# SKI Report 97:40

# Aging of Electrical Components in Nuclear Power Plants

Relationships Between Mechanical and Chemical Degradation After Artificial Aging and Dielectric Behaviour During LOCA

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Relationships Between Mechanical and Chemical Degradation After Artificial Aging and Dielectric Behaviour During LOCA

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October 1997

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This report concerns a study which has been conducted for the Swedish Nuclear Power Inspectorate (SKI). The conclusions and viewpoints presented in the report are those of the author and do not necessarily coincide with those of the SKI.

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Norstedts Tryckeri AB Stockholm 1997

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# Summary

The purpose of the study reported in SKI Technical Report 93:39 was to evaluate qualification methods for application to components intended to be installed in the containment of nuclear power plants. The study included application of Arrhenius' criterion for thermal aging as well as methodologies for updating (curve-matching, on-going qualification). This report reviews the results of complementary tests, including extended thermal aging at high temperatures and LOCA (Loss of Coolant Accident) testing of a selection of components which were aged in the program reported in SKI 93:39.

The purpose of the extended thermal aging has been to achieve some complementary points on the aging degradation curves and to ensure that the LOCA tests include some severely degraded components. The purpose of the LOCA tests is primarily to validate the suitability of using mechanical properties as indicators of the effect of aging by evaluating the correlation between the mechanical behavior after aging and the dielectric behavior at LOCA.

In addition, the complementary study also included measurement of chemical degradation by use of Oxygen Induction Time/Temperature (OIT/OITe) and microcalorimetric methods.

The complementary thermal aging was performed on cables type Lipalon, Dätwyler and Rockbestos and on o-rings type Viton and EPDM. Complete cables as well as separate insulated conductors (cable leads) were included. The conditioning included 144 and 192 days at 120°C and 24 and 48 days at 142°C. This represents very extreme conditions for the purpose of the study, in excess of what is used for qualification of cables for installation in nuclear power plants. A few solenoid coils of type Seitz 2A25 were also subjected to extended aging.

The mechanical degradation due to the extended aging of the complete cables, mainly representative of the cable jacket materials, and of the separately tested leads was determined by indenter measurements. The mechanical degradation of the cable jacket material was also determined by elongation-at-break measurements. The chlorosulphonated polyethylene (CSPE, Hypalon) insulation materials, included in the jacket material of the Rockbestos cable and the jacket and leads of the Lipalon cable, show very pronounced mechanical degradation as result of the additional conditioning. The EPDM insulation materials, included in the jacket and leads of the Dätwyler cable, are less affected. The crosslinked polyethylene insulation materials, included in the lead insulation of the Rockbestos cable, are only slightly affected.

The mechanical degradation of the o-rings due to the extended thermal aging was determined by indenter measurements on samples which were lying loose during the conditioning, and by measurement of remaining compression of samples which were placed in a special compression fixture during the conditioning. The EPDM o-rings were significantly affected by the prolonged exposure, whilst the Viton o-rings (vinylidene fluoride-hexafluoropropylene copolymer, FPM) were very little affected

The dielectric degradation of the cables and of the solenoid coils was determined by measurement of insulation resistance and capacitance and of dielectric loss factor. The earlier aging tests, reported in SKI 93:39, were performed under conditions which did not result in severe degradation of the insulation properties of the cables. The extended aging resulted in a pronounced degradation in the dielectric behavior, which is shown as significant increase in the dielectric loss factors and, in the most severe cases, in significant reduction of the insulation resistance as well as increase of the capacitance.

The use of nitrogen atmosphere during the thermal aging had not shown any significant reduction of the degradation compared to the use of air in the earlier tests, reported in SKI 93:39. An extended aging for 24 and 48 days at 142°C in nitrogen atmosphere was included in the complementary program. In this case a very significant reduction of the degradation is shown for the Lipalon and Rockbestos cables jacket materials and for the Lipalon cable lead material. For the Dätwyler cable jacket and lead materials the reduction becomes significant only after the longest exposure (48 days). For the Rockbestos cable lead (XLPE, crosslinked), the reduction is insignificant. For the EPDM o-rings the reduction was very significant whilst it was insignificant for the Viton o-rings.

The LOCA test was performed in ABB test facility LOKE, subjecting the components to overheated steam for 3 hours at 181°C and 4 BarG, followed by 160°C and 4 BarG for 3 hours and 120°C for 44 hours. The components tested included cable samples, which had been thermally aged for various duration at 95, 120 and 142°C, for the Lipalon cables also at 80°C. Cables which had been subjected to high humidity or vibration and thermal aging were included. The cable samples were subjected to ionizing radiation at 10<sup>4</sup>Gy/h for 50 hours before LOCA testing (total dose 500 kGy). Separate insulated Lipalon cable leads, which had earlier been subjected to vibration and thermal aging, and some solenoids type Seitz 2A25, were subjected to the ionizing radiation, but not to LOCA.

The effects of the radiation on the Lipalon and Dätwyler cable jackets are significant. Also the dielectric loss factors were affected for the Lipalon cables. This was the case also for the separately exposed insulated cable leads and for the solenoids.

The measurements during LOCA testing included insulation resistance and dielectric loss factor. The comparison between the values of maximum loss factors during LOCA and the indenter values before LOCA of the Lipalon cables shows a positive but rather low correlation (correlation factor approximately 0,5). Thus, it is shown by the results that the indenter values of the Lipalon cables, measured on the complete cable, are reasonably related to the dielectric behavior during LOCA.

The Dätwyler cables show no consistent relationships between mechanical condition before LOCA testing and the dielectric parameter values during LOCA. For the

Rockbestos cables there is a rather high correlation between the mechanical degradation before LOCA (in terms of indenter modulus increase) and the dielectric loss factors after LOCA (correlation factor 0,85). The loss factor couldn't be measured during LOCA for the most severily thermally aged Rockbestos cables.

Lipalon cables which were subjected to high humidity at the thermal aging show a significant reduction of insulation resistance during LOCA compared to cables which had been subjected to the same thermal aging in dry atmosphere.

The results also show that the cables which were subjected to intermittent vibration during the thermal aging indicated significantly lower insulation resistance, higher capacitance and higher loss factor at LOCA than the cables which had been subjected to the same thermal aging without vibration. The vibration levels used in the study were in excess of those occurring in normal installations in nuclear power plants.

The earlier tests, reported in SKI 93:39, had shown that a simulation of a rather long duration thermal aging at 80°C by a simulated on-going procedure gave more realistic results than simulation by a short duration thermal aging at a more elevated temperature. These results were confirmed by the results of the LOCA tests on the Lipalon cable samples used in this part of the study.

The OIT/OITe measurements were applied to unaged and aged insulation materials of the jackets and the conductor insulation of Lipalon, Dätwyler and Rockbestos cables. Microcalorimetry measurements have been applied to the conductor insulation materials. The results show that the two methods are complementary. The OIT technique can be used for studying the aging process in the materials stabilized by antioxidants (EPDM and XLPE). Microcalorimetry can be used for studying the aging process in the materials not containing any detectable antioxidants (CSPE).

It is also interesting to note that the OIT measurements were suitable for the materials which showed little or no changes in indenter values with thermal aging.

A more detailed study of OIT of the cable conductor insulation was made as a complement. A clear reduction of induction time with increased thermal aging was shown. A significant relationship between the induction times of the conductor insulation materials measured and the dielectric behavior of identical cables during LOCA could not be shown.

The study has been performed in cooperation between Ingemansson Technology AB and ABB Atom. Ingemansson has been responsible for the planning, including the analyses and interpretations of the results. The preparation of the components, the conditioning and the measurements of dielectric parameters and elongation-at-break have been the responsibility of ABB Atom. ABB Atom has also been responsible for the OIT and microcalorimetry evaluations. Ingemansson has been responsible for the indenter measurements. The study has been sponsored by Statens Kärnkraftinspektion, Forsmark Kraftgrupp AB, OKG Aktiebolag, Vattenfall AB Ringhals and Barsebäck Kraft AB. It has been governed by a steering committee, including

Tommy Appelqvist, Statens Kärnkraftinspektion Rainer Cebulla, ABB Atom Reinhold Delwall, Forsmarks Kraftgrupp AB Karel Fors, Barsebäck Kraft AB Einar Ströbäck, Vattenfall AB Ringhals Lars-Olof Ståhle, OKG AB, Oskarshamnsverken

Gunnar Ståhl, responsible for the work at ABB Atom, and Kjell Spång, responsible for the work at Ingemansson Technology AB, have also been members of the steering committee.

# Sammanfattning (summary in Swedish)

Syftet med den studie som rapporterats i SKI Technical Report 93:39 var att utvärdera metoder för kvalificering av komponenter för installation i reaktorinneslutningar. Studien inkluderade applicering av Arrhenius kriterium för termisk åldring samt metodik för uppdatering (kurvmatchning, fortlöpande kvalificering). Denna rapport redovisar resultaten av kompletterande prov, omfattande utökad termisk åldring vid höga temperaturer samt LOCA-provning (Loss of Coolant Accident) av ett urval av komponenter som åldrats enligt det program som rapporterats i SKI 93:39.

Avsikten med den utökade termiska åldringen har varit att erhålla några kompletterande punkter på kurvorna över degradering på grund av åldring och att försäkra oss om att LOCA-provningen omfattar ett antal svårt degraderade komponenter. Syftet med LOCA-provningen är i första hand att värdera lämpligheten av att använda förändringar i mekaniska egenskaper som indikatorer på effekten av åldring genom att undersöka korrelationen mellan mekaniska egenskaper efter åldring och dielektriska egenskaper under LOCA.

Den kompletterande studien har även omfattat mätning av kemiska egenskaper med hjälp av Oxygen Induction Time/Temperature (OIT/OITe) och med mikrokalorimetrisk metod.

Den kompletterande termiska åldringen genomfördes på kablar typ Lipalon, Dätwyler och Rockbestos och på o-ringar typ Viton och EPDM. Kompletta kablar och separata ledare (inkl ledarisolering) ingick. Konditioneringen omfattade 144 och 192 dagar vid 120°C samt 24 och 48 dagar vid 142°C. Detta representerar mycket extrema förhållanden för studiens ändamål, långt utöver de som används för kvalificering av kablar som installeras i kärnkraftverk. Några solenoidspolar av typ Seitz 2A25 utsattes också för utökad åldring.

Mekanisk degradering på grund av utökad åldring av kompletta kablar, i huvudsak representativ för mantelmaterialet, samt av separata ledare bestämdes med indentermätningar. Mekanisk degradering av mantelmaterialet bestämdes dessutom med brottöjningsmätningar. Hypalon (CSPE), som ingår i mantelmaterialet till Rockbestoskabeln och i såväl mantel- som ledarisoleringen till Lipalonkabeln, uppvisar en mycket uttalad degradering som resultat av den utökade åldringen. EPDM, som ingår i mantel- och ledarisolering till Dätwylerkabeln, påverkas mindre. XLPE, (tvärbunden polyetylen), som ingår i ledarisoleringen till Rockbestoskabeln, påverkas endast i liten grad.

Mekanisk degradering på grund av utökad åldring av o-ringarna bestämdes med indentermätning på exemplar som förvarades lösa under konditioneringen samt genom uppmätning av kvarstående deformation på exemplar som var monterade och utsatta för kompression i en speciell fixtur under konditioneringen. O-ringarna av EPDM påverkades signifikant av den utökade exponeringen, medan o-ringarna av Viton påverkades endast i ringa grad.

Dielektrisk degradering av kablar och solenoidspolar bestämdes genom mätning av isolationsresistanser, kapacitanser och dielektriska förlustfaktorer. Tidigare åldringsprov, rapporterade i SKI 93:39, genomfördes med en exponering som inte resulterade i någon allvarlig nedsättning i dielektriska egenskaper. Den utökade exponeringen resulterade i en påtaglig degradering av de dielektriska egenskaperna, vilket visar sig i en signifikant ökning av dielektriska förlustfaktorer och, i de svåraste fallen, i såväl signifikant reduktion av isolationsresistanserna som ökning av kapacitanserna.

Användning av kväve under den termiska åldringen hade ej visat någon signifikant minskning i degraderingen jämfört med luft vid de i SKI 93:39 redovisade proven. En utökad exponering under 24 och 48 dagar vid 142°C i kväveatmosfär inkluderades i det kompletterande programmet. I detta fall erhölls en mycket påtaglig minskning av degraderingen för mantelmaterialet till Lipalon- och Rockbestoskabeln samt för ledarisoleringen till Lipalonkabeln. För Dätwylerkabelns mantel- och ledarisolering blev minskningen signifikant endast efter den längre exponeringen (48 dagar). För Rockbestoskabelns ledarisolering erhölls ingen signifikant minskning. För o-ringarna av EPDM erhölls en mycket påtaglig minskning av degraderingen, medan den ej var signifikant för o-ringarna av Viton.

LOCA-provningen genomfördes i ABB provutrustning LOKE. Komponenterna utsattes för överhettad ånga under 3 timmar vid 181°C och 0.4 MPa, följt av 160°C och 0.4 MPa under 3 timmar och 120°C vid normaltryck under 44 timmar. Provningen omfattade kabelbitar som hade åldrats termiskt med olika varaktigheter vid 95, 120 och 142°C, för Lipalonkabeln även vid 80°C. Kablar som varit utsatta för hög fuktighet eller vibration tillsammans med den termiska åldringen var även inkluderade. Kabelbitarna utsattes för joniserande strålning vid 10<sup>4</sup>Gy/h under 50 timmar före LOCA (totaldos 500 kGy). Separata ledare till Lipalonkabel, som tidigare utsatts för vibration och termisk åldring, samt några solenoidspolar (Seitz 2A25), utsattes för joniserande strålning men ej för LOCA.

Effekten på mekaniska egenskaper av joniserande strålning på Lipalon- och Dätwylerkablarna är signifikant. För Lipalonkablarna påverkades även dielektriska förlustfaktorer. Detta gällde även separat exponerade ledare samt solenoidspolarna.

Mätningarna under LOCA inkluderade isolationsresistanser och dielektriska förlustfaktorer. För Lipalonkabeln är sambandet mellan värden på maximal förlustfaktor under LOKA och indentervärden före LOCA positivt men korrelationen är relativt låg (korrelationsfaktor ca 0,5). Resultaten visar att indentervärdet för Lipalonkabel, mätt på komplett kabel, är påtagligt men ej starkt relaterat till dielektriskt uppträdande under LOCA. Dätwylerkablarna uppvisar inget konsistent samband mellan mekaniskt tillstånd före LOCA och dielektriska värden under LOCA. För Rockbestoskablarna finns en relativt hög korrelation mellan mekanisk degradering före LOCA (ökning av indentervärde) och dielektrisk förlustfaktor efter LOCA. Det var ej möjligt att mäta förlustfaktorn under LOCA hos de hårdast termiskt åldrade Rockbestos-kablarna.

Lipalonkablar som varit utsatta för hög fuktighet i samband med den termiska åldringen visar en signifikant minskning av isolationsresistans under LOCA jämfört med kablar som utsatts för samma termiska åldring i torr luft.

Kablar som utsatts för intermittent vibration under den termiska åldringen visar signifikant lägre isolationsresistans, högre kapacitans och högre förlustfaktor under LOCA än kablar som utsatts för samma termiska åldring utan vibration. De vibrationsnivåer som använts i studien är högre än de som uppträder i normala kärnkraftinstallationer.

Tidigare prov, rapporterade i SKI 93:39 visade att en simulering av en tämligen långvarig exponering vid 80°C med fortlöpande kvalificering gav mer realistiska resultat än simulering med en kortvarig termisk åldring vid en mer förhöjd temperatur. dessa resultat styrks av resultaten av LOCA-proven på Lipalonkablar som användes för denna del av studien.

OIT/OITe mätningarna genomfördes för oåldrat och åldrat isolermaterial till mantel och ledare hos Lipalon-, Rockbestos- och Dätwylerkabeln. Mikrokalorimetrimätningarna genomfördes för materialet i ledarisoleringarna. Resultaten visar att de två metoderna kompletterar varandra. OIT-tekniken kan användas på material som stabiliserats med antioxidanter (EPDM, XLPE). Mikrokalorimetri kan användas till att studera åldringsprocesser i material som ej innehåller upptäckbara mängder av antioxidanter (CSPE).

Det är också intressant att notera att OIT-mätningarna visade sig lämpliga på material som uppvisade ringa eller inga förändringar i indentervärdena efter åldring.

En mer detaljerad studie av OIT på ledarisoleringarna genomfördes som komplement. En tydlig reduktion av induktionstiden vid ökad termisk åldring uppvisades. Ett signifikant förhållande mellan induktionstid hos ledarisolering och dielektriska egenskaper under LOCA kunde dock ej påvisas.

Studien har genomförts i samarbete mellan Ingemansson Technology AB och ABB Atom. Ingemansson Har ansvarat för planering, inklusive analys och tolkning av provresultat. Preparering av komponenterna, åldring och mätning av dielektriska parametrar och brottöjning har genomförts av ABB Atom. ABB Atom har också svarat för OIT- studierna och studierna med mikrokalorimetri. Ingemansson har ansvarat för indentermätningarna. Studien har bekostats av Statens Kärnkraftinspektion, Forsmark Kraftgrupp AB, OKG Aktiebolag, Vattenfall AB Ringhals och Barsebäck Kraft AB. Till projektet har varit knuten en Styrgrupp, bestående av

Tommy Appelqvist, Statens Kärnkraftinspektion Rainer Cebulla, ABB Atom Reinhold Delwall, Forsmarks Kraftgrupp AB Karel Fors, Barsebäck Kraft AB Einar Ströbäck, Vattenfall AB Ringhals Lars-Olof Ståhle, OKG AB, Oskarshamnsverken

Gunnar Ståhl, ansvarig för arbetet vid ABB Atom, och Kjell Spång, ansvarig för arbetet vid Ingemansson, har även ingått i Styrgruppen

# **1** Background

This report reviews the results of tests, complementary to the study reported in SKI Technical Report 93:39 (ref. /1/).

The purpose of the study reported in ref. /1/ was to evaluate the quality of methods used in qualification of components, including application of Arrhenius' criteria for thermal aging and updating (curve matching, on-going qualification) as a method to increase the realism of the testing. The study included application of different aging programs to more than one thousand component samples: cables, o-rings and solenoids.

The complementary study reported here includes:

- A. Extended thermal aging at the two highest temperatures (120 °C and 142 °C) used in the study reported in ref. /1/.
- B. LOCA testing on a selection of components which were aged in the program reported in ref. /1/.

The purpose of A is to get some complementary points on the aging curve, related to very severe degradation of the components. It shall also ensure that the LOCA tests include testing of severely degraded components.

The purpose of B is to verify the influence of the aging on the dielectric characteristics of the components during and after a LOCA event. The aims are to correlate the degradation caused by aging, measured in terms of changes in mechanical parameters (indenter value, elongation-at-break, dimensional changes), with dielectric parameter values (insulation resistance IR, dielectric loss factors etc.) during LOCA. This knowledge is important for the validation of the method of using mechanical properties as indicators of aging of electrical components.

In addition to the methods for measurement of aging characteristics of the component materials reported in ref. /1/, the complementary study also includes use of oxygen induction time (OIT) and microcalorimetric methods for determination of aging related properties of the component materials. The results of these studies are reported in details in Appendix A03 (in Swedish).

# **2** Components

## 2.1 Cables

The cables include Lipalon type FSSR7x1, Dätwyler type FEAR-PG 8x(2x1) and Rockbestos Firewall 3, RXSR-G 5x1.

The Lipalon cable has a conductor insulation and jacket of CSPE (chlorosulphonated polyethylene, Hypalon). The Dätwyler cable has a conductor insulation of black EPDM (ethylene-propylene rubber). The jacket material is blue EPDM. The Rockbestos cable

has a conductor insulation of XLPE (crosslinked polyethylene) The jacket material is CSPE.

Analysis and discussion of the cable materials were part of the OIT and microcalorimetric studies and is reported in Appendix A03 (in Swedish).

The cables and leads used for LOCA testing were selected out of the sealed cables and leads used in the testing reported in ref. /1/. They are referred to as kab d, kabvib and ledvib. They had been treated in the ends to avoid humidity intrusion from the ends and the leads were fitted with pins in one end to allow dielectric measurements, see Figure 2.1 below. The other end was sealed by means of a sealing compound. Kab d had a length of 3 m and were wound on bobbins.



Figure 2.1 Treatment of lead ends

Additional cable pieces for the aging tests (not including LOCA), were of 0,3 m length and not sealed in the ends. They were cut out from unaged cables which had been stored in climatised conditions since the performance of the earlier tests, approximately three years. They are referred to as kab m and led m.

## 2.2 O-rings

The o-rings included two types, Viton and EPDM. Both types had a diameter of 37,47 mm and a thickness of 5,34 mm. The thickness tolerance was  $\pm 0,13$  mm.

The Viton o-rings (vinylidene fluoride-hexafluoropropylene copolymer, FPM) have a hardness of 82°IRH, a tensile strength of 11 MPa and a maximum elongation (unaged) of 190%.

The EPDM o-rings have a hardness of 74°IRH, a tensile strength of 11 MPa and a maximum elongation (unaged) of 150%.

The o-rings marked Or m were mounted in a specific fixture for the test, imposing a load (compression) similar to that of normal operation. The fixture design and the mounting is shown in Figure 2.2. The compression of the o-rings when mounted in the fixture was 1,34 mm.

The o-rings marked Or e were aged under non-mounted conditions.



Figure 2.2 Fixtures used for compression of o-rings

# 2.3 Solenoids

Coils of solenoids type Seitz 2A25, 110 VDC were used in some of the tests. The connection cables were of type Rockbestos.

# **3 Testing equipment**

# 3.1 Thermal aging

The thermal aging was performed in dry heat test chambers (Heraeus), fulfilling the requirements for testing according to IEC Publication 68-2-2, test Bb: Dry Heat Testing of Non Heat-Dissipating Specimen. The variation of temperature in time was kept within  $\pm 0.5^{\circ}$ C and the spatial temperature variation was kept within  $\pm 3^{\circ}$ C.

# 3.2 Aging in nitrogen atmosphere

One of the dry heat test chambers was gas tight, used for the aging in nitrogen atmosphere. The nitrogen was fed to the chamber in the opposite side of the outlet in order to achieve a crosstreaming in the chamber. The  $N_2$  flow was regulated so that the oxygen content indicated at the outflow was << 1%. The setup is shown in Figure 3.1.



Figure 3.1 Testing in nitrogen atmosphere

#### 3.3 Ionizing radiation

The irradiation was performed in materials testing reactor R2 at Studsvik. The radiation source was spent reactor fuel elements. Ambient and material temperatures during irradiation were 35°C and 45°C. The dose rate was 10 kGy/h = 1 Mrad/h. The testing continued for 50 h; total dose 500 kGy = 50 Mrad.

The test specimens were encapsulated in plastics and sealed in a container, which was tilted 180° after 25 h. The monitoring method used was Reuter-Stokes Gamma Ionization Chamber RSG - 12A.

## 3.4 Humidity aging and vibration

Some of the test specimens had been humidity aged at the earlier tests, reported in /1/. This had been made in conditions according to IEC Publication 68-2-3 Test Ca: Damp Heat, Steady State, with a relative humidity maintained at above 95% in a temperature of  $95 \pm 2^{\circ}$ C for 24 and 48 days.

Cables marked kabvib and leads marked ledvib had been subjected to sinusoidal vibrations at the earlier tests, reported in /1/. The thermal conditioning had been interrupted once per week by sweep sinusoidal vibration testing between 2 and 200 Hz with a sweep rate of one octave per minute during 30 minutes. Two different severities were used: amplitude  $3,5 \text{ mm } 30 \text{ m/s}^2$  and amplitude  $7 \text{ mm } 60 \text{ m/s}^2$ . The cables and leads were mounted in a fixture as illustrated in Figure 3.2, which was fixed to the table of the electrodynamic vibrator used (Derritron).



Figure 3.2 Fixture used for vibration testing of cables and leads

## 3.5 LOCA testing

The device used for the LOCA testing is described in Appendix A01

# 4 Measurement parameters and methodology

## 4.1 Dielectric measurements

The dielectric measurements included measurement of insulation resistance (IR), capacitance and dielectric loss factor. Manual measurements were made before and after LOCA testing. Continuous registration of dielectric loss factor and insulation

resistance was used during the LOCA. In addition, intermittent manual measurement of insulation resistance was made during LOCA.

In order to achieve a more sensitive detection of changes, the test objects were subjected to a humidity conditioning before the dielectric values were measured. The humidity conditioning included 108h at a temperature of  $55^{\circ}C\pm 2^{\circ}C$  and a relative humidity of  $93\%\pm 3\%$ .

The loss factor measurements and the measurements of capacitance were made at 20Hz, 60Hz, 1kHz and 10kHz (5V). The measurements of insulation resistance before and after LOCA testing were made with 250V DC (1Mohm serial resistance). The measurements during LOCA were made with 50V, because of the highly degraded test objects. Figure 4.1 shows the set-up for manual insulation resistance measurements. Appendix A02 reports the measurement equipment used during the LOCA test.



Figure 4.1 Device used for manual measurement of insulation resistance

## 4.2 Mechanical measurements

The mechanical measurements included elongation at break and indenter values, for orings also deformation. The elongation at break measurements were made in accordance with IEC Publication 540. Test items of the shape in Figure 4.2 were cut out from the cable jackets and subjected to elongation in a tensile testing machine. The elongation at break was measured on aged test specimen (e) and on unaged specimen ( $e_o$ ). The results are presented in terms of the ratio ( $e/e_o$ ).



Pulling speed 25 mm/min Fixing points 50 mm apart

Figure 4.2 Test specimen used for elongation at break measurement

The remaining deformations of the o-rings marked Or m (mounted in the compression fixture during thermal aging) were measured after the thermal aging. The values reported are the ratio between the thickness of the o-rings after demounting from the fixture and the thickness before aging.

The principle of indenter measurement is described in ref. /2/. The force needed for pressing an anvil of a certain shape into the material with constant velocity is measured. The slope of the force versus the intrusion depth is defined as the indenter modulus.

The instrument and software used are described in ref. /3/. The speed used for the intrusion of the anvil into the material is with this instrument 0.5 mm/s.

In the earlier report /1/ the results of indenter measurements were presented in terms of indenter ratios, defined as the ratio between the indenter modulus of the aged specimen and the indenter modulus of the unaged specimen. This presentation is also mainly used in this report. Other users of indenter measurements often present their results in terms of indenter modulus (in N/mm).

Table 4.1 shows the modulus values of unaged samples. The values are mean values from measurements on a rather large (>10) number of samples. The deviations between values of different unaged samples were very small. The modulus values of the aged samples can be achieved by multiplying the ratios given in this report by the modulus values given in Table 4.1.

Component	Indenter modulus before aging, N/mm
Cable Lipalon	3,23
Lead Lipalon	1,04
Cable Dätwyler	3,38
Lead Dätwyler	1,28
Cable Rockbestos	1,50
Lead Rockbestos	19,2
O-ring Viton	1,42
O-ring EPDM	1,67

Table 4.1 Indenter modulus of unaged samples

The slope of the force versus intrusion is not always linear. It is then important where on the slope the indenter value is determined. For the determination of the modulus we have in this study normally selected the first part of the curve, until the anvil has travelled 2,5 mm. The maximum force was normally set below 15N. In cases of severely degraded cables or leads, the force set was reached before the anvil had traveled 2,5mm. In such cases the determination of the modulus has been based on the first part of the curve, before the slope becomes nonlinear.

A special comparative study of measurements with the indenter described in ref. /3/ and an indenter manufactured by Ogden, which is used by other researchers, was performed

on a broad range of differently aged cables and leads. The results are presented in Ingemansson Report H-14008-B, ref. /4/ (in Swedish). The correlation is high between the results from the measurements with the two indenters. The Ogden indenter is suitable for measurements in field conditions and is superior to our "old" indenter for measurements on leads (small dimensions).

Measurements of indenter values on cable jackets were made in three points on the periphery of two different sections of the cable sample, see Figure 4.3. The values presented are the mean of these six values. Measurements of indenter values on leads were made in two points on each lead sample. The small diameters and the irregular circumferences of the leads made is rather difficult to achieve representative values.



Figure 4.3 Measurement points on cable jacket

# **5** Complementary thermal aging

# 5.1 Components and measurement parameters

Table 5.1 summarizes the components and measurement parameters included in the test program for complementary thermal aging In total, 228 component samples were used for this part of the study.

Component	Marking	Measurement parameters	Number of
			components
Cable, 3m length, sealed	kab d	Dielectric loss factor, Insulation	3x8=24
Lipalon, Dätwyler, Rockbestos		resistance, capacitance, indenter value	
Cable, 30cm length, unsealed	kab m	Elongation-at-break, indenter value	3x20=60
Lipaion, Datwyler, Rockbestos			
Lead, 1m length, unsealed	led m	Indenter value	3x20=60
Lipalon, Dätwyler, Rockbestos			
O-ring unmounted	Ore	Indenter value	2x20=40
Viton, EPDM		indenter value	2720 40
O-ring, mounted	Or m	Deformation (permanent)	2x20=40
VIUI, EPDN			
Solenoid, Seitz 2A25, 110 VDC	sol	Loss factor, Insulation resistance,	4
		capacitance, coil resistance	

Table 5.1 Complementary thermal aging. Components and measures

#### 5.2 Complementary testing at very severe thermal aging conditions

The results of the earlier study, reported in /1/, indicate that the activation energy, calculated from the degradation of the components as functions of temperature and duration for the thermal aging, varies with the severity of the aging test and resulting level of degradation. It has therefore been considered to be important to determine the activation energy within a range of severity of the aging which results in a degradation of the test specimen that leads to unacceptable dielectric behavior during LOCA.

The following complementary aging programs were applied:

- Thermal aging at 120°C was made in the earlier study up to 96 days. This was complemented by additional aging tests for 144 days and 192 days
- Thermal aging at 142°C was made in the earlier study up to 12 days. This was complemented by additional aging tests for 24 days and 48 days

For the test specimen which had not been used in the earlier study (kab m and o-rings) the testing also included aging at 120°C for 48 and 96 days and at 142°C for 6 and 12 days for completeness.

The test program is shown in table 5.2. The samples have been identified by group number, referring to the thermal aging applied. Each group has normally included two identical samples, which have been identically aged. The group numbers are shown in the table.

The cable samples belonging to groups 1ba and 1bb had been thermally aged in the earlier study at 120°C for 48 days. They were now subjected to additional aging at 120°C for 96 days and 144 days, resulting in the total thermal aging time of 144 and 192 days.

The cable samples belonging to groups 8a and 10a had been thermally aged in the earlier study at 142°C for 3 days and 9 days. They were now subjected to additional aging at 142°C for 21 days and 39 days, resulting in the total thermal aging time of 24 and 48 days.

Conditioning/ measurement	Gro			roup of samples, No					Number of samples in each group			in						
	1ba	1bb	8a	10a	43	44	45	46	47	48	49	50	kab d	kab m	led m	Or m	Ore	Sol
	(kat	o d, so	l, led r	n)	(un	aged	kab m	1, o-ri	ngs)									
Indenter value					x	x	x	x	x	x	x	x	-	2	2	-	2	-
Deformation					x	x	x	x	x	x	x	x	-	-	-	2	-	-
Aging 120 <sup>o</sup> C																		
48d					x								-	2	-	2	2	-
96d	x					x							2	2	2	2	2	-
144d		x					x						2	2	2	2	2	-
192d								x					-	2	-	2	2	-
Aging 142°C																		
6d									x				-	2	-	2	2	-
12d										x			-	2	-	2	2	-
21d			x										2	-	2		-	1
24d											x		-	2	-	2	2	-
39d				x									2	-	2	-	-	-
48d												x	-	2	-	2	2	1
IR	x	x	x	x									2	-	-	-	-	-
Loss factor	x	x	x	x									2	-	-	-	-	1
Coil resistance	x	x	x	x									-	-	-		-	1
Indenter value	x	x	x	x	x	x	x	x	x	x	x	x	-	2	2	-	2	-
e/e <sub>0</sub>					x	x	x	x	x	x	x	x	-	2	-	-	-	-
Deformation					x	x	x	x	x	x	x	x	-	-	-	2	-	-

## Table 5.2 Test program for complementary thermal aging tests

The sealed cables (kab d) were unwound from the bobbins and rewound on the bobbins before the humidification for the dielectric measurements.

## 5.3 Test Results

## 5.3.1 Cables, influence of thermal aging on indenter ratios and $e/e_0$

## 5.3.1.1 Unsealed cable samples

The indenter values and elongation-at-break values were determined on the unsealed cables (kab m) before and after thermal aging.

The results of the measurements on the unsealed cable samples (kab m) are summarized in Table 5.3. The values given are the ratios between the values of the aged and unaged cable samples. Each value given represents the mean of two identically aged cables.

Temp	Duration	Cable Lipalon		Cable Lipalon Cable Dätwyler		Cable Ro	ckbestos
°C	days	ind ratio	e/e <sub>0</sub>	ind ratio	e/e <sub>0</sub>	ind ratio	e/e <sub>0</sub>
120	48	1,498	0,52	1,737	0,71	1,838	0,28
120	96	3,650	0,18	2,010	0,65	20,640	*
120	144	6,548	*	2,183	0,36	27,850	*
120	192	7,187	*	2,181	0,38	29,000	*
142	6	0,992	0,84	1,240	0,70	1,024	0,46
142	12	1,117	0,58	1,454	0,54	1,395	0,29
142	24	2,213	0,19	1,660	0,42	4,300	0,03
142	48	7,859	*	2,284	0,26	18,320	*

Table 5.3 Mean values of measured parameters, unsealed cables (kab m)

\* The cable samples were too brittle to allow test pieces to be cut out

Diagram 5.1 shows the indenter ratios as function of duration of the aging for the two temperatures 120°C and 142°C. Diagram 5.2 shows the same relationships for the  $e/e_0$  values. A regression analysis shows that the indenter ratios as function of duration can be approximated with a linear relationship. The elongation-at-break ratios shall approach zero when the duration becomes large. For this case we have assumed an exponential relationship.

The equations for the regression lines are given in the diagrams, together with the  $R^2$  values (squares of the correlation). Table 5.4 summarizes the correlations (R).

<u>Table 5.4</u> Linear correlation between duration of aging and degradation in terms of indenter ratios or elongation-at-break ratios, unsealed cables (kab m)

Cable	Linear correlation	, indenter ratio	Exponential correlation, e/e <sub>0</sub>		
	120 °C 142°C		120°C	142°C	
Lipalon	0,975	0,968	*	0,996	
Dätwyler	0,925	0,997	0,900	0,991	
Rockbestos	0,912	0,971	*	0,985	

\* less than three values

The correlations are generally high, which indicates a clear relationship between degradation, measured by the mechanical parameters, and the duration of high temperature exposure.



Diagram 5.1 Indenter ratios of thermally aged cables, kab m (unsealed)





# 5.3.1.2 Sealed cables (kab d)

The indenter modulus were determined on the sealed cables (kab d) after thermal aging. The results are summarized in Table 5.5. Each value in the table represents the mean values of two identically aged cables. The values are given in terms of the ratios between the indenter modulus of aged and unaged cable samples.

Temperature, °C	Duration, days	indenter ratio Lipalon	indenter ratio Dätwyler	indenter ratio Rockbestos
120	48	2,0585	1,1943	1,2767
120	96	2,4624	1,4017	1,8034
120	144	4,4826	(1,7580)	(25,7802)
120	192	4,8638	2,2260	28,9186
142	6	1,7780		
142	12	2,0078	1,3104	1,6206
142	24	2,1078	1,6031	3,2731
142	48	4,5913	2,2896	16,6729

Table 5.5 Mean values of measured indenter ratios, sealed cables (kab d)

Diagram 5.3 shows the indenter ratios as function of duration of the aging at 120 and  $142^{\circ}$ C. The equations for the linear regression lines are given in the diagrams, together with the R<sup>2</sup> values. Table 5.6 below summarizes the correlations (R).

<u>Table 5.6</u> Linear correlation between duration of aging and degradation in terms of indenter ratios, sealed cables (kab d)

	120°C	142°C
Lipalon	0,955	0,949
Dätwyler	0,990	0,999
Rockbestos	0,950	0,973

The correlations are very high. This shows that there is a clear relationship between the degradation, measured as the indenter values, and the duration of temperature exposure.



Diagram 5.3 Indenter ratios of thermally aged cables, kab d (sealed)

If  $t_1$  and  $t_2$  are the times to reach a certain degradation at temperatures  $T_1$  and  $T_2$  (in K), the activation energy E (in eV) in Arrhenius equation can be calculated, according to ref. /1/ as (k is the Boltzmann's constant = 0,86.10<sup>-4</sup> eV/K):

$$E = k \cdot \frac{\ln(t_2 / t_1)}{1 / T_2 - 1 / T_1}$$
 (5.1)

Diagram 5.4 shows the value of E as function of the level of indenter ratio.



<u>Diagram 5.4</u> Activation energies as function of indenter ratios, determined by comparison of degradation at 120°C and 142°C, kab d (sealed).

The variation with the degree of degradation is not very large in these cases.

5.3.1.3 Comparison of indenter ratios for sealed and unsealed cables.

Table 5.7 compares indenter ratios for sealed (kab d) and unsealed (kab m) cables.

Table 5.7 Comp	parison of indent	er ratios for l	kab d and kab m
----------------	-------------------	-----------------	-----------------

Temperature	Duration	Lipa	alon	Dätv	vyler	Rockbestos	
°C	days	kab m	kab d	kab m	kab d	kab m	kab d
120	48	1,498	2,059	1,737	1,194	1,838	1,277
120	96	3,650	2,462	2,010	1,402	20,640	1,803
120	192	7,187	4,864	2,181	2,226	29,000	28,919
142	12	1,117	2,008	1,454	1,310	1,395	1,621
142	24	2,213	2,108	1,660	1,603	4,300	3,273
142	48	7,859	4,591	2,284	2,290	18,320	16,673

It is seen from the table that the aging of the unsealed, short cable samples has a tendency to result in a higher level of degradation than the aging of the sealed, longer cable samples. The values of activation energies taken from measurements on sealed cables would be the most representative for actual conditions in nuclear power plants, since installed cables don't have open ends inside containment.

#### 5.3.2 Cables, influence of thermal aging on dielectric parameters

The results of the measurements of the insulation resistance, dielectric loss factor, and capacitance on the sealed cables (kab d) after thermal aging are summarized in Tables 5.8, 5.9 and 5.10 below. Each value shown in the tables represents the mean of two identically aged cables. Insulation resistances and capacitances are given in Mohm and pF/m, respectively (The values measured for the three meters long cables have been normalised to 1meter through multiplication by three for insulation resistance, division by three for capacitance).

The measurement of dielectric loss factor and capacitance was performed at 20, 60, 1000 and 10000 Hz on the Dätwyler and Rockbestos cables, only at 60 and 1000 Hz on the Lipalon cables. The values at 10000 Hz do not show a different picture from those at 1000 Hz and are therefore not used in the presentation below.

Temp	Time days	IR	loss factor	loss factor	capacit	capacit
°C		Mohm <sup>1)</sup>	60 Hz	1 kHz	60 Hz, pF/m	1 kHz, pF/m
120	48	1200	0,048	0,035	198	188
120	96	990	0,124	0,067	223	195
120	144	330	0,146	0,061	212	186
120	192	340	0,158	0,059	217	191
142	6	1200	0,034	0,034	199	189
142	12	1220	0,087	0,048	211	191
142	24	930	0,185	0,063	197	171
142	48	3,1	15,65	1,377	280	212

#### Table 5.8 Values of dielectric parameters, sealed Lipalon cables (kab d)

1) normalised to 1m

Significant changes of the dielectric parameter values are achieved for the aging performed at 120°C and 142°C. The dielectric loss factor, measured at 60 Hz, is the most sensitive parameter.

Table 5.9 Values of dielectric parameters, sealed Dätwyler cables (kab d)

Temp ℃	Time days	IR Mohm <sup>1)</sup>	loss factor 20 Hz	loss factor 60 Hz	loss factor 1 kHz	capacit 20 Hz, pF/m	capacit 60 Hz, pF/m	capacit 1 kHz, pF/m
120	48	375300	0,0542	0,0145	0,0097	150	148	145
120	96	405000	0,0729	0,0128	0,0088	145	142	141
120	192	405000	0,0141	0,0117	0,0085	151	150	147
142	12	344700	0,0181	0,0151	0,0117	151	149	146
142	24	281700	0,0288	0,0225	0,0143	148	146	142
142	48	132300	0,0734	0,0511	0,0195	158	151	144

1) normalised to 1m

The values at 142°C for 24 and 48 days varies for the two identically aged cables, one of them having odd values and being excluded in the table. The tendency is rather clear, but would be more unclear if the samples with the odd values were included.

<u>Table 5.10</u> Values of dielectric parameters, sealed Rockbestos cables (kab d)

Temp °C	Time days	IR Mohm <sup>1)</sup>	loss factor 20 Hz	loss factor 60 Hz	loss factor 1 kHz	capacit 20 Hz, pF/m	capacit 60 Hz, pF/m	capacit 1 kHz, pF/m
120	48	268200	0,0681	0,0020	0,0011	126	125	124
120	96	276300	0,0682	0,0023	0,0011	127	126	125
120	192	256500	0,0798	0,0148	0,0061	138	135	133
142	12	292500	0,0676	0,0021	0,0011	127	126	126
142	24	262800	0,0689	0,0038	0,0018	129	127	127
142	48	286200	0,0706	0,0144	0,0098	153	151	148

1) normalised to 1m

The tendencies for loss factors and capacitances follow the degree of thermal aging.

The relationships between dielectric loss factors at 60 Hz and duration of aging at 120°C and 142°C are shown in Diagram 5.5.



#### 5.3.3 Leads, influence of thermal aging on indenter ratios

The indenter values were determined on unsealed leads (led m) of groups 1ba, 1bb, 8a and 10a before and after additional aging. The results in terms of indenter ratios between aged and unaged leads are shown in Tables 5.11-5.13. Each value given is the mean value of two identically aged leads.

# Table 5.11 Mean values of measured indenter ratios, Lipalon cable lead

Before additional aging			After additional aging				
Temp, ℃	Time,day s	Indenter ratio	Temp, °C	Time,days (accumulated)	Indenter ratio		
120	48	2,203	120	144	21,243		
120	48	1,738	120	192	24,039		
142	3	1,858	142	24	36,684		
142	9	2,239	142	48	29,609		

Table 5.12 Mean values of measured indenter ratios, Dätwyler cable lead

Before ad	ditional agir	ıg	After additional aging			
Temp, ℃	Time,day s	Indenter ratio	Temp, ℃	Time,days (accumulated)	Indenter ratio	
120	48	0,986	120	144	1,544	
120	48	1,347	120	192	2,339	
142	3	0,917	142	24	1,475	
142	9	1,072	142	48	1,978	

Table 5.13	Mean values	of	measured	indenter	ratios.	<b>Rockbestos</b>	cable	lead
					,			

Before ad	ditional agir	ıg	After additional aging				
Temp, ℃	Time,day s	Indenter ratio	Temp, ℃	Time,days (accumulated)	Indenter ratio		
120	48	0,898	120	144	0,921		
120	48	0,922	120	192	0,982		
142	3	0,823	142	24	0,937		
142	9	0,795	142	48	0,997		

The Lipalon cable leads show a very significant degradation in terms of increased indenter ratio when subjected to prolonged aging at 120°C and at 142°C. This was also

the case for the measurements on the jacket, see table 5.3. This could be expected, since the lead insulation material is similar to that of the jacket (CSPE).

The Dätwyler cable leads show a clear degradation in terms of increased indenter ratio when subjected to prolonged aging at 120°C and at 142°C. This follows the behavior of the jacket at 142°C. The insulation material is similar to that of the jacket (EPDM).

The Rockbestos cable leads show insignificant degradation in terms of indenter ratio when subjected to prolonged aging at 120°C and 142°C. The cable conductor insulation (XLPE) is obviously very insensitive in its mechanical properties to the thermal aging, in contrast to the cable jacket material (CSPE).

**5.3.4 O -rings, influence of thermal aging on indenter ratios and thickness ratios** The indenter values were determined on 16 samples of type Viton and 6 samples of type EPDM before thermal aging of unmounted o-rings Or e. The indenter modulus are given in Table 5.14. The variation is very small, approx. 2%.

T-11-514	In Janton		~~~ d	- + man al man al	Jan . I mai a	- 4	Comment.	-	
1 able 5.14	Indenter	values	ana	sianaara	aeviation	OŢ	unagea	0 -	rings

O - ring type	Mean indenter modulus, N/mm	Standard deviation, N/mm		
Viton	1,417	0,029		
EPDM	1,667	0,037		

The values measured after thermal aging are given in Table 5.16. The indenter ratios given in the table are the ratios between the indenter values of the aged and unaged orings. Each value given represents the mean value of two identically aged orings.

Temperature, °C	Duration, days	indenter ratio,	indenter ratio,
		o-ring EPDM	o-ring Viton
120	48	0,9883	0,9861
120	96	1,0167	0,9904
120	192	1,5089	1,0835
142	12	1,0123	0,9812
142	24	1,2323	0,9607
142	48	1,4063	0,9571

Table 5.17 Mean values of measured indenter ratios, o-rings

The relationships for o-rings aged at 120°C and 142 °C are shown in Diagram 5.6, where also the regression lines are included. For EPDM there is a clear relationship,

making it possible to determine the activation energy, whilst no clear relationships occur for Viton after aging at 142 °C.



Diagram 5.6 Indenter ratios of thermally aged o-rings

The thicknesses of the o-rings were measured before mounting in the compression fixture. The measurements were repeated after demounting after thermal aging. Table 5.16 shows the ratios between the thicknesses immediately after and before the thermal aging. A measurement of the thickness was also made 1 h after demounting and 30 days after demounting. The relaxation is small - most of the compression remains after 30 days.

Temperature, °C	Duration, days	thickness ratio, o-ring Viton	thickness ratio, o-ring EPDM
120	48	0,9634	0,9164
120	96	0,9428	0,8733
120	144	0,9540	0,8385
120	192	0,9482	0,8161
142	6	0,9813	0,9560
142	12	0,9821	0,9343
142	24	0,9652	0,8803
142	48	0,9634	0,8190

Table 5.16 Mean values of measured thickness ratios, o-rings

For EPDM there is a clear relationship between degree of thermal aging and thickness ratio, making it possible to determine the activation energy, whilst no clear relationships occur for Viton. The relationships for EPDM o-rings aged at 120 and 142 °C are shown in Diagram 5.7, where also the regression lines are included.



Diagram 5.7 Thickness ratios of thermally aged o-rings type EPDM

Diagrams 5.8 and 5.9 show the results of calculations of activation energies as function of degradation level for the EPDM cables. Whilst the activation energy calculated

decreases with degradation measured as increase in indenter ratios, approaching 0,7 eV at high indenter ratios, the activation energy increases with degradation measured as decrease in thickness ratios, up to around 0,9 eV at low thickness ratios.



Diagram 5.8 O-ring EPDM. Activation energies as function of indenter ratios



Diagram 5.9 O-ring EPDM. Activation energies as function of thickness ratios

The relationships between indenter ratios and thickness ratios is shown in Diagram 5.10. Again there is little correlation between the values for o-ring Viton, due to the fact that the mechanical degradation is very small. For o-ring EPDM there is a reasonable relationship, with a linear correlation R = 0,924.



Diagram 5.10 O-rings. Relationship between indenter ratio and thickness ratio

# 5.3.5 Solenoid coils, influence of thermal aging on insulation resistance and coil resistance

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Only insulation resistance and coil resistance was measured before and after the additional aging by 21 days at 142°C to totally 24 days (group 8a) and by 39 days at 142°C to totally 48 days (group 10a). The results are shown in table 5.17.

Table 5.17 Results of measurements on solenoid coils

Aging time	Insulation resist	ance, Mohm	Coil resistance, ohm		
	before aging	after aging	before aging	after aging	
24 days	493	358	874	883	
48 days	494	81,9	895	899	

The additional aging has had an influence on the insulation resistance, primarily related to the connecting cable (Rockbestos), but not on the coil resistance.
#### 6 Influence of nitrogen atmosphere on the thermal aging

A study of the influence on the thermal aging of using nitrogen instead of air in the containment, as is the case during operation of Swedish BWR's, is reported in ref. /1/. The comparison was made at thermal aging in 95 °C for 96 and 192 days, at 120 °C for 24 and 48 days and at 142 °C for 6 and 12 days. The results didn't generally show a significant reduction of degradation due to aging in nitrogen. The oxygen present in the material from the beginning may have been enough for the oxidation process.

It is important to know if we get a more positive influence of using nitrogen atmosphere when we subject the cables to more severe thermal aging conditions. We have included testing in nitrogen atmosphere at 142 °C for 24 and 48 days. The testing has been performed on cables, leads and o-rings. The test program is shown in Table 6.1.

Conditioning/	Gro	up of sa	mples	, No		Nun	Number of samples in			
measurement							each	group		
	49	50	51	52	36	38	kab m	led m	Orm	Оте
Indenter value	x	x	x	x			2	2	-	2
Deformation	x	x	x	x			-	-	2	-
Aging 142°C										
nitrogen 18d					x		-	2	-	-
air 24d	(x)						(2)	(2)	(2)	(2)
nitrogen 24d			x				2	-	2	2
nitrogen 36d						x	-	2	-	-
air 48d		(x)					(2)	(2)	(2)	(2)
nitrogen 48d				x			2	-	2	2
Indenter value	x	x	x	x	x	x	2	2	-	2
e/e <sub>0</sub>	x	x	x	x	x	x	2	-	-	-
Deformation	х	x	x	x	х	x	-	-	2	-

#### <u>Table 6.1</u> Test program for complementary studies of the influence of nitrogen atmosphere on thermal aging

The tests on cables (unsealed, kab m) were made on new cable pieces of 30 cm length. Unaged o-rings were used for the tests on Or e and Or m. The tests on the leads were made by additional aging of samples, which had been earlier aged for 6 days (group No 36) and 12 days (group No 38) in nitrogen atmosphere. The total aging times in 142 °C for the leads were thereby extended to 24 and 48 days.

The results for the cables are shown in Table 6.2

Cable type		Indente	er ratio		Elongation-at-break ratio					
	24	days	48 (	48 days		days	48 days			
	air	nitrogen	air	air nitrogen		nitrogen	air	nitrogen		
Lipalon	2,213	1,2713	7,859	4,715	0,19	0,40	*	*		
Dätwyler	1,660	1,572	2,284	1,563	0,42	0,59	0,26	0,72		
Rockbestos	4,300	1,0875	18,320	1,4407	0,03	0,37	*	0,20		

<u>Table 6.2</u> Influence of nitrogen atmosphere on thermal aging of cables (142°C)

\* The cable samples were too brittle to allow test pieces to be cut out

There is a very significant reduction of the aging in nitrogen atmosphere compared to in air in all cases except after 24 days of aging of the Dätwyler cable. The effect is particularly drastic for the Rockbestos cable (jacket).

The results for the leads are shown in Table 6.3

Table 6.3	Influence of	f nitrogen	atmosphere on	thermal	aging o	f leads	$(142^{\circ}C)$
						,	

Cable type	Indenter ratio								
- 	24	days	48 days						
	air	nitrogen	air	nitrogen					
Lipalon	36,684	8,728	29,609	7,740					
Dätwyler	1,475	1,761	1,978	1,797					
Rockbestos	0,937	0,989	0,997	1,081					

The tendency is clear for the Lipalon lead (CSPE). A slight influence can be shown for the Dätwyler cable lead (EPDM) at 48 days (as for the jacket). For the Rockbestos cable leads (XLPE) we cannot see any significant difference; the degradation of the material is insignificant both in air and in nitrogen.

The results for the o-rings are shown in Table 6.4

<u>Table 6.4</u> Influence of nitrogen atmosphere on the thermal aging of o-rings

O-ring		Indente	er ratio		Thickness ratio					
24		days	48	48 days		days	48 days			
	air	nitrogen	n air nitroger		air nitrogen		air	nitrogen		
Viton	0,988	0,946	1,054	0,969	0,965	0,974	0,963	0,955		
EPDM	1,129	0,996	1,404	0,992	0,880	0,962	0,819	0,946		

There is a very pronounced influence of using nitrogen atmosphere on the degradation of EPDM o-rings. For the Viton o-rings the degradation is insignificant already in air. We can see a minor effect after 48 days and we would expect a more significant effect on aging at 142 °C for longer duration.

### 7 Irradiation and LOCA testing

#### 7.1 Purpose

The most important factor in the selection of parameters for evaluation of the influence of long-term exposure to various aging conditions is the relationship between the values measured after aging and the operational behavior during LOCA. The purpose of this phase of the study is therefore to establish this relationship for the parameters used for the determination of the degree of degradation due to the aging. The following has been studied

- relationship between degradation due to thermal aging and dielectric behavior during LOCA testing
- LOCA testing as part of the evaluation of the benefits of using on-going qualification as part of the program for ensuring long-term safety
- LOCA testing as part of the evaluation of the effect of including humidity and vibration in the artificial aging

The LOCA tests included irradiation, followed by subjection to the LOCA temperature/pressure profile. The irradiation was performed for cables, cable leads, orings and solenoids. The irradiation level applied ( 50h at 10kGy/h, see clause 3.3) was intended to simulate the exposure during LOCA. Only the cables (sealed, kab d) were subjected to the full LOCA test including the LOCA temperature/pressure profile.

#### 7.2 LOCA test profile

The LOCA test was performed at ABB test facility LOKE (see Appendix A01), subjecting the components to overheated steam for 3 hours at 181°C and 0.4 MPaG (gage pressure), followed by 160°C and 0.4 MPaG for 3 hours and 120°C for 44 hours.

An example of the profile of the LOCA test achieved is shown in Diagram 7.1 below.



Diagram 7.1 LOCA test profile

# 7.3 Testing of cables for evaluation of relationships between degradation due to thermal aging and dielectric behavior during LOCA testing

#### 7.3.1 Test program

Table 7.1 shows the test program for LOCA testing of cables with the purpose of studying the dielectric behavior of thermally aged cables during LOCA and of establishing the relationships between values of degradation parameters before LOCA testing and values of dielectric parameters during LOCA.

The tests included irradiation to LOCA-dose, applied before subjecting the cables to the LOCA profile. All tests were made on sealed cables (kab d).

Conditioning/measurem.	Gro	oup of	f samj	oles, l	No									
	11	3	4	13	14	16	17	16	19	lbb	9	20	8a	10a
Cable <sup>1)</sup>	L	L	L	L	LDR	LDR	LDR	LDR	LDR	LDR	L	LDR	LDR	LDR
PREHISTORY														
aging temperature °C	80	80	80	95	95	95	95	120	120	120	142	142	142	142
duration, days	192	384	576	48	96	192	384	48	96	192	6	12	24	48
Measurements:														
indenter	x	x	x	x	x	x	x	x	x	x	x	x	x	x
elongation-at-break	(x)	(x)	(x)	(x)	(x)	(x)	(x)	(x)	(x)	(x)	(x)	(x)	(x)	(x)
IR	x	x	x	х	x	x	x	x	x	x	x	x	x	x
loss factor	x	x	x	x	x	x	x	x	x	x	x	x	x	x
capacitance	x	x	x	x	x	x	x	x	x	x	x	x	x	x
coil resistance	x	x	x	x	x	x	x	x	x	x	x	x	x	x
IRRADIATION	x	x	x	x	x	x	x	x	x	x	x	x	x	x
MEAS BEFORE LOCA														
indenter	x	x	x	x	x	x	x	x	x	x	x	x	x	x
IR	x	x	x	x	x	x	x	x	x	x	x	x	x	x
loss factor	x	x	x	x	x	x	x	x	x	x	x	x	x	x
capacitance	x	x	x	x	x	x	x	x	x	x	x	x	x	x
MEAS DURING LOCA														
IR	x	x	<u>x</u>	x	x	<u>x</u>	x	<u>x</u>	x	x	x	x	x	x
loss factor	x	x	x	<u>x</u>	x	<u>x</u>	x	x	x	x	x	x	<u>x</u>	<u>x</u>
MEAS AFTER LOCA														
IR	x	x	x	x	x	x	x	x	x	x	x	x	x	x
loss factor	x	x	x	x	x	x	x	x	x	x	x	x	x	x
capacitance	x	x	x	x	x	x	x	x	x	x	x	x	x	x
indenter	x	x	x	x	x	x	x	x	x	x	x	x	x	x

<u>Table 7.1</u> Test program for evaluation of relationships between degradation due to thermal aging and dielectric parameters during LOCA

1) L=Lipalon, D=Dätwyler, R=Rockbestos

#### 7.3.2 Test results, cable Lipalon

#### 7.3.2.1 Indenter modulus before irradiation, after irradiation and after LOCA

Indenter measurements were made before irradiation, after irradiation and after LOCA. The values presented are given in terms of ratios between indenter modulus of conditioned cables and of unaged and unconditioned cables (see clause 4.2).

The results of the measurement of the indenter ratios are summarized in Diagram 7.2. The values given are the mean values of two cables tested at each combination of aging temperature and duration. The values shown on the abscissa are the temperatures and durations of the thermal aging before irradiation.



### <u>Diagram 7.2</u> Cable Lipalon. Indenter ratios before irradiation, after irradiation and after LOCA

In general, the irradiation has had a degrading effect on the mechanical behavior of the cables. The indenter values after the LOCA test are lower than before the LOCA test, due to a softening of the insulation material through the influence of the steam.

### 7.3.2.2 Insulation resistance before irradiation, after irradiation, during and after LOCA

Insulation resistance measurements were made before irradiation, after irradiation and during and after LOCA. The results are summarized in Diagram 7.3. The values given are the mean values of two cables tested at each combination of aging temperature and duration. Two values are presented from the values measured during LOCA. The first value from LOCA phase I is the lowest value measured during the first 6 hours of the

LOCA profile. The second value from LOCA phase II is the value measured at the end of the LOCA, just before the temperature and pressure are restored to room conditions.

The values are the mean values of the insulation resistances measured for the various parts, normalised to 1m through multiplication by the length of the cable tested (3m).



<u>Diagram 7.3</u> Cable Lipalon. Insulation resistance, normalised to 1m cable length, before irradiation, after irradiation, during and after LOCA (no values are given during LOCA for the cables which had been aged at 120°C for 192 days and at 142°C for 48 days. They were too damaged to make it possible to measure the insulation resistance)

The insulation resistance during LOCA decreases with duration of exposure in the thermal aging at all temperatures.

The insulation resistances decrease to values below 1 Mohm during LOCA for most of the cables. Part of this decrease is a direct effect of the variation of insulation resistance with temperature, regardless of aging. This variation was investigated by a test of unaged cables in a dry heat chamber without high pressure and humidity. The results for the insulation resistance of the Dätwyler cable (mean value of insulation resistances of the parts) are shown in table 7.2 below. The cables tested had a length of 10.5m. The insulation resistances are the values measured, multiplied by this length.

Temperature,°C	Duration, min	IR, Mohm <sup>1)</sup>	Duration, min	IR, Mohm <sup>1)</sup>
22,7		128000		
60	30	4700	90	2840
100	30	255	90	193
120	30	88	90	96
160	30	47,6	60	40,8
172	30	23,7	60	19,9
179	30	15,3	60	13,9

Table 7.2 Cable Lipalon. Temperature dependence of insulation resistance

1) normalised to 1m cable length

A comparison of the indenter ratios of the cables before LOCA and the insulation resistances during LOCA is shown in Diagram 7.4.



<u>Diagram 7.4</u> Cable Lipalon. Relationship between indenter ratios before LOCA and insulation resistance during LOCA

There is a significant tendency of lower insulation resistance for cables with higher indenter ratios, even if the correlation is low ( $R\approx 0.4$ ).







The dielectric loss factor during LOCA shows a relationship to the degree of preaging before LOCA. The loss factor increases with increased thermal aging.



Diagram 7.6 shows the relationships between indenter ratios before LOCA and loss factors during and after LOCA.

<u>Diagram 7.6</u> Cable Lipalon. Relationship between indenter ratios before LOCA and loss factors during and after LOCA

Diagram 7.7 shows the relationships between loss factors before LOCA and loss factors during and after LOCA.

There is a significantly stronger correlation between loss factor before LOCA and loss factor during LOCA (R = 0.5-0.9) than between indenter ratio before LOCA and loss factor during LOCA( $R\approx 0.4$ ).



<u>Diagram 7.7</u> Cable Lipalon. Relationships between loss factors before LOCA and loss factors during and after LOCA.

#### 7.3.2.4 Conclusions

The results of the study on the Lipalon cable is that there is a good correlation between the mechanical degradation measured by the indenter and the aging conditions. There is also a typical trend between the mechanical degradation in terms of indenter values and the dielectric parameters measured during LOCA. There is a significantly stronger trend between dielectric condition in terms of loss factor before LOCA and the same conditions during LOCA. The problem with using dielectric conditions before LOCA as measure of degradation is that significant changes are only detectable for very severely degraded cables.

#### 7.3.3 Test results, cable Dätwyler

#### 7.3.3.1 Indenter modulus before irradiation, after irradiation and after LOCA

Indenter measurements were made before irradiation, after irradiation and after LOCA. The values presented are given in terms of ratios between indenter modulus of conditioned cables and of unaged and unconditioned cables (see clause 4.2).

The results of the measurement of the indenter ratios are summarized in Diagram 7.8. The values given are the mean values of two cables tested at each combination of aging temperature and duration. The values shown on the abscissa are the temperatures and durations of the thermal aging before irradiation.



<u>Diagram 7.8</u> Cable Dätwyler. Indenter ratios before irradiation, after irradiation and after LOCA

In general, the irradiation has had a degrading effect on the mechanical behavior of the cables. Effects of the high temperature of the LOCA are also shown, especially for the rather severely degraded cables which have been subjected to a thermal aging for 48 days at 142 °C.

## 7.3.3.2 Insulation resistance before irradiation, after irradiation, during and after LOCA

Insulation resistance measurements were made before irradiation, after irradiation and during and after LOCA. The results are summarized in Diagram 7.9. The values given are the mean values of two cables tested at each combination of aging temperature and duration.

Two values are presented from the values measured during LOCA. The first value from LOCA phase I is the lowest value measured during the first 6 hours of the LOCA profile. The second value from LOCA phase II is the value measured at the end of the LOCA, just before the temperature and pressure are restored to room conditions.

The values are the mean values of the insulation resistances measured for the various parts, normalised to 1m through multiplication by the length of the cable (3m).



<u>Diagram 7.9</u> Cable Dätwyler. Insulation resistance (normalised to 1m cable length) before irradiation, after irradiation, during and after LOCA

The insulation resistance during LOCA decreases with duration of exposure in the thermal aging at 95 °C. For the cables aged at 120°C and 142°C we don't see any such effect. The explanation may be that the material which has been subjected to the high temperatures during aging has been dried out to such extent during the thermal aging that the humidification caused by LOCA is not enough to compensate for this effect on the insulation.

Since the insulation resistance doesn't show a reasonably consistent decrease with increased thermal aging, a comparison with the mechanical condition before LOCA is not meaningful.

The insulation resistances decrease to values below 1 Mohm during LOCA for most of the cables. Part of this decrease is a direct effect of the variation of insulation resistance with temperature, regardless of aging. This variation was investigated by a test of unaged cables in a dry heat chamber without high pressure and humidity.

The results for the insulation resistance of the Dätwyler cable (mean value of insulation resistances of the parts) are shown in table 7.3 below. The cables tested had a length of 10.5m. The insulation resistances are normalised to 1m through multiplication by this length.

Table 7.3	Cable Dätwyler.	Temperature de	ependence of	insulation resistance
Statement and a statement of the stateme		<b>I</b>	1 1	

Temperature,°C	Duration, min	IR, Mohm <sup>1)</sup>	Duration, min	IR, Mohm <sup>1)</sup>
22,7		158000		
60	30	44100	90	33300
100	30	7380	90	6020
120	30	4680	90	6170
160	30	5700	60	6690
172	30	6510	60	6740
179	30	5300	60	4850

1) normalised to 1m cable lenght

### 7.3.3.3 Cable Dätwyler. Dielectric loss factors before and after irradiation, during and after LOCA

Dielectric loss factor measurements were made before irradiation, after irradiation and during and after LOCA. The results are summarized in Diagram 7.10.

The dielectric loss factor during LOCA shows no direct relationship to the degree of preaging before LOCA, except for cables aged in 95°C, where a slight increase at increased aging time can be seen. Consequently, there is no significant correlation between the values of loss factors measured during LOCA and the mechanical condition of the cable.



Diagram 7.10 Cable Dätwyler. Loss factors before, during and after LOCA

Diagram 7.11 shows the relationship between loss factors before and during LOCA.



Diagram 7.11 Cable Dätwyler. Relationships between dielectric loss factors before and during LOCA

It is clear from the results presented in Diagram 7.11 that we haven't found any correlation between loss factors before and during LOCA.

#### 7.3.3.4 Conclusions

The results of the study on the Dätwyler cable are that there is a good correlation between the mechanical degradation measured by the indenter and the aging conditions, but the dielectric parameters measured during LOCA are not well related to the degree of aging. The Dätwyler cable is very resistant to thermal aging which hasn't proceeded in our tests to a degree where very significant aging related effects are shown in the dielectrical behavior during LOCA.

#### 7.3.4 Test results. Cable Rockbestos

#### 7.3.4.1 Indenter modulus before irradiation, after irradiation and after LOCA

Indenter measurements were made before irradiation, after irradiation and after LOCA. The values presented are given in terms of ratios between indenter modulus of conditioned cables and of unaged and unconditioned cables (see clause 4.2). The results of the indenter measurements are summarized in Diagram 7.12. The values given are the mean values of two cables tested at each combination of aging temperature and duration. The values shown on the abscissa are the temperatures and durations of the thermal aging before irradiation.



<u>Diagram 7.12</u> Cable Rockbestos. Indenter ratios before irradiation, after irradiation and after LOCA

In general, the irradiation has had a rather limited effect on the mechanical behavior of the cables. For the most degraded cables, LOCA has reduced the indenter modulus, probably due to softening of the insulation material by the hot steam during LOCA.

## 7.3.4.2 Insulation resistance values before irradiation, after irradiation, during and after LOCA

Insulation resistance measurements were made before irradiation, after irradiation and during and after LOCA. The results are summarized in Figure 7.13. The values given

are the mean values of two cables tested at each combination of aging temperature and duration. Two values are presented from the values measured during LOCA. The first value from LOCA phase I is the lowest value measured during the first 6 hours of the LOCA profile. The second value from LOCA phase II is the value measured at the end of LOCA. The values are the mean values of the insulation resistances measured for the various parts, normalised to 1m through multiplication by the length of the cable (3m).



<u>Diagram 7.13</u> Cable Rockbestos. Insulation resistance (normalised to 1m cable length) before and during LOCA

The insulation resistances decrease to values below 1 Mohm during LOCA for most of the cables. Part of this decrease is a direct effect of the variation of insulation resistance with temperature, regardless of aging. This variation was investigated by a test of unaged cables in a dry heat chamber without high pressure and humidity. The results for the insulation resistance of the Rockbestos

cable (mean value of insulation resistances of the parts) are shown in table 7.4.

Temperature, °C	Duration, min	IR, Mohm	Duration, min	IR, Mohm
22,7		1120000		
60	30	666000	90	721000
100	30	327000	90	304000
120	30	53400	90	42700
160	30	4250	60	5160
172	30	3360	60	3340
179	30	2360	60	2310

Table 7.4 Cable Rockbestos. Temperature dependence of insulation resistance

It can be seen in Diagram 7.13. that the insulation resistance during the LOCA test decreases below the values caused by only the temperature dependence, but there is no clear tendency of relationship to the degree of thermal aging which the cables have been subjected to before the LOCA test.

before LOCA is not meaningful. Such a comparison has therefore not been included.

# 7.3.4.3 Dielectric loss factors before irradiation, after irradiation, during and after LOCA

Dielectric loss factor measurements were made before irradiation, after irradiation and during and after LOCA. The results are summarized in Figure 7.14.



Diagram 7.14 Cable Rockbestos. Loss factors before, during and after LOCA

The dielectric loss factor couldn't be measured during LOCA for the cables which had been aged at 120°C for 192 days and at 142°C for 48 days - they were in too poor conditions for that. Only the dielectric loss factor at 60 Hz could be measured for the cables which had been aged at 142°C for 24 days. The higher loss factors after LOCA for those cables indicate that the dielectric losses during LOCA was considerably higher than for the cables which had been subjected to less severe aging.

A comparison of the indenter ratios of the cables before LOCA and the dielectric loss factors after LOCA should be reasonably representative for investigating to what extent the behavior during LOCA is related to the mechanical condition before LOCA. The result of such comparison is shown in Diagram 7.15.



Diagram 7.15 Cable Rockbestos. Relationship between indenter ratios before LOCA and loss factors after LOCA

The correlation between indenter ratios before LOCA and dielectric losses after LOCA is rather good (R = 0.85 for dielectric loss factor measured at 20 Hz, 0.88 for 60 Hz and 0.88 for 1 lkHz).

Diagram 7.16 shows the relationships between loss factors before LOCA and loss factors after LOCA



<u>Diagram 7.16</u> Cable Rockbestos. Relationship between loss factors before and after LOCA

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The correlations between the loss factors before and after LOCA are lower than between indenter modulus before LOCA and loss factor after LOCA. This is probably due to the fact that the indenter modulus is more sensitive to degradation than the loss factor and therefore shows more pronounced changes with aging before LOCA.

#### 7.3.4.4 Conclusions

The results of the study on the Rockbestos cable is that there is a good correlation between the mechanical degradation measured by the indenter and the aging conditions, but the dielectric parameters measured during LOCA are not well related to the degree of aging. A reasonable correlation is shown between the indenter modulus before LOCA and the dielectric parameters during and after LOCA.

## 7.4 Testing of solenoids for evaluation of relationships between dielectric conditions before and after LOCA

A few solenoid coils, type Seitz 2A25, were included in the LOCA test program. They were measured before irradiation, after irradiation and after LOCA, according to the program in table 7.5.

<u>Table 7.5</u> Solenoids type Seitz 2A25. Test program for evaluation of relationships between dielectric conditions before and after LOCA

Group of samples, No	1b	9	8a	10a
PREHISTORY				1
aging temperature °C	120	142	142	142
duration, days	48	6	24	48
MEAS BEFORE IRRADIATION				
IR, loss factor, capacitance and coil resistance	x	x		x
IRRADIATION	x	x	x	x
MEAS AFTER IRRADIATION				
IR, loss factor, capacitance and coil resistance	x	x	x	x
LOCA PROFILE				
MEAS AFTER LOCA				
IR, loss factor, capacitance and coil resistance	x	x	x	x

#### 7.4.1 Test results

Diagram 7.17 shows the results of measurements of resistance parameters



Diagram 7.17 Solenoid Seitz 2A25. Influence of irradiation and LOCA on insulation resistance and coil resistance

The insulation resistance increases at irradiation (due to drying) and decreases after LOCA (due to humidification). The insulation resistance after LOCA has a significant relationship with the degree of thermal aging. The coil resistance increases after irradiation and after LOCA. There is a significant relationship between the coil resistances measured before and after LOCA.

Diagram 7.18 shows the results of measurements of dielectric loss factors before irradiation, after irradiation and after LOCA.



Diagram 7.18 Solenoid Seitz 2A25. Influence of irradiation and LOCA on dielectric loss factors

There is a significant increase in loss factor with irradiation and with increased thermal aging. There is also a significant relationship of the loss factor at 60 Hz before and after LOCA.

Diagram 7.19 shows the results of measurements of capacitance before irradiation, after irradiation and after LOCA.



Diagram 7.19 Solenoid Seitz 2A25. Influence of irradiation and LOCA on dielectric loss factors

There is a slight tendency of the capacitance to increase with irradiation and a more significant tendency of it to increase with increased thermal aging.

#### 7.4.2 Conclusions

The number of solenoid coils tested is low and therefore the conclusions are based on a statistically thin material. The results indicate that values of dielectric parameters after aging can be useful in predicting the behavior at LOCA.

## 7.5 Testing of influence of ionizing radiation on degradation of leads and o-rings7.5.1 Test program

As part of the program, some leads and o-rings were subjected to ionizing irradiation, simulating the LOCA dose, but not to LOCA testing. Table 7.6 shows the test program and the results of the tests on influence of irradiation are reviewed here.

Group of samples, No	16	17	16	19	1bb-2	20	8a	10a	27	29
Component	Or	Or	Or	Or	Or	Or	Or	Or	led	led
PREHISTORY										
aging temperature °C	95	95	120	120	120	142	142	142	120	120
duration, days	192	384	48	96	192	12	24	48	48	48
vibration, mm amplitude									3,5	7,0
MEAS BEFORE IRRADIATION										
indenter	x	x	x	х	x	x	x	x	x	x
insulation resistance									X	х
loss factor									x	x
IRRADIATION (5kGy/h, 100h)	x	x	x	x	x	x	x	x	x	х
MEAS AFTER IRRADIATION										
indenter	x	x	x	x	x	x	х	х	x	х
insulation resistance									х	х
loss factor									x	x

<u>Table 7.6</u>	O-rings and leads.	Test program for	evaluation	of influence	of ionizing
radiation of	on degradation				

#### 7.5.2 Influence of radiation on degradation parameter values, cable leads

Leads, which had been used in the earlier study (ref./1/) for the evaluation of the effects of including vibration in the long-term exposure were subjected to the irradiation. The leads had been aged by exposure to  $120^{\circ}$ C for 48 days and, in sequence, sweep sine vibration at an amplitude of 3,5 mm/ 30m/s<sup>2</sup> or 7 mm/ 60m/s<sup>2</sup>. Only leads of Lipalon cables were tested.

The values of degradation parameters before and after irradiation are shown in Table 7.7. The values given are the mean values of two identical samples, subjected to identical conditions. The lengths of the leads were 0,5m.

Vib level Indenter ratio		nter ratio Loss factor, 60 Loss facto Hz 1kHz		factor, Hz	Insula resistance	ation , Mohm <sup>1)</sup>		
mm	before	after	before	after	before	after	before	after
3,5	4,163	5,919	0,022	0,275	0,021	0,099	180	20500
7	3,541	3,708	0,019	0,248	0,021	0,115	175	20250

<u>Table 7.7</u> Cable leads. Values of degradation parameters before and after irradiation.

1) normalised to 1m lead length

All values are significantly affected by the irradiation. As for the cables, the insulation resistances increased after irradiation.

#### 7.5.3 Influence of radiation on degradation parameter values, o-rings

The o-rings tested were of type EPDM and Viton. Diagram 7.20 shows the results in terms of indenter ratios before and after irradiation for o-rings type EPDM. The values shown are the mean values of two identical samples.



<u>Diagram 7.20</u> O-rings EPDM. Comparison of indenter ratios before and after irradiation.

The irradiation has a limited, but rather consistent, influence on the mechanical degradation, measured as increase in indenter ratio.

Diagram 7.21 shows the results of measurement of indenter ratios before and after irradiation for o-rings type Viton. The values shown are the mean values of two identical samples.



Diagram 7.21 O-rings Viton. Comparison of indenter ratios before and after irradiation.

The influence of irradiation is, in contrast to the influence of the thermal aging, much more pronounced for Viton than for EPDM.

### 8 Evaluation of the benefits of using on-going qualification as part of the program for ensuring long-term safety

#### 8.1 Background

In ref. /1/ a comparison is made between subjecting the test specimen to thermal aging at 142°C for 6 days with subjecting it to 80°C for 384 days, followed by 120°C for 12 days. This should simulate an on-going qualification procedure for qualifying for an actual life of 576 days at 80°C by one of the following two alternatives:

- using a highly accelerated thermal aging of 6 days at 142°C, assuming that the activation energy is 0,83 eV, which had been found for the Lipalon cable in the study
- assuming that the specimen has been qualified for 384 days initially and after 384 days of installed time extend the qualification to 576 days by a moderately accelerated thermal aging of 12 days at 120°C, which with the assumption of activation energy of 0,83 eV corresponds to 192 days at 80°C.

The indenter ratios measured on the Lipalon cable jacket were as follows:

- after thermal aging for 576 days at 80°C: 2,018
- after thermal aging for 384 days at 80°C, followed by 12 days at 120°C: 2,084 . 1,865
- after thermal aging for 6 days at 142°C:

The differences are thus 0,066 for the simulated on-going procedure, 0,153 for the highly accelerated test procedure. The simulated on-going procedure also resulted in a conservative degradation value, while the highly accelerated procedure resulted in some underestimation of the degradation.

The result was typical for the Lipalon cable. It is therefore of interest to compare the behavior of the three aged cables in LOCA.

#### 8.2 Test program

Table 8.1 shows the test program used for the evaluation

<u>Table 8.1</u> Cable Lipalon. Test program for evaluation of the benefit of on-going procedure

Conditioning/measurement	Group of	samples, N	lo	Number of samples
PREHISTORY	4	9	12	kab d
aging temperature °C	80	142	80/120	1
duration, days	576	6	384/12	
Measurements:				
indenter	x	x	x	2
IR	x	x	x	2
loss factor	x	x	x	2
IRRADIATION	x	x	x	2
MEAS BEFORE LOCA				
indenter	x	x	x	2
IR	x	x	x	2
loss factor	x	x	x	2
MEAS DURING LOCA				
IR	x	x	x	2
loss factor	x	x	x	2

#### 8.3 Test results

Table 8.2 summarizes the values of dielectric loss factors and insulation resistances measured during LOCA. The loss factors and insulation resistance values given are the maximum values measured during LOCA, normally occurring during the first 6 hours (at extreme steam temperature and pressure) of the LOCA cycle.

<u>Table 8.2</u> Cable Lipalon. Maximum loss factor values and minimum insulation resistance values measured during LOCA

temp	time	Loss factor, 60Hz			Loss factor, 1 kHz			Insul. resistance, Mohm <sup>1)</sup>		
°C	days	sample1	sample2	mean	sample1	sample2	mean	sample1	sample2	mean
80	576	28,1	26,5	27,3	4,48	4,56	4,52	0,1296	0,1260	0,1278
142	6	13,5	15,0	14,3	2,81	3,16	2,99	0,1710	0,1701	0,1705
80/120	384/12	23,0	26,5	24,8	7,40	5,85	6,63	0,2259	0,2043	0,2151

1) normalised to cable length 1m

The tendency when loss factor values are considered is an undertesting in terms of preaging for the highly accelerated case (142°C, 6 days), whilst the on-going simulation results in nearly the same values at 60 Hz, somewhat conservative values at 1 kHz. The insulation resistances show a different tendency.

The correlations between the indenter ratios measured before LOCA, presented in 8.1, and the dielectric values measured during LOCA, presented in table 8.2, are:

•	for loss factor 60 Hz	C	,887
•	for loss factor 1 kHz	. 0	,950
•	for insulation resistance	0	,306

Thus, the loss factors are well correlated to the conditions of the cables, measured as indenter values. The insulation resistances are not well correlated to the indenter values.

The conclusion is that the results of the comparison of the two methods for simulating the thermal aging conditions, achieved from the tests reported in ref. /1/, are confirmed when comparison is made with maximum loss factors during LOCA.

A comparison with insulation resistances during LOCA doesn't show similar results. The reason is presumably that the cables are not degraded to a degree at which consistent changes in insulation resistance during LOCA occur.

### 9 Testing for evaluation of the influence of humidity as part of the long-term exposure

#### 9.1 Purpose

The results of the earlier tests, presented in ref. /1/ indicated an increase in the degradation due to exposure to high humidity as part of the thermal aging. The components which had been subjected to humidity were included in the LOCA test presented here, with the purpose of evaluating to what extent the additional degradation because of the humidity factor in the aging program had an influence on the dielectric behavior during LOCA.

#### 9.2 Test program

Table 9.1 shows the test program used for study of the influence of humidity.

<u>Table 9.1</u> Cable Lipalon. Test program for evaluation of the dielectric behavior at LOCA of test specimen which have been subjected to thermal aging in humid atmosphere

Conditioning/	Group	of samp	oles, No				Number of samples
measurement	_	_					in each group
PREHISTORY	13	14	16	17	30	31	kab d
aging temp°C/RH%	95/low	95/low	95/low	95/low	95/95	95/95	
duration, days	48	96	192	384	24	48	
Measurements:	1						
indenter	x	x	x	x	x	x	2
elongation-at-break	(x)	(x)	(x)	(x)	(x)	(x)	(2)
IR	x	x	x	x	x	x	2
loss factor	x	x	x	x	x	x	2
capacitance	x	x	x	x	x	x	2
IRRADIATION	(X)	(x)	(x)	(x)	x	x	2
MEAS BEFORE LOCA							
indenter	x	x	x	x	x	x	2
IR	x	х	x	x	х	x	2
loss factor	x	x	х	х	x	x	2
capacitance	x	x	х	x	x	x	2
MEAS DURING LOCA							
IR	x	x	x	x	x	x	2
loss factor	x	x	x	x	x	x	2
MEAS AFTER LOCA							
IR	x	x	x	x	x	x	2
loss factor	x	x	x	x	x	x	2
capacitance	x	x	x	x	x	x	2
indenter	x	x	x	x	x	x	2

#### 9.3 Test results

Table 9.2 shows the results of measurement of degradation parameters after thermal and humidity aging and after only thermal aging before irradiation. Table 9.3 shows the results of the measurements after irradiation.

<u>Table 9.2</u> Results of measurement of degradation parameters, before irradiation. All measurements made after aging at  $95^{\circ}C$ 

Humidity	Duration	indenter	IR	loss	loss factor	capacitance	capacitance
%	days	ratio	Mohm <sup>1)</sup>	factor	1 kHz	pF/m, 60	pF/m, 1 kHz
				60 Hz		Hz	
low	48	1,7280	1110	0,026	0,033	195	186
low	96	1,9221	1110	0,028	0,034	199	189
low	192	2,1263	1190	0,029	0,035	199	189
low	384	2,0231	1170	0,034	0,036	196	185
>95	24	1,7057	1215	0,036	0,041	197	185
>95	48	1,7529	1215	0,037	0,042	196	184

1) normalised to cable length 1m

<u>Table 9.3</u> Results of measurement of degradation parameters, after irradiation. All measurements made after aging at  $95^{\circ}C$ 

Humidity	Duration	indenter	IR	loss factor	loss factor	capacitance	capacitance
%	days	ratio	Mohm <sup>1)</sup>	60 Hz	1 kHz	pF/m, 60 Hz	pF/m, 1 kHz
low	48	1,8879	99000	0,050	0,036	201	189
low	96	1,9946	119700	0,052	0,038	206	193
low	192	2,1165	103500	0,051	0,036	200	192
low	384	2,3523	105300	0,058	0,038	205	191
>95	24	1,9681	110700	0,066	0,048	201	186
>95	48	2,0818	99000	0,077	0,050	205	188

1) normalised to cable length 1m

The irradiation has a significant effect on the dielectric loss factor and on the capacitance at 60 Hz, both on cables thermally aged in dry air and on cables thermally aged in humid air. There is a tendency that the loss factor at 60 Hz increases more for the cables aged in humid air.

Table 9.4 shows results of measurement of dielectric parameters in terms of maximum loss factors and minimum insulation resistance values during LOCA.

Table 9.4 Results of measurement of degradation parameters during LOCA

Temp	Humidity	Duration	IR	loss factor	loss factor
°C	%	days	Mohm <sup>1)</sup>	60 Hz	1 kHz
95	low	48	0,3564	15,6	2,00
95	low	96	0,2700	17,3	2,41
95	low	192	0,1107	33,3	9,55
95	low	384	0,055	27,0	6,35
95	>95	24	0,0252	16,29	2,28
95	>95	48	0,0234	16,73	2,35

1) normalised to cable length 1m

The insulation resistances are significantly lower during LOCA for the cables which have undergone humidity exposure as part of the thermal aging. The difference is not as pronounced for the dielectric loss factor, but it is still somewhat higher for the cable pieces which were exposed to humidity during the thermal aging.

# 10 Testing for evaluation of the influence of vibration as part of the long-term exposure

#### 10.1 Purpose

The results of the earlier tests, presented in ref. /1/ indicated no significant increase of the degradation due to exposure to vibration as part of the thermal aging. The components which had been subjected to vibration were included in the LOCA test presented here, with the purpose of evaluating to what extent the additional degradation

because of possible cracks due to the vibration in the aging program had an influence on the dielectric behavior during LOCA.

#### 10.2 Test program

Table 10.1 shows the test program used for study of the influence of vibration.

<u>Table 10.</u>	<u>1</u> Test program	n for evaluatior	n of the dielect	ric behavior at	LOCA of
	test specimen	which have bee	en subjected to	vibration and	thermal aging

Conditioning	Gro	up of samples	s, No	Number of
				samples
PREHISTORY	1b	27	29	kab d/ kabvib
aging temperature/duration	120/48	120	120	
vibration	-	3,5mm	7mm	
Measurements:				
indenter	x	x	x	2
IR/loss factor/capacitance	x	x	x	2
IRRADIATION	(x)	(x)	x	2
MEAS BEFORE LOCA				
indenter	x	x	x	2
IR/loss factor/capacitance	x	x	x	2
MEAS DURING LOCA				
IR/loss factor	x	x	x	2
MEAS AFTER LOCA				
indenter	X	x	X	2
IR/loss factor/capacitance	x	x	X	2

#### 10.3 Test results 10.3.1 Cable Lipalon

#### 10.3.1.1 Irradiation

Table 10.2 shows the results of measurement of degradation parameters after thermal and vibration aging and after only thermal aging before irradiation. Table 10.3 shows the results of the measurements after irradiation. Insulation resistances and capacitances are normalised to 1m (the lengths of the cables subjected to both vibration and thermal aging were 0,5m, the length of the cables subjected to only thermal aging were 3m).

<u>Table 10.2</u> Cable Lipalon. Results of measurement of degradation parameters, before irradiation. All measurements made after aging at  $120 \,^{\circ}C$ 

Vibration	Duration days	indenter ratio	IR Mohm <sup>1)</sup>	loss factor 60 Hz	loss factor 1 kHz	capacitance pF/m, 60 Hz	capacitance pF/m, 1 kHz
•	48	2,0585	1197	0,048	0,035	198,2	563,0
3,5	48	2,1831	185	0,043	0,034	189,0	182,6
7	48	2,2875	183	0,044	0,037	187,6	176,8

1) normalised to cable length 1m

<u>Table 10.3</u> Cable Lipalon. Results of measurement of degradation parameters, after irradiation. All measurements made after aging at  $120 \,^{\circ}C$ 

Vibration	Duration days	indenter ratio	IR Mohm <sup>1)</sup>	loss factor 60 Hz	loss factor 1 kHz	capacitance pF/m, 60 Hz	capacitance pF/m, 1 kHz
	48	2,1479	106200	0,073	0,037	202,9	190,4
3,5	48	2,4215	20500	0,078	0,047	208,4	184,4
7	48	2,5284	20000	0,093	0,073	222,4	202,6

1) normalised to cable length 1m

There is no significant difference in the influence of the irradiation on cables aged with and without vibration.

#### 10.3.1.2 LOCA simulation

Diagram 10.1 shows the results of the measurements of insulation resistance (IR) during and after LOCA and capacitance after LOCA for the cables aged at 120°C for 48 days without vibration (kab d) and for the cables aged at 120°C for 48 days with vibration (kabvib). The IR values during LOCA are the lowest values, normally occurring at the end of the first phase of the LOCA with the highest temperature.





The insulation resistances are significantly lower during and after LOCA for the cables which have undergone vibration exposure as part of the thermal aging. The capacitance is significantly higher for the cables which were vibrated as part of the aging.

Diagram 10.2 shows the results of the measurements of loss factors during and after LOCA for the cables aged at 120°C for 48 days without vibration (kab d) and for the cables aged at 120°C for 48 days with vibration (kabvib). The loss factor values during LOCA are the highest values, always occurring at the end of the first phase of the LOCA with the highest temperature.



<u>Diagram 10.2</u> Cable Lipalon. Comparison of cables without and with vibration as part of the aging. Loss factors during LOCA.

The inclusion of vibration has significantly increased the loss factor.

#### 10.3.1.3 Conclusions

The inclusion of vibration in the aging of Lipalon cables results in significantly poorer dielectric behavior during LOCA. This is the case both for the insulation resistance and for dielectric losses.

#### 10.3.2 Cable Dätwyler

#### 10.3.2.1 Irradiation

Table 10.4 shows the results of measurement of degradation parameters after thermal and vibration aging and after only thermal aging before irradiation. Table 10.5 shows the results of the measurements after irradiation.

<u>Table 10.4</u> Cable Dätwyler. Results of measurement of degradation parameters, before irradiation. All measurements made after aging at 120 °C during 48 days

Vibration	indenter ratio	IR Mohm <sup>1)</sup>	loss factor 20 Hz	loss factor 60 Hz	loss factor 1 kHz	capacitance pF/m, 20 Hz	capacitance pF/m, 60 Hz	capacitance pF/m, 1 kHz
-	1,194	299700	0,0732	0,0145	0,0097	150	148	146
3,5	1,464	15500	0,4171	0,0237	0,0126	128	119	117
7	1,468	13000	0,4099	0,0226	0,0125	129	122	119

1) normalised to cable length 1m

<u>Table 10.5</u> Cable Dätwyler. Results of measurement of degradation parameters, after irradiation. All measurements made after aging at 120 °C during 48 days

Vibration	indenter ratio	IR Mohm <sup>1)</sup>	loss factor 20 Hz	loss factor 60 Hz	loss factor l kHz	capacitance pF/m, 20 Hz	capacitance pF/m, 60 Hz	capacitance pF/m, 1 kHz
	1,630	299700	0,0216	0,0175	0,0122	153	152	148
3,5	1,948	11250	0,0191	0,0148	0,0110	138	137	134
7	1,875	12500	0,0190	0,0146	0,0110	140	139	136

1) normalised to cable length 1m

There is no consistent difference in the influence on dielectric parameters of the irradiation on cables aged with and without vibration.

#### 10.3.2.2 LOCA simulation

Diagram 10.3 shows the results of the measurements of insulation resistance (IR) during and after LOCA and capacitance after LOCA for the cables aged at 120°C for 48 days without vibration (kab d) and for the cables aged at 120°C for 48 days with vibration (kabvib). The IR values during LOCA are the lowest values, normally occurring at the end of the first phase of the LOCA with the highest temperature.



<u>Diagram 10.3</u> Cable Dätwyler. Comparison of cables without an with vibration as part of the aging. Insulation resistance during and after LOCA and capacitance after LOCA

The insulation resistances are significantly lower during LOCA for the cables which have undergone vibration exposure as part of the thermal aging. The other parameters do not show any decrease in dielectric conditions due to the vibration.

Diagram 10.4 shows the results of the measurements of loss factors during and after LOCA for the cables aged at 120°C for 48 days without vibration (kab d) and for the cables aged at 120°C for 48 days with vibration (kabvib). The loss factor values during LOCA are the highest values, always occurring at the end of the first phase of the LOCA with the highest temperature.



<u>Diagram 10.4</u> Cable Dätwyler. Comparison of cables without and with vibration as part of the aging. Loss factors during LOCA.

The loss factor during LOCA is 3-5 times higher for the cables which have been subjected to vibration.

#### 10.3.2.3 Conclusions

The inclusion of vibration in the aging of Dätwyler cables results in significantly lower insulation resistances and significantly higher dielectric losses during LOCA.

#### 10.3.3 Cable Rockbestos

#### 10.3.3.1 Irradiation

Table 10.6 shows the results of measurement of degradation parameters after thermal and vibration aging and after only thermal aging before irradiation. Table 10.7 shows the results of the measurements after irradiation.

<u>Table 10.6</u> Cable Rockbestos. Results of measurement of degradation parameters, before irradiation. All measurements made after aging at 120 °C during 48 days

Vibration	indenter ratio	IR Mohm <sup>1)</sup>	loss factor 20 Hz	loss factor 60 Hz	loss factor 1 kHz	capacitance pF/m, 20 Hz	capacitance pF/m, 60 Hz	capacitance pF/m, 1 kHz
	1,277	268200	0,0681	0,0020	0,0011	126	125	124
3,5	1,663	7675	0,4352	0,0019	0,0029	113	118	107
7	1,635	7450	0,4410	0,0020	0,0010	112	106	106

1) normalised to cable length 1m

<u>Table 10.7</u> Cable Rockbestos. Results of measurement of degradation parameters, after irradiation. All measurements made after aging at 120 °C during 48 days

Vibration	indenter ratio	IR Mohm <sup>1)</sup>	loss factor 20 Hz	loss factor 60 Hz	loss factor 1 kHz	capacitance pF/m, 20 Hz	capacitance pF/m, 60 Hz	capacitance pF/m, 1 kHz
-	1,528	375300	0,0020	0,0010	0,0013	127	127	127
3,5	2,340	10000	0,0023	0,0014	0,029	126	126	125
7	2,353	9575	0,0010	0,0010	0,0027	122	123	1122

1) normalised to cable length 1m

There is no systematic trend in the influence on dielectric parameters of the irradiation on cables aged with and without vibration.

#### 10.3.3.2 LOCA simulation

Diagram 10.5 shows the results of the measurements of insulation resistance (IR) during and after LOCA and capacitance after LOCA for the cables aged at 120°C for 48 days without vibration (kab d) and for the cables aged at 120°C for 48 days with vibration (kabvib). The IR values during LOCA are the lowest values, normally occurring at the end of the first phase of the LOCA with the highest temperature.



<u>Diagram 10.5</u> Cable Rockbestos. Comparison of cables without and with vibration as part of the aging. Insulation resistance during and after LOCA and capacitance after LOCA

The insulation resistances are significantly lower during and after LOCA for the cables which have undergone vibration exposure as part of the thermal aging. The capacitance is slightly higher at 20 and 60 Hz for the cables which have been subjected to vibration.

Diagram 10.6 shows the results of the measurements of loss factors during and after LOCA for the cables aged at 120°C for 48 days without vibration (kab d) and for the cables aged at 120°C for 48 days with vibration (kabvib). The loss factor values during LOCA are the highest values, always occurring at the end of the first phase of the LOCA with the highest temperature.



<u>Diagram 10.6</u> Cable Rockbestos. Comparison of cables without and with vibration as part of the aging. Loss factors during LOCA.

The loss factor during LOCA is slightly higher for the cables which have been subjected to vibration.

#### 10.3.3.3 Conclusions

The inclusion of vibration in the aging of Rockbestos cables results in significantly lower insulation resistance, slightly higher capacitance and slightly higher dielectric losses during LOCA.

# **11 Oxygen induction time or temperature and microcalorimetry evaluation**

#### 11.1 Background

The mechanisms behind aging vary with type of insulation materials. The chemical changes due to aging depend on the composition of the material, including type of polymer, filler, softener and presence and type of antioxidant. As a complement to the studies of mechanical and dielectrical measurements, the insulation materials of the cable jackets and conductor insulations have been subjected to measurement of chemical changes due to the aging. Two methods have been applied - oxygen induction time or temperature (OIT or OITe) and microcalorimetry.

The OIT and OITe methods use a differential scanning calorimeter (DSC 7 from Perkin-Elmer). OIT indicates the time needed for consuming the remaining antioxidants in a material under elevated temperature and in pure oxygen atmosphere (induction time). OITe indicates the temperature at which a significant isothermal reaction is initiated (induction temperature). The microcalorimetry method uses an instrument capable of indicating very small heat flows associated with slow aging processes in the materials.

A first study of OIT and OITe was made in order to get qualitative indications of the applicability and comparison with the results of the mechanical degradation measurements. A second study of OIT was performed on the conductor insulation materials for the Dätwyler and Rockbestos cables, primarily for getting more quantitative data and to compare with the dielectric behavior during LOCA.

#### 11.2 Results, measurements of OIT and OITe, first study

A detailed description of the methodology used in both studies and of the results of the first study is given in Appendix A03 (in Swedish).

The EPDM materials in the Dätwyler jacket and conductor insulation as well as the PEX material in the Rockbestos conductor insulation include an antioxidant, identified as TMQ (polymerized 2,4,4-trimetyl-1,2-dihydrokinoline). The insulation materials of the Lipalon jacket and conductors and the insulation material of the Rockbestos jacket, CSPE, don't contain any significant amount of primary antioxidants. No induction time can be detected in such materials.
Table 11.1 summarizes the results of the measurements of induction times and induction temperatures on unaged and thermally aged cable samples (95°C,384 days and 142°C, 48 days).

Cable type	Insulation	Induction time	Induction temperature
Lipalon	Jacket ins.	None	Independent of aging
	Conductor ins.	None	No exoterm reaction
Dätwyler	Jacket ins.	Not well defined	Not significantly dependent on the aging
	Conductor ins.	Well defined and significant variation with the degree of aging	Significant variation of the induction temperature associated with the first of two exoterms
Rockbestos	Jacket ins.	Only detectable for the unaged sample	Significant variation with the aging
	Conductor ins.	Well defined and significant variation with the aging	Only evaluated for the unaged sample

Table 11.1 Results of measurements of OIT and OITe

A comparison with the results of the indenter measurements presented earlier in this report shows that the OIT method works where the indenter method doesn't indicate any significant changes and vice versa.

- Lipalon, jacket and conductor insulation: very significant influence of the aging on indenter measure, no influence on OIT or OITe.
- Dätwyler, conductor insulation: little influence of the aging on indenter measure, very significant influence on OIT.
- Rockbestos, jacket insulation: very significant influence of the aging on indenter measure, little influence on OITe.
- Rockbestos, conductor insulation: little influence of the aging on indenter measure, very significant influence on OIT.

The jacket insulation of the Dätwyler cable is the only application where neither indenter measure nor OIT or OITe give a very pronounced indication of the effects of aging. The results of the OIT measurements are in this case surprising, since the combination of EPDM and TMQ normally results in well defined induction times, as was the case for the Dätwyler conductor insulation.

#### 11.3 Results, measurements of OIT, second study

The results of the OIT measurements in the second study is reported in detail in Appendix A04 (in Swedish). The study included insulation materials of conductors from cables which had been subjected to the following thermal aging:

80°C for 192, 384 and 576 days 95°C for 96, 192 and 384 days 120°C for 48, 96 and 192 days 142°C for 12, 24 and 48 days

As "unaged" material was used material which had been stored in normal laboratory conditions. Six "unaged samples and various number of samples of each of the aged cable materials were tested.

Measurements were first made to find suitable test temperatures, for Dätwyler (EPDM) 205°C, for Rockbestos (cross-linked polyethylene) 235°C. Diagrams 11.1 and 11.2 summarize the results in terms of the mean values of the induction times as function of aging durations for the various aging temperatures.



<u>Diagram 11.1</u>. Conductor insulation material for cable Dätwyler. OIT as function of aging temperature and aging duration



<u>Diagram 11.2</u>. Conductor insulation material for cable Rockbestos. OIT as function of aging temperature and aging duration

For both Dätwyler and Rockbestos conductor insulations, no significant influence of the thermal aging at 80°C on the OIT was shown. For the Dätwyler conductor insulation this was the case also for cables aged at 95°C.

Conductor insulations from cables aged at higher temperatures (for Dätwyler at 120 and 142°C, for Rockbestos at 95, 120 and 142°C) show a more clear decrease of the induction time with increase in aging duration. For the material which had been aged at 142°C for 24 and 48 days, the induction time dropped to zero. This was also the case for the Rockbestos conductor insulation material aged for 192 days at 120°C.

It is of interest to compare the OIT values of conductor insulations of aged cables with the dielectric behavior of the identically aged cables during LOCA. Diagram 11.3 and 11.4 show the relationships for the Dätwyler cable and the Rockbestos cable, respectively. The points in the diagrams are related to cables aged at 95, 120 and 142°C, where values for both dielectric loss factors of cables during LOCA (phase I) and OIT values are available.



<u>Diagram 11.3</u> Relationships between OIT values of Dätwyler conductor insulation materials of aged cables and dielectric loss factors at LOCA</u>



<u>Diagram 11.4</u> Relationships between OIT values of Rockbestos conductor insulation materials of aged cables and dielectric loss factors at LOCA

The more degraded cables are supposed to have shorter OIT times and higher dielectric loss factors. But the diagrams show no clear relationships where there is a trend that lower OIT of the conductor insulation material correspond to higher loss factors of the cable during LOCA.

#### 11.4 Results of the microcalorimetry measurements

The microcalorimetric measurements were performed at Celsius Materialteknik in Karlskoga. A thermal activity monitor (TAM) from Thermometric was used. The measurement technique and the results are presented in detail i Appendix A03. A summary of the major results is given below. All measurements were made on the conductor insulation materials.

Measurements on the Lipalon conductor insulation material has been made on materials from cables which have been stored under normal laboratory conditions, cables which have been aged for 384 days at 95°C, cables which have been aged for 48 days at 142°C and cables which have been subjected to LOCA testing.

Measurements on the Dätwyler and Rockbestos conductor insulation materials have only been made on materials from cables which have been stored under normal laboratory conditions and materials which have been aged for 48 days at 142°C.

The results in terms of heat flow, measured at 65°C, 75°C and 85°C, and calculated activation energies are given in table 11.2.

	Heat flow	/, μW/g		Activation energy, eV
	65°C	75°C	85°C	
Lipalon		[	[	
stored	1,1	1,8;2,2	5,3	0,79*
aged 95°C,384d	1,6	4,9	12,9	1,05
aged 142°C,48d	49	129	216	0,95
LOCA-aged	30	77	138	0,88
Dätwyler				
stored	0,38	0,88;1,0	1,93	0,82*
aged 142°C,48d	0,21	0,75	1,53	1,00*
Rockbestos				
stored	0,56	1,36;1,57	2,72	0,80*
aged 142°C,48d	-	0,69	1,72	0,95*

<u>Table 11.2</u> Results of microcalorimetry measurements on conductor insulation materials

\* unreliable values due to low heat flow

It can be seen from the table that very significant heat flows are achieved for the aged Lipalon conductor insulation material (CSPE), for which it was not possible to achieve significant results from the OIT or OITe measurements. The two methods are shown to be complementary.

# **12** Conclusions

### 12.1 Extended thermal aging

Extended thermal aging of cables, cable leads and o-rings has been performed at 120°C (144 days and 192 days) and at 142°C (24 days and 48 days). This represents very extreme conditions, in excess of reasonable qualification for installation in the containment of nuclear power plants. The purpose has been to get the components in conditions of degradation due to thermal aging beyond the limit for proper operation in LOCA.

A good linear relationship was found between duration of aging at 120°C and at 142°C and degradation in terms of indenter values. The same was the case when compared to elongation-at-break ratios. In the latter case the analysis could only be made for some of the cables and temperatures because of problems with the very fragile cables after the most severe thermal aging. It is obviously an advantage to use indenter measurements in testing to very severe conditions, compared to using elongation-at-break ratios, since indenter values can be achieved also for highly degraded cables.

The earlier tests, presented in ref. /1/ were performed under conditions which didn't result in severe degradation of the insulating material. Therefore the measurements of dielectric parameters didn't show substantial changes with aging temperature and duration. With the additional tests presented in this report the degradation is very severe and shows up very clearly also in the dielectric loss factor values, in the most severe cases also in the insulation resistance values.

The jacket material of the Rockbestos cable (CSPE) shows very severe mechanical degradation when subjected to long term exposure to 120°C and 142°C. Also the jacket materials of the Lipalon cable and leads (CSPE) show very significant degradation, whilst the jacket material and leads of the Dätwyler cable (EPDM) are much less affected. The leads of the Rockbestos cable (XLPE) are only slightly affected.

A comparison of degradation in terms of indenter values between 3m long sealed and 0,3m long unsealed cables shows that the aging of the short, unsealed cables generally results in higher levels of degradation than the aging of the longer, sealed cables. The reason may be that the degradation process has a tendency to start at the ends and propagate successively to the rest of the cable.

The Viton o-rings (FPM) are very little affected by the subjection to 120°C and 142°C for prolonged durations. The EPDM o-rings are significantly affected, both in terms of indenter values and in terms of remaining compression.

### 12.2 Influence of nitrogen atmosphere

The use of nitrogen atmosphere instead of air during excessive thermal aging results in a very significant reduction of degradation of the Lipalon and Rockbestos cable jackets and for the Lipalon cable lead insulation material, which were severely degraded at testing in air. For the Dätwyler cable jacket and lead insulation materials the reduction becomes significant only after the longest exposure (48 days). For the Rockbestos cable lead insulation the reduction is insignificant. The influence of using nitrogen atmosphere is significant for EPDM o-rings but not for Viton o-rings. The latter type of o-rings are not being significantly affected, also not in air.

#### 12.3 Influence of ionizing radiation

The LOCA testing included an irradiation before entering into the LOCA cycle. With the dose used,  $5*10^5$  Gy, the effect on the mechanical properties of the Lipalon and Dätwyler cable jackets are significant.

Also the dielectric loss factors are generally affected for the Lipalon cables. This is the case also for the separately exposed cable leads.

# 12.4 Relationship between degradation due to thermal aging and dielectric behavior during LOCA testing, cable Lipalon

Of primary interest has been to establish the relationships between degradation due to thermal aging and the dielectric behavior during LOCA testing. There is a sinificant but not very pronounced linear relationship between indenter values measured before LOCA and the maximum dielectric loss factors at 1 kHz measured during LOCA. The correlation factor is approximately 0,5. There is a somewhat higher correlation between the indenter values as a measure of the mechanical condition before LOCA and the dielectric behavior after LOCA.

# 12.5 Relationship between degradation due to thermal aging and dielectric behavior during LOCA testing, cable Dätwyler

The results of the study on the Dätwyler cable is that there is a good correlation between the mechanical degradation measured by the indenter and the aging conditions, but the dielectric parameters measured during LOCA are not well related to the degree of aging. The Dätwyler cable is very resistive to thermal aging which hasn't proceeded in our tests to a degree where very significant aging related effects are shown in the dielectrical behavior during LOCA.

# 12.6 Relationship between degradation due to thermal aging and dielectric behavior during LOCA testing, cable Rockbestos

The results of the study on the Rockbestos cable is that there is a good correlation between the mechanical degradation measured by the indenter and the aging conditions, but the dielectric parameters measured during LOCA are not well related to the degree of aging. A rather good correlation is shown between the indenter modulus before LOCA and the dielectric parameters after LOCA (correlation factor approximately 0,85). The dielectric behavior couldn't be measured during LOCA for the most severely degraded cables, but the result indicates a relationship between mechanical degradation measured by the indenter and the dielectric conditions during LOCA.

# 12.7 Validation of earlier results on the benefit of using on-going qualification as part of aging qualification, cable Lipalon

Earlier tests, ref. /1/, had shown that a simulation of a rather long duration (576 days) thermal aging condition at 80°C by a simulated on-going procedure gave better results than a simulation by a short duration (6 days) test at very high temperature (142°C). The cables used for this test were now subjected to irradiation and LOCA. The correlation between the indenter ratios before LOCA testing and the minimum dielectric loss factors during LOCA are rather high (0,95 at 1 kHz, 0,887 at 60 Hz). The conclusions from the earlier tests are thereby verified by the behavior of the dielectric loss factors during LOCA.

#### 12.8 Influence of humidity as part of the thermal aging, cable Lipalon

The results of measurements of dielectric parameters during the LOCA tests of cables which had been subjected to high humidity (>95% RH) during the thermal aging show that the insulation resistances during LOCA are considerably lower compared to those for cables which had not been subjected to high humidity during the thermal aging.

#### 12.9 Influence of vibration as part of the thermal aging

The results of measurements of dielectrical parameters during the LOCA tests of Lipalon cables which had been subjected to intermittent vibration as part of the thermal aging show that the insulation resistances during LOCA are very much lower compared to those for cables which had not been subjected to vibration during the thermal aging. An effect was also shown on the dielectric loss factors.

For the Dätwyler cables significant effects of the inclusion of vibration in the aging is shown both on dielectric loss factors during LOCA and on the insulation resistances during and after LOCA.

For the Rockbestos cables, a significant effect of inclusion of vibration in the thermal aging is shown on insulation resistances during and after LOCA. A slight effect is also shown on dielectric loss factors during LOCA

It should be noticed that vibration levels in excess of what is expected in cables installed in the containment of nuclear power plants were used in the study.

#### 12.10 Measurement of OIT and microcalorimetry

The conductor insulation material in the Lipalon cable (CSPE) shows increasing heat flow with aging. OIT measurements are not useful in this case since the material doesn't include antioxidants. The results indicate differences in the CSPE material of the conductor insulation and in the jacket. The former seems to be better stabilized.

The conductor insulation materials of the Dätwyler cable (EPDM) and the Rockbestos cable (XLPE) are stabilized with antioxidants which are consumed at the aging. The

OIT measurements therefore gave a clear indication of the aging, whilst the heat flow, measured by microcalorimetry is very low as long as the stabilizers have not been consumed.

A detailed second study on the conductor insulation materials of the Dätwyler and Rockbestos cables indicated a clear reduction of oxidation induction time with increased thermal aging, especially in cases of severe combinations of aging temperature and duration. A relationship between OIT of the aged conductors insulations and the dielectric behavior of the cables during LOCA could, however, not be verified.

The conclusions which can be drawn from the results are that OIT is useful for indication of degree of aging as long as stabilizing materials are present in the polymer. Microcalorimetry is useful for studies on unstabilized materials and for stabilized materials where the antioxidant has been consumed. The two techniques are therefore complementary. It is also interesting to note that the OIT measurements seem to be complementary also to the indenter measurements.

#### **13 References**

/1/	Methodology for artificial aging of electrical components in nuclear power plants. Results of experimental studies. K Spång. SKI Technical Report 93:39, December 1993
/2/	Cable Indenter Aging Monitor. T.A. Shook, J.B. Gardner EPRI Report NP-5920, July 1988
/3/	Anordning för oförstörande mätning av åldring hos kablar (in Swedish). P.E. Sanborn. DNV Ingemansson PM H-12278-B, November 1988
/4/	Åldring av kablar i kärnkraftverk. Jämförande mätningar med stationär indenter och bärbar indenter (in Swedish). K. Spång Ingemansson Rapport H-14008-B, July 1997

77

, 977.7

Description of LOCA test in test loop LOKE.

A test specimen is enclosed in a autoclave as seen in figure 1. The test specimen is exposed to the specified environment. In this case KSB TBE 102-3 headed of extreme operation according to KSU TBE 102 table 1. (90°C at atmosphere pressure and 100% relative humidity).



The temperature - pressure conditions is obtained with a system controlling

- •electric heated elements at the pressure vessel
- •saturated steam generation
- •overheating of the steam
- •outlet flow control

LOCA graph, KSU TBE 102-3



		Ref	
MESSAGE		NL 96-035	
Från - From	Datum • Date	Reg.nr	Sida - Page
$\mathbf{NL}$	96-02-28	1 •	1(1)
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Staffan F	Karlsson 473	88 2	
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Measuring equipment for LOCA-testing of SKI cables.

INSULATION RESISTANCE

A.012964.100

Scanner Keithley, type 706 Atom/Lab no 5224

MUX-boards Keithley, type 7054 Atom/Lab no 2179

Powersupply EM, type TCR600S1-2 Atom/Lab no 2474

DMM Keithley, type 195A Atom/Lab no 2323 and 2325

# TEMPERATURE AND PRESSURE

DMM Keithley, type 195A Atom/Lab no 2324 and 2322

Transmitter temperature INOR, type SE42-1B Atom/Lab no 2441

Transmitter pressure Schoppe Faeser, type 15220 Atom/Lab no 6047

# DISSIPATION FACTOR AND CAPACITANCE

Measuring bridge Wayne Kerr, mod. 6425 Atom/Lab no 4288

#### CONTROLLER

Computer Macintosh, mod. Quadra 650 Atom/Lab no 5740



Appendix A03

# **Technical Report**

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Responsible Person:	Anna Kornfeldt		
Project Name:	Åldring av kabelisolation		
Document Title:	Kemiska och kalorimet	triska metoder för studi	um av åldring av
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Summary:

Microcalorimetry and oxygen induction time (OIT) have been evaluated as methods to study aging of insulation materials of cables used in nuclear power plants. Cable sheath materials and conductor insulation of Lipalon, Dätwyler and Rockbestos, which had been stored, exposed to accelerated aging and LOCA-tested, were studied.

The results showed that materials of ethylene-propylene rubber and crosslinked polyethylene are stabilized by antioxidants and OIT can be used to study the aging process. Chlorosulphonated polyethylene rubber does not contain detectable antioxidants. Microcalorimetric measurements showed increased heatflow by increased degree of material aging. Thus the two techniques are complementary for studies of cable aging.

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#### 1. BAKGRUND

Ett pågående projekt inom SKI syftar till att utvärdera metoder för att studera tillståndet hos artificiellt åldrat kabelmaterial som används inom kärnkraftverk. Huvuddelen av projektet behandlar mekaniska och dielektriska metoder. Inledande studier har även gjorts på SECRC för utvärdering av metoder som baseras på att mäta de kemiska förändringar som sker i materialet vid åldringen. Fördelarna med dessa metoder är att mycket lite material behövs för karakteriseringen och mätningarna kan göras även på ledarisoleringen. De här aktuella metoderna är mikrokalorimetri samt bestämning av kvarvarande antioxidanthalt, OIT-bestämning ("Oxygen Induction Time").

Åldringsmekanismerna är olika för olika typer av isolermaterial. De kemiska förändringar som isolermaterialet uppvisar beror på dess kemiska sammansättning, dvs typ och mängd av polymer, fyllmedel, mjukgörare och tillsats av antioxidant och antiradiant samt under vilka förhållanden materialet åldrats. Att kemiskt beskriva ett åldringsförlopp är mycket komplicerat och kräver ingående kunskaper om materialet. De här använda metoderna syftar i stället till att ge en allmän bild av graden av åldring.

OIT-metoden är ett mått på den tid det tar att förbruka kvarvarande antioxidanter i ett material vid accelererad åldring. Tiden för att förbruka kvarvarande mängd antioxidant under förhöjd temperatur och i ren syre atmosfär bestäms. Mikrokalorimetern är ett mycket känsligt instrument för att mäta värmeflöden t.ex. på grund av extremt långsamma åldringsprocesser hos material. Värmeflödet utgör summan av de endoterma och exoterma processer som pågår i materialet. Genom att göra studier i olika miljöer, exempelvis syre eller fukt, och kombinera metoden med andra metoder kan mera specifik information om materialets åldring erhållas.

Tidigare studier med mikrokalorimetri (SECRC/KD/TR-92/9386) visade att denna teknik kan användas för att studera oxidationsreaktioner hos kabelmaterial. Ur värmeflödesdata från temperaturintervallet 65-85°C kunde aktiveringsenergier och syreberoende bestämmas. Dock kunde inte aktiveringsenergin bestämmas för ett lagrat EPDM-material (etenpropengummi). Isolermaterialens reaktion med fukt, kopplade fukt/syreeffekter, liksom syrediffusionens inverkan på oxidationen kunde också studeras.

De här redovisade studierna belyser användningen av OITmätningar och mikrokalorimetri för lagrat och artificiellt åldrat isolermaterial.

#### 2. MATERIAL

Följande typer av isolermaterial har studerats:

Kabeltyp	Mantelmaterial	Ledarisolering
Lipalon	klorsulfonetengummi	klorsulfonetengummi *
Dätwyler	etenpropengummi	etenpropengummi
Rockbestos	klorsulfonetengummi	tvärbunden polyeten

Tabell 1. Isolermaterial hos de undersökt kablarna. \* uppgifter om detta material har varierat.

Ledarmaterialet hos Lipalon uppgavs av ABB Atom vara klorsulfonetengummi (Hypalon även kallat CSM eller CSPE) men i SKI Technical Report 93:39, 2.2 uppges att materialet är etenpropengummi (EPDM). Detta motiverade kemiska analyser vid SECRC, som fastställde att materialet är klorsulfonetengummi (se nedan 5.1).

Tvärbunden polyeten benämns även PEX eller XLPO/XLPE.

De mikrokalorimetriska-mätningarna har i denna studie endast gjorts på ledarisolering, medan vissa OIT-mätningar även har gjorts på mantelmaterial.

Mätningarna har gjorts på material lagrat i kontorsatmosfär samt artificiellt åldrat material. Mätningar som belyser materialvariationer har gjorts för Lipalonkabeln med mikrokalorimetri och för Dätwylerkabel med OIT-teknik.

# 3. ÅLDRING

Den accelererade åldringen har utförts vid ABB Atom. Mätningar har i denna studie gjorts efter följande åldring:

1. Termisk åldring vid 142°C i 48 dygn, för samtliga tre kabeltyper.

- 2. Termisk åldring vid 95°C i 384 dygn, för samtliga tre kabeltyper.
- 3. LOCA (Loss of Cooling Accident) test för Lipalon kabel.

#### 4. METODER

#### 4.1 OIT-mätningar

OIT-mätningarna har utförts med DSC 7 (Differential Scanning Calorimeter) från Perkin-Elmer. Två varianter har använts, dels en dynamisk mätning där en induktionstemperatur bestäms, dels en isoterm mätning där en induktionstid bestäms. Vid den dynamiska mätningen sveps temperaturen från 30°C med 10°/min med O2 som spolgas under hela mätningen. Den temperatur där en kraftig isoterm reaktion sätter in är induktionstemperaturen. Detta värde kan i sig användas för jämförelser men utgör också underlag för val av temperatur för den isoterma (eller statiska) mätningen. Vid den senare värms provet under N<sub>2</sub>-atmosfär snabbt upp till en hög temperatur (typiskt 200°C) varpå gasen switchas till O<sub>2</sub>. Tiden från att syrgasen når mätcellen fram till det att den exoterma reaktionen sätter in är induktionstiden, se bilaga 1. Det som man mäter är alltså tiden för att förbruka antioxidanterna under extremt accelererade förhållanden. Metodens tillämpbarhet varierar mellan olika material. För stabiliserade polyolefiner (PE, PEX, PP etc.) erhålls vanligen mycket väl definierade induktionstider. För vissa andra material (t.ex. en del omättade gummisorter eller ej stabiliserade polymerer) fås ingen skarp induktionstid, eller rentav inte någon exoterm reaktion alls. I en del fall kan man finna en induktionstemperatur som varierar med materialets åldringsgrad, även om någon induktionstid ej går att utvärdera.

#### 4.2 Mikrokalorimetri

De mikrokalorimetriska mätningarna har utförts vid Celsius Materialteknik Karlskoga AB. Instrumentet, TAM (Thermal Activity Monitor) från Thermometric har nyligen införskaffats och är under inkörning på ABB SECRC. För mätningen behövs 1-2 gram prov. Metodiken beskrivs utförligare i bilaga 2.

#### 4.3 Masspektrometri

För att identifiera de primära antioxidanterna som använts har masspektrometri använts. Den enklaste mätningen innebär att en liten mängd prov (1 mg eller mindre av obehandlat provmaterial) introduceras direkt i spektrometerns jonkälla och uppvärms m.h.a. en s.k. solids probe ("direktinsläpp", "direct insertion probe"). Masspektra registeras med några sekunders mellanrum. Metoden är vanligen fullt tillräcklig för att identifiera de viktigaste tillsatserna i ett någorlunda rent material. För mer komplexa prov kan det vara fördelaktigt att först extrahera materialet och sedan separera provkomponenterna med en gas- eller vätskekromatograf kopplad direkt till masspektrometern.

#### 5. RESULTAT

#### 5.1 Identifiering av Lipalon ledarisolering

Lipalon ledarisolering som enligt uppgift skulle vara EPDM visade ej det förväntade beteendet vid DSC-OIT-mätningar. Ej heller visade det sig innehålla någon av de antioxidanter man förväntar sig att finna i EPDM. Därför gjordes några analyser för att fastställa materialets identitet. Ett IR-spektrum av pyrolysprodukter visade mycket god överensstämmelse med biblioteksspektra för klorsulfonetengummi. Dock har flera gummisorter mycket likartade spektra varför även pyrolys-GC-MS gjordes på materialet. Den senare analysen visade ett helt annat spektrum av pyrolysprodukter än för ett referensmaterial av EPDM. Dessutom fanns bland de mest lättflyktiga produkterna sådana som visade mycket god spektral överensstämmelse med såväl HCl som SO<sub>2</sub>. Vi drar därför slutsatsen att <u>materialet i Lipalon ledarisolering är klorsulfonetengummi</u> vilket stämmer med tidiga uppgifter från ABB Atom.

#### 5.2 Identifiering av antioxidanter

De vanligen förekommande typerna av primära antioxidanter är s.k. hindrade fenoler (oftast parasubstituerade 2,6di*tert*butylfenoler), polymeriserad 2,4,4-trimetyl-1,2dihydrokinolin ("TMQ"), N,N'-substituerade parafenylendiaminer eller olika substituerade difenylaminer. Totalt finns tiotals olika föreningar, varav många under flera handelsnamn.

För samtliga klorsulfonetengummimaterial (Lipalon mantel och isolering, samt Rockbestos mantel) gäller att ingen av de ovan nämnda typerna av antioxidant kunde påvisas. Att döma av deras beteeende vid OIT-mätningar (se nedan) samt resultaten från mikrokalorimetriska mätningar bedömer vi det som sannolikt att de innehåller antingen ingen eller endast spårmängder av primär antioxidant av vanligt slag. Dessa material innehåller dock en stor andel olja, både mineralolja (med ett kraftigt inslag av paraffiner) och vad som utifrån masspektra förefaller vara en syntetisk olja med betydande aromatinslag. Det är tänkbart att båda dessa, kanske framför allt den senare, innehåller sådana föreningar vars oxidationsprodukter fungerar som antioxidanter, analogt med vad som händer t.ex. i en oinhiberad transformatorolja, där vissa aromatiska föreningar oxideras till fenoler vilka sedan har en inhiberande verkan på fortsatt oxidation. Oxidationsförloppet är då annorlunda jämfört med det för en inhiberad olja (eller stabiliserad polymer). Det förekommer t.ex. ingen induktionstid, utan förloppet är accelererande från första början.

I båda EPDM-materialen (Dätwyler mantel och isolering) samt i Rockbestos ledarisolering (PEX) kunde påvisas en förening som är en karaktäristisk komponent i TMQ, som är en mycket vanlig antioxidant i gummimaterial.

Direktinsläppsmetoden och GC-MS gav i samtliga fall samma besked.

5.3 OIT-mätningar Minst två mätningar har i regel gjorts på varje prov. Dubbel och trippelprov redovisas. Där flera mätningar gjorts redovisas medelvärde och standardavvikelse.

5.3.1 Lipalon Lipalon mantel.

Induktionstemp/°C
264, 265
264, 264
-

Tabell 2.

Den exoterma reaktionens induktionstemperatur tycks ej bero av åldringsgraden och reaktionen är med största sannolikhet ej betingad av förlust av en syntetisk antioxidant. För prov åldrat vid 142°C går ej att urskilja någon exoterm reaktion.

#### Lipalon isolering

Ingen som helst exoterm reaktion kunde iakttas under en dynamisk mätning, oavsett åldringsgrad.

#### 5.3.2 Dätwyler Dätwyler mantel.

Två exotermer kunde iakttas vid dynamisk mätning. Ingen av dessa tycks dock ändras med åldringsgraden. Det var endast för ej åldrat prov som någon induktionstid kunde bestämmas, och även denna var ej särskilt väl definierad.

	OIT (200°C)	Ind. temp. 1	Ind. temp. 2
ej åldrat	12.0, 10,7 , 10.5	221	326
95°	<i></i>	230	332
142°	-	223	327

Tabell 3. Dätwyler mantelmaterial

Resultaten är förvånande med tanke på att denna kombination av material och antioxidant (EPDM-gummi med TMQ) brukar ge väldefinierade induktionstider, se t.ex. Dätwyler isolering nedan.

## Dätwyler isolering.

Två exotermer kunde iakttas vid dynamisk mätning. Endast den vid lägre temperatur varierade dock med åldringsgraden. Ingen statisk OIT associerad med den högre temperaturen kunde bestämmas. Däremot erhölls en väldefinierad induktionstid för den första exotermen.

OIT (210°C)	Ind. temp. 1	Ind. temp. 2
17.0 (s=5.2, n=13)	232	326
6.1 (s=1.4, n=4)	222	322
<0.4	205	322
	OIT (210°C) 17.0 (s=5.2, n=13) 6.1 (s=1.4, n=4) <0.4	OIT (210°C) Ind. temp. 1   17.0 (s=5.2, n=13) 232   6.1 (s=1.4, n=4) 222   <0.4

Tabell 4.

Spridningen i uppmätta induktionstider är tämligen stor, åtminstone i nytt material. För mer detaljer se under Materialvariationer nedan.

#### 5.3.3 Rockbestos

#### Rockbestos mantel

	OIT (260 °C)	Induktionstemp (°C)
ej åldrat	7.0, 7.4	288, 287
95°	-	268, 277
142°		≈230 (diffus)

Tabell 5.

Eftersom det tycktes finnas en visst samband mellan induktionstemperatur och åldringsgrad gjordes ett försök att bestämma induktionstider. Det var dock endast för ej åldrat prov som någon induktionstid gick att urskilja.

#### **Rockbestos isolering**

	OIT (240°C)	Induktionstemp
ej åldrat	15.3, 16.4	267
95°	11.7, 9.3	
142°	<0.4	

Tabell 6.

Detta material verkar vara väl stabiliserat (jämför mikrokalorimetriresultaten) och visar det typiska beteendet för stabiliserade polyolefiner, med en skarpt definierad induktionstemperatur.

#### 5.3.4 Materialvariationer

Två slags variationer inom material undersöktes. Dels togs prover av ledarisolering från flera ställen längs en Dätwylerkabel, dels gjordes ett försök att påvisa skillnader mellan olika djup hos Dätwyler mantelmaterial.

#### Variationer längs kabel, samt inom korta segment

OIT för ledarisoleringen på tre slumpvis urvalda ställen längs en ej åldrad (d.v.s. kontorsmiljöåldrad) Dätwylerkabel.

pos:	1	2	3
OIT, 210°C	16.1, 19.8, 24.2, 19.9,	14.5, 18.4, 18.2	7.9, 22.0, 21.1,
(min)	21.2 medel=20.2	medel=17.0	8.8, 8.7 medel=13.7
S	2.9	2.2	7.2

Tabell 7.

Det är tveksamt om det verkligen föreligger någon signifikant skillnad mellan olika ställen på kabeln, med tanke på de stora variationer som finns mellan intillliggande individuella prover. Dessa variationer återspeglar troligen en ojämn fördelning av antioxidanter i materialet och torde endast till en mindre del ligga i mätmetoden.

#### Materialpåverkan på olika djup

Dätwyler mantelmaterial var det enda mantelmaterial för vilket det bedömdes meningsfullt att försöka studera skillnader i materialpåverkan mellan olika djup, med tanke på övriga mantelmaterials natur och dimensioner. De övriga var tunnare, dessutom av klorsulfonetengummi. Ett prov åldrat vid 95° undersöktes. Mätningarna gjordes dynamiskt då det redan var klart att ingen induktionstid kunde bestämmas för åldrade prover. Prov togs från ca. 1, 2 och 3mm djup.

djup:	1mm	2mm	3mm
induktions-	224, 230	232, 225	230, 227
temp. (°C)			

Tabell 8. Induktionstemperaturer för Dätwyler mantelmaterial på tre olika djup för en Dätwylerkabel åldrad vid 95℃.

Vi tycks alltså med denna metod ej kunna påvisa någon skillnad mellan olika djup för detta åldrade material.

Noterbart är att alla tre klorsulfonetengummimaterialen uppvisar olika beteende. Det alla har gemensamt är att eventuella exoterma reaktioner är ganska svaga och att inga användbara induktionstider går att bestämma. Dock kan för Rockbestos mantelmaterial möjligen viss information fås ur induktionstemperaturen.

## 5.4 Mikrokalorimetri-mätningar

### 5.4.1 Lipalon ledarisolering

De mikrokalorimetriska mätningarna visade ett ökat värmeflöde för de åldrade kablarna. Värmeflödet var mycket lågt för kablar som endast lagrats och bestämningen av aktiveringsenergi är därför väldigt osäker. För dessa material var den beräknade aktiveringsenergin för den oxidativa åldringen orealistiskt låg och troligen beror värmeflödet på någon annan process. Tänkbart är att den kan bero på oxidation av i materialet tillsatta oljor.

Värmeflöde µW/g			E <sub>a</sub> kJ/mol	
Lipalonkabel	65°C	75°C	85°C	Oxidativ process
Lagrad	1.1	1.8; 2.2	5.3	79 *
Åldrad 95°C 384d	1.6	4.9	12.9	105
Åldrad 142°C 48d	49	129	216	95
LOCA-åldrad	30	77	138	88
Lagrad; endast O <sub>2</sub>	1.5	3.3; 4.4	9.6	93 **

Tabell 9. Värmeflöde och aktiveringsenergier för oxidativa processerför lagrad och åldrad Lipalon ledarisolering .\* osäkra värden pågrund av lågt värmeflöde.\*\* ej endast oxidativa processer.

Intressant att notera är att värmeflödet ökade mest för Lipalonmaterial som åldrats vid 142°C i 48 dygn medan den oxidativa aktiveringsenergin för det haveritestade materialet är lägre. Det är rimligt att förvänta sig att värmeflödet ökar med ökad grad av åldring, vilket betyder att kabeln åldrad vid 142°C i 48 dygn är sämre än den LOCA-testade kabeln. Att dra några slutsatser beträffande de varierande aktiveringsenergierna är däremot svårt eftersom flera olika nedbrytningsreaktioner förväntas pågå i materialet och dessa kan variera med tid och miljö.

## 5.4.2 Dätwyler ledarisolering

Värmeflödet för ledarisoleringen i Dätwyler, dvs etenpropengummi, var mycket lågt, såväl för lagrat som åldrat kabelmaterial. Bestämningen av aktiveringsenergier är därför osäker. Orsaken är troligen att den oxidativa åldringen är mycket liten p.g.a. att det fortfarande finns kvar antioxidanter i materialet, även om de i materialet åldrat vid 142° inte gick att påvisa vid OITmätningarna.

Värmeflöde µW/g				E <sub>a</sub> kJ/mol
Dätwylerkabel	65°C	75°C	85°C	Oxidativ process
Lagrad	0.38	0.88; 1.0	1.93	82 *
Åldrad 142°C 48d	0.21	0.75	1.53	100 *

Tabell 10. Värmeflöde och aktiveringsenergier för oxidativa processer för lagrad och åldrad Dätwyler ledarisolering. \* osäkra värden på grund av lågt värmeflöde.

### 5.4.3 Rockbestos ledarisolering

Värmeflödet för ledarisoleringen i Rockbestos, dvs tvärbunden polyeten, var liksom för etenpropengummi mycket låg och värdena för aktiveringsenergier är därför osäkra. Även här finns sannolikt en rest av verksam antioxidant, t.o.m. i materialet åldrat vid 142°.

Värmeflöde µW/g				E <sub>a</sub> kJ/mol
Rockbestoskabel	65°C	75°C	85°C	Oxidativ process
Lagrad	0.56	1.36;1.57	2.72	80 *
Åldrad 142°C 48d	-	0.69	1.72	95 *

Tabell 11. Värmeflöde och aktiveringsenergier för oxidativa processer för lagrad och åldrad Rockbestos ledarisolering. \* osäkra värden på grund av lågt värmeflöde.

#### 5.4.4 Syrekänslighet

Materialens syrekänslighet har studerats dels genom att oxidationskurvorna för solida och finmalda material har jämförts och dels genom att bestämma värmeflödet för Lipalon vid olika O<sub>2</sub>-tryck.

Resultaten visar för ledarmaterial av Lipalon (klorsulfonetengummi) och Rockbestos (tvärbunden polyeten) att de processer som ger värmeflöde pågår homogent i provet och är ej specifikt kopplade till ytan. För Dätwyler (etenpropengummi) var kvoten värmeflöde (solid/mald) nära 0.5, vilket tyder på att det är skillnader på de processer som pågår vid ytan och längre in i materialet.

Värmeflödesbestämningarna vid olika syretryck gjordes på Liplon som åldrats vid 95°C. Mätningarna blir därigenom säkrare och inverkan av icke-åldringsrelaterade processer i materialet blir mindre. Mätningar gjordes vid 0 (ren kvävgas), 0.2, 0.6 och 1.0 bar O<sub>2</sub>. Resultaten visar att oxidationhastigheten för Lipalons ledarisolering är beroende av syretrycket men ej helt linjärt. Värmeflödesökningen blir något lägre vid ökat syretryck.

#### 5.4.5 Materialvariationer

Mätningar av värmeflödet hos material från Lipalonledare i ren  $O_2$  taget från 8 olika ställen på en kabelvinda gav ett medelvärde resp. standardavvikelse för värmeflödet på 9.7 och 0.4  $\mu$ W/g. Detta inkluderar givetvis både material och metodspridning.

#### 5.4.6 Termisk åldring

Försök gjordes med Lipalon ledarisolering , åldrat vid 142°C och LOCA-testat, samt Rockbestos, icke-åldrat och åldrat vid 142°C, att bestämma den termiska åldringens aktiveringsenergi, baserat på värmeflödesmätningar i ren kvävgas. Värmeflöde som funktion av omsatt energi (P(E)-plott) bestämdes. Det var emellertid inte möjligt att beräkna den termiska aktiveringsenergin och slutsatsen dras att det uppmätta värmeflödet i ren kvävgas ej är förknippat med några kemiska reaktioner i materialet.

#### 6. DISKUSSION

Resultaten visar att för Lipalon ledarisolering sker en åldringsprocess som ger ökat värmeflöde. De övriga ledarmaterialen är stabiliserade med antioxidanter och åldringen påverkar värmeflödet mycket lite medan antioxidanter finns kvar i materialet. Erfarenheter visar att oxidationsförloppet accelererar dramatiskt då antioxidanten förbrukats, med bl.a. kraftigt ökad värmeutveckling som följd. Detta gör att mikrokalorimetrin troligen också kan användas även för att bedömma stabiliserade materials kvarvarande livslängd. Genom att studera värmeflödet under viss tid vid förhöjd temperatur kan bedömmas om det är troligt att stabilisatorerna räcker för ytterliggare t.ex. 5 år. För längre tider är det inte rimligt att bedöma materialet eftersom det kräver accelerering vid alltför hög temperatur för att vara relevant för åldringsprocesserna.

De två teknikerna, OIT och mikrokalorimetri, kompletterar alltså varandra. De ger information i de två olika stadierna i ett stabiliserat materials åldring, och för ett ostabiliserat material ger mikrokalorimetrin information under hela åldringsförloppet.

Av OIT-mätningarna framgår att Dätwylers mantelmaterial inte är stabiliserat på samma sätt som ledarisoleringen. Tidigare studier av mantelmaterial från Rockbestos och Lipalon, som båda är av typ klorsulfonetengummi, visade att värmeflödet är något högre för Rockbestos och aktiveringsenergin något lägre jämfört med för Lipalon. Med hänsyn till de resultat som framkommit i denna studie bör man vara försiktig med tolkningen av dessa aktiveringsenergier. Det är troligt att de beror på andra processer i materialet än de som kan förknippas med polymeråldringen, t.ex. oxidation av i materialet tillsatta oljor.

För vissa material (Dätwyler och Rockbestos ledarisolering åldrad vid 142°) går det inte att påvisa någon induktionstid vid OITmätningar i DSC. Dock visar de låga värmeflödena vid mikrokalorimetrimätningarna att materialet ännu inte börjat oxidera snabbt. Detta kan bero på att induktionstiden är mycket nära noll, men att antioxidanten ännu inte är fullständigt förbrukad. Förklaringen ligger dock snarast i ett annat fenomen. De flesta antioxidanter förbrukas stegvis, d.v.s. även antioxidantens oxidationsprodukter har en viss antioxidantverkan. Även om de senare endast i ringa grad skulle bidra till en induktionstid bestämd vid hög temperatur kan bidraget till stabiliteten vid mer normala temperaturer vara påtagligt. Man kan sålunda få en viss frist mellan det att OIT är praktiskt taget noll till det att oxidationen under normala förhållanden sätter fart. Det förekommer också antioxidanter som redan i ursprunglig form har dålig effekt vid de höga temperaturer där OIT bestäms, men ändå har god effekt under mer normala temperaturer.

#### 7. SLUTSATSER

Resultaten visar att de kemiska åldringsprocesserna är olika för de undersökta ledarmaterialen.

Dätwyler (etenpropengummi) och Rockbestos (tvärbunden polyeten) är stabiliserade med antioxidanter som först förbrukas vid åldringen. Ett mått på materialets åldring kan därför erhållas med hjälp av OIT-mätningar, medan värmeflödet, mätt med mikrokalorimetri är mycket lågt så länge stabilisatorer finns kvar.

Ledarmaterialet i Lipalon, klorsulfonetengummi, ger ökat värmeflöde med ökad åldring och mikrokalorimetri bör vara en lämplig metod för bedömning av materialets status. Eftersom materialet saknar antioxidanter som ger mätbara OIT-värden kan OIT-mätningar inte användas för att studera åldringsstatus för klorsulfonetengummit.

Resultaten tyder också på skillnader mellan ledar- och mantelmaterial. De förra tycks vara bättre stabiliserade.

Aktiveringsenergier, i temperaturintervallet 65-85°C, kunde bestämmas för oxidativa processer i Lipalonmaterial men betydelsen av dem är svårbedömd.

Slutsatserna blir alltså att:

• OIT är en metod för åldringsstudier av stabiliserade material så länge det finns stabilisatorer kvar.

• Mikrokalorimetri är en metod för åldringsstudier av ostabiliserade material, samt för stabiliserade material där antioxidanten är förbrukad. DSC Data File: md135 Sample Weight: 4.720 mg Tue Dec 12 14:43:40 1995

PERKIN-ELMER 7 Series Thermal Analysis System

Datwyler 95gr\*384dygn #634



t el 1

STATISK (=ISOTERM) OIT, DÄTWYLER LEDARISOLERING SOM EXEMPEL

SECRC/D/TR-95/0788 bilaga 1





Appendix A04

Reporting CRC:		SECRC ABB Corporat S-721 78 Västerås S	e Research weden
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Summary:

Rockbestos and Dātwyler cables have been subjected to accelerated aging. Samples of conductor insulation from the aged cables have been examined using DSC-OIT measurements. Plots of OIT vs. aging time are presented for the different combinations of material and aging temperatures. Some companisons are made with the results from measurements of mechanical and electrical properties of material subjected to the same kind of artificial aging.

We find that the induction times (OIT) decrease during the artificial aging and reach zero before the onset of a rapid breakdown of the materials as indicated by drastic changes in the mechanical properties of samples aged under the same conditions.

DSC-OIT appears to be a useful indicator of aging status of Rockbestos and Dātwyler cable conductor insulation.

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#### ABB Corporate Research

#### 1. BAKGRUND

Det har visats att s.k. DSC-OIT-mätning är tillämpbar på ledarisolering från Dätwyler och Rockbestoskabel. Metoden innebär att man under mycket starkt accelererad åldring i syrgas, utförd under pågående mätning i en DSC (differentiell svepande kalorimeter) bestämmer induktionstiden för det exoterma oxidationsförlopp som sätter in när ett oxidationsinhiberat polymermaterial förbrukat/förlorat all antioxidant. I denna studie har sådana mätningar gjorts på material som dessförinnan i varierande grad utsatts för artificiell åldring i ugn. Syftet var att bilda ett bättre underlag för att bedöma hur denna typ av mätning skall kunna användas för att bedöma åldringsstatus hos material från fältet.

För en mer generell bakgrund se föregående rapport SECRC/D/TR-95/0788!

#### 2. MATERIAL

Följande typer av isolermaterial har studerats inom projektet:

Kabeltyp	Mantelmaterial	Ledarisolering
Lipalon	klorsulfonetengummi	klorsulfonetengummi
Dätwyler	etenpropengummi	etenpropengummi
Rockbestos	klorsulfonetengummi	tvärbunden polyeten

De viktigaste materialen ur tillförlitlighetssynpunkt torde vara de som utgör ledarisolering. De typer av etenpropengummi och tvärbunden polyeten som utgör ledarisolering hos Dätwyler respektive Rockbestos kabel är de enda av här aktuella material för vilka DSC-OIT-metoden är tillämplig (SECRC/D/TR-95/0788), varför denna studie begränsat sig till dessa två material.

# 3. ÅLDRING

Den accelererade åldringen har utförts vid ABB Atom. Mätningar har i denna studie gjorts på material åldrade enligt (gäller för båda materialen):

- 1. Termisk åldring vid 80°C i 0, 192, 384 och 576 dygn.
- 2. Termisk åldring vid 95°C i 0, 96, 192 och 384 dygn.
- 3. Termisk åldring vid120°C i 0, 48, 96 och 192 dygn.
- 4. Termisk åldring vid 142°C i 0, 12, 24 och 48 dygn

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Som nollprov (åldrat 0 dygn) har använts material lagrat i kontorsmiljö, prov med id-nr 1555 för Dätwyler, och id-nr 1556 för Rockbestos.

#### 4. METOD

OIT-mätningarna har utförts med en DSC 7 (Differential Scanning Calorimeter) från Perkin-Elmer. Provet värms under N<sub>2</sub>-atmosfär snabbt upp till testtemperaturen varpå gasen växlas till O<sub>2</sub>. Tiden från att syrgasen når mätcellen fram till det att den exoterma reaktionen sätter in är induktionstiden.

En första serie mätningar har gjorts för att finna optimal provtemperatur för respektive material. Därefter har mätningar gjorts för samtliga prover av ett visst material under så lika förhållanden som möjligt.

Provuttaget har gjorts på så vis att en kort bit isolering dragits av från ledaren. Från denna bit har sedan skurits tunna skivor på tvären med en vikt av 5mg (±10%). Prover har tagits slumpvis från de olika ledarna i varje kabelstump. En sådan fanns tillgänglig per åldringstemperatur och tid. En översikt redovisas i bilaga 1.

#### 5. RESULTAT

#### 5.1. Val av testtemperaturer

Utifrån resultat i tabellen nedan valdes 205°C som testtemperatur för Dätwyler och 235°C för Rockbestos. Längre induktionstider än 30 till 40 minuter vore alltför produktivitetshämmande, kortare tider för oåldrat material kan medföra mindre god upplösning mellan de åldrade proverna.

#### Tabell 1.

OIT för olika testtemperaturer för oåldrat material.

material	testemperatur/°C	OIT/min
Dätwyler	200	65.9, 64.2
Dätwyler	205	44.6, 43.4
Dätwyler	210	27.3, 25,3, 24.1
Rockbestos	230	54.5, 52.9
Rockbestos	235	26.6, 29.6, 37.8
Rockbestos	240	14.8, 15.8, 15.3

# 5.2. Åldringskurvor Dätwyler

För varje åldringstemperatur har ställts samman ett diagram över OIT som funktion av åldringstid.



Dätwyler aged at 95°C





Dätwyler aged at 120°C

Dätwyler aged at 142°C



# 5.3. Åldringskurvor Rockbestos

För varje åldringstemperatur har ställts samman ett diagram över OIT som funktion av åldringstid.



Rockbestos aged at 95°C




Rockbestos aged at 120°C

Rockbestos aged at 142°C



ABB Corporate Research

# 6. JÄMFÖRELSER MED ELEKTRISKA OCH MEKANISKA EGENSKAPER

Der är av stort intresse att jämföra dessa resultat med dem från mätningar av hur elektriska och mekaniska egenskaper förändras under åldring utförd under likadana betingelser. Tillgängligt material har varit SKI Technical Report 93:39 och Rapport H-14364-D (utkast daterat 96-02-29) från Ingemansson. Ett försök till sammanställning redovisas i tabellerna på de följande sidorna.

Kursiv stil anger att värdena hämtats ur rapport 93:39, övriga värden är tagna ur Rapport H-14364-D. Fet stil anger att värden avser förändring jämfört med oåldrat material (vars status vi tyvärr ej lyckats utläsa ur rapporterna i fråga). "Indenter ratio" gäller mantel om ej annat anges. Såväl indenter ratio som  $e/e_0$  har antagits vara (definitionsmässigt) lika med 1 för oåldrat material. För material som enligt uppgift var för spröda för dragprovning har  $e/e_0$  satts till noll.

# 6.1. Sammanställning av data för Dätwyler

Datwyler 80°				
tid/ dygn	0	192	384	576
R/MΩ		-16.9	-6.3	- 6 . 4
tan ∂ (60Hz)		0.005	-0.01	0.004
tan ∂ (1000Hz)		-0.007	0.004	0.002
indenter ratio (unsealed)	1	1.224	1.317	1.311
e/e0	1	0.906	0.875	0.869
indenter ratio, lead	1	1.24	1.373	1.248
OIT (medelvärde)/min	34.6	16.8	21.9	17.2

Dätwyler	8	0	c
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Dätwyler	95°
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tid/ dygn	0	96	192	384
R/MΩ		5.3	15.1	5.3
tan ∂ (60Hz)		0.017	0.002	0.006
tan ∂ (1000Hz)		0.017	0.005	0.01
indenter ratio (unsealed)	1	1.019	1.135	1.149
e/e0	1	0.92	0.685	0.784
indenter ratio, lead	1	1.379		1.464
OIT (medelvärde)/min	34.6	29.3	22.1	28.6

## Dätwyler 120°

tid/ dygn	0 .	48	96	192
R/MΩ		13.3	- 34.3	340
tan ∂ (60Hz)		0.055	0.077	0.011
tan ∂ (1000Hz)		0.046	0.054	0.0075
indenter ratio (unsealed)	1	1.74	2.01	2.18
indenter ratio (sealed)	1			1.9
e/e0	1	0.71	0.65	0.38
indenter ratio, lead	1	1.154	1.246	
OIT (medelvärde)/min	34.6	10.2	5.3	2.6

# Dätwyler 142°

Dätwyler 142°						
tid/ dygn	0	12	24	48		
R/MΩ		-986.3	310	330		
tan ∂ (60Hz)		0.088	0.014	0.068		
tan ∂ (1000Hz)		0.06	0.031	0.036		
indenter ratio (unsealed)	1	1.45	1.66	2.28		
indenter ratio (sealed)	1		1.33	1.89		
e/e0	1	0.54	0.42	0.26		
indenter ratio, lead	1	1.2				
OIT (medelvärde)/min	34.6	5.4	<0.5	<0.5		

# 6.2. Sammanställning av data för Rockbestos

# Rockbestos 80°

tid/ dygn	0	192	384	576
R/MΩ		1.9	-16.1	-39.8
tan ∂ (60Hz)		0.0002	-0.018	0.002
tan ∂ (1000Hz)		0.0016	-0.004	0.001
indenter ratio (unsealed)	1	1.283	1.414	1.48
e/e0	1	0.838	0.711	0.628
indenter ratio, lead	1	1.361	1.274	1.459
OIT (medelvärde)/min	30.0	21.6	23.1	18.5

# Rockbestos 95°

tid/ dygn	0	96	192	384
R/MΩ		12.4	12.1	12.1
tan ∂ (60Hz)		0.002	-0.021	-0.002
tan ∂ (1000Hz)		0.001	-0.002	0
indenter ratio (unsealed)	1	0.994	1.049	1.132
e/e0	1	0.705	0.602	0.466
indenter ratio, lead	1	1.164		1.182
OIT (medelvärde)/min	30.0	20.8	17.8	13.6

tid/ dygn	0	4 8	96	192
R/MΩ		13.3	21.4	330
tan ∂ (60Hz)		-0.002	-0.001	0.012
tan ∂ (1000Hz)		0	0	0.006
indenter ratio (unsealed)	1	1.838	20.64	29
indenter ratio (sealed)	1			26.95
e/e0	1	0.28	0	0
indenter ratio, lead	1	1.014	0.982	
OIT (medelvärde)/min	30.0	12.6	23.8	<0.5

#### Rockbestos 120°

#### **Rockbestos 142°**

tid/ dygn	0	12	24	48
R/MΩ		5.7	330	310
tan ∂ (60Hz)		0	0.007	0.015
tan ∂ (1000Hz)		0	0.003	0.01
indenter ratio (unsealed)	1	1.395	4.3	18.32
indenter ratio (sealed)	1		2.14	11.85
e/e0	1	0.29	0.03	0
indenter ratio, lead	1	0.933		
OIT (medelvärde)/min	30.0	5.6	<0.5	<0.5

#### 6.3. Jämförelser för 80°C och 95 °C, Dätwyler och Rockbestos

För båda materialen sker en högst måttlig minskning av OIT för prover åldrade vid 80 och 95° även sett under hela den tid försöken pågått. För Dätwyler är det frågan om det överhuvudtaget sker någon signifikant förändring alls. Dessa iakttagelser är i samklang med resultaten från indentermätningar och dragprov, där både förstyvningen och sänkningen av brottöjning visat sig vara moderata. För de elektriska egenskaperna tycks det ej ha skett några som helst signifikanta förändringar.

#### 6.4. 120°C Dätwyler

Vi ser en mycket tydlig och jämn avklingning av OIT, som dock ej minskar till under 2 minuter under den tid försöket pågått. Vi ser ingen katastrofal förändring av de mekaniska egenskaperna, även om de påverkats mer än i fallen 80 och 95°C. De elektriska egenskaperna förefaller även här i stort sett opåverkade.

# 6.5. 120°C Rockbestos

Här finns en mycket iögonfallande diskontinuitet i OITavklingningen i form av ett mycket lång induktionstid för det prov som åldrats 96 dygn. Detta prov skiljer sig knappast alls från oåldrat material trots att provet som åldrats endast 48 dygn har en gott och väl halverad induktionstid jämfört med oåldrat material. Utifrån induktionstiden för 48-dygnsprovet skulle man snarare förväntat sig en induktionstid nära noll för 96-dygnsprovet. Om vi tills vidare bortser från denna punkt i kurvan finner vi att när induktionstiden nått noll (vid 196 timmar) har de mekaniska egenskaperna försämrats dramatiskt.

Anmärkningsvärt är att de elektriska egenskaperna fortfarande är påverkade i mycket ringa grad när de mekaniska visat en mycket kraftig försämring.

#### 6.6. 142°C Dätwyler

Här har OIT nått noll redan för 24-dygnsprovet. Varken elektriska eller mekaniska egenskaper har dock börjat förändras katastrofalt ens vid 48 dygn.

# 6.7. 142°C Rockbestos

Liksom för Dätwyler har induktionstiden nått noll redan för 24dygnsprovet. Här ser vi det typiska förloppet med en skenande försämring av de mekaniska egenskaperna när antioxidanterna försvunnit. Återigen finner vi dock att de elektriska egenskaperna förändrats i ringa grad, även för det mest nedbrutna materialet.

# 7. KOMMENTARER

För Dätwyler isolering finns en stor spridning i OIT-värdena (vilket framgick redan i den förberedande studie som finns redovisad i SECRC/D/TR-95/0788). Detta gäller framförallt för måttligt åldrat material. Spridningen tycks vara avsevärt mindre för de prover för vilka en signifikant minskning av OIT uppnåtts.

Fenomenet torde i praktiken inte ha någon större betydelse. Spridningen beror på att materialet från början är tämligen inhomogent, bland annat m.a.p. innehållet av antioxidanter. Med tiden kan det ske en omfördelning av tillsatsämnen, med ett mer homogent material som följd. Detta sker samtidigt som antioxidanter förbrukas eller vandrar ut ur materialet. När förlusterna är så stora att OIT sjunkit till några få minuter har också spridningen i värdena minskat avsevärt. Med tanke på vilken funktion OIT-mätningar på verkliga fältprover är avsedda att ha, nämligen att ge en varningsignal när materialet börjar närma sig den punkt där oxidationen börjar ske snabbt, torde alltså fenomenet inte innebära något egentligt problem. Dock bör man under alla förhållanden göra multipla bestämningar även på skarpa prover från fältet.

Även för oåldrad Rockbestos isolering finns en betydande spridning hos OIT-värdena. Dock tycks det som att omfördelningen sker mycket snabbare i detta material än för Dätwyler.

Bland de kabelstumpar som fanns tillgängliga för denna studie förekom både sådana som var avtätade under åldringen och sådana som ej var det. Tidigare försök (redovisade i rapport H-14364-D från Ingemansson) visar att ej avtätade kablar åldras snabbare än avtätade. Detta innebär att resultaten här i viss mån är förvrängda, dock knappast till den grad att det påverkar några slutsatser.

I jämförelserna med resultat från mekaniska och elektriska mätningar har antagits att proverna åldrats under likvärdiga förhållanden i varje fall. Detta är givetvis en förenkling, men en nödvändig sådan för att kunna göra några jämförelser alls.

Rockbestos åldrad vid 120°C i 96 dygn avviker markant från den tydliga avklingningsbild med liten spridning i mätdata som för övrigt gäller för detta material. Vi kan inte se någon förklaring till detta. En möjligen bidragande faktor kan vara att denna kabel var åldrad med avtätade ändar, medan provet för 192 dygn hade öppna ändar. Å andra sidan är det ökningen av OIT mellan 48 och 96 dygn som är mest orimlig, och även 48-dygnsprovet var ändavtätat. Hur som helst har bortsetts från denna mätpunkt när slutsatserna dragits.

# 8. SLUTSATSER

Aven om det fortfarande finns ett tämligen begränsat material att grunda bedömningarna på kan ändå dras vissa slutsatser:

Denna studie har gjorts på material som genomgått accelererade åldringsförsök. Om resultaten från dessa har relevans för verkliga driftsförhållanden måste slutsatsen bli att OIT är användbart som ett mått på åldringsgraden för de undersökta materialen.

För Rockbestos finner vi en tydlig avklingning av OIT under åldringen samt att så länge OIT är större än noll har ingen skenande försämring av de mekaniska egenskaperna satt in. Det förefaller dock

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som att så sker ganska snabbt efter att OIT nått noll. Exakt vilken OIT som bör väljas som gränsvärde kan givetvis diskuteras. Det beror också på faktorer som ej kunnat tas med i denna begränsade undersökning. T.ex. kanske frågan bör formuleras "vilken OIT motsvarar den längst gångna åldringsgrad där materialet fortfarande klarar ett LOCA-test?".

För Dätwyler är förloppet något annorlunda. Här tycks finnas en längre tid mellan det att OIT nått noll och det att materialets mekaniska och elektriska egenskaper snabbt börjar förändras. För Dätwyler gäller således i ännu högre grad än för Rockbestos att en låg OIT är att betrakta som en <u>tidig</u> varningssignal. F.ö. torde resonemanget vara detsamma som för Rockbestos.

Temp (°C)	Tid (dygn)	Grupp nr	Dätwyler ident.	Rockbestos ident.	anm.
	1				
80	192	11	311	201	2 dm, avtätad
80	384	3	296	186	2 dm, avtätad
80	576	4	297	187	2 dm, avtätad
95	96	14	317	207	2 dm, avtätad
95	192	16	323	212	2 dm, avtätad
95	384	17	325	214	2 dm, avtätad
120	48	1 b	291	181	2 dm, avtätad
120	96	19	326	217	2 dm, avtätad
120	192	46	1550	1553	3 dm, ej avtätad
142	12	20	640	431	1 m, ej avtätad
142	24	49	1504	1465	3 dm, ej avtätad
142	48	50	1507	1467	3 dm, ej avtätad

# Kablar till OIT-mätning

96-08-13 S.K

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