ASSESSMENT OF TWO LOCAL PLANT DYES AS COLOURANTS FOR PETROLEUM PRODUCTS (PETROL, KEROSENE AND DIESEL)

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TITLE PAGE

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A PROJECT SUBMITTED TO THE DEPARTMENT OF PURE AND INDUSTRIAL CHEMISTRY, FACULTY OF PHYSICAL SCIENCES IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF THE MASTER OF SCIENCE (M.Sc) DEGREE IN FOSSIL FUEL CHEMISTRY.

CERTIFICATION

Ilojeme Odinaka Maxwell, a postgraduate student in the Department of Pure and Industrial Chemistry with Registration Number PG/M.Sc/11/59536 has satisfactory completed the requirement for the award of the degree of Master of Science in Fossil Fuel Chemistry. The work embodied in this research project is original and has not been submitted in part or full for any other diploma or degree of this or any other university.

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PROF. C.A. NWADINIGWE	Dr. A. E. OCHONOGOR
(Supervisor)	(Head of Department)
Date ----------	Date ----------

External Examiner

Date: ----------

DEDICATION

This research work is dedicated to the Almighty God. Also, to my mother, Mrs. Ilojeme Jessie, my aunty Ilojeme Patricia, my uncle Chukwugeme Augustine, my sister Ihejika Loveth, my brother Ilojeme Stanley and to my entire family.

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I am highly indebted to a celebrated erudite, astute and quintessential scholar, Prof. C.A. Nwadinigwe, who supervised this work and patiently helped me to improve on my several weaknesses with his useful advice and encouragement. I am indeed very grateful for his mentorship, tolerance and fatherly role.

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My heartfelt thanks go to my beloved family. Others are my friends, Orjioke Ego Nwakaego, Okoye Ifeanyi, Kingsley and Chinedu.

Finally, to everyone who has played one role or the other in seeing this project through the space of time. Thanks and may God bless you all.

ABSTRACT

Dyes from *Pterocarpus osun* Craib and *Lawsonia inermis* Linn. were extracted using ethanol and methanol respectively. The extracted dyes were subsequently purified using chromatographic methods. Purified pigments were characterized using Ultra-Violet Visible Spectrophotometer, Fourtier Transform Infrared Spectrophotometer and Lovibond Tintometer. The UV spectra of the dyes in petrol, kerosene and diesel showed presence of chromophores. The FTIR spectra of the dyes showed presence of phenolic O-H stretching and C=C of aromatic functional groups. The dye from *Lawsonia inermis* Linn. was remarkably stable in colouring petrol for a period of twenty-eight days but not in kerosene and diesel while the dye from *Pterocarpus osun* Craib was not stable. Thus, dye from *Lawsonia inermis* Linn. can be used as alternative source of colourant for petrol while the dye can not be used for the same purpose for kerosene and diesel due to their instability. The dye from *Pterocarpus osun* Craib cannot be used because of their instability. They are not remarkably active in colouring any of the petroleum products (petrol, kerosene and diesel).

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CHAPTER ONE

1.0 Introduction

1.1 Dyestuffs

Today, in the world of growing environmental consciousness, natural colourants have attracted the attention of everyone[1,2]. The alarming rate of high use of synthetic dyes which causes carcinogenicity, mutagenicity, green house effect and are very expensive have provided an urgent approach to the use of natural plant dyes in the petroleum industry[3,4]. Natural plant dyes have been discovered accidentally and their uses have become so much a part of manôs customs that it is difficult to imagine modern world without dyes. The art of dyeing spread widely as civilization advanced[5].

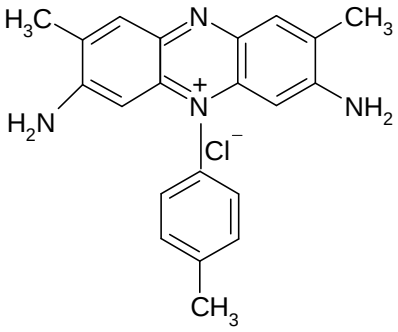
A dye is an organic compound composed of chromophore (the coloured portion of the dye molecule) and auxochrome (which slightly alters the colour) .The auxochrome makes the dyes soluble and is a site for bonding material. Dyes are molecules that can be dissolved in water or some other carrier so that they will penetrate the material[6].

For a dye to be usable in colouring materials, it must be highly coloured; must yield goods that are ñcolour fastò, or resistant to colour change or loss during use and care; and must be soluble or capable of being made soluble in water or other medium in which they are applied, or they must themselves be molecularly dispersible into the material[7].

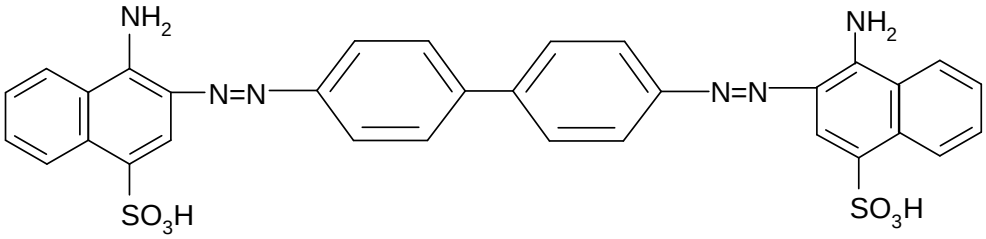
The archaeological evidences have shown that dyeing has been extensively carried out for over 5000 years, particularly in India and Phoenicia[8,9]. The dyes were obtained from animal, vegetable or mineral origin, with no or very little processing. They include cochineal obtained from dried cochineal insect, brazil-wood from *caesalphia* plant, madder from the roots of *Rubia tinctoria*, Tyrian purple from purple snail (Murex bandaris), and indigo from the leaves of *Indigofera tinctoria* and many more. The plant parts that were used are roots, bark, leaves and wood[10,11]. Colourant improve the appearance, culinary effect and promote acceptability of substrates[12].

The colour index categorizes all colouring matters according to their application and characteristics. Dyes can also be classified according to their chemical structure, shade, fastness, etc[13].

With advances in science and technology, dyes synthetically produced from coal tar and petroleum sources, and natural dyes have almost been replaced by synthetic dyes. The most significant discovery in synthetic dyes occurred in the 19th century when the first man-made dye was synthesized by a Chemist, William Henry Perkin[14]. He produced the first basic azine dye known as mauveine [1] (Mauve). The production of this dye opened door for the synthesis of many classes of dyes including the first azo dye, Congo Red [2].



[1]



[2]

Dyes are used to colour petrol, kerosene and diesel in order to improve their appearance, as well as for identification of grade, type of use or merely as a trademark of the manufacturer[15]. Solvent dyes used to colour refined petroleum products is to be able to differentiate between petrol, diesel, kerosene and jet fuels[16]. Petroleum dyes help in identification of fuel adulteration. They also help to create differentiation in various petroleum products such as leaded and unleaded, high and low octane gasoline, high and low sulphur diesel and aviation fuels attributes[17]. Other reasons for dyeing fuel, such as for aviation, supports the fuelling process itself, to ensure that the right type of fuel is used in the correct aircraft ï as the consequences of getting this wrong can be disastrous[18]. However, solubility as well as hue and fastness are the major determinant factors for dyes used in these petroleum products.

Other uses of dyes include in the colouration of food items, inks, metals, leather, textiles and paper. Other areas of use include photographic paper, as indicator in chemical analysis and biological stain and as point leak detection[19]. With the increasing demand in petroleum industry and various sectors of the economy for dyes and pigments coupled with the high cost of synthetic dyes importation, there is need to explore plant dyes and their applications will improve the socio-economy and artistic development of the nation.

Synthetic dyes have been used over the years in the petroleum industry for colouring petroleum products. This helps in check-mating adulteration, identification of grade or quality and for aesthetic purposes in the oil industry. The cost and toxicity (carcinogenity and mutagenity) of the synthetic dyes have shifted attention for alternative dyes which are cheap and environmental friendly. Dye of *Pterocarpus osun* Craib have been used to colour petroleum products (petrol, kerosene and diesel) but their stabilities have not been studied or reported while dye of *Lawsonia inermis* Linn. have not been used to colour petroleum products.

1.2 *Lawsonia inermis* Linn. plant

Lawsonia inermis (Henna) plant belongs to the family lythraceae. The plant grows at temperatures higher than 11 ^{o}C and needs about 5 years to mature. It has a height of 8-10 feet [20-22]. The leaves have been extensively used for centuries in the Middle East, the Far East and Northern Africa as dye for nails, skin, hands, hair and textile[23]. Henna is also used in treating skin problems, headache, jaundice, amebiasis and enlargement of the spleen. Leaves of *Lawsonia inermis* provide an

important cosmetic dyes and so on [24-25]. The major pigment in henna leaf is lawsone [2-hydroxyl-1,4-naphthaquinone ($C_{10}H_6O_3$)], having a fast-dyeing property[26]. *Lawsonia inermis* has been well investigated phytochemically by various researchers. The occurrence of ɓ-sitosterol glucoside, flavonoids, quinoids, naphthalene derivatives, gallic acid, coumarins, and xanthones in lawsonia leaf has been reported[27-32].

1.3 *Pterocarpus osun* Craib plant

Pterocarpus osun belongs to the family papilionaceae and it occurs throughout the tropics. The Nigerian species are trees of about 30 meters tall, with a girth size of about 2.4 meters with spreading crown and with bright yellow and usually alternate leaflets. It has distinct pinkles on twigs and young branches. The fruits are dark brown and velvety when young, with short soft and often stickly pickles mostly surrounding the centre[33,34]. The wood of *Pterocarpus osun* contains 16 % of red colour pigments, which are insoluble in water but soluble in organic acid and alkali[6]. *Pterocarpus osun* are medically useful for superficial skin diseases such as eczema and has also been used as an active ingredient in soap[35,36]. Other active constituents of *Pterocarpus osun* are tannins, phenols, saponin and flavonoids[37-40].

1.4 Statement of the problems

Synthetic dyes are no longer universally attractive in colouring petroleum products due to the numerous challenges (carcinogenic, mutagenic, toxic, cost and so on) associated with their use. The use of colourants for petroleum products cannot be

avoided because it helps in quick, visual differentiation of petrol, kerosene and diesel. It also helps in identification of grade or quality of petroleum products. Presently, the dyes used by Nigerian refineries are imported, coded and used without the source of import disclosing the exact chemical names and composition either to the refinery staff or to the public. The chemical nature used of dyes in imported petroleum products are also shrouded in secrecy. These have necessitated the search for local alternative sources of colourants for petroleum products in the oil industry.

1.5 Objectives of the study

This study has the following objectives:

i. To assess the stability of the heartwood of *P. osun* and young leaf of *L. inermis* plant extract respectively, as colourants for petroleum products (petrol, kerosene and diesel).

ii. To determine alternative sources of colouring petroleum products rather than using synthetic dyes, and

iii. To determine the stability of the dye colours when added to petroleum products.

1.6 Justification of the study

Natural dyes have been widely reported to be non-toxic, environmentally friendly and less expensive. Therefore, the assessment of two local plant dyes (*P. osun* and *L. inermis*) as colourants for petroleum products is justified.

CHAPTER TWO

2.0 Literature review

2.1 Historical development

The art of dyeing was as old as civilization. From the historical records over 5000 years, it is learnt that natural colourants were available to people during Greco-Roman periods particularly in India, Egypt and Phoenicia[8,9,41]. The use of natural dyeing materials is evident with the wall paintings and they still demonstrate the efficacy of dyeing craft that had been inherited from ancient times in India. Ancient Egyptian hieroglyphs contain a thorough description of the extraction of natural dyes and their application in dyeing[42]. Further developments extending over many thousands of years led to rather complicated dyeing process and high quality dyeing.

Natural dyes have been used since ancient times for colouring and printing fabrics[43]. Until the middle of last century, most of the dyes were derived from plants, mineral or animal sources by long and elaborate processes. Among these Indigo, Tyrian purple, Alizarin, Cochineal, Lawsone and Logwood dyes deserve special mention.

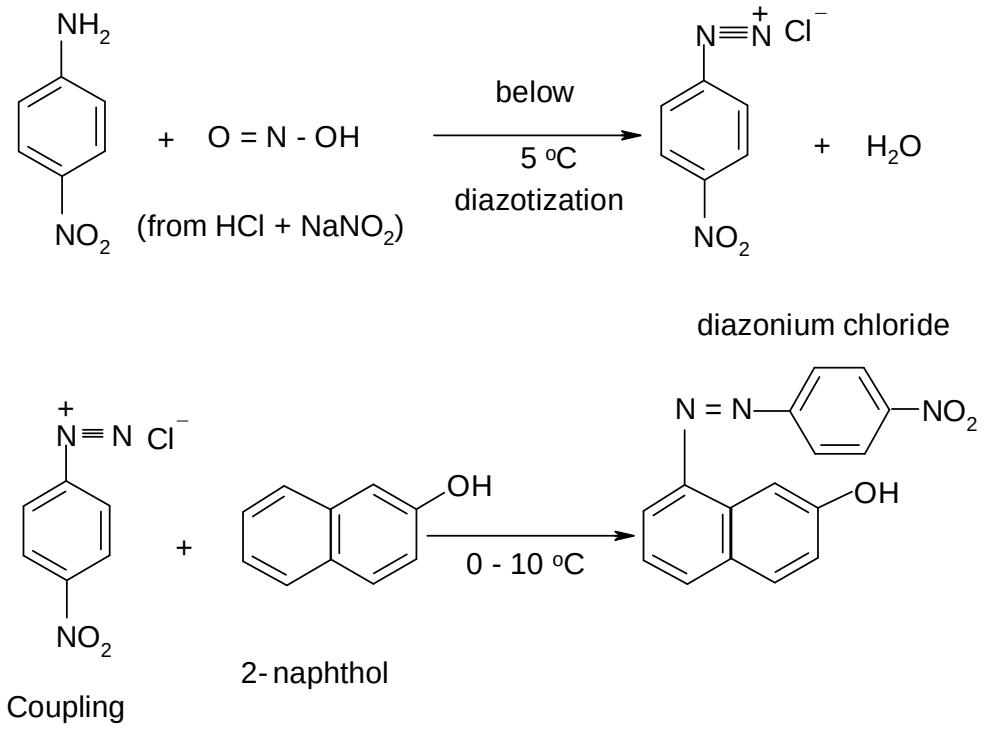
Synthetic dyes were an early result of the development of chemistry as a science. Chemistry radically changed the findings of Gebelein (1997) when in 1856, William Perkin, an English chemist working in London, made an accidental discovery of a purple dye while

trying to synthesize quinine by reacting aniline sulphate with an oxidizing agent[14].

This purple dye, which Perkin called mauve, was found to dye silk and although it was not particularly fast, it became popular. This first synthetic dye influenced the development of the synthetic dye industry. Today, industry uses synthetic dyes almost entirely because they hold their colour better than natural dyes.

Perkinôs discovery showed chemists that dyes and pigments could be produced synthetically in a laboratory. It was only a decade ago when toxicological effects of dyes during wearing dyed garments became more known and caused a great concern about the use of synthetic dyes.

In the late 1994, Germany struck a severe blow to dyestuff industries and subsequently other European countries executed ban on importation of textile and garments coloured with a series of azo dyes made from aromatic compounds, which are carcinogenic, allergic and poisonous. For instance, in the diazotization reaction hydrochloric acid reacts with sodium nitrite to form a nitrous acid, which combines with the amine to form a diazonium compounds. The diazonium compounds are active chemically and combine with various naphthoic, phenolic and amino intermediates to form the acid azo dyes through coupling.



With the present national and international awareness of environmental ecology and pollution controls, natural dyes appear to be ideal choice since they are chosen from the non-toxic lot and can be handled very easily and safely.

2.2 Colour and constituent

Colour can be defined as a sensation resulting from activation of the retina of the eye by electromagnetic vibrations, which are described as light waves[44,45].

Nature abounds with colour. Some colours such as those of hummingbird or peacock feather arise from light diffraction by the unique structure of the feathers. However, most of natureôs colours are due to the absorption of certain wavelengths of visible light by organic compounds.

Only those organic compounds that absorb in the visible region (between 400 and 700 nm) of the electromagnetic spectrum are coloured[46,47] .Light can be absorbed completely, partly or not at all by gases, liquids, or solids. That part which is not absorbed can be reflected at the surface of liquids or solids or transmitted through gases, liquids or glassy solids. The light which is emitted from a light source and the reflected or transmitted light reach the retina in the human eye.

There are light of wavelengths between 400 and 700 nm that initiate a photochemical reaction and subsequently a series of light independent reactions in the visual pigment take place. By a transfer of information between the eye and the brain, this process results in visual perception of colour. Thus, the colour which the eye records are not the colour absorbed but the colour of the reflected light or the complementary colour of the absorbed band. For instance, a substance can appear blue because it absorbs the yellow position of the spectrum only (which is the complementary colour of blue); or because it absorbs the entire visible spectrum except blue which it reflects. However, the shade will be different. Table 2.1 shows the colour absorbed at different wavelengths, and their complementary (visible) colours[46,47].

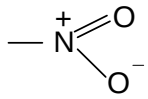
Table 2.1 Absorbed and Complementary visible colours[46,47].

Wavelength (nm)	*Colour absorbed*	*Complementary(visible) colour*
400-435	Violet	Yellow-green
435-480	Blue	Yellow
480-490	Green ï blue	Orange
490-500	Blue-green	Red
500-560	Green	Purple
560-580	Yellow-green	Violet
580-595	Yellow	Blue
595-605	Orange	Green-blue
605-750	Red	Blue-green

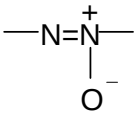
Not all coloured compounds are dyes[48]. The ability of compounds to function as dyes is closely associated with the structure of the compound. One of the earlier and still very useful explanations was given by Witt[49] who noted in 1876 that every coloured organic compound (called chromogene) contains certain unsaturated groups, fundamentally

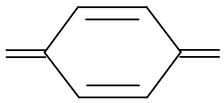
responsible for the colour to which he applied the designation chromophore (Greek, Chromo : colour, + phoros : bearer). Some of the more important chromophores are:

(a) Nitroso, —N=O

(b) Nitro, 

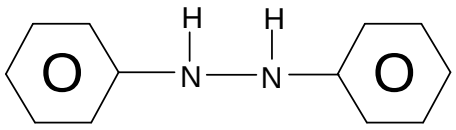
(c) Azo, —N=N—

(d) Azoxy, 

(e) Quinoid, 

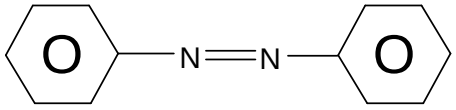
(f) Conjugated systems, (CH = CH)n

Witt further pointed out that a coloured compound becomes a dye by virtue of its possession of one or more anchoring groups called ñauxochromesò (Greek, auxo, increase). These are acidic groups such as OH, COOH, SO_3H, or basic groups like NH_2, NHR, NR_2. In a number of cases an auxochrome group not only enables the coloured compound to function as a dye (i.e. hold fast to fibers) but also modifies the colour of the compound[49,50]. For example, hydrazobenzene[3], is colourless, while azobenzene[4], is bright red. However, azobenzene is not a dye; it will not adhere to cloth. With the introduction of the amino group, the compound becomes a basic azo dye p-aminoazobenzene [5].



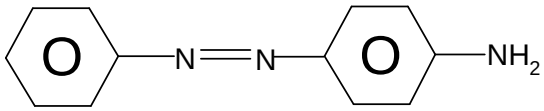
Colourless

[3]



(Red - not a dye)

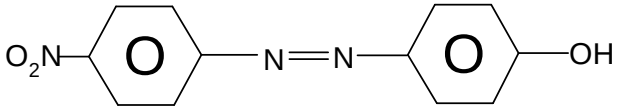
[4]



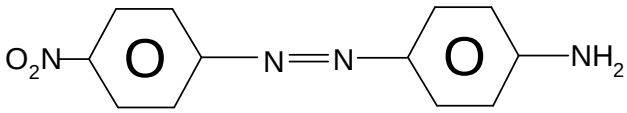
(Yellow - an azo dye)

[5]

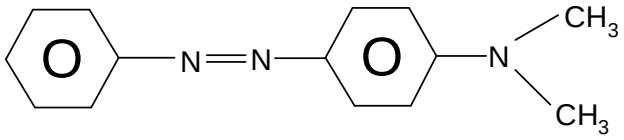
The modification of colour by the auxochromes can be seen in the following series of compounds;



(Deep yellow)



(Yellow red)



(Deep red)

The colour of a compound, in general, can be deepened by addition of substituents to increase the molecular weight. The relative position of the new substituent usually has a marked effect on the colour of the compound. Groups that deepen (increase) colour are termed ñbathochromicò groups, while those that diminish (lighten) colour are known as ñhypsochromicò groups. Bathochromic and hypsochromic effects are the shifting of the absorptive band to the longer and shorter wavelengths respectively. Deepening of colour in dye chemistry is usually taken to mean the following changes[47,51].

Yellow Ÿ Orange Ÿ Red Ÿ Purple Ÿ Blue Ÿ Green Ÿ Black

This is effectively the order of the visible complementary colour of the colour absorbed (Table 2.1)

An elaboration of the Witt theory in 1888 by Armstrong[52] regarded all coloured compounds as being quinoid in structure. However, before long it was discovered that the quinoid structure could not be given to some coloured compounds.

With the development of the theories of electronic transition, more insight was obtained on the nature and variation of colour in organic compounds. The phenomenon of absorption is associated with vibrations of electrons in the molecule responsive to the stimulation by light rays of specific oscillation frequency. When a molecule absorbs light, it undergoes transition from a lower energy state, E_o (ground state) to a

higher energy state, E_1 (excited state). From Einsteinôs equation[53], we knew that:

$E_1 - E_2 = hv$

$\Delta E = hc/\lambda$ where,

h = Plankôs constant

v = frequency of light

c = velocity of light

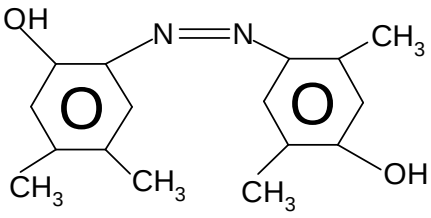
λ = wavelength of light

If the molecule consists of more than one atom, the light absorbed may bring about electronic transition, vibration or rotation. For electronic transition to occur, ΔE will be large and v will be large so that the wavelength is short. The changes in the electronic states give absorption (or emission) in the visible and ultraviolet regions of the spectrum. Hence, this determines the colour of the molecule. The more symmetrical a molecule is, the smaller the possibility of the transition dipole between different energy levels and therefore the less likely is the molecule to absorb light. Any group introduced into a molecule, which decreases the symmetry, will thus increase transition dipole and increases the intensity of the absorption. This may lead to new resonance path, hence to a shift of the band to longer wavelength. It has also been shown that resonance among charged structures affects the colour of molecules. The resonance lowers the energy of the excited state more than the ground state, hence

the greater, the resonance among various charged forms, the deeper the colour.

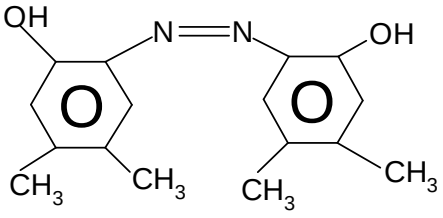
The extent of conjugation also affects the colour of an organic molecule and when a conjugated system also contains a heteroatom such as N, S, and / or O, the absorption frequency is much lower than that of the corresponding conjugated carbon compound. The effect of conjugation is well illustrated in the case of the diphenyl polygeneôs of the formula $C_6H_5(CH=CH)_nC_6H_5$. When n=1, the compound is colourless, when n =3, it is green-yellow, and when n =8, it is copperï red. As the absorption bands move from ultraviolet to blue, the colour deepens from colourless to red[51].

It has been shown that sterric factors affect the colour of organic compounds[54]. Steric hinderance of resonance in conjugated system reduces the wavelength of the molecule, hence the colour is affected. For example, the trans-form of azo-phenol [6], is coloured whereas the Cis-form [7] is colourless. The former can undergo resonance but the later cannot because a planar configuration is prevented by the spatial effect of the O-methyl groups and so resonance is inhibited hence no colour is shown.



Trans

(Coloured) **[6]**



Cis-

(Colourless) **[7]**

It is now believed that the colour of a dye is due to the interaction of the visible light with the electronic system of the dye molecule which can be affected by various factors[55,56].

2.3 Nomenclature of dye

There is no uniformity in the naming of dyestuffs[57]. In most cases, they bear the names given to them by their manufactures. Furthermore, each dyestuff may have a number of names, or different dyes may be known by the same name.

However, the commercial names of dyes are usually made up of three parts[58]. The first is a trademark used by the particular manufacturer to designate both the manufacturer and the class of dye, the second is the

colour indicated by a letter, and the third is a series of letters and number used as a code by the manufacturer to define more precisely the hue and also to indicate important properties which the dye possesses. For example, ñEric Black RX conc. Pasteò is a trade name for the colour (C.I 30235), which is direct to cotton dye, black with a reddish shade, of extra quality (improved over earlier manufacture) and standardized in strength much stronger than the ordinary type[6].

The code letters used by different manufacturers are not standardized. A few of the more widely used designation includes R for reddish, B for bluish and G for greenish shades: L for light, S for sublimation and W for wash are used for fastness properties. Dyeing properties are indicated by N for neutral and E for exhaust. There are instances where one manufacturer may designate a bluishï red dye as Red 4BL and another manufacturer uses violet 2RL for the same dye. The colour index (CI) system[58] overcomes this uncertainty by the use of a standard hue-indication chart in the shape of a hexagon with the six primary and secondary hues (yellow, orange, red, violet, blue, and green) each occupying one segment of this hexagon. A fiveï digit CI number is assigned to a dye when its chemical structure has been known by the manufacturer.

2.4 Classification of dyes

Dyes are classified in several different ways. They may be classified according to colour (yellow, red, blue etc) origin (natural or synthetic), chemical structure, substrate to which they are applied, and methods of application[19]. However the dual classification system used in colour Index (CI) complied by the Society of Dyers and Colouristic is accepted internationally throughout the dye-manufacturing and dye-using industries[58]. In this system of classification, dyes are grouped according to chemical class with a colour index number for each chemical compound, and according to usage or application class with a CI name for each dye whether made by one or several different manufacturers. Based on this, dyes are generally classified according to the method of application or their chemical constitution.

2.4.1 Classification according to methods of application

The method of application of a dye depends not only on the type and nature of the dye but also on the type of fibre to be dyed[46]. There is a marked difference in the ease with which different fibres can be dyed. Those of animal fibres such as wool, silk, leather, hair, are much more readily dyed than those of vegetable origin such as cotton, rayon, and linen[57]. This difference is because the vegetable fibres are composed of

neutral cellulose, whereas protein, the constituent of animal fibres is both acidic and basic since it is made up of amphoteric amino acids[54].

The principal application classes of dyes are as follows:

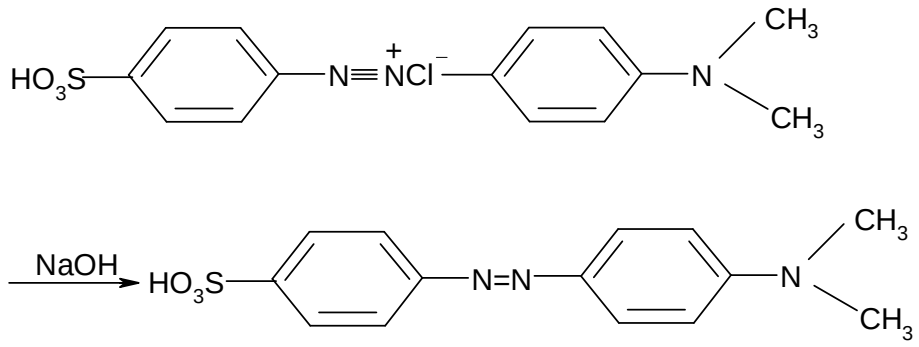
(a) Acid dyes

The term ñacidò dye has a special meaning in the textile dying. By definition, an ñacidò dye is one that dyes wool from a dye bath, which contains acid[59]. Such dyes now however, find their main application not only for wool but also for silk, polyamide, acrylic and regenerated protein fibres. They do not dye vegetable fibres directly[60].

The acid dyes are mainly sodium salts of aromatic sulphonic acids, although some contain only carboxyl and others phenolic groups[61]. They are usually applied to the fibre from an aqueous solution containing some acetic, formic or sulphuric acid; a few neutral dyeing acid dyes are also known. The dyeing of wool, silk and other protein fibres is achieved by salt formation between the basic fibre (free-NH_2 group), and the acid groups of the dye[62]. It is accomplished from aqueous medium by control of pH and temperature. The wool, for example, acting as a base, first forms a salt with the acid in the dye bath and this compound, and then reacts with the sodium salt of the dye-acid to produce a new compound, the wool-base dye acid salt.

Except for the common feature of the sulphonic acid, acid dyes vary widely in their chemical structure and belong to azo, anthroquinone,

quinophthalene, nitroarylamine and triarylmethane dyes. They vary widely in fastness to light, brilliance and tinctorial properties. An example is methyl orange [8], an acid azo dye prepared by coupling diazotized sulphanilic acid with dimethylaniline.



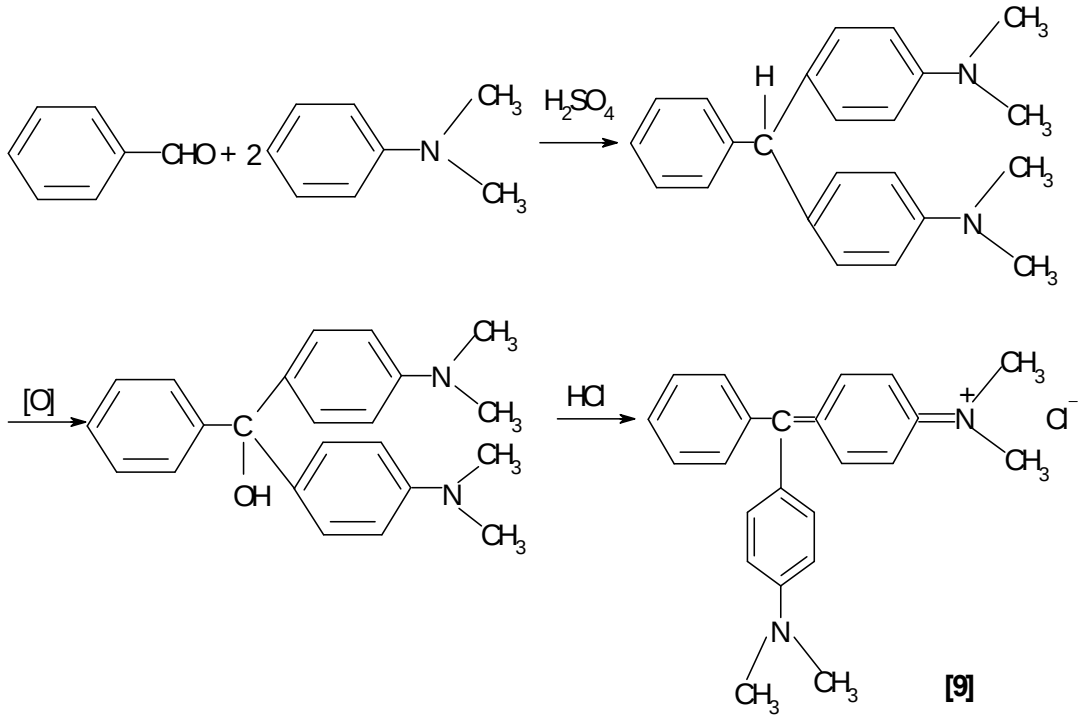
[8]

(b) Basic dyes

Basic dyes are mostly salts of colour bases with hydrochloric acid or zinc chloride. In solution the dyes yield coloured cations, hence they are frequently called cationic dyes.

They dye animal fibres directly and dye vegetable fibres in the presence of tannin (a mordant). The mechanism by which these dyes colour the substrate is believed to involve the adsorption of the dye on the fibre surfaces neutralizing the negative potential of the fibre[62]. Upon raising the temperature, the dye diffuses into the fibre and combines with acid groups in the fibre. In general, the brilliance and fastness of basic dyes are outstanding. The basic dyes chemically belong to four different groups, which include triphenylmethane derivatives, thiazines, oxazines

and azines. A typical example is Malachite Green [9], a basic triphenylmethane dye, prepared by condensing dimethlyaniline (2 molecules) with benzaldehyde (1 molecule) at 100 ^{0}C in the presence of concentrated sulphuric acid. The leuco base produced is oxidized with lead oxide in a solution of acetic acid containing hydrochloric acid, and the resulting colour base gives the malachite green with excess hydrochloric acid:



[9]

(c) Direct dyes

Direct dyes are water-soluble dyes, which exhaust into cellulose fibres, such as cotton, rayon, and linen from a salt bath. Because of this property, these dyes are called substantive dyes[58]. They also dye animal fibres directly. Direct dyes are mainly sulphonated azo compounds very

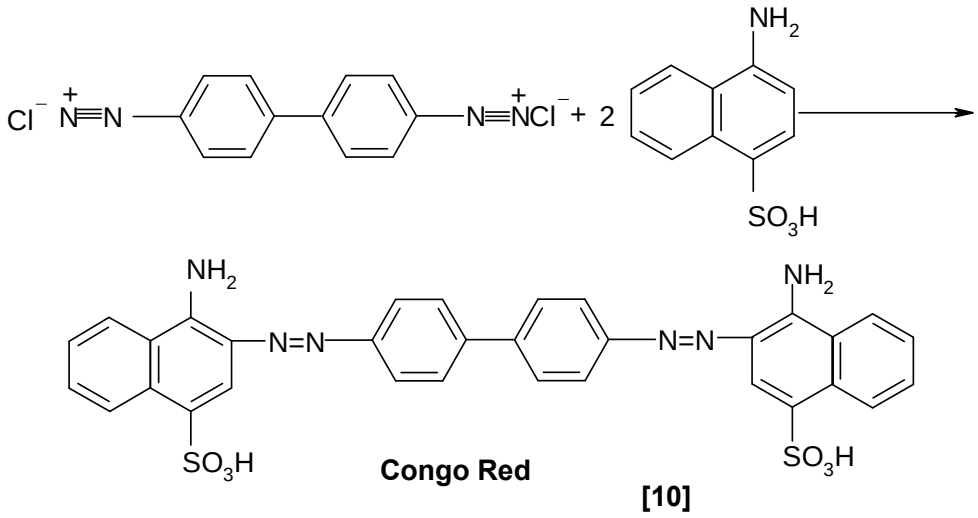
similar in constitution to the acid dyes. They differ from the acid dyes in that they are so substantive to cotton and other cellulose fibres that they fixed on the fibre from aqueous solution with the assistance of common salt or sodium sulphonate, which is added to the dye bath.

In principle, direct dyes are applied by introducing the material into the dye bath, raising the temperature of the bath to the boil and adding salt at suitable intervals. The particular procedure adopted depends on the equipment, the form of the textile and rates of exhaustion of the dyes that have been chosen[61]. The affinity of the dyestuff for the fibre depends largely on its colloidal state[62]. Large colloidal micelles are not absorbed, nor are dissociated ions; it is only the undissociated dyestuffs molecules, which are absorbed by the fibre. As the temperature of the dye bath is raised and the colloidal dye particles break down to molecular size, they are absorbed by the fibre. Salt addition promotes agglomeration and retard ionization, as also does lowering the temperature. It is for these reasons that the dyer adds salt during the progress of the dyeing cycle, and lowers the temperature of the dye bath near the end of the operation.

Direct dyes have relatively poor wash fastness, which is attributed to their tendency to ionize. The wash fastness can be improved by any of the various afterï treatment among which are: (i) diazotizing and coupling (ii) coupling with p-nitroaniline (iii) use of metal salts (iv) use of cationï

active compounds, and (v) use of cured resins or resin condensate with or without copper salts[61, 62].

A typical example of direct dye is Congo red [10] discovered in 1884 by Bottiger and prepared by coupling tetrazotised benzidine with two molecule of 1-naphthylamine-4-sulphonic:



Congo Red

[10]

(d) Mordant dyes

Mordant dyes are soluble dyes, which dye neither animal nor vegetable fibres directly, but require a mordant. The word ñmordantò is a Latin word meaning ñto biteò and is used to describe a substance incorporated with a fibre to make it more receptive to dye. The mordant could be basic or acidic. Basic mordant are used for acid dyes, while acid mordant are used for basic dyes. Basic mordant are metallic hydroxides and include hydroxides of chromium, aluminum, iron and tin. The acidic mordant generally used is tannin (tannic acid). For metal mordanting, the

fabrics is first immersed into a solution of the metallic salt and then exposed to steam, which hydrolyzes the salt to the hydroxide. The fibre is finally dipped into the solution of the dye and this produces an insoluble coloured lake, which is fast to washing. Lakes are chelate compounds formed between the metal and the dye. The colour of the lake depends on the metal used.

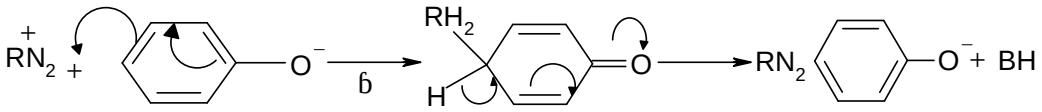
The majority of mordant dyes owe their special properties to the presence of hydroxyl groups and it is believed that dyes containing one hydroxyl group will be mordanted if this hydroxyl group is ortho to a carboxyl, nitroso, azo, or imino group. However, the sulphonic acid group cannot form lakes[60] .

(e) Ingrain dyes (Developed dyes)

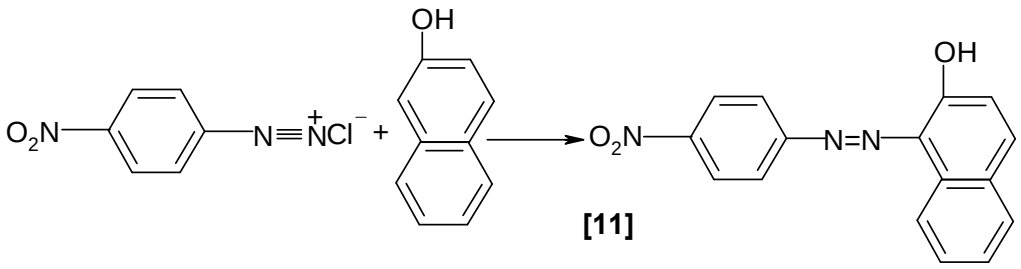
Ingrain dyes otherwise known as developed dyes are insoluble dyes, which are developed on any fibre by impregnating it with one or more of the intermediates and then producing the dye by reaction with another[51]. They are formed in situ in the substrate by some type of chemical action. The ingrain dyes are subdivided into two classes namely, the azoic dyes and the oxidation dyes.

(i)Azoic dyes: The azoic dyes are water-soluble mono- or diazo dyes which have been formed within the substrate by chemical reaction of the intermediate components. The fiber is treated with a solution of one of the components (a phenol, an amine, etc) and then passed through a

solution of the other (usually a diazonium compound) to give a coupling reaction within the fiber. The azoic dye is formed by the reaction of the strongly electrophilic diazonium ion, which attacks nucleophilic centres available in ß-ketonic acid, and aryl amides or in the ortho-and para-positions in substances such as phenols. The attack on the nucleophilic centre is followed by ejection of a proton from the complex:



Para Red [11] is an example of ingrain dye. In its application, the fabric is first wetted with an alkaline solution of ß-naphthol, then after being washed and dried, it is dipped into a solution of p-nitroaniline diazonium chloride:

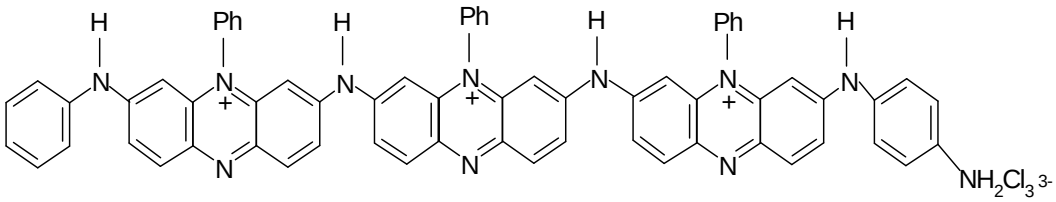


[11]

Because of the ease with which diazonium salts decompose. They have to be applied at low temperatures (0 ï 5 ^{0}C), hence azonic dyes are usually referred to as ñice coloursò. Alternatively, the diazonium salts can be employed in a ñstabilizedò form[61]. Such products are marked under the name ñfast saltò.

Azoic dyes are used primarily on cellulosic fibres but have found limited use also in wool, fur acetate, rayon, polyesters. They are used also in great quantity in printing.

(ii) Oxidation dyes: These are produced directly in the fiber or other substrates by a chemical oxidation following the impregnation of the substrate with certain aromatic amine. They are used principally for dyeing of hair fur, cotton and rayon. The principal dye of this class is aniline black [12], which are produced by chemical oxidation.



[12]

They oxidized aniline hydrochloride by oxidizing the fibre impregnated either with the amine salt, or by heating the fibre with a solution of the secondary component.

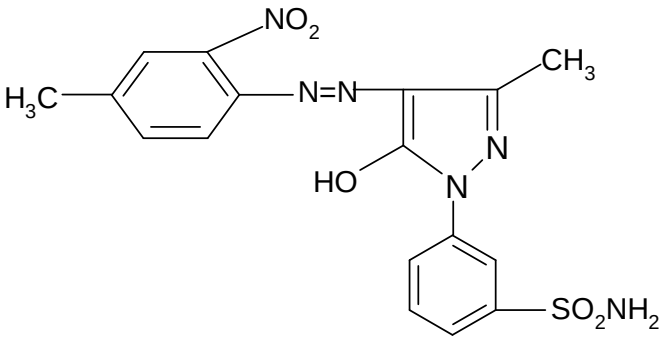
(f) Disperse dyes

Disperse dyes are water-insoluble dyes which are dispersed by suitable reagents before application to synthetic fibre. They are ionic in nature. They are generally applied in the form of dispersions from an aqueous medium and are ñdevelopedò by thermal means. The dispersing agent was originally sulphurinoleic acid, but now replaced by more effective reagents such as the sodium salt of a condensate of cresol with

naphthalene sulphonic acid and formaldehyde with a little sodium alkyl naphthalene sulphonate[61]. The dispersing agent is used to maintain the dye in a finely divided state, and to assist in dispersion in the dye bath.

To prepare the dye bath, the dye powder is pasted with cold water and dispersed by means of a dispersing agent in a limited quantity of water. The resulting dispersion is added through a sieve to the bulk of the liquor, which contains surfactant. The surfactant assists in preventing aggregation of the dye particles and their adherence to the surface of the material. Dyeing may be commenced at 40 ^{0}C and temperature raised 130 minutes to 80 ^{0}C.

The disperse dyes were originally developed for use on cellulose acetate. They now find extensive use on nylon and polyesters[62]. A typical example of a disperse dye is a pyrazolene derivative [13], which is yellow in colour.

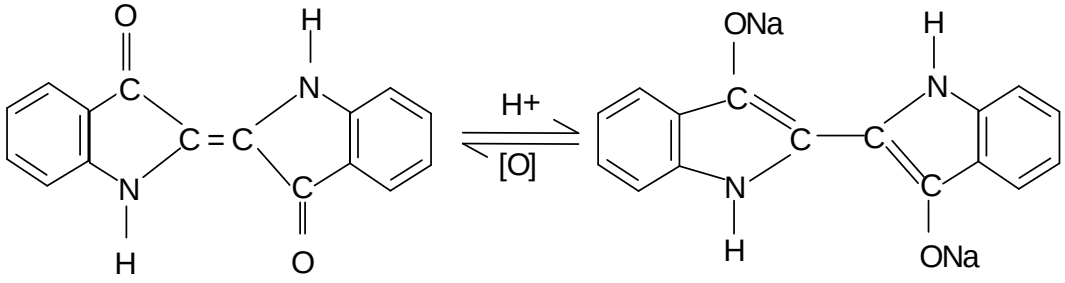


[13]

(g) Vat dyes

Vat dyes are waterï insoluble dyes which when treated with an alkaline reducing agent, usually alkaline sodium hydrosulphite ($Na_2S_2O_4$), are converted into reduced character and form soluble sodium salts, which are readily deoxidized to the dye. These reduced compounds are often white or colourless and so are called leuco-compounds. It is this soluble form of the dye, which the fibres adsorb. Once adsorbed, oxidation by air or oxidizing agents produces the insoluble form in situ in the fibre.

Chemically, the vat dyes belong to two group namely, the anthraquinone and the indigo dyes. They can dye both animal and vegetable dyes directly after reduction. Nevertheless, they are used mostly on cotton[60]. An example of vat dyes is indigo dye [14]. To dye with the indigo dye (indigotin), its paste is agitated with alkaline sodium hyposulphite in large vats. These results in the reduction of the insoluble dye to the soluble leuco-compound, indigotin-white;



Indigotin- white

[14]

The material to be dyed is soaked in the soluble leuco compound and then exposed to the air where upon the original blue dye is regenerated in the cloth. In the dye bath, the decomposition and oxidation of the reducing agent occurs with the production of acidic products: the reaction may be written as:

$$2Na_2S_2O_4 + O_2 + 4NaOH \text{ Ÿ } 4Na_2SO_3 + 2H_2O$$

(h) Sulphur dyes

Sulphur dyes are complex mixtures of uncertain composition[63]. They are produced by heating sulphur derivatives (e.g. sodium polysulphide) with certain phenols and aromatic amines. They are insoluble in water and acids but soluble in cold alkaline solution of sodium sulphide, in which the dyes are reduced to the leuco-compound. It is believed that the dye is reduced to the mercaptan derivatives[61] i.e.

$$D\text{-}S\text{-}S\text{-}D + 2H^+ \text{Ÿ } 2DSH + 2e^-$$

The material to be dyed is dipped into the leucoform and then exposed to the air, where upon the leuco-compound is oxidized to the dye. Oxidation may also be affected by immersion in a warm bath of dilute potassium dichromate.

Sulphur dyes are used extensively on cellulosic fibres because of their low price and relatively good fastness. One of the most important sulphur-dye is Vidal Black (CI Sulphur 3) prepared by heating

p-phenylenediamine, p-amino acetanilide and sulphur, with or without Benzidine and then heating the product with sodium sulphide[61].

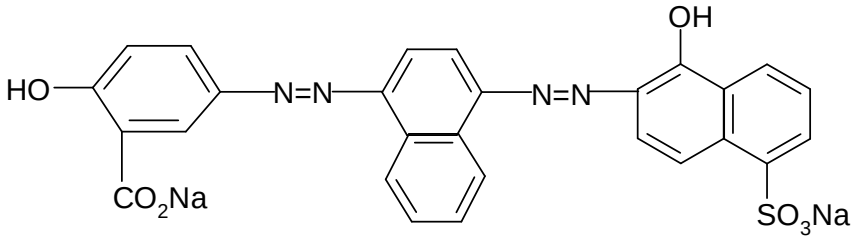
(i) Chrome dyes

Chrome dyes are capable of combining with chromium to form insoluble compounds on the fibre. These dyes may be divided into two groups, acid and premetallised chrome dyes

Acid chrome dyes are acid-dyeing colours, which can be applied in the usual way and be combined with chromium. These dyes may be applied by any of three general methods, bottom chrome, after chrome or single bath (autochrome) method. In bottom chrome method, the wool is first mordanted with sodium or potassium dichromate by boiling with the salt for some time. After washing the wool, it is then dyed with the appropriate dye in the presence of acetic acid. The essence of the dye is to neutralize any residual alkali, which may be carried over by the scoured wool or liberated in the ñchromingò process. After chrome involves a process whereby the wool is first dyed, and the colour is converted into the insoluble compound by treatment at boil with potassium dichromate ($K_2Cr_2O_7$). This method had the advantage that level dyeing may be achieved prior to chroming.

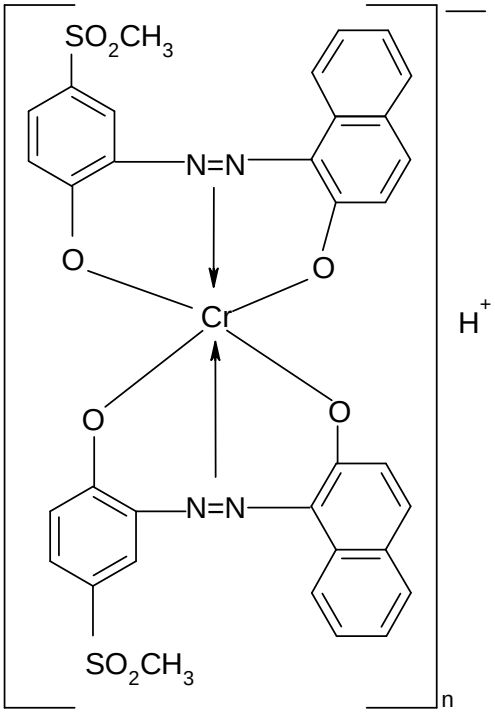
The single bath or auto chrome method involves simultaneous application of the dye and the mordant. In this case, the mordant is neutral ammonium chromate or any of the special mordants such as

metachrome, synchrine, etc. A typical example of acid chrome dye is Diamond Black F [15], which is produced by coupling diazotized 5-aminosalicyclic acid with 1-naphthylamine, and diazotizing the product with 1-naphthol-4-or 5-sulphonic acid.



[15]

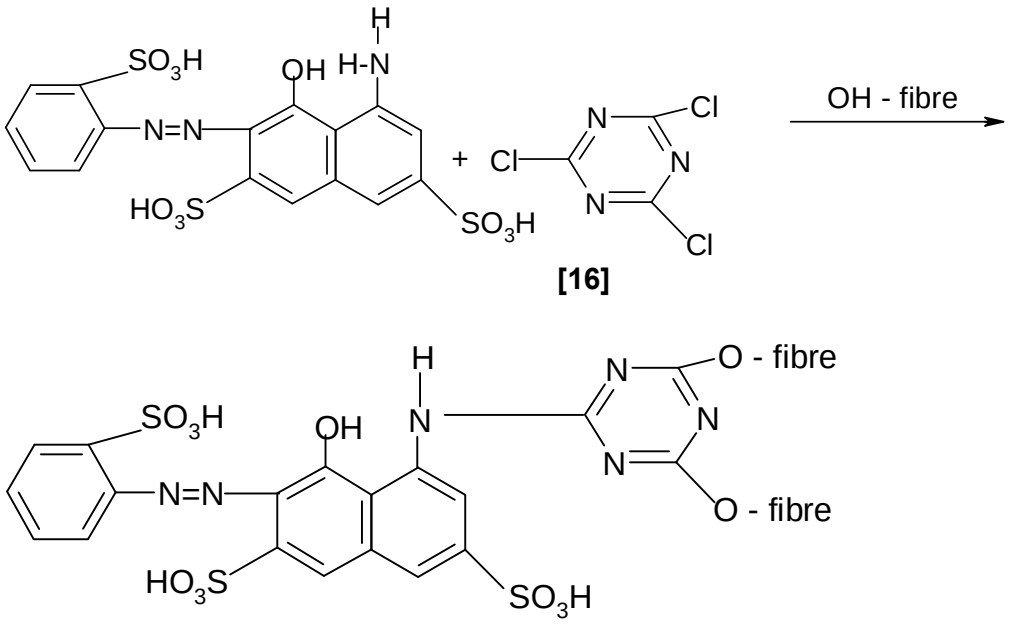
One disadvantage of acid chrome dyes is that the fibre tends to be damaged under condition of chroming. This has been overcome by using neutral dyeing premetallized dyes[60]. Premetallised dyes are preformed chromium dye complexes. The complexes are prepared by reaction of the dye with a chromium salt. (formate, fluoride etc) or coupling in the presence of a chromium salt. One group of such dyes is the Irgalans, which are 2:1 complexes (two dye molecules coordinated with the chromium atom), e.g.



These complexes are anionic and dye wool in neutral solution. They have excellent fastness to light and to washing.

(j) Reactive dyes

Reactive dyes are coloured compounds that contain functional groups capable of forming covalent bonds with active sites in fibres, such as hydroxyl groups in cellulose, aminothiol, and hydroxyl groups in wool or amino groups in polyamides. For example, the triazine derivative reactive dye [16], is a reactive dye for cellulose. The dye is stable in water and becomes attached to the cellulose fibre via hydroxyl groups on the fibre.

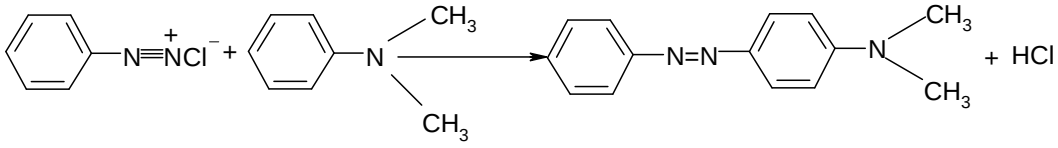


The bond formation between the functional groups in the dye and the substrate results in high wet fastness properties. The reactive dye differs from other dyes that own their wet fastness to physical absorption or mechanical retention. The simplicity of continuous application and the brightness of reactive dyes make them useful in printing as well as dyeing. They are used mostly in the dyeing of cellulose, wool and nylon. They have also found use in dyeing of silk, hair and leather.

(k) Solvent dyes

Solvent dyes are dyes that are soluble in organic solvents such as benzene, gasoline, alcohol, acetone, fats and waxes. They colour substrates by the solution of the dye in the solvent. They are sub-classified as spiritï soluble dye with principal solubility in alcohol, and oil-soluble dyes, which are soluble in benzene, vegetable and mineral oils.

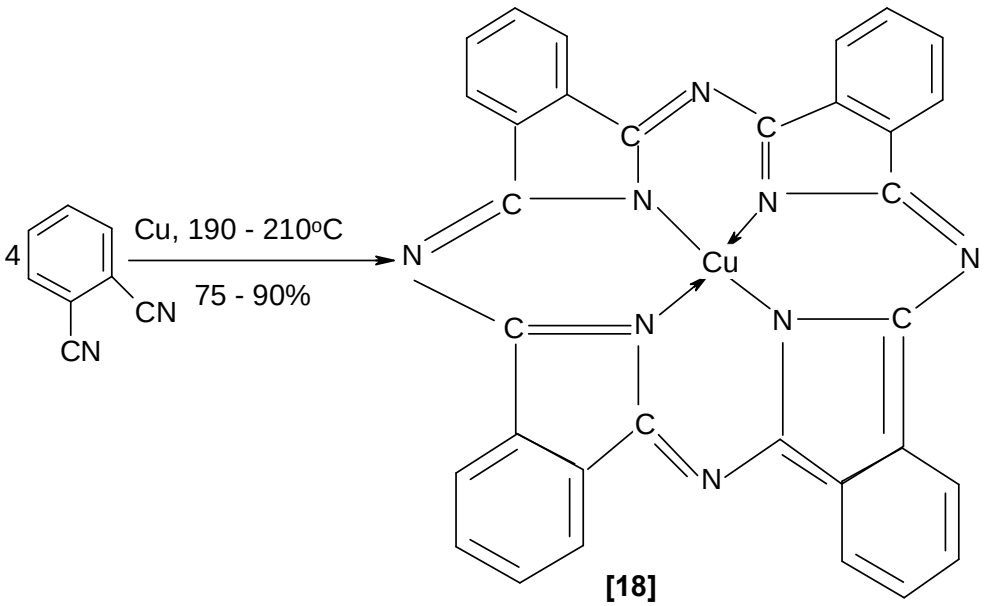
The principal uses of solvent dyes include in wood stains and varnishes, lacquers, printing and writing inks, butter, margarine, plastics and resins. Example of solvent dye is butter yellow (C.I. solvent yellow 2) [17], which are produced by the reaction of dimethylaniline with benzenediazonium chloride:



[17]

(l) Organic pigment

Organic pigments are not dyes but are insoluble coloured solids. They are insoluble in water and most organic solvents. They are used for colouring lacquers, paints, varnishes, etc. Some organic pigments have been made water-soluble and then used as dyes, hence the classification of organic pigments as dyes. An example of organic pigment dye is phthalocyanine dyes. Copper phthalocyanine [18], is readily made by the reaction of the metal with phthalonitrile:



[18]

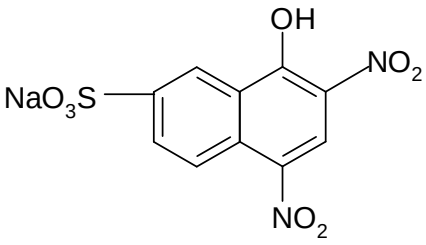
2.4.2 Classification according to chemical constitution

The classification of dyes based on their chemical structures or constitution is the most precise and scientific classification. It is the classification of interest to the research chemist and the manufacturer of dyes. The main classes of dyes based in this classification include the following:

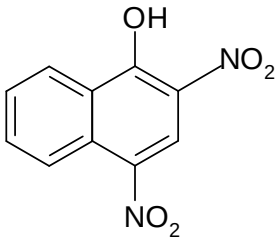
(a) Nitro dyes

The common chromophore of the group is the nitro group (-NO_2) while the autochromes can be -OH,-SO_3H, and Cl. They are generally prepared by nitration of phenols, naphthols and diphenylamine. A typical example is yellow S (-2,4-dinitro-1-naphthol-7-sulphonic acid sodium salt)[19], which is prepared by nitrating 1-naphthol-2,4,7-sulphonic acid

or 4-nitroso-1-naphthol-2,7-disulphonic acid. Another example in this group is Martius yellow.



[19]

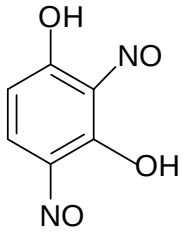


[20]

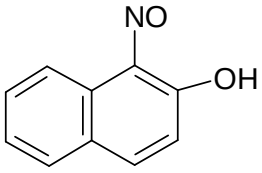
(-2,4-dinitro-1-naphthol) [20], and is prepared by nitrating 1-naphthol -2,4-disulphonic acid.

(b)Nitroso dyes

In nitroso dyes, the chromophore is the nitroso group (-N=O) and usually -OH group is the auxochrome. The nitroso dyes are usually prepared by reacting phenols or naphthols with nitrous acid-fast Green O [21], is an example of nitroso dye and it is prepared by reacting nitrous acid with resorcinol. Another example in this group is Gambine ɔ (1-nitroso-2-naphthol)[22].



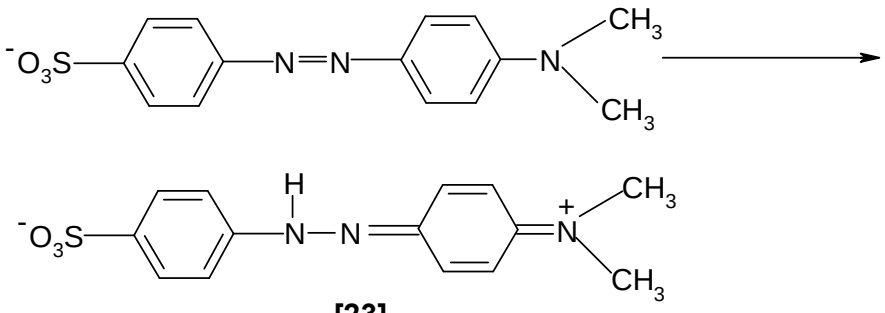
[21]



[22]

(c) **Azo dyes**

Azo dyes are characterized by the common chromophore, the azo group -N=N-, and generally have such auxochromes as -OH, $-NH_2$ and $-NR_2$. With the exception of stilbene azo dyes, all azo dyes are manufactured by reacting aromatic primary amine with nitrous acid in mineral acids (i.e. diazotization) yielding diazonium salts which are reacted with aromatic amines or phenols (i.e. coupling). The coupling with amines proceeds under acidic conditions, while phenols require alkaline media. The process of diazotization and coupling can be repeated to give diazo and polyazo dyes. The azo dyes make up the largest class of synthetic dyes because of the great number of possible synthetic alternative. Methyl orange [23], used as an indicator is an example of azo dye, and is prepared by coupling diazotized sulphanilic acid with dimethylaniline:

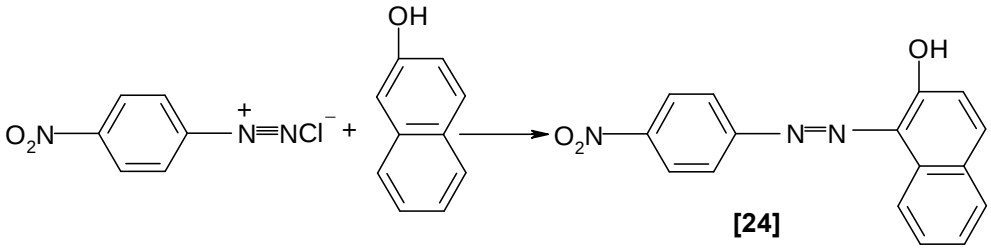


[23]

There are different types of azo dyes. These include basic, acid, direct, ingrain, mordant and stilbene azo dyes.

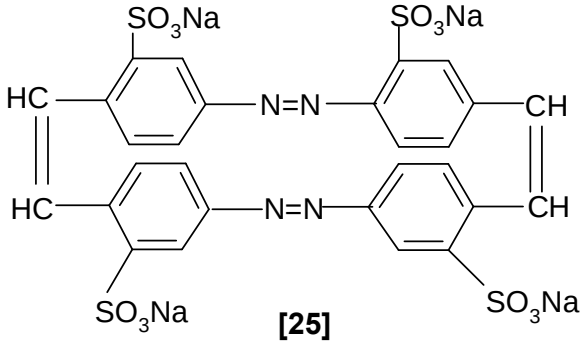
Basic azo dyes contain NH_2 or NH_2 as the auxochrome. A typical example is chrysoidine. The acid azo dyes contain a sulphonic or a carboxylic group. An example is the methyl orange [23] already mentioned above.

Para red [24] is a good example of ingrain azo dyes. Ingrain azo dyes are water-insoluble azo dyes formed in situ on the fibre. The fibre to be dyed with para red is first dipped into an alkaline solution of 2-naphthol, and then immersing it in an ice-cold bath of diazotized p-nitroaniline results in the formation of dye in the fibre:



[24]

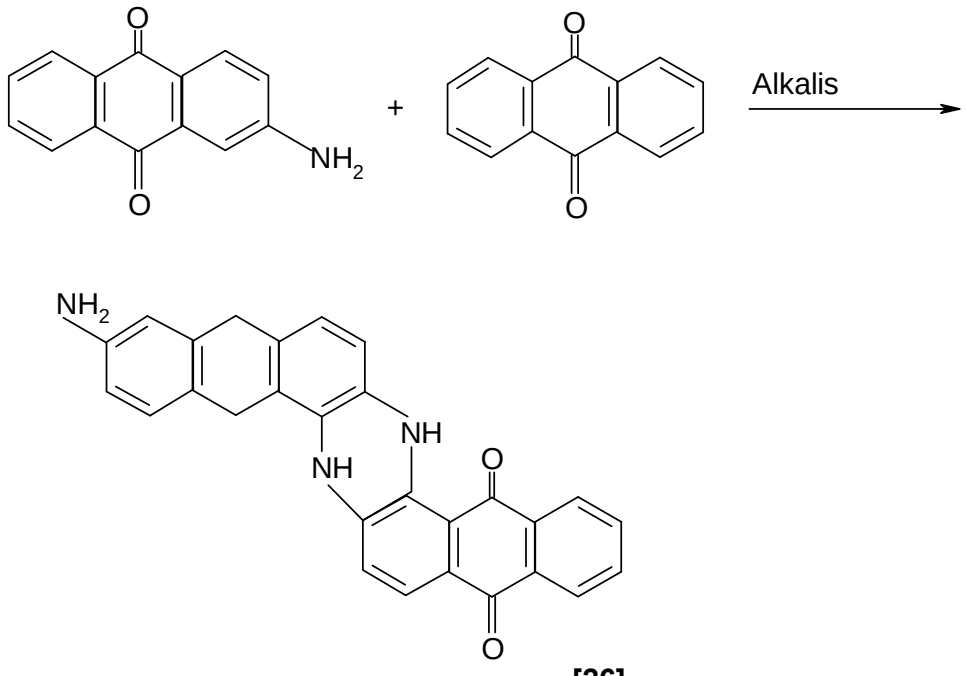
Stilbene azo dyes are direct azo dyes, which are produced without the processes, of diazotisation and coupling. A typical example is sun yellow [25], which are prepared by heating 4-aminotoluene -2-sulphonic acid with sodium hydroxide solution.



[25]

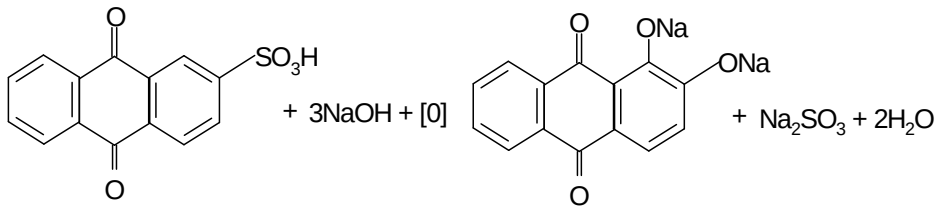
(d) Anthraquinone dyes

Anthraquinone dyes are derived from the parent substance anthraquinone. Auxochromes present in the dyes include amine, hydroxyl, alkyl amino, arylamine and acylamino groups as well as complex heterocyclic system. Dyes derived from anthraquinones are of two types, vat dyes and mordant dyes. The anthraquinone vat dyes are applied to fabrics by a process that depends on the reduction of an insoluble quinone to an alkali soluble hydroquinone and the subsequent oxidation of the hydroquinone to the quinone on the fabrics[61]. Indanthrene blue [26], is the first anthraquinone vat dyes produced, and is prepared by fusing ɓ-aminoanthraquinone with alkali.



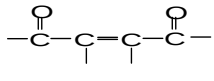
[26]

The anthraquinone mordant dyes are applied to fabrics in the form of a chelate metallic derivative of the dye, which is formed in the fabric. An important member of this group is alizarin [27], the colouring matter of madder root, which is one of the oldest of the natural dyes sources. The alizarin is now produced synthetically by sulphonating anthraquinone with fuming sulphuric acid at high temperatures, and fusing the sodium salts [28], of the product, anthraquinone-2-sulphonic acid, with sodium hydroxide and potassium chlorate at 180-200 ^{0}C under pressure.

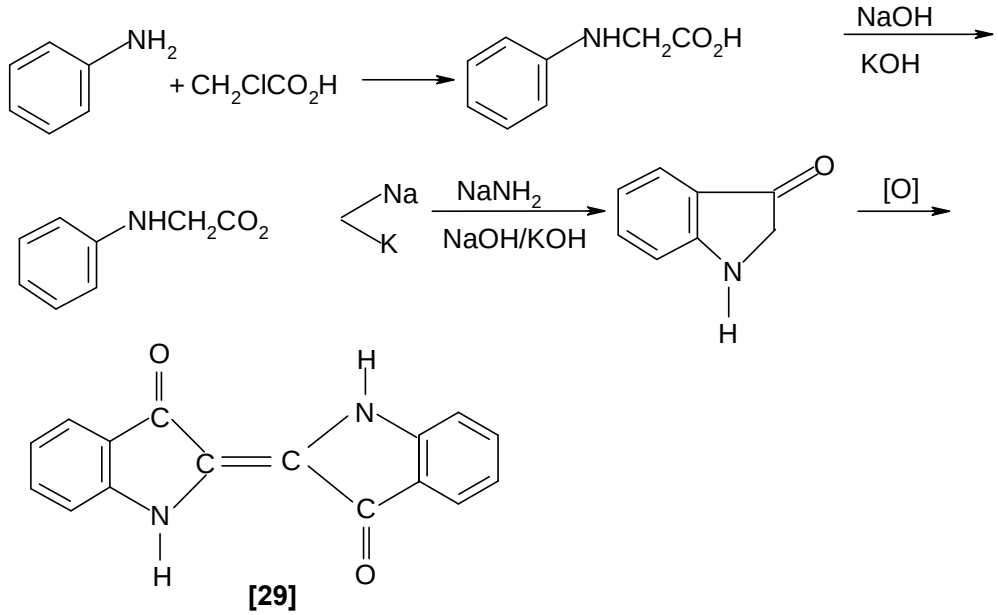


[27] **[28]**

(e) Indigold dyes

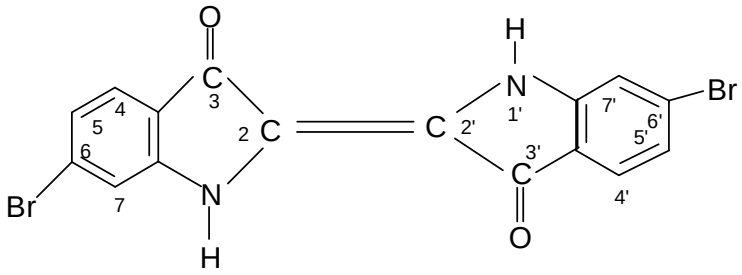
Indigold dyes contain the chromophoric group  , and -NH- group or -S- atoms as the auxochrome. The most important member of this group of dyes is indigo [29], the oldest natural dye derived from the leaf of *Indigofera tinctoria.* Various methods are now available for producing indigo synthetically. In one of such methods[60], aniline is heated with chloroacetic acid and the product, phenyglycine, converted into a mixture of its sodium and potassium hydroxides at

220-240 ^{0}C. The product, indoxyl is converted into indigo (indigotin) by atmospheric oxygen.

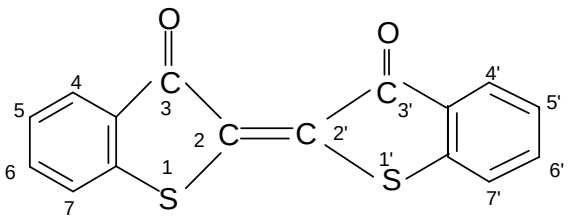


[29]

In addition to indigo, the indigo dyes embrace a large number of derivatives of indigo, such as hydrogenated and nitrated compounds, as well as many, which contain sulphur in place of imino group. The dye Tyrian purple [30], (6,6-dibromo indigo) obtained from the purple snail-Murex bandris is a derivative of indigo. Another example of indigo dye is thioindigo red [31].



[30]



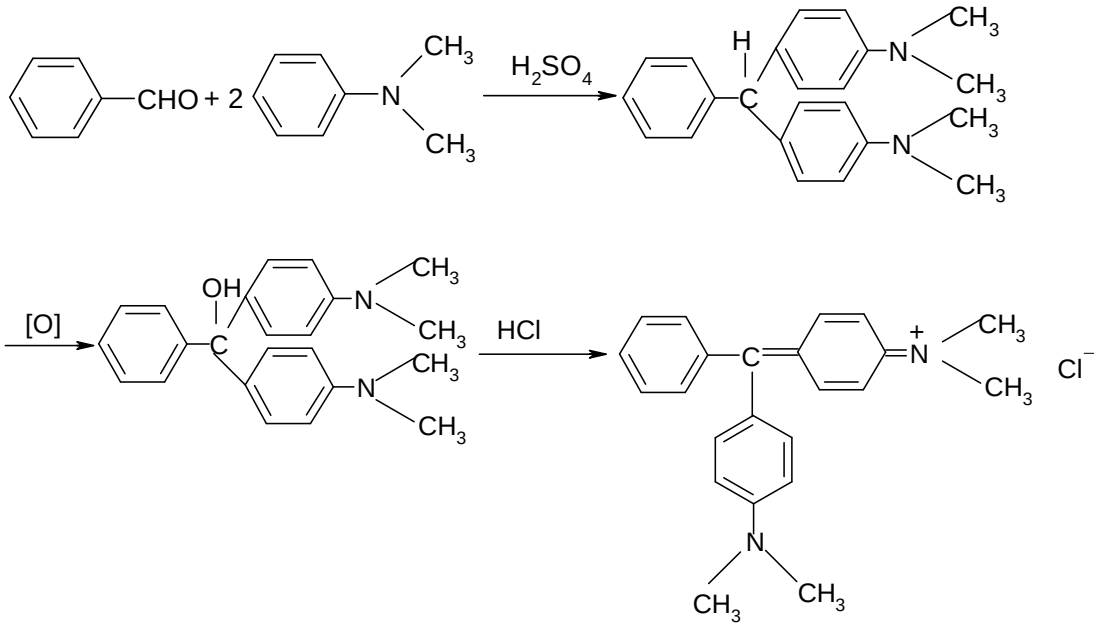
[31]

All indigo dyes are vat dyes, which are applied to the fibre by reducing the insoluble dye, in alkaline medium, with sodium hydrosulphite to water-solution ñleucoò compound. On exposure to air, the insoluble form of the dye is generated within the fibre by oxidation.

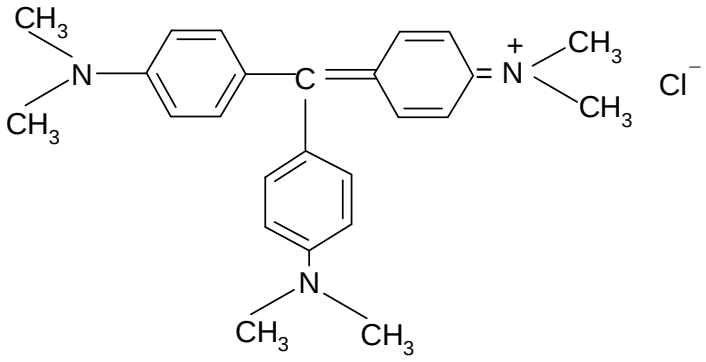
(f) Triphenylmethane dyes: In triphenylmethane dyes, three phenyl groups are joined to one carbon atom, one of the phenyl groups being in the quinoid form. Thus, the chromophoric group of this class of dyes is obtained by the introduction of NH_2, NR_2 or OH groups into the rings of triphenylmethane. The resulting compounds are colourless (the leuco-compounds) and these, on oxidation are converted into the corresponding tertiary alcohols, (the colour bases) which easily change from the colourless benzoic forms to the quinoid dyes in the presence of acid due to salt formation. The dyes are easily reconverted to the leuco-base. A typical example of triphenyl methane dyes is malachite green, which is prepared by condensing dimethylaniline with benzaldehyde at 100 ^{0}C in the presence of concentrated sulphuric acid. The leuco base formed is

oxidized with lead dioxide in a solution of acetic acid containing hydrochloric acid.

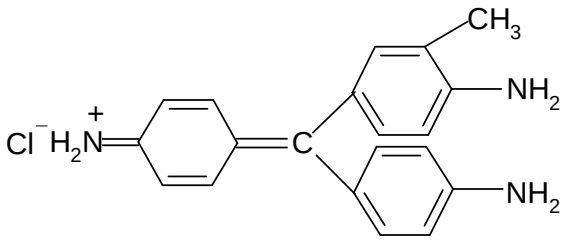
The resulting colour base gives malachite green:



Other members of this group include crystal violet [32] and rosaniline [33] (meganta or fuchsine).



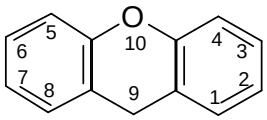
[32]



[33]

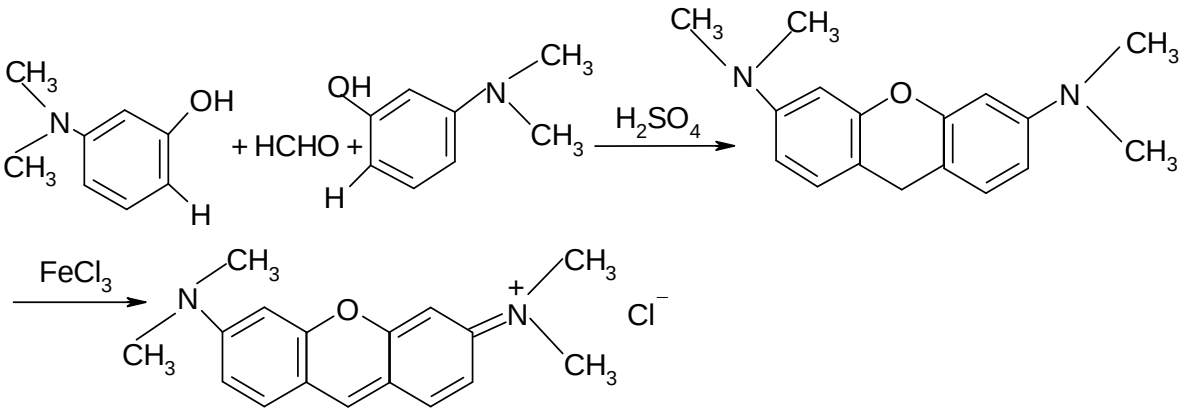
(g) **Xanthene dyes**:

Xanthene dyes are dyes derived from xanthenes [34], (dibenzo-1,4-pyran) by the introduction of auxochromes into positions 3 and 6. The properties of individual members depend largely on the substituents on the ring system.



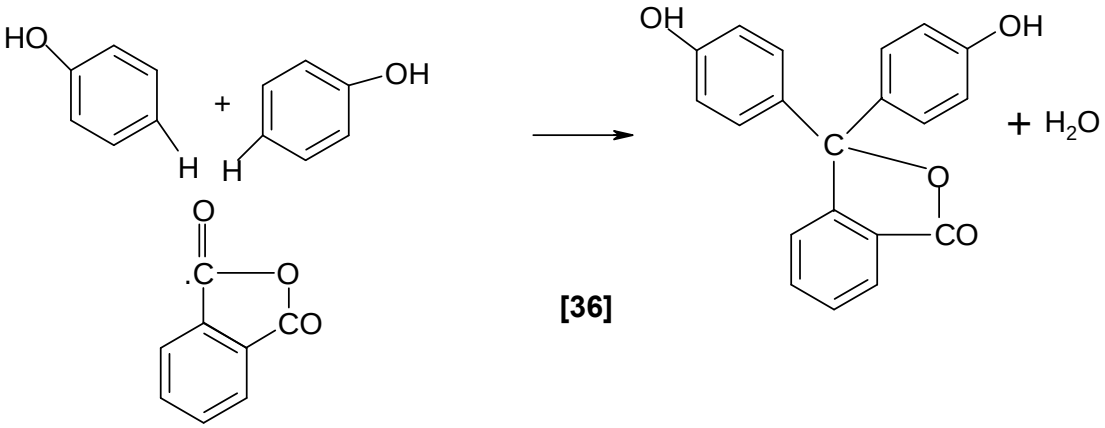
[34]

An example of the xanthene dyes is pyronine G [35], which is prepared by condensing formaldehyde with m-dimethylaminophenol in the presence of concentrated sulphuric acid. The leuco compound formed is oxidized with ferric chloride to the dye.



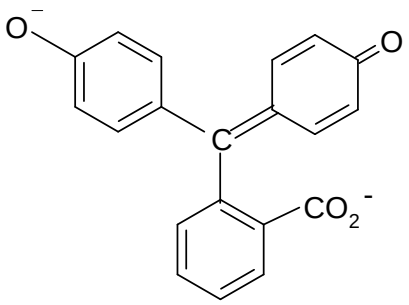
[35]

Phthalein dyes are a sub-group of the xanthene dyes produced by reacting phthalic anhydride with phenols. Phenolphthalein [36], used as an indicator is an example of this group of dyes. It is prepared by heating one molecule of phthalic anhydride with two molecules of phenol in the presence of concentrated sulphuric acid:



[36]

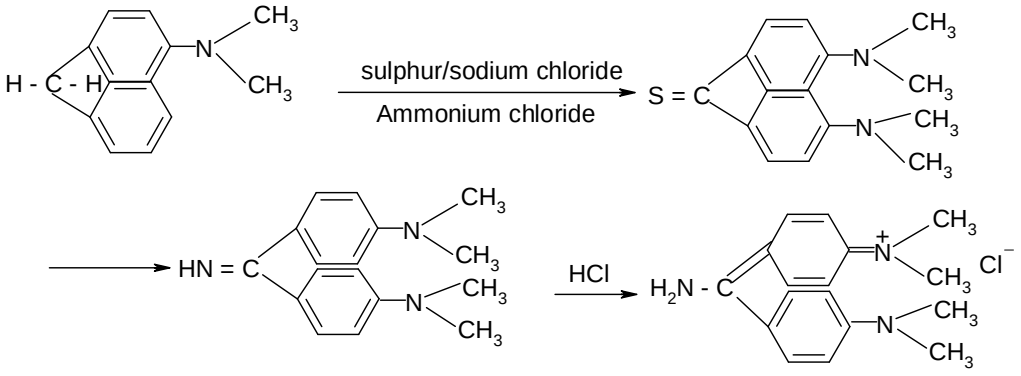
Phenolphthalein is colourless in neutral and acid solutions, even as a solid, but in alkaline solution, it forms a deep red solution.



Deep red

(h) **Diphenylmethane dyes**:

These are basic dyes, which exhibit relatively poor fastness properties. They are characterized by the chromophore $>C=NH$. Auramine [37], is an example of this group of dyes. It is prepared by heating p,p-tetra-methyldiaminodiphenylmethane with sulphur, ammonium chloride and excess sodium chloride in a current of ammonia at about 200 ^{0}C. The auramine base formed, on reaction with hydrochloric acid forms the dye.

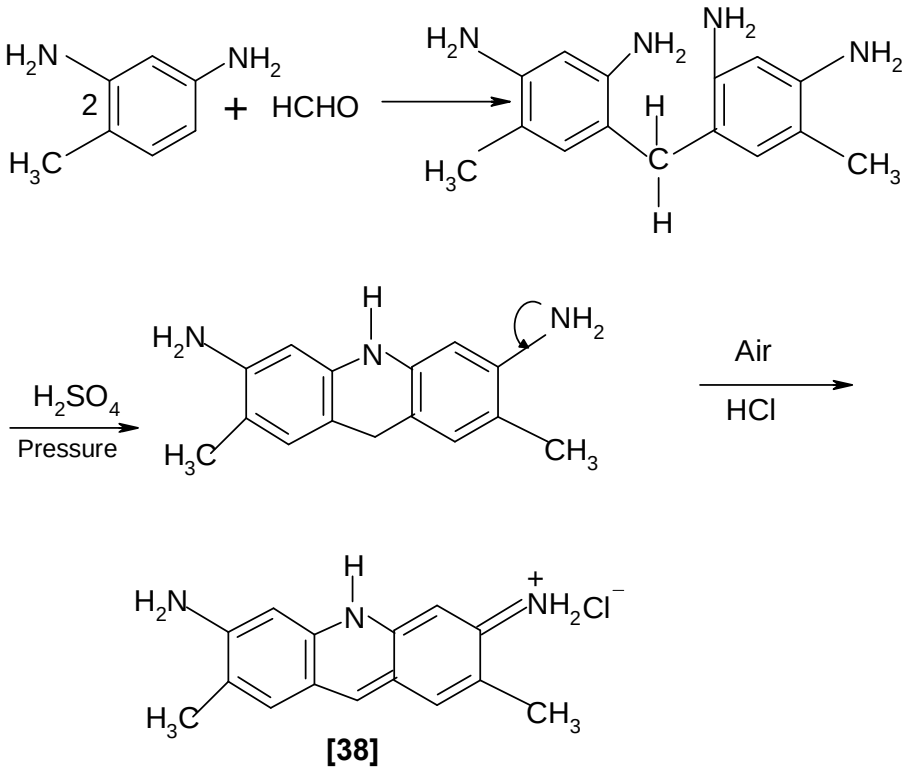


[37]

(i) Heterocyclic dyes:

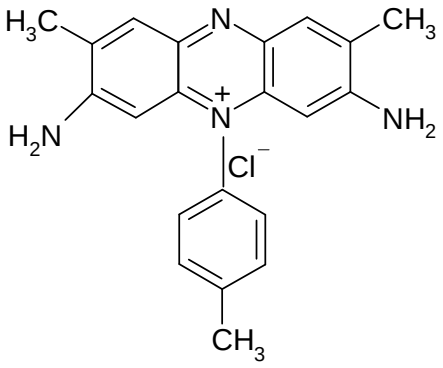
This class of dyes consists of dyes derived from heterocyclic compounds. They include the acridine, azine, thiazine, thiazole, oxazine, and quinoline dyes.

Acridine dyes are derived from acridine system. They are all basic dyes. A typical example of the acridine dyes is acridine yellow [38], prepared from 2,4-diamino toluene and formaldehyde:



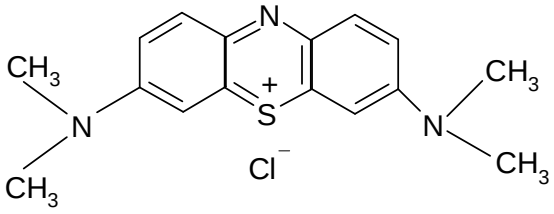
[38]

Azine dyes are derivatives of the heterocyclic phenazine system. They are mostly basic dyes. Perkinôs mauvein[39], is an example of this class of dyes.



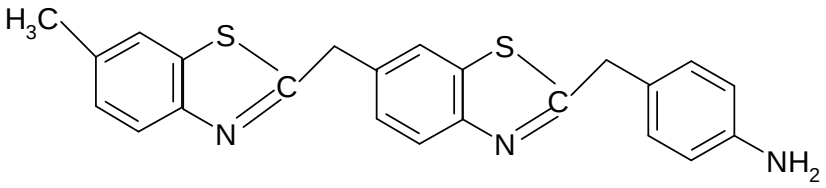
[39]

Thiazine dyes contain the thiazine nuclei, several members of this class are used as stains for tissues in biology and medicine. However, they are of less importance as colouring matter for textile fibres. An example is methylene blue [40], prepared by the oxidation of a mixture of p-aminodimethylaniline and dimethylaniline with potassium dichromate in the presence of sodium thiosulphate and aluminium sulphate.



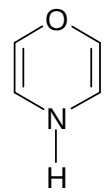
[40]

The thiazole dyes are characterized by the presence of thiazole nucleus. A typical example is primuline base [41].

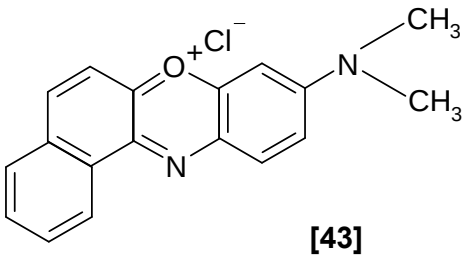


[41]

Oxazine dyes derived from the heterocyclic oxazine [42], system. Example is meldolaôs blue [43], prepared by the reaction of excess p-nitrosodimethylaniline hydrochloride with 2-naphthol in hot methanol solution.

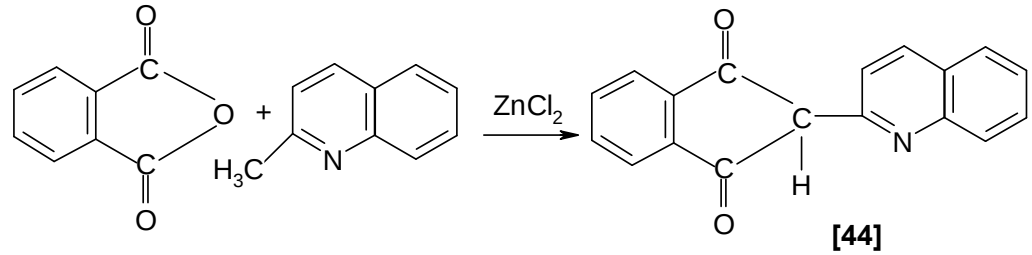


[42]



[43]

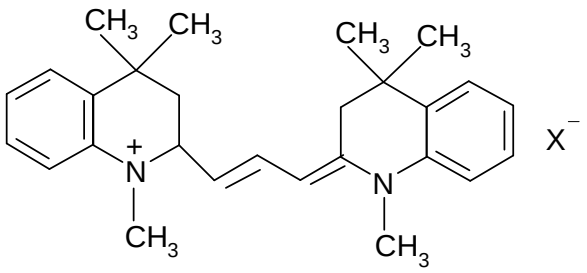
Quinoline dyes contain the quinoline nucleus, and are mainly yellow or red compound. Quinoline yellow [44], is an example of this group of dyes. It is prepared by reacting phthalic anhydride with quinaldine in the presence of zinc chloride.



[44]

Methine or cyarine dyes are characterized by a chain of methine group (-CH =), i.e by a system of conjugated double bonds. The majority of the dyes contain heterocyclic systems such as quinoline, benzothiazole

or trimethylindole. They are formed by linking these heterocycles together by means of a chain of methine groups. They are classified according to the number of methine groups as mono-, di-, tri-, tetra-, e.t.c. methines. An example is Maxilon yellow SG [45].



[45]

2.5 Natural dyes

Natural dyes comprise those colourants (dyes and pigments) that are obtained from animal and vegetable matter without chemical processing[64]. Minerals colourants such as chrome yellow, ultramarine and others are applied as natural pigments mainly in printing or produced by chemical reaction on the petroleum products. Most of the natural colours produce very colourful effects that are so amazing to behold.

Colouring matter extracted from the roots, stems, leaves and flowers of various plants have various exceptions and are also not substantive (have little or no colouring power by themselves) except when used in conjunction with mordants. Beautiful colours

that are created from natural dyes would initially appear vivid, but soon fade[65]. Lack of colour fastness resulted in the discovery of mordants-substances, which aid in the absorption of dyes.

Natural dyes fall into three categories on basis of their origin, which are as follows: plant/vegetable origin, insect/animal origin and mineral origin.

2.5.1 Plant dyes

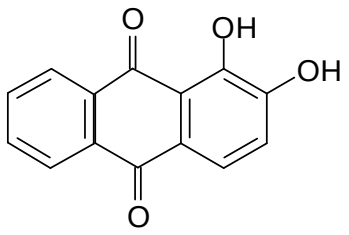
The root, leaf, back, trunk, fruit and flowers of plants are all sources of colouring pigments and dyes. Most natural dyes come from such parts of plants as the bark, flowers, leaves, and roots while the use of seeds, fruits and young shoots are other sources of natural dyes[10,66]. The outer, inner bark and heartwood of trees also produce dyes.

Natural dyes from plants include Madder, Logwood, Indigo, Brazil wood. Other dyes from plants include Lawsone, Juglone, and Saffron e.t.c.

(a) Madder

Madder commonly known as alizarin is a plant that grows in Asia and Europe as a source of bright red dye that is used on fabrics, such as linen and silk[67]. The madder plant (*Rubia tinctorum*) was the source of a brilliant red permanent dye called ñTurkey Red or ñAdrianople Redò which was very well known in 19th century domestic history for ñmadderingò wool and cotton[68]. It was used in the 1700ôs and 1800ôs to

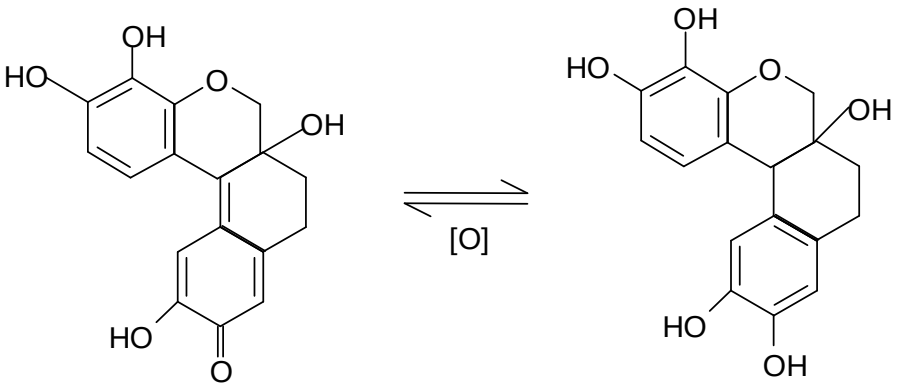
dye the red coat of British solders. Madder was cultivated and first exported from South East Europe and Turkey to the rest of Europe[68]. The wild madder (*Rubia peregrina*) is known to provide a subtler, rose pink dye. The roots of some species of the madder family were also used from the earliest period to produce a whole range of reds since the plant could be grown practically everywhere. Alizarin was the first natural dyestuff to be produced synthetically in 1869. The structure was identified as -1,2-dihydroxylanthraquinone [46].



[46]

(b) Logwood:

Logwood, a natural dye extracted from the pulp of the logwood, which grows in Central America, Mexico and the West India, is known to produce excellent dark colours and bright black and brown dyes for such materials as cotton, fur and silk for special purposes. The dye is obtained by reducing the wood into chips and extracting with boiling water (French process). It can also be obtained by extraction under 103-207 of steam (American process). The extracts are then concentrated under vacuum or evaporated to obtain crystals or solid extracts. The principal colouring bodies of logwood are haematin [47] and haematoxylin [48].



[47] [48]

(c) Indigo

Natural indigo, a dark blue dye derived from the indigo plant, which grows chiefly in India is also used to dye cotton, wool and other fibres. Yang and Narasin (1989) cited indigo as an extremely important source of blue dye that is still very popular in making special types of designs for modern fabrics such as denim.

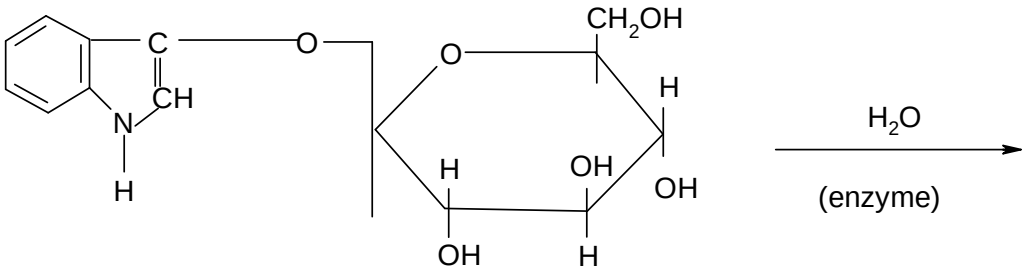
In Japan, the authors indicate that dyers revere indigo and pray to their god, Arizen Myoo, for good fortune in their work with it.

Indigo was the oldest dyes to be used for textile dyeing and printing[69]. India, China, Japan and many Asian countries have used indigo as a dye for centuries. India is believed to be the oldest centre of indigo dyeing in the old world. India was a primary supplier of indigo to Europe as early as the Greco-Roman era. The dye was also known to ancient civilizations in Mesopotamia, Egypt, Greece, Rome, Britain, Peru and Africa.

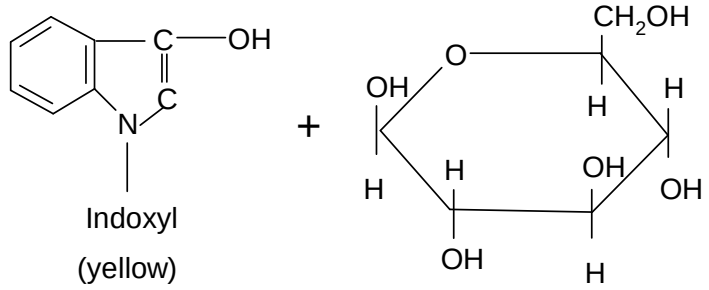
The woad tree is another important source of natural dye[68]. It is also known as Dyerôs Woad (*Isatis tinctoria*), the plant was cultivated as the source of a blue dyestuff for over 2000 years in Europe, and was only superseded around 50 years ago by indigo, a dyestuff that was first extracted from the subtropical indigofera species. Isatis is an ancient name for a healing herb, which ancient warriors painted themselves with before battle for both its psychological effect and as a means of healing the wounds of battle.

In processing woad, the leaves were fermented, dried out, re-fermented, and then rinsed in lime water. Its blue dye was more permanent than indigo. Woad dye was also made from the back of the plant up to the Middle Ages[7].

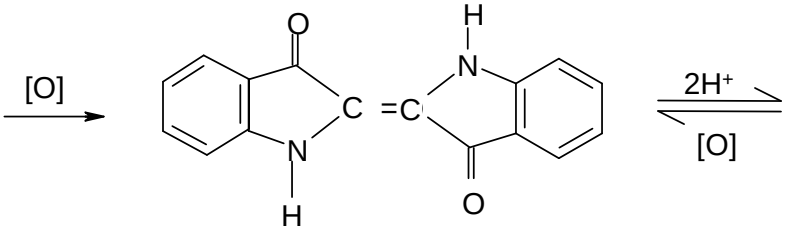
The following reactions occur during the extraction of the plant with water, the glycoside is hydrolyzed by enzymes to glucose and indoxyl, and the later is oxidized by air to the blue dye, indigo [49].



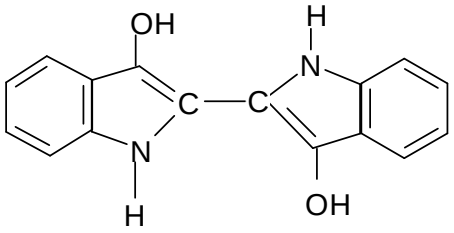
Indican



Glucose



Indigo

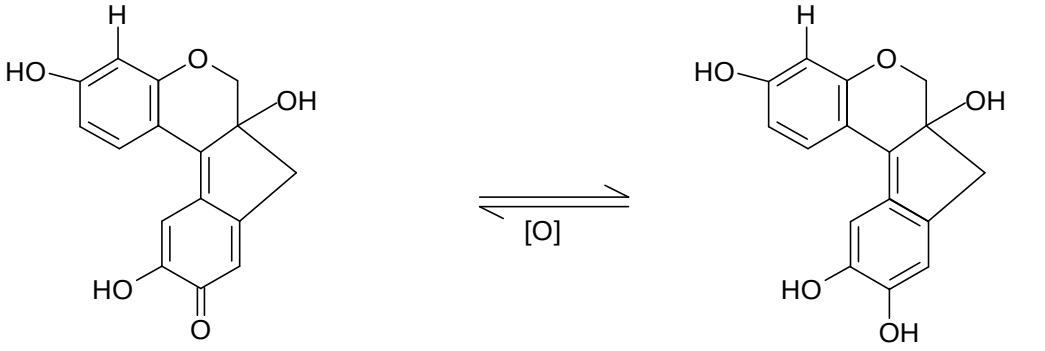


Leuco Indigo

[49]

(d) Brazil wood

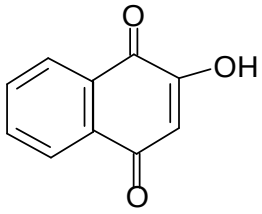
Brazil wood is a red dye obtained from various spices of *Caesalphina* plant. The tree grows mostly in Asia and Central Southern America. It was used as a mordant dye on cotton, wool and silk. The colouring matters of brazil wood are brazilin [50][51].



[50] **[51]**

(e) Lawsone

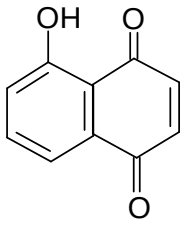
Lawsone is a naphthaquinone orange-brown dye made from a shrub henna, *Lawsonia inermis* of North Africa and the Middle East, was also used to colour leather and dye human hair[10]. The structure of lawsone has been found to be 2-hydroxyl-1,4-naphthaquinone [52].



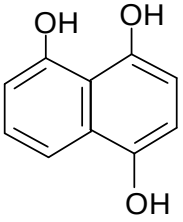
[52]

(f) Juglone

Juglone[53], is another naphthaquinone dye and is similar in structure to lawsone. The dye is found in the shells of unripe walnuts, usually in the colourless leuco form, Ŭ-juglone[54]. To obtain the dye, the shells are ground, fermented for two days and extracted with hot water. During the process, the colourless hydro form undergoes air oxidation to yield the quinine structure that dyes natural fibres a yellow-brown hue.



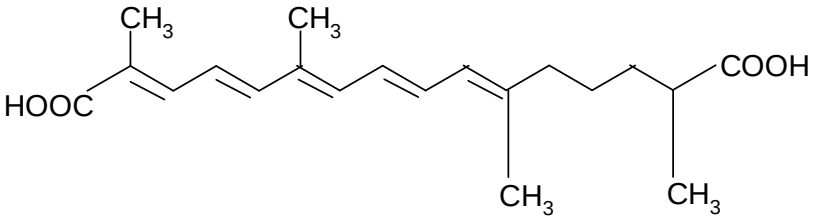
[53]



[54]

(g)Saffron

Saffron is obtained from the pistils of the *Crocus sativus* plant. It was used as a principal yellow dye of the ancients, as well as a spice in cooking, as an ingredient in medicinal formulations, as a perfume in the public baths, and as a mordant on paper with iron. The major colouring matter of saffron is crocin[55].



[55]

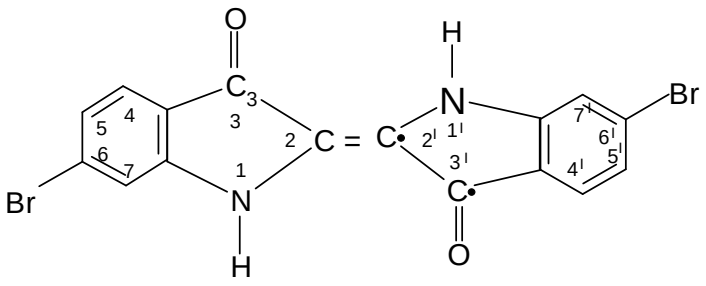
However, there are many other plant dyes but the above named ones deserve special mention. In Nigeria, plant dyes are extracted from a large number of plants like: *Baphia nitida, Cola nitida, Guinea corn husk, Morinda lucida, Daniellia oliveri, Vitex grandifolia* and so on[56].

2.5.2 Animal dyes

Natural substances such as carminia acid, kerosene and laccaic (popularly known as Lac dye) obtained from either exudation or dried bodies of insects namely cochineal and kermes respectively are well known and these acid compounds are used for dyeing purposes from ancient times. These acid compounds particularly carminic and laccaic acids find limited use for colouring food materials/products and cosmetics. Some of the examples of animal dye sources include the urine of cow, the camel dung, shell fish and mollus. Animal dyes include the following: Tyrian purple, cochineal, kermes and lac.

(a)Tyrian purple

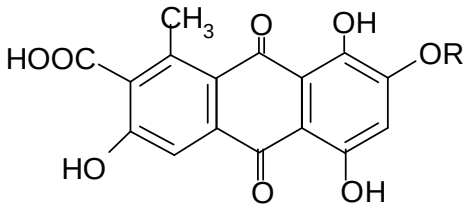
Tyrian purple or crimson is an animal dye obtained from the bodies of some types of marine snails.The dye was use as emblem of royalty and only by the Roman noble men in the ancient days[68]. It is obtained by the air oxidation of a colourless fluid expressed from the glands of the snail. The dye is the dibromo derivative of indigo, -6,6-dibromoindigo[56].



[**56]**

(b)Cochineal

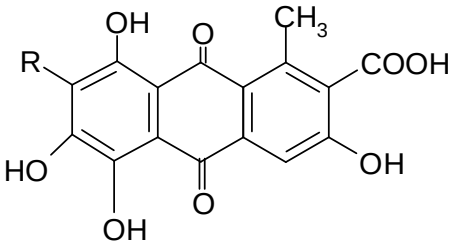
Cochineal known as Red animal dye is derived from certain species of tiny scale insects that fed on red cactus berries. The dyes were highly valued from ancient times and right through the Middle Ages. The principal colouring matter in cochineal dye is carminic acid [57]. The major use of the dye includes on tin mordanted silk. Meanwhile, it is being used in the food industry[58].



[57]

(c)Kermes:

Kermes is obtained from an oriental shield louse, particularly the female *Coccus ilices*. It is highly soluble in water but very soluble in hot water. It gives a brilliant scarlet colour when dyed on alum-mordanted fabric. The principal colouring matter in kermes dye is kerminic acid [58].

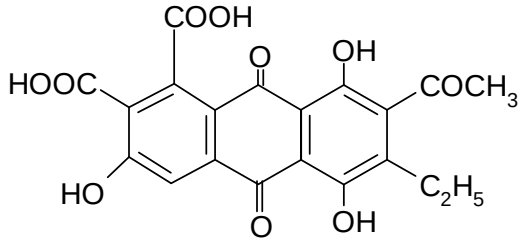


[58]

(d)Lac

This is the oldest of all animal dye. It has been used in South East Asia and India since the beginning of history. The dye is obtained from the gum lac or viscous fluid made by the insect *Coccus laccae* which bore into the bark of trees and become enclosed in an exuding juice, which hardens into a resin.

The principal colouring matter is laccaic acid [59].



[59]

2.5.3 Mineral dyes

Mineral dyes are derived from coloured clays and earth oxides.

Table 2.2 below shows the summary of mineral dyes and sources

Mineral dyes	Sources
Chrome Green	From a compound of chromium and oxygen
Chrome Red	From a compound of chromium and lead
Chrome Yellow	From a compound of chromic acid and lead
Prussian Blue	From a compound of iron and cyanide

2.6 Uses of dyes

Dyes have important and prominent uses apart from their function in colouring fabrics and other articles of daily use. In general, dyes are used in the following ways:

Textile: Dyes are used in dyeing both natural and synthetic fibres. They are also used in printing of colours on the fibres. The textile industry uses the largest amount of dyestuffs.

Paper: substantial quantities of dye are consumed by the paper industry for dyeing and pigmentation of the paper. Dyes are also applied in the coating use for duplicating paper. The most important paper dyes are selected from the direct, acid and basic groups of dyes.

Leather: Dyes are used in the colouration of leather. Natural leather is a protein substance that can dye with acid dyes among others. Finished leather also can be dyed or pigmented, the choice of colour

type depending on the nature of the finish. Surface coating of the leather is widely practiced due to closely-knit structure of the leather.

Food: Certain relatively non-toxic dyes are used to colour food products in order to enhance their appearance. Such foods include candy, cake, ice cream, butter and margarine[70].

Oils: Dyes are used to colour petrol, kerosene, diesel and lubricating oils in order to improve the appearance, as well as for identification of grade, type of use, or merely as a trademark of the manufacturer. Waxes, shoe polishes and candles are also coloured. Dyes for these purposes are selected on the basis of solubility as well as hue and fastness.

Plastics: Dyes are also used in colouration of plastics. Many plastics are sold by virtue of the aesthetic appeal of their colour. It is for example, the colour of the plastic washing-up bowl, which accounts in large measure for the fact that it is superseding metal basins in the home.

Wood: Dyes are utilized in dyeing or tinting of wood. The dyes are applied in water-alcohol, or other solvents, which evaporate after the wood has been painted with or soaked in the dye solution.

Biological Samples: The biologist employs dyes as stains for tissues and bacteria being prepared for examination under the microscope. The use of dyes in microscopic work was first reported in 1838.

Indicators: The chemist uses many dyes as indicator in analytical work. Such dyes change colour under conditions of abrupt changes in pH, or hydrogen ion concentration. Both phenolphthalein and methyl orange exhibit this change and are indicators.

Medicine: Several dyes are used in medicine as antiseptics and as remedies for specific diseases. Crystal violet is used as an external application for Staphylococcal infections and, internally, for treatment of threadworm. Brilliant green is also used externally for its antiseptic properties. Triple dye solution consisting of an aqueous solution of crystal violet, brilliant green, and acriflavine has been used for the treatment of burns and for stain sterilization[63].

Photography: Some dyes are used in photography to increase the sensitivity of the photographic film in the infrared region of the spectrum.

Pigment: Certain organic dyes are used as pigment for paints, lacquers, and printing inks. Indigo dyes serve such purpose. Copper phthalocyanine and the chlorinated derivatives are also used in pigment printing, in printing inks, artistic colours, paints and lacquers.

Miscellaneous: Other uses of dyes include in the colouration of soaps, synthetic detergent, cosmetics, hair, fur, metals, anodized aluminium, etc. During World War II various dyes were used to produce coloured signal

smokes for military identification, anthraquinone dyes are useful for this purpose.

2.7 Dyeing

Dyeing is the art of applying colouring matter to a substrate in such way that the colour appears to be a property of the substrate not readily removed by rubbing, washing or exposure to light[62]. The term dyeing is usually confined to the application of colours to textile and such related materials as furs, leather, plastic, and paper, and is used to differentiate between other colouration processes such as printing or staining. Printing and painting involves the application of coloured pigments in a vehicle to a textile or paper surface, while in staining, small amounts of colour are held on the surface of the substrate. True dyeing implies an interaction of the dye with the principles, which are responsible for bringing about a permanent union between the material to be dyed and the colouring matter applied[62].

2.7.1 Mechanism of dyeing

Natural dyes work best with natural fibres such as cotton, linen, wool, jute and sisal[70]. Among this, wool is by far the easiest to take up dyes followed by cotton, linen, silk, and then the coarse fibres such as sisal and jute. Nearly every plant will yield some colour whether we use leaves, bark, wood, roots or fruits. Nearly all require or are enhanced by

some sort of mordant. The means then is to determine which parts or which part of the plants will give not only beautiful tones but colourfast shades as well. A colouring material that has the strength to bind itself to a fibre and remain there by staining the fibre is considered the best.

So natural dyes can neither dye animal nor vegetable fibres directly but require a mordant, which depends upon the nature of the dye. If the dye is acidic, the mordant must be basic (the most common basic mordants are the salts of Cr, Al, Sn, Fe) on the other hand if the dye is basic the mordant must be acidic (the most common acidic mordant is tannin or tannic acid containing some amount of tartar emetic). The fabric to be dyed is first soaked into a solution of the metallic salt (i.e. mordant) and then steamed or other wise treated to form the insoluble metallic hydroxide. The fibre so obtained is known as mordant fibre. It is dried then placed in a solution of the dye when the latter is held by the hydroxide of the metal on the fibre by means of chelation. Such chelated complexes can be formed only when the resulting dye has a five or six membered ring which is again possible only when -OH group in the dyestuff is present ortho to one of the following groups. The chemistry of binding of dyes to fibres is complex. It involves direct bonding H-bonds hydrophobic interactions. Mordants to this effect increase binding of dye to fabric by forming a chemical bridge from dye to fibre.

The mordant has affinity for both fibre and the dye. Thus, those dyes, which do not have any affinity for a fibre, can be applied by using mordants. This improves the staining ability of any dye along the increase in fastness properties. A mordant forms an insoluble compound of the dye within the fibre. The mordant dyes include those differing widely in the origin but those form insoluble compounds with metal salts. Presence of certain functional groups in suitable position in the dyes molecule helps in coordination of the metal salt. Generally, either two-hydroxyl groups ortho to each other or one-hydroxyl group ortho to carbonyl, nitroso or azo groups are the main features of mordant dyes. They produce a wide range of hues of remarkable resistance to wet treatments but the shades lack brilliancy.

A chromium atom can combine with alizarin by covalency and co-ordinate valency to form the lake. In the first step, chromium combines with the hydroxyl groups in the same way as sodium combines with phenol to form sodium phenate. In the subsequent step, the oxygen atom of the adjacent quinone group donates a lone pair of electrons to the chromium atom and forms a co-ordinate bond. Chromium being trivalent combines with three molecules of alizarin.

2.7.2 Natural dyeing principles

Application of natural dyes in today scenario makes use of modern science and technology not only to revive the traditional technique but

also to improve its rate of production, cost effective and consistency in shades. It therefore, requires some special measures to ensure even-ness in dyeing. Many factors have to be accounted for when one works with natural dyes. They are as follows:

(a) Nature of material to be dyed

Animal proteins like wool and silk dye best in acidic conditions and are weakened by alkaline. If an animal protein is dyed in alkaline conditions, it is best to end with a diluted vinegar rinse to restore a slightly acidic pH to the fibres before they dry. Plant materials like cotton and flax dye best in alkaline (basic) conditions and are weakened by acids. If cotton is dyed in acidic conditions, it is best to end with a weak washing soda bath to restore the fibres to slightly alkaline before they dry.

(b) Measurement of mordants and dyestuffs

Most dyeing procedures specify ingredients by weight rather than measure. Recipes will also specify the amount of fibre to be dyed or the other ingredients will be expressed as a ratio to fibre weight. This is because the amount of water in the dye bath will not affect how strongly the fibre takes colour but the amount of dye in the dye bath does. Therefore, if one gram of fibre has to be dyed with one gram of dyestuff and then one wants to reproduce the same colour on 5 more grams of fibre, the amount of dyestuff should be multiplied by 5 times as well. The

water should always be enough to let the fibres move around freely, water quantity should be sufficient to dip the fabric or fibre properly.

(c)Temperature

Different dyes work better at different temperatures. Most plant dyes benefit from being heated but some (e.g. madder) change colours if allowed to boil. Sappan wood also has a tendency to change colour when heated for prolonged hours. Some dyes work best at lower temperatures (safflower and woad or indigo).

2.8 Fastness properties

A substance, which is resistance to light, water and soap is called dye. Therefore, it is a fundamental requirement that coloured textile should withstand the conditions encountered during processing, following coloration and their subsequent useful life. When a coloured textile is subjected to particular conditions e.g. light or washing one or more of several things may happen. As far as the colour of the material is concerned, there may be alteration in hue value or intensity.

In certain cases, there may be alteration in all three. Thus, a red material may become pale, yellowish and duller ï further under certain conditions e.g. during washing adjacent white material may become coloured and coloured material may acquire new colour due to the transfer of dye from the original dyed material.

The colour fastness of a coloured textile is therefore, defined as its resistance to these changes when subjected to a particular set of conditions. It follows that colour fastness must be specified in terms of the changes and expressed in terms of their magnitude.

2.9 Colour in textile industry

Dyes, also referred to as dyestuffs, are the most common way to add colour to fibres, yarns and fabrics. Manufacturers of petroleum products, textiles, food, fur, ink, leather, paper, plastics and wood also use dyes to colour their products[10]. The appeal of colour is universal. Colour is one of the most significant factors in the appeal and marketability of textile products[71].

Colour is a very important aspect of textile production because consumers generally expect two things: aesthetically pleasing colours and prints and colour performance, implying that the aesthetic aspects of a textile fabric is the prime consideration for consumers[72].

Colour is often the primary consideration in the selection and purchase of clothing and other house hold textiles and largely, the most exciting thing about textile. Colour is everywhere, in the interior of a dwelling, natural and stained wood, wall papers, upholstery fabrics, pottery, paintings, plants and flowers[73].

2.10 Colour in petroleum industry

Petroleum is a naturally occurring complex mixture made up predominantly of hydrocarbons and other compounds of carbon and hydrogen frequently containing less amounts of nitrogen, sulphur, and oxygen as well as smaller amounts of nickel, vanadium, and other elements[74]. It can occur in solid, liquid, or gaseous form as asphalt, crude oil or natural gas respectively.

Petroleum products derived from crude oil by process of refining are a convenient source of energy. They are especially used as transportation fuels, for examples in automobiles and air planes. Other refinery products include lubricants, wax, asphalt, solvents and specialities such as hydraulic fluids and others. The various fuels, and chemical markets have their own products requirements and it is the function of petroleum refineries to separate crude and other raw materials into fractions that they processed to meet product specifications.

In order to meet the products specification, many petroleum products of commerce are blended compounds or otherwise modified from primary stocks as they come from the refinery units. One of the blending and modification processes involves dyeing. Many petroleum products are dyed or coloured in other to improve the appearances as well as to identify the grade or type. Some are also dyed as a trademark.

Petroleum products that are dyed include petrol, diesel, kerosene, lubricating oil, hydraulic fluids etc[15,16].

NNPC (Nigeria National Petroleum Co-operation) for example uses a dye commonly known as ñredò dye for colouration of petrol. This dye gives the petrol dark red colouration and is mostly used for identification.

CHAPTER THREE

3.0 Materials and Methods

3.1 Reagents

- *n*-hexane
- Dichloromethane
- Ethyl acetate
- Acetone
- Benzene
- Methanol
- Ethanol
- Distilled water

All the chemicals used were of analytical reagent grade and were made by Sigma Aldrich® (Germany) while distilled water was purchased from the National Center for Energy Research and Development (NCERD) University of Nigeria, Nsukka.

3.2 Equipment

- Buckner funnel
- Buckner flask (500 cm^3)
- Filter paper (Whatmann, 11 cm)
- Electric Milling Machine made by Thomas Wiley Laboratory Mill Model 4 (U.S.A)

- Rotary Evaporator (BÜCHI ROTAVAPOR ï R, Germany).
- Edward High Vacuum Pump Machine (England)
- Freeze Drier Machine (YORCO, India)
- Conical flask (1000 cm^3)
- Mortar
- Glass funnel
- Glass beaker (1000 and 250 cm^3 respectively)
- Shunsum flask
- Measuring cylinder (500 and 250 cm^3 respectively)
- Spatula
- High sensitive analytical weighing balance (OHAUS)
- FTIR ï 8400S Fourtier Transform Infrared Spectrophotometer (SHIMADZU, Japan)
- Ultraviolet Visible Spectrophotometer (UV-2500PC series, Japan)
- Lovibond Tintometer F Model (United Kingdom)

3.3 Plant materials

The heartwood of *Pterocarpus osun* Craib and young leaf of *Lawsonia inermis* Linn. were collected from Eha-Alumona and Nsukka both in Nsukka L.G.A in Enugu State, Nigeria. The plants materials were identified by Dr. A. O. Ozioko of INTERCEED and the specimen was

submitted through voucher No Hï 1070 to the herbarium of the Institute and kept for future reference.

3.4 Extraction procedure

The heartwood of *Pterocarpus osun* Craib and young leaf of *Lawonia inermis* Linn. were washed with distilled water to remove dirt and air-dried before milling into powder with electric milling machine which has a sieving size of 1mm according to the method reported in the literature[75].

About six hundred gram (600 g) each of the plant materials were soaked in 2.5 L of 96 % ethanol and methanol respectively and extracted by cold maceration at room temperature for 48 hours. The filtrates were concentrated *in vacuo* using rotary evaporator at reduced pressure (300 Psi) and temperature (40 ^{o}C) to obtain the dry extract.

3.5 Purification of the extracts

The ethanol extract of *P. osun* was purified further using column chromatography (CC) method as previously reported in the literature[76] while the extract of *L. inermis* was purified using vacuum liquid chromatography (VLC) according to the method reported in the literature[77].

3.5.1 Column chromatographic purification of *P. osun* extract

About twenty seven gram (27.0 g) of the extract was chromatographed on silica gel (60ï 230 mesh, 250 g) packed into a glass column (15 cm long x 1.5 cm, i.d). The elution was done with ethanol (2.5 L) and the percentage yield calculated.

3.5.2 Vacuum liquid chromatographic purification of *L. inermis* extract

The methanol extract of *L. inermis* Linn. was purified using vacuum liquid chromatography (VLC). Thirty five grams (35.0 g) of the extract was purified by VLC using silica gel (230ï 400 mesh, 30 cm long x 3.0 cm i.d, 550 g) as the stationary phase and eluted with a gradient of ethyl acetate in ethanol (10:0, 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, 1:9 and 0:10, each 500 cm^3) to afford eleven sub fractions (LI_1 ï LI_{11}). LI_7 was freeze-dried and the percentage yield calculated.

3.6 Solubility of the dyes in organic solvents

The dyes (0.01 g, each) were separately dissolved in 2 cm^3 each of *n*-hexane, dichloromethane, ethyl acetate, acetone, benzene, methanol, ethanol and water in a sample bottle and was used to determined solubility of the dyes.

3.7 De-colourization of petroleum products

The de-colourization of the petroleum products used was done using activated charcoal and fullerôs earth according to the method reported in the literature[78]. The petrol, kerosene and diesel (1 L, each) were decolourised using 50 g, each of activated charcoal and fullerôs earth on Edward High Vacuum Pump. The colourless petroleum products obtained were collected in 500 cm^3 of Buckner flask and stored properly in sample bottles for analysis.

3.8 Spectroscopic analysis of the dyes

The spectroscopic analysis of the dyes was done using ultra-violet spectroscopy (UV) and infrared spectroscopy (IR).

3.8.1 Ultraviolent spectroscopy (UV)

The dyes (1.0 g, each) were separately dissolved in 3 cm^3 each of petrol, kerosene and diesel in a sample bottle. The petroleum products were used for the UV analysis for periods of twenty-eight days. The UV analysis was done on UV 2500PC spectrophotometer.

3.8.2 Infrared spectroscopy (IR)

The dyes (0.001 g, each) were mixed with NaCl and KBr disc respectively. Fourier Transform Infrared (FTIR) instrument, 8400S spectrophotometer was used to establish the functional groups present.

3.9 Colour stability analysis of the dyes

The stability of the colour of the dyes obtained was done using Lovibond Tintometer according to ASTM D 1500 method[79]. The dyes (0.001 g, each) were separately dissolved in 2 cm^3 each of petrol, kerosene and diesel in a sample bottle. They were used for the colour intensity analysis for a period of twenty-eight days.

CHAPTER FOUR

4.0 Results and Discussion

4.1 Physical data of the dyes

The physical properties of the dyes extracted from *P.osun* Craib and *L. inermis* Linn. are presented in Table 4.1 below:

Table 4.1: Physical data of the dyes

Dyes	**Solvents used for extraction**	**Physical colour of dye**	**Melting point (^{o}C)**	**Yields (%)**
***Pterocarpus osun* Craib**	96% ethanol	Dark red	130ï 132	**1.44**
***Lawsonia inermis* Linn.**	96% methanol	Orange brown	190-191	**1.50**

4.1.1 Solubility test of the dyes

The solubility of the dyes in different organic solvents shows that the dyes are very soluble in ethyl acetate, methanol and ethanol while they are sparingly soluble or insoluble in the other solvents as presented below in Table 4.1.1

Table 4.1.1: Solubility test of the dyes

Dyes	Solvents							
	***n*-hexane**	**Dichloromethane**	**Ethyl acetate**	**Acetone**	**Benzene**	**Methanol**	**Ethanol**	**Water**
***Pterocarpus osun* Craib**	-	-	+	-	-	+	+	-
***Lawsonia inermis* Linn.**	-	*	+	+	*	+	+	*

Footnote: + = soluble, * = sparingly, - = insoluble.

4.1.2 *P.osun* dye wavelengths of maximum absorption in petroleum products (petrol, kerosene and diesel) for a period of twenty-eight days were shown in Table 4.1.2-4.1.4 and presented graphically in Figure 4.1-4.1.2 respectively.

Table 4.1.2: *P.osun* dye wavelengths of maximum absorption (λ_{max}) in Petrol

Time (Days)	λ_{max}
1	753
4	667
8	777
12	740
16	668
20	768
24	747
28	669

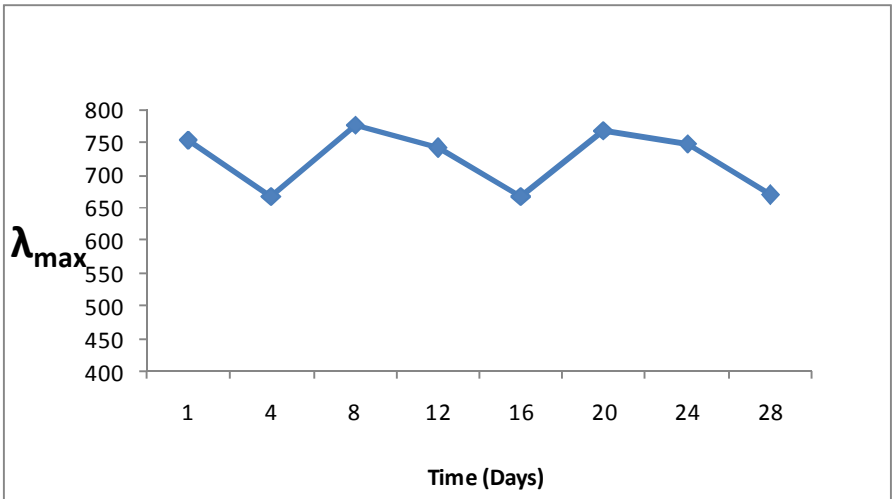


Figure 4.1: Variation of λ_{max} with time using petrol coloured with *P. osun* dye

Table 4.1.3: *P.osun* dye wavelengths of maximum absorption (λ_{max}) in Kerosene

Time (Days)	**λ_{max}**
1	670
4	776
8	670
12	670
16	670
20	670
24	694
28	670

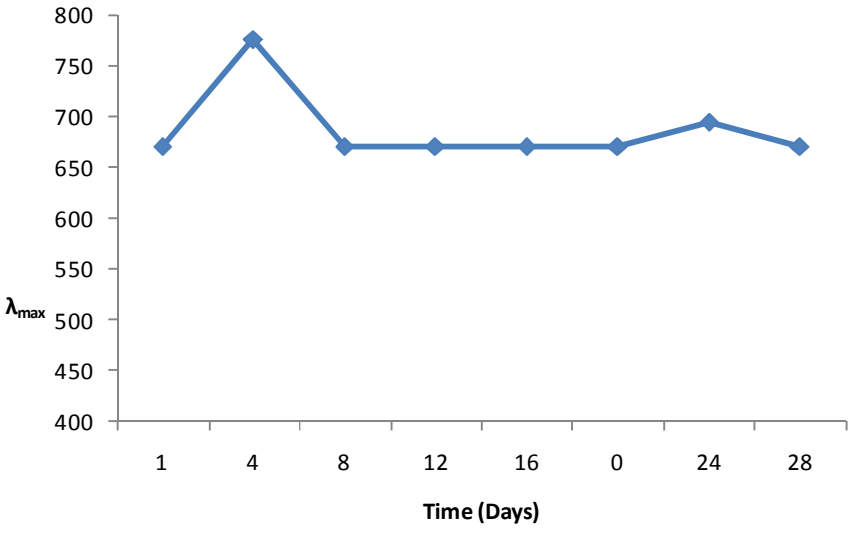


Figure 4.1.1: Variation of λ_{max} with time using kerosene coloured with *P. osun* dye

Table 4.1.4: *P.osun* dye wavelengths of maximum absorption (λ_{max}) in Diesel

Time (Days)	λ_{max}
1	671
4	670
8	669
12	670
16	757
20	761
24	750
28	671

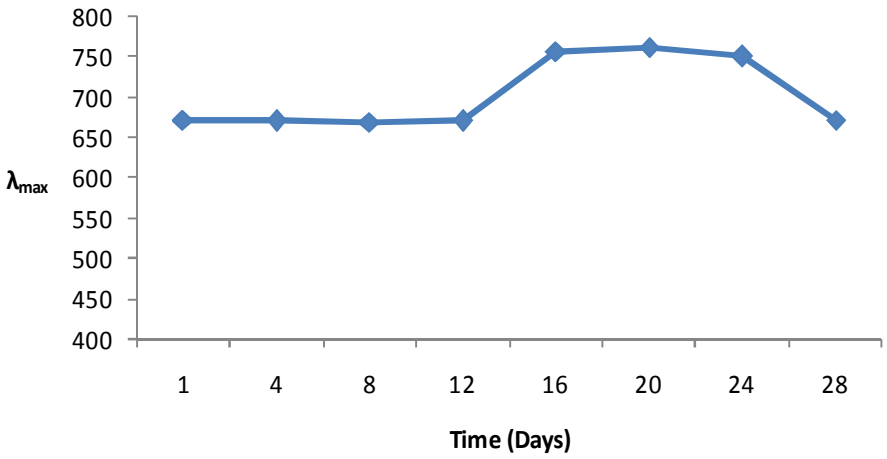


Figure 4.1.2: Variation of λ_{max} with time using diesel coloured with *P. osun* dye

4.1.3 *L. inermis* dye wavelengths of maximum absorption in petroleum products (petrol, kerosene and diesel) for the same periods were given in Table 4.1.5-4.1.7 and presented graphically in Figure 4.1.3-4.1.5 respectively.

Table 4.1.5: *L.inermis* dye wavelengths of maximum absorption (λ_{max}) in Petrol

Time (Days)	λ_{max}
1	669
4	669
8	669
12	669
16	669
20	669
24	669
28	669

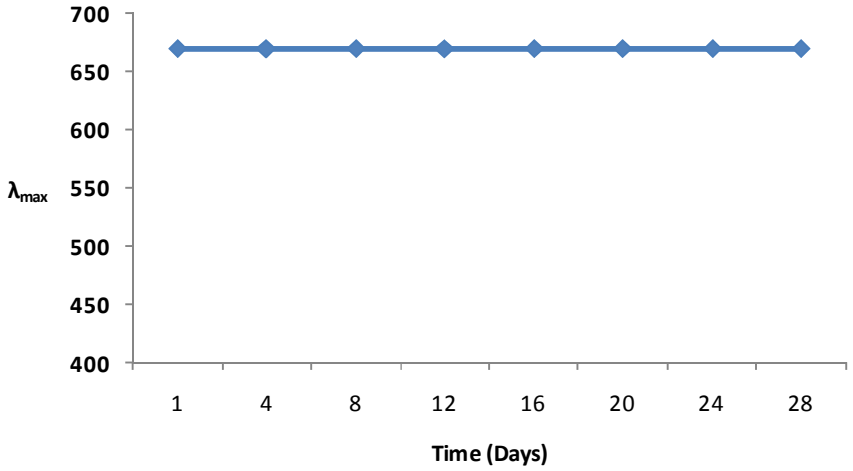


Figure 4.1.3: Variation of λ_{max} with time using petrol coloured with *L. inermis* dye

Table 4.1.6: *L. inermis* dye wavelengths of maximum absorption (λ_{max}) in Kerosene

Time (Days)	**λ_{max}**
1	777
4	748
8	742
12	723
16	670
20	746
24	777
28	670

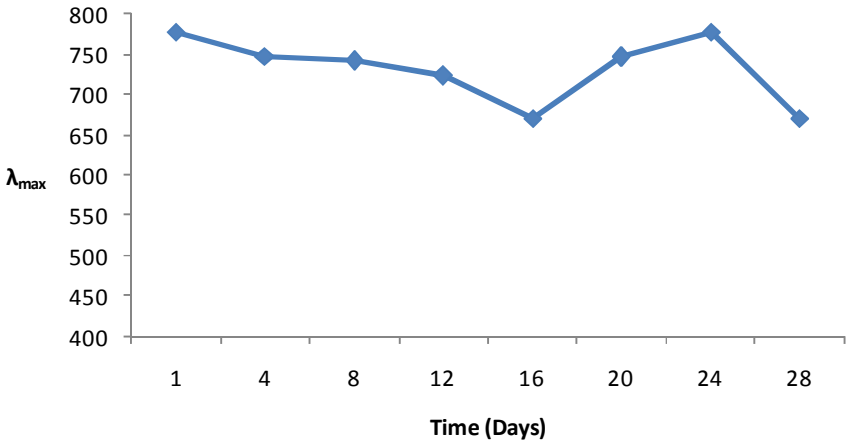


Figure 4.1.4: Variation of λ_{max} with time using kerosene coloured with *L. inermis* dye

Table 4.1.7: *L.inermis* dye wavelengths of maximum absorption (λ_{max}) in Diesel

Time (Days)	λ_{max}
1	755
4	643
8	671
12	671
16	671
20	784
24	671
28	670

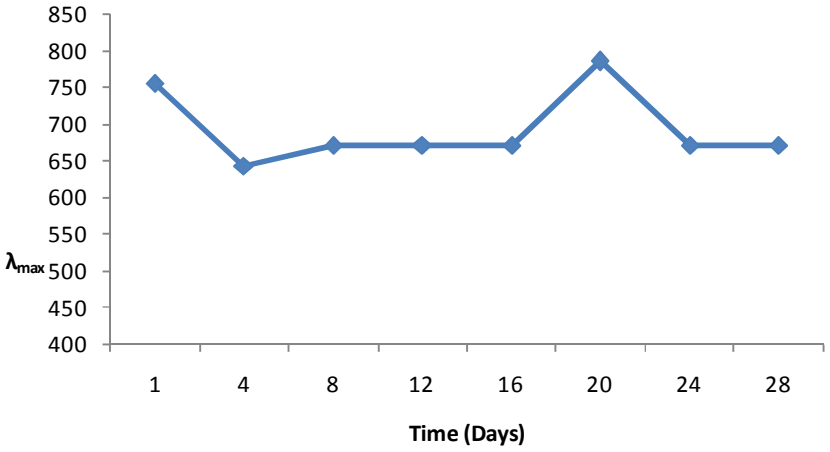


Figure 4.1.5: Variation of λ_{max} with time using diesel coloured with *L. inermis* dye

4.1.4 Colour intensity of *P.osun* dye in petroleum products

Table 4.1.8-4.1.10 and Figure 4.1.6- 4.1.8 below respectively showed results of the colour intensity of *P. osun* dye in petrol, kerosene and diesel within the specific period of twenty-eight days. From the table, it can be observed that kerosene was coloured yellow while petrol and diesel were coloured orange.

Table 4.1.8: Colour intensity of *P. osun* dye in petrol

Time (Days)	*Colour*	
	Red	Yellow
1	6	10
4	2	4
8	8	13
12	3	6
16	1	4
20	7	12
24	4	9
28	1	4

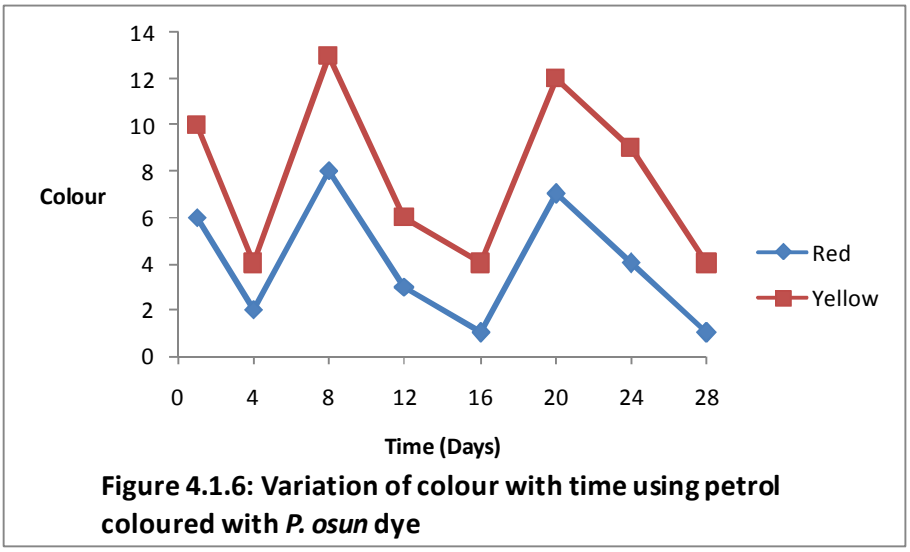


Figure 4.1.6: Variation of colour with time using petrol coloured with *P. osun* dye

Table 4.1.9: Colour intensity of *P.osun* dye in kerosene

	Colour
Time (Days)	Yellow
1	0.7
4	1
8	0.7
12	0.7
16	0.7
20	0.7
24	0.9
28	0.7

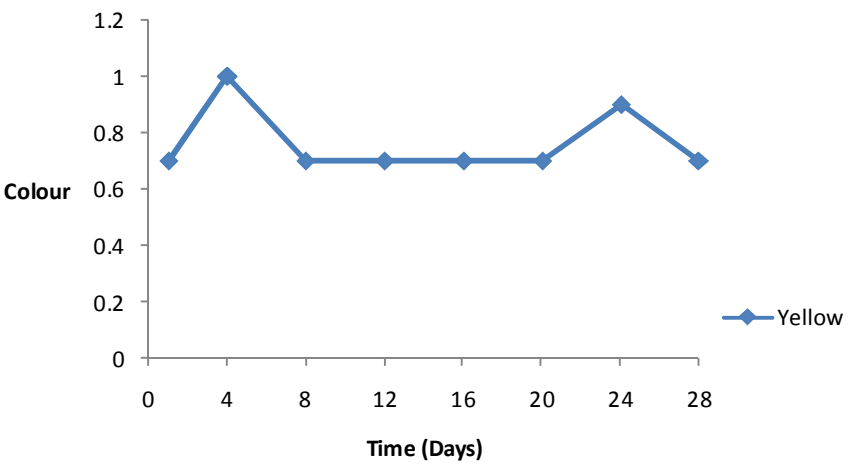


Figure 4.1.7: Variation of colour with time using kerosene coloured with *P. osun* dye

Table 4.1.10: Colour intensity of *P.osun* dye in diesel

Time (Days)	*Colour*	
	Red	Yellow
1	1	3
4	1	3
8	1	3
12	1	3
16	2	5
20	3	6
24	1	4
28	1	3

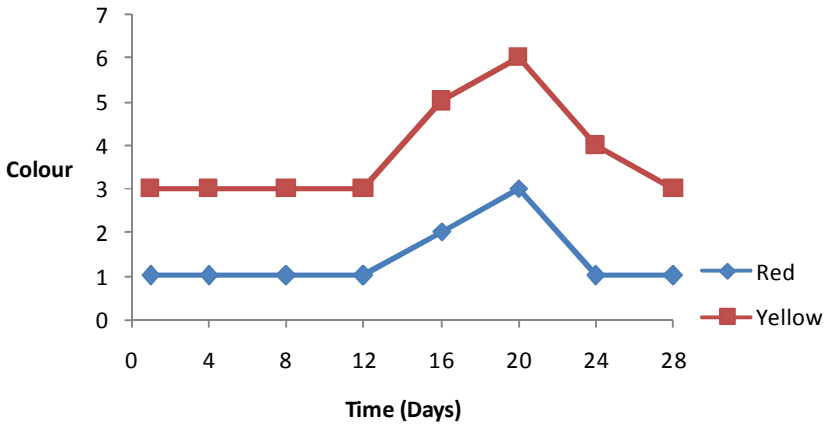


Figure 4.1.8: Variation of colour with time using diesel coloured with *P. osun* dye

4.1.5 Colour intensity of *L. inermis* dye in petroleum products

In Table, 4.1.11-4.1.13 and Figure 4.1.9-4.1.11 below respectively showed the colour intensity results of *L. inermis* dye in petroleum products using the same period of time.

Table 4.1.11: Colour intensity of *L. inermis* dye in petrol

	Colour	
Time (Days)	Red	Yellow
1	5	20
4	5	20
8	5	20
12	5	20
16	5	20
20	5	20
24	5	20
28	5	20

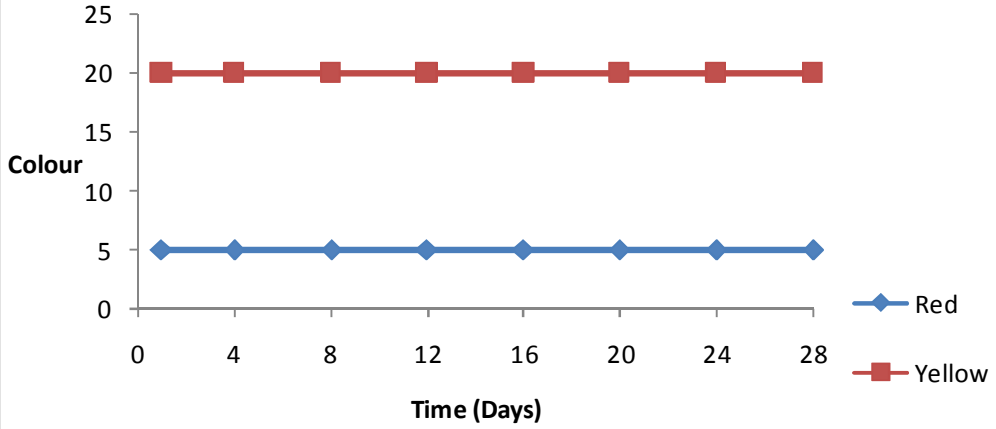


Figure 4.1.9: Variation of colour with time using petrol coloured with *L. inermis* dye

Table 4.1.12: Colour intensity of *L. inermis* dye in kerosene

	Colour
Time (Days)	Yellow
1	0.6
4	0.5
8	0.4
12	0.3
16	0.2
20	0.4
24	0.6
28	0.2

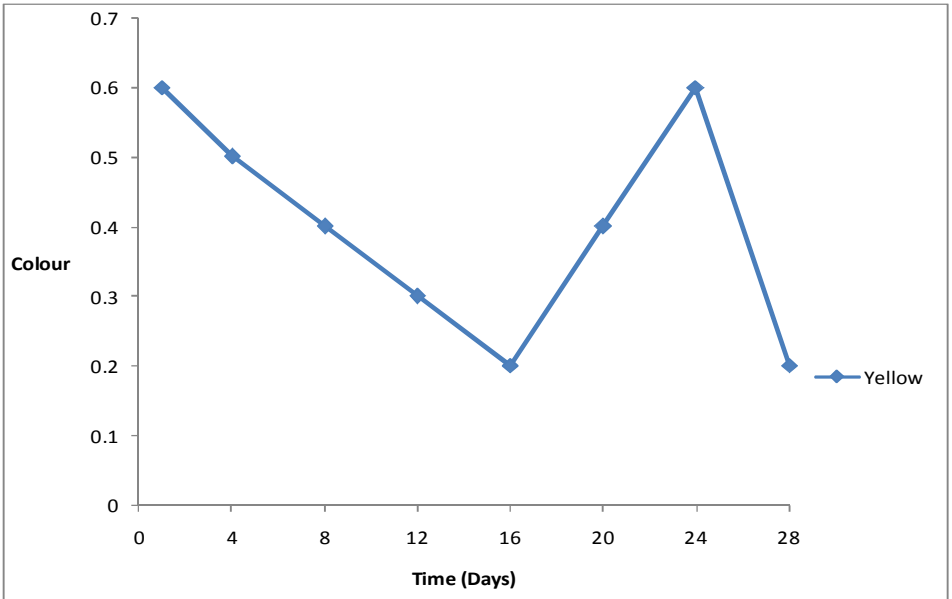


Figure 4.1.10: Variation of colour with time using kerosene coloured with *L. inermis* dye

Table 4.1.13: Colour intensity of *L. inermis* dye in diesel

	Colour	
Time (Days)	Red	Yellow
1	3	5
4	1	2
8	2	3
12	2	3
16	2	3
20	4	6
24	2	3
28	2	3

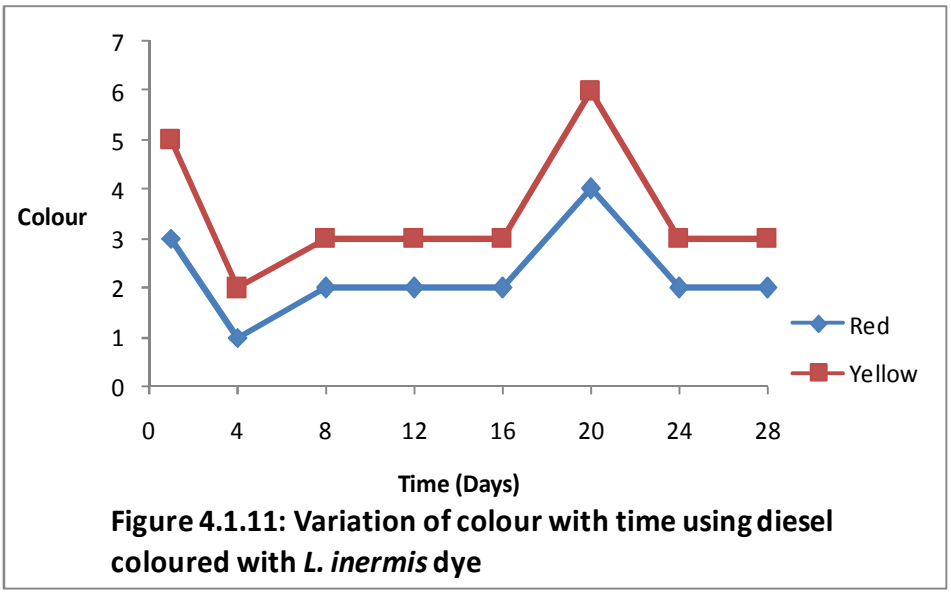


Figure 4.1.11: Variation of colour with time using diesel coloured with *L. inermis* dye

4.1.6 Infrared spectra analysis of *P. osun* dye

The infrared absorption bands and assignment of *P. osun* dye using NaCl disc were presented in Table 4.1.14 as shown below:

Table 4.1.14: FTIR of *P. osun* dye

Bands (cm^{-1})	**Assignment**
3435 br	O-H stretching
2935	CH_2, CH_3 of aliphatic group
2279	Cſ C stretching
1620, 1441	C=C of aromatic
1267, 1099	C-O, C-N stretching
772	Substitution in aromatic ring

4.1.7 Infrared spectra analysis of *L. inermis* dye

Table 4.1.15 showed the infrared absorption bands and assignment of *L.inermis* dye using KBr disc.

Table 4.1.15: FTIR of *L. inermis* dyes

Bands (cm^{-1})	**Assignment**
3380 br	O-H stretching
2930	CH_2, CH_3 of aliphatic group
2358	Cſ C stretching
1627, 1426	C=C of aromatic
1266, 1058	C-N or C-O bending

4.2 Discussion

Petroleum products are usually coloured with synthetic dyes in order to enable people differentiate them from the others and for aesthetic purposes.

Recently, interest in the use of natural dyes has been growing rapidly due to the result of stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes as previously reported[80]. Bhyan and Saikia 2004, have also reported that synthetic dyes are suspected to release harmful chemicals that are allergic, carcinogenic and detrimental to human health.

With the worldwide concern favouring the use of eco-friendly and biodegradable materials, the use of natural dyes has once again gained interest and these have been reported by a number of workers[81-83.]

Natural dyes serves as a very important colouring pigments in various area and their largest source comes from plant dyes. Therefore, many studies have been carried out using the extract from *P. osun* and *L. inermis* dyes as a colourants for different purposes.

Ezeokonkwo, M. A and Okoro, U.C 2012, previously reported that dye extracted from stem wood of *P.osun* could be used to colour petroleum products[15] but they did not determine how stable the dye were in the petroleum products while no report have been received on the stability of *L. inermis* for colouring petroleum products.

Based on the aforementioned, the stability of the dark red and orange brown dyes extracted from *Pterocarpus osun* Craib and *Lawsonia inermis* Linn. using ethanol and methanol respectively in petroleum products (petrol, kerosene and diesel) were investigated for a period of twenty-eight days. The number of days was chosen as a rough estimate of the life of the petroleum products spanning production in the refinery, transportation to the petrol filling station and disposal by the filling station.

The percentage yield of *P. osun* and *L. inermis* dyes used was 1.44 and 1.50 % and they melted at 130ï 132 ^{o}C and 190-191 ^{o}C respectively (Table 4.1).

Selection of the solvent used in the extraction, primarily depends on the availability of the solvent as well as the solubility of the dyes in the solvent (Table 4.1.1).

From results, *P. osun* dye wavelengths of maximum absorption and colour intensity in petrol, kerosene and diesel were not remarkably stable as shown in Figure 4.1, 4.1.1, 4.1.2, 4.1.6, 4.1.7 and 4.1.8 respectively. Thus, the dye cannot be used for colouring petroleum products. The *L. Inermis* dye wavelengths of maximum absorption and colour intensity in petrol as seen in Figure 4.1.3 and 4.1.9 respectively, showed that the dye wavelengths and colour in petrol were constant through the period of twenty-eight days. These were not the same for kerosene and diesel as shown in Figure 4.1.4, 4.1.5, 4.1.10 and 4.1.11 respectively.

Therefore, *L. inermis* dye was actively stable in colouring petrol because the colour does not fade when added to petrol. They cannot be used for the same purpose in kerosene and diesel because the colour fades with time when added to kerosene and diesel.

FTIR of the two plants extract (*P. osun* and *L. inermis* dyes) showed the presence of phenolic O-H stretching at 3435 cm^{-1} and 3380 cm^{-1} . They

also showed the presence of C=C of aromatic at 1620ï 1441 cm^{-1} and 1627ï 1426 cm^{-1} respectively as the functional groups present.

4.3 Contributions to knowledge

This research has made the following contributions to knowledge:

i. The dye from *L. inermis* when applied to petrol, it retains a stable colour for at least 28 days.

ii. Dye extracted from the leaf of *L. inermis* can be used as colourant for petrol while they cannot be used for the same purpose in kerosene and diesel.

iii. Refinery industries can use local plant dye from the leaf of *L. inermis* to colour petrol instead of using the imported synthetic dyes(carcinogenic, mutagenic, allergic and poisonous) whose composition, chemical names and nature are not disclosed.

iv. Oil industries should avoid the use of dye extracted from the heartwood of *P. osun* in colouring petroleum products because the colour of the dye are not stable for a reasonable length of time when applied in petrol, kerosene and diesel.

v. Dye from *L. inermis* can help in quick, visual differentiation of petrol but cannot in kerosene and diesel while the dye from *P. osun* cannot help in quick, visual differentiation of petrol, kerosene and diesel.

4.4 Conclusion

In this present work, it has been established that the dye extracted from the young leaf of *L. inermis* dye can be used as alternative source of colourant for petrol in the oil industry. They cannot be used for the same purpose for kerosene and diesel. The dye extracted from the heartwood of *Pterocarpus osun* Craib cannot be used in colouring petrol, kerosene and diesel because the colour of the dye was not stable in the petroleum products.

Hence, individual manufacturers, companies and organizations can now use these dyes extracted from the leaves of *L. inermis* as trademark to differentiate their petrol from those of other manufacturers. Most especially, this period that many nations are returning to the use of natural colourants based on the alarming rate of environmentally unfriendliness caused by synthetic dyes.

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APPENDIX 1a

UV-VISIBLE SPECTRA RESULTS OF *P. osun* EXTRACT IN PETROL FOR 28 DAYS

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/05/2015
01:55:11 PM

RawData - C:\NARICT\MAXWEL PETROL P.O.spc

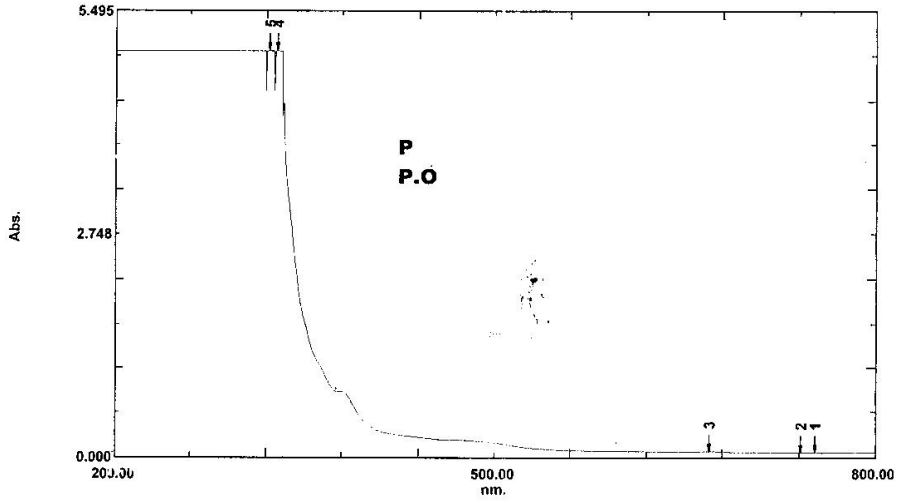
No.	P/V	Wavelength	Abs.	Description
1	⊕	752.50	0.051	
2	⊕	740.50	0.051	
3	⊕	669.50	0.067	
4	⊕	329.00	5.000	
5	⊕	322.50	5.000	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

Footnote:

P P.O= Petrol mixed with *Pterocarpus osun* dye extract.

1 b

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/09/2015

09:16:33 AM

RawData - C:\NARICT\MAXWELL PETROL.spc

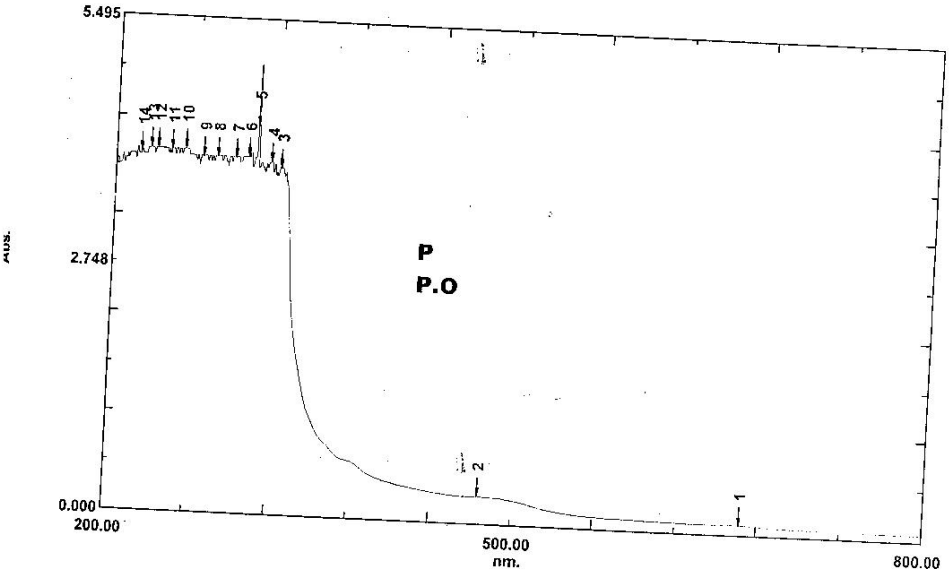
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	667.00	0.102	
2	ⓟ	475.50	0.294	
3	ⓟ	322.50	3.874	
4	ⓟ	315.00	3.935	
5	ⓟ	305.00	4.328	
6	ⓟ	299.00	3.984	
7	ⓟ	289.00	3.972	
8	ⓟ	276.00	3.972	
9	ⓟ	265.50	3.972	
10	ⓟ	252.00	4.039	
11	ⓟ	242.00	4.024	
12	ⓟ	231.50	4.039	
13	ⓟ	226.00	4.039	
14	ⓟ	219.00	3.998	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



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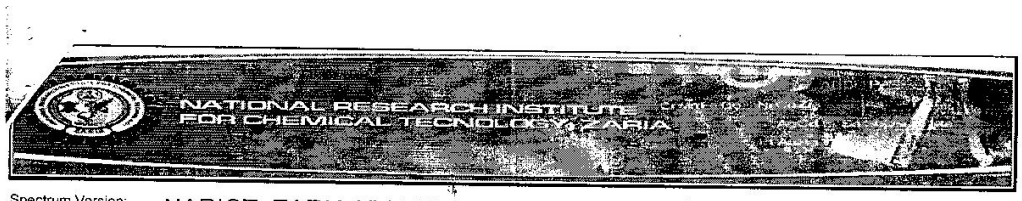

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/11/2015

09:48:24 AM

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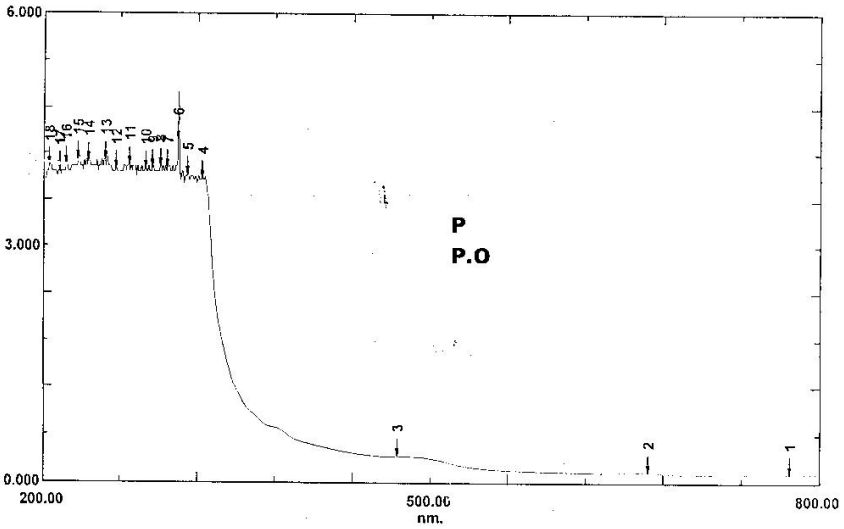
No.	P/V	Wavelength	Abs.	Description
1	⊕	777.00	0.103	
2	⊕	668.50	0.125	
3	⊕	474.50	0.321	
4	⊕	324.00	3.887	
5	⊕	312.00	3.929	
6	⊕	305.00	4.374	
7	⊕	297.00	4.022	
8	⊕	291.50	4.033	
9	⊕	285.50	4.025	
10	⊕	280.00	4.001	
11	⊕	267.00	4.048	
12	⊕	257.00	3.998	
13	⊕	248.50	4.123	
14	⊕	235.00	4.106	
15	⊕	227.50	4.127	
16	⊕	218.00	4.045	
17	⊕	212.50	3.976	
18	⊕	204.50	4.039	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

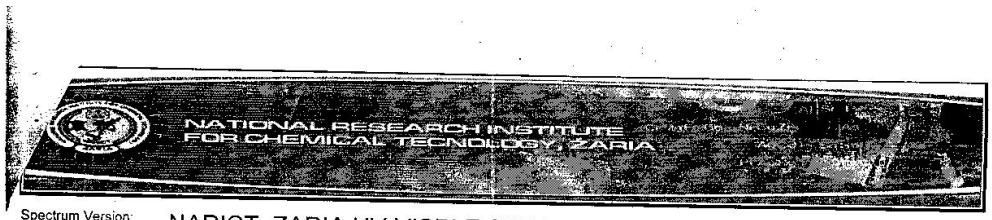
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

1d



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/16/2015
09:30:41 AM

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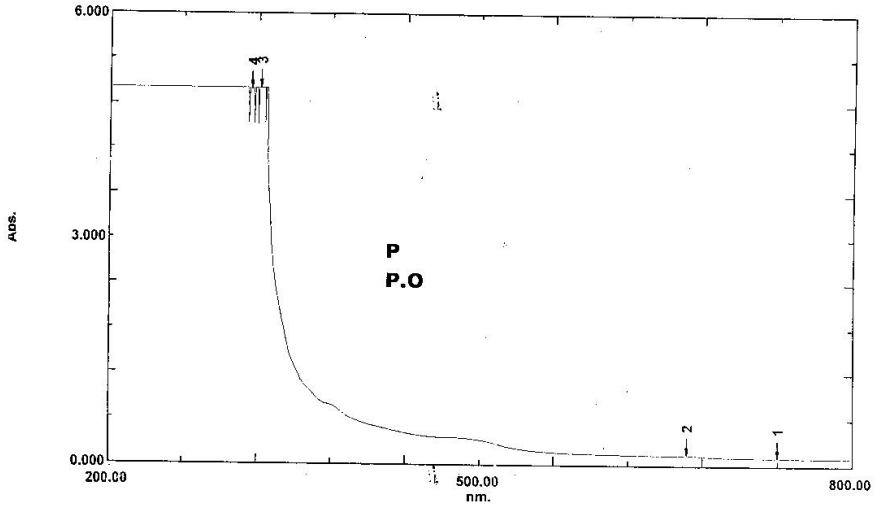
No.	P/V	Wavelength	Abs.	Description
1	⊕	740.00	0.099	
2	⊕	667.00	0.131	
3	⊕	323.00	5.000	
4	⊕	315.00	4.981	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

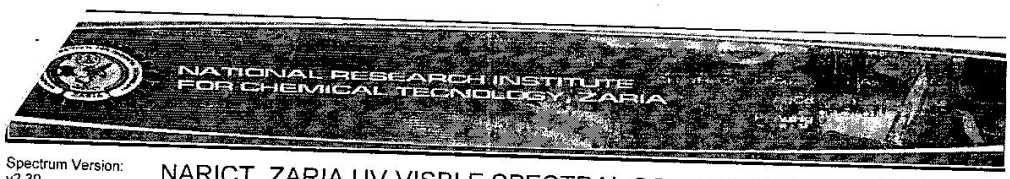
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

1e



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

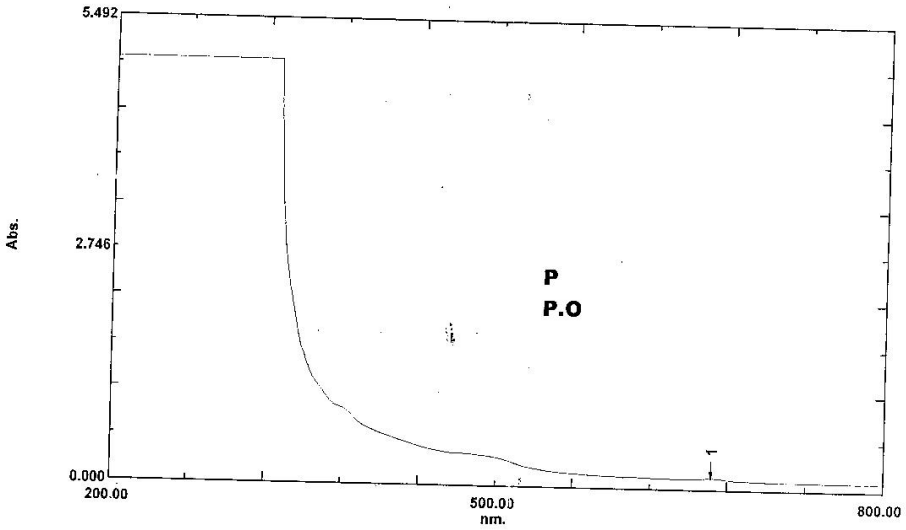
03/18/2015

03:00:21 PM

RawData - C:\NARICT\MAXWEL PETROL P.O DAY 16.spc

No.	P/V	Wavelength	Abs.	Description
1	⊕	667.50	0.133	

Measurement Properties	
Wavelength Range (nm.):	200.00 to 800.00
Scan Speed:	Fast
Sampling Interval:	0.5
Auto Sampling Interval:	Disabled
Scan Mode:	Single
Instrument Properties	
Instrument Type:	UV-2500PC Series
Measuring Mode:	Absorbance
Slit Width:	2.0 nm
Light Source Change Wavelength:	360.0 nm
S/R Exchange:	Normal
Attachment Properties	
Attachment:	None
Sample Preparation Properties	
Weight:	1g
Volume:	3ml
Dilution:	1
Path Length:	1
Additional Information:	



NALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

1f

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/23/2015

11:19:07 AM

RawData - C:\NARICT\MAXWEL PETROL DAY 20 P.O.spc

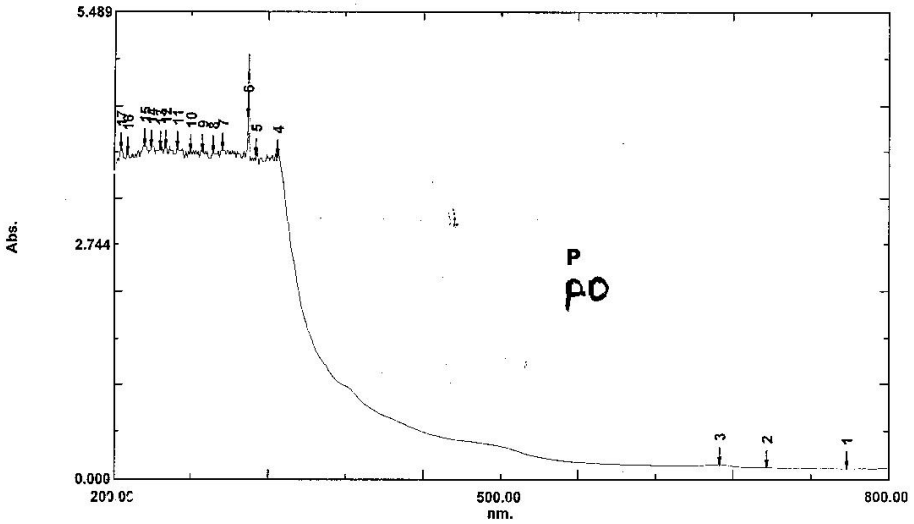
No.	P/V	Wavelength	Abs.	Description
1	P	767.50	0.114	
2	V	706.00	0.128	
3	P	669.00	0.166	
4	P	327.50	3.781	
5	P	311.50	3.796	
6	P	305.00	4.254	
7	P	285.00	3.862	
8	P	278.00	3.827	
9	P	269.50	3.853	
10	P	260.50	3.842	
11	P	250.00	3.877	
12	P	240.00	3.891	
13	P	236.00	3.879	
14	P	229.50	3.901	
15	P	224.00	3.906	
16	P	210.50	3.817	
17	P	205.50	3.859	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

1g

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/08/2015

11:44:44 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL PETROL P.O.....spc

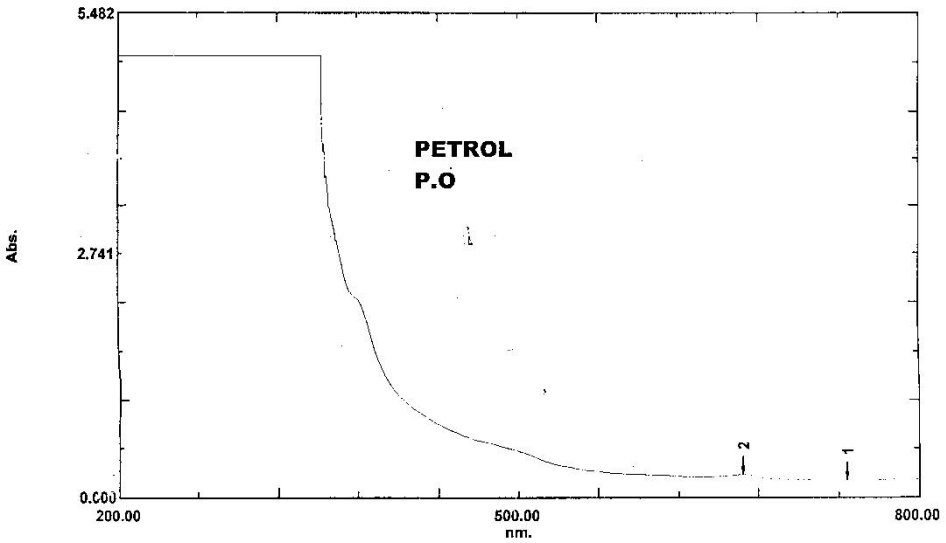
No.	P/V	Wavelength	Abs.	Description
1	⊕	746.50	0.185	
2	⊕	668.00	0.248	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

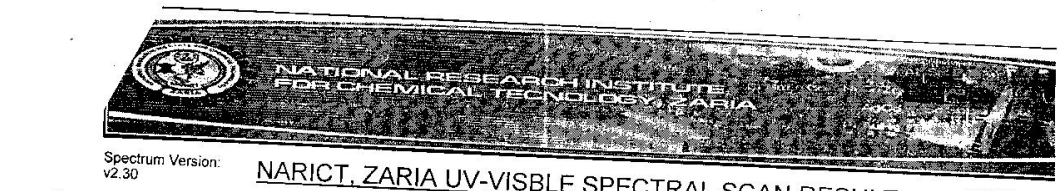
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

1h



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/13/2015
12:04:17 PM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL PETROL P.O.28.spc

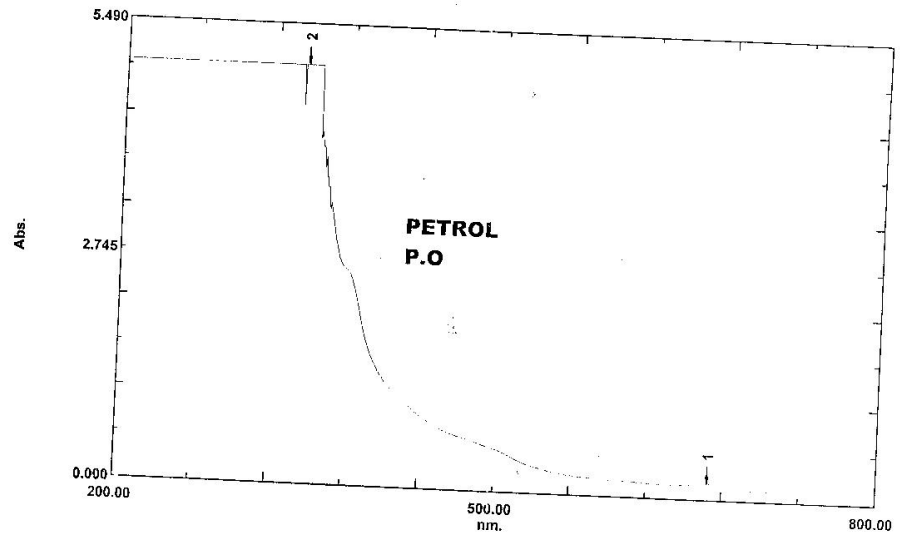
No.	P/V	Wavelength	Abs.	Description
1	ⓣ	668.50	0.181	
2	ⓣ	343.00	5.000	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

APPENDIX 2a

UV VISIBLE SPECTRA RESULTS OF *P. osun* EXTRACT IN KEROSENE FOR 28 DAYS

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/05/2015

RawData - C:\NARICT\MAXWEL (KEROSINE). P.O.spc

01:59:49 PM

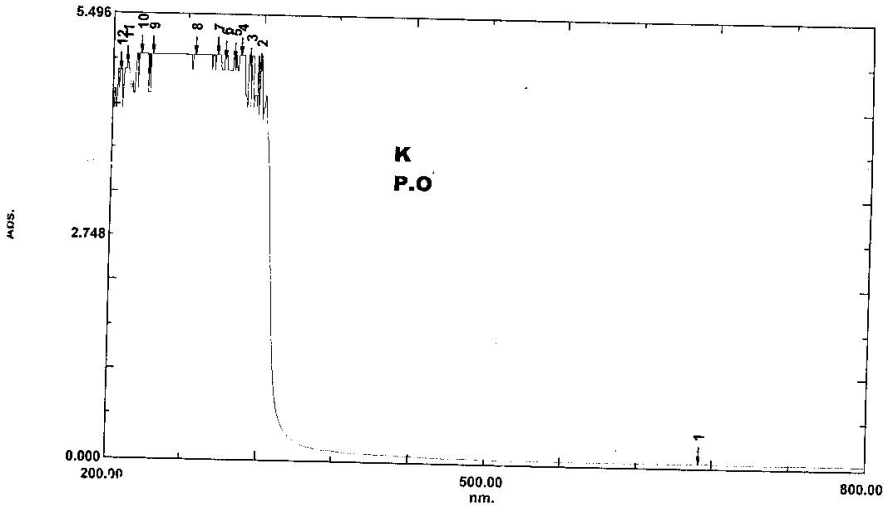
No.	P/V	Wavelength	Abs.	Description
1		669.50	0.051	
2		318.00	4.811	
3		310.00	4.873	
4		303.00	5.000	
5		297.50	4.942	
6		290.50	4.956	
7		284.50	5.000	
8		266.50	5.000	
9		232.50	5.000	
10		223.00	5.000	
11		211.50	4.883	
12		206.00	4.797	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

Footnote:

K P.O= Kerosene mixed with *Pterocarpus osun* dye extract.

2b

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/09/2015

09:13:34 AM

RawData - C:\NARICT\MAXWELL KEROSINE.spc

No.	P/V	Wavelength	Abs.	Description
1	P	775.50	0.045	
2	V	669.00	0.067	
3	P	602.50	0.057	
4	V	533.50	0.066	
5	P	471.50	0.091	
6	V	378.50	0.502	
7	P	328.50	3.810	
8	V	321.00	3.815	
9	P	313.00	3.825	
10	V	305.00	4.291	
11	P	297.50	3.889	
12	V	290.50	3.897	
13	P	285.50	3.881	
14	V	270.50	3.864	
15	P	253.50	3.877	
16	V	248.00	3.887	
17	P	239.50	3.876	
18	V	235.00	3.881	
19	P	228.50	3.868	

Measurement Properties

Wavelength Range (nm.):	200.00 to 800.00
Scan Speed:	Fast
Sampling Interval:	0.5
Auto Sampling Interval:	Disabled
Scan Mode:	Single

Instrument Properties

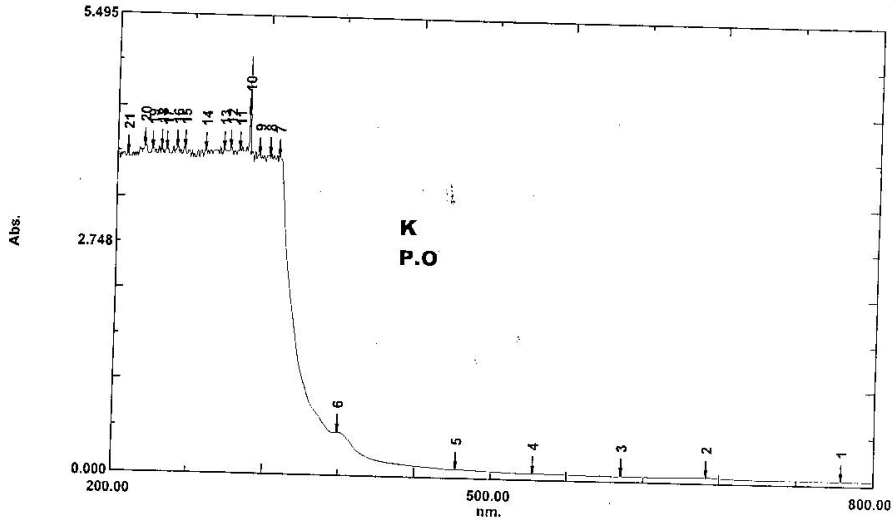
Instrument Type:	UV-2500PC Series
Measuring Mode:	Absorbance
Slit Width:	2.0 nm
Light Source Change Wavelength:	360.0 nm
S/R Exchange:	Normal

Attachment Properties

Attachment:	None

Sample Preparation Properties

Weight:	1g
Volume:	3ml
Dilution:	1
Path Length:	1
Additional Information:	



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

2c

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/11/2015

09:44:04 AM

RawData - C:\NARICT\MAXWELL KERO P.P DAY 8.spc

No.	P/V	Wavelength	Abs.	Description
1	P	669.50	0.103	
2	P	323.00	3.845	
3	P	317.00	3.875	
4	V	310.50	3.869	
5	V	305.00	4.314	
6	P	299.50	3.954	
7	P	292.50	3.947	
8	P	281.00	3.967	
9	P	269.50	3.956	
10	V	259.50	3.911	
11	P	247.00	3.969	
12	V	239.50	3.999	
13	P	226.00	3.953	
14	V	215.50	3.883	
15	V	207.00	3.914	

Measurement Properties

Wavelength Range (nm.):	200.00 to 800.00
Scan Speed:	Fast
Sampling Interval:	0.5
Auto Sampling Interval:	Disabled
Scan Mode:	Single

Instrument Properties

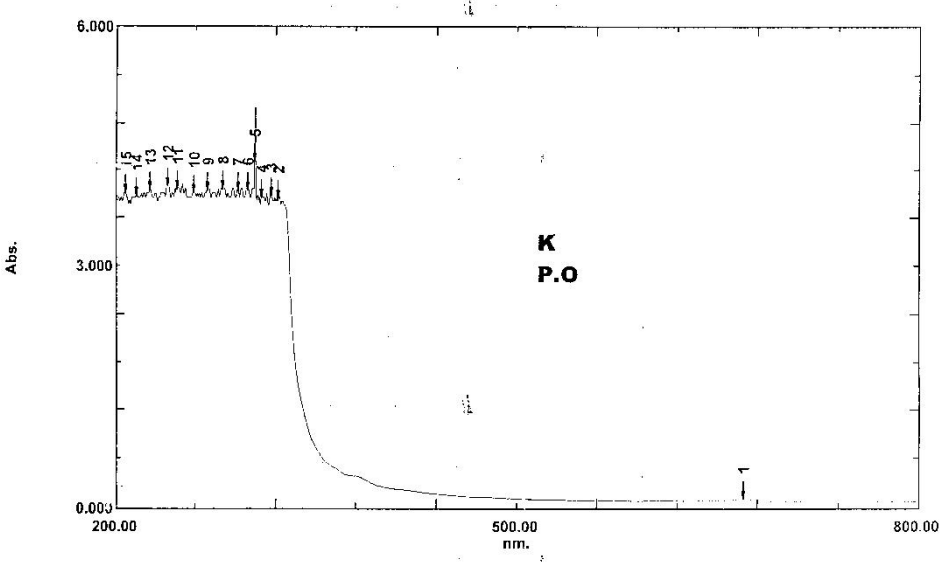
Instrument Type:	UV-2500PC Series
Measuring Mode:	Absorbance
Slit Width:	2.0 nm
Light Source Change Wavelength:	360.0 nm
S/R Exchange:	Normal

Attachment Properties

Attachment:	None

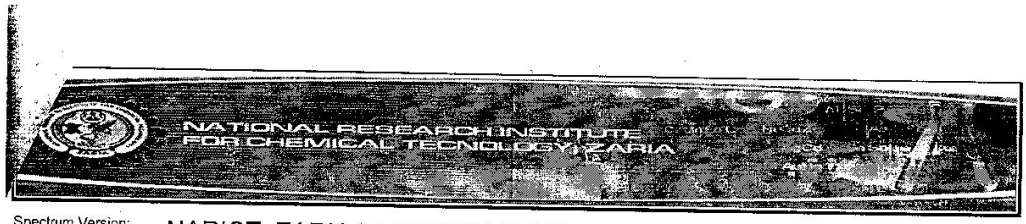
Sample Preparation Properties

Weight:	1g
Volume:	3ml
Dilution:	1
Path Length:	1
Additional Information:	



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

2d



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/16/2015

09:51:10 AM

RawData - C:\NARICT\MAXWELL KEROSINE P.O DAY 12.spc

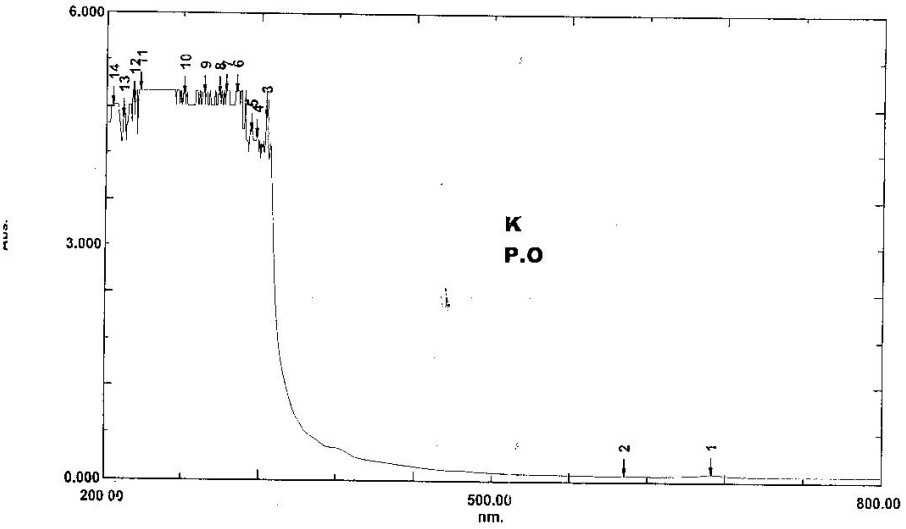
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	669.50	0.094	
2	ⓟ	602.50	0.071	
3	ⓟ	325.00	4.649	
4	ⓟ	317.50	4.408	
5	ⓟ	313.00	4.474	
6	ⓟ	302.00	4.985	
7	ⓟ	293.50	4.978	
8	ⓟ	288.00	4.956	
9	ⓟ	276.50	4.963	
10	ⓟ	261.00	4.956	
11	ⓟ	227.00	5.000	
12	ⓟ	222.00	4.887	
13	ⓟ	214.00	4.655	
14	ⓟ	205.50	4.817	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

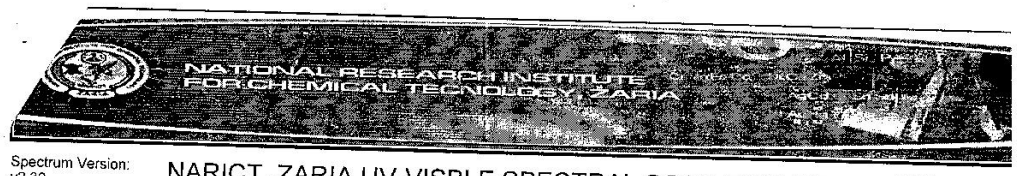
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

2e



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/18/2015

02:50:34 PM

RawData - C:\NARICT\MaXWELL KERO DAY 16 KERO P.O.spc

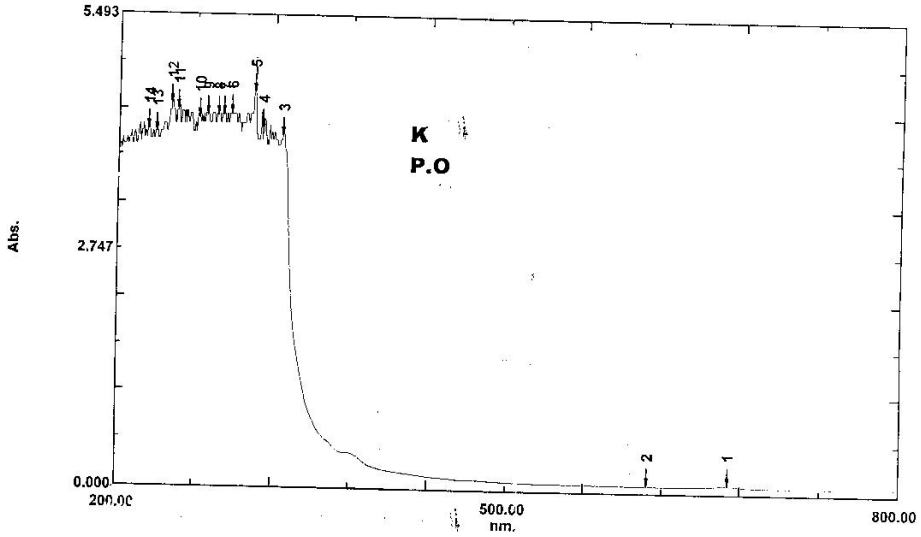
No.	P/V	Wavelength	Abs.	Description
1		670.00	0.093	
2		608.50	0.075	
3		326.50	4.083	
4		311.50	4.180	
5		305.00	4.600	
6		287.50	4.340	
7		280.50	4.322	
8		276.50	4.322	
9		268.00	4.322	
10		262.00	4.300	
11		246.00	4.396	
12		241.00	4.444	
13		229.50	4.120	
14		223.00	4.155	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

2f

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/23/2015
11:35:05 AM

RawData - C:\NARICT\MAXWELL KERO P.O DAY 20...spc

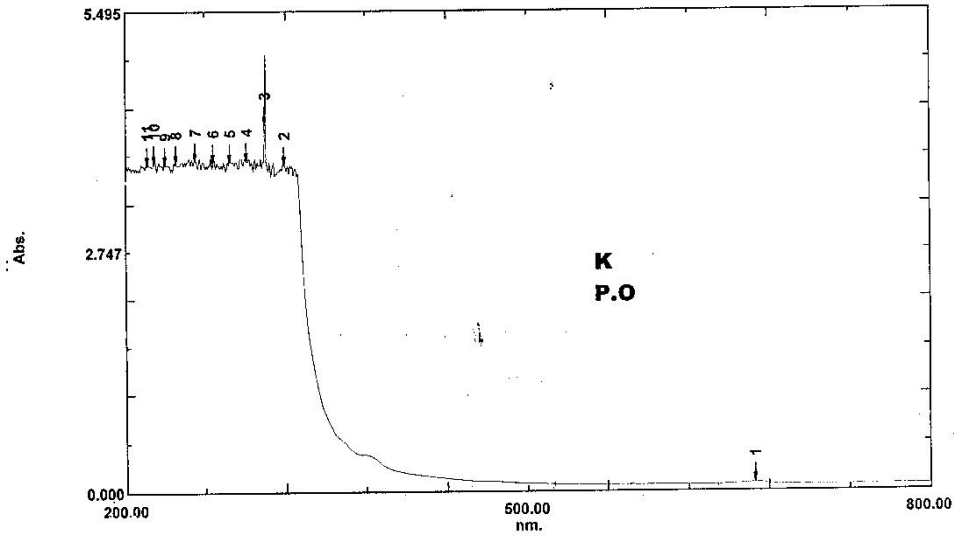
No.	P/V	Wavelength	Abs.	Description
1	⊕	669.50	0.079	
2	⊕	319.00	3.736	
3	⊕	305.00	4.207	
4	⊕	291.50	3.790	
5	⊕	279.00	3.781	
6	⊕	266.50	3.769	
7	⊕	252.50	3.790	
8	⊕	238.50	3.756	
9	⊕	230.00	3.745	
10	⊕	222.00	3.756	
11	⊕	216.50	3.748	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



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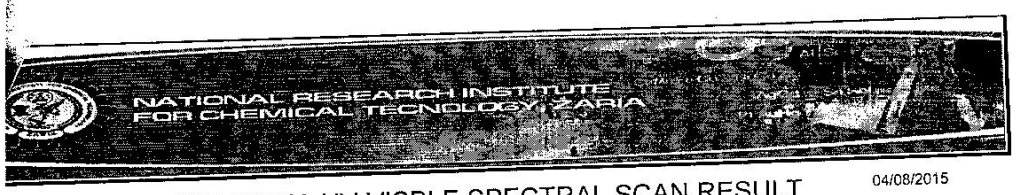
2g

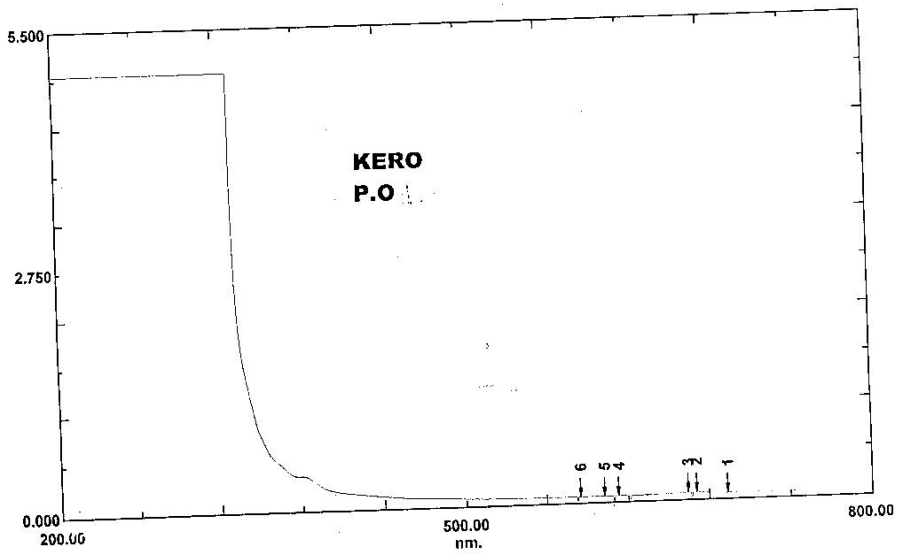


um Version: NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT 04/08/2015 11:12:08 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\File_090721_102144_111012.s

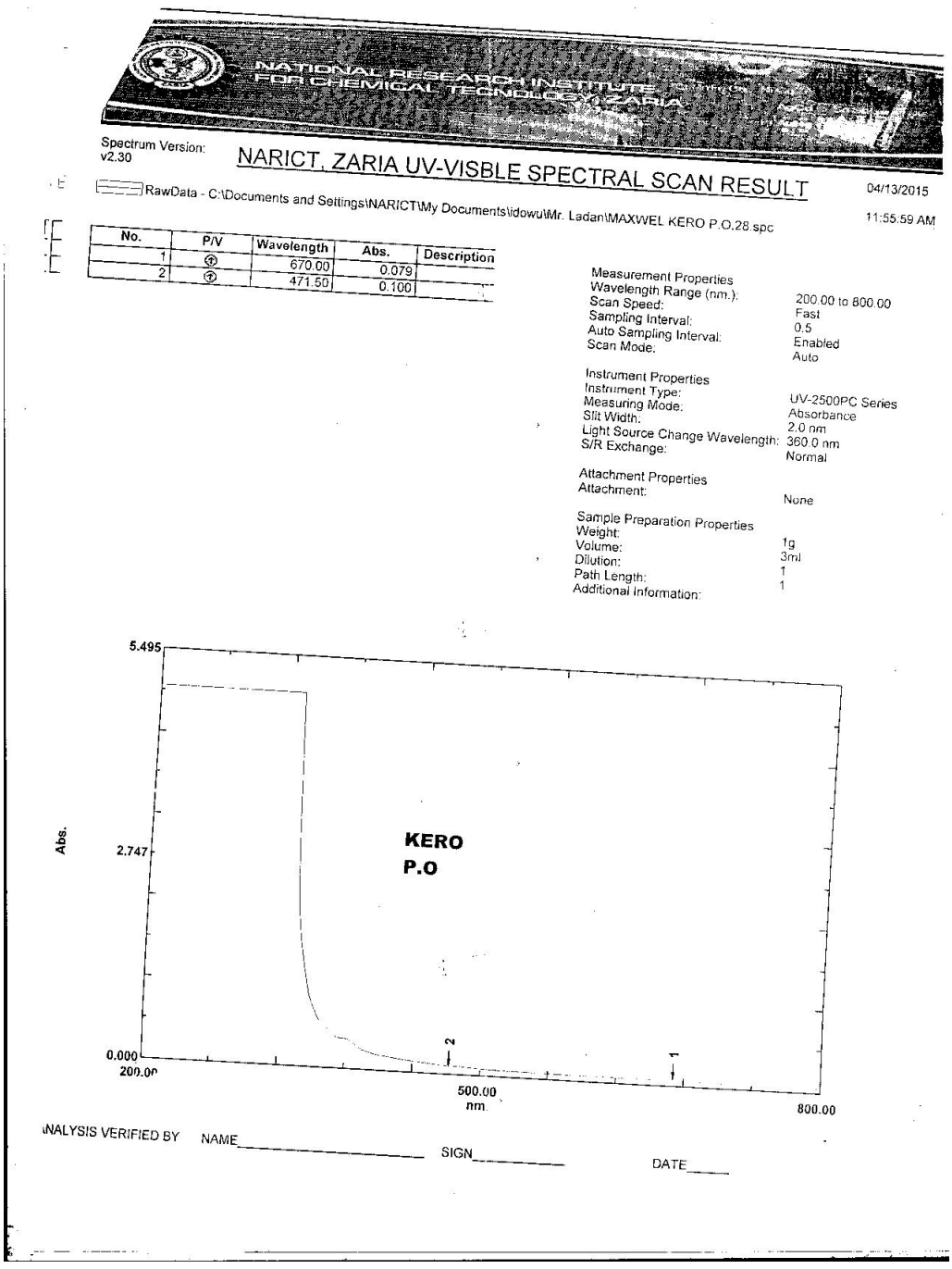
lo.	P/V	Wavelength	Abs.	Description
1	ⓟ	693.50	0.056	
2	ⓟ	670.00	0.077	
3	ⓟ	664.00	0.073	
4	ⓟ	613.00	0.061	
5	ⓟ	603.00	0.061	
6	ⓟ	585.50	0.060	

Measurement Properties	
Wavelength Range (nm.):	200.00 to 800.00
Scan Speed:	Fast
Sampling Interval:	0.5
Auto Sampling Interval:	Enabled
Scan Mode:	Auto
Instrument Properties	
Instrument Type:	UV-2500PC Series
Measuring Mode:	Absorbance
Slit Width:	2.0 nm
Light Source Change Wavelength:	360.0 nm
S/R Exchange:	Normal
Attachment Properties	
Attachment:	None
Sample Preparation Properties	
Weight:	1g
Volume:	3ml
Dilution:	1
Path Length:	1
Additional Information:	



NALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

2h


NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA
Spectrum Version: v2.30
NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT
04/13/2015
11:55:59 AM
RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL KERO P.O.28.spc
No. | P/V | Wavelength | Abs. | Description
1 | ⓟ | 670.00 | 0.079 |
2 | ⓟ | 471.50 | 0.100 |
Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto
Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal
Attachment Properties
Attachment: None
Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:
5.495
Abs.
2.747
KERO
P.O
0.000
200.00
500.00
nm.
800.00
ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

APPENDIX 3a

UV-VISIBLE SPECTRA RESULTS OF *P. osun* EXTRACT IN DIESEL FOR 28 DAYS

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/05/2015
01:49:40 PM

RawData - C:\NARICT\MAXWEL DIESEL P.O.spc

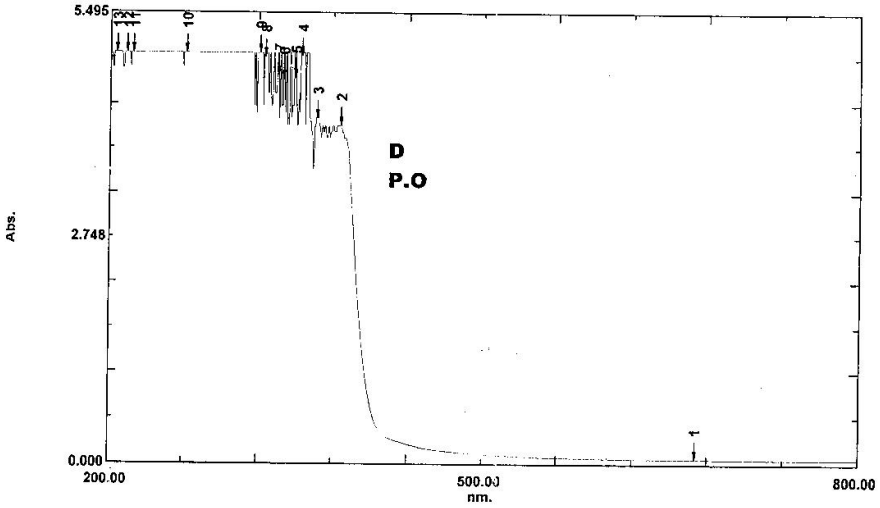
No.	P/V	Wavelength	Abs.	Description
1	⊕	670.50	0.056	
2	⊕	385.50	4.141	
3	⊕	367.00	4.220	
4	⊕	355.00	4.971	
5	⊕	349.00	4.704	
6	⊕	340.50	4.684	
7	⊕	336.00	4.740	
8	⊕	326.00	4.955	
9	⊕	321.50	5.000	
10	⊕	262.00	5.000	
11	⊕	219.00	5.000	
12	⊕	213.50	5.000	
13	⊕	205.00	5.000	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

Footnote:

D P.O= Diesel mixed with *Pterocarpus osun* dye extract.

3b

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/09/2015

09:10:14 AM

RawData - C:\NARICT\MAXWELL DIESEL P.O.spc

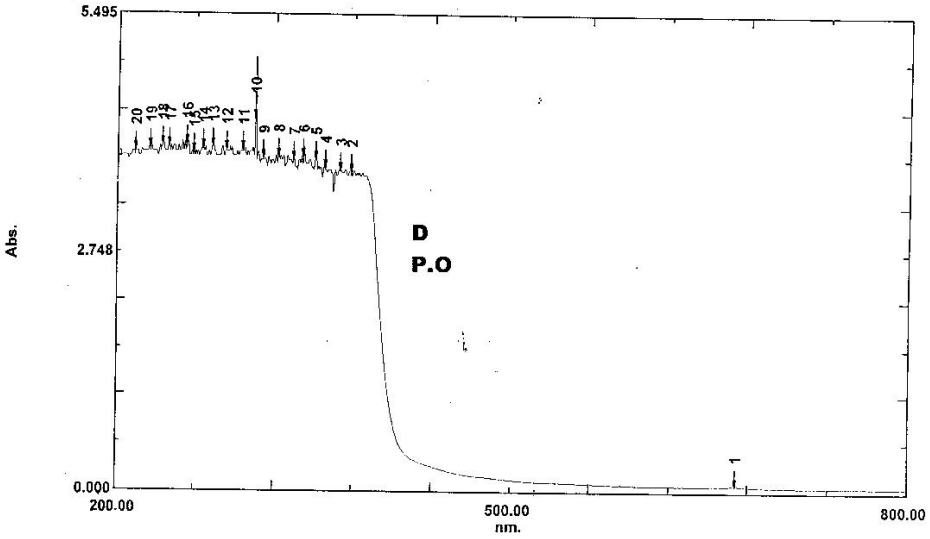
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	670.00	0.070	
2	ⓟ	377.00	3.678	
3	ⓟ	369.00	3.694	
4	ⓟ	357.50	3.723	
5	ⓟ	350.50	3.820	
6	ⓟ	341.50	3.854	
7	ⓟ	333.50	3.823	
8	ⓟ	322.50	3.853	
9	ⓟ	311.50	3.833	
10	ⓟ	305.00	4.298	
11	ⓟ	296.00	3.931	
12	ⓟ	283.50	3.933	
13	ⓟ	273.00	3.966	
14	ⓟ	265.00	3.948	
15	ⓟ	258.00	3.900	
16	ⓟ	253.00	3.991	
17	ⓟ	239.00	3.966	
18	ⓟ	234.50	3.974	
19	ⓟ	224.50	3.949	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

3c

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/11/2015
09:52:01 AM

RawData - C:\NARICT\MAXWELL DIESEL P.O DAY 8.spc

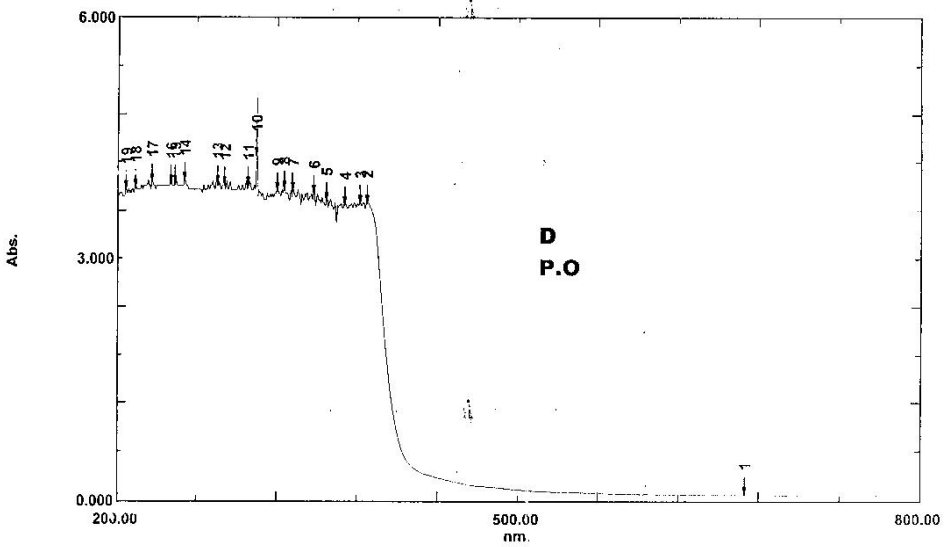
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	669.00	0.073	
2	ⓟ	387.00	3.689	
3	ⓟ	381.50	3.690	
4	ⓟ	370.50	3.680	
5	ⓟ	357.00	3.724	
6	ⓟ	347.00	3.819	
7	ⓟ	332.00	3.846	
8	ⓟ	325.50	3.857	
9	ⓟ	320.50	3.852	
10	ⓟ	305.00	4.276	
11	ⓟ	298.50	3.918	
12	ⓟ	280.50	3.922	
13	ⓟ	276.00	3.924	
14	ⓟ	251.00	3.959	
15	ⓟ	244.00	3.937	
16	ⓟ	240.00	3.937	
17	ⓟ	226.00	3.946	
18	ⓟ	214.00	3.881	
19	ⓟ	206.50	3.855	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



NALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

3d

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/16/2015
09:34:50 AM

RawData - C:\NARICT\MAXWELL DIESEL P.O.spc

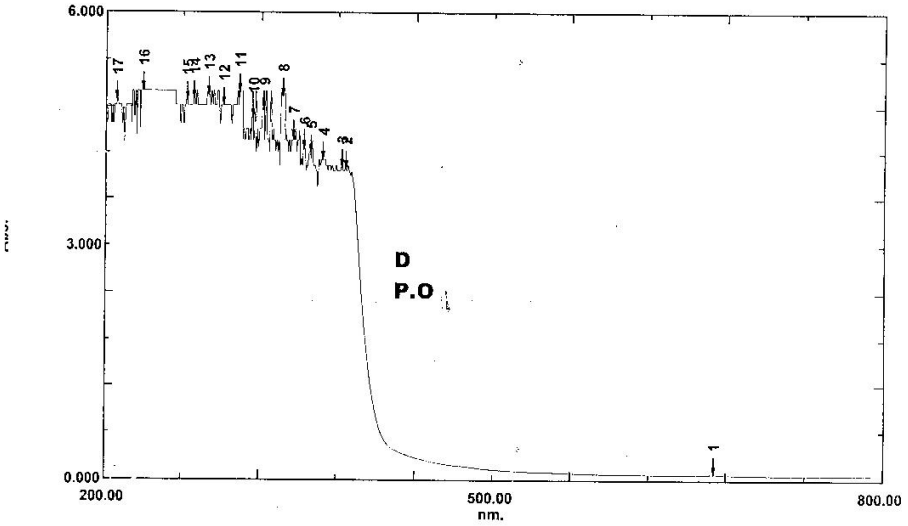
No.	P/V	Wavelength	Abs.	Description
1	⊕	670.00	0.071	
2	⊕	385.50	4.007	
3	⊕	382.50	4.025	
4	⊕	368.50	4.113	
5	⊕	359.00	4.208	
6	⊕	353.50	4.256	
7	⊕	345.50	4.402	
8	⊕	337.00	4.936	
9	⊕	322.50	4.764	
10	⊕	314.00	4.650	
11	⊕	304.00	4.978	
12	⊕	291.50	4.817	
13	⊕	279.50	4.956	
14	⊕	269.00	4.905	
15	⊕	263.00	4.874	
16	⊕	229.00	5.000	
17	⊕	208.50	4.883	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

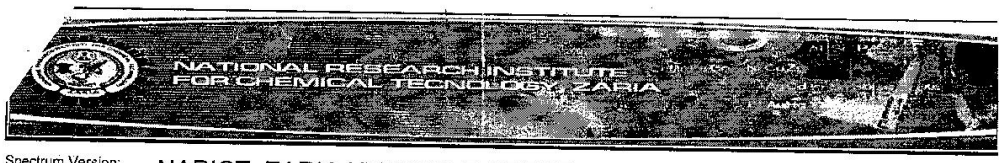
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



IALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

3e



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/18/2015
02:43:34 PM

RawData - C:\NARICT\MAXWELL DAY 16 DIESEL P.O.spc

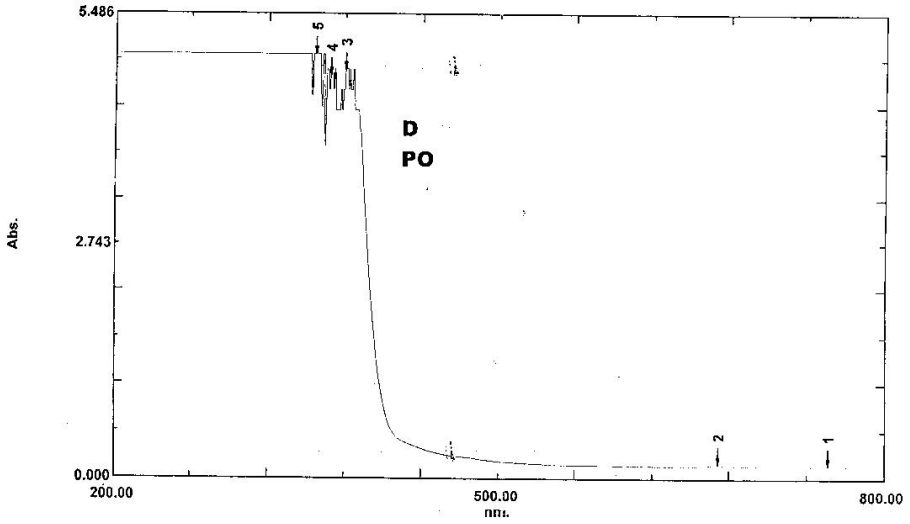
No.	P/V	Wavelength	Abs.	Description
1	⊕	756.50	0.139	
2	⊕	671.50	0.151	
3	⊕	380.00	4.816	
4	⊕	368.00	4.741	
5	⊕	356.50	5.000	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

3f

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/23/2015

11:23:12 AM

RawData - C:\NARICT\MAXWEL DIESEL DAY 20 P.O.spc

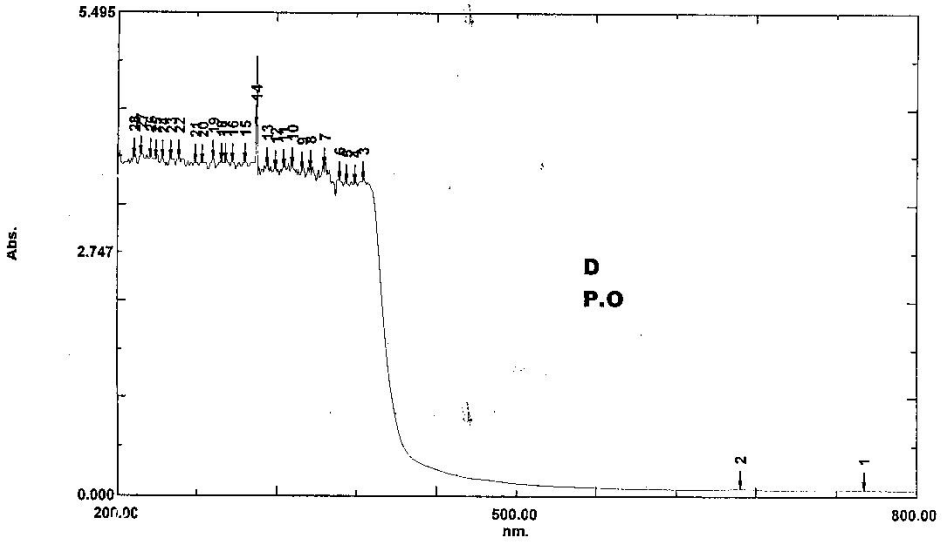
No.	P/V	Wavelength	Abs.	Description
1	P	761.00	0.060	
2	P	668.50	0.075	
3	P	385.00	3.590	
4	P	378.50	3.550	
5	P	372.00	3.553	
6	P	367.00	3.579	
7	P	356.00	3.745	
8	P	345.00	3.704	
9	P	339.50	3.695	
10	P	331.50	3.743	
11	P	326.00	3.733	
12	P	319.50	3.713	
13	P	313.00	3.738	
14	V	305.00	4.207	
15	P	296.50	3.789	
16	P	287.00	3.786	
17	P	282.50	3.792	
18	P	278.50	3.792	
19	P	272.50	3.814	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

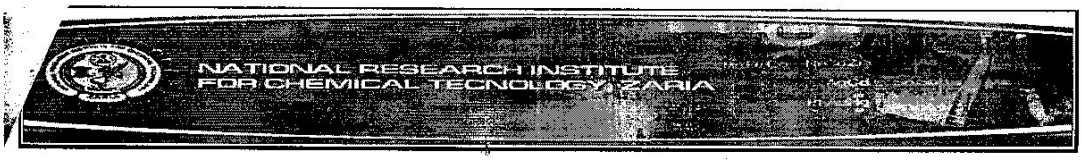
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

3g



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/08/2015
11:15:35 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL DIESEL P.O.spc

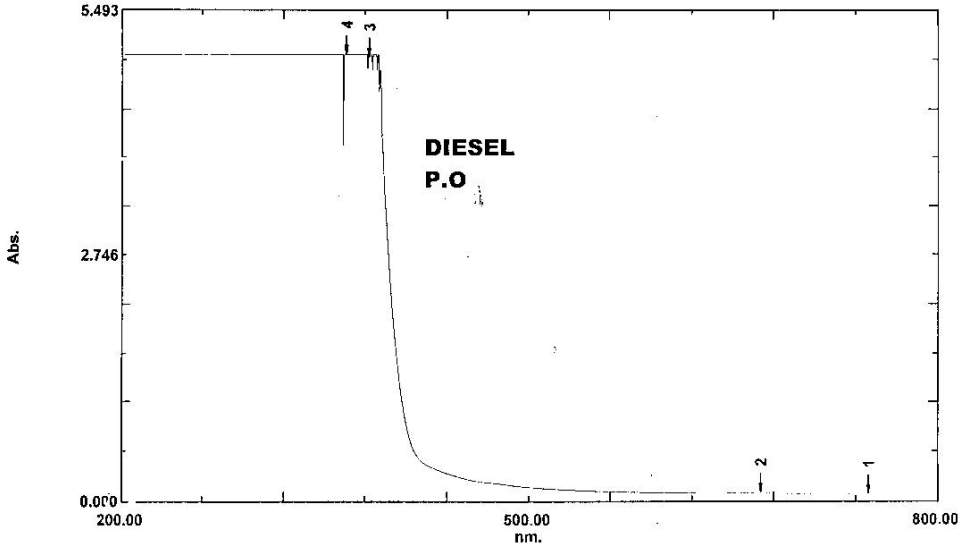
No.	P/V	Wavelength	Abs.	Description
1	①	749.50	0.076	
2	②	670.50	0.090	
3	③	382.50	4.978	
4	④	366.50	5.000	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

3h

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/13/2015

11:59:13 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL DIESEL P.O.28.spc

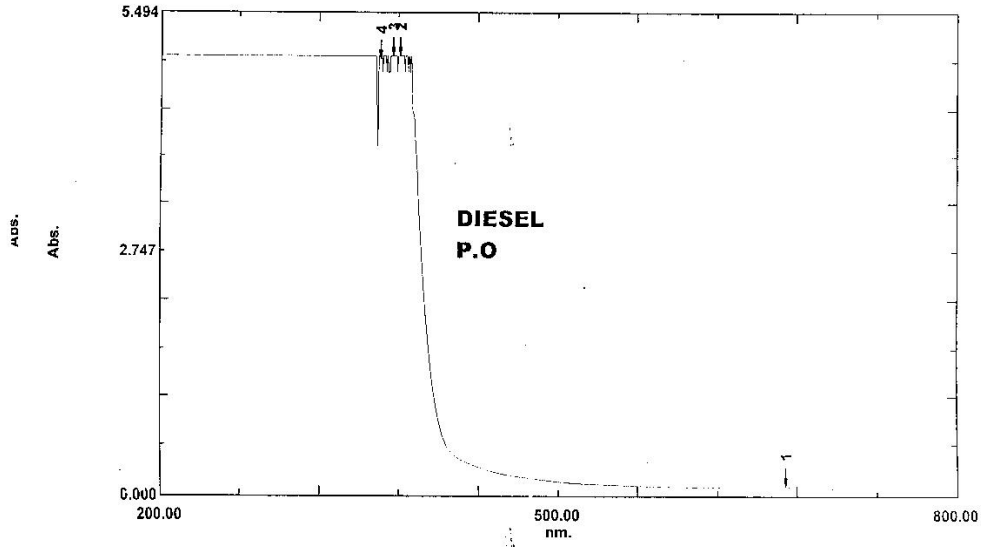
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	671.00	0.090	
2	ⓟ	381.00	5.000	
3	ⓟ	375.50	5.000	
4	ⓟ	366.50	4.978	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

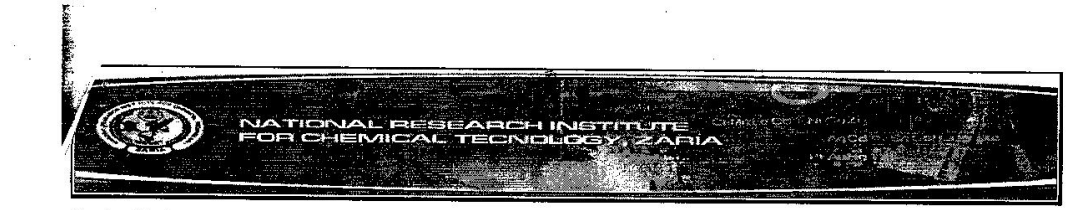
Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ S'GN_____ DATE_____

APPENDIX 4a

UV-VISIBLE SPECTRA RESULTS OF *L. inermis* EXTRACT IN PETROL FOR 28 DAYS



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/05/2015
01:40:10 PM

RawData - C:\NARICT\MAXWEL PETROL L.I.spc

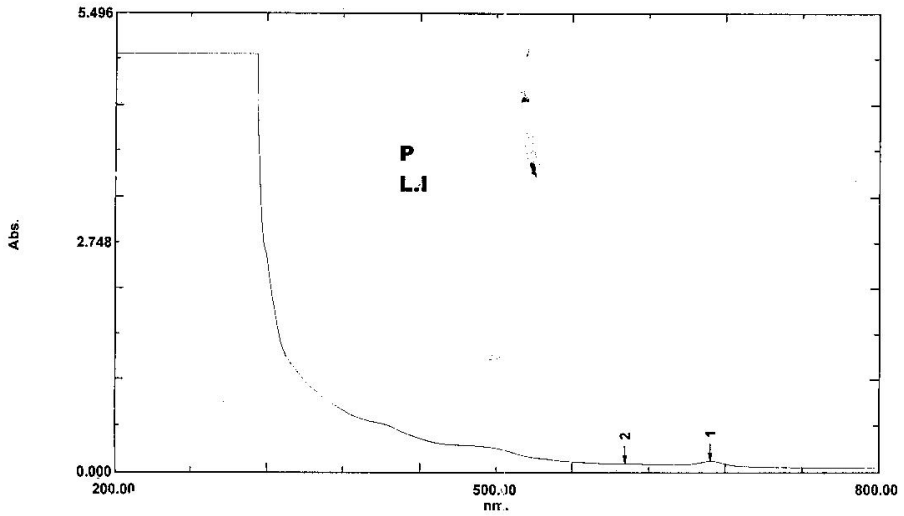
No.	P/V	Wavelength	Abs.	Description
1	P	668.50	0.124	
2	V	601.50	0.094	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

Footnote:

P L.I= Petrol mixed with *Lawsonia inermis* dye extract.

4b

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/09/2015

09:02:35 AM

RawData - C:\NARICT\MAXWELL PERTOL L.I.spc

No.	P/V	Wavelength	Abs.	Description
1	P	669.00	0.802	
2	V	609.00	0.383	
3	P	374.00	3.987	
4	V	369.00	3.996	
5	P	354.00	4.043	
6	V	347.50	4.021	
7	P	341.50	4.050	
8	V	326.50	4.006	
9	P	321.50	3.994	
10	V	305.00	4.427	
11	P	294.00	4.101	
12	V	286.00	4.095	
13	P	274.50	4.095	
14	V	269.00	4.113	
15	P	262.50	4.107	
16	V	247.50	4.191	
17	P	240.00	4.215	
18	V	229.00	4.201	
19	P	223.50	4.152	

Measurement Properties

Wavelength Range (nm.):	200.00 to 800.00
Scan Speed:	Fast
Sampling Interval:	0.5
Auto Sampling Interval:	Disabled
Scan Mode:	Single

Instrument Properties

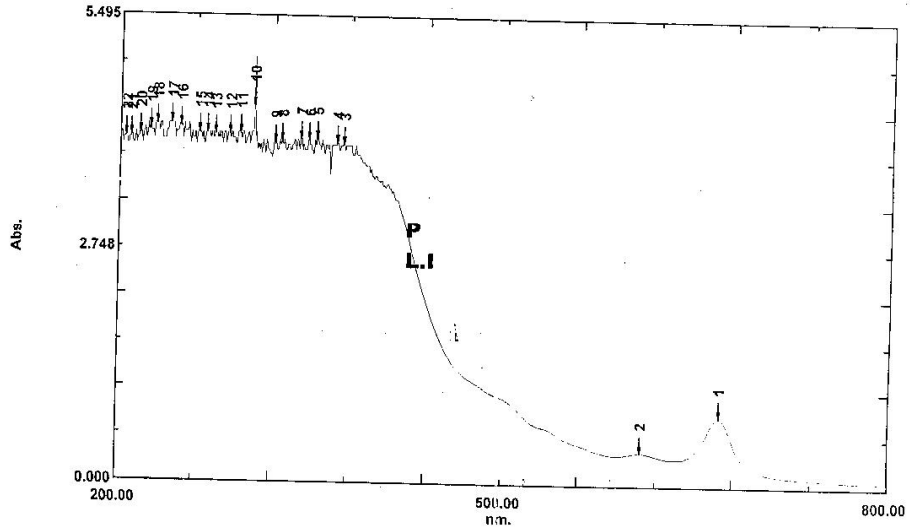
Instrument Type:	UV-2500PC Series
Measuring Mode:	Absorbance
Slit Width:	2.0 nm
Light Source Change Wavelength:	360.0 nm
S/R Exchange:	Normal

Attachment Properties

Attachment:	None

Sample Preparation Properties

Weight:	1g
Volume:	3ml
Dilution:	1
Path Length:	1
Additional Information:	



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

4c

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/11/2015

09:40:24 AM

RawData - C:\NARICT\MAXWELL PETROL DAY 8 L.I.spc

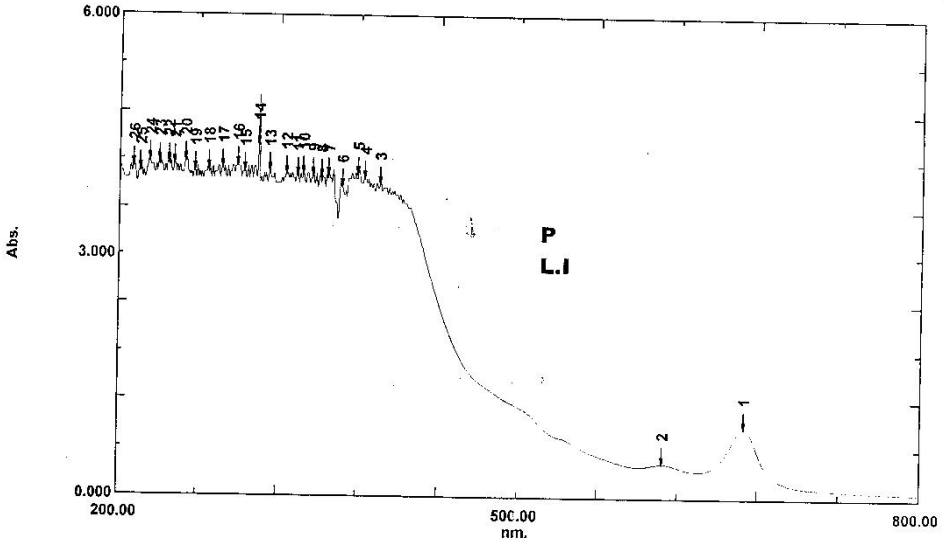
No.	P/V	Wavelength	Abs.	Description
1	⊕	669.00	0.867	
2	⊕	609.00	0.419	
3	⊕	395.00	3.895	
4	⊕	384.00	3.959	
5	⊕	379.00	4.019	
6	⊕	367.00	3.864	
7	⊕	357.00	4.002	
8	⊕	352.00	3.981	
9	⊕	345.50	4.006	
10	⊕	338.00	4.013	
11	⊕	333.50	3.999	
12	⊕	326.00	4.019	
13	⊕	313.50	4.060	
14	⊕	305.00	4.371	
15	⊕	294.00	4.060	
16	⊕	289.00	4.116	
17	⊕	278.00	4.083	
18	⊕	267.00	4.078	
19	⊕	257.00	4.057	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

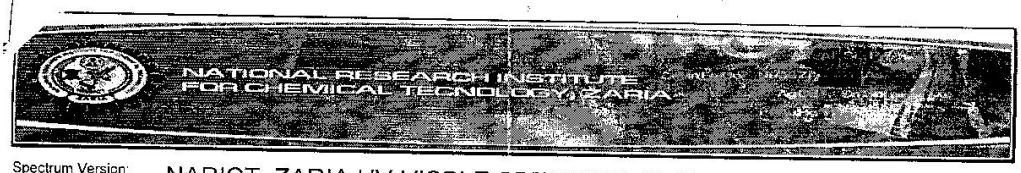
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

4d



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/16/2015
09:45:47 AM

RawData - C:\NARICT\MAXWELL PETROL L.I.spc

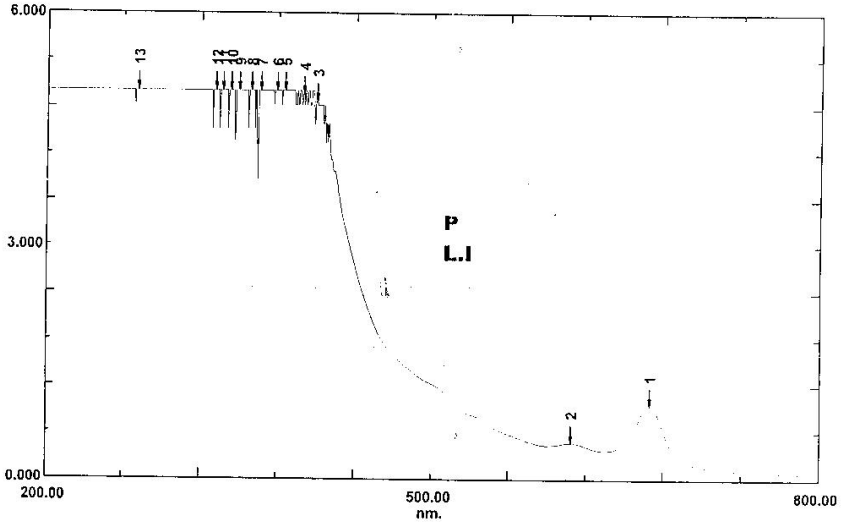
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	669.00	0.944	
2	ⓥ	608.50	0.464	
3	ⓟ	410.00	4.859	
4	ⓥ	399.00	4.942	
5	ⓟ	384.50	5.000	
6	ⓥ	378.50	5.000	
7	ⓟ	366.50	5.000	
8	ⓥ	359.00	5.000	
9	ⓟ	349.00	5.000	
10	ⓥ	343.50	5.000	
11	ⓟ	337.00	5.000	
12	ⓥ	331.50	5.000	
13	ⓟ	271.50	5.000	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

4e

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/18/2015
02:57:18 PM

RawData - C:\NARICT\MAXWELL PETROL DAY 16 L.I.spc

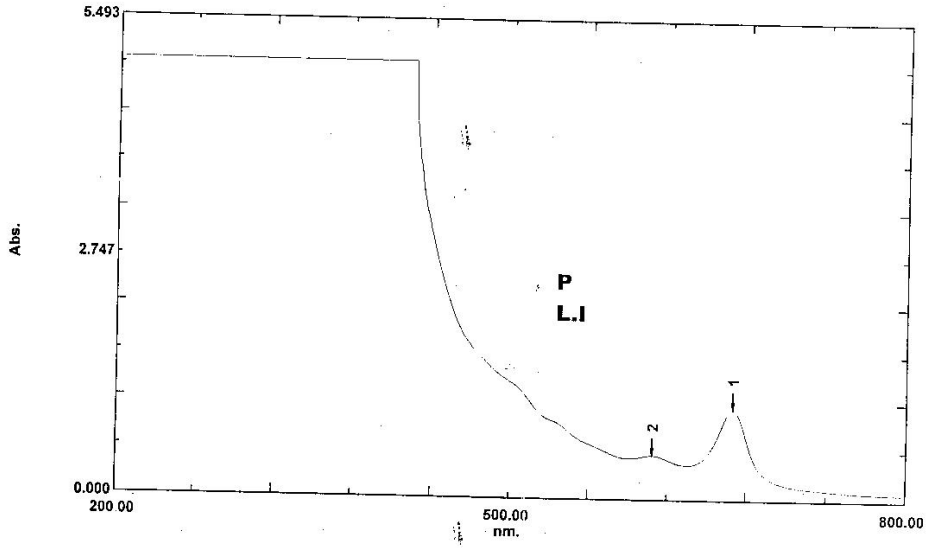
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	669.00	1.019	
2	ⓥ	608.50	0.496	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



NALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

4f

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/23/2015

11:15:22 AM

RawData - C:\NARICT\MAXWEL PETROL DAY 20 L.I.spc

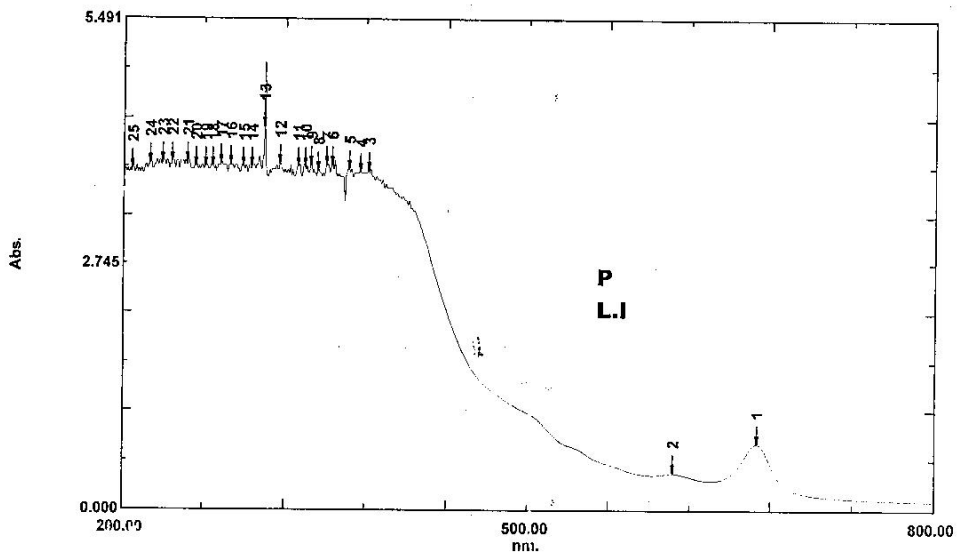
No.	P/V	Wavelength	Abs.	Description
1	P	669.00	0.736	
2	V	607.50	0.405	
3	P	381.50	3.800	
4	V	375.00	3.792	
5	P	367.00	3.825	
6	V	355.00	3.869	
7	P	351.00	3.864	
8	V	344.50	3.795	
9	P	339.00	3.871	
10	V	334.50	3.846	
11	P	329.50	3.854	
12	V	316.50	3.875	
13	P	305.00	4.294	
14	V	296.00	3.850	
15	P	289.50	3.850	
16	V	280.00	3.860	
17	P	273.00	3.883	
18	V	266.00	3.853	
19	P	261.00	3.853	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

4g

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/08/2015
11:33:26 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL PETROL L.I.spc

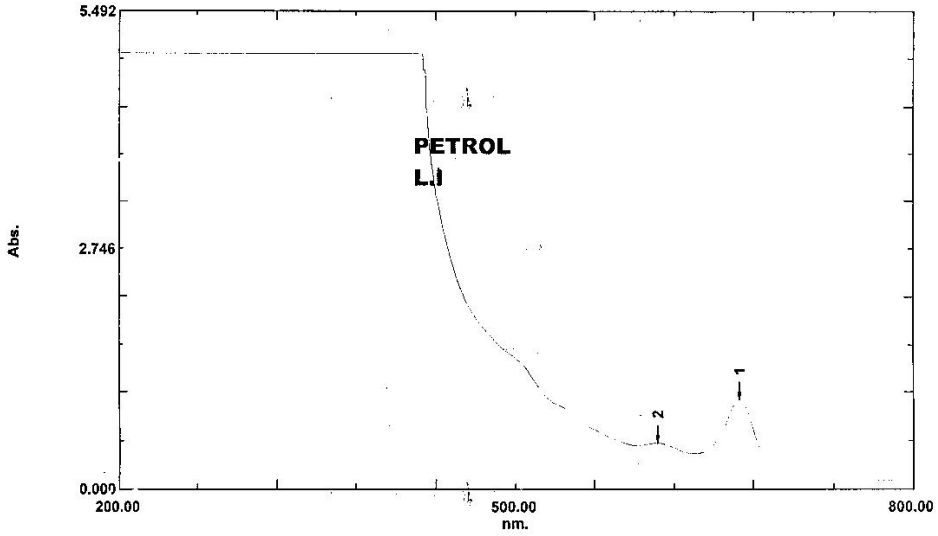
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	669.00	1.017	
2	ⓥ	607.50	0.524	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

4h

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/13/2015
11:49:21 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL PETROL L.I. 28.spc

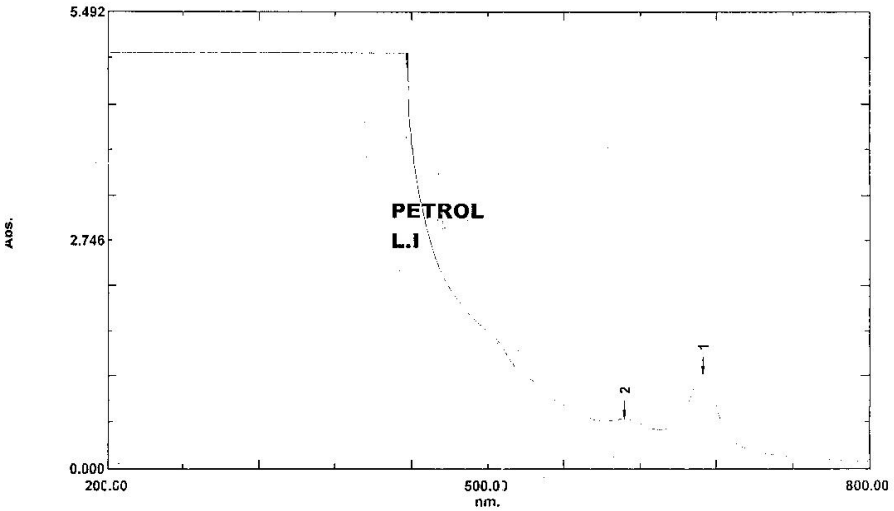
No.	P/V	Wavelength	Abs.	Description
1	⊕	669.00	1.121	
2	⊕	608.00	0.592	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



NALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

APPENDIX 5a

UV-VISIBLE SPECTRA RESULTS OF *L. inermis* EXTRACT IN KEROSENE FOR 28 DAYS

ctrum Version: .30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/05/2015
01:36:49 PM

RawData - C:\NARICT\MAXWELL KERO L.I.spc

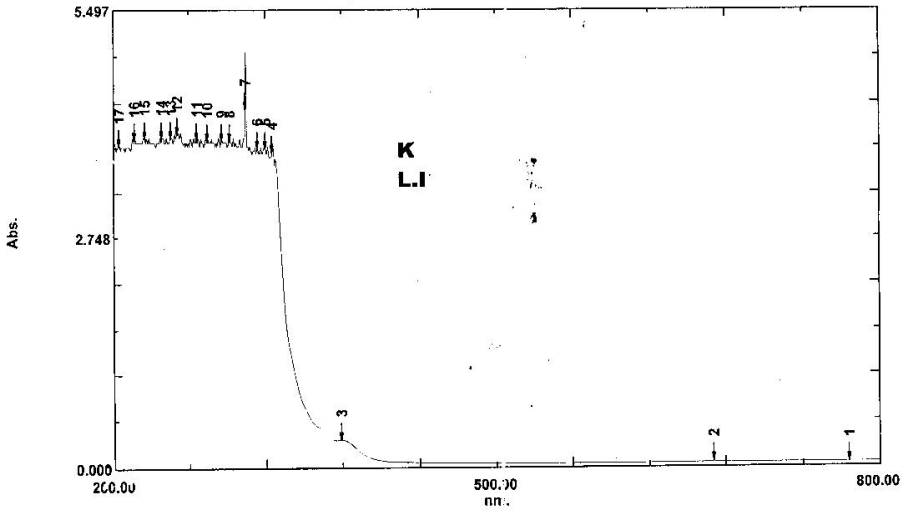
No.	P/V	Wavelength	Abs.	Description
1	ⓟ	776.50	0.039	
2	ⓥ	670.50	0.042	
3	ⓟ	378.50	0.318	
4	ⓥ	326.00	3.796	
5	ⓟ	320.50	3.838	
6	ⓥ	314.00	3.843	
7	ⓟ	305.00	4.314	
8	ⓥ	292.50	3.928	
9	ⓟ	286.00	3.933	
10	ⓥ	274.50	3.937	
11	ⓟ	266.00	3.941	
12	ⓥ	251.00	4.019	
13	ⓟ	245.50	3.971	
14	ⓥ	238.50	3.962	
15	ⓟ	224.50	3.962	
16	ⓥ	216.50	3.954	
17	ⓟ	204.50	3.869	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

Footnote:

K L.I= Kerosene mixed with *Lawsonia inermis* dye extract.

5b

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/09/2015

09:06:51 AM

RawData - C:\NARICT\MAXWELL KEROSINE L.I.spc

No.	P/V	Wavelength	Abs.	Description
1	P	747.50	0.042	
2	V	669.50	0.073	
3	P	320.00	3.838	
4	V	312.00	3.876	
5	P	305.00	4.305	
6	V	294.50	3.952	
7	P	286.50	3.948	
8	V	281.00	3.954	
9	P	273.50	3.954	
10	V	266.50	3.948	
11	P	252.50	4.015	
12	V	239.50	3.989	
13	P	228.50	3.959	
14	V	220.00	3.978	
15	P	209.00	3.915	

Measurement Properties

Wavelength Range (nm.):	200.00 to 800.00
Scan Speed:	Fast
Sampling Interval:	0.5
Auto Sampling Interval:	Disabled
Scan Mode:	Single

Instrument Properties

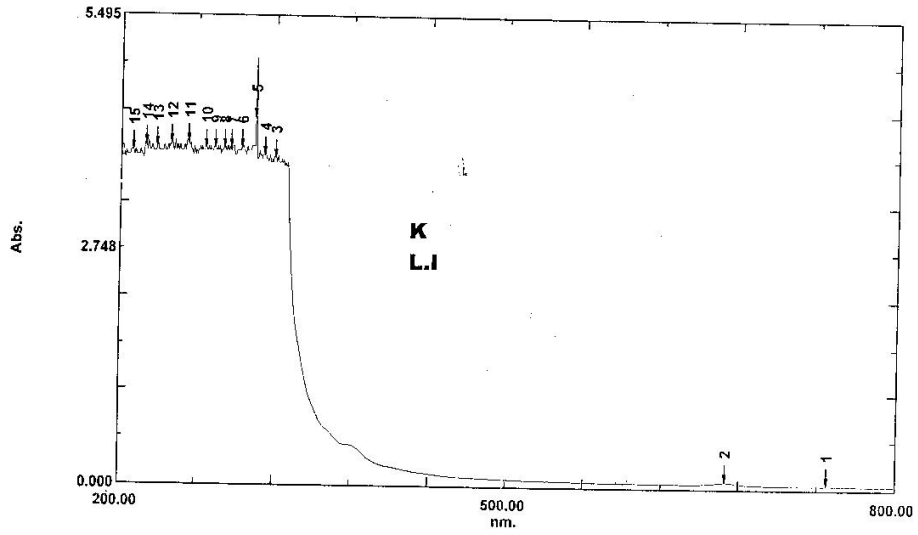
Instrument Type:	UV-2500PC Series
Measuring Mode:	Absorbance
Slit Width:	2.0 nm
Light Source Change Wavelength:	360.0 nm
S/R Exchange:	Normal

Attachment Properties

Attachment:	None

Sample Preparation Properties

Weight:	1g
Volume:	3ml
Dilution:	1
Path Length:	1
Additional Information:	



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

5c

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/11/2015

09:38:13 AM

RawData - C:\NARICT\MAXWEL KERO L.I DAY 8.spc

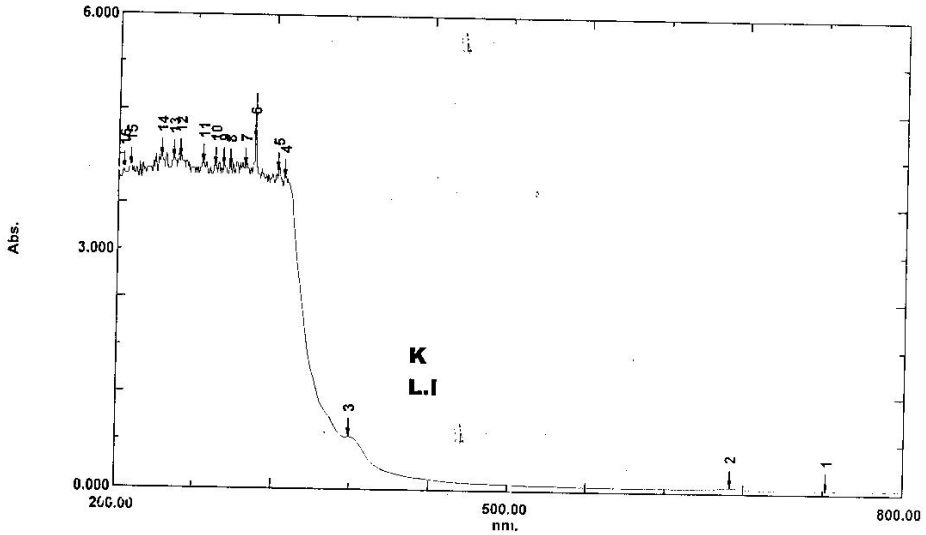
No.	P/V	Wavelength	Abs.	Description
1	P	742.00	0.040	
2	V	669.50	0.066	
3	P	378.50	0.648	
4	V	328.00	3.931	
5	P	323.00	4.016	
6	V	305.00	4.417	
7	P	298.00	4.072	
8	V	286.00	4.057	
9	P	281.00	4.063	
10	V	275.00	4.072	
11	P	265.50	4.095	
12	V	247.50	4.169	
13	P	242.50	4.155	
14	V	233.00	4.176	
15	P	209.50	4.058	
16	V	204.00	3.992	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

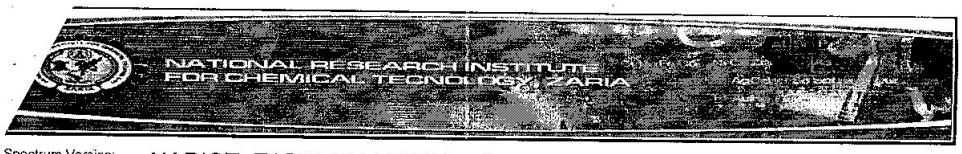
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

5d



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/16/2015

09:40:05 AM

RawData - C:\NARICT\MAXWELL PETROL L.I DAY 12.spc

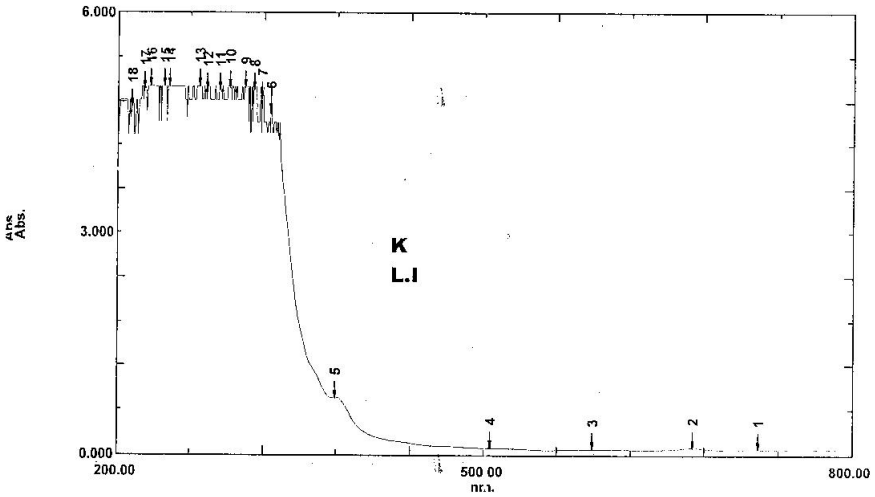
No.	P/V	Wavelength	Abs.	Description
1		723.00	0.070	
2		670.50	0.092	
3		589.00	0.065	
4		505.50	0.085	
5		379.00	0.767	
6		325.50	4.648	
7		318.50	4.821	
8		312.00	4.942	
9		305.00	4.978	
10		292.00	4.978	
11		284.50	4.956	
12		274.00	4.956	
13		267.00	5.000	
14		243.00	5.000	
15		238.00	5.000	
16		226.50	5.000	
17		221.50	4.957	
18		211.50	4.731	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

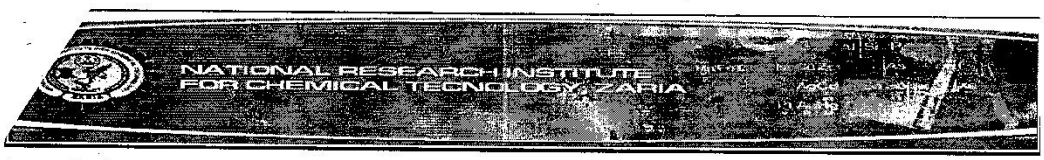
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

5e



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/18/2015
02:54:34 PM

RawData - C:\NARICT\MAXWELL KERO L.I DAY 16.spc

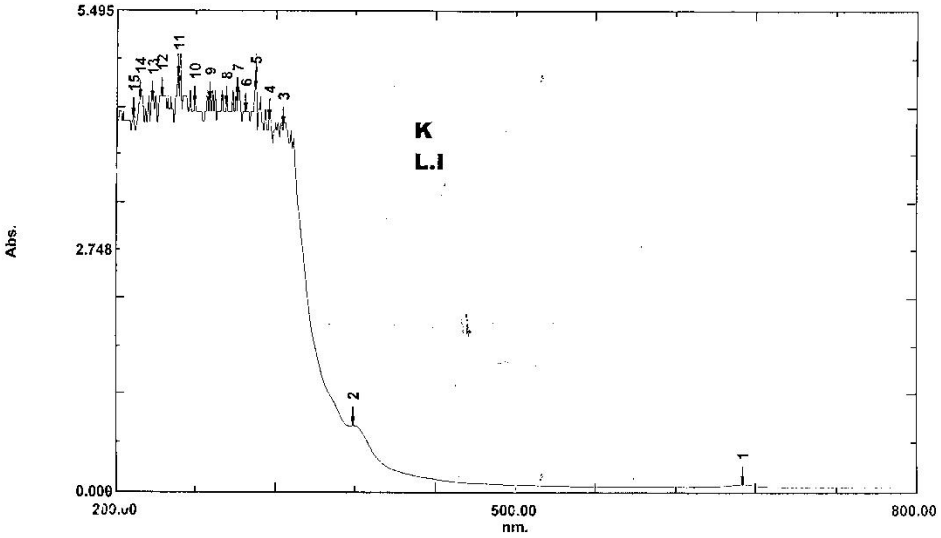
No.	P/V	Wavelength	Abs.	Description
1	⊕	670.00	0.081	
2	⊕	378.50	0.737	
3	⊕	326.00	4.187	
4	⊕	315.50	4.265	
5	⊕	305.00	4.603	
6	⊕	297.50	4.340	
7	⊕	291.50	4.521	
8	⊕	283.00	4.426	
9	⊕	270.50	4.464	
10	⊕	259.00	4.426	
11	⊕	246.50	4.748	
12	⊕	234.00	4.516	
13	⊕	227.50	4.468	
14	⊕	218.00	4.485	
15	⊕	212.50	4.275	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

5f

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/23/2015

11:31:45 AM

RawData - C:\NARICT\MAXWELL KERO L.I. DAY 20.spc

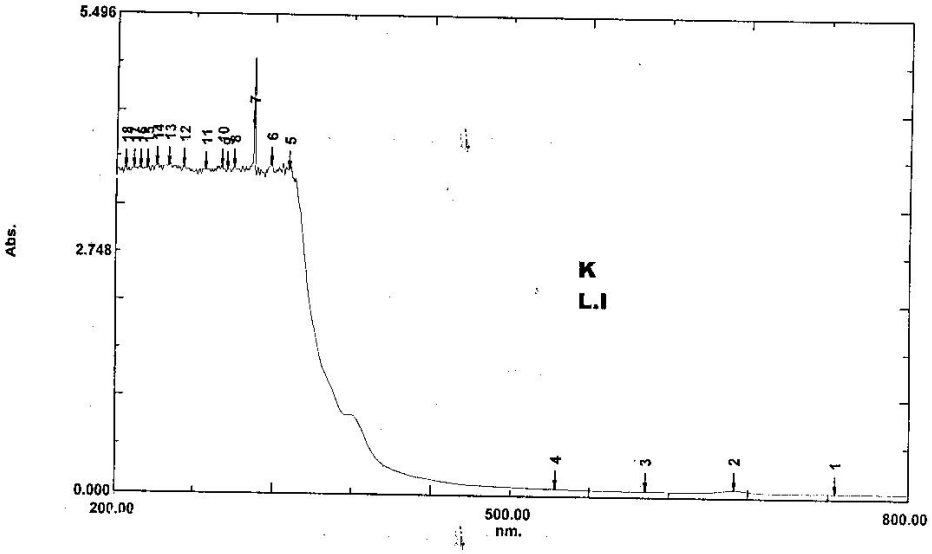
No.	P/V	Wavelength	Abs.	Description
1		745.50	0.049	
2		669.50	0.083	
3		603.00	0.057	
4		534.00	0.076	
5		331.50	3.716	
6		318.00	3.761	
7		305.00	4.190	
8		290.00	3.728	
9		285.50	3.698	
10		280.50	3.731	
11		268.00	3.703	
12		252.00	3.723	
13		241.00	3.748	
14		231.50	3.745	
15		223.50	3.719	
16		218.50	3.718	
17		213.00	3.716	
18		207.00	3.704	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

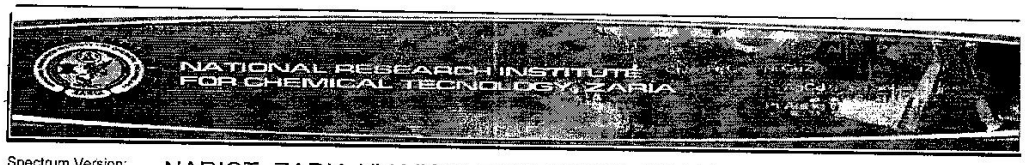
Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



NALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

5g



Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/08/2015
11:09:17 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\File_090721_102144_105802.sp
RawData - C:\MAXWEL KERO LI.spc

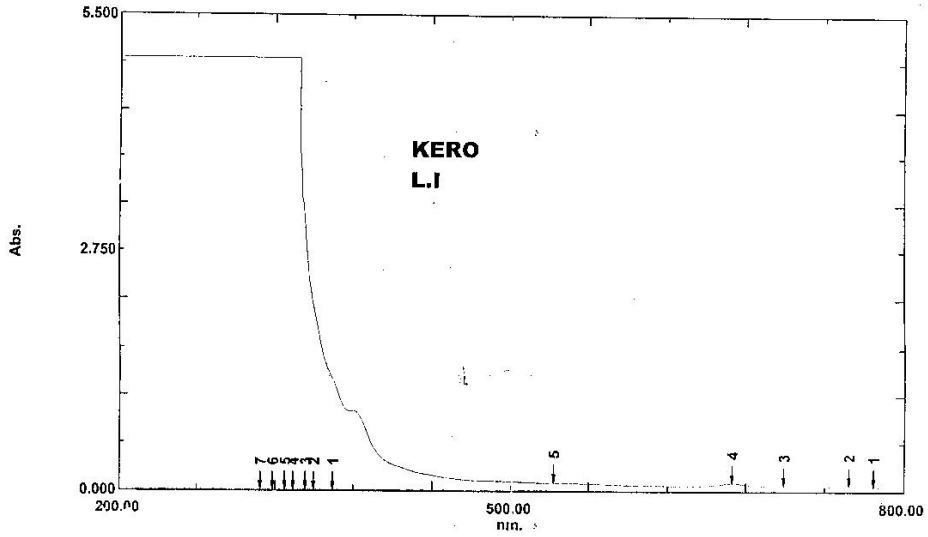
No.	P/V	Wavelength	Abs.	Description
1		777.00	0.048	
2		758.00	0.056	
3		708.50	0.056	
4		669.50	0.091	
5		533.50	0.089	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

5h

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/13/2015
11:44:40 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL KERO L.I 28.spc

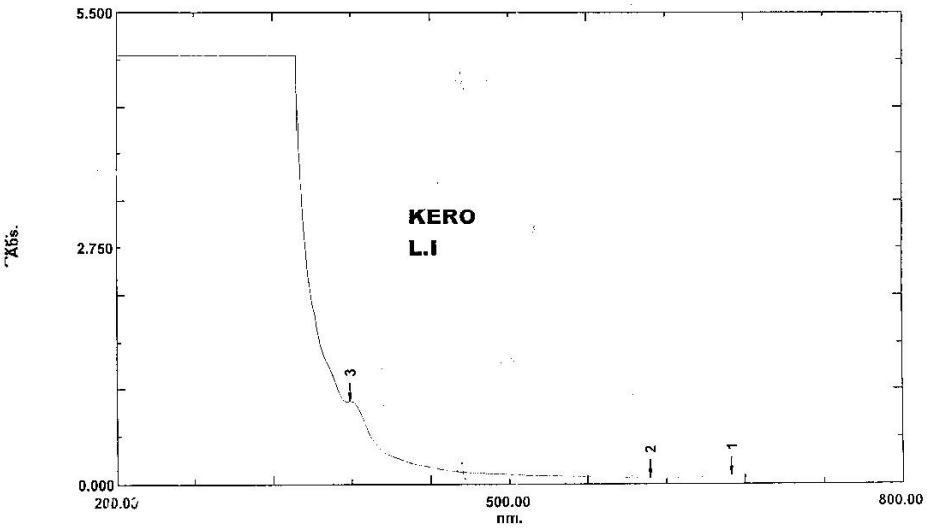
No.	P/V	Wavelength	Abs.	Description
1	⊕	669.50	0.096	
2	⊕	608.00	0.068	
3	⊕	378.50	0.960	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

APPENDIX 6a

UV-VISIBLE SPECTRA RESULTS OF *L. inermis* EXTRACTS IN DIESEL FOR 28 DAYS

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/05/2015

RawData - C:\NARICT\MAXWELL DIESEL L.I.spc

01:32:53 PM

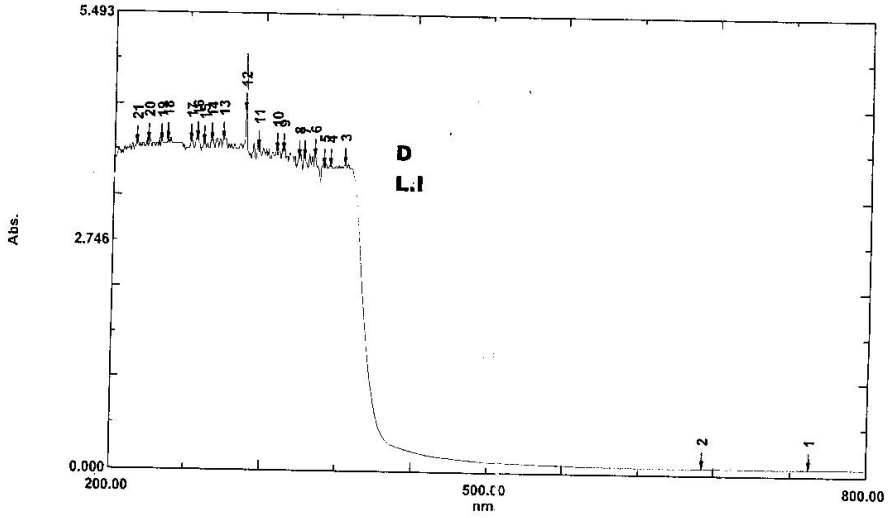
No.	P/V	Wavelength	Abs.	Description
1		754.50	0.079	
2		670.00	0.076	
3		384.00	3.683	
4		372.50	3.669	
5		367.00	3.667	
6		359.50	3.768	
7		351.50	3.762	
8		347.50	3.758	
9		336.00	3.642	
10		330.00	3.835	
11		315.00	3.864	
12		305.00	4.309	
13		287.50	3.961	
14		277.50	3.943	
15		272.00	3.920	
16		266.00	3.956	
17		261.50	3.933	
18		243.00	3.944	
19		237.50	3.929	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

Footnote:

D L.I= Diesel mixed with *Lawsonia inermis* dye extract.

6b

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/09/2015

08:58:50 AM

RawData - C:\NARICT\MAXWEL DIESEL L.I.spc

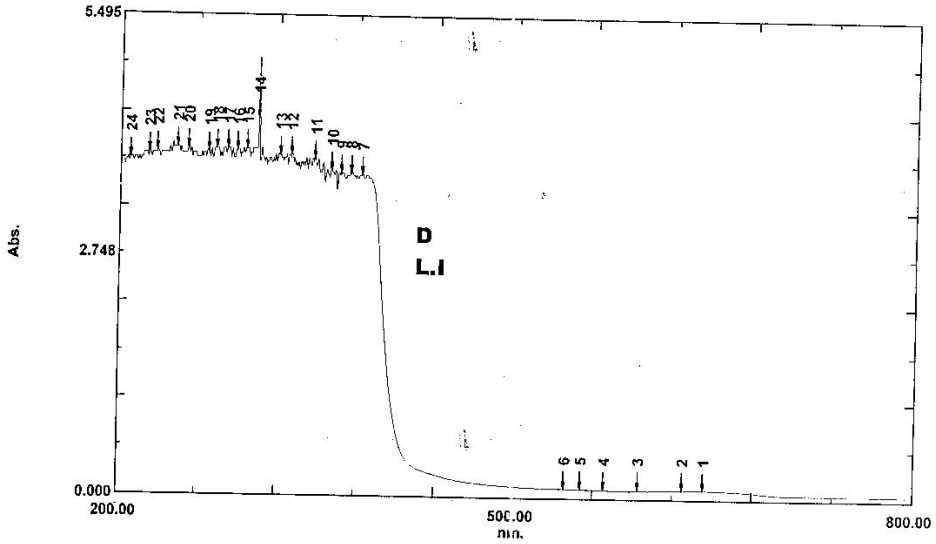
No.	P/V	Wavelength	Abs.	Description
1	⊕	643.00	0.102	
2	⊕	628.00	0.102	
3	⊕	594.00	0.091	
4	⊕	568.50	0.091	
5	⊕	550.50	0.097	
6	⊕	538.00	0.101	
7	⊕	382.50	3.673	
8	⊕	374.50	3.692	
9	⊕	367.50	3.693	
10	⊕	360.00	3.723	
11	⊕	347.00	3.847	
12	⊕	330.00	3.904	
13	⊕	321.00	3.901	
14	⊕	305.00	4.312	
15	⊕	296.50	3.963	
16	⊕	289.50	3.954	
17	⊕	282.00	3.959	
18	⊕	273.50	3.967	
19	⊕	267.00	3.937	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

6c

NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/11/2015
09:34:09 AM

RawData - C:\NARICT\MAXWELL DIESEL L.I DAY 8.spc

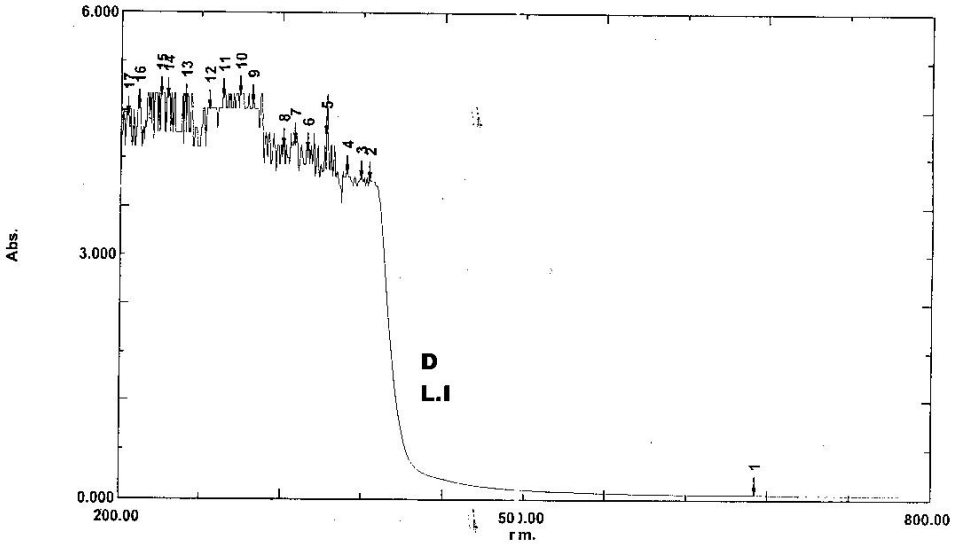
No.	P/V	Wavelength	Abs.	Description
1	P	670.50	0.051	
2	P	384.50	3.933	
3	P	378.50	3.954	
4	P	368.00	4.025	
5	P	353.00	4.494	
6	P	339.50	4.296	
7	P	329.50	4.420	
8	P	321.50	4.342	
9	P	298.50	4.883	
10	P	289.50	4.985	
11	P	277.00	4.956	
12	P	266.50	4.817	
13	P	248.50	4.872	
14	P	235.50	4.942	
15	P	230.50	4.961	
16	P	213.00	4.807	
17	P	205.00	4.731	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



ANALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

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Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/16/2015

09:55:43 AM

RawData - C:\NARICT\MAXWELL DIESEL L..I DAY 12.spc

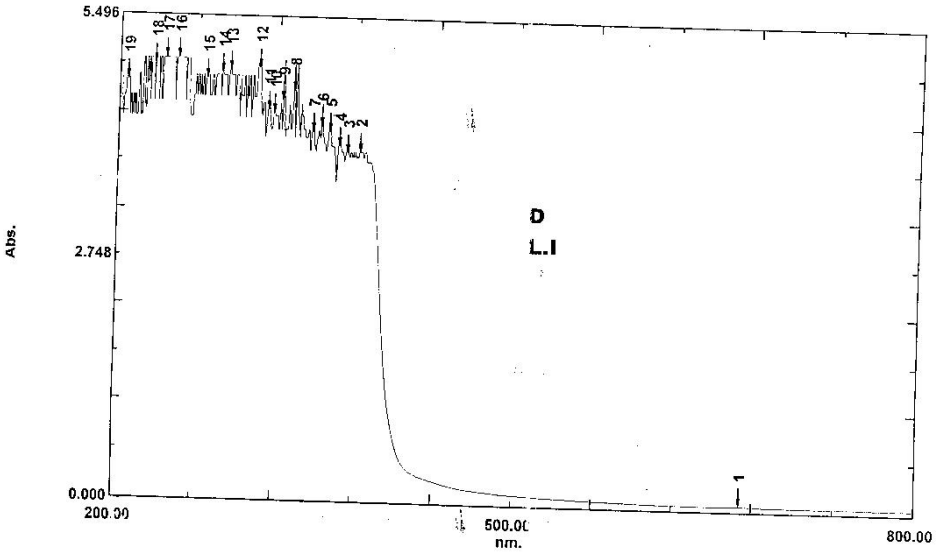
No.	P/V	Wavelength	Abs.	Description
1		670.50	0.056	
2		382.00	3.977	
3		372.50	3.959	
4		366.50	4.043	
5		358.50	4.202	
6		352.50	4.263	
7		346.50	4.194	
8		332.00	4.654	
9		324.00	4.546	
10		317.50	4.402	
11		313.50	4.438	
12		305.50	4.910	
13		284.00	4.874	
14		278.00	4.854	
15		266.50	4.779	
16		245.00	5.000	
17		235.00	5.000	
18		227.50	4.942	
19		206.00	4.751	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



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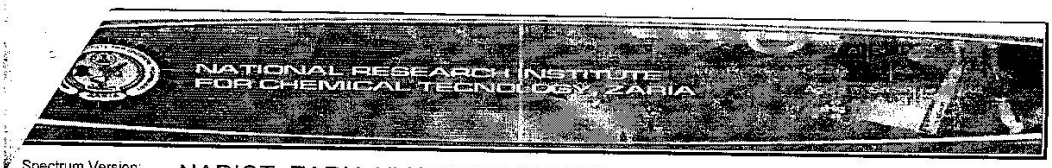


Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/18/2015

02:47:00 PM

RawData - C:\NARICT\MAXWELL 16 DAY DIESEL L.I.spc

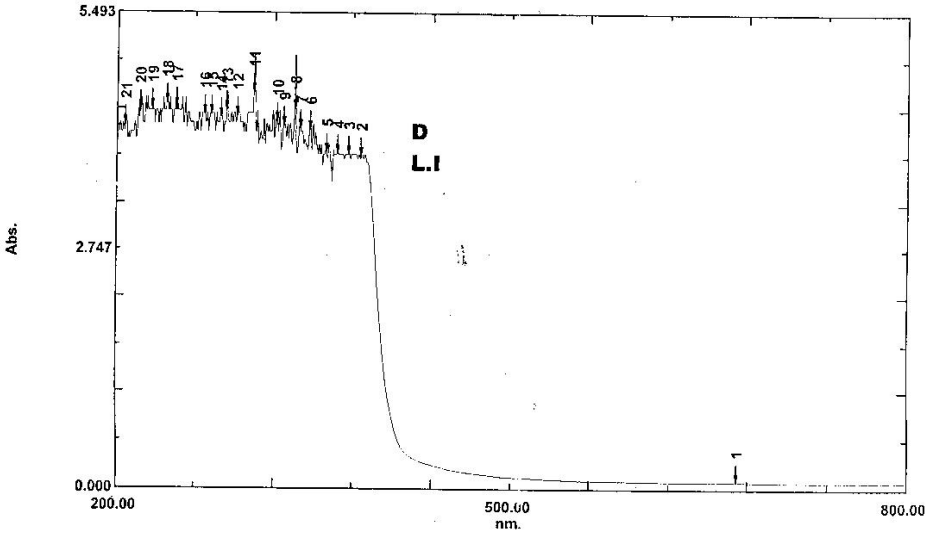
No.	P/V	Wavelength	Abs.	Description
1	⊕	671.00	0.076	
2	⊕	386.00	3.846	
3	⊕	376.50	3.874	
4	⊕	368.00	3.883	
5	⊕	359.50	3.904	
6	⊕	347.50	4.155	
7	⊕	340.00	4.166	
8	⊕	335.50	4.395	
9	⊕	328.00	4.193	
10	⊕	322.50	4.244	
11	⊕	305.00	4.575	
12	⊕	292.50	4.313	
13	⊕	284.50	4.373	
14	⊕	279.50	4.296	
15	⊕	273.00	4.331	
16	⊕	267.00	4.322	
17	⊕	245.50	4.408	
18	⊕	238.50	4.450	
19	⊕	226.50	4.390	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



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NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

03/23/2015

11:27:45 AM

RawData - C:\NARICT\MAXWEL DIESEL DAY 20 L.I.spc
RawData - C:\NARICT\MAXWEL DIESEL DAY 20 P.O.spc

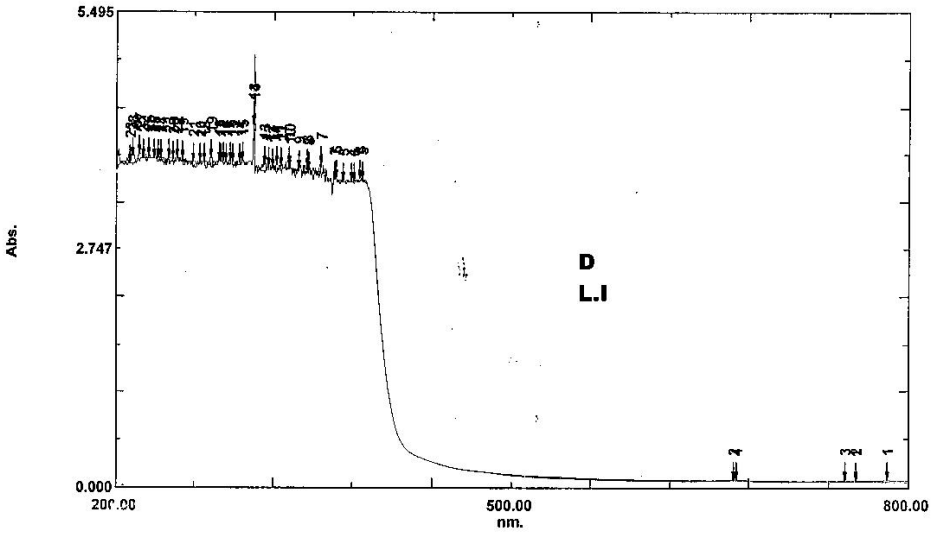
No.	P/V	Wavelength	Abs.	Description
1	⊕	784.00	0.075	
2	⊕	760.50	0.074	
3	⊕	752.50	0.074	
4	⊕	670.00	0.082	
5	⊕	387.00	3.562	
6	⊕	380.50	3.557	
7	⊕	366.50	3.576	
8	⊕	346.00	3.678	
9	⊕	339.50	3.692	
10	⊕	331.50	3.702	
11	⊕	322.50	3.744	
12	⊕	316.50	3.723	
13	⊕	305.00	4.196	
14	⊕	294.00	3.767	
15	⊕	289.00	3.773	
16	⊕	284.50	3.773	
17	⊕	279.50	3.774	
18	⊕	267.50	3.774	
19	⊕	251.00	3.802	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Disabled
Scan Mode: Single

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



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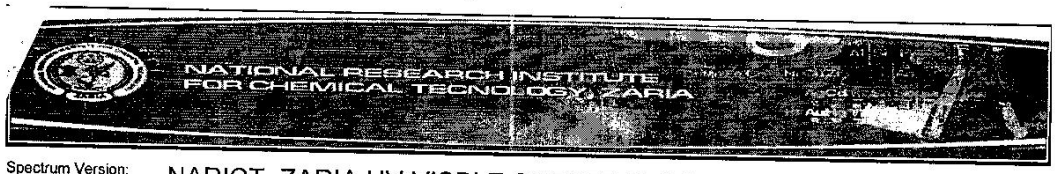


Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/08/2015

11:45:41 AM

RawData - C:\Documents and Settings\NARICT\My Documents\idowu\Mr. Ladan\MAXWEL DIESEL L.I.spc

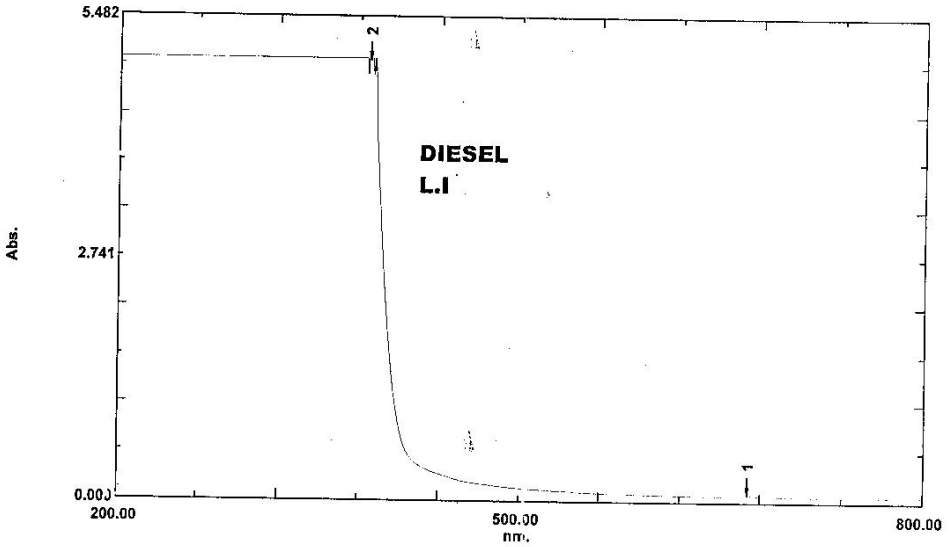
No.	P/V	Wavelength	Abs.	Description
1	⊕	670.50	0.073	
2	⊖	386.50	4.985	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



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NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, ZARIA

Spectrum Version: v2.30

NARICT, ZARIA UV-VISBLE SPECTRAL SCAN RESULT

04/13/2015

11:39:36 AM

RawData - C:\Documents and Settings\NARICT\My Documents\Idow J\Mr. Ladan\MAXWEL DIESEL L.I. 28.spc

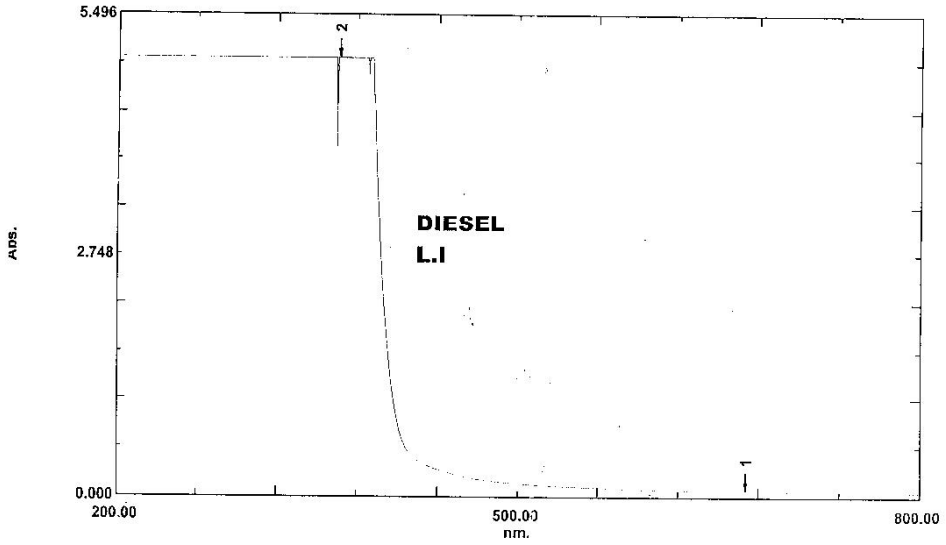
No.	P/V	Wavelength	Abs.	Description
1	⊕	670.00	0.068	
2	⊕	366.50	5.000	

Measurement Properties
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Fast
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2500PC Series
Measuring Mode: Absorbance
Slit Width: 2.0 nm
Light Source Change Wavelength: 360.0 nm
S/R Exchange: Normal

Attachment Properties
Attachment: None

Sample Preparation Properties
Weight: 1g
Volume: 3ml
Dilution: 1
Path Length: 1
Additional Information:



NALYSIS VERIFIED BY NAME_____ SIGN_____ DATE_____

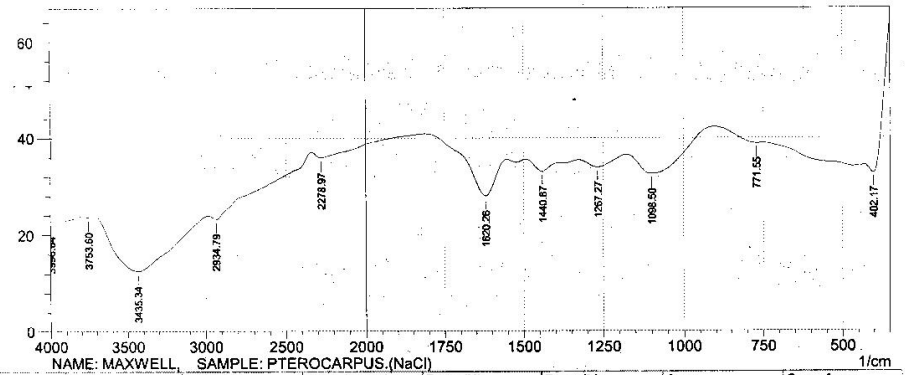
APPENDIX 7

FTIR SPECTRA RESULT OF *P. osun* EXTRACT

SHIMADZU

FTIR ANALYSIS RESULT NARICT,ZARIA

FTIR- 8400S FOURIER TRANSFORM INFRARED SPECTROPHOTOMETER



NAME: MAXWELL, SAMPLE: PTEROCARPUS.(NaCl)

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	402.17	32.6829	12.2892	431.1	349.13	32.7161	5.8736
2	771.55	38.841	0.5395	900.79	749.37	59.8402	0.4611
3	1098.5	32.5148	5.6609	1176.62	900.79	121.5936	9.7353
4	1267.27	33.9655	1.9312	1328.03	1176.62	69.0857	1.9066
5	1440.67	33.0889	2.0734	1493.92	1390.72	48.0898	1.2583
6	1620.26	28.0574	8.8777	1815.08	1552.75	119.484	9.6332
7	2278.97	35.9289	1.5582	2336.84	1815.08	218.8784	5.0464
8	2934.79	23.2555	1.7452	2985.91	2336.84	346.508	5.3012
9	3435.34	12.601	11.3883	3711.17	2985.91	553.5665	103.8677
10	3753.6	23.5946	0.0741	3759.39	3717.92	25.8634	0.0173
11	3996.64	22.7938	0.0145	4000.5	3966.74	21.6491	0.0057

Comment;
NAME: MAXWELL, SAMPLE: PTEROCARPUS.(NaCl)

User; Administrator

No. of Scans;
Date/Time; 11/5/2014 12:45:55 PM
Resolution;

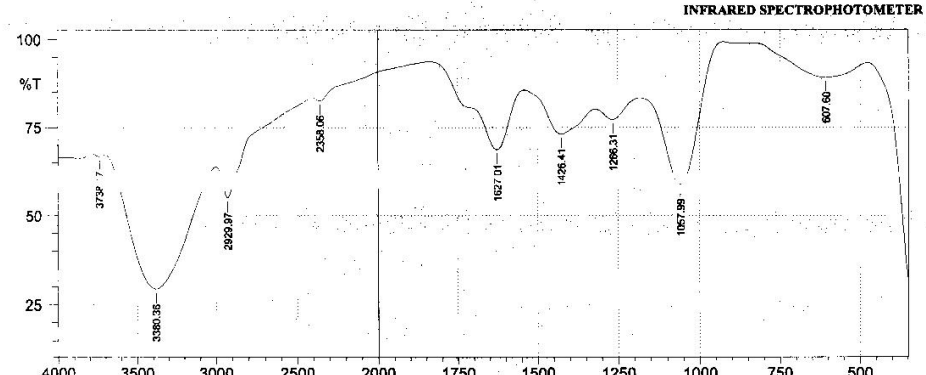
APPENDIX 8

FTIR SPECTRA RESULT OF *L. inermis* EXTRACT

SHIMADZU

FTIR ANALYSIS RESULT NARICT,ZARIA

FTIR-8400S FOURIER TRANSFORM INFRARED SPECTROPHOTOMETER



NAME: MAXWELL, SAMPLE: PLANT EXTARCT OF MTH.(KBr) L-1

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	607.6	89.22	6.188	828.45	477.4	11.722	5.633
2	1057.99	58.843	32.238	1180.47	925.86	30.092	19.707
3	1266.31	77.228	4.218	1319.35	1180.47	13.53	1.419
4	1426.41	73.116	9.697	1541.18	1319.35	23.578	5.469
5	1627.01	68.671	19.262	1839.19	1541.18	27.038	12.773
6	2358.06	82.598	1.748	2405.31	1839.19	30.225	0.072
7	2929.97	55.084	11.209	3002.3	2405.31	84.681	3.28
8	3380.36	29.469	36.229	3701.52	3003.27	247.468	119.359
9	3738.17	66.756	3.62	3787.36	3701.52	14.879	0.174

Comment;
NAME: MAXWELL, SAMPLE: PLANT EXTARCT OF MTH.(KBr)

User; Administrator

No. of Scans;
Date/Time; 11/5/2014 12:19:00 PM
Resolution;