Master of Science Thesis

ANALYSIS OF BY-PRODUCTS FROM XLPE PRODUCTION FOR CABLE INSULATION

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ABSTRACT

Polyethylene is used in the manufacturing of cables. Because of the amount of high voltages which is transmitted through the cables at elevated temperatures, crosslinking of PE is necessary for stabilizing the insulation. This is done chemically by the addition of crosslinking agents such as dicumyl peroxide. This agent starts an autonomic process when subjected to heat after extrusion. However, crosslinking generates by-products which for dielectric, mechanical and electrical test and performance considerations need to be removed from the cables, thus the process of degassing. Degassing or thermal treatment is an important process with cable manufacturing. After thermal treatments on the material XLPE different analytical techniques can be applied to measure the progress of degassing.

Analysis of XLPE cables is essential in order to determine the contents of by-products after crosslinking and degassing. Many analytical methods such as TGA, DSC, GC-MS, HPLC, FT-IR, TL and EL are used to conduct these measurements. In this project, after looking at the chemical and the physical characteristics of the interesting components of XLPE, HPLC with a UV detector was found to be best suited to carry out quantitative and qualitative analysis. This required an analytical method to be developed. An ideal wavelength of 250 nm, a buffer solution of pH 2.3 made of nano pure water and formic acid, a C 18 column and a gradient mobile phase were found to be good instrumentation settings.

Reproducibility was obtained with regards to how standard analytes appear in the chromatogram singularly and in mixed solutions. In the quantitative analysis, calibration curves were derived from data produced by standard samples. The same calibration curves were used to determine the concentrations of acetophenone, alpha cumylalcohol, alpha methylstyrene and dicumyl peroxide in both degassed and not-degassed XLPE samples. Degassing has also been carried out in the lab, in a chamber, at a fixed temperature of 70 ºC and varying times. Results have shown that the more time a sample has been degassed, the lesser the concentrations of analytes to be determined. Weight loss, a simple test for degassing, has been observed in all the cables that underwent thermal treatment.

Key words: XLPE Cables, Crosslinking by-products, degassing, methane, cumyl alcohol, acetophenone, alpha methylstyrene, dicumyl peroxide, TGA, DSC, HPLC, GC-MS, FT-IR, TL, EL

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INTRODUCTION

Polyethylene (PE) has been extensively used as an insulation material for cables of medium voltage, high voltage and even extra high voltage. Cross-linked polyethylene (XLPE) is almost universally being used in extra high voltage cables that operates at voltages up to 500 kV [1].

Polyethylene is a long chain polymer manufactured through the polymerisation of ethylene gas. At its introduction, thermoplastic PE was very popular, compared to paper insulation, as insulation for cables because of its low cost, electrical properties, processability, moisture and chemical resistance, and low temperature flexibility. A significant design issue with PE, in its thermoplastic state, was that its temperature of operation was limited to 70˚C [2]. In a power cable, a high current flow through the central conductor and the extruded insulation surrounding the conductor is subjected to high temperatures and temperature gradient. Consequently, it could not match the temperature rating of paper-oil insulated cables. This problem was solved with the advent of XLPE, which has the ability to match the thermal rating of paper-oil insulated cables and provide the freedom from hydraulic problems of oil-filled cables. When using XLPE as cable insulation, it is possible to achieve a rated maximum conductor temperature of 90 ˚C and a 250 ˚C short circuit rating. Crosslinking will increase the resistance to thermal deformation and enhance dimensional stability. The crosslinking of Low Density Polyethylene (LDPE) to form XLPE was first accomplished by Gilbert and Precopio in 1955 at the GE Research Laboratory located in Niscayuna, NY [2].

Chemical crosslinking by dicumyl peroxide, shown in figure 1, is widely used, but this method creates volatile crosslinking by-products such as acetophenone, cumyl alcohol, α-methylstyrene, and consequently methane and water. These by-products affect the insulation properties of XLPE cable and the ones such as methane constitutes a health and safety issue due to its flammability during the jointing/insulation procedures.

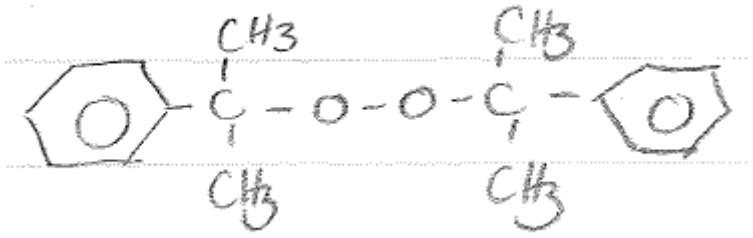

Figure 1: Molecular structure of dicumyl peroxide

Figure 2 shows the decomposition routes of dicumyl peroxide forming by-products such as acetophenone, alpha cumylalcohol and methane.

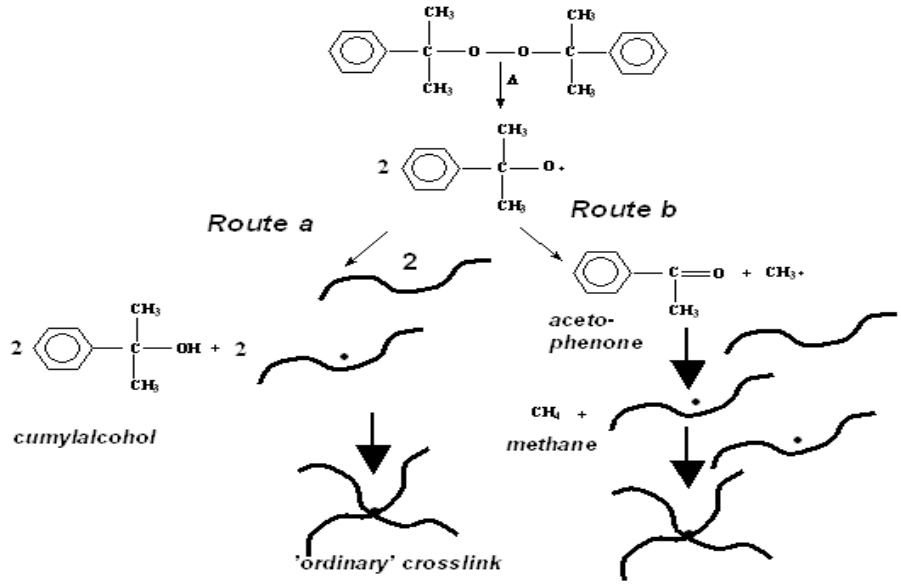

Figure 2: Peroxide initiated Crosslinking of PE [2].

In enhancing crosslinking reactions, antioxidants are widely used. The influence of antioxidants on crosslinking of polyethylene has been determined through vigorous studies and research works and volumes of information regarding this particular area of studies are been published. Antioxidants in polymeric insulation material have a radical trapping and/or peroxide decomposition function that prevent oxidative degradation. These functions may influence the crosslinking reaction of polyethylene [3]. There are many types of antioxidants used in the crosslinking of polyethylene. Some examples are the amine-, phenolic- and thioether-type shown in figure 3.

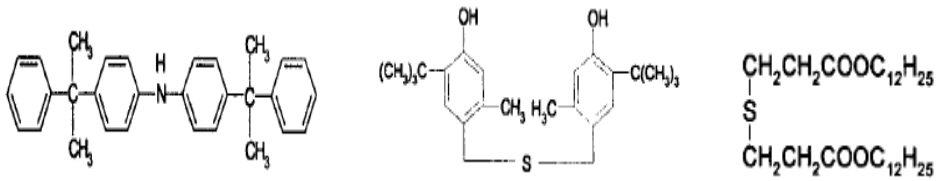

Figure 3: Molecular structures of antioxidants; amine, phenolic and thioether types.

It is well known that free radicals are formed by the thermal decomposition of dicumyl peroxide (DCP), and these free radicals abstract hydrogen from polyethylene chain to form alkyl radicals. The DCP initially decomposes to form a cumyloxy or methyl radical and acetophenone (see figure 2).

Acetophenone, a ketone, decreases the impulse breakdown strength of XLPE cables and traps space charge, especially under the application of dc voltage, to form chemically complex degradation products. However the crosslinking by-products generally produce a temporary improvement in ac breakdown strength. Because the by-products are polar they can grade the electric field around points of electric stress enhancement and jeopardise the detection of

defects during initial tests performed on the manufactured cable. Temporary improvement of cable performance also could be caused by gaseous by-products filling up voids and increasing the electrical tree inception voltage [1]. During installations temperatures can rise up to above 150 ˚C. This will lead to water being generated from cumyl alcohol, which is one of the by-products of dicumyl peroxide. As the cable cools down, the water condenses to form water filled cavities leading to water treeing. All together, the probability of insulation failure is significantly increased.

Hence, prior to installation, underground power cables, especially of the high-voltage class, must be subjected to a thermal treatment or conditioning called degassing in order to reduce the concentration of the crosslinking by-products to a negligible level [1]. Generally, one could argue that the three main considerations responsible for degassing are mechanical considerations, dielectric considerations and electric test and performance considerations. The process whereby the by-products of the crosslinking reaction are removed is almost universally termed degassing [2]. Again the removal of these by-products is of enormous importance especially for high voltage (HV) and extra high voltage (EHV) cables. Besides the fact that methane is flammable and can catch fire or create explosion during installation, its presence can also lead to issues in service as the gas pressure can create defects in the shielding and in the joints. For cables equipped with a metal barrier the gaseous products can exert a pressure, especially on the joints and terminations, and eventually cause a system failure [4]. The polar by-products such as acetophenone and cumyl alcohol contribute to dielectric losses and therefore need to be removed. It's worth noting that degassing is not only the removal of crosslinking by-products but also the redistribution of the by-products.

Degassing is a diffusion controlled process and is dependent on many parameters including temperature, thermal process history, morphology, orientation, crystallinity and degree of crystallinity, annealing, solubility and vapour pressure of the diffusion molecule(s) [4]. In practice the temperature used for the degassing operation range between 50 ˚C and 80 ˚C. However care needs to be taken not to damage the core if high degassing temperatures are used, especially for heavy cables [4]. The picture in figure 4 shows XLPE cable insulation without cable core produced at Nexans Norway AS..

Figure 4: Picture highlighting XLPE cable insulation without the core.

Untreated cross-linked cables would retain the by-products within their structure for a very long time, generally for more than a decade. Initially after cable crosslinking is completed, the by-products are evenly distributed throughout the insulation. But over time they diffuse out of the cable and are depleted more from the outer insulation layers than the inner one [1]. Any positive effects the by-products may have on the performance of the cable during testing operations will fade away with time as the by-products also diffuse away. Hence it is advantageous to degas cross-linked power cables and reduce the by-products to a low and stable level so that the electrical tests, performed prior to cable installation, would reveal the true condition of the insulation.

Methods of Degassing

To ensure that cables have the sufficient dielectric properties and that any voids are free of gas, cable manufacturers ensure that sufficient degassing (sometimes termed vaporization or conditioning) has occurred during the production process. This means that when finally tested before release, all parties can be assured that the true properties of the complete cable are measured. The increased thickness of transmission class cables and the high boiling points of the by-products imply that any natural degassing process must be augmented by high temperature treatments. Such treatments are used before the metallic sheath is applied as its presence dramatically reduces the rate of degassing [2]. It is interesting to note that the importance of degassing is recognised in the Insulated Cable Engineers Association (ICEA) specification, which requires a minimum of 5 days between testing and the finishing of the cable. The degassing of power cables is performed in large, heated chambers that are well ventilated to avoid the build up of flammable gases [1], for obvious safety reasons. These devices can consume considerable amounts of energy and factory space. Sometimes, to assist the cable in attaining the required temperature quickly, the heated chambers are augmented by conductor heating [4].

The temperatures used for practical degassing can range between 50 ˚C and 80 ˚C, with 60 ˚C to 70 ˚C being the most preferred range. The range between 70 ˚C and 80 ˚C has been shown to work reliably only for smaller MV cables. When degassing a cable, especially at high temperature, very considerable care must be exercised not to damage the core. The attendant thermal expansion and softening of the insulation have been showed to lead to undue deformation of the core [2]. The following figure shows the pattern of deformation with regard to temperature changes

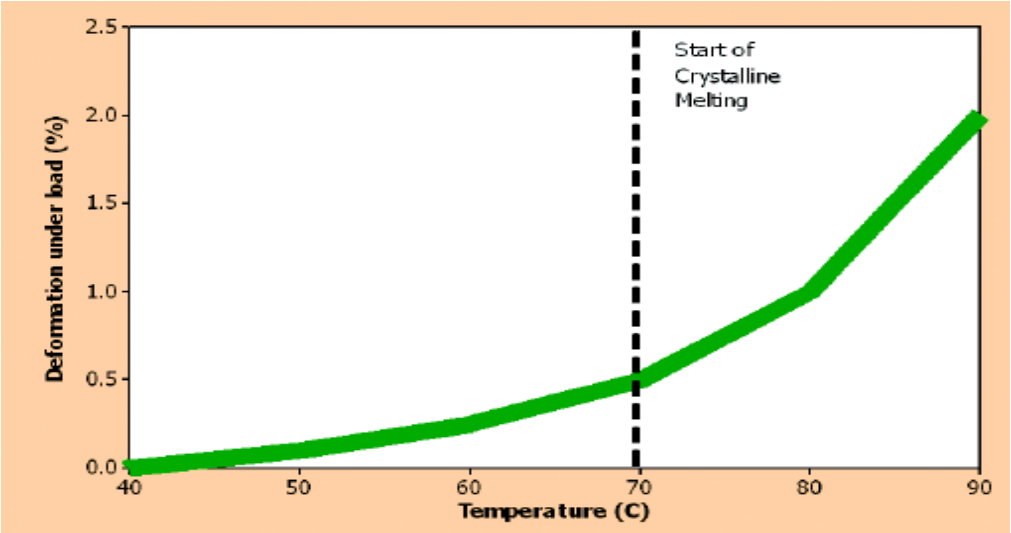

Figure 5: Effect of temperature on the expansion, and thus softening, of XLPE cable insulation (MV to EHV) [1].

It is also very common for the degassing temperature to be decreased as the cable weight is increased, this is particularly important for HV and EHV cables [2].

Measurement of Crosslinking By-Products

One of the most crucial steps in understanding degassing is the measurement of the initial state of the cables and how any treatments proceed with time. When approaching an analytical problem, there is a general sequence of steps that are followed in figuring out the protocols and analytical techniques to use [5]. The progress of degassing can be determined by applying analytical techniques:

- Optical method
- Thermogravimetric Analysis (TGA)
- Weight Loss
- Differential Scanning Calorimetry (DSC)
- Fourier Transform Infrared Spectroscopy (FT-IR)
- High Pressure Liquid Chromatography (HPLC)
- Gas Chromatography - Mass Spectroscopy (GC-MS)

Optical Method

A novel optical method has been developed to determine the concentration of by-products in chemically cross-linked polymeric materials [1]. The optical method involves the detection of thermoluminescence (TL) emitted by the cross-linked by-products during the degassing, and the intensity of the emitted light provides a direct measure of the concentration of the by-products in the material. Light emission in XLPE can occur without voltage application. During the voltage application, electroluminescence (EL) occurs above a certain threshold voltage and is caused by the injection of electrical charges from the electrodes into the polymer [1]. EL requires high voltage and can occur at cryogenic, room and high temperature. TL occurs without voltage application and can happen at room temperature for

XLPE. TL intensity depends on the temperature gradient. Figure 6 and 7 show an optical set-up for measuring the concentration of cross-linking by-products and also a graph on by-product decrement. PMT in figure 6 stands for Photomultiplier tube. PMT amplifies and detects the radiations from the chamber.

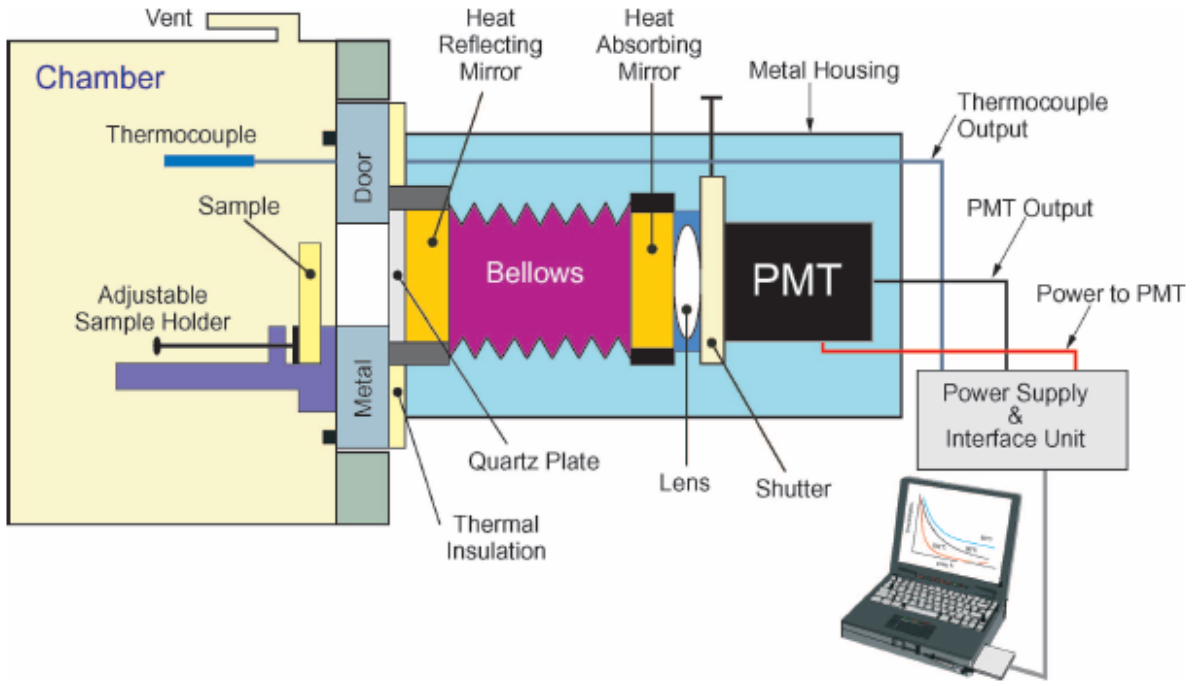

Figure 6: Setup of the optical technique for measuring the concentration of crosslinking by-products [1].

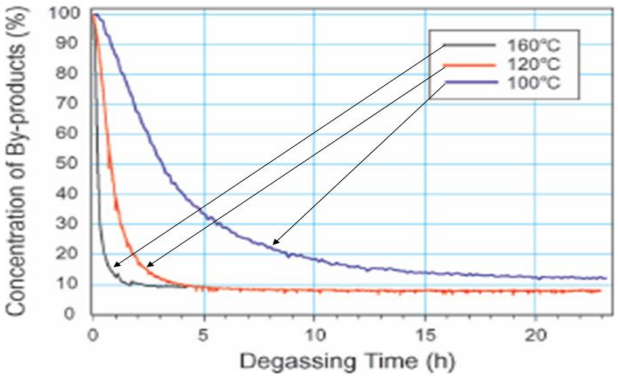

Figure 7: Effect of the degassing temperature and time on the concentration of crosslinking by-products in XLPE cables [1].

Thermogravimetric Analysis, TGA

This is a type of testing that is performed on samples to determine changes in weight in relation to change in temperature. Such analysis relies on a high degree of precision in three measurements: weight, temperature and temperature change [6]. TGA is commonly employed in research and testing in order to determine characteristics of materials such as polymers, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, solvent residues, and the estimation of corrosion kinetics in high temperature oxidation [6]. In thermogravimetric analysis the mass of a sample in a controlled atmosphere is recorded continuously as a function of temperature or time as the temperature of the sample is increased (usually linearly with time). A plot of mass or mass percent as a function of time is called a thermogram, or a thermal decomposition curve [7].
In XLPE analysis, samples should be taken from the inner, middle and outer part of the insulation. The temperature could be raised could, for example, to 175 ˚C with a minimum heating rate of 50 ˚C/min. It could be conducted for 30 minutes at a temperature of 175 ± 3 ˚C, according to HD632 S1 1998 part 1 2.4.15 [4]. Figure 8 shows TGA obtained from U.S. Navy website.

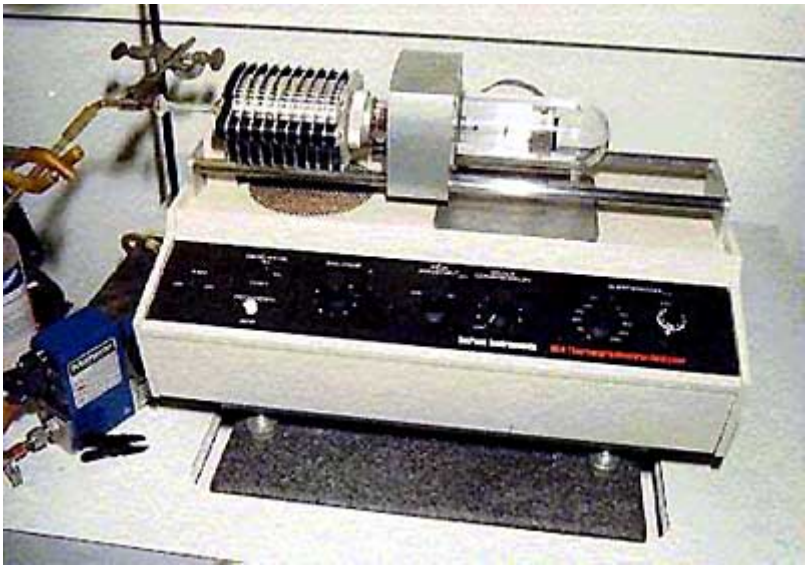

Figure 8: High resolution modulated thermal gravimetric analyzer [8]

Weight Loss

A very good technique of determining degassing is weight loss. The change in weight as a function of degassing time is plotted. It is a simple technique that requires little to perform. However, this technique does not give a qualitative or quantitative data of components in the cable.

Differential Scanning Calorimetry (DSC)

DSC is a thermal technique in which differences in heat flow into a substance and a reference are measured as a function of sample temperature while the two are subjected to a controlled temperature program [7]. The main application of DSC is in studying phase transitions, such as melting, glass transitions, or exothermic decompositions [9].
In XLPE analysis, melting, crystallisation and second melting with identified heating rates will lead to obtaining important information about material.

Fourier Transform Infrared Spectroscopy (FT-IR)

In FT-IR, spectra are collected based on the radiation that is being reflected in the sample. For the infrared region, the most important method for observing the entire spectrum at once is Fourier transform spectroscopy [10]. FT-IR instruments are known for their unique characteristics of speed, high resolution, sensitivity and unparallel wavelength precision and accuracy [7]. However in XLPE, cumyl alcohol has a very weak absorption to be monitored compared to acetophenone. Figure 9 is an example of FT-IR spectra for XLPE before and after degassing.

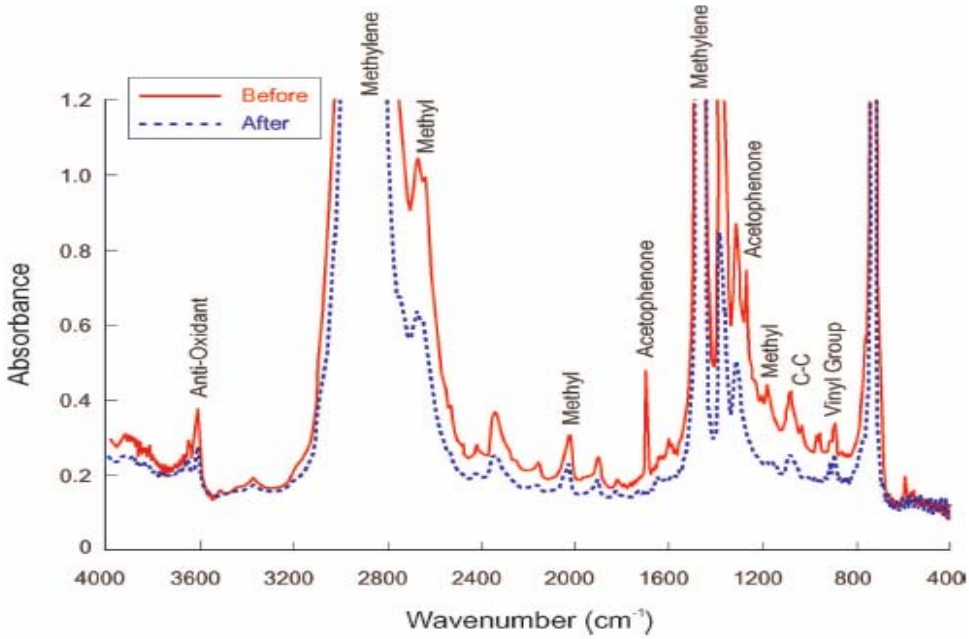

Figure 9: FT-IR of XLPE before and after degassing [1]

High Performance Liquid Chromatography

HPLC is the most widely used of all the analytical separation techniques. The reason for its popularity is its sensitivity when combined detectors such as UV or Fluorescence detectors, its ready adaptability to accurate quantitative determinations, its suitability for separating non-volatile species or thermally fragile ones, and above all, its widespread applicability to substances that are of prime interest to industry, to many fields of science and to public [7]. In determining cumyl alcohol and acetophenone in XLPE, acetonitrile could be used as the mobile phase after extraction of the sample at, for example 72 ˚C for 2 hours of which 10 μL is injected [4]. However another method will be developed in this project for the determination of XLPE by-products.

In order to determine the concentration of the various constituents in the XLPE insulation, one could employ one of the following methods.

INTERNAL STANDARD METHOD

A very important method used in gas chromatography as well as liquid chromatography is Internal Standard Method. The challenge is to find a reference compound, different from the analytes but yet very similar, that will not interfere with the natural existing components in the material one is about to produce. When a reference analyte is identified and used, the concentrations of existing components can be determined by looking at the relativity between response factors and the concentration of the reference substance, as formulated in Equation 1.

$$\frac{C_p}{C_{st}} = \frac{a_p}{a_{st}} \psi_p \qquad (1)$$

Where (a_p) is the area of the peak for the component p.

(a_{st}) is the area of the peak for the standard

(ψ_p) is the response factor for the component p

(C_p) is the concentration of the component p

And (C_{st}) is the concentration for the standard.

EXTERNAL STANDARD METHOD

In this method the solute chosen as the reference is been analysed and chromatographed separately from the sample. The results of the different chromatograms are then compared. It is vital to keep the chromatographic conditions the same throughout the whole process. In this method, the reference solutes can be the same as those present in the actual sample. The formula shown in Equation 2 could be used to obtain unknown information.

$$C_p(s) = \frac{a_p(s)}{a_p(st)} = C_p(st) \qquad (2)$$

Where:

$C_p(s)$ is the concentration of analyte in the sample

$a_p(s)$ is the area of the analyte in the sample

$a_p(st)$ is the area of the standard analyte in the reference sample

And $C_p(st)$ is the concentration of the standard analyte in the reference sample.

Figure 10 shows the HPLC instrument used in this research at University of Stavanger (UiS).

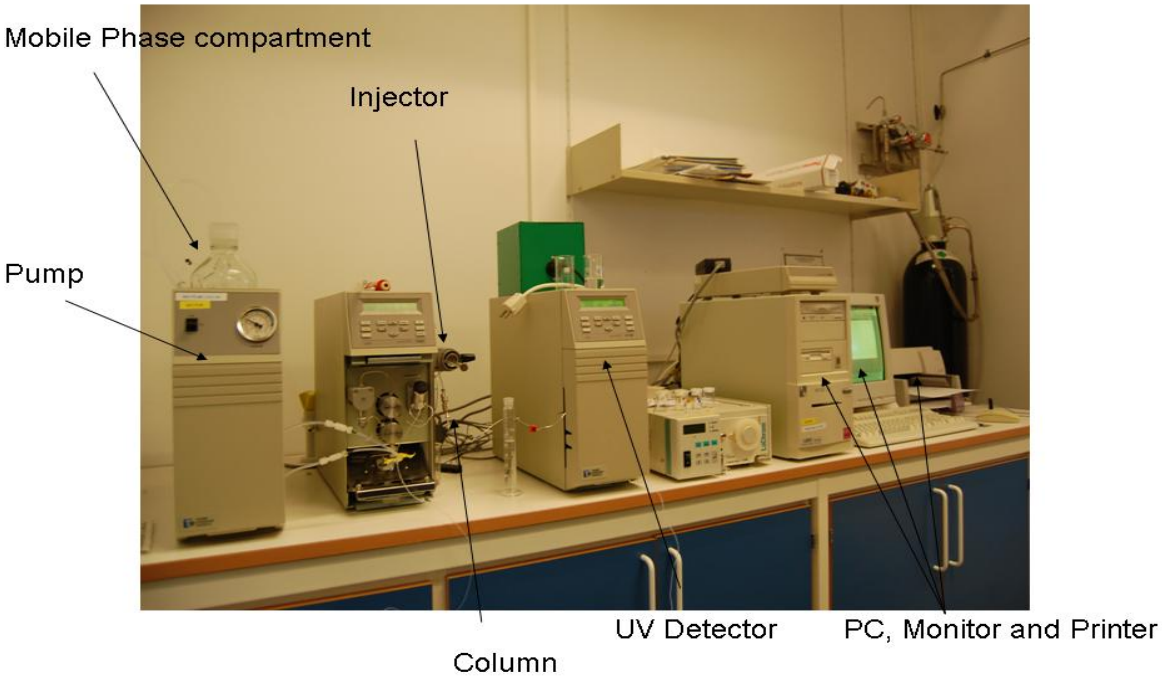

Figure 10: HPLC instrument set-up. From left to right: Pump with Mobile Phase compartment on it, injector and column, UV-detector, Thermo compartment, PC, monitor and printer.

Gas Chromatography - Mass Spectroscopy

Gas chromatography-mass spectroscopy (GC-MS) can be used for separation processes and quantitative and qualitative analysis of volatile molecules. Temperature gradient is central in a GC-MS to get a good separation. The use of MS as a detector for GC (capillary GC), is relatively simple because the mobile phase will not cause any interference and also because the analytes are volatile and already in a gas phase as it enters the MS where either electron ionisation or chemical ionisation can take place [11]. For XLPE analysis, samples could be heated at 60 ˚C for 1.5 hours and then 0.3 to 0.5 ml of the gas could be injected in the GC [4]. Figure 11 is a picture of GC-MS instrument taken at a UiS lab. Figures 12 and 13 are examples of gas chromatography-mass spectrometry spectra for untreated and degassed (one week in vacuum at 80 ˚C) XLPE.

Figure 11: GC-MS instrument set-up at University of Stavanger.

Figure 12 and figure 13 shows examples of a GC-MS chromatogram highlighting the retention time for the analyte and the abundance which is proportional with the analyte concentration.

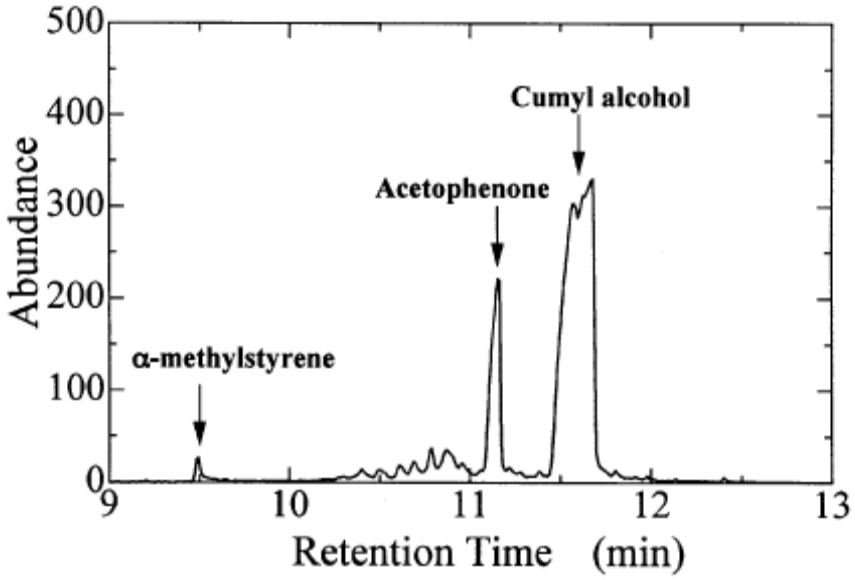

Figure 12: GC-MS chromatogram for untreated XLPE [12].

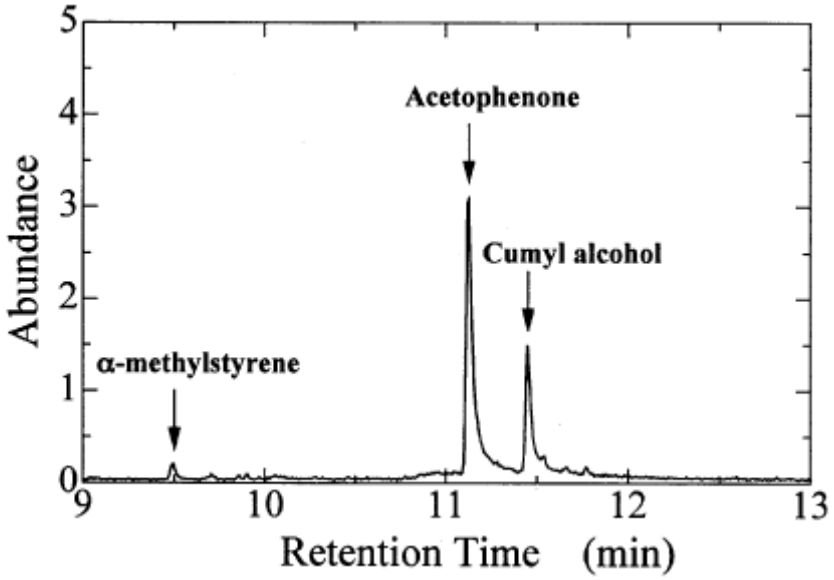

Figure 13: GC-MS chromatogram for treated XLPE [12].

Experience with a large number of analyses for many different cable designs has shown that weight loss on a full cable is the most practical and simplest way of determining the by-product level within XLPE insulations for a range of conditions. Experience has also shown that HPLC is the best method for determining the level of individual solid by-product components. Table 1 gives an overview of different methods for determining by-product concentrations of XLPE.

ANALYSIS OF METHANE

An important by-product found in XLPE cable insulation is methane. Its presence in the cable is a very serious challenge in production of high voltage cables by e.q Nexans, not only because of its role in the electrical, mechanical performance of the cable, but also its danger as to flammability and explosion. After the breakdown of dicumyl peroxide in the enhancement of crosslinking process, the amount of methane molecules nearly equals the amount of acetophenone molecules as indicated in figure 2. This will not be the case after degassing the cable.

Table 1: Experimental methods for determining by-product concentration [2].

Method	Advantages	Concerns
Weight Loss- Thermogravimetric analyser (TGA)	Fast Able to provide data for different parts of the insulation	Requires special equipment- thermogravimetric analyser Considers small insulation samples- 100 mg Uses non practical temperatures Not possible to separate different by-product species Loss of by-product during sample preparation
Weight loss- cable weight	Simple Looks at a whole cable Uses practical degassing temperatures	Not possible to separate different by-product species Takes considerable time to reach equilibrium Loss of by-products during sample preparation
Extraction high pressure liquid chromatography (HPLC)	Looks at different by-product species Good for non-gaseous by-products Fast Able to provide data for different parts of the insulation	Requires special equipment- HPLC Considers small insulation samples-1 g Calibration can be tricky Affected by variations in extraction yield Loss of by-products during sample preparation Not possible to measure gas simultaneously
Volatilization- gas chromatography mass spectrometry (GC-MS)	Looks at different by-product species Good for gaseous by-products Fast Able to provide data for different parts of the insulation	Requires special equipment- GC-MS Considers small insulation samples- 1 g Difficult to calibrate for all species at the same time Uses non-practical temperatures Effected by variations in extraction yield Loss of by-products during sample preparation
Gas Volume estimation after volatilization	Simple Looks at a whole cable Directly relevant if practical temperatures are used	Difficult to interpret – gas losses and condensation effects Often uses non-practical temperatures Species decompose/mutate during measurement Loss of by-products during sample preparation
Fourier transport infrared spectroscopy (FT-IR)	Can look at many by-products (special peaks) at the same time Minimal propagation Cross-sectional scans easy to acquire	Large effect from surface concentrations Qualitative calibration can be difficult

Degassing Calculation

Direct measurements of degassing are extremely useful and essential. However it is not practical to conduct such measurements for all different cable designs or potential treatment temperatures. Thus, it is a very common and useful practice to use computational methods to extend the fundamental understanding derived from the degassing experiments [2]. The desorption of the by-products (i.e., the change of concentration C at time and position x) of the crosslinking reaction can be simply described mathematically using Fick's Law [2].

$$\frac{\partial C}{\partial t} = \rho D \bar{V}^{2} C \qquad D = D_o \exp\left(\frac{-E_D}{RT}\right) \tag{3}$$

Where:

D= Diffusion Constant
T= Temperature
E_D = Activation Energy
ρ = Material density
R= Universal Gas Constant
D_o = Prefactor that relates the measured diffusion data at different temperatures
$\bar{V}$ = Mathematical symbol, del, describing derivations in the system

The differential equations are normally solved using either analytical solutions for simple geometries (infinite flat plane) or numerical methods for prescribed set of boundary conditions. The most common solution method uses straightforward finite-element modelling (FEM), either for the steady state case or for more complex transient case and coupled electrical thermal effects [2]. One critical aspect of calculating the distribution of by-products is the selection of correct boundary conditions for the FEM model. In principle three types can be used [2].

- Degassing occurs freely from both the outer and inner surfaces.
- Degassing occurs freely from the outer surface, but it is completely blocked from the inner surface due to the presence of the conductor and any strand blocking materials.
- Degassing occurs freely from the outer surface, but it occurs in a very constraint manner from the inner surface.

In doing assumptions for modelling one should consider experimental data as well as focus on computational simplicity. The following figure highlights by-products distribution in XLPE through computational simulation.

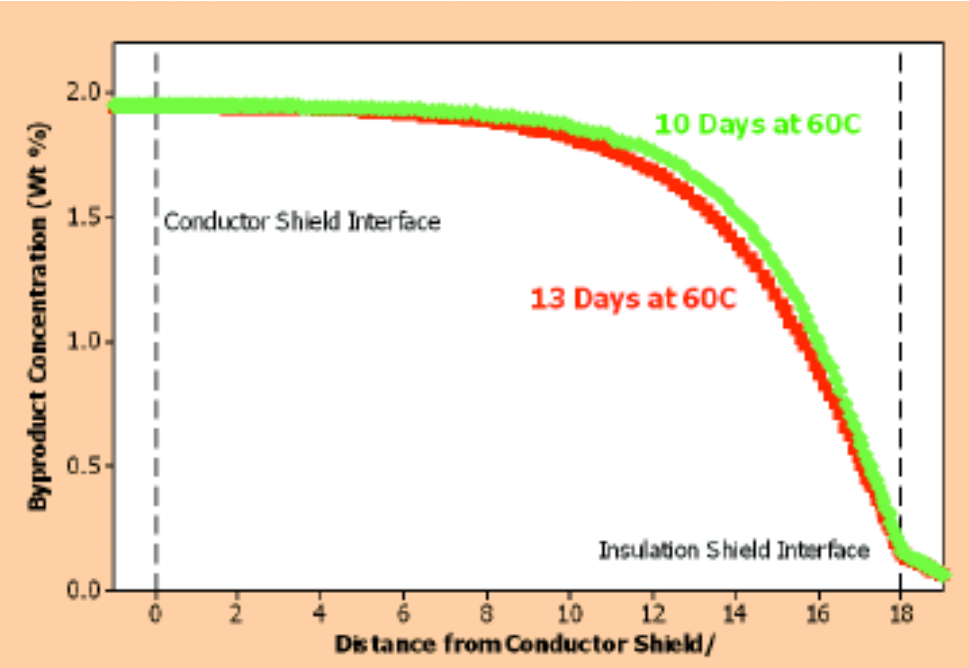

Figure 14: Spatial distribution of by-products in a HV XPLE power cable(18 mm XLPE, 1 mm semicons) calculated using FEM, including the semicon layers. The simulation has assumed that there is no loss of by-products from the inner conductor [2].

Degassing Time

Degassing time is impacted depending on the parameters employed in the process as well as the material composition. The presence of polymeric cable oversheath has a significant effect and retards the rate at which by-products are lost. If a metallic foil or sheath were included, an even lower rate of loss would be expected [2]. The by-product concentration will reduce with storage time as the by-products diffuse out of the material and this will impact the electrical performance of the cable been degassed.
Degassing is an important process to ensure the stability of electrical properties and the effectiveness of many test procedures. Equally, the times for degassing at ambient temperatures are prohibitively long. Thus, it is virtually universal to use high temperature treatments to achieve practical degassing times. The precise time/temperature conditions depend upon the details of the cable design [2].

EXPERIMENTAL METHOD

The by-products in an XLPE cable produced and samples taken in different dates will be analysed. Some of these cables have been degassed prior to been sent to the research lab at the University of Stavanger. The samples where sent through DHL services and therefore out of cooling storage for a period of 24 hours. Before the analysis, the sample cables were kept in the freezer at – 20 ºC. The following standards were ordered:

Table 2: Standards used in the analysis in the research lab at the University of Stavanger.

Chemical name	Trade name	Physical State	Supplier	CAS Number	Chemical Formula
Dicumyl Peroxide	Dicumyl Peroxide 98%	Dull white. Fine Crystals	Sigma-Aldrich Norway AS	80-43-3	C18H22O2
Alpha Cumyl Alcohol	2-Phenylpropan-2-ol	Weak colour. Fragments	Sigma-Aldrich Norway AS	617-94-7	C9H12O
Acetophenone	Acetophenone	No Colour liquid	Sigma-Aldrich Norway AS	98-86-2	C8H8O
Alpha methylstyrene	2-Phenylpropene	No colour liquid	Sigma Aldrich Norway AS	98-83-9	C9H10

After numerous considerations based on the initial UV-vis tests on all the standard products, an HPLC with a UV detector is used for the separation and detection of the analytes or the by-products. Acetophenone, alpha cumylalcohol, alpha methylstyrene and dicumyl peroxide for that matter are all aromatics with conjugated bonds making them capable of absorbing lights at a certain wavelength.

In order to know what wavelength one has to programme in the detector, UV-vis tests were run for all the standards.

A UV visible spectrophotometer instrument, CARY 50 SCAN, Instrument number 230C-8, was used to determine a spectrum for the individual standards.

Dicumyl Peroxide

A 10 mL solution of 2.200 g/L equivalent to 0.00800M DPO was made. The solution was then diluted to 0.022 g/L solutions and measurements taken.

Acetophenone

A solution of 0.99 M was diluted down to a concentration of 3.91E(-6) M by using methanol as solvent.

Alpha Methylstyrene

A solution of 0.99 M was diluted down to a concentration of 99E(-6) M by using methanol as solvent.

Alpha Cumylalcohol

0.2048 g was dissolved into a 10 ml methanol approximating 20.48 g/l. This was then diluted down to a concentration of 0.4096 g/l .

High Performance Liquid Chromatography analysis with Ultraviolet detector (HPLC-UV)

HPLC model: Thermo Separation Product, tsp.
Spectra System P2000, UV-vis detector set at 250 nm, SN4000,

Detector: Spectra System UV 1000, Deuterium lamp

Column Specifications:

- SS WAKOSIL
- 150 X 4.6 mm (Length x Diameter)
- C18 ar
- 5 µm particle size
- Injection volume 5 µL.

Buffer Composition:

- 1 liter of Nanopure water and approximately 1 mL of 97 % formic acid give it a pH of 2.3. This is the buffer solution
- In compartment A of the mobile phase unit is 95 % Methanol and 5 % buffer.
- In compartment B of the mobile phase unit is 95 % Buffer and 5 % Methanol.

Finding the best conditions for a separation by liquid chromatography involves manipulating all the equilibria that affect the reactions. The following table shows how the mobile phase gradient was set up. The time is increasing linearly for the gradients (linear gradient).

Table 3: Gradient MP set-up in the HPLC.

Time (minutes)	A (95% MEOH)	B(5% MEOH, pH 2.3)	Flow (mL/min)
0.0	50	50	0.80
10	70	30	0.80
20	100	0	0.80
30	100	0	0.80

Qualitative Analysis

First the standard chemicals for acetophenone, alpha cumylalcohol, alpha methylstyrene and dicumyl peroxide were analysed on the HPLC to find the optimal separation parameters, and the detection time for the compounds so that we can recognise how much separation one can get and also to note on individual retention times for each standard chemical.

Quantitative Analysis

Here one will be looking at the methods through which one can determine the relative or absolute amount or concentration of the various constituents to be found in the XLPE insulation material by using High Performance Liquid Chromatography, HPLC. In doing so, units of volume and concentration become important to note in order to be as close to 100 % correct as possible. Precision and good working environment is essential to stop cross contaminations.

Making of Samples, HPLC analysis and Calibration Curve

- Use a 3mL sample container.
- Weigh in 0.5 µL of alpha methylstyrene, this corresponds 5.85E(-05) grams.
- Weigh in 0.5 µL of acetophenone corresponding 5.94E(-05) grams
- Weigh in 0.0520 grams alpha cumylalcohol
- Weigh in 0.0566 grams dicumyl peroxide
- Add in 2 mL of the solvent, methanol, and shake until everything dissolves. This is the stock solution or Mix Solution. It is from the mix solution that the five calibration solutions, C1, C2, C3, C4 and C5, will be made.
- Pipette out 200 µL of Mix Solution into a new sample container, add 1.7 mL methanol and 100 µL buffer solution. This will be the first of five calibration solutions so call it C1.
- Pipette out 500 µL of C1 and mix with 400 µL methanol and 100 µL Buffer. Mark it C2.
- Pipette out 500 µL of C2 and mix with 400 µL methanol and 100 µL Buffer. Mark it C3.
- Pipette out 500 µL of C3 and mix with 400 µL methanol and 100 µL Buffer. Mark it C4.
- Pipette out 500 µL of C4 and mix with 400 µL methanol and 100 µL Buffer. Mark it C5

This technique used is based on external standard method, for the quantitative determination of the components in XLPE insulation cable. Calibrations curve were made for the four components (acetophenone, alpha cumylalcohol, alpha methylstyrene and dicumyl peroxide) by using the data obtained from the analysis of C1, C2, C3, C4 and C5, on HPLC, consisting of the standard chemicals. The curve function obtained from calibration curves would be used to calculate the concentrations of the same substances in insulation materials. In making the calibration curves, known concentrations of the standard solutions were plotted against the areas obtained from the chromatograms.

Real Tests for XLPE Cables

In total, seven cable samples were sent over to the research lab at the University of Stavanger for analysis. In this work, they will be referred to as samples I, II, III, IV, V, VI and VII. Table 4 shows the details of the cable samples.

Table 4: Different cables samples and their specifications.

SAMPLE	SPECIFICATIONS	DEGASSED
I	PT2-27.06.08	YES
II	PT2-14.07.08	YES
III	HVDC (XLPE) 2500 mm^2, 05.01.09	NO
IV	HVDC (XLPE) 2500 mm^2, 18.02.09	YES. 1008 hours, 70 ºC
V	HVDC 95 mm^2 Cu (150kV), Cable nr 1 (x-1), 10.01.09, 5:45 pm	YES. 102 hours, 70 ºC
VI	HVDC 95 mm^2, Cable nr 2 (x-2), LT 13-25	YES. 12 hours, 70 ºC
VII	HVDC 95 mm^2, Cu (150 kV), Cable nr 3 (x-3), 08.01.09	YES. 45 hours, 70 ºC

HPLC analysis of cable samples

Two analogue sample pieces were taken from each cable sample. One piece was immersed into methanol and the other one was a more diluted by adding buffer into it. The ones immersed into methanol will be used in the quantitative analysis while those with some buffer added into will be used to show how the tops change on the chromatogram. That is why the volume of buffer defers in some of the samples. One important thing in a qualitative and quantitative liquid chromatography is the solubility of analytes in the mobile phase, as well as taking into account the concentrations of the materials to be analysed in the sample.

<u>Sample I</u>

- 4.8 grams of the sample was cut into small bits and added into 25 mL methanol for extraction.
- Under mixing and at 70 ºC, extraction was carried out for 2 hours.
- The solution was filtered with a filter paper and samples prepared for HPLC analysis. Two sub samples were made out of the solution: The first sub-sample has only methanol as a solvent and the second sub-sample has methanol and buffer as solvents, at ratio of 6:4.

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Sample II

- 5.1 grams of the sample was cut into small bits and added into 25 mL methanol for extraction.
- Under mixing and at 70 ºC, extraction was carried out for 2 hours.
- The solution was filtered with a filter paper and samples prepared for HPLC analysis. Two sub samples were made out of the solution: The first sub-sample has only methanol as a solvent and the second sub-sample has methanol and buffer as solvents, at ratio of 6:4.

Sample III

- 4.8 grams of the sample was cut into small bits and added into 25 mL methanol for extraction.
- Under mixing and at 70 ºC, extraction was carried out for 2 hours.
- The solution was filtered with a filter paper and samples prepared for HPLC analysis. Two sub samples were made out of the solution: The first sub-sample has only methanol as a solvent and the second sub-sample has methanol and buffer as solvents, at ratio of 9:1.

Sample IV

- 2.9 grams of the sample was cut into small bits and added into 25 mL methanol for extraction.
- Under mixing and at 70 ºC, extraction was carried out for 2 hours.
- The solution was filtered with a filter paper and samples prepared for HPLC analysis. Two sub samples were made out of the solution: The first sub-sample has only methanol as a solvent and the second one has methanol and buffer as solvents at a ratio of 9:1.

SAMPLE V

- 1.0 grams of the sample was cut into small bits and added into 25 mL methanol for extraction.
- Under mixing and at 70 ºC, extraction was carried out for 2 hours.
- The solution was filtered with a filter paper and samples prepared for HPLC analysis. Two sub samples were made out of the solution: The first sub-sample has only methanol as a solvent and the second sub-sample has methanol and buffer as solvents at ratio of 9:1.

Sample VI

- 1.4 grams of the sample was cut into small bits and added into 25 mL methanol for extraction.
- Under mixing and at 70 ºC, extraction was carried out for 2 hours.
- The solution was filtered with a filter paper and samples prepared for HPLC analysis. Two sub samples were made out of the solution: The first sub-sample has only methanol as a solvent and the second sub-sample has methanol and buffer as solvents at a ratio of 9:1.

SAMPLE VII

- 1.1 grams of the sample was cut into small bits and added into 25 mL methanol for extraction.
- Under mixing and at 70 ºC, extraction was carried out for 2 hours.
- The solution was filtered with a filter paper and samples prepared for HPLC analysis. Two sub samples were made out of the solution: The first sub-sample has only methanol as a solvent and the second sub-sample has methanol and buffer as solvents at a ratio of 9:1.

Degassing

Two samples were degassed at a fix temperature but different intervals in an oven. These samples were later been analysed to see any difference in weight loss and also to see if the concentration of its components determined before degassing have been altered by the degassing process. These two samples are Sample III and Sample IV. Tables 5 and 6 below show the degassing settings of the samples in a total of five parallels.

Table 5: Parallels of Sample III and their degassing time and temperature

Parallels	Weight, g	Degassing time, h	Degassing temperature, ºC
1	2.627	2	70
2	0.931	6	70
3	1.722	12	70
4	2.241	48	70
5	1.643	72	70

Table 6 shows weight and degassing time for the parallels of Sample IV.

Table 6: Parallels of Sample IV and their degassing time and temperature

Parallels	Weight, g	Degassing time, h	Degassing temperature, ºC
1	1.589	2	70
2	1.690	6	70
3	1.536	12	70
4	2.516	48	70
5	1.784	72	70

RESULTS

UV spectrometric measurements of the standard chemicals dissolved in methanol for further HPLC analysis have produced the following spectra. Figure 15 shows the UV spectrum for dicumyl peroxide.

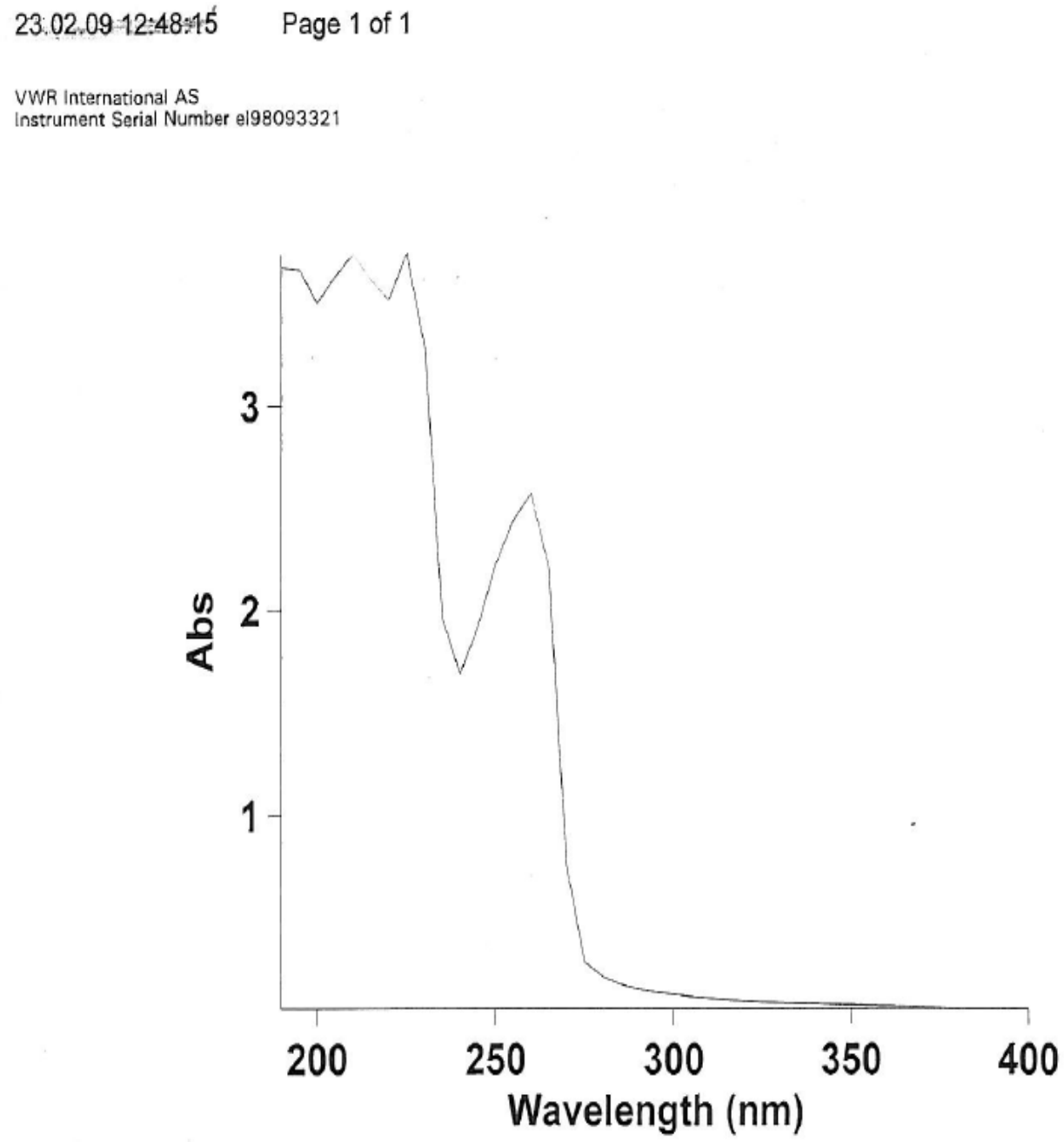

Figure 15: A UV-visible spectrum for dicumyl peroxide

Dicumyl peroxide absorbs at a wavelength of 260 nm.

Figure 16 shows spectrum for acetophenone.

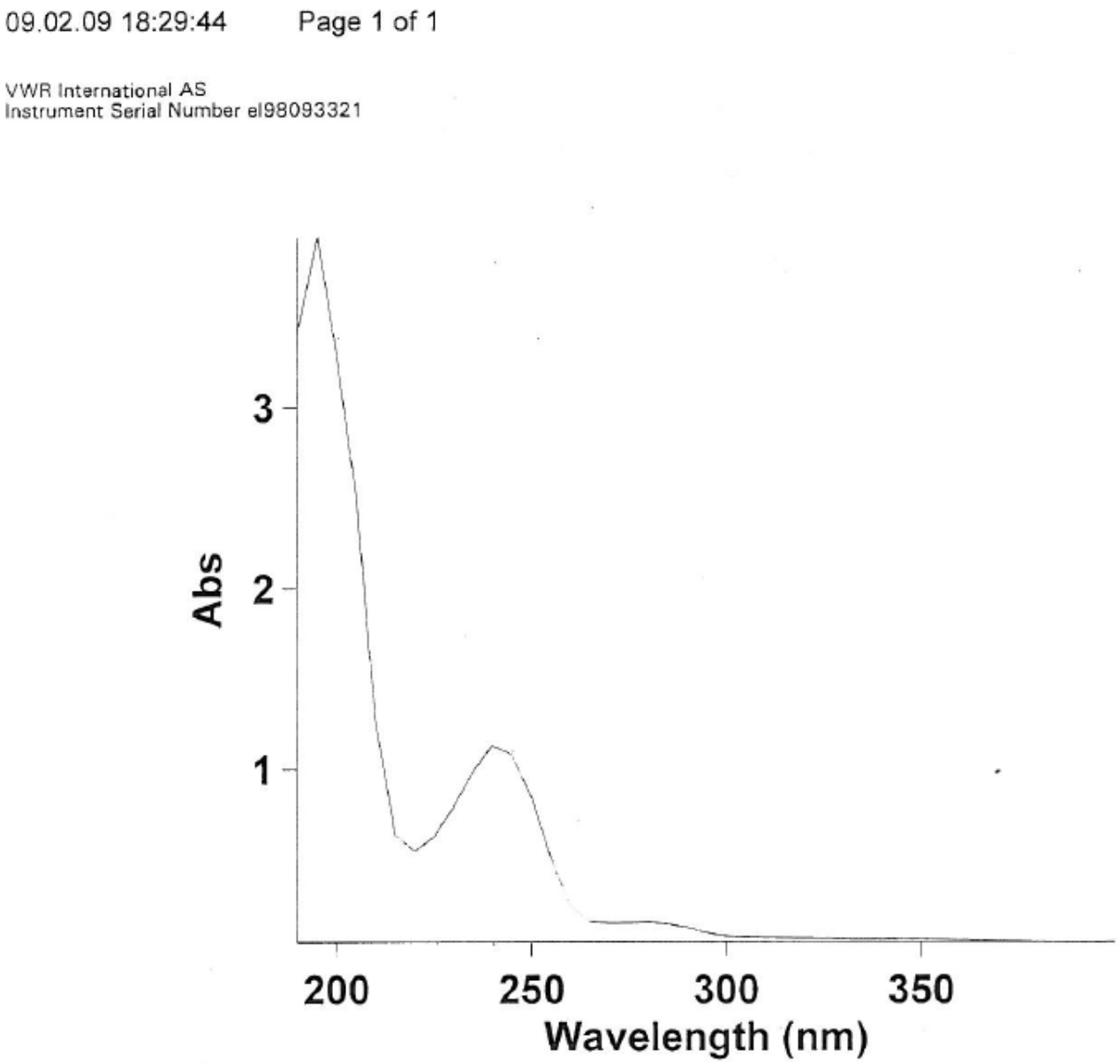

Figure 16: A UV-visible spectrum for acetophenone

Acetophenone absorbs at a wavelength 240 nm.

Figure 17 shows the UV spectrum for alpha methylstyrene.

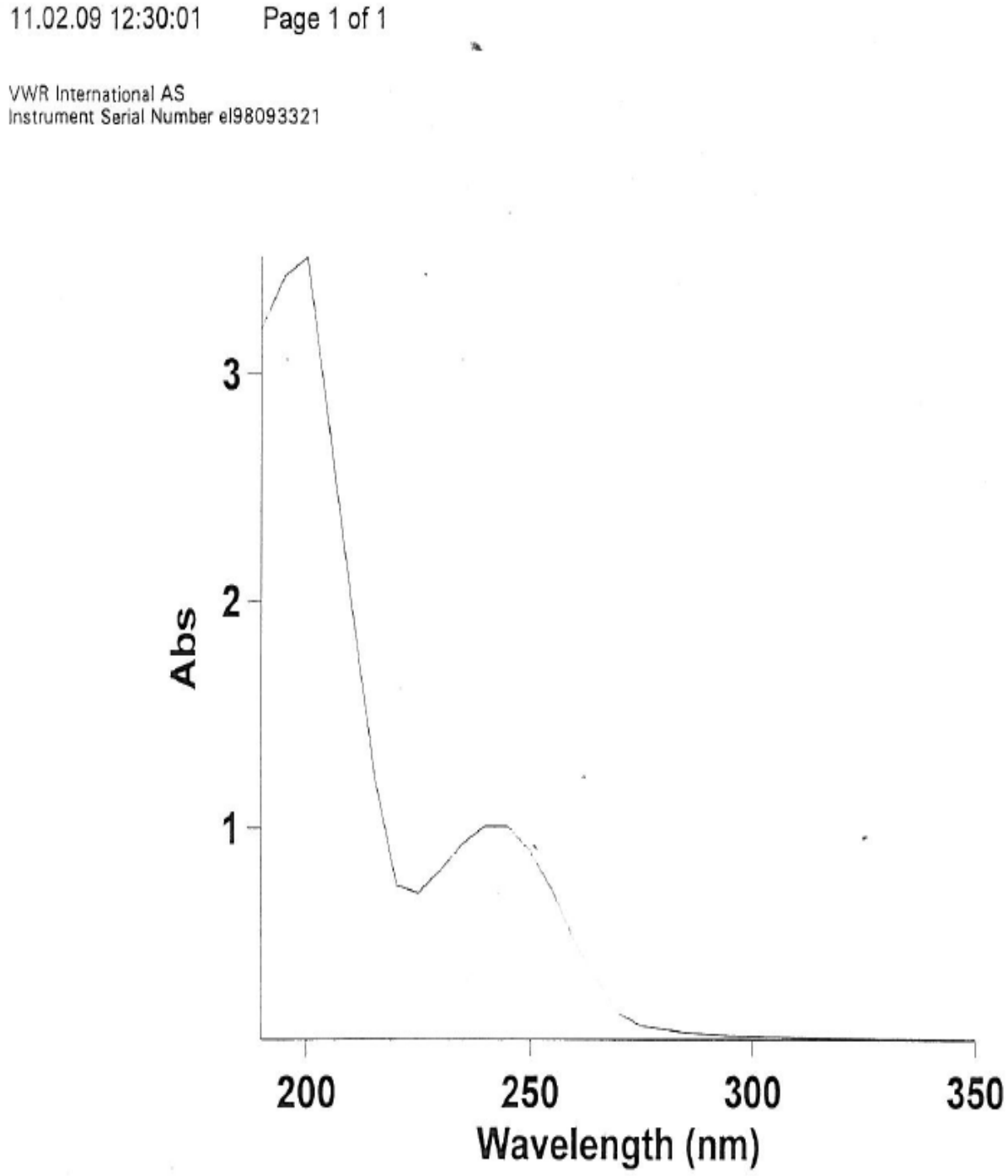

Figure 17: A UV-visible spectrum for alpha methylstyrene

Alpha methylstyrene absorbs at a wavelength 240 nm.

Figure 18 shows the UV spectrum for alpha cumylalcohol.

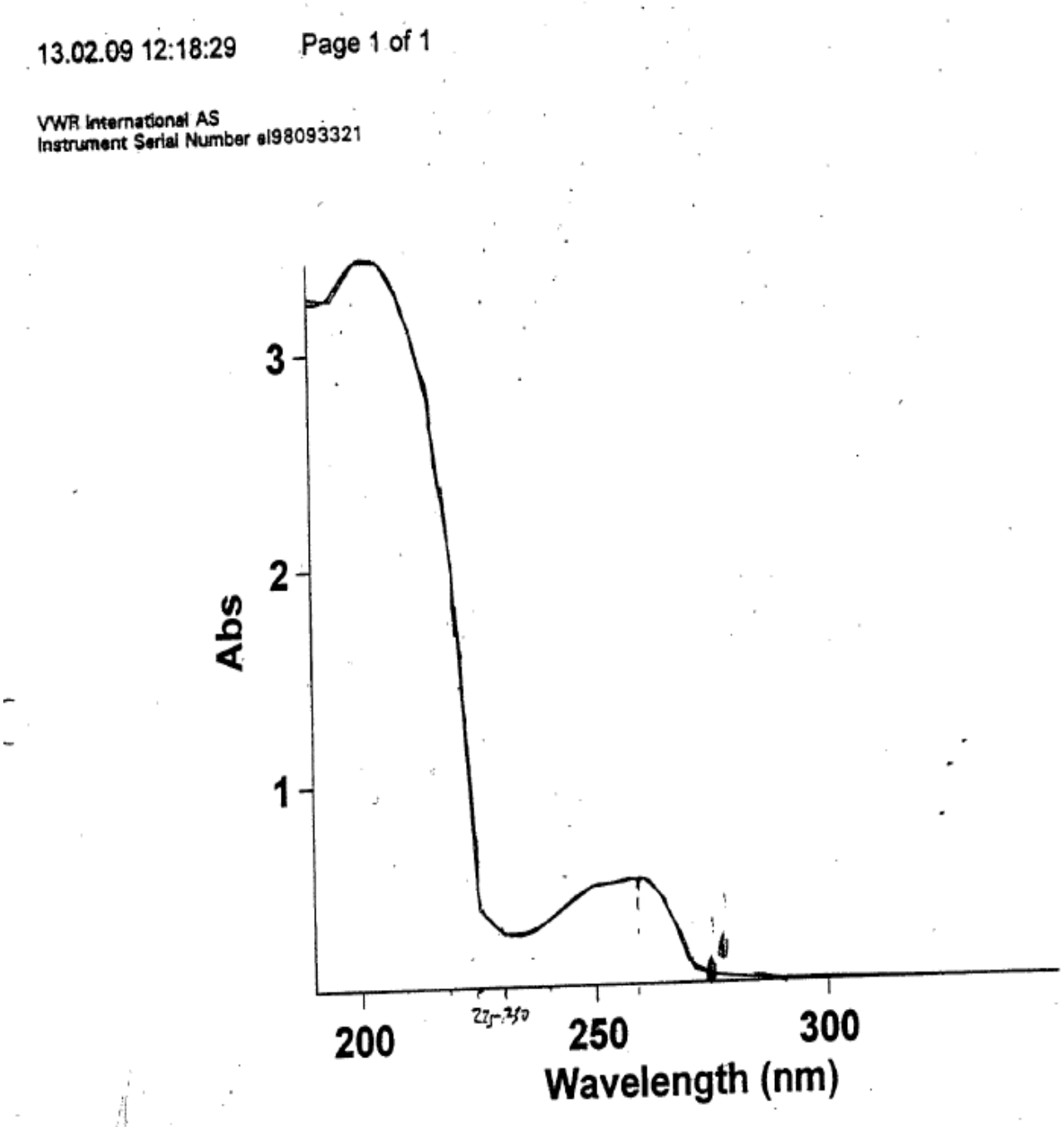

Figure 18: A UV-visible spectrum for alpha cumylalcohol

Alpha cumylalcohol absorbs at a wavelength 260 nm.

RESULTS FOR QUALITATIVE ANALYSIS

The following HPLC chromatograms from figure 19 to 22 were obtained from the qualitative analysis of the standard chemicals.

ACETOPHENONE:

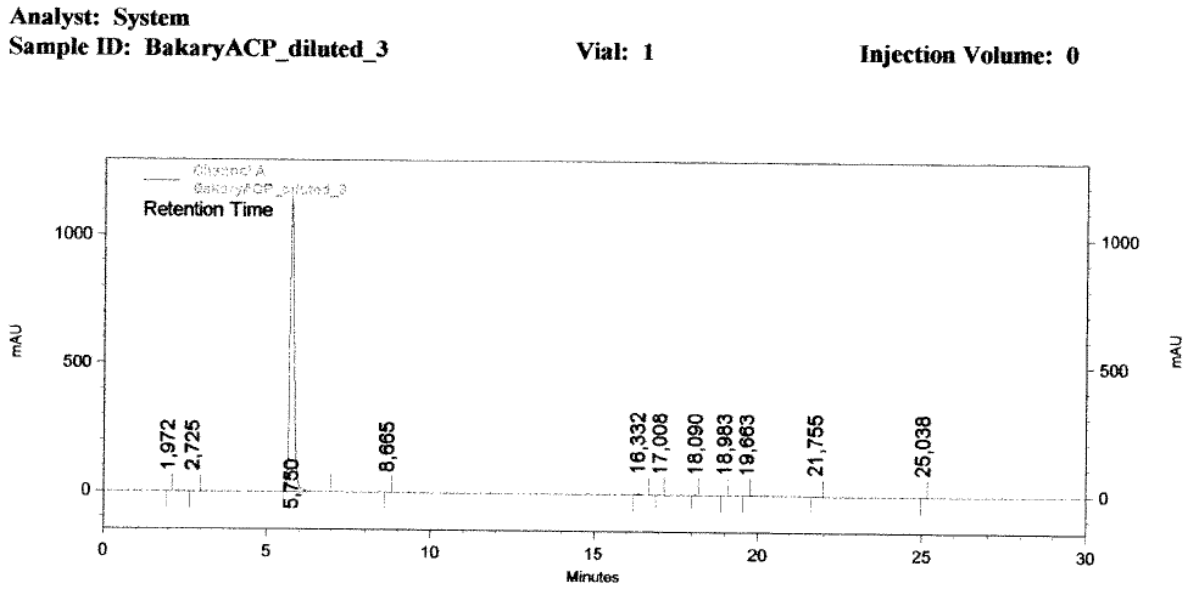

Figure 19: A HPLC-UV chromatogram showing retention time for standard Acetophenone.

ALPHA CUMYLALCOHOL:

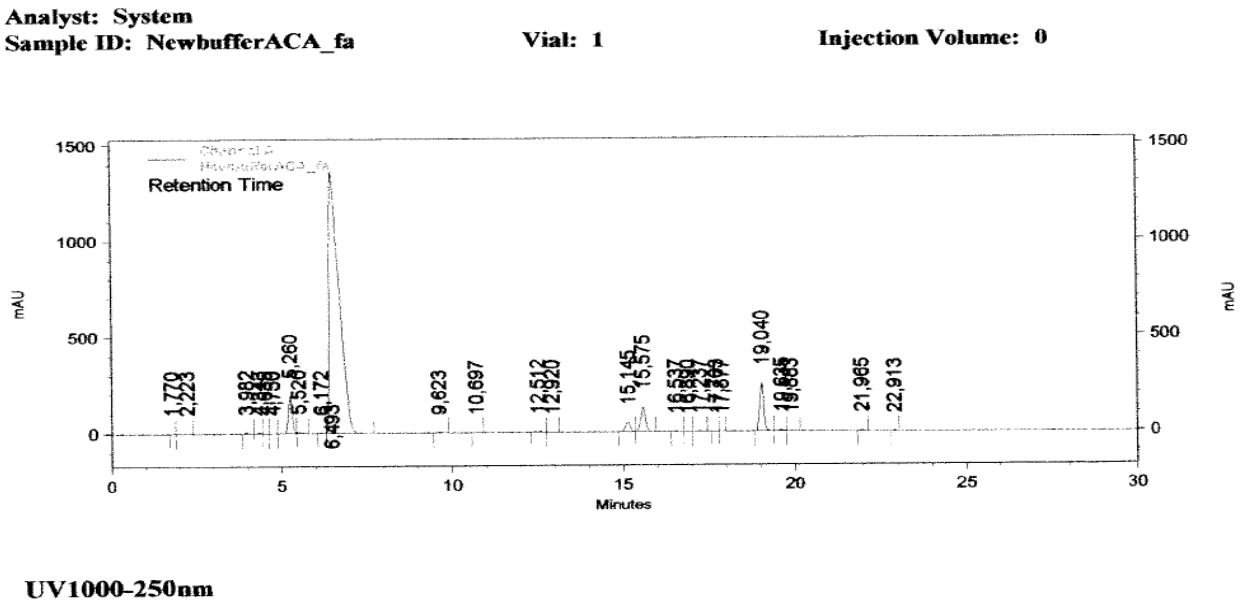

Figure 20: A HPLC-UV chromatogram showing retention time for standard Alpha Cumylalcohol

ALPHA METHYLSTYRENE:

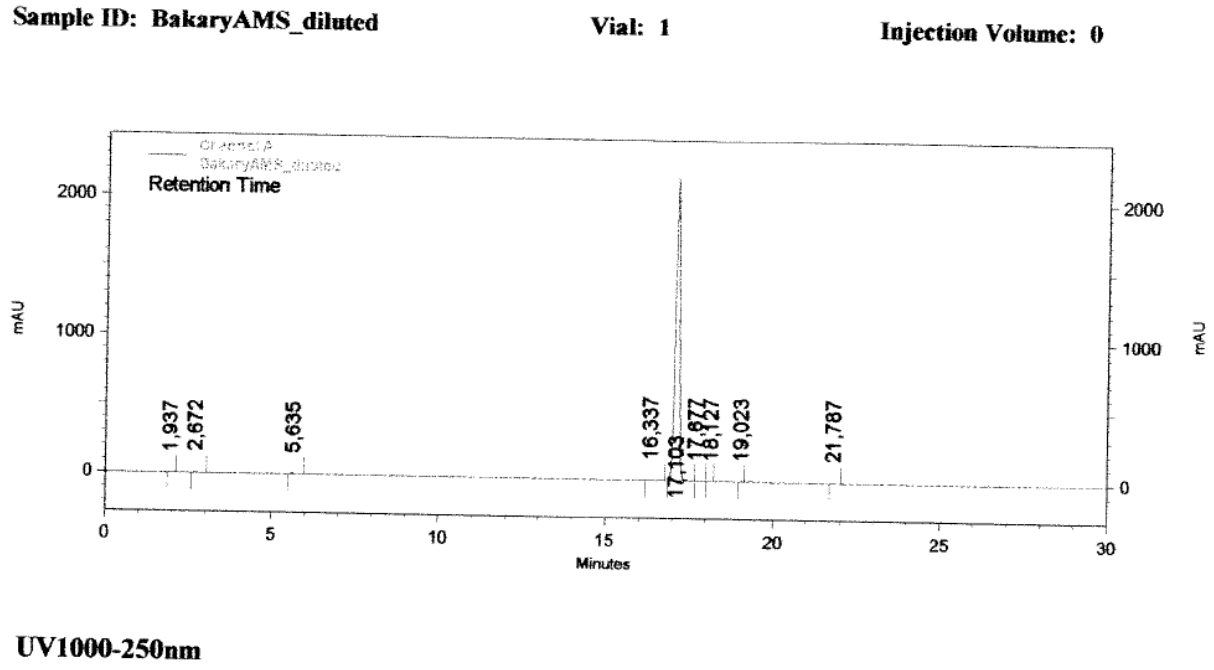

Figure 21: A HPLC-UV chromatogram showing retention time for standard Alpha Methylstyrene

DICUMYL PEROXIDE:

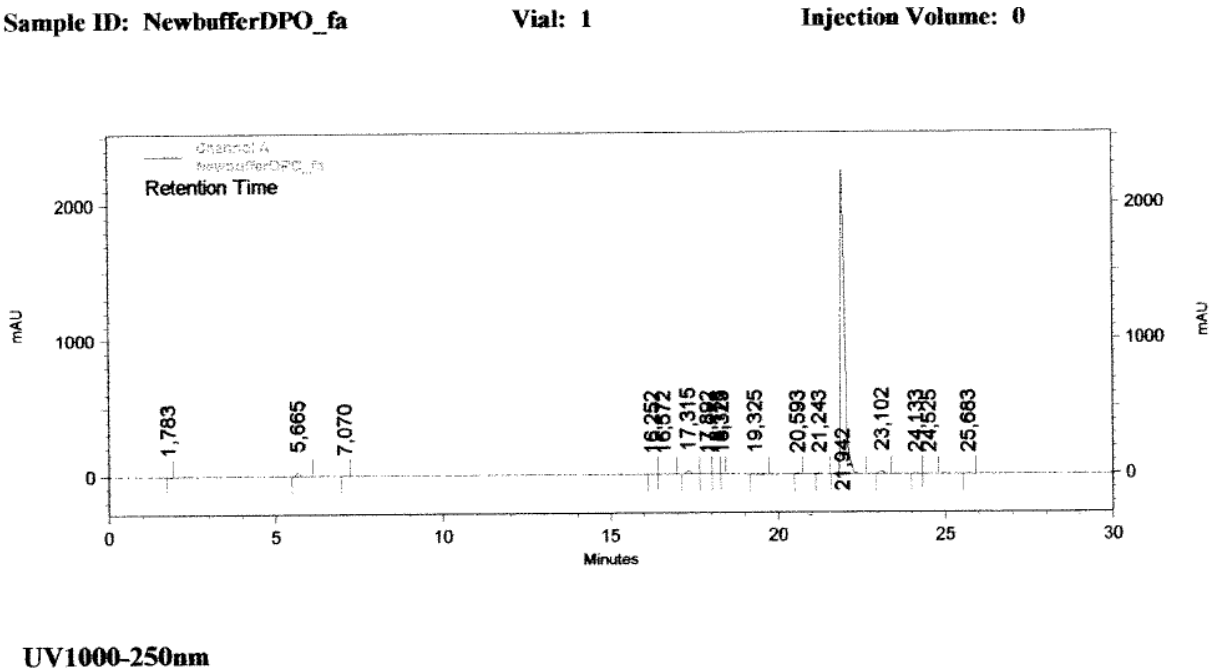

Figure 22: A HPLC-UV chromatogram showing retention time for standard Dicumyl Peroxide

A mix of all the standard chemicals was analysed so as to see if significant changes will be observed in the chromatogram. Such changes were not observed. This mix was called New Buffer Mix-analytes with Formic Acid, abbreviated as NBMFA. Figure 23 shows the chromatogram:

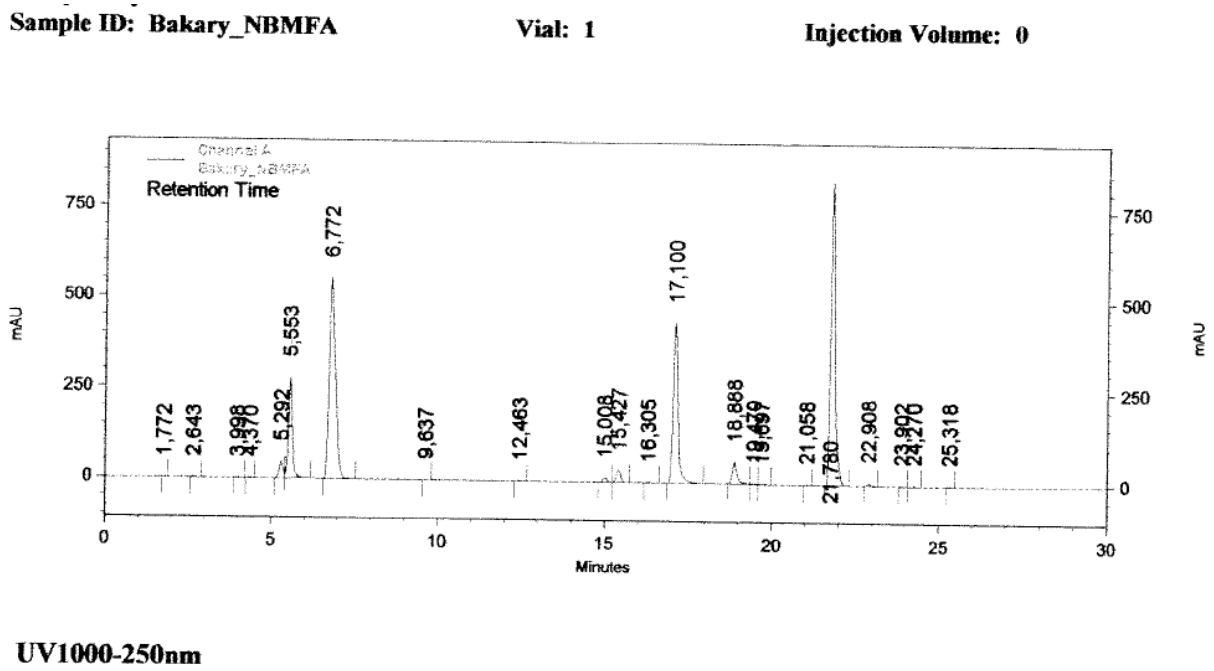

Figure 23: A HPLC-UV chromatogram showing retention times for the four standard Acetophenone, Alpha cumylalcohol, Alpha methylstyrene and Dicumyl peroxide respectively.

Data obtained from HPLC analysis given in figure 23 is included in table 7.

Table 7: Retention times of the standard analytes in the mix solution.

Analyte Mix	Retention time, minutes
Acetophenone	5.5
Alpha cumylalcohol	6.8
Alpha methylstyrene	17.1
Dicumyl peroxide	21.8

RESULTS FOR QUANTITATIVE ANALYSIS

C1, C2, C3, C4 and C5 were analysed with HPLC-UV and changes in the integrated areas of analytes noted with changing concentrations. Figures 24 to 27 show calibration curves are made based on the data obtained in the chromatograms

Table 8: Concentrations and Area of Acetophenone in the five calibration solutions

Solutions	Concentrations (g/mL) ACP	Area
c5	0.000000186	358574
c4	0.000000371	646538
c3	0.000000743	1400527
c2	0.00000149	2331260
c1	0.00000297	4901426

The data in table 8 were plotted in order to derive a formula which can be used in XLPE solutions. Figure 24 shows equation derived for calculation of acetophenone in XLPE.

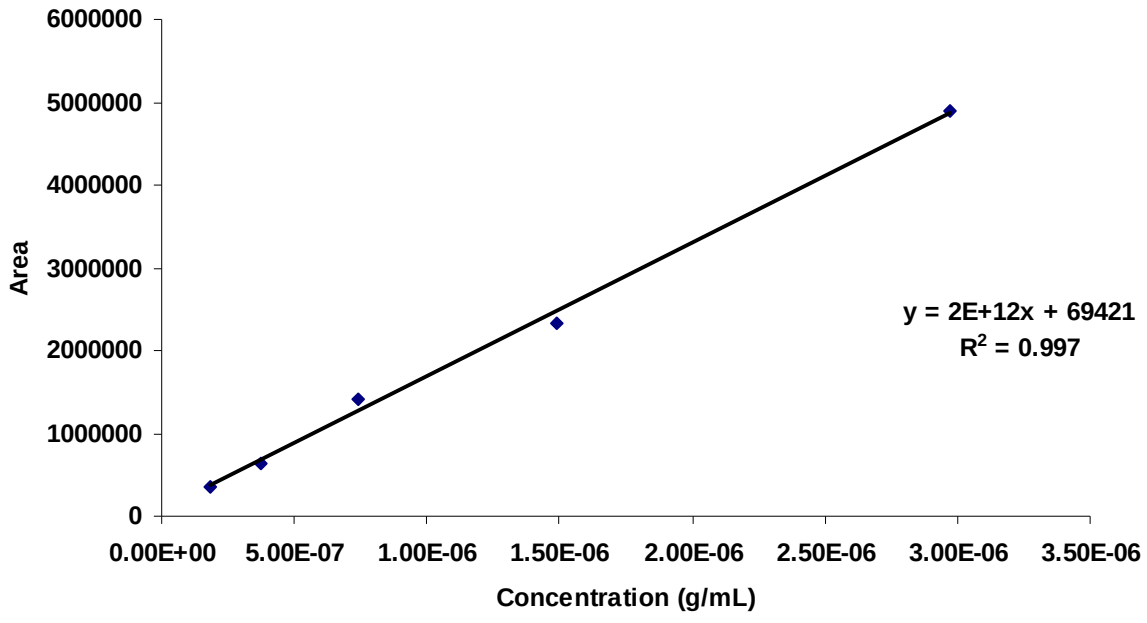

Figure 24: Standard curve for acetophenone based on HPLC analysis.

Table 9 shows the data for Alpha cumylalcohol.

Table 9: Concentration and area of alpha cumylalcohol from the five calibration solutions

Solutions	Concentrations (g/mL) ACA	Area
c5	0.00016	178076
c4	0.00033	306172
c3	0.00065	664915
c2	0.0013	1113359
c1	0.0026	2319759

The data on table 9 were plotted in order to derive a formula which can be used in XLPE solutions. Figure 25 shows the equation derived for calculation of alpha cumylalcohol in XLPE.

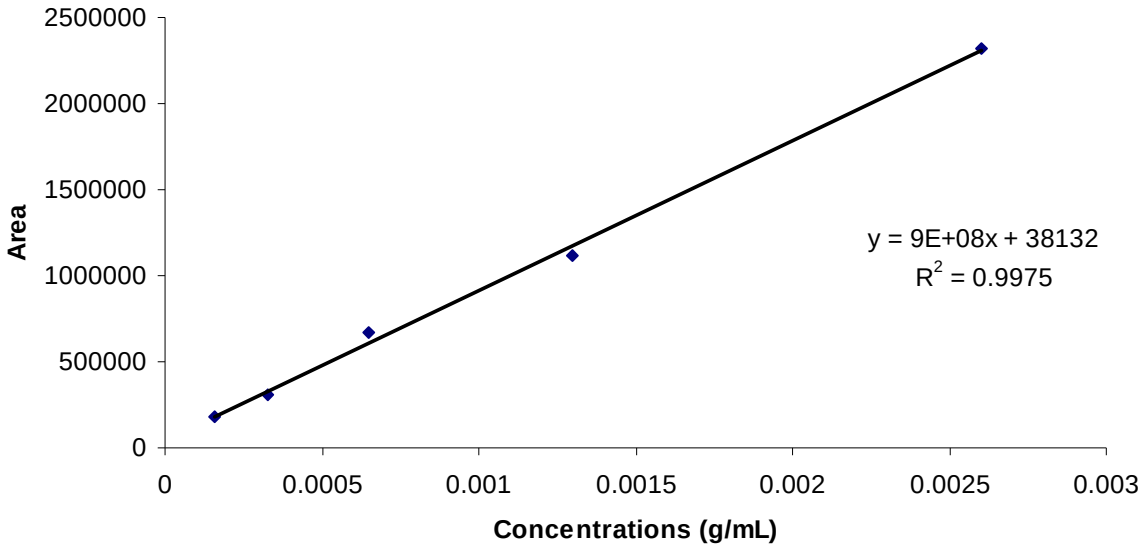

Figure 25: Standard curve for alpha cumylalcohol based on HPLC analysis.

Table 10 shows data for alpha methylstyrene.

Table 10: Concentration and area of alpha methylstyrene from the five calibration solutions

Solution	Concentration (g/mL) AMS	Area AMS
c5	1.82E-07	226741
c4	3.66E-07	386586
c3	7.31E-07	845536
c2	1.46E-06	1407239
c1	2.92E-06	2883923

The data on table 10 were plotted in order to derive a formula which can be used in XLPE solutions. Figure 26 shows the equation for dicumyl peroxide.

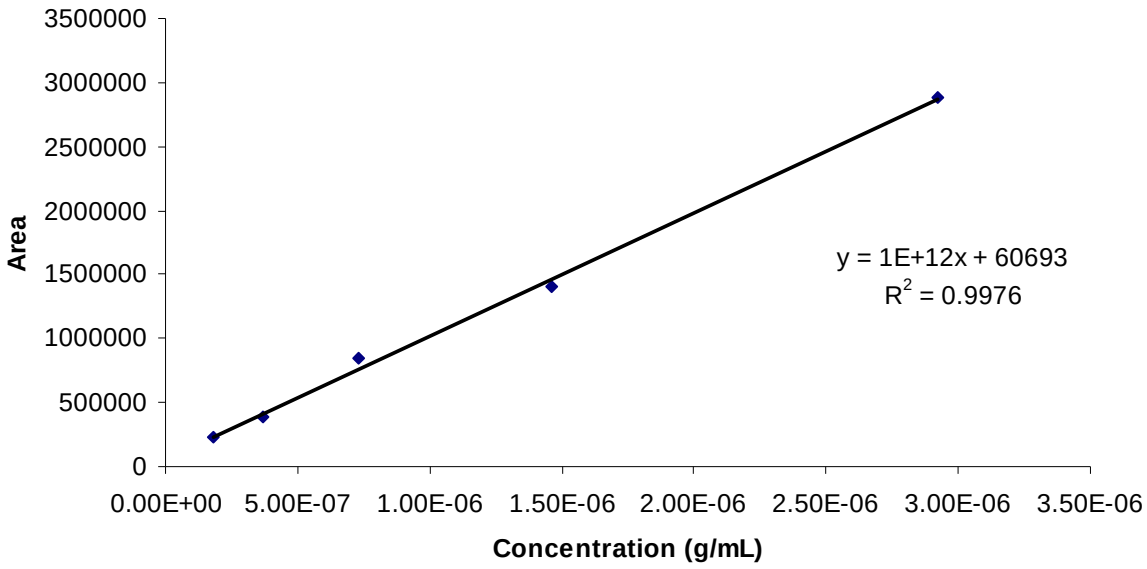

Figure 26: Standard curve for alpha methylstyrene based on HPLC analysis.

Table 11 shows data for dicumyl peroxide.

Table 11: Concentration and area of dicumyl peroxide from the five calibration solutions

Solutions	Concentrations (g/mL) DPO	Area DPO
c5	0.00018	236402
c4	0.00035	395988
c3	0.00071	862393
c2	0.00142	1423524
c1	0.00283	2807177

The data on table 11 were plotted in order to derive a formula which can be used in XLPE solutions. Figure 27 shows the equation for dicumyl peroxide as showed below.

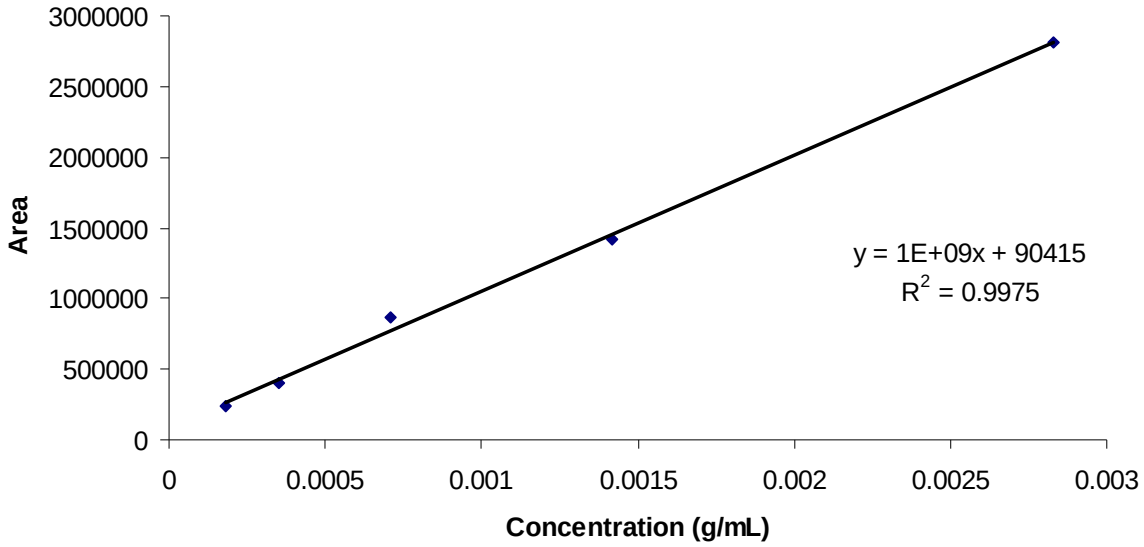

Figure 27: Standard curve for dicumyl peroxide based on HPLC analysis.

Each individual response for each analyte has been looked at separately by using the equations obtained from the calibration curves that were made based on data obtained from the analysis of the standard chemicals. An overview of the equations is presented in table 12.

Table 12: An overview of equations derived after regression analysis of individual standard chemicals.

Standard chemicals	Equation	R^2
Acetophenone	$y = 2 \times 10^{12} X + 69421$	0.997
Alpha cumylalcohol	$y = 9 \times 10^{8} X + 38132$	0.9975
Alpha methylstyrene	$y = 1 \times 10^{12} X + 60693$	0.9976
Dicumyl peroxide	$y = 1 \times 10^{9} X + 90415$	0.9975

In the equations above, y is the area integrated in the chromatogram and X represents concentrations (g/mL) of the analyte corresponding with the area. These equations for acetophenone, alpha cumylalcohol, alpha methylstyrene and dicumyl peroxide will be used to determine their concentrations in cable samples I, II, III, IV, V, VI and VII, by plotting in the integrated areas obtained from their chromatograms. The equation for Dicumyl peroxide was also used to calculate a component assumed to be either a decomposed Dicumyl peroxide or something present in the layer on top of the insulation. This component is, for identification reason, called DPO product.

RESULTS FOR REAL TESTS FOR XLPE CABLES

The following chromatogram (figure 28) shows the Sample I with pure methanol as solvent. Many other components were detected at very low concentrations.

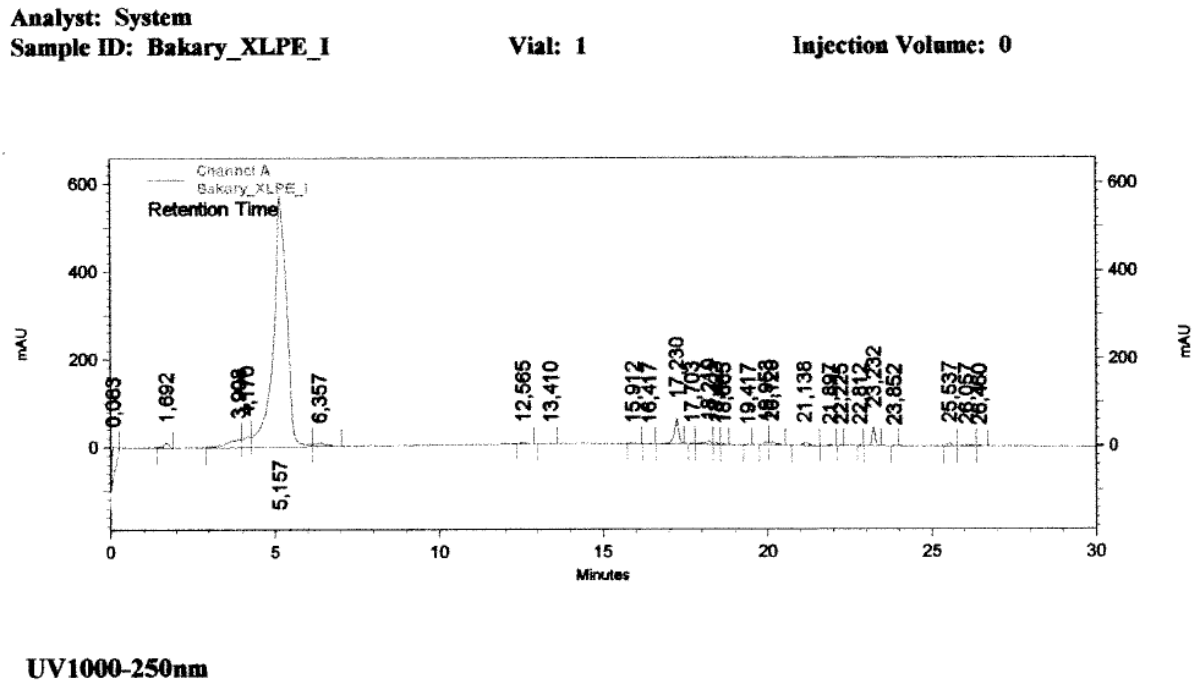

Figure 28: Sample I with methanol as only solvent

The following chromatogram (figure 29) shows sample I with methanol and buffer as solvents. One observes a better symmetry for tops produced in the chromatogram compared to the sample I in with pure methanol as solvent.

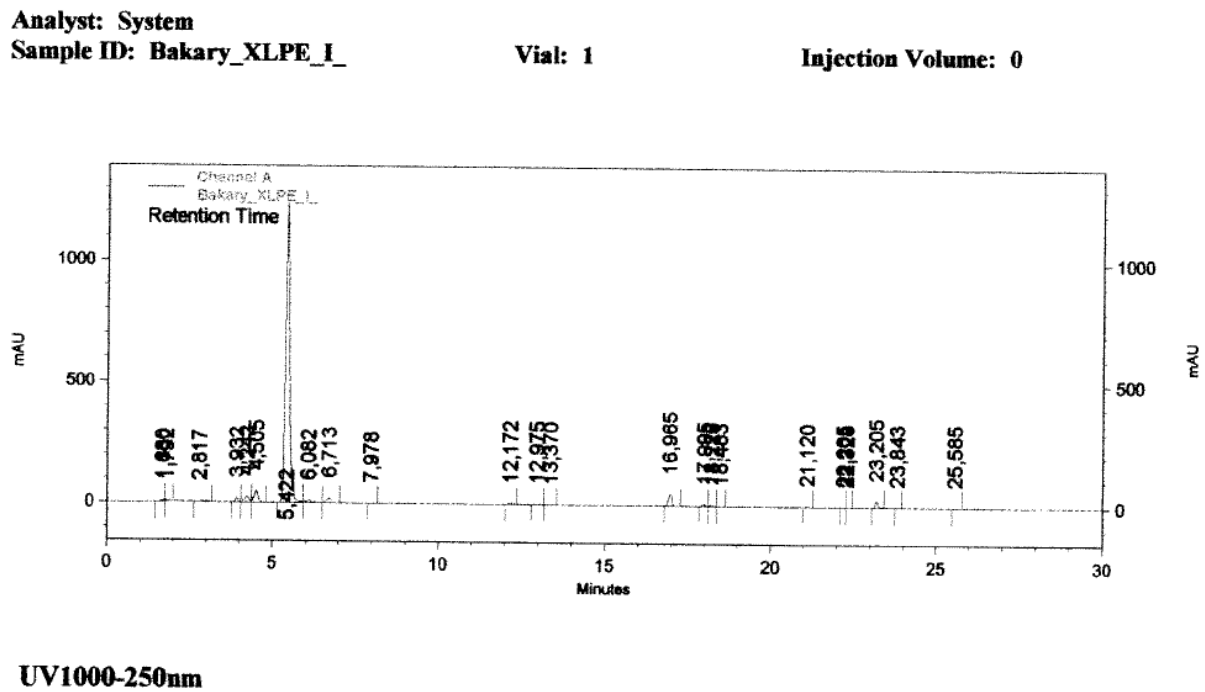

Figure 29: Sample I with methanol and buffer as solvents

The HPLC chromatograms for Samples II, III, IV, V, and VI are presented in appendix 1.

The following chromatogram (figure 30) shows the composition of sample VII with pure methanol as solvent.

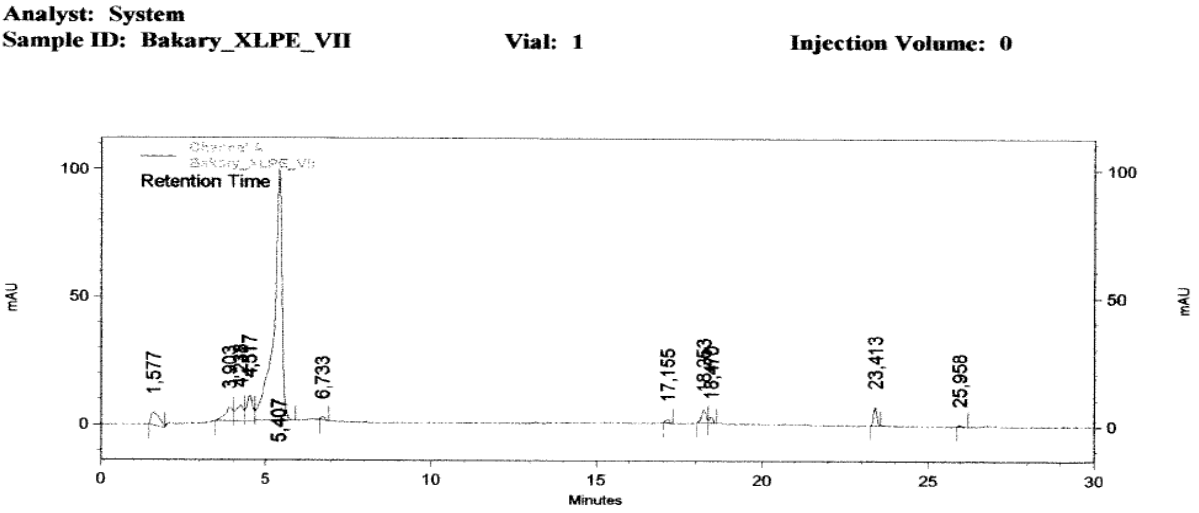

Figure 30: Sample VII with pure methanol as solvent

Figure 31 shows sample VII with methanol and buffer as solvents. Again the symmetry for the tops with both methanol and buffer are far better than samples with only methanol as solvent.

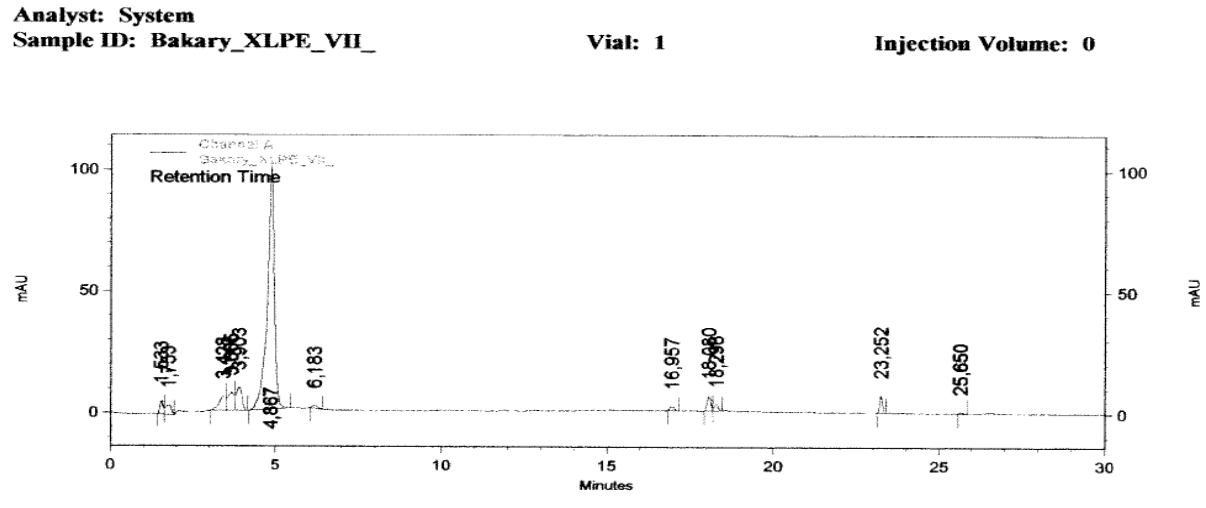

Figure 31: Sample VII with methanol and buffer as solvents

Calculations were done on Microsoft Excel by using data from chromatograms of solutions with pure methanol as the only solvent. The data is presented in table 13.

Table 13: Concentrations of acetophenone, alpha cumylalcohol, alpha methylstyrene and Dicumyl peroxide in cable insulations represented by samples I, II, III, IV, V, VI and VII.

	Acetophenone ***(mg/kg Cable insulation)***	**Alpha cumylalcohol** ***(mg/kg Cable insulation)***	**Alpha methylstyrene** ***(mg/kg Cable insulation)***	**Dicumyl peroxide** ***(mg/kg Cable insulation)***	**DPO Product**
Sample I	42.4	969	2.53	1.05	911
Sample II	46.8	1130	4.03	T	1090
Sample III	65.2	372	19.7	965	518
Sample IV	44.2	858	9.08	T	521
Sample V	28.5	667	1	T	999
Sample VI	42.4	427	43.3	ND	354
Sample VII	17.9	715	1.1	ND	917

T= Traces Detected, ND= No detection

Degassing Results

Polyethylene when cross-linked obtains the capacity to resist pressure inserted by chemicals, temperature, mechanical activities etc. In order to further strengthen its longevity and efficiency, degassing is employed and therefore by-products that were been produced during the crosslinking process are removed significantly. These can be controlled by analysing XLPE cables both before and after degassing in order to determine if the by-products are removed, and if so how much.

A thermal chamber was used to degas the samples two samples. First the chamber was heated to reach 70 ºC before the samples were placed inside. After various time intervals, they were taken out and weighted before been subjected to this research's standard extraction procedure, which is extraction in methanol at 70 ºC for 2 hours, and then analysed.

The following chromatograms (figure 32-35) were obtained after the degassing of samples III and IV and running an HPLC analysis on them. The chromatograms obtained from the HPLC analysis show a decline in components in the XLPE cables. Figure 32 and figure 33 show a decline in areas for the analytes after the cables were thermally treated for two hours.

Degassing two hours: Sample III at 70 ºC

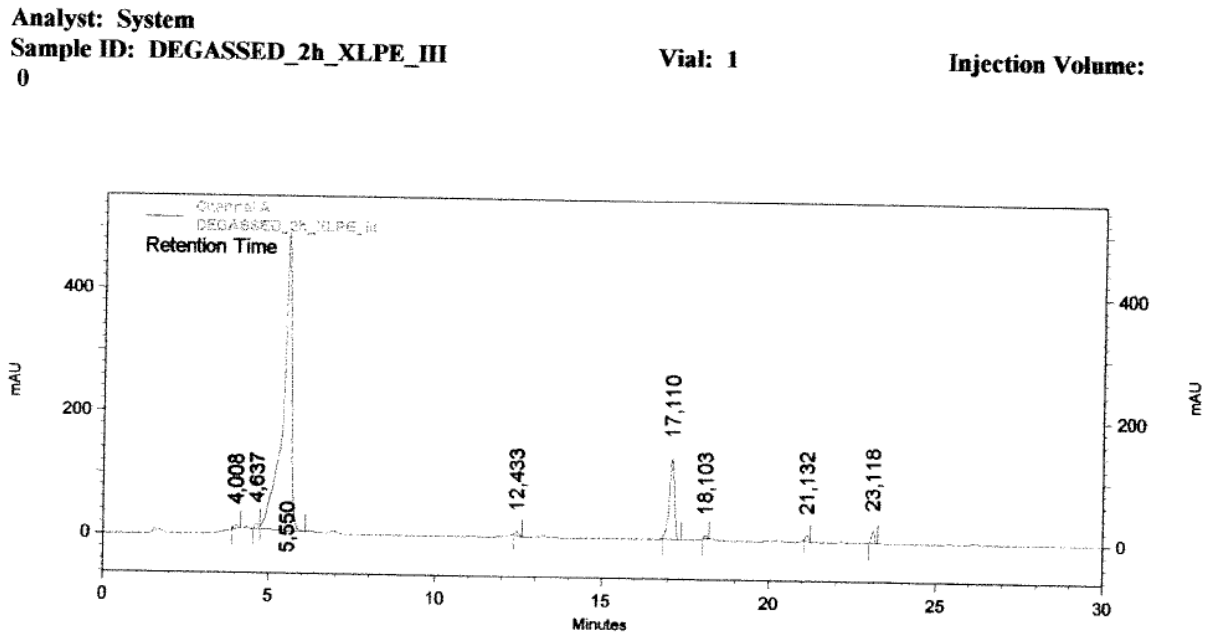

Figure 32: Sample III degassed for 2 hours at 70 ºC

Sample IV degassed for 2 hours at 70 ºC is shown in figure 33.

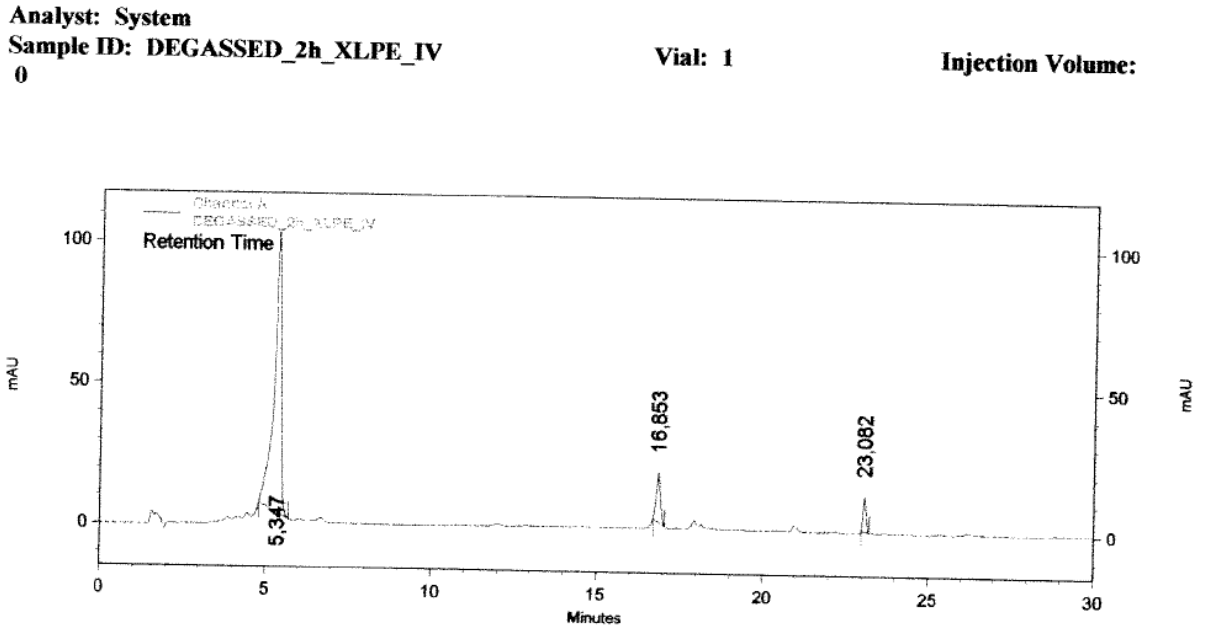

Figure 33: Sample III degassed for 2 hours at 70 ºC

HPLC chromatograms for degassing performed for 6 hours, 12 hours and 48 hours can be found in appendix 2.

Figures 34 and 35 show the removal of almost all components from the cables after the cables were thermally treated for 72 hours.

Degassing for 72 hours: Sample III at 70 ºC

Analyst: System
Sample ID: DEGASSED_72h_XLPE_III **Vial: 1** **Injection Volume: 0**

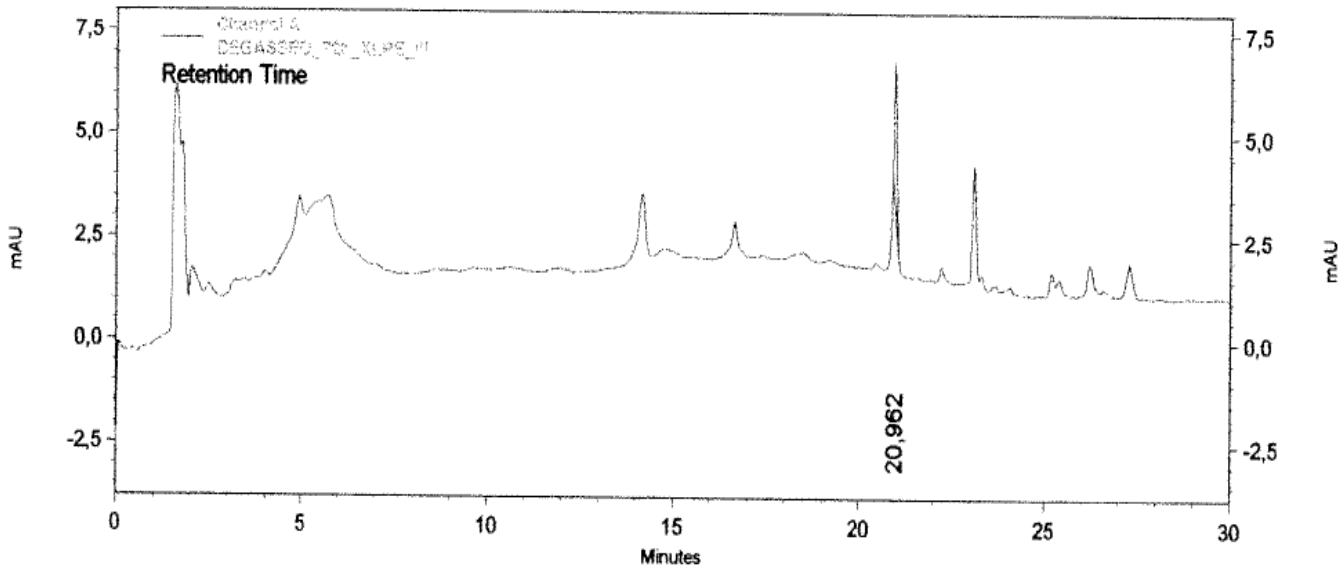

Figure 34: Sample III degassed for 72 hours

Sample IV degassed for 72 hours at 70 ºC is shown in figure 35.

Analyst: System
Sample ID: DEGASSED_72h_XLPE_IV **Vial: 1** **Injection Volume: 0**

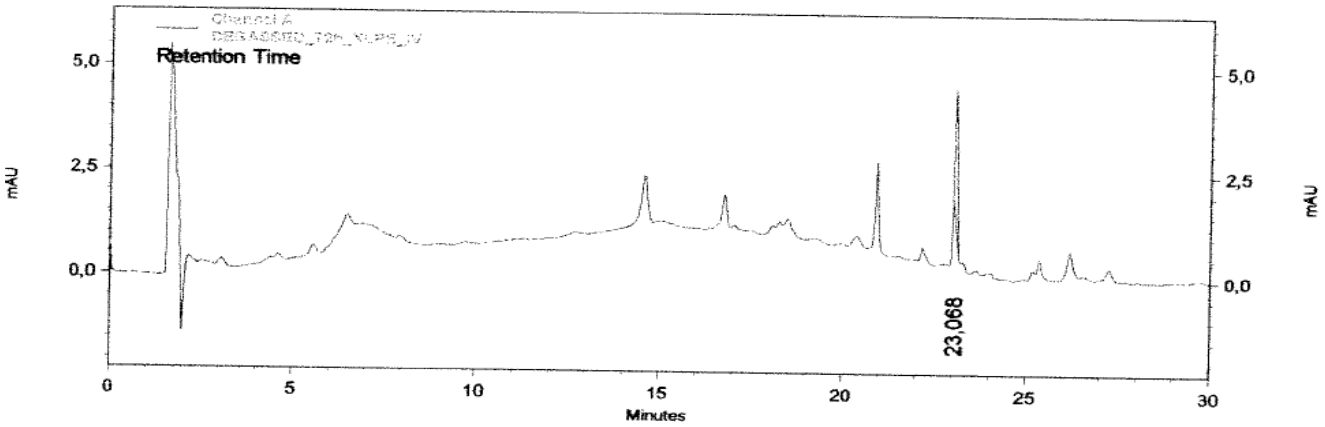

Figure 35: Sample IV degassed for 72 hours.

Table 14 shows the information for sample III after degassing.

Table 14: Parallels for sample III and information after degassing

Parallels	Weight after degassing	% weight loss	Conc. ACP (mg/kg cable)	Conc. ACA (mg/kg cable)	Conc. AMS (mg/kg cable)	Conc. DPO (mg/kg cable)	Conc. DPO product (mg/ kg cable)	Time (hours)
1	2.586	1.6	41.9	*ND*	12.4	*T*	214	2
2	0.908	2.5	1.13	*ND*	*ND*	*ND*	*T*	6
3	1.608	6.6	*ND*	*ND*	*ND*	*T*	*T*	12
4	2.104	6.1	*ND*	*ND*	*ND*	*ND*	*ND*	48
5	1.597	2.8	*ND*	*ND*	*ND*	*T*	*ND*	72

T= Traces detected
ND= No Detection

Table 15 shows the information for sample IV after degassing.

Table 15: Parallels for sample IV and information after degassing

Parallels	Weight after degassing	% weight loss	Conc. ACP (mg/kg cable)	Conc. ACA (mg/kg cable)	Conc. AMS (mg/kg cable)	Conc. DPO (mg/kg cable)	Conc. DPO product (mg/kg cable)	Time (hours)
1	1.578	0.7	11.1	*ND*	*1.44*	*ND*	*T*	2
2	1.680	0.6	4.31	*ND*	*T*	*ND*	*T*	6
3	1.526	0.7	0.18	*ND*	*ND*	*ND*	*T*	12
4	2.443	2.9	*ND*	*ND*	*ND*	*ND*	*T*	48
5	1.776	0.4	*ND*	*ND*	*ND*	*ND*	*T*	72

T= Traces detected
ND= No Detection

DISCUSSION

Cross-linked Polyethylene will always contain constituents like acetophenone, alpha cumylalcohol, alpha methylstyrene, and methane as long as the cross-linking process is chemically induced using dicumyl peroxide. With the help of degassing, these by-products can be removed to just insignificant traces thus ensuring, among other things, the cable's sufficient dielectric properties.

The degassing that has taken place in the research lab at the University of Stavanger has been carried out in a thermal chamber which by dimension is much smaller than those being used in the industry, in this case at Nexans' production unit. Again, in the lab, only small cross sections of the cables were degassed and not the whole cable. Ideally, it would be accurate to have all the parallel pieces of the cable weigh the same. That would give a more accurate picture about the deviations in cable characteristics both before and after degassing. However, the difference in sample weights also gives another picture one could not have gotten by preparing samples uniform in weights. Another way to do things is to conduct analysis based on a uniform weight but varying temperature and degassing time parameters. This is time consuming but it can lead to interesting conclusions. Degassing rate is high in the beginning of the process and becomes low with time. Most by-products are removed in the beginning of the process.

It would have been interesting to also degas and analyse the different parts of the cables like their inner, outer and the middle parts. This was not the case because of time constraints. Representative samples which are a mix of the inner, mid and outer parts were been made in all the extraction, degassing and chromatographic processes. Therefore results obtained from these experiments are broad and gives a picture of average distributions of the analytes in the cable insulation.

The results obtained from the experiments might have been impacted by experimental error including human error, instrumental error and limited sampling. For example in making samples from the cable insulations, one subjects the matrix to mechanical and temperature treatment. These can cause loss of analytes before quantitative analysis.

Extractions time was set to be two hours. However, longer extraction periods will lead to even more accurate answers regarding the true concentration of components in cable insulations. The buffer composition in the HPLC analysis is very important because HPLC is such a sensitive instrument that reacts to slightest of changes. An effect on the chromatogram that could be caused by changes in buffer composition is, typically, retention time. In this case, because one can manually identify the tops for our analytes in the chromatogram, only the areas and the concentrations in it are of ultimate interest. One should prepare enough buffer volume to last the whole HPLC analysis in order to evade effects of changes in buffer composition. One observes, from the chromatograms produced during cable sample analysis that the retention time changes with a change in the sample composition. Samples with only pure methanol as solvents and those with both methanol and buffers as solvents give slightly different retention times.

Peroxide initiated crosslinking of polyethylene means some peroxide rests will remain in the insulation. These are detected in the XLPE cables analyzed in the lab. One particular component detected by the analytical instrument has a retention time of about 23.1 minutes

and has been observed in all the XLPE samples, degassed and not degassed. If not a decomposed dicumyl peroxide, it might be something present in the membrane on the top layer of the insulation. It has been given the name DPO product, for identification. Its concentrations were determined and presented together with other by-products. Other components similar to dicumyl peroxide were detected in and around the same retention time. Some chromatograms indicate some impurities which may be due to the samples' exposure and contact with other materials in the lab and in the material technology workshop where cables were cut into pieces. These impurities may range from oil applied on the jigsaw blade, scissors that were in contact with other stuff, or even traces of antioxidants that enhanced the cross linking reactions.

The method developed and highlighted in this project is not a suitable method for analysing methane because methane is gaseous, thus suitable for instruments like gas chromatography. It is of interest for this author to look into another method suitable for methane detection and quantification. Time has, however, been an important factor for the failure to thoroughly look into the practical aspects of methane analysis. Nevertheless, two possible methods have been considered for development for the analysis of methane by using Gas Chromatography and Mass Spectrometry (GC-MS).

- Headspace is one of the methods which will allow for the application of temperature on the polymer so that volatile components like methane can be separated and analyzed in the GC column and detected by the use of MS.
- Liquid extraction and injection in GC has also been considered. Mixing of polymers in diethyl ether by ultra sound bath could be an interesting way of extracting XLPE components. By using very volatile solvent, it can be assumed that volatile components could be detected in gas chromatographic analysis.

The results obtained from this research are not been compared with the results Nexans have calculated. One thing is however certain: different analytical methods can be made and developed for quantification of by-products in cables. Decreasing by-products that were observed in the cables after degassing were expected.

CONCLUSIONS

High Performance Liquid Chromatography with a UV detector was found to be convenient for analysis of by-products from XLPE production for cable insulation. A method that included an ideal wavelength of 250 nm, a buffer solution of pH 2.3 made of nano pure water and pure formic acid, a C-18 column and a linear gradient mobile phase, was developed for analysis on HPLC. Extracting analytes from the samples was a critical and important step to do before the HPLC analysis. On the HPLC, only non-gaseous substances could be analyzed which in this case were acetophenone, alpha cumylalcohol, alpha methylstyrene and rests of chemically inductive crosslinking agent dicumyl peroxide. Reproducibility was observed in the results obtained in chromatograms of the standard chemicals which were used as reference chromatograms when XLPE was analyzed. In the XLPE analysis, by-products were found to be present in all the cable samples though at small and often insignificant concentrations. Concentration values show that samples that were not degassed have a greater concentration of by-products than those been degassed. This reconfirms the need for degassing.

The degassing process that was conducted was found to have been working well at degassing temperature of 70 ºC. By-products were observed to be reducing with increasing degassing time. The two samples that were degassed have shown that thermal treatment is an efficient way of getting rid of by-products. However one has to be mindful of not over heating cables as that might damage cable.

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APPENDIX

APPENDIX 1

Figure 36 shows sample II with methanol as only solvent.

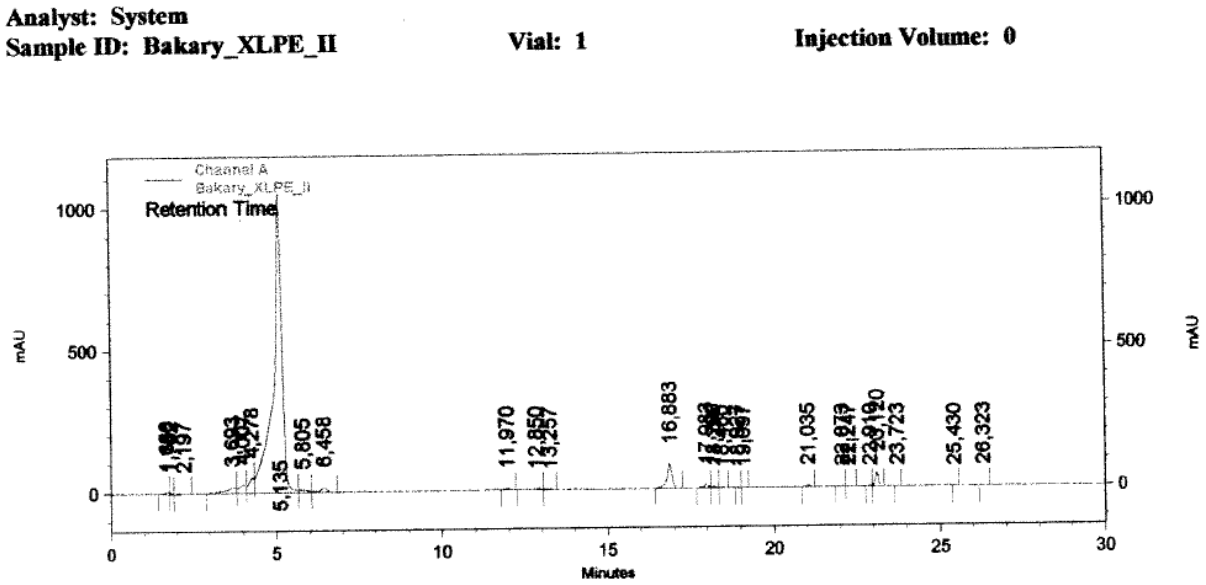


Figure 36: Sample II with pure methanol as solvent

The following chromatogram (figure 37) shows sample II with methanol and buffer as solvents.

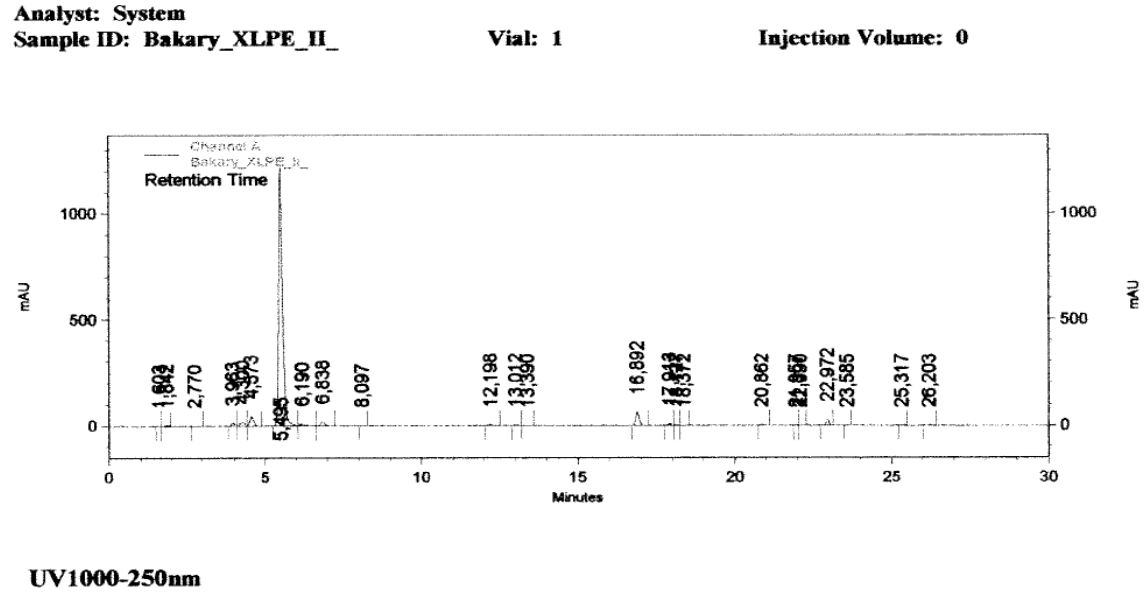


Figure 37: Sample III with methanol and buffer as solvents

Figure 38 shows composition of sample III with pure methanol as solvent.

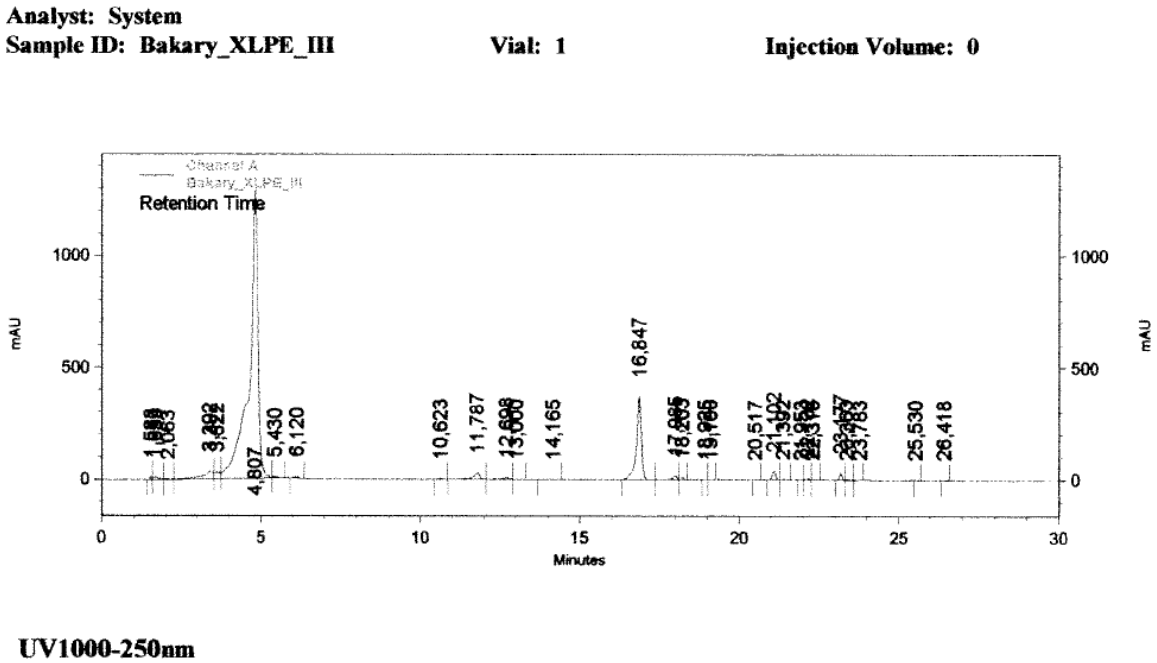


Figure 38: Sample III with methanol as solvent

Figure 39 shows sample III with methanol and buffer as solvents.

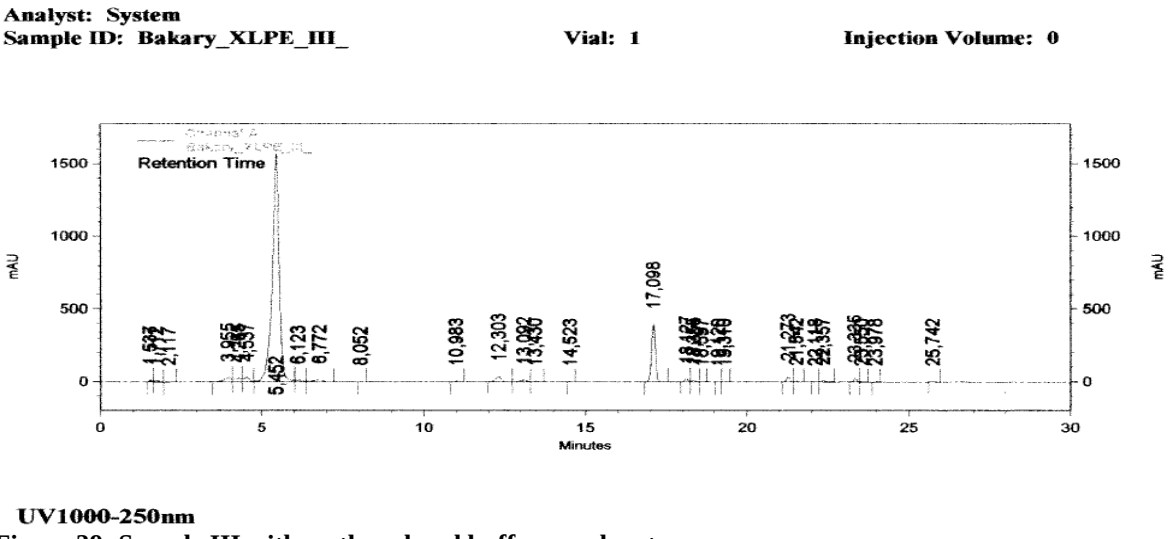


Figure 39: Sample III with methanol and buffer as solvent

The following chromatogram (figure 40) shows the composition of sample IV with pure methanol as solvent.

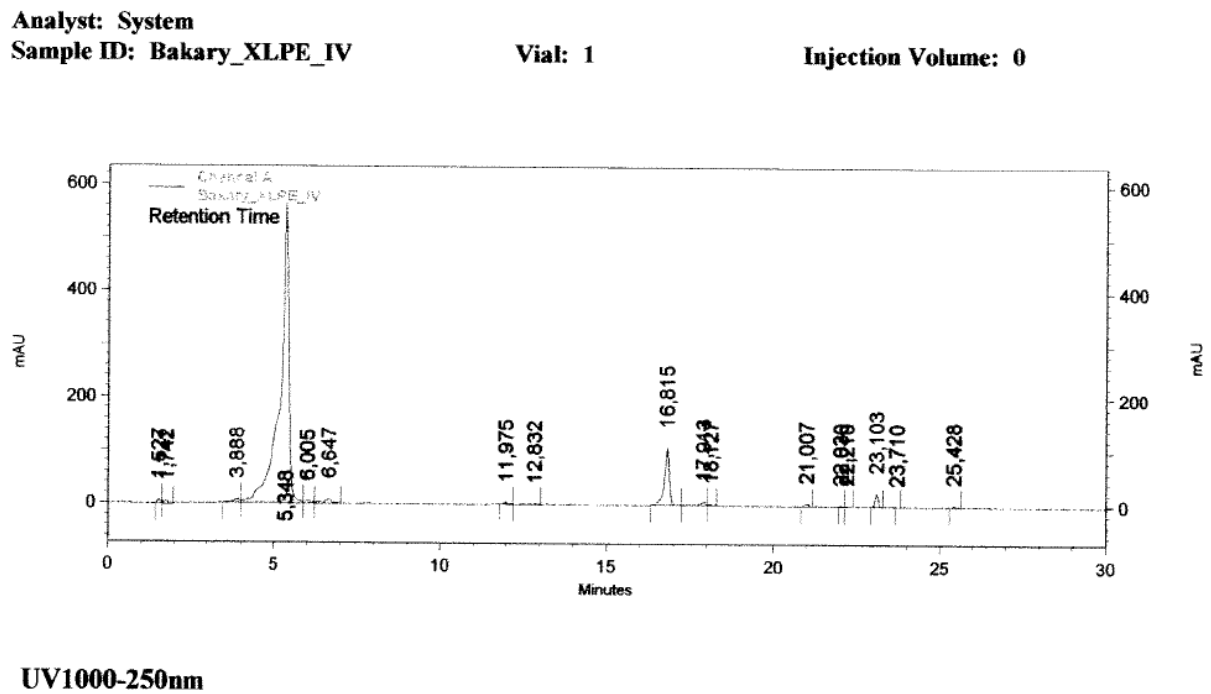


Figure 40: Sample IV with pure methanol as solvent

Figure 41 shows sample IV with methanol and buffer as solvents.

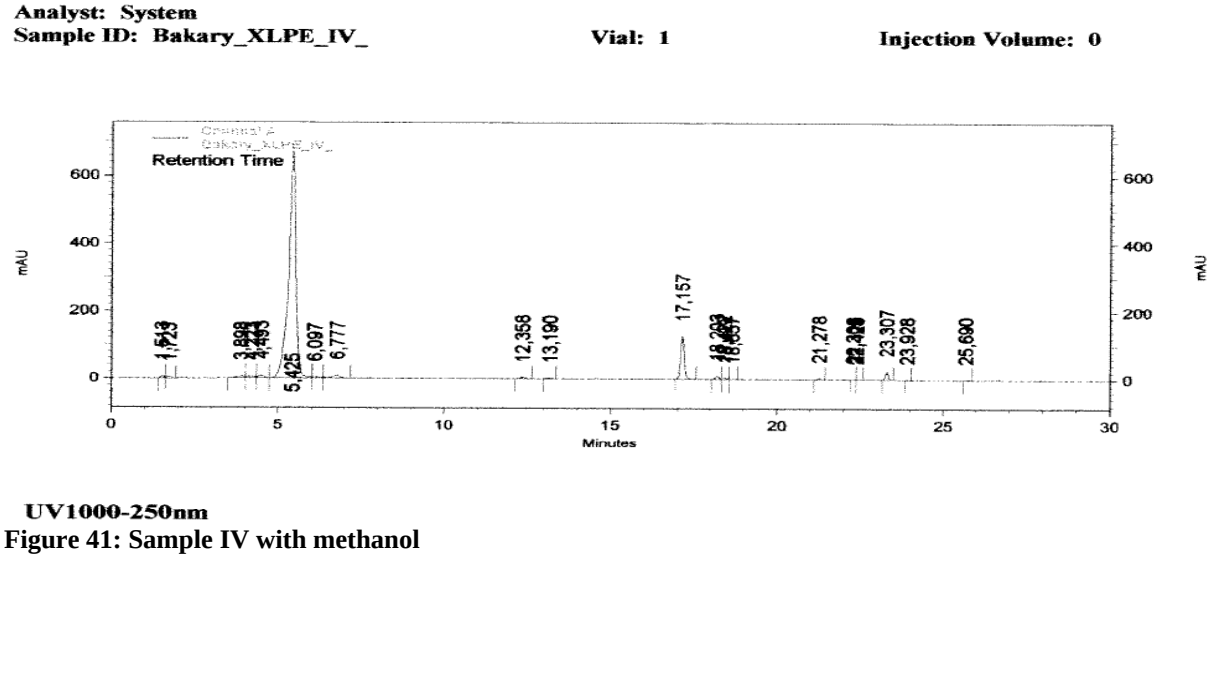


Figure 41: Sample IV with methanol

Figure 42 shows the composition of sample V with pure methanol as solvent.

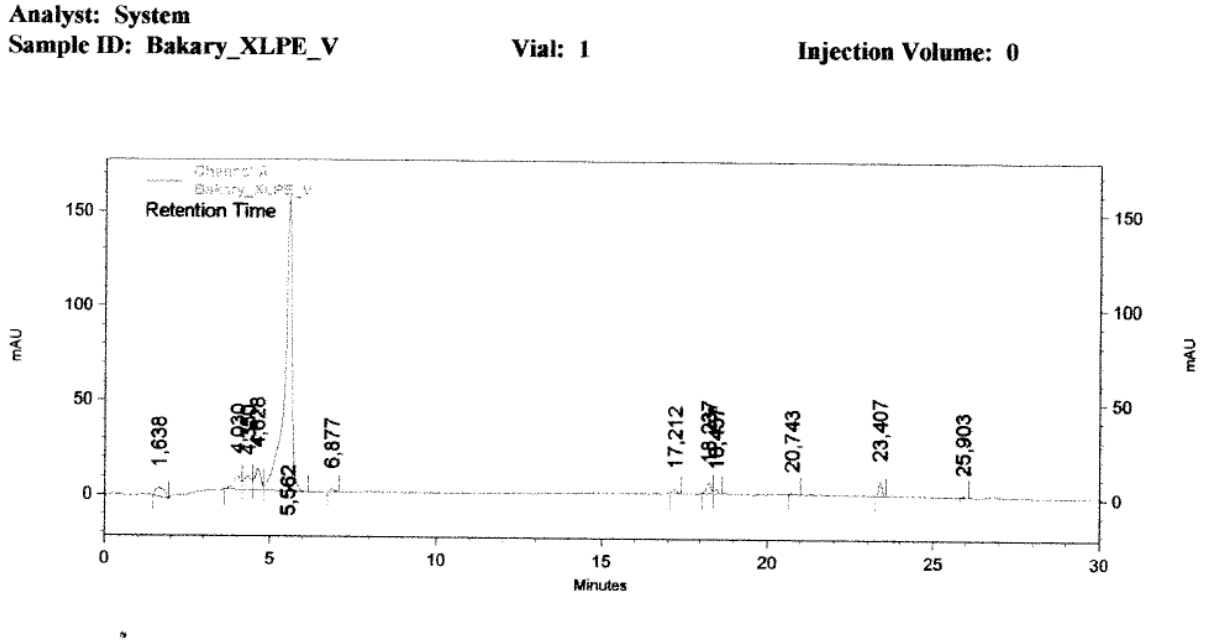


Figure 42: Sample V with pure methanol as solvent

Figure 43 shows sample V with methanol and buffer as solvents.

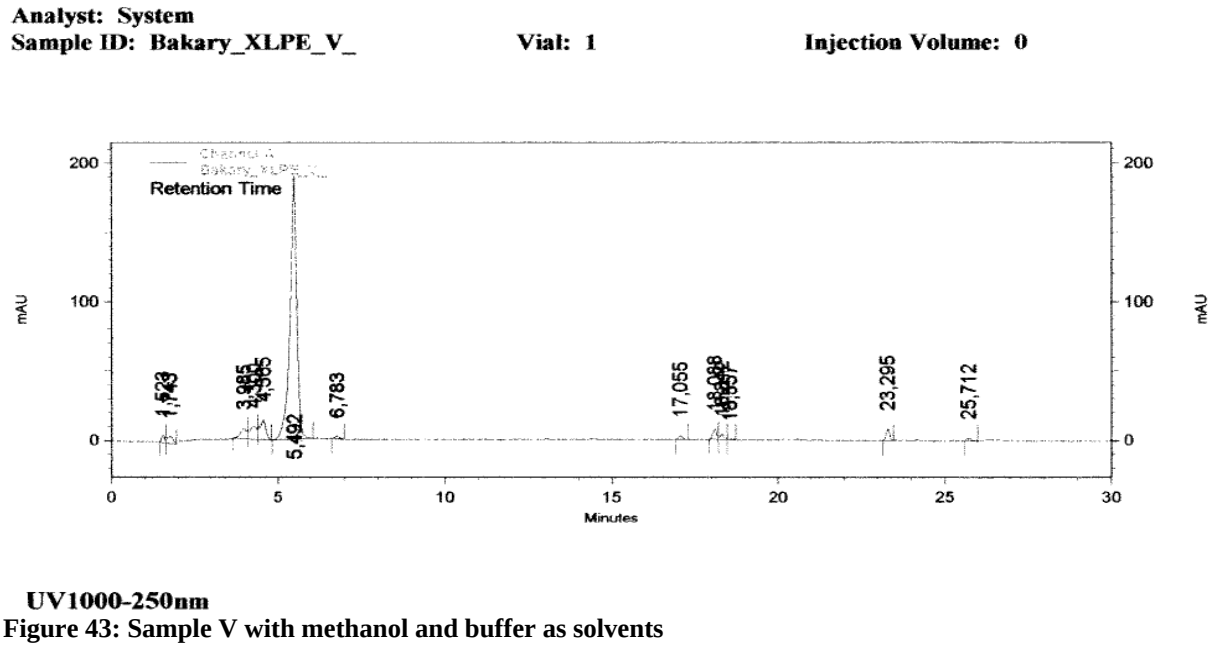


Figure 43: Sample V with methanol and buffer as solvents

Figure 44 shows the composition of sample VI with pure methanol as solvent.

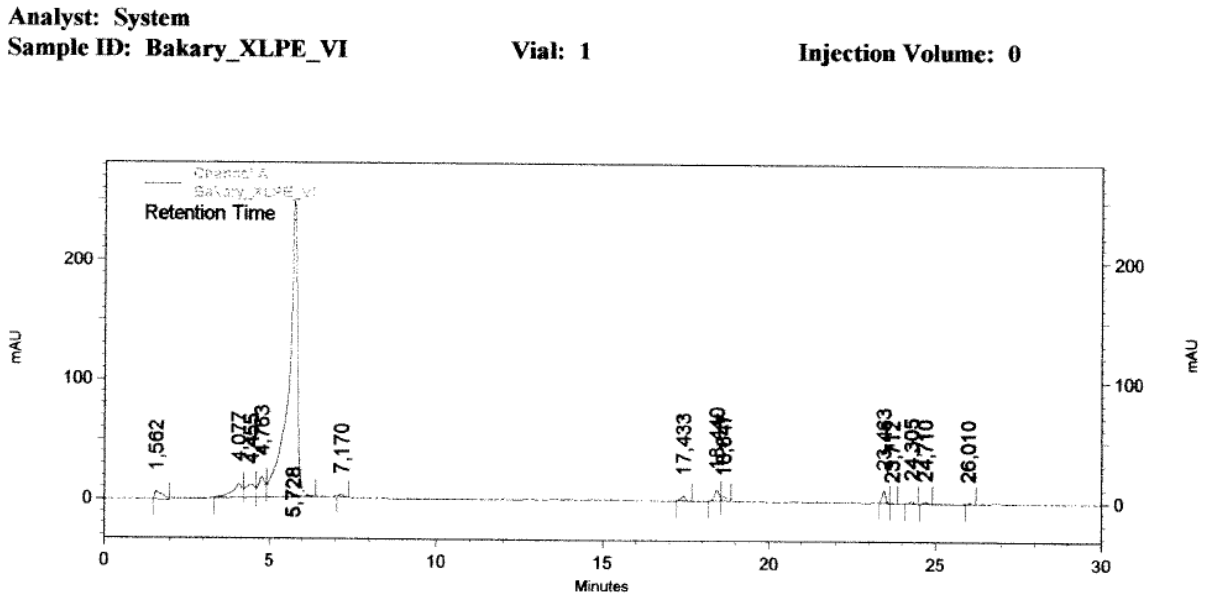


Figure 44: Sample VI with pure methanol as solvent

The following figure (figure 45) shows sample VI with methanol and buffer as solvents.

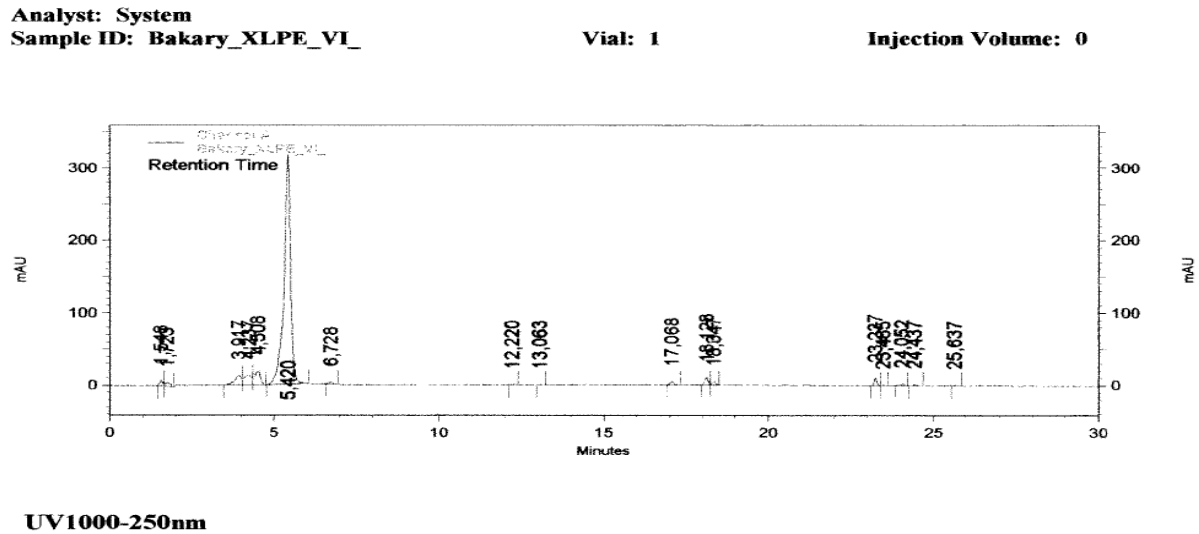


Figure 45: Sample VI with methanol and buffer as solvents

APPENDIX 2

Degassing for 6 hours:

Analyst: System
Sample ID: DEGASSED_6h_XLPE_III **Vial: 1** **Injection Volume: 0**

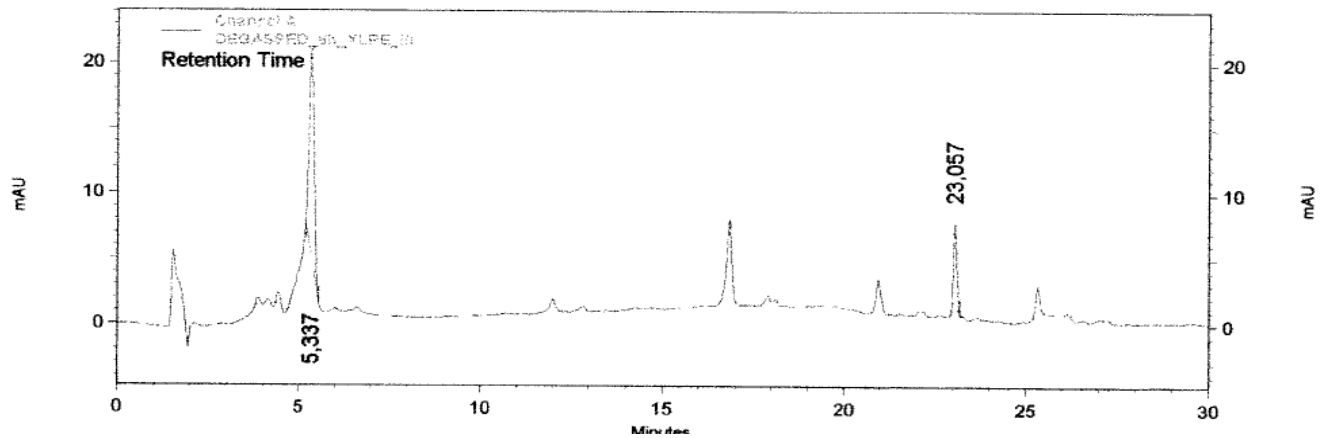


Figure 46: Sample III degassed for 6 hours at 70 °C

Analyst: System
Sample ID: DEGASSED_6h_XLPE_IV **Vial: 1** **Injection Volume: 0**

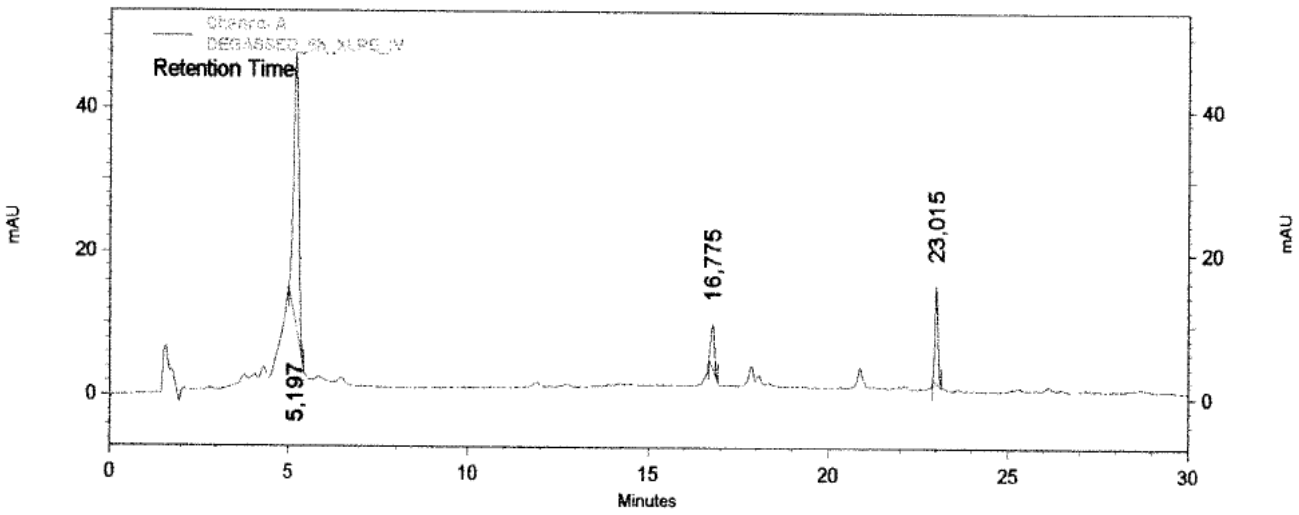


Figure 47: Sample IV degassed for 6 hours at 70 °C

Degassing for 12 hours

Analyst: System
Sample ID: DEGASSED_12h_XLPE_III **Vial: 1** **Injection Volume:**
0

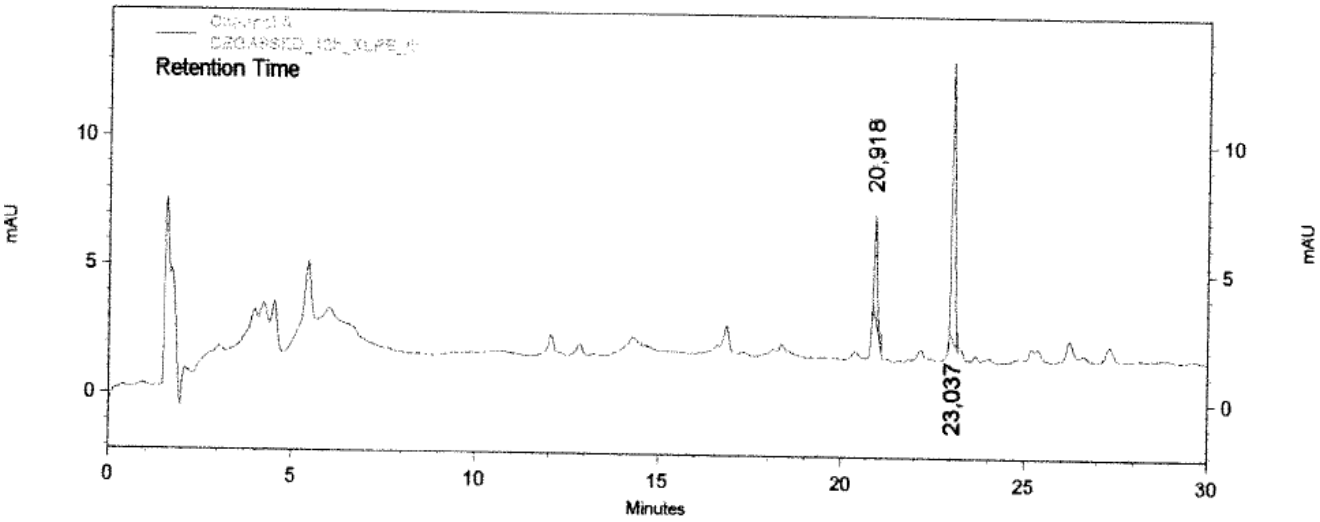


Figure 48: Sample III degassed for 12 hours at 70 °C

Analyst: System
Sample ID: DEGASSED_12h_XLPE_IV **Vial: 1** **Injection Volume:**
0

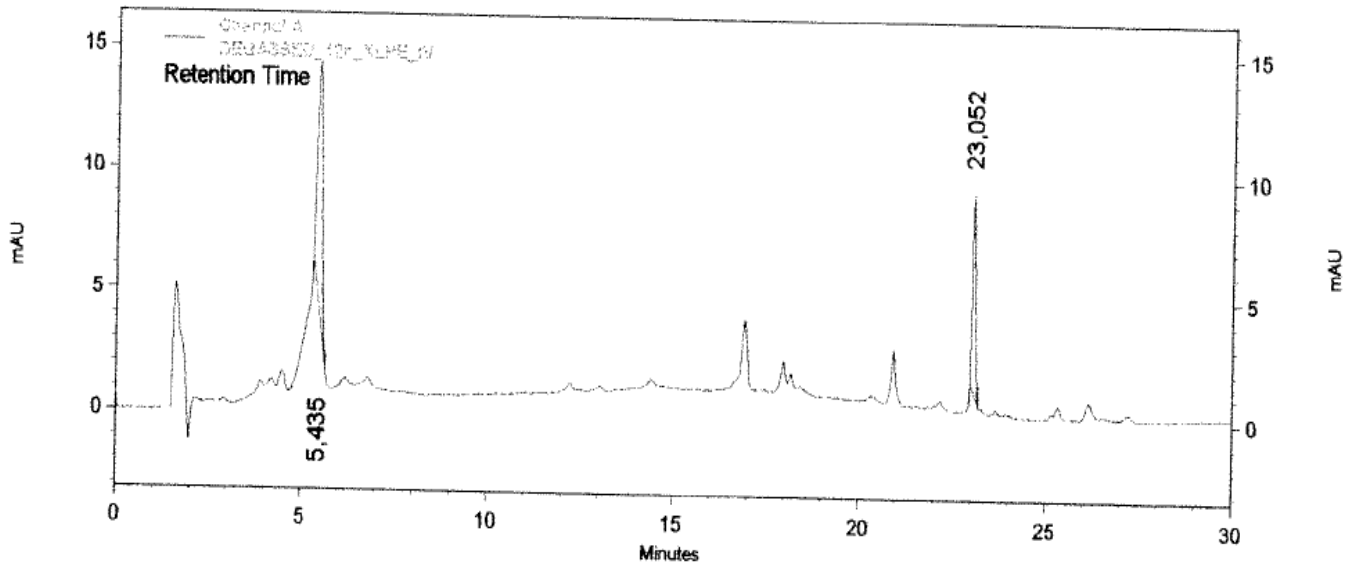


Figure 49: Sample IV degassed for 12 hours at 70 °C

Degassing for 48 hours

Analyst: System
Sample ID: DEGASSED_48h_XLPE_III_2nd **Vial: 1** **Injection Volume: 0**

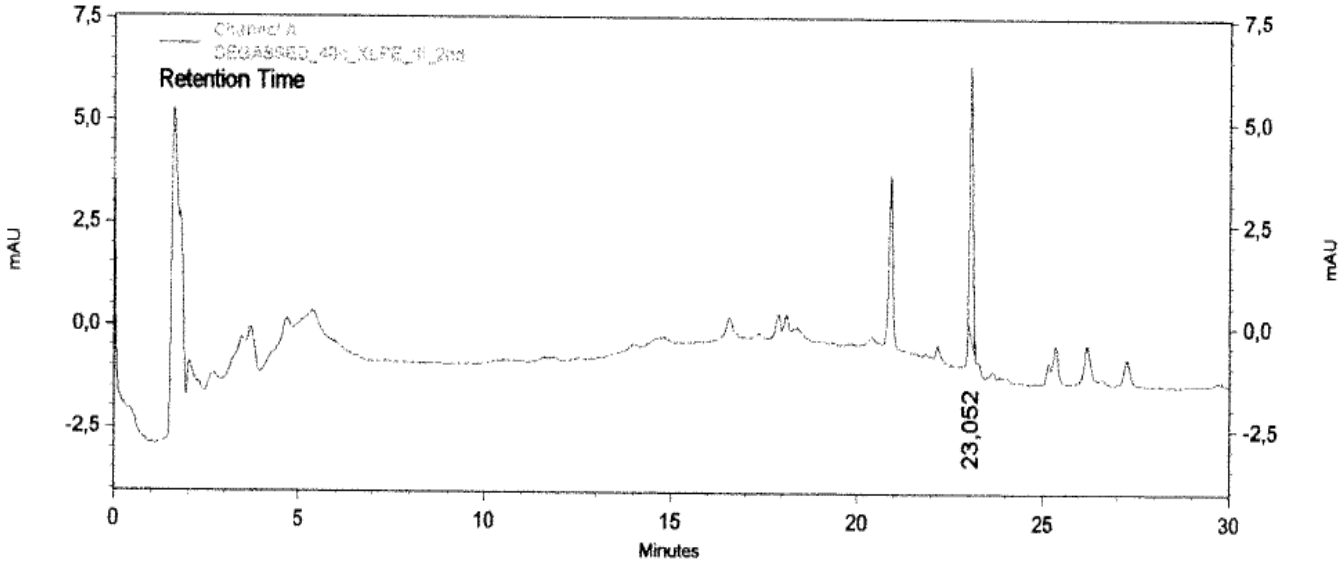


Figure 50: Sample III degassed for 48 hours. Second attempt after the first injection failed.

Analyst: System
Sample ID: DEGASSED_48h_XLPE_IV **Vial: 1** **Injection Volume: 0**

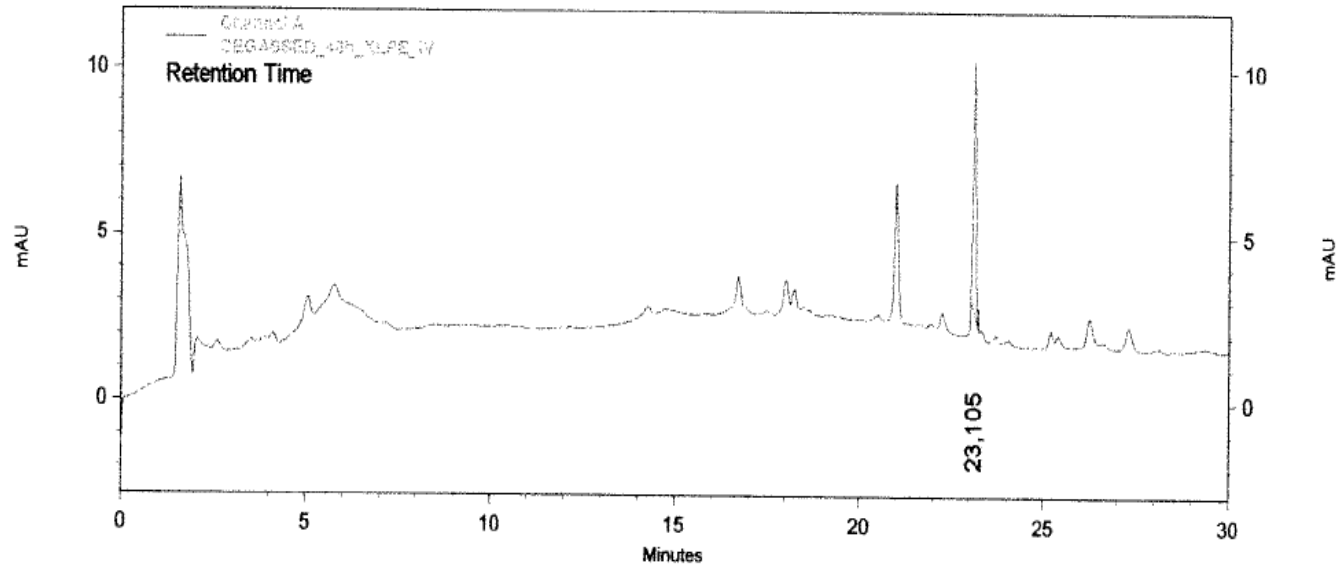


Figure 51: Sample IV degassed for 48 hours

APPENDIX 3

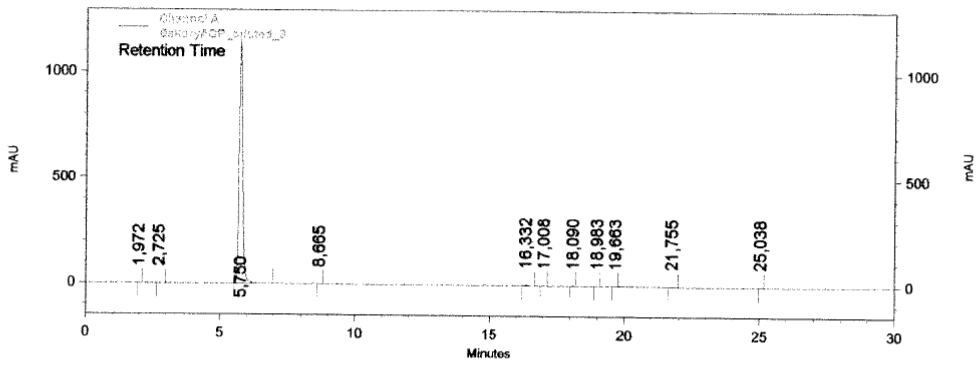
A library of detailed HPLC chromatograms, with retention time and areas, derived from the analysis of standard chemicals and degassed and non-degassed XLPE samples.

Area Percent Report

File: D:\ChromQuest\Bakary_ACP_diluted_fa.dat
Acquired: 24.03.2009 14:37:32

Printed: 24.03.2009 15:12:16

Analyst: System
Sample ID: BakaryACP_diluted_3 **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,972	1839	405	0,02	BB
	2,725	4928	693	0,05	BV
	5,750	10504969	1151607	99,32	BB
	8,665	3358	495	0,03	BB
	16,332	31366	3340	0,30	BB
	17,008	4715	662	0,04	BB
	18,090	5148	853	0,05	BV
	18,983	4599	738	0,04	BV
	19,663	2600	396	0,02	BV
	21,755	10406	1402	0,10	BB
	25,038	2441	375	0,02	BB
Totals		10576369	1160966	100,00	

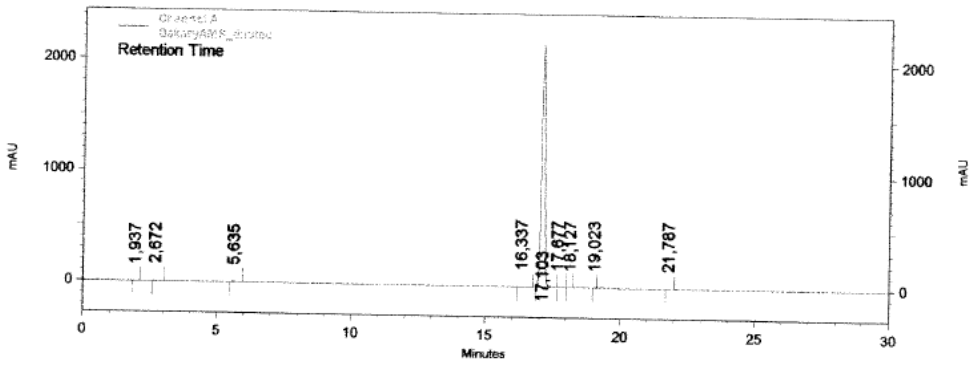
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\newbufferAMS_fa.dat
Acquired: 24.03.2009 16:34:15

Printed: 24.03.2009 17:11:21

Analyst: System
Sample ID: BakaryAMS_diluted **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,937	6170	1171	0,03	BB
	2,672	42272	5658	0,23	BB
	5,635	46624	5320	0,25	BB
	16,337	24394	2391	0,13	BB
	17,103	18579083	2167771	99,23	BV
	17,677	11805	1221	0,06	VV
	18,127	3105	515	0,02	VV
	19,023	1844	359	0,01	BV
	21,787	8667	1146	0,05	BB
Totals		18723964	2185552	100,00	

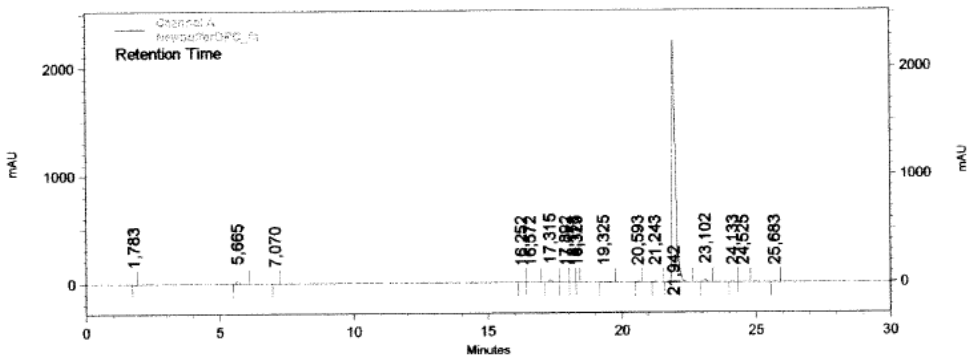
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_DPO_fa.dat
Acquired: 23.03.2009 12:20:26

Printed: 23.03.2009 12:55:53

Analyst: System
Sample ID: NewbufferDPO_fa **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,783	4892	913	0,02	BV
	5,665	239524	26044	1,17	BB
	7,070	6168	745	0,03	BV
	16,252	9545	1221	0,05	BV
	16,572	22615	2040	0,11	VB
	17,315	228251	23916	1,12	BV
	17,892	19601	1941	0,10	VV
	18,178	10008	1062	0,05	VV
	18,323	1908	367	0,01	VV
	19,325	49855	4555	0,24	BV
	20,593	5809	852	0,03	BV
	21,243	21720	2656	0,11	BV
	21,942	19654457	2246666	96,04	VV
	23,102	138731	17483	0,68	BB
	24,133	21929	2877	0,11	BV
	24,525	13411	1426	0,07	VV
	25,683	16902	1874	0,08	BV
Totals		20465326	2336638	100,00	

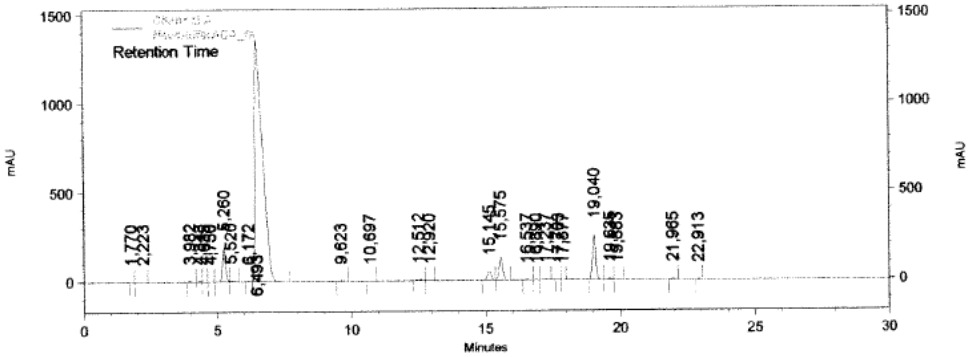
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_aca_fa.dat
Acquired: 23.03.2009 12:57:38

Printed: 23.03.2009 14:06:37

Analyst: System
Sample ID: NewbufferACA_fa **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,770	9167	1596	0,03	BV
	2,223	13594	1053	0,04	VB
	3,982	39581	5583	0,12	BV
	4,343	20255	2746	0,06	VV
	4,535	8694	1087	0,03	VV
	4,750	6115	829	0,02	VV
	5,260	1613613	194831	4,82	VV
	5,520	57190	6875	0,17	VB
	6,172	9835	1394	0,03	BV
	6,493	27813885	1360309	83,00	VV
	9,623	27304	2612	0,08	BB
	10,697	11938	1254	0,04	BV
	12,512	38303	3305	0,11	BV
	12,920	9829	1015	0,03	VV
	15,145	456483	48291	1,36	BV
	15,575	1173923	127129	3,50	VV
	16,537	19684	1998	0,06	BV
	16,890	4227	502	0,01	VV
	17,237	25073	2717	0,07	VB
	17,703	7301	990	0,02	BV
	17,877	2555	410	0,01	VV
	19,040	2009492	250481	6,00	BV
	19,635	61106	6818	0,18	VV

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_aca_fa.dat
Acquired: 23.03.2009 12:57:38

Printed: 23.03.2009 14:06:37

	19,883	19650	1697	0,06	VV
	21,965	46394	6533	0,14	BB
	22,913	5572	878	0,02	BV

Totals		33510763	2032933	100,00	

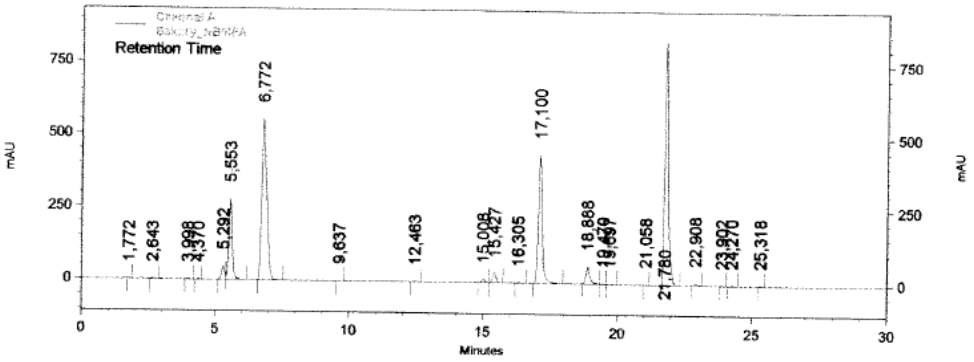
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_NBMFA.dat
Acquired: 24.03.2009 17:13:48

Printed: 24.03.2009 17:45:18

Analyst: System
Sample ID: Bakary_NBMFA **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,772	6903	1231	0,03	BV
	2,643	8854	1251	0,04	BB
	3,998	9849	1445	0,04	BV
	4,370	3857	583	0,02	VV
	5,292	381997	45902	1,65	BV
	5,553	2513419	276217	10,86	VV
	6,772	7048422	553360	30,45	BB
	9,637	3932	483	0,02	BB
	12,463	21804	2122	0,09	BV
	15,008	109470	11985	0,47	BV
	15,427	291168	31712	1,26	VB
	16,305	18220	1936	0,08	BV
	17,100	4434158	439675	19,16	BB
	18,888	603698	59037	2,61	BV
	19,470	22426	2221	0,10	VV
	19,697	13472	1101	0,06	VV
	21,058	5043	783	0,02	BB
	21,780	7582201	832252	32,76	BB
	22,908	49036	6136	0,21	BB
	23,902	8099	1166	0,03	BV
	24,270	5101	584	0,02	VV
	25,318	5490	728	0,02	BV

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

BK

Area Percent Report

File: D:\ChromQuest\Bakary_NBMFA.dat
Acquired: 24.03.2009 17:13:48

Printed: 24.03.2009 17:45:18

Totals		23146619	2271910	100,00	

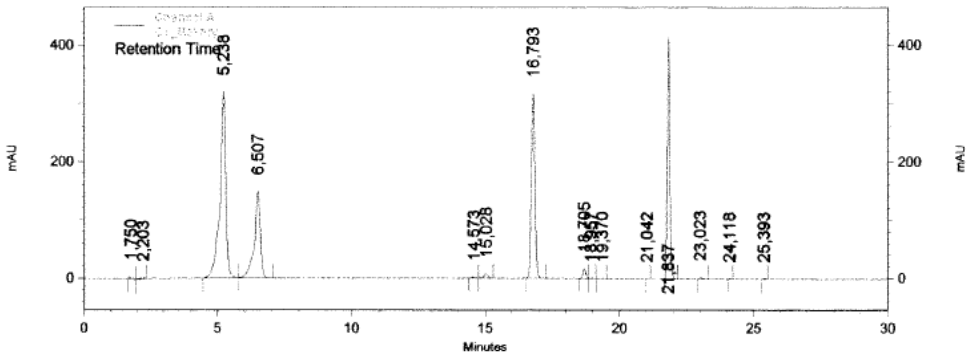
Instrument Name: Instrument 1 Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

C1

Area Percent Report

File: D:\ChromQuest\14april_c1_Bakary.dat
Acquired: 14.04.2009 17:04:30 **Printed:** 14.04.2009 17:38:53

Analyst: System
Sample ID: C1_Bakary_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,750	39157	3888	0,30	BV
	2,203	28490	1199	0,21	VV
	5,238	4901426	319212	36,99	BV
	6,507	2319759	148099	17,50	VB
	14,573	23919	2459	0,18	BV
	15,028	77538	7319	0,59	VB
	16,793	2883923	315657	21,76	BB
	18,705	134354	16906	1,01	BV
	18,957	5553	753	0,04	VV
	19,370	4128	461	0,03	VV
	21,042	1669	321	0,01	BV
	21,837	2807177	412732	21,18	BB
	23,023	19410	2760	0,15	BV
	24,118	1862	344	0,01	BV
	25,393	3858	565	0,03	BV

Totals					
		13252223	1232675	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

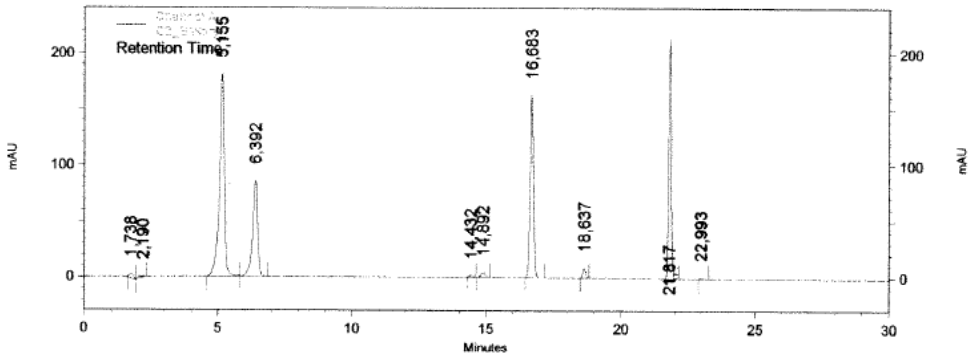
C2

Area Percent Report

File: D:\ChromQuest\14april_c2.dat
Acquired: 14.04.2009 17:41:05

Printed: 14.04.2009 18:13:32

Analyst: System
Sample ID: C2_Bakary_ Vial: 1 Injection Volume: 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,738	36925	3460	0,57	BV
	2,190	26968	1189	0,42	VV
	5,155	2331260	180674	36,05	BV
	6,392	1113359	85328	17,22	VB
	14,432	12919	1439	0,20	BV
	14,892	40478	4197	0,63	VB
	16,683	1407239	162508	21,76	BB
	18,637	64193	8414	0,99	BV
	21,817	1423524	214479	22,02	BB
	22,993	9208	1353	0,14	BV
Totals		6466073	663041	100,00	

Instrument Name: Instrument 1 Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

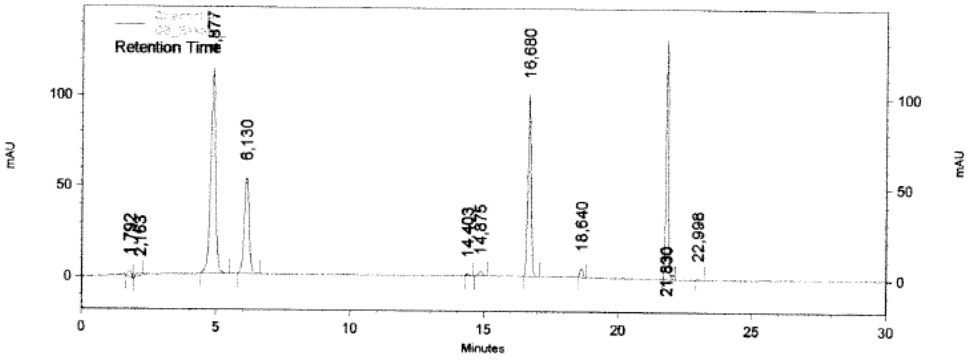
C3

Area Percent Report

File: D:\ChromQuest\14april_c3.dat
Acquired: 14.04.2009 18:14:36

Printed: 14.04.2009 18:48:58

Analyst: System
Sample ID: C3_Bakary_ Vial: 1 Injection Volume: 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,792	28485	3083	0,73	BV
	2,163	21889	1212	0,56	VV
	4,877	1400527	112839	35,92	BB
	6,130	664915	53055	17,05	BB
	14,403	7593	894	0,19	BV
	14,875	24606	2669	0,63	VB
	16,680	845536	100040	21,68	BB
	18,640	38151	5148	0,98	BV
	21,830	862393	131729	22,12	BB
	22,998	5141	793	0,13	BB
Totals		3899236	411462	100,00	

Instrument Name: Instrument 1 Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

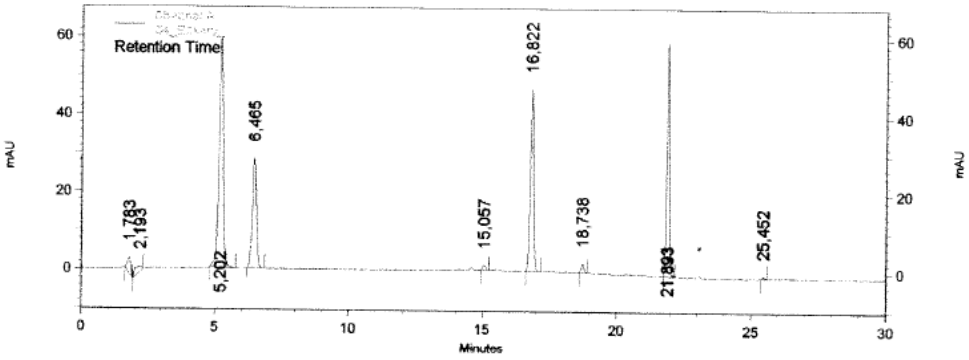
C_4

Area Percent Report

File: D:\ChromQuest\14april_c4.dat
Acquired: 14.04.2009 18:50:56

Printed: 14.04.2009 19:23:13

Analyst: System
Sample ID: C4_Bakary_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,783	35732	3955	1,95	BV
	2,193	28287	1275	1,55	VV
	5,202	646538	59363	35,35	BB
	6,465	306172	28240	16,74	BB
	15,057	9596	1181	0,52	BV
	16,822	386586	46780	21,14	BB
	18,738	16789	2326	0,92	BV
	21,893	395988	59924	21,65	BB
	25,452	3071	454	0,17	BV
Totals		1828759	203498	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

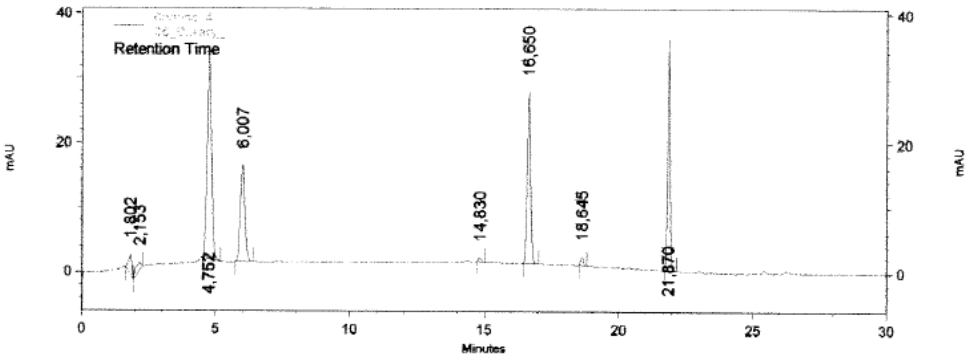
C5

Area Percent Report

File: D:\ChromQuest\14april_C5.dat
Acquired: 14.04.2009 19:25:07

Printed: 14.04.2009 19:56:32

Analyst: System
Sample ID: C5_Bakary_ Vial: 1 Injection Volume: 0



UV1000-250nm
Results (Original)

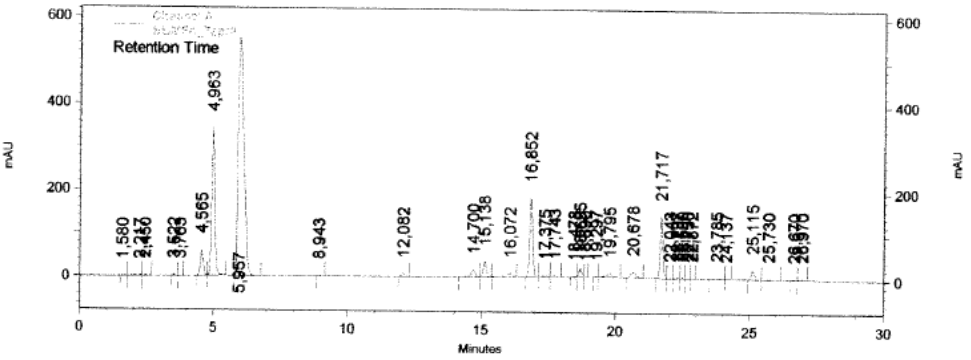
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,802	25099	2932	2,37	BV
	2,153	19375	1196	1,83	VV
	4,752	358574	31785	33,90	BB
	6,007	178076	14918	16,83	BB
	14,830	4525	584	0,43	BB
	16,650	226741	26385	21,43	BV
	18,645	9086	1258	0,86	BV
	21,870	236402	35697	22,35	BB
Totals		1057878	114755	100,00	

Instrument Name: Instrument 1 Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_7april.dat
Acquired: 07.04.2009 15:44:57
Printed: 07.04.2009 16:16:04

Analyst: System
Sample ID: NBMFA_7april **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,580	6358	730	0,04	BV
	2,217	13746	860	0,09	VV
	2,450	13123	1770	0,09	VV
	3,522	13306	2306	0,09	BV
	3,763	3278	578	0,02	VB
	4,565	453995	59229	3,00	BV
	4,963	2738695	340511	18,12	VB
	5,957	8047252	550241	53,24	BB
	8,943	5525	616	0,04	BV
	12,082	64756	6715	0,43	BV
	14,700	162296	15648	1,07	BV
	15,138	285430	34703	1,89	VV
	16,072	108928	6838	0,72	VV
	16,852	1402076	181550	9,28	BV
	17,375	29052	2338	0,19	VV
	17,743	33396	3193	0,22	VB
	18,478	5629	665	0,04	BV
	18,695	139274	19326	0,92	VV
	18,890	3200	660	0,02	VV
	19,297	5489	830	0,04	BV
	19,795	133155	8489	0,88	VB
	20,678	180798	12537	1,20	BB
	21,717	960802	140447	6,36	BV

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_7april.dat
Acquired: 07.04.2009 15:44:57
Printed: 07.04.2009 16:16:04

22,043	5098	728	0,03	VV
22,297	17509	1720	0,12	VV
22,500	8608	1289	0,06	VV
22,730	5264	787	0,03	VV
22,872	3419	594	0,02	VB
23,785	37526	1743	0,25	BV
24,137	4771	619	0,03	VV
25,115	165279	20185	1,09	BV
25,730	27587	1664	0,18	VB
26,670	10388	1083	0,07	BV
26,970	20527	1757	0,14	VV

Totals					

Area Percent Report

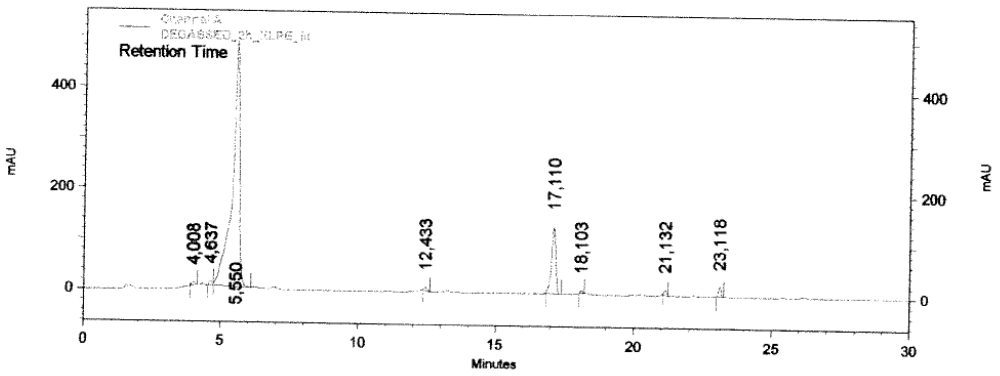
File: D:\ChromQuest\DEGASSED_2h_XLPE_III.dat
Acquired: 12.05.2009 17:53:31

Printed: 12.05.2009 18:30:07

Analyst: System
Sample ID: DEGASSED_2h_XLPE_III
0

Vial: 1

Injection Volume:



UV1000-250nm
Results (Original)

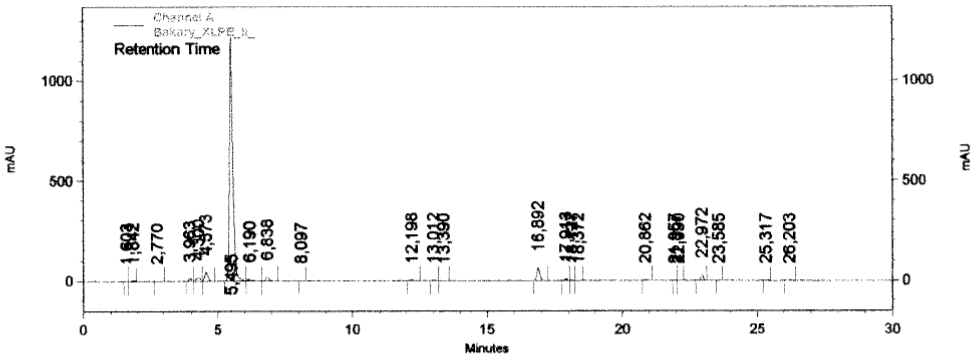
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	4,008	38297	4959	0,37	BV
	4,637	71817	7952	0,69	BV
	5,550	8756836	483988	83,81	VV
	12,433	50547	6193	0,48	BV
	17,110	1342747	131108	12,85	BV
	18,103	28438	4702	0,27	BV
	21,132	47565	7991	0,46	BV
	23,118	112541	17948	1,08	BV
Totals		10448788	664841	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_II_.dat
Acquired: 28.04.2009 15:39:57 **Printed:** 28.04.2009 16:11:35

Analyst: System
Sample ID: Bakary_XLPE_II_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,603	10581	1902	0,09	BV
	1,842	36357	3951	0,29	VV
	2,770	8280	828	0,07	BB
	3,963	101276	12882	0,82	BV
	4,300	188466	18644	1,52	VV
	4,573	374187	44581	3,02	VV
	5,495	10534356	1216154	84,93	BV
	6,190	103580	8395	0,84	VV
	6,838	164168	18469	1,32	VB
	8,097	6634	886	0,05	BB
	12,198	40294	4568	0,32	BB
	13,012	25358	3128	0,20	BV
	13,390	6809	715	0,05	VV
	16,892	469406	61480	3,78	BB
	17,913	60128	8118	0,48	BV
	18,127	27943	3793	0,23	VV
	18,372	9851	1119	0,08	VB
	20,862	32931	2879	0,27	BV
	21,957	5723	970	0,05	BV
	22,090	3734	614	0,03	VB
	22,972	173151	27459	1,40	BV
	23,585	2992	553	0,02	BV
	25,317	5803	814	0,05	BB

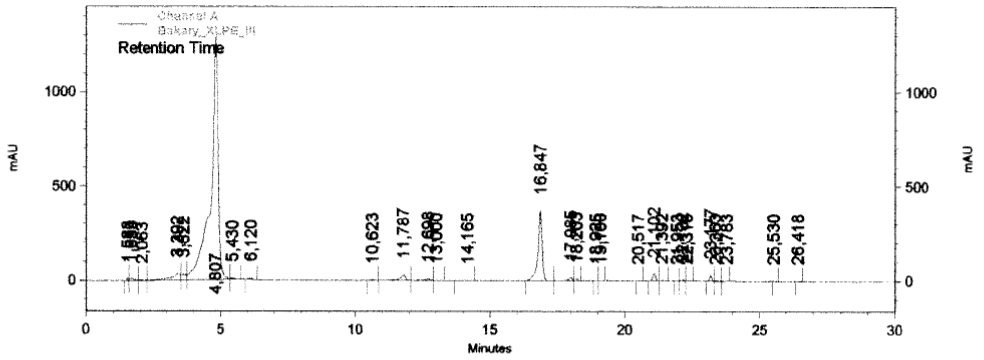
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

6.4

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_III.dat
Acquired: 28.04.2009 16:13:19 **Printed:** 28.04.2009 17:51:49

Analyst: System
Sample ID: Bakary_XLPE_III **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,533	51104	9725	0,16	BV
	1,655	117102	9411	0,37	VV
	2,063	21160	1667	0,07	VV
	3,392	680208	31759	2,12	VV
	3,622	355892	28672	1,11	VV
	4,807	25263977	1289155	78,79	VV
	5,430	73136	6192	0,23	VB
	6,120	102898	8942	0,32	BB
	10,623	19686	1750	0,06	BV
	11,787	438392	27840	1,37	VV
	12,698	178566	9443	0,56	VV
	13,000	21103	1543	0,07	VV
	14,165	14568	998	0,05	VB
	16,847	3874621	369898	12,08	BV
	17,985	183444	18111	0,57	VV
	18,203	60920	7988	0,19	VV
	18,925	9616	1347	0,03	BV
	19,100	9809	1329	0,03	VV
	20,517	3540	563	0,01	BB
	21,102	276938	37713	0,86	BV
	21,392	20919	3094	0,07	VB
	21,953	9485	1539	0,03	BV
	22,132	58734	8544	0,18	VV

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_III.dat
Acquired: 28.04.2009 16:13:19 **Printed:** 28.04.2009 17:51:49

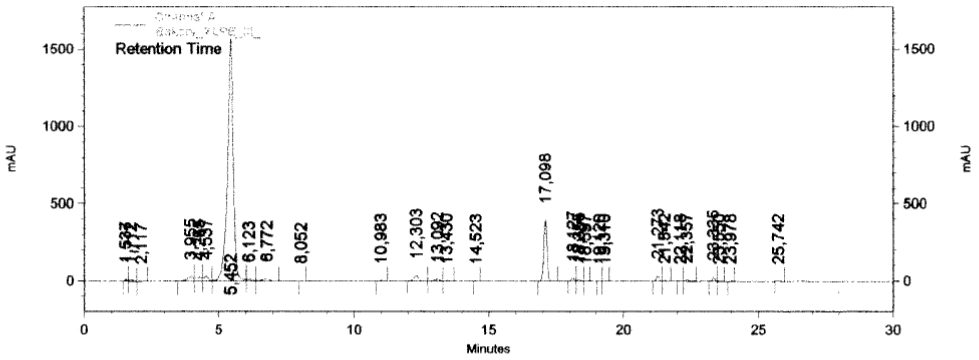
	22,318	9590	1145	0,03	VB
	23,177	190424	29870	0,59	BV
	23,363	6730	860	0,02	VV
	23,783	4046	589	0,01	VV
	25,530	3086	483	0,01	BB
	26,418	4101	519	0,01	BV
Totals		32063795	1910689	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_III_.dat
Acquired: 29.04.2009 10:27:34 **Printed:** 29.04.2009 11:02:18

Analyst: System
Sample ID: Bakary_XLPE_III_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,537	59877	9982	0,20	BV
	1,712	89852	7440	0,31	VV
	2,117	23525	1660	0,08	VV
	3,955	426693	28361	1,46	BV
	4,265	343030	21056	1,17	VV
	4,537	338911	29053	1,16	VV
	5,452	22731063	1574034	77,80	VV
	6,123	147323	10153	0,50	VV
	6,772	235098	13965	0,80	VB
	8,052	5851	717	0,02	BB
	10,983	23063	2028	0,08	BV
	12,303	397552	33794	1,36	BV
	13,092	161641	12420	0,55	VV
	13,430	26863	2161	0,09	VV
	14,523	5089	644	0,02	BV
	17,098	3418958	391684	11,70	BV
	18,127	159888	19996	0,55	BV
	18,355	74182	9275	0,25	VV
	18,597	6456	790	0,02	VV
	19,120	10128	1418	0,03	BV
	19,310	10591	1368	0,04	VV
	21,273	245793	34607	0,84	BV
	21,542	20097	2975	0,07	VB

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

9.1

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_III_.dat
Acquired: 29.04.2009 10:27:34 **Printed:** 29.04.2009 11:02:18

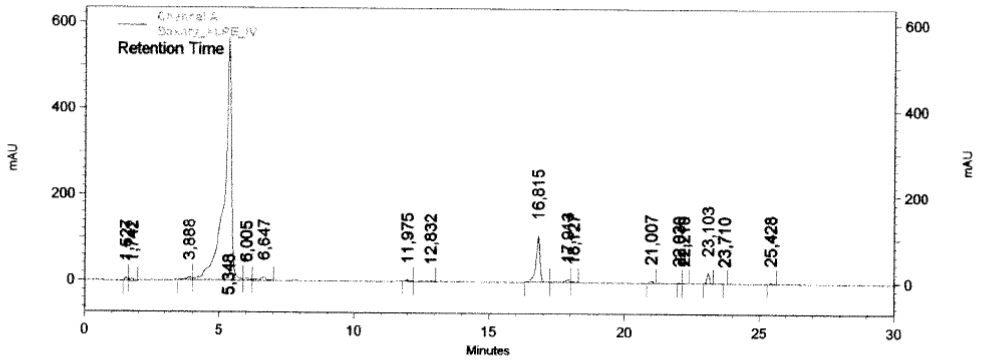
	22,118	8944	1345	0,03	BV
	22,357	60822	7323	0,21	VB
	23,335	166554	25458	0,57	BV
	23,550	5283	639	0,02	VV
	23,978	3148	516	0,01	VV
	25,742	12450	1441	0,04	BB
Totals		29218725	2246303	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_IV.dat
Acquired: 28.04.2009 17:54:27
Printed: 28.04.2009 18:52:04

Analyst: System
Sample ID: Bakary_XLPE_IV **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

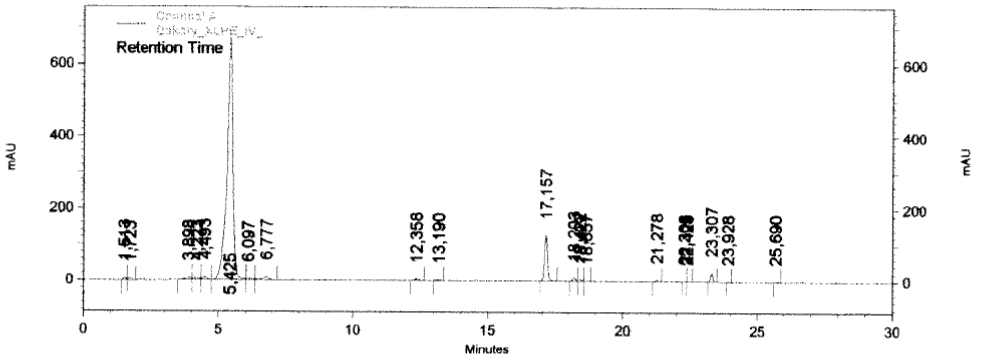
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,527	45201	5984	0,38	BV
	1,742	56652	4815	0,47	VV
	3,888	91700	6200	0,76	BV
	5,348	10176544	563003	84,65	VV
	6,005	56184	3693	0,47	VV
	6,647	126470	7758	1,05	VB
	11,975	30876	3120	0,26	BV
	12,832	22468	1563	0,19	VV
	16,815	1099461	106599	9,15	BV
	17,913	78440	7315	0,65	VV
	18,127	23540	3050	0,20	VB
	21,007	35363	4356	0,29	BV
	22,030	5590	987	0,05	BV
	22,210	3537	577	0,03	VB
	23,103	150004	23657	1,25	BB
	23,710	2255	439	0,02	BV
	25,428	17205	2235	0,14	BV
Totals		12021490	745351	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_IV_.dat
Acquired: 29.04.2009 11:05:27
Printed: 29.04.2009 12:23:23

Analyst: System
Sample ID: Bakary_XLPE_IV_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,513	43121	7313	0,35	BV
	1,723	51775	4449	0,43	VV
	3,898	96408	6704	0,79	BV
	4,223	105132	6349	0,86	VV
	4,493	103959	9003	0,85	VV
	5,425	10098925	673426	82,95	VV
	6,097	61203	4473	0,50	VV
	6,777	156236	9612	1,28	VB
	12,358	45709	4126	0,38	BB
	13,190	19422	1864	0,16	BV
	17,157	1086035	125626	8,92	BV
	18,203	70049	8875	0,58	BV
	18,422	30222	4018	0,25	VV
	18,657	3933	486	0,03	VV
	21,278	36636	4434	0,30	BV
	22,308	7168	1072	0,06	BV
	22,423	3360	588	0,03	VB
	23,307	148791	23781	1,22	BB
	23,928	2634	477	0,02	BV
	25,690	3331	481	0,03	BB
Totals					

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_IV_.dat
Acquired: 29.04.2009 11:05:27
Printed: 29.04.2009 12:23:23

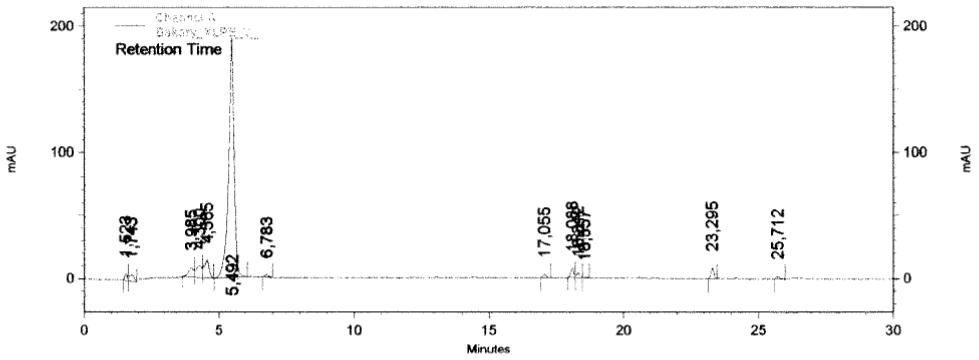
		12174049	897157	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_V_.dat
Acquired: 30.04.2009 16:07:32 **Printed:** 30.04.2009 16:43:14

Analyst: System
Sample ID: Bakary_XLPE_V_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,523	34053	4985	1,02	BV
	1,743	56137	4737	1,68	VV
	3,985	103764	7714	3,10	BV
	4,300	135868	8822	4,06	VV
	4,565	158276	13494	4,74	VV
	5,492	2658877	188983	79,55	VV
	6,783	18485	1787	0,55	BB
	17,055	22925	2719	0,69	BV
	18,088	55975	6993	1,67	BV
	18,312	27805	3341	0,83	VV
	18,557	3849	443	0,12	VB
	23,295	51549	7930	1,54	BV
	25,712	15004	1842	0,45	BV
Totals		3342567	253790	100,00	

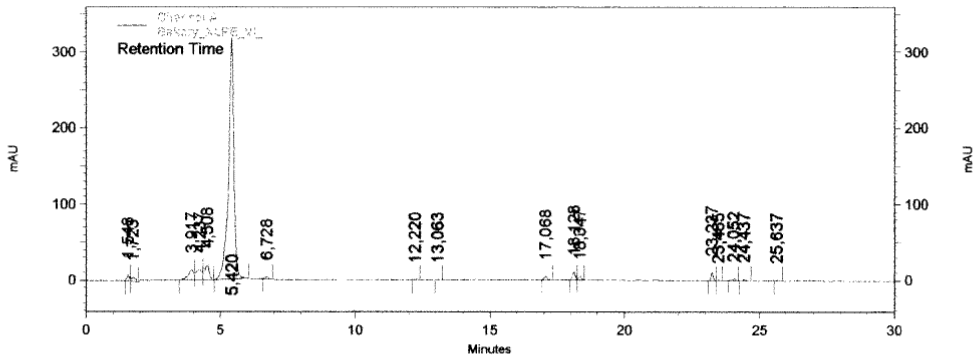
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

9:1

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_VI_.dat
Acquired: 30.04.2009 16:45:32 **Printed:** 30.04.2009 17:16:49

Analyst: System
Sample ID: Bakary_XLPE_VI_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,548	43230	7031	0,81	BV
	1,723	50920	4486	0,95	VV
	3,917	181195	12639	3,38	BV
	4,237	204161	12605	3,81	VV
	4,508	214640	18865	4,00	VV
	5,420	4377041	316577	81,63	VB
	6,728	24961	2514	0,47	BB
	12,220	5243	628	0,10	BV
	13,063	4879	603	0,09	BV
	17,068	38161	4488	0,71	BV
	18,128	77511	9937	1,45	BV
	18,347	33330	4474	0,62	VV
	23,237	67547	10404	1,26	BV
	23,485	2807	424	0,05	VV
	24,052	20085	2482	0,37	BV
	24,437	11696	1502	0,22	VV
	25,637	4619	604	0,09	BB
Totals		5362026	410263	100,00	

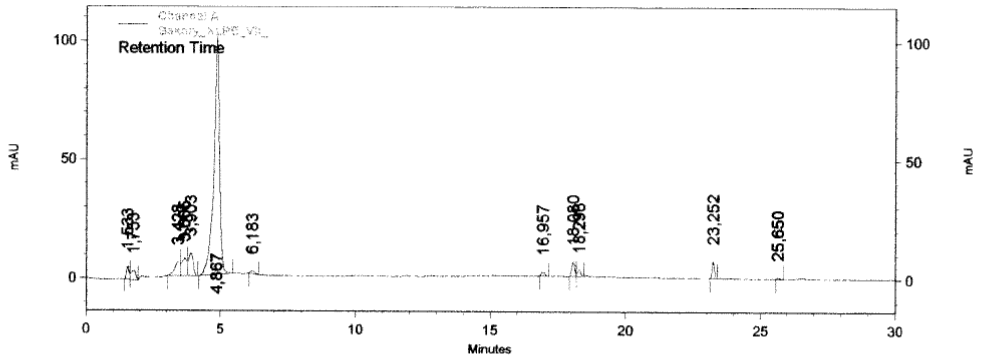
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

R:1

Area Percent Report

File: D:\ChromQuest\Bakary_XLPE_VII_.dat
Acquired: 30.04.2009 17:18:51
Printed: 30.04.2009 17:50:04

Analyst: System
Sample ID: Bakary_XLPE_VII_ **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	1,533	35513	5310	1,70	BV
	1,753	43604	3729	2,08	VV
	3,428	81915	5858	3,91	BV
	3,665	99021	7330	4,73	VV
	3,903	121355	9534	5,80	VV
	4,867	1574151	100191	75,23	VV
	6,183	11169	1069	0,53	BB
	16,957	13676	1670	0,65	BB
	18,080	44997	5903	2,15	BV
	18,298	19451	2647	0,93	VV
	23,252	43992	6989	2,10	BV
	25,650	3572	486	0,17	BV
Totals		2092416	150716	100,00	

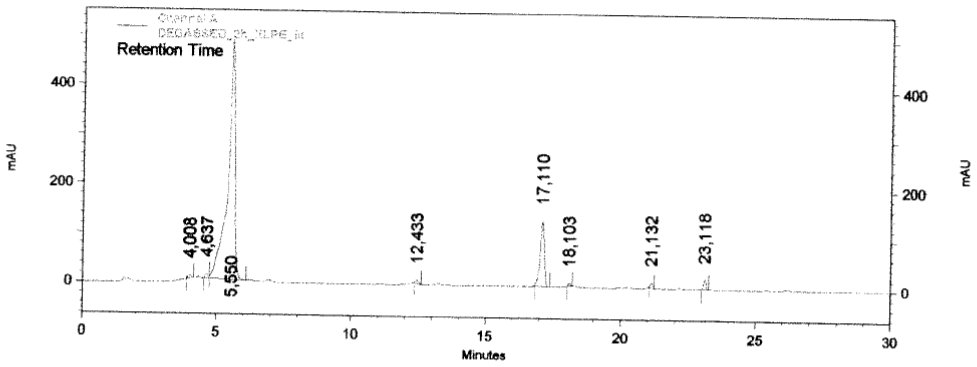
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_2h_XLPE_III.dat
Acquired: 12.05.2009 17:53:31

Printed: 12.05.2009 18:30:07

Analyst: System
Sample ID: DEGASSED_2h_XLPE_III
Vial: 1
Injection Volume: 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	4,008	38297	4959	0,37	BV
	4,637	71817	7952	0,69	BV
	5,550	8756836	483988	83,81	VV
	12,433	50547	6193	0,48	BV
	17,110	1342747	131108	12,85	BV
	18,103	28438	4702	0,27	BV
	21,132	47565	7991	0,46	BV
	23,118	112541	17948	1,08	BV
Totals		10448788	664841	100,00	

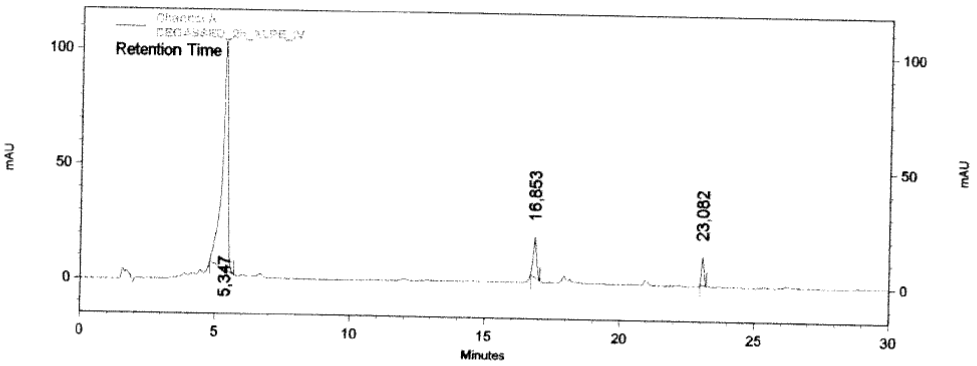
Instrument Name: Instrument 1
Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_2h_XLPE_IV.dat
Acquired: 12.05.2009 18:34:33

Printed: 12.05.2009 19:21:31

Analyst: System
Sample ID: DEGASSED_2h_XLPE_IV
Vial: 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

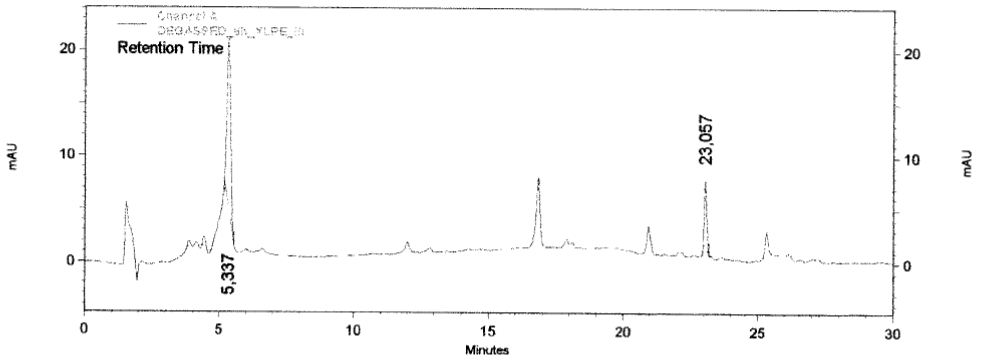
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	5,347	1474658	99990	86,79	BV
	16,853	151955	17500	8,94	BV
	23,082	72492	12015	4,27	BB
Totals		1699105	129505	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_6h_XLPE_III.dat
Acquired: 12.05.2009 21:33:48
Printed: 12.05.2009 22:05:47

Analyst: System
Sample ID: DEGASSED_6h_XLPE_III **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

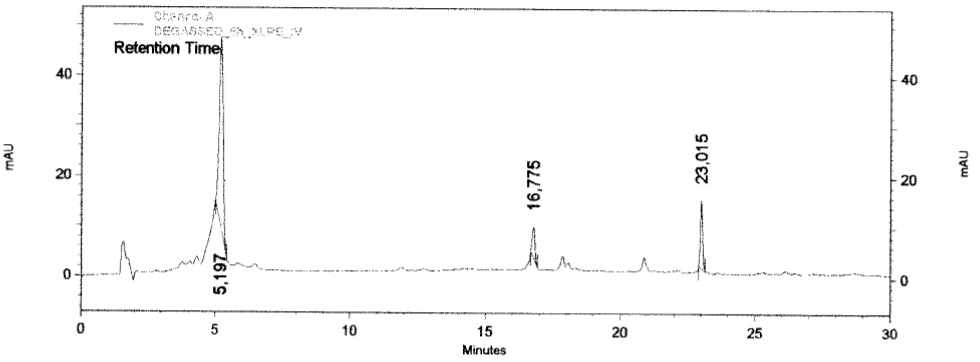
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	5,337	151571	15977	80,80	BV
	23,057	36024	6273	19,20	BV
Totals		187595	22250	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_6h_XLPE_IV.dat
Acquired: 12.05.2009 22:09:24
Printed: 12.05.2009 22:41:09

Analyst: System
Sample ID: DEGASSED_6h_XLPE_IV **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

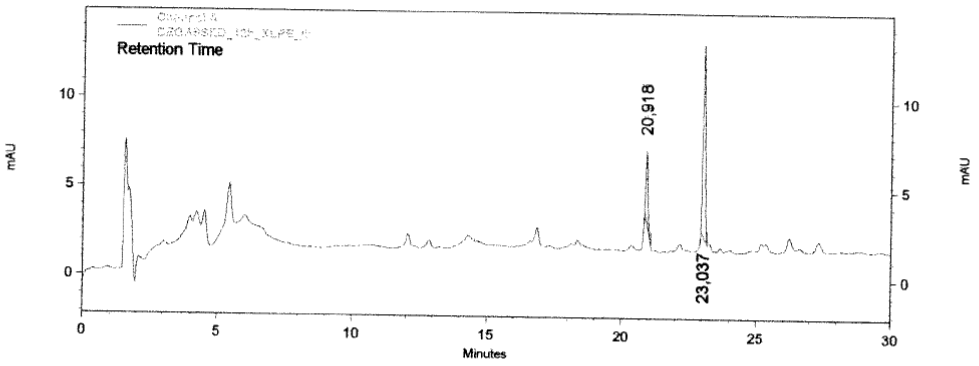
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	5,197	385984	37745	75,41	BV
	16,775	43519	6012	8,50	BV
	23,015	82329	13485	16,09	BB
Totals		511832	57242	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_12h_XLPE_III.dat
Acquired: 13.05.2009 11:54:12
Printed: 13.05.2009 12:33:42

Analyst: System
Sample ID: DEGASSED_12h_XLPE_III **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	20,918	22961	3881	25,44	BV
	23,037	67301	10737	74,56	BV
Totals		90262	14618	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

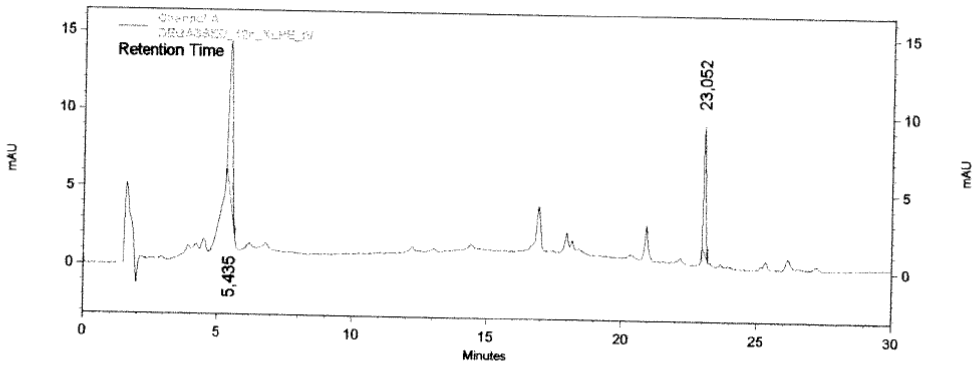
File: D:\ChromQuest\DEGASSED_12h_XLPE_IV.dat
Acquired: 13.05.2009 12:35:42

Printed: 13.05.2009 13:07:18

Analyst: System
Sample ID: DEGASSED_12h_XLPE_IV
0

Vial: 1

Injection Volume:



UV1000-250nm
Results (Original)

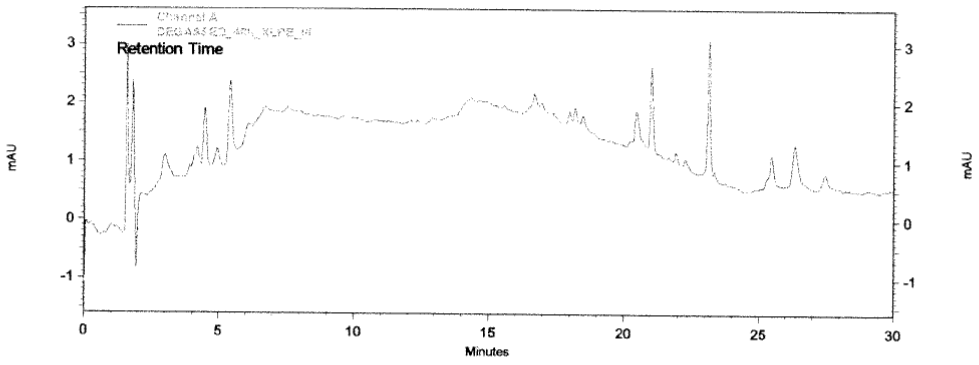
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	5,435	91769	9780	65,40	BV
	23,052	48558	8108	34,60	BV
Totals		140327	17888	100,00	

Instrument Name: Instrument 1
Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_48h_XLPE_III.dat
Acquired: 15.05.2009 14:45:13
Printed: 15.05.2009 15:17:57

Analyst: System
Sample ID: DEGASSED_48h_XLPE_III **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes

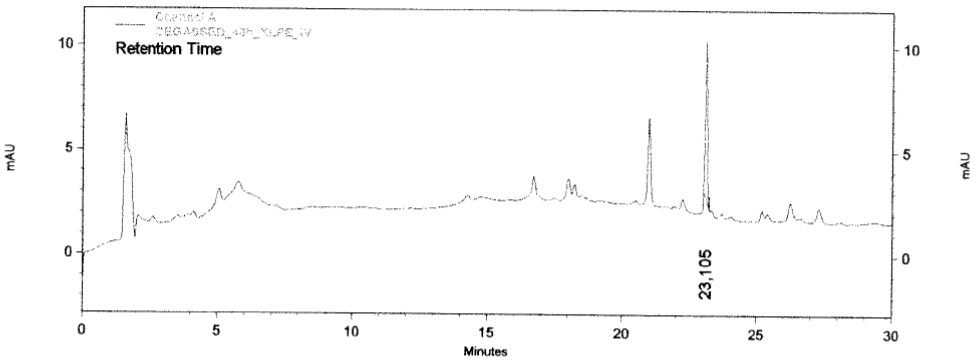
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_48h_IV.dat
Acquired: 15.05.2009 15:20:11

Printed: 15.05.2009 15:51:04

Analyst: System
Sample ID: DEGASSED_48h_XLPE_IV
Vial: 1
Injection Volume: 0



UV1000-250nm
Results (Original)

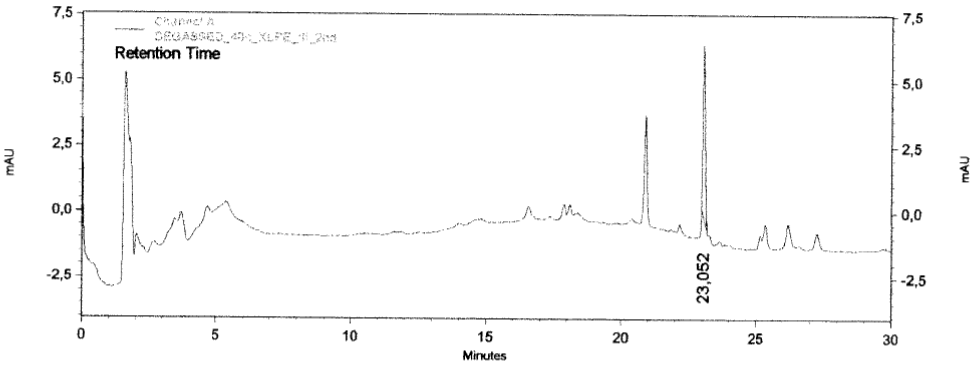
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	23,105	45433	7508	100,00	BV
Totals		45433	7508	100,00	

Instrument Name: Instrument 1
Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_48h_XLPE_III_2nd.dat
Acquired: 15.05.2009 15:52:59
Printed: 15.05.2009 16:24:14

Analyst: System
Sample ID: DEGASSED_48h_XLPE_III_2nd **Vial:** 1 **Injection**
Volume: 0



UV1000-250nm
Results (Original)

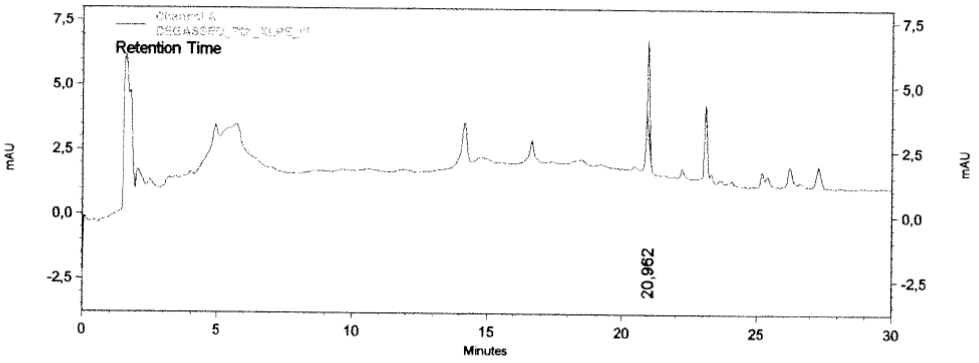
Name	Retention Time	Area	Height	Area Percent	Integration Codes
	23,052	38327	6578	100,00	BV
Totals		38327	6578	100,00	

Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_72h_XLPE_III.dat
Acquired: 15.05.2009 16:26:31
Printed: 15.05.2009 17:07:18

Analyst: System
Sample ID: DEGASSED_72h_XLPE_III **Vial:** 1 **Injection Volume:** 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	20,962	17347	3332	100,00	BB
Totals		17347	3332	100,00	

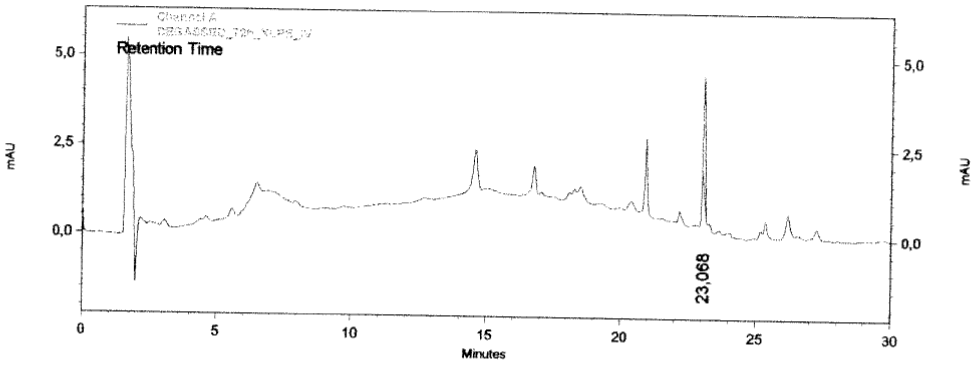
Instrument Name: Instrument 1 **Software Version:** 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq

Area Percent Report

File: D:\ChromQuest\DEGASSED_72h_XLPE_IV.dat
Acquired: 15.05.2009 17:09:36

Printed: 15.05.2009 17:55:20

Analyst: System
Sample ID: DEGASSED_72h_XLPE_IV
Vial: 1
Injection Volume: 0



UV1000-250nm
Results (Original)

Name	Retention Time	Area	Height	Area Percent	Integration Codes
	23,068	15274	3024	100,00	BV
Totals		15274	3024	100,00	

Instrument Name: Instrument 1
Software Version: 2.51
Acquisition Method: C:\Bjorn\Bakari_metode.met
Sequence: D:\ChromQuest\SEQUENCE\MultiLevel Calibration.seq