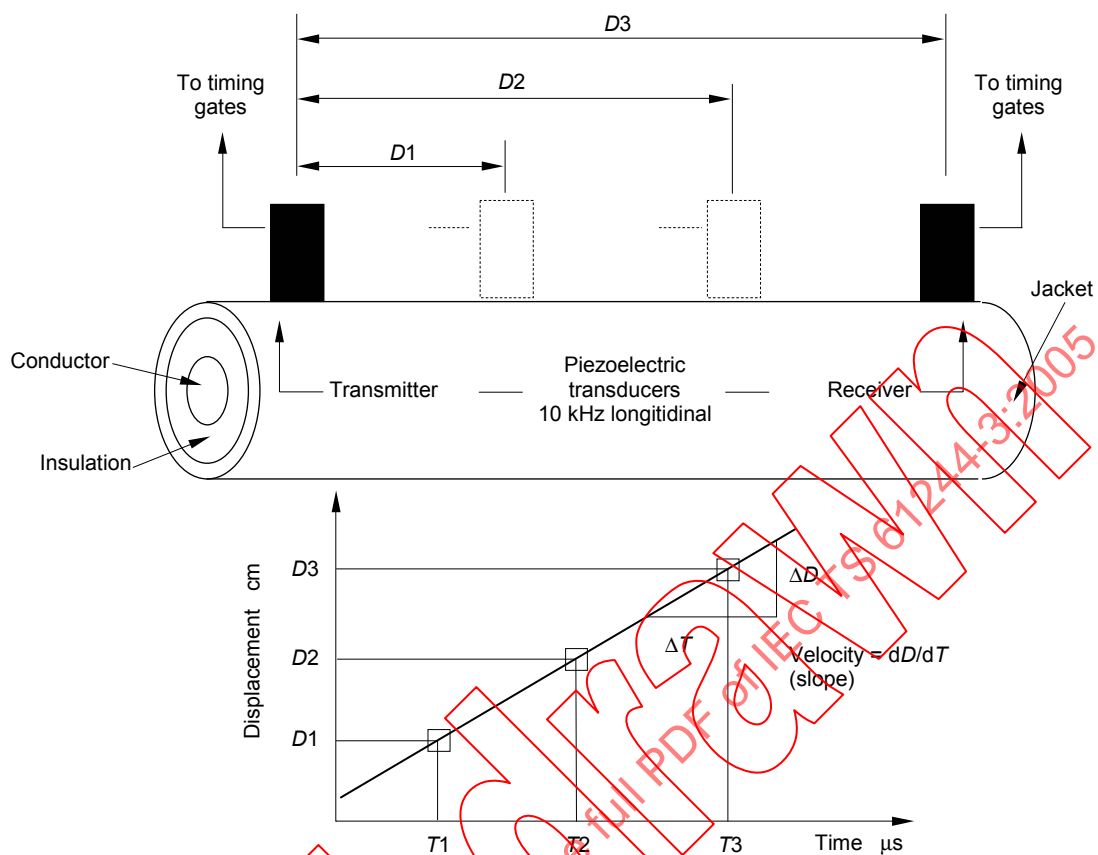
There is a wide range of techniques in use or being developed for condition monitoring in cables. These methods are summarized in Table 1, with an indication of their main limitations, current status and relative equipment cost. The most advanced techniques are published in IEC 60544-5.

No one technique is likely to be suitable for all types of cable materials or cable configurations, but sufficient techniques are available, or are being developed, to cover most of the requirements for condition monitoring in the near future.



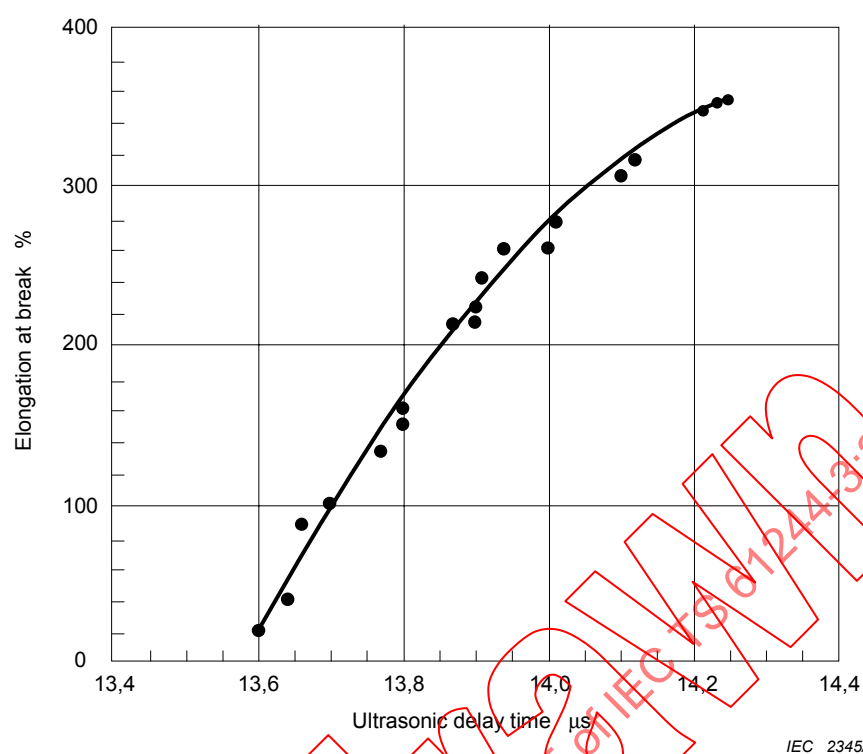
### Table 1 – Summary of currently available techniques for cable condition monitoring

[illegible]



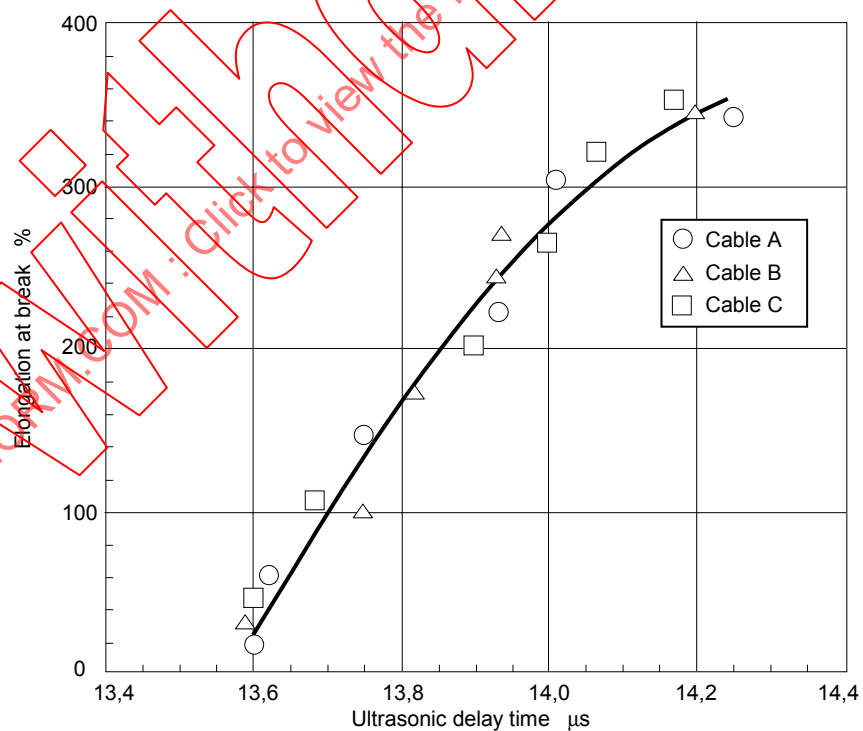
IEC 2344/05

**Figure 1 – Schematic diagram showing the operating principles of the sonic velocity meter [1]**



IEC 2345/05

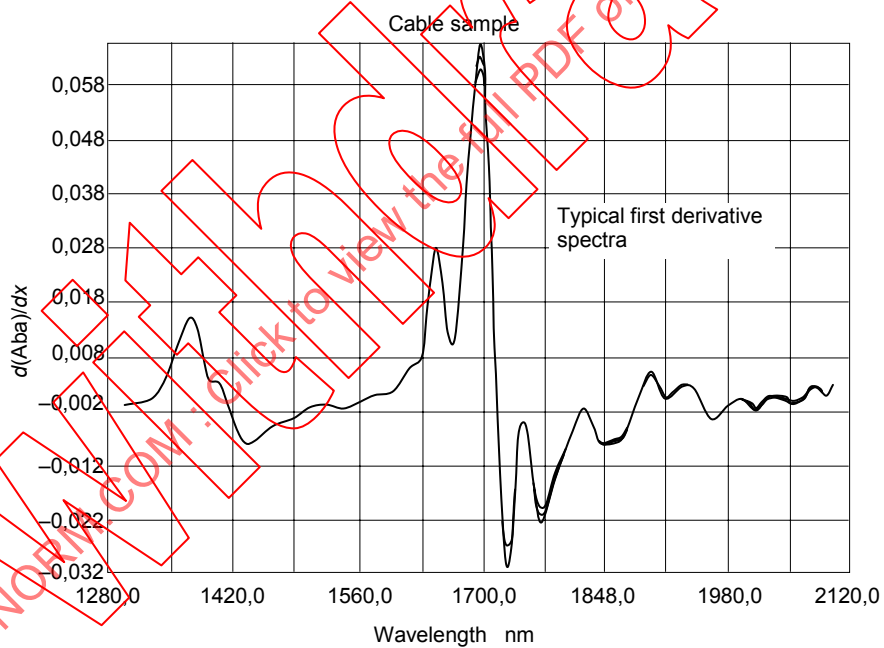
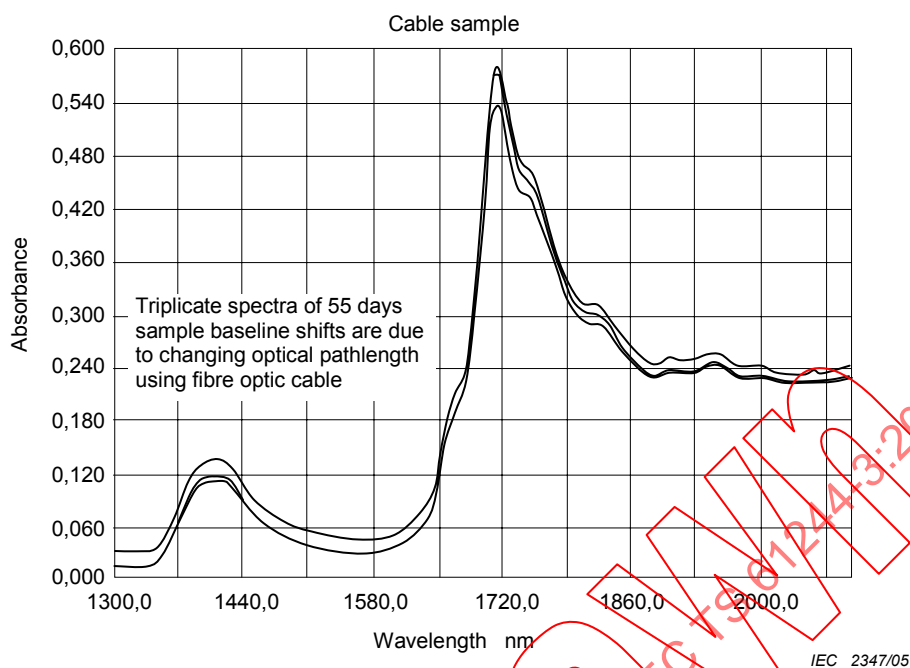
**Figure 2a – Example of the relation between the propagation time of ultrasonic waves and elongation at break for a PVC sheath material**



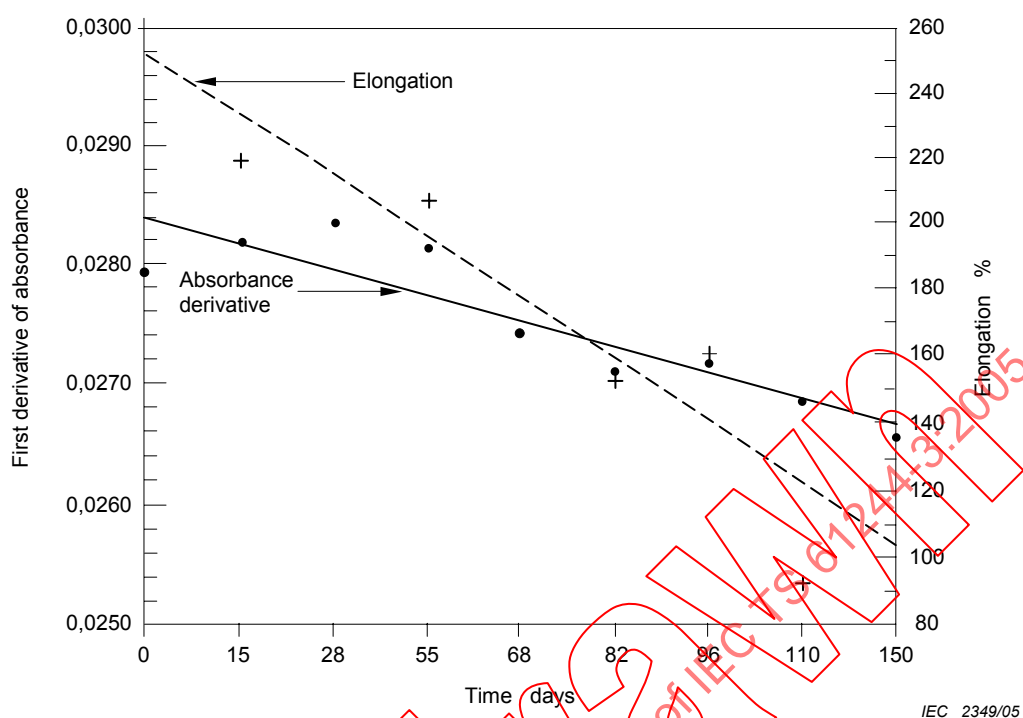
IEC 2346/05

**Figure 2b – Comparison between the master curve and various cables from different manufacturers**

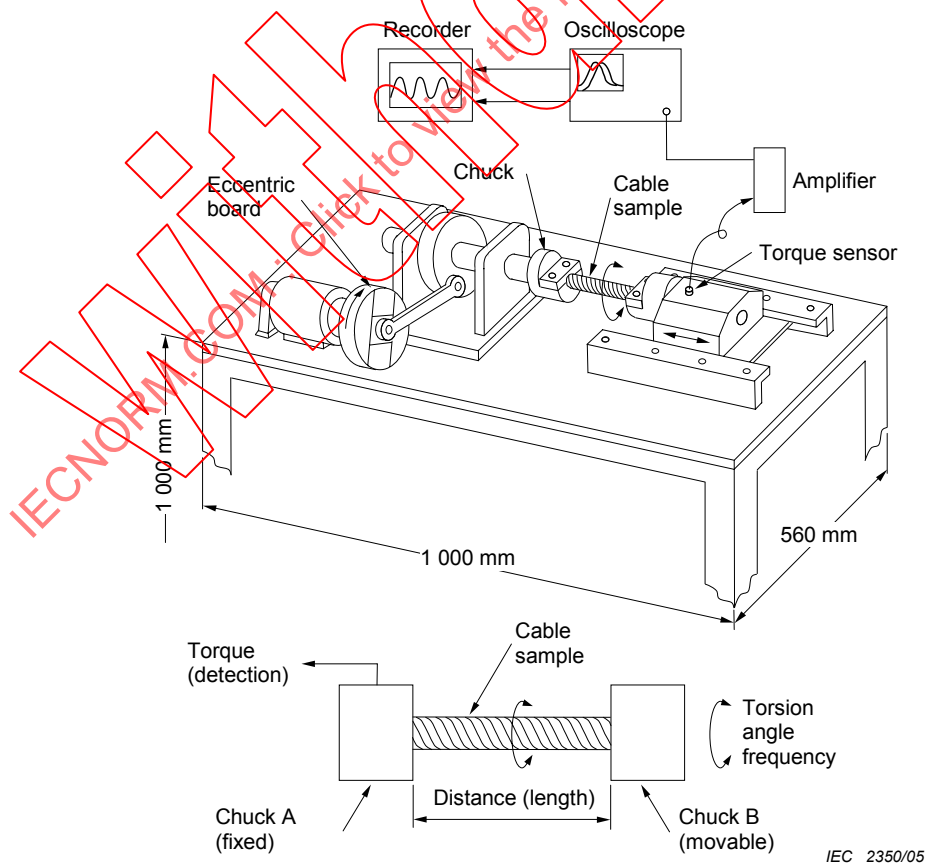
**Figure 2 – Sonic velocity test results [3]**



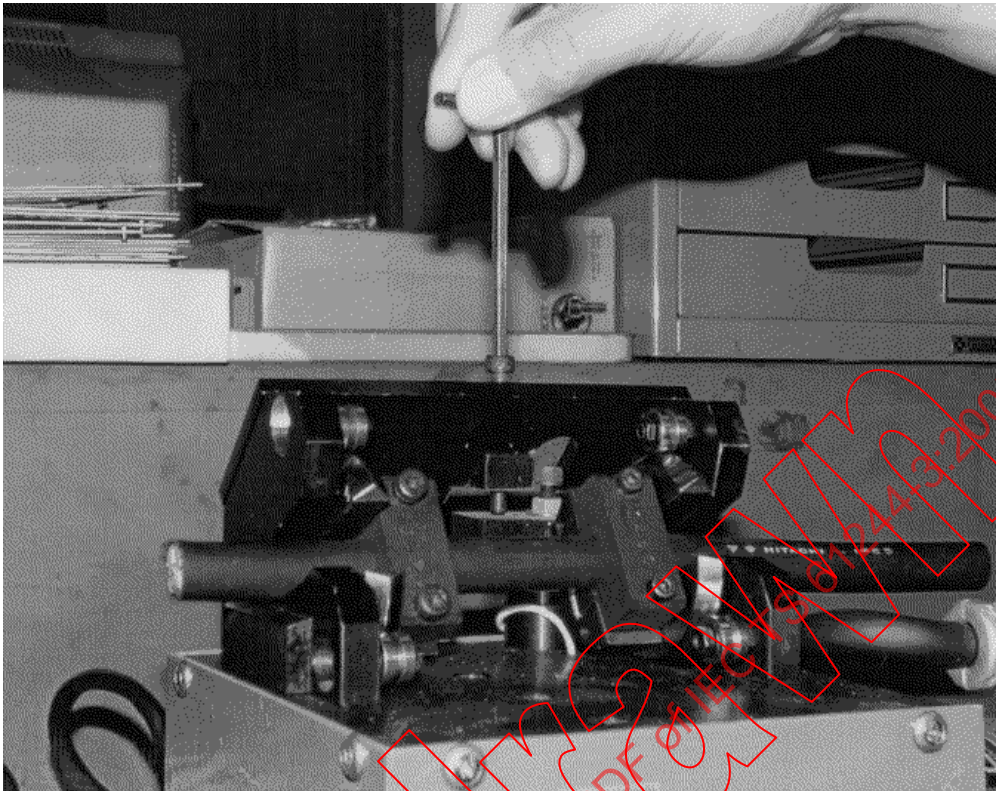
**Figure 3 – Variation in IR absorbance with wavelength for PVC material and the use of first derivative to eliminate baseline shifts [1]**



**Figure 4 – Correlation of first derivative of IR absorbance at 1 640 nm to 1 650 nm and elongation at break for thermal ageing of PVC at 110 °C [1]**

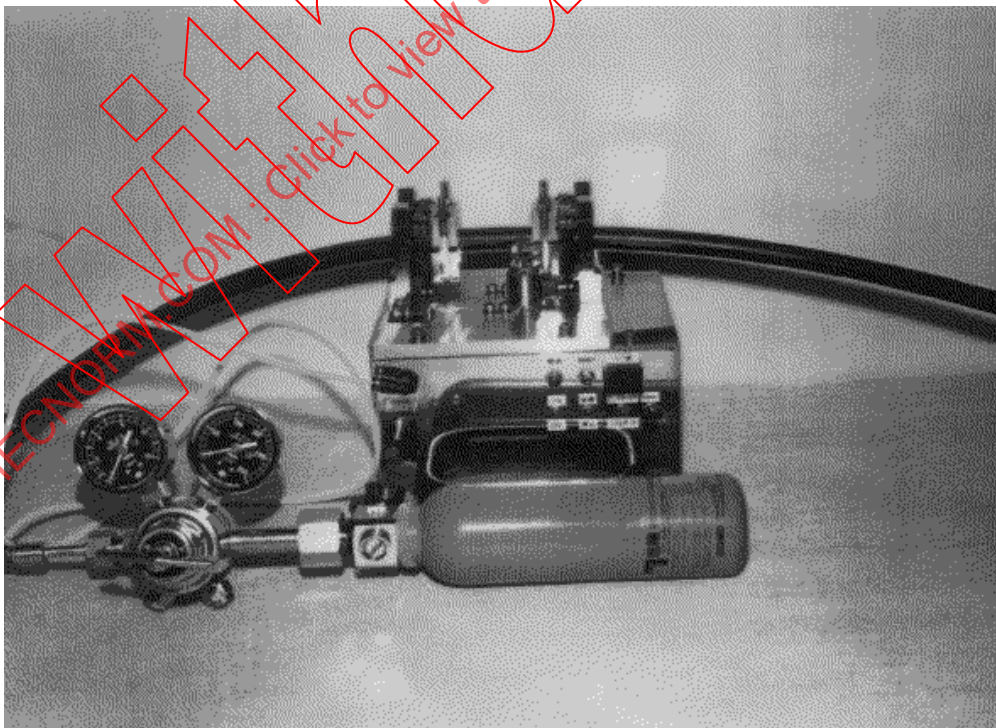


**Figure 5a – Schematic diagram of prototype device for torque measurement of cables [5]**



**Figure 5b – Prototype bench top torque tester**

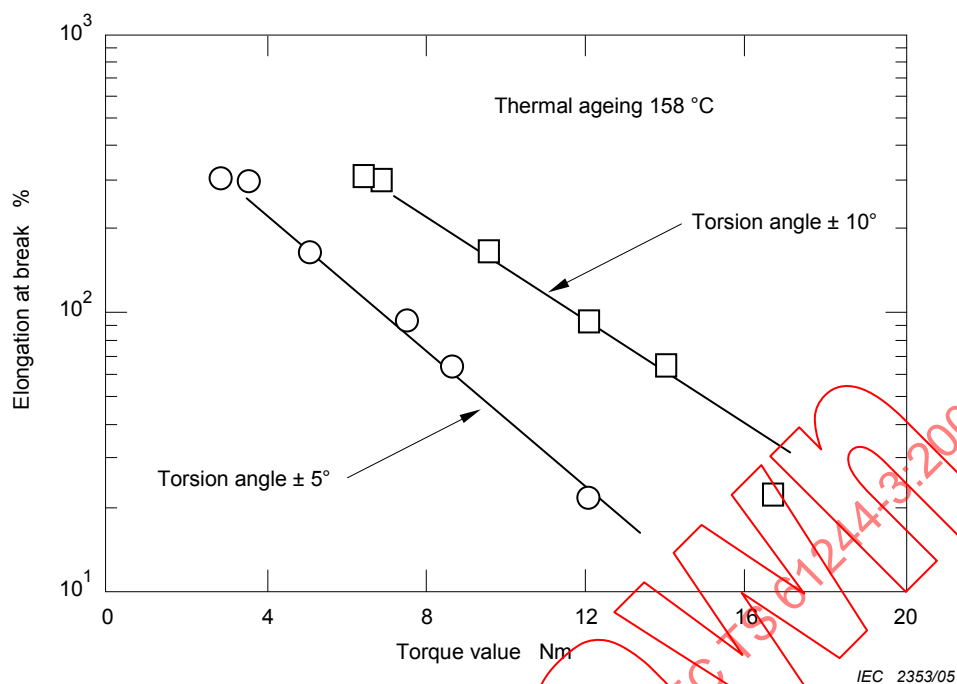
IEC 2351/05



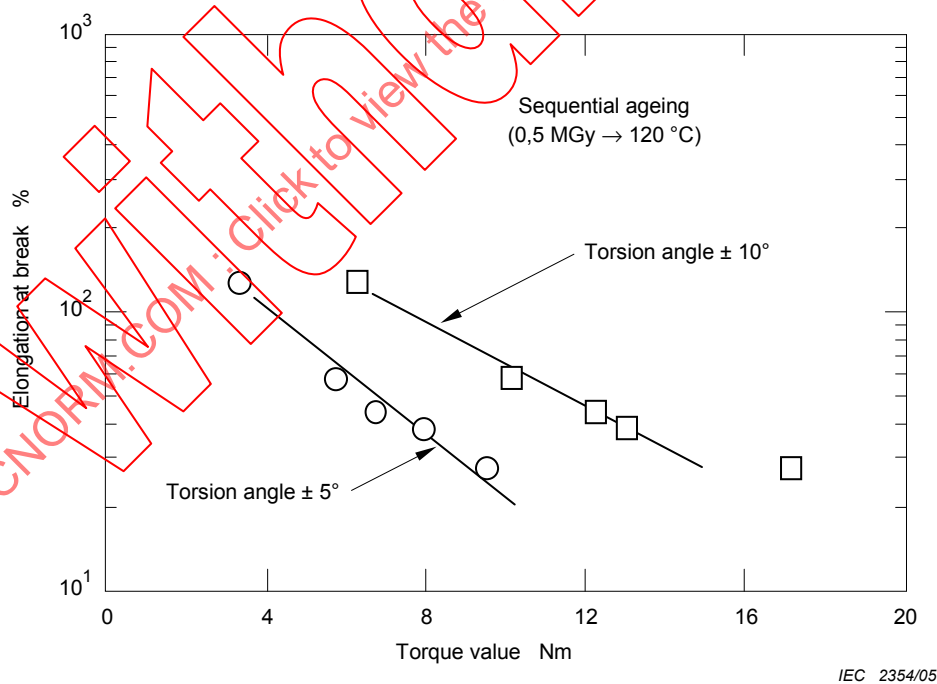
**Figure 5c – Prototype portable version of the torque tester**

IEC 2352/05

**Figure 5 – Schematic diagram and photographs of prototype torque tester**

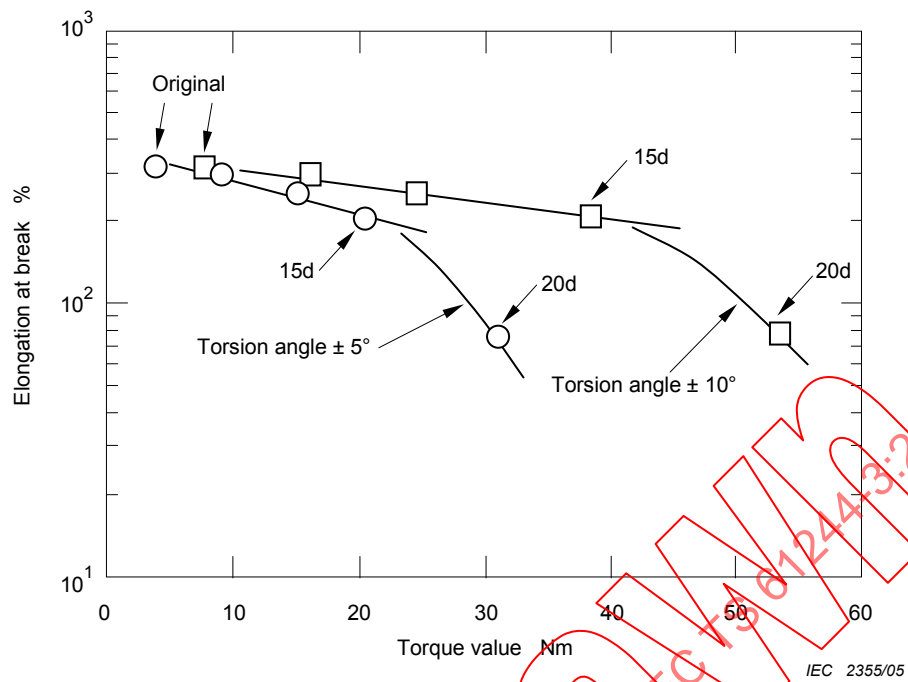


**Figure 6 – Elongation at break versus torque value for flame-retardant PVC cables thermally aged at 158 °C [5]**

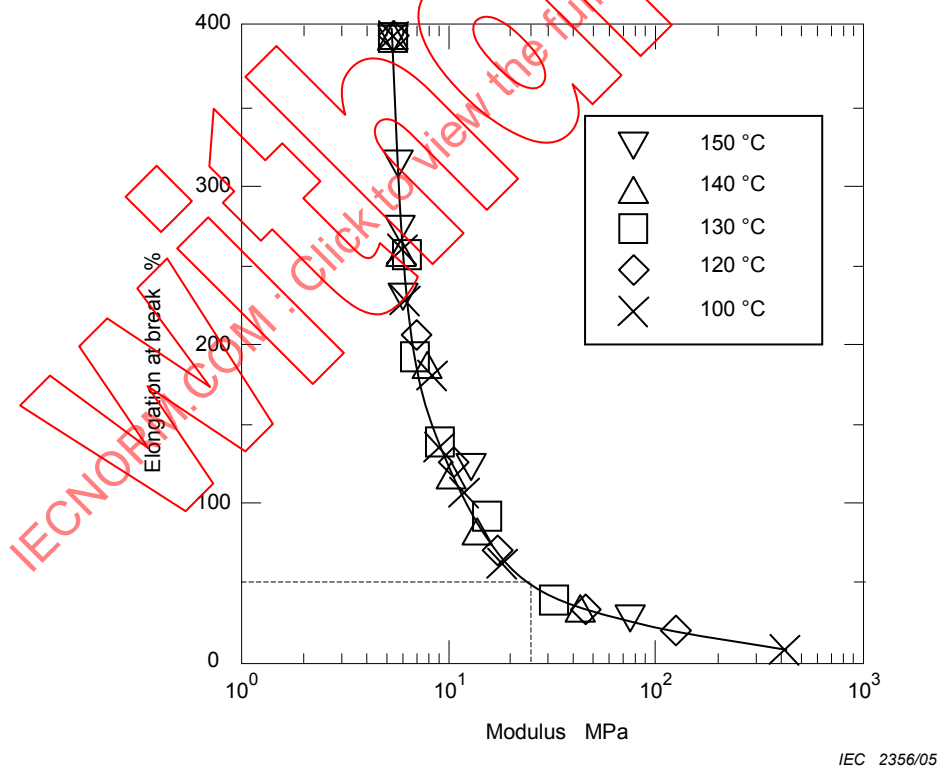


**Figure 7 – Elongation at break versus torque value for PVC cables exposed to sequential radiation ageing to 0,5 MGy and thermal ageing at 120 °C [5]**



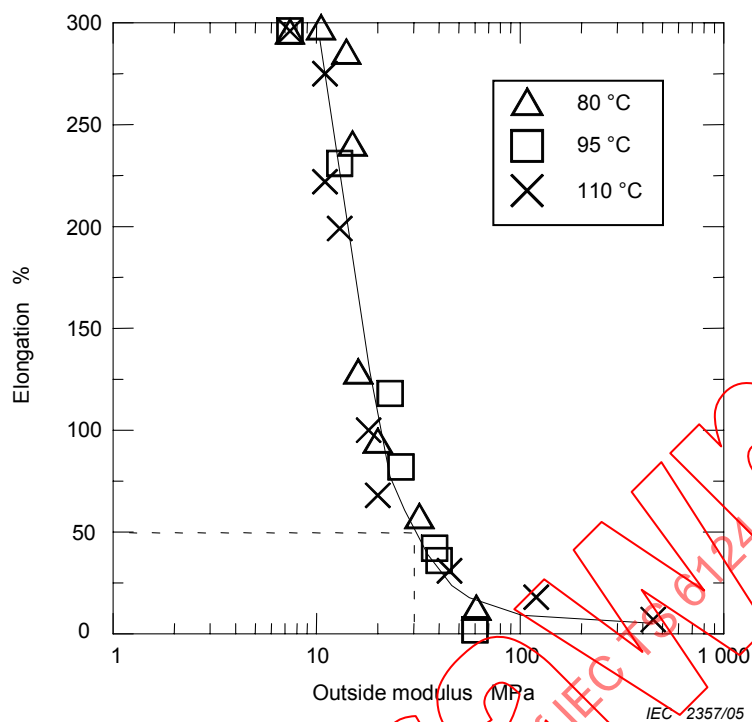


**Figure 8 – Elongation at break versus torque value for PVC cables thermally aged at 120 °C [5]**

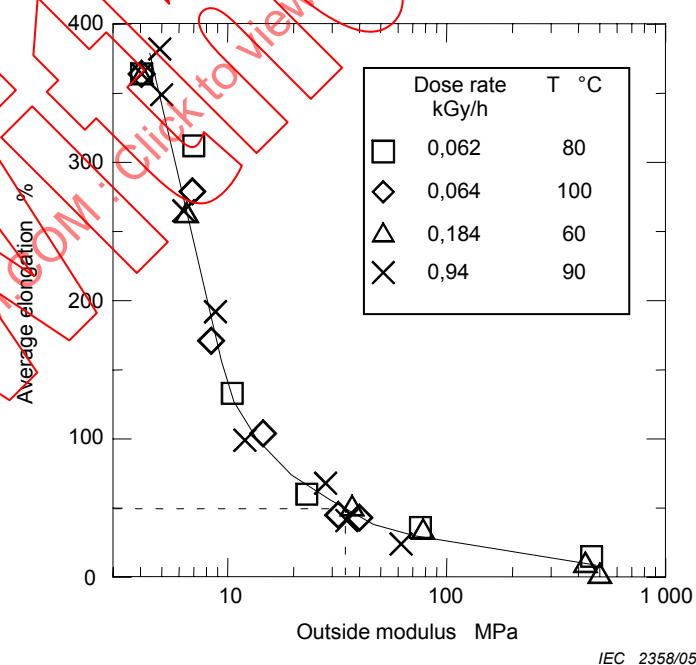


**Figure 9 – Correlation of tensile elongation with modulus measurements made using the modulus profiling apparatus for a CSPE jacket aged at the five indicated temperatures [6]**





**Figure 10 – Correlation of tensile elongation with modulus measurements made using the modulus profiling apparatus for a CP jacket aged at the three indicated temperatures (from Reference [8])**



**Figure 11 – Correlation of tensile elongation with modulus measurements made using the modulus profiling apparatus for a CSPE jacket aged at the four indicated combined environments [9]**