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Jean-Pierre Crine, S. Haridoss, K. C. Cole, A.T. Bulinski ...+2 more authors

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Crine, J. P.; Haridoss, S.; Cole, K. C. (Kenneth Chesley); Bulinski, A. T.; Densley, R. J.; Bamji, S. S.

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OXIDATION AND THERMAL RESISTANCE OF HMW-PE AND XLPE H.V. CABLES

J.-P. Crine and S. Haridoss
Institut de recherche d'Hydro-Québec
Varenes, Québec, Canada JOL 2P0

IGM88AP-101-713-G
CNRC 28894

K.C. Cole
Institut de Génie des Matériaux
Boucherville, Québec, Canada

A.T. Bulinski, R.J. Densley and S.S. Bamji
CNRC, Ottawa, Ontario, Canada

ABSTRACT - The thermal oxidation of the insulation of several unaged and aged transmission and distribution cables has been measured by FTIR spectroscopy. It is shown that all cables, even unaged ones, contain traces of oxidation which increases with aging, especially near the conductor shield. The nature of the antioxidant compound has been investigated by FTIR and UV spectroscopy and sulfur content measurements. It is shown that the amount of residual antioxidant can be related to the oxidation induction time as measured by DSC at 200°C in oxygen. The influence of various parameters on cable thermal resistance is also discussed.

INTRODUCTION

It is well-known that thermal oxidation is a detrimental aging process in the polymers used for cable applications [1]. In polyethylene (PE), the major oxidation by-products are carbonyl groups whose presence is readily determined from their IR absorbance bands in the 1710-1745 cm⁻¹ range. Extruded dielectric cables insulated with PE or crosslinked PE (XLPE) being operated mainly at ~ 50°C and sometimes up to 90°C, it can be expected that they are somewhat oxidized after some years in service. It has also been speculated that oxidation may be associated with water trees [2]. To date there is no comprehensive study of the extent of oxidation in cables aged in service and one objective of this paper is to report Fourier transform Infrared (FTIR) spectroscopy measurements performed on 19 HMW-PE and XLPE cables either unaged or aged in service.

In order to prolong the oxidation induction time, it is customary to add a given amount of antioxidant and sometimes antioxidant synergist to the polymer [1]. Santonox R has been a popular additive in the fifties and sixties but it has been replaced in the seventies by other antioxidants, such as Irganox 1035, that have a lower diffusion coefficient and higher molecular weight [1]. The role of antioxidant synergist is essentially to act as hydroperoxide decomposer during cable manufacturing [1]. Two synergists commonly used by resin suppliers are di-lauryl thio-dipropionate (DLTDP) and di-stearyl thio-dipropionate (DSTDTP). Antioxidants and synergists can be readily identified in PE by IR and UV spectroscopy [3, 6], HPLC [4] and from sulfur content measurements [5, 6]. The oxidative stability of PE which is related to the antioxidant content, can be estimated by oxygen uptake or by oxidative induction time (OIT) measurements [1]. The latter measurement is generally performed by differential scanning calorimetry (DSC) in presence of oxygen. This is an indirect method of evaluation of antioxidant content and it cannot distinguish between antioxidant and antioxidant synergist. Another objective of this study was to determine whether antioxidant content in PE and XLPE cables could be related with OIT measurements.

EXPERIMENTAL PROCEDURE

The list of the 19 tested transmission and distribution cables (unaged and aged) is shown in Table 1. They were peeled off by tool cutting on a lathe (more details in Ref. [6]) and the ribbons thus formed had a thickness in the 100-200 µm range. Samples taken at various locations in the ribbon were analysed by FTIR spectroscopy and by DSC. The samples in Table 1 are identified by six letters: the first two refer to the manufacturer or utility, the next two identify the nature of the insulation (PE or XLPE) and the last two indicate the cable aging (UA: unaged, LA: laboratory aged, FA: field aged).

TABLE 1
Cables tested

| Transmission cables | Age | Distribution cables | Age |
|---|----------------|---------------------------------------|--------------|
| SI-PE-UA } 225 kV SI-PE-LA } HMW-PE | - lab. aged | NV-PE-UA } 15 kV NV-PE-FA } HMW-PE | - 6 years |
| OT-XL-UA } 138 kV OT-XL-LA } dry-cured | - lab. aged | MP-PE-FA } 22 kV } HMW-PE | 7 years |
| SI-XL-UA } 69 kV SI-XL-LA } dry-cured | - lab. aged | FP-PE-FA } 15 kV } HMW-PE | 7 years |
| WM-XL-UA } 138 kV WM-XL-LA } steam-cured | - lab. aged | MP-XL-FA } 15 kV } steam-cured | 6 years |
| LE-XL-UA } 150 kV LE-XL-FA } steam-cured | - 6 years | FP-XL-FA } 15 kV } steam-cured | 6 years |
| DE-XL-UA } 138 kV DE-XL-FA } steam-cured | - 5 years | UE-XL-FA } 18 kV } steam-cured | 11 years |

ANTIOXIDANT AND OXIDATION MEASURED BY FTIR SPECTROSCOPY

The instrument used was a Nicolet FTIR spectrometer model 170SX and most measurements were obtained with a DTGS detector maintained at room temperature. Most absorbance spectra were recorded after 64 scans and some after 128 scans. The samples (~ 20 mm x 10 mm) being ~ 100 to 200 µm thick, there are interference fringes in the spectra. The noisy background can be reduced by mounting the sample on a polarizer tilted to the Brewster angle of the polymer (i.e. ~ 57° for PE). This simple and inexpensive procedure improves significantly the signal-to-noise ratio, and allows the identification of IR bands that otherwise could not be detected.

Identification of Antioxidants

Santonox R had been used in the past as antioxidant for cable insulation and it could be expected to be detected in the cables manufactured in the early seventies. It can be identified from its strongest IR

absorbance bands at 3510, 3320 and 1170-1190 cm^{-1} . However, none of the bands typical of Santonox R were observed in the transmission or distribution cables studied. An example is shown in Fig. 1 where it can be seen that the spectrum of a sample taken close to the conductor of MP-PE-FA (made in 1972) does fit with any

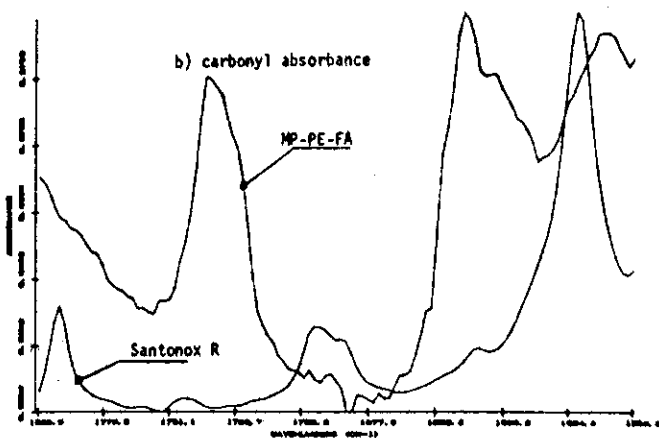
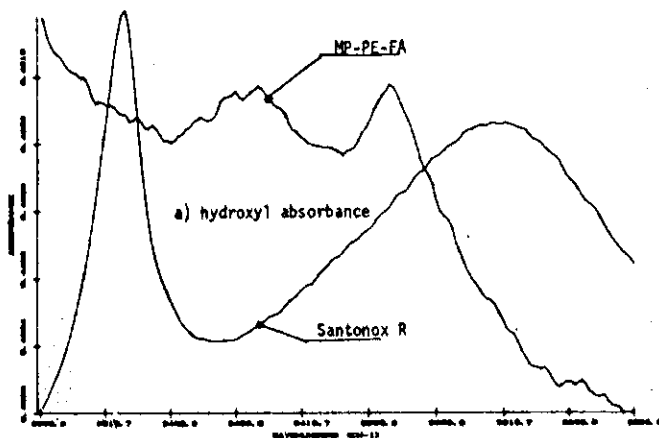


Fig. 1 Spectra of a sample taken close to the conductor of MP-PE-FA and of Santonox R. It is possible that the residual content of Santonox R, if any, is below the detection level of FTIR spectroscopy.

It appears that some of the studied transmission and distribution cables manufactured before ~ 1975 contain Irganox 1035 that can be identified from its strong absorbance band at 1733-1736 cm^{-1} (see Fig. 2). An example is shown in Fig. 3 where the absorbance band at ~ 1735 cm^{-1} in a sample taken close to the conductor of MP-PE-FA corresponds to the major absorbance band of Irganox 1035 (see Fig. 2). UV absorbance typi-

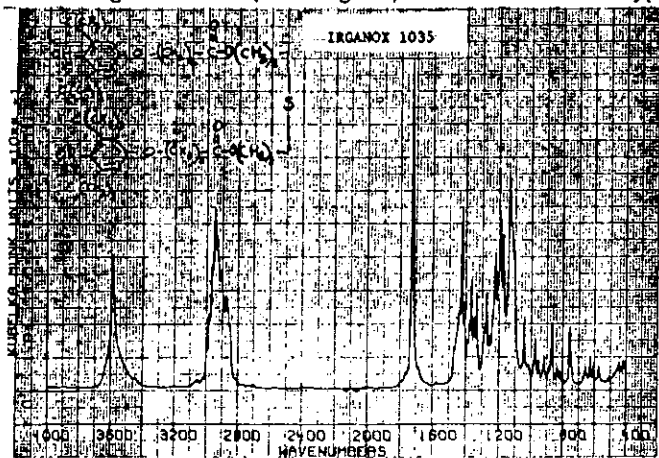


Fig. 2 - FTIR spectrum of Irganox 1035 (antioxidant).

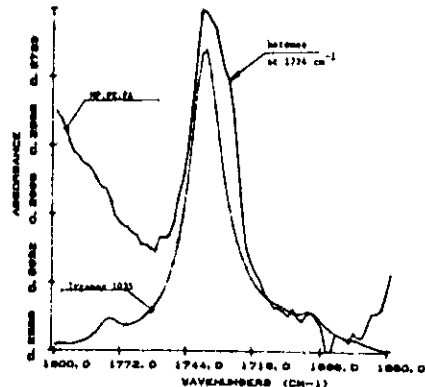


Fig. 3 Absorbance band at 1735 cm^{-1} in a sample taken in MP-PE-FA compared to the spectrum of Irganox 1035.

cal of either Santonox R or Irganox 1035 was observed (see Fig. 11); since no Santonox R IR bands were detected in that sample (Fig. 1), it seems that this insulation contains Irganox 1035. None of the DSTDP bands shown in Fig. 4 were detected and this is true for all studied cables manufactured before ~ 1975.

The distribution cables manufactured in the late seventies (MP-XL-FA and FP-XL-FA) show a strong absorbance band at 1742 cm^{-1} (see Fig. 4), typical of DSTDP (see Ref. 6). This is confirmed by the presence of a strong absorbance band at 1161 cm^{-1} . Identical observations are made with some transmission cables, especially OT-XL cables. With several cables the strong absorbance band of DSTDP at 1742 cm^{-1} completely masks the Irganox 1035 band at 1735 cm^{-1} . The antioxidant used in the SI-PE cables is readily identified by FTIR spectroscopy (see Fig. 5) and by UV spectroscopy as DPPD, an amine-type antioxidant. Thus, it is sometimes possible (especially when there is little oxidation) to identify the insulation antioxidant by FTIR spectroscopy.

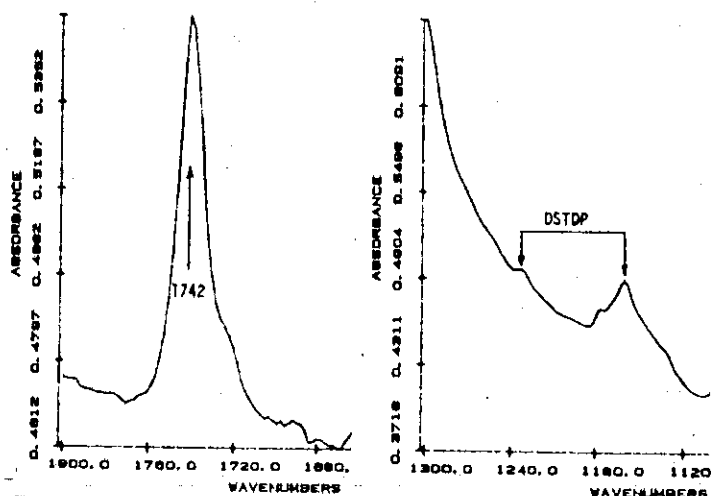


Fig. 4 - Absorbance spectra showing all the bands of DSTDP in MP-XL-FA.

Oxidation

The various carbonyl group formed during oxidation are (in sequential order of formation): ketones (absorbance at 1720 - 1724 cm^{-1}), aldehydes (absorbance at 1735 cm^{-1}), acids (absorbance at 1712 cm^{-1}). The final by-products of oxidation are esters which are formed by condensation of alcohol and acid (absorbance band at 1742 cm^{-1}) and lactones (absorbance band at 1780 cm^{-1}). However, one should be very careful before assigning bands in the 1715-1745 cm^{-1} region to oxidation, since Irganox 1035 and DSTDP absorb at 1735 and 1742 cm^{-1} , respectively. The only way to definitely assign a given band to oxidation or antioxidant is to look at other bands also. For example, the strong

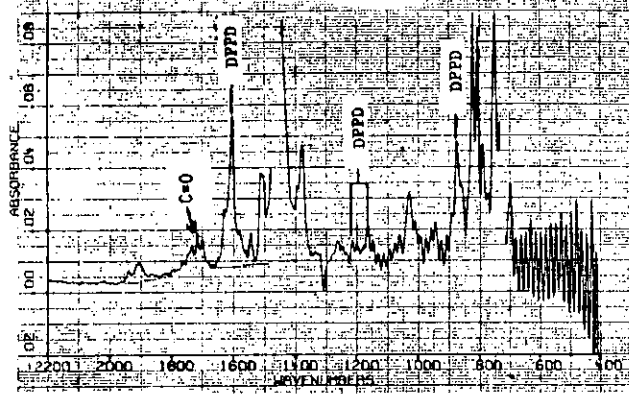


Fig. 5- FTIR spectrum of the insulation of SI-PE-LA.

ester absorbance band at 1742 cm^{-1} in MP-XL-FA (Fig. 4) can be assigned to DSTDP since other bands of this synergist are visible at 1161 and 1235 cm^{-1} . On the other hand, the strong band at 1742 cm^{-1} in WM-XL-FA (Fig. 6) should not be assigned to DSTDP since there is no (or nearly no) DSTDP absorbance at 1161 and 1235 cm^{-1} . In fact, a small band at 1724 cm^{-1} typical of ketones is visible in Fig. 6, which is another evidence for oxidation in this cable.

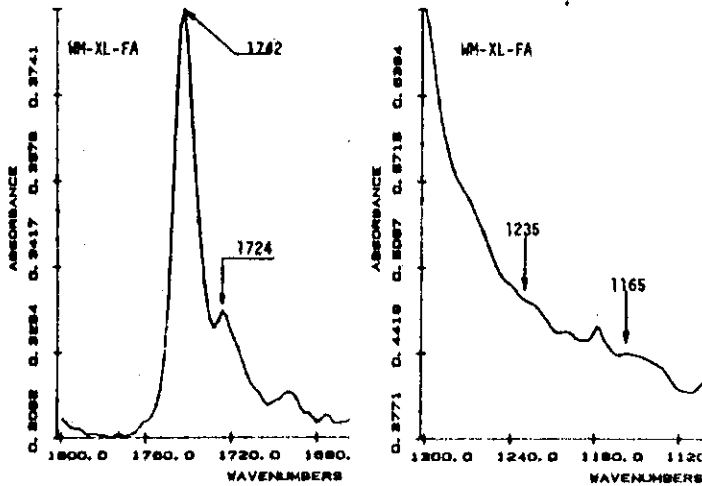


Fig. 6 Absorbance spectra of a sample taken in WM-XL-FA showing ketones and esters typical of oxidation but no DSTDP bands at 1161 and 1235 cm^{-1} .

We have examined the FTIR spectra of all cables studied and observed that:

- There are traces of oxidation in all cables, even in unaged ones, especially near the conductor shield (see Fig. 7).
- As shown in Fig. 7, aging leads to more pronounced oxidation.
- The field-aged distribution cables studied are more oxidized than the aged transmission cables (Table 2) but the former have been in service for longer time than the latter.
- There is more ketone in XLPE than in HMW-PE cables, as evidenced by the results shown in Table 2.
- The presence of carbonyl groups corresponds, as expected, to a reduction in antioxidant content. This is shown in Fig. 8 where the IR absorbance of carbonyls are plotted against two parameters representative of the antioxidant content, namely the UV absorbance of phenyl groups and the sulfur content

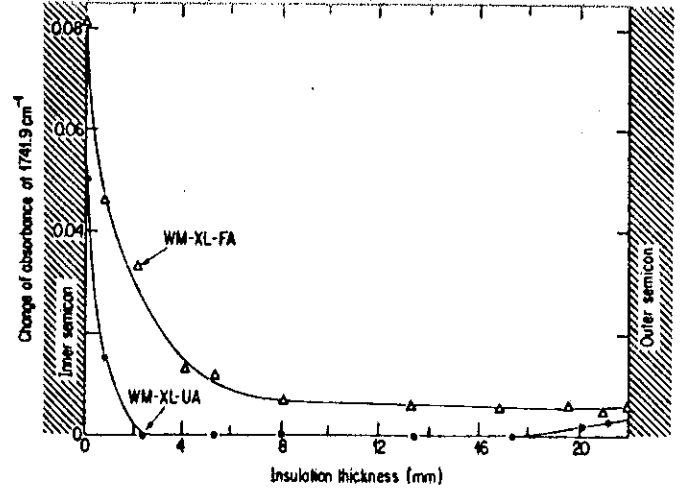


Fig. 7 Radial distribution of carbonyl absorbance at 1742 cm^{-1} in WM-XL cables. Note that the absorbance of each sample was compared to the absorbance of a sample located 8 mm from the conductor.

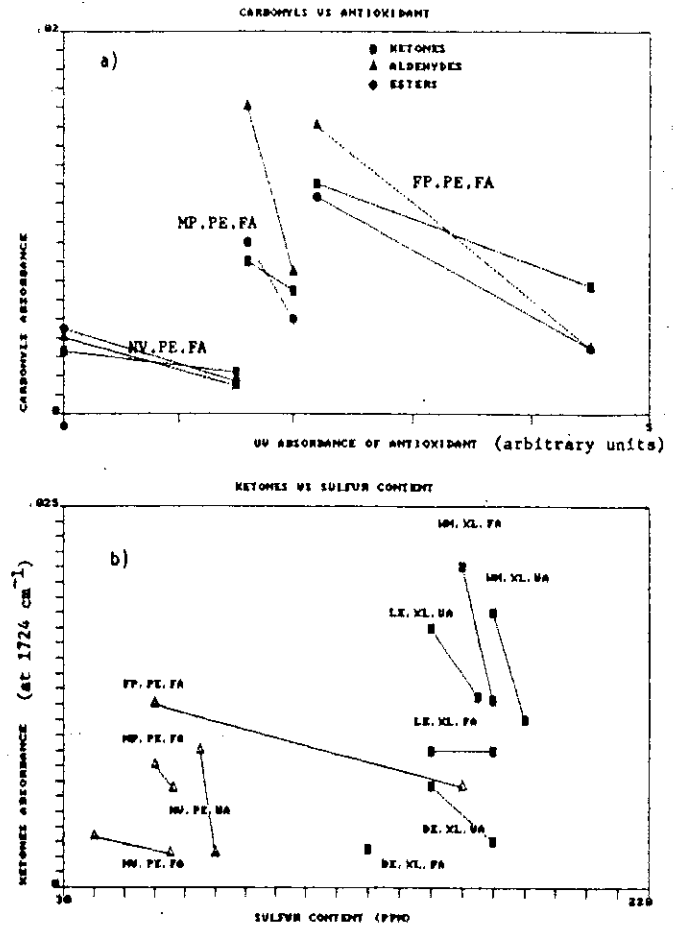


Fig. 8 - IR absorbance of carbonyl groups (i.e. oxidation) vs antioxidant content as evaluated by UV spectroscopy for HMW-PE cables (a) and sulfur content (b) in XLPE (■) and HMW-PE cables (Δ).

content. It is evident that the carbonyl content increases with decreasing antioxidant content.

- Many cables manufactured after ~ 1975 seem to be inhibited by a sulfur containing compound whose constituents appear to be Irganox 1035 and DSTDP. Older cables contain a different antioxidant system and no evidence was found for the presence of Santonox R.

FTIR absorbance of the bands assigned to oxidation functionality and antioxidant in some cables

| Distribution cables | Year | Oxidation | Oxidation/antioxidant |
|--|--------------|----------------------------------|---------------------------------|
| | Manufactured | Ketones (1724 cm^{-1}) | Esters (1742 cm^{-1}) |
| MV-PE-UA middle insul. near insul. sh. | 1971 | 0.0022 | 0.0026 |
| | | 0.0090 | 0.0170 |
| MV-PE-FA middle insul. near insul. sh. | 1971 | 0.0023 | 0.0018 |
| | | 0.0033 | 0.0045 |
| MP-PE-FA middle insul. near insul. sh. | 1972 | 0.0065 | 0.0049 |
| | | 0.0080 | 0.0090 |
| UE-XL-FA middle insul. near insul. sh. | 1972 | 0.0145 | ? |
| | | 0.0145 | 0 |
| FP-PE-FA near cond. sh. near insul. sh. | 1973 | 0.0066 | 0.0035 |
| | | 0.0120 | 0.0140 |
| Transmission cables | | | |
| DE-XL-UA near cond. sh. middle insul. | 1974 | 0.0028 | 0.0012 |
| | | 0.0067 | 0.0030 |
| DE-XL-FA middle insul. near insul. sh. | 1974 | 0.0024 | 0.0012 |
| | | 0.0025 | 0.0244 |
| LE-XL-UA near cond. sh. middle insul. | 1975 | 0.0170 | 0.0620 |
| | | 0.0110 | 0.0770 |
| LE-XL-FA near cond. sh. middle insul. | 1975 | ? | 0.0330 |
| | | 0.0090 | ? |
| WM-XL-UA near cond. sh. middle insul. | ? | 0.0180 | 0.0490 |
| | | 0.0110 | ? |
| WM-XL-FA near cond. sh. middle insul. | ? | 0.0210 | 0.0780 |
| | | 0.0126 | 0.0094 |
| SI-PE-UA near cond. sh. middle insul. | 1981 | 0.0100 | 0 |
| | | 0.0040 | 0 |
| SI-PE-LA near cond. sh. middle insul. | 1981 | 0.0060 | 0 |
| | | 0.0046 | 0 |
| SI-XL-UA near cond. sh. middle insul. | 1981 | 0.0085 | 0.0040 |
| | | 0.0050 | 0.0030 |

The traces of oxidation detected in unaged insulations were apparently generated during extrusion and curing. This is supported by the generally larger ketone content in XLPE cables (see Fig. 8). Exposition to high temperature during manufacturing or in service leads to antioxidant consumption and therefore to oxidation.

RESISTANCE TO OXIDATION FROM OIT MEASUREMENTS

Experimental Procedure

We performed two different tests, oxidation induction time (OIT) and oxidation induction temperature; both tests are designed to evaluate the oxidation resistance of polymers. In the isothermal test, samples of ≈ 10 mg were heated in aluminum pans at $10^\circ\text{C}/\text{min}$ from 22 to 200°C under nitrogen in a DuPont model 1090 Thermal Analysis System. The gas atmosphere was then changed to oxygen immediately after the temperature was reached and was maintained at 200°C . The gas flow rates were 50 cc/min as required by ASTM-D3895-80. The OIT was deduced as the onset of the exothermic reaction. In the heat ramp test the temperature of the sample is raised from 20 to $\approx 265^\circ\text{C}$ at a constant rate of $10^\circ\text{C}/\text{min}$. The samples are kept in oxygen atmosphere right from the start of the tests. The oxidation induction temperature is determined as the onset of the exothermic reaction.

OIT and oxidation temperature results depend very much on the experimental conditions, especially on the sample size and preparation. Reliable and reproducible results are obtained more easily with flat rather than with irregular samples (such as those cut with a knife). The flatness of the samples is important because it greatly affects the heat transfer between the heating pan and the sample.

Typical Results

The reproducibility of the results under the same test conditions was reasonably good and the OIT does not depend on the kind of cables (transmission or distribution) nor on the curing but is sensitive to aging as shown in Table 3. Another typical result is that the OIT of semiconductors are significantly greater than those of the insulation. For the SI-PE-UA cable, the OIT of the insulation shield is 80 ± 6 min compared to 27 min for the insulation itself. Values vary greatly from cable to cable but the ratio between the OIT of semiconductors and insulation is generally around 4.

TABLE 3
Oxidation induction time and oxidation induction temperature of the insulation of some cables

| CABLE | OIT (min) at 200°C^a | OXIDATION INDUCTION TEMPERATURE ($^\circ\text{C}$) |
|---------------------|------------------------------------|--|
| TRANSMISSION | | |
| SI-PE-UA | 27 | - |
| SI-PE-LA | 21.5 | - |
| OT-XL-UA | 21 | - |
| OT-XL-LA | 21 | 231.3 |
| WM-XL-UA | 12.2 | 225.9 |
| WM-XL-FA | 13.3 | 221.0 |
| DE-XL-UA | 11.5 | - |
| DE-XL-FA | 7.0 | - |
| DISTRIBUTION | | |
| MV-PE-UA | 11.5 | - |
| MV-PE-FA | 6.8 | - |
| FP-PE-FA | 35.5 | 242.8 |
| MP-PE-FA | 14.8 | 228.8 |
| MP-XL-FA | 15.6 | 227 |
| FP-XL-FA | 7.9 | 221 |
| UE-XL-FA | 13.6 | 230 |

a: from isothermal test.
b: from heat ramp test.

As a general rule, there is a good correlation between the measured OIT and oxidation induction temperature (Table 3). In fact, the heat ramp tests give quicker results with possibly less scatter in the values than the isothermal tests.

Correlations with other Results

The aim in performing the OIT measurements was to evaluate the ability of the insulation to successfully resist oxidation; it was therefore expected that the OIT value should be related to the remaining antioxidant compound content or activity. When the OIT value measured with some cables is plotted as a function of the antioxidant synergist content evaluated from the IR absorbance band at 1742 cm^{-1} , it appears that both parameters are linearly related (Fig. 9). Thus, the OIT indirectly measures the residual antioxidant compound content in the tested insulation.

It has been shown elsewhere [5] that the content of sulfur-containing antioxidants can be evaluated from measurements performed by neutron activation analysis (NAA). With the only exception of the SI-PE cables, all the samples in this work were inhibited with Santonox R, Irganox 1035 or DSTDP that all contain sulfur. When the residual sulfur content of the samples was plotted as a function of the OIT, it is seen that these are linearly related (Fig. 10). However, there is not a single relationship for all cables but two: one for crosslinked (transmission and distribution) and one for HMW-PE cables. This result is in excellent agreement with earlier accelerated aging measurements [5] which showed that in XLPE cables, a residual concentration of $\approx 110 \pm 15$ ppm of sulfur is always present and does not inhibit oxidation. The very approximate relation

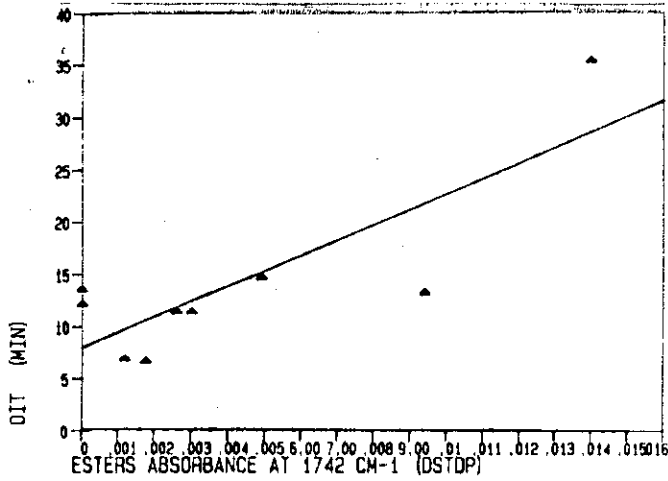


Fig. 9 -Relation between OIT and DSTDP content of several cables.

deduced in Fig. 10 for XLPE samples supports this contention. It has previously already been proposed [4, 5] that part of the antioxidant compound is grafted to the polymer molecule and this 110 ppm of sulfur seems to correspond to the grafted (i.e. non functional) portion of antioxidant, which explains why the much smaller antioxidant content left in HMW-PE insulation still

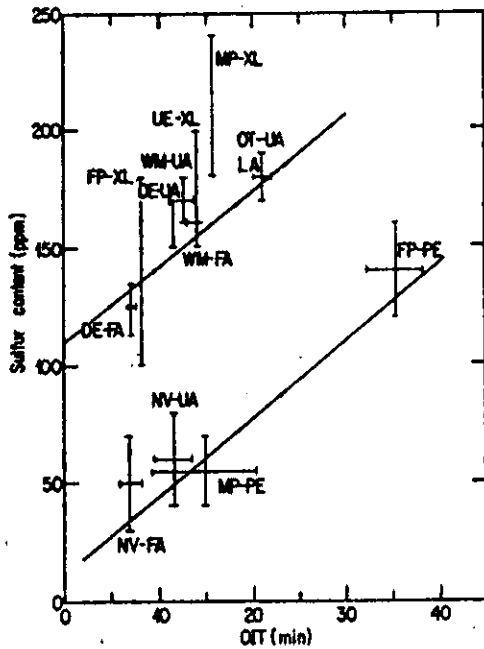


Fig. 10 - Relation between sulfur content (i.e. antioxidant compound) and OIT. Note the 2 relations for PE and XLPE.

plays an active role as oxidation inhibitor. It therefore can be concluded that OIT measurements can evaluate the antioxidant compound content in cables but the results obtained for XLPE should not be extrapolated to HMW-PE, or vice versa. In other words, two calibration curves (as in Fig. 10) are required for an approximate evaluation of the antioxidant content from OIT values. Hence there is evidence that a portion of the antioxidant that contains sulfur is grafted to the XLPE chains and has no antioxidant functionality. Also it is not certain that these results would be applicable to other cable grade insulations that contain different antioxidants.

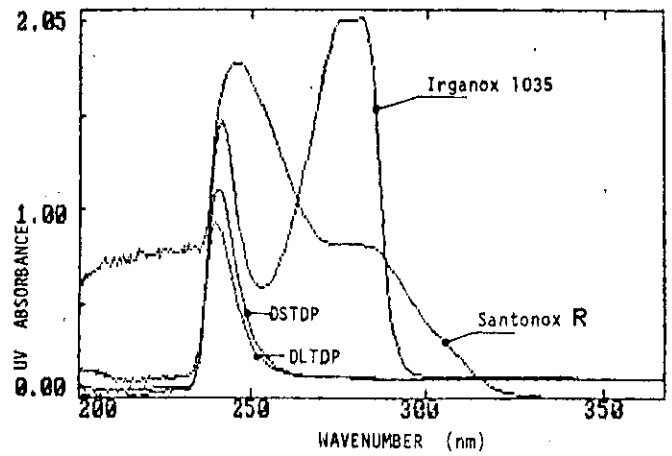


Fig. 11- UV spectra of various antioxidants and synergists.

In PE the antioxidant content can be evaluated from the UV absorbance of the phenyl groups in the antioxidant [3, 6]. This is not possible with XLPE since the curing by-products absorb in the same wavelength range. Figure 11 shows the UV absorbance of some antioxidants and synergists and it appears that the synergists do not absorb at 286 nm. We have measured the radial distribution of UV absorbance at 286 nm in various uncrosslinked cable insulations and it is compared in Fig. 12 with the radial distribution

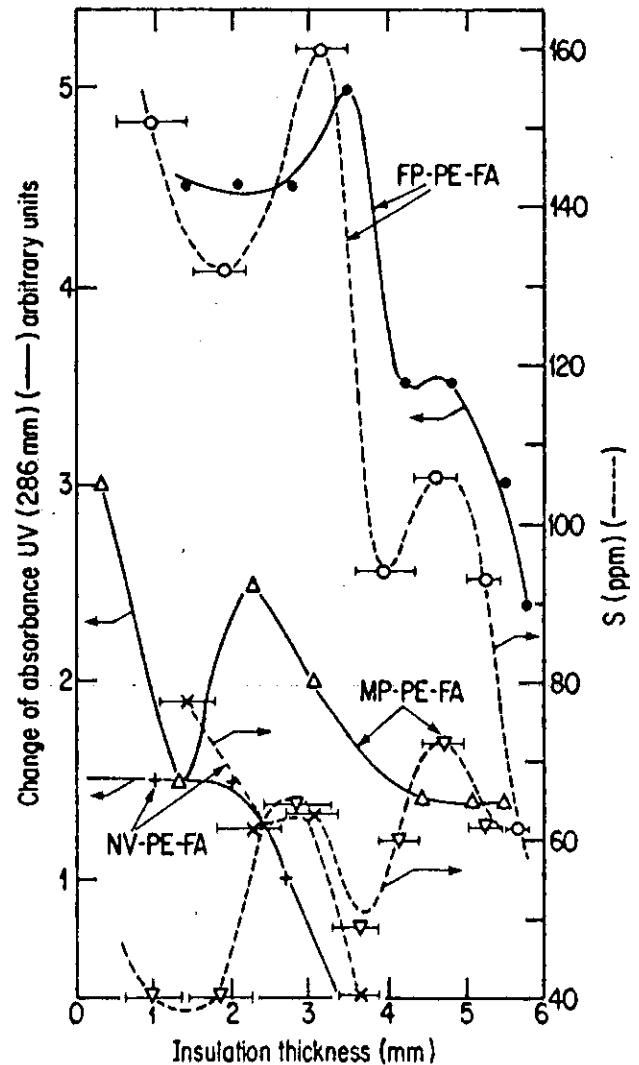


Fig. 12- Radial distribution of sulfur and phenyls (as measured by UV spectroscopy) in various HMW-PE distribution cables.

of sulfur content, as measured by NAA. It is evident that the sulfur content and the UV absorbance are related for all cables tested. Thus, UV absorbance, sulfur content, IR absorbance and OIT value are all related with the antioxidant compound content. Note that all these methods have limitations since OIT and sulfur content measurements cannot distinguish between antioxidant and synergist and UV spectroscopy is useless with XLPE. It is often very difficult to distinguish between antioxidant or synergist and oxidation in FTIR spectra. Thus, a reliable evaluation of residual antioxidant content in cable insulation may involve the combination of several analytical techniques.

CONCLUSIONS

The principal features observed in this study are as follows:

- Oxidation is present in all cables, even in unaged ones.
- Aging leads to more oxidation especially near the conductor shield.
- There are more ketones in XLPE than in HMW-PE cables (certainly because the antioxidant in XLPE is partially grafted).
- The aged distribution cables studied appear to be more oxidized than the aged transmission cables but they have been in service for longer time.
- No trace of Santonox R was detected in the cables studied.
- The antioxidant compound often used in cable insulation is a mixture of Irganox 1035 and DSTDP.

- Great care is required to distinguish oxidation from antioxidant in FTIR absorbance spectra of cable insulations.
- OIT measurements can reliably evaluate the resistance to oxidation of a cable insulation but it requires calibration curves for a quantitative estimation of antioxidant content.
- Considering the fact that unaged cables are already slightly oxidized one may wonder whether the present antioxidant compound is the most appropriate one for cable applications.

ACKNOWLEDGMENTS

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