**IAEA-TECDOC-1188** 

# Assessment and management of ageing of major nuclear power plant components important to safety:

## In-containment instrumentation and control cables

Volume II



INTERNATIONAL ATOMIC ENERGY AGENCY

December 2000

The originating Section of this publication in the IAEA was:

Engineering Safety Section International Atomic Energy Agency Wagramer Strasse 5 P.O. Box 100 A-1400 Vienna, Austria

ASSESSMENT AND MANAGEMENT OF AGEING OF MAJOR NUCLEAR POWER PLANT COMPONENTS IMPORTANT TO SAFETY: VOLUME II IN-CONTAINMENT INSTRUMENTATION AND CONTROL CABLES IAEA, VIENNA, 2000 IAEA-TECDOC-1188 ISSN 1011-4289

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Printed by the IAEA in Austria December 2000

#### FOREWORD

At present, there are over four hundred operational nuclear power plants (NPPs) in IAEA Member States. Operating experience has shown that ineffective control of the ageing degradation of major NPP components (e.g. caused by unanticipated phenomena and by operating, maintenance, design or manufacturing error) can jeopardize plant safety and also plant life. Ageing in these NPPs must be therefore effectively managed to ensure the availability of design functions throughout the plant service life. From the safety perspective, this means controlling within acceptable limits the ageing degradation and wear-out of plant components important to safety so that adequate safety margins remain, i.e. integrity and functional capability in excess of normal operating requirements.

This publication is one in a series of guidance reports on the assessment and management of ageing of the major NPP components important to safety. The reports are based on experience and practices of NPP operators, regulators, designers, manufacturers, and technical support organizations and a widely accepted Methodology for the Management of Ageing of NPP Components Important to Safety, which was issued by the IAEA in 1992.

The current practices for the assessment of safety margins (fitness-for-service) and the inspection, monitoring and mitigation of ageing degradation of selected components of Canadian deuterium uranium (CANDU) reactors, boiling water reactors (BWRs), pressurized water reactors (PWRs), including the water moderated and water cooled energy reactors (WWERs) are documented in the reports. These practices are intended to help all involved directly and indirectly in ensuring the safe operation of NPPs, and to provide a common technical basis for dialogue between plant operators and regulators when dealing with age related licensing issues. The guidance reports are directed at technical experts and managers from NPPs and from regulatory, plant design, manufacturing and technical support organizations dealing with specific plant components addressed in the reports.

The component addressed in the present report is the in-containment instrumentation and control (I&C) cables. The report presents, in two volumes, results of a Co-ordinated Research Project (CRP) on Management of Ageing of In-containment I&C Cables. Volume I presents information on current methods for assessing and managing ageing degradation of I&C cables in real NPP environments prepared by the CRP team and an application guidance for these methods prepared jointly with NPP owners/operators and regulators. Volume II contains annexes supporting this guidance with more detailed information and examples provided by individual CRP participants. For a quick overview, readers should see Section 8 of Part I, Volume I, which describes a systematic ageing management programme for I&C cables utilizing methods presented in the report; Section 9 of Part I, Volume I, which presents CRP conclusions and recommendations; and Part II providing the application guidance from the user's perspective.

The contributors to drafting and review of this TECDOC are identified at the end of this publication. Their work is greatly appreciated. In particular, the contribution of S.G. Burnay of the United Kingdom as the CRP chairman and a compiler of this report is acknowledged. The IAEA officer who was the project manager of the CRP and directed the preparation of the report was J. Pachner of the Division of Nuclear Installation Safety.

## EDITORIAL NOTE

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#### ANNEX A

#### (TO SECTION 3 OF PART I, VOLUME I: AGEING MECHANISMS)

Annex A contains additional material of relevance to Section 3 of Part I, Volume I of this report on ageing mechanisms. It includes more detailed information on the following aspects related to Section 3:

- Measurement of activation energy
- Reverse temperature effect
- Oxidation reaction scheme
- Methods for profiling heterogeneous oxidation.

#### A.1. MEASUREMENT OF ACTIVATION ENERGY

#### A.1.1. General background

Accurate values of activation energies are key to obtaining representative ageing in accelerated ageing tests. The methods recommended for determination of activation energy of thermal ageing ( $E_a$ ) and for statistical processing of the ageing data are described in series of guides [A.1–A.6]. The number of samples necessary for reliable determination of  $E_a$ , and the temperature margin for following accelerated thermal ageing are analysed and recommended in reference [A.7]. Because the limitations for the extrapolation of thermal ageing data given in IEC 216-1 [A.1] cannot usually be used for estimating to the 40-years life, other additional margins are recommended.

The 50% *relative* elongation at break is often used as a suitable end point criterion for determining the value of activation energy. Also other cable material properties sensitive to the cable functionality can be used as the end point criteria (see reference [A.6]). An example of the application of other methods for determining  $E_a$ , including a comparison of its results, is described in reference [A.8].

Most methods of measuring  $E_a$  utilise an Arrhenius plot. However, when using an Arrhenius plot over a wide temperature range, it is sometimes found to deviate from a straight line. For example, Figure A.1 shows the thermal degradation rate as a function of ageing temperature for ethylene-propylene rubber (EPR) [A.9]. The rate was determined using the reciprocal of elapsed time to 100 % elongation at break. A similar plot for a CSPE material is shown in Figure A.2 [A.10]. The rate at ambient temperature is significantly higher than was expected from data at higher temperatures. Accordingly, if one extrapolates from higher temperatures, the lifetime at near ambient temperature would be significantly overestimated. The much higher degradation rate at higher temperature compared to ambient, is probably due to surface embrittlement and cracking.

These examples indicate that the activation energy at ambient temperature should be evaluated carefully even if it is time and labour consuming, and that a sophisticated procedure to evaluate activation energy is required. Some examples of methods for measuring activation energy are given in the following sections.

#### A.1.2. Using elongation measurements

Measurements of activation energy using changes in tensile elongation properties are the most common method in use. If there are values of  $E_a$  available from more sources (e.g. from more

production batches or from other equivalent methods), the one sided confidence level of 95% for  $E_a$  value should be applied for accelerated thermal ageing. An example of statistical assessment of  $E_a$  data for a cable insulation material is given in Table A1. In this example, 12 insulations (based on an EPR/EVA compound) have been taken from a 12-core cable for testing. The equivalence of 5 end point criteria equal to 60, 50, 40, 30 and 20% of relative elongation at break is assumed within this example. The original elongation at break was about 240%, so the 20% relative end point criterion approximately corresponds to the end point criterion of 50% absolute elongation at break (which can be also applied in practice, but which yields no margin if the lifetime is read directly from the Arrhenius plot). This example shows that the possible variance of  $E_a$  found among more batches of cable cores need not be caused by a real difference among them but rather by the statistical error due to the applied test method and to the number of test samples.

In Table A.1, the different levels of the relative elongation-at-break changes have been used as the end point criteria, and then the variances among the values of  $E_a$  obtained at each level have been statistically analysed against the variances among different cable cores (i.e. production batches).

## A.1.3. Using microcalorimetry

The microcalorimetry method uses an instrument capable of measuring the very small heat flows ( $\mu$ W/g) associated with slow ageing processes. This enables measurements to be carried out at temperatures nearer to ambient temperature than are generally used in activation energy studies. The method is particularly useful for studies on unstabilised materials and for those stabilised materials where the antioxidant has been consumed. Reference [A.11] gives some examples of E<sub>a</sub> measurements made using this method on XLPE, CSPE and EPDM materials.

#### A.1.4. Using gas analysis

This method is based on measurements of oxygen consumption and  $CO_2$  evolution as a result of the oxidation that occurs when polymer materials are thermally and/or radiation aged [A.12, A.13].

Small amounts of material (approximately 0.5 g) are put into an ampoule (volume 60 mL) with a breakable seal. After degassing the ampoule is filled with oxygen (at 600 torr). The ampoules are stored at the test temperature with/without irradiation. Then the gas in the ampoule is analysed by gas chromatography to evaluate the oxygen consumed and  $CO_2$  evolved. The time required to reach a fixed amount of oxygen consumption or  $CO_2$  evolution is plotted as a function of test temperature.

Figure A.3 shows an example of the Arrhenius plot using gas analysis for the same EPDM shown in Figure A.1. The activation energy obtained was 76 kJ/mol at around ambient temperature, which coincides well with Figure A.1. This method is sensitive and therefore timesaving compared to tensile test, and the result successfully agrees with tensile data.

Irradiation to 100kGy facilitates the analysis, because irradiation consumes anti-oxidants in the compound and eliminates the induction period of the time-profiles of oxygen consumption or  $CO_2$  evolution. Irradiation also accelerates degradation, whereas the activation energy is kept almost unchanged as Figure A.4 shows.

## TABLE A1. AN ANALYSIS OF ACTIVATION ENERGY E<sub>a</sub> FOR INDIVIDUAL CABLE CORES DETERMINED FOR FIVE DIFFERENT LEVELS OF AN END POINT CRITERION [A.15]

the end point criterion: relative elongation at break,  $\varepsilon_b$  material of cores: EPR/EVA; initial  $\varepsilon_b$ : approx. 240 %

	end point criterion: relative $\varepsilon_{\rm b} = X$ (%)			without					
				differentiation	standard.				
core	X=60	X=50	X=40	X=30	X=20	of the X-value	deviation	F-test *	t-test *
yellow-green	1.157	1.079	1.056	1.049	1.010	1.070	0.054	0.391	0.180
black-1	1.124	1.093	0.985	0.940	0.979	1.024	0.080	0.977	0.894
black-2	1.053	1.102	1.050	1.027	1.012	1.049	0.034	0.084	0.323
black-3	1.151	1.020	1.013	1.024	1.036	1.049	0.058	0.459	0.518
black-4	1.084	0.836	0.746	0.857	1.074	0.919	0.152	0.041	0.181
black-5	1.055	1.048	1.011	0.978	0.958	1.010	0.042	0.177	0.401
black-6	1.096	0.976	0.960	0.955	0.991	0.996	0.058	0.467	0.282
black-7	1.033	1.031	0.985	0.977	0.976	1.000	0.029	0.047	0.118
black-8	1.162	1.104	1.044	1.012	0.995	1.063	0.069	0.744	0.392
black-9	1.175	1.104	1.031	1.019	1.007	1.067	0.071	0.798	0.344
blue-1	1.023	1.186	0.975	0.944	0.954	1.017	0.099	0.526	0.752
blue-2	1.282	1.149	1.047	0.991	0.972	1.088	0.129	0.146	0.368
arithm. mean	1.116	1.061	0.992	0.981	0.997	1.029			
stand.	0.074	0.091	0.084	0.052	0.034	0.080			
deviation									
F-test **	0.638	0.717	0.980	0.079	0.002				
t-test **	0.002	0.255	0.168	0.016	0.034				

The normal distribution of all data is supposed All of  $E_a$  values are given in eV (1 eV = 96.48 kJ/mol)

Confidence limits for the arithmetical mean:

	(	end point criter	without differentiation			
	X=60	X=50	X=40	X=30	X=20	of the X-value
95-% lower	1.081	1.018	0.952	0.956	0.981	1.008
95-% upper	1.152	1.104	1.032	1.006	1.013	1.051
98-% lower	1.072	1.007	0.942	0.950	0.977	1.002
98-% upper	1.160	1.114	1.042	1.012	1.017	1.056
99-% lower	1.066	1.000	0.935	0.946	0.974	0.999
99-%upper	1.166	1.122	1.048	1.016	1.020	1.060
99.5-% lower	1.061	0.993	0.929	0.942	0.972	0.995
99.5-% upper	1.171	1.128	1.054	1.020	1.022	1.063
99.9-% lower	1.050	0.980	0.917	0.934	0.967	0.988
99.9-% upper	1.183	1.142	1.067	1.028	1.027	1.070

The result of the F test: gives the probability, that the variances of both tested files are equal The result of the t-test: gives the probability, that the arithmetical means of both tested files are equal If the result value of the F-test was > 0.5, the double-sampling t-test supposing the equality of variances of both tested files has been used

If the result value of the F-test was < 0.5, the double-sampling t-test supposing the non-equality of variances of both tested files has been used

The lower and upper X-percent confidence limits for the arithmetical mean determine the interval in which this arithmetical mean lies with the probability equal to X

\* the individual core tested against the other all cores together.

\*\* the individual end point criterion level tested against all other levels together.



FIG. A.1. The thermal degradation rate as a function of test temperature for ethylene-propylene rubber (EPR) [A.9]. The rate was determined with respect to tensile test and normalized at 50° C (standard ageing condition).



FIG. A.2. Thermal rate constant dependence with inverse temperature for CSPE sheath material [A.10].



FIG. A.3. Arrhenius plot of oxygen gas evolved CO<sub>2</sub> for unirradiated EPR.



FIG. A.4. Arrhenius plot of oxygen gas consumption and evolved  $CO_2$  for irradiated EPR to 100 kGy.

#### A.1.5. Using chemi-luminescence

This method utilises luminescence from thermally excited ketone group (>C=O)\* [A.13, A.14]. The ketone groups are formed through oxidation of the polymer. A sample of 15 mm  $\times$  15 mm was kept in a chamber under flowing oxygen. The luminescence intensity at 350–600 nm was measured at various temperatures. Thermal treatment before measurement was found to be effective in obtaining a stable signal.

Figure A.5 shows the chemi-luminescence intensity as a function of measuring temperature for EPR unirradiated and irradiated to 500kGy. The activation energy obtained was 58 kJ/mol, which is in good agreement with tensile data at ambient temperature. This method is also sensitive and timesaving compared to tensile test, and gives successful results.

Figure A.5 also shows that irradiation up to 500 kGy enhances the intensity of the luminescence, with the activation energy kept unchanged. Irradiation to a certain dose can be convenient for the analysis in this case as well. Moreover, the constant activation energy from ambient to 140°C indicates that the apparent high activation energy measured in tensile tests (Figure A.1) comes from mechanical, not chemical factor, such as cracks at the surface.

#### A.2. REVERSE TEMPERATURE EFFECT

The reverse temperature effect is a phenomenon which has only been recognised in the last few years. It has been seen in polyolefin materials which have been radiation aged in air at temperatures below their crystalline melting point [A.16–A.18]. Under these conditions, degradation is more rapid at the lower temperatures than at higher temperatures, which is opposite to what would be expected from normal kinetics of chemical reactions (see figure A.6). However it is now realised that the reverse temperature effect is a function of the semi-crystalline nature of the polyolefins.

Polyethylene based materials, such as XLPE, are semi-crystalline and their mechanical properties are determined by their microstructure at the supermolecular level. The material contains randomly oriented crystalline regions linked by amorphous tie molecules. During radiation ageing, reactive species such as radicals are generated uniformly throughout both crystalline and amorphous regions. In the crystalline regions at temperatures well below the melting point, these species are trapped and are unable to react to form oxidative products because of the low chain mobility and the low oxygen diffusion rate in the crystalline region. Degradation then proceeds primarily through oxidative scission reactions in the amorphous regions, where both chain mobility and oxygen diffusion rates are higher. Since the amorphous regions form the tie molecules between the crystalline blocks, chain scission in these regions has a marked effect on the mechanical properties.

If the radiation ageing occurs at slightly higher temperatures, nearer the melting region for the crystalline portion, then chain mobility is high enough for the trapped species to react to form chemical crosslinks. In addition, the enhanced mobility enables some recrystallisation to occur which can reform tie molecules which were broken by oxidative scission in the amorphous regions. The combination of these effects is to effectively 'heal' some of the damage which is created by the radiation ageing. The overall macroscopic effect is a reduced rate of degradation at the higher temperature during radiation ageing.

#### A.3. OXIDATION REACTION SCHEME

The basic reaction steps in the classical oxidation reaction scheme are shown in Table A.2. Initiation of the radical chain mechanism can be either via thermal or radiation activation. Propagation and branching of the reactions can then occur with the production of unstable hydroperoxide groups as well as intermediary radicals. Termination of the chain reaction occurs with the recombination of radicals to form stable groups.



FIG. A.5. Chemi-luminescence intensity as a function of measuring temperature for EPR unirradiated and irradiated to 500 kGy.



FIG. A.6. Comparison of predicted dose required to reach 100% tensile elongation and measured dose to 100% elongation for XLPE insulation as a function of ageing.

The detailed kinetics of the individual steps within the oxidation scheme will be determined by the formulation of the polymeric compound, particularly the nature and amount of additives included in the material.

TABLE A.2. BASIC OXIDATION REACTION SCHEME

Initiation	RH	$\rightarrow$	R•
Propagation and branching	$R^{\bullet} + O_2$ $RO_2^{\bullet} + RH$ $RO_2H$ $RO^{\bullet}$	$\begin{array}{c} \rightarrow \\ \rightarrow \\ \rightarrow \\ \rightarrow \\ \rightarrow \end{array}$	$RO_{2}^{\bullet}$ $RO_{2}H + R^{\bullet}$ $RO^{\bullet} + OH^{\bullet}$ $R^{\bullet} + -CO-$
Termination	2R• 2RO <sub>2</sub> •	$\rightarrow$ $\rightarrow$ or	$RR RO_2R + O_2 ROH + -CO- + O_2$

#### A.4. METHODS FOR PROFILING HETEROGENEOUS OXIDATION

There are a number of methods which can be used to monitor heterogeneous oxidation in polymeric materials. These are briefly outlined below; for a more detailed description and examples of their use, see references [A.19, A.20].

When heterogeneous oxidation occurs in a polymer because of diffusion limited processes, there are several properties which will depend on the spatial location within the material. Any technique which can profile these spatial variations can be used to monitor diffusion limited oxidation. The most common of the profiling methods in use are:

- (a) Infrared profiling
- (b) Modulus profiling
- (c) Density profiling
- (d) X ray micro-analysis
- (e) OIT profiling

Infrared spectroscopy of thin film samples gives detailed chemical information, such that any oxidation sensitive IR peak can be used to monitor oxidation. By cutting thin slices progressively from the surface of the sample through the thickness of the sample, measurement of the IR peak can give a profile of the oxidation. The most common peak used for this is that relating to the carbonyl (C=O) region, which is generally found at approximately 1720 cm<sup>-1</sup>. This method is particularly useful for polyolefins.

Modulus profiling makes use of the changes in mechanical properties which occur on oxidation of a polymer. The compressive modulus can be measured using a micro-indentation technique which enables measurements to be made as a function of distance across a cross-section through the sample. This approach is particularly useful for elastomeric materials.

Density profiling usually uses a density gradient column to measure the density of successive slices cut from a sample, each slice being parallel to the sample surface. It is based on the fact

that oxidation reactions often lead to significant and easily measurable increases in density. This method is often used with polyolefins.

X ray micro-analysis can be used to locate oxidation products, such as carboxyl and peroxide groups, by exposing a cross-sectional surface to KOH/isopropanol solution. The oxidation products are converted to potassium-containing species which can be detected by an electron probe X ray microanalyser. This technique has been demonstrated on an EPDM material.

OIT-profiling also uses slices cut parallel to the sample surface, which are then tested for OIT. Oxidation profiles can be obtained because OIT is directly related to the relative amount of anti-oxidant in the sample. Examples of such profiling analysis for an EPR/EVA compound can be found in reference [A.8].

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#### ANNEX B

#### (TO SECTION 4 OF PART I, VOLUME I: MANAGING AGEING OF CABLES THROUGH ENVIRONMENTAL QUALIFICATION)

Annex B contains additional material of relevance to Section 4 of Part I, Volume I of this report on environmental qualification. It includes more detailed information on the following aspects related to Section 4:

- On going qualification
- French strategy for cable ageing management
- Czech strategy for cable ageing management.

#### **B.1. ON GOING QUALIFICATION**

#### **B.1.1. Basic principles**

Formal on going qualification is a repetitive procedure intended to overcome the concerns with using high acceleration factors and of qualifying for a long life. The principle is to start repetitive testing after a certain installation time, e.g. 10 to 15 years, and subsequently demonstrate continued qualification by repeated ageing and testing (with or without DBE). The procedure, if successful, is repeated until the cables are replaced for any reason. Representative samples which have been submitted to realistic ageing need to be selected and the condition monitoring methods need to be defined.

Underpinning all of the procedures is the requirement for environmental monitoring in the plant to obtain a detailed knowledge of temperature, radiation dose rate, humidity, etc. in areas of the plant where safety related cables are positioned. Environmental monitoring has already been discussed in Section 5.2 of the report.

The principle of on going qualification is to use reasonably short life qualification (typically 10 to 15 years) at initial EQ and to extend the qualification by repeated accelerated ageing and testing at the end of the initially qualified life. By limiting the qualified life, lower acceleration factors can be used in the ageing tests, giving a more realistic simulation of service conditions.

Before the end of the initial qualified life, representative samples of the installed cables are taken out of containment and subjected to accelerated ageing followed by a DBE. The ageing is again designed to qualify for a limited life e.g. 7 to 10 years. The cable samples can be from a cable deposit, from installed cables or possibly from long term laboratory tests. If the results of the testing are successful then the qualified life of the cables is extended. This procedure is repeated at the end of the extended qualified life and, if successful, the qualified life is again extended. This procedure is repeated until the tests show that the qualified life cannot be extended, or the installed cables are replaced for other reasons, or the power plant is shut down. Testing of cables during on going qualification should include a range of CM methods for assessing cable degradation, as well as the DBE testing.

On going qualification can be applied to all types of cable materials. The main requirement is to ensure that the cable samples used for re-qualification are representative of the most severe conditions in the plant. The methodology is mainly suitable for new power plant or for new cable installations in older power plant. It can also be applied to older plant provided installed cables can be removed for testing and replaced by identical un-aged cables.

On going qualification need not affect plant operation since samples are required for testing at relatively long time intervals which can be scheduled into normal maintenance periods. The timing should be such that the qualified life of the cables being tested covers the next planned maintenance so that any cables which fail the extended test can be replaced within their qualified life.

## **B.1.2.** Cable deposits

One of the advantages of a cable deposit for on going qualification is that the cables age under real plant conditions but can be checked and monitored without major disruption to plant operations.

## B.1.2.1. Cable deposits installed in 'hot spot' areas

The cable deposit can be placed in a position in the nuclear power plant that is exposed to a higher dose rate and/or higher temperature than typical cable positions. Experience has shown that the loop line between the reactor pressure vessel and the steam generator is suitable for this purpose in PWRs and the reactor water cleanup system is suitable in BWRs. In German designed PWRs, the dose rate at this position is 1.3 to 1.5 times higher than the values prevailing at the most exposed cable positions [B.1].

The deposit should include cables which are representative of the types used in containment. The number of samples and their total length must also be assessed to ensure that enough material is available for the removal of samples over a period of up to 40 years. The intervals at which samples are to be removed and the type of tests that are to be performed are of major importance in this respect. For example, samples of about 30 cm in length are quite satisfactory for measurements of elongation. On the other hand, a minimum length of between 2 and 3 m is required for a DBE test with all electrical measurements.

At intervals, samples are removed from the cable deposit and prepared for tests. The tests to be performed normally consist of the following:

- The elongation at break of each sample is measured first.
- Determination of the actual DBE resistance of the sample.
- On completion of the DBE test, the elongation at break is measured again.

In order to simplify testing and conserve the supply of sample material in the deposit, the DBE resistance is initially examined using a modified "steam test. As long as the measured elongation at break is >50% of the initial value in the as-new condition, a cable sample approximately 30 cm long is exposed to the steam test. This consists of the specified DBE temperature, pressure and moisture conditions, but electrical measurements are not performed. The complete DBE test with all the normal electrical measurements is only performed when the elongation drops to <50% of initial value.

The assessment of suitable sampling intervals can best be explained with the aid of the schematic diagram presented in Figure B.1. In the upper section, the accelerated dose in the cable deposit (solid line) is compared to the dose at the most exposed real cable position (dotted line) as a function of time. The data for this graph are obtained from dosimetry results for real cable positions and for the cable deposit.



FIG. B.1: Determination of lead times for a cable deposit.

The results of the elongation at break test obtained from the cable deposit samples are plotted over the same time scale in the lower section of Figure B.1 (solid line). Assuming that there is no significant dose rate effect, the elongation values determined from the cable deposit can be shifted by the lead times ( $t_{v1}, t_{v2} \dots$  etc.) to determine the variation in elongation values with time for cables in the rest of the plant. Since the acceleration factor for the cable deposit is small relative to installed cables, dose rate effects are likely to be insignificant.

For the types of cable currently in use, the first samples from the deposit might be removed five years after the start of plant operation since the initial qualification tests provide an acceptable confidence interval for at least this period. The determination of further sampling times is dependent on the lead time of the cable deposit, as shown in the upper section of Figure B.1, and with changes in the test results.

The above description assumes the most favourable case, that is the installation of a deposit prior to or within 5 years of commissioning of the power plant. The conditions that are encountered in practice, however, may require modifications. For instance, a deposit can also be installed in a plant that is more than 5 years old. For this purpose, the cable samples to be placed in the deposit must be artificially aged in the laboratory with the lowest possible dose rate in order to attain the necessary lead time.

#### **B.1.2.2.** Cable deposits installed in representative areas

To evaluate the differences between natural and artificial ageing, the Electric Power Research Institute developed the natural versus artificial ageing programme that was implemented by the University of Connecticut. In this programme, sets of cable specimens and components were placed in 15 different power plant locations in 1985 and 1986 with ambient environments ranging from 23°C to 61°C [B.2]. Nine different power plants were involved in

the programme, which is still on going. Periodically, a set of specimens is removed from the plant and the environmental data for the period is obtained. Cables commonly used in U.S. nuclear plants were included in the programme having insulations comprised of EPR and XLPE and jackets of Neoprene or CSPE. The cable materials are tested for changes in elongation-at-break and density. To date, the only material with appreciable changes is the Neoprene that is located in the elevated temperature locations (50°C or greater). So far, there are no identifiable changes in elongation-at-break and density properties for the other materials [B.2].

## **B.1.3. Real time aged cables**

The cable deposit method described in section B1.2 is primarily suitable for new nuclear power plants. Because of the radiological lead time of a cable deposit, its installation after plant commissioning is still a viable proposition up to about 5 years after commissioning when it should be possible to catch up with the actual radiation dose of the cables at real positions.

However, after 5 years an alternative is to evaluate the actual long term resistance by removing cable samples from the plant. The disadvantages of this sampling procedure are that it constitutes an intervention into the plant and that the samples have to be replaced accordingly. In addition, removal of samples may damage other cables adjacent to the sampled cables.

As in the cable deposit method, environmental monitoring of temperature and dose rate within the plant is a pre-requisite (see section 5.2). Once the environmental conditions are known, a cable position can be selected in the NPP at a suitable hot spot. In practice, such radiation hot spots are usually in the direct vicinity of the loop lines (PWR) or in the reactor water cleanup system (BWR).

Naturally aged cable samples are normally irradiated quite inhomogeneously, e.g. a cable loop converging on the loop line. Before removing the cable from the plant, the local geometry must be observed and the cable sample must be clearly marked to allow later test results to be interpreted correctly in terms of the actual radiation dose received.

Performance of the appropriate tests and examinations, determination of the sampling times for further specimens and verification of the test interval are all based on the procedures explained in section B.1.2 for the cable deposit method.

In order to use this method for formal on going qualification, cable samples have to be taken repeatedly from the power plant, which is a further disadvantage. The problem is whether it will be possible to find further samples whose ageing behaviour is identical or similar to that of the first sample. In PWR plants, for instance, appropriate identical cable positions are located in the vicinity of the various loop lines. Test programmes of this type have been performed in a number of plants.

Sampling of real time aged cable from plant is most appropriate for older NPPs, where it is not feasible to use a cable deposit and where unaged material is unavailable. The methodology can be applied to any of the cable types or materials. Careful selection of the locations within the plant for sampling will give some lead time over the majority of cables in the plant but the data are not likely to be as reliable as a well-designed cable deposit.

#### B.2. FRENCH STRATEGY FOR CABLE AGEING MANAGEMENT

All of the strategies for the management of ageing of cables inside the containment building of nuclear power plants have a basic concept in common: the initial qualification of the equipment. The environmental conditions that were assumed at initial qualification are chosen with a view to guarantee a conservative level for the tests, so that the main functional characteristics of the cable are assured over the required time scale. Yet, chemical and physical phenomena which govern the ageing of polymers are complex and directly depend on the environmental conditions. The accelerated simulation of environmental conditions that is carried out in qualification tests raises the question of how representative such tests are with respect to natural ageing. In order to confirm cable predictive lifetime (one of the issue of the initial qualification as performed on the actual NPP installations) and to validate long term behaviour (50 years), studies and laboratory ageing tests need to be initiated. The complementary laboratory ageing tests are carried out with lower acceleration factors, which have been determined after the analysis of the ageing mechanisms involved (using a predominance diagram – Section 3.2) in order to simulate the natural ageing process more realistically.

For the last 15 years, 3 programmes of long term ageing have been initiated, on 3 types of cables installed in French NPPs: plasticised PVC (K2), EPR/CSPE (K1) and halogen-free cables based on EVA  $(K1)^1$ . The approach used has five major objectives:

- (a) to simulate accelerated ageing under several conditions (temperature, dose rate)
- (b) to identify the ageing mechanisms involved (predominance diagram)
- (c) to develop a kinetic model in order to predict the lifetime of the cable in operating conditions
- (d) to build a chart for potential on-site applications (indenter measurement)
- (e) to carry out thermodynamic behaviour tests

The quality of the data collected during these research and development projects allow us to increase our knowledge on the long term behaviour of the cable materials (50 years), with middle-term studies in laboratory (about 5 years). A very low dose rate simulation (0.5, 1 and 2 Gy/h) was even performed on EPR/CSPE over a period of 10 years. Moreover, several studies carried out on samples of cables taken from several French plants have confirmed some points of the studies related to ageing mechanisms, residual state, and accident behaviour.

A second series of actions comes as a complement to the management of the cables in NPPs. It concerns all the maintenance operations which are performed in operating units, in normal zones (most of installed cables) and in more severe zones. The association of electrical controls with visual inspection allows us to make sure that the cables are operating well, and to detect possible faults. In this particular case, the maintenance team uses an expert analysis guide, which is a practical diagnosis tool drawn up from data from long term programmes and experimental feedback from operating units that has been gathered over more than 30 years.

<sup>&</sup>lt;sup>1</sup> French cable categories for use in NPPs are:

K1: equipment installed in-containment with important to safety functions during accident;

K2: equipment installed in-containment without important to safety functions during accident;

K3: equipment installed outside containment.

#### B.3. CZECH APPROACH TO CABLE AGEING MANAGEMENT

The Czech approach to cable ageing management has been to use a linear modelling method combined with condition monitoring using microsampling methods. Their experience of using these CM methods is described in Appendices D.2 and D.5. The linear model is described in detail in Annex E.1.

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#### ANNEX C

#### (TO SECTION 5 OF PART I, VOLUME I: IDENTIFYING CABLES OF CONCERN)

Annex C contains additional material of relevance to Section 5 of Part I, Volume I of this report on identifying cables of concern. It includes more detailed information on the following aspects related to Section 5:

- Distributed temperature measurements using fibre optics
- RADIATION monitoring
- Evaluation of hot spots using indenter measurements.

#### C.1. DISTRIBUTED TEMPERATURE MEASUREMENTS USING FIBRE OPTICS

This procedure is based on Raman scattering and described in Figure C.1. Pulsed laser light of frequency  $v_0$  is input from one end and scattered in the optical fibre at position x. The back-scattered light is observed at the entrance at time (2x)/v, where v is the speed of light in the material of the fibre. The scattered light contains Stokes light (frequency  $v_0$ -v, where v is the lattice eigen-frequency, intensity Is) and Anti-Stokes light (frequency  $v_0$ +v, intensity Ia). The ratio of Ia/Is is a function of temperature.

$$Ia/Is = (v_0 + v)^4 / (v_0 - v)^4 \exp[-hvv/(kT)]$$

where h is Planck's constant, v is velocity of light in the material, k is Boltzman's constant and T is the temperature in K, respectively. Therefore, if one measures Ia/Is at time t after the pulse, the temperature at position x = v(t/2) along the fibre is obtained. The detail is given in Reference [C.1].



FIG. C.1. Schematic illustration of optical fibre used for temperature measurement. Incident pulsed-light travels from the left end at velocity of v, and is back scattered at position x. Time elapsed from incidence to detection is t.

In a non-radiation environment, reliability of this procedure has been demonstrated with a spatial resolution of 1m and accuracy within 1°C [C.1]. The potential advantages of this procedure consist in its flexibility and the capability to monitor temperature continuously along the fibre length with a reasonable spatial resolution. This temperature monitoring procedure is already at a practical stage for various industrial plants including NPPs [C.1–C.3].

Irradiation induces colour centres in optical fibre, and the colour centres tend to cause reduced transmittance of light and a change in the intensity-ratio, which result in incorrect temperature measurement. The yield of colour centre formation is enhanced at higher dose rate. Therefore, the following is necessary.

- (a) the fibre type should be selected for less formation of colour centres,
- (b) the wavelength of the source-light should be selected so that the Stokes and anti-Stokes frequencies are not absorbed by the colour centres,
- (c) the dose rate dependence of the colour-centre formation should be investigated.

Radiation resistance towards gamma rays has been tested at JAERI Takasaki with Hitachi cable Co. Ltd [C.4–C.6] and at the SCK-CEN Nuclear Research Centre in Belgium [C.7, C.8]. Both Ge doped fibre and quartz-core fibres were tested under combined radiation and thermal ageing. The response from fibres for two kinds of laser-lights (854nm and 1047nm) was monitored and compared to the temperature measured using a thermocouple.

Ge doped fibre was found to be radiation sensitive, showing severe deviation from the real temperature (thermocouple) after 38 hours at 1 Gy/h (3.8 Gy total dose). On the other hand, quartz-core fibre was found to be reliable even after 1200 hours at 1 Gy/h (1.2 kGy total dose). Using 1047nm-laser was found to be more suitable than 854nm-laser because of the lower loss in transmittance at the higher wavelength. Compared at a fixed dose, the loss in transmittance is worse at the higher dose rate of 10 Gy/h. This could affect the use of the fibre though its effect may be minor because the real dose rates in NPPs are generally lower. It was also found that hydrogen gas environment can reduce loss in transmittance and examination is currently under progress at JAERI Takasaki [C.4].

## C.2. RADIATION MONITORING

## C.2.1. Alanine dosimetry

Temperature dependence of the response and long term stability (in the order of a year) is of concern. Because the radical formation and stability is temperature-dependent, it is assumed that the temperature during irradiation would be monitored by some other procedure (such as those described in Section 5.2.2).

Systematic investigation of the characteristics of alanine dosimeters has been performed at JAERI Takasaki [C.9, C.10] and at CERN [C.11–C.13]. Alanine dosimeters were irradiated at various temperatures in the range 25–80°C at two dose rates to doses up to 0.1–1000 Gy, then stored at 25°C or at the same temperature as irradiated. Measurements at CERN have also been made at cryogenic temperatures [C.11].

The response increased with dose with a good linearity in the dose range of 0.1-50 kGy. The effect of irradiation temperature on the response was also measured and found to be +0.1% per °C, except for long term (in the order of 1000 hours) irradiation above

 $60^{\circ}C$  [C.11–C.13]. Under these higher temperature conditions, the radicals decay. The temperature coefficient for elevated temperature is similar to that seen at low temperatures down to 4 Kelvin: 0.2% per °C [C.11]. Other authors have observed a temperature dependence in the range 0.1–0.25%/°C for the temperature range usually encountered in NPPs [C.14].

The radical decay behaviour in alanine dosimeters has been investigated at various combinations of irradiation temperature and storage temperature. The decay of response was almost negligible when the dosimeters were stored below 40°C (the only exception was for those samples irradiated at 80°C and stored at 25°C which showed a slight decay). When stored above this temperature, the decay must be taken into account. The decay behaviour is only dependent on the storage temperature and humidity, being independent of dose and dose rate.

Therefore, if the irradiation temperature, storage temperature and time after irradiation are known, one can estimate the "initial" response at the irradiation temperature. Then the dose is determined by taking the irradiation temperature into account, and eventually dose rate is estimated.

## C.2.2. Radiation monitoring in NPP — Examples

Figure C.2 provides an overview of the measured doses at cable locations within a modern 1300 MW PWR, which have been sorted into several categories based on the extrapolated dose at 40 years. The doses were measured with the aid of alanine dosimeters during one fuel cycle, which in this case corresponded to one year operation at 100% reactor power. The dosimeters had been fixed at those cable positions inside the plant with the shortest spatial distance to any primary loop line. The measured dose figures then were integrated to the 40 years life time of the power plant, by taking the number of operating days at nominal power for the assumed life time of 40 years into consideration. It is apparent that at more than 2/3 of the cable positions the integrated dose would be expected to be < 1 Gy. At approximately 25 positions the radiation level is higher than 30 kGy.

Figure C.3 shows how the measured dose varies as a function of distance from the primary loop line. Here the integrated dose (in kGy) for a 40 year service life is plotted over the spatial distance to the nearest primary loop line of the cables. It can be seen that, for the temperature sensors which are directly inserted into the primary loop line, the integrated dose peaks at about 150 kGy. The sensor cables are less than 10 centimetres away from the surface of the thermal insulation of the primary loop line. At a distance of about two meters the integrated dose falls below 25 kGy.

In order to identify the locations where increased radiation level could be expected, Figure C.4 was also plotted. It shows the measured dose levels in kGy (integrated to a 40 year service life) plotted as a function of the different rooms within the power plant where the cables are installed. Only three general areas can be identified where the radiation levels are significant. The first two areas are clearly pronounced and can be found in the vicinity of the hot and cold leg of the primary loop lines. The third area is only very weak and represents the area surrounding the steam generators.



FIG. C.2. Measured operational doses within a typical PWR (1300 MW) for a range of cable locations.



FIG. C.3. Measured operational doses in kGy in the vicinity of the primary loop lines of PWR (1300 MW).

The results of this dosimetry has been verified by several others and they can be summarised as follows for this type of PWR:

- (a) Typically, significant radiation levels occur only at a comparatively small number of safety relevant cables.
- (b) Significant radiation levels are usually restricted to a small sector measuring not more than approximately 2 to 3 meters away from the primary loop lines and the steam generators.



FIG. C.4. Measured operational doses in kGy in a 1300 MW PWR, as a function of area within the plant.

Long term dosimetry programmes have also been performed in modern BWRs. The general radiation level here is significantly lower. Maximum doses are about 50% lower than in comparable PWRs and the same could be observed for the number of cable positions that are exposed to elevated radiation levels.

Generally, thermal and fast neutron fluences are minor compared with the gamma dose rate, in terms of the effects on cable materials. Fluence of thermal and fast neutrons within containment can be measured by using cobalt and nickel foils, respectively [C.15].

#### C.2.3. Radiation monitoring in particle physics research facilities

At CERN, intensive R&D work is being carried out for the design and building of the future Large Hadron Collider (LHC) and its associated experimental detectors for physics research. Electronics and optical links will have to be installed in these devices, where due to the high-energy of the particles and to the high-frequency of the interactions, high radiation doses are expected (up to several tens of kGy per year). For these R&D programmes, radiation fields need to be monitored accurately, including dose and particle-fluence measurements. For the latter, activation foils can be used [C.4], but semi-conductor dosimeters are easier [C.7]. In polymer insulators, radiation degradation depends mainly on the total absorbed dose, and on dose rate, independently of the radiation type (see e.g. [C.18, C.19]). It is therefore only necessary to record the absorbed dose to organic components. It is to be noted that temperature does not need to be monitored in the accelerator tunnel, as it is usually close to ambient with very little variations.

For the survey of absorbed doses in the present particle accelerators, several types of dosimeters have been used on a routine basis for more than 30 years [C.20, C.21]. In particular, alanine powder has been incorporated in a rubber and extruded like a cable; this allows dose distribution measurements [C.12]. This dosimeter has proven to be accurate and reliable [C.13]. Its behaviour at cryogenic temperature has also been studied [C.11].

#### C.3. EVALUATION OF HOT SPOTS USING INDENTER MEASUREMENTS

Two types of hot spots exist — real and theoretical. A real hot spot is one in which a severe environment exists with respect to a cable's capabilities. A theoretical hot spot is one that has been identified through a calculation or evaluation. A theoretical hot spot may be real or it may be an overly conservative estimation of actual conditions. For example, theoretical hot spots often result from thermal calculations of ohmic heating of cables covered by fire barriers. These calculations contain many conservative assumptions and generally result in elevated temperature estimates with some results indicating temperatures of 110 to 140°C. Condition monitoring tests may be performed on cables having real and theoretical hot spots. For cables with real hot spots, the condition monitoring results can be used to determine the extent of the affected zone. With this determination, the cable can be sectioned and spliced at a new or convenient junction box such that the affected section can be replaced periodically. For cables with theoretical hot spots, the actual cable can be subjected to CM testing. If the results indicate severe ageing, then the calculation is correct and the cable must be replaced or closely monitored to assure acceptability of its condition. If the result indicates a lack of any significant ageing, then the calculation has excessive conservatism and the hot spot is not real.

The indenter has been used to evaluate both types of hot spots. The cables under test had either CSPE or Neoprene jackets. For both materials, indenter modulus measurements provide very good indication of thermal ageing. In one case where cables were located near a large, high-temperature header, the indenter was used to map the extent and severity of the damage and helped locate a section of damaged thermal insulation that was adding significantly to the level of thermal stress. In another case, after an extended abnormal elevated thermal condition, a cable was used to determine the boundaries of the heat-affected zone for the containment structure. A secondary result of the testing was the confirmation that only a small section of the cable had been damaged and splicing of a replacement segment could be performed rather than repulling the entire cable. The savings from not having to replace the entire circuit paid for the testing [C.22].

With respect to the theoretical hot spots, indenter testing of cables that were supposedly exposed to 110 to 140°C conditions for 10 years indicated that the cable jackets were in like new condition [C.22]. The material was CSPE which would have been extremely embrittled had such high temperatures existed for such long periods. The high temperatures had been indicated by thermal calculations related to ohmic heating within the confines of fire barriers.

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## ANNEX D

## (TO SECTION 6 OF PART I, VOLUME I: CONDITION MONITORING)

Annex D contains additional material of relevance to Section 6 of Part I, Volume I of this report on condition monitoring. It includes more detailed information on the following aspects related to Section 3, including examples of plant experience with condition monitoring methods:

- Plant experience using the indenter in Canadian NPP
- Experience with OIT/OITP in Dukovany NPP (Czech Republic)
- Prediction of residual lifetime of PE insulation using OITP measurements
- Prediction of residual lifetime of PVC insulation using TGA measurements
- Experience with correlation between insulation and jacket materials for PVC/PVC and PVC/XLPE cables in Dukovany NPP
- Experience with cables in a high-energy physics laboratory
- Experience with correlation of jacket and insulation materials for a range of PVC jacketed cables
- Example of method to correlate indenter modulus with DBE survivability
- Round-robin test programme on CM methods for cables.

## D.1. PLANT EXPERIENCE USING THE INDENTER IN A CANADIAN NPP

A cable ageing monitoring programme was initiated to provide a surveillance method to support the qualified life of important to safety cables, and as a means of generally predicting the useful life of cables throughout the station.

The programme was implemented in the following phases:

- Phase 1: Selection of a primary method of broadly sampling cables.
- Phase 2: Select initial sample base and retrieve field data.
- Phase 3: Baseline (fingerprint) the correlation between the indenter modulus and elongation at break for the relevant cable types as well as condition indicators from supplementary monitoring methods.
- Phase 4: Broaden the field cable sample base over time and implement a formalised system of regularly retrieving and analysing data.

It was recognised that phases 3 and 4 would demand a significant amount of time and effort to complete, however, it was felt that it was important to begin retrieving field data as early as possible to permit more accurate predictive analysis earlier in the life of the plant. In order to take advantage of a planned extended outage, Phases 1 and 2 were undertaken. These two phases are discussed in more detail below [D.1].

#### **D.1.1. PHASE 1** — Selection of primary method of cable ageing monitoring

The indenter was selected as the primary method of retrieving ageing data due to the following advantages:

- (a) useful acceptance criteria are achievable
- (b) non-destructive
- (c) non-intrusive
- (d) easily portable
- (e) widely used in the nuclear power industry

## D.1.2. PHASE 2 — Selection of initial sample base and retrieval of data

D.1.2.1. Cable selection criteria

- (a) Select generic types of power and control cables (i.e. FRPVC/FRPVC, FRPVC/FRPVC, FRPVC/PVC) that represent the largest volume of station cables, with emphasis on EQ cables.
- (b) Cable type must be positively identified in the field.
- (c) Cable dimensions must be accommodated by indenter probe and sufficient slack must be available to provide adequate clearance to attach the clamp.
- (d) Select cables known to have a significant continuous electrical loading, or frequent inrush transients.
- (e) Select same and similar cables in different locations with different environments.
- (f) Select similar cables in same location/environment.
- (g) Select cables installed prior to commissioning.

D.1.2.2. Location selection criteria

- (a) Confine selection to the reactor building and focus on EQ cables and worst perceived environments.
- (b) Provide a broad cross-section of the reactor building environment including radiation and thermal hot spots.
- (c) Include areas where there is a high concentration of cables.
- (d) Locations must be accessible as much as possible (to minimise staging support).
- (e) Select locations where spare cables are available for supplementary destructive testing.
- (f) Identify secondary sample sites (e.g. Specific cable trays, conduits, junction box entrances) within the larger primary sites.
- D.1.2.3. Cable identification and marking

Each selected cable is uniquely identified with an ID# which denotes the primary location, secondary location and serial number of the cable sample. For example, cable sample R6A4 would be located in primary site "R6" (R/B West F/M Vault, elevation 45' + 20), at secondary site "A" (cable tray CP4015), cable sample #4. This identification system permits easy sorting of data by locations. A general arrangement of relevant locations, with sample site locations clearly identified, is documented to enable quick location of the site analysis and future field measurements.

Each cable sample area is marked with two laminated cable ID# tags, positioned 300 mm to 500 mm apart between which indenter measurements are to be taken. This sample zone permits multiple sample points on a single cable while ensuring that future sampling is confined to the same small defined area.

## D.1.2.4. Database development

A relational database was developed to record cable sample description and location information. This database was first used to provide a form to use as a guide and checklist for performing field measurements, as well as a means for recording problems or other relevant feedback. Data retrieved via the indenter software will be transferred to the cable ageing monitoring database for long term tracking and trending analysis.

#### D.1.2.5. Preparation for field measurements

All staging, ladders, safety harnesses, etc, were available and in place and shop support was reserved. A key factor in minimising effort to prepare for and execute field indenter measurement was the early involvement of operations staff.

Many of the field cables selected for sampling carry special safety and safety related systems circuits. Many more cables are "black snakes" located in the middle of main cableway trunks where the end devices are not easily determined. In the early stages of Phase 2 a presentation was given to senior Operations personnel demonstrating the non-intrusive nature and safety features of the indenter. This avoided the need to incorporate work plans and post maintenance testing into the field work. This also facilitated quick approval of work permits. It may be prudent, however, to ensure the indenter and a sample of cable is nearby when requesting a work permit in the event that an individual shift supervisor requires further convincing.

#### D.1.2.6. Experience gained

- (a) Relaxation modulus used to sample XLPE insulation was ineffective due to mechanical noise induced by vibration from operating machinery. The possibility of providing filtering is being investigated.
- (b) The probe clamping arrangement can be improved to provide more consistent grip on cables. The present arrangement is of limited use at cable bends.
- (c) Cable sampling areas should be cleaned prior to taking measurements to prevent interference from surface contaminants.
- (d) The indenter provides a convenient and effective method of retrieving cable age trending data from a broad population of field samples.

#### D.2. EXPERIENCE WITH OIT/OITP IN DUKOVANY NPP (CZECH REPUBLIC)

Cable condition monitoring was performed in the framework of the cable qualification of the NPP Dukovany and it was hampered by limited availability of technical documentation, lack of stored original cables and control deposit specimens from the NPP Dukovany operation. These conditions were reflected in the design of the cable qualification methodology for NPP Dukovany cables [D.2]. The initial work had to proceed from the identification of the cable polymeric materials. On the basis of these results, the activation energies of cable materials were determined. Since the vast majority of the cable materials were manufactured from polyvinylchloride (PVC) mixtures, a number of measures have been included to account for the dose rate effect on the ageing rate as well as the influence of the sequence of radiation/thermal ageing on the accelerated cable ageing under operational conditions.

The biggest problem in the cable CM programme at NPP Dukovany proved to be the scarcity of control deposit cable specimens from NPP operation and the unavailability of stored cables. The situation was solved by applying the method based on the measurement of the oxidation-induction time (OIT) or oxidation-induction temperature (OITP) of the microsamples of the polymeric cable materials by differential scanning calorimeter (DSC).

Applying this method, the time of the thermo-oxidative degradation onset at constant temperature (oxidation induction time — OIT) or the temperature at the thermo-oxidative degradation onset (oxidation induction temperature — OITP) are recorded. The OIT is related

to the amount of antioxidant remaining in a polymer, and thus to the age or to the remaining life of polymer [D.3, D.4].

The results obtained by DSC are compared with those obtained by the measurement of elongation at break as a common technique for assessing the cable (Figure D.1). To assess the lifetime of a particular cable in power plant, a small sample of the cable can be removed and tested for OIT/OITP. Comparison of this value with that for the an unaged cable, together with the dependence of the thermo-oxidative stability on ageing caused by radiation and thermal ageing, can establish the extent of degradation of the cable under simulated operation condition.

OIT is also used for direct prediction of service lifetime of polymers [D.5]. As it was mentioned above, OIT is linked to the content of antioxidants and so it has a proportionality to the service lifetime. But the effectiveness of antioxidants, as measured by the OIT at high temperatures ( $\approx 200$  °C), may differ as a function of temperature [D.6]. This may be due to a number of causes including changing mechanisms, loss of antioxidants due to volatilisation at OIT test temperatures, etc. Therefore, the lifetime assessment of plastics determined only by OIT has a high uncertainty and cannot be recommended for the direct service lifetime prediction of NPP cables. Hence, the correlation of OIT response with the elongation at break of insulation materials (or with some other property directly reflecting the cable functionality) is necessary (see Figures D.1 and D.2).

The most difficult problem existing in cable condition monitoring is sampling from cables installed in NPP. The collection of all necessary permits and the knowledge of the hot spots may not be enough for successful sampling. The existence of cable protection layers (like steel braiding, Pb-armouring or fire barriers) is a common problem. Samples for OIT from such cables can be taken sometimes from their connections (but these are often not located at the hot spots) or there can be found a location where the cable protection is damaged, e.g. a sharper bend. Another problem is the inaccuracy and errors in the cable labelling. Hence, it is recommended to analyse the sample material before OIT testing (e.g. by use of IR spectroscopy or DSC) and to compare it with a reference material.

The sampling must be performed by a smooth scraping with a special device which must completely exclude the possibility of cable damage (especially a deep cut). Another reason for very careful planning of samples is the fact that almost all cables are energised. The removal of a small sample of thickness up to 50  $\mu$ m and of sample mass in the range of 1–2 mg from the cable surface does not affect the mechanical properties [D.7].

Before the sampling, the cable surface should be cleaned. The use of any solvent is not recommended; it is better to mechanically clean the surface by using cotton-wool or a duster. The location of the sampling should be marked to find it in the future. The use of a contrast colour is acceptable.

## OIT experimental effects

The OIT method is very sensitive to a number of experimental conditions [D.3, D.8]. For this reason it is necessary to have a standardised methodology where following parameters must be taken into consideration:

- (a) *Temperature* is one of the most important factors. Slight changes in test temperature can have strong effect on the OIT value. This parameter can have (for polyethylene sample) a bias of 8 % per °C [D.6]. Therefore correct calibration is essential for the identified test temperature.
- (b) Oxygen flow rate provides a necessary minimum which is about 50 ml/min. The flow rates must be also limited to a very narrow range of about  $\pm$  5 ml/min [D.8]. In Perkin-Elmer DSC apparatus, the use of a special flow through cover is recommended.
- (c) *Sample pan material* copper, stainless steel and platinum (all commercially available sample pans) catalyse the oxidation reaction of the test specimen. To avoid this problem, standard commercial aluminium pans, which show no catalytic effect, should be used [D.9].



FIG. D.1. The DSC response versus absolute elongation at break for the PVC jacket after its 0-, 10-, 20- and 30-years simulation of service ageing inside containment of the NPP Dukovany.



FIG. D.2. The change of elongation at break as a function of the simulated service ageing inside containment of NPP. The same cable as in Figure D.1.
- (d) *Type of sample pans* open, with holes or non-hermetically sealed can influence results.
- (e) *Sample mass and form:* The most precise OIT methods call for small (under 5 mg) and uniform samples. Also in the NPP, it is only possible to take very small samples and from the cable surface only. Hence, it is better for the comparison of results, when the sampling is performed by a smooth scraping and the sample mass does not exceed 2 mg.
- (f) *Time:* Experimental conditions should be selected so that the OIT values are longer than 15 minutes. Because of the long term DSC stability as well as the testing time required, the test time should not exceed about 200 min.
- (g) *End point selection:* The onset of oxidation is taken as the end point for the OIT measurements. Two methods for OIT determining have been established the method of extrapolated onset and the first-deviation-from-baseline method. Because some materials seem to have a multi-staged oxidation, the end point established by these two methods may give different results.

Main advantages of OIT method

- (a) Essentially a non destructive technique only very small samples from the NPP cable surface are needed
- (b) Very simple sampling
- (c) Samples can be removed even from an energised cable
- (d) Feasible regular monitoring of cable conditions
- (e) Applicable for on going qualification of cables.

The OIT method enabled the evaluation of the condition of the cable materials from the microsamples taken in-situ from the cables installed inside containment. The residual service life of the individual cable types was then assessed using the methodology described in references [D.2].

D.3. PREDICTION OF RESIDUAL LIFETIME OF PE INSULATION USING OITP MEASUREMENTS

Experience shows that the oxidation induction temperature at a constant temperature rate *(OITP)* for polyethylene materials is subject to the dependence [D.10]:

$$1/OITP = 1/OITP_i + K \cdot t,$$

where  $OITP_i$  is the OITP onset for an unaged sample, t is the time of ageing, and K is the constant of the rate of OITP change, which depends on service conditions and rate of temperature.

(D1)

From equation (D1), it is easy to get an expression for the prediction of residual lifetime by using OITP measurements during ageing of the polyethylene material.

$$t_s = \frac{t_m (OITP_m - OITP_k)}{(OITP_i - OITP_m)} \cdot \frac{OITP_i + 273}{OITP_k + 273},$$
 (D2)

where  $t_s$  is the residual service life,  $t_m$  is the service life at the moment of monitoring,  $OITP_m$  is the OITP value at the moment of monitoring,  $OITP_k$  is the OITP value when failure is observed (for example, cracking for XLPE). All of the OITP values are expressed in °C.

Note that lifetime prediction using OITP onset leads to lowered service life, since the effective activation energy for thermal oxidation increases when moving through the crystalline melting point back to ambient temperature. Consequently, extrapolation from the results of OITP measurements, obtained at temperatures well above the melting temperature, to service conditions can give slightly lowered lifetimes.

# D.4. PREDICTION OF RESIDUAL LIFETIME OF PVC INSULATION USING TGA MEASUREMENTS

Thermal/radiation ageing of PVC insulation must be assessed by using at least two parameters, one sensitive to the remaining concentration of plasticizer, the other sensitive to the degree of destruction of polymeric links. By using TGA analysis, it is possible to determine the level of destruction of polymeric chains by the parameter TG5% and define the remaining concentration of plasticizer by comparisons of thermogravimetric curves of plasticized insulating PVC micro samples of non-plasticized PVC [D.11].

The parameter TG5% is quantitatively defined as the temperature on the thermogravimetric curve at which the microsample mass decreases by 5% relative to the initial mass. A measurement technique for the determination of the remaining concentration of the plasticizer (concentrations of the low molecular components  $C_{lmc}$ ) is based on the comparison of two thermogravimetric curves: without a plasticizer and with a plasticizer. This parameter can be determined in term of the mass percentage calculated by the following formula:

$$C_{lmc} = B - B' \cdot \frac{B - A}{B' - A'},\tag{D3}$$

where A, A', B, B' are parameters obtained from the thermogravimetric curves in accordance with Figures D.3a and D.3b.

The change in TG5% for samples of cables aged under radiation ageing, at operating temperatures as low as 70°C, is described by the expression:

$$TG5\% = TG5\%(0) \cdot (1 - r \cdot P^n \cdot t),$$
 (D4)

where TG5%(0) is the value of TG5% for an unaged sample, *r* and *n* are the constants for the given material, *P* is the dose rate, *t* is the ageing time.



FIG. D3a. TGA and differential TGA curves for plasticized polyvinylchloride (plasticizerdioctylphthalate) [Ref. D.11].



FIG. D3b. TGA and differential TGA curves for PVC without plasticizer [Ref. D.11].

Measurement of the TG5% value must be carried out in a crucible of a thermogravimetric analyser in the absence of oxygen, for instance, in nitrogen. Equation (D4) also holds true when using open crucibles in air in the absence of removal of the products of decomposition. The value of TG5%(0) also strongly depends on presence of oxygen in the crucible during decomposition of the sample. With increasing concentration of oxygen, the value of TG5%(0)decreases.

Content of oxygen in	TG5%
atmosphere of the crucible	
90	268
30	277
~0	290

The change in the remaining concentration of the low-molecular component  $C_{lmc}$  during the process of thermal radiation ageing is described by the expression below [D.11].

$$\begin{cases} C_{lmc} = C_{lmc}(0) \exp\left[-\left(k_t + k_r P^n / C_{lmc}(0)\right) \cdot t\right] \text{at the } D \ge D_{\mathbf{c}} \\ C_{lmc} = C_{lmc}(0) \exp\left(-k_t \cdot t\right) \text{ at the } D < D_{\mathbf{c}} \\ k_t = k_t(0) \exp\left(-E_T / RT\right) \\ k_r = k_r(0) \exp\left(-E_R / RT\right), \end{cases}$$
(D5)

where  $C_{lmc}(0)$  is the initial concentration of plasticizer (LMC), *P* is the dose rate, *n* is the constant for the given material,  $k_t$  is the constant of thermal ageing rate,  $k_r$  is the rate of radiation ageing;  $E_T$ ,  $E_R$  and  $k_t(0)$ ,  $k_r(0)$  are their activation energies and preexponents, respectively. D<sub>c</sub> is the critical absorbed dose, below which the contribution of thermal radiation ageing becomes essential (for instance, decrease in concentration of plasticizer is registered by chromatographic or some other method).

Figure D.4 and D.5 show the kinetics of the parameters TG5% and  $C_{lmc}$ , for the PVC jacket of cable KVVGng during accelerated testing.



FIG. D.4. Time dependence of TG5% in PVC jacket (KVVGng cable) under thermal and radiation ageing [Ref. D.12].



FIG. D.5. Time dependence of residual concentration of low molecular components in PVC jacket of KVVGng cable under thermal and radiation ageing [Ref. D.12].

Therefore, using only thermogravimetric analysis, it is possible to carry out condition monitoring and define a residual lifetime of PVC jackets of cables. For the determination of the residual lifetime, it is necessary to use the formulae:

$\int C_{lmc} = C_{lmc}(0) \cdot \exp(-K_{ef1} \cdot t)$	(D6)
$TG5\% = TG5\%(0) \cdot (1 - K_{ef2} \cdot t),$	(D0)

and know the initial and critical values of the parameters  $C_{lmc}$  and TG5% (at which failure is observed). For many industrial PVC insulating materials  $C_{lmc}(0)=30\%$ , critical value of  $C_{lmc}(k)=15\%$ . Numerical values of magnitudes TG5%(0) and TG5%(k) depend on the composition of the atmosphere in crucibles during TGA measurements.

Practical experience with the particular cable materials tested (with PVC sheath and insulation materials) shows that under thermal and radiation ageing on cables, the rate of insulation degradation is higher than the rate of sheath ageing [D.12]. Qualitatively, it may be explained by formation of active products of PVC degradation, which are removed from the surface of a cable sheath, but accelerates insulation degradation under the sheath. Figure D.6 and D.7 presents the results of comparison of PVC sheath service life with PVC insulation service life for a cable KVVGng (the main plasticizer for this cable insulation and sheath is dioctylphthalate), which were obtained from accelerated laboratory tests.

The results presented in Figure D.6 and D.7 show that the service life of PVC insulation can be ten times less than the service life of PVC jacket under ionising radiation. With an increase in operating temperature, the contribution of ionising radiation to the degradation of PVC cable decreases. Under higher absorbed dose rates, which are used in accelerated tests, degradation of the jacket can be faster than degradation of the insulation (Figure D.7).

It is qualitatively understandable that when increasing the molecular weight of the plasticizer, the service life of PVC cable as a whole would increase, since the desorption of plasticiser with a higher molecular weight will be slower under the same operating temperature [D.12]. The results presented in Figures D.6 and D.7 were obtained for PVC insulation and jacket with dioctylphthalate plasticizer, which has the lowest molecular weight of plasticizers used in the cable industry. Therefore, practical application of the ratio of service life of sheath and insulation of PVC cables to those of other plasticizers will give an underestimated service life of insulation, if it is evaluated from the service life of sheath.



Fig. D.6. Ratio of sheath service life to insulation service life of KVVGng cable as defined by parameter TG5% as a function of dose rate [Ref. D.12].



 $1 - 30^{\circ}C, 2 - 40^{\circ}C, 3 - 50^{\circ}C, 4 - 60^{\circ}C, 5 - 70^{\circ}C.$ 

# D.5. EXPERIENCE WITH CORRELATION BETWEEN INSULATION AND JACKET MATERIALS FOR PVC/PVC AND PVC/XLPE CABLES IN DUKOVANY NPP

The procedure used for the assessment was as follows:

- (a) Cable jacket and insulation are aged in the laboratory for periods corresponding to 10, 20 and 30 years of NPP operation.
- (b) The mechanical properties (especially elongation at break) of jacket and insulation are measured. The data can be plotted as a function of jacket elongation at break vs. insulation elongation at break (Figures D.8 and D.11). Each point on this plot must correspond to the same time of simulated operation time. Another possible plot is elongation at break as a function of simulated service operation (Figures D.9 and D.12).
- (c) The thermo-oxidative stability of the jacket is determined from OIT/OITP tests on the laboratory aged samples and plotted as a function of elongation at break (Figures D.10 and D.13).
- (d) Sampling from the jacket surface in the NPP and measurement of the OIT/OITP of the jacket. From the plot of OIT/OITP vs. elongation at break, it is possible to assess the actual elongation at break of a cable jacket that is in service. From the insulation–jacket correlation one can find the corresponding elongation at break of the insulation and so the extent of its degradation and its residual service life can be established.

Examples of cross-plots of jacket elongation vs. insulation elongation are shown in Figure D.8 (for PVC/PVC cable) and in Figure D.9 (for PVC/XLPE cable). In the PVC insulated cable, there is good correlation between the jacket properties and that of the violet insulation, but the correlation is less good for the yellow insulation. This highlights the need for tests on different colours of insulation in any practical assessment.

For the specific cables tested, the PVC insulation degrades either at the same rate as the jacket material (violet insulation) or less rapidly (yellow insulation). The XLPE insulated cable

Fig. D.7. Ratio of sheath service life to insulation service life of KVVGng cable as defined by parameter  $C_{lmc}$  as a function of dose rate and operating temperature [Ref. D.12].

shows a useful correlation with the jacket properties, even though the insulation degrades faster than the jacket.

From 14 cables tested for EQ, the sheath degraded faster than the insulation in 4 cases, the insulation degraded faster than the sheath in 6 cases, and approximately the same rate of degradation has been found in the remaining 4 cases. This clearly demonstrates that each cable has to be evaluated individually. OIT/OITP results are not transferable from one type of cable to the other one, unless you are sure that it is the same cable type, prepared from the same cable compound and from the same cable producer. Sometimes differences between cable batches may also be found to be significant due to the history of cable polymer processing and quality control.



FIG. D.8. Correlation of elongation at break between the jacket and the insulation. This PVC cable has two types of PVC insulations. The data of this plot have been obtained after the separate simulation of the same time of NPP operation for insulation and jacket.



FIG. D.10. The DSC response as a function of the absolute elongation at break for the jacket from Figures D.8 and D.9.



FIG. D.9. Correlation of elongation at break between the jacket and the insulation. The same PVC cable as in Figure D.8.



FIG. D.11. Correlation of elongation at break between the jacket (PVC) and the (XPE) insulation. The jacket and the insulation have been separately aged to the time corresponding to 0, 10, 20 or 30 years of operation in an NPP.



FIG. D.12. Correlation of elongation at break between the jacket and the insulations. There has been simulated the operation temperature of  $55^{\circ}C$  + dose rate of 1 kGy/year. The same cable as in Figure D.11 (PVC jacket and XPE insulation).



FIG. D.13. The DSC response as a function of the absolute elongation at break for the jacket from Figs D.11 and D.12..

#### D.6. EXPERIENCE WITH CABLES IN A HIGH-ENERGY PHYSICS LABORATORY

From a survey of radiation damage in the Super Proton Synchrotron at CERN, it appeared in 1979 that the degradation of PE-based and PVC-based cable insulation materials was worse in naturally aged samples than in accelerated tests; degradation was observed at doses below 100 kGy [D.13]. After 10 years of operation and in-situ ageing, it has been observed that the end point doses for PVC and PE is around 100 to 200 kGy if they are used as jacket materials, and around 500 to 800 kGy if they are used as insulations [D.14].

This was rather optimistic with regard to the insulation materials. More recently, after 10 years of operation of the large electron-positron collider (LEP), it has been observed that the insulation (made of coloured PE) of two multi-conductor cables (both irradiated at 120 kGy) were already crumbling, while the characteristics of their polyolefin jackets remained acceptable [D.15]. Moreover, as it is generally observed, the red insulations show a stronger degradation than the other colours.

At the end of the year 2000, the LEP will definitively be stopped and most of the cables (namely all I&C cables, which are all insulated with flame-retardant halogen-free materials) will be taken out before the next Large Hadron Collider is installed in the 27 km tunnel. This will be a good opportunity to test insulating materials which have been aged for 12 years in a radiation environment.

D.7. EXPERIENCE WITH CORRELATION OF JACKET AND INSULATION MATERIALS FOR A RANGE OF PVC JACKETED CABLES

In Ontario Hydro NPPs, safety related cables contain predominantly PVC type jackets while insulations vary from PE, PVC, FRXLPE to FREPR. The condition of many cables have been analysed, which were removed from these nuclear stations and accelerated aged samples.

Analysis was based on measurements of the elongation at break values of insulation and jacket materials. These cables were subjected to a variety of environments in the field such as radiation, thermal, and combined radiation plus thermal environments. Our observations are that the relationship between the condition of insulation and PVC jacket is dependent on both the type of insulation material being evaluated and the ageing environment. Examples are given below:

## D.7.1. Low density PE insulated cables with PVC jacket

Cables exposed to a predominantly radiation environment showed embrittlement of PE insulation while the PVC jacket showed elongation values in the region of 75–200% absolute. Whereas another identical PE insulation in a primarily thermal environment had elongation value in the 500% region while the jacket was embrittled. This data suggest that for cables used inside containment, assessment of the condition of PVC jacket alone does not indicate the condition of the PE insulation. However, outside containment, condition of the jacket is a conservative indicator of the PE condition.

### **D.7.2. PVC insulated cables with PVC jacket**

Cables exposed to predominantly radiation, or combined radiation and thermal environment showed that the insulation aged faster than the jacket. For example, in a cable removed from a predominantly radiation environment, insulation and jacket had elongation values of 70% and 228% respectively. For an identical cable exposed to additional heat, results showed elongation values of 36% and 84% respectively for the insulation and jacket. However, the opposite effect was found in a primarily thermal environment. Accelerated ageing data indicate that for a 75°C rated cable, the jacket is expected to age faster than the insulation. Hence in this situation, by monitoring the condition of the jacket one should be able to determine the condition of the cable insulation. But if the cable is exposed to both the radiation and thermal environment, the jacket will not provide the actual condition of the insulation.

#### **D.7.3. FREPR insulated cable with PVC jacket**

An FREPR cable jacketed with PVC exposed to mostly thermal environment in the field showed embrittlement of the jacket while the insulation had elongation value in the 80% region. Similar behaviour occurs if the cable is exposed to radiation or combined radiation and thermal environments. Hence in this case, condition of the jacket will provide a conservative indication of the insulation condition.

# D.8. EXAMPLE OF METHOD TO CORRELATE THE INDENTER MODULUS WITH THE DBE

As part of the development effort for the indenter, an in-plant demonstration and a series of tests on 17 different configurations of cables aged in increments of the original manufacturers' qualification ageing were performed [D.16]. The ageing was performed in 25% increments allowing a plot of indenter modulus versus fraction of thermal life expended to be developed. During the course of the accelerated ageing, materials such as CSPE experience a factor of 10 change in indenter modulus from the unaged to fully aged state. This makes discrimination between degrees of ageing readily possible. When an indenter measurement is taken in the plant, the result may be compared to the curve and the fraction of

thermal life may be determined. The concept of fraction of qualified life expended was coined during this programme to eliminate the dependency on the Arrhenius model for developing a time based qualified life. When the fraction of qualified life expended reaches 1, the equivalent of 100% of ageing has occurred and the cable must be replaced. If one takes the reciprocal of the fraction of qualified life expended and multiplies it by the age of the cable, one obtains a rough approximation of the total life the cable can obtain while remaining within the boundaries of qualification. This assumes that the environments remain approximately the same through the entire life of the cable.

The following example is from a study, reported in reference [D.17]. Cables with a CSPE (Hypalon) insulation and jacket of were subjected to thermal ageing for various duration and at various temperatures (80°C from 192 to 578 days, 95°C from 48 to 384 days, 120°C from 48 to 96 days and 142°C from 6 to 24 days). The cables were then subjected to ionising irradiation (from spent fuel elements) for 50 hours at a dose rate of 10 kGy/h. After this the indenter modulus were measured. The cables were then subjected to a LOCA test (3 hours at 181°C and 0.4 MPaG gauge pressure, followed by 160°C and 0.4 MPaG for 3 hours and 120°C for 44 hours). The insulation resistance was measured continuously during the LOCA test.

Figure D.14 shows the result in terms of the relationship between the indenter modulus before LOCA and the lowest value of the insulation resistance during LOCA. The linear correlation was 0.76, which shows that there is a useful relationship between the degradation measured by indenter modulus and the dielectric behaviour during LOCA for this CSPE insulated cable.



Comparison between indenter modulus before LOCA and IR values during LOCA

FIG. D.14. Correlation between indenter modulus and insulation resistance during a LOCA test on Lipalon cable [Ref. D.17].

#### D.9. ROUND-ROBIN TEST PROGRAMME

(This section contains the text of the report on round-robin testing of cables [D.18], but does not contain the full Appendices to that report which contain all of the tabulated results and examples of the raw data from the tests. The Appendices referred to in Section D9 do not form a part of this TECDOC; the reader should consult Ref. D.18, if desired.)

#### **D.9.1.** Introduction

The IAEA co-ordinated research project (CRP) on management of ageing of I&C cables in containment originally started in 1993. The first CRP, which ran until 1995, developed a document outlining methods available for the management of cable ageing<sup>1</sup>. A number of technical issues were identified that needed to be resolved to further develop a suitable guidelines document. The second CRP, which started in Dec 1996, aims to resolve some of these issues<sup>2</sup>. One of the issues identified was the need for practical methods of monitoring the condition of cables in nuclear power plant (NPP). Although a number of condition monitoring methods have been identified and tested in the laboratory, only a limited number are currently at a stage of development where they could be usefully employed for field testing. As part of the second CRP, a 'round-robin' test programme has been set up to look at the most developed of the condition monitoring methods. Although most NPP which are interested in condition monitoring of cables would use a single test laboratory for their programme, there is a need for baseline data for common materials. It is therefore important to be able to compare data obtained from different test laboratories.

The 'round-robin' test programme is aimed at assessing the reproducibility of various methods for monitoring degradation in polymeric cable materials and in improving standardisation of the test methods. Some of these test methods may be suitable for routine condition monitoring of cables within a NPP. A range of cable materials have been included in the programme, representing the main types currently in use in nuclear plant. The samples were all prepared by the same laboratory and sent to the participating laboratories for testing. The test methods used by each of the participants have been standardised where possible.

Section D.9 covers the data obtained from the test programme and some analysis of the factors affecting reproducibility. The preliminary results were discussed at the second research co-ordination meeting held in Bordeaux in June 1998 and additional test data identified. These additional test data have been included in this report.

Section D.9.2 covers details of the materials used in the programme, sample preparation, ageing conditions and the test methods agreed for the 'round-robin' testing. Section D.9.3 covers the results of the testing with details of all of the test results and some analysis of the data. Section D.9.4 addresses the variability of the test methods between the participating test laboratories and methods by which this may be improved. Summaries of the experimental data are given in the Appendices.

#### **D.9.2.** Experimental methods

D.9.2.1. Materials included in the test programme

Cable samples (typically 6 m long) for the test programme were supplied by several of the countries participating in the co-ordinated research project on cable ageing. The cables represent types which are currently in use in nuclear power plant and cover the major generic classes of polymeric material typically used for cable insulation and jacketing. These cables are listed in Table D.1 and their construction is detailed in Table D.2.

<sup>&</sup>lt;sup>1</sup> IAEA-TECDOC-932 (March 1997), "Pilot study on the management of ageing of instrumentation and control cables – results of a co-ordinated research project 1993-1995"

<sup>&</sup>lt;sup>2</sup> "Co-ordinated research project on management of ageing of in-containment cables" – report on the IAEA Research Co-ordination Meeting 9-13 Dec 1996, IAEA, Vienna

Identification	Supplying	Manufacturer	Insulation	Sheath material
no. (1)	country		material <sup>(2)</sup>	(2)
1126	Russia	Podolsk kabel	PVC	PVC
1127	Czech	Not known	PE	PVC
	Republic			
1128	USA	Samuel Moore	EPDM+CSPE	CSPE
1129	USA	AIW	EPR+CSPE	CSPE
1130	USA	Okonite	EPR	CSPE
1131	Sweden	ABB	EPDM+CSPE	CSPE
1132	Canada	Pirelli Canada	XLPE	PVC
1133	Canada	Canada Wire	EPR	PVC
1134	Canada	Canada Wire	PVC	PVC
1135	Japan	Hitachi Cable	XLPE	PVC
1136	UK	Pirelli	EPR/EVA	EVA/EVA
1137	UK	Pirelli	XLPE	EVA/EVA
1138 & 1139	Germany	Siemens	EPR	EVA
1140 & 1141	Germany	Siemens	XLPE	EVA
1142 & 1143	Germany	HEW	SiR	SiR

#### TABLE D.1. CABLES SUPPLIED FOR THE ROUND-ROBIN TEST PROGRAMME

<sup>(1)</sup> Where two numbers are given, the cables supplied were identical types but of different sizes

<sup>(2)</sup> Abbreviations used:

PVC	polyvinyl chloride
PE	polyethylene
XLPE	cross-linked polyethylene
CSPE	chlorosulphonated polyethylene
EPR	ethylene propylene rubber
EPDM	ethylene propylene diene rubber
SiR	silicone rubber
EVA	ethylene vinyl acetate
where ty	wo materials are shown, the insulation is a dual layer material

The generic types of polymer represented by the cables supplied were as follows:

- PVC polyvinyl chloride
- PE polyethylene
- XLPE cross-linked polyethylene
- CSPE chlorosulphonated polyethylene
- EPR ethylene propylene rubber
- EPDM ethylene propylene diene rubber
- SiR silicone rubber
- EVA ethylene vinyl acetate

# TABLE D.2. DETAILS OF CABLE TYPES

Cable no.	Supplying country	Manufacturer	Cable diameter (mm)	Construction, insulation (type, colour, thickness) and conductors	Sheath (type, colour, manufacturer's coding)	Other components (shield, armour, bedding layers)
1126	Russia	Podolsk kabel	15	19 core PVC on solid Cu conductors — white, yellow, purple	PVC (1.5 mm) black - KVVGng	
1127	Czech Rep.	Not known	22	7 core PE on solid Cu conductors — clear, red, yellow (1 mm)	PVC (2 mm) blue + white (1.5 mm), not bonded - KPOBOV	Cu tape and clear bedding layer
1128	USA	Samuel Moore	9	3 core EPDM + CSPE jacket on stranded Cu — white (0.7 mm)	CSPE (1.5 mm) black - Q28	Metallic tape
1129	USA	AIW	16.5	4 core EPR + CSPE jacket on stranded Cu — black (1 mm)	CSPE (2 mm) black with white cloth layer on inside - D14	
1130	USA	Okonite	18	1 core EPR on multistrand uncoated Al — black (2 mm)	CSPE (0.5 mm) bonded to insulation layer — R04	
1131	Sweden	ABB Atom	11	7 core CSPE on stranded Cu — grey (0.7 mm)	CSPE (2 mm) blue - type Lipalon FSSR 7*1	
1132	Canada	Pirelli Canada	15	4 core XLPE 5 on stranded Cu - white (0.7 mm)	PVC (1 mm) black - EQ96	Wrapping tape and filler cords
1133	Canada	Canada Wire	17	6 core EPR 3 on solid Cu - black, white, blue, orange (0.5 mm)	PVC (1.5 mm) black - EQ43	Braided Cu, black bedding layer (1.5 mm)
1134	Canada	Canada Wire	19	12 core PVC 15 on stranded Cu - red, yellow, black, brown, white, blue, green orange (0.7 mm)	PVC (1.5 mm) black - EQ45	
1135	Japan	Hitachi Cable	11	4 core XLPE on stranded Cu - black, white, green, red (0.5 mm)	PVC (1.5 mm) black	Metallic tape, fibre cords between cores
1136	UK	Pirelli	21	7 core EPR/EVA on stranded Cu - white/grey (0.7 mm)	EVA/EVA (2 mm) grey/white	Al spiral armour, grey EVA bedding layer

TABLE D.2	(cont.)
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1137	UK	Pirelli	40	4 core XLPE on solid Al - black, red_vellow blue (1 mm)	EVA/EVA (4 mm) black/white	Al spiral armour, white EVA bedding layer
1138	Germany	Siemens	15	4 core EPR on solid Cu - blue, black, green, brown (1 mm)	EVA (2 mm) black - Sienopyr FRNCX NHXHX-I	White bedding layer bonded to sheath
1139	Germany	Siemens	16	4 core EPR on solid Cu - blue, black, green, brown (1 mm)	EVA (2 mm) black - Sienopyr FRNCX NHXSHXO-J	White bedding layer bonded to sheath
1140	Germany	Siemens	9	4 core XLPE on stranded Cu - white with coloured bands (0.7 mm)	EVA (1 mm) grey - JE- H(ST)H BD SI FRCNX	
1141	Germany	Siemens	18	2 x 20 core XLPE on solid Cu - multi colours	EVA (1 mm) grey - JE- H(ST)H BD SI FRCNX	
1142	Germany	HEW	16	5 core SiR on stranded Cu	SiR (2.5 mm) dark grey - HEW KABEL FRNCX-X	
1143	Germany	HEW	22	20 core SiR on stranded Cu	SiR (3 mm) dark grey	Braid

These represent most of the types of polymeric material used in nuclear power plant. Other materials are in use in specific countries but in limited quantities.

D.9.2.2. Sample preparation

All samples for round-robin testing were prepared by AEA Technology from the cables samples supplied by the participants, not from sheet material. Several different types of sample were prepared, dependent on the tests to be carried out. The sample types were:

- whole cable samples (for indenter measurements, torque testing)
- tensile test samples
- thermal analysis samples (for TGA, OIT, OITP)

The whole cable samples consisted of 100 mm lengths cut from the cable for indenter measurements on sheath materials. Similar lengths of individual insulated cores were also prepared for indenter measurements on insulation materials. For torque testing, longer samples were prepared (300 mm long).

Where possible, standard tensile test samples were cut from the cables using a dumbbell shaped cutter with dimensions to ISO standard, with either a 20 mm or 30 mm gauge length. This was possible for all of the sheath materials and for the insulation from cable 1137. All other insulation samples for tensile testing consisted of tubes of insulation approximately 100 mm long, with the conductor removed. All tensile samples were prepared before being aged.

For all of the thermal analysis tests, small pieces of sheath and insulation material approximately 20 mm long were supplied, from which samples could be cut.

D.9.2.3. Ageing conditions

For the purpose of the round-robin testing, which is intended only to examine the reproducibility of test measurements, thermal ageing was used, not radiation ageing. Each material type was aged in air at a temperature intended to introduce some degradation into the material. All of the samples were aged for the same time period (1008 hours); note that the tensile samples were stripped from the cables and prepared before ageing was carried out. The ageing temperatures used for each material are shown below.

Material	Ageing
type	temperature
	(°C)
PE	100
XLPE	110
PVC	120
CSPE	120
EPR	120
EPDM	120
SiR	120

The PVC and CSPE materials were aged in separate ovens from the EPR, EPDM and SiR samples. Whole cable samples were aged at the temperature appropriate to the sheath material.

Because of the variability in degradation behaviour between materials, even of the same generic group, these ageing conditions did not necessarily produce a large change in properties, but it was necessary to restrict the scope to keep within a manageable test programme.

### D.9.2.4. Test methods

The test methods which were to be compared in the round-robin testing were:

- tensile tests (elongation and strength)
- indenter measurements
- oxidation induction time (OIT)
- oxidation induction temperature (OITP)
- thermo-gravimetric analysis (TGA)

In addition to these test techniques, some participating laboratories have also carried out additional testing using other methods. These include torque testing (Japan) and insulation resistance.

Tensile tests were included in the test programme since they are normally used as the prime indicator of degradation in cable materials and form the basis for definition of the failure criterion in many cases. The other techniques are used for monitoring cable condition but as yet there are no recognised failure criteria based on these particular test methods.

The basic test method to be used for each of the main tests was agreed at the first research coordination meeting held in Vienna, Dec 1996. The agreed methods are summarised in the following sections.

## D.9.2.4.1. Tensile testing

The test method for tensile tests specified the following parameters to be used. The distance between the grips should be 30mm for the tubular insulation samples, and 25mm or 35mm for the jacket samples (dependent on the gauge length supplied — see Table D.3). The cross-head or pulling speed should be 25 or 50 mm/min (or nearest equivalent available on the test machine being used). These cross-head speeds are slower than are usually used for routine testing (200 to 250 mm/min is more typical for pneumatic grips), but can be used with standard mechanical grips. Samples should preferably be gripped using pneumatic grips where available. For the tubular insulation samples, inserts or end tabs need to be used in the gripped sections (see Table D.3 and Figs. D.15 and D.16).

The test report should include the following information:

- identify the colour tested for insulation samples
- report the elongation at break and tensile strength (based on initial cross-section of samples)
- report the number of samples tested, mean values and standard deviation
- identify any tests which are not typical of the batch

Cable	Insulation/s	Sample type	Gauge	Crosshead	Test
no.	heath	(tube/dumbell)	length	spacing	method
			(mm)	(mm)	
1126	Insulation	Tube	30	30	1 (end tab)
1127	Insulation	Tube	30	30	1/2 (end tab
					and insert)
1128	Insulation	Tube	30	30	1 (end tab)
1129	Insulation	Tube	30	30	2 (insert)
1131	Insulation	Tube	30	30	1 (end tab)
1132	Insulation	Tube	30	30	2 (insert)
1133	Insulation	tube	30	30	1 (end tab)
1134	Insulation	tube	30	30	1 (end tab)
1135	Insulation	tube	30	30	1 (end tab)
1136	Insulation	tube	30	30	1 (end tab)
1137	Insulation	dumbell	30	35	
1139	Insulation	tube	30	30	1 (end tab)
1141	Insulation	tube	30	30	1 (end tab)
1143	Insulation	tube	30	30	1 (end tab)
1126	sheath	dumbell	20	25	
1127	sheath	dumbell	30	35	
1128	sheath	dumbell	20	25	
1129	sheath	dumbell	30	35	
1130	sheath	dumbell	30	35	
1131	sheath	dumbell	20	25	
1132	sheath	dumbell	20	25	
1133	sheath	dumbell	20	25	
1134	sheath	dumbell	20	25	
1135	sheath	dumbell	20	25	
1136	sheath	dumbell	30	35	
1137	sheath	dumbell	30	35	
1139	sheath	dumbell	30	35	
1141	sheath	dumbell	30	35	
1143	sheath	dumbell	30	35	

### TABLE D.3. TEST PARAMETERS RECOMMENDED FOR TENSILE TESTS

#### D. 9.2.4.2. Indenter measurements

The cable indenter is a means of measuring the compressive modulus of a cable material by driving a well-defined probe shape into the surface under computer control and monitoring the force-displacement curve. The indenter modulus is taken from the slope of the force-displacement curve at low loads. The value of the modulus obtained will often be determined by the region of the curve selected, since many of the polymers used in cables show a non-linear curve.

The test method used a nominal value of maximum force of 9 N for all cable types (except cable no. 1142/1143, which is a silicone cable which used 4.5 N as maximum force limit). The probe velocity used was 5 mm/min and a minimum of 4 measurements were made circumferentially around the cable at 2 locations. The recommended force ranges over which the modulus was calculated are given in Table D.4.

Tensile Test Procedure (Method 1) For Insulation Specimens



FIG. D.15. Tensile test sample preparation - end-tabs.

TABLE	D.4.	FORCE	RANGES	RECOMMENDED	FOR	ANALYSIS	OF
INDENTI	ER DAT	ΓA					

	Force range (N/mm)					
Cable no.						
	Sh	leath	Inst	ulation		
	Lower range	Upper range	Lower range	Upper range		
1126	1.5 - 4.5		1.5 – 4.5			
1127	1.5 - 4.5		1 - 4			
1128	2.5 - 5.5		1 - 4			
1129	1-3	4.5 - 9	1-3	4.5 - 9		
1130	1 - 2	4.5 - 9				
1131		4.5 - 9		3.5 - 7.5		
1132		4.5 - 9	1-3	4.5 - 9		
1133	1 - 4	6-9		4.5 - 9		
1134	1-3	4.5 - 9		2 7		
1135	1 – 3	5-7		7 – 9		
1136	1 - 4	7-9	1.5 - 3.5			
1137	1 – 3	6 - 9	1 – 3	6 – 9		
1138/9	1 – 3	4.5 - 9	1 – 3	6 - 9		
1140/1	1 – 3		1.5 – 3			
1142/3	1 - 3.5		1-2			

Where two ranges are given, the lower range would usually be used but both ranges have been analysed in this series of tests.

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The test report should include the test temperature, modulus values based on the upper and lower force ranges quoted in Table D.4, and examples of the full test curves.

## D.9.2.4.3. Oxidation induction time (OIT)

Samples were prepared from the bulk material supplied to the participating laboratories. Sample weight was in the range 8 to 12 mg and were taken from the surface for jacket materials, or as a cross-section for insulation materials (solvents should not be used to clean the surface of the cable samples). The sample should be chopped using a scalpel or razor blade to pieces < 1 mm and placed in sample pans which are open or have lids with holes to allow easy access of oxygen.

To get reproducible results using the OIT test, it is very important to use a standardised thermal history. The test method recommended is to initially increase the temperature to  $10^{\circ}$ C below the test temperature in nitrogen at a ramp rate of  $50^{\circ}$ C/min (the recommended test temperatures to be used with each material are shown below). A ramp rate of  $5^{\circ}$ C/min was used for the final ramp to the test temperature and the gas flow switched to oxygen on stabilising at the test temperature. This is normally done by incorporating a hold of 2 minutes at the nominal test temperature before switching the gas flow.





FIG. D.16. Tensile test sample preparation — inserts.

Note that it is very important for reproducibility of OIT tests to ensure that exactly the same heating cycle is used for each test. The oxygen flow rate should be 50–100 ml/min. If possible, the sample temperature should be held constant at the test temperature, rather than the furnace temperature.

Material	OIT test			
	temperature			
	(C)			
EPR/EPDM	210			
XLPE	190			
EVA	200			
CSPE	200			

The test report should include the instrument used for the tests (eg. DuPont model 951) and the calibration method used (eg. indium/lead/tin standard sample). The sample code number and colour should be included. The threshold for oxidation onset should be defined at 0.1 W/g relative to the baseline (if heat flows are too small for an onset to be defined at this value of threshold, use 0.05 W/g). The onset time is defined by the intersection of the tangent to the test curve taken at the threshold with the extrapolated baseline (Figure D.17).





FIG. D.17. Definition of oxidation onset time in OIT test.

# D.9.2.4.4. Oxidation induction temperature (OITP)

Sample preparation for OITP tests is the same as that used for OIT tests. The test should be carried out at a temperature ramp rate of 10°C/min in an oxygen flow rate of 50–100 ml/min.

The test should state the instrument used for tests and the calibration method used and note the sample code number and colour. The threshold for oxidation onset is defined at 0.1 W/g relative to the baseline (if heat flows are too small for an onset to be defined at this value of

threshold, use 0.05 W/g). The onset temperature is defined by the intersection of the tangent to the test curve taken at the threshold with the extrapolated baseline (Figure D.18).

The maximum temperature in the OITP test only needs to be high enough to determine the onset value.

## D.9.2.4.5. Thermo-gravimetric analysis (TGA)

Sample preparation for TGA tests is the same as that used for OIT tests. The test should be carried out at a temperature ramp rate of  $10^{\circ}$ C/min in an oxygen flow rate of 50–100 ml/min. The maximum temperature in the TGA test only needs to be high enough to determine the onset value.

The test report should state the instrument used for tests and the calibration method used and note the sample code number and colour. The onset temperature should be taken as the temperature at which 2.5% and 5% weight loss is reached (Figure D.19).





FIG. D.18. Definition of oxidation onset temperature in OITP test.

## D.9.2.4.6. Torque testing

Torque measurements were only made by one laboratory (JAERI) but have been included for comparison with the other techniques. The torque tester uses lengths of whole cable which are gripped in a pair of chucks spaced 50 mm apart. One end of the cable is held fixed and the other is twisted by 10° at a frequency of 0.5 Hz (Figure D.20). The peak-to-peak torque is monitored at the third complete cycle. The instrument used can carry out such tests on cables with diameters from 9 mm to 17 mm. Some of the cables were too large in diameter to be tested (cables 1127, 1130, 1134, 1136 and 1137).



Schematic of output from TGA

FIG. D.19. Definition of 5% weight loss temperature in TGA test.



FIG. D.20. Schematic layout of the torque tester (courtesy of JAERI).

#### D.9.2.5. Statistical test method

In comparing the results of the round-robin tests from the different test laboratories, some simple statistical analysis has been carried out. Since all of the samples were prepared by the same laboratory under the same conditions, the assumption has been made that all of the samples come from the same population. An estimate of the population mean and variance can be taken from the total data obtained in the tests.

For test batches of mean value x, standard deviation s and sample number n, the population mean X is given by

$$X = (n_1.x_1 + n_2.x_2 + \dots + n_k.x_k) / N$$

where  $N = n_1 + n_2 + .... n_k$ 

The best estimate of the population variance  $\sigma^2$  is given by

$$\sigma^2 = T/(N - k)$$

where  $T = n_1 s_1^2 + n_2 s_2^2 + \dots + n_k s_k^2$  and k is the number of batches tested.

The t-test is a means of identifying whether a sample is representative of a population and can be used to assess whether the round-robin tests from different laboratories give results which are representative. The parameter t is defined for each sample batch from each test laboratory as

t = error in mean/standard error of mean =  $|X - x| . \sqrt{(n-1)} / \sigma$ 

The larger the difference between the means of the sample and the population, the greater the value of t. The parameter t has its own probability distribution with any specified value of t being exceeded with a calculable probability which takes into account the degrees of freedom (N - k).

This probability distribution is often used in the form of a graph such as that shown in Figure D.21, which shows the value of t as a function of the numbers of degrees of freedom (N - k) for different levels of probability. For example, Figure D.20 shows that for a test series where (N - k) = 10, there is only a 1% probability that a sample with a t value of 3.2 belongs to the population tested. The difference between this set of data and the estimated population mean is regarded as being statistically significant.

#### **D.9.3.** Results of round-robin tests

#### D.9.3.1. Participating organisations

The test laboratories and organisations participating in the round-robin testing are listed in the table below. Each of the participants tested the materials and test techniques which were of particular interest to their own programmes on cable ageing.

Contact names for each of the participating organisations are in Appendix 7. More details of the individual test data may be available from these contacts.

Country	Organisation	Tensile	Indenter	OIT	OITP	TGA	Other
Canada	AECL,	~			~		
	NBP (Pointe LePreau),		~				
	Ontario Hydro		~	~		~	
Czech Rep.	NRI, Rez	~		~	~		
France	EdF/Cis Bio		~	~	<b>~</b>		
	International						
Germany	Siemens KWU	~					
India	Bhabha Atomic Res.	✓					
	Centre						
Japan	JAERI, Takasaki	~				~	✓ <sup>(1)</sup>
Romania	Eurotest SA	~				~	
Russia	Minatom, RISI	~	<ul> <li>✓</li> </ul>			~	
Sweden	ABB Atom,	✓		~			
	Ingemansson		~				
Switzerland	CERN	~					
UK	AEA Technology		~				
USA	EPRI/PSE,						
	Brookhaven Nat. Lab.			~	~		

<sup>(1)</sup> torque testing

#### D.9.3.2. Tensile test results

#### D.9.3.2.1. Test data

Appendix 1 contains the full tensile test results. Section A1.1 gives the tabulated data for each cable with the tables containing the following information for the data from each test laboratory.

- Mean elongation value
- Standard deviation
- No. of samples tested
- Calculated t-values
- Cross-head speed used in the test
- Colour of sample tested (where given)

In addition, the estimated population mean X and standard deviation  $\sigma$  are given for each material, as defined in section D.9.2.5, in the final summary table in Appendix 1.1. In calculating the t-values for each sample batch, the population mean and variance have been calculated from the total data set for each material (as described in section 2.5) and compared with the sample means. For those materials where the degrees of freedom (N – k) are >20, t values of greater than about 2.5 indicate that the sample is significantly different from the estimated population mean. For this series of tests, this implies that some aspect of the test method used has affected the values obtained.

The data are also shown in graphical format in Appendix A1.2 as bar charts of mean values  $(x_n)$  with an error bar of  $\pm$  one standard deviation  $(s_n)$ .

Appendix A1.3 also contains some examples of the type of tensile test curves obtained on sheath samples (data from JAERI, Japan). The plots show the type of stress-strain curves obtained on a range of materials, both as-received and after ageing.

Comments on the behaviour of the different generic types of material are given in the following sections. The cable code numbers corresponding to the generic types are given in the table below.

Generic type	Cable numbers
PVC	1126I, 1126S, 1127S, 1132S, 1134I, 1134S,
	1133S, 1135S
PE	1127I
XLPE	1132I, 1135I, 1137I, 1140/1I
EPDM	1128I, 1131I
EPR	1129I, 1130I, 1133I, 1136I, 1138/9I
CSPE	1128I <sup>*</sup> , 1128S, 1129I <sup>*</sup> , 1129S, 1130S, 1131S
EVA	1136I <sup>*</sup> , 1136S, 1137S, 1138/9S, 1140/1S
SiR	1142/3I, 1142/3S

— present only as a thin outer layer on the insulation

Brief comments on the behaviour of the different material types are given below based on the t values obtained from the estimated population mean and standard deviations. These can be skewed very noticeably if there is a single data set which is very different from the others. For example, in cable 1129 sheath material, if the single high value of mean elongation is removed, t values for the other data sets reduce from 4.1 maximum to less than 2.3.

## D.9.3.2.2. CSPE materials

For many of the results, t values tend to be high indicating that variations in test procedures are affecting the results obtained. However, there appears to be no systematic variation associated with test parameters which were reported, such as cross-head speed. For these materials, only one colour was present for each material so there were no pigment effects observed. Within individual test laboratories, the coefficient of variation (s/x) for individual batches is typically 6% for as-received material and 11% for aged material.

## D.9.3.2.3. Ethylene propylene (EPR and EPDM) materials

These materials also show a significant proportion of high t values. For these materials, only one colour was present for each material so there were no pigment effects observed. Within individual test laboratories, the coefficient of variation (s/x) for individual batches tends to increase after ageing for the EPDM-based materials but is unchanged in the EPR-based materials. Typical values of s/x for unaged material are 6 to 8% for the EPDM and 8 to 10% for the EPR.

# D.9.3.2.4. EVA materials

These materials show slightly less variability between laboratories, as indicated by t values which are usually < 2.5, but this may just be a reflection of the higher standard deviation values seen in these materials. Typical coefficient of variation values are 12% in the asreceived material and 15% in the aged material. For these materials, only one colour was present for each material so there were no pigment effects observed.

# D.9.3.2.5. PVC materials

The PVC based materials showed a large variability between laboratories, as indicated by the t values, particularly for the as-received material. The aged samples tend to show lower t values, but this is probably due to the higher standard deviations observed. Coefficients of variation are typically 7% in as-received material and 19% in aged material. For these materials, only one colour was present for each material so there were no pigment effects observed.

## D.9.3.2.6. PE and XLPE materials

Some of the polyethylene based materials show a very large variation between laboratories, as indicated by the t values. This might be related to the different colours which were tested. Within individual test batches, coefficients of variation can range from 2% to 30% for the same material tested by different laboratories. The PE-based materials show some of the largest variations between laboratories.

## D.9.3.2.7. Silicone materials

Most of the test batches from the silicone sheath material tested have t values <2.5 but the insulation material generally has t values >2.5. This may just be a reflection of the difference in coefficient of variation, which is typically 6% for the insulation but 12% in the sheath material. For these materials, only one colour was present for each material so there were no pigment effects observed.

#### D.9.3.2.8. General comments

Considerable variability has been observed in the tensile test data obtained from the participating test laboratories. From the information reported, the cause of the variation cannot be established. However, there are a number of factors which may be sources of variation; not all of these were reported in this test programme.

- Method of gripping the samples in the test machine
- Method used to measure tensile elongation
- Cross-head speed
- Effect of different pigments in the samples
- Location of the failure point
- Type of test machine used and method of calibration
- Variability of material composition or structure
- Test temperature

The gripping of samples for tensile testing can affect the reproducibility of results and the mode of failure. It is usually recommended that pneumatically operated grips are used with polymer samples, but many laboratories use friction grips which are mechanically tightened where the grip force is not monitored. The grip faces may be smooth or may be ridged to enhance gripping.

In determining the elongation at break of a tensile sample, measurements may be made using optical extensioneters or use the cross-head movement. The gauge length used to measure elongation also needs to be specified. In this series of tests, the method to be used for measuring elongation was not specified.

Most of the tests in this programme used a cross-head speed of 50 mm/min, but some laboratories used 100 or 200 mm/min. The elongation measured is often a function of testing speed for polymeric materials and slower testing speeds usually give more reproducible results. However, the practicalities of testing timescales are such that most laboratories would use a cross-head speed of 200 mm/min or more for routine testing and only use 50 mm/min or less for more detailed studies.

The use of different pigments within nominally the same material may introduce variations in the material's properties. There was not enough information in this programme to establish whether pigment effects were significant.

If samples fail within the grip or immediately adjacent to the grip, the data generated are usually assumed to be non-representative and are excluded from any assessment of mean properties. This was a particular concern in the tubular insulation samples used for tensile tests.

The type of test machine used and its method of calibration need to be included in any test report.

Variability of the properties of the material can also contribute to the variations observed in a round-robin test programme such as this. Although all samples were prepared by the same test laboratory from the same length of cables (typically 6 m long), there is still scope for variation arising from inhomogeneity of the material. Another factor may be the influence of the cable storage conditions. For cables which are normally stored as a coil, samples cut from the inner side of the coil will have been exposed to different stress levels to those taken from the outside of the coil.

The mechanical properties of polymeric cable materials can be strongly dependent on the temperature at which they are tested. Some of the variability observed in the tensile test data may be as a result of different test temperatures in the laboratories. This would be particularly important if the test temperature is outside of the recommended range of 20–25°C.

Overall, the tensile test results highlight the need for more detailed reporting of the test conditions used and a detailed specification of the test method to be used before comparisons can be made between data from different test laboratories.

#### D.9.3.3. Indenter test results

Only a limited number of laboratories were able to carry out indenter measurements, but all used the same make of indenter instrument. The data tables for each of the cable materials

which were tested are shown in Appendix 2.1. Each table contains the mean values and standard deviations, the temperature at which the indenter measurements were made and the force ranges used for analysis. For some materials, which showed non-linear plots of force against displacement, two ranges were used for analysis. The force ranges finally selected for analysis (Table D.4) were decided after consideration of the shape of the force-displacement plots for each of the material types.

# D.9.3.3.1. Temperature effects

In some of the materials there appears to be an effect of sample temperature on the values of indenter modulus obtained, even with a standardised test procedure. The sample temperatures during measurements were monitored by the test labs and were in the range 17°C to 26°C. The temperature dependence of the indenter data are shown graphically in Appendix A2.2 for each of the materials.

In most of the cable materials tested, there is a small but significant decrease in the indenter modulus with increasing temperature. The limited data available indicate that the modulus changes linearly with temperature over the limited temperature range considered. There is some indication that the temperature dependence becomes more marked after ageing. Cable 1126 was unusual in that the PVC sheath and insulation both show an increase in modulus with increasing temperature after ageing. However, this was for a heavily degraded material where the measured modulus values are very high and show a large standard deviation.

The PVC, EVA and some of the XLPE materials tend to show the largest effects of temperature on IM values. Most of the CSPE, EPDM and EPR materials show only a small effect. The SiR materials appear to be independent of temperature over this range.

# D.9.3.3.2. Effect of ageing

In general, the indenter modulus increases with ageing. PVC and CSPE materials tend to show the largest increases in indenter modulus with ageing. However, where there is significant plasticizer migration on ageing, softening of the surface can occur and indenter modulus values can be lower in aged samples. This effect was seen in the sheath material of cable 1127.

In the aged materials in particular, the indenter values are very variable within one laboratory, as shown by the high standard deviation values. In some cases, eg. cable 1126 and 1128 sheath, this is because the cable is not very well packed, with significant air gaps. Some examples of the variability between individual indenter curves on the same material, before and after ageing, are shown in Appendix 2.3 (data courtesy of Ingemansson, Sweden).

Some of the PE and XLPE materials also show a significant change in indenter value after ageing. This was slightly unexpected, in that the indenter is not normally considered suitable for these materials.

## D.9.3.3.3. General comments

The results obtained for indenter measurements show a good correlation between laboratories provided the same force ranges are used for analysis. This particular test method had the advantage of being carried out on a single type of test instrument so instrumental variations

were limited. The tests have highlighted the need to report the temperature of the sample and the force ranges used for analysis when comparing data between laboratories. Where two ranges were used for measurement of the indenter modulus, the lower force range was generally found to give more consistent results.

Because of the temperature dependence of the indenter modulus in most of the cables that were tested, statistical analysis (in the form of a t-test) was not appropriate.

#### D.9.3.4. OIT tests

A summary of the test parameters used by each laboratory for OIT testing is given in Appendix 3.1, including the method used for determining the onset of oxidation. Most laboratories used the intersection of a tangent to the threshold value with the baseline. For some instruments, this was not possible and an automatic software command was used which generated a tangent to the steepest part of the oxidation exotherm. This method generally gives higher values than the tangent at a fixed threshold. Where a fixed threshold was used, but a tangent was not drawn, the values will also be higher.

Data tables for all of the cable materials tested are shown in Appendix 3.2. Each table contains the mean values and standard deviations of the oxidation induction time, the test temperature and the colour of sample tested. There were very few materials which were tested by more than 3 laboratories. Most test data were obtained on the EPR, EPDM, EVA and XLPE materials, with only a few laboratories attempting to test PVC or CSPE materials.

## D.9.3.4.1. Interpretation of OIT thermograms

There were some general problems associated with OIT measurements. The test temperatures selected were too low for onset measurements to be made in a reasonable time scale for some materials. This was particularly noticeable in the 1137 insulation and 1138/9 sheath materials.

In a number of materials, baseline determination was also difficult because of sloping or very variable baselines. In others, the heat flows observed were very low making onset determination difficult and in some materials multiple onsets were observed. These are summarised in Table D.5, for those materials which were tested.

Some examples of raw data plots are shown in Appendix 3.3 (data from Ontario Hydro and NRI) for EPR, EPDM and XLPE insulation materials. Example 1 shows a well-defined onset and a reasonable baseline. Examples 2 and 3 show some of the problems associated with identifying what is a suitable baseline. Example 4 shows an OIT plot where the heat flows are very low and example 5 shows multiple onsets. The type of variability observed between samples within the same test laboratory is shown in example 6.

Typical variations in determining the OIT onset time within individual laboratories are 16%.

## D.9.3.4.2. Ethylene propylene based materials (EPR and EPDM)

Only a few of the materials tested showed consistent thermograms with flat baselines and a well defined single onset. Most of these materials showed multiple onsets. Where there are multiple onsets, it is still possible to use OIT provided that the same onset is used. For example, in 1133 insulation the data between the 3 laboratories which carried out tests is

Cable ID	Material	Baseline	Heat flow	Onsets
1126 I	PVC	variable		poorly defined
1126 S	PVC	sloping		
1127 I	PE	variable		multiple onsets, poorly
1100 1				defined
11281	EPDM+CSPE	good		poorly defined
1129 I	EPR+CSPE	good	low	well defined, multiple onsets
1129 S	CSPE	sloping		poorly defined
1130 I	EPR	good		well defined
1130 S	CSPE	not established		not visible after 160 min
1131 I	EPDM+CSPE	good	very low	
1131 S	CSPE	variable		difficult to define
1132 I	XLPE	good		well defined
1132 S	PVC	good	very low	multiple onsets
1133 I	EPR	good		multiple onsets
1133 S	PVC	sloping		poorly defined
1135 I	XLPE	good	low	poorly defined
1136 I	EPR+EVA	good		multiple onsets
1136 S	EVA+EVA	variable		poorly defined
1137 I	XLPE	good		well defined
1137 S	EVA+EVA	variable	low	multiple onsets
1138/9 I	EPR	good		multiple onsets
1138/9 S	EVA	good		well defined
1140/1 I	XLPE	good		well defined
1140/1 S	EVA	variable		multiple onsets

#### TABLE D.5. TYPE OF THERMOGRAMS OBTAINED IN OIT TESTS

reasonably consistent. Overall the consistency between laboratories is poor compared with the variation observed within laboratories.

## D.9.3.4.3. Polyethylene based materials (PE and XLPE)

These tend to give thermograms with good baselines and a single well defined onset, but low heat flow was observed in several materials, making onset determination more difficult. The low heat flows observed were possibly due to too low a temperature being selected for this series of tests. Overall the consistency between laboratories is poor compared with the variation observed within laboratories.

## D.9.3.4.4. EVA materials

Most of the EVA materials had poor baselines and multiple onsets making consistent determination of onset times very difficult. Only one material (1138/9 sheath) showed clear thermograms. Unfortunately there were very few test results on this material but the limited data available indicate a reasonable consistency between two laboratories using the same type of test equipment.

### D.9.3.4.5. CSPE and PVC materials

All of these materials tend to give variable baselines and poorly defined onsets. OIT testing would not normally be recommended on these types of materials because of the corrosive degradation products which are generated during the test. The data from those few laboratories which attempted to carry out OIT tests indicate that the test method is not particularly useful.

## D.9.3.4.6. General comments

There were insufficient data available to carry out statistical analysis (in the form of a t-test) on the results obtained.

OIT tests are only likely to give reproducible results where there is a good baseline and a single well-defined onset. This limits there use to XLPE and some EPR materials. The temperature used for OIT testing needs to be established for each material individually if test times are to be kept to a practical limit of 60 to 90 minutes. In this series of tests, the generic values of test temperature used were not necessarily suitable.

Practical limits to sampling of cables within a NPP may require smaller samples (1 to 2 mg) to be used with the OIT test. For those materials which show a low heat flow, this may limit the usefulness of the test method.

#### D.9.3.5. OITP tests

The test parameters and data tables for all of the cable materials which were subjected to OITP tests are shown in Appendix 4.1 and 4.2. Each data table contains the mean values and standard deviations for the onset temperature and the colour tested. The data available are very limited, with only a few materials being tested by more than 2 laboratories.

#### D.9.3.5.1. Interpretation of thermograms

There were fewer problems in making OITP measurements than with OIT measurements, mainly because baselines tend to be more consistent and the oxidation exotherm has a higher heat flow and is better defined. Even where the onset is not as well defined, onset measurements are still reasonably straightforward. In some materials a dip in the baseline is seen immediately prior to the oxidation onset, which can cause some problems in defining the appropriate baseline to use. Where the material contains significant amounts of crystalline material (eg. in XLPE), an endothermic melting peak is also seen, before the oxidation exotherm is reached. TableD.6 identifies the type of thermograms observed for each material.

Some examples of raw data plots from OITP tests are shown in Appendix 4.3 for EPR, EPDM, XLPE and CSPE materials. The first two examples show the type of well-defined baseline and onsets that are typical for many amorphous (example 1) and semi-crystalline (example 2) cable materials. In example 2, the endothermic melting peak is seen at temperatures below 100°C. Example 3 shows the more gradual onset typical of the CSPE materials. Example 4 shows an endothermic dip immediately before the oxidation exotherm. The main concern here is in the definition of the baseline, whether to use an extrapolation of the lower temperature baseline or whether to use the lowest part of the dip.

Cable ID	Material	Baseline	Melting	Onsets	
			endotherm		
1127 I	PE	good	yes	Gradual	
1127 S	PVC	good		Gradual, multiple onsets	
1128 I	EPDM+CSPE	variable		Sharp	
1128 S	CSPE	good		Gradual	
1129 I	EPR+CSPE	good		Gradual	
1129 S	CSPE	sloping		Gradual	
1130 I	EPR	sloping		Sharp	
1130 S	CSPE	sloping		Gradual	
1131 I	EPDM+CSPE	variable		Gradual	
1131 S	CSPE	variable		Gradual, multiple onsets	
1132 I	XLPE	good	yes	Sharp	
1133 I	EPR	sloping	small	Sharp	
1135 I	XLPE	good	yes	Sharp	
1135 S	PVC	good		Multiple small onsets	
1136 I	EPR+EVA	sloping		Endothermic before sharp onset	
1136 S	EVA+EVA	sloping	small	Sharp	
1137 I	XLPE	good	yes	Sharp	
1137 S	EVA+EVA	sloping	small	Endothermic before sharp onset	
1138/9 I	EPR	sloping		Endothermic before sharp onset	
1138/9 S	EVA	good		Endothermic before sharp onset	
1140/1 I	XLPE	good	yes	Sharp	
1140/1 S	EVA	sloping		Endothermic before sharp onset	

#### TABLE D.6. TYPE OF THERMOGRAMS OBTAINED IN OITP TESTS

OITP onset temperatures from EdF tend to be consistently higher than that from other laboratories because of the difference in the method used for their calculation. The French test equipment had analysis software which determines onset from extrapolation at the maximum slope of the exothermic reaction, whereas in the other laboratories, analysis could be carried out at a fixed deviation from the baseline.

Typical variations in the measurement of OITP onset temperatures within individual laboratories are 3°C.

#### *D.9.3.5.2. Ethylene propylene based materials (EPR and EPDM)*

The EPDM materials associated with CSPE outer layers tend to show rather variable baselines which makes determination of the onset more difficult. The other EPR materials tend to show good baselines, often sloping and with an endothermic dip immediately prior to the oxidation exotherm eg. 1138/9 insulation. Some of the variability between laboratories for these materials may be associated with the definition of the baseline.

## *D.9.3.5.3.* Polyethylene based materials (PE and XLPE)

These are all semi-crystalline materials and show a melting endotherm. All of the XLPE materials tested show a good baseline and a sharp oxidation onset. The PE material shows a

rather more gradual onset. The values obtained are reasonably consistent between laboratories provided the method of measuring onset temperature is taken into account.

# D.9.3.5.4. EVA materials

These materials tend to show a sloping baseline and usually have an endothermic dip immediately before a sharp oxidation exotherm. The very limited data indicate a reasonable consistency between laboratories provided the method of measuring onset temperature is taken into account.

## D.9.3.5.5. CSPE and PVC materials

This method is not recommended for PVC based materials because of the corrosive degradation products generated during the test. The CSPE materials tested tend to show sloping baselines with a gradual onset. The very limited data indicate considerable variation between laboratories. The single PVC that was tested showed good baselines but multiple onsets.

## D.9.3.5.6. General comments

There were insufficient data available to carry out statistical analysis (in the form of a t-test) on the results obtained.

Interpretation of OITP thermograms is usually easier than in OIT tests, but in both test methods, the baseline used needs to be specified in the test report to aid comparison between test laboratories.

Practical limits to sampling of cables within a NPP may require smaller samples (1 to 2 mg) to be used with the OITP test. For those materials which show a low heat flow, this may limit the usefulness of the test method. D.9.3.6. TGA tests

Test parameters and data summary tables for all of the cable materials tested are shown in Appendix 5.1 and 5.2. Each table contains the mean values and standard deviations, and the sample colour tested. The TGA test is usually used as an alternative to OIT and OITP testing for PVC based materials. In this series of tests, a few additional tests were also carried out on other materials.

TGA measurements are generally well behaved, with consistent baselines and significant changes in weight at the onset of oxidation, making determination of the onset temperature relatively straightforward. An example is shown in Appendix 5.3.

The main source of variation in the TGA onset temperatures was found to be the oxygen flow rate through the test cell. This ranged from zero flow (in a closed cell system) to 50 ml/min of air to 100 ml/min of oxygen. There was a direct correlation between the oxygen content and the measured TGA onset for the PVC materials, the onset being lowest in the samples tested in flowing oxygen. It has not been possible to assess the variability between laboratories because of these differences in the test method.

The oxygen content also affects the changes observed on ageing. In PVC, the TGA onset increases on ageing when measured in oxygen but decreases with ageing for TGA measured in a closed cell.

Within the individual laboratories, the TGA onset temperature typically shows variations of 2°C, up to 5°C.

#### D.9.3.7. Torque testing

The changes in torque on ageing are shown in Appendix 6 for each of the cable samples tested. For most of the samples, there is an increase in torque on ageing. The initial torque measured is a function of both the material properties and the construction of the cable. Smaller diameter cables and/or a small number of cores tend to give low initial torque values, whereas the presence of bedding layers, braids and armouring tend to give large initial torque values.

#### D.9.3.8. Comparison of test methods

Although the test programme was not designed to compare the effectiveness of the different condition monitoring methods used, some information can be obtained by comparing the effect of ageing on the various test parameters. These are only rough comparisons, between the estimated population means for tensile elongation, which is normally regarded as being the prime indicator of ageing degradation, and the other parameters. In comparing the data, some approximations have had to be made. The elongation values are taken from the pooled estimates of population means. The indenter values are also taken from the pooled estimates, without taking into account any variations arising from differences in temperature. The OIT ratios were calculated from the mean values for each laboratory and then averaged for each material.

The different test methods have been compared in the table below. The ratios of elongation, indenter modulus, OIT and torque before and after ageing have been included in the table, together with the estimated change in onset temperature for the OITP and TGA tests.

For completeness, the correlation between changes in elongation and the other test methods are shown in Figs. D.21–D.25 for each of the generic material types.

Cable ID	Material	Typical change in properties						
		Elongation	Indenter	OIT/	ΔΟΙΤΡ	ΔTGA <sup>*</sup>	Torque	
		e/e <sub>0</sub>	IM/IM <sub>0</sub>	OIT <sub>0</sub>	(°C)	(°C)	test T/T <sub>0</sub>	
1128 S	CSPE	0.23	2.00		-16	-1	4.23	
1129 S	CSPE	0.48	1.43	0.04	-30	0	1.18	
1130 S	CSPE	1.08	4.67	0	+3	+1		
1131 S	CSPE	0.37	1.51		-16	-4	1.46	
1131 I	EPDM	0.37	1.83	0.04	-9	+16		
1128 I	EPDM+CSPE	0.92	1.16	0.40	-19	-4		
1130 I	EPR	0.40		0.76	-15			
1133 I	EPR	0.86	1.00	0.95	+8	0		
1136 I	EPR	0.94	1.20	0.12	-40	+2		
1138/9 I	EPR	0.85	1.39	0.80	-2	+29		
1129 I	EPR+CSPE	0.25	2.19	0.22	-25	-6		
1136 S	EVA	0.83	0.99		+21			
1137 S	EVA	0.71	1.63	0	+8	0		
1138/9 S	EVA	1.06	1.03	0	-8	-1	1.37	
1140/1 S	EVA	0.92	1.05	0.62	-9	-2	1.76	
1127 I	PE	0.04	1.14	0	-15	-23		
1126 I	PVC	0.55	7.03	0		+15		
1126 S	PVC	0.07	6.69	0.63		+17	3.44	
1127 S	PVC	0.89	0.90		0	-4		
1132 S	PVC	0.58	1.44	0.71		+16	1.28	
1133 S	PVC	0.37	1.71	0.42		+2	1.20	
1134 I	PVC	0.79	1.29			-3		
1134 S	PVC	0.47	1.79			+3		
1135 S	PVC	0.89	0.90		-22	-1	1.54	
1142/3 I	SiR	0.90	1.00					
1142/3 S	SiR	0.83	1.02				1.25	
1132 I	XLPE	0.94	1.22	0.64	-10	+27		
1135 I	XLPE	0.93	1.32	0.65	-3	+2		
1137 I	XLPE	0.96	0.97	0.11	-15	+9		
1140/1 I	XLPE	0.99	2.20	0.43	+3	-7		

\* tests in flowing oxygen only



FIG. D.21. Correlation between indenter modulus ratio and elongation ratio.



FIG. D.22. Correlation between OIT ratio and elongation ratio.


FIG. D.23. Correlation between change in OITP onset temperature and elongation ratio.



FIG. D.24. Correlation between change in TGA onset temperature and elongation ratio.

#### Correlation - Elongation/torque



FIG. D.25. Correlation between torque ratio and elongation ratio.

# **D.9.4.** Conclusions

The main conclusion that has come out of this series of round-robin tests has been the need for more detailed specification of the test procedures for condition monitoring methods. This includes the information which needs to be reported with any data generated from such tests.

At this stage, the most robust of the techniques tested appears to be indenter measurements. These have shown a reasonable level of variability between laboratories once the analysis procedure was standardised and generally correlate well with ageing degradation for most materials.

Specific recommendations on the information needed for the different tests are given below. The following information should be included in the test report in addition to the test data.

#### **Tensile tests:**

- Type of test machine used
- Calibration procedure
- Method of gripping samples and type of grip face
- Test temperature (particularly if outside the range 20–25°C)
- Cross-head speed
- Method of measuring elongation

#### Indenter tests:

- Test temperature
- Probe speed
- Force range used for analysis (it would be helpful to establish standard ranges for materials commonly used in cables)

• Calibration procedure (it would be helpful to have standard samples available for calibration purposes)

# **OIT tests:**

- Type of instrument used
- Method of calibration
- Sample weight and preparation method
- Type of sample pan (whether open or closed)
- Oxygen flow rate used
- Temperature profile used to reach oxidation temperature
- Method of establishing baseline (a raw data plot would be helpful)
- Method of establishing oxidation onset

# **OITP tests:**

- Type of instrument used
- Method of calibration
- Sample weight and preparation method
- Type of sample pan (whether open or closed)
- Oxygen flow rate used
- Temperature ramp rate and starting temperature
- Method of establishing baseline (a raw data plot would be helpful)
- Method of establishing oxidation onset

# **TGA tests:**

- Type of instrument used
- Method of calibration
- Sample weight and preparation method
- Type of sample pan (whether open or closed)
- Oxygen flow rate used
- Temperature ramp rate and starting temperature
- Method of establishing oxidation onset

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# ANNEX E

# (TO SECTION 7 OF PART I, VOLUME I: PREDICTIVE MODELLING OF CABLE AGEING)

Annex E contains additional material of relevance to Section 7 of Part I, Volume I of this report on predictive modelling. It includes more detailed information on the following aspects related to Section 7. (For each of the main modelling methods, information is given on the test procedure, limitations of the model and some examples of its application to cable materials).

- Simple linear model for cable materials
- Power law extrapolation method
- Superposition of time dependent data
- Superposition of DED data
- Prediction of lifetime of rubber cable jacket materials.

# E.1. SIMPLE LINEAR MODEL FOR CABLE MATERIALS

The aim of predictive modelling is to estimate either the residual life of in-service (i.e. already installed) cables or the expected life of new cables as closely as possible. Predictive modelling is based on the simulation of in-containment environmental conditions by an accelerated test. Linear predictive modelling of cable life is based on linear fitting and neglecting the synergistic effects. This approach enables life prediction to be carried out as easily (and quickly) as possible. But, the likelihood of such ageing results matching real time ageing is lower than in the case of using the more sophisticated models.

This disadvantage can be often offset by its simplicity. It is why this linear approach for thermal, radiation, and combined thermal-radiation ageing is also often recommended in Guides and Standards [E.1E.4] for application of material life testing, radiation resistance testing, and initial environmental qualification (type testing) of cables.

The assumptions and requirements of the linear model are as follows.

- (a) All significant environmental conditions have to be determined for each cable type and assessed, whether they are of severe impact or whether they can be neglected.
- (b) The cable degradation caused by all individual environmental conditions is assumed to be additive, so that each stress can be simulated separately (i.e. synergistic effects are neglected). But, if a synergistic effect is known and simultaneous accelerated ageing is not possible, additional margin should be applied during accelerated ageing.
- (c) If the influence of the order of application of individual environmental ageing conditions is known, the individual conditions should be applied in such sequence to obtain the most severe degradation.
- (d) The simulation of individual environmental conditions may be performed either:
  - by a single block of ageing or
  - by splitting the simulated cable life into several intervals (e.g. 4 blocks of 10 years for simulation of 40-years NPP service) and applying these 4 blocks of simulation using the same order for each individual environmental condition as in the single ageing block).
- (e) The linear extrapolation for thermal ageing is assumed to be acceptable, using plots of log time versus 1/T coordinates, i.e. the Arrhenius equation is assumed to be valid over

the range of temperatures and times. The limitations for using Arrhenius equation and life extrapolation are discussed more detailed in reference [E.1] and also in Section 3 of Part I, Vol. I.

- (f) The linear extrapolation (interpolation) for radiation ageing to lower dose rates is assumed to be acceptable in using time versus cumulative dose (i.e. no dose rate effect occurs).
  - Homogeneous oxidation conditions should be achieved during radiation. The recommended dose rate for accelerated radiation ageing for simulating the inservice cable doses under normal NPP operation is equal to or less than 0.1 kGy/h [E.2].
  - Irradiation at cable service temperature (usually 50–60°C) is preferred, the irradiation at room temperature is also acceptable. If the influence of irradiation temperature is known, this should be taken into consideration, i.e. non-linear approach should be applied. Either general theoretical models can be applied [E.5, E.6] or special models could be incorporated (if some anomalies on temperature dependence of radiation degradation are known) [E.7–E.9].
- (g) The DBE survivability should be tested by DBE and post-DBE simulations performed both, before and after simulation of normal service NPP life of cable. When the cable life is simulated in blocks, the DBE simulation can be (but need not to be) performed after each ageing block to test the DBE survivability after 10, 20 and 30 years of cable life as well.

The final stage of linear modelling consists of the construction (mathematically and/or graphically) of the predictive curves based on the extrapolation of data from accelerated ageing (end point criterion values versus simulated NPP service life) from which the lifetimes could be estimated. Examples of such procedures are given in the Annex D, Sections D.2 and D.5, where some typical results from the Czech NPP Dukovany are shown. Here only the basic features are outlined:

- (a) The changes in elongation at break as a function of the simulated service life are fitted and extrapolated (or interpolated) to the point, where the fitted curve crosses the y coordinate equal to 50 % of *absolute* elongation at break; the corresponding x co-ordinate gives the expected lifetime.
- (b) For each time period (usually 10-, 20-, and 30-years) of the simulation, the absolute elongation at break is correlated with another non-destructive property which can be measured on NPP cables (e.g. OIT and/or OITP). These enable comparison of results from the modelling based on accelerated ageing experiments with OIT/OITP data coming from in-situ sampling.
- (c) As the cable insulations are not always accessible, correlation has to be made between the cable jacket and insulation properties (i.e. for elongation at break, and for OIT/OITP or any other non-destructive condition indicator) see Annex D, Section D.5.

#### E.2. POWER LAW EXTRAPOLATION METHOD

This method is based on the extrapolation of test data obtained under isothermal conditions in air over a range of dose rates. The upper limit to the dose rate is such that homogeneous oxidation conditions are achieved (but see Section E.1.2). The test data obtained at the different dose rates are used to determine end point criteria which are extrapolated graphically to the service dose rate.

#### E.2.1. Test procedure

The maximum dose rate at which homogeneous oxidation will occur in the test material must be assessed. Once the maximum dose rate has been established, at least two (preferably 3) other dose rates should be selected, each dose rate preferably being one order of magnitude lower than the previous value.

For the types of materials for which this procedure is recommended (i.e. elastomers and thermoplastics), elongation at break e is generally the property measured. To establish the end points at each dose rate, the relative elongation  $e/e_0$  is plotted against absorbed dose ( $e_0$  is the initial value of the elongation at break). A number of end point criteria can be interpolated from the graph (Figure E.1); typical end point criteria might be  $e/e_0 = 0.75$  or 0.5. A sufficient number of absorbed doses must be used to enable the end point criterion at each dose rate to be established without extrapolation.

The dose at which the end point criterion is reached, i.e. the dose to equivalent damage DED, is plotted against the dose rate in a log/log plot (Figure E.2). This plot is found to be linear in some materials e.g. some polyolefins, enabling extrapolation to lower dose rates. The end point dose is then given by

$$DED = K.D^n \qquad \dots (E1)$$

where D is the dose rate; K and n are empirical constants specific to the material tested. The constant n is usually in the range 0 to 0.3.



FIG. E.1. Interpolation of the end point dose (schematic).



FIG. E.2. Extrapolation of end point dose to lower dose rates (schematic).

# E.2.2. Limitations

This procedure can be a useful method for estimating the behaviour of some polymers at low dose. For all materials it must break down at dose rates low enough for thermal ageing to become dominant. On a log-log plot of DED versus dose rate used for extrapolation, the thermal ageing limit would be represented by a line of slope = 1, i.e. constant time conditions, whereas the slope of the extrapolated data is generally < 1. Extrapolation to dose rates within the thermally dominated region would give unrealistically high values for the predicted DED (as shown schematically in Figure E.3). This problem can be partially accounted for if separate thermal ageing data are available; these would allow determination of the appropriate thermal only result. If the additional data indicate that thermal effects will dominate, the thermal results can be used for predictions. The power law extrapolation method also cannot be used for materials which exhibit complex dose rate effects in the homogeneous oxidation region.

Although the power law extrapolation method assumes that homogeneous oxidation conditions have been obtained in all of the experiments, it appears to be useful in some materials at dose rates where heterogeneous oxidation would be expected to occur. This may arise because cracks generated in the thin oxidised layer can then propagate through the bulk unoxidised material, so that the observed macroscopic properties are determined by the degradation in the oxidised surface layer.

# E.2.3. Examples

Some examples of the use of the power law extrapolation method are given for several types of polyolefins, as follows:

# Polypropylene

This material was tested over a dose rate range of 4.45 to 2000 Gy/hr. In Figure E.4, plots of relative elongation at break  $e/e_o$  are shown for polypropylene monofilaments irradiated in air [E.10]. Using an end point criterion of  $e/e_o = 0.5$ , the values of the end point dose DED can be interpolated from these plots. These DED values are shown in Figure E.5; it can be seen that over this dose rate range there is a linear relationship between log(DED) and log(dose rate), with a slope of 0.26. For this material, homogeneous oxidation would have been obtained at all dose rates because of the thickness (0.4 mm) of the samples.

### Crosslinked polyethylene

For this XLPE cable material [E.10], tested over the dose rate range 100 to  $1.8 \times 10^5$  Gy/hr, data for  $e/e_0 = 0.5$  are shown in Figure E.6. This is an example where heterogeneous oxidation would have occurred at the two highest dose rates. However for this material, the slope of the plot remains the same at all of the dose rates tested.



FIG. E.3. Limitations – extrapolation of DED near thermal ageing limit.



FIG. E.4. Elongation at break of polypropylene, irradiated in air-from [E.9].



FIG. E.5. Extrapolation of end point dose from data in Fig. E.4.



FIG. E.6. Extrapolation of end point dose for XLPE – from [E.10].

#### E.3. SUPERPOSITION OF TIME DEPENDENT DATA

The second procedure which can be used to extrapolate to lower dose rates makes use of additional data obtained at elevated temperatures under irradiation. The method uses the superposition principle which has been used extensively for thermal ageing (time-temperature superposition) and here is expanded to time-temperature-dose rate superposition for combined thermal-radiation environments [E.11, E.12].

The superposition procedure in thermal ageing uses time dependent data taken under isothermal conditions at different temperatures. The higher temperatures are assumed to accelerate the degradation of properties in a uniform manner so that data can be shifted on the time axis to form a single curve at the reference temperature by the application of a multiplicative shift factor. In combined thermal-radiation ageing environments a similar procedure can be used to shift time dependent data obtained under constant temperature and constant dose rate; this is shown schematically in Figure E.7. By using elevated temperatures to accelerate degradation, data are obtained which are relevant to dose rates lower than those which are accessible experimentally.

In thermal ageing, the functional relationship between the shift factor b(T) and the temperature often takes a simple form, such as the Arrhenius relationship. The empirically derived activation energy from such a functional relationship represents an effective activation energy for the overall thermal degradation. For combined thermal-radiation ageing, the shift factors a(T,D) which are determined from the time dependent data do not always show a simple functional relationship with temperature and dose rate. In some cases an empirical relationship can be determined [E11, E.12], which enables extrapolation to lower dose rates to be carried out. Some examples of polymers where this procedure has been used are given in Section E.2.3.



log Ageing time

FIG. E.7. Superposition principle for combined thermal-radiation ageing (schematic).

#### E.3.1. Test procedure

The damage parameter used should reflect the use that is to be made of the material; for example, for a seal material, compression set would be an appropriate parameter whereas, for a cable material, elongation at break would be more suitable.

Data should be obtained at a minimum of three dose rates and at least two, preferably three, temperatures at each of these dose rates. For each of these temperature/dose rate conditions, measurements should be made at a minimum of three times. In addition, similar data on

unirradiated material should be obtained for at least three temperatures. This is the minimum data set for this method; more accurate assessment of the shift factors will be obtained if more data are available.

The first stage in the evaluation of the data is to superpose plots of the damage parameter versus log (time) obtained at constant temperature conditions on unirradiated material to yield a master curve (Figures E.8a and E.8b). The values of the shift factor at each temperature are determined and used to superpose the data. For ease of assessment, the reference temperature  $T_{ref}$  is usually chosen to be one of the temperatures used in the combined thermal-radiation ageing measurements.



log Ageing time

FIG. E.8a. Determining the shift factors b(T) for thermal ageing (schematic).



log [b(T). Ageing time]

FIG. E.8b. Superposition of data to form master curve (from Figure E.8a).

In the second stage of the evaluation, the time dependent data obtained under combined thermal-radiation ageing conditions are superposed on the master curve as shown in Figure E.9. The shift factors a(T,D), at temperature T and dose rate D, required to superpose the data are determined for each temperature-dose rate condition. At this stage of the evaluation, values of a(T,D) are known for the matrix of temperatures and dose rates used.

The following empirical relationship between the shift factor a(T,D) and the temperature and dose rate [E.4] can be used for a number of polymers.

$$a(T,D) = \exp -\underline{E} \left( \underline{1} - \underline{1} \right) \left\{ 1 + k.D^{x}.\exp \underline{Ex} \left( \underline{1} - \underline{1} \right) \right\} ...(E2)$$
  
R T T<sub>ref</sub> R T T<sub>ref</sub>

where T is the temperature in K,  $T_{ref}$  is the reference temperature in K, D is the dose rate and E, k and x are the model parameters. The parameter E is the value of the activation energy for thermal-only ageing. The parameters k and x are determined by fitting the values of a(T,D) obtained experimentally to the empirical equation (E2); at T =  $T_{ref}$ , the equation simplifies to

$$a(T_{ref}, D) = 1 + k.D^{x}$$
 ...(E3)

The parameter x usually takes the value  $x \le 1$ . This is shown schematically in Figure E.10; the parameter x is the limiting value of the slope of the plot at high dose rates. The parameter k determines the position of the curve on the dose rate axis.

Having determined the parameters E, k and x from the experimental data, the empirical model can then be used to calculate the DED at lower dose rates or temperatures. This can be determined using the equation,

$$DED = \underline{D.t_m} \qquad \dots (E4)$$
  
a(T,D)

where  $t_m$  is the time required to reach the selected damage level at the reference conditions of  $T = T_{ref}$  and D = 0, and a(T,D) is calculated from equation (E2). This is shown schematically in Figure E.11; the limiting slope of the log-log plot of DED versus dose rate is (1-x) at high dose rates .



FIG. E.9. Determination of shift factors a(T,D) for combined thermal-radiation ageing (schematic).



log Dose rate

FIG. E.10. Fitting experimental values of the shift factors a(T,D) to the model (equation E2).



log Dose rate

FIG. E.11. Calculated DED values using equation E3.

## E.3.2. Limitations

Despite its empirical nature, the general form of the superposition model (equation E2) has been found to be of practical use in radiation environments. Some examples of the use of the model are given in section E2.3.

This empirical model can only be used for those materials where the shape of the damage parameter versus log (time) curve does not change with temperature and dose rate. In practice, this limits its use to those materials where a single mechanism, e.g. oxidation, dominates both thermal and radiation degradation. If the curve shape changes, superposition of data is not possible and the method cannot be used. The procedure can satisfactorily model the change in DED as the material moves from the homogeneous region into the thermal degradation region at low dose rate. The procedure should not be used to extrapolate through a thermal transition of the polymer.

### E.3.3. Examples

#### EPDM elastomer:

This is a formulated elastomer used as a seal material, where compression set has been used as the damage parameter. This material exhibits little temperature dependence (for  $T < 90^{\circ}C$ ) under combined thermal-radiation ageing at dose rates in the range 35 to 1000 Gy/h [E.12]. Using the a(T,D) values obtained in this dose rate range to estimate the model parameters shows that at low dose rates, where thermal effects dominate, the superposition model predicts a marked temperature dependence. Long term tests at dose rates of 3.5 Gy/h indicate that the model can satisfactorily predict the behaviour of this seal material (Figure E.12).

Using the model to calculate the expected DED values as a function of dose rate shows that, for this material, DED is independent of dose rate over a wide dose rate range. By using data from tests carried out at elevated temperature, the equivalent degradation at 20°C at dose rates down to  $1.8 \times 10^{-3}$  Gy/h can be assessed (Figure E13). Note that, in this material, thermal effects dominate at the lowest dose rates emphasising the need for thermal ageing data when using the power law extrapolation method described in section E.1.



FIG. E.12. Experimental data for an EPDM elastomer fitted to the superposition model, equation E2.



FIG. E.13. Calculated DED values for 50% compression set of EPDM elastomer, compared with experimental data.



FIG. E.14. Calculated DED values for 50% compression set for a nitrile elastomer, compared with experimental data.

# Nitrile elastomer:

This material is also used as a seal but, unlike the EPDM elastomer of the previous example, it shows a marked temperature dependence under irradiation at dose rates in the region 100 to 1000 Gy/h [E.12]. The superposition model has been used to fit the data from this material

and can be used to calculate DED values as a function of dose rate (Figure E.14). In this nitrile elastomer, degradation at 40°C for dose rates < 350 Gy/h is dominated by thermal degradation as indicated by the line with a slope of one on the plot of DED versus dose rate in Figure E.14.

## Ethylene vinyl acetate polymer:

This is a dual layer EVA/EVA cable jacketing material which has been fitted to the empirical model. Unlike the previous two examples where the model parameter x took a value of 1, this material requires an x value of 0.7 to fit the data to the model [E.13]. A log/log plot of DED versus dose rate then shows a marked dose rate dependence even at high dose rates (Figure E.15); the slope of the plot at high dose rate will be (1-x).



FIG. E.15. Calculated DED values for  $e/e_0$  for an EVA cable jacket material.

# E.4. SUPERPOSITION OF DED DATA

This procedure also makes use of data obtained at elevated temperature under combined thermal-radiation ageing. Time-temperature-dose rate superposition in this case is applied to log-log plots of DED versus dose rate rather than damage parameter versus log (time). The shift factors obtained from superposition of DED versus dose rate data are often a simple function of temperature like the Arrhenius relationship [E.14] and can often be mechanistically rationalised in terms of the underlying oxidation mechanisms [E.15]. This procedure can be applied to a wide range of materials, including those for which superposition of time dependent data is inappropriate.

# E.4.1. Test procedure

Data at several dose rates and at least two temperatures are needed for superposition to be carried out. Sufficient data needs to be obtained at each temperature — dose rate condition for

the DED value to be determined; DED can be assessed for a number of damage levels. It is important to identify any temperature-dose rate conditions which give heterogeneous oxidation conditions.

For each temperature/dose rate condition the DED value is determined from a plot of the damage parameter versus dose as shown in Figure E1. These DED values are then plotted versus log (dose rate), excluding any data which is not homogeneously oxidised (Figure E16). The data points are shifted horizontally on the dose rate axis by using shift factors calculated from the Arrhenius relationship (see section 3.2.2 in Vol.1). The activation energy E is determined by trial and error until superposition of all of the data is obtained (Figure E.17). In some cases the empirical activation energy determined from the superposition process is found to be equal to that for thermal-only ageing. If a single value of E can be determined which will yield superposed data at different damage levels, then that value of E can be used to extrapolate to lower dose rates.



FIG. E.16. DED values under combined thermal-radiation conditions (schematic).



FIG. E.17. Superposition of DED data (schematic).

### E.4.2. Limitations

Although this procedure can satisfactorily be used to extrapolate data in the homogeneous region and in the thermally dominated region, it cannot be used if the temperature range of interest is at or near a thermal transition of the polymer.

The uncertainty in the value of the activation energy determined in this manner will be dependent on a number of factors. When dose rate effects are small, i.e. DED varies little with dose rate, there will be large uncertainties. At dose rates where thermal effects are dominant, the uncertainty in E is much less and will approach that for thermal-only degradation.

# E.4.3. Examples

Some examples of the use of superposition of DED data are given in the following sections.

#### *Neoprene cable jacket:*

Measurements of tensile elongation have been made on this material over a range of combined thermal-radiation environments [E.14]. The activation energy determined from superposition of DED data was found to be the same as that determined from thermal ageing. For this particular material, superposition occurs for both homogeneous and heterogeneous data (Figure E.18). It is suggested that this arises because, in heterogeneous samples, cracks which originate in the oxidised surface regions can propagate through the less oxidised bulk material away from the surfaces. This would make the elongation relatively insensitive to the homogeneity of oxidation in the samples. The superposed data in Figure E18 can be seen to asymptotically approach the line representing thermal-only degradation, indicating that the thermal environment dominates the degradation in this material at dose rates < 1 Gy/h at  $45^{\circ}$ C.



FIG. E.18. Superposition of DED data for a Neoprene cable jacket material (from ref. [E.14]).

#### PVC cable jacket:

This particular PVC cable jacketing material has been studied extensively under combined radiation-thermal conditions [E.14, E.15]. An activation energy of 96 kJ/mole yields excellent superposition of the data over a wide range of temperature and dose rate. The superposed data has an unusual S-shaped curve where dose rate effects tend to decrease at low dose rates (Figure E.19). This is typical of a complex dose rate dependence in the homogeneous oxidation region. In contrast to the two previous examples, the activation energy determined from superposition does not equal the thermal only activation energy which is 142 kJ/mole for this material. Detailed studies of the kinetics of degradation in this material have shown that the dose rate effects and enhanced degradation at elevated temperatures are due to the rate-determining breakdown of hydroperoxide species produced under irradiation [E.15, E.16]. The activation energy for this rate-determining step has been shown to be 96 kJ/mole i.e. identical to the empirical value found from superposition. The shape of the superposed curve in Figure E.19 is also consistent with the theoretical modelling of the detailed kinetics of the chemical reactions involved.



FIG. E.19. Superposition of DED data for a PVC cable jacket material with a complex dose rate dependence (from refs [E.15, E.16]).

#### E.5. PREDICTION OF LIFETIME OF RUBBER CABLE JACKET MATERIALS

Elongation at break is a parameter often used for the evaluation of condition and for lifetime prediction. Despite the fact that this parameter does not always show monotonic changes with ageing time, it possesses a high sensitivity to ageing for rubber cable insulation. In a study of thermal ageing of rubber cables [E.18], it was established that the main mechanism of ageing is a realignment of the crosslinked networks with changes in the correlation between physical and chemical nodes. Simultaneously, a degree of spatial heterogeneity of the network also changes bringing about changes in elasticity. Under the influence of radiation, the ageing process is complicated by the appearance of radiation-induced processes of scission and crosslinking. It is obvious that a full understanding of the mechanisms at the level of the basic kinetic equations is impractical, because there are too many unknown parameters.

In such a situation, it is reasonable to use semi-empirical models. They will reflect the main kinetics, but contain a limited number of parameters and are therefore applicable over a limited range of ageing conditions. The dependency of the parameters on the ageing factors should be assessed experimentally in each case, e.g. using Arrhenius law.

For rubber insulated cable, an empirical equation for changes in elongation under thermal and radiation ageing takes the form [E.19]:

$$ELB = \{b - (bd - ac)\{1 - \exp[-(c+d)t]\}/(c+d)\}\exp[-(e+f)\cdot t],$$
(E5)

where *a* and *b* are the values proportional to concentrations of "physical" and "chemical" nodes, respectively; *c* and *d* are the rate constants of direct  $(a \rightarrow b)$  and reverse  $(b \rightarrow a)$  reactions; *t* is the time; *e* and *f* are the components of the kinetic parameter for thermal and radiation degradation, *e* is also dependent on irradiation temperature. The parameter *f* depends on dose rate and also depends on temperature if synergistic effects are present.

In the general case, the parameters in equation (E5) shows the following dependence on degradation factors:

$$d = d_o \exp(-E_d / RT)$$
(E6)  

$$c = c_o \exp(-E_c / RT)$$
  

$$e = e_o \exp(-E_e / RT)$$
  

$$f = f_o P^n \exp(-S_f / RT),$$

where  $E_d$ ,  $E_c$ ,  $E_e$  are the effective activation energies of the processes, which are described by the appropriate parameters;  $exp(-S_f/RT)$  is a coefficient accounting for synergistic effects;  $d_0$ ,  $c_0$ ,  $e_0$  and  $f_0$  are the pre-exponents of the parameters; P is the dose rate; n is an empirical index, R is the gas constant, T is the temperature.

In the presence of synergistic effects,  $S_f > 0$ ; in the absence of synergism,  $S_f = 0$ . If ageing is retarded in simultaneous exposure to heat and radiation,  $S_f < 0$ . The experience of using equation (E6) has shown that the kinetic parameter *c*, specifying the transformation of "physical" nodes to "chemical" ones, is independent of temperature.

The application of equation (E5) is only feasible if every dependence in equation (E6) can be experimentally proved. Examples of applying equation (E5) to a cable with EPR jackets (produced by Filergie Company) as defined by the data from [E.20] are presented in Figure E.20 and Table E.1.

TABLE E.1. SERVICE LIFE (YEARS) OF A CABLE WITH EPR JACKET (PRODUCED BY FILERGIE COMPANY).

Dose rate	Temperature, °C					
Gy/hour	40	45	50	55	60	70
0,1	271	156	91,5	54	33	12,6
1	200	137	86	53	32,5	12,5



FIG. E.20. Kinetics of change in elongation at break of cable jacket samples made of EPR during thermal radiation ageing.

Table E.1 presents the results of service life prediction regarding a cable with EPR jacket (produced by Filergie Company) at the reliability level of 0.95. Reduction in the value of ELB equal to 50% was defined as a cable failure.

Equation (E5) can be used for the determination of residual lifetime, when service conditions are known, but the initial value of ELB of the material is unknown. In this case, the task is solved as a prediction task, i.e. samples removed from service are subjected to accelerated testing, these results being applied to assessment of residual lifetime.

It is obvious, that the use of the entire programme of accelerated tests required for determination of the parameters in equation (E5) is not practical. The task is greatly simplified, if evaluation is conducted at sufficiently advanced stages of ageing. If t >> 1/(c + d), equation (1) is transformed to a simple exponential model:

$$ELB = g \cdot \exp[-(e+f) \cdot t], \tag{E7}$$

where g = (a+b)c/(c+d), which is dependent on the temperature only. If testing is carried out at one temperature (the operating temperature), then the parameter *e* may be assumed equal to zero. As a minimum, it is possible to use two dose rates.

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